Apparent anisotropy effects of upper critical field in high-textured superconducting Nb-Ti tapes

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Abstract. Analysis of the voltage-field characteristics of superconducting niobium-titanium tapes is the objective of this work. We used both the original cold-rolled and heat-treated tapes, whose structure and texture we had studied in detail. We observed anisotropy in the upper critical field and the reduction of the transition width with increasing angle between the plane of tape and the direction of the magnetic field. In addition, we have registered a significant difference of the upper critical field values, obtained from the voltage-field characteristics and field dependence of the pinning force. Considering our results, we suggest an explanation of the observed features within the macro-inhomogeneity model.

1. Introduction

One of the key problems of applied superconductivity is electrodynamics of high pinning superconductors (HPSC) [1]. Usually the nano-sized defects (the so-called “pinning centers”) are formed in HPSC during a manufacturing process. Such defects cause high current-carrying capacity and, on the other hand, they often lead to the broadening of transitions and to critical current anisotropy against magnetic field and major directions (e.g. as rolling direction, direction of broaching etc.), which is noticeable even in body-centered-cubic Nb-Ti material [2]. Obviously these features should be considered in device designing process.

It is well known that Nb-Ti is suitable and reproducible material for application and study of HPSC electrodynamics [1-3]. A complex structure of thin ribbon-like α-Ti precipitates is formed in commercial multi-filamentary Nb-Ti wires [4]. In contrast, the cold-rolled Nb-Ti tape is simpler object and thus allows investigating of native effects related to the variations of the pinning centers system. Therefore, this paper analyses changes in material microstructure and voltage-field characteristics (VFC) of cold-rolled and heat-treated Nb-Ti tapes.

2. Methods and samples

Two kinds of samples were investigated. First series of samples was fabricated from long-length 80-mm width and 10-μm thick cold-rolled tape of the Nb-50wt.%Ti, manufactured at the metallurgic factory in 1978 [2]. The second one was made from the same tape, and additionally annealed in high

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vacuum at 385°C during 25 hours to precipitate non-superconducting α-Ti nano-particles. We expected that such heat treatment should change pinning system significantly.

Flat samples with bridge geometry along the rolling direction were made by laser cutting (the bridge was 1.0 mm in width and 6.7 mm in length). The VFCs of the samples were obtained by direct transport method. The sample was fixed on a rotating probe, which allowed them to rotate in an external field and change the angle θ between the sample plane and the field direction. The current direction was perpendicular to the field at all times. Measurements were carried out in liquid helium in the superconducting magnet with field up to 13 Tesla.

The criterion for the upper critical magnetic field value ($H_{c2}$) was half of the resistance in the normal state ($\rho_n$) and the width of the transition was determined by the difference in field values at 0.9 and 0.1 fracture of the $\rho_n$. VFCs were studied at various angles θ to investigate the $H_{c2}$ anisotropy.

Titan S-TWIN 80-300 (FEI) microscope was used to carry out SEM investigation and to determine the average size of the grains. The detailed texture analysis was performed using Bruker D8 Discover diffractometer with Cu-Kα radiation and was verified on Kurchatov Synchrotron Radiation Source. The set of pole figures were used to construct the orientation distribution function (ODF) [11].

3. Results and discussion

3.1. Micro-structural and textural studies

Typical SEM images of cross-sections of cold-rolled and heat-treated tapes are shown in Figure 1. The Nb-Ti grains resemble stacks of thin long ribbons extended along the rolling direction (RD). The main grains sizes in all directions were determined by averaging of statistical data from several dozen of images. Calculated main grains sizes were: 65 nm in the direction of normal to the plane of the tape; 0.4 μm in the transverse direction; and more than 1 μm in the RD. Pronounced grain boundaries were observed only in parallel to the plane (Fig. 1). Distributions of Nb-Ti grain sizes were similar in cold-rolled and heat-treated tapes.

Figure 2 shows the pole figures of Nb-Ti phase for the reflections (110), (200) and (222) in the cold-rolled tape. Analysis of ODF showed almost uniform distribution of the orientations from {100} <110> to {112} <110>, which is typical for the rolled β-Ti alloys [5]. Heat treatment does not change the texture of the main Nb-Ti phase. Additional α-Ti reflections arise on X-ray patterns.

![Figure 1. SEM cross-section images of samples: a) cold-rolled, rolling direction is horizontal; b) cold-rolled, rolling direction is perpendicular to the image plane; c) heat-treated, rolling direction is horizontal; d) heat-treated, rolling direction is perpendicular to the image plane. Black spots are clearly visible on images of heat-treated samples and were interpreted as α-Ti precipitates.](image)

3.2. Voltage – field characteristics

Figure 3 shows the VFCs for cold-rolled and heat-treated samples at the applied field orientated perpendicular to the tape ($\theta = 90^\circ$) and in plane of the tape ($\theta = 0^\circ$). The differences in the $H_{c2}$ values for such orientations are about 0.3 T and 0.2 T for the cold-rolled and heat-treated samples. The anisotropy of $H_{c2}$ for Nb-Ti tapes (processed with complicated thermo-mechanical treatment) was also...
observed in [3] where authors suggested an explanation of the effect: the α-Ti precipitations form the weak links between the Nb-Ti grains due to their elongated shape and a small thickness comparable to the coherence length $\xi$. Theory [6] predicts that anisotropy arises due to weak links. This assumption is not supported by our results, as we have observed the most pronounced anisotropy in cold-rolled samples, which had very small amount of α-Ti precipitations.

Another possible source of anisotropy is high degree of texture. Indeed, in single crystals with a cubic symmetry one can expect variations in physical properties in directions [100], [110], and [111]. Such anisotropy of $H_{c2}$ was observed in the niobium single crystal [7]. According to [7], the $H_{c2}$ increases in following sequence of directions: [100] → [110] → [111]. From the pole figure (222) (Fig. 2) it is clear that at $\theta = 43^\circ$ the majority of the grains are oriented in the [111] direction, and one can expect the maximum value of $H_{c2}$ at this angle. However, $H_{c2}$ reaches the maximum value when the field is in the plane of the tape ($\theta = 0^\circ$) (see Table 1). This means that apparent $H_{c2}$ anisotropy does not depend on crystalline orientation of grain in Nb-Ti tapes.

Table 1. $H_{c2}$ and transition width at different angles $\theta$ for two cold-rolled samples.

| Sample# | Measuring current, mA | $H_{c2}$, T (Transition width, T) |
|---------|------------------------|----------------------------------|
|         | $\theta = 0^\circ$     | $\theta = 43^\circ$ | $\theta = 90^\circ$ |
| 1       | 5                      | 11.89 (0.434)          | -                 | 11.58 (0.279) |
|         | 5                      | 12.06 (0.363)          | -                 | -               |
| 2       | 1                      | 12.10 (0.387)          | 11.75 (0.225)     | -               |
|         | 0.1                    | 12.11 (0.354)          | 11.75 (0.195)     | -               |
|         | 0.01                   | 12.12 (0.347)          | 11.74 (0.189)     | -               |
|         | 0.001                  | 12.12 (0.334)          | 11.74 (0.183)     | -               |

Finally, to analyse the influence of the measuring current on $H_{c2}$, we carried out series of measurements using different currents in the range between 1 μA to 5 mA (Table 1). Both the $H_{c2}$ and transition width values do not depend significantly on the current, so we suggest that the observed anisotropy is directly linked to anisotropy of $H_{c2}$ rather than any other kind of anisotropy (e.g. critical current anisotropy).
Figure 3. On the left: The magnetic field dependence of pinning force $F_p$. On the right: The magnetic field dependence of the normalized resistance (VFCs at measuring current 5 mA). The legend shows symbols indicating the orientation of the field and the type of samples.

Figure 3 also shows the magnetic field dependences of the pinning force $F_p$ ($F_p$ calculated as $I_c*B$, where $I_c$ was determined by a standard criterion of $1 \mu V/cm$). Extrapolation of these curves into a higher field shows that in the absence of inflections the pinning force should equate to zero at the field above 10.7 T.

All these features can be explained by the macro-inhomogeneity model [8] where the length of an electron’s free path is locally reduced at the Nb-Ti grain boundaries, which leads to reduction of the coherence length $\xi$ and increase of $H_{c2}$. The similar microscopic mechanism determines the pinning in the cold-rolled tape at the field below ~10.7 T [9]. Above this field only the Nb-Ti grain boundaries form percolating superconducting paths; while significant part of the Nb-Ti grain matrix is not superconducting. This explains the lack of dependence of $H_{c2}$ anisotropy on crystallographic orientation of grains.

From this point of view Nb-Ti tape in the high field can be roughly represented as a stack of superconducting films (grain boundaries) with thicknesses of about $\xi$. The distance between them is equal to the mean grain thickness $d$ (~65nm) which is significantly larger than $\xi$ (~5 nm). Therefore the films are not coupled, which is in contrast with the model [3]. So observed anisotropy $H_{c2}$ is similar in nature to the well-known $H_{c2}$ anisotropy observed in thin superconducting films. As seen from Figure 1 the $\alpha$-Ti precipitates are concentrated basically at the grain boundaries, violating their flatness, and reducing the anisotropy. As a result, the anisotropy is more pronounced in cold-rolled rather than heat-treated tape. This is also consistent with the experimental results of [3], where authors observed the growth of anisotropy with the increasing intensity and degree of rolling.

The macro-inhomogeneity model implies that there are at least two superconducting components in Nb-Ti tape: the grain boundaries and the grain bodies, which have different average values of $H_{c2}$. It should be noted that the method used to determine $H_{c2}$ does not give the “true” $H_{c2}$ value for grain boundaries because they are shunted by grain bodies. We cannot calculate correctly the shunting effect, since a magnetic field dependence of grains resistance as well as statistical dispersion of $H_{c2}$ in the grains are unknown. Quantitative estimation of the relationships between $H_{c2}$ values of grain bodies and grain boundaries is complicated task which is beyond the frame of this work.
The macro-inhomogeneity model also explains the augmentation in the transition width with \( \theta \) reduction (see Table 1). Indeed, there is dispersion in the grain boundaries orientation in our samples (see Figure 1). In accordance with the angle dependence of \( H_{c2} \) for the non-connected stack of thin films [10] the derivative \( dH_{c2}/d\theta \) increases quickly with the angle reduction. So for the fixed dispersion \( \delta \theta \) (i.e. fixed misorientation degree of grain surfaces) corresponding dispersion of the upper critical field \( \delta H_{c2} \) increases with decreasing of angle \( \theta \).

4. Conclusion
In this study the upper critical field anisotropy in Nb-Ti cold-rolled and heat-treated tapes was observed by the direct transport method. The apparent anisotropy is not related to the crystallographic texture and has been observed in both kinds of samples. It was shown that this anisotropy is also not directly related to \( \alpha \)-Ti precipitations. The extrapolated upper critical field obtained from the field dependence of pinning strength is of about 1 Tesla lower than the one derived from voltage-field characteristics. The transition width decreases with the increasing angle between the tape and the direction of the magnetic field. All these features can be explained by the macro-inhomogeneity model. It assumes that in the high magnetic field near \( H_{c2} \) only boundaries of Nb-Ti grains are in superconducting state. So Nb-Ti tape can be roughly represented as a stack of thin non-coupled superconducting films with thicknesses of about the coherence length.

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