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Magnesium Diboride Superconductors

for Magnet Applications

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# Magnesium Diboride Superconductors

## for Magnet Applications

Magnesium Diboride Superconductors  
for Magnet Applications

H. van Weeren



MAGNESIUM DIBORIDE SUPERCONDUCTORS  
FOR MAGNET APPLICATIONS



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The work described in this thesis was performed at the Low Temperature Division, the Special Chair for High Current Superconductivity at the Faculty of Science and Technology, University of Twente, The Netherlands.

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# MAGNESIUM DIBORIDE SUPERCONDUCTORS FOR MAGNET APPLICATIONS

## PROEFSCHRIFT

ter verkrijging van  
de graad van doctor aan de Universiteit Twente,  
op gezag van de rector magnificus,  
prof. dr. W.H.M. Zijm,  
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door

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te Hoorn



Dit proefschrift is goedgekeurd door de promotor en assistent promotor

prof. dr. ir. H.H.J. ten Kate  
dr. M. Dhallé



# Nomenclature

Symbol	Meaning	Unit
$A$	Area	[m <sup>2</sup> ]
$A_m$	Cross-sectional area of the matrix	[m <sup>2</sup> ]
$A_{sc}$	Cross-sectional area of the superconductor	[m <sup>2</sup> ]
$B$	Magnetic field	[T]
$B_0$	Applied magnetic field	[T]
$B^*$	Irreversibility field	[T]
$B_0^*$	Irreversibility field @ $T = 0$ K	[T]
$B_{100}$	Magnetic field at which $J_c = 100\text{A}/\text{mm}^2$	[T]
$B_c$	Thermodynamic critical field	[T]
$B_{c2}$	Second critical field	[T]
$B_{c2,0}$	Second critical field @ $T = 0$ K	[T]
$B_n$	Exponential decay field of $n$ -value	[T]
$B_{n,0}$	Exponential decay field of $n$ -value @ $T = 0$ K	[T]
$B_{\text{offset}}$	Offset field in resistive transition	[T]
$B_{\text{offset},0}$	Offset field @ $T = 0$ K	[T]
$B_{\text{onset}}$	Onset field in resistive transition	[T]
$B_{\text{onset},0}$	Onset field @ $T = 0$ K	[T]
$B_p$	Exponential current decay field	[T]
$B_{p,0}$	Exponential current decay field @ $T = 0$ K	[T]
$c$	Volumetric heat capacity	[Jm <sup>-3</sup> K <sup>-1</sup> ]
$d$	Diameter	[m]
$D$	Diffusivity	[m <sup>2</sup> s]
$D_m$	Magnetic diffusivity	[m <sup>2</sup> s]
$D_T$	Thermal diffusivity	[m <sup>2</sup> s]
$E_Y$	Youngs' modulus	[GPa]
$E$	Electric field	[V/m]
$E_c$	Electric field criterion for $I_c$ determination	[V/m]
$f$	Superconductor filling factor	[%]
$f_i$	Fraction of the $i^{\text{th}}$ component in the composite	[%]

*Continued on next page*

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Symbol	Meaning	Unit
$h$	Height	[m]
$h_t$	Heat transfer coefficient	[Wm <sup>-2</sup> K <sup>-1</sup> ]
$H_v$	Vickers hardness	[-]
$I$	Transport current	[A]
$I_c$	Critical current	[A]
$I_{c,0}$	Critical current @ $T = 0$ K and $B = 0$ T	[A]
$I_0$	Total transport current	[A]
$I_m$	Current in the matrix	[A]
$I_{sc}$	Current in the superconductor	[A]
$J$	Transport current density	[Am <sup>-2</sup> ] <sup>1</sup>
$J_c$	Critical current density	[Am <sup>-2</sup> ] <sup>1</sup>
$J_{c,0}$	Critical current density @ $T = 0$ K; $B = 0$ T	[Am <sup>-2</sup> ] <sup>1</sup>
$J_{c,0}^T$	Critical current density @ $B = 0$ T	[Am <sup>-2</sup> ] <sup>1</sup>
$J_0$	Total transport current density	[Am <sup>-2</sup> ] <sup>1</sup>
$J_m$	Current density in the matrix	[Am <sup>-2</sup> ] <sup>1</sup>
$J_{sc}$	Current density in the superconductor	[Am <sup>-2</sup> ] <sup>1</sup>
$K_{I_c}$	Strain dependence of the critical current	[-]
$K_{I_{c,0}}$	Strain dependence of the critical current @ $T = 0$ K; $B = 0$ T	[-]
$K_{T_c}$	Strain dependence of the critical temperature	[-]
$K_{B_0}$	Strain dependence of the irreversibility field	[-]
$l$	Length	[m]
$l_{MPZ}$	Minimum Propagation Zone length	[m]
MQE	Minimum Quench Energy	[J]
$n$	$n$ -value in the relation $E \propto I^n$	[-]
$n_0$	$n$ -value at $B = 0$ T	[-]
$P$	Power / Heat flux	[W]
$P_{\text{initial}}$	Injected power	[W]
$p_{\text{diss}}$	Dissipating power density	[Wm <sup>-3</sup> ]
$p_{\text{initial}}$	Injected power density	[Wm <sup>-3</sup> ]
$Q$	Heat	[J]
$Q_{\text{initial}}$	Thermal disturbance	[J]
$R$	Electrical resistance	[ $\Omega$ ]
$R_t$	Thermal resistance	[KW <sup>-1</sup> ]
$R_o$	Initial outer radius of conductor	[m]
$R_i$	Initial interface radius	[m]
$R_{\text{fil}}$	Initial filament radius	[m]
$R_{fo}$	Final outer radius	[m]

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*Continued on next page*

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<sup>1</sup>The units for current density listed in this table are the official SI units. However, throughout the thesis the more practical unit [Amm<sup>-2</sup>] is used.

Symbol	Meaning	Unit
$R_{fi}$	Final interface radius	[m]
$t$	Time	[s]
$T$	Temperature	[K]
$T_0$	Operating temperature	[K]
$T_c$	Critical temperature	[K]
$T_{c,0}$	Critical temperature @ $B = 0$ T	[K]
$T_{cs}$	Current sharing temperature	[K]
$T_{ns}$	Normal state recovery temperature	[K]
$T_{offset}$	Offset temperature in resistive transition	[K]
$T_{onset}$	Onset temperature in resistive transition	[K]
$T_s$	Temperature at which heat generation starts	[K]
$V$	Voltage	[V]
$v_{nz}$	Normal zone propagation velocity	[ms <sup>-1</sup> ]
$w$	Width	[m]
$z$	Longitudinal coordinate along a conductor	[m]
<b>Greek Symbols</b>		
$\alpha$	Thermal expansion coefficient	[-]
$\Delta$	Superconducting gap energy	[J]
$\epsilon$	Emissivity	[-]
$\epsilon$	True strain	[-]
$\epsilon_f$	True strain at final diameter	[-]
$\epsilon$	Mechanical axial strain	[%]
$\epsilon_{applied}$	Applied axial strain	[%]
$\epsilon_{rev}$	Reversible strain limit	[%]
$\epsilon_{pre}$	Strain induced by thermal pre-compression	[%]
$\gamma_{k,l}$	$l^{\text{th}}$ root of the $k^{\text{th}}$ order Bessel function $\Upsilon_k$	[-]
$\kappa$	Thermal conductivity	[Wm <sup>-1</sup> K <sup>-1</sup> ]
$\kappa_m$	Thermal conductivity of the matrix	[Wm <sup>-1</sup> K <sup>-1</sup> ]
$\kappa_{sc}$	Thermal conductivity of the superconductor	[Wm <sup>-1</sup> K <sup>-1</sup> ]
$\nu$	View factor	[-]
$\mu_0$	Magnetic permeability of vacuum ( $4\pi \cdot 10^{-7}$ )	[Hm <sup>-1</sup> ]
$\Lambda$	Current transfer length	[m]
$\rho$	Electrical resistivity	[ $\Omega$ m]
$\rho_m$	Electrical resistivity of the matrix	[ $\Omega$ m]
$\rho_{sc}$	Normal state electrical resistivity of the filaments	[ $\Omega$ m]
$\rho_{fil}$	Filament density	[kgm <sup>-3</sup> ]
$\rho_{fil,0}$	Initial filament density	[kgm <sup>-3</sup> ]
$\rho_{fil,th}$	Theoretical filament density	[kgm <sup>-3</sup> ]

*Continued on next page*

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<b>Symbol</b>	<b>Meaning</b>	<b>Unit</b>
$\sigma$	Mechanical stress	$[\text{Nm}^{-2}]$
$\sigma_{\text{SB}}$	Stefan-Boltzmann constant ( $5.669 \cdot 10^{-8}$ )	$[\text{Wm}^{-2}\text{K}^{-4}]$
$\sigma_y$	Yield strength	$[\text{Nm}^{-2}]$
$\tau_T$	Thermal diffusion time	$[\text{s}]$
$\tau_m$	Magnetic diffusion time	$[\text{s}]$

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# Chapter 1

## Introduction

---

*Magnesium diboride has several attractive features justifying its development into a new technical superconductor for use in magnet systems. It is relatively simple to synthesize, its raw-material costs are low, conductors can be made with existing technologies and operated at higher temperatures. But just like any other technical superconductor its applicability in magnets depends on several performance aspects on a fundamental- as well as a technological level. These issues are introduced in this chapter.*

## 1.1 Magnesium Diboride

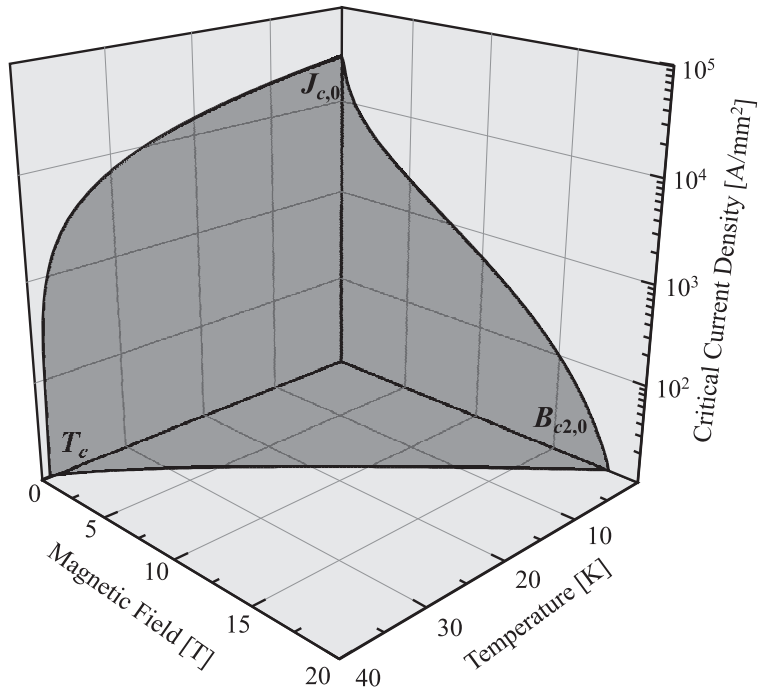
Magnesium diboride was first synthesised in the 1950's but it took until 2001 before its superconducting properties were discovered [1].  $\text{MgB}_2$  is sometimes considered as a medium- $T_c$  superconductor. With low- $T_c$  materials it has its metallic character in common, its relatively simple crystal structure and superconducting pairing that occurs through a BCS-like phonon mediated electron-electron interaction [2]. However, its  $T_c$  of 40 K is much higher than all previously known metallic superconductors. Moreover, like the high- $T_c$  cuprates, it has a layered structure leading to significantly anisotropic properties [3]. Finally,  $\text{MgB}_2$  shows two-gap superconductivity, a phenomenon that was considered theoretically possible in the past [4], but is experimentally observed for the first time in this material [5].

In terms of prospects for applications, its  $T_c$  makes  $\text{MgB}_2$  attractive for operation at  $\sim 20$  K, a temperature that is reached relatively easily with cooling machines, cutting out the need for a liquid cryogen and simplifying the cryostat. Compared to high- $T_c$  superconductors  $\text{MgB}_2$  is attractive mostly because of its relative ease of synthesis. Together with the abundance of magnesium and boron, simpler production causes its presently estimated cost-to-performance ratio to be about 1 €/kA-m at an operating temperature of 4.2 K and a magnetic field of 2 T, instead of 50-200 €/kA-m for high- $T_c$  conductors at 77 K and self-field [6, 7].

## 1.2 Magnesium Diboride in Magnets

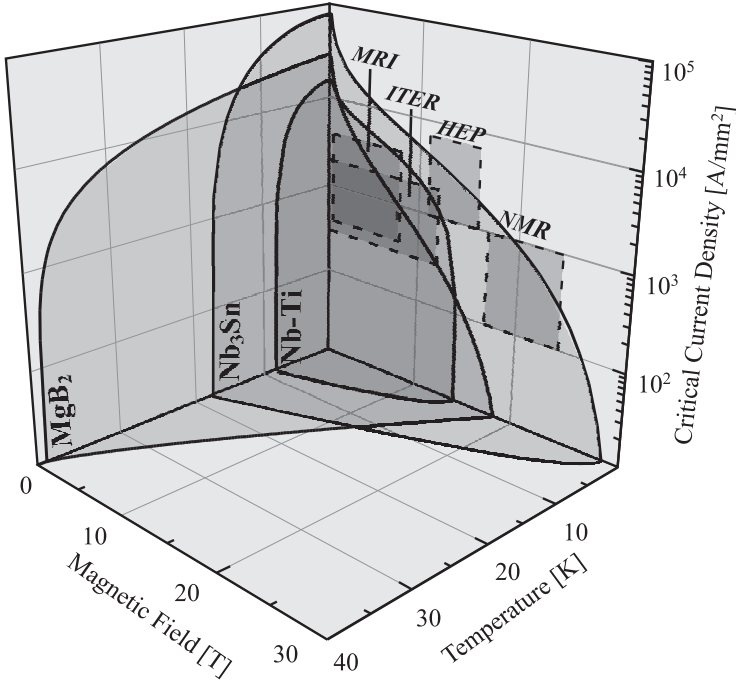
A major interest in the use of  $\text{MgB}_2$  superconductors is in medium-field magnets. Superconductors in magnet systems drastically reduce power consumption, but the application of a superconducting magnet is restricted by the critical surface of the material used. As an example, the critical surface of an optimised  $\text{MgB}_2$  conductor is presented in figure 1.1 [8]. Three critical parameters indicate the intersection of the critical surface with the current-, field- and temperature axes: the critical current density ( $J_{c,0}$ ), the upper critical field ( $B_{c2,0}$ ) and the critical temperature ( $T_c$ ). Below the critical surface, the material is in the superconducting state, above it is resistive.

When considering superconducting electromagnets purely from a performance point of view, a high- $T_c$  material would be the conductor of choice. Indeed, a conduction-cooled Magnetic Resonance Imaging (MRI) demonstrator operating at 20 K with coils wound from  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  was successfully constructed [9], while a record-breaking hybrid solenoid system was recently announced generating 25 T, using a  $\text{Bi}_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_x$  insert coil to provide 5 T extra inside a resistive magnet of 20 T [10]. However, both  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  and  $\text{Bi}_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_x$  are considered too expensive for large magnet systems, while  $\text{YBa}_2\text{Cu}_3\text{O}_x$  coated conductors are not yet commercially available in sufficient length [11]. At present the vast majority of superconducting magnets are made with Nb-Ti wires or, if higher fields are required, a combination of Nb-Ti outer coils and  $\text{Nb}_3\text{Sn}$  inner high-field coils.



**Figure 1.1:** Critical surface of a MgB<sub>2</sub> conductor characterised by the three critical parameters: the critical current density ( $J_{c,0}$ ), the upper critical field ( $B_{c2,0}$ ) and the critical temperature ( $T_c$ ) (based on the data in [8]).

The critical surfaces of state-of-the-art Nb<sub>3</sub>Sn [12] and Nb-Ti [13,14] are compared with that of the present “record” MgB<sub>2</sub> conductor [8] in figure 1.2. The performance of Nb<sub>3</sub>Sn is superior in terms of  $T_c$  and  $B_{c2,0}$  to that of Nb-Ti, but economical arguments dictate the use of Nb-Ti in less demanding applications. Whereas the cost of commercial Nb-Ti is about 1 €/kA-m (at 4.2 K/5 T), Nb<sub>3</sub>Sn is more complicated to produce and its costs fall in the range of 5-25 €/kA-m (at 4.2 K/12 T), depending on the fabrication process [6]. The operational requirements in terms of current density and magnetic field of actual large superconducting magnet applications are also projected in the current-field plane of figure 1.2. Note that superconducting research magnets are excluded since they can be considered as a “niche”-application in terms of both number of units and system size (and thus conductor lengths required) compared to superconducting magnets for MRI, Nuclear Magnetic Resonance (NMR), High Energy Physics (HEP) and Nuclear fusion (ITER). Among these application areas, the conductor choice for HEP and ITER magnets is presently mostly determined by performance and

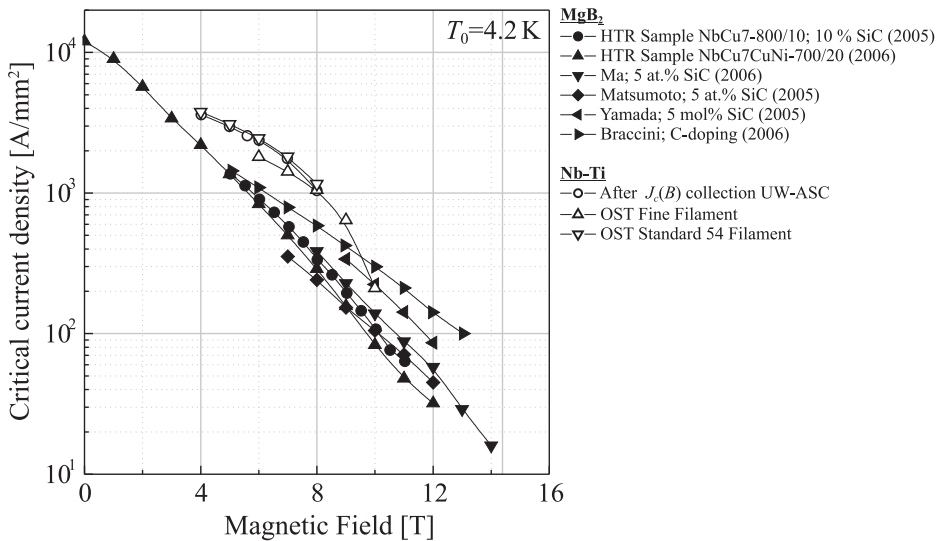


**Figure 1.2:** Comparison of the critical surfaces of MgB<sub>2</sub> and the most widely used superconducting materials Nb<sub>3</sub>Sn and Nb-Ti (after [12–14]). The current-field projections of important applications, such as Magnetic Resonance Imaging (MRI), Nuclear Magnetic Resonance (NMR), High Energy Physics (HEP) and Fusion Energy (ITER) are also indicated.

other technical issues. MRI and NMR, on the other hand, are typical commercial products in the sense that a large and very competitive market dictates choices not only in technical but also in cost terms. As the present current-field behaviour of Nb-Ti and MgB<sub>2</sub> at 4.2 K is quite similar, both materials are of interest for applications such as MRI and medium performance research magnets. For commercial markets cost is a key issue. Indeed, MgB<sub>2</sub> will only be a true alternative for Nb-Ti if the production and running cost of a MgB<sub>2</sub> magnet system is less than that of a Nb-Ti-based magnet system generating a similar magnetic field. An indication for the potential for MgB<sub>2</sub> to be an economical technical superconductor is the fact that its raw materials costs are about 0.80 €/m, which is about half the cost of Nb-Ti [6].

The present  $J_c(B)$  performance at 4.2 K of several commercial and laboratory-scale MgB<sub>2</sub> conductors [8, 17–23] is compared to that of state-of-the-art commer-





**Figure 1.3:** Collection of critical current density versus magnetic field characteristics of Nb-Ti and MgB<sub>2</sub> conductors from several authors. Nb-Ti data from [15,16], MgB<sub>2</sub> data obtained from [17–23]. Note: HTR stands for HyperTech Research.

cial Nb-Ti [15, 16] in figure 1.3. For fields lower than  $\sim 10$  T, the  $J_c(B)$  performance of MgB<sub>2</sub> falls slightly below that of Nb-Ti so that the cost-to-performance ratio is estimated as the earlier mentioned numbers of 1 €/kA-m at 2 and 5 T for MgB<sub>2</sub> and Nb-Ti respectively.

However, the microstructure of Nb-Ti is considered to be close to optimal, with a very fine distribution of  $\alpha$ -Ti pinning sites (on the scale of  $\sim 100$  nm) and a carefully tuned composite conductor layout in terms of number and size of the filaments, superconductor filling factor, matrix conductivity and strength. MgB<sub>2</sub> in comparison is considered to be in an early stage of development and further scope for improvement seems realistic. In this respect it is quite likely that the cost-to-performance ratio of MgB<sub>2</sub> will improve further and become comparable to or even better that of Nb-Ti. An important improvement of the  $J_c(B)$  performance was first reported by Dou *et al.* in 2002 [24] who doped MgB<sub>2</sub> with SiC and increased  $J_c$  in higher magnetic fields with a factor of 5-10 compared to un-doped samples, while the present-day record laboratory scale conductor by Braccini *et al.* [8] is a carbon doped conductor with a technical upper field limit at 4.2 K of 13 T, above which  $J_c < 100$  A/mm<sup>2</sup>. Furthermore, even the latest un-doped wires from HyperTech Research (e.g. sample NbCu7CuNi-700/20 from [19] in figure 1.3) have similar  $J_c(B)$  characteristics to most SiC doped samples. Altogether this is indicative for the steady progress of MgB<sub>2</sub> conductors made since 2001.

Although the improvement of the cost-to-performance ratio can be expected to

continue it should also be mentioned that  $\text{MgB}_2$  conductors, like  $\text{Nb}_3\text{Sn}$ , require a heat-treatment which adds to the cost of magnet production. In this sense, it will be beneficial if  $\text{MgB}_2$  conductors permit a react-and-wind technique, i.e. that the conductor is heat treated prior to magnet winding. However, this will impose mechanical constraints on the conductor behaviour.

The high  $T_c$  of 40 K allows a  $\text{MgB}_2$ -based magnet system to be operated at higher temperatures. The  $J_c(B,T)$  performance of the  $\text{MgB}_2$  conductor from [17] shows that it can carry about 1 kA/mm<sup>2</sup> at a magnetic field of 4 T and an operating temperature of 20 K. These values directly meet the current-field requirements of MRI systems depicted in figure 1.2. Since 20 K is easily reached with present state-of-the-art cryocoolers, the cooling cost can be reduced significantly by the construction of a conduction-cooled MRI magnet system instead of using a relatively expensive liquid helium cryostat at 4.2 K.

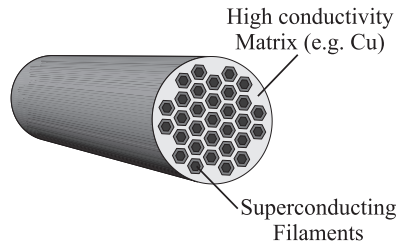
Another possible area of application at 20 K is the foreseen emergence of hydrogen technology in the area of sustainable energy. In order to maximise mass density (and thus chemical energy density),  $\text{H}_2$  might well be stored in the form of a cryogenic liquid, i.e. at or below its boiling point of 20.28 K. This raises the intriguing prospect of combining cryogenic chemical energy storage with various superconducting applications. For example, a  $\text{H}_2$  operated fuel cell that converts the chemical energy released in the reaction  $2\text{H}_2 + \text{O}_2 \rightarrow 2\text{H}_2\text{O}$  to electrical energy works best with constant loads [25]. For applications that require peak loads, a buffer is required. Since batteries, that are presently used in prototypes, are often limited in terms of in- and output power, Superconducting Magnetic Energy Storage (SMES) can fulfill this buffer role as it is capable of storing and delivering large power levels [26].

Since the high-field  $J_c(B,T)$  performance of  $\text{MgB}_2$  at 4.2 K might surpass that of Nb-Ti, it is likely to be used in combination with  $\text{Nb}_3\text{Sn}$  conductors as well. NMR –another very competitive market– typically uses Nb-Ti/ $\text{Nb}_3\text{Sn}$  hybrid magnet systems (see for example [27]).  $\text{Nb}_3\text{Sn}$  is used in the high-field insert coils, while Nb-Ti is used in the low-field outsert coils. Replacing Nb-Ti in the outsert by  $\text{MgB}_2$  might enable it to generate a higher field, requiring a less voluminous insert coil. This way cost can be significantly reduced since  $\text{Nb}_3\text{Sn}$  is a relative expensive material.

### 1.3 Design of Practical Superconductors

The design of a practical superconductor is dictated by the requirements of stable and safe operation. In practice a superconductor is a composite with a large number of fine superconducting filaments embedded in a highly conducting matrix, as depicted in figure 1.4.

Two important requirements that are treated in this thesis are: 1) the superconductor should be thermally stable and it should be possible to protect it against thermal instabilities; 2) the superconductor should be able to withstand



**Figure 1.4:** Typical cross-section of a superconducting wire in which the superconducting filaments are embedded in a highly conducting matrix.

the electromagnetic forces that act on it during magnet operation<sup>1</sup>.

### 1.3.1 Thermal Stability

The lossless transport of very high current densities by a superconductor can, in a certain sense, be seen as a metastable process. When a superconductor carries a current, a small heat release of several micro-joules may already be enough to increase the local temperature well above  $T_c$  [28]. These small heat releases have, for example, a mechanical origin when the magnet is energised and large Lorentz forces start to act on the windings. The resulting “hot-spot” is in the ohmic state (the normal zone) and generates more heat. If heat is generated faster than it is drained, the zone will expand and eventually the whole wire will revert to the normal state, while continuing to carry a high current. When this happens in a magnet, the process is referred to as a quench. In such a process the temperature can rise dangerously fast and the wire might even locally melt or evaporate.

The possibility that an expanding normal zone occurs is inversely proportional to the minimum quench energy MQE. MQE is determined by the thermal margin that needs to be overcome to bring the superconductor locally in the normal state; by the corresponding heat, determined by the volumetric heat capacity  $c$ ; by the initial heat generation  $\rho J^2$ ; and by the degree of heat drainage, determined by both the volumetric heat capacity  $c$  and the thermal conductivity  $\kappa$ .

The impact of a normal zone is determined by the speed of its propagation along the conductor. A faster propagation implies that the generated heat is quickly distributed over a larger volume so that the local temperature rise remains limited. Furthermore, protection schemes that drain the energy stored in the magnet and thus prevent magnet damage typically trigger above a certain voltage level [28]. A faster propagating normal zone means that the voltage across a magnet section rises faster, the normal zone is detected earlier and the protection circuit is triggered faster. The velocity of the normal zone is determined by the

<sup>1</sup>There are further requirements like sufficiently low AC-loss. This issue is not treated in this thesis. Interested readers are referred to [28] for a general introduction and to [29] for the first AC-loss studies on MgB<sub>2</sub> conductors.

**Table 1.1:** Comparison of thermal stability related parameters of Nb<sub>3</sub>Sn, MgB<sub>2</sub> and Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub>.

	Nb <sub>3</sub> Sn		MgB <sub>2</sub>		Bi <sub>2</sub> Sr <sub>2</sub> Ca <sub>2</sub> Cu <sub>3</sub> O <sub>x</sub>
	$T = 4.2$ K	$T = 4.2$ K	$T = 20$ K	$T = 20$ K	$T = 77$ K
Matrix	Cu		Cu (Fe) <sup>b</sup>		Ag
$f$	[%]	55	55 <sup>c</sup>		30
$B^a$	[T]	10	3		0
$T_c(B)^a$	[K]	13	35		110
$J_c(B)^a$	[·100A/mm <sup>2</sup> ]	40	20	6	2
$\kappa^a$	[W/mK]	384	173 (3)	691 (35)	436
$\rho^a$	[·10 <sup>-9</sup> Ωm]	0.3	0.6 (15)	0.6 (14)	5
$c^a$	[·10 <sup>3</sup> J/m <sup>3</sup> K]	1.2	0.7 (1.7)	34 (20)	1550
$D_T$	[·10 <sup>-3</sup> m <sup>2</sup> /s]	329	243 (1.5)	21 (1.7)	0.3
MQE	[·10 <sup>-3</sup> J]	0.1	3.3 (0.04)	90 (0.14)	611

<sup>a</sup> The data presented in this table are obtained from [19,30–36]. The magnetic field  $B$  is chosen in such a way that the  $J_c$  values of the conductors are comparable.

<sup>b</sup> As will be shown later Cu is for chemical reasons not the ideal matrix material for MgB<sub>2</sub>, therefore the values for ARMCO Fe are also added between brackets.

<sup>c</sup> This filling factor is chosen for comparison with the Nb<sub>3</sub>Sn conductor and is the envisaged value. However at present the filling factor for typical MgB<sub>2</sub> conductors is closer to 30%.

conductors' thermal diffusivity  $D_T = \kappa/c$ ; by the heat generation  $\rho J^2$  in the normal zone; and by the thermal margin.

Both MQE and the velocity of a normal zone thus depend on the choice of matrix material. A highly conducting matrix causes heat to drain faster, which explains its use in composite conductors. The operating temperature of a conductor is also a key parameter since all relevant material properties are temperature dependent. For comparison, table 1.1 shows values of stability-related parameters for Nb<sub>3</sub>Sn, MgB<sub>2</sub> and Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> composite conductors at their usual operating temperatures. The effective volumetric heat capacity of the three materials increases drastically with operating temperature. Correspondingly, the diffusion constant decreases, while MQE increases. This results in a clear qualitative difference between the behaviour of low- $T_c$ - and high- $T_c$  conductors. The minimum quench energy of Nb<sub>3</sub>Sn is relatively small compared to typical thermal disturbances that can occur inside a magnet, increasing the risk of a quench. However, due to the large thermal diffusivity the generated heat is rapidly distributed along the conductor, limiting the temperature rise. Moreover, the normal zone voltage across the conductor is easily measured. In Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub> the situation is reversed. MQE is large compared to typical disturbances, so there is a low risk that the conductor will quench. However, if such an event does occur, heating remains localised because of the low thermal diffusivity and a rapid growth of the local

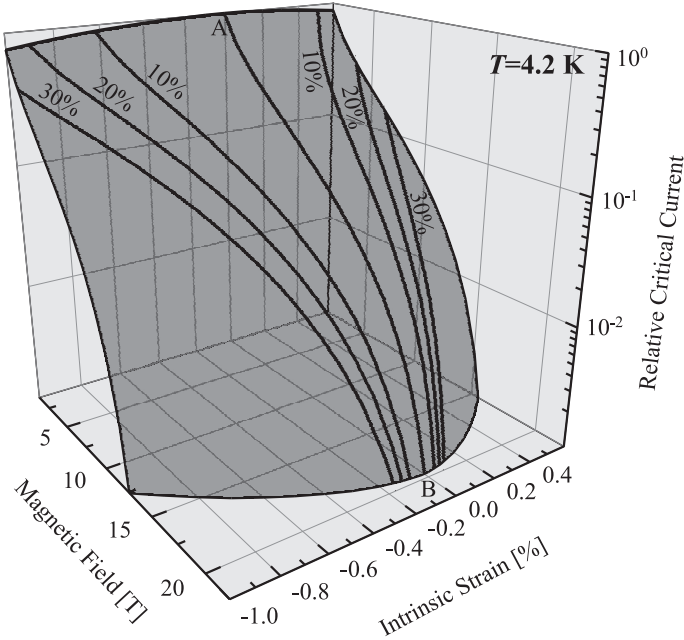
temperature can be expected. The normal zone is difficult to detect because of its slow expansion. At an operating temperature of 4.2 K the characteristics of  $\text{MgB}_2$  are quite similar to  $\text{Nb}_3\text{Sn}$ , depending on the matrix material. However, the envisaged operating temperature of  $\text{MgB}_2$  can also be about 20 K. Thus, to gauge the suitability of  $\text{MgB}_2$  conductors for magnet systems running at  $\sim 20$  K, conductor properties will need to be carefully modeled and measured. Such study is the subject of chapter 4 of this thesis.

### 1.3.2 Mechanical Strain

Besides thermal stability, the response to mechanical stress can also impose stringent boundary conditions on the design of a practical superconductor. Strain is the response of a material to external stress, which at high levels causes irreversible changes of the superconducting properties due to damage in the superconducting filaments, especially in brittle materials like  $\text{Nb}_3\text{Sn}$ ,  $\text{MgB}_2$  and high- $T_c$  conductors. At lower strain levels the superconducting properties change reversibly. Hydrostatic pressure experiments on superconducting materials indeed show a  $T_c$  reduction when the pressure is increased [37, 38]. This effect can to first order be explained by BCS theory through a reduced electron-phonon coupling energy due to lattice deformation [39].

Although the effects of hydrostatic pressure (i.e. lattice deformations) on the superconducting properties were already evident in the early 1960's [37], it took until the mid-1970's before technical superconductors were not only characterised by their critical current, upper critical field and critical temperature but also by their response to mechanical stress or strain [40]. Nb-Ti superconductors were subjected to longitudinal stress and a degradation of  $J_c$  was observed that could not be attributed to the reduction in cross-sectional area of the filaments induced by the conductor's elongation. In Nb-Ti this effect is small: a 10 % reduction of  $I_c$  requires an applied strain of about 2 % or 800 MPa of applied stress.  $\text{Nb}_3\text{Sn}$  and other A15 compounds are much more sensitive to strain. Early measurements by Ekin *et. al.* [41] showed 10 % degradation of  $I_c$  at only 0.2 % strain or an equivalent stress of 100 MPa, which is roughly a factor of 10 lower than in Nb-Ti. In view of the technical relevance of the strain response of superconductors, Ekin described a four dimensional  $J$ - $B$ - $T$ - $\varepsilon$  critical surface for A15 based technical superconductors which is described by a scaling law [42, 43]. An example of the  $J$ - $B$ - $\varepsilon$  critical surface of a commercial  $\text{Nb}_3\text{Sn}$  conductor [43] is reproduced in figure 1.5. The  $A - B$  line shows the  $J_c(B)$  curve at zero strain state, whereas the curves on both sides are the loci at which the critical current at  $B = 1$  T is degraded by 10, 20 and 30% with respect to its unstrained value.

Meanwhile, other models have been developed to describe the strain response in A15 based practical superconductors [12, 44]. Although they accurately predict the strain response of the whole critical surface with just a small number of observations, they all have in common that they are empirical and do not explain the physical background of the strain response of the critical parameters. Microscopic models that describe the pressure dependence of  $T_c$  are based



**Figure 1.5:**  $J$ - $B$ - $\varepsilon$  critical surface of a multifilamentary  $\text{Nb}_3\text{Sn}$  superconductor (after [43]). Line  $A - B$  represents the maximum strain-free value of the critical current as a function of magnetic field. The loci to the left and right of the  $A - B$  line represent 10, 20 and 30% degradation of  $I_c$  at  $B = 1$  T with respect to its strain free value. Note that the critical current is normalised over its strain-free value at  $B=1$  T.

on hydrostatic pressure and thus hydrostatic strain. For technical applications, however, non-hydrostatic terms have a significant influence and must be included. Recently, Markiewicz [45, 46] presented a full three-dimensional, strain-invariant analysis of the strain dependence of  $T_c$  for  $\text{Nb}_3\text{Sn}$  conductors. The central assumption of this analysis is that the strain dependence of  $T_c$  results from the strain dependence of the phonon frequencies in the  $\text{Nb}_3\text{Sn}$  lattices. The model correctly predicts the strain dependence of practical  $\text{Nb}_3\text{Sn}$  superconductors based on physical assumptions, rather than being empirical.

Strain in practical superconductors is caused by three effects: firstly, thermal stresses and strains built up in a composite superconductor when it is cooled down from the heat treatment temperature ( $> 600$  °C) to the operating temperature, due to differences in thermal contraction coefficient of filaments and matrix. Secondly, especially in react-and-wind type of applications, conductors are subjected to a range of bending, torsion and tension when they are first bundled into

a cable and subsequently wound into a coil. Thirdly and arguably most important, stresses build up due to the considerable Lorentz forces generated by the combination of high current and high magnetic field.

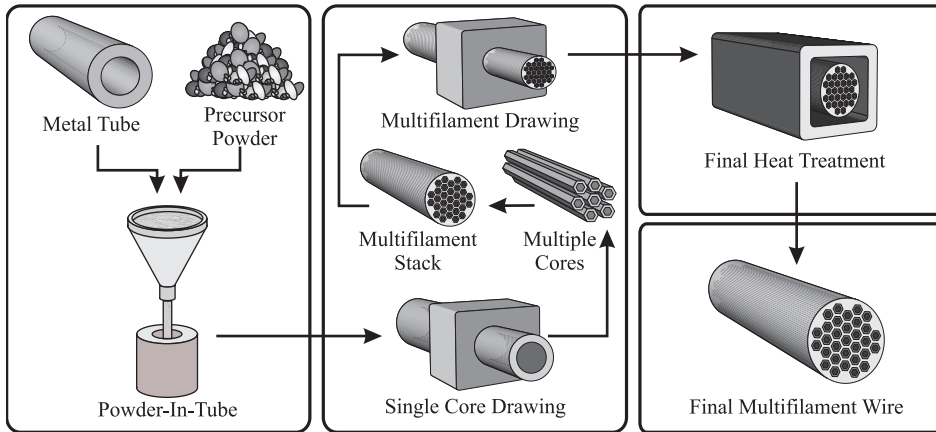
To illustrate the technical relevance of the strain response of the critical parameters, consider a solenoid of 8 T with inner and outer radii of about 73 and 100 mm, respectively. The maximum stress that is exerted by the magnetic field in the windings is approximately 120 MPa [47]. For a Nb<sub>3</sub>Sn wire this corresponds to an axial strain of about 0.35 % [48], which reduces its critical current by about 20 % [49]. Moreover, this strain level is already in the irreversible regime, so that the filaments are damaged.

Evidently, for proper magnet engineering the strain sensitivity of MgB<sub>2</sub> should be studied in order to predict changes to the critical surface of the conductors under the influence of electromagnetic forces. MgB<sub>2</sub> and Nb<sub>3</sub>Sn are alike in the sense that they are both brittle materials so that the irreversible regime can be expected to occur at low strain levels in comparison to Nb-Ti. For this reason it is important that not only the response in the reversible regime is explored, but also that the maximum reversible strain level is studied. These studies are the topic of chapter 5.

## 1.4 Fabrication of MgB<sub>2</sub> Conductors

Practical superconductors consist of several filaments of superconducting material embedded in a highly conducting matrix. Wires are usually reduced in size by extrusion or drawing, depending on the desired reduction and batch length. For the ductile alloy Nb-Ti, this is a relatively easy process. MgB<sub>2</sub>, on the other hand, is like Nb<sub>3</sub>Sn a hard intermetallic compound, which is more difficult to deform. For Nb<sub>3</sub>Sn this problem is avoided with the Bronze, Internal Tin and Powder-In-Tube processes [12]. Essentially a billet is assembled that contains niobium and tin as separate phases. Due to the ductility of the separate elements, it is relatively easy to deform a billet into a wire. In the final wire form, the Nb<sub>3</sub>Sn is phase formed during a diffusion heat treatment. Similar strategies are adopted for MgB<sub>2</sub>.

Only months after the discovery of superconductivity in MgB<sub>2</sub>, the first trials were made to produce practical superconducting wires. One of the first attempts consisted of a boron clad tungsten wire that was exposed to magnesium vapour at 950 °C [50, 51]. The resulting 160 μm diameter wire was of very high purity. A successful variation of this method is the “coated conductor” route, in which a boron clad Hastelloy substrate is exposed to magnesium vapour [52]; the “liquid infiltration” technique, in which a boron tube is filled with magnesium and reacted [53]; and, recently, a solid state diffusion route, in which boron powder is deformed inside a Fe-Mg alloy tube and then reacted [54]. However, for long length production of MgB<sub>2</sub> wires the Powder-In-Tube process (PIT) might be a more convenient and proven method. Figure 1.6 shows a simplified schematic of the PIT fabrication process of a multi-filament wire. In this example deformation



**Figure 1.6:** Simplified representation of the Powder-In-Tube fabrication method. The precursor powder can be pre-reacted  $\text{MgB}_2$  in the ex-situ route or a mixture of Mg and B in the in-situ route.

is performed by drawing. However, other deformation routes are also applied, such as 2-axial rolling, groove rolling and flat rolling [55–59]. Besides the deformation method, there are two main strategies regarding the precursor powder: 1) *the ex-situ route*, in which pre-reacted  $\text{MgB}_2$  powder is put into a tube; and 2) *the in-situ route* where magnesium and boron powders are well mixed and put into a tube. In both cases the powder-tubes are deformed into single-core wires. The wires are then stacked into a multi-filament billet. After deformation of the billet into a wire, it is heat treated. In the ex-situ route the purpose of the heat treatment is to increase the cross-section of the grain-to-grain current path by sintering the powder grains. With the in-situ route the  $\text{MgB}_2$  phase is formed during this heat treatment.

Since the tube in which the powder is filled will be part of the matrix in the final wire, it should preferably be a material with high thermal and electrical conductivity, such as copper or aluminium. Moreover, such materials are often ductile and easy to deform. In  $\text{Nb}_3\text{Sn}$  conductors copper is usually applied. However,  $\text{MgB}_2$  reacts strongly with materials such as copper, aluminium or silver [56,60–62]. Materials that are chemically compatible with  $\text{MgB}_2$  are iron, steel or nickel, but these metals are not excellent conductors and rather hard. Another requirement is that optimal  $J_c(B,T)$  performance implies a high degree of electrical connectivity between the grains. Chapter 2 will show how grain-to-grain connectivity depends strongly on various process parameters.

To have a broader overview of possibilities and trends in terms of the  $J_c(B,T)$  performance, a literature study is presented of reported  $\text{MgB}_2$  conductors prepared with different deformation techniques and precursor powders. Conclusions drawn from this study are the topic of chapter 3.



## 1.5 Scope of the Thesis

In this thesis the focus is on the question whether  $\text{MgB}_2$  conductors are suitable for use in superconducting magnets. To arrive at a conclusion several questions have to be addressed:

*1) Is the  $J_c(B,T)$  performance of  $\text{MgB}_2$  conductors suitable for magnet applications?*

The answer to this question depends on the preparation route of the conductors. High  $J_c(B,T)$  values require a high densification of the powder core in order to increase the cross-sectional area of the current path. The degree of densification depends on the deformation method, matrix material and precursor powder. How combinations of these parameters affect the  $J_c(B,T)$  performance is the subject of chapter 2. Results are put in a broader perspective in chapter 3 with a literature survey that reveals prospects and trends in the development of  $\text{MgB}_2$  conductors.

Since a magnet system must be protected against thermal instabilities to avoid a burnout, the second question is:

*2) Given the conductors matrix materials and operating temperature, can a  $\text{MgB}_2$  magnet be protected against thermal instabilities?*

Choice in composition of the matrix is complicated by the fact that  $\text{MgB}_2$  reacts strongly with most high-conductivity metals. Therefore poorly conducting materials are typically present in the composite. Together with the prospect of a relatively high operating temperature, this can compromise the ease of normal zone detection. On the other hand, a higher operating temperature may decrease the possibility of normal zone to occur. The influence of matrix composition and operating temperature on the normal zone propagation and minimum quench energy are presented in chapter 4.

Since electromagnetic forces in magnets can be significant and affect the critical parameters of  $\text{MgB}_2$  conductors, the third question is:

*3) Given a  $\text{MgB}_2$  conductor with a certain matrix, does it support the electromagnetic forces acting on a magnet?*

$\text{MgB}_2$  is a brittle material so that this question should be separated in two parts: 1) is the strain level at which the irreversible regime starts high enough to prevent damage of the filaments?; and 2) to what extent are the critical parameters influenced by strain in the reversible regime? These issues are addressed in chapter 5 by mapping the strain response of several  $\text{MgB}_2$  conductors in the reversible regime and by exploring the irreversible strain limit.

Finally, in chapter 6 the main conclusions of this thesis are summarised. The coherence between the chapters is illustrated in figure 1.7.

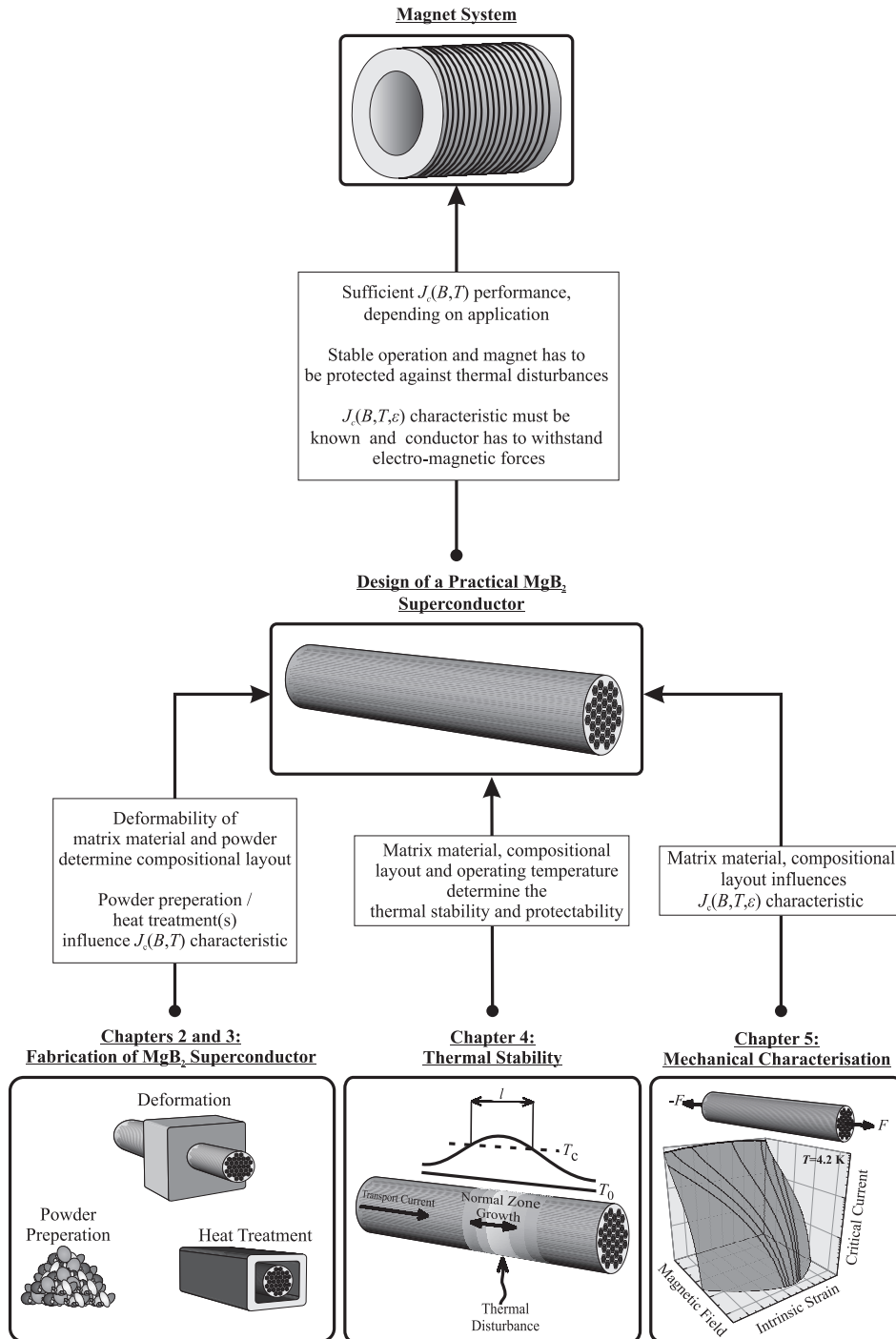


Figure 1.7: Coherence of chapters in the thesis.

## Chapter 2

# Preparation of Ex-Situ Magnesium Diboride Conductors

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*In this chapter the development of ex-situ PIT conductors using different precursor powders, matrix materials and deformation methods is discussed. Precursor powder preparation with a relatively simple method is presented. Causes of conductor fracture during deformation are explored and possibilities to prevent them are discussed. Finally, the combination of parameters that offers the best  $J_c(B,T)$  performance is presented.*

## 2.1 Introduction

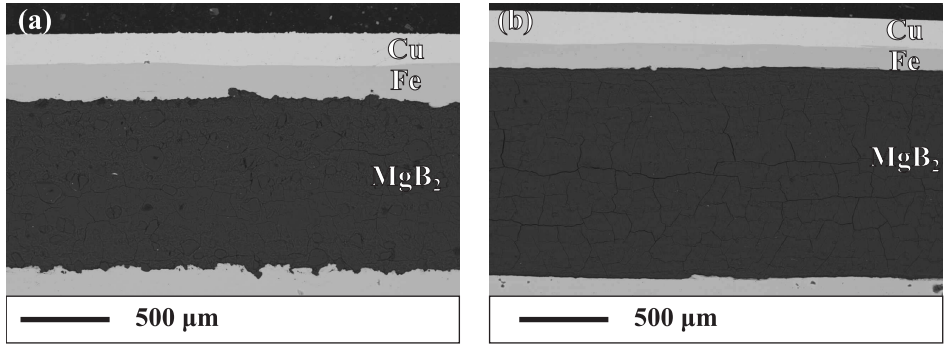
The two main development routes for technical PIT  $\text{MgB}_2$  conductors are the in-situ and ex-situ techniques (see also §1.4). The advantage of using the in-situ route is that the soft Mg acts as a lubricant for powder flow in the filament during deformation. As will be shown in §3.4.2 this results in a high filament density and high critical current densities. Low-level doping can also be better controlled in in-situ conductors so that the field retention of  $J_c$  can be significantly enhanced [24]. On the other hand, ex-situ conductors show better  $n$ -values due to more homogeneous morphology. Furthermore, recent developments show that effective pinning centers can also be introduced in ex-situ powder [8], increasing the practical upper field limit at which  $J_c = 100 \text{ A/mm}^2$  to as high as 13 T.

In this chapter the development of ex-situ PIT conductors is described. A key requirement in the deformation of ex-situ conductors is to reach sufficient filament density with the hard  $\text{MgB}_2$  powder in order to obtain high critical current density values. Parameters that influence filament densification are the deformation method, the yield strength of the matrix material and the powder morphology.

For high current density  $\text{MgB}_2$  conductors it is important that the grains are small ( $\sim 1 \mu\text{m}$ ). Not only do small grains lead to improved grain-boundary pinning and enhanced  $J_c$  values [63], but they also prevent stress concentrations in the matrix material, which can cause early breakage of the conductor. The technique to reduce the particle size of the as-purchased powder is presented in §2.2. Moreover, to further enhance the deformation capacity the effect of adding the soft metal Pb is also tested. Lead additions also influence the current carrying capability of ex-situ conductors. The preparation of  $\text{MgB}_2/\text{Pb}$  mixtures and their influence on the superconducting properties is discussed in §2.2.

To gauge which deformation method is best suited for ex-situ conductors, wires were fabricated both by drawing and two-axially rolling, while tapes were fabricated with the flat-rolling technique. Different matrix materials were used to test their influence on the powder densification in the filaments. One option tested is a bi-metal Cu/Fe composite in which the relatively soft and highly conducting Cu acts as a thermal stabiliser, while the relatively hard Fe provides the strength for powder densification. How combinations of the various parameters including Pb additions affect the filament densification and maximum achievable area reduction is the subject of §2.3.

The critical current density of as-deformed ex-situ  $\text{MgB}_2$  PIT conductors can be significantly enhanced by a heat-treatment that causes recrystallisation and sintering of the  $\text{MgB}_2$  grains [64]. The effects of the final heat treatment on the microstructure and superconducting properties of the differently deformed conductors with different matrix materials and varying concentrations of Pb are discussed in §2.4.1. The best-performing conductors presented in this work are compared to conductors reported elsewhere over the years 2003-2006.



**Figure 2.1:** (a) SEM image of the longitudinal cross-section of a  $\text{MgB}_2/\text{Fe}/\text{Cu}$  as-drawn sample with as-purchased powder and (b) a similar sample with 48-hours milled powder.

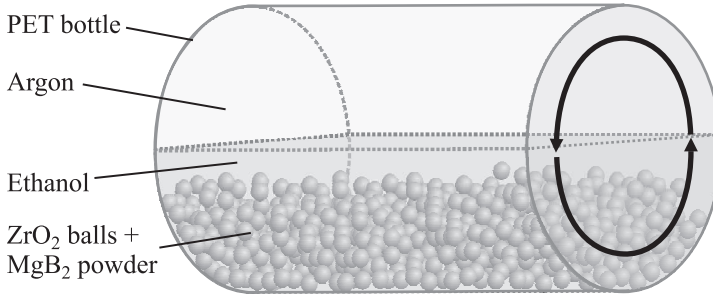
## 2.2 Powder Processing

To optimise the effective current path in PIT conductors, the powder density in the filaments after deformation should be as high as possible. The maximum density that can be obtained is influenced by the force that the matrix materials can exert on the powder core and by the flow characteristics of the powder. The latter depends on the morphology and mechanical properties of the powder particles<sup>1</sup>.

Since  $\text{MgB}_2$  is a relatively hard material, particle size distribution is a key issue. This is illustrated in figure 2.1, where as-drawn wires with an identical matrix and deformation history but with different starting powders are compared. The tube in figure 2.1a was filled with as-purchased powder with particles up to  $\sim 100 \mu\text{m}$ , while the tube in figure 2.1b was filled with powder particles up to  $\sim 2 \mu\text{m}$  in size. The as-purchased powder results in a rougher core/matrix interface as the large particles penetrate into the Fe. This gives rise to local stress concentrations and may cause the appearance of micro-cracks in the core or even premature breaking of the wire during drawing. The conductor made with the finer powder shows a much smoother interface. Also in light of development of multi-filament conductors [19,65] with filament sizes of  $< 50 \mu\text{m}$ , it is essential to reduce the particle size.

Apart from improved connectivity and thus higher  $J_c$  values, particle size reduction might also lead to better field retention of the critical current. It was found by [66] that smaller grains increase the irreversibility field  $B^*$ , which may

<sup>1</sup>In the context of powder processing, the general term “particle” is used since the powders do not only consist of single-crystalline grains, but also of hard agglomerates and polycrystalline lumps of the various materials.



**Figure 2.2:** Schematic drawing of the shear milling process used to reduce the  $\text{MgB}_2$  particle size.

be associated with improved grain boundary pinning<sup>2</sup>.

In this work, the particle size of the as-purchased  $\text{MgB}_2$  is reduced using a relatively simple but effective shear milling technique or by particle selection through sieving. The details and efficiency of these methods are discussed in §2.2.1.

The method used to reduce the particle size should not influence the purity of the powder, since this might deteriorate the superconducting properties. These issues are discussed in §2.2.2.

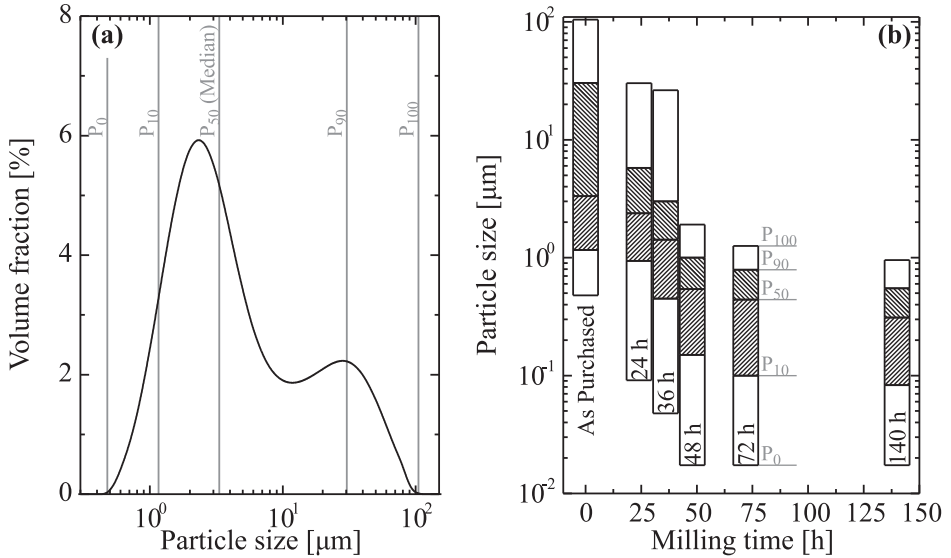
Another method to achieve higher density and better grain-to-grain connectivity is the addition of soft metals. These additions may act as a lubricant for powder flow and thus prevent crack formation. Moreover, when finely distributed in-between the grains, the metal might influence the grain connectivity through proximity coupling [67]. In this work Pb is used to test the effect of soft metal additions. The details are described in §2.2.3.

## 2.2.1 Reducing Particle Size

### Shear Milling

With the shear milling technique used in this work particles are reduced in size by impact as well as shear stresses [68]. Other milling techniques commonly used for  $\text{MgB}_2$ , such as planetary ball milling, mainly reduce particle size by impact of the milling balls. The shear milling technique is schematically shown in figure 2.2. The powder is placed in a cylindrical container together with a relative large amount of milling balls. A fluid is added to the mixture, which increases the efficiency of the milling process by reducing the packing of particles and preventing the formation of inactive corners in the container. The container is rotated so that

<sup>2</sup>Pinning mechanisms in  $\text{MgB}_2$  conductors are further discussed in §3.4.



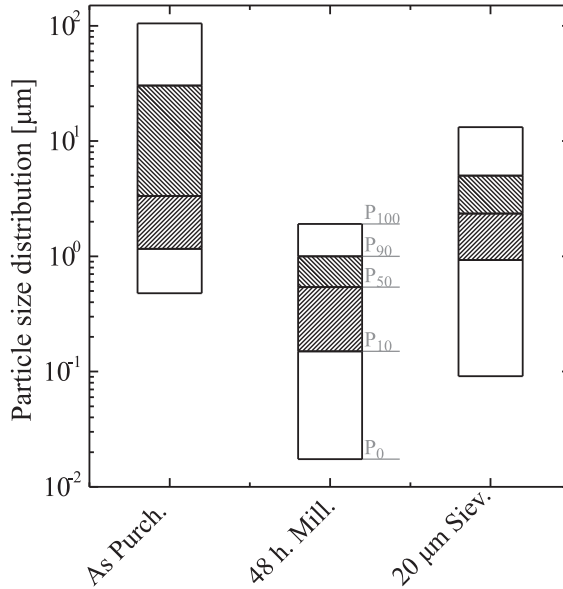
**Figure 2.3:** (a) particle size distribution of as purchased  $\text{MgB}_2$  powder; and (b) the particle size distribution (plotted as floating columns) as a function of milling time. Indicated are the 0, 10, 50 (median), 90 and 100 % percentiles.

the milling balls cascade. The powder particles in-between the milling balls are crushed through impact and shear stress.

The chosen suspension fluid is ethanol, which is widely available and does not react with  $\text{MgB}_2$ . For the milling balls,  $\text{ZrO}_2$  with a diameter of  $\approx 3$  mm is used. The Vickers hardness  $H_v$  of the milling balls is about 1300, exceeding that of single crystal  $\text{MgB}_2$  ( $H_v \sim 1100$  [69]). The mixture  $\text{MgB}_2$ :ethanol: $\text{ZrO}_2$  with weight ratio 5:10:90 is prepared in an Ar-filled glove-box to prevent oxygen contamination. The mixture is poured in a polyurethane container, which is then rotated on a rolling bench. After milling, the balls are removed and the  $\text{MgB}_2$ /ethanol suspension is sealed in a closed container. To dry the powder, this container is connected to a vacuum pump and the ethanol is evaporated.

The particle size distribution of the powders, represented as a volume fraction versus particle size, is measured with a Malvern Instruments Mastersizer 2000, with the  $\text{MgB}_2$  powder suspended in ethanol. Since gas bubbles and agglomerates appear as large particles, the intensity and duration of degassing and ultrasonic vibration of the suspension is varied until the distribution remains constant. As an example, the particle size distribution of the as-purchased powder is shown in figure 2.3a. The 0, 10, 50 (median), 90 and 100 % percentiles ( $P_0$ ,  $P_{10}$ ,  $P_{50}$ ,  $P_{90}$  and  $P_{100}$ ) of the volume fraction are also indicated.

How the particle size distribution changes as a function of milling time is shown in figure 2.3b, with particle size distributions represented as floating columns of



**Figure 2.4:** Comparison of the particle size distribution of as purchased, 48 h shear milled and 20  $\mu\text{m}$  sieved powders.

the percentiles. During the first 48 h the maximum particle size steadily reduces from 100  $\mu\text{m}$  to 2  $\mu\text{m}$ . After that, the particle size distribution changes relatively little. To minimise effects of contamination due to wear of the milling balls, the standard milling time is set to 48 h.

Note that this shear milling technique not only efficiently reduces the particle size, but also that its yield is virtually 100 %. Furthermore, it can be scaled-up to larger volumes relatively simply.

### Sieving

A disadvantage of any milling method is contamination associated with wear of the milling tools. Figure 2.3a shows that, although the maximum particle size in the as-purchased powder is too large, 50 vol.% of the particles are already smaller 3  $\mu\text{m}$ . In principle sieving the powder should still result in a reasonable yield, without the risk of excessive contamination.

For sieving,  $\sim 100$  g as-purchased  $\text{MgB}_2$  is suspended in 1 l of ethanol. This suspension is then poured through a cascade of sieves with mesh sizes of 90, 63, 40 and 20  $\mu\text{m}$ . Reducing the mesh size even further significantly reduces the yield due to excessive clogging of the finer meshed sieves. After sieving, the powder is dried in a similar fashion as with the milled powder. The resulting particle size distribution is plotted in figure 2.4 and compared to the distributions of as-purchased and 48 h milled powders. Compared to milling, sieving is less effective



in terms of particle size reduction. Also, the yield of  $\sim 20\%$  is relatively low, mainly due to clogging of the sieves. Nevertheless, in the remainder of this work sieved powders are used, since the milling procedure with ethanol described above turns out to cause excessive contamination as discussed in the next paragraph.

## 2.2.2 Superconducting Properties and Contamination

### Magnetisation Measurements

Magnetisation measurements were carried out on pellets pressed from four differently treated powders to check whether various processing steps introduce contamination. The first powder was the as-purchased one, the second the shear milled, the third the sieved and the last one the as-purchased after soaking in ethanol for 48 h and drying. The latter powder served to distinguish between prolonged exposure to ethanol and the actual milling or sieving procedures.

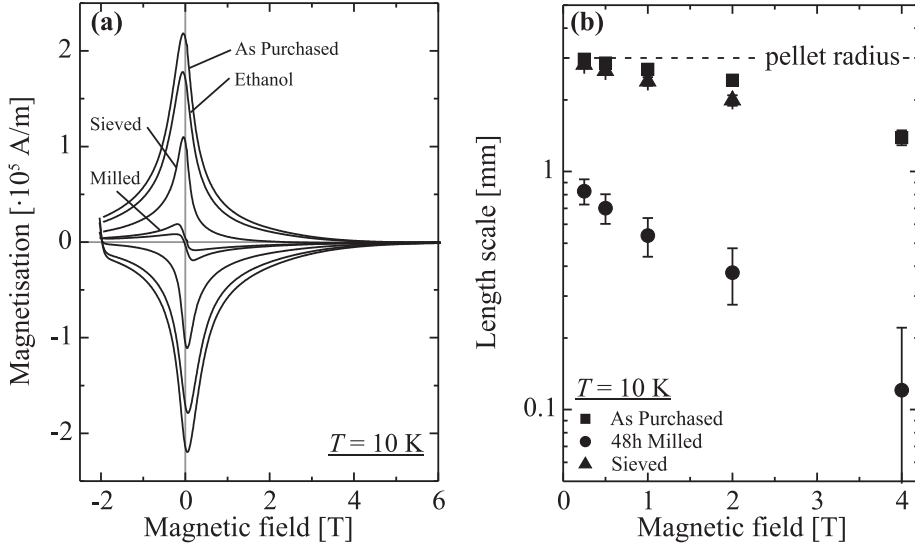
Each of the four powder types was prepared from two different batches of commercial Alpha-Aesar  $\text{MgB}_2$ . The eight powder samples were pressed into pellets of 6 mm diameter and 2 – 3 mm thickness with a force of 3 kN, corresponding to a pressure of  $\sim 1$  GPa. Magnetisation loops were recorded at  $T = 10$  K with a VSM, using a field ramp rate of 2 T/min.

The magnetisation loops of four pellets from the same batch are shown in figure 2.5a. Differences with corresponding loops recorded for the other batch are negligible. Soaking the as-purchased powder in ethanol has no significant influence on the superconducting properties: differences between the loops for the as-purchased and soaked powder can simply be attributed to the uncertainty in the pellets' volume, which is about  $\sim 10\%$ . The 45% smaller magnetisation loop of the pellet made from sieved powder can probably be attributed to a higher porosity of the pellet. The mass density of the pellets of as-purchased or soaked powders is  $75 \pm 3\%$  of the theoretical  $\text{MgB}_2$  density ( $\approx 2.62$  g/cm<sup>3</sup> [64]), while that of the milled or sieved powder pellets is  $72 \pm 2\%$ . The difference can be related to the narrower particle size distributions of the milled and sieved powder (2.4), which leads to less efficient packing in the pressed pellets. Correspondingly, the intergranular  $J_c$  will decrease and the magnetisation loop will become smaller. However, it is difficult to explain the reduction of the milled powders' loop by an order of magnitude compared to that of the as-purchased one with such a porosity argument.

To understand the nature of the reduction after milling, the length scale  $\Lambda$  over which coherent macroscopic current loops flow is determined. A magnetisation loop of a polycrystalline superconductor contains contributions of both inter- and intragranular current flows so that the magnetic moment  $\Delta m$  can generally be described as follows [70]:

$$\Delta m = (2/3)V_{\text{core}}(J_{\text{inter}}\Lambda + J_{\text{intra}}a_0), \quad (2.1)$$

where  $V_{\text{core}}$  is the volume of the superconducting core,  $J_{\text{inter}}$  and  $J_{\text{intra}}$  the inter- and intragranular current densities,  $a_0$  the typical grain size and  $\Lambda$  the typical



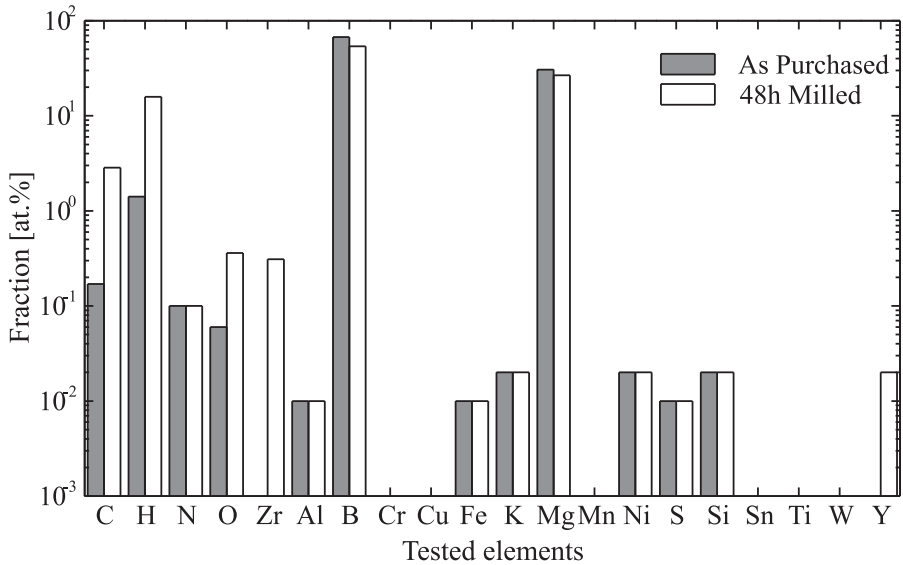
**Figure 2.5:** (a) shows magnetisation measurements at  $T = 10$  K of the as purchased  $\text{MgB}_2$ ,  $\text{MgB}_2$  that is submerged in ethanol for 48 h, 48h milled  $\text{MgB}_2$  and  $20 \mu\text{m}$  sieved  $\text{MgB}_2$ . (b) shows the corresponding length scales of the super-currents in the pellets.

size of intergranular current loops. For homogeneous samples  $\Lambda$  has the value of the sample radius  $R$  and the first term in equation (2.1) dominates provided that

$$\frac{J_{\text{inter}}}{J_{\text{intra}}} > \frac{a_0}{R} (\approx 0.001 - 0.01).$$

For inhomogeneous samples,  $\Lambda$  will decrease below the sample size. The actual length scale in the pellets is determined with an independent experiment, through detailed analysis of the  $m(H)$  behaviour after field sweep reversal. The initial repenetration slope  $dm/dH \propto \Lambda^3$  is independent of the magnitude of the critical current density [71].

The length scale  $\Lambda$  of the different powder pellets as a function of magnetic field is shown in figure 2.5b. Both the as-purchased and the sieved powder show a length scale of the order of the pellet radius, indicating homogeneous intergranular current flow. This is not the case for the milled powder, in which the length scale becomes nearly an order of magnitude lower than the pellet radius. Together with the strong reduction of the loop amplitude, this indicates a strong suppression of the intergranular currents in the milled powder pellet.



**Figure 2.6:** Fraction of tested elements found in as-purchased MgB<sub>2</sub> and 48 h milled MgB<sub>2</sub>.

## Chemical Analyses

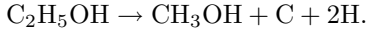
To clarify the mechanism behind the reduction of the intergranular  $J_c$ , various chemical analyses are performed.

First, the chemical composition of the milled and the as-purchased powders is compared. The concentration of the relatively light elements C, H, N, O was determined with a Carlo-Erba Elemental Analyser, while all other elements were determined with ICP measurements. As shown in figure 2.6, the milled powder has a significantly higher concentration of Zr, O and Y. This is a direct reflection of the wear of the Y<sub>2</sub>O<sub>3</sub> stabilised ZrO<sub>2</sub> balls. Furthermore, the C and H concentration in the milled powder is an order of magnitude higher than in the as-purchased powder. The only possible source of these two elements is the ethanol in which the powder is milled, indicating a chemical reaction.

Independently, the gas present in the polyurethane container after milling was analysed. This was motivated by the observation that after 48 h of milling the pressure inside the container had increased significantly, again indicating a chemical reaction. Gas chromatography revealed a final atmosphere of ~ 50 % H<sub>2</sub> and ~ 50 % Ar, where the initial gas in the bottle only contained Ar and traces of O<sub>2</sub> and H<sub>2</sub>O (< 1 ppm).

The order of magnitude higher concentration of C in the milled powder together with the H<sub>2</sub>-gas in the polyurethane bottle leads to the conclusion that

the ethanol is partially converted into methanol, releasing  $\text{H}_2$  gas and C:



Based on this chemical reaction, it is estimated that  $\sim 0.5$  vol.% of the ethanol is converted into methanol<sup>3</sup>. Thus the mechanism behind intergranular current suppression by milling can be summarised as follows: during shear milling in ethanol, the ethanol is partly converted into methanol leaving C as impurity in-between the  $\text{MgB}_2$  grains. In order to preserve the superconducting properties while still reducing the particle size with this effective shear milling technique, a more inert fluid should be found.

Isopropanol was tested as an alternative, but also in this case gas chromatography showed a final gas atmosphere still containing about  $\sim 35$  %  $\text{H}_2$ . Also ICP measurements after replacing  $\text{ZrO}_2$  balls with SiC ones lead to a similar C concentration as the ones shown in figure 2.6.

### 2.2.3 Soft Metal Additions

Advantages of soft metal additions in ex-situ conductors can be three-fold. First, soft metal additions can significantly enhance the critical current density [72, 73] due to formation of S/N/S junctions between the normal metal and  $\text{MgB}_2$ . Effectively this leads to better grain-to-grain connectivity and an enhancement of the self-field  $J_c$ . Second, soft metals may improve the powder flow as it acts as a lubricant during deformation, reducing the friction between the  $\text{MgB}_2$  grains and reducing the risk of crack formation in the filaments. Third, soft metal additions generally have a higher thermal and electrical conductivity than  $\text{MgB}_2$  and can therefore act as an internal thermal stabiliser [74].

In this work, Pb powder with a typical grain size of  $< 100 \mu\text{m}$  is mixed in an Ar atmosphere with  $20 \mu\text{m}$  sieved  $\text{MgB}_2$  powder. To test the effect of Pb additions on the deformation behaviour of conductors, critical current density and thermal stability, powder mixtures were prepared with Pb concentrations varying from 0 to 15 vol.%. These powders were used in different types of conductors (rolled or drawn) and the resulting properties are discussed in paragraphs 2.3 and 2.4.

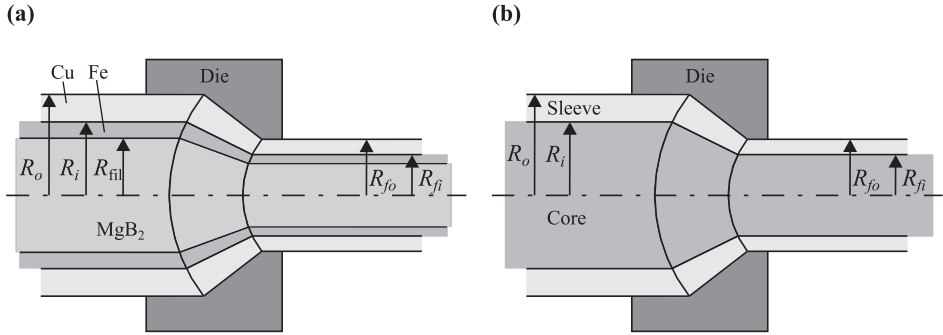
## 2.3 Deformation

The maximum achievable powder densification in the PIT process depends on the morphology of the powder, but also on the deformation route and the choice of matrix material.

Different deformation processes impose different powder flow profiles so that the maximum achievable powder density may differ as well. In case of drawing, densification is obtained by the radial force that the die exerts on the conductor, but the axial pulling force counteracts this. For flat-rolling on the other hand, only

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<sup>3</sup>It is assumed that the H found in the  $\text{MgB}_2$  powder is bound to C.



**Figure 2.7:** Schematic view of co-deformation of a  $\text{MgB}_2$  conductor with a Cu/Fe matrix (a) and a bi-metal rod (b) by drawing.

a transverse force is involved. To test which deformation procedure is best suited for the deformation of ex-situ conductors, three methods were tested: drawing, flat-rolling and two-axial rolling.

The maximum densification is also determined by the amount of force that the matrix material can transmit to the powder. The choice of materials is limited by requirements of chemical compatibility and thermal stabilisation. Therefore two types of matrix were used: 1) a combination of Cu and Fe, in which Cu provides the thermal stability and Fe acts as a reaction barrier; and 2) a thicker Fe matrix that provides more strength for increased powder densification. Although pure Fe has a lower conductivity, in combination with a  $\text{MgB}_2/\text{Pb}$  powder mixture it may still yield a conductor with sufficient thermal stability.

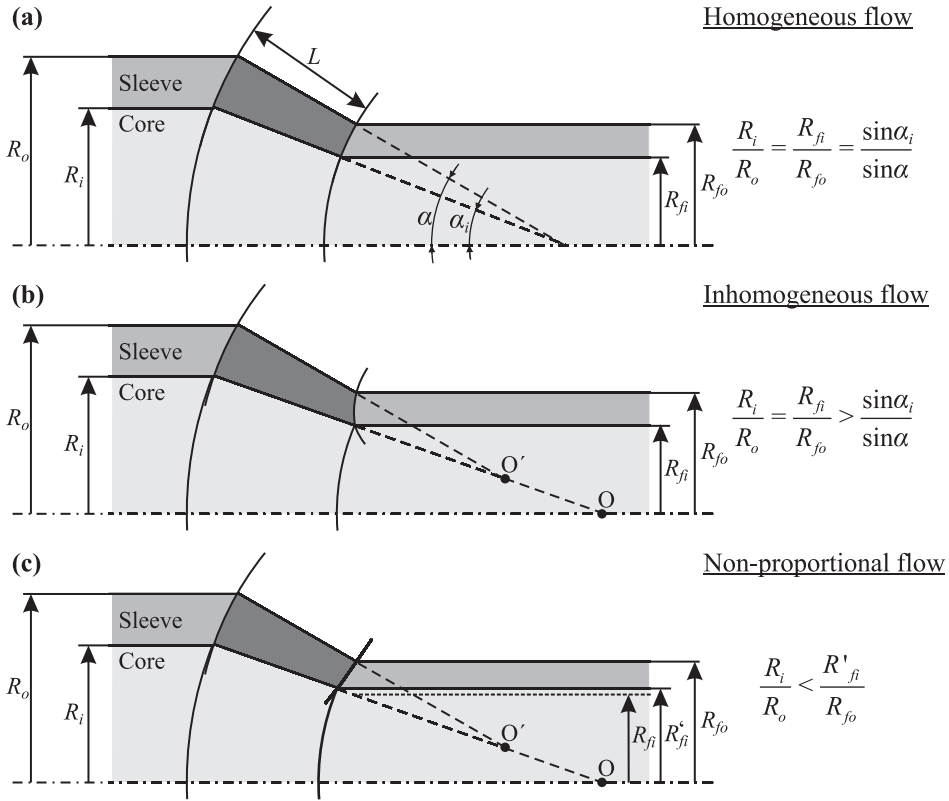
Critical in each deformation method is the hardness evolution of the matrix material, which in the worst case leads to fracture before sufficient powder density has been reached. Conductor processing is further controlled by various details of the deformation process and matrix layout, especially in the case of the Cu/Fe combination. Which parameters are important for “successful” deformation is explored in §2.3.1.

In order to test the powder densification, both the evolution of the filament density and the  $J_c$  of the as-deformed conductors are monitored. The results are discussed in §2.3.2.

### 2.3.1 Parameters Controlling Deformation Processes

#### Drawing

To explore the parameter space for the drawing process, a model developed by Avitzur *et. al.* [75] for the deformation of bi-metal rods is applied (see figure



**Figure 2.8:** Flow modes during co-drawing two different metals. (a) shows homogeneous flow; (b) inhomogeneous flow; and (c) non-proportional flow.

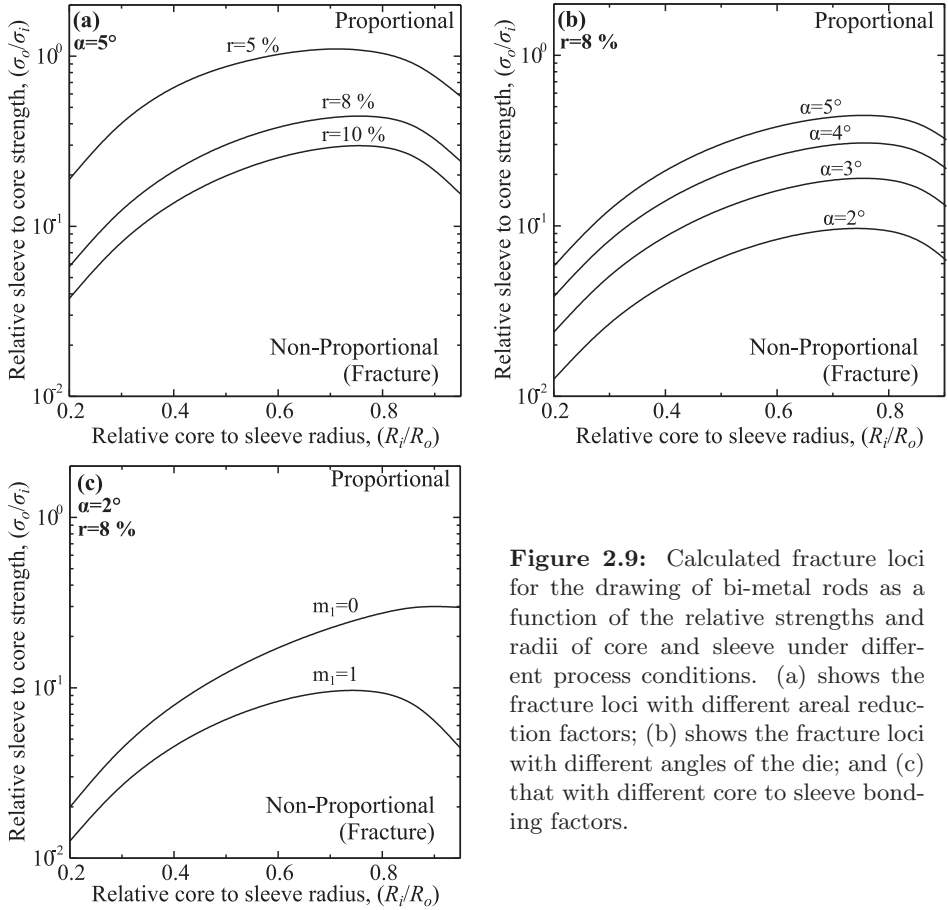
2.7b)<sup>4</sup>. The model is used as a simplified representation of PIT conductors with a matrix consisting of two metals with different hardness, such as Cu/Fe, and does not include the powder core (figure 2.7a). It predicts the existence and location of a boundary between fracture and successful drawing of a bi-metal rod and is a function of various drawing parameters.

During drawing, a bi-metal rod can follow three types of flow patterns. When the two metals have comparable hardness, the rod flows homogeneously (figure 2.8a). In this case the radii of the sleeve and core have the same relative reduction so that:

$$\frac{R_i}{R_o} = \frac{R_{fi}}{R_{fo}}, \quad (2.2)$$

with  $R_i$  and  $R_o$  the outer radii of the core and sleeve before deformation and  $R_{fi}$

<sup>4</sup>Only the essence of Avitzur's model is presented here. For a full discussion, the reader is referred to [75–77].



**Figure 2.9:** Calculated fracture loci for the drawing of bi-metal rods as a function of the relative strengths and radii of core and sleeve under different process conditions. (a) shows the fracture loci with different areal reduction factors; (b) shows the fracture loci with different angles of the die; and (c) that with different core to sleeve bonding factors.

and  $R_{fo}$  after. Furthermore, the velocity field of the sleeve and core point towards the same origin  $O$  so that the angle of deformation of the core  $\alpha_i$  is related to the semicone angle of the die  $\alpha$  as:

$$\frac{\sin(\alpha_i)}{\sin(\alpha)} = \frac{R_i}{R_o}. \quad (2.3)$$

When the core is harder than the sleeve, it tends to deform less (figure 2.8b). As a result the angles  $\alpha_i$  and  $\alpha$  are no longer related anymore by (2.3), but rather:

$$\frac{\sin(\alpha_i)}{\sin(\alpha)} < \frac{R_i}{R_o}. \quad (2.4)$$

The velocity field of the core and sleeve point to different origins. This type of flow is referred to as inhomogeneous flow, but it can still be proportional so that (2.2) still holds. However, depending on the drawing parameters, inhomogeneous

flow can also lead to relative thinning of the sleeve and thus non-proportional flow:

$$\frac{R_i}{R_o} < \frac{R_{fi}}{R_{fo}}. \quad (2.5)$$

If powder densification inside the core can be neglected, the volume remains constant and (2.5) implies that the sleeve elongates more than the core, so that the latter undergoes a tensile force. This is when fracture of the core may occur.

Whether the flow is non-proportional for a given combination of process parameters is determined by searching the  $R_{fi}/R_{fo}$  ratio that corresponds to the minimum power required for drawing. The process parameters that can be investigated in the model are: the area reduction  $r = 1 - (R_{fo}/R_o)^2$ ; the semicone angle of the die  $\alpha$ ; the length of the bearing of the die  $L$ ; the friction between the outer surface of the sleeve and the bearing of the die; the bonding strength between core and sleeve  $m_1$ ; the relative core to sleeve radius ( $R_i/R_o$ ); the relative yield moduli of sleeve and core ( $\sigma_o/\sigma_i$ ); and the outgoing velocity of the rod.

As an example, calculated fracture loci are plotted in figure 2.9 as a function of ( $R_i/R_o$ ) and ( $\sigma_o/\sigma_i$ ). In all cases, the harder the core ( $\sigma_o/\sigma_i < 1$ ), the more likely it is to fracture. This is especially true for intermediate cores sizes ( $R_i/R_o \approx 0.5$ ). As the relative core size increases ( $R_i/R_o \rightarrow 1$ ), the flow may still be inhomogeneous, but will tend towards proportional so that the critical ( $\sigma_o/\sigma_i$ ) value decreases.

Figure 2.9a shows how the fracture locus shifts with increasing area reduction. At a constant semicone angle  $\alpha$ , an increase in  $r$  implies a longer die bearing  $L$ , which promotes homogeneous flow. Vice versa, increasing the semicone angle at constant area reduction results in a shorter  $L$  so that fracture is promoted (figure 2.9b). Furthermore, 2.9c shows that better bonding of the sleeve and core ( $m_1 = 1$ ) also promotes homogeneous flow.

Despite the strong simplification made by neglecting the specific properties of the powder core, it will be shown that this model does allow a qualitatively correct choice of process parameters such as the ratio of core to sleeve radius or the relative sleeve to core strength.

### *Comparison with Experiments*

The wires that were drawn are listed in table 2.1. Five different matrix layouts are used, while the powders are either pure  $\text{MgB}_2$  or contained 10 vol. % Pb. Drawing was done with the facilities at Shape Metal Innovation and at the University of Geneva. Area reduction per pass is typically 10 %, while the semicone angle of the dies is typically  $\sim 5$  %. The listed final radius  $R_f$  is determined as the radius at which further drawing is impossible due to repeated breaking of the wire. The effective true strain  $\epsilon_f$  at the final radius is also listed so that samples



**Table 2.1:** Overview of drawn conductors, together with their final dimensions.

Wire #	Matrix	Pb [vol. %]	Initial				Final	
			$R_o$ [mm]	$R_i$ [mm]	$R_{fil}$ [mm]	$(R_i/R_o)$	$R_{fo}$ [mm]	$\epsilon_f$
1-D	Cu/Fe	-	6.00	4.00	2.50	0.67	1.7	2.6
1-Pb10-D	Cu/Fe	10	6.00	4.00	2.50	0.67	1.7	2.6
2-D	Cu/Fe	-	5.00	4.00	2.50	0.80	1.5	2.5
2-Pb10-D	Cu/Fe	10	5.00	4.00	2.50	0.80	1.3	2.8
3-D	Cu/Fe	-	5.50	4.75	4.25	0.86	0.8	3.8
3-D-II	Cu/Fe	-	5.50	4.75	4.25	0.86	0.6	4.4
3-Pb10-D	Cu/Fe	10	5.50	4.75	4.25	0.86	0.7	4.2
4-D	Cu	-	5.50	-	4.75	-	1.8	2.3
4-Pb10-D	Cu	10	5.50	-	4.75	-	1.9	2.2
5-D	Fe	-	-	4.00	2.50	-	0.6	3.9

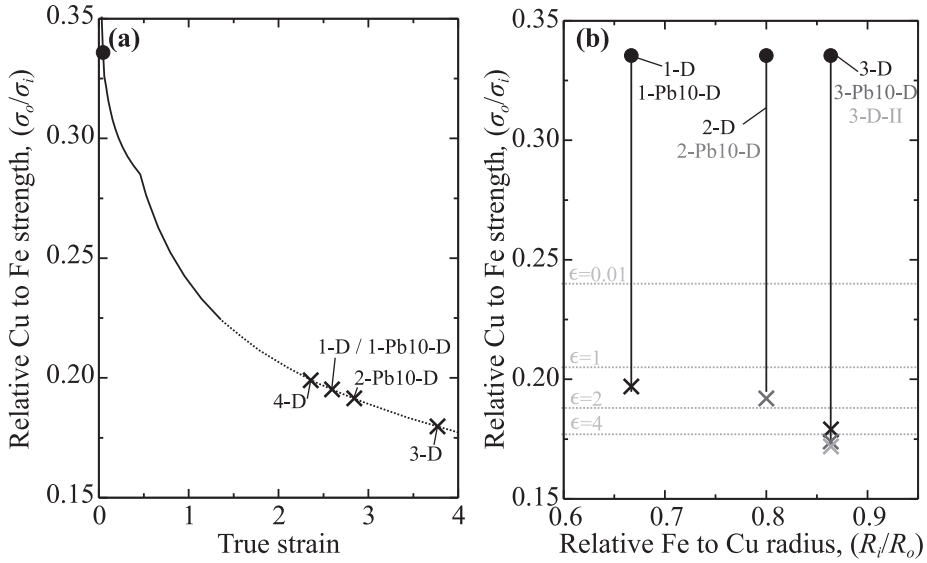
with different initial radii can be compared. It is defined as:

$$\epsilon_f = 2 \ln \left( \frac{R_o}{R_{fo}} \right). \quad (2.6)$$

It can readily be seen that wires with different core to sleeve ratios indeed tend to arrive at different true strain values, as predicted by the model discussed above. It should be noted that Fe work-hardens faster than Cu [78,79], so that the relative yield modulus ( $\sigma_o/\sigma_i$ ) changes from one drawing step to the next. The drawing process starts above the fracture locus shown in figure 2.9 but ( $\sigma_o/\sigma_i$ ) will steadily decrease so that fracture can be expected at some point.

The relative Cu to Fe strain hardening curve is plotted in figure 2.10a. The curve is calculated as the ratio of true stress to true strain data of annealed Cu to those of ARMCO Fe [78,79]. Note that this curve is a rough approximation of the actual relative work-hardening of the Cu/Fe matrices, as the actual yield strengths of the wires are not measured. However, for all conductors similar annealed OFHC Cu is used and the initial value for  $\sigma_o$  is assumed to be 60 MPa [78,80]. For samples 1-D, 1-Pb10-D, 2-D and 2-Pb10-D as-annealed 99.5 % pure Fe tubes are used, while for samples 3-D, 3-D-II and 3-Pb10-D as-drawn ARMCO Fe tubes were annealed at 550 °C for 2 hr. For  $\sigma_i$  the starting value 180 MPa of annealed ARMCO Fe is used. Thus, drawing starts at ( $\sigma_i/\sigma_o \approx 0.33$ ), as indicated by the dot in figure 2.10a.

Converting the curve of 2.10a to a plot of ( $\sigma_i/\sigma_o$ ) versus ( $R_i/R_o$ ), results in figure 2.10b. The dots again indicate the estimated starting point of each

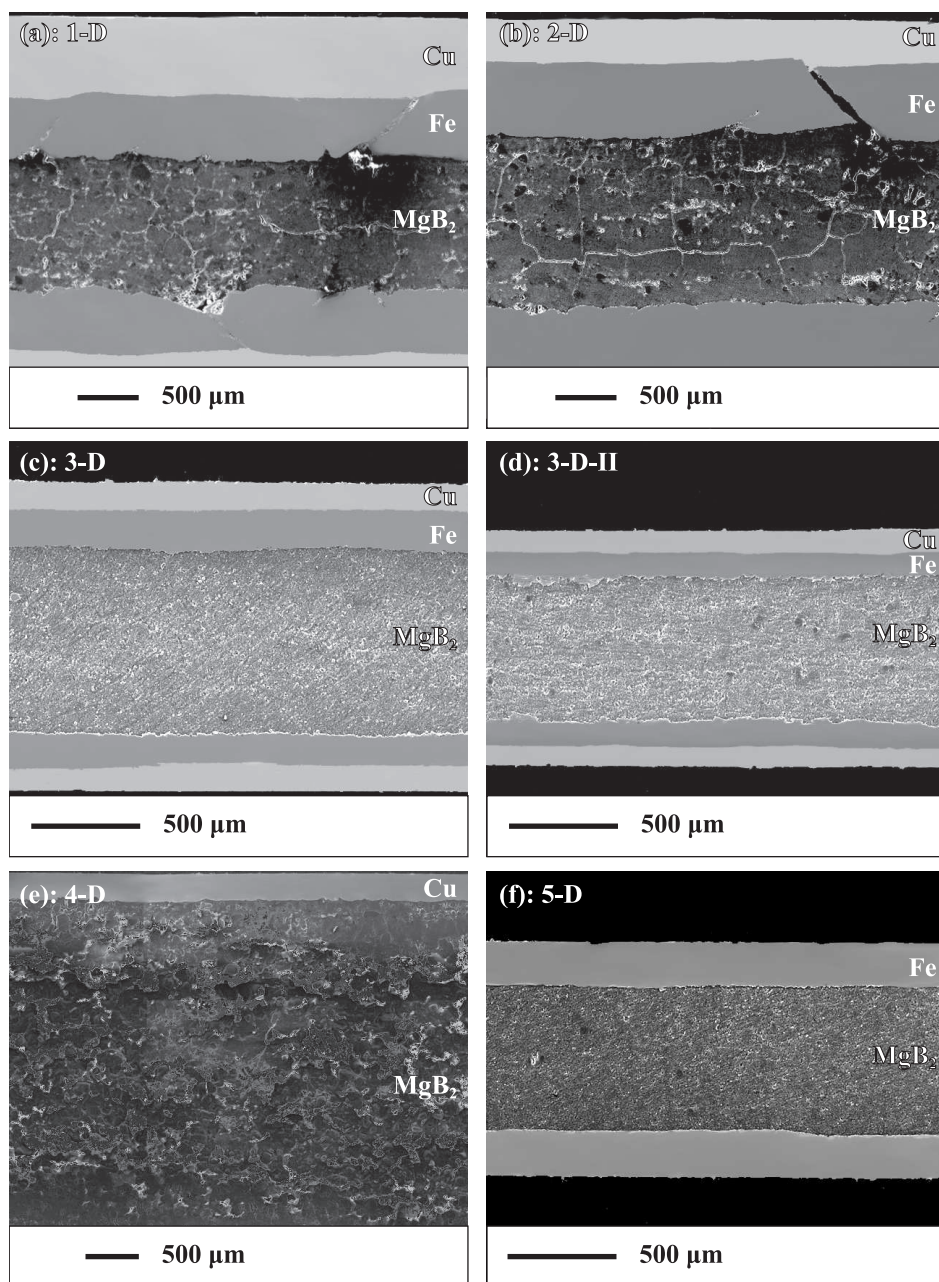


**Figure 2.10:** (a) shows the relative Cu to Fe strength ratio plotted against true strain (calculated from [78, 79]). The dot indicates the estimated relative strength of the starting tubes, the crosses the final true strain of selected wires. (b) shows the same data, but this time plotted in a similar way as the model predictions of figure 2.9.

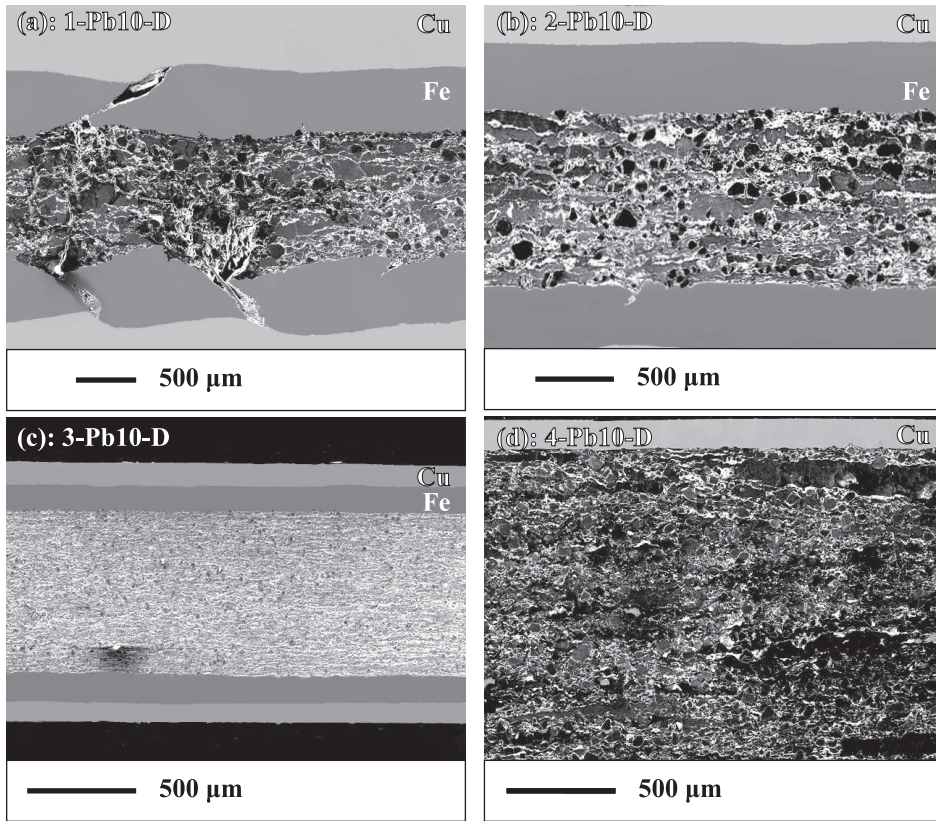
conductor, while the crosses indicate where the wires broke repeatedly. Just as predicted by the model, the wires with relatively high ( $R_i/R_o$ ) ratio can be drawn further, corresponding to a lower critical ( $\sigma_i/\sigma_o$ ) value. It should be noted that no quantitative conclusions can be drawn from figure 2.10b as the actual relative work hardening and initial ( $\sigma_i/\sigma_o$ ) of the Cu/Fe matrices were not measured.

Figure 2.11 shows the SEM micrographs of the longitudinal cross-section of the as-drawn samples with pure  $MgB_2$  at their final diameter. Samples 1-D and 2-D with the lower ( $R_i/R_o$ ) values clearly show how Fe fractures inside the Cu sleeve. The  $45^\circ$  fracture angle indicates shear stress to be the main cause of fracture [81]. In contrast, sample 5-D has the same Fe matrix but no Cu outer sleeve and could be drawn to a  $\sim 70\%$  higher strain level. The final true strain level of this wire was not determined by fracture, but simply by the lack of smaller dies. Together, these observations illustrate that Fe in the composite tubes fractures due to non-proportional flow, in which the Cu sleeve exerts a tensile force on the Fe core.

Also in samples 3-D and 3-D-II this can be the mechanism of failure, even if no internal fracture planes are observed in the longitudinal cross-sections. Samples 1-D and 2-D have a  $\sim 30 - 65\%$  larger Cu thickness so that the Cu sleeve is still able to support the drawing force by itself after the Fe has fractured. For samples



**Figure 2.11:** SEM micrographs of the longitudinal cross-section of as-drawn wires, all with pure  $\text{MgB}_2$  cores but different matrix layout at their final dimensions.

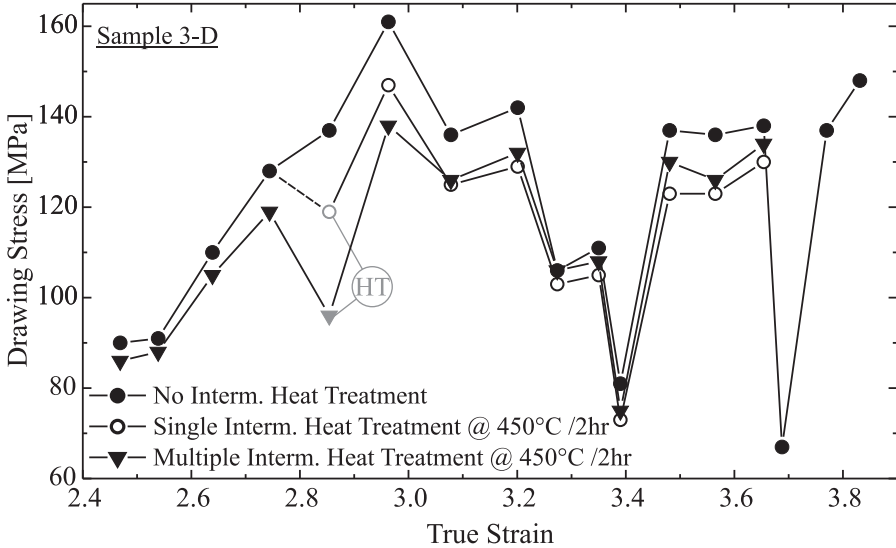


**Figure 2.12:** SEM micrographs of the longitudinal cross-section of as-drawn wires, all with 10 vol.% Pb-containing powder cores but with different matrix layout at their final dimension. The bright regions in the powder core correspond to Pb-rich areas.

3-D, 3-D-II, the Cu is likely to be too thin so that the whole wire breaks when the Fe first fails. Note also that sample 3-D-II could be drawn to a 16 % higher  $\epsilon_f$  level than sample 3-D, due to the use of different dies.

For sample 4-D, with just a Cu matrix, only a relatively small true strain of 2.3 could be reached. Also in this sample the Cu is relatively thin and unable to support the drawing force.

The longitudinal cross-sections in figure 2.12 are those of samples that contain 10 vol.% Pb. Also here drawing failure is caused by fracture of the Fe, which is clearly visible in figures 2.12a and b. Between samples 1-D and 1-Pb10-D, no difference in the maximum achievable true strain  $\epsilon_f$  is observed, indicating that the relative strain hardening of Cu and Fe dominates the deformation behaviour. On the other hand, samples 2-Pb10-D and 3-Pb10-D could be drawn to  $\sim 12$  %



**Figure 2.13:** Influence of intermediate softening heat treatment on the drawing force of sample 3-D.

higher  $\epsilon_f$  levels than their Pb-free counterparts. For higher ( $R_i/R_o$ ) values, Pb additions thus indeed seem to promote proportional flow. For sample 4-Pb10-D, no difference in  $\epsilon_f$  is observed compared to 4-D. Just like with 1-Pb-10, the properties of the matrix (the ultimate strength of Cu) determine its overall behaviour irrespective of the powder characteristics.

The drawing experiments show that the maximum achievable true strain  $\epsilon_f$  is mainly limited by the level of relative Cu to Fe strain hardening. In an attempt to increase  $\epsilon_f$ , pieces cut from sample 3-D were subjected to several softening heat treatments in-between the drawing steps. Figure 2.13 shows the effect of the intermediate heat treatments on the overall drawing stress.

The wire subjected to a single intermediate heat treatment was cut from the untreated sample 3-D at a true strain value of  $\sim 2.75$  and heat treated at  $450^\circ\text{C}/2$  hr. Another piece was subjected to multiple heat treatments at  $450^\circ\text{C}/2$  hr each time  $\epsilon$  increased by 1. In figure 2.13, the indicated heat treatment of this piece is the last one, since no force measurements could be performed at diameters  $d$  larger than 3.5 mm. Heat treatments at  $550^\circ\text{C}/2$  hr were also tested, with similar results.

The intermediate heat treatment clearly affects the drawing stress, reducing its value by  $\sim 14\%$  for the single- and  $\sim 28\%$  for the multiple heat treated pieces compared to the untreated sample. Although the matrix materials are indeed softened by the heat treatments, the effect disappears rapidly. In fact, the drawing stress coincides again with that of the untreated sample after only 4



**Table 2.2:** Overview of Two-Axially-Rolled and Flat-Rolled conductors.

Cond #	Matrix	Pb [vol.%]	$A_o^\dagger$ [mm <sup>2</sup> ]	$A_f$ [mm <sup>2</sup> ]	$f$ [%]	$\epsilon_f$
Two-Axial-Rolling:						
3-T	Cu/Fe	-	$\pi \times 5.50^2$	1.40 <sup>2</sup>	49	3.9
3-T4	CuNi/Cu/Fe	-	5.20 <sup>2‡</sup>	1.10 <sup>2</sup>	22	7.0 <sup>¶</sup>
3-Pb10-T	Cu/Fe	10	$\pi \times 5.50^2$	1.40 <sup>2</sup>	51	3.9
3-Pb10-T16	CuNi/Cu/Fe	10	7.96 <sup>2‡</sup>	2.45 <sup>2</sup>	-	6.2 <sup>¶</sup>
Flat-Rolling:						
5-FR	Fe	-	$\pi \times 4.00^2$	4.11 $\times$ 0.38	34	3.5
5-Pb05-FR	Fe	5	$\pi \times 4.00^2$	4.01 $\times$ 0.38	33	3.5
5-Pb10-FR	Fe	10	$\pi \times 4.00^2$	4.08 $\times$ 0.38	32	3.5
5-Pb15-FR	Fe	15	$\pi \times 4.00^2$	4.02 $\times$ 0.37	30	3.5

<sup>†</sup> Initial dimensions of samples 3-T and 3-Pb10-T are the same as those of samples 3-D and 3-Pb10-D in table 2.1, while the initial dimensions of samples 5-FR to 5-Pb15-FR are identical to that of sample 5-D.

<sup>‡</sup> Initial cross-sectional area of the assembly of mono-cores and CuNi outer tube.

<sup>¶</sup> Cumulative true strain build up from the deformation of the mono-core and that of the stacked assembly.

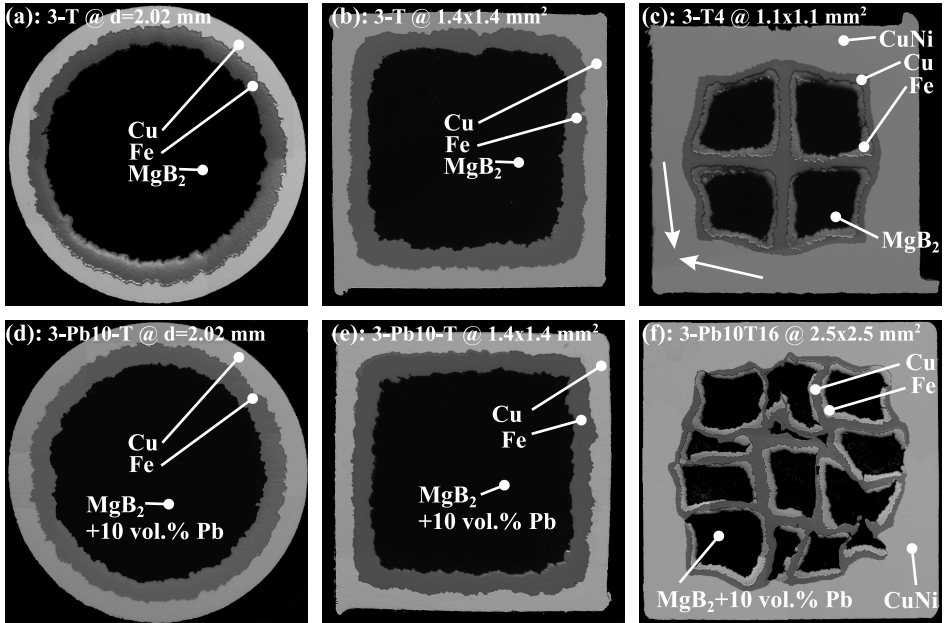
drawing steps, when the true strain is increased by  $\sim 0.4$ . As a consequence, the heat treated pieces could not be drawn any further than the untreated samples. Clearly, to reach higher true strain levels more intermediate heat treatments would be necessary. Furthermore, as shown below the powder density reduces after heat treatment, supposedly due to the altered force balance between the powder core and the softened matrix.

### Two-Axial Rolling and Flat Rolling

The samples fabricated using two-axial and flat rolling are listed in table 2.2. Two-axial rolling was performed at the Institute of Electrical Engineering, Bratislava, while tapes were flat-rolled at the University of Geneva.

For samples 3-T and 3-Pb10-T the same Cu and Fe tubes are used as for wires 3-D, 3-D-II and 3-Pb10-D (table 2.1). The samples were drawn to a diameter of 2.02 mm and then two-axially rolled to  $1.4 \times 1.4$  mm<sup>2</sup> with an area reduction per pass of 5 to 10%. Attempts to roll further led to splitting of the matrix, i.e. longitudinal cracks appearing at the surface of the samples.

From the two mono-core square wires, the two multifilamentary conductors 3-T4 and 3-Pb10-T16 were fabricated. Pieces of samples 3-T and 3-Pb10-T were

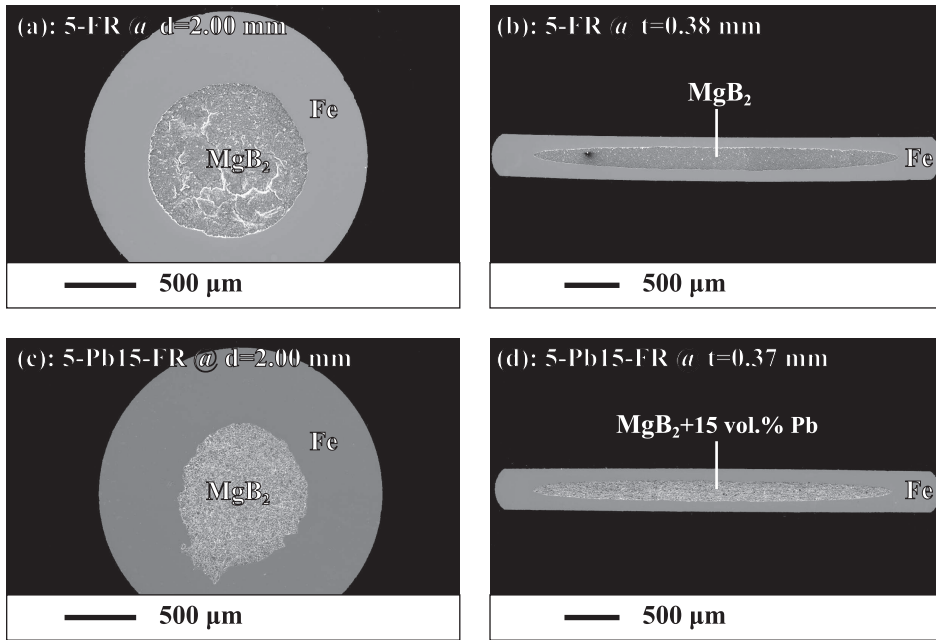


**Figure 2.14:** Optical micrographs of the two-axially rolled samples in different deformation stages. (a) and (d) show samples 3-D and 3-Pb10-D, drawn to  $d = 2.02$  mm; (b) and (e) show the mono-core two-axially rolled samples 3-T and 3-Pb10-T; (c) and (f) show the multifilament two-axially-rolled samples. The arrows in (c) indicate the flow of the matrix material.

stacked into a Cu-Ni outer tube in a  $2 \times 2$  and a  $4 \times 4$  arrangement. As discussed above, Pb-containing wires can be drawn to higher true strain levels than samples without Pb. Therefore, the amount of filaments for sample 3-T4 was limited to 4, while sample 3-Pb10-T16 contained 16 filaments. The multifilamentary wires were rolled to  $1.1 \times 1.1$  mm<sup>2</sup> and  $2.5 \times 2.5$  mm<sup>2</sup>, respectively. As with the square mono-core wires, their final dimensions were limited by splitting of the surface.

Figure 2.14 shows optical micrographs of the cross-section of the two-axially rolled wires at different deformation stages. To ensure successful sintering the Fe matrix should remain thick enough ( $\sim 30\mu\text{m}$ ) to prevent reaction between Cu and MgB<sub>2</sub> [82]. However, in both mono-core and multifilamentary conductors the filaments tend to take a circular shape due to the flow of matrix material towards the corners [83], as indicated by the arrows in figure 2.14c. In the case of sample 3-T4 the non-uniform matrix deformation leads to a Fe reaction barrier that is too thin at some places (down to  $12\mu\text{m}$ ). As discussed below, this leads to a strong reaction between MgB<sub>2</sub> and Cu during the final heat treatment.

In the 16-filaments conductor, on the other hand, figure 2.14f shows that the individual filaments are inhomogeneously deformed: of the 16 filaments only



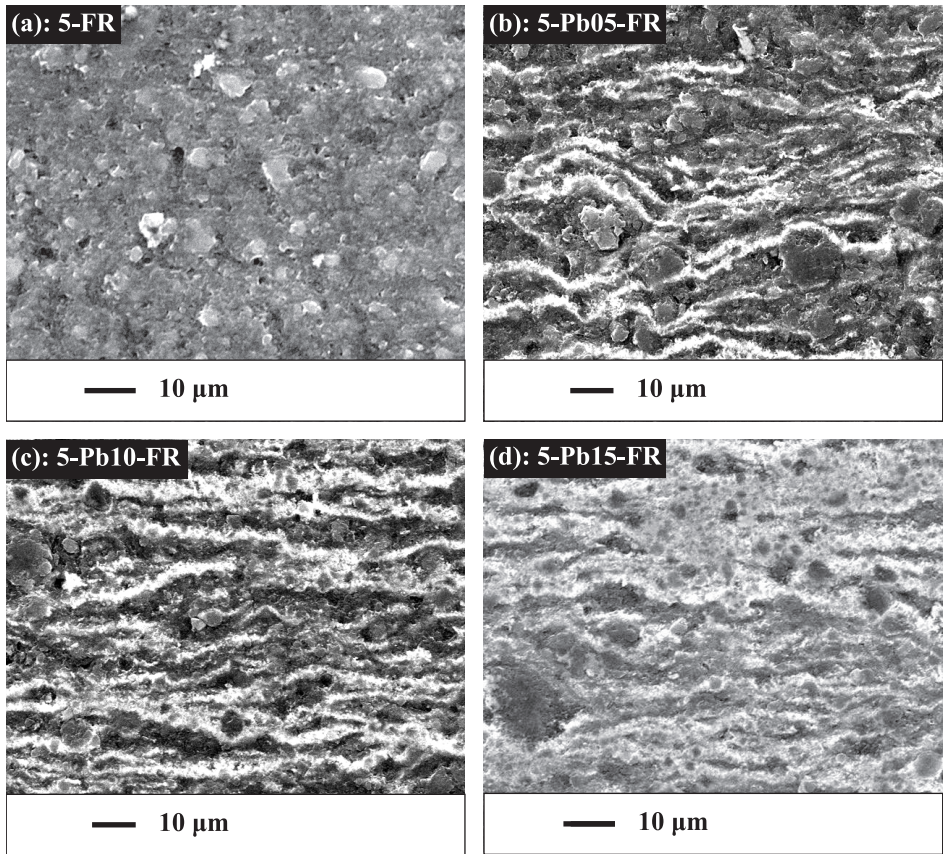
**Figure 2.15:** SEM micrographs of the flat-rolled samples without Pb ((a) and (b)) and with 15 vol.% Pb (c) and d)). The bright regions in images (c) and (d) indicate Pb-rich areas.

13 can be observed in this particular section. Also this is caused by the flow of matrix material towards the corners, in combination with the relatively thin Cu/Fe matrix in-between the filaments.

Cross-sections of the flat-rolled samples 5-D and 5-Pb15-D at different deformation stages are shown in figure 2.15. The Fe tubes used for these samples are the same as in sample 5-D. Similar to wire 5-D, the flat-rolled conductors are first swaged from a diameter of 8 mm down to 3.85 mm and then further drawn to 2 mm. At this point, the conductors were flat-rolled to their final thickness of  $\sim 0.38$  mm. To account for the work hardening of Fe the area reduction per pass was gradually reduced from 7 % at the first deformation steps down to 1 % for the last.

Figure 2.16 shows longitudinal cross-sections of the 4 samples with different Pb concentrations. In the SEM images, Pb shows up as a bright minority phase inside the grey  $\text{MgB}_2$  filament. At lower concentrations it is clearly visible how Pb is distributed as stripes in the rolling direction of the tapes. The density of these stripes increases with the Pb concentration. This indicates that the softer Pb indeed flows in-between the harder  $\text{MgB}_2$  particles and acts as a lubricant during the deformation. Furthermore, table 2.2 shows a clear trend towards lower





**Figure 2.16:** SEM micrographs of longitudinal cross-sections of the as-rolled flat tapes with 0, 5, 10 and 15 vol.% Pb. The rolling direction is horizontal.

filling factors for higher Pb concentrations. Since the initial filling factor  $\rho_{\text{fil},0}$  was 39 % for all tapes, this is an indication for the higher powder densification that is obtained. In the next paragraph, the powder densification that can be obtained with different deformation routes is further discussed.

### 2.3.2 Powder Densification

#### Normalised density versus True Strain

The different deformation procedures, matrix materials and Pb concentrations lead to a different filament density of the final conductor and thus to different critical current densities. To keep track of the powder densification during deformation, the length and cross-sectional area change at each deformation pass is

recorded. The critical current density of green samples in different deformation stages is also measured, which will be discussed in the next paragraph<sup>5</sup>.

Since the volume of the matrix remains constant, the filament density  $\rho_{\text{fil}}$  at any stage of deformation can be estimated as:

$$\frac{\rho_{\text{fil}}}{\rho_{\text{fil},0}} = \frac{f_0 (l_0/l)}{(A/A_0) - (1 - f_0) (l_0/l)}, \quad (2.7)$$

with  $\rho_{\text{fil},0}$  the initial powder density after filling the tube;  $l_0$  the initial length;  $A_0$  the initial overall cross-section; and  $f_0$  the initial filling factor. The overall cross-sectional area  $A$  and the length  $l$  at intermediate stages of deformation are simply measured.

The density evolution is presented as a percentage of the theoretical filament density ( $\rho_{\text{fil}}/\rho_{\text{fil,th}}$ ) versus the true strain  $\epsilon$ . The theoretical filament density  $\rho_{\text{fil,th}}$  of the samples with Pb is determined as:

$$\rho_{\text{fil,th}} = (1 - f_{\text{Pb}}) \rho_{\text{MgB}_2} + f_{\text{Pb}} \rho_{\text{Pb}}, \quad (2.8)$$

with  $f_{\text{Pb}}$  the volume fraction of Pb in the powder and  $\rho_{\text{MgB}_2}$  or  $\rho_{\text{Pb}}$  the densities of MgB<sub>2</sub> or Pb ( $\rho_{\text{MgB}_2} = 2.62 \text{ g/cm}^3$  and  $\rho_{\text{Pb}} = 11.3 \text{ g/cm}^3$ ). The true strain is determined from the reduction of cross-sectional area:

$$\epsilon = \ln \left( \frac{A_0}{A} \right). \quad (2.9)$$

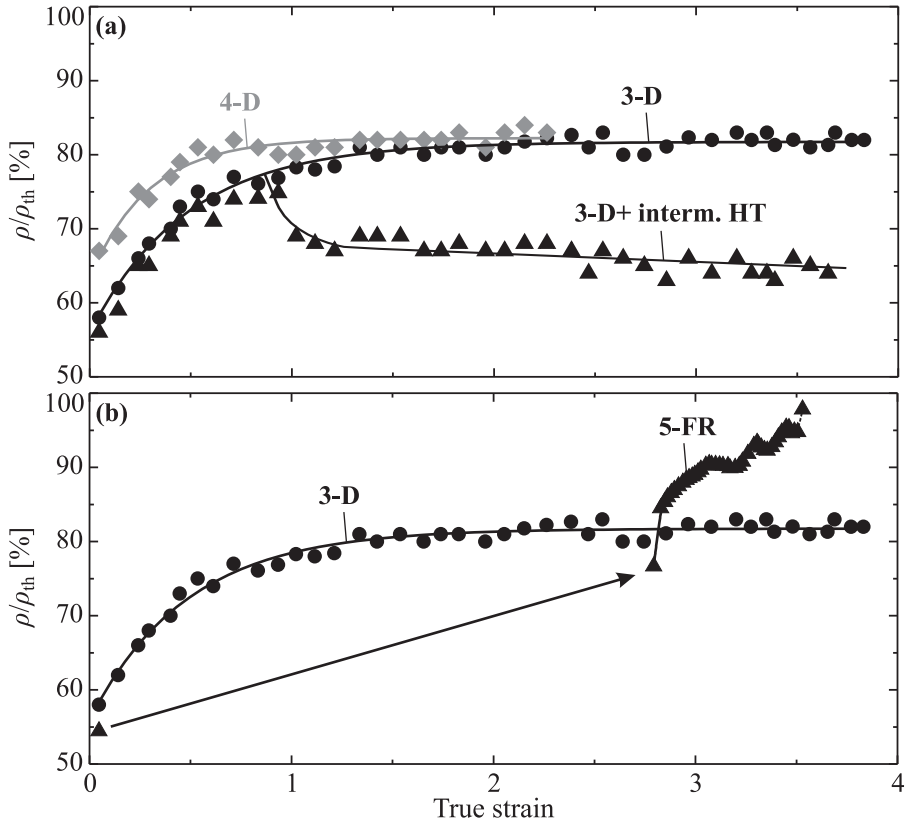
Figure 2.17a shows how the density evolves during the drawing of wires 3-D and 4-D with Cu/Fe and Cu matrix. For both wires, the density saturates at approximately 82 % of its maximum theoretical value, even though sample 4-D has a higher initial density than 3-D. The plot also indicates the effect of the softening heat treatment on the evolution of the filament density of wire 3-D. As discussed above, heat treatments were applied at 450°C for 2 hours at true strain values of 1, 2 and 3. Already after the first heat treatment at  $\epsilon = 1$ , the density decreases by about 10 %. This clearly shows the strong relation between matrix hardness and powder densification.

Figure 2.17b shows how densification during drawing and flat rolling differ by comparing sample 3-D and 5-FR. It should be noted that during initial swaging and drawing of sample 5-FR, no length change was recorded. The first point of the density evolution during rolling is just after the last drawing step at a diameter of 2 mm ( $\epsilon = 2.8$ ). The filling factor  $f_0$  at this point is determined from the cross-sectional SEM image in figure 2.15a, while the initial powder density  $\rho_{\text{fil},0}$  is determined from the weight and volume of a piece cut of the wire.

Up to  $\epsilon = 2.8$  sample 5-FR has gained a similar density as sample 3-D, despite its harder and thicker Fe matrix and the fact that it is also swaged (up to  $\epsilon = 1.5$ ;

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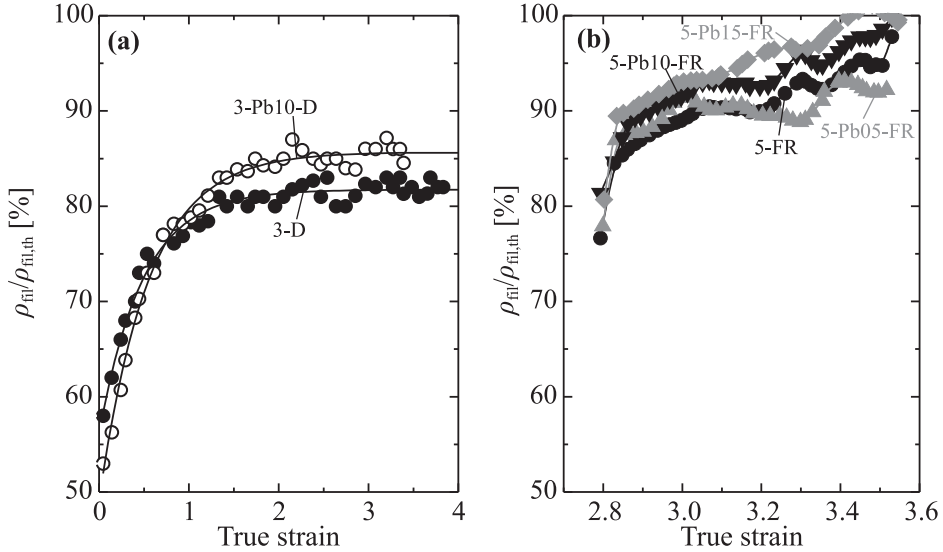
<sup>5</sup>Note that for the two-axially rolled samples the densification was not recorded. Furthermore, these samples were prepared from milled powder with a poor  $J_c$ . Therefore, densification of the two-axially rolled samples is not discussed.



**Figure 2.17:** (a) shows the densification during drawing of samples 3-D and 4-D. Also shown is the effect of the softening heat treatment on the densification of sample 3-D. (b) shows the densification during the drawing of sample 3-D and the rolling of sample 5-FR. The **solid lines** are a guide to the eye.

$d = 3.85$  mm). However, already during the first rolling step the density increases by  $\sim 10$  % and then continues to increase with true strain, while with drawing it saturates. The enhanced densification during flat rolling may result from the absence of a pulling force which counteracts the densification.

The difference in densification during drawing of samples with and without Pb is shown in figure 2.18a. In both samples 3-D and 3-Pb10 the density saturates. However, the maximum level is  $\sim 86$  % in 3-Pb10-D, which is 4 % higher than in 3-D. Also for the flat-rolled tapes in figure 2.18b, a trend towards higher density with increasing Pb concentration can be observed. This suggests that the soft Pb fills the voids in-between the hard  $MgB_2$  grains.



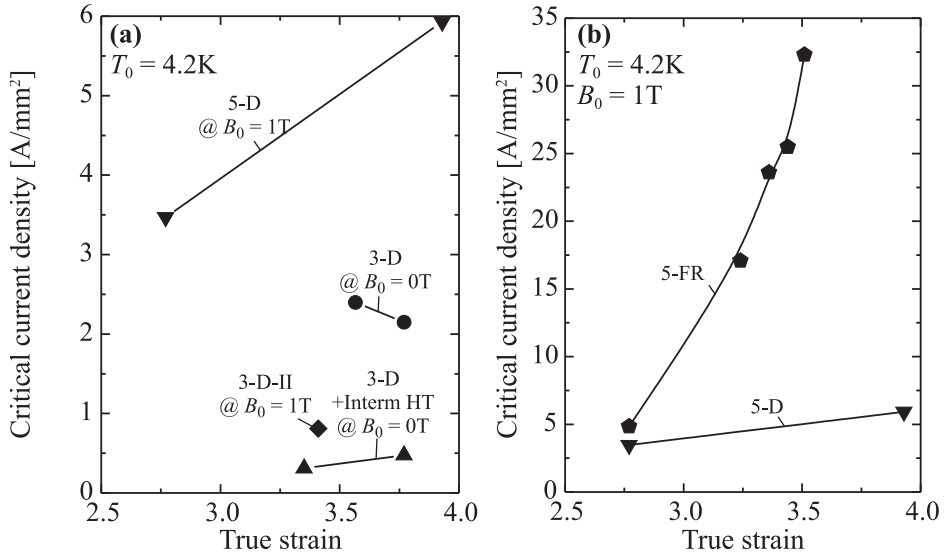
**Figure 2.18:** Influence of Pb additions on the densification. (a) shows the densification of the drawn wires 3-D and 3-Pb10-D and (b) of the flat rolled tapes 5-FR to 5-Pb15-FR. The solid lines are a guide to the eye.

### Critical current versus True Strain

To keep track of the evolution of  $J_c$  during drawing and flat-rolling, pieces of the conductors were cut at several stages of deformation.  $J_c$  of the as-deformed samples was measured in a magnetic field parallel to the tape surface and at  $T_0 = 4.2$  K. The voltage taps were placed  $\sim 1$  cm apart and an electric field criterium of  $100 \mu\text{V}/\text{m}$  was used.

In figure 2.19a the drawn samples with either Fe or Cu/Fe matrix are compared. Samples 5-D and 3-D-II were subjected to the same swaging/drawing deformation and are both measured at  $B = 1$  T. The harder and thicker Fe matrix of sample 5-D causes a higher densification and higher  $J_c$  than the relatively thin Cu/Fe matrix of sample 3-D-II. Similarly, the piece of sample 3-D that was subjected to intermediate heat treatments has a significantly lower  $J_c$  value than the one without. Comparison with figure 2.17 shows that this is likely due to the reduced filament density after heat treatment.

Figure 2.19b shows how the  $J_c$  value of samples 5-D and 5-FR evolves during deformation. Both conductors were subjected initially to the same swaging and drawing deformation up to  $d = 2$  mm ( $\epsilon = 2.77$ ). After that point, sample 5-D was further drawn, while sample 5-FR was flat-rolled. As expected, both samples have a similar  $J_c$  value at  $\epsilon = 2.77$ . However, further flat-rolling clearly increases  $J_c$  to a larger extent than continued drawing. This is in agreement with the



**Figure 2.19:** Evolution of the critical current density as function of true strain of as-deformed samples. (a) samples with different matrix materials and varying hardness; (b) samples that are either drawn or flat-rolled.

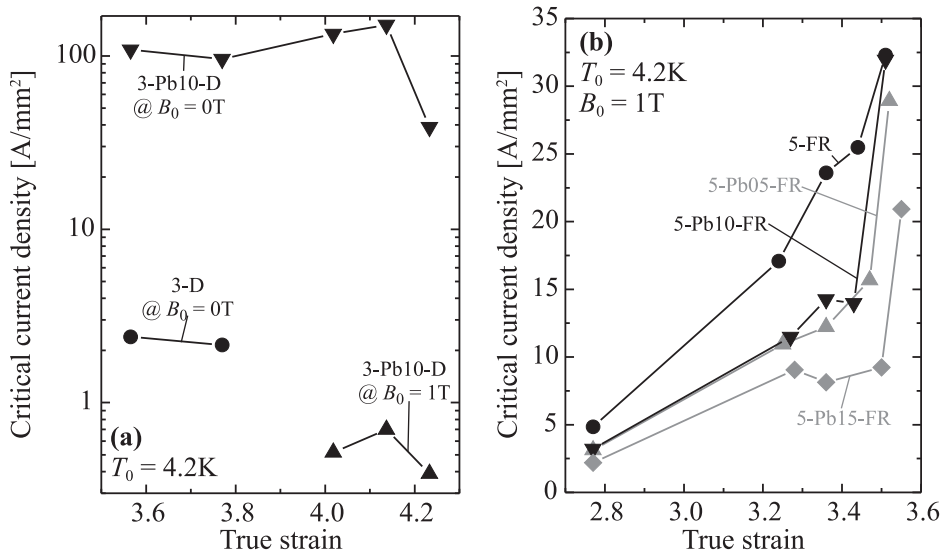
difference in the density evolution during the two processes as shown in figure 2.17b.

The influence of Pb on the evolution of the  $J_c$  value in as-deformed samples is shown in figure 2.20. In figure 2.20a it is shown that sample 3-Pb10-D has a  $J_c$  value at  $B = 0$  T that is nearly two orders of magnitude larger than that of sample 3-D. At  $B = 1$  T, however, its  $J_c$  has decreased significantly. This shows that at  $B = 0$  T, most of the current is carried by the Pb, which is superconducting at this field. At  $B = 1$  T, Pb is not superconducting anymore and only the MgB<sub>2</sub> grains carry a supercurrent. Similarly, it is shown in figure 2.20b that at  $B = 1$  T, the flat-rolled samples with higher Pb concentrations tend to have lower critical current densities. These two observations lead to the conclusion that Pb in the normal state hampers the MgB<sub>2</sub> grain-to-grain connectivity and thus lowers  $J_c$ .

Similar to the samples without Pb, the  $J_c$  value of the Pb-containing wires is approximately constant during drawing, but increases with progressive flat-rolling.

## 2.4 Final Heat Treatment

It is generally observed that the final heat treatment of as-deformed ex-situ conductors causes an increase in  $J_c$  by approximately one order of magnitude [64]. In



**Figure 2.20:** Evolution of the critical current density as function of true strain of as-deformed samples with and without Pb. (a) drawn samples (note the logarithmic scale of the critical current density); (b) flat rolled samples.

this paragraph, the interplay between microstructure and critical current behind this improvement is discussed.

In §2.4.1 the influence of the heat treatment on the microstructure is presented. As mentioned above, different deformation routes result in different final filament densities and thus the microstructure might evolve differently during heat treatment. Possible chemical reactions of the  $MgB_2$  with matrix materials are also discussed. Furthermore, it is shown how the heat treatment influences the microstructure of filaments in Pb-containing samples.

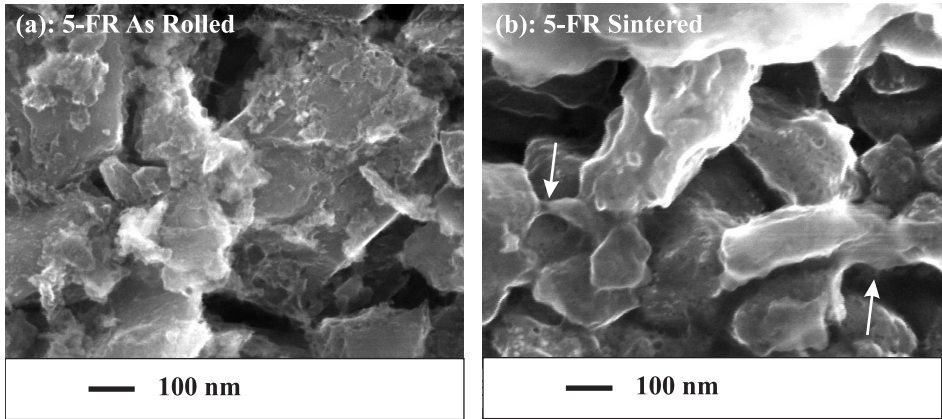
Then, in §2.4.2 the influence of the heat treatment on the critical current densities is discussed for differently deformed samples with or without Pb.

All final heat treatments discussed in this paragraph were at  $950^\circ C$  for 0.5 hr in vacuum. The Pb-containing samples were vacuum-sealed in a quartz tube together with a lump of metallic Pb to prevent excessive Pb evaporation out of the filaments.

## 2.4.1 Microstructure

### Sintering

Figure 2.21 shows the microstructure of the  $MgB_2$  core in tape 5-FR before (a) and after heat treatment (b). The microstructure clearly changes during the heat



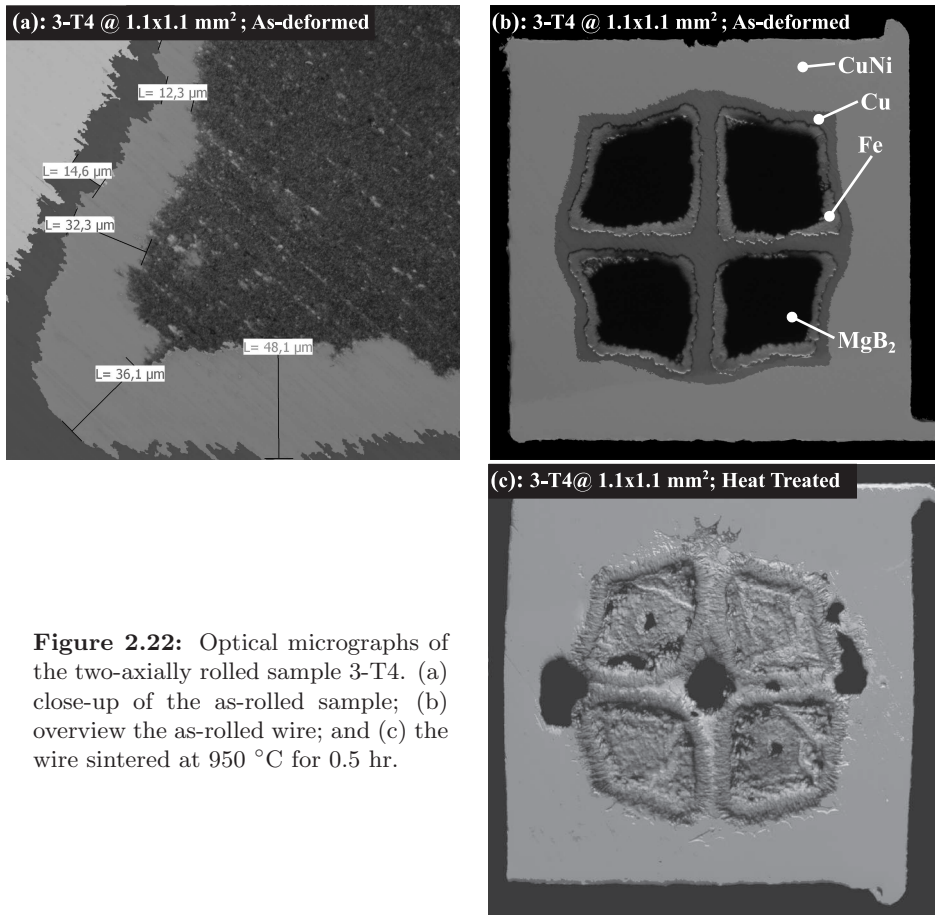
**Figure 2.21:** HRSEM micrographs of the  $\text{MgB}_2$  filament of sample 5-FR. (a) for the as-rolled sample and (b) for the tape sintered at  $950\text{ }^\circ\text{C}$  for 0.5 hr. The **arrows** in (b) indicate locations where neck formation has occurred.

treatment. First, it can be seen that the  $\text{MgB}_2$  grains have recrystallised as the grain surfaces are rounded and smoother than before heat treatment. Furthermore, the arrows in figure 2.21b indicate sites where “necks” between grains have formed, which is typical for sintering [84]. In this way, the heat treatment strongly improves the grain-to-grain connectivity and thus increases  $J_c$ . Note that this is in direct contrast to [85], where no sintering was observed in bulk  $\text{MgB}_2$  samples up to  $T = 1100\text{ }^\circ\text{C}$ .

### Reaction Layer

The main purpose of Fe in the matrix is to prevent the strong reaction between Cu and  $\text{MgB}_2$  [56, 60–62]. However, Fe and  $\text{MgB}_2$  also react to some extent, which at  $950\text{ }^\circ\text{C}$  for typical heat treatment times of 0.5 hr leads to the formation of interface layers of 12 to  $35\text{ }\mu\text{m}$  thickness [86]. Therefore, the Fe reaction barrier should be about 3 times as thick to prevent contact between Cu and Mg [82]. As discussed above, the multifilamentary two-axially rolled conductor 3-T4 has a relatively thin Fe reaction barrier, in some places down to  $12\text{ }\mu\text{m}$  (see figure 2.22a). Figure 2.22b and c show sample 3-T4 before and after heat treatment. The Fe reaction barrier was clearly too thin, causing Cu to diffuse into the filaments leaving voids in the matrix. Even if in this particular conductor the filaments were deformed non-uniformly and the Fe barrier became too thin only at some places, the requirement of a thick reaction barrier may impose filament size limits on the development of multifilamentary conductors with a Cu/Fe matrix.





**Figure 2.22:** Optical micrographs of the two-axially rolled sample 3-T4. (a) close-up of the as-rolled sample; (b) overview the as-rolled wire; and (c) the wire sintered at 950 °C for 0.5 hr.

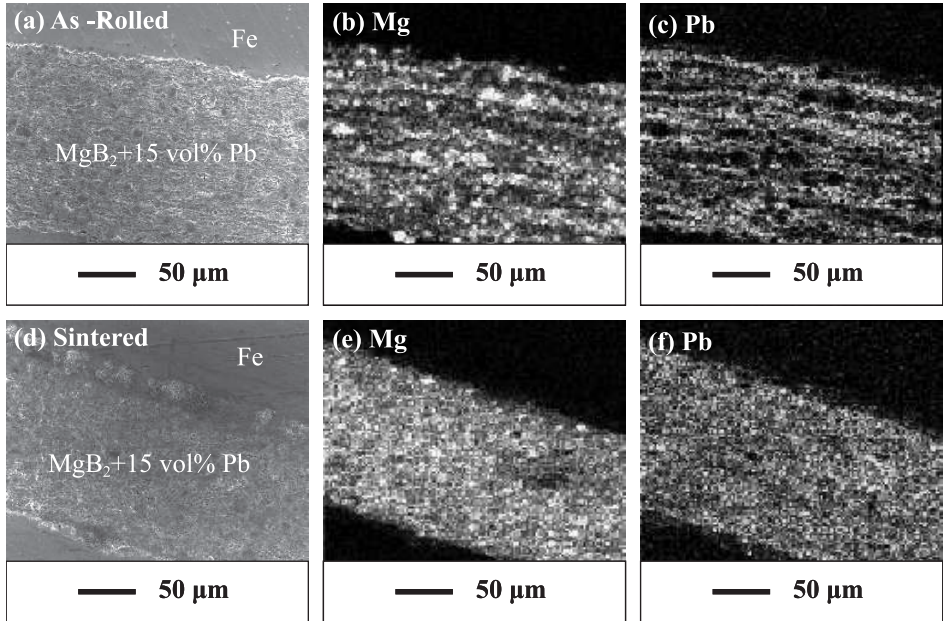
### Lead Addition

The motivation to investigate Pb-additions was not just improved deformation, but also possible  $J_c$  enhancement. Lead could improve intergrain connectivity by the formation of S/N/S junctions, but might also facilitate sintering. The envisaged mechanism is that the presence of a liquid phase significantly increases diffusivity and thus enhances neck growth and grain-to-grain connectivity.

Lead melts at  $T \approx 330$  K so that it can redistribute in the filament well before the sintering temperature 950 °C is reached. In this way, enhanced neck growth of the  $MgB_2$  grains would occur uniformly throughout the filament. Furthermore, in an O-poor environment no chemical reaction of Pb with either Fe, Mg and B should occur, since the only binary phase in this system,  $Mg_2Pb$ , is not stable above 550 °C [87].

Figure 2.23 shows the EDX chemical mapping of Mg and Pb in sample 5-Pb15-FR before and after heat treatment (a to c and d to f, respectively). In the





**Figure 2.23:** EDX mapping of the as-rolled ((a) to (c)) and sintered tape 5-Pb15-FR ((d) to (f)). (a) and (d) show the SEM images of the mapped area; (b) and (e) the distribution of Mg in the filament; and (c) and (f) the distribution of Pb in the filament.

as-rolled tape, the Pb stripes in the rolling direction are visible as discussed in the context of figure 2.15. After the heat treatment, Pb is redistributed across the filament.

HRSEM micrographs, did not reveal any improvement of the neck growth between the  $\text{MgB}_2$  grains. Nevertheless, the distributed Pb in-between the  $\text{MgB}_2$  grains may still improve the  $J_c(B)$  characteristic through the formation of S/N/S junctions or through thermal stabilisation.

## 2.4.2 Critical Current Density

In the previous paragraph it was shown that the  $\text{MgB}_2$  grains in sample 5-FR indeed show neck growth and that sintering thus enhances the grain-to-grain connectivity. However, as-drawn conductors show a significantly lower filament density than flat-rolled ones so that  $J_c$  after heat treatment is expected to be lower.

In figures 2.24a and b it is shown how the  $J_c(B)$  characteristic of the drawn wires 3-D-II and 5-D improves with heat treatment. Wire 5-D has a relatively hard Fe matrix compared to the softer Cu/Fe matrix used in sample 3-D-II. It

was already shown that this results in a higher filament density and an as-drawn  $J_c$  value of a factor 10 higher than in sample 3-D-II. This difference remains the same after sintering: both conductors show a factor 10 improvement of  $J_c$  after heat treatment irrespective of the as-drawn filament density.

A measure for the homogeneity in a conductor is the slope of its voltage-current characteristic:

$$E = E_c \left( \frac{J}{J_c} \right)^n, \quad (2.10)$$

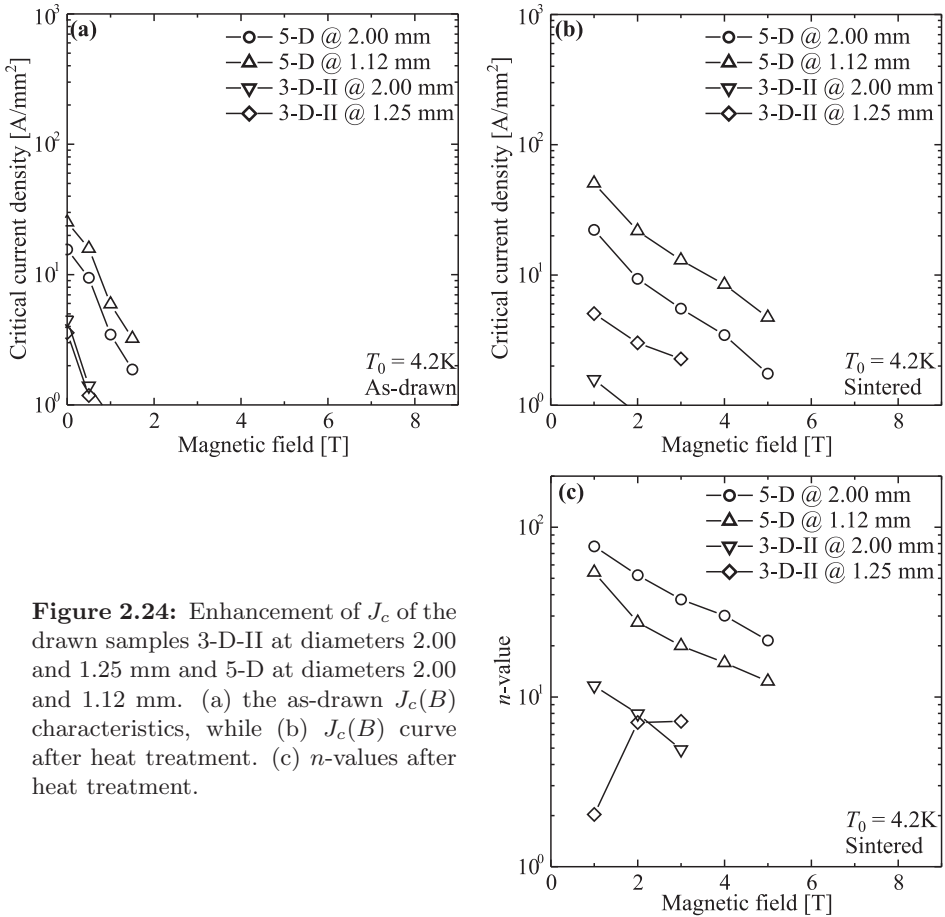
with  $E$  the electric field,  $E_c$  the electric field at which the critical current density  $J_c$  is defined,  $J$  the current density in the superconducting filaments and  $n$  the exponent of the power law. A smooth slope is characterised by a low  $n$ -value and indicates poor homogeneity in the filaments. The relatively high  $n$ -values of sample 5-D are typical for ex-situ  $\text{MgB}_2$  conductors and are about a factor of 10 higher than those for sample 3-D-II. Furthermore, sample 3-D-II does not show a clear field dependence of the  $n$ -value. Together, these observations demonstrate a relatively poor homogeneity of the filament in sample 3-D-II.

In figure 2.25, it is shown that also in the flat-rolled tapes a significant improvement is obtained in the flat-rolled samples during heat treatment. For sample 5-FR with no Pb,  $J_c$  at 4.2 K, 4 T increases by a factor 20. However, the apparent  $J_c(B)$  characteristic of the Pb-free reacted tape shows a clear plateau at fields lower than 6 T, caused by lack of thermal stabilisation due to the poor conductivity of the pure Fe matrix. This poor thermal stabilisation causes thermal runaways at high currents, which will be further elaborated in chapter 4. The plotted data of sample 5-FR below 6 T are strictly speaking not critical current density values, but rather quench current densities at which thermal runaways occur. As will be shown in §3.2.1 the “true”  $J_c(B)$  dependence in most  $\text{MgB}_2$  conductors can be well approximated with:

$$J_c(B) = J_{c,0}^T \exp \left[ -\frac{B}{B_p(T)} \right], \quad (2.11)$$

with  $J_{c,0}^T$  the zero field extrapolation of the critical current at a given temperature and  $B_p$  the characteristic current decay field. Fitting the  $J_c(B)$  curve of sample 5-FR with (2.11) through the  $J_c$  curve above 6 T results in the gray dotted line. Assuming the  $J_c(B)$  expression holds and using it to estimate “non-quench”  $J_c$  values below 6 T, an improvement of a factor  $\sim 100$  is attained during heat treatment. Compared to the drawn samples this is a relatively large gain, indicating that the higher as-rolled filament density in the tapes causes more contact areas between the grains that can serve as necking sites during sintering and thus improve  $J_c$  to a larger extent.

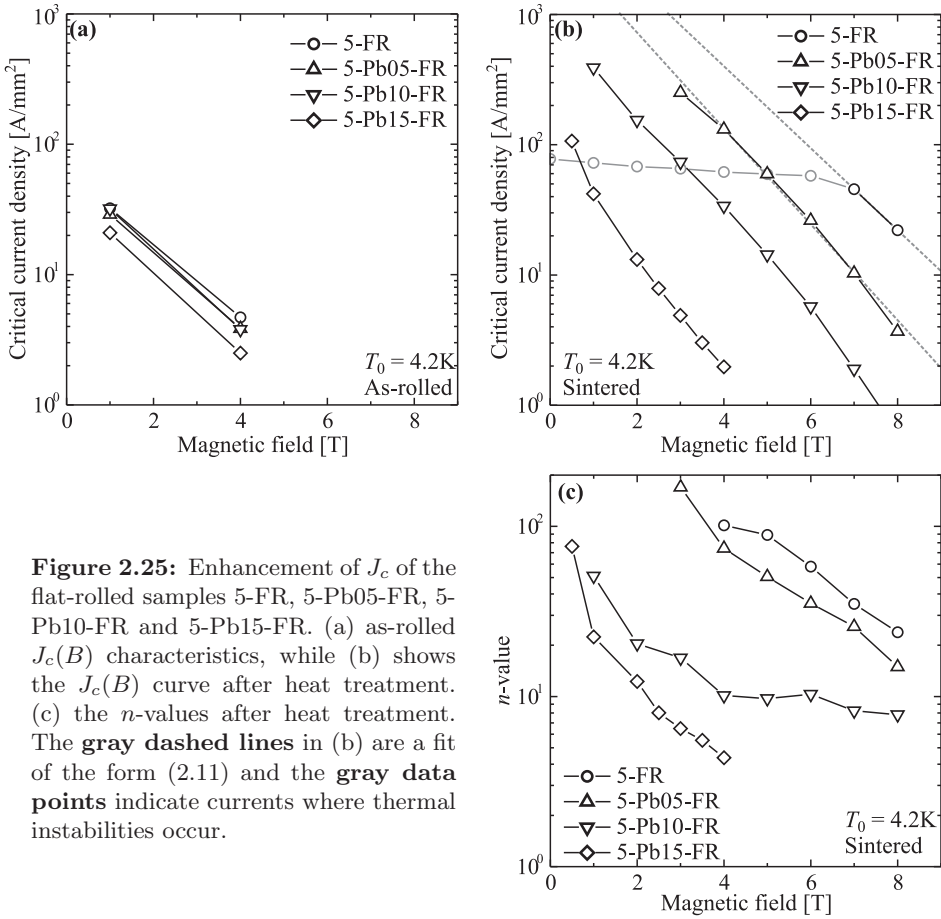
The main advantage of using Pb becomes clear by comparing the Pb-free sample 5-FR with the Pb-containing tapes 5-Pb05-FR and 5-Pb10-FR. The latter two samples do not show a plateau and  $J_c$  continues to increase towards lower field. This clearly shows that Pb works as an internal thermal stabiliser. On



**Figure 2.24:** Enhancement of  $J_c$  of the drawn samples 3-D-II at diameters 2.00 and 1.25 mm and 5-D at diameters 2.00 and 1.12 mm. (a) the as-drawn  $J_c(B)$  characteristics, while (b)  $J_c(B)$  curve after heat treatment. (c)  $n$ -values after heat treatment.

the other hand, the  $J_c(B)$  curves of these tapes indicate poorer grain-to-grain connectivity, since the estimated  $J_c(B)$  curve of sample 5-FR obtained with (2.11) is generally higher than that of the Pb-containing samples. Lead thus hampers the connectivity rather than improving it. Furthermore, in figure 2.25c, it is shown that the  $n$ -values decrease with increasing Pb concentration, again indicating poorer homogeneity in the filaments.

Further proof that Pb indeed compromises the homogeneity of current flow in the filaments is also strikingly seen in the voltage-current characteristics at high currents and low magnetic fields. Figure 2.26a shows the voltage-current characteristic of the heat treated wire 1-Pb10-D at self-field. Voltage jumps are visible which are very similar to the ones occurring during a thermal runaway. However, after such a voltage jump a new stable level is reached at a significantly lower voltage level than that corresponding to a completely normal conducting wire. Furthermore, when the current is ramped down again, a hysteresis effect

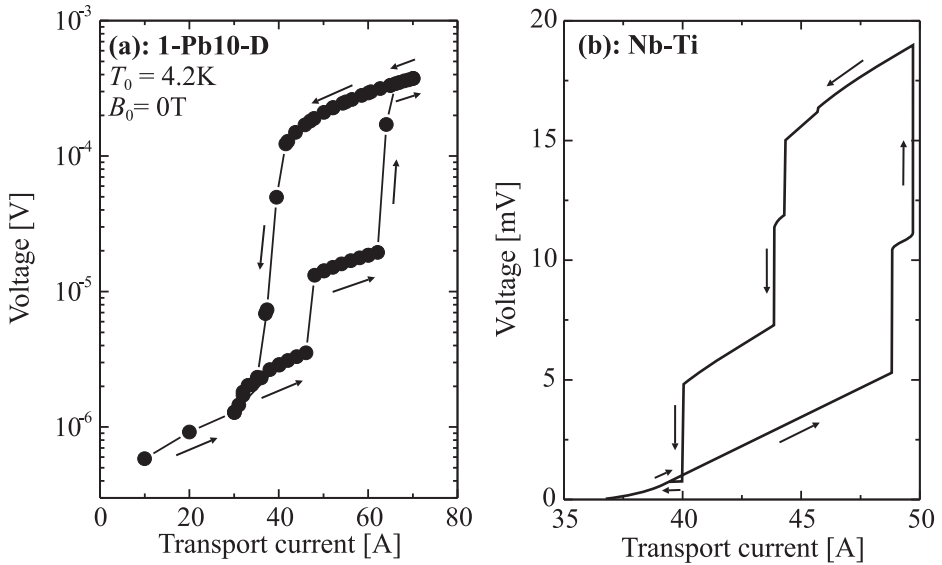


**Figure 2.25:** Enhancement of  $J_c$  of the flat-rolled samples 5-FR, 5-Pb05-FR, 5-Pb10-FR and 5-Pb15-FR. (a) as-rolled  $J_c(B)$  characteristics, while (b) shows the  $J_c(B)$  curve after heat treatment. (c) the  $n$ -values after heat treatment. The **gray dashed lines** in (b) are a fit of the form (2.11) and the **gray data points** indicate currents where thermal instabilities occur.

becomes apparent. These effects were also observed by Boschmann in Nb-Ti conductors [88], although at 10 to 1000 times higher voltage levels, and are explained by the existence of static normal zones in poorly conducting parts of the filament (figure 2.26b). In order to improve the grain-to-grain connectivity while maintaining the enhanced thermal stability, further tests with Pb concentrations between 1-5 vol.% are needed.

### Comparison with Published Conductors

In chapter 3, a complete literature survey of MgB<sub>2</sub> conductor data published over the years 2003-2006 is presented in detail. Here, some of the data is already used to compare tapes 5-FR and 5-Pb05-FR to samples produced elsewhere. The characteristic values  $J_{c,0}^T$  and  $B_p$  defined in (2.11) are used to compare between samples. In addition a third parameter  $B_{100}$  is used, which is the magnetic field at which  $J_c = 100\text{ A/mm}^2$  and can be seen as a rough upper field limit for the use



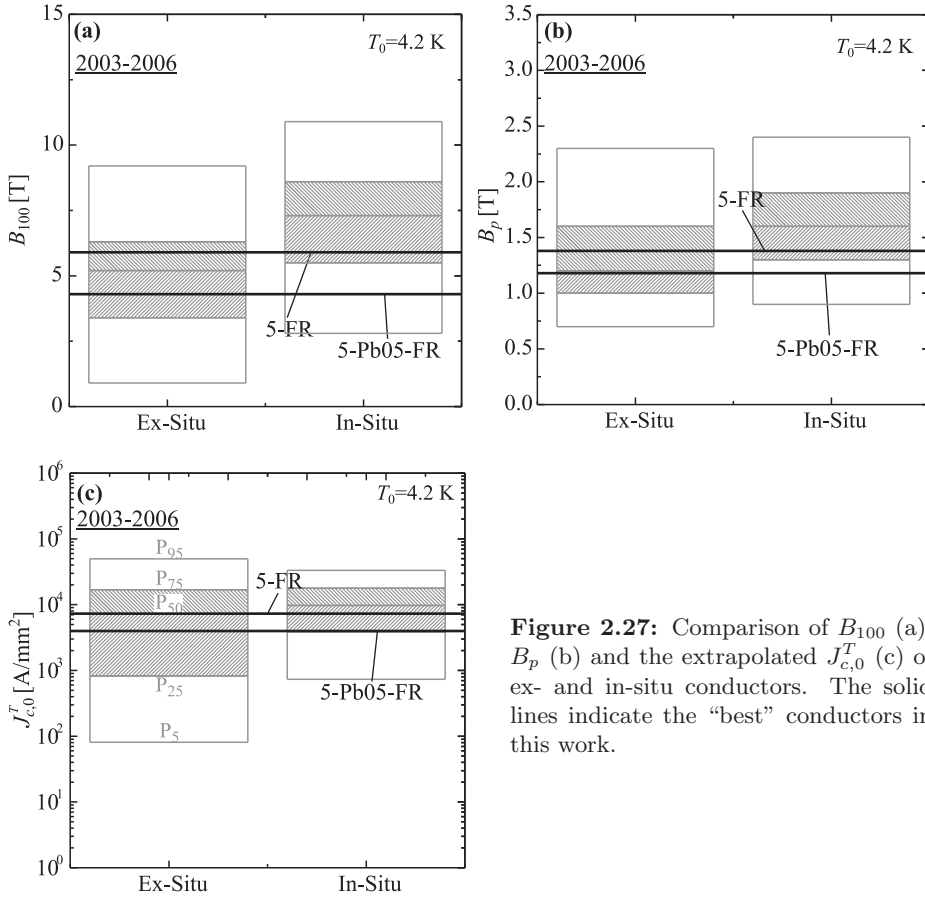
**Figure 2.26:** Hysteresis in the voltage-current characteristic of the heat treated Pb-containing wire 1-Pb10-D (a) and that of a Nb-Ti conductor (b) after Boschmann [88]. Note the log-scale in (a) and the factor 10-1000 higher voltage scale of (b).

of technical  $\text{MgB}_2$  superconductors. Note that  $B_{100}$  depends on both  $J_{c,0}^T$  and  $B_p$ .

Figure 2.27 shows the distributions of  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  values of the published ex- and in-situ conductors. As explained in chapter 3, the distributions are plotted as floating columns indicating the 5, 25, 50, 75 and 95 percentiles. The solid lines indicate the values of the three characteristic parameters of samples 5-FR and 5-Pb05-FR.

Compared to other published ex-situ conductors, sample 5-FR shows an above-average performance in terms of all three parameters as their values all lie above the median values  $P_{50}$ . For the Pb-containing sample 5-Pb05-FR, its  $B_p$  value is slightly lower than the median value. However its  $J_{c,0}^T$  value is about a factor 2 lower than the median and as a result also its  $B_{100}$  value is lower than the median. As will be discussed in §3.4.2, this is a general tendency of ex-situ conductors with additions, indicating compromised connectivity.

Compared with the in-situ conductors, the characteristic parameters of ex-situ samples 5-FR and 5-Pb05-FR all lie below the median values. As discussed in §3.4.1 and §3.4.2 in-situ conductors show a general tendency towards higher  $B_p$  values, associated with pinning or  $B_{c2}$  enhancement through doping. This trend results also in higher  $B_{100}$  values.



**Figure 2.27:** Comparison of  $B_{100}$  (a),  $B_p$  (b) and the extrapolated  $J_{c,0}^T$  (c) of ex- and in-situ conductors. The solid lines indicate the “best” conductors in this work.

## 2.5 Conclusion

To draw PIT conductors down to small diameters using the ex-situ route it is important to reduce the particle size of the as-purchased  $\text{MgB}_2$  powder. With finer particles stress concentrations in the matrix during deformation can be avoided. Furthermore  $J_c$  can be improved due to enhanced grain boundary pinning. Shear milling is a suitable method to effectively reduce the particle size. With this relatively simple technique the maximum particle size of the as-purchased  $\text{MgB}_2$  is reduced from  $\sim 100 \mu\text{m}$  to  $\sim 2 \mu\text{m}$  after 48 h milling. However, the milling fluid should be inert to prevent reaction with  $\text{MgB}_2$ . The tested ethanol turns out to decompose into methanol, leaving C as impurities in-between the grains and reducing the intergrain critical current density.

The combined requirements of sufficient densification, no reaction with  $\text{MgB}_2$  and enough thermal stability suggest a bi-metal matrix. The outer metal provides the thermal stability, while the inner material acts as a reaction barrier between

the chemical incompatible thermal stabiliser and the  $\text{MgB}_2$  filament. Highly conducting thermal stabilisers such as Cu are usually soft materials, while reaction barriers are usually hard. For drawing this may lead to fracture of the reaction barrier due to non-proportional flow during plastic deformation since the harder reaction barrier tends to deform less than the softer stabiliser. As a result, the thermal stabiliser exerts a tensile force on the reaction barrier. However, barrier fracture can be prevented by using either materials with similar yield strengths and work hardening rates or by a high barrier-to-stabiliser radius ratio. After deformation the reaction barrier should still be thick enough to prevent reaction between stabiliser and  $\text{MgB}_2$ . This may impose limits on the development of Cu/Fe/ $\text{MgB}_2$  conductors with finer filaments.

The densification of the  $\text{MgB}_2$  filament during drawing saturates at  $\sim 80\%$  of the maximum theoretical density, while with flat-rolling the filament density continues to increase to  $\sim 95\%$ . Compared to drawing, this leads to a factor 5-10 higher as-deformed  $J_c$  values. The as-deformed filament density turns out to be also important for the efficiency of the grain-to-grain neck growth during sintering. After heat treatment,  $J_c$  in the drawn wires has improved by a factor of 10 compared to the as-drawn value, while for the flat-rolled samples  $J_c$  improves by a factor of 50-100. For the fabrication of ex-situ conductors with high current densities, flat-rolling is a more suitable technique than drawing.

The advantage of using a mixture of hard  $\text{MgB}_2$  powder with soft Pb is that Pb acts as a lubricant during deformation, indicated by a  $\sim 7\%$  higher filament density compared to samples with pure  $\text{MgB}_2$ . As a result, Pb-containing wires can be drawn to  $\sim 10\%$  higher true strain levels. The effect of Pb on the superconducting properties is mainly in enhancing thermal stability. However, grain-to-grain connectivity and thus  $J_c$  is compromised by the addition of Pb. Further optimisation of the Pb concentration between 1 and 5 vol.% are needed to increase the grain-to-grain connectivity while maintaining the enhanced thermal stability.

In this work the conductors with the best  $J_c$  values are fabricated using flat-rolling and have a Fe matrix with a filament containing either pure  $\text{MgB}_2$  or a mixture with 5 vol. % Pb. Compared to ex-situ conductors that were reported over the years 2003-2006, the tape with pure  $\text{MgB}_2$  performs above average in terms of the zero-field extrapolation of  $J_c$  as well as field retention of  $J_c$ . The tape with Pb shows reduced field retention and zero field extrapolation of  $J_c$  compared to the one with pure  $\text{MgB}_2$ , but also shows better thermal stability at lower fields and high currents.





## Chapter 3

# Critical Current

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*A literature study is presented of  $J_c(B,T)$  data for conductors reported over the years 2003-2006. Statistical analysis is used to reveal general trends and prospects for further development. The main mechanism behind the progress made during these years is discussed and the prospects for further development with the different deformation routes is explored. Also the different effects of additions in ex-situ conductors and in in-situ conductors is presented. Finally it is shown that non-carbon and carbon based additions differently affect the field retention of  $J_c$ , possibly on a fundamental level.*

### 3.1 Introduction

The development of practical  $\text{MgB}_2$  superconductors has followed different approaches that vary regarding several aspects.

A first aspect is the precursor powder. Conductors are developed using the ex-situ and in-situ routes [89,90]. In both routes progress in the field-dependence of the critical current has been achieved by optimising the starting powder in terms of powder purity and morphology [65,90,91]. Also, substantial improvement of the  $J_c(B)$  performance has been made with additions to the  $\text{MgB}_2$  powder, such as nanometric SiC [24].

A second aspect is the deformation route. The two commonly applied processes are drawing into wires and flat-rolling into tapes [65,89,90,92]. Both conductor types are fabricated using the in- and ex-situ techniques, with and without additions. Important in view of AC-losses, thermal stability and mechanical behaviour is the development of multi-filament conductors. At present all conductor types are successfully fabricated [19,65], with increasing number of filaments and thus reduction of their size.

The development of a new superconducting material into a practical conductor only makes sense if it will offer specific advantages over existing technology.  $\text{MgB}_2$  may become an economical conductor for use in medium-range magnetic fields, similar to Nb-Ti but able to operate also at a higher temperature. Indeed,  $\text{MgB}_2$  conductors with a high-field  $J_c(B)$  performance at 4.2 K better than that of Nb-Ti wires have already been reported [8,17], see figure 1.3.

In the present chapter it is discussed whether this development can reasonably be expected to continue further, or whether the  $J_c(B)$  performance of  $\text{MgB}_2$  conductors already appears to approach intrinsic limits. A second question to be addressed is whether any of the development lines sketched above is more successful than others.

In an attempt to answer these questions, the literature regarding published transport  $J_c(B)$  characteristics of  $\text{MgB}_2$  conductors was surveyed over the years 2003 to 2006. Characterising the  $J_c(B)$  curves with only three figures of merit allows to perform a statistical analysis of trends in the development of  $\text{MgB}_2$  conductors. With such statistical analysis it is also possible to distinguish between conductor-types categorised by ex-situ versus in-situ or tape versus wire, regardless of laboratory-specific fabrication conditions. Also, distribution functions of the three figures of merit allow to reveal possible intrinsic limits, based on the  $J_c(B)$  data so far.

It should be noted, however, that a meaningful statistical analysis requires a sufficient amount of samples (typically  $\geq 15$ ). It is not possible with this method to study trends in less common conductor-types, such as those fabricated using the liquid infiltration technique [53] or “coated conductor” routes [50,52,93]. Another important remark concerns “record” performance conductors. By its very nature, a statistical analysis will not reveal the combination of factors leading to such “best” performance. Instead, it will indicate trends and thus help to clarify the general potential of certain development routes.

In the survey described in this chapter, the published conductors are first discriminated according to their publication year, which allows to study the progress over the years in §3.3.

To study the effect of different precursor powders, the conductors are also categorised by either in-situ or ex-situ fabrication route. Also the group of conductors that contain pure MgB<sub>2</sub> and those also containing additions are distinguished. How the  $J_c(B)$  characteristics among these categories tend to differ is subject of §3.4.

From a deformation point of view samples are categorised as wires or tapes and mono- or multi-filament. In §3.5 it is discussed how the  $J_c(B)$  curves of wires and tapes tend to differ. Whether further deformation and down-scaling of the filaments in multi-filament conductors tend to affect the  $J_c(B)$  characteristic is also considered.

The chosen  $J_c(B)$  parametrisation is presented in §3.2 together with the method and overall results of the literature survey.

## 3.2 Literature Survey

In §3.2.1 it is illustrated how the  $J_c(B)$  dependence of the conductors is parametrised with three “figures of merit”: the extrapolated self-field  $J_c$ -value and two characteristic fields  $B_p$  and  $B_{100}$ . Which published  $J_c(B)$  curves are included in the survey and which not is described in §3.2.2 together with the overall results. In order to study the influence of several variables on the  $J_c(B)$  behaviour (year, in versus ex-situ, pure MgB<sub>2</sub> versus additions) distribution functions of the three figures of merit are also introduced in this paragraph.

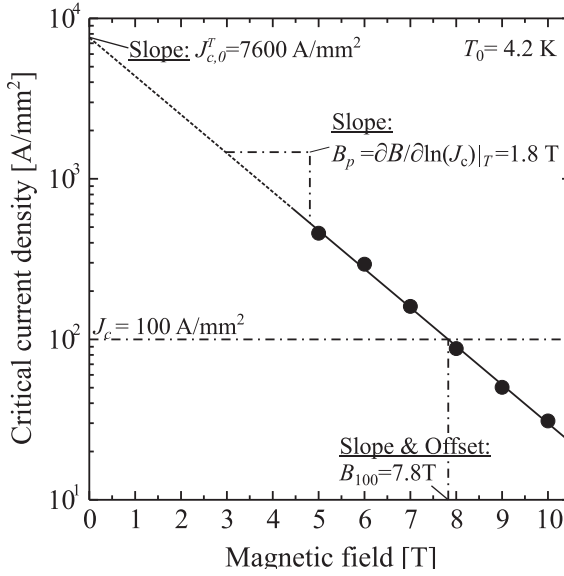
### 3.2.1 Critical Current Characterisation

To describe the critical current density of MgB<sub>2</sub> conductors as a function of magnetic field and temperature the following approximation [94] is fitted to the published  $J_c(B)$  results (see also figure 3.1):

$$J_c(B, T) \approx J_{c,0}^T \exp \left[ -\frac{B}{B_p(T)} \right] \text{ with } J_{c,0}^T(T) = J_{c,0} \left( 1 - \frac{T}{T_c} \right) \quad (3.1)$$

$$\text{and } B_p(T) = B_{p,0} \left( 1 - \frac{T}{T_c} \right),$$

where  $J_{c,0}^T$  is the temperature-dependent zero-field extrapolation of the critical current density;  $J_{c,0}$  the zero-temperature zero-field extrapolation of  $J_c$ ;  $B_p = \partial B / \partial \ln(J_c)$  the temperature-dependent exponential current decay field; and  $B_{p,0}$  its zero temperature extrapolation. With approximation (3.1), the parameters  $J_{c,0}^T$  and  $B_p$  fully describe the  $J_c(B)$  curve of a given conductor at a given temperature.  $J_{c,0}^T$  is the maximum critical current density at that temperature. A-priori, it might be determined by the grain-to-grain connectivity or effective cross-section



**Figure 3.1:** Example of fitted  $J_c(B)$  data [95]. Indicated are the zero field extrapolation of the critical current density  $J_{c,0}^T$ ; the slope  $-1/B_p$  of the  $J_c(B)$  curve; and the magnetic field  $B_{100}$  where  $J_c$  falls below a value of  $100 \text{ A/mm}^2$ .

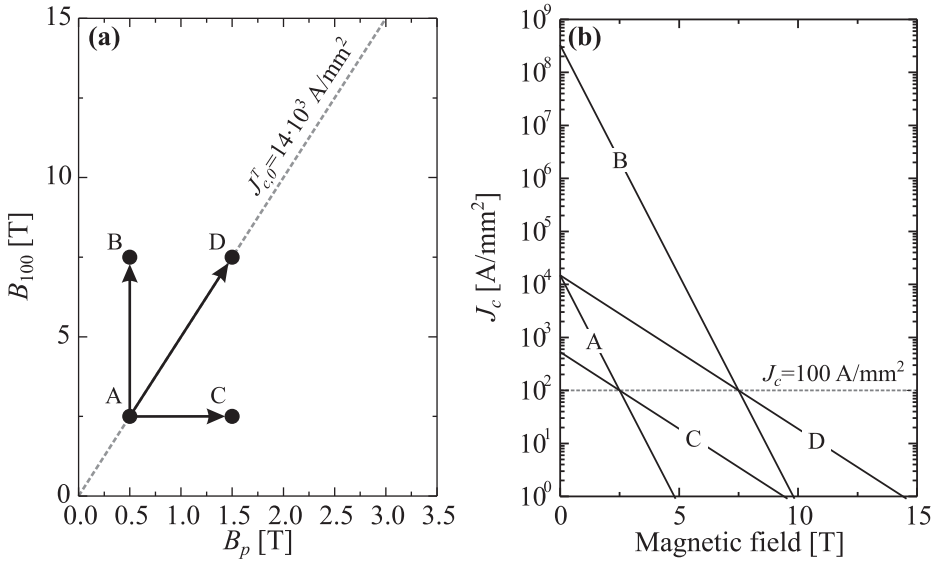
in the filaments, but also by the intra-granular pinning of vortices.  $B_p$  describes the field retention of  $J_c$  and is influenced by both flux-pinning and  $B_{c2}$ . Note that  $B_p$  is also the magnetic field at which the pinning force  $F_p = J_c B$  is maximum.

Figure 3.1 illustrates how the parameters  $J_{c,0}^T$  and  $B_p$  are extracted from the data published in [95]. These  $J_c(B)$  data are typical in the sense that the authors only published results at higher fields ( $B \geq 5 \text{ T}$ ). At low fields insufficient thermal stabilisation often causes thermal runaways or a homogeneous rise of the sample temperature so that the apparent  $J_c(B)$  curve tends to “level off” towards zero field.

This implies that the parameter  $J_{c,0}^T$  usually needs to be extrapolated, introducing uncertainty on the obtained results. Therefore a third parameter  $B_{100}$  was extracted from the data, defined as:

$$B_{100} \equiv B_p(T) \ln \left( \frac{J_{c,0}^T}{100 [\text{A/mm}^2]} \right). \quad (3.2)$$

$B_{100}$  is the field at which the value of  $J_c$  falls below  $100 \text{ A/mm}^2$ . The value  $100 \text{ A/mm}^2$  is an arbitrary choice, but can reasonably be seen as a lower limit for the usefulness of technical superconductors.  $B_{100}$  can therefore be considered as a practical upper field limit for the use of a given superconductor. The advantage of using  $B_{100}$  is that in most papers it falls within the published data range.



**Figure 3.2:** (a) shows the  $B_{100}$ - $B_p$  plot in which the four hypothetical conductors A, B, C and D are indicated. The dotted line indicates the zero field extrapolation of  $J_c$  of conductors A and B. (b) shows their corresponding  $J_c(B)$  curves.

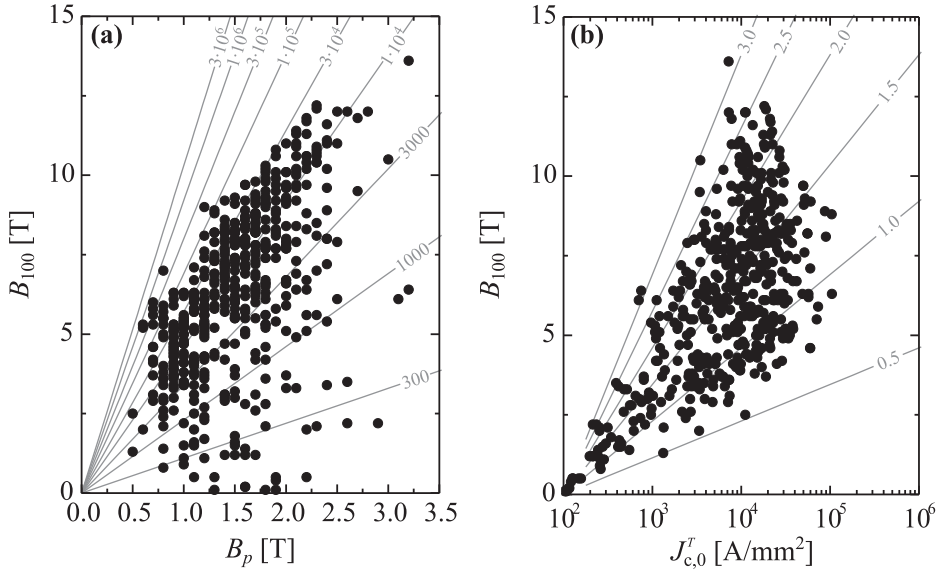
Note that its value depends on grain-to-grain connectivity as well as on the field retention of  $J_c$ . Obviously, the three parameters  $J_{c,0}^T$ ,  $B_p$  and  $B_{100}$  are not independent.

To illustrate their relation, figure 3.2a shows the  $B_p$  and  $B_{100}$  values of four hypothetical conductors A-D, while figure 3.2b shows their corresponding  $J_c(B)$  curves. Conductor A has relatively low  $B_p$  and  $B_{100}$  values, corresponding to a steep decay of  $J_c$  with magnetic field and a relatively low overall  $J_c$ . Conductor B has the same  $B_p$  value as conductor A. However, its  $B_{100}$  value is higher due to a higher overall  $J_c$ <sup>1</sup>. Thus, conductor B has a better connectivity than conductor A. Conductor C shows a higher  $B_p$  value, corresponding to a better field retention of  $J_c$ . However, the  $B_{100}$  values of conductors A and C are similar due to its lower  $J_{c,0}^T$ . Compared to conductor A, conductor D has the same  $J_{c,0}^T$  value but a better field retention, resulting in a higher  $B_{100}$ . Thus, it is pinning or a higher  $B_{c2}$  causing conductor D to perform better than A.

### 3.2.2 Result of the Literature Survey

For the survey, the literature database INSPEC was used to collect a publication record [96]. The search term “(MgB<sub>2</sub> wire) or (MgB<sub>2</sub> tape) or (MgB<sub>2</sub> conductor)”

<sup>1</sup>Note that the zero field extrapolation of  $J_c \sim 10^8$  A/mm<sup>2</sup> in this hypothetical sample is unrealistically high.

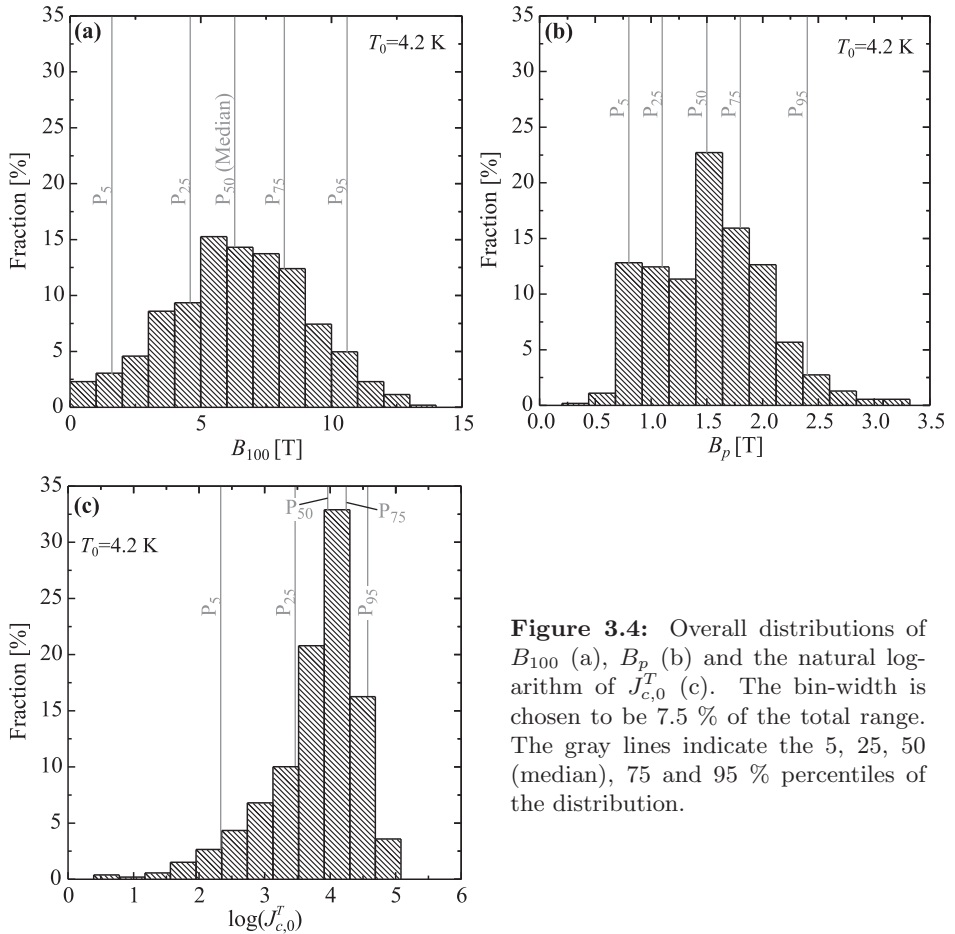


**Figure 3.3:** (a) shows the  $B_{100}$  versus  $B_p$  plot in which all investigated conductors are indicated. The gray lines indicate constant  $J_{c,0}^T$  values (units are [A/mm<sup>2</sup>]). (b) shows the equivalent  $B_{100}$  versus  $J_{c,0}^T$  plot. The gray lines indicate constant  $B_p$  values.

yields about 300 articles over the years 2003-2006. Earlier conductor development, covering 2001 and 2002, is described in the review [56]. Approximately 110 of the 300 papers present transport  $J_c(B)$  or  $I_c(B)$  data together with information on the filling factor. Overall, the survey comprises  $J_c(B)$  curves of approximately 550 different MgB<sub>2</sub> conductors. The publications used are [19–22, 53, 55, 58, 65, 74, 89–92, 95, 97–189] and selected properties of the conductors are tabulated in appendix A. Note that this survey focusses on the  $J_c(B)$  curves at 4.2 K. Only < 5 % of published  $J_c(B)$  characteristics were measured at the anticipated operating temperature of  $\sim 20$  K, which would lead to poor statistics in the further analysis. Nevertheless, a general comparison between the available data at 20 K and those at 4.2 K is presented in §3.6.

The overall result of the literature survey is summarised in figures 3.3a and b, showing  $B_{100}$  as a function of  $B_p$  or of  $J_{c,0}^T$ , respectively. With respect to the self-field current, the majority of conductors tend to lie grouped around  $J_{c,0}^T = 10^4$  A/mm<sup>2</sup>, while  $B_{100}$  and  $B_p$  values are rather scattered and a trend is relatively difficult to deduce.

To study correlations between the three figures of merit and various selected properties or processing conditions (in- versus ex-situ), distributions are determined. Figure 3.4a to c show the overall distributions of  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$ . Also indicated in the plots are the 5, 25, 50 (median), 75 and 95 % percentiles of each



**Figure 3.4:** Overall distributions of  $B_{100}$  (a),  $B_p$  (b) and the natural logarithm of  $J_{c,0}^T$  (c). The bin-width is chosen to be 7.5 % of the total range. The gray lines indicate the 5, 25, 50 (median), 75 and 95 % percentiles of the distribution.

distribution, which will be indicated by  $P_5$ ,  $P_{25}$ ,  $P_{50}$ ,  $P_{75}$  and  $P_{95}$ . As an example, the  $P_{25}$  value of  $B_{100}$  lies at 4.6 T which means that 25 % of the published conductors have a  $B_{100}$  value  $\leq 4.6$  T.

Of the three figures of merit  $B_{100}$  is arguably the most important one from a technological point of view. Therefore, in the remainder of this chapter variations in  $B_{100}$  are discussed in terms of variations in  $B_p$  and  $J_{c,0}^T$ . However, equation (3.2) shows that variations in  $B_{100}$  depend on variations in  $\ln(J_{c,0}^T)$  rather than  $J_{c,0}^T$  itself. For this reason figure 3.4c shows the distribution of  $\log(J_{c,0}^T)$ .

The first impression in figure 3.3b that the  $J_{c,0}^T$  value of the conductors are grouped around the  $J_{c,0}^T = 10^4$  A/mm<sup>2</sup> line is reflected in figure 3.4c by a strong peakedness in the  $J_{c,0}^T$  distribution. This distribution is also strongly asymmetric. Below the median  $P_{50} \sim 10^4$  A/mm<sup>2</sup> the distribution shows a tail towards lower  $J_{c,0}^T$  values, while above the median there is a strong drop-down. This is an

indication that the top 50 % of the conductors display  $J_{c,0}^T$  values near a maximum.  $B_{100}$  and  $B_p$ , shown in figure 3.4a and b, have a relatively wider distribution and a less pronounced peakedness.

Note that in figures 3.4a to c all conductors are lumped together, i.e. no distinction is made yet between in- and ex-situ, doped and un-doped and tapes and wires. In the following paragraphs these different variables will be distinguished. However, first in §3.3 the evolution of  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  over the years 2003-2006 will be discussed.

### 3.3 Evolution over the Years

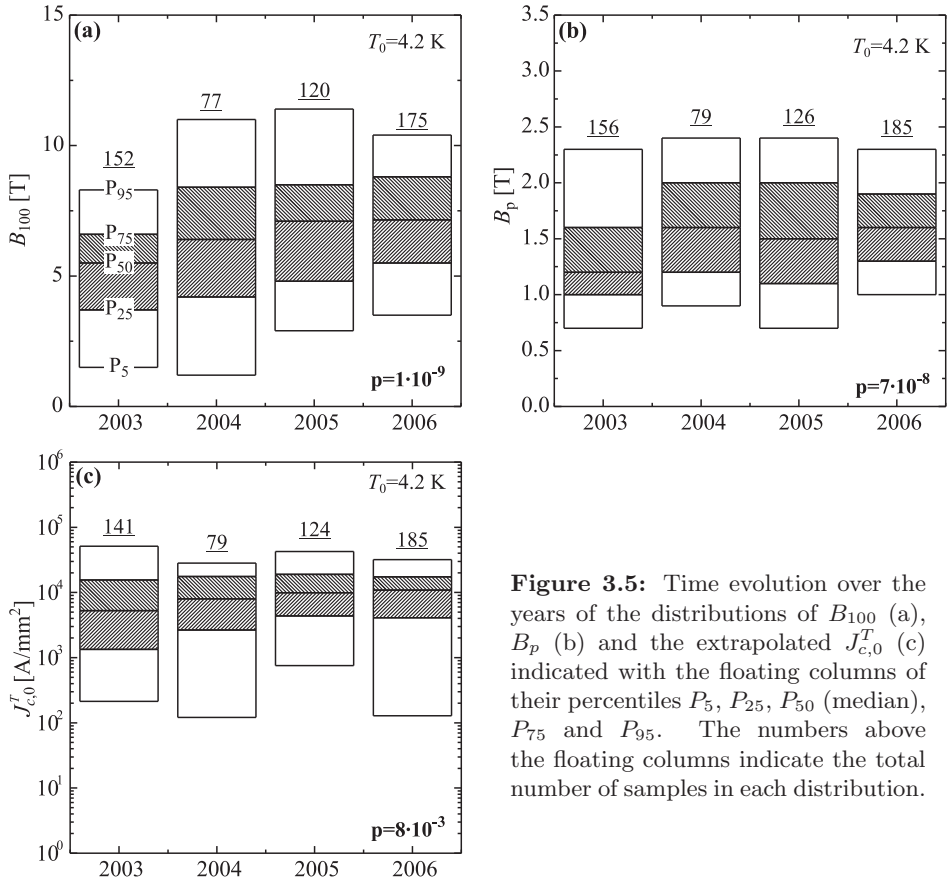
Figures 3.5a to c show how the distributions of the  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  evolve over the years 2003-2006. The distributions are presented as floating columns, indicating the  $P_5$ ,  $P_{25}$ ,  $P_{50}$ ,  $P_{75}$  and  $P_{95}$  values. The numbers on top of each column indicate the total number of samples in each year. No distinction is made between the precursor powder, mono- or multi filament and tapes or wires. Note that the numbers of samples in each year are not always the same for  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$ , partly because unrealistically high values of  $J_{c,0}^T (> 10^6 \text{ A/mm}^2)$  are disregarded and partly because some conductors had self-field  $J_c$  values below  $100 \text{ A/mm}^2$ , so that  $B_{100}$  is meaningless.  $B_p$ , however, could be determined for all samples.

Figures 3.5a to c show that the median value of all parameters appears to increase over the years. Whether such a variation is significant or not can be determined with the so-called Kruskal-Wallis test [190]. This method tests the significance of the differences between non-uniform distributions of *groups* (in this case the samples reported in a year) that also have unequal variances. It is similar to the better-known ANOVA test of variance, which is only applicable to normal distributions that have similar variances. The main outcome of the test is a *p-value* that indicates the probability that observed differences are purely coincidental. When the p-value is large, the probability is high so that the differences between groups are insignificant. A low p-value indicates that coincidence can be rejected so that the differences are significant. In practice a threshold of 5 % is often adopted, i.e.  $p \leq 5 \%$  implies a significant difference.

The p-values of the time evolution of each parameter are also indicated in the figures. All are well below the practical threshold of 5 % so that the time evolution is significant for all parameters. From 2003 to 2006, the median values of both characteristic fields  $B_{100}$  and  $B_p$  increase by about 30 %, while the median value of  $\log(J_{c,0}^T)$  increases by about 18 %. However, the distribution of  $J_{c,0}^T$  is much wider than that of  $B_{100}$  and  $B_p$ . As a result also its p-value is higher than that of the characteristic fields (although still lower than 5 %) and the evolution in  $J_{c,0}^T$  is thus less significant.

Assuming that  $J_{c,0}^T \approx 10^4 \text{ A/mm}^2$  and only varies little over the years, a 30 % increase in  $B_p$  would indeed induce a 30 % increase of  $B_{100}$  (similar to conductors A and D in figure 3.2). Thus, the main improvement over the years appears

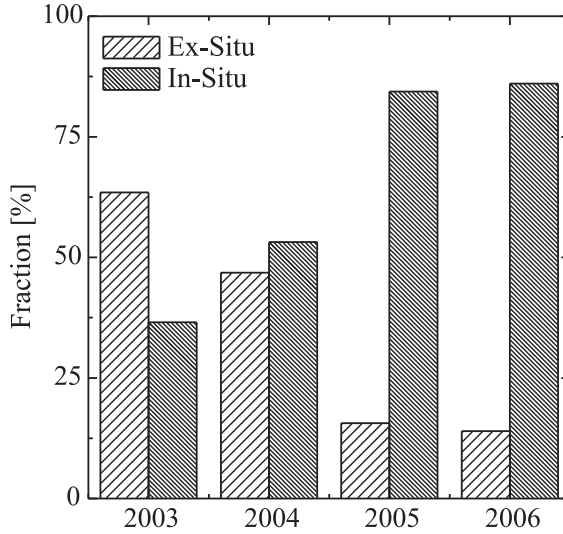




**Figure 3.5:** Time evolution over the years of the distributions of  $B_{100}$  (a),  $B_p$  (b) and the extrapolated  $J_{c,0}^T$  (c) indicated with the floating columns of their percentiles  $P_5$ ,  $P_{25}$ ,  $P_{50}$  (median),  $P_{75}$  and  $P_{95}$ . The numbers above the floating columns indicate the total number of samples in each distribution.

to be the enhancement of  $B_p$ , which corresponds to enhanced pinning or higher  $B_{c2}$  values.

Related to this observation is figure 3.6, where the evolution of the fraction of ex- and in-situ prepared conductors are plotted over the years 2003-2006. No distinction is made yet between samples that contain pure MgB<sub>2</sub> or samples that also contain additions (e.g. SiC). Samples that are fabricated with another procedure, such as the liquid infiltration technique [53] or the hybrid technique used by [22] are disregarded in this comparison. Strikingly, in 2003 60 % of published conductors were ex-situ, while in 2006 this amount is reduced to about 10 %. As will be shown in the next paragraph, in-situ conductors typically display significantly higher  $B_p$  and  $B_{100}$  values, mainly since enhancement of  $B_p$  and  $B_{100}$  through additions in in-situ conductors turns out to be more effective than in ex-situ samples.



**Figure 3.6:** Evolution over the years of published  $J_c(B)$  curves (at 4.2 K) of ex- and in-situ conductors.

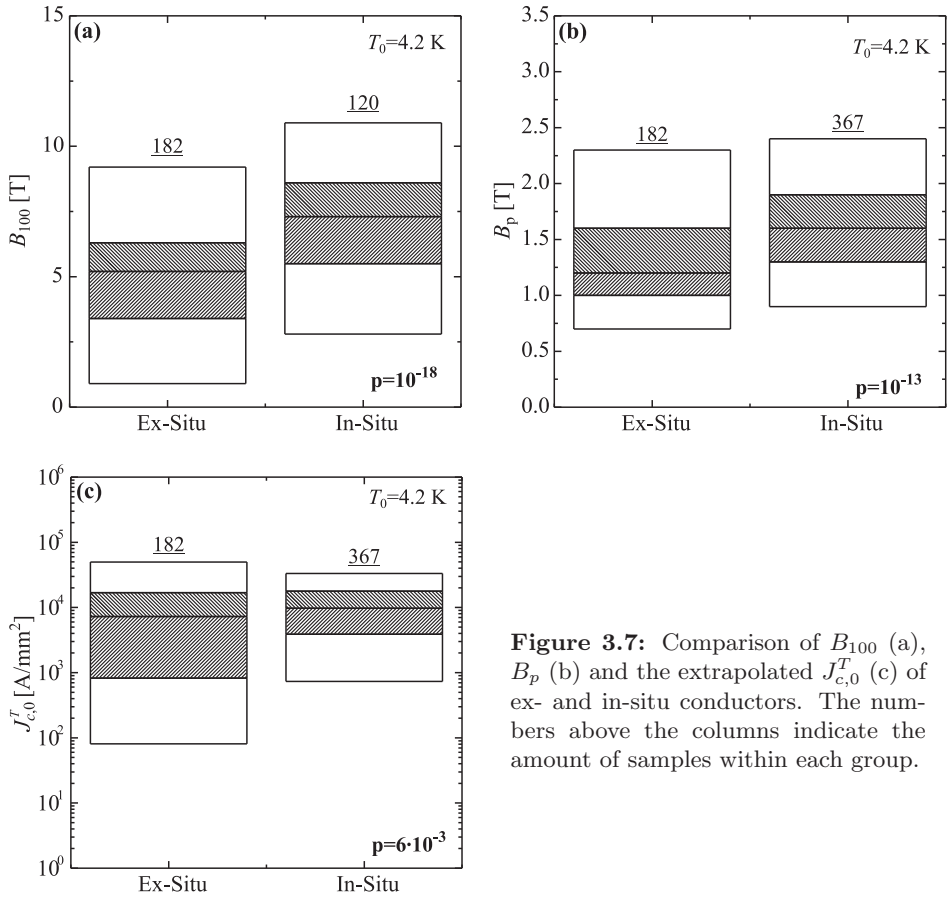
## 3.4 Influence of Precursor Powder

The influence of the precursor powder on the  $J_c(B)$  behaviour is described in this paragraph, distinguishing between conductors that contain pure ex- or in-situ powder and those that contain additions.

A comparison between ex-situ and in-situ conductors is the subject of §3.4.1. In §3.4.2 it is discussed how additions in ex-situ conductors tend to have a different effect than in in-situ conductors. Also, it turns out that carbon based additions in in-situ conductors tend to differ significantly from those with non-carbon based additions. This will be the subject of §3.4.3.

### 3.4.1 Ex-Situ versus In-Situ

A comparison between  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  of grouped ex- and in-situ conductors is shown in figures 3.7a to c. The median values of the two characteristic fields  $B_{100}$  and  $B_p$  are significantly higher by  $\sim 30\%$  for in-situ conductors. The median value of  $\log(J_{c,0}^T)$  of in-situ conductors is only  $\sim 5\%$  higher, while the significance of the difference is lower than for the two characteristic fields. Thus the higher typical  $B_{100}$  value observed for in-situ conductors is mainly caused by a higher  $B_p$ , i.e. better pinning or a higher  $B_{c2}$ . It should be stressed that within each group, no distinction is made yet between conductors that contain pure  $\text{MgB}_2$  and those with additions. This distinction is made below in §3.4.2 and will clarify the



**Figure 3.7:** Comparison of  $B_{100}$  (a),  $B_p$  (b) and the extrapolated  $J_{c,0}^T$  (c) of ex- and in-situ conductors. The numbers above the columns indicate the amount of samples within each group.

origin of the significantly different median  $B_p$  values.

Also worth noting is that the  $J_{c,0}^T$  values of ex-situ conductors display a wider distribution than in-situ ones, which is quantified by the 40 % higher standard deviation.

### 3.4.2 Additions in Ex-Situ and In-Situ Conductors

In an attempt to improve the  $J_c(B)$  behaviour of conductors, additions to the  $\text{MgB}_2$  precursor powder are extensively studied. In this paragraph statistical analysis is applied to show how  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  tend to vary with additions.

The first additions in  $\text{MgB}_2$  were chemical substitutions to enhance  $T_c$  (see the review by Buzea *et. al.* [191] and references therein). Examples of studied dopants are Si, Li, Ni, Fe, Co and Mn. However, as most of these tend to lower  $T_c$ , straightforward doping is rarely applied in conductors.

A breakthrough came in 2002, when Dou *et. al.* [24] reported that nanometric

SiC added to the precursor powder substantially improved the resulting  $J_c(B)$  characteristic. Since then, many other nanometric additions such as  $\text{SiO}_2$ ,  $\text{ZrSi}_2$  and  $\text{B}_4\text{C}$  have been tried. The results are described below.

Another type of additions are soft metals like Pb or In [72, 73]. These materials facilitate easier deformation, especially in the case of ex-situ conductors. Improvement of  $J_c(B)$  is explained as a possible proximity effect in an ensemble of S/N/S junctions, which would enhance the grain-to-grain connectivity [72]. Since this type of additions is already discussed in chapter 2, the remainder of this chapter will focus on the nanopowder additions.

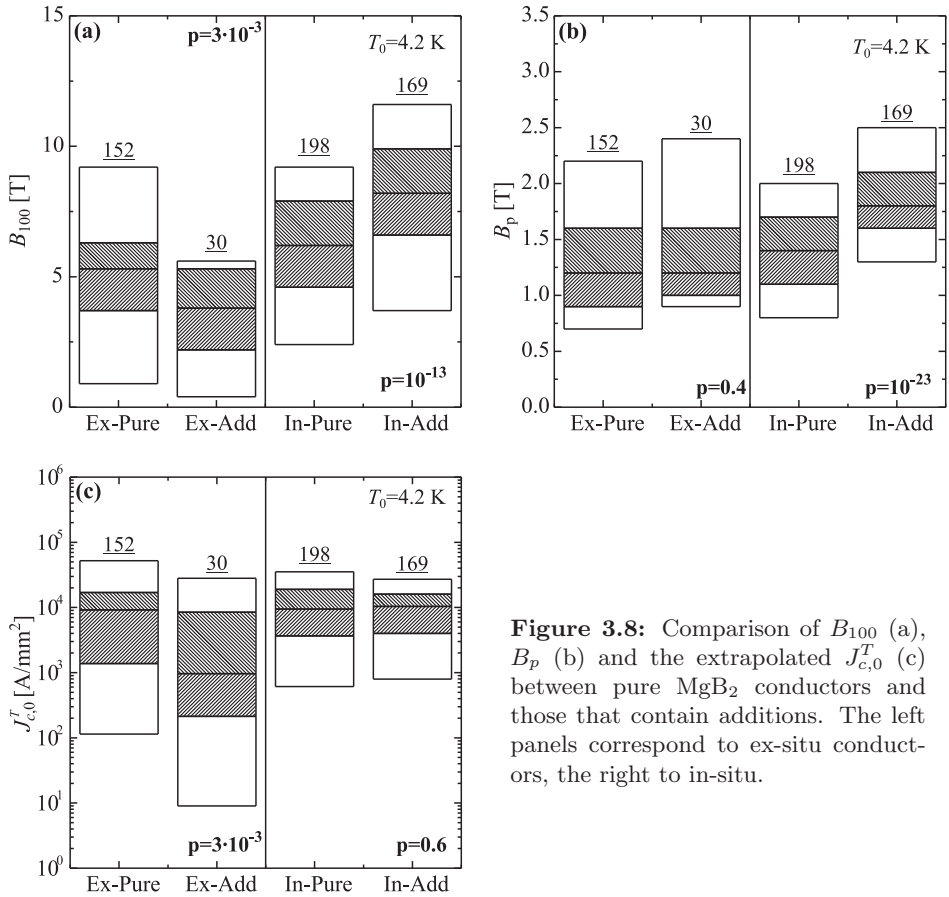
A priori, improvement of the  $J_c(B)$  characteristic following from nanometric additions might be explained through enhanced pinning or through an increase of  $B_{c2}$ . In the first scenario nanometric particles are incorporated into the  $\text{MgB}_2$  crystallites as a whole and directly or indirectly form pinning centers. An analogous system is top seeded melt grown  $(\text{RE})\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  that contains the secondary phase  $(\text{RE})_2\text{BaCuO}_5$  as nano-inclusions [192]<sup>2</sup>. Although this material has important applications in powerful trapped-field magnets and has been intensively studied for several years, the detailed mechanism of pinning enhancement is still under debate. Often cited possibilities are core pinning at oxygen-vacancy clusters in the vicinity of the inclusions or the presence of strain fields at the  $(\text{RE})\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}/(\text{RE})_2\text{BaCuO}_5$  interface that cause local variations of the order parameter. Also in  $\text{MgB}_2$  TEM studies show that nanometric SiC additions are indeed incorporated in the  $\text{MgB}_2$  crystallites, e.g. [193], so that similar pinning mechanisms may also apply here.

Another possible mechanism is pinning of magnetic flux at grain boundaries. In  $\text{Nb}_3\text{Sn}$  this is believed to be the primary pinning mechanism, since the maximum pinning force strongly correlates with grain size [12]. Also in  $\text{MgB}_2$  smaller grains were found to enhance the irreversibility field  $B^*$ , which is associated with improved pinning [66]. In this context, Mikheenko *et. al.* suggested an indirect role of nanometric additions: that of grain-growth inhibitor [63]. It was found that polycrystalline  $\text{MgB}_2$  samples containing nanometric particles systematically have smaller grains. As a consequence the amount of grain boundaries increases and pinning is improved.

In the second scenario, in which  $J_c(B)$  is enhanced by increasing  $B_{c2}$ , the nanometric particles are thought to act as a source for low-level doping. As an analogy, in  $\text{Nb}_3\text{Sn}$  conductors Ta and Ti additions are incorporated in the crystal structure on Nb sites [12]. As a consequence the electronic mean free path decreases, resulting in a higher normal state resistivity and a lower effective coherence length below  $T_c$ . In this so-called dirty limit, the smaller coherence length causes  $B_{c2}$  to increase [67]. In  $\text{MgB}_2$ , low-level C substitution on B sites is believed to play a similar role, but with a much stronger effect due to the two-gap nature of  $\text{MgB}_2$  [194–196]. This scenario is supported by observations in C doped single crystals, where point-contact spectroscopy reveals strongly increased coupling between the two gaps [197]. In bulk samples and conductors, X-ray

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<sup>2</sup>RE stands for Rare Earth.



**Figure 3.8:** Comparison of  $B_{100}$  (a),  $B_p$  (b) and the extrapolated  $J_{c,0}^T$  (c) between pure MgB<sub>2</sub> conductors and those that contain additions. The left panels correspond to ex-situ conductors, the right to in-situ.

diffraction experiments have also shown that C can replace B leading to improved  $J_c(B)$  characteristics [175,198].

In §3.3 it was already suggested that the overall increase in  $B_{100}$  values over 2003 to 2006 can to a large extent be attributed to additions, specifically in in-situ conductors. Below, a statistical analysis is performed to demonstrate that additions indeed tend to behave fundamentally different in in- or ex-situ conductors and to clarify the role of connectivity, pinning and  $B_{c2}$  enhancements in these trends.

Figures 3.8a to c show how  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  are all differently affected by additions in ex-situ and in-situ conductors. In general, additions in ex-situ conductors leave  $B_p$  unchanged, indicating that neither pinning nor  $B_{c2}$  changes. It should be stressed once more, however, that this statistical study only clarifies trends. Indeed, the record  $B_{100}$  and  $B_p$  values ( $\sim 13$  T and  $\sim 3$  T, respectively) at the writing of this thesis are reported in carbon-added ex-situ tapes [8].

The  $B_{100}$  distribution of ex-situ conductors, on the other hand, changes significantly with additions, decreasing by  $\sim 25\%$ . Since  $B_p$  tends to remain constant this is directly related to the significant decrease with  $\sim 25\%$  of the  $\log(J_{c,0}^T)$  distribution. A reasonable explanation for these observation might be that additions in ex-situ conductors tend not to be incorporated inside the  $\text{MgB}_2$  crystallites and thus leave  $B_p$  unchanged. Instead, they remain between the grains, reducing connectivity and thus lowering  $J_{c,0}^T$  and  $B_{100}$ .

The trends are drastically different in the case of in-situ conductors. Here, additions do significantly improve the median value of  $B_p$ , by as much as  $\sim 30\%$ . Thus, in general they clearly improve pinning or increase  $B_{c2}$ . In contrast, the  $\log(J_{c,0}^T)$  distribution of in-situ conductors does not change significantly. This latter observation allows to draw two interesting conclusions: 1) in contrast to the ex-situ process additions in in-situ conductors are easier incorporated inside the  $\text{MgB}_2$  crystallites during formation of the phase; and 2)  $J_{c,0}^T$  is not influenced by the increase in pinning and thus appears to be “purely” determined by connectivity. The observed increase of  $\sim 30\%$  in the  $B_{100}$  distribution is therefore only due to the improved  $B_p$  values.

### 3.4.3 Non-Carbon versus Carbon Based Additions in In-Situ Conductors

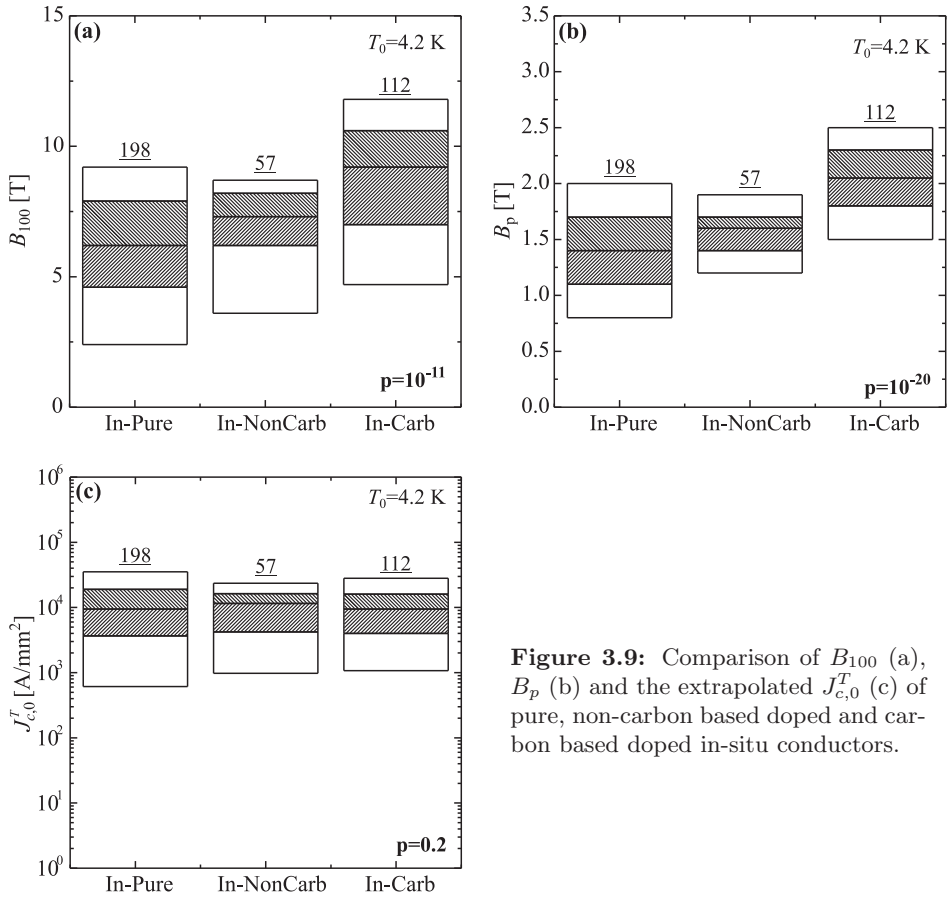
In an attempt to make a possible distinction between improved pinning or enhanced  $B_{c2}$  in in-situ conductors with additions, the statistical analysis is further refined by considering carbon based and non-carbon based additions separately. Figure 3.9a to c show how  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  tend to change when either non-carbon or carbon based additions are added to in-situ conductors.

It is clear that in general both non-carbon and carbon based additions leave  $J_{c,0}^T$  unchanged, again indicating that it is a parameter that is mainly determined by connectivity.

Both types of additions improve  $B_p$  and  $B_{100}$ . However, the median values of the characteristic fields is significantly higher for carbon based additions than for non-carbon based additions ( $\sim 50\%$  and  $\sim 15\%$  respectively). This observation might indicate that carbon based additions improve  $B_{c2}$  and possibly also pinning, while non-carbon based additions improve only pinning. Which pinning mechanism is dominant can not be revealed with this statistical analysis.

## 3.5 Influence of Cross-Sectional Layout

The cross-sectional layout resulting from different deformation routes may also influence  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$ . Not only the filament density and grain-to-grain connectivity are affected by the deformation procedure but flat rolling may also induce texture in the filaments’ microstructure.  $\text{MgB}_2$  is an anisotropic superconductor with different  $B_{c2}$  values depending on the direction of the field with respect to the crystallographic c-axis [191]. Therefore, textured conductors can



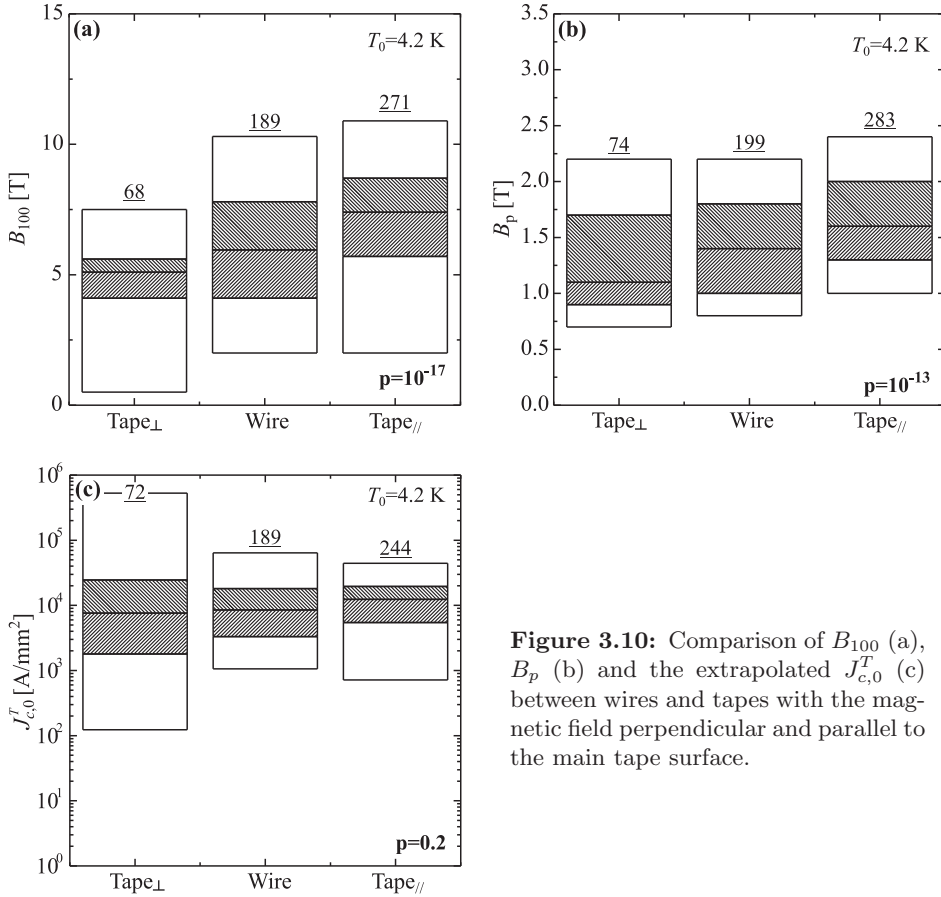
**Figure 3.9:** Comparison of  $B_{100}$  (a),  $B_p$  (b) and the extrapolated  $J_{c,0}^T$  (c) of pure, non-carbon based doped and carbon based doped in-situ conductors.

also have different  $B_p$  and  $B_{100}$  values depending on the field orientation. In §3.5.1 it is shown how the three parameters differ between tapes and wires.

Also, one may expect possible differences between mono- and multi-filament conductors. Multi-filament conductors are usually assembled from already deformed mono-core conductors and filament size is reduced with further deformation. This could lead to a higher  $J_{c,0}^T$  due to a higher filament density. On the other hand, multi-filament conductors have a larger MgB<sub>2</sub>-matrix interface area. This may lead to a lower effective cross-sectional area through chemical reaction during heat treatments. Possible variations of  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  between mono- and multi-filament conductors are the subject of §3.5.2.

### 3.5.1 Tape versus Wire

Figures 3.10a to c show the  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  distributions of wires and tapes. In this comparison no distinction is made with regard to the precursor powder and



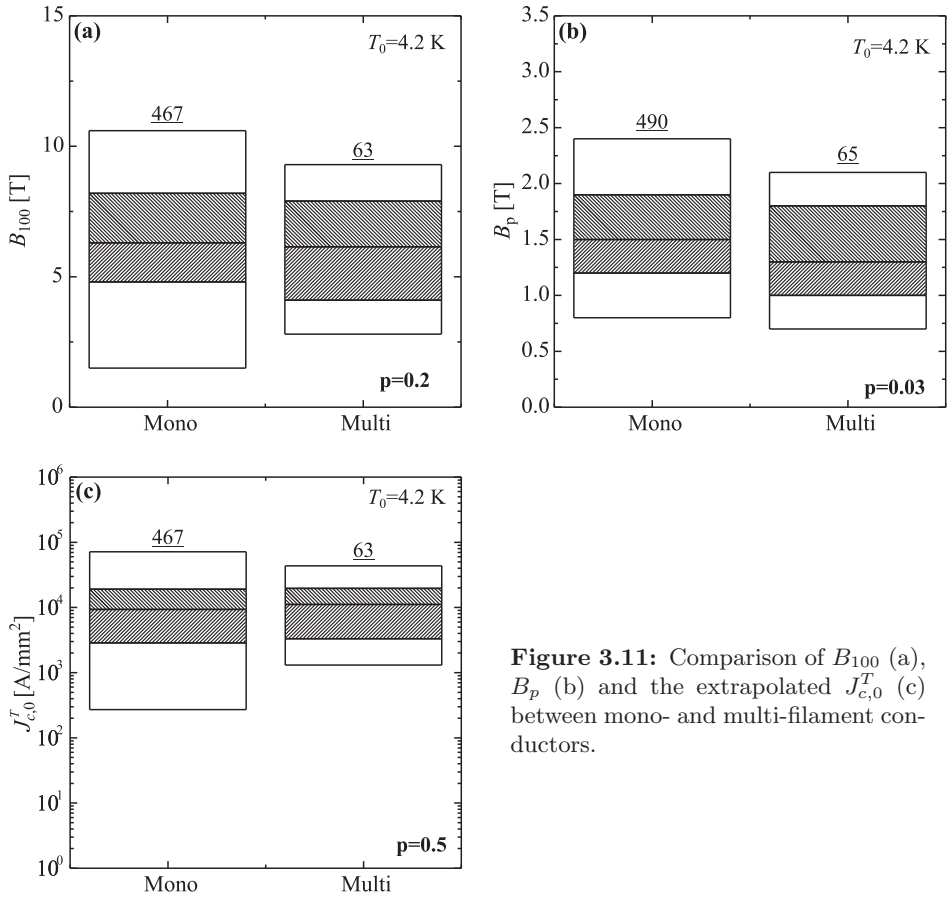
**Figure 3.10:** Comparison of  $B_{100}$  (a),  $B_p$  (b) and the extrapolated  $J_{c,0}^T$  (c) between wires and tapes with the magnetic field perpendicular and parallel to the main tape surface.

the number of filaments.

It is shown in [156] that the filamentary microstructure in flat-rolled tapes exhibits a certain degree of texture. Due to the anisotropy of  $B_{c2}$ ,  $B_p$  values in parallel magnetic field will then differ from those in perpendicular field, depending on the degree of texture. For this reason the data of tapes are separated into  $J_c(B)$  curves measured with the field perpendicular to the main plain of the tape and those measured in parallel field.

Figures 3.10a and b show that the median  $B_p$  and  $B_{100}$  values of tapes measured in parallel field are indeed significantly higher ( $\sim 50\%$ ) than those in perpendicular field. Together with the insignificant difference between the median  $J_{c,0}^T$  values this observation confirms the conclusion of [144] that the direction-dependent  $B_p$  and  $B_{100}$  values are caused by texture through the anisotropy of  $B_{c2}$ . As expected, the  $B_p$  and  $B_{100}$  values of wires fall in-between those of tapes in parallel and perpendicular field, demonstrating that texture in wires can be



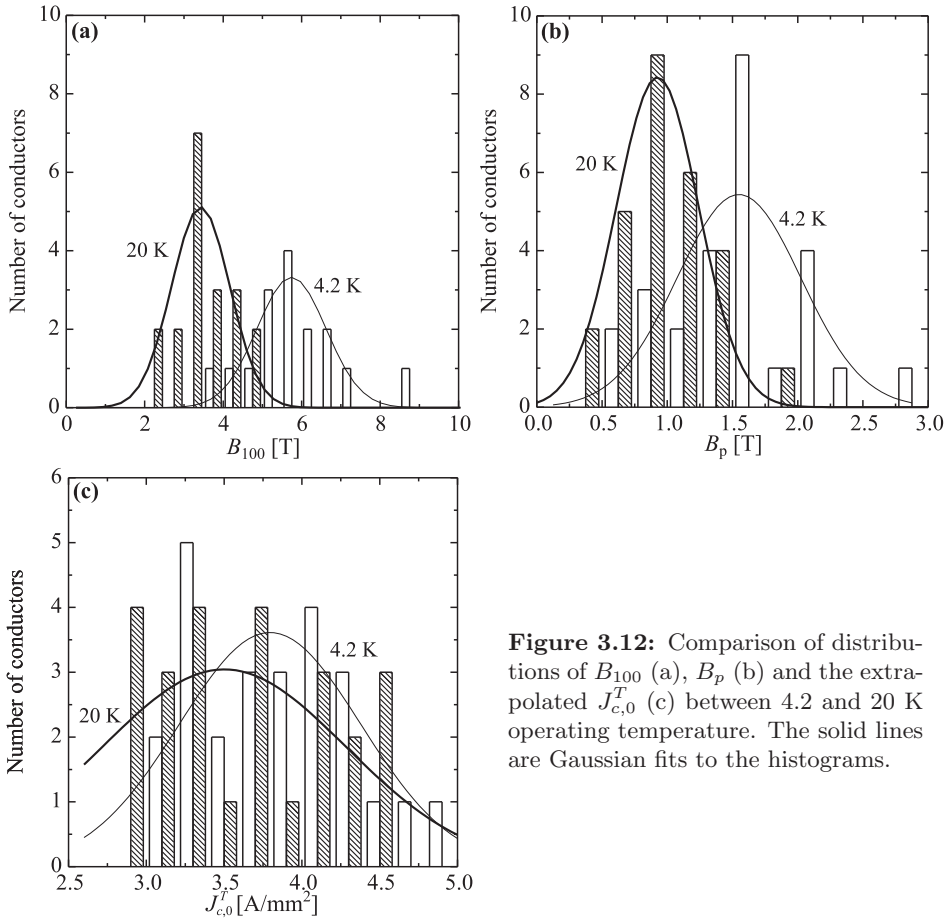


**Figure 3.11:** Comparison of  $B_{100}$  (a),  $B_p$  (b) and the extrapolated  $J_{c,0}^T$  (c) between mono- and multi-filament conductors.

neglected. Also note the absence of a significant difference in  $J_{c,0}^T$  between wires and tapes, which implies that the grain-to-grain connectivity is similar in both.

### 3.5.2 Mono versus Multi-Filaments Conductors

A comparison of  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  between mono- and multi-filaments conductors is presented in figures 3.11a to c. Only the median value of  $B_p$  of multifilamentary conductors is slightly lower than that of the mono-filament conductors. However, the significance of this difference is rather low since the p-value is close to the threshold of 5 %. The absence of major differences between the  $J_c(B)$  behaviour of mono- and multi-filamentary conductors is encouraging, since it indicates that further reduction in filament size should be possible. Note that more detailed statistical analysis in terms of filament size and number was not possible, due to an insufficient number of samples.



**Figure 3.12:** Comparison of distributions of  $B_{100}$  (a),  $B_p$  (b) and the extrapolated  $J_{c,0}^T$  (c) between 4.2 and 20 K operating temperature. The solid lines are Gaussian fits to the histograms.

### 3.6 Influence of Temperature

As mentioned in §3.2, only  $\sim 5\%$  of the 550 conductors in this survey were characterised at 20 K. Because data at this temperature is available of only 30 conductors, no statistical study was performed of possible performance differences between in- and ex-situ conductors or of the effect of low level doping at 20 K. Clearly, since 20 K is the anticipated operating temperature of several MgB<sub>2</sub>-based applications, more studies are needed at this temperature.

Nevertheless, the available data of these 30 conductors can be used to see what the effect of temperature is on the values of  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$ , regardless of precursor powder and deformation route. The distribution functions of these parameters are compared between 4.2 and 20 K in figure 3.12. For both characteristic fields, a clear shift towards lower values can be observed. On average, the ratio of  $B_{100}$  at 20 and 4.2 K is  $\langle B_{100}(20\text{ K})/B_{100}(4.2\text{ K}) \rangle = 0.45 \pm 0.21$ , with the indicated uncertainty the standard deviation of the ratios of individual conduct-

ors. Similar values are found for  $B_p$  with  $\langle B_p(20 \text{ K})/B_p(4.2 \text{ K}) \rangle = 0.50 \pm 0.25$  and  $J_{c,0}^T$  with  $\langle J_{c,0}^T(20 \text{ K})/J_{c,0}^T(4.2 \text{ K}) \rangle = 0.61 \pm 0.40$ . These ratios are in reasonable agreement with the analytical approximation of the critical surface (3.1). As a rule of thumb, it can be stated that the performance both in terms of field and current decreases by  $\sim 50 \%$  between 4.2 and 20 K. Note that the conductor with the highest  $B_{100}$  value of 13 T at 4.2 K found in the literature survey may thus be expected to have a technical upper field limit of  $\sim 6 - 7$  T at 20 K.

## 3.7 Conclusion

To study trends and prospects in the development of practical MgB<sub>2</sub> conductors, a statistical study of the transport  $J_c(B)$  behaviour of published conductors over the years 2003 to 2006 was conducted.

With respect to the self-field  $J_c$ , it is found that the top 50 % of published values fall in a relatively small range of  $10^4$  to  $10^5$  A/mm<sup>2</sup>. Moreover, self-field  $J_c$  values do not correlate with the pinning-related parameter  $B_p$ , so that  $J_{c,0}^T$  appears to be mainly determined by grain-to-grain connectivity within the filaments. Together, these two observations can indicate that  $10^5$  A/mm<sup>2</sup> forms an upper limit imposed by grain-to-grain connectivity.

The progress made over the years is mainly in the field retention of  $J_c$ . This progress is gauged by the magnetic field at which  $J_c = 100$  A/mm<sup>2</sup>, which has increased by about 25 %. Related with this is the observation that the focus of research has shifted from ex-situ to in-situ conductors, of which a substantial part contain additions that improve the field retention of  $J_c$ .

In general, carbon additions in in-situ conductors improve the field retention of  $J_c$  by about 50 %. The mechanism behind this improvement seems to be an enhancement of  $B_{c2}$ , but possibly also enhanced pinning. Non-carbon based additions tend to improve the field retention to a lesser extend, by about 15 %. It is suggested that only pinning is affected in this case.

In contrast, additions in ex-situ conductors tend not to influence the field retention, but instead worsen the grain-to-grain connectivity by about 25 %.

Differences between wires and tapes can be explained with the textured nature of the filaments in the latter. As to connectivity, no significant differences are found.

In view of further development of multi-filament conductors, no significant differences were found between mono-filament conductors and the relatively small number of multifilamentary wires and tapes available at present.

For the operation of MgB<sub>2</sub> at 20 K, it is shown that the  $J_c(B,T)$  performance decreases by roughly 50 % in terms of both field and current compared to 4.2 K.

Overall, the main progress from 2003 to 2006 has been obtained with in-situ conductors, in which low-level doping seems to be better controllable. It should be noted that all conclusions mentioned above concern the ‘‘average’’ behaviour of MgB<sub>2</sub> conductors: an important exception is formed by the record  $B_{100}$  value of 13 T at 4.2 K, which was reported for an ex-situ conductor. In contrast to the

self-field  $J_c$  value, an upper limit in the field retention seems not to be reached yet and realistic predictions are difficult due to the ongoing progress and the lack of understanding of the precise mechanism behind improvements obtained with carbon-based dopants. Further insights in this area may well lead to further enhancement of the field retention.

## Chapter 4

# Normal Zone Development

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*The occurrence of a normal conducting zone and the following heating effect can cause permanent damage to a superconducting magnet. The probability of such an event and the ability to protect a MgB<sub>2</sub>-based magnet from damage is discussed in this chapter in terms of the minimum quench energy MQE and the normal zone propagation velocity  $v_{nz}$ . Both depend on the conductors thermal properties and on operating conditions such as transport current, magnetic field and temperature. Especially the latter is important for MgB<sub>2</sub> because of its possible use at about 20 K rather than 4.2 K. A new experimental set-up is developed to investigate intrinsic MQE and  $v_{nz}$  values as a function of current, field and temperature. How MQE and  $v_{nz}$  depend on the three operating parameters is discussed. As a tool for magnet and conductor design a new algorithm is developed that takes into account the temperature dependent conductor properties and detailed filament properties. Calculations with the algorithm shows excellent agreement with the measurements.*

## 4.1 Introduction

When a thermal disturbance occurs in a practical superconductor, the temperature rises locally and the critical current drops accordingly. If the temperature rise is large enough, the critical current drops below the transport current. The superconducting filaments cannot carry the full transport current anymore and current is redistributed over filaments and matrix. The conductor now locally generates heat. When the heat is generated faster than it can be dispersed, an “avalanche” effect occurs since temperature rises even further, more current diffuses in the matrix, the normal zone grows and still more heat is generated. Such an avalanche effect may even cause the conductor to burn out, in which case the superconducting magnet will be permanently damaged.

Being able to predict the response of a superconductor to a thermal disturbance is thus very important in order to protect a superconducting device from permanent damage. Three parameters are of importance:

1. the amount of heat that is needed to trigger the avalanche effect, referred to as the “minimum quench energy” MQE. This parameter is a measure for the stability of a conductor.
2. the “normal zone propagation velocity”  $v_{nz}$ , i.e. how fast a normal conducting zone grows along the conductor. A large  $v_{nz}$  has the advantage that heat is distributed over a relatively large volume so that the temperature rise remains limited. Furthermore, a fast-growing normal zone also means that the voltage rise across the conductor is easier to detect and that appropriate measures can be taken faster.
3. the temperature at the position of the thermal disturbance, referred to as the “hot-spot temperature”. This is ultimately the factor that will decide whether permanent damage will occur in a conductor.

These parameters can, to a large extent, be controlled by the choice of the matrix material. MQE, for example, is determined by the volumetric heat capacity of the composite  $c$  (a higher heat capacity means that more heat is required); by the thermal conductivity  $\kappa$  that determines the cooling rate<sup>1</sup>; and by the electrical resistivity  $\rho$  that determines how much heat is generated for a given current density.  $v_{nz}$  is determined by the matrix’ thermal diffusivity (the ratio of  $\kappa$  and  $c$ ) and, just like MQE, the amount of generated heat.

Apart from the matrix’ properties, the properties of the superconducting filaments also play an important role. The critical temperature, for example, determines the thermal margin and thus how far the temperature needs to be raised before current starts to diffuse into the matrix. Furthermore, the average current density in the conductor section determines how much heat is generated.

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<sup>1</sup>In this thesis only the adiabatic case is considered, in which cooling occurs through longitudinal redistribution of heat along the conductor. In most applications, also lateral cooling to neighbouring conductors or to a cryogen plays a role. However, it is the “intrinsic” adiabatic behaviour that characterises a given conductor and that will be the subject of this study.

As mentioned in §1.3.1, the parameters differ quite substantially between low- $T_c$  and high- $T_c$  conductors. Accordingly, their response to a thermal disturbance also differs substantially. In conductors like Nb-Ti and Nb<sub>3</sub>Sn, MQE values are relatively low ( $\sim \mu\text{J}$ ), due to their relatively low critical temperatures ( $\sim 9$  and  $\sim 18$  K for Nb-Ti and Nb<sub>3</sub>Sn respectively) and the fact that their heat capacity at the usual operating temperature (4.2 K) is relatively low. As a consequence, a relatively small amount of heat is needed to initiate a propagating normal zone. However, since these conductors are usually stabilised with a highly conducting Cu matrix,  $v_{nz}$  values are relatively high (10 to 100 m/s [199,200]), which means that heat is quickly distributed and the temperature rise remains limited. Magnets made of these conductors are relatively easy to protect, since the voltage rises fast. Values of MQE and  $v_{nz}$  for low- $T_c$  conductors can in most cases be accurately predicted with the aid of analytical formulae [28,88,201].

In high- $T_c$  conductors, the situation is reversed. The conductors can be operated at temperatures as high as 77 K, at which the heat capacity is relatively large (roughly 3 orders of magnitude larger than at 4.2 K). Furthermore, their  $T_c$  is relatively high ( $\sim 110$  K for Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub>,  $\sim 90$  K for YBa<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub>). Together, these factors lead to higher MQE values ( $\sim 10$  J [202]). On the other hand, due to the high heat capacity, thermal diffusion is low and the response of a conductor to a thermal disturbance can be approximated by heating in a static normal zone [203]. Consequently, the voltage rise is slow and it is relatively difficult to detect a normal zone in high- $T_c$  conductors.

MgB<sub>2</sub> can be regarded as a medium- $T_c$  conductor. At the envisaged operating temperature of 20 K, its thermal margin and heat capacity are in-between those of low- and high- $T_c$  conductors. Therefore, typical values of MQE and  $v_{nz}$  can be expected to be also in-between those of low- and high- $T_c$  conductors. This would suggest that the response to a thermal disturbance may be approximated with the “classical” analytical formulae for low- $T_c$  conductors. However, as discussed in chapter 2, materials that are chemically compatible with MgB<sub>2</sub> are usually relatively poor electrical and thermal conductors. In contrast to low- $T_c$  conductors, the normal state resistivity of MgB<sub>2</sub> filaments is often comparable to that of the matrix and neglecting heat generation in the filaments (as in the analytical approximations) can not be justified. Accounting for heat generation in the filaments means also that the voltage-current characteristic of the filaments needs to be considered explicitly.

In this chapter, the minimum quench energy and normal zone propagation velocity of MgB<sub>2</sub> conductors with varying matrix materials and  $n$ -values are studied. This is done with the aid of a numerical model that also accounts for the heat generation in the superconducting filaments. To validate the model, experiments are performed on samples with varying cross-sectional layouts (matrix materials and shape).

However, before discussing MQE and  $v_{nz}$  of MgB<sub>2</sub> conductors, in §4.2 the diffusion processes of current- and heat in a composite conductor during and after the occurrence of a thermal disturbance are discussed. The diffusion processes are in principle 3-dimensional, but they will be modeled 1-dimensionally. It will

be discussed under which conditions the 1-dimensional assumption is justified.

Next, to provide a better understanding of the mechanisms determining  $v_{nz}$  and MQE, the analytical formulae that were developed for low- $T_c$  conductors are first discussed in §4.3. Then the details of the numerical model are discussed and it is shown to what extent heat generation in MgB<sub>2</sub> conductors deviates from the “classical” approximations.

Validation of the numerical model is performed with measurements of MQE and  $v_{nz}$  in a quasi-adiabatic set-up. This allows to eliminate extra complications due to lateral cooling terms and also allows to vary the baseline temperature of the conductor straightforwardly. The development of the experimental set-up and measurement procedure are subject of §4.4.

The results of MQE and  $v_{nz}$  measurements as a function of transport current, temperature and magnetic field are presented in §4.5. Results are then compared with the parallel-paths calculations in order to validate the numerical model.

Lastly, the parallel current paths model is used to predict the influence of matrix material and  $n$ -value on MQE,  $v_{nz}$  and hot-spot temperature.

## 4.2 Diffusion Process

As outlined in §4.1, a thermal disturbance in a superconducting composite can be followed by an avalanche effect when heat generation exceeds cooling. The avalanche effect is driven by the diffusion of current- and heat. In this paragraph, the diffusion process is discussed in more detail. In §4.2.1 the diffusion equation of current and heat are presented. Although diffusion is 3-dimensional, stability of a superconducting composite is usually treated as a 1-dimensional problem. This is also the case in this work and in §4.2.2 the circumstances for which this assumption is justified are discussed.

### 4.2.1 Diffusion Equations

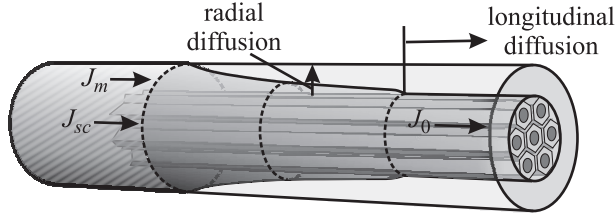
Figure 4.1 shows a composite superconductor in which current diffuses *radially* into the matrix when a thermal avalanche effect is initiated. The net effect of the avalanche is a *longitudinal* propagation of the normal zone. The speed at which the normal zone propagates along the conductor is referred to as the “normal zone propagation velocity”  $v_{nz}$ .

Diffusion of heat is determined by the balance between heat conduction through the composite, the amount of heat that can be stored (thermal inertia), the heat generation and the initial thermal disturbance. The most general form of such a heat balance is:

$$\underbrace{c \frac{\partial T}{\partial t}}_{\text{inertia}} = \underbrace{\nabla(\kappa \nabla T)}_{\text{conduction}} + \underbrace{p_{\text{initial}}}_{\text{thermal dist.}} + \underbrace{p_{\text{diss}}}_{\text{heat diss.}}, \quad (4.1)$$

in which  $c$  is the volumetric heat capacity,  $T$  the temperature,  $t$  the time,  $\kappa$  the thermal conductivity,  $p_{\text{initial}}$  the heat flux associated with the thermal disturb-





**Figure 4.1:** A composite superconductor in which the current diffuses into the matrix due to a thermal disturbance.

ance and  $p_{\text{diss}}$  is the heat dissipation. The heat flux generated by the thermal disturbance can be written as:

$$p_{\text{initial}} = \frac{1}{V} \left( \frac{dQ_{\text{initial}}}{dt} \right), \quad (4.2)$$

with  $Q_{\text{initial}}$  the thermal disturbance and  $V$  the volume in which this heat is deposited.

The source term  $p_{\text{diss}}$  in equation 4.1 results from the diffusion of current throughout the sample. Current diffusion is described by Maxwell equations. For the normal conducting matrix Ohm's law is included in this description, which leads to the following diffusion equation for current:

$$\mu_0 \frac{\partial J_m}{\partial t} = \rho_m \nabla^2 J_m, \quad (4.3)$$

with  $\mu_0$  the permeability of vacuum,  $J_m$  the current density in the matrix and  $\rho_m$  the resistivity of the matrix. For the filaments, which behave non-Ohmic, the current-diffusion equation needs to be modified. Assuming that the voltage-current relation of the superconducting filaments can be described by the power law:

$$E = E_c \left( \frac{J_{sc}}{J_c} \right)^n, \quad (4.4)$$

equation 4.3 becomes:

$$\mu_0 \frac{\partial J_{sc}}{\partial t} = E_c \nabla^2 \left( \frac{J_{sc}}{J_c} \right)^n, \quad (4.5)$$

with  $E$  the electric field,  $E_c$  the electric field at which the critical current density  $J_c$  is defined,  $J_{sc}$  the current density in the superconducting filaments and  $n$  the exponent of the power law.

### 4.2.2 Dimensionality of the Diffusion Processes

Treating the diffusion equations in a superconducting composite 1-dimensionally and using “lumped” (averaged over the radial direction) material properties such as  $\kappa$  and  $c$  assumes that the temperature over the cross-section of the composite is homogeneous. In order to verify whether this is a reasonable assumption, a criterion for 1-dimensional modeling of dynamic stability can be adopted [28], which states that the characteristic thermal diffusion time in the filaments  $\tau_{T,f}$  must be smaller than the characteristic magnetic diffusion time in the composite  $\tau_{m,c}$ :

$$\tau_{T,f} < \tau_{m,c}. \quad (4.6)$$

This requirement is based on the temperature dependence of  $J_c$  in (4.5). When (4.6) is not fulfilled, radial temperature gradients across the filaments are not negligible so that  $J_c$  and the heat generation become radially inhomogeneous.

To verify whether (4.6) is satisfied, diffusion equations (4.1) and (4.3) are solved in radial direction assuming cylindrical coordinates. Furthermore, the material properties  $c$  and  $\kappa$  are assumed to be temperature independent. The solutions then have the form:

$$T(r, t) = \sum_{l=1}^{\infty} T_l(\gamma_{1,l} \frac{r}{R}) \exp\left(-\frac{\gamma_{1,l}^2 D_T}{R^2} t\right) \quad \text{with} \quad D_T = \frac{\kappa}{c}; \quad (4.7)$$

$$J(r, t) = \sum_{l=1}^{\infty} J_l(\gamma_{1,l} \frac{r}{R}) \exp\left(-\frac{\gamma_{1,l}^2 D_m}{R^2} t\right) \quad \text{with} \quad D_m = \frac{\rho}{\mu_0}, \quad (4.8)$$

where  $T_l(\gamma_{1,l}(r/R))$  and  $J_l(\gamma_{1,l}(r/R))$  are multiples of the zeroth order Bessel function;  $\gamma_{1,l}$  is the  $l^{\text{th}}$  root of the first order Bessel function of the first kind [204]; and  $R$  is the radius of the system under consideration. Decay time constants are associated with the time-dependent exponential factors in (4.7) and (4.8). The slowest decay ( $l = 1$ ) determines the overall characteristic diffusion time  $\tau$ . Thus the characteristic times of thermal diffusion in the filaments and magnetic diffusion in the composite are:

$$\tau_{T,f} = \frac{R_f^2}{\gamma_{1,1} D_{T,f}}; \quad (4.9)$$

$$\tau_{m,c} = \frac{R_c^2}{\gamma_{1,1} D_{m,c}}, \quad (4.10)$$

where  $R_f$  and  $R_c$  are the radii of the filament and composite, respectively. With these characteristic diffusion times, requirement (4.6) becomes:

$$\left(\frac{R_f}{R_c}\right)^2 < \frac{D_{T,f}}{D_{m,c}}. \quad (4.11)$$

Whether or not a 1-dimensional approximation is justified for a given conductor can now be judged straightforwardly. Suppose two monofilament MgB<sub>2</sub> conductors that have  $R_c = 0.5$  mm and  $R_f = 0.3$  mm (filling factor  $f=36$  %) and that operate at 20 K. Conductor 1 is stabilised with a Fe matrix while conductor 2 is stabilised with Cu. In appendix B the thermal and electrical properties of relevant materials are presented. The electrical resistivities for conductor 1 are  $\rho_{\text{Fe}} = 7 \cdot 10^{-9}$   $\Omega\text{m}$  and  $\rho_{\text{MgB}_2} = 64 \cdot 10^{-9}$   $\Omega\text{m}$ , so that its lumped electrical resistivity becomes  $\rho_{\text{lumped}} = 10 \cdot 10^{-9}$   $\Omega\text{m}$ . The volumetric heat capacity and thermal conductivity of MgB<sub>2</sub> at 20 K are  $c_{\text{MgB}_2} = 3892$  J/m<sup>3</sup>K and  $\kappa_{\text{MgB}_2} = 5$  W/mK. This leads to:

$$\frac{D_{T,f}}{D_{m,c}} = 0.16,$$

while the ratio of filament and composite radii is:

$$\left(\frac{R_f}{R_c}\right)^2 = 0.36.$$

Clearly, the requirement is not met with this combination of materials and filling factor and a significant radial temperature gradient is present. For conductor 2, which has a Cu matrix with a resistivity of  $\rho_{\text{Cu}} = 0.2 \cdot 10^{-9}$   $\Omega\text{m}$  (at  $B = 0$  T and a residual resistivity ratio RRR=100), the criterion is clearly met since the lumped electrical resistance is reduced to  $\rho_{\text{lumped}} = 0.3 \cdot 10^{-9}$   $\Omega\text{m}$ . The ratio of thermal and magnetic diffusivities now becomes:

$$\frac{D_{T,f}}{D_{m,c}} = 5.8,$$

while the ratio of filament and composite radii is not changed.

In this example the matrix resistivity is the decisive factor for the fulfilment of requirement (4.6). However, reducing the radius of the filament (left hand side of inequality (4.11)) can also help to justify the 1-dimensional assumption.

It should be noted that this analysis is rather crude in the sense that it neglects that filaments are for some time during the diffusion in the superconducting state. Current diffusion is then described by equation (4.5) and the diffusion “constant” can no longer simply be written as  $D_m = \rho/\mu_0$ , but effectively depends on the current density itself. Since equation (4.5) is non-linear, a full validation of the 1-dimensional approximation requires a 2-dimensional numerical model that also accounts for the superconducting state of the filaments.

### 4.3 Minimum Quench Energy and Normal Zone Propagation

As discussed in §4.2.1, a normal conducting zone in which the current is distributed over both filaments and matrix can only continue to exist if its initial volume

–induced by  $Q_{\text{initial}}$ – is large enough so that the total heat generation is larger than the cooling. The minimum value of  $Q_{\text{initial}}$  required to initiate such a zone is the MQE. When  $Q_{\text{initial}} \geq \text{MQE}$ , an “avalanche” effect occurs since the imbalance between heat generation and cooling increases the temperature even further and the normal zone grows with a velocity  $v_{nz}$ .

In §4.3.1 a straightforward analytical model that provides a clear understanding of MQE and the  $v_{nz}$  is discussed.

The simple model assumes temperature- and field independent material properties and an abrupt diffusion of all the transport current from the filaments into the matrix. These are rather crude assumptions that can lead to a significant disagreement with the measurements, especially in the case of  $\text{MgB}_2$ , where a smooth transition from lossless transport to Ohmic heat generation can occur. Furthermore, the large temperature margin in  $\text{MgB}_2$  ( $T_c \sim 40$  K) requires that one takes into account the temperature dependence of the relevant material properties. Therefore, a 1-dimensional numerical model was developed that accounts for: 1) the continuous temperature dependence of  $I_c$ ; 2) the detailed voltage-current transition of the filaments; and 3) the temperature- and field dependence of the material properties. The details of the refinements are discussed in §4.3.2, while their implications on the heat generation, MQE and  $v_{nz}$  are presented in §4.3.3.

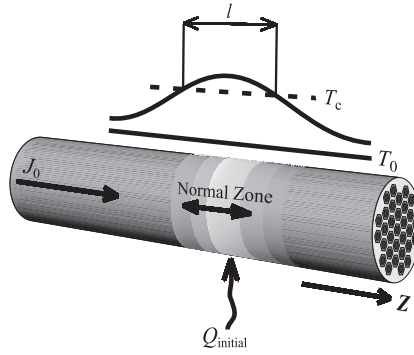
### 4.3.1 Analytical Description

#### Minimum Quench Energy

An instructive analytical approximation for the magnitude of MQE was presented by Wilson [28]. The basis of this analytical description is that for a normal zone to propagate,  $Q_{\text{initial}}$  must be large enough so that: 1) the temperature increases above  $T_c$ <sup>2</sup>, all the current  $I_0$  flows in the matrix (with current density  $J_m = I_0/A_m$ ) and Ohmic heat is generated ( $p_{\text{diss}} = J_m^2 \rho_m$ ); and 2) the length of this region must be large enough so that the total Ohmic heat generation integrated over the entire length exceeds the cooling<sup>3</sup>. Figure 4.2 schematically shows a conductor that initially carries a current density  $J_0$  in the filaments and in which a thermal disturbance  $Q_{\text{initial}}$  induces a temperature rise above  $T_c$  over a length  $l$ . If this length is too short, the heat will simply diffuse and the temperature will drop again below  $T_c$ . The minimum length that is needed for this normal zone to propagate (for heat generation to exceed cooling) is defined as the “minimum propagation zone”  $l_{\text{MPZ}}$ . Equating Ohmic heat generation and

<sup>2</sup>The choice of  $T_c$  as the temperature at which current “switches” from the filaments to the matrix [205] is rather arbitrary. Other possible choices are discussed in §4.3.3.

<sup>3</sup>These assumptions ( $T > T_c$  and sharp current redistribution) are rather crude and will be refined later (§4.3.2). However, they provide a clear understanding about the physics without complicating the issue with second order effects.



**Figure 4.2:** A normal zone that is created in a current-carrying wire by a small thermal disturbance.  $l$  indicates the length where the temperature exceeds  $T_c$ , and heat is generated.

cooling,  $l_{\text{MPZ}}$  can be written as:

$$J_m^2 \rho_m A_m l_{\text{MPZ}} = \frac{2\kappa_m A_m (T_c - T_0)}{l_{\text{MPZ}}} \Leftrightarrow l_{\text{MPZ}} = \left\{ \frac{2\kappa_m (T_c - T_0)}{J_m^2 \rho_m} \right\}^{1/2}, \quad (4.12)$$

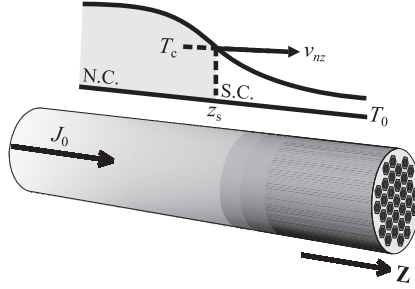
with  $\rho_m$  the resistivity of the matrix;  $\kappa_m$  the thermal conductivity of the matrix; and  $A_m$  the cross-sectional area of the matrix. Once  $l_{\text{MPZ}}$  is known, MQE becomes simply the heat needed to raise the temperature above  $T_c$  over a length  $l_{\text{MPZ}}$ :

$$\text{MQE} = A \int_{l_{\text{MPZ}}} \int_{T_0}^{T_c} c(T) dT dz, \quad (4.13)$$

with  $A$  the cross-section of the whole conductor.

### Normal Zone Propagation Velocity

Once a normal zone is established that propagates along the conductor with a velocity  $v_{nz}$ , two regions can be distinguished: 1) the normal conducting part of the wire ( $T > T_c$ ) in which Ohmic heat is generated; and 2) the part of the wire that is still superconducting ( $T < T_c$ ) but which drains heat from the normal conducting zone (see figure 4.3). The two regions are separated by a moving interface at  $z_s$  where the temperature is equal to  $T_c$  and current  $I_0$  is assumed to diffuse instantly into the matrix. Following Iwasa [47], an expression for  $v_{nz}$  can be derived by formulating two separate diffusion equations for the normal- and



**Figure 4.3:** A current carrying conductor in which a normal zone propagates with velocity  $v_{nz}$ .  $T_s$  is the transition temperature above which heat is generated and  $z_s$  indicates the position of the normal-superconducting interface.

superconducting zone, which are coupled by the heat flow across the interface:

$$\begin{aligned} c_n \frac{\partial T}{\partial t} &= \frac{\partial}{\partial z} \left( \kappa_n \frac{\partial T}{\partial z} \right) + p_{\text{diss}} \quad (\text{n.c.}); \\ c_s \frac{\partial T}{\partial t} &= \frac{\partial}{\partial z} \left( \kappa_s \frac{\partial T}{\partial z} \right) \quad (\text{s.c.}), \end{aligned} \quad (4.14)$$

where the indices  $n$  and  $s$  of  $c$  and  $\kappa$  refer to the volumetric heat capacity and thermal conductivity in the normal- and superconducting state, respectively;  $p_{\text{diss}}$  is the Ohmic heat generation.

The two heat balance equations (4.14) are solved in a moving frame of reference that follows the normal-superconducting interface ( $z_s$ ) with a velocity  $v_{nz}$  in the  $+z$  direction. Thus, a coordinate transformation  $z' = z - z_s = z - v_{nz}t$  is applied to (4.14). Furthermore, only the area adjacent to the interface needs to be considered, so that the material properties  $c$  and  $\kappa$  can be assumed constant. This leaves the following ordinary differential equations:

$$\begin{aligned} v_{nz} c_n \frac{dT}{dz'} + \kappa_n \frac{d^2 T}{dz'^2} + p_{\text{diss}} &= 0 \quad (\text{n.c.}); \\ v_{nz} c_s \frac{dT}{dz'} + \kappa_s \frac{d^2 T}{dz'^2} &= 0 \quad (\text{s.c.}). \end{aligned} \quad (4.15)$$

Using the boundary conditions that the heat flux across the interface is continuous and that the temperature at the interface equals  $T_c$ , the two heat balance equations leave the following expression for  $v_{nz}$ :

$$v_{nz} = \left\{ \frac{p_{\text{diss}} \kappa_n}{c_n c_s (T_c - T_0)} \right\}^{1/2} = \left\{ D_{T,n} \frac{p_{\text{diss}}}{c_s (T_c - T_0)} \right\}^{1/2}, \quad (4.16)$$

with  $D_{T,n}$  the thermal diffusion constant of the normal conducting zone.

Thus  $v_{nz}$  is determined by  $p_{\text{diss}}$ , the rate of Ohmic heat generation in the normal zone; by  $D_{T,n}$ , the speed of heat diffusion from the normal zone into the superconducting zone; and by  $c_s(T_c - T_0)$  the amount of heat required to maintain the temperature gradient in the superconducting region.

It should be noted that most authors ([28, 201]) use one single value of  $c$  and  $\kappa$  for the superconducting and normal conducting regions, i.e.  $c_n = c_s = c$  and  $\kappa_n = \kappa_s = \kappa_m$ , with  $\kappa_m$  the matrix' thermal conductivity. Using the Wiedemann-Franz law  $\rho_m \kappa_m = L_0 T_c$  (4.16) becomes:

$$v_{nz} = \frac{J_m}{c} \left\{ \frac{\rho_m \kappa_m}{(T_c - T_0)} \right\}^{1/2} = \frac{J_m}{c} \left\{ \frac{L_0 T_c}{(T_c - T_0)} \right\}^{1/2}, \quad (4.17)$$

where the Lorentz number  $L_0 = 2.45 \cdot 10^{-8} \text{ W}\Omega/\text{K}^2$ .

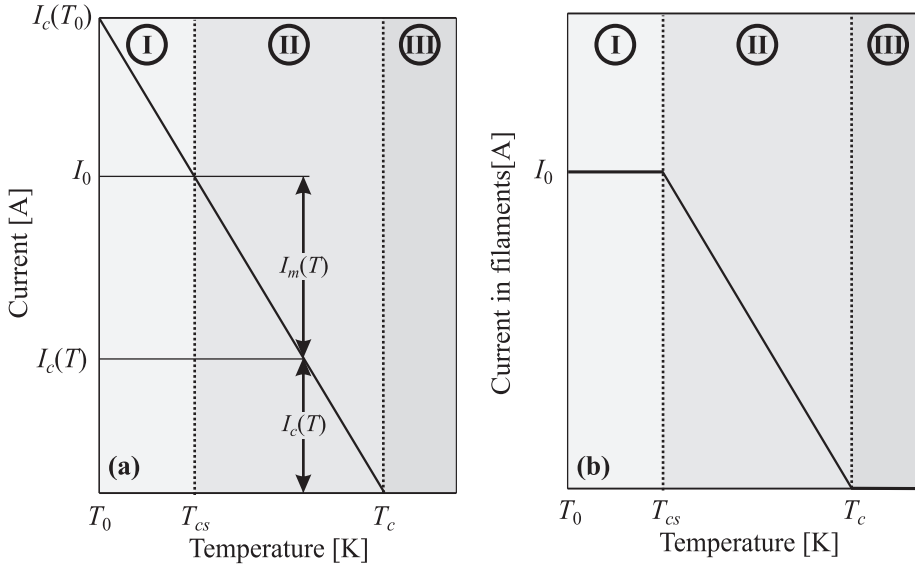
### 4.3.2 Numerical Models

Although equations (4.13) and (4.16) provide a good qualitative insight in the parameters that influence MQE and  $v_{nz}$ , serious simplifications were applied in their derivation that can lead to a quantitative discrepancy with the measured values. Arguably the most serious simplification is the assumption that the complete transport current  $I_0$  instantly diffuses into the matrix as soon as the temperature reaches  $T_c$ . This assumption disregards the fact that  $I_c$  is a continuous function of the temperature (that  $I_c$  does not become infinite at  $T < T_c$ ) and also that the voltage-current transition has a finite  $n$ -value. Significant deviation between the model and the measurements may result, especially in the case of  $\text{MgB}_2$  that exhibits relatively low  $n$ -values at high magnetic field. Furthermore, when accounting for the continuous  $I_c(T)$  function and a finite  $n$ -value, the temperature dependence of the heat capacity, thermal conductivity and the electrical resistivity must be included.

To include all these effects, a 1-dimensional numerical model was developed, based on the explicit finite difference scheme of equation (4.1)<sup>4</sup>. In the remainder of this paragraph, several refinements are discussed. As a first refinement step the continuous temperature dependence of  $I_c$  is implemented in the numerical model, which leads to the concept of ‘‘current sharing’’ between filaments and matrix [207]. As a second refinement step, the voltage-current relation of the filaments is included in the model by considering filaments and matrix as an electrical parallel circuit. As a last step, the field- and temperature dependence of the thermal- and electrical properties are taken into account. The actual implications of the refinements on MQE and  $v_{nz}$  are discussed separately in §4.3.3.

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<sup>4</sup>For a thorough discussion on numerical finite difference solutions of partial differential equations, see for example [206]. The details of this particular numerical model are discussed in [199, 200].



**Figure 4.4:** (a) shows the  $I_c(T)$  line of  $\text{MgB}_2$ . Due to a thermal disturbance  $I_c(T)$  drops below the transport current  $I_0$ . As a consequence,  $I_0$  is shared between the matrix, carrying a current  $I_m$  and the filaments that carry a current  $I_c(T)$ . (b) shows how the current in the filaments decreases with increasing temperature. Region (I) indicates the region where  $I_0$  is transported lossless by the filaments; (II) indicates the region where current is shared between the matrix and filaments; and (III) the region where all the transport current flows in the matrix.

### Current Sharing Model

When a conductor is operating at its nominal temperature  $T_0$  –with a critical current  $I_c(T_0)$ –, the transport current  $I_0$  is carried lossless by the filaments (region (I) in figures 4.4a and b). If the temperature of the conductor is raised by a thermal disturbance  $I_c$  will drop, following the  $I_c(T)$  line in figure 4.4a. At the current sharing temperature  $T_{cs}$ , the critical current equals the transport current  $I_0$ . When this temperature is exceeded (region (II)), the filaments cannot carry all the transport current anymore. Instead, current is shared between the filaments and matrix. The filaments continue to carry a part  $I_c(T)$  of the current lossless, while the excess current  $I_m(T) = I_0 - I_c(T)$  is carried Ohmic by the matrix. At the moment where the temperature exceeds  $T_c$ ,  $I_c$  has become zero and the transport current is carried only by the matrix.

It has to be stressed that in this current sharing model only the excess current  $I_m(T)$  generates Ohmic heat. This assumption is reasonable as long as a conductor has a sharp voltage-current transition and when the electrical resistivity of its matrix is much lower than the normal state resistivity of its filaments. This is the case for Nb-Ti and  $\text{Nb}_3\text{Sn}$  conductors that usually have high  $n$ -values and a low



resistive Cu matrix. Accordingly, this straightforward current sharing concept works very well for these materials [208], but not necessarily for MgB<sub>2</sub>.

### Parallel-Paths Model

Presently some MgB<sub>2</sub> conductors suffer from relatively low  $n$ -values. Furthermore, since MgB<sub>2</sub> reacts strongly with low-resistive materials such as Cu or Al other metals are commonly used with a resistivity that is often similar to that of the filaments. The heat generation in the partly normal conducting filaments can therefore not be neglected anymore. To account for this, the current sharing concept described above is further refined by including also the voltage current relation of the superconducting filaments:

$$E = E_c \left( \frac{J_{sc}}{J_c} \right)^n. \quad (4.4)$$

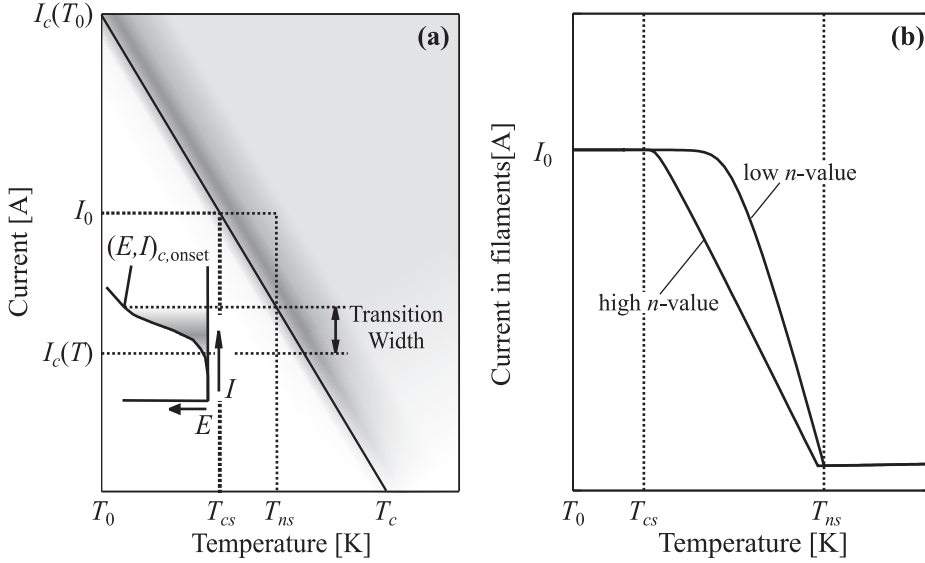
$E_c$  is the voltage criterium at which the critical current density  $J_c$  is defined and  $J_{sc}$  is the current density in the superconductor. Furthermore, an electric field  $E_{c,onset}$  and current density  $J_{c,onset}(= I_{c,onset}/A_{sc})$  are defined at which dissipation in the normal- and superconducting state of the filaments become equal:

$$E_{c,onset} = E_c \left( \frac{J_{c,onset}}{J_c} \right)^n = \rho_{sc} J_{c,onset}, \quad (4.18)$$

with  $\rho_{sc}$  the normal-state resistivity of the filaments. The temperature at which  $I_{c,onset} = I_0$  is defined as the current dependent normal state temperature  $T_{ns}$ .

Figure 4.5a shows the  $I_c(T)$  line on which the voltage-current transition (4.4), (4.18) is projected, while figure 4.5b shows how the current in the filaments reduces with increasing temperature in the case of a low- and a high  $n$ -value. Considering a conductor with a high  $n$ -value, the voltage-current relation is sharp and the current in the filaments starts to decrease when  $T \approx T_{cs}$ , following the  $I_c(T)$  line, just like in the current sharing model. However it should be stressed that with this model, the current in the filaments *does* generate heat, which was not the case in the simpler current sharing model. When the temperature has reached  $T_{ns}$ , the filaments switch to Ohmic behaviour (4.18). Furthermore, due to the finite resistance of the filaments, the current in the filaments does not reduce to zero when  $T > T_{ns}$  but continues to be distributed between the filaments and matrix. In a conductor with a low  $n$ -value, on the other hand, the relatively “smooth” voltage-current transition causes most of the current  $I_0$  to be carried by the filaments up to a temperature that is significantly higher than  $T_{cs}$ .

Overall, by including the voltage-current relation and in the case that  $\rho_{sc} \sim \rho_m$ , the clear boundaries (I)-(II) and (II)-(III) in the current sharing model (figure 4.4a and b) become poorly defined.



**Figure 4.5:** (a) shows the  $I_c(T)$  line of  $\text{MgB}_2$ . The shaded area around the  $I_c(T)$  line represents the transition width (it is the voltage-current transition projected on the  $I_c(T)$  line (**inset**)). (b) shows how the current in the filament reduces with increasing temperature in the case of a low- and a high- $n$ -value.

The detailed distribution of  $I_0$  during the transition from lossless to Ohmic current transport is defined by considering the filaments and matrix as parallel-paths:

$$J_0 = \begin{cases} (1-f) \frac{E}{\rho_m} + f \left[ J_c \left( \frac{E}{E_c} \right)^{1/n} \right] & E < E_{c,\text{onset}}; \\ (1-f) \frac{E}{\rho_m} + f \frac{E}{\rho_{sc}} & E > E_{c,\text{onset}}, \end{cases} \quad (4.19)$$

where  $f$  is the superconductor filling factor,  $\rho_m$  is the electrical resistivity of the matrix and  $\rho_{sc}$  is the normal state resistivity of the superconductor. The accompanying ohmic heat generation term becomes:

$$p_{\text{diss}} = EJ_0. \quad (4.20)$$

A comparison of the heat generation terms in the parallel-paths model (4.20); the current sharing model; and the analytical approximation as well as the implications for MQE and  $v_{nz}$  are discussed in §4.3.3.

### Temperature- and Field Dependence of the Thermal- and Electrical Properties

The heating function (4.19), (4.20) requires knowledge of the temperature- and field dependence of  $J_c$  as well as the  $n$ -value. Therefore equations (3.1) and (4.21) are used to describe the  $J_c(B,T)$  and  $n(B,T)$  surfaces [94]:

$$\frac{J_c(B,T)}{J_{c,0}} = \left(1 - \frac{T}{T_{c,0}}\right) \exp \left[ -\frac{B}{B_{p,0} \left(1 - \frac{T}{T_c}\right)} \right]; \quad (3.1)$$

$$\frac{n(B,T)}{n_0} = \left(1 - \frac{T}{T_{c,0}}\right) \exp \left[ -\frac{B}{B_{n,0} \left(1 - \frac{T}{T_c}\right)} \right]. \quad (4.21)$$

The transition from lossless- to Ohmic current transport can become a very gradual function of temperature, especially at low  $n$ -values and highly resistive matrices. Therefore the temperature dependence of the material properties  $c$ ,  $\kappa$  and  $\rho$  must be accounted for. Indeed, these material properties can be strong functions of the temperature. For example, the electrical- and thermal conductivities in pure metals such as Cu undergo a transition from electron-impurity scattering to electron-phonon scattering. This is reflected in a strong deflection of their temperature dependence below 50 K<sup>5</sup>. Furthermore  $c$ ,  $\kappa$  and  $\rho$  can be a strong function of the magnetic field as well, not only in the superconducting filaments but also in the metallic matrix materials.

### Output of the Numerical Model

The principle output of the numerical model is the time evolution of the temperature profile along the conductor. Two examples of such profiles are plotted in figures 4.6a and b. Figure 4.6a shows how the normal zone collapses in time when the thermal disturbance  $Q_{\text{initial}}$  is lower than MQE. Figure 4.6b shows the opposite case, in which the normal zone propagates along the conductor.

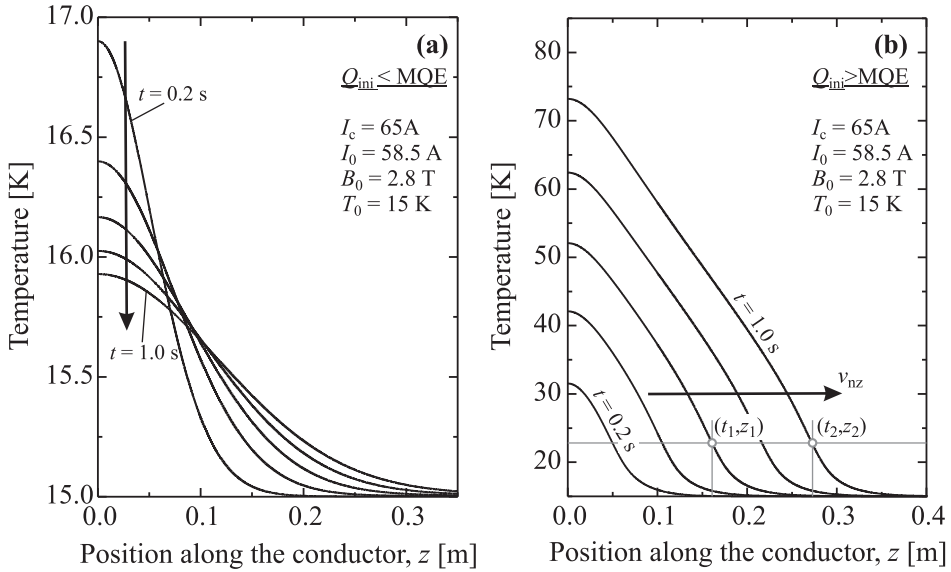
Similar to the actual experiment (§4.4.5), the value of MQE is found by varying  $Q_{\text{initial}}$  and searching for the boundary between a collapsing and a propagating normal zone. The search routine is truncated when the difference between the values of  $Q_{\text{initial}}$  at a propagating- and collapsing normal zone is smaller than  $\sim 1\%$ .

Once the normal zone is propagating (figure 4.6b), the numerical code calculates  $v_{nz}$  following:

$$v_{nz} = \frac{z_2 - z_1}{t_2 - t_1}. \quad (4.22)$$

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<sup>5</sup>In fact, for pure metals the Wiedemann-Franz law used in equation (4.17) is not valid in the temperature range from 10 to 250 K due to the different effect of the electron-phonon scattering on  $\rho$  and  $\kappa$  ([209]).



**Figure 4.6:** Calculated time evolution of the temperature profile along sample HTR1 (see table 4.1) for (a) a collapsing normal zone and (b) a propagating normal zone.  $(t_1, z_1)$  and  $(t_2, z_2)$  are the time-space coordinates from which  $v_{nz}$  is determined using equation (4.22). MQE of this sample 10 mJ

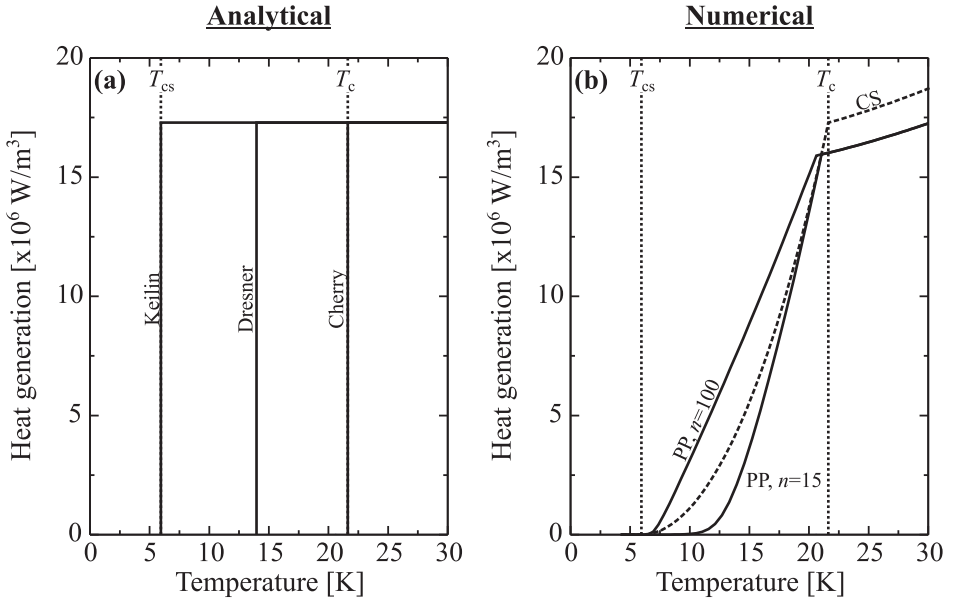
The position on the temperature profile at which  $z_1$  and  $z_2$  are determined is the average of the “classical”  $T_{cs}$  and the temperature  $T_{ns}$  at which  $I_{c,onset}$  meets the transport current  $I_0$ .

### 4.3.3 Comparison Between the Models

The main refinement of the parallel-paths model with respect to the current sharing model and to the analytical approximations (4.12) and (4.16) is that the filaments can also generate heat. Furthermore, compared to the analytical approximations, the current sharing- and parallel-paths models account for the temperature- and field dependence of the material properties ( $c$ ,  $\kappa$  and  $\rho$ ). The refinements mainly influence the heat generation during a transition. In this paragraph it will be shown how the heat generation varies from one model to another and how this influences the prediction of MQE and  $v_{nz}$ .

#### Heat Generation

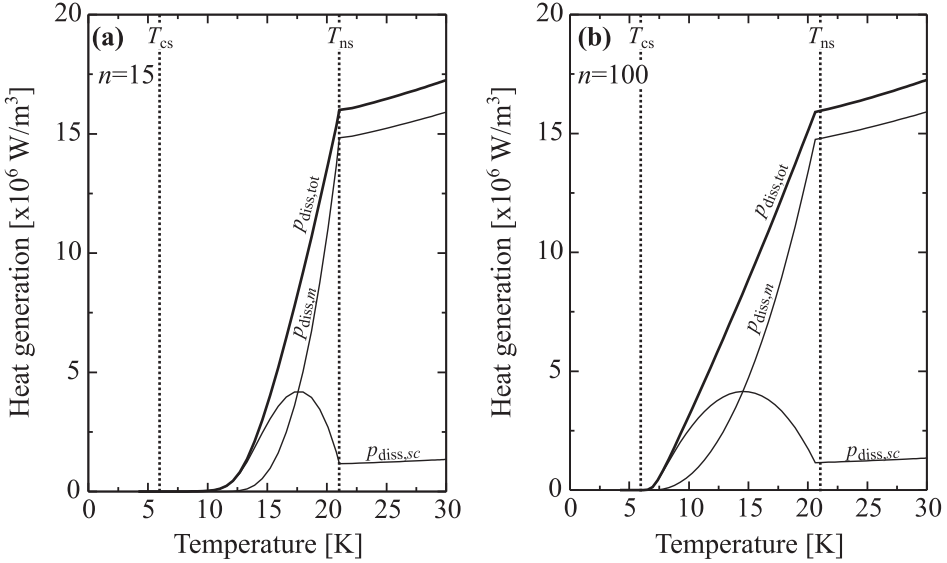
Figure 4.7a shows the heat generation in the analytical approximations (4.12) and (4.16) as a function of temperature. The approximations are represented by three step-like heating functions that are labeled by the authors that proposed them



**Figure 4.7:** (a) compares the heat generation as a function temperature of the analytical approximation (Keilin at  $T_{cs}$ , Dresner at  $\frac{1}{2}(T_{cs} + T_c)$  and Cherry at  $T_c$  [88]). Note that here  $T_c$  is an effective critical temperature and is thus  $B$ -dependent. (b) shows the heating function of the current sharing- and of the parallel-paths model at an  $n$ -value of 15 and 100. In this example, a MgB<sub>2</sub>/Fe conductor is assumed with  $f = 42$  %, operating at  $B_0 = 8$  T,  $T_0 = 4.2$  K and  $I_c = 40$  A.

(Cherry, Keilin and Dresner [88]). As already discussed in §4.3.2, current diffusion – and therefore heat generation – starts at  $T_{cs}$ . Therefore the assumption of Cherry that current abruptly diffuses into the matrix at  $T = T_c$  (the assumption used in (4.12) and (4.16)) results in an underestimation of the heat generation. As an alternative, Keilin replaced  $T_c$  with the current sharing temperature, so that all the current abruptly diffuses into the matrix at  $T = T_{cs}$ . This clearly leads to an overestimation of the heat generation, since at  $T_{cs}$  the critical current of the filaments does not become zero and the filaments still carry a sizeable fraction of the current. As a compromise, Dresner used the intermediate temperature  $\frac{1}{2}(T_{cs} + T_c)$  as the temperature where heat generation starts. Although Dresner's choice might seem a reasonable compromise, compared to both the current sharing and the parallel-paths model it is still a crude approximation that does not account for the detailed distribution of the current between the filaments and the matrix.

Figure 4.7b compares the heat generation in the parallel-paths model in the case of an  $n$ -value of either 15 or 100 with that in the current sharing model. For a conductor with  $n = 15$ , the current sharing model overestimates the heat generation with respect to the parallel-paths model. In contrast, the current

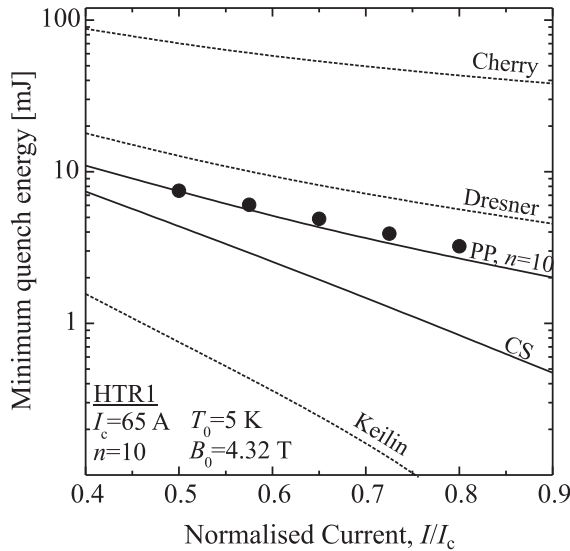


**Figure 4.8:** Heating function of the parallel-paths model, showing the individual contributions of the filaments and matrix. (a) shows the case for a  $n$ -value of 15, while (b) shows the case for a  $n$ -value of 100.

sharing model underestimates dissipation for a conductor with a high  $n$ -value.

To clarify the differences, the heating function of the parallel paths model is re-plotted in figure 4.8a ( $n = 15$ ) and b ( $n = 100$ ), this time also showing the separate contributions of the filaments and the matrix. The heat generation in the matrix simply follows  $p_{\text{diss,m}}(T) = J_m(T)^2 \rho_m(T)$ . Heat generation in the filaments, on the other hand, follows  $p_{\text{diss,sc}}(T) = J_{sc}(T)E(J_{sc}(T))$ , which results in a peaked curve between  $T_{cs}$  and  $T_c$ . As defined before,  $T_{cs}$  is the temperature at which the voltage starts to rise, which causes the current to diffuse into the matrix. The maximum in the curve  $p_{\text{diss,sc}}$  corresponds to the temperature at which the effective resistance of the filaments equals that of the matrix. When the temperature increases further, the effective resistance becomes higher than that of the matrix and the current in the filaments decreases fast. Above the normal state temperature  $T_{ns}$ , the filaments are normal conducting and current continues to be shared between filaments and matrix according to the parallel-paths connection between two Ohmic resistances. As a consequence even in this regime, dissipation in the filaments is not zero since in typical MgB<sub>2</sub> conductors the normal state resistivity of the filaments and matrix are comparable.

As shown in figure 4.5b, the “smooth” voltage-current relation of the filaments for low  $n$ -values causes the transport current to be carried virtually lossless by the filaments well above  $T_{cs}$ . Accordingly, significant heating starts at a higher temperature than in the case of the current sharing model. For  $n = 100$ , on the



**Figure 4.9:** Comparison of measured MQE( $I/I_c$ ) values for conductor HTR1 (data points) with the analytical (dashed lines) and the numerical models (solid lines). Further details of sample HTR 1 can be found in table 4.1. Note that for the analytical approximations  $\kappa$  and  $\rho$  are taken at  $T = \frac{1}{2}(T_{cs} + T_c)$ .

other hand, the sharp voltage-current relation causes the current in the filaments to decrease almost linearly with temperature, just like in the current sharing model (see figures 4.4b and 4.5b). The reason that the heating function of the parallel-paths model does not converge with that of the current sharing model at high  $n$ -values is the heat generation in the filaments, which is neglected in the current sharing model. In fact, the heat generation in the filaments causes the total heat generation to become an almost linear function of temperature that, within the range  $T_{cs} < T < T_c$ , is higher than in the current sharing model.

### Minimum Quench Energy

Figure 4.9 compares experimental MQE data as a function of current with different model calculations. Comparing the different models, it becomes clear that the details of current redistribution –and the associated heating functions– are very important as they lead to results that differ two orders of magnitude. As shown by equation (4.12),  $l_{MPZ}$  and MQE depend on  $p_{diss}^{-1/2}$ . As a result, the underestimation of  $p_{diss}$  following from Cherry's assumption (no heat generation below  $T_c$ ) leads to a seriously overestimation of MQE. The opposite is true for Keilin's assumption, which results in predicted values of MQE that are too low. In fact, by assuming  $T_{cs}$  as the temperature at which current diffuses abruptly into the matrix, MQE  $\rightarrow$

0 when  $I_0$  approaches  $I_c$ . This is caused by the fact that for  $I \rightarrow I_c$ ,  $T_{cs}$  approaches the baseline temperature  $T_0$  and the temperature margin vanishes.

The overestimation of  $p_{\text{diss}}$  (see figure 4.7a and b) of Dresner's assumption with respect to the parallel-paths model results in a predicted MQE value that is too low. The reason that MQE is in fact overestimated is the assumption of a relatively simple "block" shaped temperature profile in the minimum propagation zone ( $T(z) = \frac{1}{2}(T_{cs} + T_c)$  over  $l_{\text{MPZ}}$ ). Furthermore, equation (4.12) requires the choice of  $\kappa$  and  $\rho$  at some "appropriate" temperature, which introduces an extra uncertainty.

Just like the parallel-paths model, the current sharing model does account for the strong temperature dependence of  $c$ . The underestimation of MQE in the current sharing model is a direct result of neglecting the voltage-current relation of the filaments. As shown by figure 4.7b, a low  $n$ -value causes significant heating to start at a higher temperature than in the current sharing model.

### Normal Zone Propagation Velocity

In figure 4.10a measurements of  $v_{nz}$  as a function of current for sample HTR1 are compared with the model calculations. Clearly, by including the details of current distribution the parallel-paths model results in a good agreement with the experimental data.

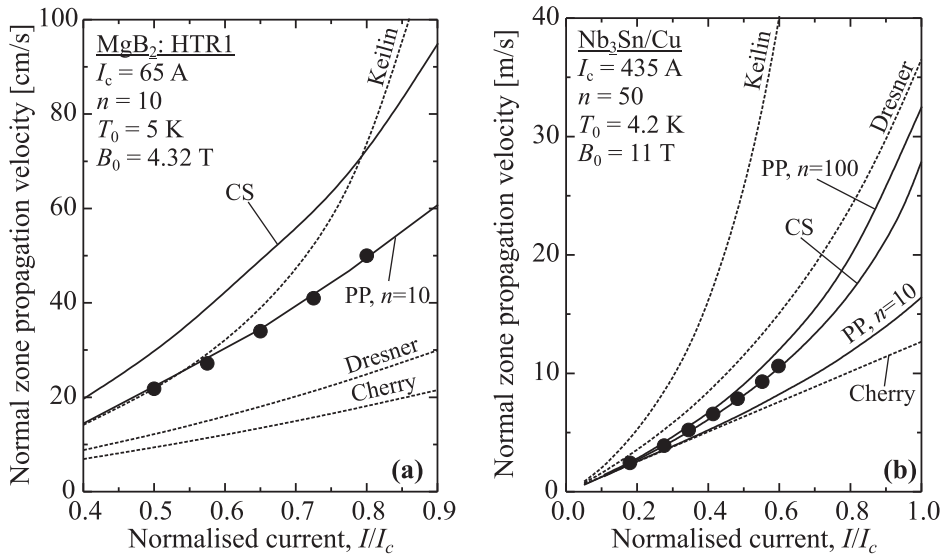
With the analytical approximation, using Cherry's assumption results in an underestimation of  $v_{nz}$ , due to the omission of heat generation for  $T < T_c$ . In contrast, the overestimation of  $p_{\text{diss}}$  in Keilin's approximation results in a seriously overestimated  $v_{nz}$ . Similar to MQE, by assuming  $T_{cs}$  as the temperature at which *all* the current diffuses in the matrix, the temperature margin vanishes as  $I_0 \rightarrow I_c$ , which in this case results in  $v_{nz} \rightarrow \infty$ .

Figures 4.7a and b show that with Dresner's assumption, the total Ohmic heat generation over the temperature range  $T_{cs}$  to  $T_c$  is slightly overestimated with respect to the current sharing and parallel-paths model. On first sight, this would suggest that also the predicted values of  $v_{nz}$  would be too high. The reason that  $v_{nz}$  is actually underestimated is the assumption that all material properties are temperature independent. Clearly, in order to get a better agreement, an "appropriate" temperature has to be chosen at which  $c$ ,  $\kappa$  and  $\rho$  are taken. However, in the case of  $\text{MgB}_2$ , this leads to the problem that  $c$  increases by two orders of magnitude in the range between 5 and 40 K, which causes the results of the analytical predictions to depend strongly on the choice of this "appropriate" temperature.

Similar to MQE, the overestimation of  $v_{nz}$  in the current sharing model is a direct result of neglecting the voltage-current relation of the filaments. The relatively low  $n$ -value of this conductor under these operating conditions causes significant heat generation to start at a higher temperature than in the current sharing model (see figure 4.7b).

For comparison,  $v_{nz}$  measurements as a function of current for a  $\text{Nb}_3\text{Sn}/\text{Cu}$  conductor with an  $n$ -value of 50 are presented in figure 4.10b. As expected from





**Figure 4.10:** Comparison of the measured  $v_{nz}(I/I_c)$  (data points) with the analytical (dashed lines) and the numerical models (solid lines). (a) shows the results of MgB<sub>2</sub> conductor HTR1 (table 4.1) and (b) of a Nb<sub>3</sub>Sn/Cu conductor. Note that for the analytical approximations  $c$ ,  $\kappa$  and  $\rho$  are taken at  $T = \frac{1}{2}(T_{cs} + T_c)$ .

the heating functions parallel-paths model calculations with  $n$ -values of 10 and 100 result in an under- and overestimated  $v_{nz}$  respectively. Interestingly, however, the conductor's intermediate  $n$ -value (compared to 10 and 100) causes the results of current sharing model to agree rather well with the measurements. Contrary to MgB<sub>2</sub>, the normal state resistivity of Nb<sub>3</sub>Sn filaments is significantly higher than the matrix resistivity, so that in this material the current sharing approximation (absence of dissipation in filaments) is justified.

## 4.4 Experiment

As discussed in section 4.3, prediction of  $v_{nz}$  and MQE can be inaccurate due to the 1-dimensional approach of most models, uncertainties in the boundary conditions (liquid helium cooling, thermal contacts) and uncertainties in the thermal and electrical properties of the conductors. Moreover, the electrical and thermal properties vary with magnetic field and temperature. To verify the predictive value of the numerical model, it is necessary to measure  $v_{nz}$  and MQE not only as a function of transport current but also of magnetic field and temperature.

In §4.4.1 the principle of the voltage-time-of-flight method with which  $v_{nz}$  is measured is explained. Furthermore, some details of the quench heater that ini-

tiates the normal zone are treated.

It is useful to measure  $v_{nz}$  and MQE under adiabatic conditions. This yields the intrinsic properties of the conductor, avoiding complicated cooling terms –that depend on the detailed boundary conditions– and straightforwardly simulates the cryogen-free operation of MgB<sub>2</sub> conductors. In order to perform such measurements a quasi-adiabatic set-up was constructed, which is described in §4.4.2.

With the quasi-adiabatic set-up it is possible to measure  $v_{nz}$  and MQE as a function of temperature. Measurements at elevated temperature require a careful thermal design of the experiment in order to ensure a homogeneous baseline temperature along the sample. The thermal design of the experimental set-up is therefore discussed separately in §4.4.3.

In §4.4.4 the instrumentation is discussed that is used to accurately measure  $v_{nz}$  and MQE data under different operating conditions ( $I_0, T_0, B_0$ ).

Lastly,  $v_{nz}$  and MQE depend on the cross-sectional layout of the conductors (thermal/electrical conductivity, heat capacity, filling factor, number of filaments, etc.). Therefore, conductors with various layouts were selected to study  $v_{nz}$  and MQE. In §4.4.6 an overview is given of their relevant properties and the main differences between them are discussed.

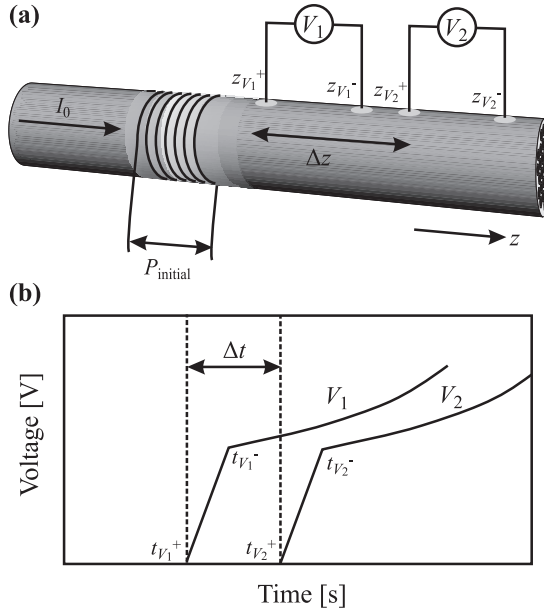
#### 4.4.1 Measurement Technique

The voltage-time-of-flight method is a widely used technique to measure the normal zone propagation velocity  $v_{nz}$  [208,210,211]. A conductor sample is equipped with a quench-heater (in this case a manganin wire wound around the sample) and with several voltage taps that are spaced a distance  $\Delta z$  apart and connected to an oscilloscope (figure 4.11). The conductor is biased with a steady current and a propagating normal zone is initiated with a block shaped heat pulse  $Q_{\text{initial}} (= P_{\text{initial}}\Delta t)$ . The propagating normal zone front passes the voltage contact at position  $z_{V_1+}$  at time  $t_{V_1+}$  and the voltage signal  $V_1$  starts to rise. At  $t_{V_1-}$  the volume between  $z_{V_1+}$  and  $z_{V_1-}$  is completely in the normal state. For  $t > t_{V_1-}$  any further voltage rise is only due to the increase of electrical resistance with temperature. The same process holds for voltage signal  $V_2$ . The normal zone propagation velocity  $v_{nz}$  is then calculated from the time lag  $\Delta t$  between voltage signals  $V_1$  and  $V_2$ :

$$v_{nz} = \frac{\Delta z}{\Delta t} = \frac{z_{V_2+} - z_{V_1+}}{t_{V_2+} - t_{V_1+}} = \frac{z_{V_2-} - z_{V_1-}}{t_{V_2-} - t_{V_1-}}. \quad (4.23)$$

An alternative method, uses pick-up coils rather than voltage taps to trace the position of the normal zone front [212,213]. Current diffusion from the filaments to the matrix around the normal zone front causes a change in flux distribution, which induces a voltage signal in the pick-up coils. Similarly to the previous method, the normal zone propagation velocity is calculated from the time lag between the voltage signals of two consecutive pick-up coils.

With the voltage-time-of-flight method it is in principle possible to measure also the minimum quench energy MQE, simply by varying  $Q_{\text{initial}}$  and searching



**Figure 4.11:** Principle of the Voltage-Time-of-Flight method. (a) shows a conductor that carries a transport current density  $J_0$ . The thermal disturbance  $P_{\text{initial}}$  is injected into the conductor with a wire wound heater to initiate a normal zone. Voltage taps  $V_1$  and  $V_2$  are soldered to the wire, a distance  $\Delta z$  apart. They are connected to an oscilloscope to measure the time dependent voltage signal that is associated with the normal zone propagation. (b) shows a typical voltage signal of a propagating normal zone. The signal between  $t_{V_1^+}$  and  $t_{V_1^-}$  (or  $t_{V_2^+}$  and  $t_{V_2^-}$ ) are associated with the normal zone growth in-between  $z_{V_1^+}$  and  $z_{V_1^-}$  ( $z_{V_2^+}$  and  $z_{V_2^-}$ ). The voltage rise for  $t > t_{V_1^-}$  ( $t > t_{V_2^-}$ ) is associated with the increase of resistance with temperature.

for the transition point between a collapsing- and a propagating normal zone. The main uncertainty in this kind of measurements is the amount of heat that is actually transferred to the conductor itself. In this respect, three requirements are important for heater design: 1) the enthalpy of the heater itself (the temperature integral of the volumetric heat capacity over the heater's volume) needs to be small compared to MQE. If this is not the case, the generated heat is distributed over both sample and heater, which leads to a significant overestimation of MQE; 2) the thermal conductivity between heater and sample needs to be high so that the generated heat does not remain localised in the heater itself, which would again lead to an overestimation of MQE; 3) thermal contacts between heater and other parts of the experimental set-up (heat conduction through heater wiring) should be kept minimal, since once more this heat loss would lead to a significant overestimation of MQE.

An optimised heater for MQE measurements that fulfills all of the design requirements above is presented in [214]. A small tip-heater ( $d \sim 0.6$  mm) is pressed against the sample with a clamping system. Heating takes place at the point where the tip-heater is pressed against the sample via the electrical resistance of the heater-sample contact. Control of the heater-sample resistance is achieved by using graphite paste between heater and sample and by the use of a clamping system that allows to control the force with which the heater is pressed on the sample. Furthermore, the tip-heater is made with a low thermal conductivity material (graphite or machinable glass (Macor)) that is coated with several micrometers of silver paint or Ti/Ag to provide electrical conductance. With this method, heat diffusion into the tip heater is limited, thus minimising the overestimation of MQE.

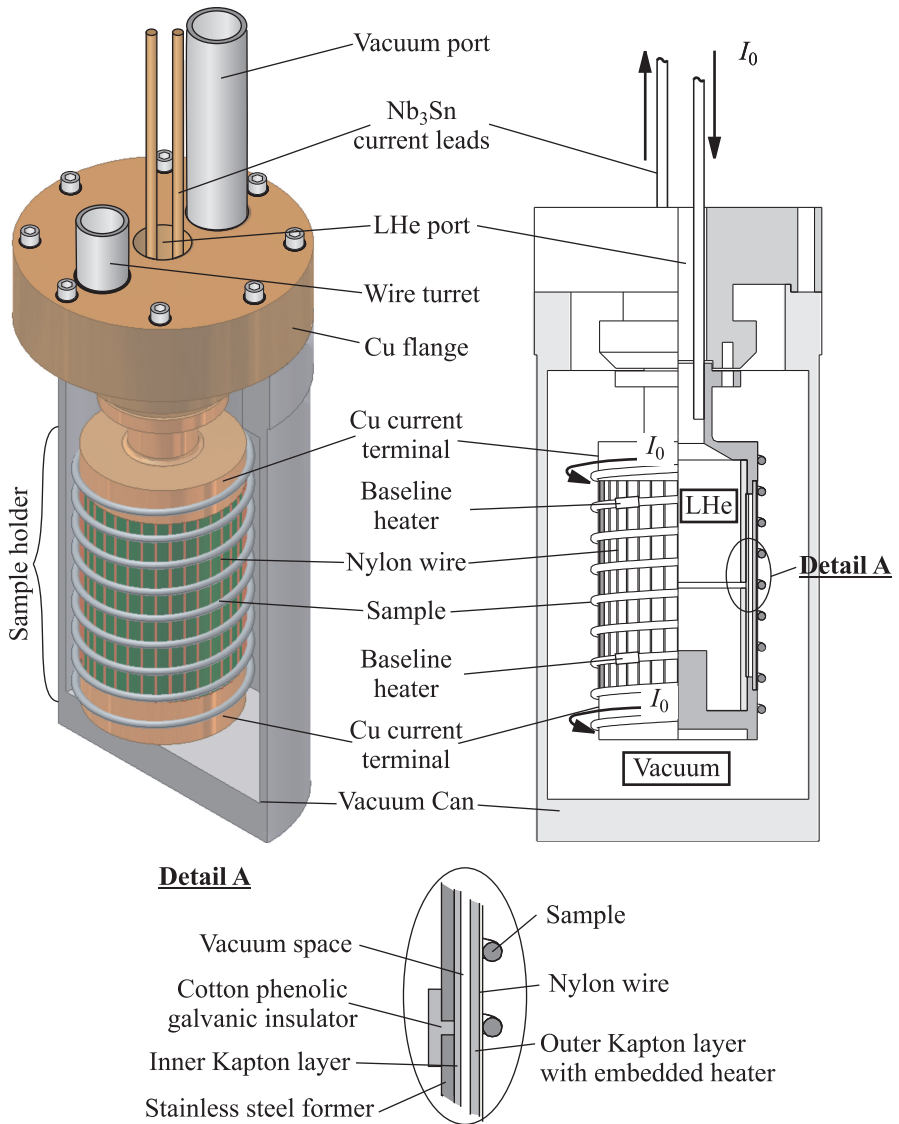
Although the heater enabled accurate MQE measurements in Nb-Ti samples, the similar design was not adopted here, since its use seriously limits sample throughput as for each sample shape (wire or tape) a different clamping system would have to be used. Moreover, the size of the clamping system makes it difficult to mount the whole in a vacuum can, limiting the ability to vary the temperature of the sample in a controllable manner.

A relatively simple manganin wire-wound heater is used. The total length of the manganin wire is about  $\sim 45$  cm, its diameter  $\sim 0.1$  mm and its resistance  $\sim 25 \Omega$ . The heater wire is tightly wrapped around the sample into a bifilar winding to minimise self-induction. It is then fixed to the sample with GE varnish. Clearly, this heater design leads to an overestimation of MQE, since its enthalpy is large compared to that of the average sample. The enthalpy of the manganin heater is approximately  $\sim 0.3$  mJ [32] in the range between 4 and 40 K, whereas the MQE of the  $\text{MgB}_2/\text{Fe}$  conductor from table 1.1 for example is about 0.04 mJ at 4.2 K. The generated heat will thus be distributed over both the sample and the heater. Nevertheless, this type of heater was used in all experiments described below, mainly because of its ease of handling and flexibility. Similarly to [215], the intrinsic MQE values are estimated by comparing the experimentally obtained values with the results of the numerical calculations, thus estimating the effective heat transfer from the quench-heater to the sample. The corresponding heat “loss” is typically about 80 % of the total heat input. The MQE values presented in the corresponding figures are 20 % of the measured ones.

#### 4.4.2 Design

If the normal zone propagation velocity and minimum quench energy are measured in LHe, an extra cooling term would have to be added to the heat balance equation (4.1). This cooling term seriously complicates the analysis of  $v_{nz}$  and MQE, since transient effects of LHe film boiling at the sample’s wetted perimeter have to be accounted for. These transient effects are caused by the relatively slow formation and decay of He vapour films<sup>6</sup>. Moreover, as explained in §4.1, direct LHe cooling

<sup>6</sup>The influence of transient effects on the stability of superconductors is extensively discussed in [201]



**Figure 4.12:** Overview of the quasi-adiabatic setup to measure the normal zone propagation velocity. The sample is placed in a vacuum can and the normal conducting Cu current terminals to which the sample is soldered are cooled with liquid helium that is allowed inside the sample holder through the LHe port. In order to change the baseline temperature, two resistors are positioned at the ends of the sample and a third wire-wound heater (manganin) is embedded in the outer Kapton layer. To limit heat conduction from this embedded heater to the LHe reservoir inside the holder the outer- and inner Kapton layers are separated with a vacuum space. The nylon wire thermally detaches the sample from the outer Kapton layer.

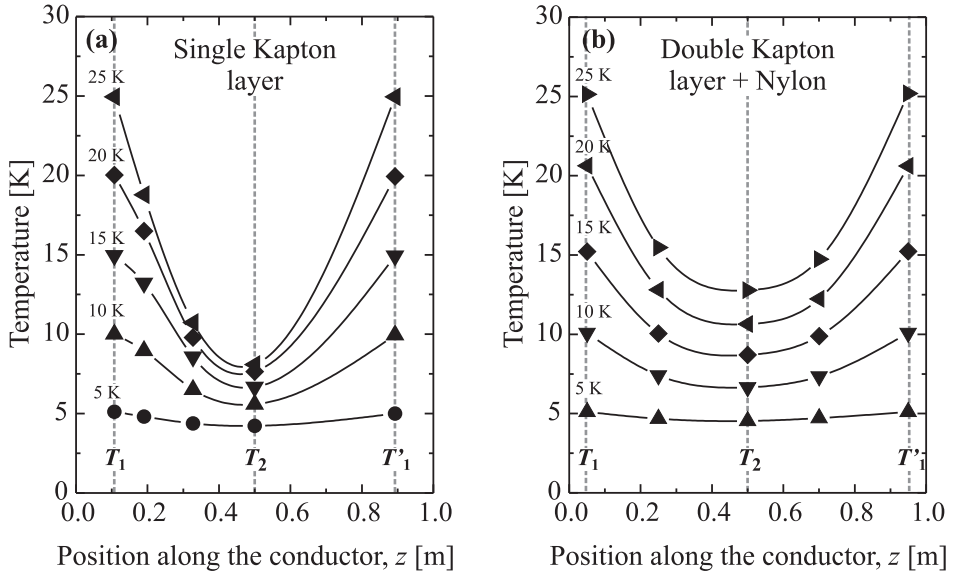
might not be the most relevant operating condition for  $\text{MgB}_2$  conductors. To avoid complications of transient heat-transfer effects and to simulate cryogen-free operation of  $\text{MgB}_2$ , the sample is isolated from LHe by placing it in a vacuum can. A schematic overview of the experimental set-up is given in figure 4.12.

An as-deformed sample is wound on a reaction holder and subsequently sintered (ex-situ) or reacted (in-situ) at the required temperature. After heat treatment, the helically shaped sample is transferred to the sample holder and soldered to the Cu current terminals. The transport current is fed to the current terminals with two  $\text{Nb}_3\text{Sn}$  current leads. To provide additional cooling of the normal conducting current terminals, LHe is allowed inside the hollow central core of the sample holder through a LHe port. The sample is equipped with a quench-heater and voltage taps to trace the position of the normal zone front (as described in the previous paragraph). Additional voltage taps monitor the voltage over the whole sample, while two background heaters and three Cernox thermometers regulate the operating temperature of the sample. The wiring for the instrumentation is fed through the wire turret, which is vacuum sealed with Stycast 2850FT.

The sample holder consists of an upper and lower stainless steel former, which are galvanically separated with a piece of cotton phenolic. The galvanic separation prevents current from flowing through the steel former during the superconducting to normal transition. To prevent LHe from leaking out of the hollow core into the vacuum can, Kapton foil (inner Kapton layer) is wound around the former and glued with Stycast 2850FT. A vacuum space is left between the inner and outer Kapton layers to decrease the heat transfer from the embedded heater in the outer Kapton layer to the LHe. The outer Kapton layer is also made of coiled Kapton foil, and a manganin wire heater is co-wound to provide additional baseline heating. More details on the thermal design are presented in §4.4.3. Nylon spacer wires minimise the mechanical contact area between the sample and the outer Kapton layer. This limits heat leak from the sample to the outer Kapton layer during the propagation of the normal zone, which otherwise would result in an underestimation of  $v_{nz}$ . Lastly, the mechanical connection between the Cu flange and the vacuum can and sample holder assembly is provided with a pressed In wire seal ( $d \sim 1$  mm), which is impermeable for LHe.

### 4.4.3 Controlling Heat Flows

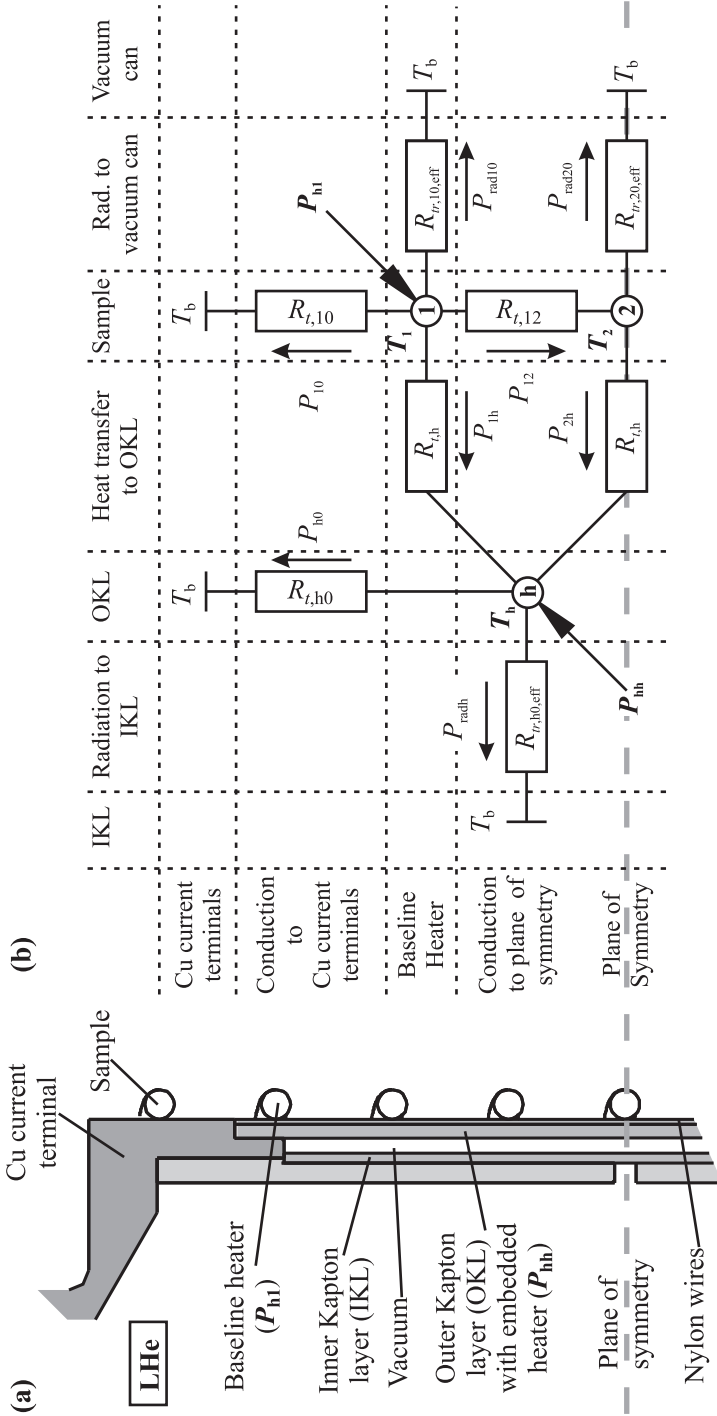
To measure  $v_{nz}$  and MQE of  $\text{MgB}_2$  conductors at elevated temperatures, temperature regulation had to be added to the set-up. In a first attempt the temperature of the  $\text{MgB}_2$  sample was regulated by two baseline heaters,  $P_{h1}$  and  $P'_{h1}$ , and two temperature sensors,  $T_1$  and  $T'_1$ , which were directly mounted on the sample near the top- and bottom Cu current leads respectively. To reduce the lateral heat leak, the sample was not fixed with Stycast 2850FT, but loosely wound on the Kapton layer. The temperature profile over the sample was then monitored with sensors  $T_1$  and  $T'_1$  on both ends of the sample, a central sensor  $T_2$  in the middle of the sample and with two extra auxiliary thermometers between  $T_1$  and  $T_2$ . The resulting temperature profiles over the sample at different heater powers is



**Figure 4.13:** Temperature profiles over the sample with (a) the original sample holder (with a single Kapton layer) and (b) with the second version of the sample holder with Nylon wires and a vacuum space between the inner and outer Kapton layer. The different profiles in each graph represent the different baseline temperatures to which  $T_1$  and  $T'_1$  are regulated with heaters  $P_{h1}$  and  $P'_{h1}$  respectively.

depicted in figure 4.13a. Clearly, a large lateral heat leak between sample and holder resulted in a temperature gradient that can be as large as  $\sim 70\%$  (when  $T_1 = 25$  K). This is too large for  $v_{nz}$  and MQE measurements, since  $I_c$  over a typical MgB<sub>2</sub> conductor tested at a nominal temperature of 25 K and 1 T would vary about 300 % along its length. For an acceptable  $I_c$  gradient over the sample of  $\sim 5\%$ , the maximum temperature gradient should be about  $\sim 1\%$ .

To decrease the lateral heat leak, a new holder was made in which the single Kapton layer was replaced by an inner- and outer Kapton layer with a vacuum space in-between. Furthermore, Nylon spacer wires were mounted on the outer Kapton layer, further minimising the thermal contact between sample and holder. This layout was similar to the final version of the sample holder, depicted in 4.12. Increasing the temperature of the sample in a similar manner as before resulted in the temperature profiles shown in figure 4.13b. Although the homogeneity of the temperature over the sample was improved with these measures, the gradient over the sample at 25 K remained still about  $\sim 50\%$ , i.e. still too large.



**Figure 4.14:** (a) Shows part of the cross-section of the sample holder, while (b) shows its equivalent thermal network.



To solve this problem, a careful thermal analysis of the sample holder assembly was made, as summarised in the next two paragraphs. For more details, the reader is referred to appendix C.

### Thermal Analysis

The thermal analysis is performed by modeling the holder as an equivalent thermal network (see figure 4.14). It is assumed that the problem is symmetric around the midplane of the sample holder (designated as the plane of symmetry) and that the outer Kapton layer is isothermal at temperature  $T_h$ . Also the Cu current leads, the inner Kapton layer and the vacuum can are assumed to be at a fixed temperature of  $T_b = 4.2$  K. The assumptions leave three nodes at which the temperature needs to be solved as a function of the baseline heater input  $P_{h1}$ :

**node 1:** the point on the sample where heater  $P_{h1}$  is mounted, close to the Cu current terminal and at a temperature  $T_1$ ;

**node 2:** the point on the sample at the plane of symmetry, with temperature  $T_2$ ;

**node h:** the outer Kapton layer with temperature  $T_h$ .

At each node the heat fluxes are balanced, yielding three equations. The heat fluxes themselves are determined by:

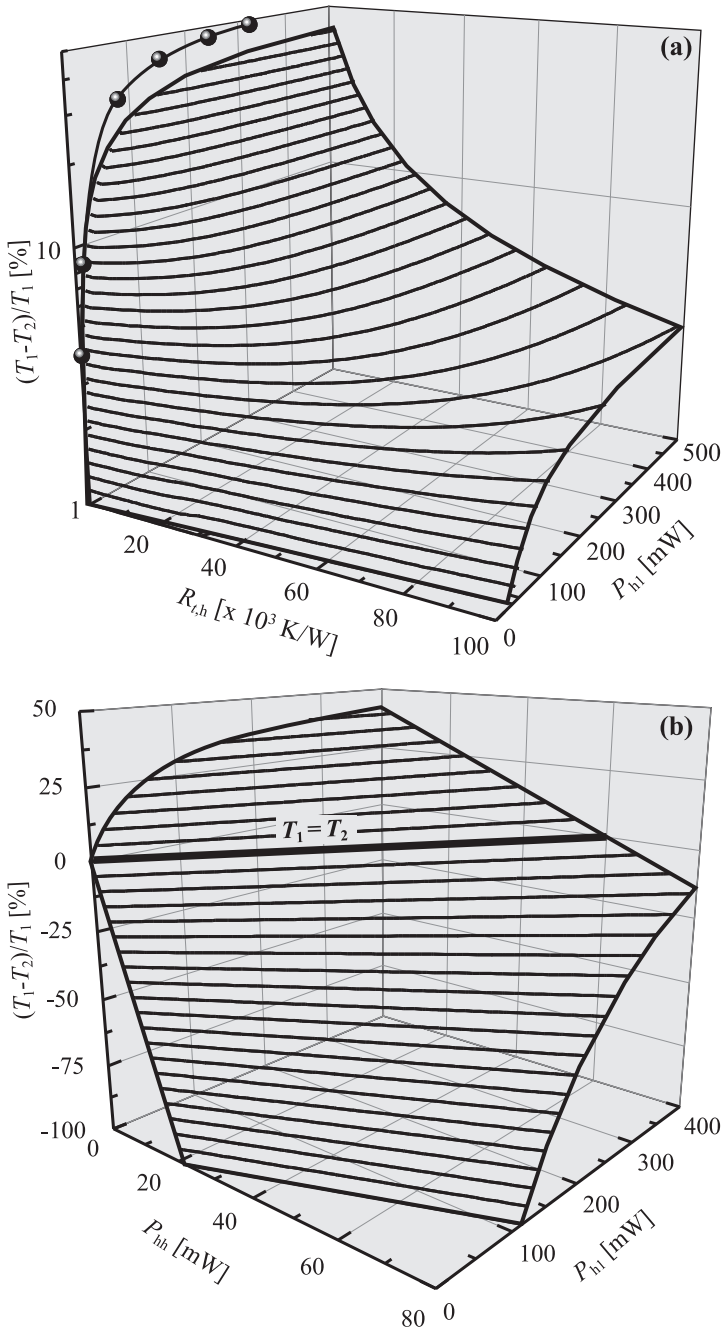
$$P = \frac{\Delta T}{R_t}. \quad (4.24)$$

$\Delta T$  and  $R_t$  are the temperature drop and the effective thermal resistance ([K/W]), in each branch of the thermal network. In principle, all the thermal resistances can be estimated using the relevant thermal properties of each branch (thermal conductivity  $\kappa$ , cross-sectional area  $A$  and length  $l$  for conductivity and emissivity  $\epsilon$ , Stefan-Boltzmann constant  $\sigma_{SB}$  and surface area  $A$  for radiation). Overall, this leaves a set of three linear equations, expressing heat balances at each node and three unknown temperatures ( $T_1$ ,  $T_2$  and  $T_h$ ). The only thermal resistance which is a-priori unknown is that of the sample to the outer Kapton layer ( $R_{t,h}$ ), because of the poorly defined thermal contact between the sample and the Nylon wires. Instead,  $R_{t,h}$  is determined by fitting the linear equations to the measured temperature profile in figure 4.13b.

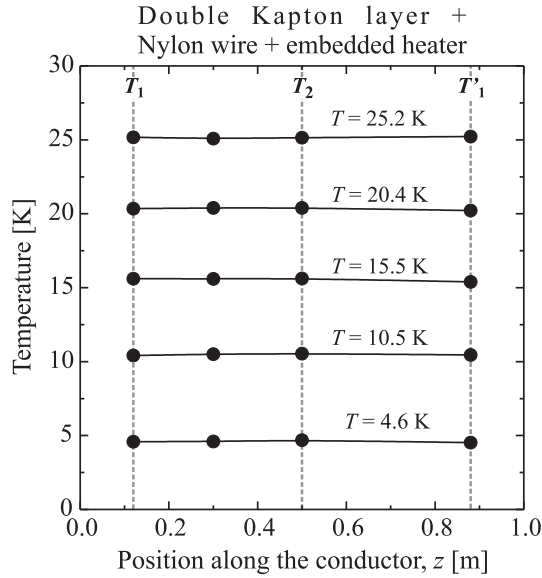
With this model, two possible strategies to minimise the thermal gradient over the sample were explored: 1) increasing  $R_{t,h}$  further; and 2) minimising the lateral heat leak by increasing temperature  $T_h$  of the outer Kapton layer.

### Results

Figure 4.15a shows the calculated temperature gradient between  $T_1$  and  $T_2$  as a function of both the power of the baseline heater at node 1 ( $P_{h1}$ ) and  $R_{t,h}$ . The data-points in the graph are the measured temperature gradients from the profiles in figure 4.13b. The best fit between data and calculations corresponds to a value



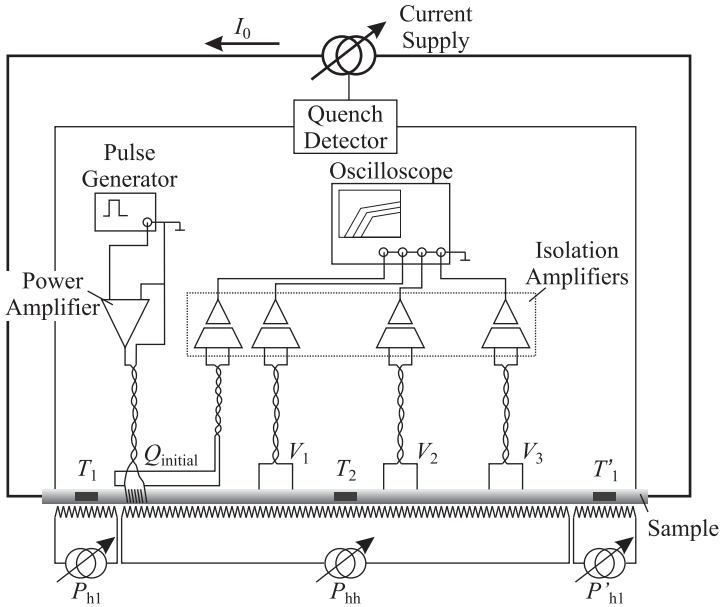
**Figure 4.15:** (a) Calculation of the relative temperature difference as a function of the heater power  $P_{h1}$  and the thermal resistance between sample and outer Kapton layer ( $R_{t,h}$ ). The data points indicate the measurements (see figure 4.13b) at an assumed  $R_{t,h}$  of 1000 K/W. Note the logarithmic scale of the  $(T_1 - T_2)/T_1$ -axis. (b) Calculation of the relative temperature difference as a function of the embedded heater power in the outer Kapton layer ( $P_{hh}$ ) and the heater power at node 1 ( $P_{h1}$ ).



**Figure 4.16:** Measured temperature profiles along the sample in the final thermal design of the sample holder. The various profiles represent the different temperatures to which  $T_1$ ,  $T_2$  and  $T'_1$  are regulated to by  $P_{h1}$ ,  $P_{hh}$  and  $P'_{h1}$  respectively.

for  $R_{t,h}$  of about 1000 K/W. If this value could be increased to  $1 \cdot 10^5$  K/W, the maximum temperature gradient would be about 3 %. This corresponds to an  $I_c$  gradient of about 14 %. Although this would be a clear improvement of the temperature homogeneity, it is still too high. Moreover,  $R_{t,h} \sim 1 \cdot 10^5$  K/W corresponds to a free-hanging sample with only radiation as a lateral heat loss mechanism. In conclusion, increasing  $R_{t,h}$ , even to an unrealistically high level, would not lead to an acceptable temperature gradient.

The second option –implemented in the final design– is to increase the temperature of the outer Kapton layer with a third heater  $P_{hh}$  on the outer Kapton layer so that the lateral heat leak is minimised. The model calculations in figure 4.15b show that with this third heater it is possible to invert the temperature gradient, such that  $T_2 > T_1$ . This means that the temperature gradient can be regulated to zero. As discussed in §4.4.2, this concept is implemented by embedding a manganin heater in the outer Kapton layer across its full length. The measured temperature profiles in figure 4.16 show that with this design the gradient has indeed reduced significantly to about  $\sim 0.5$  % at 25.2 K, corresponding to an  $I_c$  gradient of  $\sim 2$  %, which is only limited by the waiting time acceptable for full temperature stabilisation.



**Figure 4.17:** Schematic view of the instrumentation. Note that the instrumentation for the magnet field is not included.

#### 4.4.4 Instrumentation

The instrumentation used to measure MQE and  $v_{nz}$  as a function of temperature, field and transport current is described. Figure 4.17 shows a schematic overview.

As discussed in the previous section, 3 heaters ( $P_{h1}$ ,  $P'_{h1}$  and  $P_{hh}$ ) are used to increase the baseline temperature of the sample above 4.2 K. Each heater has its own current source so that residual temperature gradients across the sample can be minimised. The temperature of the sample is monitored with three Cernox thermometers ( $T_1$ ,  $T'_1$  and  $T_2$ ).

The current source providing the sample current  $I_0$  is connected to a quench detector that monitors the voltage over the whole sample. If the voltage exceeds 50 mV while a normal zone is propagating through the sample, the quench detector switches the current supply off, preventing the sample from burning out.

The quench heater that initiates the normal zone ( $P_{initial}$ ) is a wire-wound heater with a resistance of  $\sim 25 \Omega$ . The heater envelops a sample length of about  $\sim 15$  mm. The heat pulse has a block shape and is generated by a programmable function generator that controls a power amplifier. The typical length of the heat pulse is about  $\sim 0.5$  s depending on the operating conditions ( $I_0$ ,  $T_0$ ,  $B_0$ ).

In order to increase the resolution of the voltage traces of the propagating normal zone (see figure 4.11b), the voltage signals  $V_1$  through  $V_3$  are amplified before they are fed into the oscilloscope. The amplifiers also galvanically isolate

the oscilloscope from the sample to prevent ground loops, which might otherwise cause artefacts in the voltage traces.

### 4.4.5 Measurement Procedure

As explained in §4.4.1, the minimum quench energy (MQE) is determined by varying  $Q_{\text{initial}}$  and finding the transition point between a collapsing- and a propagating normal zone. The maximum difference between the value of  $Q_{\text{initial}}$  at which the normal zone collapses and the value at which the normal zone propagates is chosen in such a way that the uncertainty in the measured value of MQE (as opposed to the absolute uncertainty in MQE, discussed in §4.4.1) is about  $\sim 5\%$ . Once the normal zone is propagating,  $v_{nz}$  is also measured.

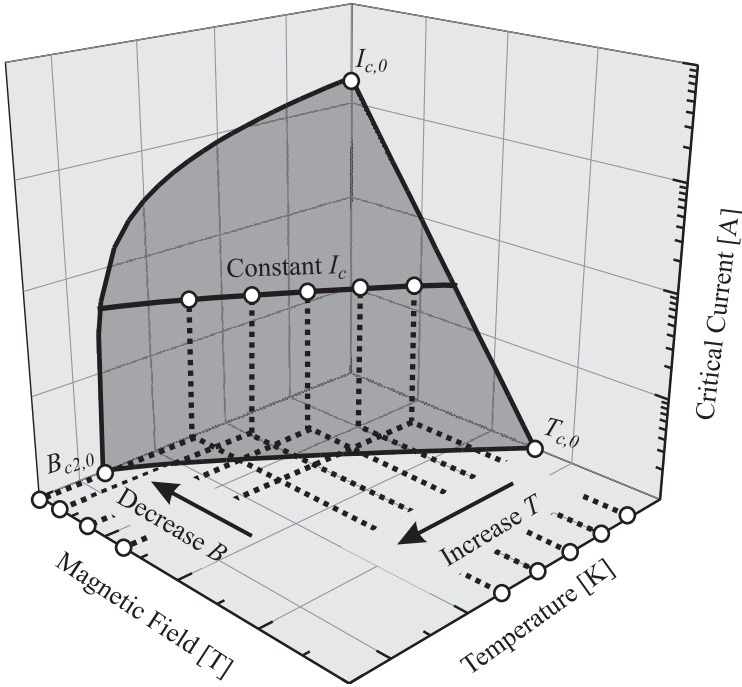
Both  $v_{nz}$  and MQE depend on the operating conditions  $(I_0, T_0, B_0)$ .  $I_0$  determines the value of the heating term in (4.16), while  $T_0$  and  $B_0$  influence the material properties  $c$ ,  $\kappa$  and  $\rho$ . Moreover, all three parameters together determine the temperature margin in (4.16) through:

$$T_s - T_0 \approx [I_0 - I_c(B_0, T_0)] \left( \frac{dI_c(B_0, T)}{dT} \right)^{-1}. \quad (4.25)$$

Ideally,  $v_{nz}$  and MQE should therefore be characterised as a function of all three parameters. This, however, requires a prohibitively large amount of data-points to be measured, which is time consuming especially in view of the MQE search routine. In order to keep the measurement time reasonable, a strategy is chosen in which the parameters  $B_0$  and  $T_0$  are coupled in such a way that the critical current  $I_c$  is kept constant. This procedure is schematically illustrated in figure 4.18. At each  $(B_0, T_0)$  setting,  $v_{nz}$  and MQE are measured as a function of the transport current  $I_0$ .

To adjust  $B_0$  in such a way that  $I_c$  remains constant when  $T_0$  is changed, the critical surface of a conductor should first be measured over the relevant  $(B_0, T_0)$  range. The initial  $I_c$  measurements also serve to verify whether handling damage has occurred during the preparation of the normal zone experiment. Since the adiabatic nature of the experimental set-up makes it less suited for  $I_c$  measurements at elevated temperatures (thermal runaways),  $I_c(B)$  is only measured at 4.2 K, directly cooled with LHe and without vacuum can. In order to estimate the whole critical surface,  $T_c$  is also measured by applying a transport current of 1 mA to the sample during the cool-down of the set-up (with the vacuum can installed). The resistive transition is determined by monitoring the voltages  $V_1$ ,  $V_2$  and  $V_3$  together with temperatures  $T_1$ - $T_3$ <sup>7</sup>. The cool-down of the sample is slow and homogeneous enough to measure  $T_c$  within  $\sim 2$  K accuracy. With the measured  $I_c(B)$  curve at 4.2 K and critical temperature  $T_c$  the two remaining parameters  $I_{c,0}$  and  $B_{p,0}$  in the approximate descriptions of the critical surface

<sup>7</sup>See §5.2.3 for a more thorough discussion on  $T_c$  measurements.



**Figure 4.18:** Overview of the measurement procedure. To keep the critical current  $I_c$  constant while  $T$  is increased, the magnetic field  $B$  has to be decreased accordingly.

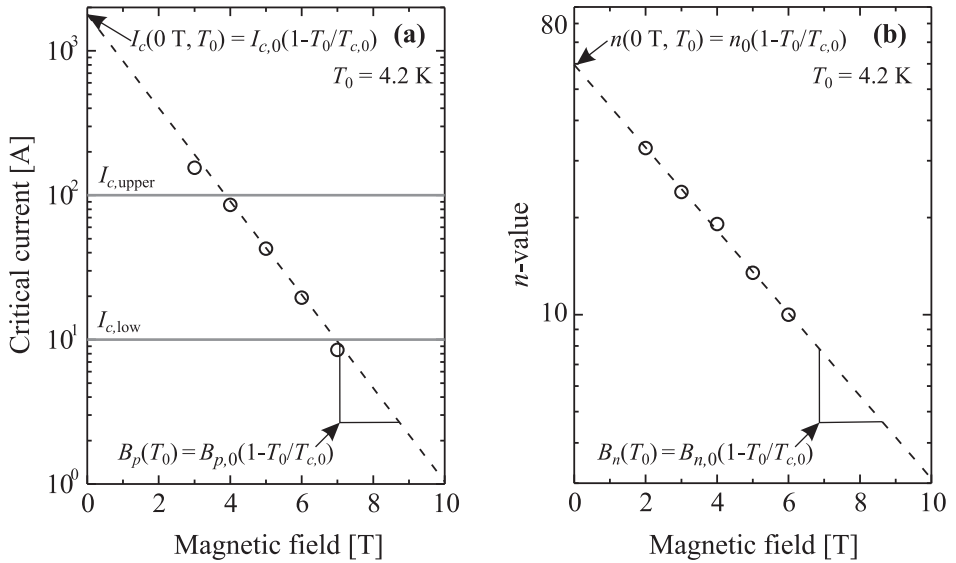
(3.1) can be determined:

$$I_{c,0} = I_c(0 \text{ T}, 4.2 \text{ K}) \left(1 - \frac{4.2 \text{ K}}{T_c}\right)^{-1} \quad \text{and} \quad (4.26)$$

$$B_{p,0} = B_p(4.2 \text{ K}) \left(1 - \frac{4.2 \text{ K}}{T_c}\right)^{-1}.$$

$I_c(0 \text{ T}, 4.2 \text{ K})$  is determined via the extrapolation of  $\ln(I_c(B))$  to 0 T, while the slope of the  $\ln(I_c(B))$  curve determines  $B_p(4.2 \text{ K})$ . An example of such an  $I_c(B)$  curve measured at 4.2 K and is fitted with equation (3.1) is plotted in figure 4.19a.

Knowledge of the critical surface in the relevant temperature-field range is not only important for the  $v_{nz}$  and MQE measurement procedure, but also serves as input data for the numerical model. For modeling, the  $n$ -value as a function of  $T$  and  $B$  also needs to be known. Equation (4.21) can be used in an analogous



**Figure 4.19:** (a) shows an  $I_c(B)$  measurement at 4.2 K for sample HTR1 (see §4.4.6) from which  $I_{c,0}$  and  $B_{p,0}$  are determined with equation (3.1\*) (dashed line). The gray lines show the upper and lower  $I_c$  limits for the  $v_{nz}$  and MQE measurements. (b) shows an  $n(B)$  curve for sample FZK from which  $n_0$  and  $B_{n,0}$  are determined.

manner to obtain  $n_0$  and  $B_{n,0}$ :

$$n_0 = n(0 \text{ T}, 4.2 \text{ K}) \left(1 - \frac{4.2 \text{ K}}{T_c}\right)^{-1} \quad \text{and} \quad (4.27)$$

$$B_{n,0} = B_n(4.2 \text{ K}) \left(1 - \frac{4.2 \text{ K}}{T_c}\right)^{-1} .$$

An example of a fitted  $n(B)$  curve at 4.2 K is shown in figure 4.19b.

Figure 4.19a also shows a minimum and maximum value for the critical current. This is the  $I_c$  range in which  $v_{nz}$  and MQE are measured. The upper value is the point where the  $I_c$  data start to deviate from the exponential dependence (3.1). As explained in chapter 3, this is assumed to be the point where the sample heats up due to intrinsic thermal instability. The lower value for  $I_c$  corresponds to the practical maximum value MQE ( $\approx 5 \text{ J}$ ) that can be measured with the set-up. Measurements at lower  $I_c(B_0, T_0)$  values would require heat pulses MQE  $> 5 \text{ J}$ , which might burn out the quench-heater.

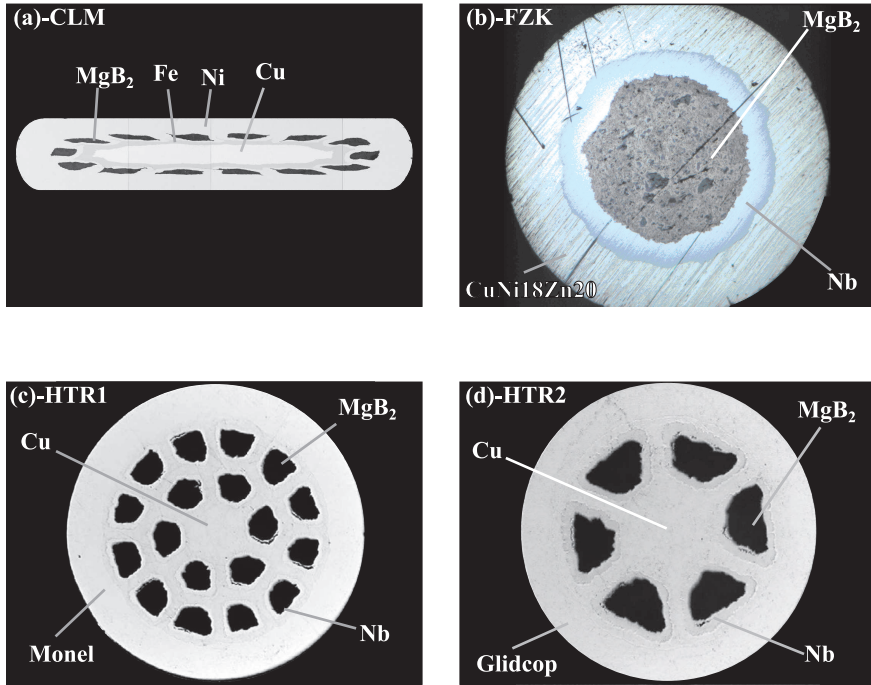
### 4.4.6 Samples

As discussed in §4.2 and §4.3, both  $v_{nz}$  and MQE are influenced by the conductors cross-sectional layout (number, shape and size of the filaments), the heat capacity and the electrical/thermal conductivity of the materials in the composite conductor. The  $n$ -value and the critical current density also plays a significant role. The samples studied here are different but representative. Their cross-sections are depicted in figure 4.20a-d, while table 4.1 lists their layout properties as well as the parameters describing their critical surface and  $n(B,T)$  dependence.

The design of a superconductor is, amongst other considerations, a trade-off between thermal stability and the requirement that the filaments should be sufficiently dense to obtain a high  $J_c$  (see chapter 2 and §4.1). Sufficient stability requires the incorporation of a high conductivity material in the matrix, such as Cu or Al. On the other hand, as discussed in chapter 2, to obtain a high filament density a high strength matrix material is required, which is usually poorly conducting. Another trade-off arises since high conductivity materials tend to react with MgB<sub>2</sub> while chemically compatible materials are usually relatively poor conductors. The necessity to include poorly conducting materials in the matrix will influence the diffusion of current and heat and thus  $v_{nz}$  and MQE. In the conductors studied here different strategies are applied to ensure sufficient filament density and to prevent reaction between filament and matrix, while providing at the same time thermal stability. Consequently the conductors will react differently to thermal instabilities. In order to obtain a high densification, the filaments of sample CLM are embedded in a hard, but compared to Cu poorly conducting, Ni matrix. The Ni matrix, together with the Fe, also serves as a reaction barrier between the filaments and the Cu stabiliser that is placed at the center of the sample. Thus, in conductor CLM filament densification and stability are provided by using multiple materials in the matrix. In contrast, in sample FZK, both filament densification and stability are provided by the relatively poorly conducting, but high strength, CuNi18Zn20 alloy. Reaction between Cu and MgB<sub>2</sub> is prevented by a Nb reaction barrier. Similarly to tape CLM in the HTR wires filament densification and stability are provided by using different materials in the matrix. In HTR1 the high conductivity Cu directly surrounds the filaments (with a Nb reaction barrier in-between) to provide stability, while sufficient filament density is attained by using a Monel (Cu-Ni) outer tube. The same strategy is applied in sample HTR2, but in this case the Monel outer tube is replaced by Glidcop, which is a dispersion strengthened Cu [216]. This material has significant advantages over Cu alloys, since Cu is strengthened by Al<sub>2</sub>O<sub>3</sub> particles rather than alloyed with other metals. The inter-particle distance is large enough to preserve largely the high conductivity of the Cu.

The variety of layouts allows to study the predictive value of the 1D model. For example, in §4.2 the limits of the 1D model were discussed by comparing the radial diffusion with the longitudinal diffusion ( $v_{nz}$ ) of current and heat. Radial diffusion can be relatively slow if the filaments are thick compared to the matrix or if they are surrounded by a relatively thick diffusion barrier, since the resistivity of





**Figure 4.20:** Cross-sections of the investigated conductors. (a) sample CLM, a 14-filaments ex-situ tape by Columbus Superconductors (Genova, Italy). (b) sample FZK, a monofilament in-situ wire by Forschungszentrum Karlsruhe (Karlsruhe, Germany). (c) sample HTR1, an 18-filaments in-situ wire by Hypertech Research (Columbus, Ohio, USA). (d) sample HTR2, a 6-filaments in-situ wire also fabricated by Hypertech Research. Details on dimensions and compositional layout of the conductors are given in table 4.1.

barrier materials is typically relatively high. This will lead to an inhomogeneous distribution of the current and temperature in the cross-section of the sample. The samples CLM and HTR1 form two extremes. Sample HTR1 has a relatively straightforward layout, with finely distributed filaments. Thus current and heat are distributed relatively fast and homogeneous over the cross-section. In this case the 1D model can be expected to describe  $v_{nz}$  and MQE relatively good. On the other hand, sample CLM has from a diffusion point of view a more “complicated” layout. Although it also has relatively small filaments, its tape form and the fact that the filaments are cooled only from one side by the Cu stabiliser leads to asymmetric current and heat distributions, so that deviations between the 1D model and the measurements are expected to be larger. Similarly, sample FZK has a single, relatively thick, filament. Since cooling occurs through CuNi18Zn20, rather than copper, this will lead to a relatively slow radial diffusion.

**Table 4.1:** Overview of the samples with their layout properties, parameters describing their critical surface and parameters describing  $n(B,T)$ .

	CLM	FZK	HTR1	HTR2
Shape	Tape	Wire	Wire	Wire
Powder	Ex-situ	In-situ	In-situ	In-situ
$d$	[mm]	-	0.9	0.83
$w$	[mm]	0.7	-	-
$h$	[mm]	3.6	-	-
$A$	[mm <sup>2</sup> ]	2.5	0.6	0.5
$f$	[%]	9	26	17
Matrix	Ni/Cu/Fe	Nb/CuNiZn	Nb/Cu/Monel	Nb/Cu/GC <sup>a</sup>
$f_i$	[%]	67/14/10	19/55	19/22/41
# Filaments		14	1	18
$T_{c,0}$	[K]	38	35	40
$J_{c,0}$	[A/mm <sup>2</sup> ]	$1.3 \cdot 10^3$	$0.9 \cdot 10^3$	$21.7 \cdot 10^3$
$B_{p,0}$	[T]	1.4	1.9	1.4
$n_0$		40	67	34
$B_{n,0}$	[T]	8.3	3.4	5.5

<sup>a</sup> GC stands for Glidcop.

Finally, the samples differ significantly in critical current density and  $n$ -value, which leads to a quantitatively different heat generation process during a thermal instability (see §4.3.2).

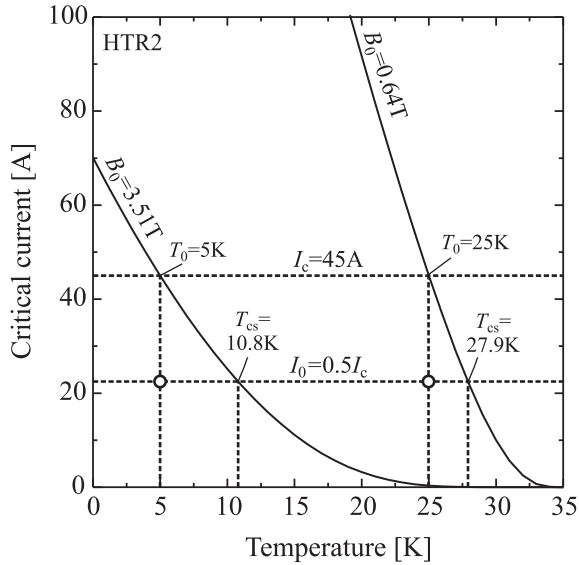
## 4.5 Results

The results of the MQE and  $v_{nz}$  experiments are presented together with the parallel-paths model calculations.

In §4.5.1 the MQE and  $v_{nz}$  behavior of the conductors under study at varying transport current, baseline temperature and magnetic field are compared. It shows how the different matrix materials and cross-sectional layouts result in a different response to a thermal disturbance.

In order to demonstrate the predictive value of the parallel-paths model, the experimental MQE and  $v_{nz}$  data are compared with the model calculations in §4.5.2. In this paragraph also the limits of the 1-dimensional model are analysed and it is shown how uncertainties in the relevant parameters such as  $c$ ,  $\kappa$  and  $\rho$  can lead to discrepancies between measurements and model.

Since the matrix materials in MgB<sub>2</sub> conductors vary widely (from highly conducting Cu matrices to poorly conducting Fe matrices) and also their  $n$ -values can vary significantly, the parallel-paths model is used to investigate the influence



**Figure 4.21:**  $I_c(T)$  lines of sample HTR2 according to equation (3.1) at  $B_0 = 3.51$  T and  $B_0 = 0.64$  T. The critical current for both cases is set to  $I_c = 45$  A so that the  $(B_0, T_0)$  settings are (3.51 T, 5 K) and (0.64 T, 25 K). In this example sample HTR2 operates at  $I_0 = 0.5 I_c$  and the operating points  $(I_0, B_0, T_0)$  are indicated by the **open circles**.

of these parameters on the response of a thermal disturbance of a hypothetical conductor. In this analysis, not only the variation of MQE and  $v_{nz}$  are calculated but also the time evolution of the hot spot temperature since its time evolution will decide whether a given conductor will burn out.

As mentioned in §4.4.1, the quench heater used in this study has a rather limited efficiency of about 20 %, which will be derived in §4.5.2. Throughout this paragraph, the “corrected” value of the measured MQE will be used unless stated otherwise.

### 4.5.1 Temperature- and Field Dependence

The measurement procedure as presented in §4.4.5 is used. In order to compare MQE and  $v_{nz}$  of samples CLM, FZK, HTR1 and HTR2 (see table 4.1) under similar conditions, the critical current of all conductors is kept constant at 45 A. Thus, to study the behaviour of the conductors at elevated temperatures, the baseline temperature is increased from 5 to 25 K whilst the magnetic field is reduced in such a way that  $I_c$  remains constant. To illustrate this, figure 4.21 shows two  $I_c(T)$  lines at  $B_0 = 3.51$  T and 0.64 T of sample HTR2. These magnetic field values are chosen so that  $I_c$  remains constant at 45 A at  $T_0 = 5$  K and 25 K.

### Minimum Quench Energy

The minimum quench energy as a function of transport current at different  $(B_0, T_0)$  settings of samples CLM, HTR1 and HTR2 are displayed in figures 4.22a, c and e.

As a first observation, the minimum quench energies range from  $\sim 2$  to 250 mJ, which is a factor  $\sim 1000$  times higher than typical values measured in low- $T_c$  conductors [214]. These differences are mainly caused by the fact that temperature margins are generally larger than in low- $T_c$  conductors.

Considering the temperature dependence of MQE in more detail, figures 4.22(b), (d) and (f) show a clear non-monotonic behaviour. Since not just the temperature, but also the field  $B_0$  is varied, all four parameters ( $\kappa$ ,  $\rho$ ,  $c$  and temperature margin) in equations (4.12) and (4.13) are affected. With reference to the lumped material properties presented in appendix B, the following can be observed:

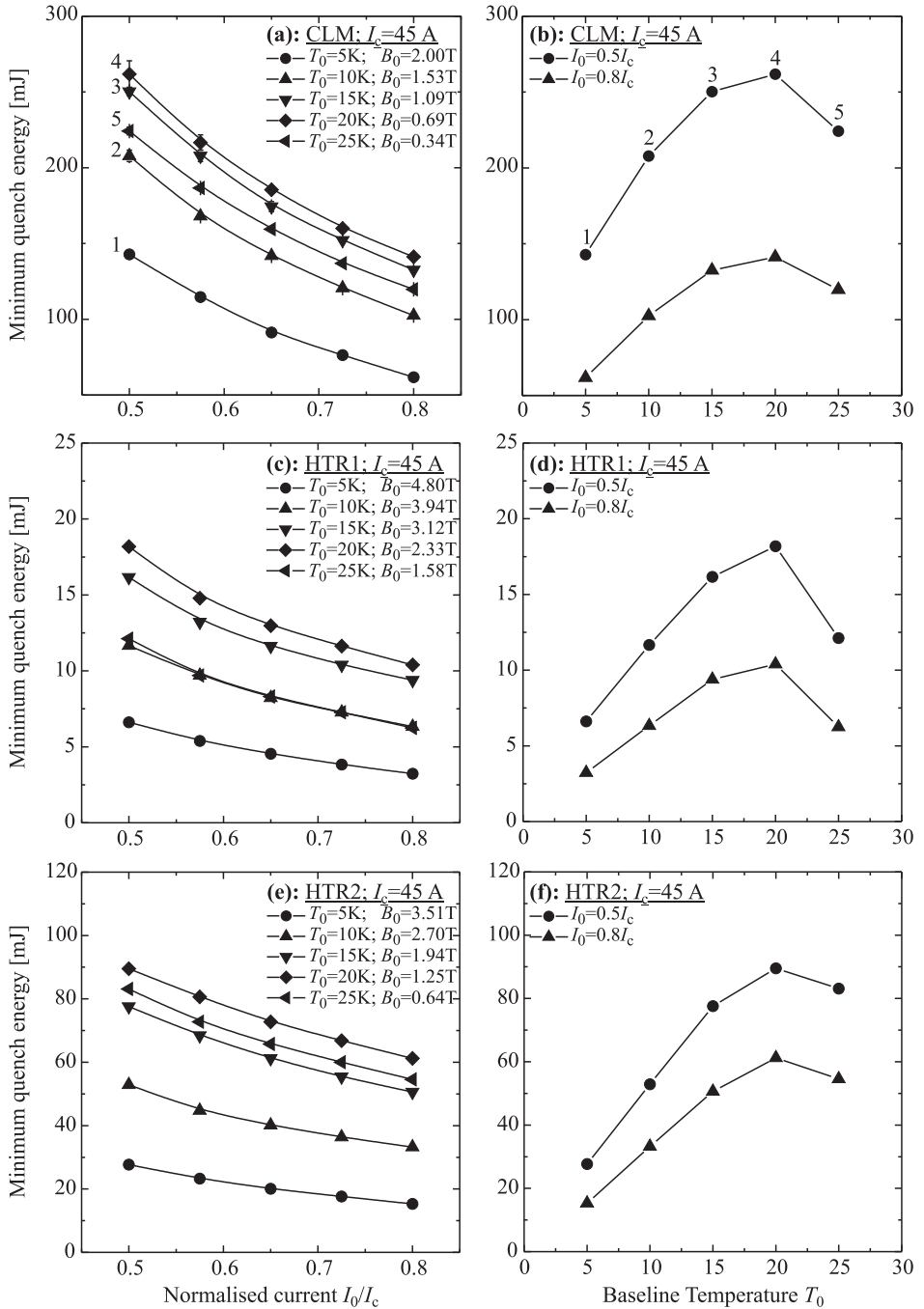
1. The volumetric heat capacity  $c$  increases by approximately two orders of magnitude when the temperature is increased from 5 to 25 K.
2.  $\kappa$  increases by a factor 3 to 10, depending on the sample, when temperature is increased from 5 to 25 K. Furthermore, due to the inclusion of pure Cu in samples CLM, HTR1 and HTR2, the lumped  $\kappa$ -value depends on magnetic field and increases a factor 2 when  $B_0$  is reduced from 5 to 0 T. Sample FZK, on the other hand, only contains “dirty” materials (materials with a low mean free paths for electron transport) and therefore  $\kappa$  is not affected by a changing magnetic field.
3. In contrast to the thermal conductivity,  $\rho$  is virtually temperature independent in the range from 5 to 25 K. However, since samples CLM, HTR1 and HTR2 contain pure Cu, their electrical resistivity depends on  $B_0$  and decreases a factor of 2 when  $B_0$  is reduced from 5 to 0 T.
4. Increasing the temperature  $T_0$  whilst decreasing  $B_0$  to keep  $I_c$  constant results in steeper  $\partial I_c / \partial T|_{B=B_0}$  curves (figure 4.21) and therefore smaller temperature margins (equation (4.25)). Using for instance Dresner’s assumption (see §4.3.3) that heat generation starts at  $T_s = \frac{1}{2}(T_{cs} + T_c)$ , the temperature margin typically reduces by a factor of  $\sim 2$  between 5 and 25 K.

It is now possible to gauge the influence of the various temperature dependencies on the measured temperature dependence of MQE. As discussed in §4.3.1, the value of MQE can be estimated analytically with:

$$l_{\text{MPZ}} = \left\{ \frac{2\kappa_m(T_c - T_0)}{J_m^2 \rho_m} \right\}^{1/2} \quad \text{and} \quad (4.12)$$

$$\text{MQE} = A \int_{l_{\text{MPZ}}} \int_{T_0}^{T_c} c(T) dT dz. \quad (4.13)$$

$\rho$  and  $\kappa$  enter the expression for  $l_{\text{MPZ}}$  only as a square root. The temperature margin also appears in this square-root expression, but furthermore influences



**Figure 4.22:** (a), (c) and (e) show MQE measurements of samples CLM, HTR1 and HTR2 as a function of the normalised current at an  $I_c$  of 45 A and at different temperature- and field combinations. (b), (d) and (f) show the same data, this time plotted as a function of the baseline temperature  $T_0$  at  $I_0 = 0.5I_c$  and  $I_0 = 0.8I_c$ . Note that with changing  $T_0$  also  $B_0$  changes. The **solid lines** are a guide to the eye.

the value of MQE through the integration boundaries in (4.13). The volumetric heat capacity  $c$  enters (4.13) directly. From this, it is clear that the temperature dependence of MQE will be determined mainly by the balance between the decreasing temperature margin and the strongly increasing heat capacity.

Between 5 K and 20 K, it turns out to be the latter effect that is dominant in all 3 conductors and MQE is an increasing function of temperature. Above 20 K, on the other hand, it is the decreasing temperature margin that dominates and in all 3 samples MQE decreases again going from  $T_0 = 20$  K to 25 K (figures 4.22b, d, f).

Comparing the individual conductors shows that sample CLM is the most stable conductor with MQE values ranging from 100 to 300 mJ, which is 3 to 10 times higher than MQE in samples HTR1 and HTR2. Similar values for the same conductor have also been measured and calculated by other authors [211, 215, 217], although under different operating conditions. The high MQE values are mainly caused by the relatively small filling factor of this conductor ( $\sim 9\%$ ), which causes the overall current density to be relatively small ( $J_e \sim 20$  A/mm<sup>2</sup> at an  $I_c$  of 45 A) and thus significantly reduces Ohmic heat generation.

Samples HTR1 and HTR2 only differ in matrix materials, while their filling factor is approximately the same. The matrix of sample HTR1 contains the relatively poor conductor Monel, which is replaced by the relatively highly conducting Glidcop in sample HTR2. The effect of this replacement is reflected in MQE values that are a factor  $\sim 5$  higher than in sample HTR1. Clearly, using Glidcop significantly enhances MQE, while still providing enough strength for wire deformation.

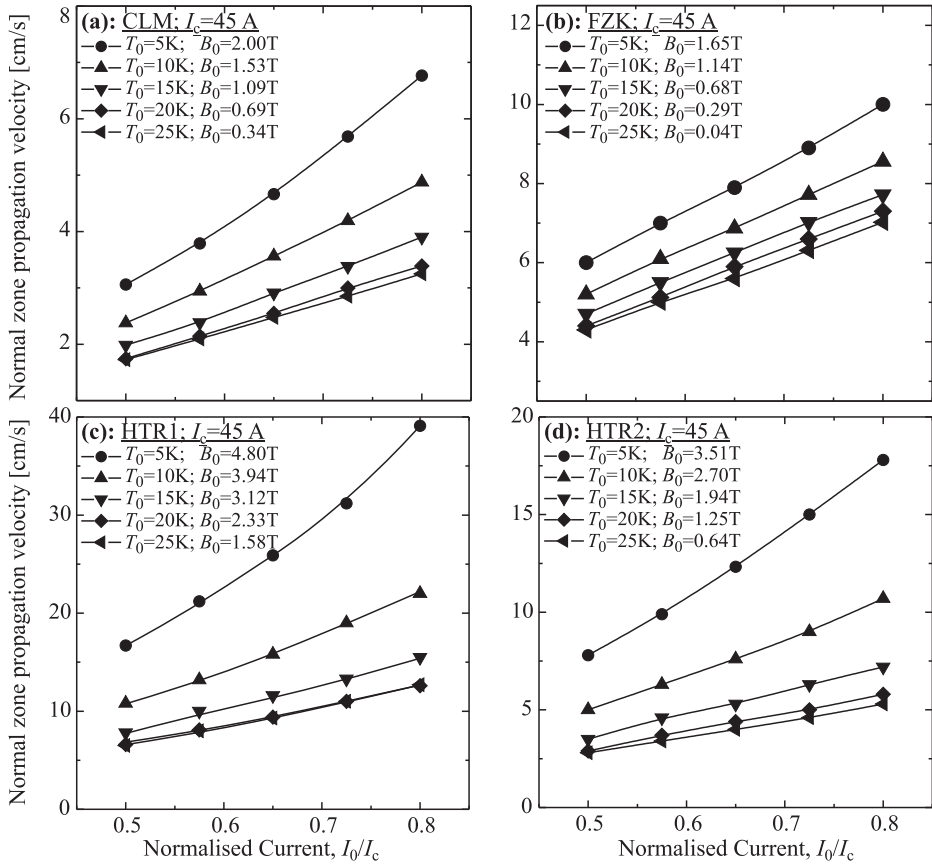
### Normal Zone Propagation Velocity

Figures 4.23a-d show the  $v_{nz}$  measurements of samples CLM, FZK, HTR1 and HTR2 as a function of transport current at several  $(B_0, T_0)$  settings and a constant critical current of 45 A.

The same reasons that cause MQE values in these MgB<sub>2</sub> conductors to be significantly higher than in low- $T_c$  conductors, also cause the  $v_{nz}$  values to be significantly lower. The  $v_{nz}$  values fall in the range from 1 to 50 cm/s, compared to 10 to 100 m/s in low- $T_c$  conductors [48, 199, 208, 214]. Just like with MQE, this difference originates mainly from higher temperature margins and also higher  $c$  at elevated temperatures. The temperature dependence of  $v_{nz}$  is governed by similar considerations. The analytical approximation

$$v_{nz} = \frac{J_m}{c} \left\{ \frac{\rho_m \kappa_m}{(T_c - T_0)} \right\}^{1/2} \quad (4.17)$$

shows that there is a competition between the increasing heat capacity and the decreasing temperature margin. Between 5 K and 20 K, the temperature dependence of  $c$  dominates and  $v_{nz}$  decreases with temperature. Approaching  $T_c$ , however, this balance changes and when the baseline temperature  $T_0$  is increased further from 20 to 25 K,  $v_{nz}$  ceases to decrease. Again similarly to MQE, the



**Figure 4.23:**  $v_{nz}(I_0/I_c)$  measurements at an  $I_c$  of 45 A and at different  $(B_0, T_0)$  combinations of samples CLM (a); FZK (b); HTR1 (c); and HTR2 (d). The **solid lines** are a guide to the eye.

temperature margin decreases and in this range starts to dominate over the temperature dependence of the heat capacity.

Comparing the four samples, it is clear that sample CLM, which is the most stable one in terms of MQE, also has the lowest  $v_{nz}$ . Again, it is the relatively low filling factor of this conductor that results in relatively low current densities and consequently relatively low heat generation. In contrast, sample FZK has a cross-section that is almost a factor 4 lower than of sample CLM, so that  $v_{nz}$  can be expected to be significantly higher. The reason that  $v_{nz}$  of the two samples are nevertheless rather similar is that even when the lumped resistivity of sample FZK is a factor of 4 higher than that of sample CLM, its lumped thermal conductivity is a factor 40 lower (see figures B.3b and B.2b in appendix B). Equation (4.17) then shows that  $v_{nz}$  in sample FZK is suppressed by the poor thermal diffusivity of

the matrix material. Accordingly, since heat remains localised it can be expected that the temperature at the point of disturbance (hot spot temperature) will rise faster than in sample CLM. In §4.5.3 this will be verified with model calculations.

Comparing samples HTR1 and HTR2, it can be seen that their  $v_{nz}$  values differ by a factor  $\sim 2$ . Clearly, the replacement of Monel with Glidcop reduces  $v_{nz}$ , since both current densities as well as temperature margin of the two samples are similar. However, based on the available thermal conductivity and electrical resistivity data of Glidcop this reduction is unexpected. Due to the presence of a pure Cu core in both conductors, the changes in the overall  $\rho$  and  $\kappa$  values are limited. The lumped electrical resistivity of sample HTR2 is approximately a factor 3 lower (figure B.3b), while the lumped thermal conductivity is factor of 3 higher (figure B.2) than in sample HTR1 at a temperature  $T_s = \frac{1}{2}(T_{cs} + T_c)$ . Furthermore, at this temperature the heat capacities of the two conductors are about the same. Equation (4.17) then predicts similar propagation velocities. The discrepancy between analytical prediction and observed data is likely due to the rather high uncertainty in the thermal conductivity and electrical resistivity data of Glidcop. As discussed in appendix B, data of Glidcop is found only for temperatures above 100 K [216] and data below 100 K is extrapolated, which causes significant uncertainties.

### 4.5.2 Comparing Measurement and Model

In order to demonstrate the predictive value of the parallel-paths model calculations, the experimentally determined MQE- and  $v_{nz}$  values are compared with the a-priori model calculations.

It is also shown that for the mono-filamentary conductor FZK the 1-dimensional model is unable to predict  $v_{nz}$  and MQE accurately, since this conductor does not fulfill requirement (4.6) and a significant radial temperature gradient occurs across the superconducting core.

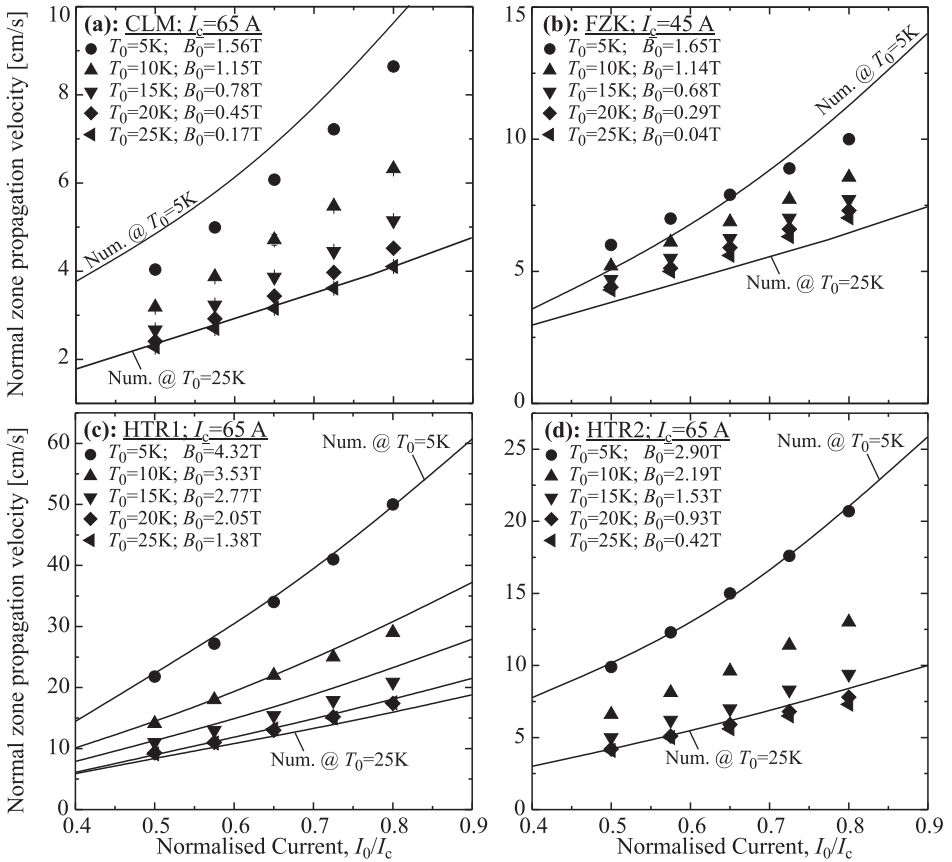
Finally, it is shown by how far uncertainties in the material properties influence the model predictions of MQE and  $v_{nz}$ .

#### Normal Zone Propagation Velocity

In figures 4.24a-d, the parallel-paths model calculations for  $v_{nz}$  with the measured data of samples CLM, FZK, HTR1 and HTR2 are compared.

For samples CLM, FZK and HTR1 the model results are a-priori calculations, involving no free parameters. Even so, satisfactory agreement between measurements and calculations is generally observed. For sample HTR2 initial attempts to model  $v_{nz}$  with extrapolated data proved unsatisfactory (typically deviating  $\sim 10\%$ ). Therefore  $\kappa$  of this conductor was used as an adjustable parameter. The final thermal conductivity of Glidcop was set to 75% of that of Cu with an RRR of 100 at 5 T. Compared to the published data of Glidcop above 100 K, this is about 15% lower.

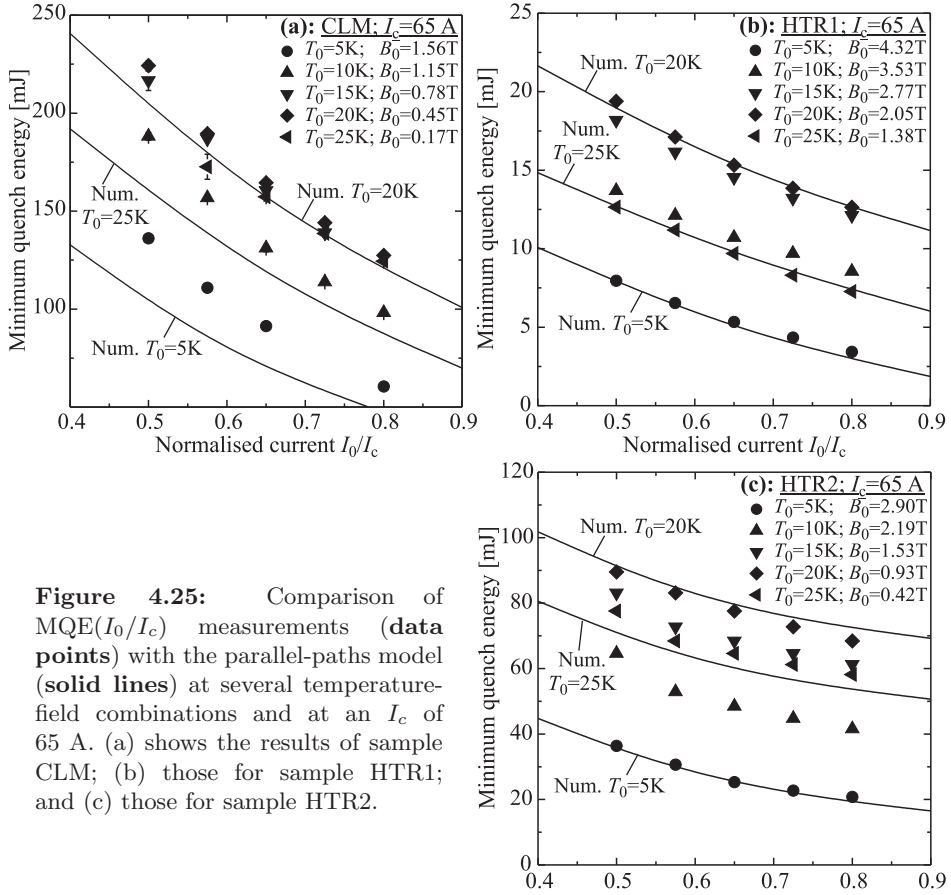




**Figure 4.24:** Comparison of  $v_{nz}(I_0/I_c)$  measurements (data points) with parallel-paths model calculations (solid lines) at several  $(B_0, T_0)$  settings for samples CLM (a); FZK (b); HTR1 (c); and HTR2 (d).

For sample CLM, the measured data at lower temperatures are systematically  $\sim 10\%$  lower than the a-priori calculations. This indicates a lower Ohmic heating than expected. As shown in figure 4.20a, sample CLM has a Cu stabiliser in the center of the composite, while the filaments are directly surrounded by the poor conducting Ni and Fe. This relatively complicated lay-out can cause a different distribution of current- and heat fluxes than assumed in the simple 1-dimensional parallel paths approximation.

For the mono-core sample FZK, the measured data at  $T_0 = 5$  K shows a different trend than the calculations. As will be shown below, sample FZK does not satisfy requirement (4.6) for 1-dimensional modeling and a temperature gradient is expected to exist within the superconducting core. In this case the 1-dimensional parallel-paths model no longer fully describes the thermal stability. Furthermore,



**Figure 4.25:** Comparison of MQE( $I_0/I_c$ ) measurements (data points) with the parallel-paths model (solid lines) at several temperature-field combinations and at an  $I_c$  of 65 A. (a) shows the results of sample CLM; (b) those for sample HTR1; and (c) those for sample HTR2.

in the absence of the relevant material properties of its CuNi18Zn20 matrix, these properties are estimated based on the available data for a CuNi30 alloy.

### Minimum Quench Energy

In figures 4.25a-c MQE( $I_0/I_c$ ) measurements on samples CLM, HTR1 and HTR2 with the corresponding model calculations at several ( $B_0, T_0$ ) settings are compared.

As explained in §4.4.1, the quench heater that is used is a relatively simple wire-wound heater. The main uncertainty therefore is the amount of heat that is actually transferred from the heater to the sample: a large amount of heat remains localised in the heater itself since it has a similar enthalpy as the sample. A comparison between measured data and model calculations of MQE can therefore also serve as a determination of the “efficiency” of the quench heater.

For all three conductors the calculated MQE values fall within  $20 \pm 5$  % of

the measured ones. Thus, despite the uncertainties caused by the mounting of the quench heater (how tight the heater wire is wound and the amount of GE varnish used), the efficiency of the heat transfer from quench heater to sample is relatively constant.

Taking into account this 20 % efficiency, it follows from figures 4.25a-c that the calculated and measured results for MQE of sample HTR1 agree within  $\sim 5\%$  over the whole measured  $(I_0, B_0, T_0)$  range.

The calculated MQE results for sample CLM tend to be systematically lower than the measured data. Not only may this be caused by the uncertainty in the heat transfer from quench heater to sample but, just as with the  $v_{nz}$  calculations, it is also possible that the heat flux- and current distribution in the relative complex cross-section are not fully described by a simple 1-dimensional model.

In contrast to samples CLM and HTR1, the numerical results of HTR2 are not based on a-priori calculations, but are generated by fitting the calculated- to measured  $v_{nz}$ , using  $\kappa$  of Glidcop as a fitting parameter. Nevertheless, when this fitted  $\kappa$  is used together with the  $20 \pm 5\%$  efficiency of the heater this leads to satisfactory agreement between measured- and calculated MQE values over the measured  $(I_0, B_0, T_0)$  range.

### Validity of the 1D Model

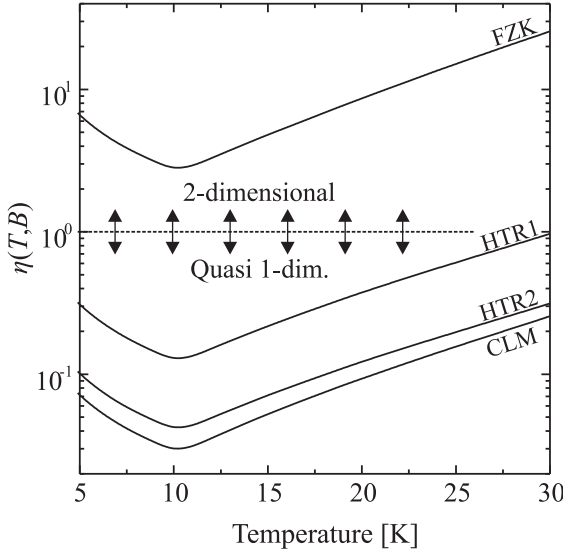
In §4.2.2 it was discussed that for 1-dimensional modeling to be accurate (assuming lumped values for the composite volumetric heat capacity, thermal conductivity and electrical resistivity) the thermal diffusion time of the filaments ( $\tau_{T,f}$ ) must be smaller than the magnetic diffusion time of the whole composite ( $\tau_{m,c}$ ), see equation (4.6). If this is not the case, significant radial temperature gradients occur across the filaments so that  $J_c$  and heat generation become radially inhomogeneous. Assuming round filaments- and conductors, this requirement was written as:

$$\eta(T, B) = \left(\frac{R_f}{R_c}\right)^2 \left(\frac{D_{m,c}(T, B)}{D_{T,f}(T, B)}\right) < 1, \quad (4.11)$$

with  $\eta(T, B)$  a temperature- and field dependent factor. The  $T$  and  $B$  dependence originates from the fact that  $c$ ,  $\kappa$  and  $\rho$  all depend on temperature and field.

Figure 4.26 shows the factor  $\eta$  as a function of temperature for the four samples. The relevant material properties of sample FZK are field independent, while the presence of high-purity Cu in samples CLM, HTR1 and HTR2 causes a significant field dependence of  $\eta$ . As a worst case, the plotted values of  $\eta$  for these samples are taken at 5 T, which is outside the field-range that is applied to the samples. For the magnetic diffusivity, the lumped electrical resistivity is taken with the MgB<sub>2</sub> filaments in the normal state.

Clearly, sample FZK does not fulfill the requirement for 1-dimensional modeling, mainly due to the poor electrical- and thermal conductivity of its matrix material. It should be noted, however, that the deviation between measurements



**Figure 4.26:** Temperature dependence of requirement (4.11) for 1D approximation of MQE and  $v_{nz}$  for the four samples. Data of samples CLM, HTR1 and HTR2 are taken at  $B_0 = 5$  T.

and model calculations is still rather small (see figure 4.24d), especially considering the uncertainties in the relevant material properties of its CuNi18Zn20 matrix.

The figure also shows that sample CLM does fulfill requirement (4.11), despite the fact that the comparison between measurements and model calculations show some discrepancy (figures 4.24a and 4.25a). As argued above, the deviations might well be due to the relatively complex cross-section and corresponding current- and heat flux distribution in this conductor. The magnetic diffusivity  $D_{m,c}$  in requirement (4.11) accounts for the lumped  $\rho$  of the whole composite, while it is not excluded that electrical field gradients occur across the sample. To verify such a scenario, a 2 or 3-dimensional model [217] needs to be used to simulate these MQE and  $v_{nz}$  measurements.

### Uncertainties in Materials Properties

The main uncertainties originate in the unknown thermal conductivity and electrical resistivity of some matrix materials. In this paragraph, the influence of uncertainties in the lumped volumetric heat capacity  $c$ , in the matrix thermal conductivity  $\kappa_m$  and in the matrix electrical resistivity  $\rho_m$  on the calculated MQE and  $v_{nz}$  values are compared. As an example, model calculations of sample HTR1, which show a good agreement with the measurements, are used to study the effects. Figures 4.27a and b show how the predictions for MQE and  $v_{nz}$  change

when  $c$ ,  $\kappa$  and  $\rho$  of the matrix are estimated 20 % too high with respect to the literature values.

When MQE is calculated with values for  $c$  that are estimated too high, the calculated value of MQE is also  $\sim 20$  % higher than measured. This can be expected, since in such a case 20 % more heat is needed to bring the temperature of a length  $l_{\text{MPZ}}$  of the conductor above  $T_c$  (equation (4.13)). When  $\kappa_m$  is estimated 20 % too high, the longitudinal cooling at both ends of the normal zone is overestimated. This results in a  $\sim 9$  % overestimation of MQE. For a 20 % higher  $\rho_m$ , the heat generation is overestimated, resulting in a 9 % underestimation of MQE. Using the analytical approximations (4.12) and (4.13) the influence of the uncertainty in  $c$ ,  $\kappa_m$  and  $\rho_m$  can also be deduced analytically:

$$\frac{\Delta\text{MQE}}{\text{MQE}} = \frac{\Delta c}{c} + \frac{\Delta\kappa_m}{2\kappa_m} - \frac{\Delta\rho_m}{2\rho_m}, \quad (4.28)$$

with  $\Delta\text{MQE}$ ,  $\Delta c$ ,  $\Delta\kappa_m$  and  $\Delta\rho_m$  the absolute uncertainty of the corresponding parameters. Using the 20 % overestimated properties in (4.28), it can be seen that this equation describes the influence on the model calculations rather well, despite the fact that it neglects the detailed heating function.

Uncertainties of 20 % in the thermal conductivity and electrical resistivity of MgB<sub>2</sub> (rather than the matrix resistivity  $\rho_m$ ) have in this example only a negligible influence on MQE, mainly because of the relatively low fill factor and the presence of highly-conducting Cu in the matrix.

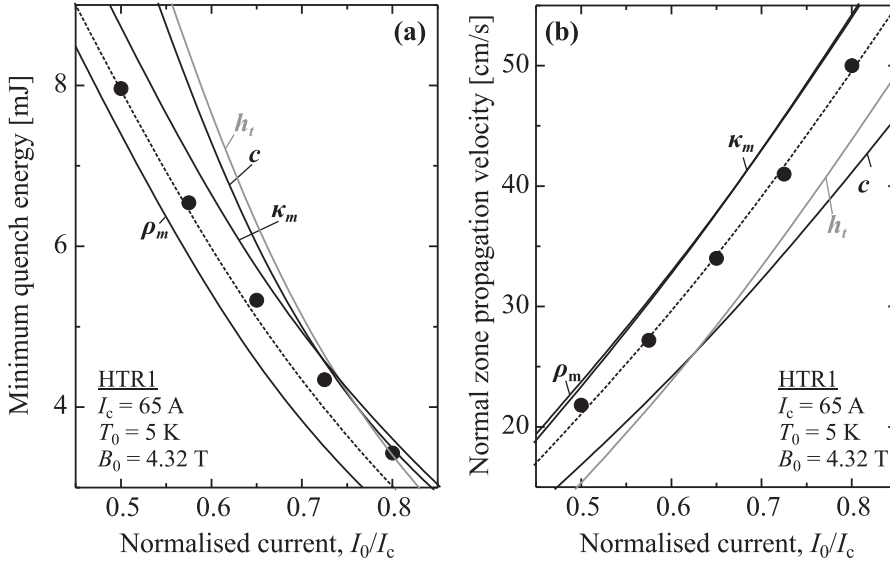
A calculation of  $v_{nz}$  with a  $c$ -value that is 20 % too high leads to a 15 % underestimation of  $v_{nz}$ , which is expected since  $c$  directly influences the diffusion of heat. In contrast, the overestimation of  $\kappa$  leads to an expected diffusivity that is too high and  $v_{nz}$  is overestimated by  $\sim 9$  %. As expected from equation (4.17) a similar effect occurs when the matrix resistivity  $\rho_m$  is estimated too high, also leading to a  $\sim 9$  % overestimation of  $v_{nz}$ . Using the analytical expression for  $v_{nz}$  (4.17), the influence of uncertainty in the material properties becomes:

$$\frac{\Delta v_{nz}}{v_{nz}} = -\frac{\Delta c}{c} + \frac{\Delta\kappa_m}{2\kappa_m} + \frac{\Delta\rho_m}{2\rho_m}, \quad (4.29)$$

with  $\Delta v_{nz}$  the absolute uncertainty of  $v_{nz}$ . Similar as with equation (4.28), the observed uncertainty is described rather well with (4.29).

For similar reasons as with MQE, an uncertainty in the thermal conductivity and electrical resistivity of MgB<sub>2</sub> has, for this specific conductor, negligible influence on  $v_{nz}$ .

A further factor introducing uncertainty is the lateral heat leak from the conductor to the sample-holder during the experiments, which can lead to a higher MQE and lower  $v_{nz}$  than one would expect from an ideal adiabatic case. As discussed in §4.4.3 and in appendix C, the thermal resistance from sample to the sample-holder is relatively low ( $R_{t,h} \approx 1000$  K/W), compared to the longitudinal thermal resistance of the sample itself ( $R_{t,12} \approx 2750$  K/W). Figures 4.27a and b show also how this lateral heat leak influences MQE and  $v_{nz}$ .



**Figure 4.27:** Calculated influence on MQE (a) and  $v_{nz}$  (b) of estimating the volumetric heat capacity  $c$ , thermal conductivity of the matrix  $\kappa_m$  and electrical resistivity of the matrix  $\rho_m$  20 % too high. Also shown is the influence of a lateral heat leak from sample to sample-holder with a heat transfer coefficient  $h_t$  of 40 W/m<sup>2</sup>K (curves labeled as “ $h_t$ ”). The dashed lines are the simulations as presented in figures 4.24c and 4.25b. The data points show the measured MQE and  $v_{nz}$ .

The assumed heat transfer coefficient is  $h_t = 40$  W/m<sup>2</sup>K, which leads to a 17 % increase in MQE and a 18 % reduction of  $v_{nz}$  compared to the adiabatic situation. It should be noted, however, that the measured thermal resistance between sample and sample-holder of  $R_{t,h} \approx 1000$  K/W corresponds to a heat transfer coefficient of only  $h_t = 2$  W/m<sup>2</sup>K, so that the actual experimentally realised differences with the MQE and  $v_{nz}$  in the ideal adiabatic case are negligible.

### 4.5.3 Influence of Matrix Material and $n$ -value

Having established the reliability of the 1-dimensional numerical model by directly comparing it to the experimental results, the model can now be used to study the influence of various parameters on the stability in an explicit way, i.e. varying only one parameter while keeping all others constant.

Clearly, the choice of matrix material significantly influences the conductor’s response to a thermal disturbance, with highly conducting materials significantly enhancing thermal stability in terms of MQE. In the experimental results presented above however, direct comparison between different matrix materials is some-

what complicated, since the matrix materials also influence the  $J_c$ - and  $n$ -value of a conductor. In this paragraph parallel-paths model simulations are presented on an “imaginary” conductor, keeping the  $J_c(B,T)$  and  $n(B,T)$  performance as well as the filling factor constant but assuming two different matrix materials, namely relatively poorly conductive Fe and highly conducting Cu. For the simulations the  $J_c(B,T)$ ,  $n(B,T)$  surfaces and filling factor of sample HTR1 are adopted.

Furthermore, sample HTR1 shows relatively low  $n$ -values of  $\sim 10$ , which is not uncommon in  $\text{MgB}_2$  conductors. However, very high  $n$ -values of  $\sim 100$  can also occur [119]. To study the influence of the  $n$ -value on both MQE and  $v_{nz}$ , two hypothetical samples are modeled, one with a constant  $n$ -value of 10 and one with an  $n$ -value of 100. Both conductors are assumed to have a matrix and  $J_c(B,T)$  surface identical to sample HTR1.

In the experiments temperature and field were set in such a way that the critical currents were relatively low. Experimentally, one of the limiting factors is the intrinsic thermal instability of the samples at currents larger than  $\sim 100$  A, which at low temperature and field results in a reduction of the measured “ $I_c(B)$ -curve” below the exponential behaviour (3.1). It is assumed that the conductors follow the  $J_c(B,T)$  and  $n(B,T)$  dependence of equations (3.1) and (4.21) up to higher currents. This idealised hypothetical case allows to study the response to a thermal instability of a  $\text{MgB}_2$  conductor at relatively high currents.

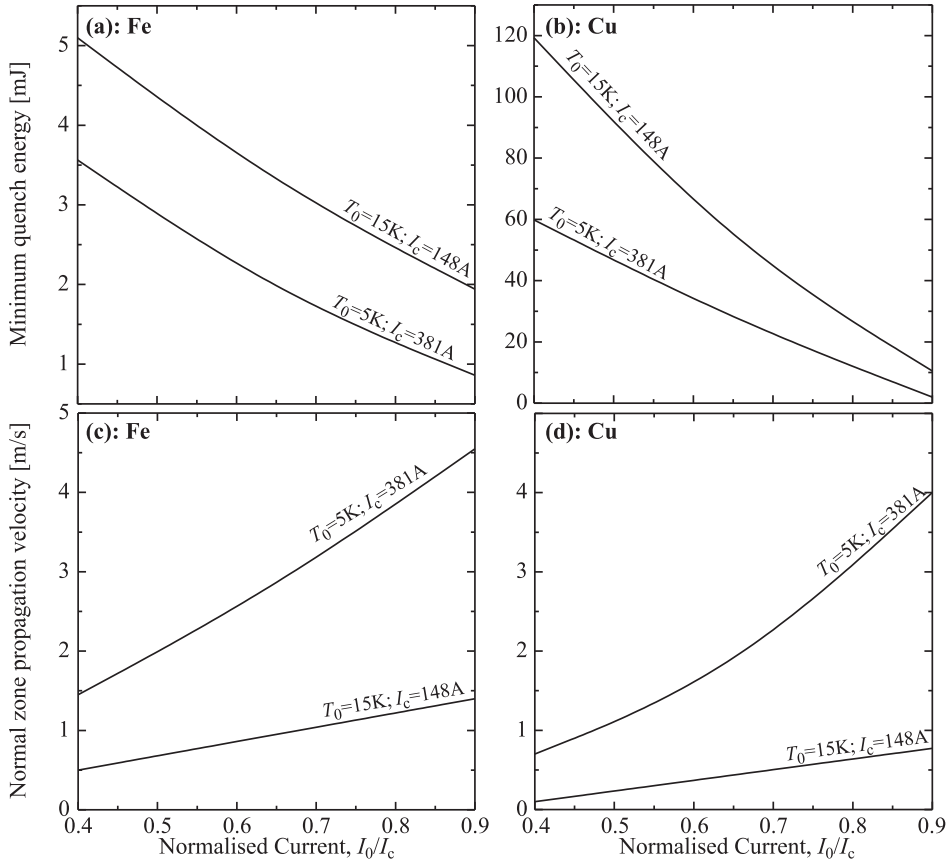
### Influence of Matrix Conductivity

Figures 4.28a and b show how the replacement of a relatively poorly conducting Fe matrix with a Cu one affects MQE at an applied field of 2 T and  $T_0 = 5$  K ( $I_c = 381$  A) or  $T_0 = 15$  K ( $I_c = 148$  A). For both temperatures, replacing Fe with Cu increases MQE by more than an order of magnitude and thus significantly enhances the thermal stability of the conductor.

Note that in the case of the Fe-stabilised conductor, the calculated MQE values are significantly lower than the values measured for sample HTR1 (figure 4.25b), which is caused by the higher current as well as by the lower conductivity of the Fe matrix with respect to the Nb/Cu/Monel matrix of HTR1. Interestingly, the Cu-stabilised conductor has significantly higher MQE values than those measured on sample HTR1, even if the assumed currents are 3 to 6 times higher.

In contrast to MQE,  $v_{nz}$  is hardly affected by replacing the Fe matrix with Cu, as shown in figures 4.28c and d. This can be understood from the analytical approximations (4.13) and (4.17), since a decrease in electrical resistivity is combined with an increase in thermal conductivity. As a result, the reduced heat generation ( $p_{\text{diss}} = J^2\rho$ ) is counterbalanced by the increased thermal diffusivity ( $D_{T,n} = \kappa/c$ , see equation (4.16)). Thus, provided that  $c$  remains constant (as is roughly the case with Fe and Cu at these temperatures), replacing a poorly conducting matrix material with a highly conducting one does not automatically imply that  $v_{nz}$  increases. For MQE on the other hand, the decrease in  $\rho$  and the corresponding increase in  $\kappa$  reinforce each other (4.12).

Compared to the  $v_{nz}$  measurements on sample HTR1 (figure 4.24b), the cal-



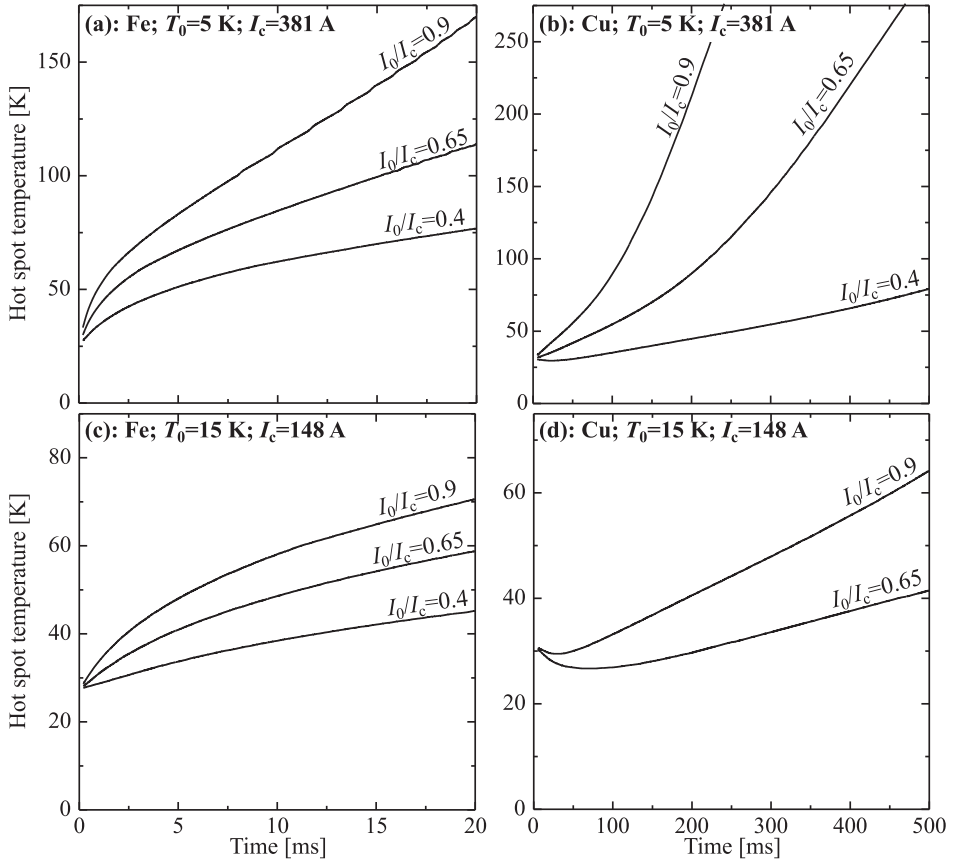
**Figure 4.28:** Comparison of MQE and  $v_{nz}$  for a Fe stabilised  $MgB_2$  conductor ((a) and (c)) and a Cu stabilised  $MgB_2$  conductor ((b) and (d)).

culated  $v_{nz}$  values at relatively high current increased to about  $\sim 1$  m/s rather than  $\sim 10$  cm/s. Clearly, a higher current causes a higher heat generation, so that  $v_{nz}$  increases.

The fact that  $v_{nz}$  in a Fe- or a Cu-stabilised conductor are similar has immediate consequences for the hot spot temperature. Since the propagation velocity remains similar, at any given time the volume in which heat is generated will also be similar, but the amount of heat generated itself will be almost two orders of magnitude higher in the Fe-stabilised conductor than in the Cu stabilised one. The time evolution of the hot spot temperature is presented in figures 4.29a-d.

Note that the time scales of the plots showing the Fe stabilised ((a) and (c)) and the Cu ((b) and (d)) stabilised conductors differ by more than two orders of magnitude. This means that the time lag between a thermal instability and a possible burnout is significantly increased by replacing Fe with Cu.





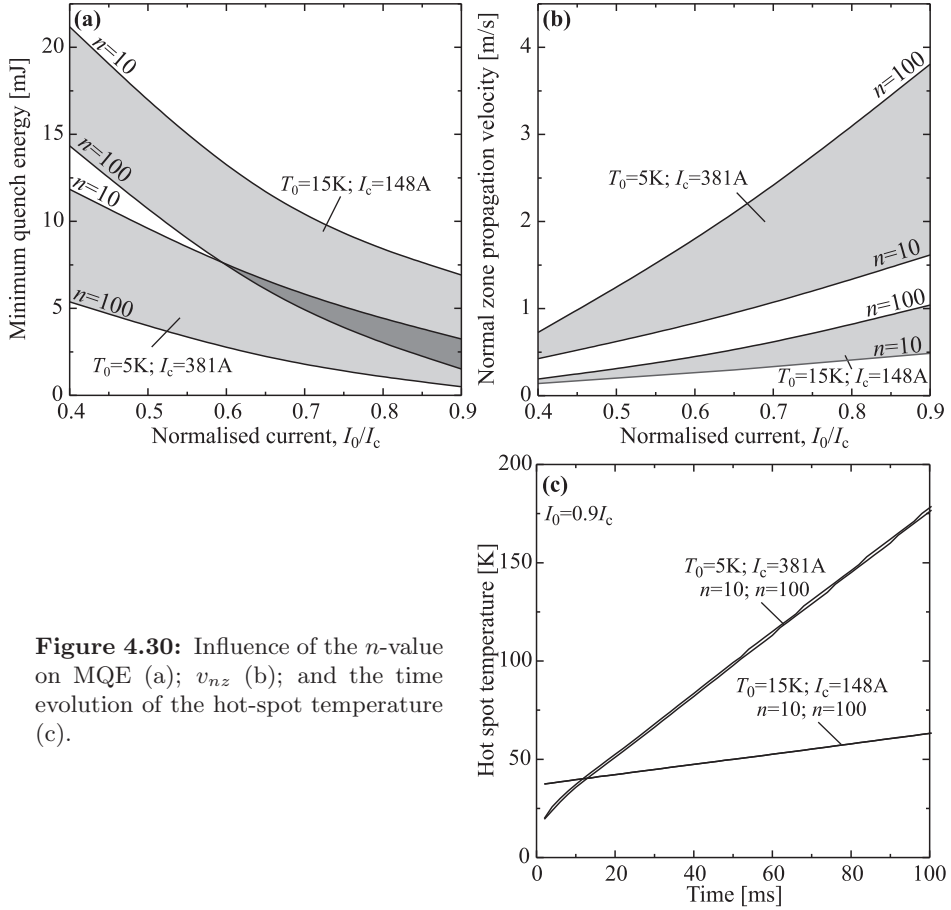
**Figure 4.29:** Comparison of the time evolution of the hot spot temperature of an Fe stabilised  $\text{MgB}_2$  conductor at 5 K and 15 K (a) and (c) and that of a Cu stabilised one (b) and (d). Note the different time scales between Fe and Cu.

When the baseline temperature  $T_0$  is increased from 5 to 15 K, the hot spot temperature increase becomes slower due to the reduced transport current as well as the increased heat capacity.

### Influence of the Superconductors' $n$ -value

In figures 4.30a and b it is shown how the  $n$ -value influences both MQE and  $v_{nz}$ .

Figure 4.8 showed how in conductors with a higher  $n$ -value, significant heating starts already at a lower temperature than with a relatively low  $n$ -value. Due to the higher heating of the conductor with  $n = 100$ , the MQE of this conductor is about a factor 2 lower than that of the conductor with  $n = 10$  at  $T_0 = 5$  K. However its  $v_{nz}$  at this temperature is a factor 4 higher. At  $T_0 = 15$  K, MQE for the



**Figure 4.30:** Influence of the  $n$ -value on MQE (a);  $v_{nz}$  (b); and the time evolution of the hot-spot temperature (c).

conductor with  $n = 100$  is about 25 % lower, while its  $v_{nz}$  is about 2 times higher. Thus, with increasing  $n$ -value  $v_{nz}$  increases twice as fast than MQE decreases.

Figure 4.30c shows the time evolution of the hot spot temperature for both  $n = 10$  and  $n = 100$ . Clearly, the two  $n$ -values result in virtually identical hot spot temperature increase. Indeed, different  $n$ -values only have an effect on the heat generation in the region immediately adjacent to the normal-superconducting interface. For larger times, the hot spot is relatively far from this interface and it is the normal state resistivity of the filaments that determines heat generation.

## 4.6 Conclusion

The influence of material properties and operating temperature on the normal zone behaviour in MgB<sub>2</sub> conductors was studied both experimentally and with a numerical parallel-paths model.

Representing a composite superconductor as electrical parallel-paths between superconducting filaments and normal conducting matrix makes it explicitly clear that the driving force of current diffusion during a thermal instability is the electric field. Moreover, the parallel-paths model accounts for the heat generation in all parts of the composite conductor, including the superconducting filaments.

Compared to analytical approximations and the “classical” current sharing model, this new model leads to a more detailed heating function in which the  $n$ -value of the superconductor becomes an important parameter. This is especially relevant for MgB<sub>2</sub> conductors, since their  $n$ -values vary widely depending on the fabrication route and operating conditions. In combination with a poorly conducting matrix material, heat generated in the MgB<sub>2</sub> filaments becomes a significant contribution to the overall heat generation.

In order to measure intrinsic MQE and  $v_{nz}$  values, a new quasi-adiabatic experimental set-up was developed, which is unique in the sense that three relevant operating parameters, current, magnetic field and temperature can be varied.

MQE measurements on three different MgB<sub>2</sub> conductors yield values ranging between 2 and 300 mJ, which is a factor  $\sim 1000$  higher than typical values observed for low- $T_c$  conductors. This difference is mainly caused by the larger thermal margin of the MgB<sub>2</sub> conductors and indicates that MgB<sub>2</sub> conductors are more stable than low- $T_c$  conductors.

Increasing the baseline temperature (while decreasing the magnetic field to keep  $I_c$  constant) reveals a non-monotonic temperature dependence of MQE, due to a competition between increasing heat capacity and decreasing temperature margin. Between 5 and 20 K, all conductors show an increase of MQE with temperature due to the rapid increase of  $c$ . Between 20 and 25 K, the reduced thermal margin dominates and MQE starts to decrease in all samples. Thus, MgB<sub>2</sub> conductors tend to be the most stable around 20 K, with MQE values ranging between 20 and 300 mJ.

$v_{nz}$  measurements on four different samples yield values between 1 and 100 cm/s, which is a factor 100 lower than those observed in low- $T_c$  conductors. This indicates that it will be more difficult to detect a normal zone in MgB<sub>2</sub> conductors than in low- $T_c$  conductors. Similar to MQE, this is caused by the larger thermal margin. When the baseline temperature is increased (and  $B_0$  decreased),  $v_{nz}$  decreases even further due to the increasing heat capacity, but reaches a minimum at 20 K with typical  $v_{nz}$  values of  $\sim 50$  cm/s.

Parallel-paths model calculations of MQE and  $v_{nz}$  generally show a good agreement with the measurements. For wires with a poorly conducting matrix or conductors that have a relatively complex lay-out, the 1-dimensional model results deviates somewhat from the measurements. This is likely due to radial thermal- or electric field gradients, so that a 2-dimensional model is expected to yield more accurate predictions.

Uncertainties in the model calculations are mainly caused by the absence of relevant data for some matrix materials. Ideally, the properties of these materials should be measured. This is especially the case for Glidcop, which is an interesting material that shows relatively good stabilising properties (high  $\kappa$  and low  $\rho$ ) and

is at the same time strong enough for powder densification.

With the 1-dimensional model, the influence of varying matrix properties can be separated from possible variations in  $J_c(B,T)$  and  $n(B,T)$ . It turns out that the matrix conductivity has a strong influence on MQE but hardly changes  $v_{nz}$ . A direct consequence of this latter observation is that the temperature in a wire with a relatively poorly conducting matrix will rise much faster than when a high-conductivity material is used. Thus, just like with any other technical superconductor, a highly conducting matrix is not only beneficial for stability but also reduces the risk of conductor burnout

The parallel-paths model predicts also that a high  $n$ -value of about 100 results in a higher Ohmic heat generation in the filaments than in a similar conductor with an  $n$ -value of about 10. At 15 K, this causes  $\sim 25\%$  lower MQE values and a factor  $\sim 2$  higher  $v_{nz}$ . However, the time evolution of the hot spot temperature is not affected the  $n$ -value since only the normal state resistivity causes heat generation at the centre of the normal zone. Thus at the expense of somewhat reduced stability, the detection of a normal zone becomes easier in conductors with high  $n$ -values.

Although MQE and  $v_{nz}$  are important factors in the normal zone behaviour of a superconductor, the time evolution of the hot spot temperature is also of significant practical importance. It can be readily studied with parallel-paths calculations, but to verify the calculated hot spot temperature it should also be measured experimentally.

## Chapter 5

# Mechanical Behaviour

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*Knowledge of the strain dependence of the critical surface is important for the design of MgB<sub>2</sub>-based magnets. The strain dependence of the critical current is studied experimentally and shows two distinct regions. Just like Nb<sub>3</sub>Sn, MgB<sub>2</sub> in compressive and moderate tensile strain shows a reversible behaviour and, just like Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>x</sub>, above a conductor-dependent reversible strain limit a steep and irreversible degradation of the critical current sets in. The reversible strain limit strongly depends on the degree of pre-compression that the matrix exerts on the filaments. In the reversible regime, the strain dependence of the critical current seems to have an electronic origin since also the critical temperature and the irreversibility field vary with strain. It is shown that the reversible strain dependence of the critical surface can be described with the strain dependence of just three critical parameters: the zero-field, zero-temperature extrapolation of the critical current density, the critical temperature and the zero-temperature extrapolation of the irreversibility field.*

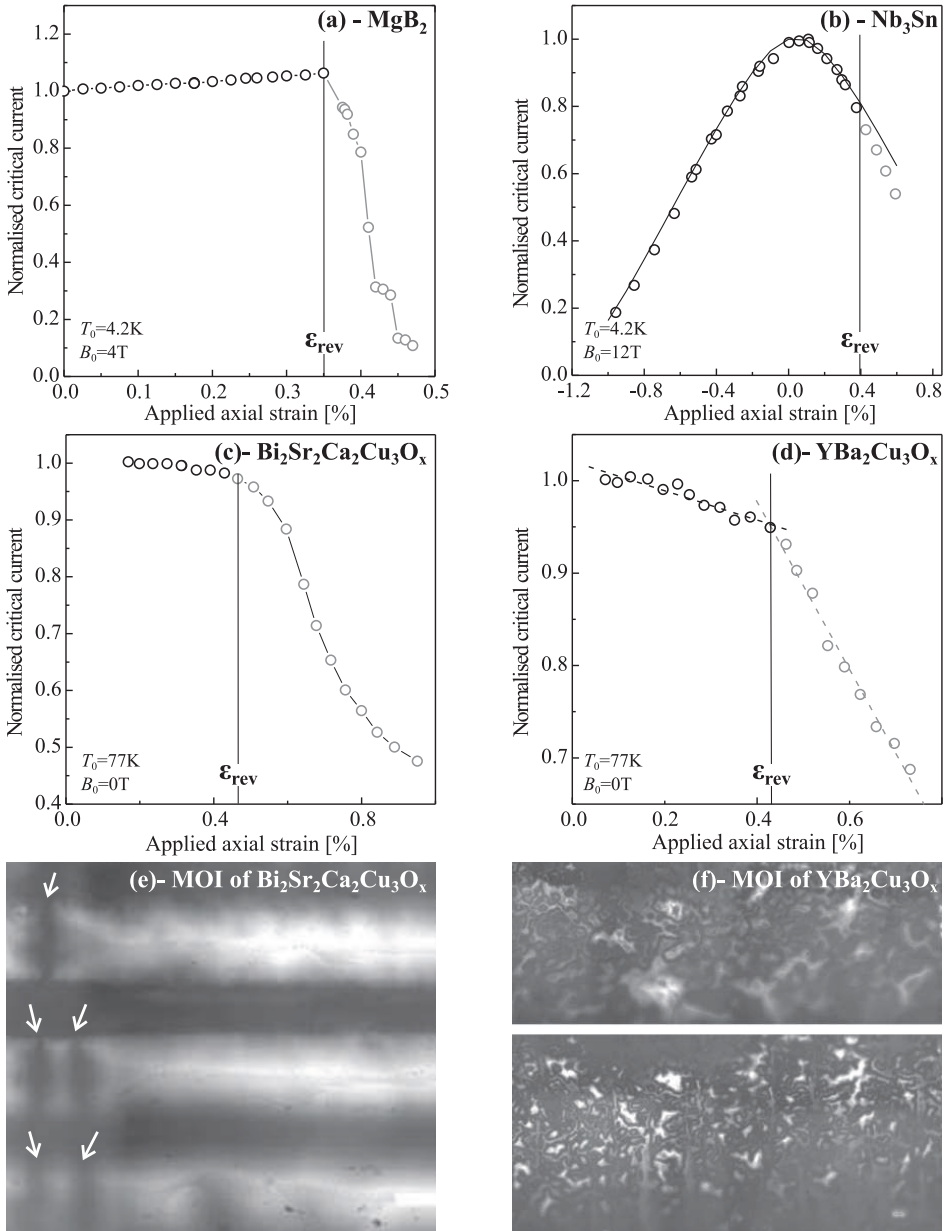
## 5.1 Introduction

Understanding the behaviour of superconductors subjected to mechanical strain is of fundamental as well as technical relevance. Among the first measurements of the behaviour of strained  $\text{MgB}_2$  conductors were those by Kitaguchi *et. al.* [218] and by Kováč *et. al.* [219]. A typical result of the critical current dependence on strain is shown in figure 5.1a. At sufficiently low strain the critical current depends linearly and reversibly on strain. Above a certain strain level called the reversible strain limit  $\varepsilon_{\text{rev}}$ , the critical current degrades steeply and this decrease is irreversible.

Comparing these observations to the well-studied strain dependence of  $I_c$  of other technical superconductors, i.e.  $\text{Nb}_3\text{Sn}$ ,  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  and  $\text{YBa}_2\text{Cu}_3\text{O}_x$  (figures 5.1b to d), three remarks can be made: 1) All these materials show a reversible-to-irreversible crossover at a strain level  $\varepsilon_{\text{rev}}$ . Indeed, they are all brittle materials and at high strain will develop microstructural cracks. In the ceramic  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  this was shown indirectly with an ultrasonic fracturing technique [220, 221], or through microscopic investigations of strained tapes [222]. Later, Van Der Laan [223] confirmed this cracking process in both strained  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  and  $\text{YBa}_2\text{Cu}_3\text{O}_x$  tapes with an in-situ magneto-optical technique (figure 5.1e and f). Similar cracks were observed with electron microscopy in  $\text{Nb}_3\text{Sn}$  conductors subjected to sufficiently high axial strain [224]. 2) just like  $\text{MgB}_2$ ,  $\text{Nb}_3\text{Sn}$  shows a reversible  $I_c(\varepsilon)$  dependence below  $\varepsilon_{\text{rev}}$ , which is believed to have an electronic origin [45, 46] and can be described by Ekin's scaling law [42] or later refinements (for an overview of such models see [12]). The strain scaling refinement by Ten Haken [44] is displayed as the solid line in figure 5.1b. In contrast,  $I_c$  appears to be virtually strain independent below  $\varepsilon_{\text{rev}}$  in  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  [225], while in  $\text{YBa}_2\text{Cu}_3\text{O}_x$  coated conductors sometimes modest [226, 227], but recently also a strong [228] reversible strain dependence is reported. 3) it was shown, e.g. by Passerini *et. al.* [222], that the value of  $\varepsilon_{\text{rev}}$  in  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  conductors is closely related to the pre-compressive strain that the matrix exerts on the filaments due to differential thermal contraction during cool-down. For  $\text{Nb}_3\text{Sn}$ , on the other hand, the thermal pre-compression is not related to  $\varepsilon_{\text{rev}}$ , but rather to the position of the peak in the  $I_c(\varepsilon)$  curve, while it is believed that microstructural cracks start to occur when the  $I_c(\varepsilon)$  data starts to deviate from its scaling [229]. As a further interesting observation, figures 5.1b to d also illustrate how the two ceramic conductors show an abrupt and steep degradation with strain, while the  $\text{Nb}_3\text{Sn}$  conductor merely slowly deviates from its scaling curve.

The strain dependence of the critical current in  $\text{MgB}_2$  appears to be between that of  $\text{Nb}_3\text{Sn}$  and that of the ceramic conductors. Like  $\text{Nb}_3\text{Sn}$ ,  $\text{MgB}_2$  has a clear reversible  $I_c(\varepsilon)$  regime, presumably indicating an electronic origin; and, just like the ceramic conductors, it has an abrupt degradation of  $I_c$  above  $\varepsilon_{\text{rev}}$ .

This chapter will focus on two issues: the factors determining the value of  $\varepsilon_{\text{rev}}$  will be explored; and the reversible  $I_c(\varepsilon)$  regime will be mapped out in detail, leading to a scaling law for this regime. In §5.2, the experimental techniques to apply axial strain to a conductor and measure the critical parameters are discussed



**Figure 5.1:** Comparison between the strain response of the critical current in  $\text{MgB}_2$  (a),  $\text{Nb}_3\text{Sn}$  (b),  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  (c) and  $\text{YBa}_2\text{Cu}_3\text{O}_x$  (d), after [49,219,223]. The solid line in (b) is calculated with a scaling relation for the  $I_c(\epsilon)$  data. All the conductors display irreversible behaviour when a composite-dependent reversible strain limit  $\epsilon_{\text{rev}}$  is exceeded. The figures at the bottom are magneto optical images (MOI): (e) reveals cracks in strained  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  filaments on the left part of the image, indicated by the arrows. The upper image of (f) shows an unstrained  $\text{YBa}_2\text{Cu}_3\text{O}_x$  coated conductor and the bottom image shows a strained sample in which cracks occur. Both MO images are adapted from [223].

and the samples selected for this study are described. §5.3 presents in detail the strain dependence of the critical current. Here the origin of the reversible strain limit is investigated and a general scaling of the reversible strain regime is introduced. Next the strain dependence of the critical temperature  $T_c$  and the irreversibility field  $B^*$  are described in §5.4 and §5.5 respectively. Combining the strain dependence of the critical current, the critical temperature and the irreversibility field, a scaling relation of the reversible strain response of the entire critical surface is developed in §5.6.

## 5.2 Experiment

As indicated above, the strain dependence of the critical parameters of  $\text{MgB}_2$  conductors can have either a microstructural or an electronic origin. In order to study this, strain has to be applied to a  $\text{MgB}_2$  conductor both in the compressive and tensile regime. Moreover, tensile stress needs to be high enough to be able to reach the reversible strain limit. In §5.2.1 several techniques are discussed that are used to apply strain to superconductors and to measure the critical parameters as a function of strain. It concludes with the U-shaped bending spring set-up used in this study.

It is explained in §5.2.2 how with this U-spring experiment incomplete current transfer to the  $\text{MgB}_2$  filaments can lead to an anomalous ohmic contribution in the voltage-current characteristic. A procedure to obtain the correct value of critical current and  $n$ -value is introduced.

In §5.2.3 the procedure is described to extract the variation of the critical temperature  $T_c$  and the irreversibility field  $B^*$  data with strain from the resistive transition as function of temperature and magnetic field respectively.

Lastly, the critical parameters may depend on the geometry and precursor powder of the conductor (i.e. tape/wire, matrix material, number of filaments). Therefore, several conductors have been selected to study the strain behaviour of the critical parameters. §5.2.4 describes the samples and the properties that are investigated.

### 5.2.1 Experimental Set-Up

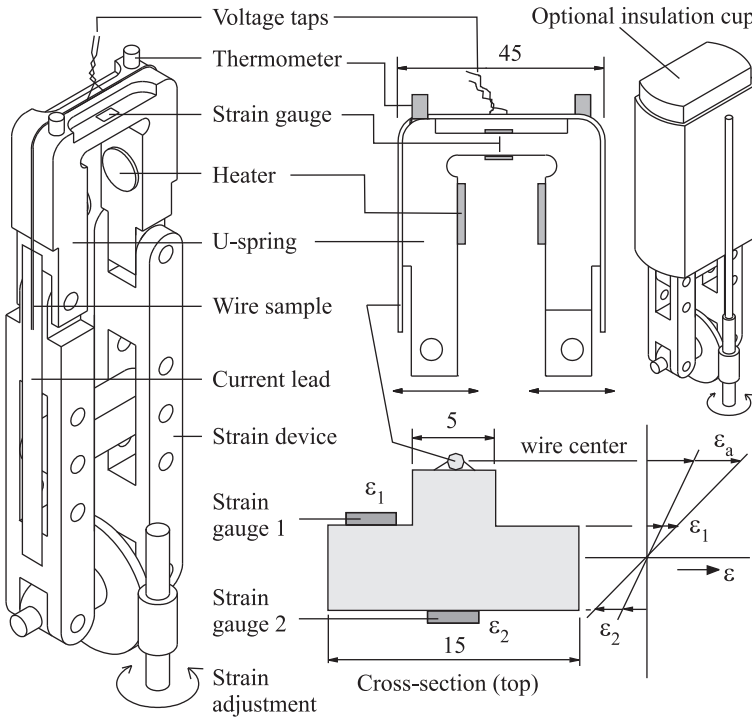
Several experimental methods to characterise the strain response of superconductors can be employed. In the first and most straightforward technique, the sample is clamped at both ends and an axial pulling force is applied (see for example [42]). The main advantages of this method are that it does not require a special sample shape, that the stress needed to reach a given strain level in the sample can be measured directly and that the experiment introduces no extra thermal pre-strain in the conductor. However, the sample can only be strained in tension and not in compression, limiting the experimental strain window considerably. A second type of device is the so called Walter's spring. A sample is mounted on a spiraled substrate and is subjected to axial strain by applying a torsion force to the sub-



strate [230]. The main advantage of this probe is the relatively long sample length ( $\sim 1$  m), which enables measurements at low electric field to be performed relatively easy. By soldering the sample over its full length on the substrate, it can easily be brought into the compressive strain regime. Moreover, the temperature can be varied by positioning the sample in a flow cryostat or a vacuum can. A drawback of this method is its complex mechanical structure, causing sample preparation and temperature regulation to be relatively difficult. The temperature response is relatively slow so that care has to be taken to avoid thermal runaways. Consequently, at elevated temperatures the maximum current that can be applied to the sample is limited [12]. To overcome this problem Godeke [49] recently developed a probe that has the same functionality as the Walter's spring, but with a structure that is "simplified" by reducing the amount of turns to one, limiting the sample length to  $\approx 10$  cm. Due to its shape, this device is referred to as the "Pacman". The simplified mechanical structure of the Pacman, in comparison with the Walter's spring, allows a relatively fast temperature regulation so that the risk of thermal runaways is limited. Both the Walter's spring and the Pacman device require the sample to be heat treated in a specific shape to avoid initial straining (bending) of the sample during mounting of the experiment.

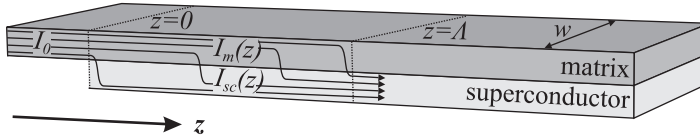
To avoid this complication, in this study a fourth method is used based on a straight beam section as part of a U-shaped bending spring [44, 231, 232]. This probe has been used for over a decade at the University of Twente. The major advantages are the simple sample layout, the relative ease with which the sample can be compressed, and the ease of temperature regulation. A disadvantage is the fact that the maximum length of the sample is limited by the bore of the magnet. The typical sample length of  $\approx 4$  cm so that current entrance can have a significant influence, depending on the matrix material of the studied sample. Moreover, due to the limited sample length the electric field resolution is low compared to the Walters' spring or the Pacman-device. Nevertheless, this device is used for its fast temperature regulation (thermal response time  $\sim 1$  s), which is especially important for characterising  $\text{MgB}_2$  conductors as typical temperatures are relatively high (up to 50 K).

A schematic overview of the U-shaped bending spring and its components is depicted in figure 5.2. The device is made of Ti-6Al-4V, which allows to apply high strain levels due to its high elastic strain limit of about 1.2 % [12]. Moreover, this material introduces a small thermal pre-compression of  $-0.05$  % to typical  $\text{MgB}_2$  samples [219]. The sample is mounted by soldering it over its full length onto a copper buffer layer on top of the straight beam section of the device. In this way, the sample can be brought into the compressive regime without buckling. Force is applied to the legs of the U-spring by an H-shaped lever system, which legs are adjusted by a left- and right threaded rod and wormgear. This bends the straight beam section of the U-spring so that the sample is axially strained in the compressive or in the tensile regime, depending on the direction of the rotation. In order to measure a superconducting transition (i.e. voltage-current for  $I_c$ , voltage-temperature for  $T_c$  or voltage-field for  $B_{c2}$ ), a pair of voltage taps are soldered to the sample, approximately 10 mm apart. To monitor the strain state of



**Figure 5.2:** U-shaped bending spring. Strain is applied by a rotating wormgear that drives a left- and right threaded rod pushing the legs of the device apart. A helium gas environment is created by placing the insulator cup over the device so that temperature can be regulated through a set of heaters and thermometers. Strain in the sample is monitored by extrapolating the readings of the two strain gauges.

the sample, two strain gauges are mounted on the U-spring so that their readings can be extrapolated to the wire axis position. Measurements above 4.2 K can be performed by covering the entire device with a polyamide cup creating a helium gas environment. The devices' mechanical structure causes the heat leaks and heat generation paths to be nearly symmetric. Residual temperature gradients along the sample can be minimised by using two thermometers and two heaters. For current transport to the sample, etched  $\text{Ag}/\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  current leads are used to limit the the heat leak from the sample to the helium bath (4.2 K), while allowing enough transport current to be able to measure critical currents. Turbulent convection of the helium gas in the polyamide cup plays a significant role at higher temperatures, increasing temperature fluctuations and thus rendering measurements of the critical parameters less precise. The sample is therefore isolated with a piece of neoprene foam to minimise the effect of temperature fluctuations to about 30 mK in the range of 4.2 K to 60 K operating temperature.



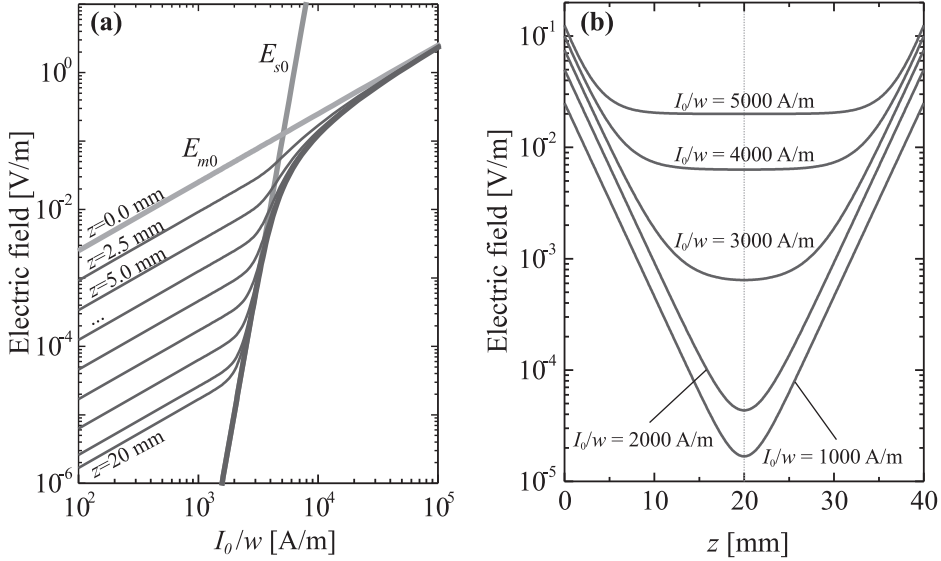
**Figure 5.3:** Current transfer in a superconductor-normal conductor composite, modeled in a “coated conductor” layout. The transport current  $I_0$  first enters the matrix at  $z = 0$  at which current is only flowing in the matrix. Along the entrance length, from  $z = 0$  to  $z \approx \Lambda$ , current is diffusing from the matrix into the superconductor (i.e.  $I_m$  decreases while  $I_{sc}$  increases). Within the entrance length an electric field is generated even for  $I_0 < I_c$ . For  $z \gg \Lambda$  nearly all the current is flowing in the superconductor so that no electric field is generated.

The total response time of the thermal system of the U-spring device is  $\sim 1$  s, allowing compensation for dissipation in the sample itself during the measurement of a voltage-current transition. With this method, critical currents up to 200 A have been measured at temperatures up to 20 K in  $\text{MgB}_2$  conductors [142] and up to 60 K in  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  samples.

### 5.2.2 Critical Current and $n$ -value

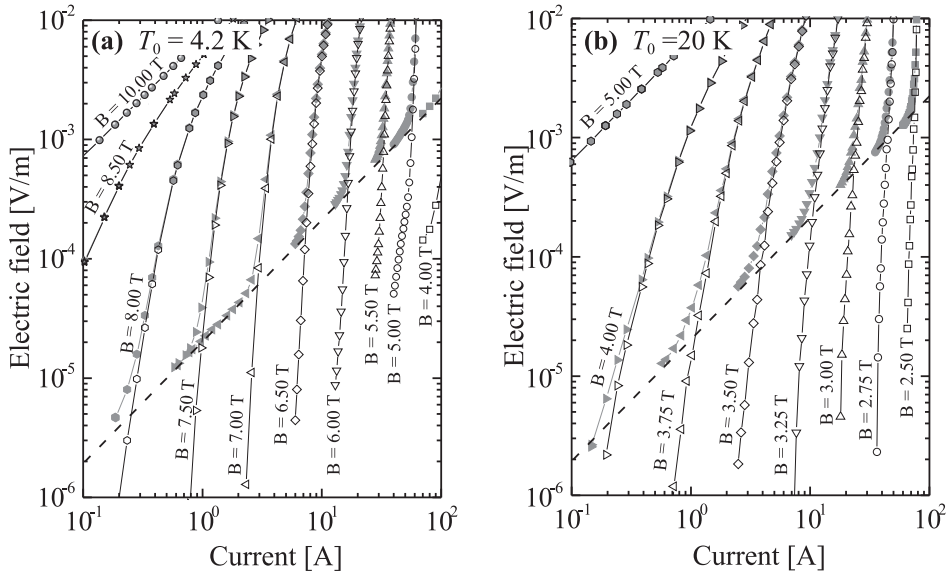
Determination of the critical current and the  $n$ -value from voltage-current data obtained with the U-spring requires a correction procedure. The short sample length together with the presence of a solder layer all along the sample cause current transfer effects to have a significant influence on the voltage-current transition.

Transfer effects are caused by current that passes non-uniformly through the matrix before it is carried by the superconducting filaments, generating an ohmic electric field. Schematically this process is depicted in figure 5.3. A composite conductor is modeled in a quasi 1-dimensional layout with width  $w$ . At the point of current injection at  $z = 0$  the transport current  $I_0$  is flowing in the matrix so that an Ohmic electric field is generated. Then, for  $z > 0$ , the current gradually diffuses from the matrix into the superconductor, dividing the total transport current  $I_0$  into  $I_m(z)$  and  $I_{sc}(z)$ . Across this length an electric field is generated by  $I_m(z)$  even when the total transport current  $I_0$  is lower than  $I_c$  of the superconductor. At distances much larger than the characteristic transfer length  $z \gg \Lambda$  nearly all the transport current has diffused into the superconductor so that no electric field is generated. A simplified model for current transfer that allows to calculate the characteristic entrance length is presented by Wilson [28]. Recently Dhallé [233] extended this model to include the non-linearity of the voltage-current transition of the superconductor (the power law  $E \propto J^n$ ). Typical results are depicted in figure 5.4. Figure 5.4a shows the voltage-current relations of the components in the composite.  $E_{m0}$  is the voltage-current relation of purely the matrix ( $\propto I_0$ )



**Figure 5.4:** (a) Calculations of the electric field in a parallel superconductor-normal conductor composite when a current is applied.  $E_{m0}$  and  $E_{s0}$  represent the electric field in the matrix ( $E_{m0} \propto I_0$ ) and a “pure” superconductor ( $E_{s0} \propto I_0^n$ ). The thin lines represent the electric field close to the point where current is injected.  $E_\infty$  represents the “intrinsic” voltage-current transition of the composite superconductor. In (b) the electric field profile along the conductor is displayed at various currents. Note that samples on the U-spring are indeed about 40 mm long, thus an ohmic contribution can be expected when the voltage-current transition is measured. A transfer length of  $\Lambda = 2.5$  mm is assumed.

and  $E_{s0}$  is the voltage-current relation of the bare superconductor ( $\propto I_0^n$ ). The thin lines represent the electric field in the matrix of a composite, such as the one depicted in figure 5.3, at various distances from the injection point.  $E_\infty$  is the electric field in the matrix of the composite far away from the injection point ( $z \rightarrow \infty$ ). As mention before, all the transport current at the injection point ( $z = 0$ ) is flowing in the matrix so that the electric field in the matrix of the composite corresponds with that of a “pure” normal conductor. With increasing distance from the injection-point, the amount of current flowing in the matrix ( $I_m(z)$ ) decreases, whilst current in the superconductor  $I_{sc}(z)$  increases, so that the ohmic contribution decreases along the conductor. At sufficiently large distances ( $z \gg \Lambda$ ), the ohmic contribution disappears for  $I_0 \ll I_c$  as all the transport current is carried by the superconductor ( $E_\infty$ ). When the total current  $I_0$  is increased and  $I_c$  is exceeded the electric field approaches  $E_{m0}$  since current diffuses back into the matrix, similarly to the current sharing process described in chapter §4.3.2.



**Figure 5.5:** Voltage-current characteristics of a  $\text{MgB}_2/\text{Fe}$  tape from University of Geneva at various magnetic fields and at temperature 4.2 K (a) and 20 K (b) measured on the U-spring sample holder (**solid symbols**). The ohmic contribution of the entrance effect is indicated by the **dashed line**, which is subtracted from the data to obtain the intrinsic voltage-current characteristic of the conductor (**open symbols**). These characteristics are fitted with the second order polynomial of (5.1) in order to obtain  $I_c$  and the  $n$ -value.

A profile of the electric field in the matrix along the length of the composite at different currents is depicted by 5.4b. The sample length that is assumed in this plot is approximately the length of a typical sample measured with the U-spring. The voltage-taps are placed at about  $z = 15$  mm and 25 mm, but even at the center of the sample at low currents a significant contribution of the entrance effect in the measured voltage-current transition can be expected. Indeed, when a reasonable value of  $E_c$  (e.g.  $10^{-4}$  V/m) is chosen, the data has been corrected in order to determine  $I_c$  and the  $n$ -values that are representative for long length samples.

An example of a voltage-current transition at various magnetic fields of a  $\text{MgB}_2/\text{Fe}$  tape measured with the U-spring is indicated by the solid data points in figure 5.5. Similarly to figures 5.4a and b, a significant ohmic contribution (indicated by the dashed line) is measured that is caused by current flow in the matrix. Strikingly, the same ohmic limit acts as a lower boundary for all voltage-current curves irrespective of the external magnetic field or of the temperature. This is easily understood when considering that the matrix material is pure Fe, which has a resistivity virtually independent of temperature and field

in the range under consideration. The value of the corresponding resistance (in this case  $\sim 20\mu\Omega/\text{m}$ ) thus depends only on the resistivity of Fe and on geometrical factors. Consequently, this point of the voltage-current characteristics is not representative for the intrinsic properties of the superconductor. In order to determine the intrinsic  $J_c$  and  $n$ -value, first the ohmic contribution is linearly fitted and the corresponding line is subtracted from the raw data, yielding the intrinsic voltage-current transition (i.e.  $E_\infty$ ) of the superconductor indicated by the open symbols. This (double logarithmic) intrinsic voltage-current curve is then written as a Taylor expansion around  $\ln(E) = \ln(E_c)$ :

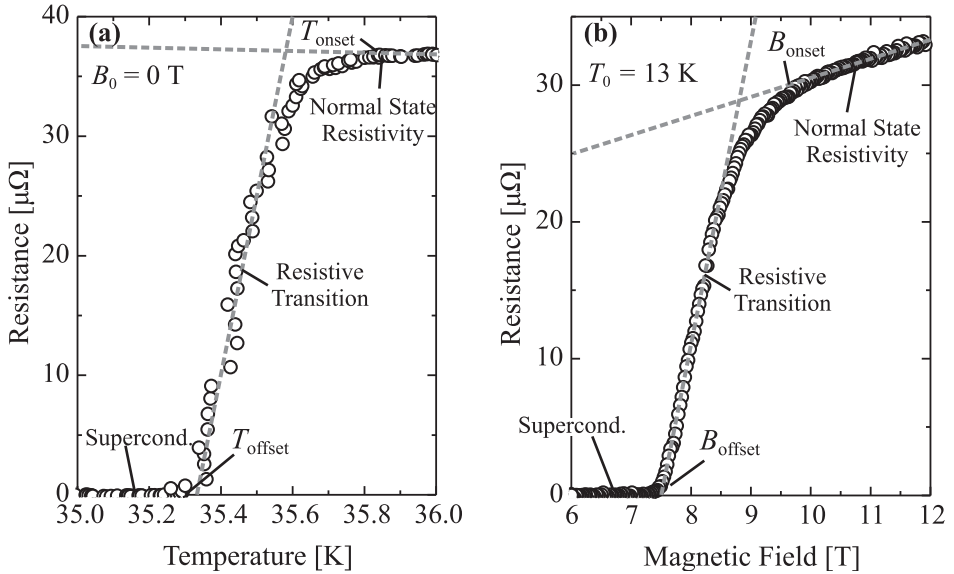
$$\ln J = a_0 + a_1 (\ln(E) - \ln(E_c)) + \frac{a_2}{2} (\ln(E) - \ln(E_c))^2. \quad (5.1)$$

A second-order expansion is used to take into account the curvature of the transition that is caused by the diffusion of current back into the matrix at currents  $J \gg J_c$ . The critical current density is obtained by fitting the second-order polynomial (5.1) to the data and taking  $J_c = \exp(a_0)$ , while its uncertainty can be found from  $\Delta J_c = J_c \Delta a_0$ . The  $n$ -value (the local slope of the voltage-current characteristic on a log-log plot) is simply given by  $n = a_1$ ;  $\Delta n = \Delta a_1$ .

### 5.2.3 Resistive Transition

If the critical current density in MgB<sub>2</sub> shows a reversible strain dependence, it might well be of an electronic origin. This suggests that not only  $I_c$  changes reversibly, but also the critical temperature  $T_c$  and the irreversibility field  $B^*$ . To verify if this is the case, in this study  $T_c$  and  $B^*$  are probed by resistive transition measurements, of which two examples are displayed in figure 5.6a and b ( $R$ - $T$  and  $R$ - $B$ , respectively). Both plots can roughly be separated into three parts: first there is the superconducting region at temperature (or field) below  $T_{\text{offset}}$  ( $B_{\text{offset}}$ ); secondly, above  $T_{\text{offset}}$  ( $B_{\text{offset}}$ ) the resistivity starts to rise towards the normal state (the resistive transition itself); and thirdly above  $T_{\text{onset}}$  ( $B_{\text{onset}}$ ) the superconductor is in the normal state. In other words,  $T_{\text{onset}}$  is defined as the temperature at which, during cool-down, the resistivity starts to drop below its normal-state value, while  $T_{\text{offset}}$  is the temperature at which the resistive transition is “complete” and the resistivity becomes zero. It should be noted that the resistive transition is not infinitely sharp. Not only are  $T_{\text{onset}}$  and  $T_{\text{offset}}$  well-separated temperatures, the on- and offset points are also “rounded” in such a way that their determination depends to some extent on the precision of the resistivity measurement. There are essentially three mechanisms that can determine the width of the transitions: thermal fluctuations, magnetic flux motion and sample inhomogeneity.

Even in the absence of any significant magnetic field and in pure samples such as for instance single crystals, thermal fluctuations will cause “smearing out” of both  $T_{\text{onset}}$  and  $T_{\text{offset}}$ . Above  $T_c$ , the paired electron state has a higher energy than unpaired ones. However, when the temperature is sufficiently close to  $T_c$ , this energy difference becomes comparable to  $k_B T$  so that there is a non-zero

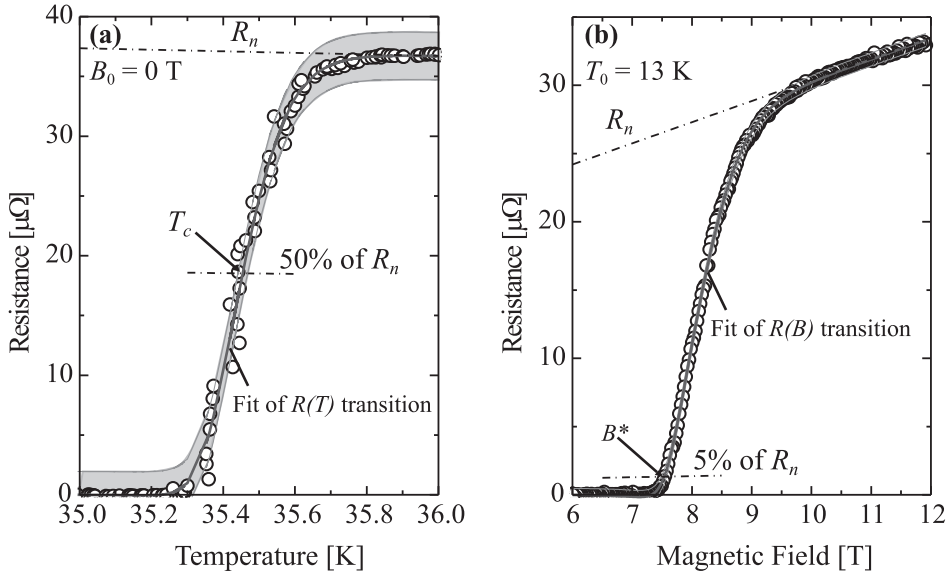


**Figure 5.6:** Example of the resistive transitions as function of temperature (a) and magnetic field (b) for an ex-situ  $\text{MgB}_2$  conductor fabricated at the Institute of Electrical Engineering in Bratislava, Slovakia.

probability for electrons to pair already. This so-called fluctuation conductivity can start to contribute already well above  $T_c$  and thus determines the value of  $T_{\text{onset}}$  [234]. Vice versa, the appearance of resistance at  $T_{\text{offset}}$  can occur well below  $T_c$  since thermal fluctuations can already break up electron-pairs.

The first occurrence of resistance at  $B_{\text{offset}}$ , well below the second critical field  $B_{c2}$ , in figure 5.6b is assumed to be caused by the motion of flux-lines, which is a dissipative process. The motion of flux lines is hampered by flux pinning [67] with a corresponding pinning energy. In general, this pinning energy is a decreasing function of temperature, field and current, which can become zero well below the  $B_{c2}(T)$  line. When this happens, the critical current becomes zero and a finite resistivity will be measured irrespective of the transport current. The corresponding magnetic field is called the irreversibility field  $B^*$ . When measured with a sufficiently low current density, the offset field in a  $R(B)$  curve such as the one in figure 5.6b will approximately correspond to this irreversibility field,  $B_{\text{offset}} \approx B^*$ . Due to the dissipative nature of the flux motion the exact position of  $B_{\text{offset}}$  depends on the transport current and if the transport current is too high,  $B_{\text{offset}}$  will be lower than  $B^*$ . For the same reason  $B_{\text{onset}}$  also depends on the transport current. But, when the current is sufficiently low,  $B_{\text{onset}}$  will approximate the point where the value of the superconductivity gap reduces to zero (when the field is ramped up) and thus approximates  $B_{c2}$ ,  $B_{\text{onset}} \approx B_{c2}$  [234].

A third factor influencing the shape of the  $R(T)$  and  $R(B)$  curves is sample ho-



**Figure 5.7:** The same  $R$ - $T$  and  $R$ - $B$  transitions as shown in figure 5.6, but indicating the points where the critical temperature  $T_c$  and irreversibility field  $B^*$  are defined. It also indicates the fit (solid line) of the measured data with expression (5.2) and the 99 % prediction band (gray area) from which the uncertainty in  $T_c$  and  $B^*$  are determined.

mogeneity. The filaments in a practical  $\text{MgB}_2$  superconductor are polycrystalline with the crystallites randomly ordered. Since the critical parameters of  $\text{MgB}_2$  in the direction of the  $ab$ -plane differ from those in the  $c$ -axis direction [235], crystallites will display a different behaviour depending on the (local) direction of field and current. Consequently, the width of the transitions can also be caused by distributed critical temperatures, critical fields and irreversibility fields [236]. Furthermore, inevitable non-superconducting impurities in the filaments can induce resistance developing before the intrinsic critical parameters are crossed. For example, Godeke [12] reported a significant reduction of the  $R$ - $B$  transition width in  $\text{Nb}_3\text{Sn}$ -PIT conductors when the reaction time is increased, which is attributed to a more homogeneous Sn distribution throughout the cross-section of the filament.

In order to characterise the strain dependence of the resistive transitions first  $T_c$  is defined at the 50 % intersection of the normal state resistivity with the  $R$ - $T$  transition. Secondly,  $B^*$  is defined as the field at which the  $R$ - $B$  curve intersects with the 5 % of the normal state resistivity curve. Figure 5.7a and b show the same resistive transitions as in figure 5.6 but include  $T_c$  and  $B^*$  with the 50 and 5 % normal state resistivities at which they are defined.

The measured transitions with temperature are obtained by applying a DC



current of 100 mA that toggles polarity at each data point, to compensate for offset or inductive voltages. While applying this current, the temperature is varied and magnetic field is fixed to obtain the  $R$ - $T$  curves such as that shown in figure 5.7a. In order to avoid apparent hysteresis between increasing- and decreasing temperature, a relatively low temperature ramp rate of 50 mK/min is used. Vice versa, to obtain the  $R$ - $B$  curve (figure 5.7b) the temperature is fixed while the magnetic field is varied at a ramp rate of about 0.2 T/min. This ramp rate is low enough to avoid a measurable hysteresis between ramping the magnetic field up and down.

In order to estimate  $T_c$  and  $B^*$  from the measured transitions, they are fitted with the following function:

$$R(x) = (a_1x + a_2)\frac{1}{2} \left\{ 1 - \frac{w_1}{x_{c1} - x_{c2}} \ln \left[ 2 \cosh \left( \frac{x - x_{c1}}{w_1} \right) \right] + \frac{w_2}{x_{c1} - x_{c2}} \ln \left[ 2 \cosh \left( \frac{x - x_{c2}}{w_2} \right) \right] \right\} \quad (5.2)$$

with,

$$x = T \text{ or } x = B.$$

This expression is essentially a step function that can be asymmetric, in which  $(a_1x + a_2)$  is the linear normal state resistivity, and  $x_{c1}$  and  $x_{c2}$  are centered at the offset and onset of the transition, respectively. Moreover, equation (5.2) allows the offset and onset to have a certain width (in contrast to a sudden increase of the resistance), which are proportional to  $w_1$  and  $w_2$  respectively. Then,  $T_c$  and  $B^*$  are the intersections of this fit with 50 % and 5 % of their normal state resistivities, respectively ( $0.5 \times (a_1x + a_2)$  and  $0.05 \times (a_1x + a_2)$ ). The uncertainty of  $T_c$  and  $B^*$  are obtained through the 99 % prediction band of equation (5.2), indicated by the gray area in figure 5.7a.

### 5.2.4 Samples

As pointed out before, type and uniformity of matrix materials can be of great influence on the critical parameters (see also §2.3.2) as well as the thermal stability (§4.5). The samples listed in table 5.1 have different matrix materials that are chosen either to provide enough strength to obtain a high densification of the filaments during deformation (using Ni, Fe or the alloys CuNiZn and AgMg) or to obtain sufficient thermal stabilisation (Cu). As will be shown below, the choice of matrix material also influences the strain response of the critical current. This is not unusual, already in the introduction (§5.1) it was mentioned that in A15 and ceramic superconductors the thermal expansion coefficient of the matrix material influences the point where the strain-dependence of the critical parameters switches from reversible to irreversible behaviour ( $\varepsilon_{\text{rev}}$ ).

Another parameter that varies from sample to sample and that can influence the strain response of the critical parameters is the powder itself, which can be either ex-situ/in-situ or doped/non-doped. For example, both  $B^*$  and  $B_{c2}$  are

**Table 5.1:** Overview of the investigated samples and their layout properties.  $f$  and  $f_i$  are the filling factor and the fraction of the  $i^{\text{th}}$  component in the matrix.

Sample	Powder	$f$ [%]	Matrix	$f_i$ [%]	# Filaments	Shape	Dimensions
CLM <sup>a</sup>	ex-situ	9	Ni/Cu/Fe	67/14/10	14	Tape	$A=0.7 \times 3.6 \text{ mm}^2$
FZK1 <sup>b</sup>	in-situ	50	Fe	50	Mono	Wire	$\varnothing = 1.2 \text{ mm}$
FZK2 <sup>b</sup>	in-situ	13	Cu/CuNiZn/Nb	56/24/7	7	Wire	$\varnothing = 0.9 \text{ mm}$
HTR <sup>c</sup>	in-situ	16	Nb/Cu/Monel	15/29/40	7	Wire	$\varnothing = 0.8 \text{ mm}$
IEE1 <sup>d</sup>	ex-situ	25	Fe	75	4	Square	$A = 1.2 \times 1.2 \text{ mm}^2$
IEE2 <sup>d</sup>	in-situ+SiC	26	AgMg/Nb	44/30	4	Square	$A = 1.1 \times 1.1 \text{ mm}^2$
UG1 <sup>e</sup>	ex-situ	30	Fe	70	Mono	Tape	$A = 0.4 \times 3.9 \text{ mm}^2$
UG2 <sup>e</sup>	in-situ	29	Fe	71	Mono	Wire	$\varnothing = 1.1 \text{ mm}$

<sup>a</sup> Columbus Superconductors, Genova (Italy)

<sup>b</sup> Forschungszentrum Karlsruhe, Karlsruhe (Germany)

<sup>c</sup> HyperTech Research, Columbus, Ohio (USA)

<sup>d</sup> Institute of Electrical Engineering, Bratislava (Slovakia)

<sup>e</sup> University of Geneva, Geneva (Switzerland)

enhanced by SiC addition so that their strain dependence might well be different from that in non-doped samples.

Also the geometry of the conductors will influence their strain response. The investigated samples are either rolled to tapes, drawn into wires or two-axially-rolled into square wires. The different deformation processes affect the texture of the MgB<sub>2</sub> grains [156] thus influencing the effective critical parameters (see also §5.2.3) and so the strain dependence. Moreover, when axial strain is applied to a conductor, also a transverse strain will be built up, as described by an effective Poisson ratio of the composite. The influence of the transverse strain components on the critical parameters of the superconducting filaments will depend on the geometry of the conductor [44] and thus the number of filaments.

### 5.3 Strain Dependence of the Critical Current

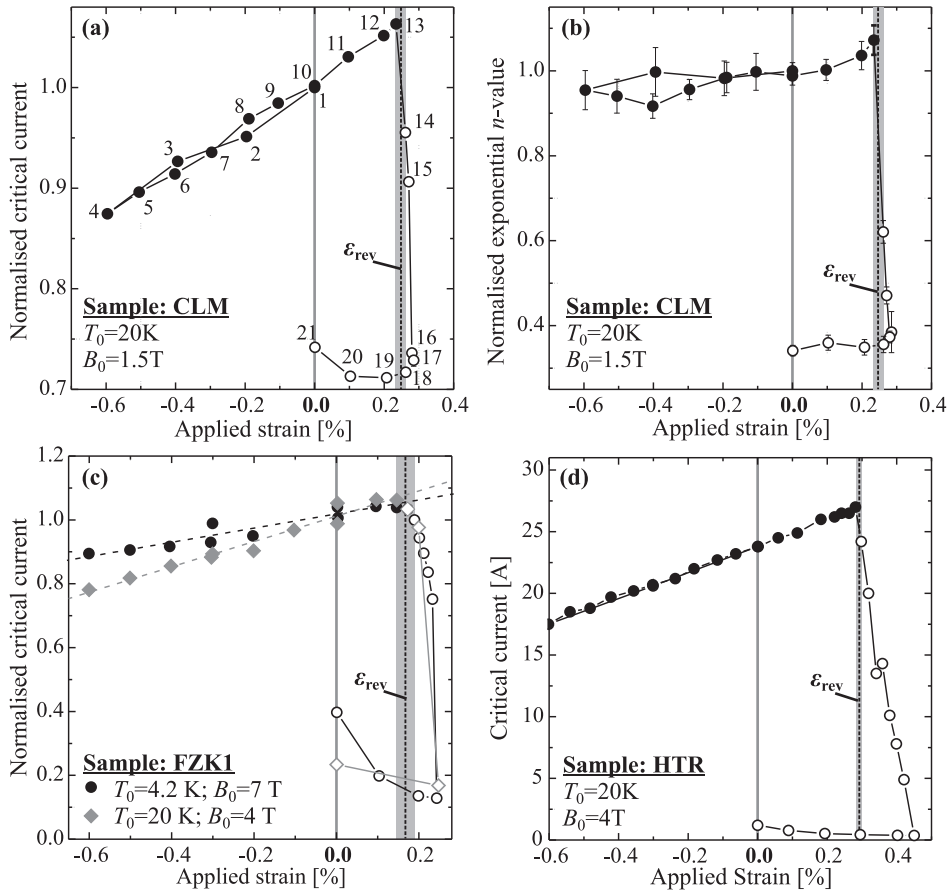
Figures 5.8a, c and d show the strain dependence of the critical current of samples CLM, FZK1 and HTR from table 5.1.

The sequence of the measurements is indicated by the numbers next to the data-points in figure 5.8a. The critical current is first measured at zero applied strain (data-point 1). Then, the applied strain is stepwise decreased into the compressive regime down to a minimum of approximately -0.6 %. At each step the critical current is measured (data-points 2 to 4). Next, the applied strain is stepwise increased back into the tensile regime (data-points 3 to 18). Finally, the strain is decreased again to 0 % (data-points 19 to 21).

The samples all show a similar behaviour in the sense that they all exhibit reversible linear strain response for compressive and sufficiently low tensile strain (data-points 1 to 13), while, when the tensile strain is increased above a certain reversible strain limit  $\varepsilon_{\text{rev}}$  (indicated by the gray band), the critical current decreases abruptly (data-points 13 to 18). When the applied strain is finally released again (data-points 19 to 21), the critical current does not recover. This behaviour is general for MgB<sub>2</sub> conductors and is also observed by other authors [218, 237]. These observations do not only hold for the critical current, but also for the  $n$ -value as indicated by figure 5.8b.

Comparing figures 5.8a, c and d shows that the reversible strain limit  $\varepsilon_{\text{rev}}$  is sample dependent ( $\varepsilon_{\text{rev}} \approx 0.23$  %, 0.18 % and 0.30 % for sample CLM, FZK1 and HTR respectively), while the two different measurements of sample FZK1 (figure 5.8c, taken from two different pieces of the same wire) show that the value of  $\varepsilon_{\text{rev}}$  seems to be independent of temperature and magnetic field. Taken together with the irreversible  $I_c(\varepsilon)$  and  $n(\varepsilon)$  behaviour above  $\varepsilon_{\text{rev}}$ , the fact that the value of  $\varepsilon_{\text{rev}}$  does not depend on  $T$  or  $B$  suggests that, just like in ceramic superconductors,  $\varepsilon_{\text{rev}}$  is strongly related to the degree of thermal pre-compression of the filaments by the matrix. In §5.3.1 this will be further investigated.

Although  $\varepsilon_{\text{rev}}$  does not seem to depend on the operating conditions, the strain response of the critical current in the reversible regime clearly does (figure 5.8c). This is reminiscent of the strain response of A15 materials as discussed in §1.3.2



**Figure 5.8:** General observations of the strain dependence of the critical current (a, c and d) and the  $n$ -value (b). The dashed lines indicate the reversible strain limit  $\epsilon_{\text{rev}}$  at which reversible strain response changes to irreversible behaviour. The corresponding gray bands indicate the uncertainty of  $\epsilon_{\text{rev}}$ , which is the window described by the last data-point in the reversible regime and the first data-point in the irreversible regime. (a) Strain response of the normalised critical current of sample CLM. The numbers indicate the order in which the critical current is measured as function of applied axial strain. (b)  $n$ -value as function of strain of sample CLM. (c) Strain dependence of the normalised critical current of sample FZK1 at two combinations of field and temperature. (d) Strain dependence of the critical current of sample HTR.

and §5.1 and suggests an electronic origin of the reversible strain response also for MgB<sub>2</sub>. A detailed study of the reversible strain response of  $I_c$  as a function of  $T$  and  $B$  will be presented in §5.3.2.

### 5.3.1 Reversible Strain Limit

In order to verify the correlation between the reversible strain limit and the thermal pre-compression in the superconducting filaments (like in BSCCO/Ag composites), the thermal strain exerted by the matrix on the MgB<sub>2</sub> filaments has to be estimated. This is done by considering the different thermal expansion coefficients  $\alpha$  of the constituent materials:

$$\alpha \equiv \frac{1}{l} \left( \frac{dl}{dT} \right), \quad l(T) \approx l(T_0)[1 + \alpha(T - T_0)]. \quad (5.3)$$

In a simple 1-dimensional approach, if two materials  $A$  and  $B$  with different thermal expansion coefficients  $\alpha_A$  and  $\alpha_B$  are welded at a temperature  $T_0$  and subsequently cooled down to temperature  $T = T_0 + \Delta T$ , they will each be constrained in their thermal contraction by the other material and therefore each be subjected to a thermal strain  $\varepsilon_{A,B}$ :

$$\begin{aligned} \varepsilon_A &= \frac{l(T) - l_A(T)}{l_A(T)}, & l_A(T) &\approx l(T_0)[1 + \alpha_A \Delta T] \\ \varepsilon_B &= \frac{l(T) - l_B(T)}{l_B(T)}, & l_B(T) &\approx l(T_0)[1 + \alpha_B \Delta T], \end{aligned} \quad (5.4)$$

with  $l(T)$  the length of the composite at temperature  $T$  and  $l_{A,B}(T)$  the length of either material if they were allowed to contract freely. The actual length of the composite can then be calculated by demanding that the forces exerted by the two materials on one another must be balanced:

$$\begin{aligned} f_A \sigma_A(T) + f_B \sigma_B(T) &= 0 \\ f_A E_{Y,A} \varepsilon_A(T) + f_B E_{Y,B} \varepsilon_B(T) &= 0, \end{aligned} \quad (5.5)$$

with  $f_{A,B}$  the volume fractions,  $\sigma_{A,B}$  the thermal stresses and  $E_{Y,A,B}$  the Youngs' moduli of materials  $A$  and  $B$ . Substituting equation (5.4) into (5.5) and considering  $\alpha_{A,B} \Delta T \ll 1$ , the actual length change of the whole composite and the thermal strain in each component can be expressed as function of their mechanical- and thermal properties:

$$\begin{aligned} l(T) &\approx l(T_0) \left[ 1 + \frac{f_A E_{Y,A} \alpha_A + f_B E_{Y,B} \alpha_B}{f_A E_{Y,A} + f_B E_{Y,B}} \Delta T \right], \\ \varepsilon_A &\approx \frac{f_B E_{Y,B}}{f_A E_{Y,A} + f_B E_{Y,B}} (\alpha_B - \alpha_A) \Delta T, \\ \varepsilon_B &\approx \frac{f_A E_{Y,A}}{f_A E_{Y,A} + f_B E_{Y,B}} (\alpha_A - \alpha_B) \Delta T. \end{aligned} \quad (5.6)$$

As indicated in table 5.1, some conductors have more than two components. To account for this, (5.6) can be readily expanded to  $N$  components:

$$\begin{aligned} \sum_{i=1}^N f_i E_{Y,i} \varepsilon_i(T) &= 0, \\ l(T) &\approx l(T_0) \left[ 1 + \frac{\sum f_i E_{Y,i} \alpha_i}{\sum f_i E_{Y,i}} \Delta T \right], \\ \varepsilon_i(T) &\approx \frac{\sum f_i E_{Y,i} (\alpha_i - \alpha_j)}{\sum f_i E_{Y,i}} \Delta T. \end{aligned} \quad (5.7)$$

Note that equations (5.6) and (5.7) provide only rough estimates for temperature variations  $\Delta T$  of a few hundreds of Kelvin. Properties such as  $\alpha$  and  $E_Y$  are temperature dependent, so that equations (5.3), (5.4), (5.6) and (5.7) need to be integrated over the relevant temperature range. Furthermore, the thermal constraints can become so important that for some materials (e.g. Cu) they may exceed the elastic limit, so that the temperature dependent yield strength needs to be taken into account. Unfortunately, the temperature dependence of the Youngs' modulus  $E_Y$  and the yield strength  $\sigma_y$  are not available in the literature for all the materials that are analysed in this study. Therefore, (5.7) is used to estimate the thermal pre-compression. An example of a more thorough approach for  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x/\text{Ag}$  conductors can be found in [222].

The values of the thermal expansion coefficient  $\alpha$  and Youngs' modulus  $E_Y$  that are used are listed in table 5.2 and the estimates of the thermal pre-compression are listed in table 5.3. The estimation of the thermal pre-compression is separated into  $\varepsilon_{\text{pre I}}$  and  $\varepsilon_{\text{pre II}}$ . The former is the pre-compression that builds up during cool-down from the heat treatment- to room temperature, whereas the latter is the extra thermal strain building up during the cryogenic cool-down to 4.2 K. The reason for this separation is that  $\alpha$  tends to zero between  $T \approx 100 - 150$  K for most materials [80]. If its room temperature value were used throughout this low-temperature range, the thermal contraction would be strongly overestimated. Instead, for the cryogenic cool down the "integrated" thermal contraction  $\varepsilon_{293 \rightarrow 4.2 \text{ K}} = (l(4.2 \text{ K}) - l(293 \text{ K}))/l(293 \text{ K})$  is used. For the samples that are soldered on a U-shaped bending, spring  $\varepsilon_{\text{pre I}}$  is further split into the cool-down from heat treatment to the soldering temperature of 220 °C and from soldering temperature to room temperature. When the sample is soldered to a spring, the thermal contraction is nearly completely determined by the spring, since the cross-sectional area of the spring is much larger than the one of the sample:  $A_{\text{spring}} (\approx 100 \text{ mm}^2) \gg A_{\text{sample}} (\approx 1 \text{ mm}^2)$ .

The pre-compression estimates for the samples that are measured in this study and those measured by others, listed in table 5.3, are shown as a function of the measured reversible strain limit  $\varepsilon_{\text{rev}}$  in figure 5.9. Clearly  $\varepsilon_{\text{rev}}$  and  $\varepsilon_{\text{pre}}$  are related, since all data lie grouped around the line  $\varepsilon_{\text{rev}} = -\varepsilon_{\text{pre}}$ . So, to first order, the reversible strain limit of  $\text{MgB}_2/\text{metal}$  composites is determined by the level of thermal pre-compression. Just like in ceramic superconductors, the filaments

**Table 5.2:** Thermal and mechanical properties that are used in the estimation of the thermal pre-strain that the matrix exert on the filaments.  $\alpha_{273\text{K}}$  and  $E_{Y,273\text{K}}$  are the thermal expansion coefficient and the Youngs' modulus at 273 K respectively.  $\varepsilon_{293 \rightarrow 4.2\text{K}}$  is the relative change in length during a cool down from room temperature to 4.2 K.  $\Delta\alpha_{273\text{K}}$  and  $\Delta\varepsilon_{293 \rightarrow 4.2\text{K}}$  are the differences in the values of  $\alpha_{273\text{K}}$  and  $\varepsilon_{293 \rightarrow 4.2\text{K}}$  between the given material and  $\text{MgB}_2$ .

Material	$\alpha_{273\text{K}}$ [ $10^{-6}\text{K}^{-1}$ ]	$\varepsilon_{293 \rightarrow 4.2\text{K}}$ [%]	$E_{Y,273\text{K}}$ [GPa]	$\Delta\alpha_{273\text{K}}$ [ $10^{-6}\text{K}^{-1}$ ]	$\Delta\varepsilon_{293 \rightarrow 4.2\text{K}}$ [%]	Source
$\text{MgB}_2$	$8.0 \pm 0.2$	$-0.14 \pm 0.01$	$151 \pm 10$	-	-	[238, 239]
AgMg	$11.7 \pm 0.2$	$-0.40 \pm 0.05$	$100 \pm 10$	$10.9 \pm 0.4$	$-0.26 \pm 0.02$	[240]
Cu	$16.0 \pm 0.2$	$-0.32 \pm 0.01$	$121 \pm 10$	$8.0 \pm 0.4$	$-0.18 \pm 0.02$	[80]
CuNi18Zn20	$16.0 \pm 0.2$	$-0.25 \pm 0.05$	$128 \pm 10$	$8.0 \pm 0.4$	$-0.11 \pm 0.02$	[241]
CuZn30	$19.0 \pm 0.3$	$-0.39 \pm 0.01$	$125 \pm 10$	$11.0 \pm 0.5$	$-0.25 \pm 0.02$	[80]
Fe	$12.0 \pm 0.2$	$-0.24 \pm 0.01$	$180 \pm 10$	$4.0 \pm 0.4$	$-0.10 \pm 0.02$	[242]
Monel-400	$13.0 \pm 0.2$	$-0.22 \pm 0.01$	$186 \pm 10$	$5.0 \pm 0.4$	$-0.06 \pm 0.02$	[243]
Ni	$12.0 \pm 0.2$	$-0.22 \pm 0.01$	$200 \pm 10$	$4.0 \pm 0.4$	$-0.08 \pm 0.02$	[80]
Nb	$6.7 \pm 0.2$	$-0.14 \pm 0.01$	$105 \pm 10$	$-1.3 \pm 0.4$	$-0.00 \pm 0.02$	[80]
SS	$15.0 \pm 0.3$	$-0.30 \pm 0.01$	$190 \pm 10$	$7.0 \pm 0.5$	$-0.16 \pm 0.02$	[80]
Ti6AlV4	$8.9 \pm 0.2$	$-0.17 \pm 0.01$	$115 \pm 10$	$0.9 \pm 0.4$	$-0.03 \pm 0.02$	[80]

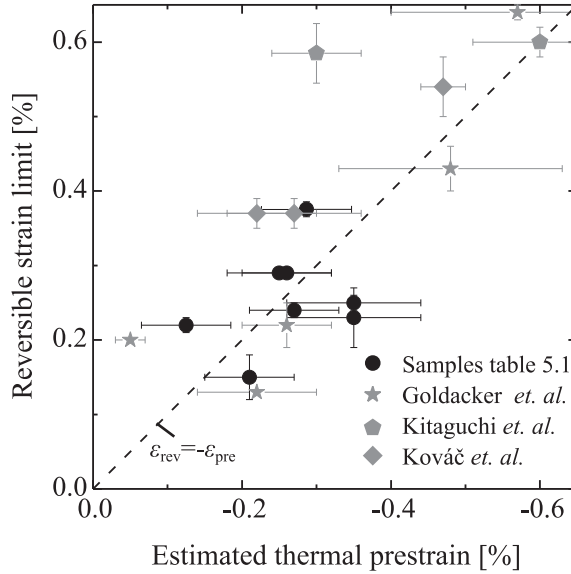
**Table 5.3:** Estimates of the thermal pre-strain in the MgB<sub>2</sub> filaments of the samples listed in table 5.1 and of samples measured by Kováč [219], Kitaguchi [151, 218] and Goldacker [108, 237].  $\epsilon_{\text{pre I}}$  is the thermal strain building up between sintering- and room temperature. It is split in two ranges; cool down from heat treatment (HT)- to soldering temperature (ST) and from soldering- to room temperature (RT). The heat treatment temperatures are typically 700 °C for in-situ and 950 °C for ex-situ conductors.  $\epsilon_{\text{pre II}}$  is the strain building up from the cool down from room temperature to 4.2 K. (CuZn), (SS) and (Ti) indicate a brass, stainless steel or titanium bending spring, respectively.

Sample	Composition	$f_i$ [%]	$\epsilon_{\text{pre I(HT} \rightarrow \text{RT)}}$ [%]		$\epsilon_{\text{pre II(293} \rightarrow \text{4.2K)}}$ [%]	$\epsilon_{\text{pre, total}}$ [%]
			(HT $\rightarrow$ ST)	(ST $\rightarrow$ RT)		
CLM	MgB <sub>2</sub> /Fe/Cu/Ni(Ti)	9/10/14/66	-0.30 $\pm$ 0.06	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.35 $\pm$ 0.09
FZK1	MgB <sub>2</sub> /Fe(Ti)	50/50	-0.16 $\pm$ 0.03	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.21 $\pm$ 0.06
FZK2	MgB <sub>2</sub> /Nb/Cu/CNZ(Ti) <sup>a</sup>	13/7/56/24	-0.30 $\pm$ 0.06	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.35 $\pm$ 0.09
HTR	MgB <sub>2</sub> /Nb/Cu/Monel(Ti)	16/15/29/40	-0.20 $\pm$ 0.04	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.25 $\pm$ 0.07
IEE1	MgB <sub>2</sub> /Fe(Ti)	25/75	-0.23 $\pm$ 0.05	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.28 $\pm$ 0.06
IEE2	MgB <sub>2</sub> /Nb/AgMg(Ti)	26/30/44	-0.08 $\pm$ 0.02	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.13 $\pm$ 0.06
UG1	MgB <sub>2</sub> /Fe(Ti)	30/70	-0.21 $\pm$ 0.05	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.26 $\pm$ 0.06
UG2	MgB <sub>2</sub> /Fe(Ti)	29/71	-0.22 $\pm$ 0.05	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.27 $\pm$ 0.06
Kováč.	MgB <sub>2</sub> /Fe(CuZn)	47/53	-	-0.22 $\pm$ 0.01	-0.25 $\pm$ 0.02	-0.47 $\pm$ 0.09
	MgB <sub>2</sub> /Fe(Ti)	47/53	-0.17 $\pm$ 0.05	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.22 $\pm$ 0.08
	MgB <sub>2</sub> /Fe(Ti)	25/75	-0.22 $\pm$ 0.06	-0.02 $\pm$ 0.01	-0.03 $\pm$ 0.02	-0.27 $\pm$ 0.09
Kitag.	MgB <sub>2</sub> /SS(SS)	0.4/0.6	-	-0.14 $\pm$ 0.01	-0.16 $\pm$ 0.02	-0.30 $\pm$ 0.06
	MgB <sub>2</sub> /Fe/Cu/Ni(SS) <sup>b</sup>	9/10/14/66	-0.30 $\pm$ 0.06	-0.14 $\pm$ 0.01	-0.16 $\pm$ 0.02	-0.60 $\pm$ 0.09
Gold.	MgB <sub>2</sub> /Fe	49/51	-0.20 $\pm$ 0.04	-	-0.06 $\pm$ 0.02	-0.26 $\pm$ 0.06
	MgB <sub>2</sub> /Fe/SS	30/33/37	-0.39 $\pm$ 0.12	-	-0.09 $\pm$ 0.03	-0.48 $\pm$ 0.15
	MgB <sub>2</sub> /Fe/SS	22/24/54	-0.46 $\pm$ 0.13	-	-0.11 $\pm$ 0.04	-0.57 $\pm$ 0.17
	MgB <sub>2</sub> /Fe	58/42	-	-	-0.05 $\pm$ 0.02	-0.05 $\pm$ 0.02
	MgB <sub>2</sub> /Fe	58/42	-0.17 $\pm$ 0.06	-	-0.05 $\pm$ 0.02	-0.22 $\pm$ 0.08

<sup>a</sup> CNZ stands for CuNi18Zn20

<sup>b</sup> Note that this sample is in fact sample CLM measured on a stainless steel U-shaped bending spring.

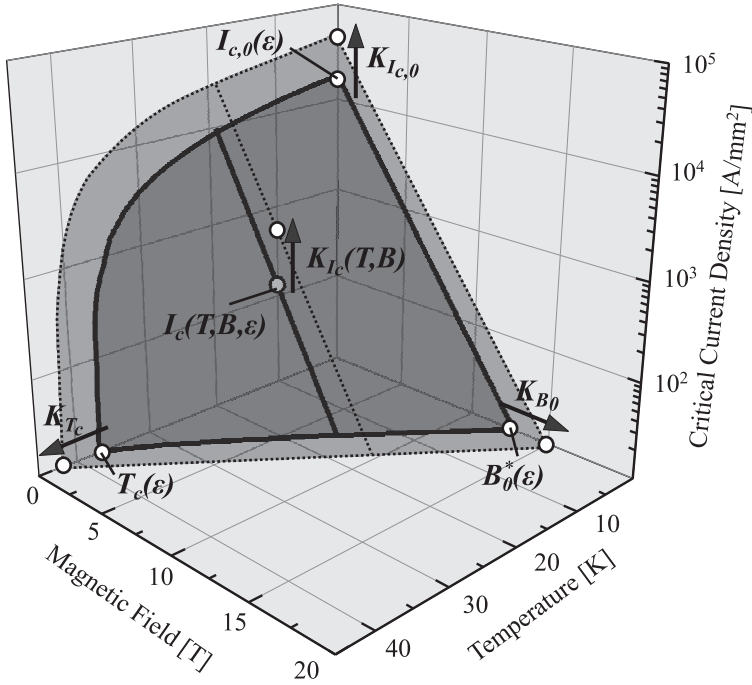




**Figure 5.9:** Measured reversible strain limit  $\varepsilon_{\text{rev}}$  of  $\text{MgB}_2$ /metal composites, versus the estimated thermal pre-compression strain  $\varepsilon_{\text{pre}}$ .

will not be damaged by an applied tensile stress as long as this stress does not fully compensate the thermally induced compressive stress (as long as the internal strain state of the filaments remains compressive).

However, note that the thermal pre-compression alone cannot fully explain the sample dependence of the reversible strain limit. Kováč *et al.* [59] measured the strain response of the critical current of samples with the same matrix materials and fill factors, but different in layout (aspect ratio and number of filaments). If thermal pre-compression alone would determine the reversible strain limit,  $\varepsilon_{\text{rev}}$  should be similar for these samples. Instead, they found significantly different reversible strain limits ( $\varepsilon_{\text{rev}} \approx 0.20 - 0.35$  %) depending on the shape of the samples. Experimental artefacts such as a strain gradient across the sample could be excluded. The value of such a strain gradient would be  $\approx 7$  % of the mean strain value, which cannot explain the observed difference. Similarly, also samples UG1 and UG2, with  $\varepsilon_{\text{rev}} \approx 0.29$  and  $0.23$  % respectively, differ significantly. They both have a Fe matrix with approximately the same filling factor but UG1 is an ex-situ tape, whereas UG2 is an in-situ wire. A similar discrepancy between in- and ex-situ conductors was also observed by Kováč [244]. A possible explanation for these deviations is a difference in filament density, which results in differences in the effective Youngs' modulus. For example, as discussed in chapter 2,  $\text{MgB}_2$ /Fe ex-situ samples that are rolled into tapes have a significantly higher filament density than similar samples that are drawn into wires. Also softer materials in the precursor powder (Pb additions such as discussed in §2.3.2 or Mg in



**Figure 5.10:** Schematic representation of the strain response of the critical surface of a MgB<sub>2</sub> conductor.  $I_c(B, T, \varepsilon)$  is the temperature-, field- and strain-dependent critical current with  $K_{I_c}(B, T)$  its strain response as described by equation (5.9).  $I_{c,0}(\varepsilon)$ ,  $T_c(\varepsilon)$  and  $B_0^*(\varepsilon)$  are the strain dependent “corner points” of the critical surface and  $K_{I_{c,0}}$ ,  $K_{T_c}$  and  $K_{B_0}$  are their corresponding strain responses. Note that the strain response of the critical surface is exaggerated for clarity.

the in-situ route) lead to higher effective densities. To verify if indeed the filament density influences the level of pre-compression, a more systematic study on the variation of the Youngs’ modulus as a function of filament density is needed.

### 5.3.2 Reversible Regime

The linear strain response of the critical current in the reversible regime can be described as:

$$I_c(B, T, \varepsilon) = I_c(B, T, 0)[1 + K_{I_c}(B, T)\varepsilon], \quad (5.8)$$

with  $K_{I_c}(B, T)$  the normalised slope of the reversible strain response:

$$K_{I_c}(B, T) \equiv \frac{1}{I_c(B, T, 0)} \left( \frac{\partial I_c(B, T, \varepsilon)}{\partial \varepsilon} \right)_{B, T}. \quad (5.9)$$

In §5.1 the electronic origin of the reversible strain dependence in Nb<sub>3</sub>Sn was briefly discussed. If the strain response of  $I_c$  also has an electronic origin in MgB<sub>2</sub>, then the critical temperature  $T_c$  and the irreversibility field  $B_0^*$  might be expected to be similarly strain dependent. Indeed, Lorenz [245] and Tang [246] reported a  $T_c$  suppression in MgB<sub>2</sub> bulk samples when applied to hydrostatic pressure. Kitaguchi [247] also observed similar effects in axially strained MgB<sub>2</sub> tapes. At the University of Twente it was found that the irreversibility field  $B_0^*$  is also strain dependent [142]. The aim of the remainder of this chapter is to show that the reversible strain response of the critical current  $K_{I_c}$  at any field and temperature can be expressed in terms of just three scaling parameters: 1) the zero field-, zero temperature-limit of the strain response of the critical current  $K_{I_{c,0}}$ ; 2) the strain response of the critical temperature  $K_{T_c}$ ; and 3) the strain response of the irreversibility field at zero temperature  $K_{B_0}$ . In fact, these three parameters describe the reversible strain behaviour of the three “corner points”  $I_{c,0}$ ,  $T_c$  and  $B_0^*$  of the critical surface, as displayed in figure 5.10. Mathematically, they are defined as:

$$\begin{aligned} I_{c,0}(\varepsilon) &\equiv I_c(B = 0, T = 0, \varepsilon); \\ T_c(\varepsilon) &\equiv T(I_c = 0, B = 0, \varepsilon); \\ B_0^*(\varepsilon) &\equiv B(I_c = 0, T = 0, \varepsilon), \end{aligned} \quad (5.10)$$

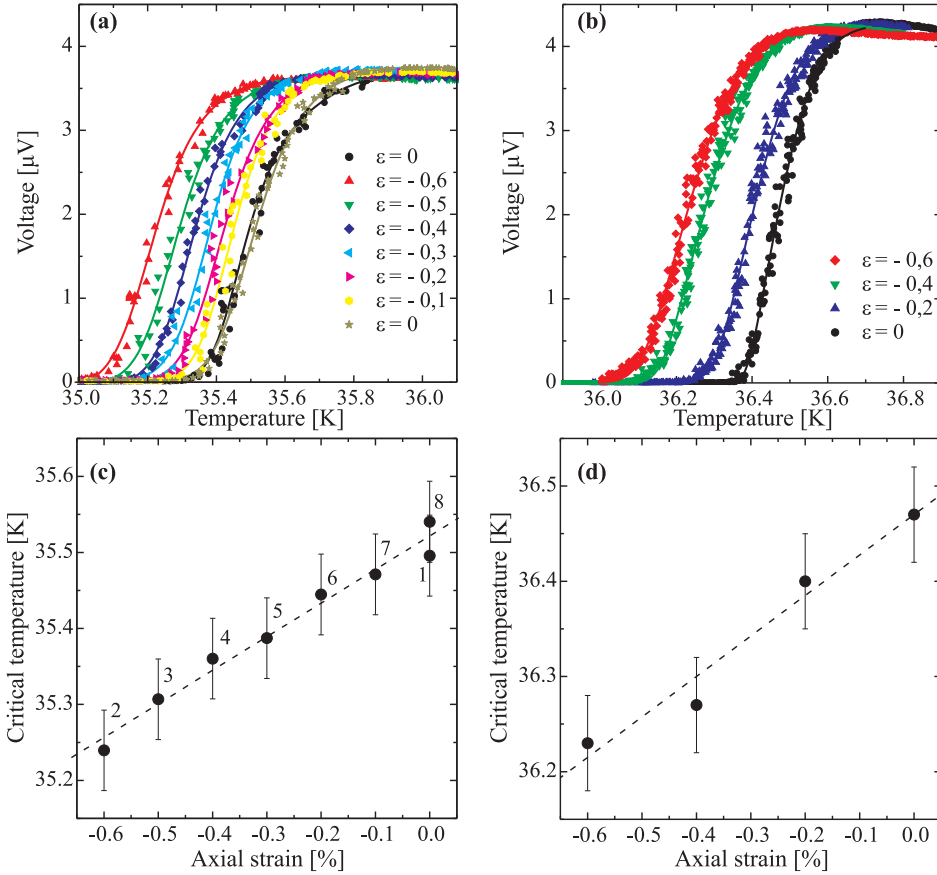
where  $T_c(B, I_c, \varepsilon)$ ,  $B(I_c, T, \varepsilon)$  and  $I_c(B, T, \varepsilon)$  are three equivalent descriptions of the strain-dependent critical surface. Analogous to the strain response of the critical current at any  $T$  and  $B$  (expression (5.9)), the strain responses of the three “corner points” of the critical surface can be described with the normalised strain derivative:

$$\begin{aligned} K_{I_{c,0}} &\equiv \frac{1}{I_{c,0}(0)} \frac{dI_{c,0}(\varepsilon)}{d\varepsilon} = \frac{1}{I_c(0, 0, 0)} \left( \frac{\partial I_c(B, I_c, \varepsilon)}{\partial \varepsilon} \right)_{T=0; B=0}; \\ K_{T_c} &\equiv \frac{1}{T_c(0)} \frac{dT_c(\varepsilon)}{d\varepsilon} = \frac{1}{T(0, 0, 0)} \left( \frac{\partial T(B, I_c, \varepsilon)}{\partial \varepsilon} \right)_{B=0; I_c=0}; \\ K_{B_0} &\equiv \frac{1}{B_0^*(0)} \frac{dB_0^*(\varepsilon)}{d\varepsilon} = \frac{1}{B(0, 0, 0)} \left( \frac{\partial B(I_c, T, \varepsilon)}{\partial \varepsilon} \right)_{I_c=0; T=0}. \end{aligned} \quad (5.11)$$

In §5.6 it will be shown how to construct a relation of the form

$$K_{I_c}(B, T) = g \left( \frac{B}{B_0^*}, \frac{T}{T_c(0)}; K_{I_{c,0}}; K_{T_c}; K_{B_0} \right), \quad (5.12)$$

that describes the strain dependence of the entire critical surface in terms of these three partial derivatives, reflecting the behaviour of its corner points. However, first the reversible strain dependence of the critical temperature and the irreversibility field, i.e. the second and third partial derivatives in (5.11), will be examined in detail.



**Figure 5.11:** Reversible variation of  $T_c$  with axial strain. (a) and (c) are measured on sample IEE1 and (b) and (d) are measured on sample FZK1.

### 5.4 Strain Dependence of the Critical Temperature

To verify the validity the scaling relation (5.12), samples IEE1 and FZK1 were fully characterised in terms of the strain dependence of the critical current, -temperature and the irreversibility field. The latter will be treated in §5.5. In this paragraph the behaviour of  $T_c$  is described.

The resistive transitions as a function of axial strain for both samples are displayed in figures 5.11a and b. Clearly, the transitions shift when strain is applied to those samples. Plotting the critical temperature against axial strain (figures 5.11c and d) reveals a linear  $T_c(\epsilon)$  dependence. Moreover, the numbers next to the data-points in figure 5.11c indicate the order in which strain is applied. It is clear

that the  $T_c$  variation with strain is also reversible. The linear strain response of the critical temperature allows to determine  $K_{T_c}$  through linear regression. Since the transitions shift as a whole, the strain response does not depend on the exact criterion used to determine  $T_c$ . In figures 5.11c and d, a criterion of 50 % of the normal resistivity is used, while the uncertainty of  $T_c$  is determined with the 99 % prediction band as discussed in §5.2.3.

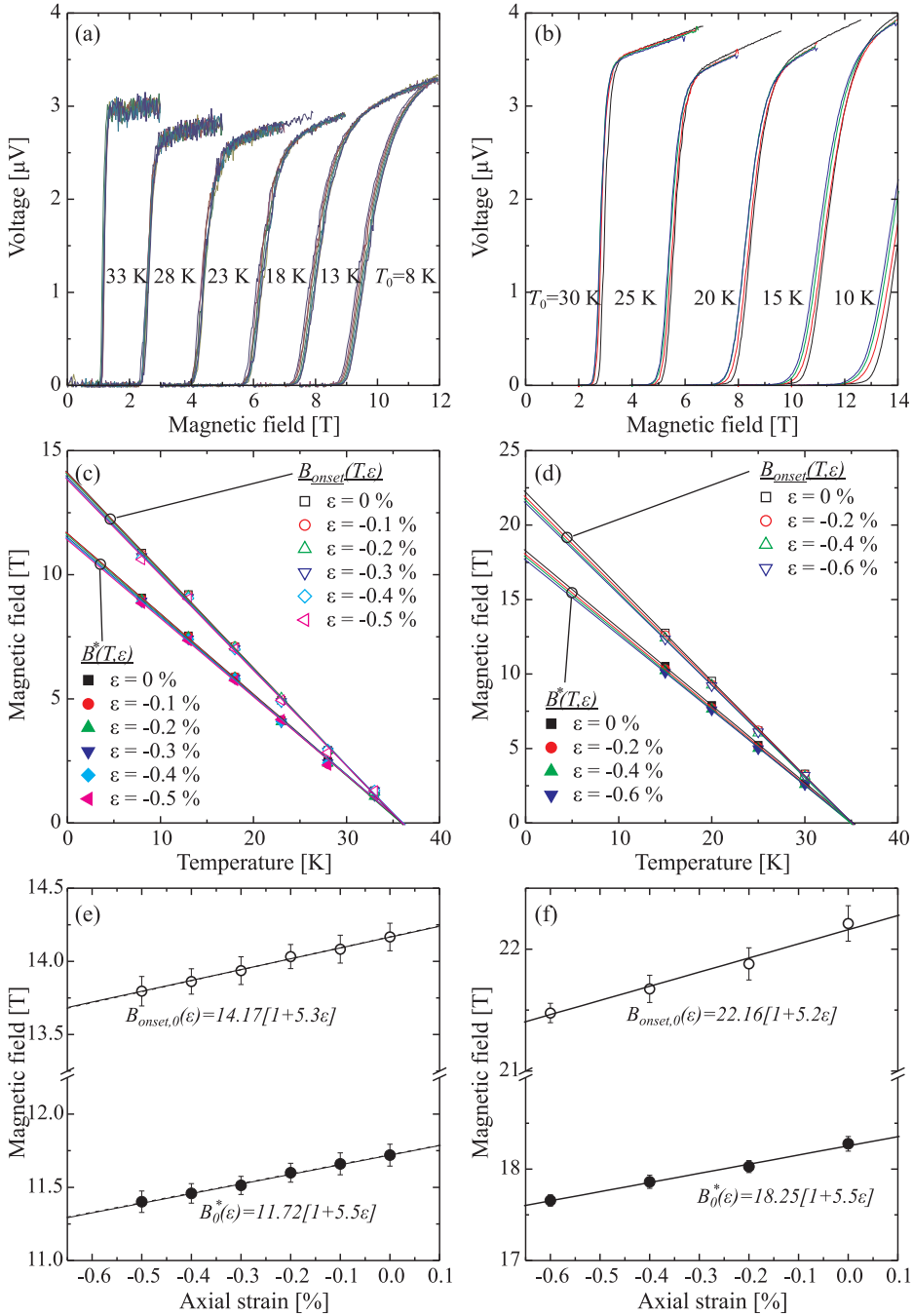
For sample IEE1, an ex-situ multi-filamentary square wire,  $K_{T_c}$  is 1.3, whereas for sample FZK1, an in-situ mono-filamentary wire,  $K_{T_c}=1.2$ . The  $K_{T_c}$  values found here are comparable to those found by Kitaguchi [247] who found a value of  $K_{T_c}=1.4$  for an ex-situ  $\text{MgB}_2/\text{Fe}$  tape. These observed values of  $K_{T_c}$  correspond to a relatively small effect, since a 1 % change in strain induces a change in  $T_c$  of only  $\approx 1.3$  % ( $\Delta T_c \approx 0.5$  K). Note that the similarity between the  $K_{T_c}$  values measured for samples FZK1, IEE1 and for the sample reported by Kitaguchi is quite remarkable, since the conductors differ substantially. For example, in-situ and ex-situ conductors differ significantly in homogeneity and purity, which can influence the electron-phonon coupling, so that the strain response of  $T_c$  of these conductors can be expected to differ. Furthermore, as discussed in §5.2.3, the superconducting properties of a polycrystalline  $\text{MgB}_2$  conductor are for a large part determined by distributed properties of the individual crystallites. Since  $\text{MgB}_2$  grains in tapes tend to exhibit more texturing [156], this would cause the “overall” strain response of  $T_c$  to behave differently in these conductors. Lastly, the fact that these conductors vary in layout (number of filaments, dimensions and shape) causes the transversal Poisson contraction to deform the conductors differently. Again, this can affect the strain response of  $T_c$ .

## 5.5 Strain Dependence of the Irreversibility Field

Analogous to the determination of  $K_{T_c}$ ,  $K_{B_0}$  is found by measuring the magneto-resistive transitions at different levels of applied strain. The data are shown in figure 5.12a and b, for samples IEE1 and FZK1. Each family of  $V(B)$  curves corresponds to a constant temperature and represents a set of selected compressive strain values. Next, the irreversibility field is determined using the criterion 5 % of the normal state resistivity. In this way the temperature dependence of the irreversibility field at selected strain levels is obtained. The curves are depicted in figures 5.12c and d for samples IEE1 and FZK1 respectively. The irreversibility field at zero temperature  $B_0^*$  is found through linear extrapolation of the  $B^*(T)$  curves, obtaining the strain dependence of  $B_0^*$  shown in figures 5.12e and f (samples IEE1 and FZK1 respectively).

Just like  $T_c$ ,  $B_0^*$  turns out to be linearly dependent on strain, so that  $K_{B_0}$  can readily be obtained through linear regression. Furthermore,  $K_{B_0}$  is also surprisingly similar for both conductors, being  $K_{B_0} = 5.5$  for both samples. The strain response of  $B_0^*$  is stronger than that of  $T_c$ , since a 1 % change in strain induces a change of  $\approx 5$  % in  $B_0^*$  (for sample FZK1, this corresponds to 1 T).

Also plotted in figures 5.12c and d is the magnetic field at the onset of su-



**Figure 5.12:** Reversible variation of the irreversibility field with axial strain. (a),(c) and (e) correspond to sample IEE1; (b)(d)(f) to sample FZK1. (a) and (b) are the magneto-resistive transition at selected temperatures and strains. (c) and (d) are the corresponding temperature dependent irreversibility- and onset fields. (e) and (f) are the strain dependence of the zero-temperature extrapolation of the irreversibility field and onset field

perconductivity  $B_{\text{onset}}$  as a function of temperature at different strain states. Extrapolating these data to zero temperature results in the strain dependence of  $B_{\text{onset},0}$ , plotted in figures 5.5e and f. Clearly, strain shifts the magneto-resistive transition as a whole, so that the strain response of  $B_{\text{onset},0}$  is similar to that of  $B_0^*$  for both samples, just like the resistive transition with temperature shown in figure 5.11. A-priori, this is rather unexpected.

As discussed in §5.2.3, the measured  $B_{\text{onset},0}$  approximates the second critical field  $B_{c2,0}$ , while  $B_{\text{offset},0}$  should correspond to irreversibility field  $B_0^*$ . If this is indeed the case then  $B_{\text{onset},0}$  should also be proportional to the thermodynamic critical field  $B_c(0)$ . As suggested by several experiments [2, 248],  $\text{MgB}_2$  is a BCS-like material with  $B_c(0)$  proportional to the gap energy  $\Delta(0)$ . Summarising this mathematically, one would expect

$$B_{\text{onset},0} \approx B_{c2,0} = \sqrt{2\kappa}B_c(0) = \sqrt{8\pi N(0)\kappa}\Delta(0), \quad (5.13)$$

in which  $\kappa$  is the Ginzburg-Landau parameter, defined as the ratio of the London penetration depth to the coherence length; and  $N(0)$  is the density of states. The irreversibility field  $B_0^*$ , on the other hand, can be seen as a crude measure of the effective pinning potential. As such, in a first approximation it is expected to be proportional to the condensation energy [67]. The latter is proportional to the square of  $\Delta(0)$ , leading to the expectation

$$B_0^* \propto \Delta^2(0). \quad (5.14)$$

Combining (5.13) and (5.14) shows that  $B_0^*$  is proportional to the square of  $B_{\text{onset},0}$ , i.e.

$$B_0^* \propto B_{\text{onset},0}^2. \quad (5.15)$$

Consequently, when strain is applied to a sample, a-priori the variation of  $B_0^*$  is expected to be twice that of  $B_{\text{onset},0}$ :

$$\frac{1}{B_0^*} \left( \frac{dB_0^*(\varepsilon)}{d\varepsilon} \right) = \frac{2}{B_{\text{onset},0}} \left( \frac{dB_{\text{onset},0}(\varepsilon)}{d\varepsilon} \right). \quad (5.16)$$

As suggested before, this clearly is not the case in the conductors under study, since the measured slopes of  $B_0^*(\varepsilon)$  and  $B_{\text{onset},0}(\varepsilon)$  are virtually equal.

In order to understand the discrepancy between the first order approximation described above and the actual measurements, it would be insightful to study the strain or pressure dependence of  $B_0^*$  and  $B_{\text{onset},0}$  in  $\text{MgB}_2$  single crystals. In this way any ‘‘averaging’’ of the anisotropic superconducting properties of the individual crystallites over the whole polycrystalline filaments [236] can be ruled out. These conditions will allow, for example, to verify the assumption that  $B_{c2}$  and  $B_c$  indeed respond similar to strain, which is implicitly assumed in expression (5.13) and is not necessarily the case, for instance if the Ginzburg-Landau parameter  $\kappa$  itself is strain dependent.

## 5.6 Scaling the Strain Dependence of the Critical Current

With both the strain dependence of the critical temperature  $T_c$  and the zero-temperature extrapolation of the irreversibility field  $B_0^*$  determined, it is now possible to see whether the strain response of the critical current can indeed be scaled, i.e. whether a relation of the form (5.12) can be found. Such scaling implies that the critical current at any field and temperature can be described according to

$$\frac{I_c(B, T, \varepsilon)}{I_{c,0}(\varepsilon)} = f\left(\frac{B}{B_0^*(\varepsilon)}, \frac{T}{T_c(\varepsilon)}\right), \quad (5.17)$$

with  $f$  a function that does not depend explicitly on  $\varepsilon$ . Combining with (5.9) yields

$$K_{I_c}(B, T) = K_{I_{c,0}} - \frac{1}{f} \left( \frac{\partial f}{\partial t} \right)_b t K_{T_c} - \frac{1}{f} \left( \frac{\partial f}{\partial b} \right)_t b K_{B_0}$$

with  $t \equiv \frac{T}{T_c}; b \equiv \frac{B}{B_0^*}$ . (5.18)

This expression has the form of (5.12) and describes the strain response of the entire critical surface in terms of the variations of its three ‘‘corner points’’ only. In order to verify if (5.18) indeed describes the observed strain behaviour of the critical current at any field and temperature, three possible cases are considered:

1. The field-temperature scaling relation  $f$  of the critical current (5.17) is known analytically;
2. The field-temperature scaling relation  $f$  of the critical current (5.17) is only known numerically;
3. The field-temperature scaling relation  $f$  of the critical current (5.17) is known analytically *and* the three scaling parameters  $K_{I_{c,0}}$ ,  $K_{T_c}$  and  $K_{B_0}$  are known.

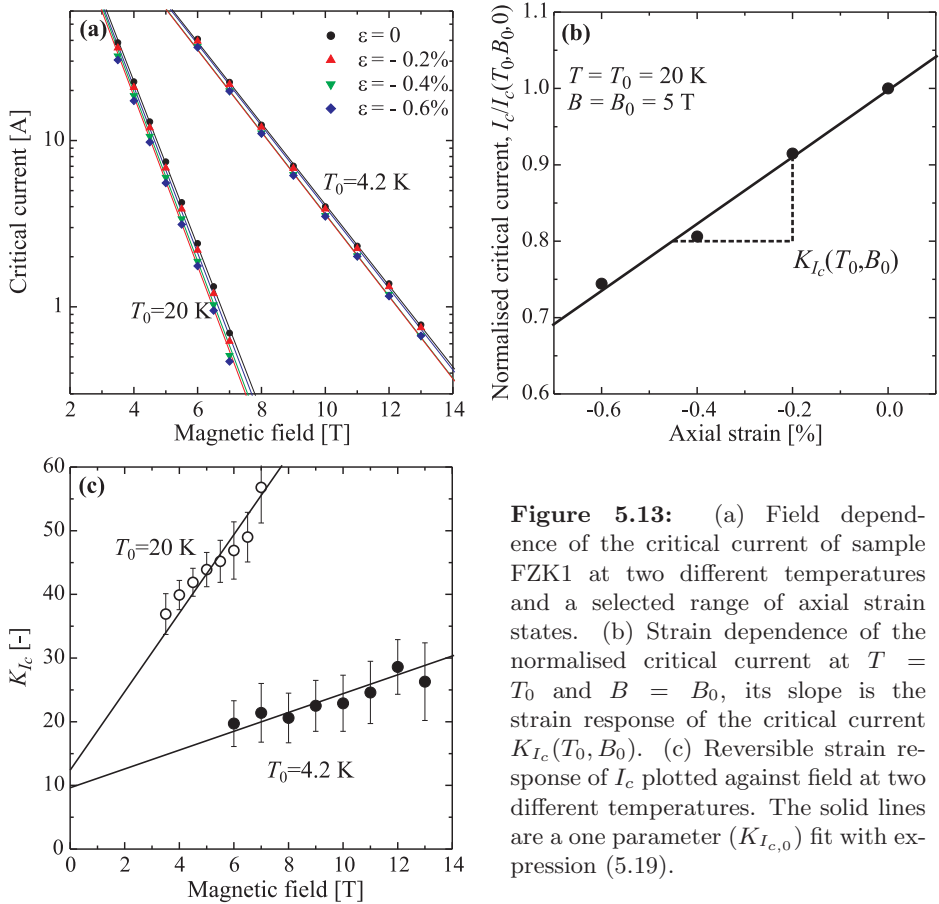
### 5.6.1 Scaling Relation is known Analytically

In §3.2.1, an analytical expression (equation (3.1)) was introduced that approximates the experimentally observed critical surface of bulk  $\text{MgB}_2$ , as well as that of most  $\text{MgB}_2$  composite conductors:

$$\frac{I_c(B, T)}{I_{c,0}} = \left(1 - \frac{T}{T_c}\right) \exp\left[-\frac{B}{B_p(T)}\right], \quad (3.1)$$

with  $B_p(T) = B_{p,0} \left(1 - \frac{T}{T_c}\right)$ .





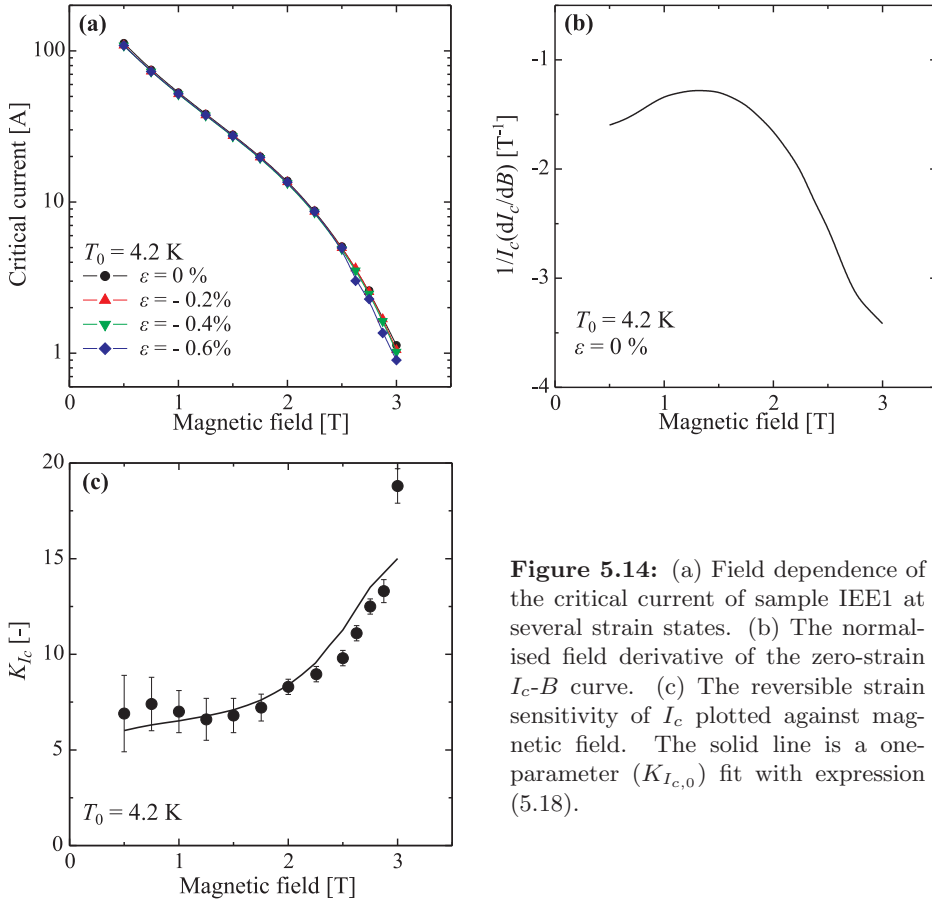
**Figure 5.13:** (a) Field dependence of the critical current of sample FZK1 at two different temperatures and a selected range of axial strain states. (b) Strain dependence of the normalised critical current at  $T = T_0$  and  $B = B_0$ , its slope is the strain response of the critical current  $K_{I_c}(T_0, B_0)$ . (c) Reversible strain response of  $I_c$  plotted against field at two different temperatures. The solid lines are a one parameter ( $K_{I_c,0}$ ) fit with expression (5.19).

Indeed, (3.1) has the form of (5.17). Moreover, as shown in figure 5.13a, the scaling relation (3.1) satisfactorily describes the critical current data of the unstrained sample FZK1 using  $I_{c,0} \approx 5100$  A,  $T_c = 35.3$  K and  $B_{p,0} = 2.02$  T.

So with expression (3.1) as a satisfactory scaling relation, equation (5.18) can be written as:

$$K_{I_c}(B, T) = K_{I_{c,0}} + \frac{T}{T_c - T} \left( 1 + \frac{B}{B_p(T)} \right) K_{T_c} + \left( \frac{B}{B_p(T)} \right) K_{B_0}. \quad (5.19)$$

Also plotted in figure 5.13a are the  $I_c(B, T)$  data measured at four different levels of compressive strain. Linear regression of  $I_c(\varepsilon)$  at constant temperature and field yields the strain response of the critical current  $K_{I_c}$ , which as an example is plotted in figure 5.13b for  $T = 20$  K and  $B = 5$  T. As already noted in §5.3, it is clear that the  $K_{I_c}$  depends on both magnetic field and temperature. Knowing  $K_{T_c} (=1.2)$  and  $K_{B_0} (=5.5)$  from §5.4 and §5.5 respectively, expression (5.19) can be fit to the measured data with only one remaining free parameter  $K_{I_{c,0}}$ . The



**Figure 5.14:** (a) Field dependence of the critical current of sample IEE1 at several strain states. (b) The normalised field derivative of the zero-strain  $I_c$ - $B$  curve. (c) The reversible strain sensitivity of  $I_c$  plotted against magnetic field. The solid line is a one-parameter ( $K_{I_{c,0}}$ ) fit with expression (5.18).

solid lines in figure 5.13c are the result of a least squares fit of expression (5.19) to the measured  $K_{I_c}$  data, yielding a value of  $K_{I_{c,0}}$  of 9.3. It is clear that expression (5.19) is indeed able to describe the field and temperature dependence of the strain response of the critical current.

### 5.6.2 Scaling Relation is known Numerically

In contrast to sample FZK1, figure 5.14a shows that sample IEE1 does not exhibit an exponential dependence of the critical current with magnetic field. In fact, it shows a poor performance in magnetic field with a strong downturn of  $I_c(B)$  at 4.2 K for fields above 2 T (see also figure 5.14b), probably caused by handling damage during sample mounting. Consequently, it is difficult to describe the field-temperature dependence of the critical current analytically. This data are presented to show that also for this case, expression (5.18) can still be used to describe the strain response of the critical current, using numerical data.

The second term on the right hand side in (5.18) (the temperature dependence) can be estimated to be of the order 0.1 at 4.2 K, while the strain dependence of the critical current  $K_{I_c}$  is measured to be of the order 10 (figure 5.14c), so that this second term can be neglected at  $T = 4.2$  K. The normalised field derivative of the critical current is calculated numerically from the measured  $I_c(B)$  dependence and is plotted in figure 5.14b.

Knowing that  $K_{B_0} = 5.5$  and  $B_0^* = 11.7$  T from §5.5, expression (5.18) can again be fit to the  $K_{I_c}(B)$  data with  $K_{I_{c,0}}$  as a single free parameter. Using  $K_{I_{c,0}} = 5.3$  results in the solid line in figure 5.14c. Clearly, also in this case expression (5.18) describes the measured  $K_{I_c}(B)$  data well. It can be seen that  $K_{I_c}(B)$  does not show a linear behaviour like sample FZK1, which is a direct reflection of the downturn in the  $I_c(B)$  curve.

### 5.6.3 Scaling Relation is known Analytically and the Scaling Parameters are known

In §5.6.1 and §5.6.2 it was shown how (5.18) indeed applies to the measured strain response of  $I_c(B, T)$  of samples FZK1 and IEE1, respectively. Of course, the practical use of a scaling relation such as (5.18) lies in its predictive value. This is demonstrated in this paragraph.

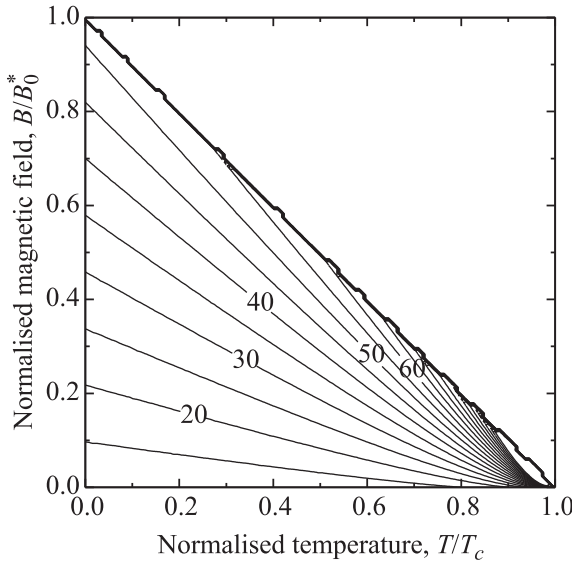
When a (hypothetical) conductor is considered of which function (5.17) is known, as well as the three scaling parameters  $K_{I_{c,0}}$ ,  $K_{T_c}$  and  $K_{B_0}$ , then expression (5.18) can readily be used to *predict* the strain response of the critical surface. Using expression (3.1) as the analytical description of the critical surface, so that expression (5.18) can be written in the form of (5.19), and taking  $K_{I_{c,0}} = 10$ ,  $K_{T_c} = 5$  and  $K_{B_0} = 1$  for the three scaling parameters (similar to sample FZK1), results in a strain response of the critical surface  $K_{I_c}$  as depicted in figure 5.15.

Interestingly, the strain response of the critical current can show relatively high values ( $K_{I_c} \sim 50$  or even higher) compared to the values of the strain responses of  $T_c$ ,  $B_0^*$  and  $I_{c,0}$ , especially close to the line  $B^*(T)$ . This is a direct result from the strong field dependence of  $I_c$ , together with the fact that the current decay field  $B_p$  is strongly dependent on the temperature (i.e.  $B_p \rightarrow 0$  when  $T \rightarrow T_c$ ), which causes the last term in (5.18) to dominate.

## 5.7 Conclusion

The strain response of the critical current is found to exhibit two distinct regimes: 1) in the compressive strain regime or for moderate tensile strains, the critical current shows a linear and reversible dependence on strain; and 2) when a certain sample-dependent reversible strain limit  $\varepsilon_{\text{rev}}$  is exceeded, the critical current shows a steep and irreversible degradation.

By estimating the thermal pre-compression  $\varepsilon_{\text{pre}}$  that the matrix and sample holder exert on the  $\text{MgB}_2$  filaments, it was found that the reversible strain limit is, to first order, determined by this thermal pre-compression strain.



**Figure 5.15:** Calculated strain response  $K_{I_c}$  of a hypothetical conductor as a function of the normalised field and -temperature, of which the field-temperature scaling of  $I_c$  is known as well as the three scaling parameters  $K_{I_{c,0}}$ ,  $K_{T_c}$  and  $K_{B_0}$ . The parameters are chosen such that this hypothetical conductor is similar to sample FZK1. The magnetic field is normalised to the zero-temperature extrapolation of the irreversibility field  $B_0^*$ , while the temperature is normalised to the critical temperature  $T_c$ .

Measurements of the resistive and magneto-resistive transitions of strained samples shows that both the critical temperature  $T_c$  and the irreversibility field  $B^*$  are strain dependent. Just like the critical current, they depend linearly and reversibly on axial strain.

The studied conductors show a remarkably similar strain dependence in both the  $T_c$  and  $B^*$ , despite the fact that these conductors are inherently different through their precursor powder (in-situ versus ex-situ), matrix material and layout (shape, dimensions and number of filaments). All these aspects can influence texture, homogeneity and purity of the filaments, but apparently they do not affect the effective strain dependence of  $T_c$  and  $B^*$ .

It is also found that the strain dependence of the magneto-resistive transitions does not follow the expected behaviour based on simple BCS-like arguments, which would predict the strain sensitivity of  $B_0^*$  to be twice that of  $B_{c2,0}$ . The reason for this is at present unclear and further studies of strained single crystals or single crystals under hydrostatic pressure are suggested to clarify this issue.

Combining the strain dependence of  $I_c$ ,  $T_c$  and  $B^*$ , it was found that the slope  $K_{I_c}(B, T)$  of the linear and reversible  $I_c(\varepsilon)$  regime at any temperature and mag-

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netic field can be described with the strain sensitivities of the zero-temperature, zero-field extrapolation of the critical current ( $K_{I_{c,0}}$ ), the critical temperature ( $K_{T_c}$ ) and the zero-temperature extrapolation of the irreversibility field ( $K_{B_0}$ ).



## Chapter 6

# Conclusion

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*In the previous chapters several aspects related to the application of technical MgB<sub>2</sub> conductors in magnets were studied. These include whether the  $J_c(B,T)$  performance is suitable for magnet operation; prospects for further development of the material; and how MgB<sub>2</sub> conductors respond to thermal instabilities and electro-mechanical forces. The main findings and conclusions are brought together in this chapter and are used to address the general suitability of MgB<sub>2</sub> superconductors for magnet applications.*

This thesis work was motivated by the discovery of superconductivity in  $\text{MgB}_2$ , a material with several attractive features that, taken together, make it a useful and practical superconductor. At an estimated materials cost of about 0.80 €/m for filaments and matrix together  $\text{MgB}_2$  constitutes a relative low-cost superconductor, provided that the manufacturing cost can be kept low. Furthermore, with a critical temperature of 40 K it can be operated at a higher temperature than Nb-Ti and  $\text{Nb}_3\text{Sn}$ . Compared to high- $T_c$  conductors  $\text{MgB}_2$  is a relatively simple compound. Lastly,  $\text{MgB}_2$  conductors can be made with the powder-in-tube technology already used for  $\text{Nb}_3\text{Sn}$  and high- $T_c$  conductors.

In order to gauge the applicability of  $\text{MgB}_2$  conductors in magnets, three areas of attention were defined: 1) cost-efficient fabrication of  $\text{MgB}_2$  conductors with suitable  $J_c(B,T)$  performance; 2) the possibility to protect  $\text{MgB}_2$ -based magnets from thermal instabilities; and 3) the influence of mechanical deformations in magnets on the superconducting properties of  $\text{MgB}_2$  conductors. These issues were formulated as questions in the introductory chapter 1 and will be answered here.

*1) Is the  $J_c(B,T)$  performance of  $\text{MgB}_2$  conductors suitable for magnet applications?*

Experience with previously developed practical superconductors learns that the  $J_c(B,T)$  performance of a conductor depends on the degree of electrical connectivity that can be obtained between the crystallites in the filament and on the field retention of  $J_c$ , which is determined by flux-line pinning and the value of  $B_{c2}$ . In  $\text{MgB}_2$  these factors depend strongly on the deformation technique, mechanical strength of the matrix material and the precursor powder (in- and ex-situ, pure  $\text{MgB}_2$  or doped). The literature study in chapter 3 on the  $J_c(B)$  performance of reported conductors in the years 2003-2006 reveals that the top 50 % of the conductors have self-field extrapolated critical current density values falling in a relatively narrow range of  $J_{c,0}^T = 10^4 - 10^5$  A/mm<sup>2</sup> at 4.2 K. The self-field extrapolation of  $J_c$  appears to be a purely connectivity determined parameter as it is not correlated with its field retention, i.e. pinning or changes in the second critical field  $B_{c2}$ . Thus, the upper limit of  $J_c$  seems to be  $10^4 - 10^5$  A/mm<sup>2</sup> at 4.2 K and is imposed by an upper limit in the connectivity.

Less clear to quantify at present is an upper limit of the in-field performance, i.e. the field retention of  $J_c$ . First, the field retention of  $J_c$  has continued to improve between 2003 and 2006. The median value of technical upper field limit  $B_{100}$  above which  $J_c$  drops below 100 A/mm<sup>2</sup> has increased by 25 % over the four years to a present value of 7 T. Related to this is a general shift of focus towards in-situ conductors in which low level doping is better controlled and the field retention has increased accordingly. A second reason that a possible upper limit in the field retention is still difficult to gauge is the fact that the mechanism of carbon-based doping is at present unclear. Comparison of non-carbon and carbon based dopants shows that the latter tends to increase the field retention by  $\sim 50$  % compared to undoped conductors, while non-carbon based dopants tends to increase the field retention by only  $\sim 15$  %. This could suggest that



carbon based dopants improve both pinning and  $B_{c2}$ , while the non-carbon based dopants only increase pinning. Further study of the effect of carbon-based dopants will reveal the mechanism of the enhancement and might well open possibilities to improve the field retention even further. At the time of writing this thesis, the conductor with the best field retention showed a  $B_{100}$  value of 13 T.

Literature data concerning higher temperatures is sparse, a point which could be addressed better in future work. Nevertheless, generally speaking both  $J_{c,0}^T$  and  $B_{100}$  decrease with a factor of  $\sim 2$  when going from 4.2 to 20 K. High-temperature applications at 20 K with still sufficient current density at fields of 3-5 T are thus already feasible with state-of-the-art conductors, while also here ongoing development is likely to push this field limit up.

In view of the  $J_c(B,T)$  performance, this work addresses the question which combination of deformation techniques, matrix materials and precursor powders is the most successful. In chapter 2 the fabrication of ex-situ PIT conductors using different matrix- materials and layout and different deformation techniques is described. It was found that with wire drawing alone using either a Fe or a bi-metal Cu/Fe matrix insufficient powder densification is reached to obtain high  $J_c$  values. Significantly better  $J_c$  performance was found by using after wire drawing also the flat-rolling technique with a Fe matrix.

Critical current density values of the ex-situ conductors enhances significantly after a heat treatment at 950 °C, by a factor of  $\sim 10$  in drawn wires and  $\sim 100$  in flat rolled tapes. The  $MgB_2$  grains show the neck growth behaviour typical for sintering, causing the grain-to-grain connectivity to increase. Another way to enhance the apparent  $J_c(B,T)$  performance is the addition of soft metals such as Pb. The mechanism behind this improved performance is an increase in thermal stability at higher currents, i.e. at low magnetic field. Also, Pb acts as a lubricant during drawing so that powder flow is improved, allowing for a higher overall area reduction compared to Pb-free conductors. However, the grain-to-grain connectivity is compromised with Pb additions.

These observations together indicate that for the ex-situ route with pure  $MgB_2$  powder, the most suitable technique is flat-rolling. The fact that drawing alone leads to insufficient powder density is mainly caused by the poor flow characteristics of hard particles at higher density. Also in the literature study it is found that most successful ex-situ conductors are fabricated with rolling techniques (i.e. flat-rolling or two-axial rolling), in contrast to the in-situ route where the relatively soft Mg facilitates powder flow and wires are successfully fabricated with drawing. For applications at low magnetic field, Pb-containing ex-situ conductors can be useful, in which Pb serves not only as lubricant during deformation but also as an internal stabiliser especially when a high strength and therefore usually poorly conducting matrix material is required. The effect of Pb can be further optimised by investigating lower concentrations between 1 and 5 %.

2) *Given the conductors matrix materials and operating temperature, can a  $\text{MgB}_2$  magnet be protected against thermal instabilities?*

The key parameters needed to answer this question are the propagation velocity of the normal conducting zone  $v_{nz}$  and the minimum quench energy MQE. A high  $v_{nz}$  value implies that the heat generated in the normal zone is distributed over a large volume and that the temperature rise remains limited. Also, a higher normal zone voltage is generated, detection becomes easier so that appropriate measures can be taken faster. As described in chapter 4, these parameters were studied both experimentally and numerically. Since  $\text{MgB}_2$  is particularly beneficial for applications in conduction-cooled magnet systems operating at a temperature of  $\sim 20$  K, it is important to characterise and model the normal zone development under relevant conditions. For this purpose a versatile quasi-adiabatic experimental set-up is developed with which intrinsic normal zone development can be measured as function of temperature, magnetic field and transport current.

Typical propagation velocities of the normal zone in  $\text{MgB}_2$  conductors range from 1 to 100 cm/s, depending on the conductors' matrix layout, filament properties and experimental boundary conditions. Compared to high- $T_c$  conductors, in which normal zone development can be described as static heating, detection is relatively simple. Compared to low- $T_c$  conductors, on the other hand,  $v_{nz}$  is typically a factor 100 lower, mainly due to the higher temperature margin. At a higher operating temperature  $v_{nz}$  decreases further due to the increased heat capacity. Even for samples with very different matrix materials and tested at a wide range of current densities  $v_{nz}$  reaches a minimum value ranging from 1 to 50 cm/s at  $T \approx 20$  K. Thus, normal zone detection in  $\text{MgB}_2$  conductors is certainly possible but will require more care than in low- $T_c$  conductors.

On the other hand, the probability that a normal zone occurs, which is inversely proportional to MQE, is a factor  $\sim 1000$  lower than in low- $T_c$  conductors. Again this is mainly due to the higher thermal margin. Similarly to  $v_{nz}$ , MQE increases with temperature due to the increasing heat capacity and typically reaches a maximum at 20 K. At higher temperatures MQE decreases again due to the decreasing thermal margin. The maximum MQE values range from 20 to 300 mJ, depending on the conductors' matrix material and current densities. Thus,  $\text{MgB}_2$  tends to be the most stable at an operating temperature of 20 K.

As a tool for magnet design, classical analytical models fail to predict  $v_{nz}$  and MQE accurately, mainly because they fail to take into account the filament behaviour in detail and the temperature dependence of the material properties. These factors affect the heating function during the super- to normal-conducting transition strongly and should be included. Including the voltage-current relation ( $n$ -value) and the temperature dependence a new numerical algorithm was developed that predicts the experimental data with  $\sim 5$  % accuracy. This model is not just a valuable aid for magnet design, but also allows to calculate  $v_{nz}$  and MQE of hypothetical conductors, so that selected parameters can be optimised in the very first stage of conductor design.

Compared to conductors with relatively low  $n$ -values of about 10, the ones

that have high  $n$ -values of about 100 show MQE values that are  $\sim 25\%$  lower, while their  $v_{nz}$  is a factor of  $\sim 2$  higher. Thus, at the expense of somewhat reduced stability, the detection of a normal conducting zone becomes easier. The conductivity of the matrix material mainly influences MQE: a conductor with a Fe matrix, for example, shows a factor 20 lower MQE value than one with a Cu matrix at a similar current density. At the same time  $v_{nz}$  and thus the ease of protection is hardly influenced due to the competing effects of resistivity (heat generation) and thermal conductivity (heat drainage). Thus, further development of MgB<sub>2</sub> conductors should continue to focus also on the implementation of highly conducting matrix materials.

3) *Given a MgB<sub>2</sub> conductor with a certain matrix, does it support the electromagnetic forces acting on a magnet?*

Also the answer to this question depends largely on the choice of matrix material. In chapter 5 it is described that the strain dependence of the critical current has two distinct regions. For compressive and moderate tensile strains, the critical current depends linearly and reversibly on strain. When a certain sample-dependent reversible strain limit is exceeded, the critical current degrades steeply and irreversibly. The reversible strain limit is, to first order, determined by the thermal pre-compression that the matrix exerts on the filaments. The maximum strain that a conductor can tolerate can thus be increased by choosing a matrix material that has a relatively large thermal expansion coefficient compared to the filaments.

The reversible linear regime is also important since strain-induced reversible changes in the current carrying capability can become significant, especially at higher magnetic field and temperature. It is shown that in this regime not only  $J_c$  changes linearly with strain but also the critical current  $T_c$  and the irreversibility field  $B^*$ . The strain dependence of  $J_c$  has a clear electronic origin, of which the exact mechanism is still unclear. Nevertheless, it was shown that the strain dependence of the whole critical surface for a given conductor can be predicted with only three parameters: the strain sensitivity of the zero-temperature, zero-field extrapolation of  $I_c$ ; that of the critical temperature; and that of the zero-temperature extrapolation of the irreversibility field.

*In summary, the answer to our initial questions is that present-day MgB<sub>2</sub> already constitutes a practical material for magnet applications requiring a peak magnetic field of 7-10 T at 4.2 K or 3-5 T at 20 K and these field limits are likely to increase further. From a technical point of view, thermal stability and electro-mechanical issues will, just like with other technical superconductors, require careful design which is best addressed already in the stage of material development. It is my hope that the tools and insights laid down in this thesis will form a useful contribution to these developments.*



## Appendix A

# Properties of Published Magnesium Diboride Conductors

In chapter 3 a literature survey of the transport  $J_c(B)$  performance of MgB<sub>2</sub> conductors published over the years 2003 to 2006 is presented. Selected properties of the conductors are tabulated in this appendix. The conductors are categorised by year, shape, precursor powder, number of filaments, final deformation, matrix material and additions. The values of the figures of merit,  $B_{100}$ ,  $B_p$  and  $J_{c,0}^T$  at 4.2 K, with which the statistical analysis in chapter 3 is conducted are also tabulated.

**Table A.1:** Overview of references and selected properties of published MgB<sub>2</sub> conductors. The **shape** of the conductor can be a Wire, Tape or Other; **MgB<sub>2</sub> precursor** can be Ex-situ, In-situ or Other; the **number of filaments** can be either Mono or MULTI in which case the number of filaments is also listed; **final deformation** is DRawn, Extruded, Swaged, FlatRolled, GrooveRolled, 4Rolled or Other. In the columns **matrix** and **additions** the elements are denoted by their usual symbols. Other materials that occur in these columns are Monel Steel, Stainless Steel and Carbon Nano Tubes. Irradiated samples are also noted as additions.

First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field			
									B <sub>100</sub> [T]	B <sub>p</sub> [T]	J <sub>c,0</sub> <sup>T</sup> [A/mm <sup>2</sup> ]	B <sub>100</sub> [T]	B <sub>p</sub> [T]	J <sub>c,0</sub> <sup>T</sup> [A/mm <sup>2</sup> ]	
Arda	[139]	2005	W	I	M	DR	Fe/Mon.		12.7	1.4	7000000				
Bhatia	[122]-1	2004	W	I	M	DR	Cu		1.5	1.1	400				
Bhatia	[122]-2	2004	W	I	M	DR	Cu		4.2	1.7	1100				
Bhatia	[122]-3	2004	W	I	M	DR	Fe		7.7	1.9	6200				
Bhatia	[122]-4	2004	W	I	M	DR	Cu		6.4	3.2	700				
Braccini	[8]	2006	T	E	M			C							
Chen	[141]-1	2005	W	I	M	DR	Fe		13.6			13.6	3.2		7200
Chen	[141]-2	2005	W	I	M	DR	Fe		4.7			5.1	1.6		2700
Chen	[141]-3	2005	W	I	M	DR	Fe		4.7			4.7	1.5		2000
Chen	[141]-4	2005	W	I	M	DR	Fe		5.8			5.8	1.5		5500
Chen	[141]-5	2005	W	I	M	DR	Fe		4.2			4.2	1.4		1900
Chen	[141]-6	2005	W	I	M	DR	Fe	CNT	6.5			6.5	1.5		6800
Chen	[141]-7	2005	W	I	M	DR	Fe	CNT	6.4			6.4	1.7		5100
Chen	[141]-8	2005	W	I	M	DR	Fe	CNT					1.7		6500
Chen	[141]-9	2005	W	I	M	DR	Fe	CNT					1.7		5800
Chen	[141]-10	2005	W	I	M	DR	Fe	SiC	7.0			7.0	2.3		2400
Chen	[141]-11	2005	W	I	M	DR	Fe	SiC	5.9			5.9	2.0		1700
Chen	[141]-12	2005	W	I	M	DR	Fe	SiC					2.1		2800
Civale	[97]-1	2003	W	E	M	DR	SS	SiC			300				2000
Civale	[97]-2	2003	W	E	M	DR	SS		1.5	1.5		1.5	1.5		
Civale	[97]-3	2003	W	E	M	DR	SS		1.7		< 100				
Collings	[92]-1	2003	W	I	M	DR	Fe/Mon.		6.0	1.5	6200				
Collings	[92]-2	2003	W	I	M	DR	Fe/Mon.		5.3	1.8	2000				
Dhaille	[142]-1	2005	W	I	M	DR			5.2	1.0	15300				
Dhaille	[142]-2	2005	W	E	MU	DR			5.3	1.8	1100				
Dou	[98]-1	2003	W	I	M	DR	Fe		7.8	1.5	15700				
Dou	[98]-2	2003	W	I	M	DR	Fe	SiC	12.0	2.8	7300				

*Continued on next page*

First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field			
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	
Eisterer	[143]-1	2005	W	I	M	DR	Fe/SS			7.2	1.4	16400			
Eisterer	[143]-2	2005	W	I	M	DR	Fe/SS			8.8	1.7	16300			
Eisterer	[144]-1	2005	W	I	M	DR	Fe/SS	Irr.		7.2	1.4	16400			
Eisterer	[144]-2	2005	W	I	M	DR	Cu			3.4	0.8	6500			
Eisterer	[144]-3	2005	W	I	M	DR	Cu	Irr.		5.6	1.2	10800			
Eyidi	[99]-1	2003	T	E	M	FR	Fe			5.8	1.2	12500			
Eyidi	[99]-2	2003	W	E	M	DR	SS/Fe			5.4	1.9	1800			
Fabbric.	[100]	2003	T	E	MU4	4R	Fe			2.5	0.5	11100			
Fang	[101]	2003	T	E	M	FR	Fe								
Fang	[145]-1	2005	T	I	M	FR	Fe			2.9	0.7	7100			
Fang	[145]-2	2005	T	I	M	FR	Fe								
Fang	[146]	2005	W	I	M	GR	Cu			5.6	2.2	1500			
Feng	[102]	2003	W	I	M	DR	Fe								
Feng	[103]	2003	W	I	M	DR	Fe			7.6	1.4	26600			
Feng	[123]-1	2004	W	I	M	DR	Fe			6.1	0.9	76100			
Feng	[123]-2	2004	W	I	M	DR	Fe			9.5	2.7	3400			
Feng	[123]-3	2004	W	I	M	DR	Fe			10.5	3.0	3500			
Feng	[123]-4	2004	W	I	M	DR	Fe			8.2	1.9	6600			
Feng	[123]-4	2004	W	I	M	DR	Fe			6.8	1.7	5600			
Fischer	[104]-1	2003	T	E	M	FR	Fe			9.1	1.8	15700			
Fischer	[104]-2	2003	T	E	M	FR	Fe			7.7	2.0	4700			
Fischer	[104]-3	2003	T	E	M	FR	Fe			8.0	2.0	5135			
Fischer	[104]-4	2003	T	E	M	FR	Fe			9.0	1.2	133200			
Fischer	[124]-1	2004	T	E	M	FR	Cu			3.3	2.1	500			
Fischer	[124]-2	2004	T	E	M	FR	Cu			2.2	2.9	200			
Fischer	[124]-3	2004	T	E	M	FR	Cu			2.0	2.2	300			
Fischer	[124]-4	2004	T	E	M	FR	Fe			9.1	1.8	16600			
Fischer	[124]-5	2004	T	E	M	FR	Fe			7.7	1.9	5400			
Fischer	[124]-6	2004	T	E	M	FR	Fe			8.9	1.3	87300			
Fischer	[124]-7	2004	T	E	M	FR	Fe			9.2	1.8	17000			
Fischer	[124]-8	2004	T	E	M	FR	Fe			9.2	1.6	29600			
Fischer	[124]-9	2004	T	E	MU19	FR	Fe			10.0	2.1	11700			
Fischer	[124]-10	2004	T	E	M	FR	Fe			6.6	2.0	2700			
Fischer	[124]-11	2004	T	E	M	FR	Fe			10.1	2.0	16900			
Fischer	[124]-12	2004	T	E	M	FR	Fe			8.3	1.9	7300			
Fischer	[124]-13	2004	T	E	M	FR	Fe			9.9	1.8	21500			
Flukiger	[55]-1	2003	T	E	M	FR	Fe			6.7	1.1	42000			

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field		
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\perp}$ [ $\text{A}/\text{mm}^2$ ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\parallel}$ [ $\text{A}/\text{mm}^2$ ]
Flukiger	[55]-2	2003	T	E	M	FR	Fe		1.4	1.6	200	1.4	1.6	200
Flukiger	[55]-3	2003	T	E	M	FR	Fe		5.2	0.9	38400	5.2	0.9	38400
Flukiger	[55]-4	2003	T	E	MU7	FR	Fe		4.6	0.7	59600	4.6	0.7	59600
Fu	[105]-1	2003	W	I	M	DR	Fe		6.1	1.1	33300			
Fu	[105]-2	2003	W	I	M	DR	Ta/Cu		2.0	0.6	3300			
Fu	[125]	2004	T	E	M	FR	CuNi		0.1	1.9	100			
Fuji	[106]-1	2003	T	I	M	FR	Fe	WB	7.5	1.6	12500	7.5	1.6	12500
Fuji	[106]-2	2003	T	I	M	FR	Fe	WB	8.8	1.7	15500	8.8	1.7	15500
Fuji	[106]-3	2003	T	I	M	FR	Fe	WB	8.3	1.7	14500	8.3	1.7	14500
Fuji	[107]-1	2003	T	I	M	FR	St.		1.8	1.5	300	1.8	1.5	300
Fuji	[107]-2	2003	T	I	M	FR	St.		5.2	1.8	1900	5.2	1.8	1900
Fuji	[107]-3	2003	T	I	M	FR	St.		7.1	1.6	8600	7.1	1.6	8600
Fuji	[107]-4	2003	T	I	M	FR	St.		5.6	1.5	3800	5.6	1.5	3800
Fuji	[107]-5	2003	T	I	M	FR	St.		6.1	1.4	9200	6.1	1.4	9200
Fuji	[107]-6	2003	T	I	M	FR	St.		6.2	1.3	10700	6.2	1.3	10700
Fuji	[107]-7	2003	T	I	M	FR	St.			0.3	100			
Fuji	[107]-8	2003	T	I	M	FR	St.		3.1	1.4	900	3.1	1.4	900
Fuji	[107]-9	2003	T	I	M	FR	St.		3.3	1.2	1700	3.3	1.2	1700
Fuji	[107]-10	2003	T	I	M	FR	St.		2.9	1.1	1300	2.9	1.1	1300
Fuji	[107]-11	2003	T	I	M	FR	St.		2.3	1.2	600	2.3	1.2	600
Fuji	[107]-12	2003	T	I	M	FR	St.		7.1	1.6	9500	7.1	1.6	9500
Fuji	[107]-13	2003	T	I	M	FR	St.		8.2	1.5	19600	8.2	1.5	19600
Fuji	[107]-14	2003	T	I	M	FR	St.		6.7	1.5	7600	6.7	1.5	7600
Fuji	[107]-15	2003	T	I	M	FR	St.		8.1	1.6	18200	8.1	1.6	18200
Fuji	[107]-16	2003	T	E	M	FR	St.		6.1	1.1	22900	6.1	1.1	22900
Fuji	[107]-17	2003	T	E	M	FR	St.		7.4	1.6	10100	7.4	1.6	10100
Giunchi	[53]-1	2003	W	O	M	DR	St./Nb							
Giunchi	[53]-2	2003	W	O	MU7	S	St./Nb		7.5	1.4	18300	7.5	1.4	18300
Goldacker	[108]-1	2003	W	E	M	DR	Fe		5.3	0.6	1007600	5.3	0.6	1007600
Goldacker	[108]-2	2003	W	E	M	DR	Fe/SS		6.0	0.7	958200	6.0	0.7	958200
Goldacker	[108]-3	2003	W	E	M	DR	Fe/SS		6.0	1.1	26700	6.0	1.1	26700
Goldacker	[108]-4	2003	W	E	M	DR	Fe/SS		6.1	1.2	15900	6.1	1.2	15900
Goldacker	[126]	2004	W	E	M	DR	Fe/SS		5.2	1.0	18800	5.2	1.0	18800
Goldacker	[127]-1	2004	W	I	M	DR	Fe/SS		4.0	1.1	3400	4.0	1.1	3400
Goldacker	[127]-2	2004	W	I	M	DR	Fe/SS		3.9	0.9	9700	3.9	0.9	9700
Goldacker	[127]-3	2004	W	I	M	DR	Fe/SS		3.7	0.9	6000	3.7	0.9	6000
Goldacker	[127]-1	2004	W	I	M	DR	Fe/SS		1.5	1.5	< 100	1.5	1.5	< 100
Goldacker	[127]-2	2004	W	I	M	DR	Fe/SS		1.2	1.5	200	1.2	1.5	200
Goldacker	[127]-3	2004	W	I	M	DR	Fe/SS		3.0	1.4	900	3.0	1.4	900

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field			
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	
Goldacker	[127]-4	2004	W	I	M	DR	Fe/SS		1.4	0.8	600				
Goldacker	[127]-5	2004	W	I	M	DR	Fe/SS		2.6	0.9	2200				
Goldacker	[127]-6	2004	W	E	M	DR	Fe/SS		1.1	1.0	300				
Goldacker	[127]-7	2004	W	E	M	DR	Fe/SS		4.9	1.2	5400				
Goldacker	[127]-8	2004	W	I	M	DR	Fe/SS		5.6	1.2	9700				
Goldacker	[127]-9	2004	W	I	M	DR	Fe/SS		5.6	1.2	12500				
Goldacker	[127]-10	2004	W	I	M	DR	Fe/SS		5.8	1.3	7500				
Goldacker	[127]-11	2004	W	I	M	DR	Fe/SS		7.8	1.6	14200				
Goldacker	[128]-1	2004	W	I	M	DR	Fe		5.8	1.2	12900				
Goldacker	[128]-2	2004	W	E	M	DR	Fe		5.5	1.0	19400				
Goldacker	[128]-3	2004	W	E	M	DR	Fe		3.1	1.7	600				
Goldacker	[129]-1	2004	W	I	M	DR	Fe/SS		7.3	1.5	13600				
Goldacker	[129]-2	2004	W	I	M	DR	Fe/SS		8.0	1.5	21200				
Grasso	[58]-1	2003	T	E	M	FR	Ni		2.6	1.7	500				
Grasso	[58]-2	2003	T	E	M	FR	Ni		5.4	2.4	1000				
Grasso	[58]-3	2003	T	E	M	FR	Ni		6.9	1.7	5200				
Grasso	[58]-4	2003	T	E	M	FR	Ni		5.7	1.1	17800				
Grasso	[58]-5	2003	T	E	M	FR	Ni		4.0	0.8	17000				
Grasso	[65]	2004													
Hascicek	[130]	2004													
Hassler	[109]-1	2003	T	E	M	FR	Cu					3.3	2.0	500	
Hassler	[109]-2	2003	T	E	MU16	FR	Cu					2.1	2.3	300	
Hassler	[109]-3	2003	T	E	M	FR	Cu					2.2	2.6	200	
Hassler	[165]-1	2006	T	E	M	FR	Fe					9.3	1.5	52100	
Hassler	[165]-2	2006	T	E	M	FR	Fe					9.7	1.6	49800	
Hassler	[165]-3	2006	T	E	M	FR	Fe					9.7	1.6	49800	
Hassler	[165]-4	2006	T	E	M	FR	Fe					3.1	1.1	1600	
Hassler	[165]-5	2006	T	E	M	FR	Fe					9.6	2.0	13600	
Hata	[166]-1	2006	T	I	M	FR	Fe					7.9	1.5	23000	
Hata	[166]-2	2006	T	I	M	FR	Fe	SiC				10.4	2.3	9600	
Hata	[166]-3	2006	T	I	M	FR	Fe	SiC				11.0	2.5	7800	
Hishinuma	[167]-1	2006	W	I	M	O	Fe	MgCu <sub>2</sub>	8.2	1.7	11600				
Hishinuma	[167]-2	2006	W	I	M	O	Fe	MgCu <sub>2</sub>	7.8	1.6	15000				
Hishinuma	[167]-3	2006	W	I	M	O	Fe	MgCu <sub>2</sub>	6.3	1.2	23400				
Hishinuma	[167]-4	2006	W	I	M	O	Fe	MgCu <sub>2</sub>	6.1	1.1	23500				
Holubek	[147]	2005	T	E	M	4R	Fe		4.5	0.9	14500				

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field		
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\perp}$ [ $\text{A}/\text{mm}^2$ ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\parallel}$ [ $\text{A}/\text{mm}^2$ ]
Jiang	[91]-1	2005	T	I	M	FR	Fe		8.3	1.4	36100			
Jiang	[91]-2	2005	T	I	M	FR	Fe		8.6	1.4	42500			
Jiang	[91]-3	2005	T	I	M	FR	Fe		9.2	1.4	60200			
Jiang	[91]-4	2005	T	I	M	FR	Fe		6.4	1.4	8900			
Jiang	[91]-5	2005	T	I	M	FR	Fe		8.2	1.4	30800			
Jiang	[91]-6	2005	T	I	M	FR	Fe		9.1	1.5	50700			
Jiang	[91]-7	2005	T	I	M	FR	Fe		5.0	1.6	2400			
Jiang	[91]-8	2005	T	I	M	FR	Fe	SiC	9.7	2.0	11500			
Jiang	[91]-9	2005	T	I	M	FR	Fe	SiC	10.2	2.2	10400			
Jiang	[91]-10	2005	T	I	M	FR	Fe	SiC	11.0	2.1	18000			
Jiang	[91]-11	2005	T	I	M	FR	Fe	SiC	7.2	2.4	2100			
Jiang	[91]-12	2005	T	I	M	FR	Fe	SiC	9.2	2.1	7900			
Jiang	[91]-13	2005	T	I	M	FR	Fe	SiC	9.7	2.1	10600			
Jiang	[91]-14	2005	T	I	M	FR	Fe	SiC	6.1	2.5	1100			
Jiang	[148]-1	2005	T	I	M	FR	Fe		8.2	1.5	23800			
Jiang	[148]-2	2005	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	8.5	1.7	17700			
Jiang	[148]-3	2005	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	8.7	1.7	16000			
Jiang	[148]-4	2005	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	8.7	1.7	16000			
Jiang	[148]-5	2005	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	8.3	1.7	13400			
Jiang	[148]-6	2005	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	7.8	1.7	11100			
Jiang	[148]-7	2005	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	7.2	1.6	10300			
Jiang	[149]-1	2005	T	I	M	FR	Fe		7.9	1.4	25000			
Jiang	[149]-2	2005	T	I	M	FR	Fe	MgO	8.2	1.4	30900			
Jiang	[149]-3	2005	T	I	M	FR	Fe	MgO	8.5	1.5	32600			
Jiang	[149]-4	2005	T	I	M	FR	Fe	MgO	8.3	1.5	26000			
Jiang	[149]-5	2005	T	I	M	FR	Fe	MgO	8.0	1.5	22300			
Jiang	[168]-1	2006	T	I	M	FR	Fe		8.2	1.5	23600			
Jiang	[168]-2	2006	T	I	M	FR	Fe	SiC	9.7	1.9	15400			
Jiang	[168]-3	2006	T	I	M	FR	Fe	SiC	10.2	2.1	13600			
Jiang	[168]-4	2006	T	I	M	FR	Fe	SiC	10.8	2.3	11500			
Jiang	[168]-5	2006	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	8.5	1.7	17200			
Jiang	[168]-6	2006	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	8.7	1.7	15900			
Jiang	[168]-7	2006	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	8.7	1.7	15900			
Jiang	[168]-8	2006	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	8.2	1.7	12700			
Jiang	[168]-9	2006	T	I	M	FR	Fe	$\text{Si}_3\text{N}_4$	7.8	1.7	10700			
Kikuchi	[131]	2004	W	I	M	O	Fe		2.4	0.8	2400			

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field			
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	
Kikuchi	[150]	2005	W	I	M	DR	Fe		4.1	1.1	4400				
Kim	[169]-1	2006	W	I	M	DR	Fe		6.6	1.3	19700				
Kim	[169]-2	2006	W	I	M	DR	Fe	CNT	9.2	2.0	9400				
Kim	[170]-1	2006	W	I	M	DR	Fe		6.8	1.4	15000				
Kim	[170]-2	2006	W	I	M	DR	Fe		6.8	1.2	33200				
Kim	[170]-3	2006	W	I	M	DR	Fe	CNT	8.8	1.7	17400				
Kim	[170]-4	2006	W	I	M	DR	Fe	CNT	9.2	1.9	14000				
Kim	[170]-5	2006	W	I	M	DR	Fe	CNT	7.9	1.5	20100				
Kim	[170]-6	2006	W	I	M	DR	Fe	CNT	7.0	1.4	17000				
Kim	[170]-7	2006	W	I	M	DR	Fe	CNT	8.7	1.6	27000				
Kim	[170]-8	2006	W	I	M	DR	Fe	CNT	10.1	1.9	21700				
Kitaguchi	[132]-1	2004	T	I	M	FR	Fe	SiC				10.7	2.3	11900	
Kitaguchi	[132]-2	2004	T	I	M	FR	Fe					7.9	1.5	18900	
Kitaguchi	[249]-1	2005	T	E	MU	FR	Ni/Fe/Cu					4.3	1.0	9100	
Kitaguchi	[249]-2	2005	T	E	MU	FR	Ni/Fe/Cu					3.7	0.7	18900	
Ko	[110]-1	2003	T	E	M	FR	SS					4.7	1.0	9300	
Ko	[110]-2	2003	T	E	M	FR	SS	Cu				4.2	1.1	5100	
Ko	[110]-3	2003	T	E	M	FR	SS	Cu				4.2	1.1	5100	
Ko	[110]-4	2003	T	E	M	FR	SS	Cu				4.3	1.2	3600	
Ko	[110]-5	2003	T	E	M	FR	SS							100	
Ko	[110]-6	2003	T	E	M	FR	SS	Cu				3.4	2.4	400	
Kondo	[152]-1	2005	T	I	M	FR	Fe			2.0	100				
Kondo	[152]-2	2005	T	I	M	FR	Fe	SiC	2.9	1.4	800				
Kondo	[152]-3	2005	T	I	M	FR	Fe	SiC	1.2	1.4	200				
Kondo	[152]-4	2005	T	I	M	FR	Fe	SiC	7.7	2.0	4500				
Kondo	[152]-5	2005	T	I	M	FR	Fe	SiC	7.6	1.7	8800				
Kondo	[152]-6	2005	T	I	M	FR	Fe	SiC	5.5	1.6	2900				
Kondo	[152]-7	2005	T	I	M	FR	Fe	SiC	6.9	2.1	2700				
Kondo	[152]-8	2005	T	I	M	FR	Fe	SiC	5.1	2.2	1100				
Kondo	[152]-9	2005	T	I	M	FR	Fe	SiC	5.5	1.5	3900				
Kovac	[74]-1	2004	T	E	M	4R	NiFe	SiC	5.3	1.2	8500				
Kovac	[74]-2	2004	T	E	M	4R	NiFe	Hf	5.6	1.0	22900				
Kovac	[74]-3	2004	T	E	M	4R	NiFe	W	5.3	1.2	8500				
Kovac	[74]-4	2004	T	E	M	4R	NiFe	Nb	5.5	1.0	28000				
Kovac	[74]-5	2004	T	E	M	4R	NiFe	Zr	5.3	0.9	37900				
Kovac	[74]-6	2004	T	E	M	4R	NiFe	Nb <sub>2</sub> O <sub>5</sub>	5.0	1.0	14700				

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field		
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^*$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^*$ [A/mm <sup>2</sup> ]
Kovac	[74]-7	2004	T	E	M	4R	NiFe	Ti	5.0	1.0	16000			
Kovac	[74]-8	2004	T	E	M	4R	NiFe		4.3	1.1	5300			
Kovac	[74]-9	2004	T	E	M	4R	NiFe	Ti <sub>2</sub> O <sub>5</sub>	3.3	0.9	3400			
Kovac	[74]-10	2004	T	E	M	4R	NiFe	BN	0.4	1.9	100			
Kovac	[74]-11	2004	T	E	M	4R	NiFe	V <sub>2</sub> O <sub>5</sub>	0.2	1.6	100			
Kovac	[74]-12	2004	T	E	M	4R	NiFe	SrCO <sub>3</sub>	2.3		< 100			
Kovac	[153]-6	2005	T	E	MU	4R	FeNiCo		4.7	0.9	20800			
Kovac	[153]-7	2005	W	E	MU	DR	FeNiCo		3.6	0.8	7300			
Kovac	[153]-8	2005	T	E	MU	4R	FeNiCo		4.1	0.7	25100			
Kovac	[153]-9	2005	T	E	MU	4R	FeNiCo							
Kovac	[153]-10	2005	T	E	M	4R	FeNiCo		6.3	0.9	104800			
Kovac	[154]-1	2005	T	I	M	4R	FeNiCo		2.4	1.2	700			
Kovac	[154]-2	2005	T	I	M	4R	FeNiCo		4.8	1.0	10400			
Kovac	[154]-3	2005	T	I	M	4R	FeNiCo		4.7	0.9	14800			
Kovac	[154]-4	2005	T	I	M	4R	FeNiCo		3.5	1.0	3400			
Kovac	[154]-5	2005	T	I	M	4R	FeNiCo		0.5	1.1	200			
Kovac	[154]-6	2005	T	I	M	4R	FeNiCo			1.2	< 100			12400
Kovac	[154]-7	2005	T	I	M	4R	Fe		2.9	0.8	4800			
Kovac	[154]-8	2005	T	I	M	4R	Fe		5.1	0.9	27900			
Kovac	[154]-9	2005	T	I	M	4R	Fe		4.9	0.8	33800			
Kovac	[154]-10	2005	T	I	M	4R	Fe		5.0	0.8	35200			
Kovac	[171]-1	2006	T	E	MU4	4R	Fe		4.9	0.9	29400			
Kovac	[171]-2	2006	T	E	MU4	4R	Fe		4.3	0.9	15100			
Kovac	[171]-3	2006	T	E	MU4	4R	Fe		3.6	0.8	7300			
Kovac	[171]-4	2006	T	I	MU4	4R	Nb/AgMg	SiC	6.5	1.8	3800			
Kovac	[171]-5	2006	T	I	MU4	4R	Nb/AgMg	SiC	6.5	1.7	5000			
Kovac	[171]-6	2006	T	I	MU4	4R	Nb/AgMg	SiC	5.5	1.7	2700			
Kovac	[172]-1	2006	T	E	MU6	4R	Fe		6.5	1.4	9200			
Kovac	[172]-2	2006	T	E	MU6	4R	Fe		6.7	1.4	10800			
Kovac	[172]-3	2006	T	E	MU6	4R	Fe		6.7	1.4	11400			
Kovac	[172]-4	2006	T	E	MU6	4R	Fe		6.2	1.3	10100			
Kovac	[172]-5	2006	T	E	M	4R	Fe		5.9	1.3	10800			
Kovac	[173]-1	2006	T	I	M	4R	Nb		5.4	1.0	22800			
Kovac	[173]-2	2006	T	I	M	4R	Ta		5.2	1.0	18500			
Kovac	[173]-3	2006	T	I	M	4R	Fe		4.9	0.9	21000			
Kovac	[173]-4	2006	T	E	M	4R	Fe/Cu		6.3	1.0	48800			

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field			
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	
Kovac	[173]-5	2006	T	E	M	4R	Ta/Fe								
Kovac	[173]-6	2006	T	E	M	4R	Nb/Fe								
Kumakura	[111]-1	2003	T	E	M	O	SS				5.2	1.2		6800	
Kumakura	[111]-2	2003	T	E	M	O	FeC				3.7	1.5		1300	
Kumakura	[111]-3	2003	T	E	M	O	Cu-Ni				1.2	1.7		200	
Kumakura	[111]-4	2003	T	E	M	O	Cu				0.2	1.6		100	
Kumakura	[111]-5	2003	T	E	M	GR	SS				3.9	1.5		1400	
Kumakura	[111]-6	2003	T	E	M	O	SS				4.2	0.7		28700	
Kumakura	[111]-7	2003	T	E	M	O	Fe				5.6	0.7		267600	
Kumakura	[111]-10	2003	T	E	M	O	Fe							1.4	100
Kumakura	[111]-11	2003	T	E	M	O	Fe				5.6	1.2		13300	
Kumakura	[111]-11	2003	T	E	M	O	Fe-C				7.4	1.5		12800	
Kumakura	[111]-12	2003	T	E	M	O	SS				6.3	1.8		3200	
Kumakura	[133]-1	2004	T	I	M	FR	Fe				7.9	1.5		17200	
Kumakura	[133]-2	2004	T	I	M	FR	Fe	SiC			11.0	2.0		22500	
Kumakura	[133]-3	2004	T	I	M	FR	Fe	SiC			11.0	2.4		8900	
Kumakura	[155]-1	2005	T	I	M	FR	Fe				8.0	1.6		16700	
Kumakura	[155]-2	2005	T	I	M	FR	Fe							7.3	1.3
Kumakura	[155]-3	2005	T	I	M	FR	Fe				7.1	1.4		16500	
Kumakura	[155]-4	2005	T	I	M	FR	Fe				10.6	2.2		12000	
Kumakura	[155]-5	2005	T	I	M	FR	Fe	SiC			10.6	2.2		12000	
Kumakura	[155]-6	2005	T	I	M	FR	Fe	SiC			10.6	2.2		12000	
Kuroda	[174]-1	2006	T	E	M	FR	Fe				0.1	1.3		100	
Kuroda	[174]-2	2006	T	E	M	FR	Fe	Fe						1.3	100
Kuroda	[174]-3	2006	T	E	M	FR	Fe	Fe						1.3	100
Kuroda	[174]-4	2006	T	E	M	FR	Fe	Fe						1.9	100
Kuroda	[174]-5	2006	T	E	M	FR	Fe	Fe						1.6	<100
Kuroda	[174]-6	2006	T	E	M	FR	Fe	Fe						1.9	<100
Kuroda	[174]-7	2006	T	I	M	FR	Fe	Fe						1.6	13800
Kuroda	[174]-8	2006	T	I	M	FR	Fe	Fe			8.0	1.6		3900	
Kuroda	[174]-9	2006	T	I	M	FR	Fe	Fe			6.2	1.7		800	
Kuroda	[174]-10	2006	T	I	M	FR	Fe	Fe			2.1	1.8		300	
Kuroda	[174]-11	2006	T	I	M	FR	Fe	Fe			0.1	1.8		100	
Lezza	[90]-1	2004	T	E	M	FR	Fe				6.1	1.1		25800	
Lezza	[90]-2	2004	T	E	M	FR	Fe				6.8	1.4		14300	
Lezza	[90]-3	2004	T	E	M	FR	Fe				7.0	1.2		29000	
Lezza	[156]-1	2005	T	E	M	FR	Fe				5.2	0.6		431000	

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field		
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\perp}$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\parallel}$ [A/mm <sup>2</sup> ]
Lezza	[156]-2	2005	T	E	M	FR	Fe		0.6	1042000		1.3	16000	
Lezza	[156]-3	2005	T	E	M	FR	Fe		0.8	70600		1.4	13900	
Lezza	[156]-4	2005	T	E	M	FR	Fe		0.9	31500		1.2	17000	
Lezza	[175]-1	2006	W	I	M	DR	Fe		7.2	11400				
Lezza	[175]-2	2006	W	I	M	DR	Fe		6.7	1.3	21600			
Lezza	[175]-3	2006	W	I	M	DR	Fe		6.2	1.0	57000			
Lezza	[175]-4	2006	W	I	M	DR	Fe	B <sub>4</sub> C	6.4	2.2	1700			
Lezza	[175]-5	2006	W	I	M	DR	Fe	B <sub>4</sub> C	7.9	2.1	4000			
Lezza	[175]-6	2006	W	I	M	DR	Fe	B <sub>4</sub> C	9.1	1.8	14700			
Lezza	[175]-7	2006	W	I	M	DR	Fe	B <sub>4</sub> C	6.9	1.6	7600			
Lezza	[175]-8	2006	W	I	M	DR	Fe	B <sub>4</sub> C	6.4	1.5	8100			
Lezza	[175]-9	2006	W	I	M	DR	Fe	B <sub>4</sub> C	9.3	1.7	28100			
Lezza	[175]-10	2006	W	I	M	DR	Fe	B <sub>4</sub> C	8.6	1.8	13300			
Lezza	[175]-11	2006	W	I	M	DR	Fe	B <sub>4</sub> C						
Ma	[112]-1	2003	T	I	M	FR	Fe	ZrSi <sub>2</sub>				7.7	1.8	6600
Ma	[112]-2	2003	T	I	M	FR	Fe	ZrSi <sub>2</sub>				6.7	2.0	2800
Ma	[112]-3	2003	T	I	M	FR	Fe	WSi <sub>2</sub>				6.7	1.8	3900
Ma	[112]-4	2003	T	I	M	FR	Fe	ZrB <sub>2</sub>				5.6	1.8	2300
Ma	[134]-1	2004	T	I	M	FR	Fe					5.6	1.8	2200
Ma	[134]-2	2004	T	I	M	FR	Fe	WSi <sub>2</sub>				6.6	2.0	2600
Ma	[20]-1	2006	T	I	M	FR	Fe					5.0	1.2	6900
Ma	[20]-2	2006	T	I	M	FR	Fe	ZrSi <sub>2</sub>				6.7	2.0	2500
Ma	[20]-3	2006	T	I	M	FR	Fe	SiC				10.8	1.9	27000
Ma	[176]-1	2006	T	I	M	FR	Fe					5.7	1.6	3700
Ma	[176]-2	2006	T	I	M	FR	Fe					6.0	1.7	3100
Ma	[177]-1	2006	T	I	M	FR	Fe					6.1	1.2	18700
Ma	[177]-2	2006	T	I	M	FR	Fe					9.3	1.8	16000
Ma	[177]-3	2006	T	I	M	FR	Fe	C				9.3	2.0	9700
Ma	[177]-4	2006	T	I	M	FR	Fe	C				8.8	2.2	5200
Ma	[177]-5	2006	T	I	M	FR	Fe	C				8.1	2.3	3600
Ma	[177]-6	2006	T	I	M	FR	Fe	C				7.9	2.5	2500
Ma	[177]-7	2006	T	I	M	FR	Fe	C				10.1	2.0	14900
Ma	[177]-8	2006	T	I	M	FR	Fe	C				8.0	1.4	33700
Ma	[177]-9	2006	T	I	M	FR	Fe	C				10.6	1.9	26900
Ma	[177]-10	2006	T	I	M	FR	Fe	C				11.3	2.2	17700
Malagoli	[113]-1	2003	T	E	M	FR	Ni		5.5	0.8	122500	7.5	1.7	7800

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field		
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]
Malagoli	[113]-2	2003	T	E	MU7	FR	Ni		8.5	2.3	3800	4.6	1.8	1200
Matsumoto	[114]-1	2003	T	I	M	FR	Fe					7.4	1.9	5300
Matsumoto	[114]-2	2003	T	I	M	FR	Fe	SiO <sub>2</sub>				5.8	1.9	2200
Matsumoto	[114]-3	2003	T	I	M	FR	Fe	SiO <sub>2</sub>				2.7	1.2	1000
Matsumoto	[114]-4	2003	T	I	M	FR	Fe	SiC				4.7	1.4	2500
Matsumoto	[114]-6	2003	T	I	M	FR	Fe	SiC				5.5	1.5	4300
Matsumoto	[114]-7	2003	T	I	M	FR	Fe	SiC				6.6	1.8	4100
Matsumoto	[114]-8	2003	T	I	M	FR	Fe	SiC				7.6	1.2	51300
Matsumoto	[114]-9	2003	T	I	M	FR	Fe	SiO <sub>2</sub>				5.2	1.3	4700
Matsumoto	[114]-10	2003	T	I	M	FR	Fe	SiC				9.4	1.7	22600
Matsumoto	[114]-11	2003	T	I	M	FR	Fe	SiC				8.1	1.2	89400
Matsumoto	[114]-12	2003	T	I	M	FR	Fe	SiC				8.2	1.3	55600
Matsumoto	[114]-13	2003	T	I	M	FR	Fe	SiC				8.5	1.5	35500
Matsumoto	[114]-14	2003	T	I	M	FR	Fe	SiC				7.3	1.7	7500
Matsumoto	[114]-15	2003	T	I	M	FR	Fe	SiC				8.2	1.4	28300
Matsumoto	[114]-16	2003	T	I	M	FR	Fe	SiC				5.9	0.9	73300
Matsumoto	[114]-17	2003	T	I	M	FR	Fe	SiC				6.3	1.0	57100
Matsumoto	[114]-18	2003	T	I	M	FR	Fe	SiC				4.4	0.9	11700
Matsumoto	[115]-1	2003	T	E	M	FR	SS					6.0	1.5	5300
Matsumoto	[115]-2	2003	T	E	M	FR	SS					7.2	1.7	6800
Matsumoto	[115]-3	2003	T	E	M	FR	SS					6.4	2.1	2100
Matsumoto	[115]-4	2003	T	E	M	FR	SS					5.8	0.7	312200
Matsumoto	[115]-5	2003	T	E	M	FR	SS					5.9	0.8	139100
Matsumoto	[115]-6	2003	T	E	M	FR	SS					6.3	0.9	128900
Matsumoto	[115]-7	2003	T	E	M	FR	SS					5.7	1.4	5900
Matsumoto	[115]-8	2003	T	E	M	FR	SS					6.4	1.6	5100
Matsumoto	[115]-9	2003	T	E	M	FR	SS					6.1	3.1	700
Matsumoto	[115]-10	2003	T	E	M	FR	SS					7.9	1.5	18500
Matsumoto	[115]-11	2003	T	E	M	FR	SS					7.8	1.7	11400
Matsumoto	[115]-12	2003	T	E	M	FR	SS					8.4	1.6	17600
Matsumoto	[135]-1	2004	T	I	M	FR	Fe	WSi <sub>2</sub>				8.4	1.6	17500
Matsumoto	[135]-2	2004	T	I	M	FR	Fe	ZrSi <sub>2</sub>				10.4	2.3	9700
Matsumoto	[135]-3	2004	T	I	M	FR	Fe	Mg <sub>2</sub> Si				6.4	1.7	4200
Matsumoto	[135]-4	2004	T	I	M	FR	Fe							
Matsumoto	[135]-6	2004	T	I	M	FR	Fe	SiC						
Matsumoto	[135]-7	2004	T	I	M	FR	Fe	SiO <sub>2</sub>						

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field		
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\perp}$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\parallel}$ [A/mm <sup>2</sup> ]
Matsumoto	[135]-8	2004	T	I	M	FR	Fe	SiC	11.6	2.4	13500	11.6	2.4	13500
Matsumoto	[135]-9	2004	T	I	M	FR	Fe	SiC	10.9	2.1	18500	10.9	2.1	18500
Matsumoto	[135]-10	2004	T	I	M	FR	Fe	SiC	8.2	1.4	28200	8.2	1.4	28200
Matsumoto	[21]-1	2005	T	I	M	FR	Fe			1.8	1300		1.8	1300
Matsumoto	[21]-2	2005	T	I	M	FR	Fe		6.2	1.6	5400	6.2	1.6	5400
Matsumoto	[21]-3	2005	T	I	M	FR	Fe		7.4	1.8	6500	7.4	1.8	6500
Matsumoto	[21]-4	2005	T	I	M	FR	Fe		8.0	1.6	14500	8.0	1.6	14500
Matsumoto	[21]-5	2005	T	I	M	FR	Fe		7.3	1.4	18600	7.3	1.4	18600
Matsumoto	[21]-6	2005	T	I	M	FR	Fe		8.2	1.5	23600	8.2	1.5	23600
Matsumoto	[21]-7	2005	T	I	M	FR	Fe	SiC	8.0	2.4	3000	8.0	2.4	3000
Matsumoto	[21]-8	2005	T	I	M	FR	Fe	SiC	9.6	2.4	5300	9.6	2.4	5300
Matsumoto	[21]-9	2005	T	I	M	FR	Fe	SiC	10.2	2.4	6300	10.2	2.4	6300
Matsumoto	[21]-10	2005	T	I	M	FR	Fe	SiC	8.9	2.4	4000	8.9	2.4	4000
Matsumoto	[21]-11	2005	T	I	M	FR	Fe	SiC	7.8	2.0	4900	7.8	2.0	4900
Matsumoto	[21]-12	2005	T	I	M	FR	Fe	SiC	9.7	2.2	7700	9.7	2.2	7700
Matsumoto	[21]-13	2005	T	I	M	FR	Fe	SiC	8.3	2.2	4000	8.3	2.2	4000
Nakane	[22]-1	2006	T	I	M	FR	Fe		8.4	1.6	17600	8.4	1.6	17600
Nakane	[22]-2	2006	T	O	M	FR	Fe		7.1	2.3	2400	7.1	2.3	2400
Nakane	[22]-3	2006	T	O	M	FR	Fe		8.8	2.3	4400	8.8	2.3	4400
Nakane	[178]-1	2006	T	E	M	FR	Fe		0.5	2.2	100	0.5	2.2	100
Nakane	[178]-2	2006	T	O	M	FR	Fe		7.4	2.2	3000	7.4	2.2	3000
Nakane	[178]-3	2006	T	O	M	FR	Fe		7.9	2.6	2100	7.9	2.6	2100
Nakane	[179]-1	2006	T	O	M	FR	Al		8.3	1.6	18200	8.3	1.6	18200
Nakane	[179]-2	2006	T	O	M	FR	Al		4.8	2.1	1000	4.8	2.1	1000
Nakane	[179]-3	2006	T	O	M	FR	Al		4.5	1.2	4300	4.5	1.2	4300
Nakane	[179]-4	2006	T	E	M	FR	Al							
Pachla	[157]-1	2005	W	E	M	E	Cu/Fe		4.1	0.9	9900	4.1	0.9	9900
Pachla	[157]-2	2005	W	E	M	E	Fe	SiC	5.4	1.0	17700	5.4	1.0	17700
Pachla	[157]-3	2005	W	E	M	E	Fe	SiC	2.8	1.6	600	2.8	1.6	600
Pachla	[157]-4	2005	T	E	M	4R	Fe		3.2	1.5	900	3.2	1.5	900
Pachla	[157]-5	2005	T	E	M	4R	Cu/Fe		4.6	0.9	17000	4.6	0.9	17000
Pachla	[157]-6	2005	T	E	M	4R	Fe	SiC	5.1	1.1	9000	5.1	1.1	9000
Pachla	[180]-1	2006	W	I	M	E	Fe		1.0	1.0	5700	1.0	1.0	5700
Pachla	[180]-2	2006	W	I	M	E	Fe	SiC	4.9	2.1	1100	4.9	2.1	1100
Pachla	[180]-3	2006	W	I	M	E	Fe	SiC	2.6	2.6	300	2.6	2.6	300
Pachla	[180]-4	2006	W	I	M	E	Fe	SiC	4.2	1.8	1100	4.2	1.8	1100

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field		
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]
Pachla	[180]-5	2006	W	I	M	4R	Fe		3.8	1.0	5700			
Pachla	[180]-6	2006	W	I	M	4R	Fe		5.1	2.0	1200			
Pachla	[180]-7	2006	W	I	M	4R	Fe	SiC	3.5	2.6	400			
Pachla	[180]-8	2006	W	I	M	4R	Fe	SiC		1.8	700			
Pachla	[180]-9	2006	W	I	M	4R	Fe		5.2	1.1	9900			
Perner	[158]-1	2005	T	I	M	FR	Fe					9.2	1.7	23500
Perner	[158]-2	2005	T	I	M	FR	Fe					7.7	2.0	5200
Perner	[158]-3	2005	T	I	M	FR	Fe					8.1	1.9	6400
Perner	[158]-4	2005	T	I	M	FR	Fe					8.8	1.3	104000
Ruan	[116]-1	2003	T	I	M	FR	Fe					6.1	1.2	17800
Ruan	[116]-2	2003	T	I	M	FR	Fe					6.1	1.5	6200
Schlachter	[89]-1	2003	W	E	M	S	Fe		0.5	1.3	100			
Schlachter	[89]-2	2003	W	E	M	S	Fe		4.7	1.0	9500			
Schlachter	[89]-3	2003	W	I	M	S	Fe		4.5	0.9	15700			
Schlachter	[89]-4	2003	W	I	M	S	Fe		4.6	0.9	21200			
Schlachter	[89]-5	2003	W	I	M	S	Fe		5.1	0.9	22900			
Schlachter	[89]-6	2003	W	I	M	S	Fe		3.6	0.9	4900			
Schlachter	[89]-7	2003	W	I	M	S	Fe		3.4	0.9	4800			
Schlachter	[89]-8	2003	W	I	M	S	Fe		3.0	0.8	3900			
Schlachter	[89]-9	2003	W	I	M	S	Fe		2.6	0.8	2700			
Schlachter	[89]-10	2003	W	I	M	S	Fe		2.1	0.8	1300			
Schlachter	[95]-1	2006	W	I	M	S	Cu		5.2	0.9	32000			
Schlachter	[95]-2	2006	W	I	MU7	S	Cu		4.7	1.0	10900			
Schlachter	[95]-3	2006	W	I	MU7	S	Cu		4.4	0.9	11300			
Schlachter	[95]-4	2006	W	I	MU7	S	Cu		3.5	1.0	2900			
Schlachter	[95]-5	2006	W	I	MU7	S	Fe		8.3	1.8	9900			
Schlachter	[95]-6	2006	W	I	MU19	S	Fe		8.4	1.6	17800			
Schlachter	[95]-7	2006	W	I	MU31	S	Fe		7.1	1.4	16600			
Schlachter	[95]-8	2006	W	I	MU19	S	Cu		3.8	1.0	4100			
Schlachter	[95]-9	2006	W	I	MU19	S	CuAg		3.5	1.0	3800			
Schlachter	[95]-10	2006	W	I	MU19	S	CuSn		4.4	1.3	3200			
Schlachter	[95]-11	2006	W	I	MU19	S	CuSn		3.6	1.2	1900			
Schlachter	[95]-12	2006	W	I	MU19	S	CuSn		4.3	1.3	2900			
Schlachter	[95]-13	2006	W	I	M	S	Nb/Cu/SS		7.8	1.8	7600			
Schlachter	[95]-14	2006	W	I	M	S	Nb/Cu/NiZn		7.4	1.8	6400			
Schlachter	[95]-15	2006	W	I	M	S	Cu		5.2	0.9	31300			

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field		
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\perp}$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\parallel}$ [A/mm <sup>2</sup> ]
Schlachter	[95]-16	2006	W	I	M	S	Nb/Cu		0.5	1.9	100			
Schlachter	[181]-1	2006	W	I	M	S	SS		7.9	1.3	37300			
Schlachter	[181]-2	2006	W	I	M	S	Nb/Cu/SS		9.1	1.7	23700			
Serquis	[117]-1	2003	W	E	M	DR	SS		6.0	1.1	30600			
Serquis	[117]-2	2003	W	E	M	DR	SS		7.4	1.2	41600			
Shcherbak.	[182]-1	2006	W	I	M	DR/GR	Fe		7.7	1.4	25300			
Shcherbak.	[182]-2	2006	W	I	M	DR/GR	Fe	SiC	10.3	1.8	29100			
Shcherbak.	[182]-3	2006	W	I	M	DR/GR	Fe	SiC	11.1	2.4	9700			
Shcherbak.	[182]-4	2006	W	I	M	DR/GR	Fe	SiC	9.6	2.1	9200			
Shcherbak.	[182]-5	2006	W	I	M	DR/GR	Fe	SiC	10.4	2.2	12000			
Shcherbak.	[182]-6	2006	W	I	M	DR/GR	Fe	SiC	10.2	1.8	30700			
Soltanian	[159]-1	2005	W	I	M	DR	Fe		7.1	1.1	59900			
Soltanian	[159]-2	2005	W	I	M	DR	Fe	SiC	12.0	2.5	11200			
Soltanian	[159]-3	2005	W	I	M	DR	Fe		12.0	2.6	11000			
Soltanian	[159]-4	2005	W	I	M	DR	Fe	SiC	11.4	2.1	22300			
Soltanian	[159]-5	2005	W	I	M	DR	Fe	SiC	11.3	2.1	21200			
Song	[118]	2003	T	E	M	FR	SS							
Sumption	[160]	2005	W	I	MU	DR	Nb/Cu/Mon.		7.3	2.1	3500			
Sumption	[19]-1	2006	W	I	M	DR	NbCu		3.7	2.0	800			
Sumption	[19]-2	2006	W	I	M	DR	NbCu		5.6	2.3	1200			
Sumption	[19]-3	2006	W	I	MU6	DR	Mon/NbCu		8.4	2.0	7300			
Sumption	[19]-4	2006	W	I	MU6	DR	Mon/NbCu		7.9	2.0	5200			
Sumption	[19]-5	2006	W	I	MU18	DR	Mon/NbCu		7.9	2.0	5300			
Sumption	[19]-6	2006	W	I	MU18	DR	Mon/NbCu		7.9	1.8	7600			
Sumption	[19]-7	2006	W	I	MU36	DR	Mon/NbCu		9.5	1.9	14600			
Sumption	[19]-8	2006	W	I	MU6	DR	Mon/NbCu		8.6	1.9	10100			
Sumption	[19]-9	2006	W	I	MU6	DR	Mon/NbCu		9.0	1.8	15500			
Sumption	[19]-10	2006	W	I	MU18	DR	Mon/NbCu		8.8	1.9	11000			
Sumption	[19]-11	2006	W	I	MU6	DR	Mon/NbCu		8.4	2.1	5600			
Sumption	[19]-12	2006	W	I	MU6	DR	Mon/NbCu		6.3	1.7	3500			
Sumption	[19]-13	2006	W	I	MU6	DR	Mon/NbCu		7.1	1.6	8400			
Sumption	[19]-14	2006	W	I	MU6	DR	Mon/NbCu		7.7	1.8	8200			
Sumption	[19]-15	2006	W	I	MU6	DR	Mon/NbCu		7.3	1.3	19000			
Sumption	[19]-16	2006	W	I	MU6	DR	Mon/NbCu		6.6	1.8	4000			
Sumption	[19]-17	2006	W	I	MU6	DR	CuNi/NbCu		9.5	1.8	20000			
Sumption	[19]-18	2006	W	I	MU6	DR	CuNi/NbCu		9.3	1.8	17000			

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field			
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	
Sumption	[19]-19	2006	W	I	MU6	DR	Mon/NbCu		9.1	2.0	12000				
Sumption	[19]-20	2006	W	I	MU6	DR	Mon/NbCu		9.0	1.8	13000				
Sumption	[19]-21	2006	W	I	MU18	DR	Mon/NbCu		7.8	1.8	10000				
Sumption	[19]-22	2006	W	I	MU18	DR	Mon/NbCu		6.1	1.8	4300				
Sumption	[19]-23	2006	W	I	MU18	DR	Mon/NbCu		2.8	1.8	600				
Sumption	[19]-24	2006	W	I	MU18	DR	Mon/NbCu			1.8	13000				
Sumption	[19]-25	2006	W	I	MU18	DR	Mon/NbCu			1.4	2300				
Sumption	[19]-26	2006	W	I	MU6	DR	Mon/NbCu		8.9	1.6	21300				
Suo	[119]-1	2003	T	E	M	FR	Fe					6.7	1.1	38200	
Suo	[119]-2	2003	T	E	M	FR	Fe					6.3	1.1	29800	
Suo	[119]-3	2003	T	E	M	FR	Fe					1.6	1.5	300	
Suo	[119]-4	2003	T	E	M	FR	Fe					3.0	1.5	700	
Suo	[119]-5	2003	T	E	M	FR	Fe					5.1	0.9	32400	
Suo	[119]-6	2003	T	E	M	FR	Fe					5.9	1.2	12400	
Suo	[119]-7	2003	T	E	M	FR	Fe					5.4	1.2	9200	
Suo	[119]-8	2003	T	E	M	FR	Fe					4.4	1.7	1400	
Suo	[119]-9	2003	T	E	M	FR	Fe					6.3	1.3	13600	
Suo	[119]-10	2003	T	E	M	FR	Fe					5.9	1.2	12800	
Suo	[119]-11	2003	T	E	M	FR	Fe					5.7	1.1	14900	
Suo	[119]-12	2003	T	E	MU7	FR	Fe					4.6	0.7	59800	
Suo	[119]-13	2003	T	E	MU9	FR	Fe					3.7	1.1	2900	
Tachikawa	[120]-1	2003	T	E	M	FR	Ni	In				2.2	1.0	800	
Tachikawa	[120]-2	2003	T	E	M	FR	Ni	In				2.5	1.1	1000	
Tachikawa	[120]-3	2003	T	E	M	FR	Ni	In				3.1	1.4	1000	
Tachikawa	[120]-4	2003	T	E	M	FR	Ni					0.8	0.8	300	
Tachikawa	[120]-5	2003	T	E	M	FR	Ni	Sn				1.5	1.0	400	
Tachikawa	[120]-6	2003	T	E	M	FR	Ni	Sn				1.7	1.2	400	
Tachikawa	[120]-7	2003	T	E	M	FR	Ni	Sn				2.0	1.1	600	
Tachikawa	[120]-8	2003	T	E	M	FR	Ni	Cu				1.2	1.6	200	
Tachikawa	[120]-9	2003	T	E	M	FR	Ni					0.9	1.0	< 100	
Tachikawa	[136]	2004													
Takahashi	[183]	2006	W	I	M	DR	Cu		8.9	1.9	10900				
Tanaka	[137]-1	2004	W	E	MU6	DR	SS/Cu		3.0	0.9	2700				
Tanaka	[137]-2	2004	W	E	MU6	DR	SS/Cu		3.4	1.0	2800				
Tanaka	[137]-3	2004	W	E	MU6	DR	SS/Cu		3.4	1.0	3000				
Tanaka	[137]-4	2004	W	E	MU6	DR	SS/Cu		3.8	1.1	2800				

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field			
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\perp}$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^{\parallel}$ [A/mm <sup>2</sup> ]	
Tanaka	[161]	2005	W	I	M	DR	Fe/Cu		7.4	1.3	30800				
Tanaka	[162]	2005	W	I	M	DR	Fe/Cu		7.5	1.3	33500				
Xu	[163]-1	2005	W	I	M	DR	St.		4.0	0.9	8719				
Xu	[163]-2	2005	W	I	M	DR	St.		3.9	0.9	9762				
Xu	[163]-3	2005	W	I	M	DR	St.		3.7	0.9	6230				
Xu	[163]-4	2005	W	I	M	DR	St.		3.7	0.9	6230				
Xu	[163]-5	2005	W	I	M	DR	St.		3.1	0.7	7395				
Xu	[184]-1	2006	W	I	M	DR	Fe		4.1	1.0	5100				
Xu	[184]-2	2006	W	I	M	DR	Fe		4.1	0.9	8100				
Xu	[184]-3	2006	W	I	M	DR	Fe		4.4	1.0	7100				
Xu	[184]-4	2006	W	I	M	DR	Fe		3.8	1.2	2600				
Xu	[185]-1	2006	W	I	M	DR	St.	TiB <sub>2</sub>	3.7	1.2	2500				
Xu	[185]-2	2006	W	I	M	DR	St.	TiB <sub>2</sub>	4.3	1.2	4100				
Xu	[185]-3	2006	W	I	M	DR	St.	TiB <sub>2</sub>	4.2	1.2	2900				
Xu	[185]-4	2006	W	I	M	DR	St.	TiB <sub>2</sub>	4.1	1.1	3800				
Yamada	[164]-1	2005	T	I	M	FR	Fe					9.8	1.7	30000	
Yamada	[164]-2	2005	T	I	M	FR	Fe	SiC				11.8	2.2	21000	
Yamada	[164]-3	2005	T	I	M	FR	Fe	SiC				12.1	2.3	19000	
Yamada	[164]-4	2005	T	I	M	FR	Fe	SiC				11.8	2.7	7800	
Yamada	[11]-1	2004	T	I	M	FR	Fe					12.2	2.3	18200	
Yamada	[11]-2	2004	T	I	M	FR	Fe					11.7	2.2	21400	
Yamada	[11]-3	2004	T	I	M	FR	Fe					9.8	1.8	22700	
Yamada	[11]-4	2004	T	I	M	FR	Fe					8.0	2.4	2900	
Yamada	[11]-5	2004	T	I	M	FR	Fe					5.8	1.6	3800	
Yamada	[186]-1	2006	T	I	M	FR	Fe					8.4	1.4	32000	
Yamada	[186]-2	2006	T	I	M	FR	Fe					10.1	2.1	12000	
Yamada	[186]-3	2006	T	I	M	FR	Fe	Thioph.				9.9	1.9	16700	
Yamada	[186]-4	2006	T	I	M	FR	Fe	Napth.				10.1	1.8	26500	
Yamada	[186]-5	2006	T	I	M	FR	Fe	Benz.				10.9	2.3	11000	
Yamada	[186]-6	2006	T	I	M	FR	Fe	SiC				10.6	2.1	15400	
Yamam.	[121]-1	2003	W	E	M	GR	Fe		5.3	0.9	34200				
Yamam.	[121]-2	2003	W	E	M	GR	Fe		5.7	0.8	92300				
Yamam.	[121]-4	2003	W	E	M	GR	Fe		5.3	0.7	207500				
Yamam.	[121]-5	2003	W	E	M	GR	Fe		3.4	1.1	2500				
Yamam.	[121]-6	2003	W	E	M	GR	Fe		5.1	1.2	7900				
Yamam.	[121]-7	2003	W	E	M	GR	Fe		5.3	1.0	19600				

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First Author	Ref	Year	Shp.	Prc.	# Fil.	Fin. Def.	Mat.	Add.	Perpendicular field			Parallel field			
									$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	$B_{100}$ [T]	$B_p$ [T]	$J_{c,0}^T$ [A/mm <sup>2</sup> ]	
Yamam.	[121]-8	2003	W	E	M	GR	Fe		7.0	0.8	435600				
Yamam.	[121]-9	2003	W	E	M	GR	Fe		5.5	0.8	71900				
Yamam.	[121]-10	2003	W	E	M	GR	Fe		5.3	0.8	81000				
Yamam.	[121]-11	2003	W	E	M	GR	Fe		1.1	1.2	300				
Zhan	[187]-1	2006	T	I	M	FR	Fe					6.2	1.1		27600
Zhan	[187]-2	2006	T	I	M	FR	Fe					7.2	1.4		19200
Zhan	[187]-3	2006	T	I	M	FR	Fe	MoSi <sub>2</sub>				6.9	1.3		20500
Zhan	[187]-4	2006	T	I	M	FR	Fe	MoSi <sub>2</sub>				6.9	1.3		21800
Zhan	[187]-5	2006	T	I	M	FR	Fe	MoSi <sub>2</sub>				6.2	1.5		6800
Zhan	[187]-6	2006	T	I	M	FR	Fe	MoSi <sub>2</sub>				5.9	1.4		6300
Zhan	[187]-7	2006	T	I	M	FR	Fe					5.8	1.1		15700
Zhan	[187]-8	2006	T	I	M	FR	Fe	MoSi <sub>2</sub>				7.0	1.5		10600
Zhan	[187]-9	2006	T	I	M	FR	Fe	MoSi <sub>2</sub>				6.5	1.5		8700
Zhan	[187]-10	2006	T	I	M	FR	Fe	MoSi <sub>2</sub>				6.1	1.4		7100
Zhan	[188]-1	2006	T	I	M	FR	Fe					5.8	1.1		15700
Zhan	[188]-2	2006	T	I	M	FR	Fe					7.4	1.5		13400
Zhan	[188]-3	2006	T	I	M	FR	Fe	ZrB <sub>2</sub>				7.8	1.5		16400
Zhan	[188]-4	2006	T	I	M	FR	Fe	ZrB <sub>2</sub>				8.2	1.7		14000
Zhan	[188]-5	2006	T	I	M	FR	Fe	ZrB <sub>2</sub>				7.4	1.5		13400
Zhan	[189]-1	2006	T	I	M	FR	Fe					6.2	1.1		26000
Zhan	[189]-2	2006	T	I	M	FR	Fe	SiC				9.6	2.0		12000
Zhan	[189]-3	2006	T	I	M	FR	Fe	SiC				8.6	1.6		24200
Zhan	[189]-4	2006	T	I	M	FR	Fe	Si/N/C				7.9	1.6		12700



## Appendix B

# Heat Capacity, Thermal Conductivity and Electrical Resistivity

The temperature- and field dependence of the volumetric heat capacity, thermal conductivity and electrical resistivity of the materials present in the samples studied in chapter 4 are presented in this appendix. The data are collected from literature and from the commercial database Cryocomp [32]. For some materials the properties were not found in the available literature. This is typically the case for specific alloys, in which case data is used of a similar alloy.

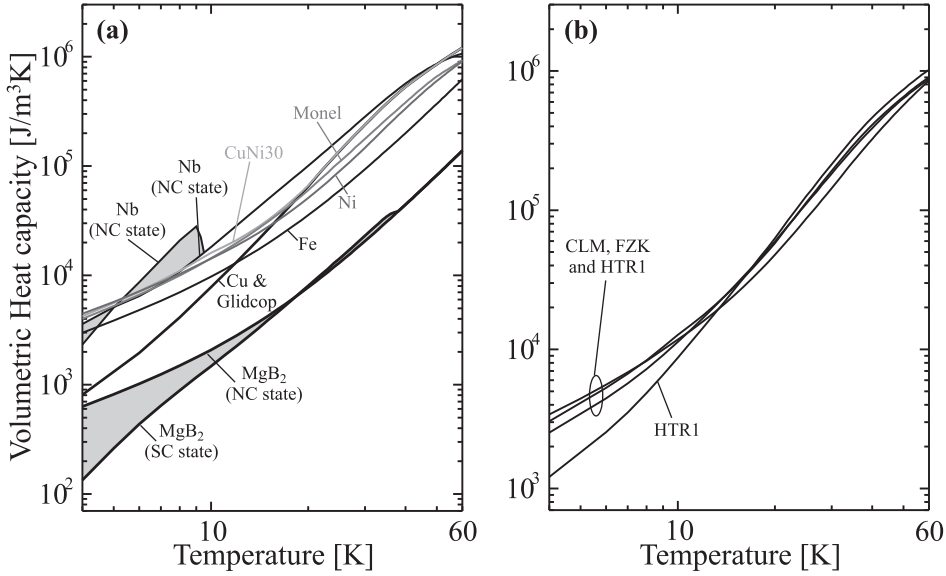
### Volumetric Heat Capacity

Figure B.1a shows the temperature dependence of the volumetric heat capacity of all the materials present in samples CLM, FZK HTR1 and HTR2.

The volumetric heat capacities of Cu, CuNi30, Nb, Ni and Monel are taken from Cryocomp. The database not only provides the temperature dependence of the heat capacity but also the field dependence when the material is a superconductor, such as Nb. It should be noted that data for CuNi30 are used for sample FZK, since there are no data available for the CuNi18Zn20 alloy in this sample. It can be seen that the data for CuNi30 follow those of Cu above  $\sim 20$  K, while below this temperature they follow the data for Ni. Comparing the heat capacity of CuNi30 and Cu80Zn20 (brass, not shown in the figure), which does not contain Ni, it follows that alloying with Zn has a limited effect on the total heat capacity as the data of Cu80Zn20 follow those of Cu even below  $\sim 20$  K<sup>1</sup>. Therefore it is assumed that the temperature dependence of CuNi30 approaches that of CuNi18Zn20.

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<sup>1</sup>For clarity volumetric heat capacity data of Cu80Zn20 is not presented here, but is available in [32, 80].



**Figure B.1:** (a) shows the volumetric heat capacity of the materials that are used in the samples in chapter 4. (b) shows the “lumped” volumetric heat capacity of those samples.

The presented heat capacity of Fe is that of Armco, which is a high purity Fe ( $\geq 99.8\%$  [241]) and the data are obtained from [250].

Heat capacity data for Glidcop are acquired from [216]. However, data below 100 K are not presented in this publication and not found in other papers. However, it is shown in [216] that above 100 K the heat capacity of Glidcop is only about 2 % higher than of Cu. Since Glidcop is a Al<sub>2</sub>O<sub>3</sub> dispersion-hardened copper, it can be expected that its heat capacity remains similar to that of Cu also below 100 K. Therefore the Glidcop data in the range between 4 and 60 K used in this study are taken to be identical to those for Cu.

The heat capacity data for MgB<sub>2</sub> used here are those published by [31], which agree within 10 % with data published by other authors [251, 252].

Figure B.1b shows the “lumped” volumetric heat capacity of samples CLM, FZK, HTR1 and HTR2 studied in chapter 4. The lumped data are calculated by taking the weighted averages of the volumetric heat capacities of the individual materials that constitute the conductors:

$$c_{\text{lumped}} = \sum_i f_i c_i, \quad (\text{B.1})$$

with  $f_i$  and  $c_i$  the volume fraction and heat capacity of the  $i^{\text{th}}$  component in the sample. The fractions of each component in the four samples are listed in table 4.1 in chapter 4. It should be noted that due to the relatively small fraction



of  $\text{MgB}_2$  or Nb in the composites, the differences in their heat capacity in the superconducting and normal states are neglected in the lumped heat capacity.

### Thermal Conductivity

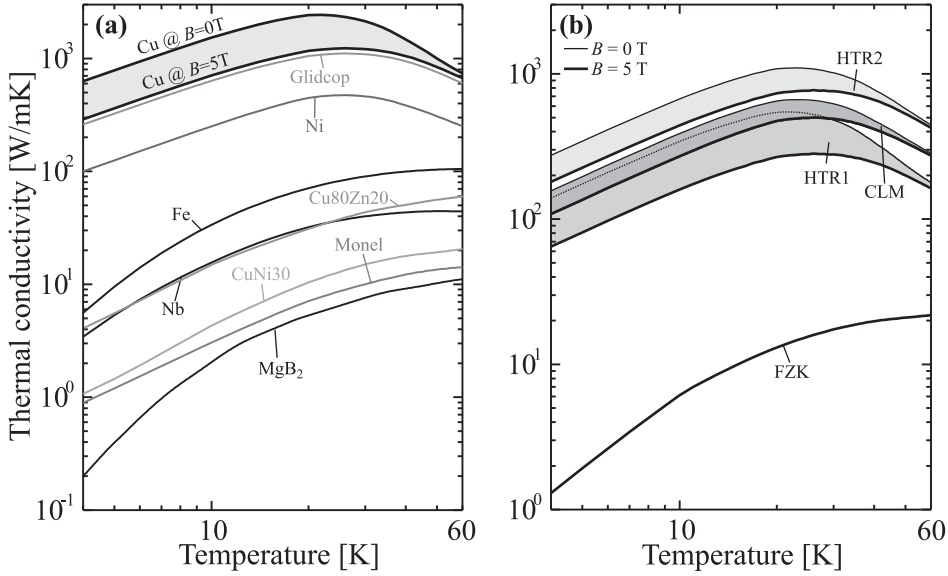
Figure B.2a shows the thermal conductivity of the materials used in samples CLM, FZK, HTR1 and HTR2.

Similar to the volumetric heat capacity  $c$ , the thermal conductivities  $\kappa$  of Cu (RRR = 100), CuNi30, Nb, Ni and Monel are obtained from Cryocomp. Also the thermal conductivity data for CuNi18Zn20 are not found in literature. Therefore the data for CuNi30 are substituted to estimate the temperature dependence of the thermal conductivity of CuNi18Zn20 in sample FZK. For comparison, also the thermal conductivity of Cu80Zn20 is plotted in figure B.2a. Clearly, alloying Cu with Ni has a stronger effect on the thermal conductivity than alloying it with Zn. Therefore it is assumed that the thermal conductivity of CuNi18Zn20 resembles that of CuNi30. Nevertheless, to improve the predictive value of the parallel-paths model in chapter 4, the thermal conductivity should ideally be measured.

The thermal conductivity of Fe is taken to be that of the high purity Armco. The data are obtained from [80].

Just like with  $c$ , the temperature dependence of  $\kappa$  for Glidcop is taken from [216], in which data are only given above 100 K. Compared to Cu, the thermal conductivity of Glidcop is approximately 10 % lower above 100 K. In figure B.2a, it is assumed that it continues to be 10 % lower than the thermal conductivity of Cu with RRR = 100 at 5 T down to 4 K. Nevertheless, as discussed in §4.5.2, it was necessary to use the thermal conductivity of Glidcop as a fitting parameter in order to have a good agreement between the model calculations of MQE and  $v_{nz}$  and the experiments, due to the large uncertainty involved in this extrapolation. For example, the peak in the thermal conductivity of Cu that results from the transition from electron-phonon to electron-impurity scattering [209] is now also included in the thermal conductivity of Glidcop. In reality, it is unsure how strong this peak will be present in Glidcop. Also, it is unknown to what extent the strong field dependence of the thermal conductivity of Cu is also present in Glidcop. The magnitude of this peak and the field dependence of the thermal conductivity of Cu are strongly related to the purity of Cu (RRR value). For Glidcop no data are available on how the thermal conductivity relates to the purity of the Cu of which it is made of. In order to improve the predictive value of the parallel-paths model in chapter 4, the thermal conductivity of Glidcop should ideally also be measured in the range from 4 to 100 K.

The thermal conductivity data for  $\text{MgB}_2$  are obtained from [30]. Compared to measurements by [253], the values are about 20 % lower. The discrepancies are presumably caused by differences in the samples or by measurement uncertainties [30]. The differences in published results are typical for the uncertainty in the estimation of the thermal conductivity of the filaments, since different preparation routes (in-situ or ex-situ and densification) can result in different thermal conductivities.



**Figure B.2:** (a) shows the thermal conductivity of the materials used in the samples studied in chapter 4. (b) shows the “lumped” thermal conductivity of the samples.

Figure B.2b shows the lumped thermal conductivity of the samples CLM, FZK, HTR1 and HTR2. Similar to the heat capacity, the lumped values of the thermal conductivity are calculated as the weighted average of the data for the individual components:

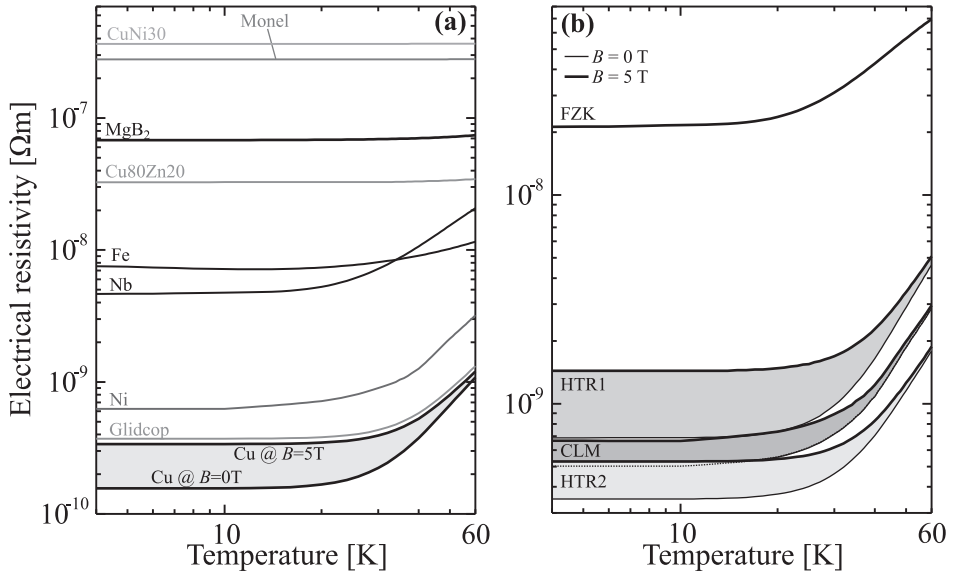
$$\kappa_{\text{lumped}} = \sum_i f_i \kappa_i, \quad (\text{B.2})$$

with  $\kappa_i$  the thermal conductivity of the  $i^{\text{th}}$  component. Due to the relatively large fraction of Cu in samples CLM, HTR1 and HTR2, the field dependence of the lumped thermal conductivity becomes significant. This field dependence is accounted for in the numerical model discussed in chapter 4.

### Electrical Resistivity

Figure B.3a displays the electrical resistivity of the materials used in samples CLM, FZK, HTR1 and HTR2.

Data for Cu (RRR = 100), CuNi30, Nb, Ni and Monel are obtained from Cryocomp. Once more, the electrical resistivity of CuNi30 is used to characterise that of CuNi18Zn20. Comparing the CuNi30 data with that of Cu80Zn20 it can be seen that, just like for the thermal conductivity, Ni has a much stronger effect



**Figure B.3:** (a) shows the electrical resistivity of the materials used in the samples studied in chapter 4. (b) shows the “lumped” electrical resistivity of those samples.

on the resistivity of Cu alloys than Zn. Therefore it is assumed that the electrical conductivity of CuNi18Zn20 resembles that of CuNi30.

Electrical resistivity data for Fe (Armco) are obtained from [80].

The temperature dependence of the electrical resistivity of Glidcop is taken from [216]. Just like with the thermal conductivity, electrical resistivity data in this paper are only given above 100 K. Above this temperature the electrical resistivity is approximately 10 % higher than that of Cu and it is assumed that it continues to be so down to 4 K (with respect to Cu with RRR = 100 and at  $B = 5$  T). The uncertainty in the electrical resistivity is, similar to the thermal conductivity, the unknown purity of the Cu of which Glidcop is made of; its relation to the overall resistivity of Glidcop; and its response to a magnetic field.

Normal-state electrical resistivity data for MgB<sub>2</sub> are obtained from [30]. Compared to the data from [254] these data are a factor of 3 lower, which can again be attributed to the different preparation routes.

Also the lumped electrical resistivity of the samples is presented here in figure B.3b. It should be noted, however, that the lumped resistivity of the matrix materials and the normal-state electrical resistivity of the filaments are taken separately in the parallel-paths model, since the superconducting-to-normal transition of the filaments is accounted for. The overall lumped resistivity serves as an indication for overall resistivity of a sample in the normal state. They are calculated

through the weighted average of the electrical conductivities ( $1/\rho$ ):

$$\frac{1}{\rho_{\text{lumped}}} = \sum_i \frac{f_i}{\rho_i}, \quad (\text{B.3})$$

with  $\rho_i$  the electrical resistivity of the  $i^{\text{th}}$  component in the composite conductor. The field dependence of the lumped resistivity of samples CLM, HTR1 and HTR2 is caused by the presence of a relatively large fraction Cu and is accounted for in the numerical model.

## Appendix C

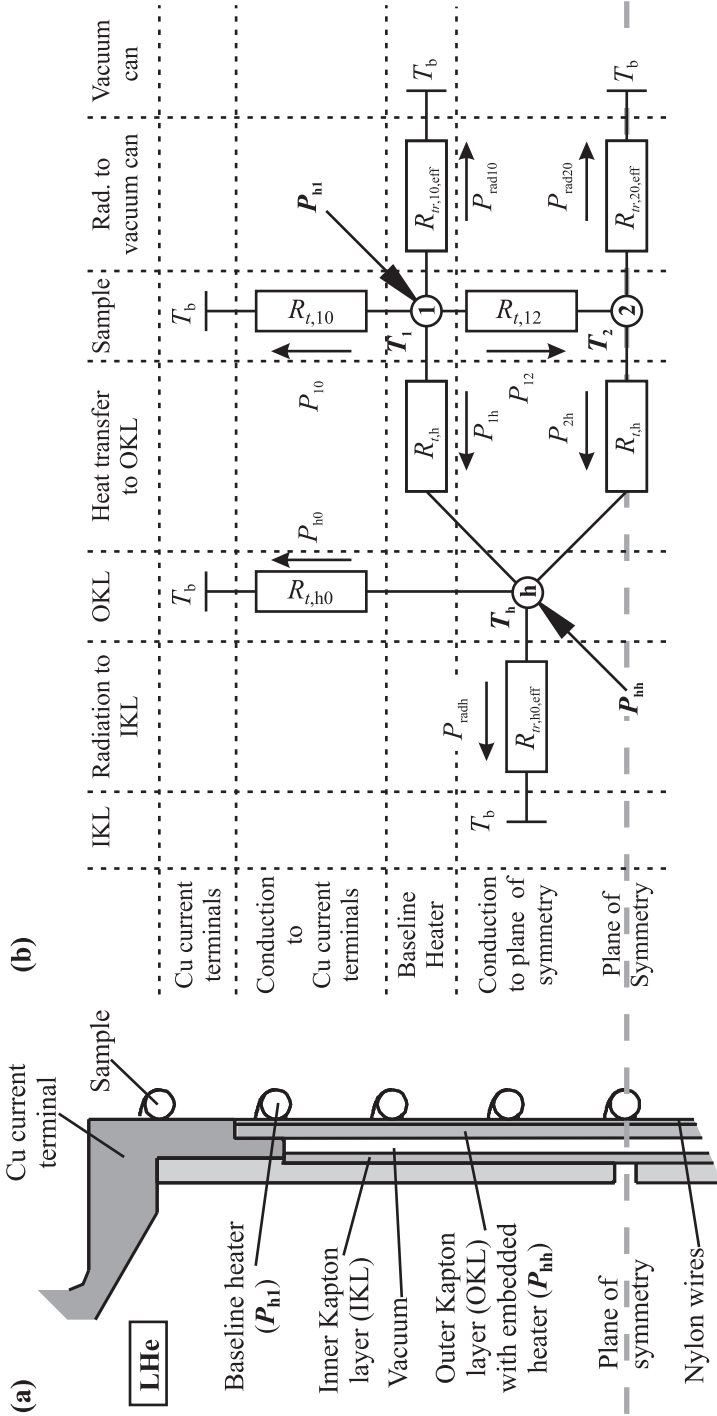
# Design of the Thermal Stability Experiment

In §4.4.3, the modification of the existing stability set-up –designed for use at 4.2 K only– to higher temperatures is described. The main problem was to minimise lateral heat-leak from the wires sample to the sample holder. A first modification to improve the homogeneity of the temperature over the sample was to minimise the thermal contact with the sample holder using Nylon wires (figure C.1a). Furthermore, the Kapton layer was separated into an inner- and outer Kapton layer with a vacuum space in-between them. With this improvement, the homogeneity of the temperature over the sample was still insufficient. The only way to further improve the homogeneity to an acceptable level turned out to be an embedded heater in the outer Kapton layer. This conclusion is based on an analytical thermal model of the sample holder / sample assembly. In this appendix the thermal model is discussed in detail and it is shown how it led to this conclusion.

### Analytical Model

The analytical model is based on an equivalent thermal network of the sample holder / sample assembly. The relation between the actual assembly and the equivalent network is shown in figure C.1. The model makes the following assumptions:

1. The problem is symmetric around the mid-plane of the sample holder, indicated as the “plane of symmetry”;
2. The inner Kapton layer, the Cu current terminals and the walls of the vacuum can all have a fixed temperature  $T_b = 4.2$  K.



**Figure C.1:** (a) Shows a part of the cross-section of the sample holder, while (b) shows the equivalent thermal network.

With these assumptions, three nodes are defined at which the temperature is to be solved as a function of the heater inputs  $P_{h1}$  on the sample and  $P_{hh}$  embedded in the Kapton:

**node 1:** the point on the sample where the sample heater  $P_{h1}$  is mounted, close to the Cu current terminal and at a temperature  $T_1$ ;

**node 2:** the point on the sample where it crosses the plane of symmetry, with temperature  $T_2$ ;

**node h:** the outer Kapton layer with temperature  $T_h$ . This is also the location of the embedded heater  $P_{hh}$ .

The three unknown temperatures  $T_1$ ,  $T_2$  and  $T_h$  are solved by balancing the heat fluxes at these three nodes:

$$\begin{array}{rcccccc} P_{h1} & -P_{10} & -P_{rad1} & -P_{12} & -P_{1h} & = 0 & \text{(node 1)} \\ & & -P_{rad2} & +P_{12} & -P_{2h} & = 0 & \text{(node 2)} \\ P_{hh} & -P_{h0} & -P_{radh} & +P_{1h} & +P_{2h} & = 0 & \text{(node h)} \end{array} \quad (C.1)$$

Node 1 receives heat from heater  $P_{h1}$  mounted on the sample. From this node heat is conducted through the sample to the Cu current terminal ( $P_{10}$ ) and to node 2 ( $P_{12}$ ). A radiative heat flux goes from node 1 to the vacuum can ( $P_{rad1}$ ). Furthermore, the lateral cooling of the sample to the outer Kapton layer is represented by  $P_{1h}$ . Node 2 receives a conductive heat flux from node 1 ( $P_{12}$ ). Heat is lost by radiation the vacuum can ( $P_{rad2}$ ) and via lateral cooling to the outer Kapton layer ( $P_{2h}$ ). The outer Kapton layer (node h) receives heat from the embedded heater ( $P_{hh}$ ) and incoming heat fluxes from nodes 1 and 2 ( $P_{1h}$  and  $P_{2h}$  respectively). Furthermore, heat is distributed via conduction to the Cu current terminal ( $P_{h0}$ ) and via radiation to the inner Kapton layer ( $P_{radh}$ ).

The heaters  $P_{h1}$  and  $P_{hh}$  are resistive so that the heat that they generate can be derived from their electrical resistance and from the current with which they are fed.

The conductive heat fluxes,  $P_{10}$ ,  $P_{12}$ ,  $P_{1h}$ ,  $P_{2h}$  and  $P_{h0}$ , are estimated using the effective thermal resistances  $R_t$  of the corresponding conduction paths:

$$\begin{array}{l} P_{\text{cond}} = \frac{\Delta T}{R_t}, \\ \text{with } R_t = \frac{l}{A\kappa}. \end{array} \quad (C.2)$$

$\Delta T$  is the temperature drop across the conduction path;  $l$  and  $A$  are its length and cross-sectional area, respectively; and  $\kappa$  its thermal conductivity.

Radiative heat flux in this problem always occurs between a surface at a fixed LHe temperature of  $T_b = 4.2$  K (inner Kapton layer or vacuum can) and a surface  $x$  with a variable -a-priori unknown- temperature (sample temperatures  $T_1$  and  $T_2$  and outer Kapton layer temperature  $T_h$ ). These radiative heat fluxes are

described as:

$$P_{\text{rad}} = c_{\text{rad}} [T_x^4 - T_b^4], \quad (C.3)$$

$$\text{with } c_{\text{rad}} = \frac{\sigma_{\text{SB}}}{\frac{1 - \epsilon_x}{A_x \epsilon_x} + \frac{1}{A_x \nu} + \frac{1 - \epsilon_b}{A_b \epsilon_b}}.$$

$T_x$  is the unknown temperature  $T_1$ ,  $T_2$  or  $T_h$ ;  $T_b$  is the LHe temperature; the proportionality constant  $c_{\text{rad}}$  depends on the Stefan-Boltzmann constant ( $\sigma_{\text{SB}} = 5.669 \cdot 10^{-8} \text{ W m}^{-2} \text{ K}^{-4}$ ), the surface areas  $A_x$  and  $A_b$ , the emissivity of the two surfaces ( $\epsilon_x$  and  $\epsilon_b$ ) and the geometric view factor  $\nu$  [255]. Note that for the radiation terms the temperature of the sample between the Cu current lead and node 1 is assumed to be at temperature  $T_1$  and between node 1 and node 2 at temperature  $T_2$ .

The view factor  $\nu$  from the outer- to the inner Kapton layer is assumed to be that of two infinitely long concentric cylinders. In this case, the outer cylinder radiates to the inner cylinder and the view factor takes the form:  $\nu = A_b/A_x$  [255]. The view factor from the sample to the vacuum can is rather complicated because of the sample's helical shape. To simplify the problem, the view factor is again assumed to be that of two infinitely long concentric cylinders. In this case the inner cylinder (the sample) radiates to the outer cylinder (the vacuum can) and the view factor  $\nu = 1$ . Note that with this assumption the view factor (thus  $P_{\text{rad}}$ ) is overestimated.

In order to simplify the solution of (C.1), the radiative heat fluxes are linearised with respect to the unknown temperature  $T_x$  by developing (C.3) in a first order Taylor expansion:

$$P_{\text{rad}} \approx c_{\text{rad}} (T_{x,0}^4 - T_b^4) + 4c_{\text{rad}} T_{x,0}^3 (T_x - T_{x,0})$$

$$= \frac{T_x}{R_{tr,x0}} - \frac{T_b}{R_{tr,0x}}, \quad (C.4)$$

with

$$R_{tr,x0} = [4c_{\text{rad}} T_{x,0}^3]^{-1};$$

$$R_{tr,0x} = \left[ c_{\text{rad}} \left( T_b^3 + 3 \frac{T_{x,0}^4}{T_b} \right) \right]^{-1}.$$

$R_{tr,x0}$  and  $R_{tr,0x}$  can be regarded as the ‘‘thermal resistances’’ close to surfaces  $x$  and  $b$  respectively<sup>1</sup>.  $T_{x,0}$  is a temperature that needs to be estimated a-priori and

<sup>1</sup>Note that for ease of representation, the radiative heat transfer in figure C.1 is represented by a single effective resistance, i.e.:

$$\frac{T_x}{R_{tr,x0}} - \frac{T_b}{R_{tr,0x}} \equiv \frac{T_x - T_b}{R_{tr,x0,\text{eff}}}.$$



is taken to be the average estimated temperature of the whole radiation surface under consideration. For example, in table C.1 in the radiation term from the part of the sample between the current terminal and node 1 to the vacuum can (column S1 → vacuum can)  $T_{x,0}$  is listed with a value of 15 K, which is an approximate average of the temperature of  $T_1$  at node 1 and the bath temperature  $T_b$ .

With equations (C.2) and (C.4), the set of linear equations (C.1) can be written as:

$$\begin{aligned} P_{h1} - \left( \frac{T_1 - T_b}{R_{t,10}} \right) - \left( \frac{T_1}{R_{tr,10}} - \frac{T_b}{R_{tr,01}} \right) - \left( \frac{T_1 - T_2}{R_{t,12}} \right) - \left( \frac{T_1 - T_h}{R_{t,h}} \right) &= 0 \\ &- \left( \frac{T_2}{R_{tr,20}} - \frac{T_b}{R_{tr,02}} \right) + \left( \frac{T_1 - T_2}{R_{t,12}} \right) - \left( \frac{T_2 - T_h}{R_{t,h}} \right) = 0 \quad (\text{C.5}) \\ P_{hh} - \left( \frac{T_h - T_b}{R_{t,h0}} \right) - \left( \frac{T_h}{R_{tr,h0}} - \frac{T_b}{R_{tr,0h}} \right) + \left( \frac{T_1 - T_h}{R_{t,h}} \right) + \left( \frac{T_2 - T_h}{R_{t,h}} \right) &= 0 \end{aligned}$$

Reorganising (C.5) leaves the following linear equation:

$$\begin{pmatrix} -1 & 1 & 1 \\ \frac{R_{t,1\text{tot}}}{R_{t,10}} & \frac{1}{R_{t,12}} & \frac{1}{R_{t,h}} \\ 1 & -1 & 1 \\ \frac{R_{t,12}}{R_{t,12}} & \frac{R_{t,2\text{tot}}}{R_{t,12}} & \frac{R_{t,h}}{R_{t,h}} \\ 1 & 1 & -1 \\ \frac{R_{t,h}}{R_{t,h}} & \frac{R_{t,h}}{R_{t,h}} & \frac{R_{t,\text{htot}}}{R_{t,h}} \end{pmatrix} \begin{pmatrix} T_1 \\ T_2 \\ T_h \end{pmatrix} = \begin{pmatrix} -P_{h1} - \frac{T_b}{R_{t,10}} - \frac{T_b}{R_{tr,01}} \\ -\frac{T_b}{R_{tr,02}} \\ -P_{hh} - \frac{T_b}{R_{t,h0}} - \frac{T_b}{R_{tr,0h}} \end{pmatrix}, \quad (\text{C.6})$$

with

$$\begin{aligned} R_{t,1\text{tot}} &= \frac{1}{R_{t,10}} + \frac{1}{R_{tr,10}} + \frac{1}{R_{t,12}} + \frac{1}{R_{t,h}}; \\ R_{t,2\text{tot}} &= \frac{1}{R_{tr,20}} + \frac{1}{R_{t,12}} + \frac{1}{R_{t,h}}; \\ R_{t,\text{htot}} &= \frac{1}{R_{t,h0}} + \frac{1}{R_{tr,h0}} + \frac{2}{R_{t,h}}. \end{aligned}$$

In this equation, the temperatures  $T_1$ ,  $T_2$  and  $T_h$  are the parameters to be solved; the heater powers  $P_{h1}$  and  $P_{hh}$  are the independent variables and all the thermal resistances are known through the relevant material properties (see equations (C.2) and (C.4)) and are worked out –as an example– for a given wire in table C.1. The only remaining thermal resistance that is a-priori unknown is the resistance between the sample and the outer Kapton layer  $R_{t,h}$ . This resistance consists of poorly defined contacts between the sample and the Nylon wires. Therefore  $R_{t,h}$  is determined by fitting  $T_1$  and  $T_2$  in equation (C.6) to the measured temperature  $T_1$  and  $T_2$ , using  $R_{t,h}$  as a free parameter.

**Table C.1:** Estimate of the thermal resistivities in the sample holder / sample assembly at a baseline temperature of 25 K. The sample is a 19 filament MgB<sub>2</sub>/CuSn wire with  $d = 0.9$  mm and a filling factor  $f$  of 17 %. The data of the material properties are obtained from [30, 32, 255, 256].

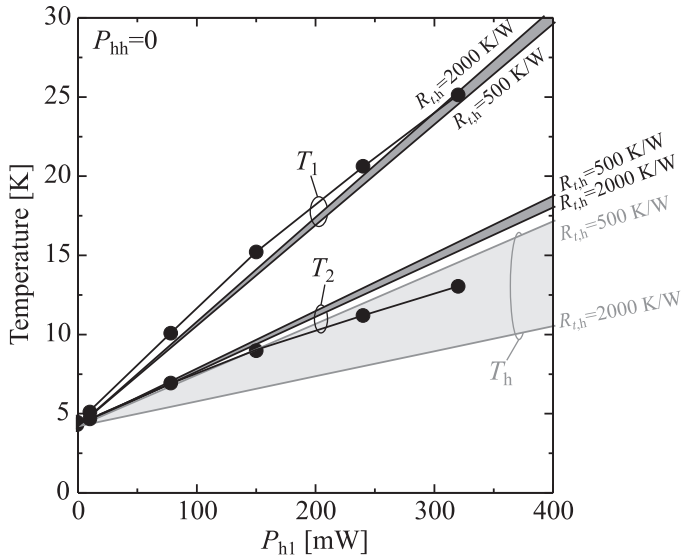
		Node 1 ↓ Curr. term.	S1 <sup>†</sup> ↓ Vac. can	Node 1 ↓ Node 2	S2 <sup>†</sup> ↓ Vac. can	Node h (OKL) ↓ Curr. term.	Node h (OKL) ↓ IKL
<b>Conduction</b>							
$A$	[mm <sup>2</sup> ]	0.64	-	0.64	-	65	-
$l$	[mm]	10	-	490	-	3	-
$\kappa$	[W/mK]	280	-	280	-	0.1	-
$R_t$	[K/W]	56	-	2752	-	459	-
<b>Radiation</b>							
$T_{x,0}$	[K]	-	15	-	20	-	12
$A_x$	[mm <sup>2</sup> ]	-	304	-	2485	-	3100
$A_b$	[mm <sup>2</sup> ]	-	471	-	3847	-	2900
$\epsilon_x$	[-]	-	0.7	-	0.7	-	0.9
$\epsilon_b$	[-]	-	0.8	-	0.8	-	0.9
$\nu$	[-]	-	1	-	1	-	1.1
$R_{tr,x0}$	[·10 <sup>6</sup> K/W]	-	5.31	-	0.27	-	0.11
$R_{tr,0x}$	[·10 <sup>6</sup> K/W]	-	1.98	-	0.08	-	0.03

<sup>†</sup> S1 is the sample between Cu current lead and node 1. S2 is the sample between node 1 and node 2.

### Temperature Profile across the Sample

The measurements are performed with a 19 filament MgB<sub>2</sub>/CuSn wire with a diameter of 0.9 mm and a filling factor of 17 %. Node 1 with the sample heater  $P_{h1}$  is located approximately 3 mm from the upper Cu current terminals, which correspond to approximately 10 mm along the conductor. At this point an 80  $\Omega$  resistor is mounted on the wire that acts as heater  $P_{h1}$ . Close to this point a Cernox thermometer ( $T_1$ ) is mounted on the wire. To ensure symmetry around the midplane of the holder, a similar heater ( $P'_{h1}$ ) and thermometer ( $T'_1$ ) are mounted  $\approx 3$  mm from the bottom Cu current terminal (node 1'). At node 2 a third Cernox thermometer is mounted to monitor  $T_2$ . The relevant parameters estimated for this configuration are listed in table C.1.

$T_1$ ,  $T_2$  and  $T'_1$  are then measured as a function of the heater powers  $P_{h1}$  and  $P'_{h1}$ , while  $P_{hh}$  is switched off. In this way the temperature gradient between  $T_1$  and  $T_2$  is representative for the radial heat loss along the sample. The results of



**Figure C.2:** Measured temperatures  $T_1$  and  $T_2$  as a function of the heater power  $P_{h1}$ , with  $P_{hh} = 0$  (data-points). The **dark gray bands** indicate the calculations with  $R_{t,h}$  ranging from 500 to 2000 K/W. Also plotted (**light gray band**) is the corresponding temperature of the outer Kapton layer  $T_h$ .

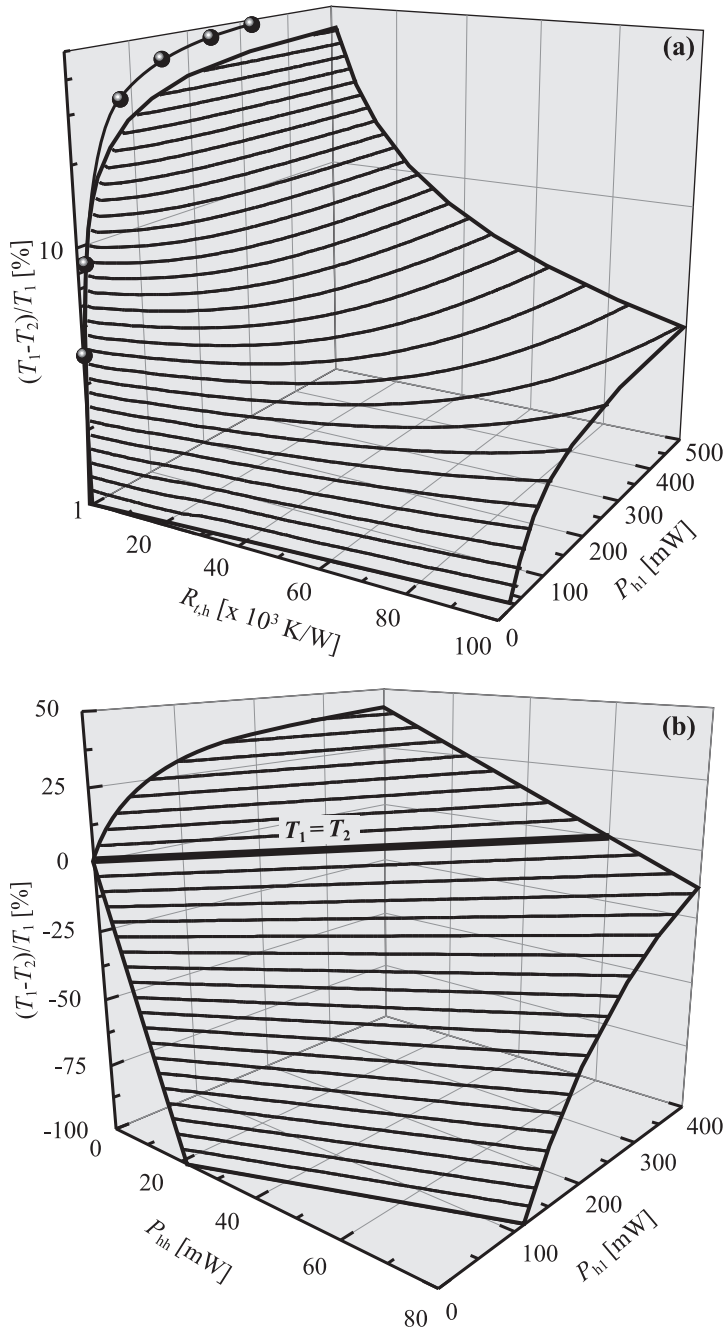
the measurements are plotted in figure C.2, along with the model calculations. It can be seen that the model describes both  $T_1$  and  $T_2$  as a function of  $P_{h1}$  reasonably well when  $R_{t,h}$  is taken to be in the range between 500 and 2000 K/W. The deviation between the calculations and the data can be attributed to the simplifications that were assumed in the model. For instance, the downward curvature of the measured data is not described by the model. This is likely due to the linearisation of the radiative heat fluxes and to the increasing heat conduction through the wiring at higher temperatures, which is not accounted for in the model.

Note that the resulting value of  $R_{t,h}$ , ranging between 500 and 2000 K/W, is relatively low compared to the thermal resistance of the sample between nodes 1 and 2 ( $R_{t,12} \approx 2750$  K/W). Consequently, in spite of the Nylon spacers a significant amount of heat is lost to the underlying outer Kapton layer, resulting in a temperature gradient along the sample between nodes 1 and 2. In order to improve the homogeneity of the temperature, either  $R_{t,h}$  should be increased or the outer Kapton layer should be heated ( $P_{hh}$ ). Both options are considered below.

## Results

Figure C.3a displays the calculated effect of increasing  $R_{t,h}$  further. The calculated relative temperature gradient between  $T_1$  and  $T_2$  is plotted as a function of the heater power ( $P_{h1}$ ) and the thermal resistance between sample and outer Kapton layer ( $R_{t,h}$ ). Included in the figure (data points) are the measured temperature gradients (from figure C.2) at  $R_{t,h} \approx 1000$  K/W. Increasing the value of  $R_{t,h}$  by a factor  $\sim 100 \sim 1 \cdot 10^5$  K/W still leaves a maximum temperature gradient of about 3 %. Although this would be a significant improvement, table C.1 shows that this value of  $R_{t,h}$  would be similar to that of the radiative thermal resistances. In other words, even a free-hanging sample would still show a significant temperature gradient. So increasing  $R_{t,h}$ , even to unrealistically high levels, would not result in an acceptable homogeneity over the sample.

Figure C.3b shows the effect of using an embedded heater  $P_{hh}$  in the outer Kapton layer instead of increasing  $R_{t,h}$ . In this graph, the relative temperature gradient between  $T_1$  and  $T_2$  is plotted against the heater powers  $P_{h1}$  and  $P_{hh}$  (assuming that  $R_{t,h} \approx 1000$  K/W, as derived from the measurements at  $P_{hh} = 0$ ). This figure shows that, for sufficiently high  $P_{hh}$ -values, the temperature gradient can even be inverted with  $T_2 > T_1$ . This implies that in principle,  $P_{h1}$  and  $P_{hh}$  can be adjusted in such a way that  $T_1 = T_2$ . The model shows that for this particular sample, a homogeneous profile is achieved when  $P_{hh} \approx 0.15P_{h1}$ . Also worth noting is that using only  $P_{hh}$  (along the line  $P_{h1} = 0$ ), would leave a negative gradient (i.e.  $T_2 > T_1$ ) and would not lead to an acceptable temperature homogeneity. Thus to eliminate the temperature gradient, both the heaters on the sample's ends ( $P_{h1}$  and  $P'_{h1}$ ) and the embedded heater ( $P_{hh}$ ) have to be used.



**Figure C.3:** (a) Calculated relative temperature difference as a function of the heater power  $P_{h1}$  and the thermal resistance between sample and outer Kapton layer ( $R_{t,h}$ ). The data points indicate the measurements (see figure C.2) at an assumed  $R_{t,h}$  of 1000 K/W. Note the logarithmic scale of the  $(T_1 - T_2)/T_1$ -axis. (b) Calculated relative temperature difference as a function of the heater power at the outer Kapton layer ( $P_{hh}$ ) and the heater power at node 1 ( $P_{h1}$ ).



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# Summary

With the discovery of its superconducting properties in 2001, MgB<sub>2</sub> is the latest superconductor for use in large scale applications such as superconducting magnets.

Compared to other metallic superconductors like Nb-Ti and Nb<sub>3</sub>Sn, it has the advantage of a higher  $T_c$  (40 K), which enables magnets to operate at higher temperatures ( $\sim 20$  K) and cuts down cooling costs. Compared to high- $T_c$  conductors such as YBa<sub>2</sub>Cu<sub>3</sub>O<sub>*x*</sub> and Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>*x*</sub> on the other hand, it is a relatively simple compound and therefore easier to synthesise. Together with the abundance of magnesium and boron, this causes its presently estimated cost-to-performance ratio to be a factor of  $\sim 100$  lower than that of high- $T_c$  conductors.

MgB<sub>2</sub> conductors are fabricated with the Power-In-Tube technology, a technique that is already applied for Nb<sub>3</sub>Sn and Bi-based high- $T_c$  superconductors. In this study the ex-situ route is chosen, in which pre-reacted MgB<sub>2</sub> powder is sealed in a metal tube that is subsequently drawn into wires or rolled into tapes. The resulting conductors consist of a composite metal matrix embedding MgB<sub>2</sub> filaments.

Such a composite conductor can break already in an early stage of wire drawing. A first possible cause is the particle size of the MgB<sub>2</sub> powder. In the as-purchased powder, the largest particles ( $\sim 100 \mu\text{m}$ ) cause stress concentrations in the matrix during deformation. An effective technique to reduce the particle size is shear milling, in which the as-purchased powder is suspended in a fluid and milled with a relatively large number of small milling balls ( $d \sim 3 \text{ mm}$ ). Due to shear stresses, the maximum particle size reduces to  $\sim 2 \mu\text{m}$  after 48 h of milling. However, care has to be taken with the choice of suspension fluid. Ethanol and isopropanol leave carbon in-between the MgB<sub>2</sub> grains, compromising the superconducting properties of the MgB<sub>2</sub> filament.

A second possible cause of premature wire breakage during drawing is the choice of matrix materials, which is not only dictated by ease of deformation but also by a number of functional requirements. The matrix should be a highly conducting material such as Cu or Al to provide stabilisation against thermal disturbances. Unfortunately, MgB<sub>2</sub> tends to react strongly with such materials so that a practical wire typically has a bimetal matrix consisting of a chemically compatible inner layer and a highly conducting outer layer. These type of materials often have a different hardness, which may lead to breakage of the hardest of

them due to different flow characteristics of the individual metals. Model calculations and experiments show that careful selection of the volume fractions of the matrix metals can prevent premature breakage.

One important parameter dictating the critical current density of the conductor is the maximum attainable filament density, since it determines the degree of grain-to-grain connectivity. For the ex-situ route, drawing alone leads to insufficient powder density due to the poor flow characteristics of the hard  $\text{MgB}_2$  particles. With tape rolling significantly higher filament densities can be achieved, resulting in a factor of 5-10 higher critical current density.

Addition of Pb to the  $\text{MgB}_2$  filaments can facilitate powder flow by acting as a lubricant during deformation. This is demonstrated by the higher maximum reduction that can be achieved during drawing. Pb also enhances the thermal stability of the conductors at high currents and low magnetic fields.

To illustrate trends and prospects in the development of  $\text{MgB}_2$  conductors a statistical study of critical current versus magnetic field data published over the years 2003-2006 was conducted. The main progress in the development of  $\text{MgB}_2$  conductors is found in the field retention of the critical current. Between 2003 and 2006 the usable field range has, on average, increased by 25 %. This progress is mainly a consequence of the trend towards in-situ conductors, in which unreacted Mg and B are put into the matrix as separate phases. In this type of conductor low-level doping can better be controlled and increases the field retention of the critical current. It was found that carbon-based dopants tend to enhance the field retention of the critical current more than non carbon-based dopants. This could suggest that carbon based dopants improve the second critical field as well as magnetic flux line pinning, while non-carbon based dopants only enhance pinning.

One of the requirements on  $\text{MgB}_2$  conductors used in magnets is imposed by the need to protect the device against thermal instabilities. Already small heat releases of the order of  $\mu\text{J}$  can cause the conductors' temperature to rise locally above its critical temperature, creating a normal-conducting zone. The heat released in this zone can lead to a burnout of the conductor, unless the normal conducting zone is detected in time, protection circuits can be triggered and the current switched off.

The probability that a normal zone occurs is inversely proportional to the minimum quench energy. Measurements show the minimum quench energy of  $\text{MgB}_2$  conductors to be typically a factor  $\sim 1000$  higher than in low- $T_c$  conductors. Thus, the probability that a thermal disturbance induces a normal zone is significantly lower than in low- $T_c$  conductors.

The ease of protection against burnout depends on the speed with which the normal zone propagates. High- $T_c$  conductors typically display quasi-static heating and detection is difficult. In low- $T_c$  conductors protection is easier due to the relatively fast propagation of the normal zone, with several m/s. In  $\text{MgB}_2$  conductors the normal zone propagates a factor of  $\sim 100$  slower than in low- $T_c$  conductors.



Thus, protection is considerably easier than with high- $T_c$  conductors but requires more care than in low- $T_c$  conductors.

In magnets large Lorentz forces act on the superconducting windings, which influence the superconducting properties. Application of  $\text{MgB}_2$  conductors in magnets requires that they withstand these electro-mechanically induced strains.

The strain dependence of the critical current show two regimes. For compressive strains and at low tensile strain levels,  $\text{MgB}_2$  conductors show a linear and reversible strain dependence of the critical current, while at higher tensile strain levels the wire is damaged and the critical current degrades steeply and irreversibly.

The boundary between the two regimes is, to first order, dictated by the thermal pre-strain that the matrix exerts on the filaments. This implies that the maximum strain that an  $\text{MgB}_2$  conductor can tolerate can be increased by choosing a matrix material that has a relatively large thermal expansion coefficient.

In the linear regime, the strain dependence of the critical current has an electronic origin. Important for magnet design is that in this reversible regime the strain dependence of the critical current at any given temperature and magnetic field can be predicted by knowing only three parameters: the strain sensitivity of the zero-temperature, zero-field extrapolation of the critical current; that of the critical temperature; and that of the zero-temperature extrapolation of the irreversibility field.

The general conclusion of this thesis is that at present  $\text{MgB}_2$  already constitutes a practical superconductor for magnet applications requiring peak fields of 7-10 T at 4.2 K or 3-5 T at 20 K. It is likely that these fields will increase further. From a technical point of view, thermal stability and electro-mechanical issues can be addressed by careful conductor design, just like with any other technical superconductor.



# Samenvatting (Summary in Dutch)

Met de ontdekking van supergeleidende eigenschappen in  $\text{MgB}_2$  in 2001, is het de nieuwste supergeleider voor gebruik in grootschalige toepassingen zoals magneten.

Vergeleken met andere metallische supergeleiders zoals Nb-Ti en  $\text{Nb}_3\text{Sn}$  heeft  $\text{MgB}_2$  het voordeel van een hogere  $T_c$  (40 K). Hierdoor kunnen magneten gebruikt worden bij een hogere temperatuur ( $\sim 20$  K), met lagere kosten voor koeling. Aan de andere kant, in vergelijking met keramische hoge- $T_c$  geleiders zoals  $\text{YBa}_2\text{Cu}_3\text{O}_x$  en  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  is  $\text{MgB}_2$  een relatief eenvoudige verbinding en dus makkelijker te synthetiseren. Samen met de ruime beschikbaarheid van magnesium en boor betekent dit dat de “prijs-kwaliteit” verhouding een factor  $\sim 100$  lager liggen dan bij hoge- $T_c$  geleiders.

$\text{MgB}_2$  geleiders worden gemaakt met de zogenaamde Poeder-In-Buis technologie. Deze techniek wordt al geruime tijd gebruikt voor  $\text{Nb}_3\text{Sn}$  en hoge- $T_c$  geleiders op basis van bismuth. In dit onderzoek is de ex-situ route gekozen waarbij een metalen buis met voorgereageerd  $\text{MgB}_2$  poeder wordt gevuld en vervolgens wordt getrokken tot draden of gewalst tot tapes. De resulterende geleider bestaat dan uit een composietmatrix met daarin  $\text{MgB}_2$  filamenten.

Een dergelijk samengestelde geleider kan tijdens het draadtrekken al in een vroeg stadium breken. Een mogelijke oorzaak is de deeltjesgrootte van het  $\text{MgB}_2$  poeder. In het aangekochte poeder kunnen tijdens het draadvormen de grootste deeltjes ( $\sim 100 \mu\text{m}$ ) spanningsconcentraties veroorzaken in de matrix. Een effectieve techniek om de deeltjesgrootte te reduceren is malen door middel van afschuifspanningen. Hierbij wordt het poeder in suspensie gebracht en vervolgens gemaald, waarbij een groot aantal kleine maalballetjes ( $d \sim 3 \text{ mm}$ ) afschuifspanningen veroorzaken in de deeltjes. Na 48 uur malen is de maximum deeltjesgrootte gereduceerd tot  $\sim 2 \mu\text{m}$ . Voorzichtigheid is geboden bij de keuze van het suspensievloeistof. Bijvoorbeeld ethanol en isopropanol laten koolstof achter tussen de  $\text{MgB}_2$  deeltjes waarbij de supergeleidende eigenschappen verslechteren.

Een andere mogelijke oorzaak van vroegtijdige breuk tijdens het draadtrekken is de keuze van het matrixmateriaal. Deze keuze wordt niet alleen bepaald door het gewenste trekgedrag, maar ook door een aantal functionele vereisten. Een van

de vereisten is dat de matrix een lage thermische en elektrische weerstand heeft, zodat de geleider thermische stabiel is. Voorbeelden van zulke metalen zijn Cu en Al.  $MgB_2$  reageert echter sterk met deze materialen, zodat praktische geleiders typisch een bi-metalen matrix hebben met een chemisch compatibel binnenste laag en een goed geleidende buitenste laag. Vaak hebben deze metalen echter ook verschillende hardheden die kunnen leiden tot breuk vanwege te sterk afwijkende vloeikarakteristieken. Modelberekeningen en experimenten tonen aan dat een zorgvuldige keuze van de volumefracties van de matrix metalen vroegtijdige breuk kan voorkomen.

Een belangrijke parameter die de kritieke stroomdichtheid in de geleider bepaalt is de maximaal haalbare dichtheid van het filament. Dit bepaalt in welke mate individuele deeltjes elektrisch contact hebben. Slechts trekken van ex-situ draad leidt tot onvoldoende poederdichtheid vanwege het gebrekkige vloeigedrag van de harde  $MgB_2$  poederdeeltjes. Het rollen tot een tape daarentegen leidt tot aanmerkelijk hogere filamentdichtheden, wat resulteert in kritieke stroomdichtheden die een factor 5-10 hoger zijn.

Toevoeging van Pb aan het  $MgB_2$  poeder als smeermiddel tijdens de deformatie leidt tot gunstiger vloeigedrag. Dit resulteert in een hogere maximale reductie tijdens het trekken. Een ander voordeel van het toegevoegde Pb is een verhoogde thermische stabiliteit van de geleiders bij hogere stromen en lager magnetische velden.

Om tendensen en vooruitzichten in de ontwikkeling van  $MgB_2$  geleiders te bestuderen werd een statistische studie uitgevoerd omtrent de veldafhankelijkheid van de kritieke stroom in geleiders waarover gepubliceerd werd tussen 2003 en 2006. In die jaren is het veldafhankelijkheid van de kritieke stroom gemiddeld verbeterd met 25 %. Deze vooruitgang is toe te schrijven aan de trend richting in-situ geleiders waarbij ongereageerd Mg en B als aparte materialen in de matrix worden gebracht. In dit type geleider kan het toevoegen van doteringen beter gecontroleerd worden en als gevolg daarvan verbetert de veldafhankelijkheid van de kritieke stroom. Doteringen die gebaseerd zijn op koolstof verbeteren de veldafhankelijkheid van de kritieke stroom meer dan doteringen die op andere materialen zijn gebaseerd. Dit kan erop duiden dat dotering gebaseerd op koolstof zowel de tweede kritieke veld als de pinning kracht van de magnetische fluxlijnen verbetert, terwijl andere doteringen alleen de pinning kracht verhogen.

Een van de vereisten voor  $MgB_2$  geleiders in magneten wordt bepaald door het feit dat het instrument beveiligd moet kunnen worden tegen thermische instabiliteit. Kleine warmte ontwikkelingen van de grootte-orde  $\mu J$  kunnen de temperatuur van de geleider lokaal doen stijgen boven de kritieke temperatuur. Hierbij wordt een normaal geleidende zone gecreëerd. De warmteontwikkeling in deze zone kan leiden tot doorbranden van de geleider, tenzij de normale zone op tijd wordt gedetecteerd, beveiligingscircuits worden geactiveerd en de stroom wordt uitgeschakeld.

De kans dat een normaal geleidende zone ontstaat is omgekeerd evenredig met

de “minimum quench energy”. Metingen laten zien dat de “minimum quench energy” van  $\text{MgB}_2$  geleiders typisch een factor  $\sim 1000$  hoger is dan typische waarden voor lage- $T_c$  geleiders. Dit betekent dat de kans dat een thermische verstoring een normale zone veroorzaakt significant kleiner is.

Hoe goed de geleider beveiligd kan worden tegen doorbranden wordt bepaald door de snelheid waarmee de normale zone zich uitbreidt. In hoge- $T_c$  geleiders is beveiliging relatief moeilijk omdat ze lokaal en quasi-statisch kunnen opwarmen na een thermische verstoring. In lage- $T_c$  geleiders daarentegen is beveiliging relatief makkelijk door de snelle propagatie van de normale zone met enige meters per seconde. In  $\text{MgB}_2$  geleiders propageert de normale zone met een factor  $\sim 100$  langzamer dan in lage- $T_c$  geleiders. Dit betekent dat beveiliging aanzienlijk makkelijker is dan in hoge- $T_c$  geleiders, maar meer zorgvuldigheid vereist dan in lage- $T_c$  geleiders.

In magneten ondervinden de supergeleidende windingen hoge mechanische belasting vanwege de Lorentz krachten en de supergeleidende eigenschappen worden hierdoor beïnvloed. Het toepassen van  $\text{MgB}_2$  geleiders in magneten vereist dat ze bestand zijn tegen deze elektro-mechanisch geïnduceerde rek.

De rekafhankelijkheid van de kritieke stroom heeft twee regimes. Voor druk (compressie) en lage rektoestanden heeft de kritieke stroom een lineaire en reversibel rekafhankelijkheid. Bij grotere rekwaarden beschadigt de draad en neemt de kritieke stroom abrupt en irreversibel af.

De grens tussen de twee gebieden wordt, in eerste aanleg, bepaald door de thermische voorspanning die de matrix uitoefent op de filamenten. Dit houdt in dat de maximale rek die een  $\text{MgB}_2$  geleider kan verdragen kan worden verhoogd door een matrix materiaal te gebruiken met een relatief hoge thermische uitzettingscoëfficiënt.

In het lineaire gebied heeft de rekafhankelijkheid van de kritieke stroom een elektronische oorsprong. Belangrijk voor het ontwerpen van magneten is dat in dit reversibele gebied de rekafhankelijkheid van de kritieke stroom voor elke gegeven temperatuur en elk veld voorspeld kan worden met slechts drie parameters: de rekgevoeligheid van de naar 0 K en 0 T geëxtrapoleerde kritieke stroom; van de kritieke temperatuur; en van het naar 0 K geëxtrapoleerde “irreversibiliteits veld”.

De algemene conclusie van dit proefschrift is dat de huidige  $\text{MgB}_2$  geleiders al een praktische materiaal vormen voor toepassing in magneten waarbij een maximaal veld wordt vereist van 7-10 T bij 4.2 K of 3-5 T bij 20 K. Bovendien is het erg waarschijnlijk dat deze veldwaarden verder zullen stijgen. Vanuit technisch oogpunt zullen  $\text{MgB}_2$  geleiders, net als alle andere praktische supergeleiders, zorgvuldig ontworpen moeten worden om te voldoen aan eisen omtrent thermische stabiliteit en electro-mechanische kwesties.



# Dankwoord

“Ben je goed genoeg om te promoveren?” Een typische vraag van Herman ten Kate, ergens gedurende mijn afstuderen. Een flinke hoeveelheid  $MgB_2$  geleiders, allerhande metingen en een flink proefschrift later ben ik toegekomen aan schrijven van het dankwoord. Het moge duidelijk zijn wat mijn antwoord op Hermans vraag was.

Terugkijkend, realiseer ik me dat het een erg multidisciplinair promotieonderzoek is geworden: van de preparatie van  $MgB_2$  geleiders tot onderzoek naar de applicatie in magneten. Een dergelijk breed onderzoek is iets wat ik erg leuk vind om te doen. Dat betekent automatisch dat er ook veel mensen zijn die meegewerkt hebben aan het project en die ik hier wil bedanken.

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Na 6 jaar wereldwijd onderzoek naar praktische  $\text{MgB}_2$  supergeleiders waren we ook wel eens nieuwsgierig naar het toekomstperspectief en trends van dit materiaal. Dit vergde een grondige statistische analyse waarvoor ik speciaal Esther wil bedanken, die mij in no-time het één en ander bijgebracht over statistiek.

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Hoewel het er soms wel op lijkt, gaat het leven van een AIO niet alleen over promoveren. Ik wil mijn vrienden bedanken voor de leuke tijd die ik met ze heb en die mijn promovendusleven een stuk aangenamer hebben gemaakt. Daarnaast wil ik speciaal mijn ouders, Chris en Trudy, bedanken voor hun interesse en steun. Als laatste wil ik mijn lieve Irene bedanken voor haar steun, gezelligheid en liefde!

Harald.