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REVIEW

Nickel base substrate tapes for coated superconductor applications

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Abstract The development and status of Ni base alloy substrate tapes for coated superconductor applications have been reviewed. Rolling assisted Biaxially Textured Substrates or RABiTS^{TM} method comprising cold rolling and annealing of Ni gives rise to sharp cube texture ({100} ⟨100⟩) component which is required for subsequent epitaxial growth of the buffer and YBCO layers. However, pure Ni seems to be rather unsuitable for this purpose since its mechanical strength as well as the stability of the cube texture deteriorate at the deposition temperature of YBCO. Refractory metals, particularly, W and Mo hold the potential to be used as micro-alloying element to impart strength as well as to preserve the cube texture and maintain its stability at high temperatures. Cr and V at higher alloying level are suited for non-magnetic applications. Recent works have suggested the possibility of using substrates with multilayer architecture and this needs to be investigated further.

Introduction

Ever since the discovery of high temperature oxide superconductors in 1986, a world wide research endeavor has been directed towards developing long, flexible conductors with high critical current density (J_c) for a wide range of industrial applications, particularly, in the power generation and distribution systems like power transmission cables, transformers etc. However, oxide superconductors are brittle ceramic materials and therefore not amenable to common forming operations. An elegant way of overcoming this difficulty is to use High Temperature Superconducting (HTS) materials like Yttrium Barium Copper Oxide (YBCO) in the form of thin film on some substrate material. This approach has come to be known as the second generation coated conductor technology (2G coated conductor technology) as opposed to the first generation conductors (1G conductors) which are prepared through multifilamentary wire technique.

During the early periods of these efforts it has been observed that randomly oriented polycrystalline HTS materials have critical current densities below 500 A/cm^2. In contrast, oriented YBCO films grown epitaxially on single crystal oxide substrates such as (001) oriented $SrTiO_3$ exhibit J_c values in excess of 1 MA/cm^2 at 77 K [1]. This huge difference in critical current density values between randomly oriented HTS materials and single crystal like epitaxial film is attributed to the existence of large enough misorientation angles at the grain boundaries in the former. The value of J_c decreases significantly across the grain boundary as the misorientation angle increases with "weak link" behavior observed for misorientation angle ~10 and above [2, 3]. Feldmann et al. [4] with the help of the Magneto-Optical imaging and Electron Back Scatter Diffraction (EBSD) studies have established that flux penetration starts at grain boundary misorientation angles greater than 4°. Therefore, in order to achieve

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high J_c values the HTS wires or tapes must have a high degree of grain alignment i.e. the conductor must have a pronounced crystallographic texture over its entire length.

One approach in the production of high-J_c HTS tapes is to deposit a thick epitaxial HTS film on a substrate material that has a high degree of in-plane and out-of-plane crystallographic texture that can be produced in long lengths. Epitaxial HTS films on single crystal oxides satisfy the requirements for high J_c, but it is not economical to produce long lengths of such substrates. A very novel method to produce such substrate materials has been developed at Oak Ridge National Laboratory, USA through thermomechanical processing of metallic materials [5]. In this method, base metals such as Ni or Cu is rolled to a high degree of deformation and then recrystallized to obtain a very sharp, well developed cube texture, on which epitaxial growth of a buffer layer and of YBCO film can be achieved. Such substrates which are biaxially textured and have chemically compatible surfaces for epitaxial growth of YBCO or other superconducting films have been referred to as Rolling Assisted Biaxially Textured Substrates (RABiTS™). A schematic diagram of the RABiTS™ process has been shown in Fig. 1 [6]. Current densities above 10^6 A/cm^2 have been reported in case of YBCO coated on such substrates [7].

The first metals used to produce biaxially textured substrates for coated conductor applications are Ag and Ni [5]. However, Ag is technically not attractive due to its lack of strength, cost and inability to develop a sharp cube texture. Amongst the metallic elements Ni has been widely used as a substrate material because of good oxidation resistance and good lattice match with YBCO. However, YBCO cannot be directly deposited on a Ni substrate because of the chemical reaction between Ni and YBCO at high temperatures. To suppress the diffusion of Ni into YBCO, buffer layers are epitaxially grown on Ni. These buffer layers serve two purposes. They maintain the sharp crystallographic texture of the substrate material and act as diffusion barriers to prevent the diffusion of substrate material into the HTS material and the resulting chemical reaction. The primary objective therefore is to develop a substrate material with a strong cube texture which is stable at the deposition temperatures of the buffer and superconducting layers. In addition, there are several other desirable criteria such as the high mechanical strength of the substrate material which is very essential during actual fabrication process involving a reel-to-reel deposition of the buffer and superconducting layers. Pure Ni does not have the requisite tensile strength in the annealed condition. For this reason there has been a progressive shift towards using alloyed Ni base substrates rather than pure Ni. It remains a challenge to identify the specific alloying elements and the total alloy contents needed for specific applications. The present paper reviews the developmental activities and current status of the Ni base substrate materials for coated superconductor applications.

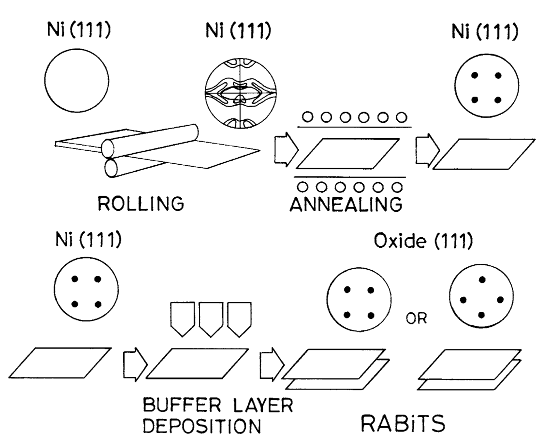


Fig. 1 Schematic representation of the RABiTS™ process [© (1999), reprinted with permission from Elsevier] [6]

Pure nickel as a substrate material

Rolling and annealing of fcc metals such as Cu, Ni and Al lead to the formation of {100} ⟨100⟩ or "cube texture" as defined in Fig. 2. The cube texture is very sharp; it is mostly a single component texture i.e. it develops with a large fraction of grains near the ideal cube orientation, with little misorientation angle among them. In addition, Ni is a relatively high melting point metal and is resistant to high temperature oxidation. This helps in the epitaxial growth of the YBCO film on intermediate buffer layers.

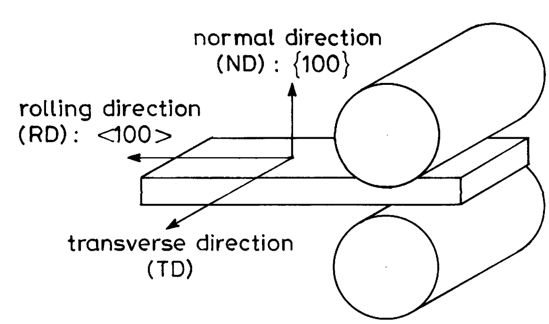


Fig. 2 Definition of the notation for the cube texture [© (1998) IOP Publishing Ltd., reprinted with permission from the publisher and authors] [7]

Cube texture in Ni can be formed after heavy cold rolling and subsequent annealing, as the primary recrystallization texture [8]. The intensity of cube texture in Ni has been found to depend on a number of factors amongst which the purity of Ni is the most important one. Presence of impurities even in ppm level can adversely affect the recrystallization behavior and consequent texture development [9]. The most detrimental effect has been found to be associated with the presence of sulfur [10]. It influences the recrystallization characteristics of Ni significantly. Eickemeyer et al. [11] have amply demonstrated that the purity of Ni has a significant bearing on the sharpness of the cube texture. They measured the intensity of the cube component in two Ni tapes with 99.98 and 99.94% purity, after heavy cold rolling and annealing. The relative intensity of the cube component was found to be significantly higher in case of the Ni tape with greater purity, as evident from Fig. 3. The less pure Ni (99.94%) tape was found to be more prone to secondary recrystallization.

Goyal et al. [5] were the first to study in detail the possibility of using pure Ni as a substrate material. They cold rolled pure Ni to more than 90% deformation, followed by recrystallization annealing at 1,000 °C for 4 h. The development of a very sharp single component cube texture has been reported by them after this heat treatment. The full width half maximum (FWHM) values of the X-ray ω and φ-scans have been reported as 6° and 7°, respectively. Investigations on the misorientation angle distribution in millimeter size regions of the substrate with over 100 interconnected grain boundaries have revealed that over 95% of the grain boundaries have misorientation angle less than 10° i.e. there were very few high angle grain boundaries and most of the grain boundaries were of low angle type only. The grain size was found to be in the range of 50–100 μm and the as rolled substrate was found to possess a fairly small surface roughness (~10 nm) which was suitable for epitaxial film growth. The subsequent deposition of YBCO with different combinations of buffer layers yielded critical current density exceeding 10^5 A/cm^2 in certain configurations.

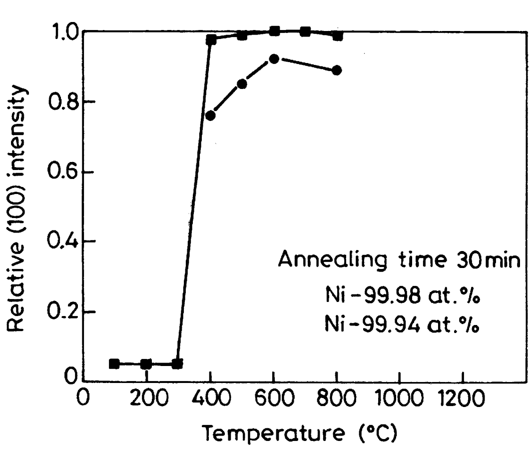


Fig. 3 Effect of Ni purity on the intensity of cube texture developed after annealing [© (2000), reprinted with permission from Elsevier] [11]

Specht et al. [7] studied the optimum rolling and annealing conditions for the formation of a sharp cube texture in such Ni substrates. They conducted investigation on pure Ni over a range of annealing temperature (300–1375 °C). It was found that at 300 °C annealing temperature the primary recrystallization texture consists of 94% cube component ({100} ⟨100⟩) and 6% cube twin component ({221} ⟨221⟩). However, with higher annealing temperature and time the twin component gradually disappears and practically no twin component is observed above the annealing temperature of 1200 °C. Higher annealing temperature has been found to lead to increased surface roughness. According to these researchers a substrate thickness of 125 μm leads to the formation of an optimum texture.

The strengthening of the cube component at higher annealing temperatures has also been reported by other investigators [11, 12]. Zhou et al. [13] have reported that for an ultra pure Ni sample (99.999%) and for a commercially pure Ni tape (99.95%) strong cube component is present after annealing at 1200 °C. In addition, for the ultra pure Ni tape the sharpness of the cube component is found to increase with an annealing process using a continuous heating ramp up to 1200 °C.

The influence of cold rolling variables on the sharpness of cube texture in Ni substrates has been evaluated by Zhou et al. [14]. They have subjected the Ni tapes to different amounts of total reduction using a range of reduction per pass. It has been observed by these authors that the cube texture upon annealing is more intense when the final deformation is more than 95%. The sharpness of the cube texture is also influenced by the reduction per pass. Small reductions per pass increase the intensity of the cube component by decreasing the amount of lateral spread.

However, pure Ni has some major disadvantages for being used as a substrate material. In the deposition temperature range for the buffer and YBCO films on these substrates (~700–900 °C), abnormal grain growth or secondary recrystallization i.e. a preferential growth of minor texture component at the expense of the major cube texture component, can take place thereby destroying the cube texture altogether. De Boer et al. [15] have reported the influence of grain growth on the texture of Ni tapes annealed at three different temperatures, namely, 500, 550 and 600 °C, as shown in Fig. 4. They observed that after moderate grain growth, which is characterized by slow growth of the cube textured grains (Fig. 4a, b), some grains start to grow very fast (Fig. 4c). This abnormal grain growth becomes very prominent above 600 °C, with large grains highly misoriented towards the cube grains, growing at the expense of cube grains. Upon further annealing the sharp cube texture developed at 500 °C (Fig. 5a), is found to be destroyed altogether, and the grains which grow to sizes of about 1 mm or more are randomly oriented as can be seen from the discrete (111) pole figure of those giant grains in Fig. 5b.

Makita et al. [16] studied the development of recrystallization textures in both high purity (99.99%) and less purity Ni (99.95%) samples and reported that secondary recrystallization occurs once the grain size reaches some final value. Contrary to the above findings, Specht et al. [7] have found that no secondary recrystallization occurs and cube texture continues to strengthen even after annealing at 1375 °C although the final grain size is reached at about 600 °C.

A necessary condition for abnormal grain growth to occur is the existence of highly mobile high angle grain boundaries. Due to this, only those grains which are highly misoriented towards the surrounding cube grains can only grow. Other than the misorientation angle the mobility of grain boundaries depends upon the impurities present either as solute or second phase particles. Solutes which are segregated at the grain boundaries interact with the grain boundaries so that the migrating grain boundaries have to drag the solute along with it, resulting in a decrease of grain boundary mobility. It is well-known for a long time that fine precipitated second phase particles have a pinning effect on grain boundaries which is known as the Zener drag [17].

Abnormal grain growth may take place if somehow the mobility of the grain boundary is enhanced. This may occur at higher temperatures where due to increased solubility the segregated solute is taken into solution and thus the grain boundary segregation becomes considerably less. The grain boundary mobility may also increase due to the phenomenon of Ostwald ripening, where the pinning force will decrease due to increased interparticle distance. In both the mechanisms some boundaries may become mobile earlier than the others leading to the preferential growth of only a few grains.

In addition to secondary recrystallization or abnormal grain growth, another major problem is grain boundary grooving at high annealing temperatures. Due to this phenomenon, atoms at the grain boundaries, which meet the surface of the tape, diffuse along the surface or through the bulk out of the grain boundary region causing a ditch at the surface and an enrichment of the material on both sides of the ditch, as shown in Fig. 6. Groove formation may occur by an evaporation–condensation mechanism or surface diffusion process [18]. The groove profile depends upon the formation mechanism, the processing conditions, annealing temperature and time, grain boundary and surface energy, and the diffusion coefficients of the

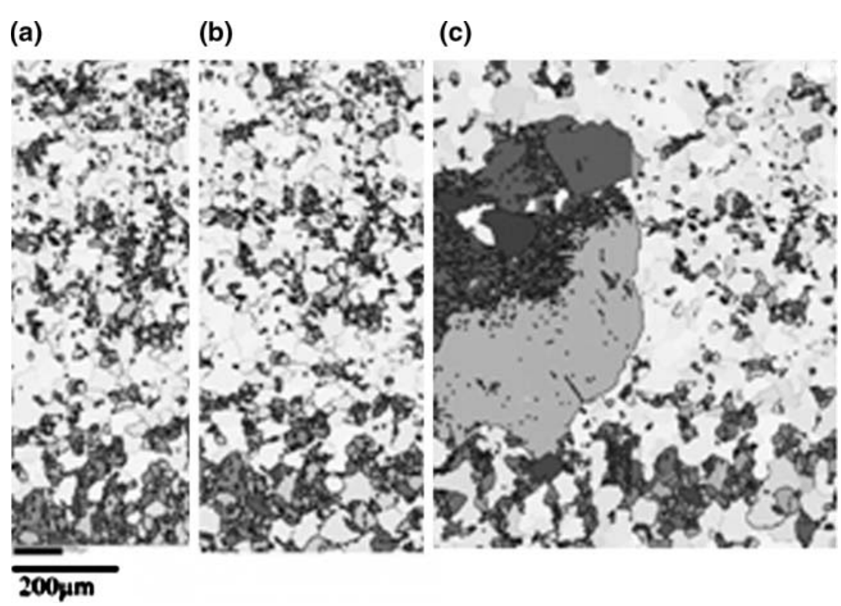


Fig. 4 Anomalous grain growth (secondary recrystallization) in Ni. EBSD-mappings of a sample annealed successively at increasing temperatures: (**a**) 500 °C/30 min, (**b**) 550 °C/30 min and (**c**) 600 °C/30 min. Dark grains are highly misoriented with respect to the exact cube orientation. Grain boundaries with misorientations between 5° and 10° are marked by a gray line, grain boundaries with misorientation angles above 10° by a fat black line in the EBSD-maps [© (2001), reprinted with permission from Elsevier] [15]

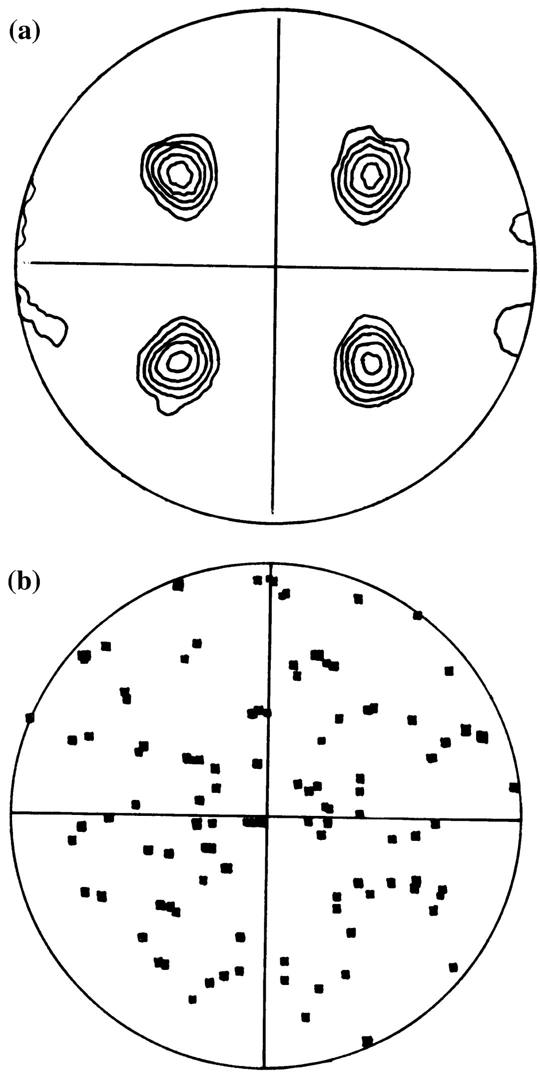


Fig. 5 {111}-pole figures determined by EBSD of (**a**) the as recrystallized Ni Sample annealed at 500 °C/30 min and (**b**) the orientations of giant grains grown due to abnormal grain growth [© (2001), reprinted with permission from Elsevier] [15]

atom species through grain boundaries and the surface. Amongst these, the processing conditions and annealing temperature and time are found to have the most important contributions. Gladstone et al. [19] have conducted AFM and EBSD studies on the grooving phenomenon in cube textured Ni tapes. They have observed that there is a significant correlation between the groove depth and the misorientation angle at the grain boundaries. The depths of the grooves at low angle grain boundaries are rather low as compared to those at high angle boundaries and the Σ3 or the coincident lattice site boundaries, which usually show a wide average range of groove depth. The typical values for low angle grain boundary grooves are 300 Å and some high angle grain boundaries have depths greater

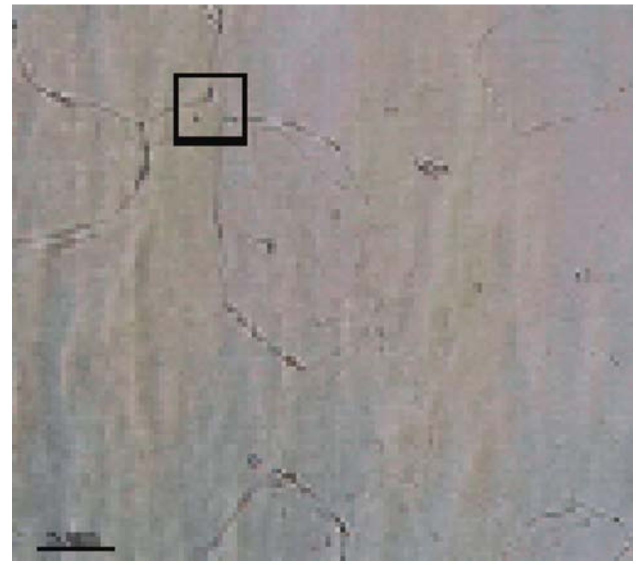


Fig. 6 Optical micrograph of a typical region of a cube texture Ni tape showing thermal etching at the grain boundaries [© (2001) IEEE, reprinted with permission from IEEE] [19]

than 1,200 Å. The grain boundary groove depth is found to depend on the annealing atmosphere also. Ni annealed in Ar–4% H_2 atmosphere shows lower average groove depth than Ni annealed in vacuum. The dependence of groove depth on misorientation angle and annealing atmosphere can be appreciated from Fig. 7.

Pure Ni has low tensile strength in annealed condition which limits the possibility of coating very thin Ni tapes, typically in the range of 20–30 μm that is necessary to achieve a high current density. Also, Ni is ferromagnetic with a Curie temperature of 627 K. Thus at the liquid nitrogen temperature (77 K) Ni is sufficiently ferromagnetic. In alternating current applications this may lead to a significant energy loss due to hysteresis.

It can now be clearly understood that pure Ni has some inherent problems associated with it. It was therefore felt necessary to design such substrate materials which have high strength, high texture stability and also suitable for alternating current applications. The classical metallurgical approach to the above problems is solid-solution strengthening of pure Ni through alloying element addition. There has been tremendous research thrust in recent years to develop strong as well as non-magnetic Ni base substrate tapes which can retain their texture sharpness at the deposition temperatures of buffer and YBCO layers. A number of metallurgical systems based on Ni have so far been identified which seem potentially suitable to meet these requirements.

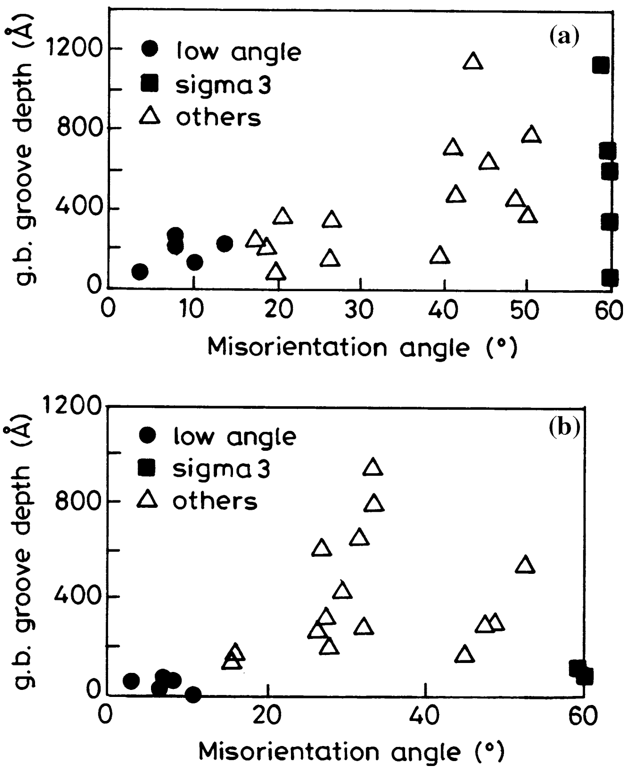


Fig. 7 Plot showing the relationship between the grain boundary groove depth and the misorientation for Ni tape (**a**) recrystallized in vacuum at 800 °C for 4 h and (**b**) recrystallized in Ar–1%H_2 at 800 °C for 4 h [© (2001) IEEE, reprinted with permission from IEEE] [19]

Nickel base alloy substrates

There are certain alloy design issues which must be addressed while developing suitable Ni base alloy substrate. Amongst them the most important ones are:

- Development of sharp cube texture in Ni base alloys.
- Type and amount of alloying elements to be added.

It has long been established that the addition of most of the alloying elements to Ni reduces the stacking fault energy (SFE) which influences the rolling texture and subsequently the recrystallization texture upon annealing. High and Medium stacking fault energy FCC materials tend to develop a pure metal or copper type rolling texture. In contrast, low SFE materials develop a prominent brass type rolling texture. Ray [20] has studied the "texture transition" behavior in the Ni–Co alloy system as a function of SFE. In fact, high alloying addition may completely suppress the cube component after recrystallization. Thus it is of prime importance to ensure that solution hardening due to alloying addition does not interfere with the formation of a sharp cube component in the recrystallized material.

Selection and level of alloying elements

From the above discussion it should be clear that the alloying elements added to Ni must satisfy the following conditions:

- The stability of the cube texture in Ni is known to get diminished due to the presence of alloying elements. Therefore, the type and level of alloying elements should be such as not to adversely affect the recrystallization and requisite texture formation.
- Since the tapes to be used should be in a very thin substrate form (~20–30 μm) they should be sufficiently strong mechanically for a reel-to-reel deposition process at high temperatures. Therefore, the alloying elements should enhance the mechanical strength of Ni.
- Since abnormal grain growth may occur at the deposition temperature range, leading to the destruction of the cube texture in Ni, texture stability must be guaranteed at the deposition temperatures, usually in the range of 700–900 °C.
- The alloying elements should suppress the actions of undesirable impurity atoms, not deliberately added, and allow attainment of a sharp cube texture. They should also ensure a high thermal stability against secondary recrystallization at the processing temperature.

Potential Ni based alloy systems to be used as substrate material

The suitable alloying elements to meet the above mentioned criteria are the ones belonging to the group VA and group VIA of the Periodic Table as identified in Fig. 8. The reasons behind the selection of these elements are:

- All these elements are high melting point metals i.e. so called refractory metals. Due to their high melting points they are able to increase the primary recrystallization temperature of Ni. Table 1 gives an idea of the melting points of these metals in comparison to that of pure Ni.
- These elements are able to strengthen the grain boundaries in nickel even when present in a micro-alloying level (≤2,000 wt ppm).
- The chemical affinity of these elements, particularly Mo or Ta, towards deleterious impurities, namely

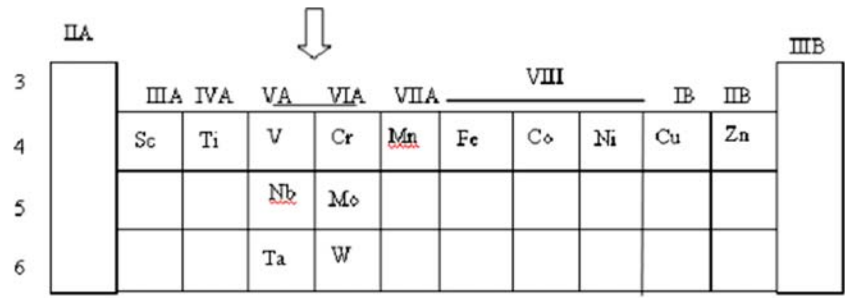


Fig. 8 Preferred micro-alloying elements for RABITS^{TM} substrates are indicated by arrow head [© (1998) IOP Publishing Ltd., reprinted with permission from the publisher and authors] [7]

Table 1 Comparison of the melting points of different refractory metals with respect to Ni

Element	$T_{m,\ element}/T_{m,\ nickel}$
Tungsten	2.13831
Tantalum	1.903935
Molybdenum	1.675926
Vanadium	1.26331
Chromium	1.261574
Niobium	1.591435

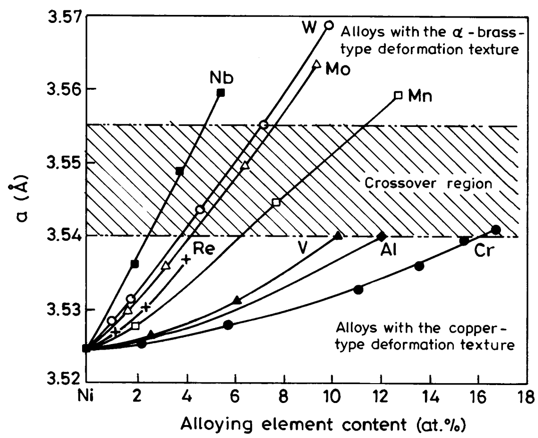


Fig. 9 Texture transition in Ni with the addition of different alloying elements (© 2004 by Schastlivtsev, Ustinov, Rodionov, Sokolov, Gervas'eva, Khlebnikova, Nosov, Sazonova, Vasil'ev, Vladimirova, Abaleshev, Gierlowski, Lewandowski, Szymczak. With kind permission of Springer Science and Business Media) [22]

sulfur, is stronger than that of Ni. So they can form sulphides and suppress the detrimental effect of sulfur on the development of recrystallization texture in Ni.

- Significant solid solubility of these metals exists in Ni which is helpful in solid-solution strengthening and consequent increase in the tensile strength of the derived tapes.
- The metals particularly Cr, V and Mo when added in larger quantities can suppress the ferromagnetic property of Ni beyond 77 K [21].

Micro-alloyed Ni tapes

Effect of micro-alloying on texture formation and texture stability

The beneficial effects of micro-alloying Ni with refractory metals on the development and stability of the cube texture have been reported by many investigators. Schastlivstev et al. [22] have studied the development of cold rolling textures in a series of Ni–W, Ni–Mo, Ni–Cr, Ni–V and Ni–Re alloys. In all those alloys there is a critical amount of alloying element addition beyond which the rolling texture changes from Copper type to Brass type. This is in accordance with the well-known phenomenon of "texture transition" in fcc metals and alloys discussed earlier. They correlated the observed transition behavior with the change in lattice parameter of the different alloy systems studied (Fig. 9).

The cold rolling textures of Ni–W alloys have also been studied in detail by Sarma et al. [23]. These authors have reported that texture transition in Ni–W

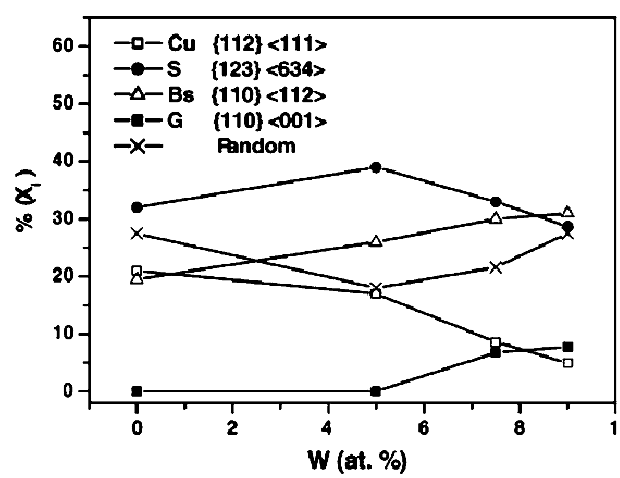


Fig. 10 Volume fractions of different rolling texture components (C, S, B and G) as a function of W content [© (2004), reprinted with permission from Elsevier] [23]

alloys takes place in the vicinity of 7.5 at.% W (Fig. 10).They subsequently studied the recrystallization behavior of Ni–W alloys with up to 10 at.% of W subjecting them to single stage and two stage annealing treatments [24]. In the two step annealing, the samples were initially held at lower temperatures and then taken to higher temperatures such that the total time was same in both single and two step annealing. It was observed that almost 100% cube texture can be obtained in Ni–5 at.% W alloy with the intensity sharply diminishing with higher W content and that the two stage heat treatment yields much intense cube component than the single stage heat treatment. The higher intensity of the cube component in the two stage annealed material has been attributed to the fact that a lower temperature favors the nucleation of a large number of cube grains which can rapidly grow at higher temperature at the expense of other orientations.

Eickemeyer et al. [25] have studied the influence of small additions (~0.1%) of Mo, W, Nb and Ta to Ni in both low and high temperature annealing regimes. The recrystallization textures in these alloys have been characterized by them using X-ray diffraction technique, i.e. θ–2θ scan and the FWHM values of (111) pole figures, as well as by EBSD. They have observed that in comparison to pure Ni (99.94 at.% purity) micro-alloying yielded an increased (100) X-ray reflection intensity after annealing up to 1100 °C as shown in Fig. 11. The evaluation of the pole figures and EBSD mappings of the Ni–0.1 at.% W, Ni–0.1 at.% Mo, Ni–0.1 at.% Nb and Ni–0.1 at.%Ta revealed strong cube texture in all these materials. The measured relative intensity of the Ni–0.1 at.% tantalum has been found to be 1.0 over the annealing temperature range of 800–1100 °C. After annealing at 850 °C, the micro-alloyed Ni tapes with 0.1 at.% niobium also offered the maximum relative intensity value of 1.0 with FWHM values ranging between 6.7 and 7.5°. Consequently, the four refractory metals are found to be very effective as micro-alloying elements with pure nickel, enhancing the temperature region in which the recrystallized cube texture becomes considerably more stable.

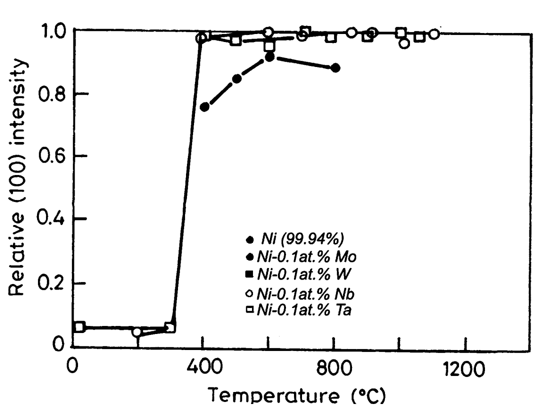


Fig. 11 Influence of micro-alloying of Ni with Mo, W, Nb and Ta on the relative (100) X-ray reflection intensity after annealing at different temperatures for 30 min [© (2001) IOP Publishing Ltd., reprinted with permission from the publisher and authors] [25]

Goyal et al. [26] have studied the influence of minor additions of W (3 at.%) and Fe (2 at.%) to Ni, for the production of strong biaxially textured substrates. The material was deformed in excess of 98% reduction in thickness by rolling and then subsequently annealed at temperatures between 1000 and 1400 °C under Ar–4%H_2 atmosphere for 1 h. For lower annealing temperatures (~up to 1100 °C) they have reported the presence of a significant amount of rolling texture present in the material. For annealing temperatures above 1200 °C they found the substrate to be highly cube textured. After annealing at 1400 °C, the texture was found to sharpen further as revealed by the FWHM values and grain boundary misorientation distribution (GBMD) measurements. GBMD results obtained with the help of EBSD studies have proved that most of the grain boundaries in this condition are of low-angle type essential for achieving a high J_c value. High critical current densities, up to 1.9 MA/cm^2 at 77 K, self-field have been demonstrated on this substrate using a multilayer buffer configuration (YBCO/CeO_2/YSZ/Y_2O_3/Ni–3 at.% W–1.7 at.% Fe).

Varesi et al. [27] have studied the texture development in three Ni alloys, namely, Ni–11 at.% V, Ni–13 at.% Cr and Ni–5 at.% W. They have reported that Ni–V and Ni–Cr substrates have a greater number of cube twin and highly misoriented grains in comparison to Ni–5 at.% W. From optical microscopic analysis they determined the volume fractions of the twinned cube grains to be 0.17, 0.15 and 0.05, respectively, for the above three alloys. The ratios of the area representing the cube oriented grains with respect to the substrate area were found to be 0.96, 0.97 and 0.99, respectively, for the above three cases.

Eickmeyer et al. [28] have further investigated the effect of microstructural conditions, particularly the initial grain size, for achieving a perfect cube texture in Ni–5 at.% W alloy. Starting with two Ni–5 at.% W alloys with different grain sizes, they found that the resulting cube texture after heavy cold rolling and annealing is rather more well developed and stronger in the material with lower initial grain size. Subsequent coating of the tapes with CeO_2 buffer and superconducting YBCO layers has resulted in the attainment of critical current densities up to 0.2 MA/cm^2 at 77 K and zero field on tapes of 20 cm in length [29].

However, possibility of abnormal grain growth or secondary recrystallization and existence of deep thermal grooves at the grain boundaries can substantially interfere with the epitaxial growth of the buffer layers on these substrates. Therefore, suppression of these two phenomena would appreciably improve the conditions during subsequent buffer layer deposition.

Effect of micro-alloying on secondary recrystallization and grain boundary grooving

During prolonged annealing of the recrystallized tape at higher temperatures, the material may undergo both normal and abnormal grain growth (secondary recrystallization). Abnormal grain growth occurs due to the very fast growth of some grains by way of fast movement of grain boundaries. This may result in the destruction of the cube texture, accompanied by the development of a strong non-cube component, which deteriorates the desired final properties of the tapes. This phenomenon has been amply demonstrated by De Boer et al. [15] for pure Ni, starting from an annealing temperature as low as 600 °C (Fig. 12). They have further shown that abnormal grain growth in Ni can be effectively prevented by alloying with 0.1 at.% Mo. Interestingly, grains having more than 16° misorientation to the cube orientation are consumed during the normal growth of the cube oriented grains. They observed that the highly misoriented grains disappear while low angle grain boundaries between cube oriented grains remain quite stable. Therefore, normal grain growth is found to be beneficial for the development of a sharp cube component.

De Boer et al. [15] suggested two possible mechanisms that may be responsible for the inhibition of abnormal grain growth in Ni alloyed with Mo, Mn or W. The first one is a solute drag effect in which the solutes preferentially segregate around the high angle grain boundaries which are high energy sites. The grain boundary movement is thereby arrested at higher temperatures. Another mechanism that may be responsible for the inhibition of abnormal grain growth is the formation of small precipitates by Mo or W reacting with other impurities that may be present in Ni which can very efficiently pin the grain boundary movement even at higher temperatures.

Eickmeyer et al. [25] studied the effects of micro-alloying W and Mo with Ni (99.99 and 99.94 at.% purity) on thermal grooving during high temperature annealing. They have reported that whereas on the high purity nickel surface the grain boundaries are clearly attacked during annealing at 850 °C under hydrogen gas atmosphere, the extent of thermal etching or grooving of the grain boundaries in micro-alloyed low purity Ni tapes is either considerably less, or totally absent (Fig. 13). The beneficial effect of small concentrations of tungsten in Ni by way of increased resistance against grain boundary grooving also holds for higher alloy contents. The above authors have found that nickel alloyed with 5 at.% W reveals no grooved grain boundaries even after annealing at 1000 °C in hydrogen atmosphere for over 45 minutes.

Effect of micro-alloying on substrate strength

Since the YBCO superconductor coating on the substrate does not withstand a strain above 0.5% in compression and 0.2% in tension without degradation, the stress at low strains (e.g. 0.2% yield strength) is more critical for the substrate material in such applications [30]. Goyal et al. [26] have reported that Ni substrate with minor alloying additions of W and Fe is

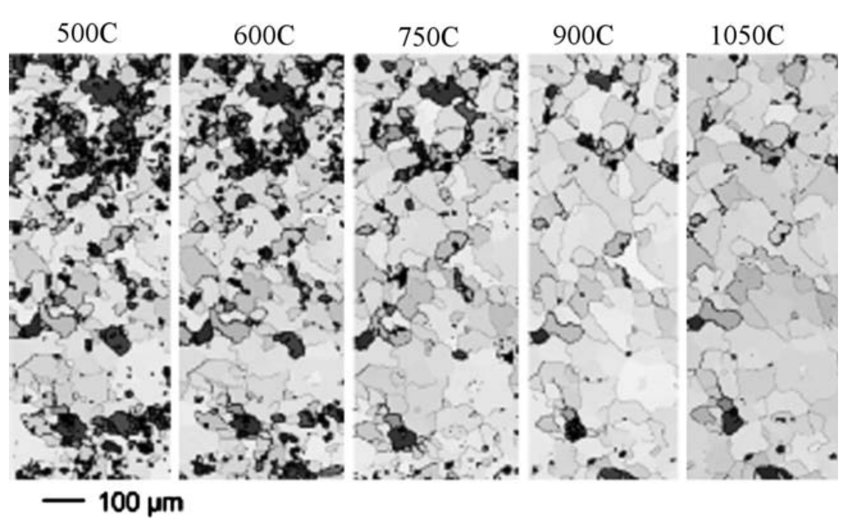


Fig. 12 Normal grain growth in Ni–0.1 at.%Mo. EBSD mapping of a sample area annealed successively at increasing temperatures for 30 min. The highly misoriented dark shaded grains disappear due to normal grain growth [© (2001), reprinted with permission from Elsevier] [15]

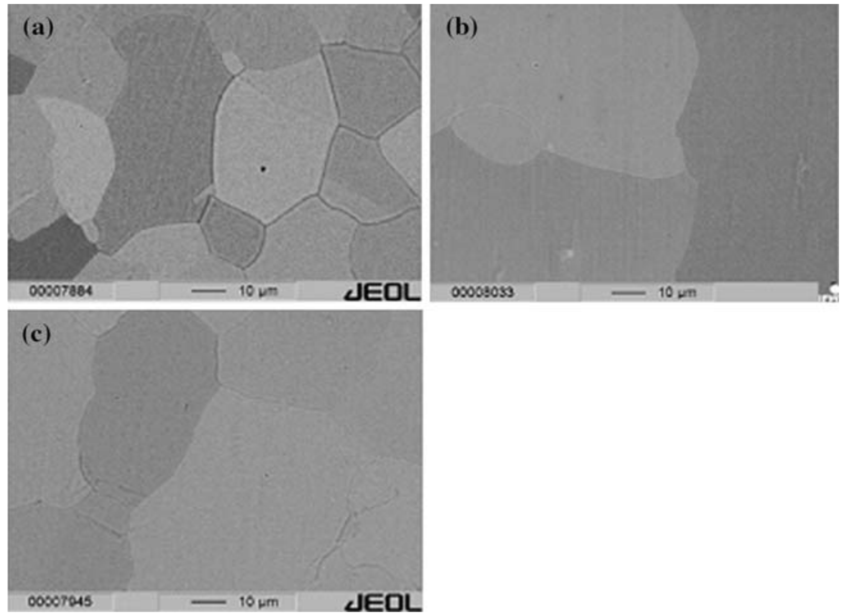


Fig. 13 Scanning electron micrographs of RABiTS™ tape surface to manifest effect of micro-alloying on grain boundary grooving: (**a**) Ni (99.99%), (**b**) Ni (99.94%) + 0.1 at.% W and (**c**) Ni (99.94%) + 0.1 at.% Mo [© (2001) IOP Publishing Ltd., reprinted with permission from the publisher and authors] [25]

significantly stronger than the pure Ni (99.99%) substrates. The average yield strength of Ni–3 at.% W–1.7 at.% Fe at 0.02 and 0.2% strain are 143 and 154 MPa, respectively, whereas pure Ni (99.99%) attains strength levels of only 40 and 58 MPa at the same strain levels. Rupich et al. [31] have reported a tensile strength of 178 MPa for a Ni–5 at.% W alloy at room temperature. This is much higher than the tensile strength of 34 MPa for a comparable pure unalloyed Ni substrate. At the operating temperature of 77 K (liquid N_2 temperature) for these substrate materials the yield strength of Ni–5 at.% W is more than four times higher than that for pure Ni, whereas the Young's modulus and proportional limit of elasticity are higher by a factor of 1.8 and 2.25, respectively. However, the strain-hardening of pure Ni is found to be greater than that of Ni–5 at.% W alloy (Fig. 14) [32]. Improvement of mechanical strength by micro-alloying is believed to be mostly due to solid solution hardening. Increase in strength of Ni because of solution hardening depends on the specific alloying elements via their atomic diameter mismatch with Ni. The relatively larger solute atom diameters of Mo (+11%), W (+12%), Nb (+16%) and Ta (+17%) [33] should lead to a much stronger effect as compared to the elements of the fourth period, such as Cr (+2%) or V (+7%) [25]. For a continuous reel-to-reel deposition process of the buffer and superconducting layers the substrates should have reasonably good high temperature mechanical strength. Unfortunately, the strengths of Ni-refractory metal tapes at the deposition temperatures of these layers have not been investigated to any significant extent so far.

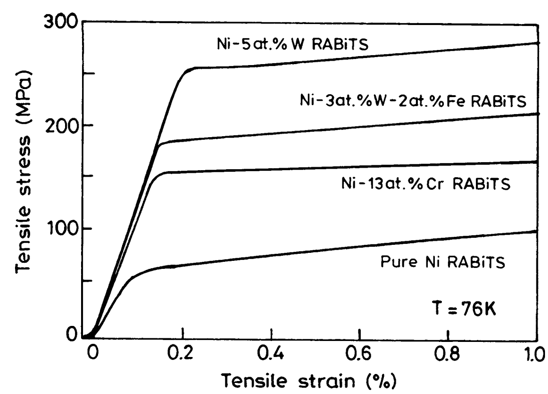


Fig. 14 Comparison of stress–strain curves at liquid N_2 temperature of pure Ni, Ni–13 at.% Cr, Ni–3 at.% W–2 at.% Fe and Ni–5 at.% W alloy RABiTS™ tapes [© (2003) IEEE, reprinted with permission from IEEE] [32]

Solution hardened Ni tapes for magnetic applications

Selection of alloying elements for suppressing the Curie temperature

An obvious consideration for using Ni as a substrate material for alternating current applications is that pure Ni is ferromagnetic with a Curie temperature of 627 K and a saturation magnetization of 57.5 emu/g at T = 0 K. Therefore, Ni is sufficiently ferromagnetic at the liquid N_2 temperature of 77 K. The ferromagnetism of Ni adds to the complexity of design of the high field magnets. In addition, the use of Ni as substrate in alternating current applications may lead to increased energy losses due to hysteresis loss in the substrate material. The actual energy loss per cycle of applied magnetic field H is given by the area enclosed within the magnetization loop $M(H)$, and this varies with the magnetic "hardness" of the ferromagnet. Keeping these considerations in view it is obviously desirable to develop suitable alloys with reduced ferromagnetism which can be successfully biaxially textured at the same time.

The potential alloying elements to reduce ferromagnetism of pure Ni are Cr, V, Si, Al and Ti (Fig. 15) although the solubility of Ti, Al and Si in Ni is not high enough to suppress the Curie temperature below 77 K [34]. Cr and V, however, are mostly preferred as alloying elements for such applications since only Ni–Cr and Ni–V alloys are found to develop a strong cube texture against the general observed tendency that the cube texture intensity goes down drastically with higher alloying content.

Thompson et al. [35] have studied a series of Ni–Cr alloys with increasing Cr content up to 13 at.%. They found that mass magnetization $M(T)$ along with the Curie temperature decrease steadily with increasing Cr content, as can be seen from Fig. 16. The reported values of hysteresis energy loss/cycle of a Ni–7 at.% Cr alloy investigated by them, are 1400 and 2000 erg/cm^3 under a coercive field of 4 Oe at 77 K with the applied magnetic field parallel and perpendicular to the plane of the foil, respectively (Fig. 17a). In contrast, the hysteresis loss/cycle of pure biaxially textured Ni is found to be 1.82×10^4 erg/cm^3 with the field perpendicular to the plane of the foil under a coercive field of 7 Oe (Fig. 17b). For coated conductor applications, materials will be used either at large magnetic fields or to generate such magnetic fields. Therefore the field dependent magnetization is a very important consideration. Ni–Cr alloys with lower Cr content, typically with 7–9 at.%, are found to be ferromagnetic, whereas Ni–11 at.% Cr is paramagnetic with significantly lower magnetization even comparable to that of annealed 304 type stainless steel. Ni–13 at.% Cr alloy exhibits even lower magnetization at the temperatures of 40 and 77 K, as can be seen from Fig. 18.

Development of texture in highly alloyed Ni substrates

As has been pointed out earlier, the basic requirement for substrate materials for coated conductor application is the development of a strong cube texture. Since

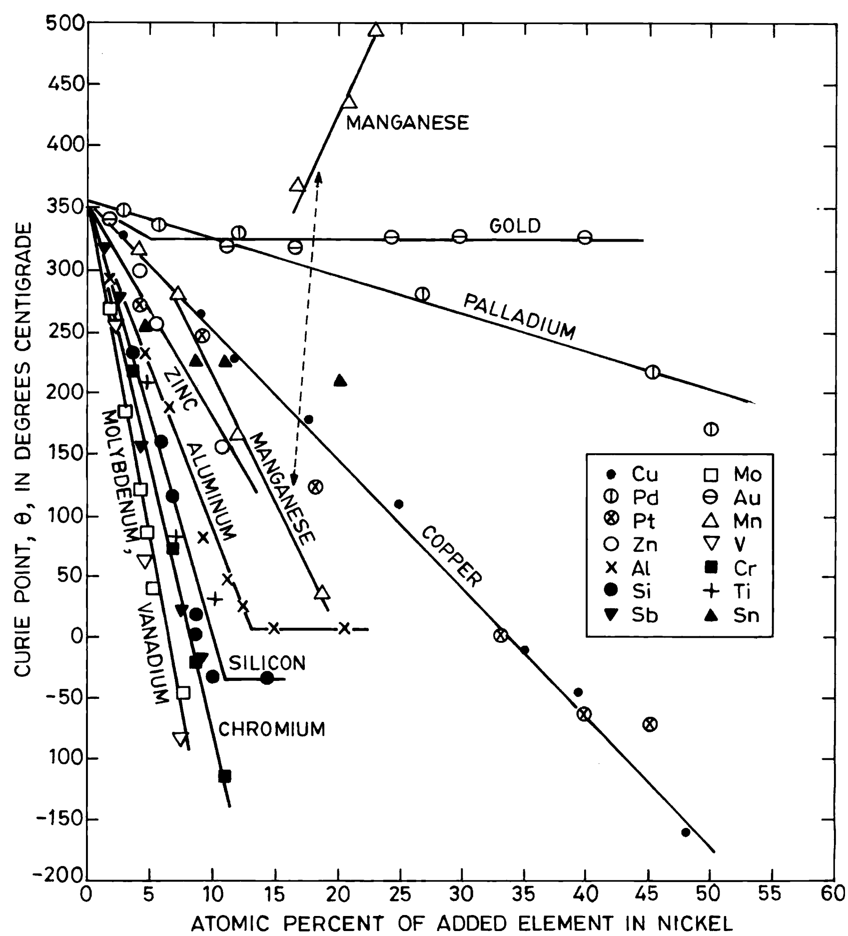


Fig. 15 Dependence of the Curie temperature on different alloying elements [34] (Reprinted with permission from the NRC Research Press)

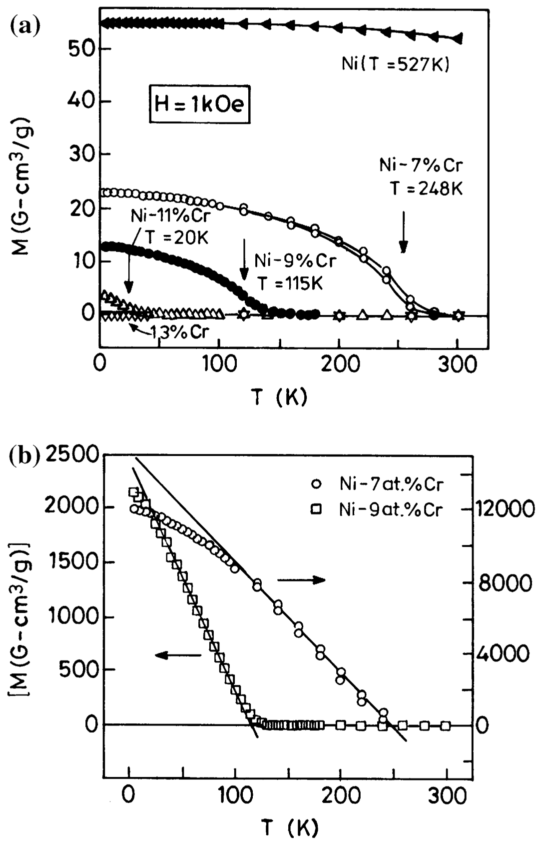


Fig. 16 (**a**) The Mass magnetization of Ni–Cr alloys vs temperature measured in an applied magnetic field H = 1 KOe applied parallel to the plane of the foils. Nominal Cr concentration is given in at.% and (**b**) A plot of M^3 vs. temperature T. Straight lines show the extrapolation to M = 0 used to define the Curie temperature [© (2001), reprinted with permission from Elsevier] [35]

the alloying addition necessary for non-magnetic substrates is generally on the higher side, it is therefore important to ensure that the higher alloying content

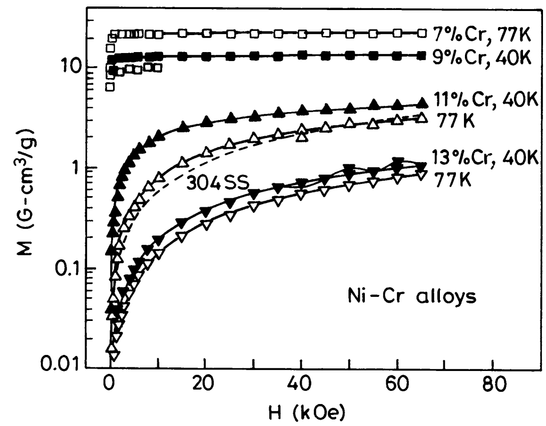


Fig. 18 The field dependence of the magnetization of different Ni–Cr alloys [© (2001), reprinted with permission from Elsevier] [35]

does not impair the cube texture of the substrate materials. De Boer et al. [34] have studied several Ni based systems to determine the preferred alloying elements which can promote a sharp cube texture even when present in larger enough amounts. According to their studies, non-magnetic alloys such as Ni–13 at.% Cr and Ni–9 at.% V can develop a strong cube texture after recrystallization (Fig. 19). In fact, very sharp cube texture component has been reported by them in Ni–13 at.% Cr and Ni–9 at.% V alloy recrystallized at 900 °C. This has been confirmed by measuring misorientation distribution using the EBSD technique (Fig. 20a). The intensity of the cube texture improves with increasing annealing temperatures until the onset of abnormal grain growth at temperatures exceeding 950 °C (Fig. 20b). Thompson et al. [35] also reported that Ni–Cr alloys can be thermo-mechanically processed to develop a very strong cube texture component up to a Cr content of 13 at.%. The (111) X-ray

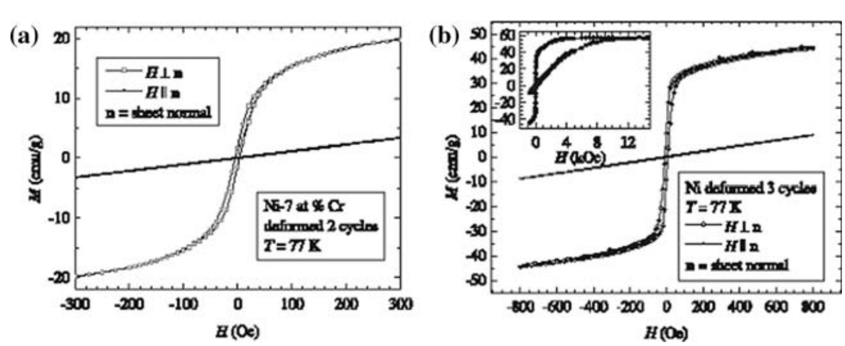


Fig. 17 (**a**) Magnetization loops (expanded scale) for a deformed $Ni_{93}Cr_7$ foil at 77 K, with magnetic field applied parallel or perpendicular to the plane of the foil. The magnetization is relatively reversible, with limited hysteretic energy loss/cycle and (**b**) magnetization loops for a deformed biaxially textured Ni foil at 77 K, with magnetic field applied parallel or perpendicular to the plane of the foil inset: magnetization in large fields [© (2001), reprinted with permission from Elsevier] [35]

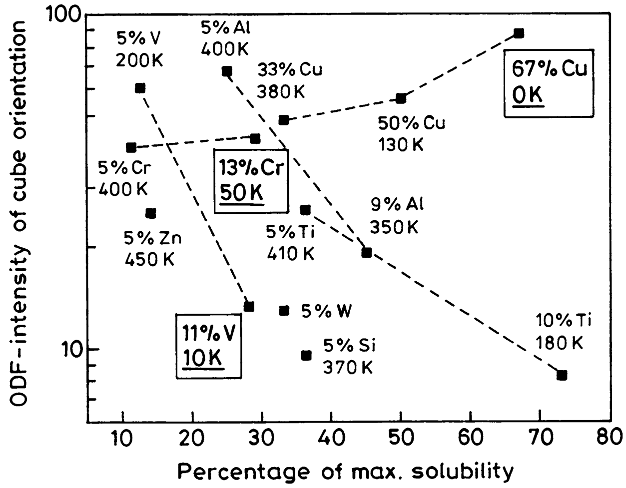


Fig. 19 Dependence of cube intensity of several Ni-alloys on the alloying content scaled to the absolute maximum solubility. The legends on the data point stands for the absolute content in at.% and the estimated Curie temperature (Reprinted with permission from NRC Research Press) [34]

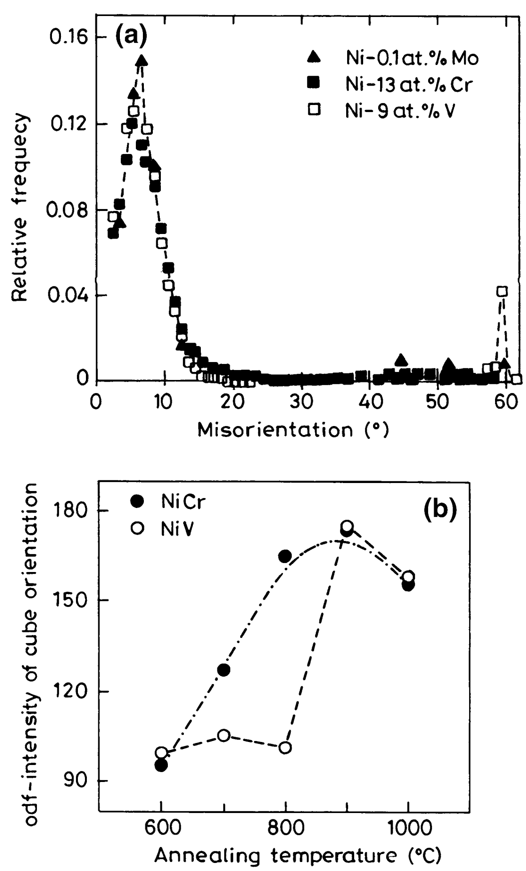


Fig. 20 (a) Texture development of the alloys Ni–9 at.% V and Ni–13 at.% Cr with annealing temperature and (b) misorientation distribution of different Ni alloys (Reprinted with permission from NRC Research Press) [34]

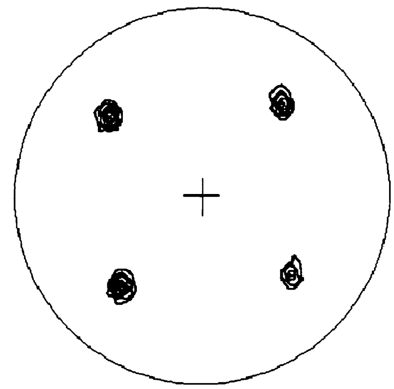


Fig. 21 (111) pole figure of a Ni–13 at.% Cr alloy substrate annealed at 1,050 °C for 2 h [© (2001), reprinted with permission from Elsevier] [35]

pole figure of a Ni–13 at.% Cr alloy annealed at 1050 °C for 2 h has been shown in Fig. 21. It can be noted that all the four crystallographically equivalent peaks of {111} corresponding to the {100} ⟨100⟩ cube orientation are present in the pole figure indicating the presence of a very sharp cube texture.

The main difficulty of using Cr or V as an alloying element in Ni is that both these elements are very prone to oxide formation, especially at the buffer/YBCO deposition temperature of around 700–900 °C. The formation of Cr/V oxide is detrimental to the epitaxial growth of the buffer and YBCO layers on the Ni alloy substrates. The alloying elements in Ni alloy substrates have different affinities towards oxygen and the oxidation behavior is also strongly affected by the environmental conditions, surface finish, grain size, sample thickness, size and alloy composition etc. It has been found that very often the simple kinetic rate equation for oxygen diffusion is not followed and the thickness of the scale changes in a very complex way [36]. Tuissi et al. [37] have studied the oxidation rate of Ni–Cr–W and Ni–Cr–V alloys by thermogravimetric analysis (see Fig. 22). As compared to the binary alloys of Ni–Cr and Ni–V, the ternary alloy systems Ni–Cr–W and Ni–Cr–V showed substantially lower weight gain (~ <0.03% g/cm^2). It may be noted in the figure that pure Ni shows oxidation resistance better than both Ni–Cr and Ni–V alloys. The weight gain for the Ni–Cr sample can certainly be attributed to the incomplete formation of protective Cr_2O_3 layer. Optical microscopy studies on Ni–Cr–W and Ni–Cr–V alloys have revealed more uniform scale formation in case of the Ni–Cr–W alloy in contrast to the Ni–Cr–V alloy. Both Ni–Cr and Ni–V alloy systems show non-uniform oxidation, principally at the grain boundaries where Cr_2O_3 grows easily (see Fig. 23). It appears that addition of W improves the oxidation resistance of Ni–Cr alloys.

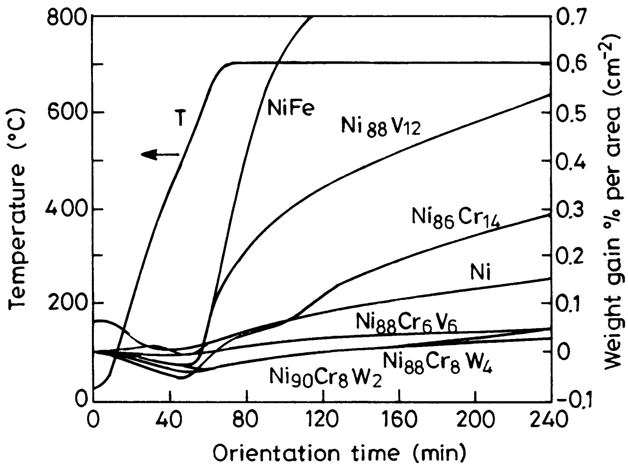
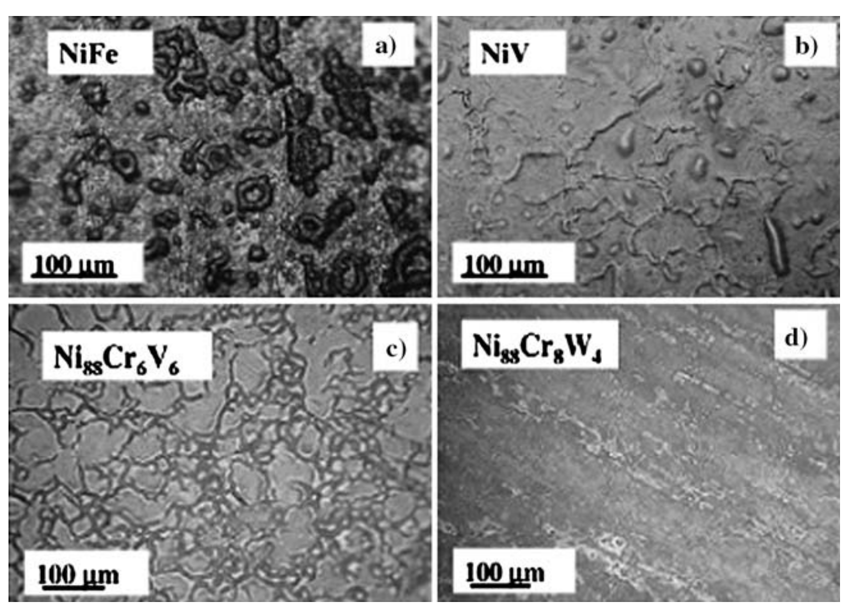


Fig. 22 Oxidation behavior of pure Ni and different Ni base alloys [© (2002), reprinted with permission from Elsevier] [37]

A recent approach is to produce textured NiO on the surface of Ni–Cr alloys through surface oxidation epitaxy [38]. Boffa et al. [39] have found that it is possible to grow (001)-oriented NiO buffer layer on cube textured substrate through controlled oxidation during the annealing process itself. This has been further established by Lockman et al. [40] who have reported in detail the oxidation behavior of Ni–10 at.% Cr and Ni–13 at.% Cr alloys. They found that air oxidation of Ni–Cr foils at 1,050 °C can produce a smooth, highly textured NiO surface oxide which can efficiently act as the first buffer layer in a superconducting coated architecture.

Goyal [41] have suggested a composite architecture for the possible RABiTS^{TM} tapes to overcome the surface oxidation problem. Such composite substrates essentially have an outer layer which will develop strong cube texture after annealing and will be more resistant to oxide formation. The core of the substrate will consist of an alloy stronger than the outer layer. Sarma et al. [42] also have adopted a similar strategy. They have investigated a composite substrate material Ni–3 at.% W (outer layer)/Ni–10 at.% Cr–1.5 at.% Al (core) to overcome the surface oxidation problem. Ni–3 at.% W alloy is used as the outer layer since Ni–W alloys develop strong cube texture up to 5 at.% W and they also possess good oxidation resistance. On the other hand, Ni–10 at.% Cr–1.5 at.% Al is used as the inner core as Cr can suppress the ferromagnetism beyond 77 K as discussed earlier. The outer layer and inner core are so chosen that the strength levels do not differ to a significant extent to prevent inhomogeneous deformation which may lead to cracking. Al is used in the substrate material to produce Al_2O_3 by controlled internal oxidation to increase the strength level further. EBSD studies have shown that the substrate develops a strong cube texture when annealed at 950 °C after heavy cold deformation. Majority of the grain boundaries are reported to have misorientation angle less than 10° with maximum in the misorientation distribution close to 7° (low-angle grain boundaries) as shown in Fig. 24. The composite showed reasonably good tensile properties with yield strength exceeding by a factor of four when compared to pure unalloyed Ni and about 40 MPa more than that of Ni–10 at.% Cr–1.5 at.% Al (see Fig. 25). There is reasonable conformity in the strength levels of the inner core and the outer core. Another study involving a

Fig. 23 Optical Micrograph of oxidized surfaces of (**a**) Ni–Fe, (**b**) Ni–V, (**c**) $Ni_{88}Cr_6V_6$, (**d**) $Ni_{88}Cr_8W_4$ [© (2002), reprinted with permission from Elsevier] [37]



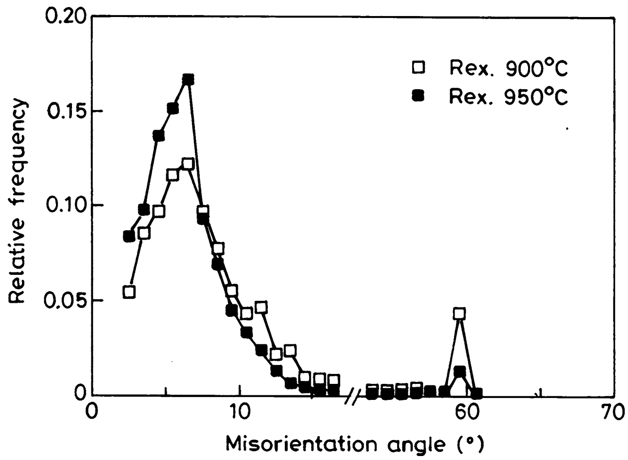


Fig. 24 Grain boundary misorientation distribution of the Ni–3 at.% W/Ni–10 at.% Cr–1.5 at.% Al composite after recrystallization at 900 and 950 °C for 30 min [© (2003), reprinted with permission from Elsevier] [42]

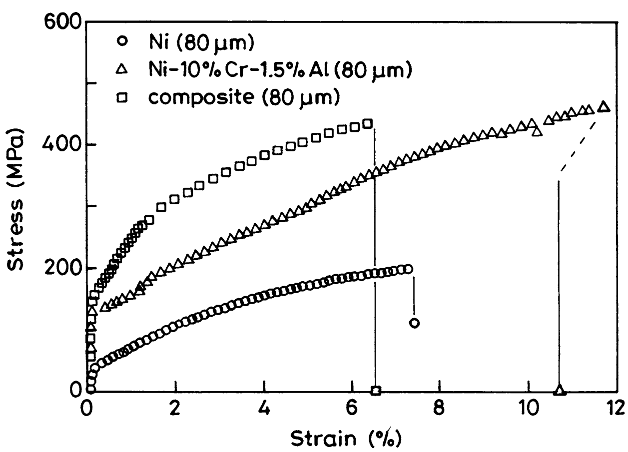


Fig. 25 Stress–strain curves of the recrystallized substrates [© (2003), reprinted with permission from Elsevier] [42]

composite of configuration Ni–4.5 at.% W/Ni–15 at.% Cr has also been reported by these authors [43]. The significant point in their study is that a highly alloyed material at the core of the substrate, which does not develop cube texture, does not inhibit the development of a sharp cube texture at the outer layer. Since the final tape thickness is quite small (~40 μm) in their study, with only a few grains expected in the thickness direction, this observation is important from the point of view of usability of such multilayered substrates. Thus, laminated or multilayer architecture appears to be a technologically very attractive solution although it demands further and dedicated effort at its current level of development.

Nast et al. [44] employed an electroplating technique to make composite substrate tapes of architecture Ni/Ni–W and Ni/Ni–Cr with better oxidation resistance. The original substrates were made from alloys of Ni–Cr and Ni–W which were cold rolled to more than 98% thickness reduction and annealed at different temperatures and for different lengths of time to produce a strong cube component. These substrates were electroplated with nickel to develop a very thin nickel layer followed by a post annealing treatment at 700 °C to develop cube texture epitaxially in the thin nickel layer.

Recent developments

The proper processing route to produce the materials for use as substrates is one of the major issues as far as the practical applications are concerned. So far the alloys to be used as substrates have been prepared by conventional melting and casting route and to a much lesser extent by the powder metallurgy (P/M) techniques. Unfortunately, there have been very few serious studies available to establish the supremacy of one process over the other. However, it might appear to be quite reasonable that since the purity of Ni is one of the most important issues for developing a strong cube component, the powder metallurgy technique may offer a stiff challenge to the melting route which inadvertently introduces some deleterious impurities. Though it has been reported in literature that alloys prepared through melting and casting route develop sharp cube texture after cold deformation and annealing, at the same time the requirement of strict quality control during the whole procedure cannot be overstated. In addition, the initial grain size can be much better controlled in the P/M technique by using very fine powder and of course, the processing temperatures encountered are expected to be much lower in the case of the powder metallurgy route.

Recently Lim et al. [45] have made a comparative study of Ni substrates prepared through powder metallurgical route and also the melting and casting route. They have ensured a very high level of purity in the latter through Plasma Arc Melting (PAM) method such that it is comparable to that in case of the powder metallurgy route. They have found that the intensity of the cube component of P/M Ni substrate after cold rolling and annealing is not very sensitive to temperature of annealing as compared to the Ni substrate prepared through the melting and casting route. Sharp cube component develops at a lower temperature for P/M Ni substrate and this does not sharpen very much upon annealing at higher temperatures. On the other hand, the sharpness of the cube texture component increases with increasing temperature for the cast Ni substrate until the onset of secondary recrystallization which leads to a marked deterioration of the cube texture intensity. Both the intensity and stability of the cube component are found to be higher in the P/M Ni-substrate having a smaller grain size as compared to the PAM Ni substrate.

Bhattacharjee et al. [46, 47] have studied the possibility of preparing mechanically strong substrates with sharp cube texture through the conventional powder metallurgy route. The process comprises unidirectional pressing and sintering operation to get a dense compact suitable for subsequent heavy deformation. Their investigation has been focused upon developing single layer as well as multilayer substrate tapes through powder metallurgical processing. The multilayer architecture that were studied for that purpose is Ni/Ni–5 at.% Mo and Ni/Ni–5 at.% W. The idea behind adopting such multilayer configuration is that the upper layer of pure Ni will provide the sharp cube texture after cold rolling and annealing and the base layer of either Ni–5 at.% W or Ni–5 at.% Mo will provide the necessary strength in the annealed conditions. The two powder layers were separately filled in a die of rectangular cross section, and then pressed and sintered. These compacts were subsequently cold rolled to ~95% reduction in thickness with the final thickness lying between 120 and 150 μm. These cold rolled tapes were then annealed at different temperatures. The typical cross-sectional micrographs of a tape in cold rolled and annealed condition have been shown in Fig. 26. It can be noticed from the figure that the two constituent layers have maintained their individual identity throughout the whole processing cycle and the interface between the two different layers is sharp and continuous.

Lee et al. [48] have investigated the possibility of employing cold-isostatic pressing (CIP) followed by sintering and cold rolling for preparing the initial materials. These substrates develop sharp cube texture intensity after annealing at 1,000 °C. Ji et al. [49] have studied the difference in cube texture intensity in uniaxial and CIP compacts prepared from very pure Ni (~99.99%). They have observed that the sharpness of the cube texture after annealing at the same temperature and for the same time is significantly more in the latter case. The difference is attributed to the fact that materials which are prepared through CIP have higher and more uniform density as compared to the uniaxially pressed compacts. They have concluded that a more homogenous and denser compact will give rise to a highly homogenous microstructure after sintering which will increase the sharpness of the cube texture after cold rolling and annealing.

As an alternative to the melting route involving casting, cold-rolling and annealing, a novel process of electrodeposition of Ni under applied magnetic field has very recently been reported [50, 51]. The magnetic field, which is 0.4 T in these works, is applied concurrently during electro deposition to develop sharp cube texture intensity. However, the main problem with electrodeposition is that often the deposited film has either pinholes or corrosion pits which deteriorate the mechanical properties of such tapes. In addition, electrodeposition may not be a very suitable process for quick production of long length of tapes due to the sluggish nature of the electrochemical deposition.

Wang et al. [52, 53] have investigated a process based upon ion beam structure modification of rolled Ni tapes for sharp cube texture development. The Ni tapes are rolled to 98.7% in thickness reduction and subsequently exposed to ion beam irradiation replacing the annealing stage. The major parameter that affects the sharpness of the cube texture and surface morphology after irradiation is the inclination of the beam with the specimen. Local heating effect due to ion beam bombardment is thought to play a significant role in the development of the cube texture. However, it is quite possible to damage specimens through creation of point defects during ion beam bombardment. In addition, often the surfaces of the ion beam bombarded specimens contain an amorphous layer due to excessive irradiation which is a very common problem

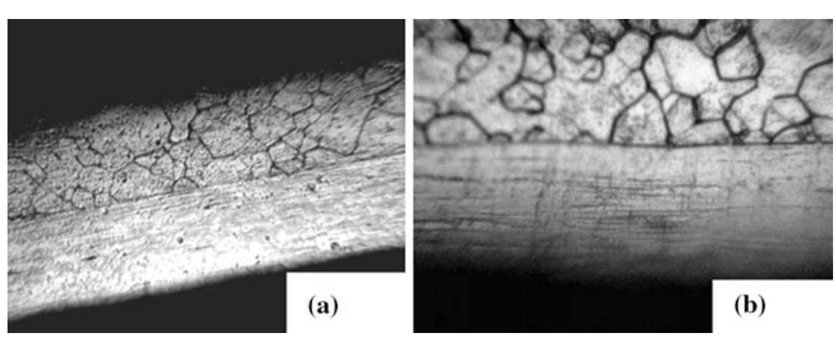


Fig. 26 Cross-sectional micrographs of (**a**) Ni/Ni–5at.% W and (**b**) Ni/Ni–5 at.% Mo annealed at 800 °C for 1 h [47]

encountered in TEM thin foil specimen preparation through Ion beam thinning. Although no such problem has been cited by these authors, still, the utility of replacing an industrially established heat treatment process like annealing by ion beam structural modification to produce long length of tapes is questionable.

Eickemeyer et al. [54] have investigated the possibility of employing drawing operation through rigid dies as well as through freely rotating rolls as an alternative processing route for preparing such substrates. A comparison has been made between these Drawing Assisted Biaxially Textured Substrate (DABiTS®) tapes and RABiTS™ tapes (see Fig. 27) in terms of the intensity of cube texture developed after annealing at the same temperature and for the same duration. Interestingly, it has been observed by these authors that drawing is a more effective forming operation than rolling. Even at intermediate stages of deformation followed by annealing at the same temperature and for the same duration, the intensity of cube texture is found to be more in case of the tapes prepared by drawing as compared to the tapes prepared by rolling. Amongst the two drawing operations studied by these authors, drawing through rigid dies appears to be superior to drawing through loose rolls.

Future prospects

The second generation coated conductor technology based on the RABiTS™ method at its current state of development appears to be a potentially attractive route for using HTS coated tapes for a range of technological applications, particularly, in the power generation and distribution systems such as power transmission cables, electric motors and generators, transformers, synchronous condensers and fault current limiters. In comparison to multifilamentary HTS wires which currently dominate the superconducting wire market, the RABiTS™ tapes are projected to involve a substantially lower fabrication cost and this has been one of the key driving forces for accelerated research in coated conductor technology [55]. One estimate puts the fabrication cost of HTS wires involving the RABiTS™ method to two to five times lower than first generation multifilamentary wires [56]. Wires based upon the RABiTS™ method have already penetrated the HTS wire market and the demand is expected to grow in near future [56].

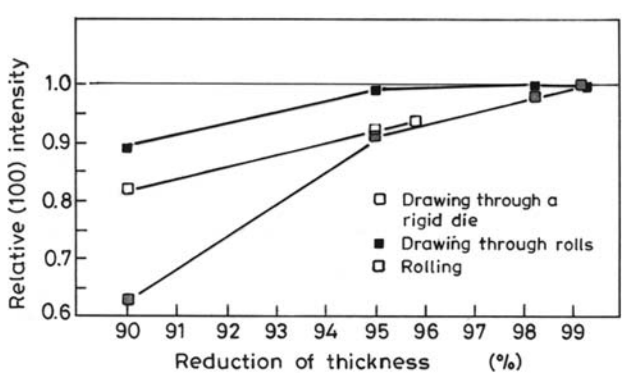


Fig. 27 Relative X-ray intensity of (100) reflections of nickel tapes after different cold forming procedures and subsequent recrystallization at 600 °C for 30 min [© (2004), reprinted with permission from Elsevier] [54]

Summary

Biaxially textured Ni base tapes prepared by the RABiTS™ method have been established as techno-economically viable solution for preparing substrate materials for coated superconductor applications. However, proper alloy design is the key issue to their successful usage. The properties of these alloy substrates are to be tailored in such a way that these substrates develop very sharp cube texture component after recrystallization with good texture stability and reasonably good mechanical strength at high temperatures. At its current state of development the most potential alloying elements are W and Mo at the micro-alloying level and Cr at higher levels of alloying for non-magnetic applications requiring a very low hysteresis loss. Another technologically useful method appears to be the development of Ni base functionally graded composite materials. Of late a number of alternative processing routes have been put under investigation, but further research is necessary to establish their supremacy and industrial scalability.

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