Features of microstructure and magnetic flux dynamics in superconducting Nb-Ti tapes with strong anisotropic pinning

Sergey Shavkin*, Valentin Guryev, Vitaly Kruglov, Alexey Ovcharov, Igor Likhachev, Alexandre Vasiliev, Alexey Velizhanin, and Yan Zubavichus

National Research Centre “Kurchatov Institute”, Kurchatov sq. 1, 12318, Moscow, Russia

Abstract. Results of micro-structural, phase and texture analysis of Nb-Ti tapes and results of magnetic flux dynamics fixed by Magneto-Optical Imaging (MOI) method are presented in order to investigate origin and consequences of the anisotropic pinning model proposed, and implemented earlier experimental results including Guided Vortex Motion (GVM) phenomenon.

1 Introduction

All practically used low-temperature (conventional) superconducting materials including them with the cubic lattice structure (Nb-Ti alloy, Nb₃Sn compound, etc.) demonstrate pronounced anisotropy of the electromagnetic superconducting properties, caused by the directional nature of manufacturing processes (rolling, drawing, etc.) which lead to formation of the non-isotropic field of natural or artificial pinning centers and hence to anisotropic current-carrying capacity [1, 2].

The macroscopic electrodynamics of such superconductors with anisotropic (i.e. depending on the directions of a magnetic field and Lorentz force) pinning was proposed phenomenologically, on the basis of empirical studying of critical Lorentz forces. Interaction of the vortex structure (magnetic flux) with the anisotropic complex of pinning centers in the superconductor is described by a global potential well which depth depends on the direction of a magnetic induction and described by the tensor field. Force counteraction of system of the pinning centers to driving of a magnetic flux is described by a derivative of this tensor field on coordinates. It is also the tensor field. The proposed macroscopic approach allowed to avoid a problem of summation of the individual pinning forces.

For thin Nb-50%wt.Ti cold-rolled tapes this formalism allowed to calculate the value of critical Lorentz force for the arbitrary orientation of a magnetic field and transport current, measured by 2D Voltage Current Characteristic method [2], and to explain quantitatively the measured value of the angle between magnetic flux movement direction (and corresponding direction of the electric field induced by this movement) and the direction of the transport current (this is so-called “guided vortex motion” phenomenon - GVM) [3]. It is important that combination of critical currents measurements and GVM angle measurements give a way to restore a shape of pinning surface cross section.

The angle between electric field and current usually is very large (around some dozens of degrees) in high-anisotropy pinning superconductors.

In this paper we presented micro-structural, phase and texture analysis of Nb-Ti tapes (both cold-rolled and heat-treated) and results of magnetic flux dynamics fixed by Magneto-Optical Imaging (MOI) method in order to investigate origin and interesting consequences of such an anisotropic pinning.

2 Samples and methods

Two kinds of samples were investigated by structural methods. First series of samples were fabricated from long-length 80-mm width and 10-µm thick cold-rolled tape of the Nb-50wt.%Ti, tape manufactured at the metallurgic factory in 1978 [2]. Second series of samples (heat-treated) were initially the same tape, and additionally they had been annealed in high 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.

Detailed TEM and SEM investigation of Nb-Ti tapes microstructure were done using Titan S-TWIN 80-300 (FEI) microscope with resolution up to 0.08 nm.

Detailed phase analysis by wide angle X-ray diffraction and Small angle X-ray scattering (SAXS) measurements were carried out at Kurchatov Synchrotron Radiation Source (STA and DIKSI stations) in transmission geometry using 2D detectors. The texture analysis was performed using conventional Bruker D8 Discover diffractometer with Cu-Kα radiation.

MOI experiments were performed in University of Oslo, Norway. Small (up to 2 mm in length) rectangular slices of cold-rolled Nb-Ti tape were mounted in the optical cryostat and the temperature were ~ 5.5-6K in an perpendicular external field up to 60 mT, generated by a pair of non-superconducting electromagnets outside the
cryostat A Bi-doped YIG film with in-plane anisotropy was used as the Faraday active indicator for MOI experiments [4].

3 Results and discussion

3.1 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 look like stacks of thin long ribbons extended along tape 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; 440 nm in the direction perpendicular to the RD; and more than 1 μm in the rolling direction. Pronounced grain boundaries were observed only in parallel to the plane. Black spots are clearly α-Ti precipitates. Distributions of Nb-Ti grain sizes were similar both for cold-rolled and heat-treated tapes.

![Fig. 1. SEM cross-section images for Nb-Ti samples: a) cold-rolled, RD is horizontal; b) cold-rolled, RD is perpendicular to image plane; c) heat-treated, RD is horizontal; d) heat-treated, RD is perpendicular to image plane.](image)

X-ray diffraction measurements were performed at the «Structural Materials Science» beamline at the Kurchatov Synchrotron Radiation Source using the following parameters:
- Transmission (Debye-Scherrer) geometry,
- Beam size 200x200 mkm,
- X-ray wavelength λ=0.688862 Å,
- Si(111) monochromator to ΔE/E~2·10⁻⁴,
- FujiFilm ImagingPlate 2D detector.

Figure 2 shows diffraction patterns of cold-rolled and heat-treated tapes for two typical quadrants emphasizing features of diffraction peaks in rolling direction (RD) and in perpendicular to RD. It is clear that significant texture exists in both samples.

![Fig. 2. Synchrotron XRD 2D images for Nb-Ti samples: a) cold-rolled, RD is vertical; b) cold-rolled, RD is horizontal; c) heat-treated, RD is vertical; d) heat-treated, RD is horizontal.](image)

All diffraction lines in Figure 2 can be indexed in terms of cubic β-phase of NbTi (main phase) and small amount of α-Ti phase. Volume fracture of α-Ti phase can be estimated as about 5% for heat treated sample and less than 1% for initial cold-rolled sample.

It should be noted that α-Ti precipitates which mainly are located nearby grain boundaries (see Figure 1) are also high textured and moreover the texture in α-Ti succeeds the texture of main cubic NbTi phase. Possibly it is due to diffusion nature of α-Ti precipitates formation mechanism during heat-treatment of NbTi alloy.

Small angle X-ray scattering (SAXS) 2D images for both types of samples are presented in Figure 3.

![Fig. 3. Small angle X-ray scattering (SAXS) 2D images for Nb-Ti samples: a) cold-rolled, b) heat-treated. Rolling direction (RD) is shown by arrows.](image)
One can see “eye-shape” of the SAXS images which points at anisotropic form of precipitates along RD and in perpendicular to RD. For heat-treated Nb-Ti tape the brightness of SAXS image is much higher (more than one order of magnitude) in contrast with cold-rolled tape. This can be explained by significant increasing of $\alpha$-Ti precipitates volume in heat-treated Nb-Ti tape.

Figure 4 shows the pole figures of Nb-Ti phase registered by standard X-ray diffraction for the reflections (110), (200) and (222) in the cold-rolled Nb-Ti tape [5]. Analysis of ODF showed almost uniform distribution of the orientations from \{100\} <110> to \{112\} <110>, which is typical for the rolling of Nb-Ti alloys [6]. Heat treatment does not change texture of the main Nb-Ti phase.

**3.2 Magneto-optical study**

Features of magnetic flux penetration (field is normal to sample plane) into cold-rolled Nb-Ti tape were observed in MOI experiment with optical cryostat in polarized light. The penetration picture in general corresponds to model of a critical state taking into account anisotropy of a pinning in cases when current flow along RD and in perpendicular to RD. Due to small inhomogeneity of a superconductor the path of a magnetic flux movement can be observed that allowed to estimate the angle between the direction of flux movement and the Lorentz force originated this driving which is normal to sample edge.

In Figure 5 the evolution of the magnetic flux penetration into Nb-Ti sample with an angle of 10 degrees to rolling direction is shown. Some interesting peculiarities can be marked on these images.

- the flux penetration corresponds to a model of a critical state in perpendicular geometry;
- it is visible, that in case of a full flux penetration (in field more than 45 mT) the anisotropy factor of the critical current density is about 2 (see caption of a the Figure 5 for details). The similar value of anisotropy factor was obtained from direct transport measurements (see Figure 6) at low field end [2].
- it is visible, that the flux starts to penetrate into superconductor as "branches". It can be explained by some heterogeneity of a material.
- it is visible, that the direction of such a "branches" does not coincide with tape rolling direction and/or perpendicular to sample edge. For example, it is about 73 deg. for MO image at 30 mT in Figure 5, which corresponds to GVM angle of 90-73=17 deg. This angle is very close to calculated GVM angle for anisotropy factor of 2 derived from anisotropic pinning model [2] (see Figure 7).
4 Discussion and conclusions

In previous papers [1, 2] the macroscopic electrodynamics of hard superconductors with anisotropic pinning was proposed phenomenologically, on the basis of empirical studying of critical Lorentz forces. For Nb-50%wt.Ti tapes this formalism allowed to calculate the value of critical Lorentz force for the arbitrary orientation of a magnetic field and transport current, and to define the direction of magnetic flux movement. GVM phenomenon was experimentally investigated by analysis of 2D Voltage-Current Characteristics.

In this paper we present a number of various modern structural analytical methods (including SEM, wide angle and small angle X-ray diffraction) and texture analysis in connection with the same Nb-Ti material. In cold-rolled Nb-Ti tape the pinning force anisotropy can be explained mainly by anisotropy of grain boundaries density. 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 rolling direction. Pronounced grain boundaries observed only in the tape plane and this results to very high pinning force if magnetic field lies in the tape plane. In contrary when magnetic field is perpendicular to the tape plane the pinning force drops dramatically [2]. In-plane anisotropy factor of pinning force can be explained by difference of main grain size (and correspondent grain boundaries density) in rolling direction and in perpendicular to rolling direction. Volume fracture of α-Ti precipitates in cold-rolled tapes is negligible (less than 1%) so precipitates do not influence on global pinning system significantly. After annealing in high vacuum at 385°C during 25 hours the volume fracture of precipitates increases up to 5% and the precipitates mainly are located nearby grain boundaries. It is obvious that critical current anisotropy in Nb–Ti tape after such a heat-treatment should change significantly. Detailed analysis of transport and magnetic measurements of heat-treated Nb-Ti samples will be published additionally.

Magneto-Optical Imaging (MOI) method was implemented to verify if the model of pining anisotropy can be applied at low magnetic field (less than 0.1 T) and in perpendicular geometry (taking into account known limitations of the method). Good quantitative agreements with previous transport measurements [2] were obtained.

The authors are very grateful to Prof. E. Yu. Klimenko for discussions of the results and to Prof. T. H. Johansen for arranging of MOI investigations. The SEM and X-ray measurements structural and texture were performed on equipment of the Resource Center of Probe and Electron Microscopy, Resource Center of X-ray methods (Kurchatov Complex of NBICS-Technologies, NRС “Kurchatov institute”) and on Kurchatov Synchrotron Radiation Source.

References

1. E. Yu. Klimenko, Superconductivity Theory and Applications ed. by A. M. Luiz. InTech (2011)
2. E. Yu. Klimenko, S. V. Shavkin, P. V. Volkov, Zh. Eksp. Teor. Fiz 112 (1997)
3. A. K. Niessen, C. H. Weijesfeld, J. Appl. Phys. 40 (1969)
4. T. H. Johansen, M. Baziljevich, H. Bratsberg, Y. Galperin, P. E. Lindelof, Y. Shen and P. Vase, Phys. Rev. B, 54, N22 (1996)
5. V. V. Guryev, S. V. Shavkin, V. S. Kruglov, P. V. Volkov, A. L. Vasiliev, A. V. Ovcharov, I. A. Likhachev, E. M. Pashaev, R. D. Svetogorov, Y. V. Zubavichus, J. of Phys.: Conf. Ser. 747 (2016)
6. M. Gepreel, Recent Developments in the Study of Recrystallization ed. P Wilson, chapter 4, InTech (2013)