JOURNAL OF MATERIALS SCIENCE 16 (1981) 2154–2166

Microstructure dependence and origin of local energy release in b c c technical superconductors during strain

G. PASZTOR*, C. SCHMIDT
Kernforschungszentrum Karlsruhe, Institut für Technische Physik, Postfach 3640, 7500 Karlsruhe, West Germany

Dynamic stress effects in b c c technical superconductors NbTi and NbZr were studied using two measuring techniques. In short sample training experiments the load at which normal transition due to instantaneous local energy release occurred in successive straining cycles was measured. The acoustic emission was monitored during strain at 4.2 K and is explained by a stress induced shear tranformation. A time correlation was found between normal transition and acoustic emission, although the shear transformation does not furnish the required energy for transition into the normal state. The experiments strongly indicate that the mechanism inducing normal transition is micro-yielding, induced by shear transformation.

1. Introduction

The training effect of superconducting magnets unfortunately seems to remain a problem for the magnet designer during the eighties. Training means that the proposed design current of the magnet is reached only after a number of energizing cycles connected with premature normal transitions at successively higher current values. Mechanical effects in the superconductor itself [1–3] have been suggested among others to be responsible for training. Short sample training experiments [4] tend to support the view that the energy release during low temperature deformation of superconductors may be *one* cause of training of superconducting magnets.

Most of the previous investigations have dealt with the effect of stress on the superconducting properties [5–7] and very little work has been directed towards understanding the mechanism of energy release during deformation of superconductors. Evans [1] has put forward the idea that serrated yielding could be the mechanism inducing normal transitions whereas Heim [2] proposed that training is associated with the inelastic deformation of the copper matrix. Although these ideas appeared to be attractive, a doubt remained as pointed out in a previous work [8].

The possibility of a direct investigation of the origin of stress-induced energy release in superconductors was demonstrated by short sample training experiments [4, 8, 9]. The present work is an attempt to determine more thoroughly the effects of metallurgical history and alloy composition on the stress-induced energy release in the b c c superconducting NbTi and NbZr alloys.

2. Experimental procedure

The experiments described in this paper were performed on a series of NbTi and NbZr alloy superconductors. All samples were single core conductors where the copper matrix was removed on the test length. The NbTi samples are listed in Table I. Some of the conductors are commercial type conductors, the others are developmental conductors. The samples taken from a special fabricated batch of Nb–60 wt% Ti are characterized in Table II. The NbZr samples are of commercial compositions containing between 25 and 50 wt% zirconium (see Table III). The metallurgical treatment is described as far as is known, and the

*Present address: Brown, Boveri and Cie., Dept. IMS-L, CH-8050 Zürich, Switzerland.

 0022–2461/81/082154–13$03.30/0

TABLE I Investigated NbTi alloys. The Nb–50 wt % Ti is commercial, the other are developmental conductors. Supplier: Vacuumschmelze Hanau

Sample	Diameter (mm)	I_c (5T) (A)	I_c (0) (A)	Condition*	Characteristic microstructure
Nb–50 wt % Ti	0.27	130	476	Current-optimized 4 x(HT + CW)	Dislocation cell structure α-Ti precipitations
Nb–39 wt % Ti	0.33	48	313	Current-optimized 4 x(HT + CW)	Dislocation cell structure
Nb–39 wt % Ti	0.33	21	118	Cold worked	Dislocation cell structure
Nb–46.5 wt % Ti	0.33	128	480	Current-optimized 2 x(HT + CW)	Dislocation cell structure α-Ti precipitations
Nb–46.5 wt % Ti	0.33	66	255	Cold worked	Dislocation cell structure
Nb–49.5 wt % Ti	0.27		495	Current-optimized 3 x(HT + CW)	Dislocation cell structure α-Ti precipitations
Nb–49.5 wt % Ti	0.27		73	Cold worked	Dislocation cell structure
Nb–52 wt % Ti	0.34		561	Current-optimized	Dislocation cell structure α-Ti precipitations
Nb–52 wt % Ti	0.34		101	Cold worked	Dislocation cell structure
Nb–65 wt % Ti	0.4	214	675	Current-optimized 2 x(HT + CW)	Dislocation cell structure α-Ti precipitations

*HT: Heat treated in the two-phase region.
CW: Subsequently cold worked.

resultant characteristic microstructure is outlined. Table IV gives the interstitial concentrations of the samples.

The measurements were made in an apparatus described in detail earlier [10]. This is a tensile machine, in which wire samples immersed in liquid helium can be strained while carrying a transport current. The load was applied to the sample holder through a pull-rod by means of an electromagnet installed at the top of the cryostat. The load was monitored by a piezoelectric stress gauge and the strain by a parallel-plate capacitance gauge. A piezoelectric transducer, resonant at 150 kHz, was fixed to the sample holder to permit recording of acoustic signals during strain [4]. The transducer output signal was fed into a pre-amplifier with a fixed gain of 40 dB, then passed through a band-pass filter and further amplified by a variable-gain amplifier. An electronic counter was used to count the number of oscillations, whose amplitudes exceeded a fixed threshold.

TABLE II Processing history and current densities of the Nb–60 wt % Ti samples. NbTi diameter 0.38 mm. Supplier Atomics Int. USA

Sample number	Heat treatment	Degree of cold work (%)	j_c ($B = 0$) (10^5 A cm^{-2})
1	450° C/2 h	75	5.3
2	450° C/2 h	86	5.2
3	450° C/2 h	91	5.0
4	450° C/2.5 h	91	5.1
5	450° C/3.5 h	91	5.4
6	450° C/2 h	96.3	3.7
7	450° C/2 h	98.4	1.8

TABLE III NbZr conductors

Sample	Diameter (mm)	j_c ($B = 0$) 10^5 A cm^{-2}	Supplier
Nb–25 wt % Zr	0.27	11.0	Vacuumschmelze
Nb–25 wt % Zr	0.25	12.4	Westinghouse
Nb–33 wt % Zr	0.25	3.7	Atomics Int.
Nb–50 wt % Zr	0.3	8.7	Teledyne Wah Chang

TABLE IV Analysis of the interstitial content of some investigated alloys. The analysis was carried out twice for all samples, the second value is given in case it differs from the first

Sample	O_2 (wt ppm)	N_2 (wt ppm)	H_2 (wt ppm)
Nb–39 wt % Ti optimized	500	< 100	21/24
Nb–46.5 wt % Ti optimized	1000	< 100	20/30
Nb–50 wt % Ti optimized	300/400	< 100	22/22
Nb–52 wt % Ti optimized	500/600	< 100	20/30
Nb–60 wt % Ti (No. 7)	300	< 100	67/70
Nb–65 wt % Ti optimized	600	< 100	17/22
Nb–25 wt % Zr Westingh.	500	< 100	40/50
Nb–33 wt % Zr	500/600	< 100	89/97
Nb–50 wt % Zr	500/600	< 100	60/80

The acoustic emission data together with load or strain were recorded on a digital storage oscilloscope and stored on magnetic tape.

In a typical experiment investigating short sample training, the sample was strained at a constant stress rate of about $5 \times 10^8 \mathrm{N\,m^{-2}\,sec^{-1}}$, while the sample was supplied with a constant transport current below the critical value, I_c, e.g. 95% of I_c. At a certain stress level the sample became normal. The voltage signal appearing on the sample was used to switch off the transport current as well as the load, which fell back to zero with the time constant of the electromagnet, i.e. 80 msec. After the sample had returned to the superconducting state, the current was applied again and straining repeated. The stress at which the normal transition occurred usually increased from step to step, i.e. a training effect of the conductor was found. The difference relative to magnetic training was that in an energized magnet the current, the field and the load increased together while, in our experiments, the transport current was maintained at a constant value and the external magnetic field, B, was zero.

3. Results

3.1. Effects of alloy composition and metallurgical history on stress-induced energy release in NbTi and NbZr

3.1.1. NbTi alloys

The composition of the alloy is of importance in considering the stress effects in NbTi superconductors. According to the NbTi phase diagram [11], compositions with 44 wt % or less Ti undergo no phase separation. Alloys with higher Ti contents, on the other hand, become two-phased when subjected to heat treatment in the two-phase $\alpha + \beta$ region: the nucleation and growth of a second phase precipitate (α-Ti) can occur in these alloys.

A training effect was encountered both in the two-phase and the single-phase β-alloys. Fig. 1 shows training curves of alloys with various Ti-contents subjected to precipitation heat treatments which improve the current density, j_c. Fig. 1 also shows alloys which are only cold worked to the same wire size (see Table I). Two

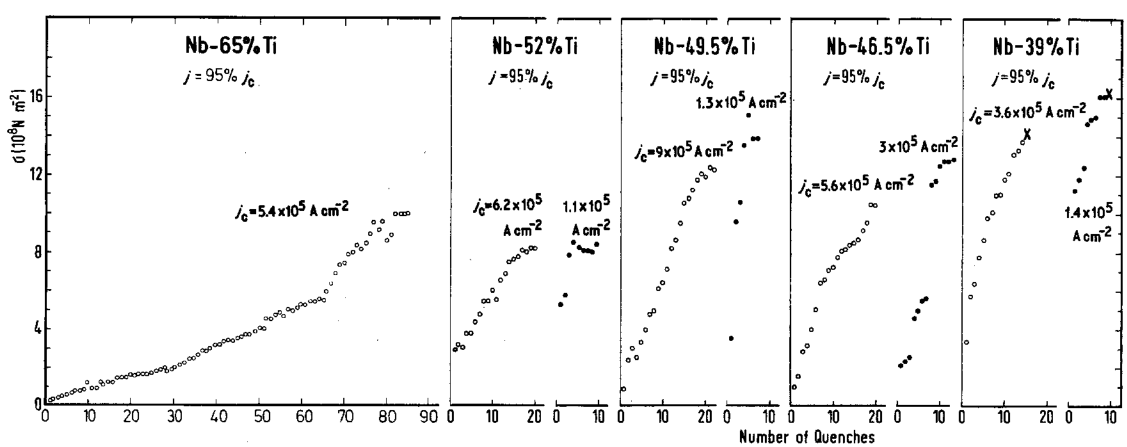

Figure 1 Short sample training of current-optimized (○) and cold worked (●) NbTi conductors with different Ti-contents. No external field, $j = 95\% j_c$. Samples in liquid helium (×: failure of specimen).

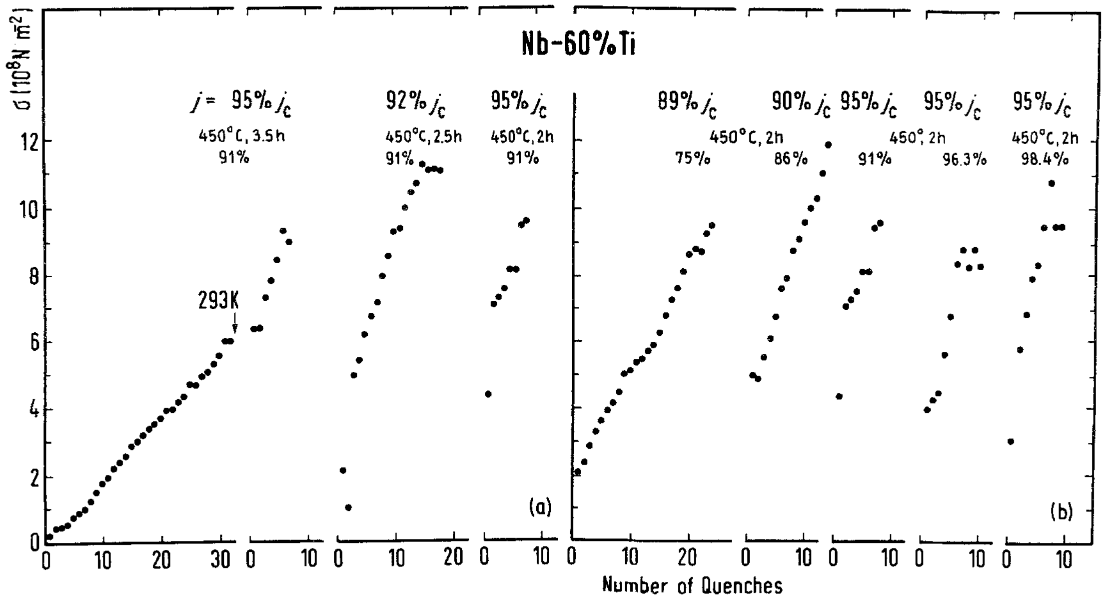

Figure 2 Effect of metallurgical history on short sample training of Nb–60 wt % Ti (a) dependence on annealing time, constant degree of cold work (91%) and (b) dependence on final cold work, constant annealing conditions (450° C for 2 h). No external field.

results are apparent from the training behaviour of NbTi alloys. There is firstly the decrease of the training effect in current-optimized samples with compositions of $\lesssim$ 50 wt % Ti as compared to the Nb–65 wt % sample. Secondly, there is a marked decrease of training of those samples which were not subjected to heat treatments.

The role of the metallurgical history was investigated in more detail in a series of experiments with the Nb–60 wt % Ti samples (see Table II and McInturff and Chase [12]). The main feature of the processing of this two-phase alloy is that the wires were precipitation heat-treated at intermediate size, then further cold drawn to the final diameter of 0.38 mm. Two parameters were varied, the duration of precipitation annealing and the degree of final cold work. Precipitation heat-treatments of 2, 2.5 and 3.5 h at 450° C were followed to a constant degree of cold work of 91%, while a second series of samples were heat-treated for 2 h and then cold worked, covering a range between 75 and 98.4%.

Fig. 2a shows the dependence of the training effect on the annealing time. Decreasing the duration of annealing results in a strong reduction of training and in a slight reduction of j_c (see Table II).

The effect of final cold work on training is represented in Fig. 2b. The curves show that high degrees of cold work also diminish training. The significant point to note in these experiments is that metallurgical treatments which optimize j_c (see Table II) also result in enhanced training.

3.1.2. NbZr alloys

Training experiments were carried out on commercial NbZr superconductors with different Zr contents (see Table III). Among these alloys only the Nb–33 wt % Zr conductor showed training (Fig. 3). In all other samples no transition to the normal state was found, even at strains of 2.5% and currents of 98% j_c. The lack of metallurgical history data of the NbZr conductors investigated makes it difficult to discuss this behaviour. However, examination of Table III shows that all alloys, except for Nb–33 wt % Zr, have high j_c ($B = 0$) values. This proves that the microstructure which controls j_c differed in Nb–33 wt % Zr compared with the remaining samples.

It has been suggested that the monotectoid reaction in the NbZr system resulting in two b c c phases occurs only in the presence of oxygen or nitrogen interstitials [13]. In practice, no differences in the gas contents of the NbZr alloys (see Table IV), which could indicate different kinetics of the transformation processes were found. Also, transmission electron microscopy (TEM) studies gave no hint of the presence of precipitates, which agrees with a study by Santhanam [14] of Nb–25 wt % Zr with a comparable gas content.

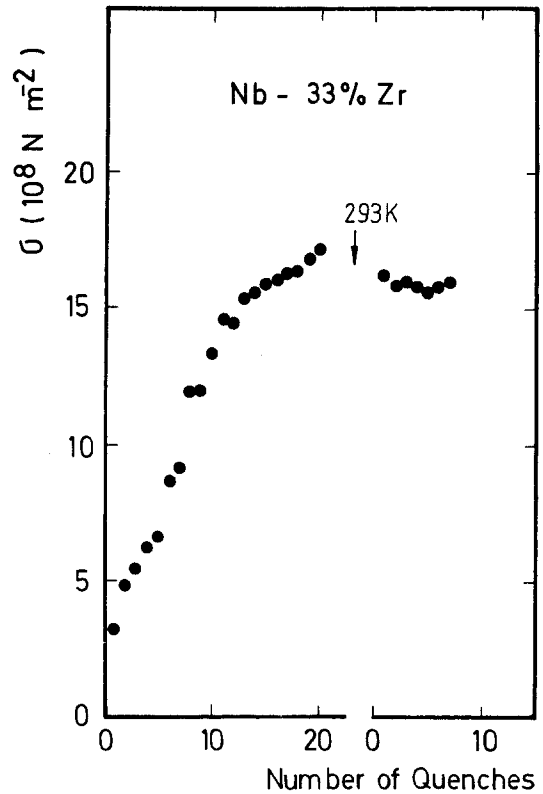

Figure 3 Short sample training of the Nb–33 wt % Zr, $j = 95\% j_c$. After some 20 steps training was interrupted, the sample warmed up to 293 K, cooled down and again examined.

For this reason it may be assumed that it is not precipitates, but the dislocation structure which plays the dominant role in current carrying capacity and in the stress effects.

3.2. Reactivation and inhibition of the source of premature quenches

3.2.1. Reactivation

To gain a better understanding of the process controlling the stress-induced energy release at 4.2 K, the effect of annealing on the reversibility of training was investigated. For this purpose, NbTi samples were trained in a second run after warming up to room temperature. The ratio of the number of training steps after and before the temperature cycle was used as a measure of the reversibility of training. Fig. 4 shows the results for the different NbTi compositions. While Nb–65 wt % Ti exhibits the same training behaviour as before the temperature cycle, the temperature increase to 293 K is not sufficient in alloys with 39–60 wt % Ti to completely reactivate the mechanism inducing training.

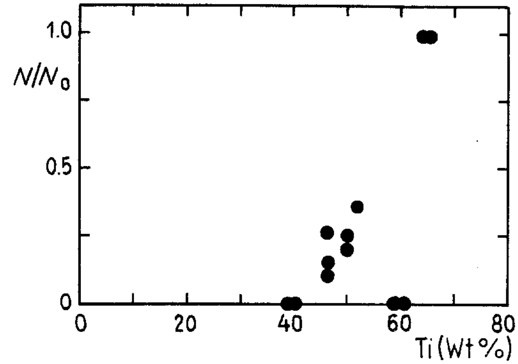

Figure 4 Reversibility of short sample training of different NbTi compositions after a temperature cycle to room temperature. N_0 and N are the number of training steps in the first run and after warming up to 293 K, respectively.

The reversibility of training in Nb–65 wt % Ti was interpreted as proof of a complete recovery of the mechanism of energy release, which was eliminated by straining at 4.2 K in the first run. The recovery temperature of the microstructure of this alloy is above 77 K, because heating to this temperature did not give rise to any reversibility of the training, as was found in another experiment. Naturally, the question arose whether or not a heat-treatment above 293 K would be able to achieve recovery of the microstructure in alloys with $\leqslant 60$ wt % Ti and, in this way, also attain a reversible training behaviour in these alloys. This was studied using Nb–46.5 wt % Ti (Fig. 5). After

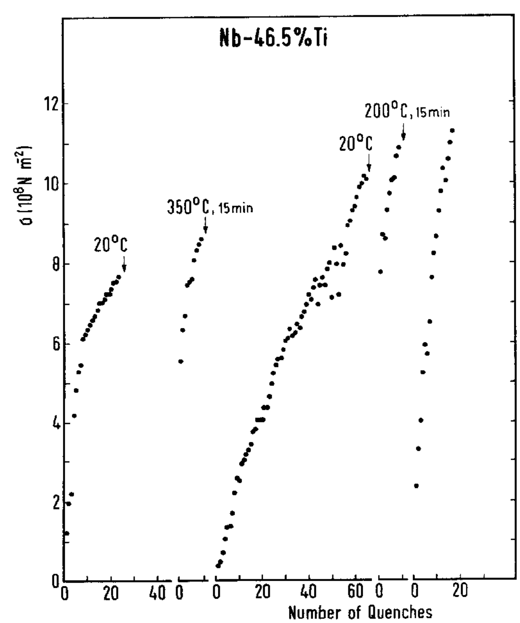

Figure 5 Reactivation of the source of normal transitions in Nb–46 wt % Ti as a function of annealing temperature. $j = 95\% j_c$.

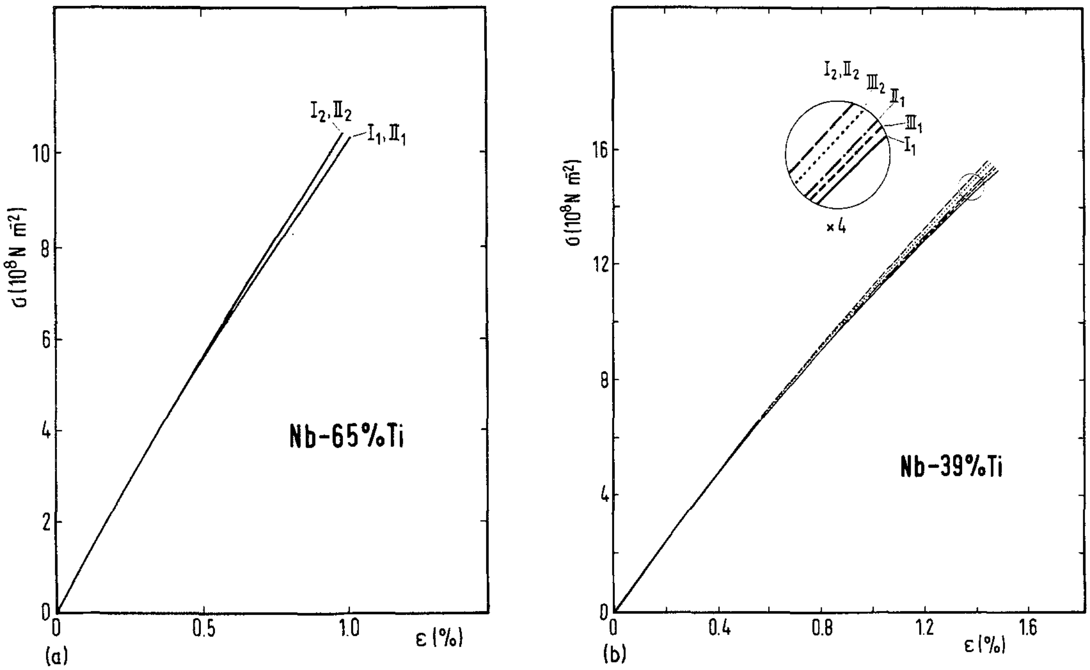

Figure 6 The first two (1,2) stress–strain curves of (a) Nb–65 wt % Ti and (b) Nb–39 wt % Ti at 4.2 K after a first (I) and second (II) cool down from room temperature (for example, II_1: first loading after the second cool down). The Nb–39 wt % Ti sample was also strained after a third cool down (III_1 and III_2).

some 20 steps, (Curve 1) training was interrupted and the sample was warmed up to room temperature. After cooling the sample exhibited the training irreversibility known from Fig. 4 (Curve 2). The training process was interrupted once more and the sample heated to 350° C for 15 min. The training curve (Curve 3) obtained after this treatment shows that not only complete reversibility was attained but that the energy source leading to training was even intensified. After some 60 training steps the sample was heated to 293 K, cooled down and examined again (Curve 4). The results further show (Curve 5) that the degree of recovery is very much a function of temperature.

The reversibility of training in NbTi alloys, as shown in Fig. 4, is also reflected in the stress–strain curves. This is shown in Fig. 6a and b, where the first two stress–strain curves before and after a temperature cycle to 293 K overlap considerably in the case of Nb–65 wt % Ti, unlike the Nb–39 wt % Ti sample, which exhibits irreversible training.

3.2.2. *Inhibition*

From the behaviour of trained NbTi superconductor after thermal cycling it might be expected that straining of a conductor at room temperature before the training experiment in alloys with ⩽ 60 wt % Ti may lead to a reduction in the mechanism initiating training, because the recovery temperature is above room temperature. This is in fact true; Nb–50 wt % Ti single core and multifilament conductors, which had been prestrained at 293 K, showed reduced training relative to non-strained conductors [4]. Accordingly, it is unimportant to suppress the energy source in these superconductors whether straining occurs at 4.2 or at 239 K. Superconducting magnet training can be reduced by suitable prestraining of the superconductor before winding [15, 16].

However, in NbTi compositions in which the mechanism inducing training recovers below 293 K, prestraining is not an effective means of preventing the occurrence of premature normal transitions. This was confirmed with a sample of Nb–65 wt % Ti: 1% straining at 293 K in four cycles did not produce any significant change in the training curve shown in Fig. 1.

3.3. Correlation of acoustic emission from NbTi superconductors with premature quenches

Acoustic emission under mechanical load in superconductors at 4.2 K has already been

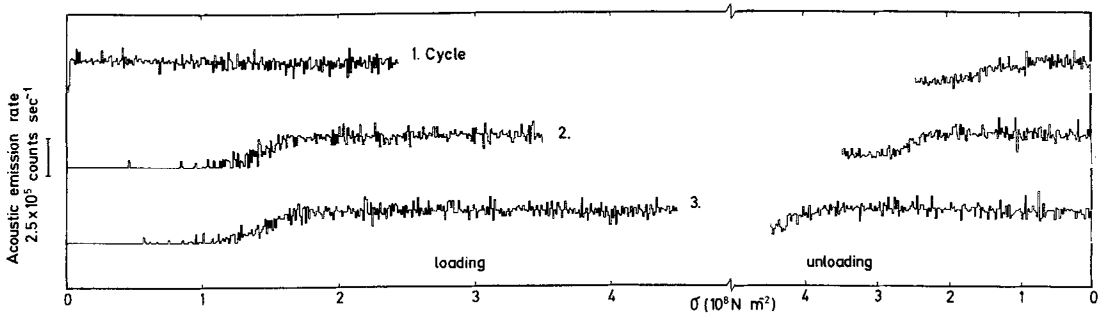

Figure 7 Acoustic emission rate from Nb–25 wt % Zr as a function of stress during three loading–unloading cycles at 4.2 K. Amplification 98 dB.

observed in earlier work [3, 8]. The emission of acoustic waves begins in NbTi immediately upon loading, but shows a stress-irreversible behaviour, i.e. emission always begins only after the previous maximum stress level has been exceeded. Above a certain load, however, there also appears a stress-reversible component during loading as well as unloading [4]. This anelastic behaviour appears also as a hysteresis in the stress–strain curve.

The situation with NbZr is qualitatively similar, but the stress-reversible component appears at lower stresses and is more pronounced than in NbTi. Fig. 7 shows the emission rate in Nb–25 wt % Zr for 3 strain cycles during load and unload. In the second and third cycle no emission occurs before the onset of the stress-reversible component at a stress of $10^8 \mathrm{N\,m^{-2}}$. The occurrence of a stress-irreversible acoustic activity in mechanically-stressed superconductors leads to the questions of whether or not there is a connection between the mechanism underlying these acoustic signals and the training of short samples and how could such correlation be proved experimentally. Let us begin with indirect indications, namely studies of the correlation between the reversibility of acoustic activity and of training (i) after room-temperature cycling and (ii) after prestraining the superconductor at room temperature before the experiment.

As shown by the experiments on NbTi alloys described in Section 3.2, reactivation of the mechanism initiating training by thermal cycling depends on the composition of the alloy. Therefore, the acoustic emission was measured before and after a room-temperature cycle and it was compared with the reversibility of training. The results of acoustic emission in Nb–65 wt % Ti and Nb–39 wt % Ti samples are shown in Fig. 8a and b. The plot gives the total number of oscillations during load and unload. This presentation allows a better quantitative comparison than plotting the counting rate. The decrease of acoustic emission in the case of Nb–65 wt % Ti after a thermal cycle does not correspond to the behaviour of the mechanism initiating training, which remains unchanged (reversible training). On the other hand some acoustic emission is still present in Nb–39 wt % Ti after thermal cycling, while training in this alloy is completely irreversible (see Fig. 4). The reduced acoustic emission after a room-temperature cycle was seen for all samples.

A qualitatively similar result was obtained in measurements of samples prestrained at room temperature. Fig. 9 shows the result for a Nb–50 wt % Ti sample. While prestraining completely suppresses training [4], the acoustic emission is only reduced by less than a factor of 2. It is seen again that the acoustic activity does not obey the same laws as the mechanism initiating premature normal transitions.

For a direct study of the correlation between acoustic emission pulses and premature normal transitions experiments were performed in which the emission was recorded together with the voltage trace at the moment of normal transition. In a number of experiments with increasingly better time resolutions no emission was found for times of the order of milliseconds before the normal transition, apart from occasional random pulses. At the time of the voltage rise there was strong emission in all cases, if amplification was sufficient, but it should be noted that emission as a consequence of normal transition must be expected to occur because of the sudden flux redistribution and heating of the sample. The statement that emission is due to the event initiating the normal transition is true only if emission occurs before the voltage rise. Here time of flight of the acoustic signal from the sample to the transducer must

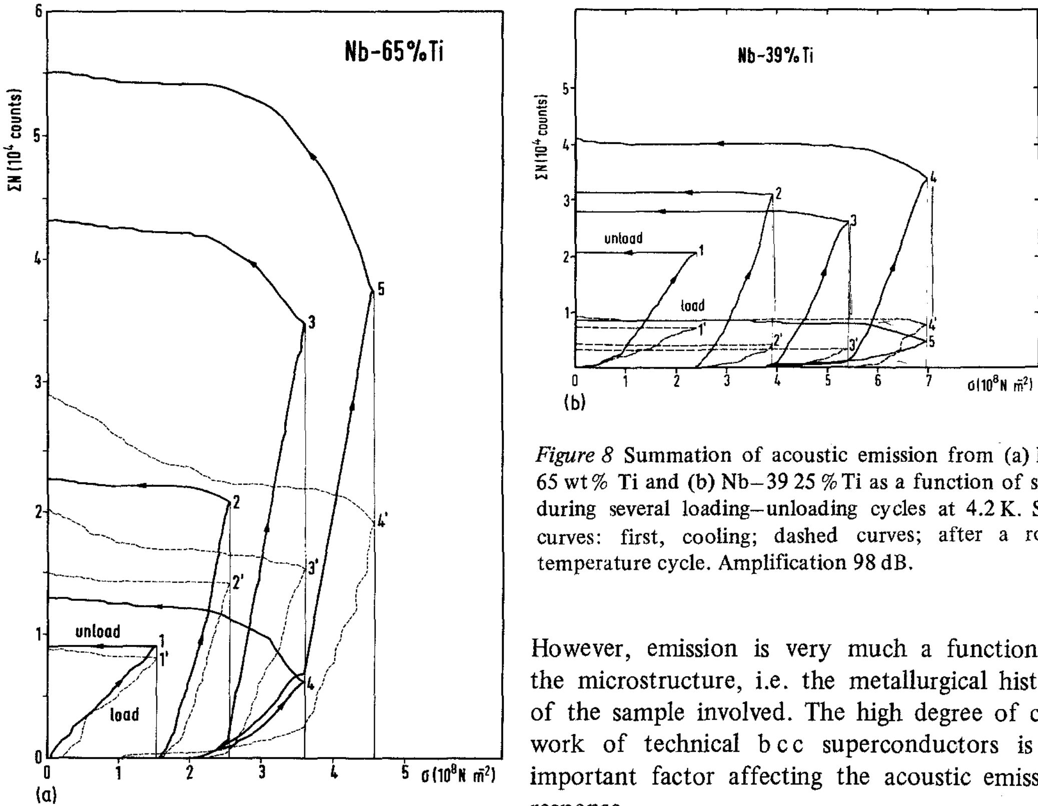

Figure 8 Summation of acoustic emission from (a) Nb–65 wt% Ti and (b) Nb–39 25 % Ti as a function of stress during several loading–unloading cycles at 4.2 K. Solid curves: first, cooling; dashed curves; after a room-temperature cycle. Amplification 98 dB.

also be taken into account; it is $t \sim 0.04$ m per (5000 m sec^{-1}) = 8 μsec. Fig. 10 shows the acoustic emission (signal at the output of the main amplifier) for three successive normal transitions. The zero point of the time scale is located at 8 μsec after the rise of the voltage signal, which is shown for the first normal transition; this takes into account the delay brought about by the time of flight of the acoustic signal. A small acoustic signal above the noise level is seen to occur a few microseconds before normal transition. Several microseconds following the quench a major acoustic emission starts, which must be regarded as a consequence of normal transition.

Thus, a correlation is found to exist between acoustic emission and premature normal transition, although training and emission respond differently to the same metallurgical treatment of the conductor, as seen previously. In Section 4 this apparent conflict will be discussed in more detail.

3.4. The source of acoustic emission in NbTi under strain at 4.2 K

The most frequent causes of acoustic emission in mechanically stressed metals are dislocation motion, twinning and phase transformations. However, emission is very much a function of the microstructure, i.e. the metallurgical history of the sample involved. The high degree of cold work of technical b c c superconductors is an important factor affecting the acoustic emission response.

Acoustic emission originating from dislocations involves an interaction between dislocations and obstacles in the metal. In the model used by Engle [17] the dislocations piling up at obstacles cause an increase in local strain energy. When the force exerted on the obstacle exceeds a given value, the dislocations piled up break away and the strain energy causes lattice vibrations which appear as acoustic signals.

However, a minimum free dislocation line-length is necessary for the detection of acoustic emission [18]. This lower limit is determined by the sensitivity of the sensor, i.e. the minimum displacement at the surface of sample that can be detected by a piezoelectric transducer. An estimate for aluminium with a dislocation density of 10^6 cm^{-2} indicated a minimum free dislocation length of $\sim 10\,\mu$m, which was in agreement with experiments [18]. Since the free dislocation line-length decreases with a degree of cold work, it seems reasonable to conclude that dislocation motion in technical superconductors with dislocation densities of $\sim 10^{12}$ cm^{-2} cannot lead to acoustic emission.

It therefore appears consistent to connect lattice shearing with acoustic emission, in agreement with earlier studies which indicated a stress-

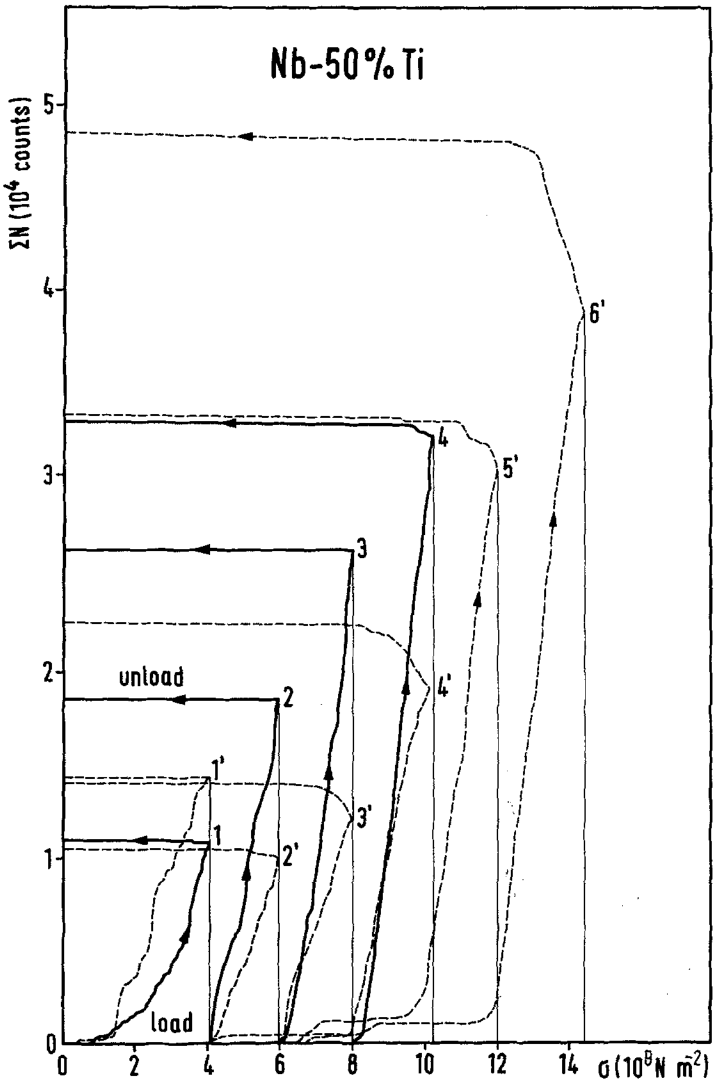

Figure 9 The effect of prestraining at 293 K on the summation of acoustic emssion from Nb–50 wt % Ti at 4.2 K. Solid curves: sample not prestrained; dashed curves: prestrained sample at room temperature with 1.2 GN m^{-2} in four cycles. Amplification 98 dB.

induced lattice transformation in NbTi at 4.2 K [3, 19]. Mainly twinning or martensite transformation have to be taken into account. Both are diffusionless processes involving shearing of the lattice planes; while martensite transformation generates a new crystal structure, twinning reproduces the existing lattice, but in a different orientation. These two possibilities will be discussed in the light of the acoustic emission results.

The formation of martensite by titanium-rich NbTi alloys is well documented [20–22]. Transformation could be induced by rapid cooling of Nb–65 wt % Ti from the β-field below the martensitic transformation temperature $M_S \sim 175°$ C, or by application of stress above M_S [23]; on heating, reverse transformation could be induced. The possibility of stress-induced growth of martensite at low temperatures in NbTi alloys with ~ 50 wt % Ti was recognized by Easton and Koch [24]. However, the M_S curve can no longer be valid for technical NbTi superconductors because the increased density of defects in the parent phase may increase the restraining energy on martensitic transformation [25]. This argument is consistent with the absence of acoustic emission from Nb–65 wt % Ti during strain at room temperature. Presumably, the high degree of initial cold work depresses the M_S temperature of this alloy. Consequently the temperature M_d below which deformation-induced martensite forms (usually M_d lies above M_S) will be also depressed. Since the microstructure influences the formation of the martensitic phase, the investigation of the acoustic activity of samples with different

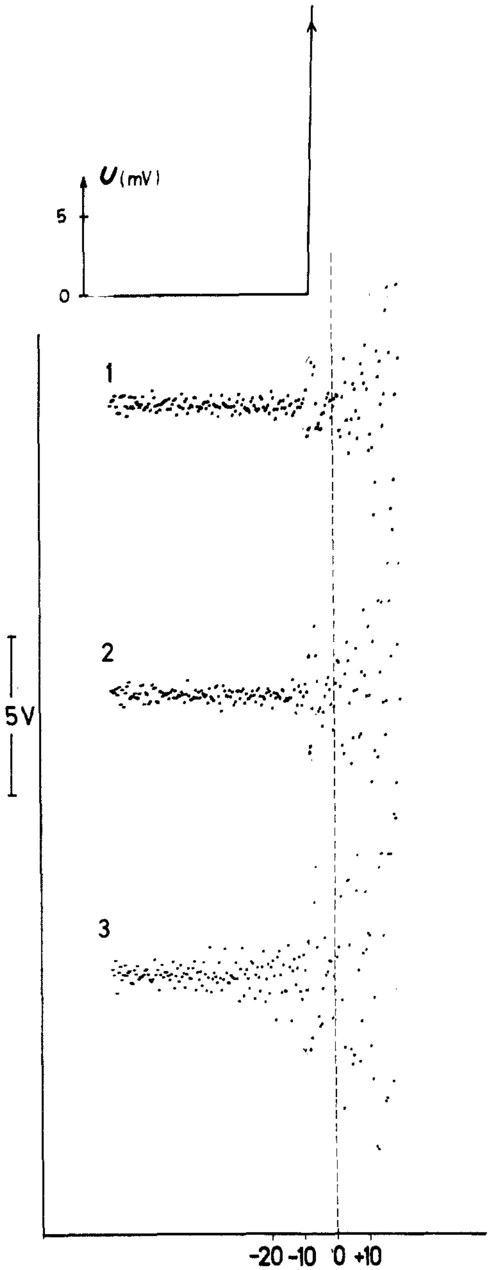

Figure 10 Acoustic emission pulses at the moment of normal transition for three successive quenches of a Nb–50 wt % Ti conductor. Amplification 90 dB.

metallurgical treatments may prove to be a way of distinguishing between martensite formation and twinning. The Nb–52 wt % Ti alloy was selected for this purpose. According to the phase diagram this alloy is able to form a second phase in addition to the b c c structure. The second phase can be either α-Ti precipitates or, if we consider the possibility of a stress-induced martensite transformation at 4.2 K, mentioned above, an orthorombic α''-martensite. Depending on the metallurgical treatment, the α- or the α''-phase dominates, but the sum of $\alpha + \alpha''$ must remain more or less constant. In other words, a sample in which the formation of α-Ti is avoided will be more susceptible to stress-induced formation of α''-martensite at 4.2 K than a sample of the same composition which had been subjected to precipitation annealing.

Fig. 11 shows the acoustic emission of two Nb–52 wt % Ti samples, one with and the other without α-Ti precipitates. In both cases the summation of acoustic emission is comparable. If martensite formation were the source of acoustic emission, the sample without α-Ti should exhibit a much higher emission, because the formation of martensite would be preferred.

The experiment thus suggests that twinning is the primary cause of acoustic emission in NbTi. However, it must be emphasized that it is impossible to distinguish between twinning and martensite formation from the shape of the signal of the emission; this is merely indirect proof. The generation of stress waves results in both cases from a sudden shearing of a volume of the crystal lattice, in which process the growth of the sheared region proceeds almost at sonic velocity.

It should be pointed out that twinning in NbTi correlates well with the behaviour of other martensitic alloys under stress (i.e. TiMo and FeBe). In both cases deformation-induced twinning was observed which recovered when the load was removed [26, 27]. As mentioned by these authors, the twinning system was not the $\{112\}\ \langle 111\rangle$ normally found in other b c c metals and alloys. Evidence for a partial reversion of twinning on subsequent heating was provided by experiments with NbZr alloys deformed at 77 K [28].

For martensite formation and twinning the more general term of "shear transformation" will be used. As shall be seen in the discussion, it is of secondary importance for the explanation of the training phenomenon whether lattice shearing occurs by martensite formation or twinning.

4. Discussion

The training effect in short sample superconductors has been investigated. Premature normal transitions during strain are caused by local release of energy (transformation of strain energy into

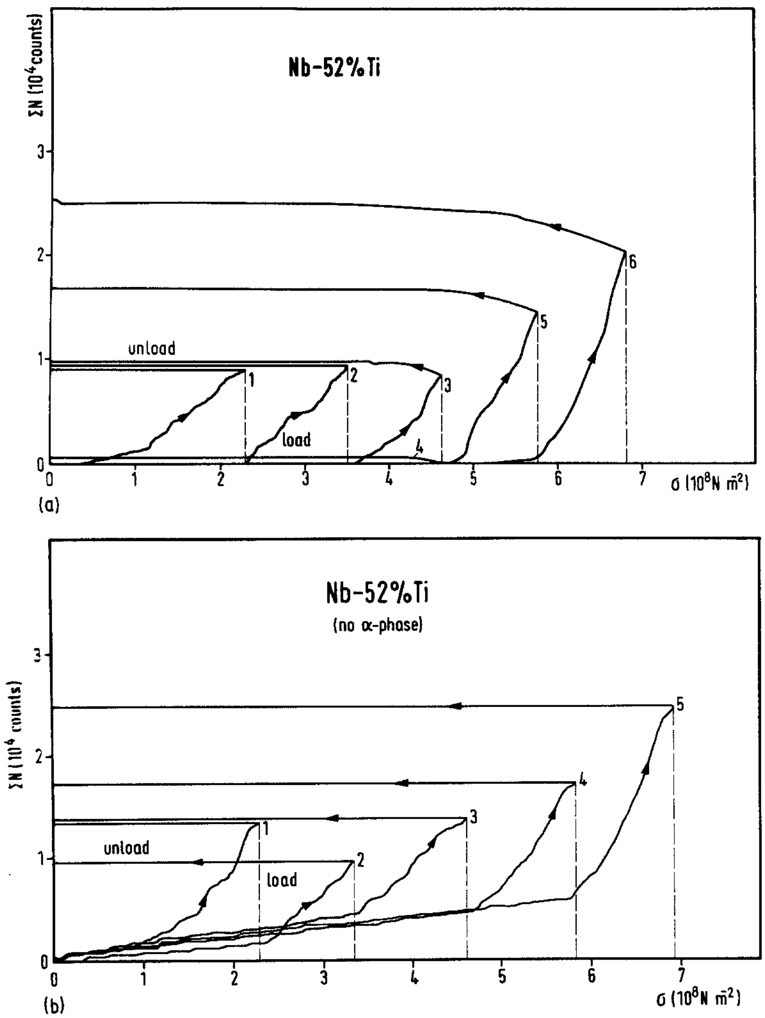

Figure 11 Summation of acoustic emission from Nb–52 wt % Ti samples as a function of stress during several loading–unloading cycles at 4.2 K (a) specimen subjected to precipitation heat treatment and (b) specimen only cold worked.

heat pulses), which occur at random over the length of the sample and which are stress-irreversible. That means in a subsequent straining cycle energy release occurs only after the previous maximum stress has been exceeded. The acoustic emission of the samples during strain was also studied and explained by shear transformation (twinning or martensite formation).

The training and acoustic emission for a number of NbTi and NbZr alloys after subjecting the samples to different metallurgical and thermal treatments were compared. The different response of the two effects on prestraining and on thermal cycling, as shown for the NbTi samples in Section 3.3, leads to the conclusion that training and acoustic emission are caused by two different mechanisms. In other words, training is not explained by the energy release of the shear transformation, which causes the acoustic activity.

This becomes even more evident looking at the results of the NbZr samples. All the alloys investigated (see Table III) lead to reversible acoustic emission similar to that shown in Fig. 7. On the other hand, premature normal transitions during strain could be induced only in the Nb–33 wt % Zr sample (see Fig. 3). Since twinning is the major mode of deformation in NbZr alloys at low temperatures [28], it is reasonable to associate the acoustic activity of these alloys at 4.2 K with this mechanism.

No training was observed in the Nb–25 wt % Zr and Nb–50 wt % Zr samples, which proves that twinning does not furnish sufficient energy to induce a normal transition. Anyway, training could not be explained by the mainly reversible acoustic emission of NbZr, since a stress-irreversible process is required, which transforms strain energy into heat pulses.

On the other hand, a time correlation was found between acoustic emission and the onset of normal transition (see Fig. 10). Therefore it must be assumed that a connection exists between the shear transformation and another mechanism involved. Recent experiments with the Nb–60 wt % Ti samples suggest that this second mechanism is dislocation microyielding [29].* Although dislocation microyielding does not constitute a detectable source of acoustic emission, this, together with shearing, supplies the necessary energy for transition into the normal state.

Therefore a model which accounts for the observed correlation between acoustic emission and training, despite their different origin, is proposed. The acoustic emission results from shear transformation (probably twinning) which forms in a volume of macroscopic size. The growth of the sheared region occurs with nearly sonic velocity. It leads, within a fraction of a microsecond, to a relaxation of the material since the overall strain of the sample cannot follow the rapid local deformation because of its mass inertia. The stress relaxation manifests itself in a stress wave which is recorded with the acoustic sensor, but it does not liberate enough thermal energy to induce a normal transition. However, the relaxation of the sheared region leads to an increased load on the rest of the sample cross-section, since the total load on the sample remains unchanged. In this way the yield stress of the unsheared region may be exceeded locally and microyielding occurs†. These microyield drops are too small to be detected on the stress–strain curve but dissipate a certain amount of strain energy as local heat pulses. When the sheared region exceeds a critical size, the amount of heat generated by microyielding is sufficient to induce a normal transition. This energy was calculated to be of the order of 10^{-7} J for samples investigated in this work [4].

In this model the shear transformation induces a normal transition indirectly, without itself supplying the necessary energy. Thus the model explains why thermal cycling and prestraining affects training and acoustic emission differently. In the case of partly reversible acoustic emission after thermal cycling, training can be irreversible if microyielding is suppressed by work hardening. In the same way training can be suppressed for some alloys by prestraining at room temperature, while acoustic emission is only slightly reduced: the shear transformation seems not to be very sensitive to prestrain but is unable to induce microyield drops in the strain work-hardened alloy.

The reduced training of cold-worked NbTi alloys is in agreement with the model described. Subjecting alloys to a heat treatment permits recovery of the dislocation structure and thus enhances training. The degree of recovery seems to be very sensitive to the annealing time, as shown in the experiments of Fig. 2a. The proposed model can also account for the effect of final cold work on training. The reduced training in alloys with a higher degree of cold work can be attributed to hardening effects.

It is difficult to state whether or not there is a correlation between the expected increased tendency to lattice instability in titanium-rich alloys and the enhanced training of Nb–65 wt % Ti. This is because the mechanism responsible for training is not identical to the lattice shearing, as shown in Section 3.3.

An open question is the low recovery temperature of the dislocation microstructure in Nb–65 wt % which is below room temperature (reversible training). It may be argued that the heavily cold-drawn condition and the low deformation temperature are of significance. Further investigations will be required to clarify the role of alloy composition and metallurgical history with respect to the recovery process of the source of premature quenches.

Acknowledgements
The authors wish to thank D. S. Easton, D. C. Larbalestier, A. D. McInturff, H. Hillmann and R. K. Williams for supplying the wire samples.

References
1. D. EVANS, Rutherford Laboratory Report RL-73-092 (1973).
2. J. R. HEIM, Fermi National Accelerator Laboratory Report, No. TM-334-B (1974).

*In these experiments it was shown that the chemical removal of a 20 to 40 μm thick surface layer of the sample after a training run led to a reactivation of the quench inducing mechanism. This was explained by the disappearance of the piled-up dislocations in this surface layer which act as obstacles to further dislocation movement.

†It should be mentioned that in the case of martensite transformation, there is another possible source of microyielding. The growth of the martensite phase leads to a dilatation accompanying the pure shear, which may cause the yield stress of the parent phase to be exceeded locally.

3. D. S. EASTON and C. C. KOCH, in "Shape Memory Effects in Alloys", edited by Jeff Perkins (Plenum Press, New York, 1975) p. 431.
4. G. PASZTOR and C. SCHMIDT, *J. Appl. Phys.* **49** (1978) 886.
5. J. W. EKIN, R. F. FICKETT and A. F. CLARK, *Adv. Cryog. Eng.* **22** (1977) 449.
6. J. W. EKIN and A. F. CLARK, Joint MMM – Intermag Conference on Magnetism and Magnetic Materials – 1976, AIP Conference Proceedings No. 34 (American Institute of Physics, New York, 1976) p. 81.
7. J. W. EKIN, *IEEE Trans. Magn.* **MAG-13** (1977) 127.
8. C. SCHMIDT and G. PASZTOR, *ibid.* **MAG-13** (1977) 116.
9. *Idem, Appl. Phys. Lett.* **28** (1976) 463.
10. *Idem, Rev. Sci. Instrum.* **48** (1977) 597.
11. U. ZWICKER, *Metall.* **18** (1964) 941.
12. A. D. McINTURFF and G. G. CHASE, *J. Appl. Phys.* **44** (1973) 2378.
13. C. W. BERGHOUT, *Phys. Letts* **1** (1962) 292.
14. A. T. SANTHANAM, D. M. MOON, M. P. MATHUR, M. ASHKIN and D. W. DEIS, *J. Appl. Phys.* **46** (1975) 4951.
15. C. SCHMIDT and B. TURCK, *Cryogenics* **17** (1977) 695.
16. C. H. DUSTMANN and H. KÖFLER, *ibid.* **17** (1977) 667.
17. R. B. ENGLE, PhD Thesis, Michigan University, Ann Arbor (1966).
18. A. B. L. AGARWAL, J. R. FREDERICK and D. K. FELBECK, *Met. Trans.* **1** (1970) 1069.
19. D. T. READ, *Cryogenics* **18** (1978) 579.
20. L. W. BERGER, D. N. WILLIAMS and R. I. JAFFEE, *Trans. ASM* **50** (1958) 384.
21. B. A. HATT and V. G. RIVLIN, *Brit. J. Appl. Phys. (J. Phys. D)* **1** (1968) 1145.
22. B. OBST, D. PATTANAYAK and P. HOCHSTUHL, *J. Low Temp. Phys.* **41** (1980) 595.
23. C. BAKER, *Met. Sci. J.* **5** (1971) 92.
24. D. S. EASTON and C. C. KOCH, *Adv. Cryog. Eng.* **22** (1977) 453.
25. R. P. REED and J. F. BREEDIS, *ASTM STP* **387** (1966) 60.
26. M. J. BLACKBURN and J. A. FEENEY, *J. Inst. Met.* **99** (1971) 132.
27. R. H. RICHMAN, "Deformation Twinning", (Gordon and Breach, New York, 1964) p. 237.
28. P. E. FLEWITT and D. E. B. PALMER, *J. Less-Common Met.* **32** (1973) 57.
29. G. PASZTOR, KfK-Report Nr. 2879, Kernforschungszentrum Karlsruhe (1979).

Received 19 November 1980 and accepted 26 January 1981.