Texture development in long lengths of NiFe tapes for superconducting coated conductor

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A highly oriented cubic texture (full width at half maximum $<10°$) has been formed in long length NiFe tapes. The X-ray diffraction (XRD), electron back-scattered patterns (EBSP) and optical microscopy (OM) techniques have been used in assessing the surface and volume texture and also the surface morphology of these kilometer long NiFe substrates. This texture was formed under a range of conditions including dynamic annealing in a reduced atmosphere and static annealing in hydrogen and in vacuum. Heat treatment for excessive times in vacuum tends to roughen the surface and should be avoided. Mechanical polishing can induce an additional undesirable texture, but electropolishing gives smooth tapes with good texture.

1. Introduction

For more than 15 years thin films of yttrium barium copper oxide, $YBa_2Cu_3O_7$, (YBCO) have demonstrated promising superconducting properties for use at liquid nitrogen temperatures with critical current density, J_c, (at 77 K) higher than 10^6 A/cm^2 and high irreversibility field, H_{irr}(77 K). Recent progress in the development of biaxially oriented epitaxial buffer layers [1, 2] on biaxially oriented flexible metallic substrates [3] has opened up possibilities for the application of YBCO in electric utility and high magnetic field dc devices such as MRI and NMR magnets. While naturally occurring defects in YBCO are capable of providing high critical current J_c values, weak links such as high angle grain boundaries (GBs) can considerably affect the overall critical current [4–6].

Several different types of deposition techniques such as Pulse Laser Deposition, Sputtering, CVD, MOCVD, Thermal Evaporation, Sol-Gel, and Liquid Phase Epitaxy have been used to manufacture short pieces of the high temperature superconducting tapes on a laboratory scale [7, 8]. There is a range of Ni-based binary and ternary alloys that can be used as a substrate. Some of them are magnetic for example Ni [3], NiFe [5] and some of them are non-magnetic for example NiCu, NiV, NiCr, NiW [9–12] depending on the alloy composition, and of course, the temperature of the application envisaged. As we know NiFe and also Ni (used extensively by Oak Ridge National Lab.) is a ferromagnetic material and may not be the best for ac applications. However the recently patented method of decoupling filaments by using magnetic-superconducting heterostructures may make some ac applications economically justified [13]. For the major magnet manufactures such as Oxford Instruments plc, since their applications are DC type, the magnetism of the metallic substrates is not a critical

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issue. A more important issue is the NiFe tapes have superior mechanical and structural properties compared with pure Ni substrates.

However, the current challenge is to find an economic route for large-scale production of the conductors including highly textured mechanically strong and thin metallic substrates in the form of a tape. This challenge is being addressed in a multi-partner Brite Euram industrial project called **Mu**ltifunctional Flexible High Temperature **S**uperconducting **T**ape (MUST) [14]. The design of the final conductor under consideration consists of: 1) long lengths of well-oriented NiFe substrate, 2) yttria stabilised zirconia (YSZ) as an epitaxial buffer layer grown epitaxially on the metallic substrate, 3) yttrium barium copper oxide (YBCO) as the superconductor grown epitaxially on the buffer layer, 4) silver as a cryogenic and electric stabilisation layer and finally 5) polymer as the top layer for insulation and stress relief. This architecture is schematically presented in Fig. 1.

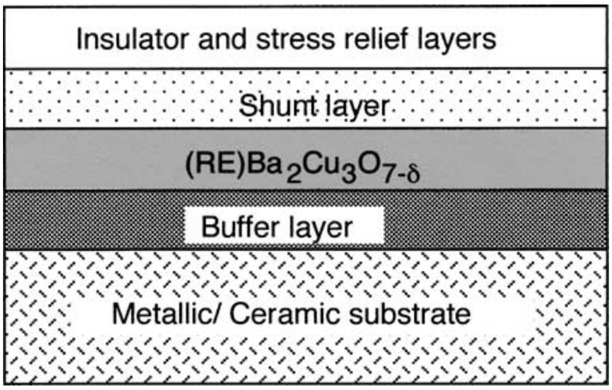


Figure 1 Schematic cross section of a fully engineered (RE)-123 coated conductor tape. Such a tape is typically 10 mm wide and is required to have a uniform high critical current over long lengths. The key conductor components are the metallic or ceramic substrate material, the buffer layer and the superconducting layer; the latter has to be biaxially textured throughout so that, although granular, the misorientation from grain to grain is less than a few degrees.

The substrate must be thin because a very important consideration in most applications is a high value of engineering critical current density, J_{eng}, defined as a critical current, I_{c}, carried by the superconducting layer divided by the total cross section of the conductor which includes the substrate and all the functional layers. A low value of the overall critical current density J_{eng} will reduce the exploitation potential of fully engineered conductors. The thickness of non-superconducting layers and any deterioration of J_{c} in the superconductor with increasing thickness of the superconducting layer, t_{sup}, leads to a maximum in the dependence of J_{eng} with t_{sup} which may occur at the higher t_{sup} than usually envisaged [6]. If the $J_{\mathrm{c}}(t_{sup})$ dependence on the t_{sup} is expressed as $J_{\mathrm{c}}(t_{sup}) = J_{\mathrm{c}}(t_{sup} - 0)\exp[-Ct_{sup}]$, where C is an empirical fitting parameter, then the dependence of J_{eng} on the thickness of the superconducting layer may be expressed as:

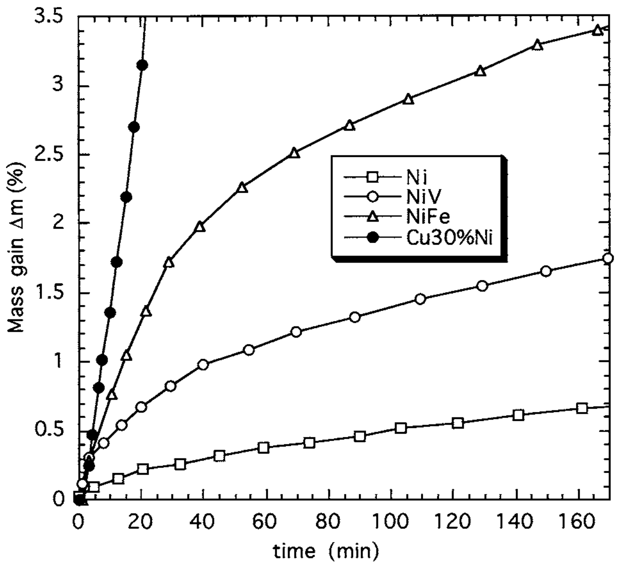


Figure 3 Thermogravimetric data representing oxidation of the different Ni-alloys developed in frame of European collaboration; The axis represent time during which the samples were exposed to oxygen at constant temperature 700°C.

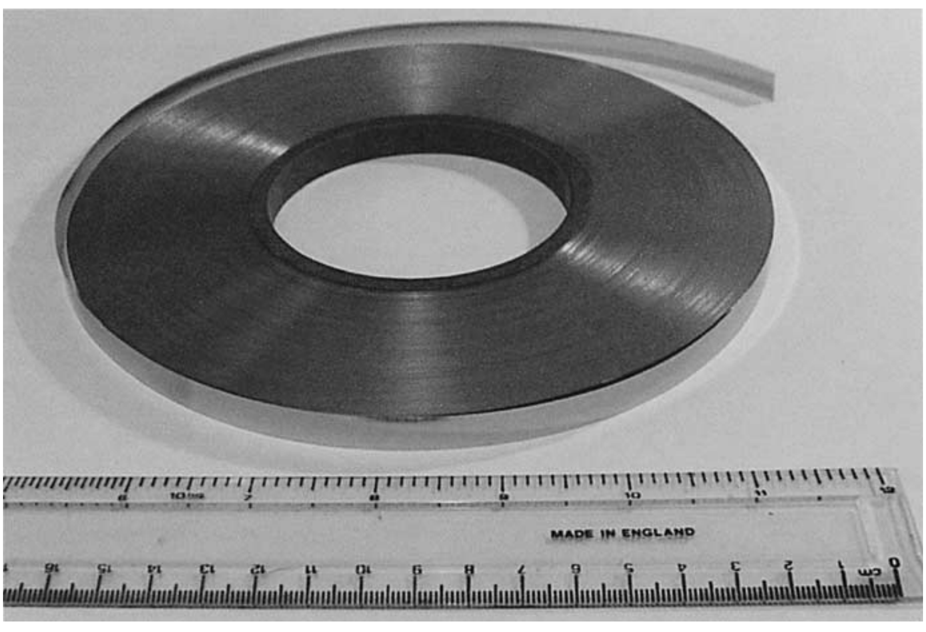


Figure 2 View of the NiFe tape 760 m long, 10 mm wide and 25 μm thick.

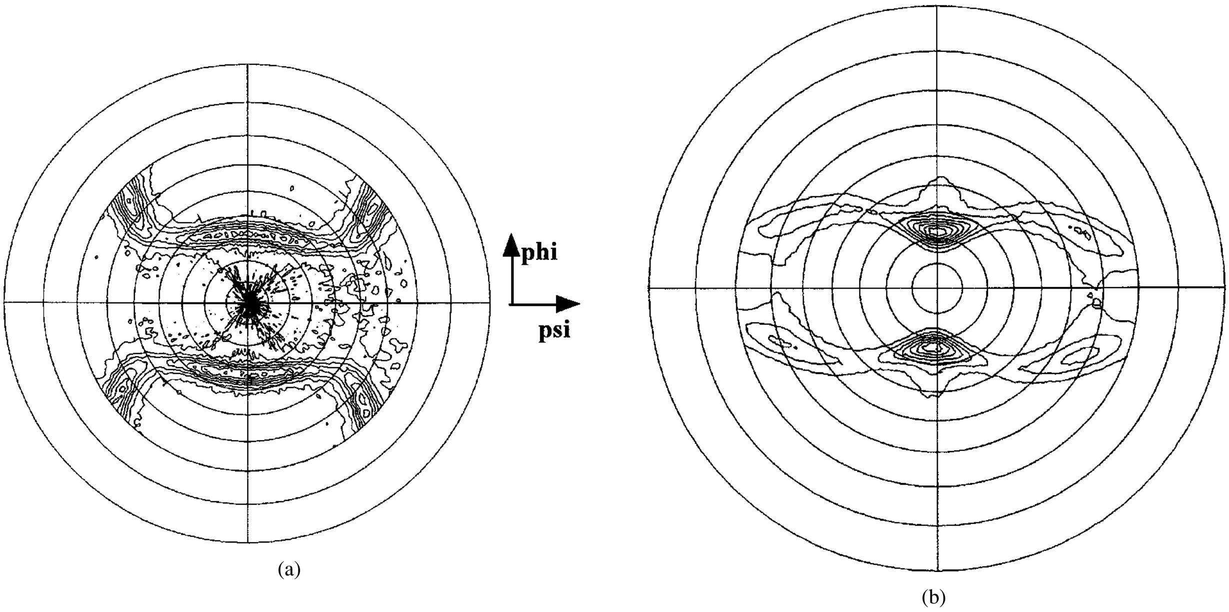


Figure 4 X-ray pole figures of cold rolled NiFe tape, 25 μm thick: a) (200) (linear scale $I_{\max} = 914$) and b) (111) (linear scale $I_{\max} = 3361$). All X-ray pole figures have roll direction vertical.

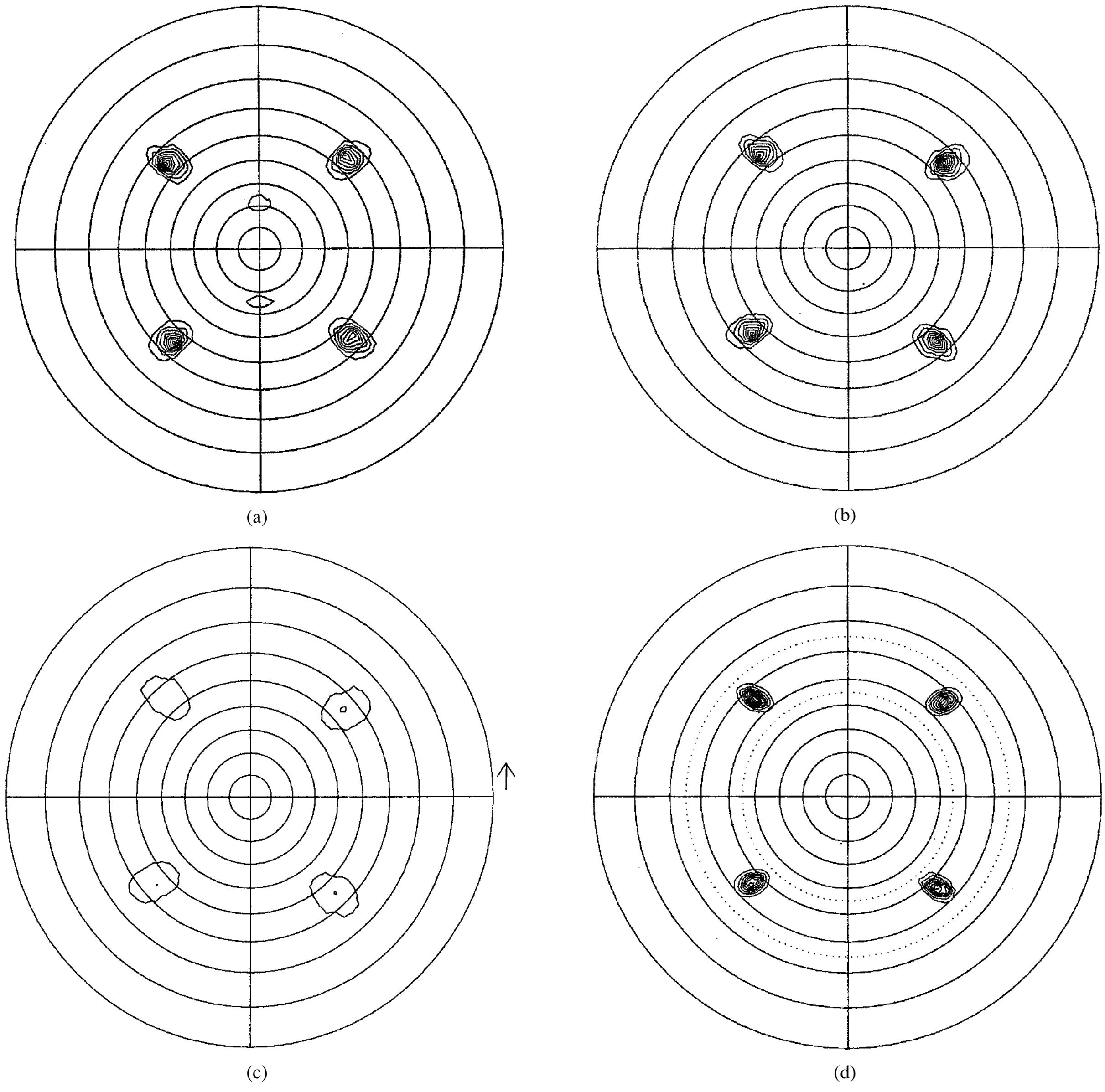


Figure 5 (111) X-ray pole figures of the polycrystalline NiFe tape, 25 μm thick, annealed at: a) 500°C b) 800°C (linear scale), c) 800°C (log scale) and d) 1050°C (linear scale); (linear scale $I_{\max}$ are 4062, 7502 and 7628 respectively); (log scale $I_{\max} = 7000$).

$$J_{\text{eng}}(t_{sup}) = J_{\text{c}}(t_{sup})\{t_{sup}/[t_{sub} + t_{buf} + t_{sup} + t_{stab} + t_{ins}]\}$$

where t_{sub}, t_{buf}, t_{stab}, t_{ins} are thicknesses of the substrate, buffer layer, stabilising (shunt) layer and insulating layer respectively. Taking as an example the 1 μm thick superconducting coatings characterised by $J_{\text{c}}(t_{sup}) \sim 10^6$ A cm^{-2}, but deposited on the 100 μm thick Ni-based substrates, this will not differ, in respect of overall critical current density from the ordinary powder-in-tube (PIT) conductor which has $J_{\text{eng}}(t_{sup}) \sim 10^4$ A cm^{-2}. Therefore for the complex coated conductor technique to gain a significant advantage, one has to use the thinnest possible substrates but simultaneously preserve their mechanical strength. The essential condition for development of biaxially oriented epitaxial buffer layers and superconducting layers on flexible metallic substrates is the degree of texture in the metallic substrate itself. The so-called cube texture, with crystallographic axes parallel to the sample axes, forms an ideal textured metallic substrate. This can be obtained in most FCC metals by cold rolling to a reduction of greater than about 95% followed by heat treatment [15–17]. Cold rolling gives a characteristic, but quite complex, texture described as (110)[112], (112)[111], (123)[422] and (146)[211]. (An ideal texture has (hkl) planes parallel to the sheet and the

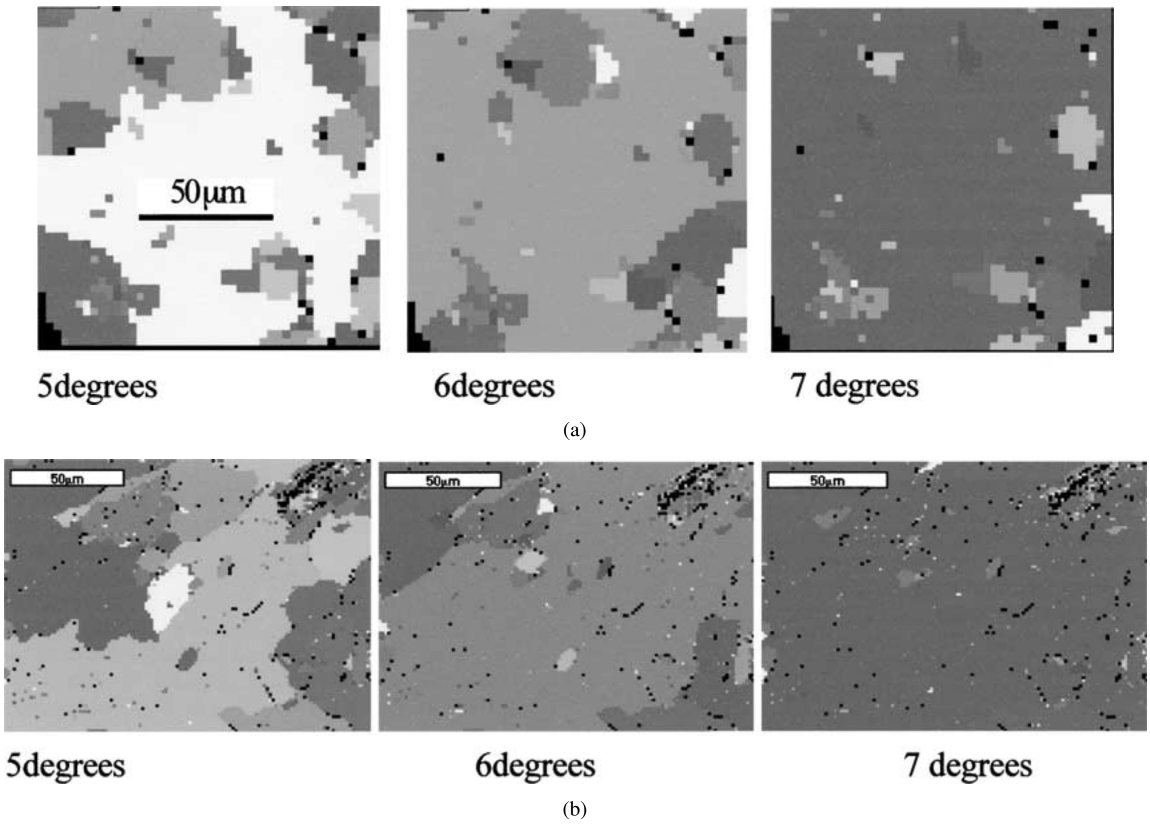


Figure 6 EBSP data of the NiFe tape, 25 μm thick, after treatment at 1050°C for a) 2 hours, b) 48 hours. Misorientation angles are marked correspondingly on the figures.

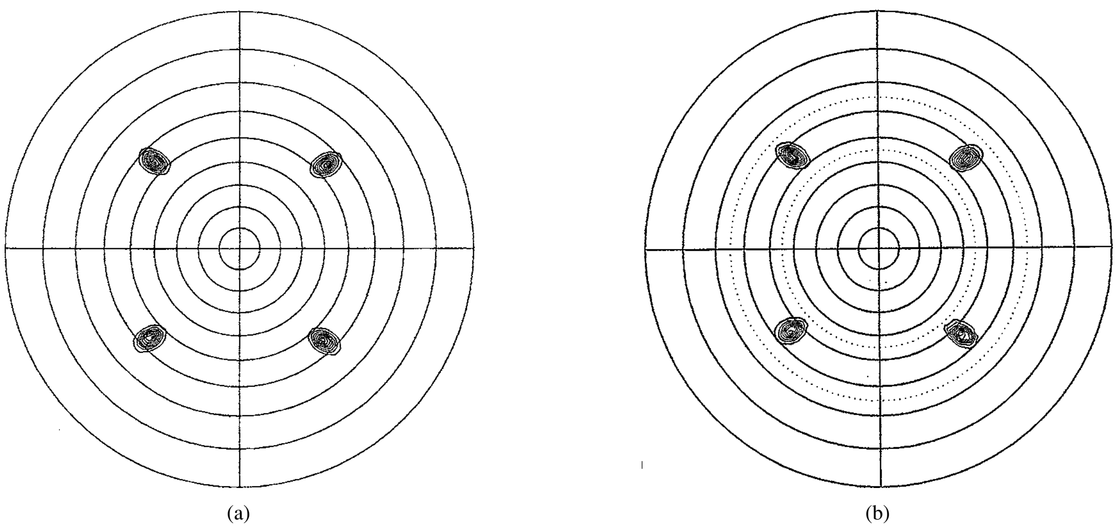


Figure 7 (111) X-ray pole figures for NiFe tape, 25 μm thick, after treatment at 1050°C for a) 2 hours, (linear scale $I_{\max} = 6085$); b) 48 hours, (linear scale $I_{\max} = 7628$).

vector [uvw] parallel to the rolling direction.) Some of the work of deformation is stored as defects and this energy provides the driving force for recovery and recrystallisation [17, 18]. The heat treatment required to give the optimum highly oriented cube texture is likely to depend on the grain size and the level of impurities. In the current publication we will try to draw attention to the preparation and characterisation of the surface and volume texture and also surface morphology of NiFe substrates using different characterisation techniques such as X-ray diffraction (XRD), electron back-scattered patterns (EBSP) and also optical microscopy (OM).

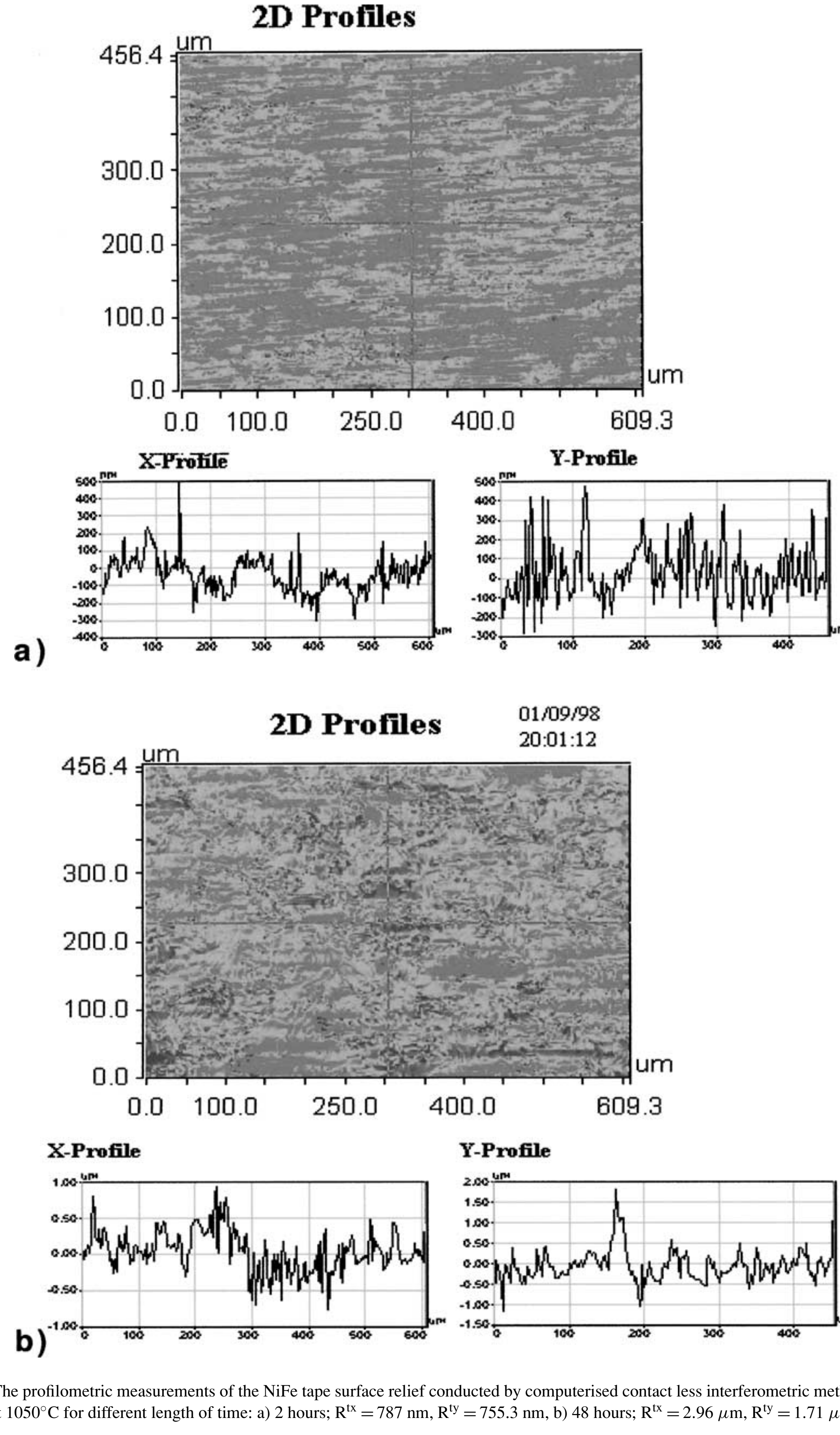


Figure 8 The profilometric measurements of the NiFe tape surface relief conducted by computerised contact less interferometric method on the tape annealed at 1050°C for different length of time: a) 2 hours; $R^{tx} = 787$ nm, $R^{ty} = 755.3$ nm, b) 48 hours; $R^{tx} = 2.96$ μm, $R^{ty} = 1.71$ μm.

2. Experimental conditions for manufacture and texture development of NiFe tapes

A range of the Ni-based materials from the Cu-Fe-Ni, Cr-Ni-Fe ternary phase diagrams, were considered for the manufacture of the long pieces of the metallic substrates for the Brite Euram MUST project. Among the available Ni and Ni binary alloys suitable for cube texture development are NiFe [5], NiV [10, 11], NiCr [10, 11], NiCu [9, 11], NiW [12]. The only material potentially scalable for the YBaCuO long lengths coated conductor development for our project was NiFe, since the remaining alloys materials were processed as a 100 μm thick, and relatively short experimental tapes. The current development of Ni-based alloys is continually progressing delivering longer and thinner tapes. The thickness of the tape and length required for the reel-to-reel unit under design was 25 μm thick, 25 mm wide and 25 m long (minimum) piece (the distance between the reels was 15 m) [19]. The width 25 mm was chosen to increase the production speed by slitting the finally deposited conductor to narrower tapes for winding future magnets. For this purpose the NiFe50% tape was cast in the form of 2 tonnes ingot and deformed and cold-rolled to the 3–5 mm thin stripes, followed by the final rolling to thicknesses of 50 μm, 25 μm and 13 μm and a width of 10 cm. Each final roll

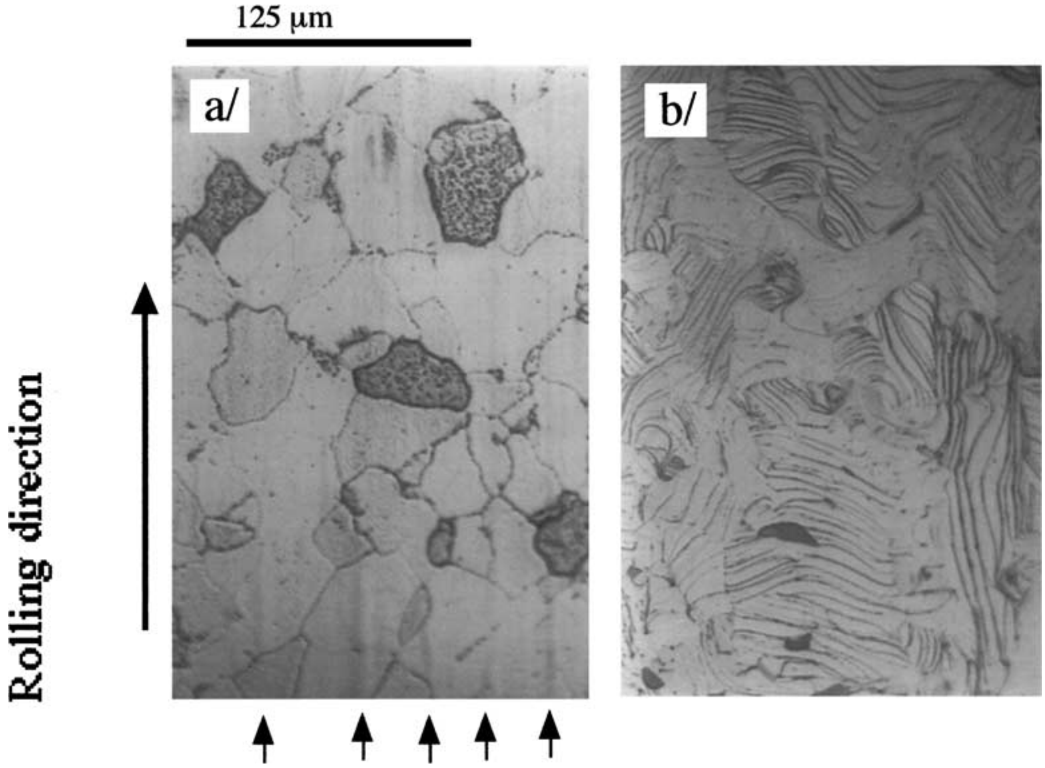


Figure 9 Optical micrographs of surface of the NiFe tape, 25 μm thick, annealed for different length of time in vacuum at 1050°C: a) 2 hours, b) 48 hours. Arrows represent the macro-modulations induced by rollers.

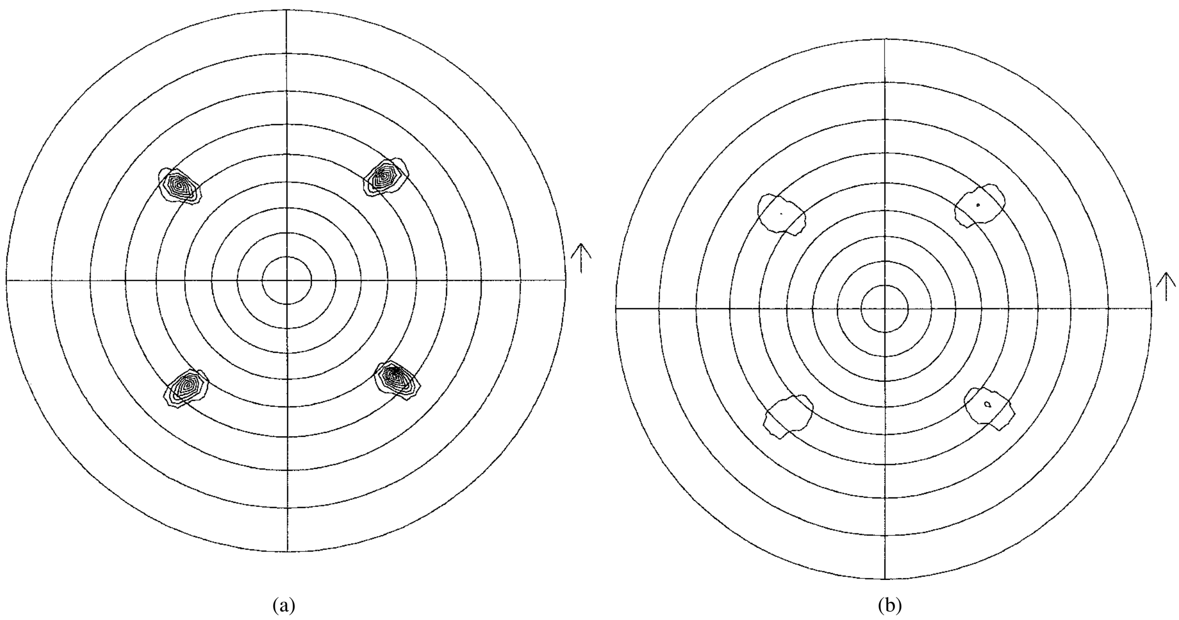


Figure 10 (111) X-ray pole figures of the polycrystalline NiFe tape, 25 μm thick, dynamically annealed a) (linear scale, $I_{max} = 20706$), b) (log scale $I_{max} = 20000$).

weighed ~20 kg and was approximately 1 km long. The deformation process was conducted in a factory environment by Carpenter Technologies (UK). The final tape was slitted to 25 mm and 10 mm wide tapes for our research development, see Fig. 2.

Due to the relatively high susceptibility of NiFe to oxygen see Fig. 3, [20] recrystallisation of the tapes was conducted statically under an atmosphere of hydrogen, and vacuum at temperatures range 600°C–1150°C for variable time ranging from 2–48 h, where dynamic annealing was conducted in an atmosphere of hydrogen and nitrogen gas mixtures at elevated temperatures 350°C–750°C for 15 minutes. The dynamic heat treatment of the ~1 km long pieces was carried out at optimised temperature, processing speed and reducing H2/N2 atmosphere. The optimisation of dynamic annealing was conducted in a factory environment to provide a reliable source of long highly textured NiFe substrate material for our industrially oriented rotating target continuous high rate sputtering deposition unit developed in the context of the project [19].

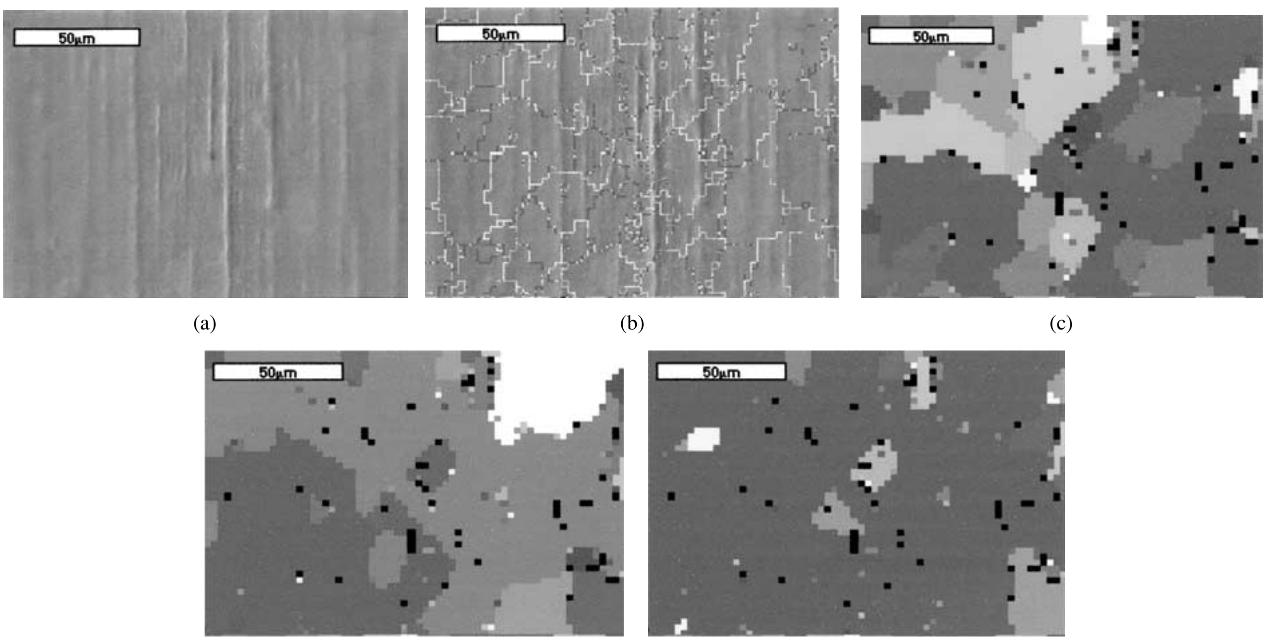
Surface polishing was carried out in Cambridge by mechanical polishing of as-rolled tapes before recrystallisation, whereas Europa Metalli conducted the electropolishing mainly on the dynamically annealed 25 mm wide tapes in an experimental unit which was purpose build.

X-ray pole figures were collected in a standard Schultz texture cradle with Cu K_α radiation out to the limit of $psi = 70°$. The sample was oscillated and thus the area sampled was about 6 × 10 mm to a depth of about one micron. (Both the area and the depth are somewhat dependent on the angle of incidence). Electron back-scattered patterns (EBSP) were obtained and analysed with the Oxford Instruments OPAL hardware and software [21]. In this technique the pattern from each sample point contains the information to reconstruct full pole figures, and also inverse pole figures. Various maps are also produced using this technique. One representation distinguishes one grain from another by colouring the grains differently if they are misoriented from their immediate neighbours by more than a user-defined angle. Another representation shows the grain boundaries in different colours depending on the ranges of misorientation angles defined by the user. Pole figures and inverse pole figures can also be constructed from the EBS data. The size of the electron probe must be less than the grain size: a larger probe improves counting statistics or reduces counting time but if the probe samples more than one grain at a time the pattern cannot be analysed. Many points have to be collected to give a representative sample. The area sampled was typically ~200 μm × 300 μm. This is a surface sensitive technique with a typical sampling depth of ~70 nm. In addition, the surface morphology was investigated by optical microscopy and non-contact surface interferometery.

3. Results and discussion

3.1. Recrystallisation

Fig. 4 shows (200) and (111) pole figures of the cold rolled NiFe alloy with thickness 25 μm. The complex texture is consistent with data in the literature [15–18, 22] and is described as a mixture of (110)[112], (112)[111], (123)[422] and (146)[211]. The effect of heat treatment, for 2 hours at increasing temperatures, on the texture of 25 μm thick tape is shown in Fig. 5. After treatment at 500°C for 2 h, the bulk of the material had a sharp cubic texture ((111) max at $psi = 55$ and $phi = 45°$) but some cold rolled texture was still present, with the intensity of cold rolled peaks ~15% of cubic texture peaks. The cold rolled texture almost



(d) (e)

Figure 11 The NiFe 25 μm thick tape dynamically annealed a) SEM picture of the tape surface fragment, b) SEM picture of the tape surface fragment with superimposed combined EBSP misorientation map presented in the following pictures c) misorientation EBSP map for 4 degrees, d) misorientation EBSP map for 5 degrees e) misorientation EBSP map for 6 degrees; most of the grains are less than 6 degrees misaligned from normal to the surface of the substrate. *Colours used for "decoration" of the individual grains or crystallites are arbitrary for each individual picture to emphasise the boundary of the given degree of misalignment.*

disappeared after treatment at 800°C (peaks ~1% of cubic ones) and above this temperature the cold rolled texture was not observable. No major changes were observed after heating at higher temperatures. Between 500° and 1050°C the out-of-plane orientation, *psi*, remained the same with a full width at half maximum (FWHM) of about 8°, but there was a small decrease in the in-plane orientation, *phi*, from 9° to 7°. Fig. 5c was typical of the X-ray pole figures obtained for these tapes under a wide variety of conditions where the annealing temperature was greater than 800°C.

In particular, the X-ray pole figures of heat-treated samples 13 μm, 25 μm and 50 μm thick were identical. As the final overall critical current of the conductor is influenced mainly by the thickness of the metallic substrates, the 13 μm substrate should be used for deposition of the buffer and superconductor if there are no other mechanical limitations. However, it appeared during our extensive experiments in a modular sputtering system [19] that from the mechanical point of view it would be better to use 25 μm thick tape. However in future if there is a need for further improvement of the overall current density value of coated conductors, 13 μm thick tapes can be used. Optical microscopy conducted on both sides of 25 μm thick NiFe tapes has demonstrated that the polycrystalline tape is effectively only one grain thick. Therefore deposition conditions of the buffer and superconducting layers on either side of the substrate should not differ and the final conductor should possess similar properties on either side of the metallic substrate. As a result J_{ceng} of the coated conductor can potentially be doubled.

The EBSP data displayed in Fig. 6a (2 hours annealing) and b (48 hours annealing) showed that some subtle changes arose after prolonged annealing. The overall map of orientation as well as information about misorientation from grain to grain provides information about potential percolative current paths which can be derived on the base of the assumption that epitaxial growth of the subsequent buffer layers and superconducting layers will follow the original texture induced by substrates [23, 24]. The X-ray data in Fig. 7 did not reveal any influence of annealing time on orientation. It appears that these differences may relate to an increase in roughness with prolonged heat treatment. Fig. 8 shows a profilogram of these samples. The average surface roughness increased from 750 nm to 2 μm. This can be understood more clearly by looking at the optical micrographs in Fig. 9 where thermal etching of the tape after annealing in vacuum is very pronounced. Because of the difficulties in annealing long pieces of 25 μm thick tape under vacuum and at the same time preserving the quality of the surface finish, a process for continuous recrystallisation was investigated. The optimised dynamic annealing was conducted in an atmosphere of hydrogen and nitrogen gases at a temperature of 650°C on 25 μm and 13 μm thick and 25 mm and 10 mm wide as-rolled kilometer long tapes. Every

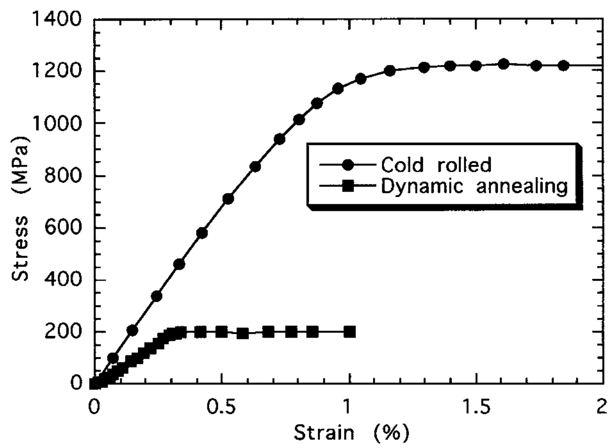
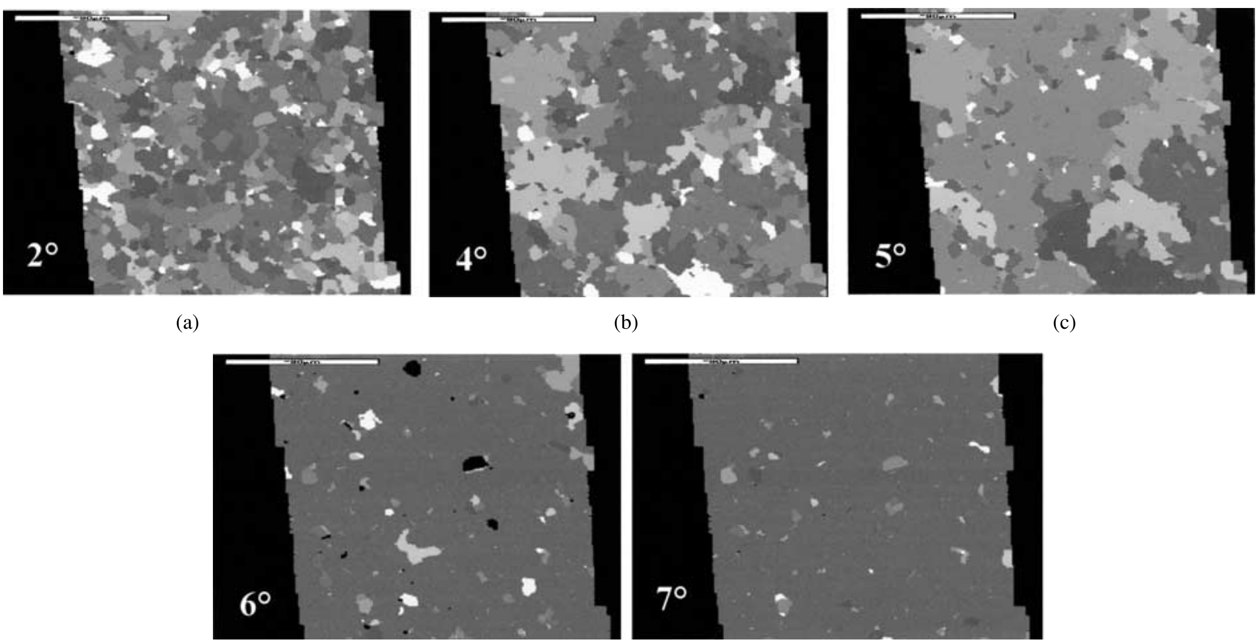


Figure 13 Stress–strain curves of the NiFe tape, 25 μm thick tape: a) ●–Cold rolled, b) ■–Dynamically annealed in protective atmosphere.



(a) (b) (c) (d) (e)

Figure 12 Large area EBSP misorientation map for a) 2 degrees, b) 4 degrees, c) 5 degrees, d) 6 degrees, e) 7 degrees. The width of the investigated area ~500 μm. (courtesy of Dr Cecile Prouteau).

section of the tape was exposed to the recrystallisation process for 15 minutes.

Thus we have established a benchmark or "standard substrate" for the project; a dynamically annealed nickel iron substrate with an excellent in-plane and out-of-plane alignment, Fig. 10. This material was deemed to be representative of that used for the buffer layer deposition experiments [19]. If "idealised" buffer layers and a final YBCO layer were to be deposited on this sample, with texture following exactly that of the substrate, then Fig. 11 shows that the current would percolate easily for the "True Grain ID" map of 6 degrees

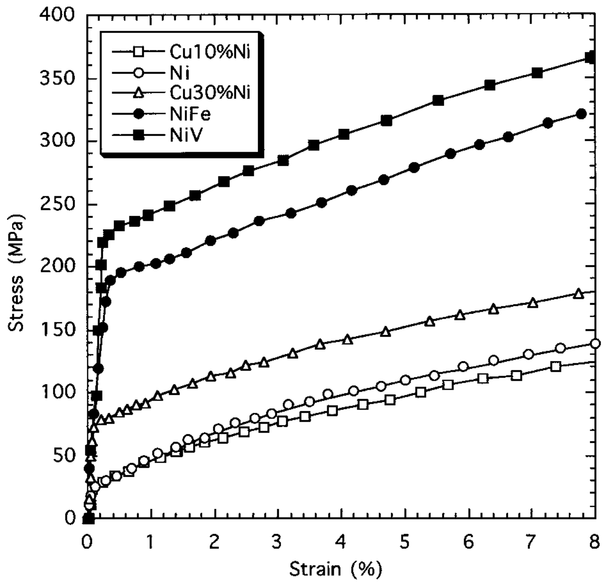


Figure 14 Stress–strain characteristics of the selected Ni-alloys developed in frame of European collaboration.

i.e. One can easily imagine the essentially unobstructed passage of current from one side to an opposite side of the area mapped percolation when a figure of 6 degrees is tolerated for the grain boundary angles within the mapped area. The question that naturally arises is "Is this area truly representative of the substrate material?" One can only answer this question by sampling larger areas, but the acquisition times for equivalent resolution clearly increases as the square of the linear dimensions of the mapped area. Although in principle this can sometimes be done, this is not a practical procedure for routine analysis, and in any case maps with the dimensions of the current tape widths cannot be obtained in one set-up operation. However, within OpalMap there is a montage facility which effectively combines data sets for OPAL maps obtained in adjacent areas by using OpalAuto and the Autostage software which controls the stage movements [21]. Using these facilities, large area OPAL maps of the same substrate material using the montage facility in conjunction can be obtain with automated stage. An example of such a montage is shown in Fig. 12 and this confirms the good percolation characteristics obtained for smaller areas of "standard substrate". Fig. 12 represents an enormous amount of data processed from the backscattered pattern recognition routines, and several such montages were made from different parts of the large area "standard substrate" tape to confirm the percolation characteristics and establish the essential uniformity of this excellent material. Using this continuously annealed NiFe tape a multilayer [NiFe-CeO_2-YSZ-CeO_2-$YBa_2Cu_3O_7$] composite conductor was manufactured by biaxially textured epitaxial growth process in context of the MUST project [25].

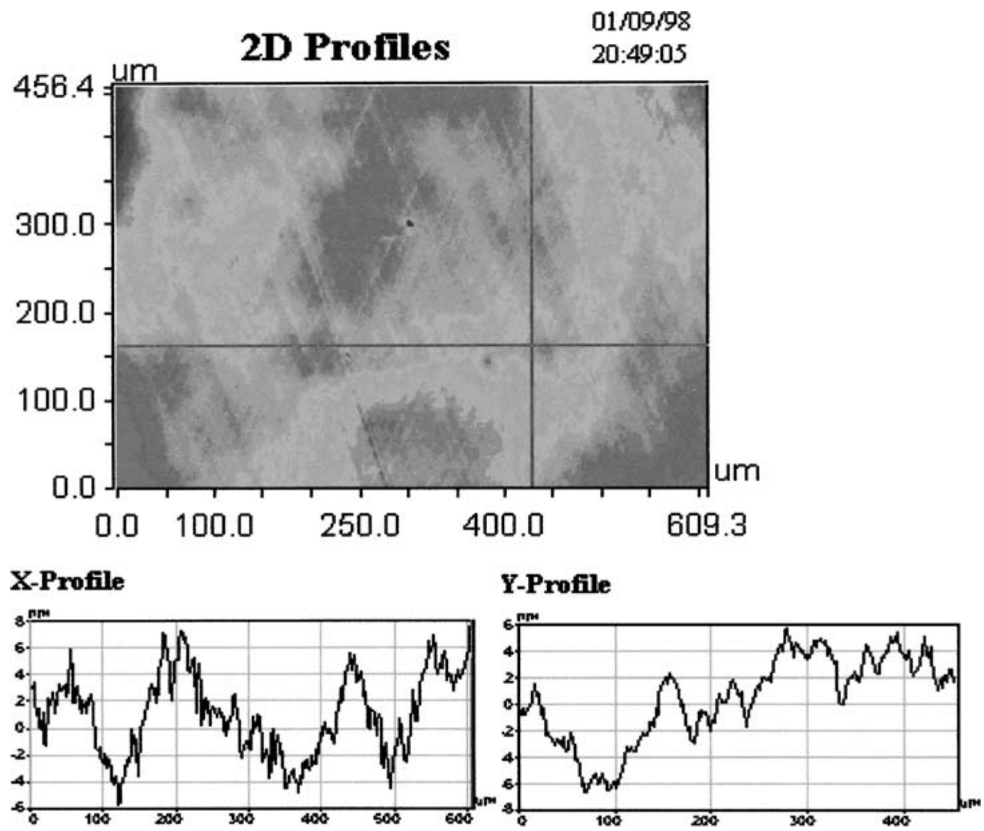
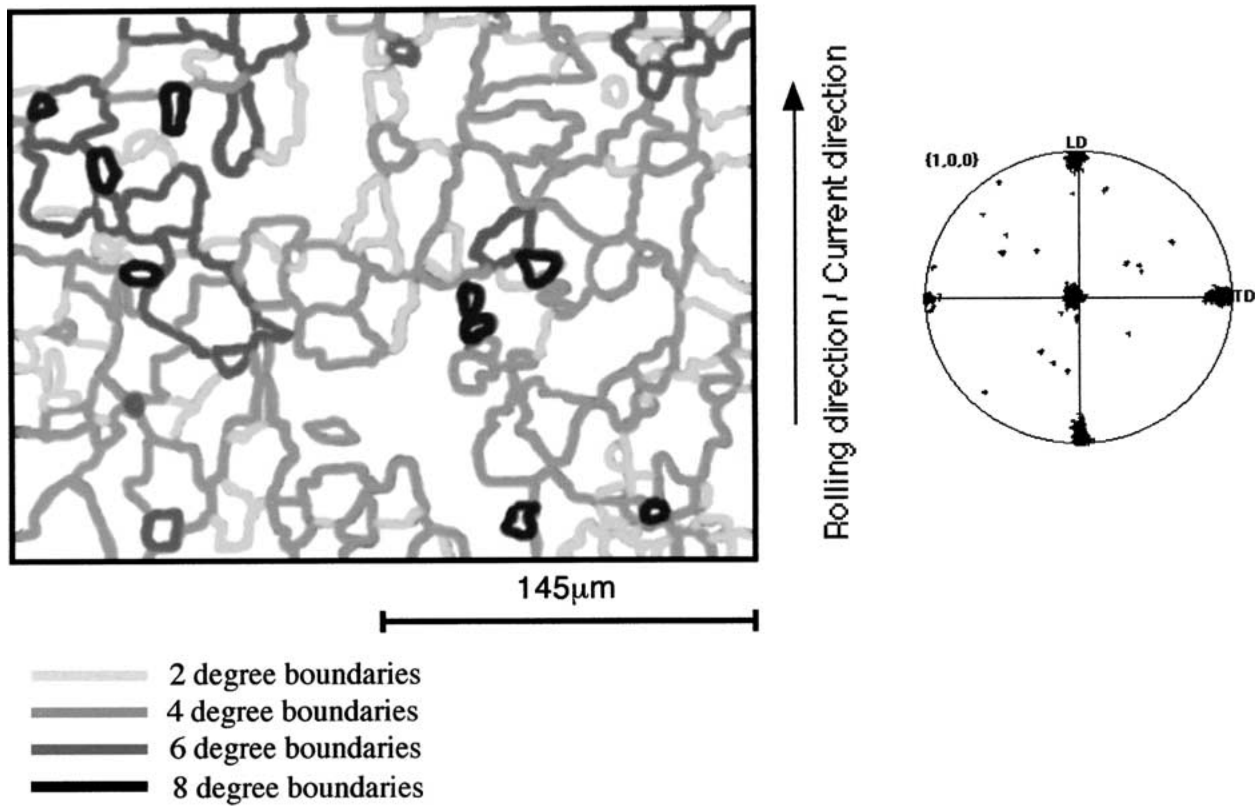


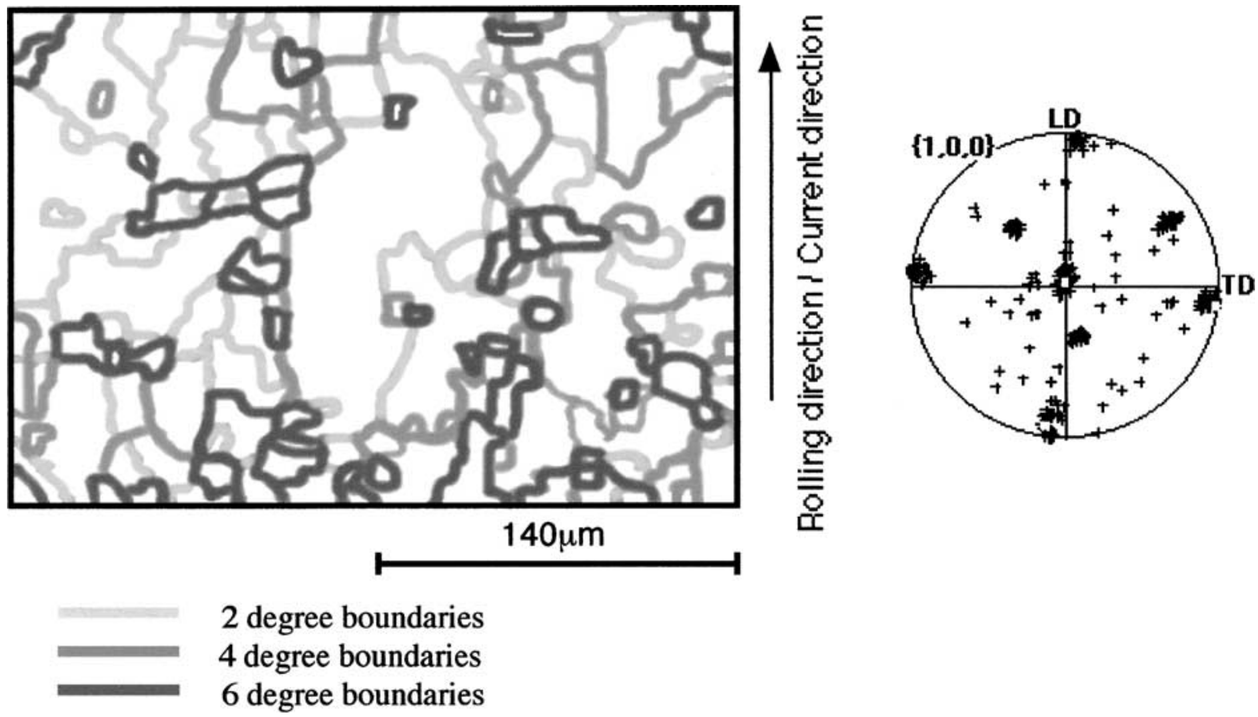
Figure 15 Profilometric measurements of the mechanically polished NiFe tape.

3.2. Mechanical properties

There is a deterioration in mechanical properties of the NiFe tape after the annealing process, as presented in Fig. 13. Systematic stress-strain measurements of the annealed tape substrate versus substrate thickness showed that by reducing the thickness below the actual grain size of the substrate, the slope of the stress-strain curve (modulus) increases and the yield point also increases. Compared with other Ni-alloys, NiFe has a lower elastic modulus and so it supports higher applied deformations, Fig. 14. This is an advantage if such tapes need to be bent around a smaller radius, as in the fabrication of small coils for high field magnets, for example.



(a)



(b)

Figure 16 Collective image of the grain boundary in NiFe 25 μm thick tape annealed at 1100°C for 4 hours characterised by different degree of disorientation from normal to the tape surface; a) unpolished surface, b) polished surface. The pole figures on the right of the collective images represent degree of alignment. One may notice on the pole figure of Fig. 16b an additional texture was developed after mechanical polishing, however the very high degree cubicity was maintained. (Image processing was conducted by Cambridge University).

3.3. Polishing

The important issue for the uninterrupted epitaxial growth of the buffer layers and superconducting layers on a highly textured tape is the smoothness of the sample surface. Therefore an experiment was conducted on the as-rolled 25 μm thick NiFe tape after polishing one side of the tape on the diamond lapping/polishing unit developed for continuous polishing of 25 mm wide tapes, using 0.25 μm diamond paste. Mechanical polishing can be used to improve the surface finish of our heavily-rolled tapes only if recrystallisation and texture formation is conducted afterwards. Fig. 15 shows the effect of mechanical polishing. Macro-modulations from the rollers were removed by the polishing. The X-ray and EBSP data, Fig. 16, demonstrated that an additional texture was developed after mechanical polishing. However the high degree of cubicity after recrystallisation was maintained and thus mechanical polishing followed by recrystallisation should be used with great caution.

An alternative option investigated was an electropolishing technique which has greater flexibility and can be used on cube textured tapes. An initial study on "statically" electropolishing was concluded with a set of parameters (electrolyte composition, temperature, time, current). Initial work on Ni and NiFe tapes brought the roughness down from 0.20 μm to $\sim$0.1 μm and formed the base for the development of the pilot continuous electropolishing treatment unit by Europa Metalli. Trials indicated that to preserve uniform electric field distribution and to provide the optimum electropolishing, cleaning and drying conditions, the orientation of the tape should be vertical, see Fig. 17.

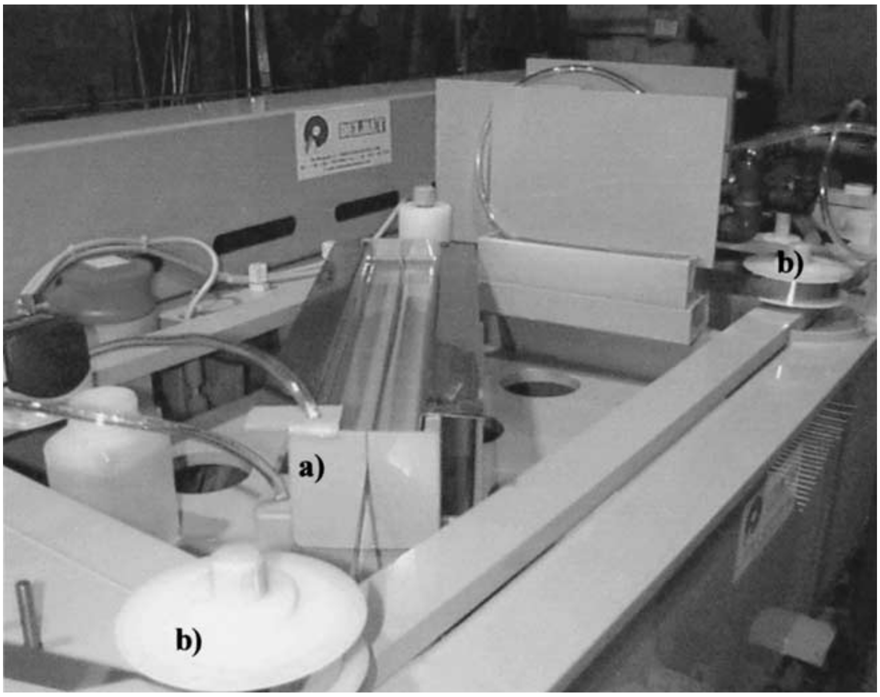


Figure 17 Electropolishing unit at EM where long lengths of 25 mm wide and 25 μm thick NiFe tapes were electropolished a) view of the treatment tank, b) drying and winding system. (courtesy of Europa Metalli S.p.a. Superconductors Division).

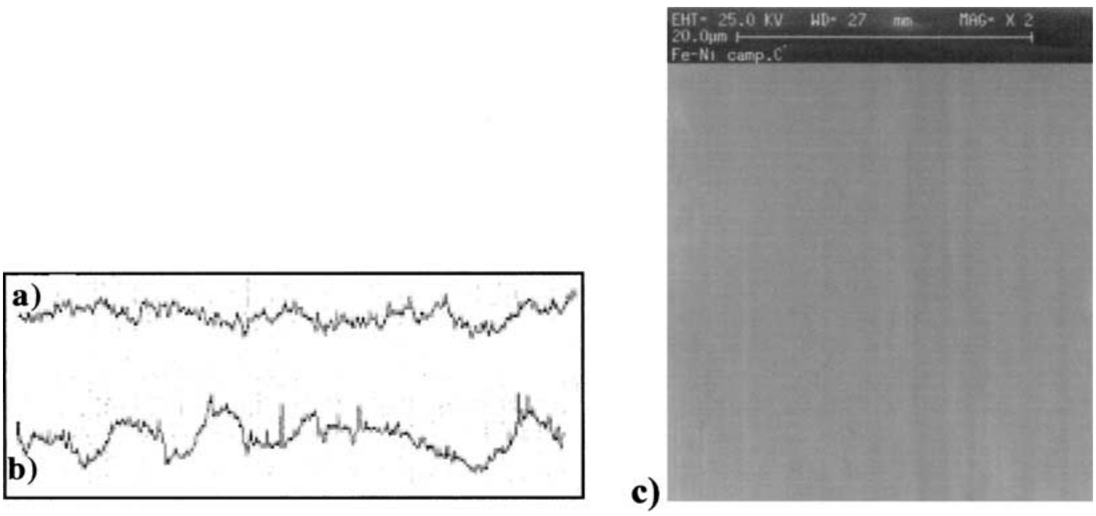


Figure 18 The profilometric measurements of the dynamically annealed NiFe tape surface relief conducted by contact method a) after electropolishing, b) before electropolishing, c) SEM picture of the tape surface after electropolishing.

From all these observations we can argue that the most reliable technique to achieve the roughness level requested over the longer lengths without microstructure interruption is electropolishing. The profilometric measurements along the length of the NiFe (50%50%) tapes are presented in Fig. 18. Continuous electropolishing of the long lengths of dynamically annealed tape improved the surface smoothness.

4. Conclusions

A highly oriented (FWHM < 10°) cubic texture has been formed in long length 13 μm, 25 μm, 50 μm thick and 10 mm and 25 mm wide tapes NiFe tapes. This texture is formed under a range of conditions but heat treatment for excessive times in vacuum tends to roughen the surface and should be avoided. Also, mechanical polishing can induce an additional undesirable texture, whereas electropolishing does not. This suggested that the morphological differences of the surface relief are of the highest importance. In extreme case a rough surface can also reduce the quality of the epitaxial growth and can even obscure the texture development. However we must stress that the surface texture and purity are critical to subsequent film deposition processes and therefore good EBSP data of the metallic substrates is an essential prerequisite for the subsequent deposition of high quality superconducting layers. These NiFe highly textured tapes produced in kilometer lengths prove capable of providing a basis for the epitaxial growth of the buffer and superconducting layer [25].

One may expect that NiFe is shortly going to make its mark in the current R&D market for superconducting conductor industry in Europe.

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References

1. X. D. WU, S. R. FOLTYN, P. N. ARENDT, W. R. BLUMENTHAL, I. H. CAMPBELL, J. D. COTTON, J. Y. COULTER, W. L. HULTS, M. P. MALEY, H. F. SAFAR and J. L. SMITH, *Appl. Phys. Lett.* **67** (1995) 2397.
2. M. PARANTHAMAN, A. GOYAL, F. A. LIST, E. D. SPECHT, D. F. LEE, P. M. MARTIN, QING HE, D. K. CHRISTEN, D. P. NORTON, J. D. BUDAI and D. M. KROEGER, *Physica C* **275** (1997) 266.
3. GOYAL A. GOYAL, D. P. NORTON, J. D. BUDAI, M. PARANTHAMAN, E. D. SPECHT, D. M. KROEGER, D. K. CHRISTEN, Q. HE, B. SAFFIAN, F. A. LIST, D. F. LEE, P. M. MARTIN, C. E. KLABUNDE, E. HARTFIELD and V. K. SIKKA, *Appl. Phys. Lett.* **69** (1996) 1795.
4. J. E. EVETTS and B. A. GLOWACKI, *Cryogenics* **28** (1988) 641.
5. B. A. GLOWACKI, M. VICKERS and E. MAHER, *Materials World* **6**(11) (1998) 683.
6. J. E. EVETTS and B. A. GLOWACKI, *Supercond. Sci. Technol.* **13** (2000) 443.
7. A. GOYAL, D. P. NORTON, D. M. KROEGER, D. K. CHRISTEN, M. PARANTHAMAN, E. D. SPECHT, J. D. BUDAI, Q. HE, B. SAFFIAN, F. A. LIST, D. F. LEE, E. HATFIELD, C. E. KLABUNDE, P. M. MARTIN, J. MATHIS and C. PARK, *J. Mater. Res.* **12** (1997) 2924.
8. Y. YAMADA, *Supercond. Sci. Technol.* **13** (2000) 82.
9. A. TUISSI, R. CORTI, E. VILLA, A. P. BRAMLEY, M. E. VICKERS and J. E. EVETTS, *Inst. Phys. Conf. Ser.* No. 167, **1** (2000) 399.
10. E. VILLA, A. TUISSI, R. TOMOV and J. E. EVETTS, *International Journal of Modern Physics B* **14** (25–27) 3145.
11. N. REGER, B. DEBOER, J. EICKEMEYER, R. OPTIZ, B. HOLZAPFEL and L. SCHULTZ, *Inst. Phys. Conf. Ser.* No. **167** (2000) 331.
12. J. EICKEMEYER, D. SELBMANN, R. OPITZ, E. MAHER and W. PRUSSEIT, in Proceedings of 6th International Conference on Materials and Mechanisms of Superconductivity and High-Temperature Superconductors, Houston, Texas, USA, February 20–25, 2000.
13. M. MAJOROS, B. A. GLOWACKI and A. M. CAMPBELL, *Physica C* **334** (2000) 129.
14. Web address: http://www.cus.cam.ac.uk/~bag10/MUST.html
15. C. S. BARRETT, "The Structure of Metals" (McGraw-Hill, 1952, 1966).
16. I. L. DILLAMORE, *Metallurgical Reviews* **10** (1965) 271.
17. K. LUCKE and O. ENGLER, *Materials Science and Technology* **6** (1990) 1113.
18. R. W. CAHN (ed.), "Materials Science and Technology," Vol. 15: Processing Metals and Alloys (VCH Verlagsgesellschaft mbH, 1991).
19. J. DENUL, I. VAN DRISSCHE, H. TE LINTELO, R. DE GRYSE, A. TSETSEKOU, S. HOSTE, B. A. GLOWACKI, R. GARRE, E. MAHER, J. GOOD, E. RANUCCI, E. LARRAURI and S. HOSTE, *Inst. Phys. Conf. Ser.* No. 167 (2000) 335.
20. T. J. JACKSON, B. A. GLOWACKI and J. E. EVETTS, *Physica C* **296** (1998) 215.
21. Web address: http://www.Oxford-Instruments.com
22. A. GOYAL, S. X. REN, E. D. SPECHT, D. M. KROEGER, R. FEENSTRA, D. NORTON, M. PARANTHAMA D. F. LEE and D. K. CHRISTEN, *Micron* **30** (1999) 463.
23. N. A. RUTTER, B. A. GLOWACKI and J. E. EVETTS, *Supercond. Sci. Technol.* **13** (2000) L25.
24. N. A. RUTTER and B. A. GLOWACKI, *IEEE Trans. Applied Superconductivity* **11** (2001) 2730.
25. R. I. TOMOV, A. KURSUMOVIC, M. MAJOROS, D.-J. KANG, B. A. GLOWACKI and J. E. EVETTS, *Superconductor Science and Tecnology*, in press.

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