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MICROSTRUCTURE OF SUPERCONDUCTING CABLE COMPONENTS

Svetlana Barannikova[1,2,3], Galina Shlyakhova[1], Lev Zuev[1,2] and Anatoly Malinovskiy[3]

[1]Institute of Strength Physics & Materials Science, SB RAS, Russia; [2]National Research Tomsk State University, Russia; [3]Tomsk State University of Architecture and Building, Russia

ABSTRACT: Using atomic force, optical and electron microscopy methods, the changes in the microstructure and phase composition were investigated for the alloy Nb47%Ti used for the manufacture of superconducting cable employed as current-carrying elements in the magnetic system for International Thermonuclear Experimental Reactor. The test samples were prepared from the superconducting wire at an intermediate step of the drawing process for the area reduction Ø1.3→Ø1.2 mm. The effect of cold drawing and intermediate annealing on the properties of as-worked Nb-Ti alloy was assessed. Local strain zones were found to occur in the rupture area. The shape and chemical composition of Nb-Ti wire was examined for both a defect-free area and the rupture area. A Nb diffusion barrier was found to occur in the copper matrix of Nb-Ti wire.

Keywords: Superconductors, Microstructure, Plasticity, Localization

1. INTRODUCTION

Materials traditionally used in electrical engineering have been to some extent supplanted by superconductive materials, which are cost-effective and suitable for a wide range of applications, in particular, nuclear energetics [1]. Thus Nb-Ti alloys are worthy of special attention [2]-[4]. These are used for the manufacture of superconducting cable employed as current-carrying elements in the magnetic system for International Thermonuclear Experimental Reactor. The cable houses several thousand strands of superconducting wire in a copper matrix, each strand being 2…5 mμ in diameter [2]. Such cables must be capable of meeting a number of stringent requirements, i.e. stability of criticality parameters and of current-voltage characteristics, strength-resistance to breaking and structural uniformity of the superconducting wire, uniform cross-section geometry of wire [5]-[7].

By the manufacture of superconducting Nb-Ti wire, the most important stage is cold drawing with the area reduction of the original bar Ø60 …70 mm→Ø0.1…1.0 mm [8]. As-produced superconducting wire is expected to have high strength resistance to breaking as well as structural uniformity, i.e. pre-set density of micro-defects for controlling formation of pinning centers. The goal of the given work is examination of the influence of the severe plastic deformation (SPD) treatment by drawing on the structure of superconducting Nb-Ti wire.

2. HEADINGS MATERIALS AND METHODS

The structural investigations were performed using test samples of the Nb-Ti alloy containing 47.5% Ti (by mass) or 63.7% Ti (at. percent). The test samples were prepared from the superconducting cable wire at the intermediate stage of cold drawing with the area reduction of the original bar Ø1.33→Ø1.2 mm. To provide for high space resolution level, several methods were used for investigation of the composition and structure of the alloy: optical microscopy (Olympus GX 71 units); raster electron microscopy (Philips SEM 515 unit); atomic force microscopy (Solver PH47-PRO unit) and contact method. The element distribution was determined for material on the border of copper matrix and Nb-Ti cable core, using the method of raster electron microscopy (Quanta 200 3D ion-electron unit equipped with detectors of secondary and back-scattered ions).

3. RESULTS AND DISCISSION

The coated cable construction houses several thousand strands of superconducting Nb-Ti wire in composite copper matrix (Fig. 1). The composite might contain resistive or diffusion barriers, stabilizing coating or armoring [2]. Metallography investigation was performed using cross-sections of superconducting Nb-Ti wire strands, which had a round form with the average diameter ~10 μm. However, the intermediate layer adjacent to the copper matrix all the Nb-Ti wire strands have rhomb-like shape with13-µm and 11-µm diagonals, which might be attributed to the SPD treatment by cold drawing.

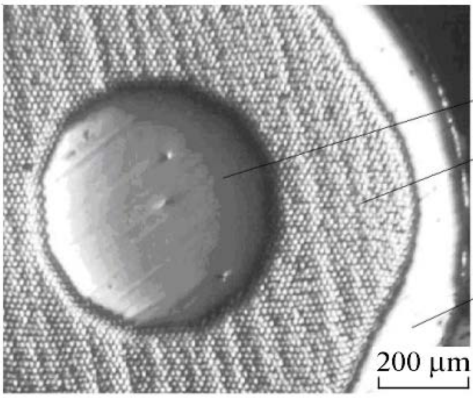

Fig. 1 The cross-section of superconducting Nb-Ti cable wire strands at the intermediate stage of cold drawing with the area reduction Ø1.3→Ø1.2 µm: 1 – copper matrix; 2 – Nb-Ti wire strands and 3 – copper core

Using atomic force microscopy in conjunction with contact method in constant force mode, cross-section relief was examined for different cable zones [9], [10]. The scanning revealed a diffusion Nb barrier, which is clearly defined by the relief hills observed in the area of Nb-Ti strands and copper matrix (Figs. 2*a* and *3a*). A profilogram plotted by the linear-intersept method shows a smooth relief of low-amplitude lines of Ni-Ti strands and copper matrix (Fig. 2*b* and *3b*), which separate high-amplitude maximums 250…260 nm wide corresponding to the Nb barrier (Fig. 2*c* and *3c*).

The SPD treatment would cause grain refinement in the copper matrix. As a result, a submicrocrystalline structure is formed with average grain size ~800 nm, which also contains individual grains and conglomerates composed of up to eight grains. The most heavily deformed area is located along the copper core-intermediate Ni-Ti layer border with the grain sizes varying from ~2120 nm to ~310 nm. The intermediate copper matrix layers separating Nb-Ti wire strands contain equiaxed grains having average size ~800 nm, while the outer copper layers have average grain size ~1050 nm.

Using electron microscopy methods, material structure was examined in the intermediate layer on the Ni-Ti strands-copper core border; a specific defect was found to occur in the material (Fig. 4), which is due to the rupture of superconducting strands. The investigations were carried on using a raster electron microscope Carl Zeiss EVO 50, which was equipped with an attachment designed for X-ray dispersion microanalysis (Oxford Instruments). It was found that the outer layer and the core are comprised of copper; however, elements from the intermediate Ni-Ti layer would penetrate into the cable's copper core as well as intermediate-outer layer interphase. The chemical composition of intermediate layer wire strands on the core border is 35.66% (at) and 63.07% (at) for Nb and Ti, respectively; the wire strands in the point of rupture have the same chemical composition. In the former case, intermediate layer wire strands have a round form and in the latter, an elongated one.

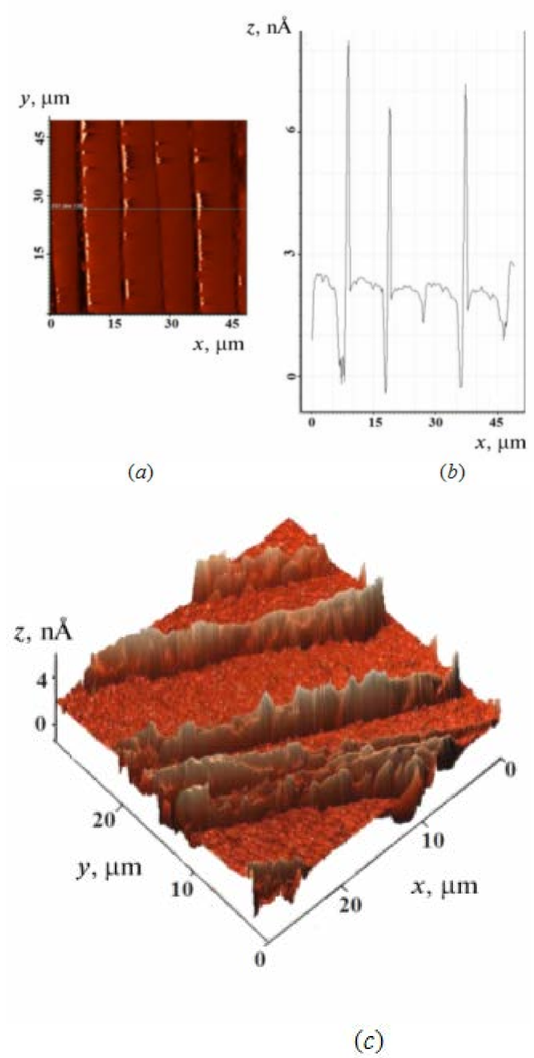

Fig. 2 The relief of longitudinal cross-section showing diffusion Nb barrier within unpolished defect-free area of Nb-Ti strands and copper matrix (*a*); a profilogram of the same area (*b*); 3D image (*c*)

Using scanning microscopy in secondary electrons mode in conjunction with characteristic X-ray radiation on a unit Quanta 200 3D, the composition of Nb-Ti strands was examined and found to be uniform.

Energy dispersive X-ray composition microanalysis was used to identify the elemental composition of material. In the area examined, i.e. intermediate Nb-Ti strands layer-copper matrix-intermediate Nb-Ti strands layer, the following elements were found to occur: Nb, Ti and Cu.

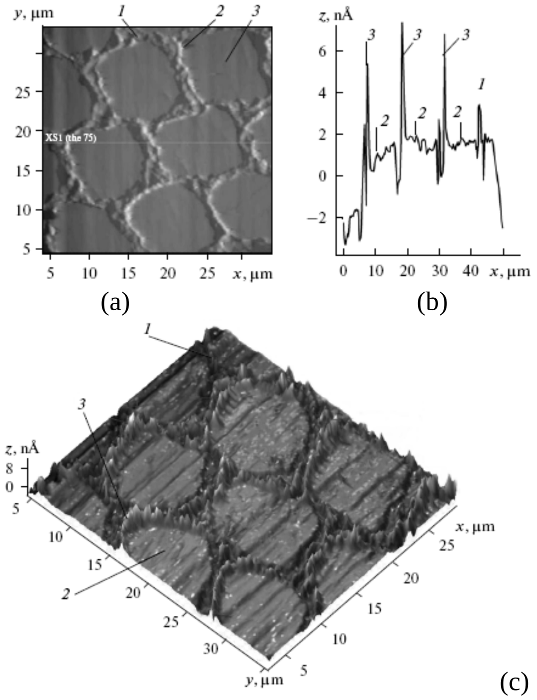

Fig. 3 The relief of transverse cross-section showing Nb barrier around the wires in a conductor matrix: (*a*), (*c*) 2D and 3D images, respectively; (*b*) profilogram of a 30 × 30 μm region. (*1*) Copper matrix, (*2*) Nb–Ti wires, and (*3*) niobium barrier

The non-homogeneous space distribution of characteristic X-ray photons of Ti, Nb and Cu in the Nb-Ti strands and the copper matrix suggests that a diffusion layer occurs in the direction across the Nb-Ti strands-copper matrix border. The maximal and the minimal number of characteristic X-ray photons of Nb and Ti are observed, respectively for the Nb-Ti strands and the copper matrix. Exactly the converse situation is observed for the copper matrix, i.e. the maximal number of characteristic X-ray photons is observed for Cu and the minimal number of the same photons, for Ti and Nb. In the case of intermediate diffusion layer, the number of characteristic X-ray photons of Nb would first remain constant, which supports the evidence for the occurrence of Nb barrier, which was obtained by the AFM method. Then the number of characteristic X-ray photons of Nb and Ti would either grow or decrease depending on whether the number of the same photons of Cu grows or decreases.

Using ion beam, the fine structure of cable components was examined by the thin-foil method. The Nb-Ti strands are found to have a heavily deformed structure; grain boundaries were indistinct, evidence of relaxation unavailable. Individual dislocations within the grains of Nb-Ti strands could not be resolved. The Nb barrier on the Nb-Ti strands-copper matrix border could be defined only partially in the point of rupture. The material within the matrix was in a deformed and relaxed state. Individual dislocations would occur within the grains; however, no dislocation clusters are observable.

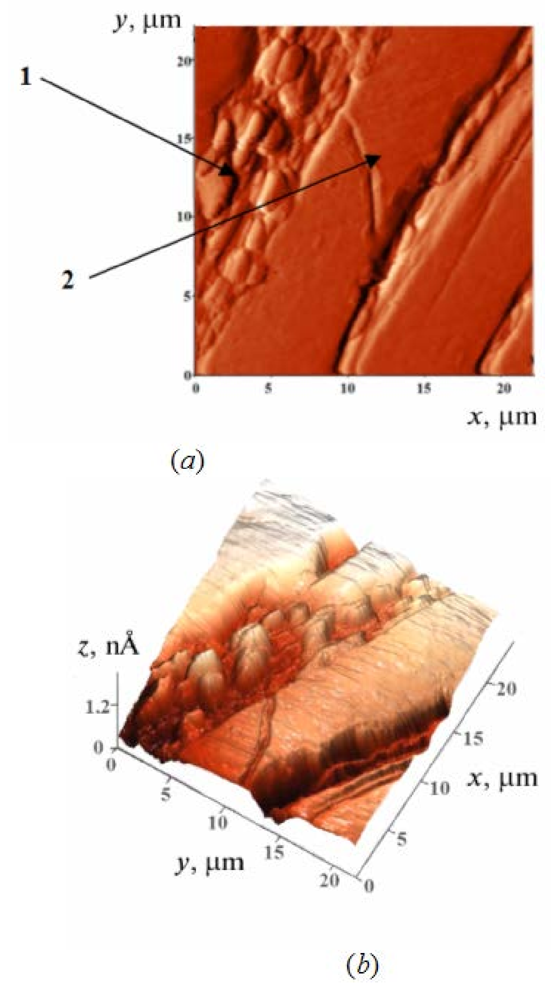

Fig. 4 Longitudinal section showing rupture of unpolished Nb-Ti strands in as-received defect-free state (*a*); 3D image (*b*); 1– copper matrix; 2 – Nb-Ti strands

The Nb barrier would become noticeable on going along the Nb-Ti strands-copper matrix border; it has submicrocrystalline structure, small-size grains have a low degree of non-equiaxiality (Fig. 5).

The morphology of material within the localized plastic deformation zone was investigated. Several layers of material 0.5-mm each were removed with the aid of abrasive from the latter zone to be examined by metallography technique. It was found that all the Nb-Ti strands of the inner layer adjacent to the copper core have a round shape. The SPD treatment by drawing has left traces in the intermediate layer of the region adjacent to the copper core. The Nb-Ti strands in the outer layer are rhomb-shaped.

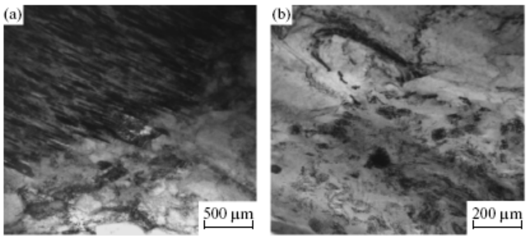

Fig. 5 Fine structure of superconducting cable: (a) boundary between Nb–Ti fiber and matrix; (b) boundary between copper matrix and niobium barrier

The cross-section topography was examined by the methods of optical and atomic-force microscopy. After the region adjacent to the copper core had been polished with abrasive to a depth of 0.5 mm, Nb-Ti strands on its border would assume irregular shape and would form a localized deformation zone similar to that observed for as-received Nb-Ti strands which have not been polished (Fig. 6*a*, *b* and Fig. 7*a*, *b*). In the point of rupture the intermediate copper matrix layer separating Nb-Ti strands had average grain size ~850 nm.

The statistic treatment data suggests that the average grain size of the defect-free zone of the copper matrix has average grain size ~800 nm, which is comparable to that of copper in the point of rupture, i.e. ~850 nm. The profilogram obtained for the cross-section polished to a depth of 0.5 mm shows a Nb barrier in the point of rupture, which surrounds Nb-Ti strands within the copper matrix. This presents a series of high-amplitude sharp maxima having width ~250 nm, which is similar to that observed for as-received material without polishing. A similar Nb barrier surrounding Nb-Ti strands in the location of breakage was observed for material that had been polished to a depth of 1 mm.

Metallography investigation was performed for Nb-Ti strands cross-sections, which were subjected to etching and polishing to a depth of 1 mm. It was found that Nb-Ti strands appear to be unchanged by comparison with as-received unpolished material. However, the localization zone has a different aspect (Fig. 6*c* and Fig. 7*c*). The lobe-like strands coalesce to form one strand, which suggests that the strands are of different thickness. The Nb-Ti strands within the localization zone assume a round shape.

The structure of section surface, which had been polished to a depth of 2mm, was examined in an optical microscope. The general aspect of localization zone is found to have changed significantly relative to as-received defect-free state. The Nb-Ti strands in the point of rupture gradually form a conglomerate (Fig. 6*d* and Fig. 7*d*); the near-lying Nb-Ti strands have a round shape, which is characteristic for defect-free zones. It should be noted that another defect was found to occur in the vicinity of boundary between the intermediate layer of Nb-Ti strands within the copper matrix and the copper core, i.e. two Nb-Ti strands of irregular shape, which had smaller size relative to the near-lying ones.

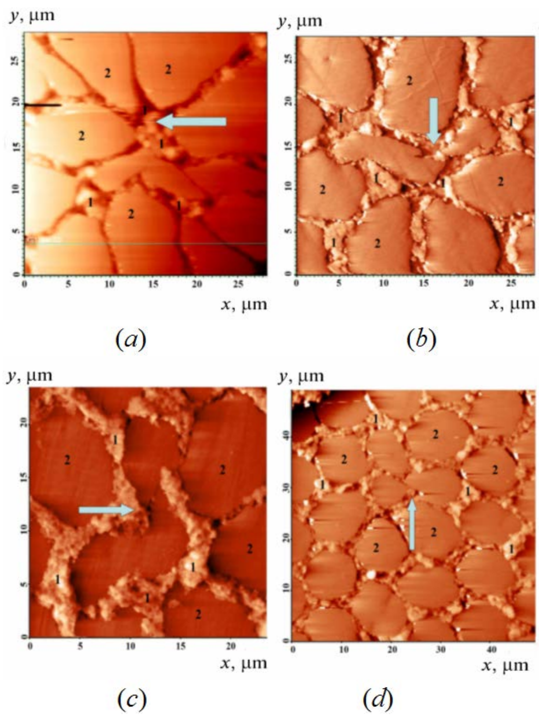

Fig. 6 The AFM image of the zone of localized plastic deformation at the point of breakage of Nb-Ti strands (designated by arrows): 1–copper matrix; 2–Nb-Ti strands; depth of polishing: (*a*) 0 mm; (*b*) 0.5 mm; (*c*) 1 mm; (*d*) 2 mm

Using the contact method of AFM technique, we examined the cross-section surface of Nb-Ti strands which had been subjected to etching an polishing to a depth of 2 mm.

In this case, a Nb barrier is found to occur on both the inner and the outer surface of the Nb-Ti strands in copper matrix. As noted above, a similar Nb barrier had been observed previously in a defect-free area of as-received material; this shows in profilograms as a series of high-amplitude narrow maxima having width ≤ 250 nm.

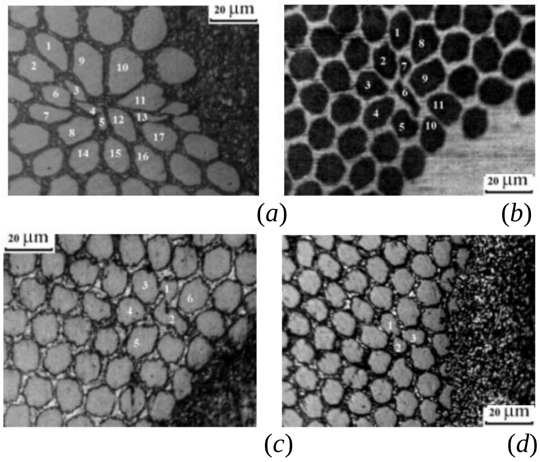

Fig. 7 The optical microscopy image of the localized plastic deformation zone in the site of breakage of Nb-Ti strands; depth of polishing: (*a*) 0 mm; (*b*) 0.5 mm; (*c*) 1 mm; (*d*) 2 mm

The evolution of localized plasticity zone is illustrated in Figs. 6 and 7. Analysis of the curve was made in order to define in what way a change in the cross-section area of Nb-Ti strands in the point of rupture is affected by the depth of polishing (Fig. 8). The sections of Nb-Ti strands polished to a depth of 2mm were examined; the results obtained suggest that the SPD treatment by drawing caused neck formation within the localization zone observed for the Nb-Ti strands.

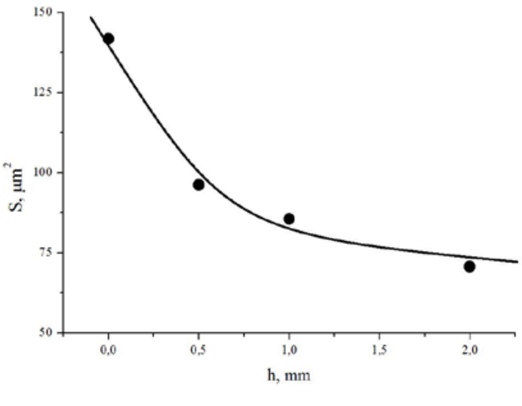

Fig. 8 A change in Nb-Ti strands cross-area (S) in the point of rupture (within the localization zone) *vs* depth of polishing: h = 0 mm; 0.5 mm; 1 mm; 2 mm

Thus, the area next to the cable copper core is found to contain localized deformation zone in the point of rupture of Nb-Ti strands. Moreover, the Nb barrier is present only partially in the same area, which is indicative of a varying extent of deformation in the sections, which were polished to depths varying from 0.5, 1 and 2 mm. A major drawback to the conventional approach employed in the analysis of the ultimate tensile strength of superconductors on the base of Nb-Ti alloy is the conceptual representation of plastic deformation as a steady-state and homogeneous process [11].

The experimental evidence for the nature of plastic deformation strongly suggests that at the early stages of straining the plastic deformation would exhibit an inhomogeneous behavior, which is liable to cause formation of localized plasticity nuclei [12]; in one of these nuclei rupture of cable strands would occur. The macro-scale localization of deformation has been studied in sufficient detail. On the base of experimental evidence a one-to-one correspondence has been established between the work hardening law, which is acting at the given flow stage of the stress-strain curve, on the one hand and the space-time distribution plastic distortion tensor components observed for the deforming material on the other hand [12], [13].

4. CONCLUSION

Analysis was made of the effect of the SPD treatment by drawing on material structure of the multi-strand superconducting cable on the base of Nb-Ti alloy. The following results have been obtained.

- Deformation localization zones are found to occur in the point of rupture of the cable. Sections of Nb-Ti strands were polished to a depth of ≤ 2 mm to reveal that the adjacent Nb-Ti strands have irregular shape.
- A variation in the shape and size of Nb-Ti strands is found to occur in a defect-free area of the intermediate layer; on the copper core border Nb-Ti strands have a round shape and diameter ~ 10 µm and those on the copper matrix border are rhomb-shaped with diagonals ~ 13 and 11 µm.
- The SPD treatment by drawing causes grain refinement; a submicrocrystalline structure will form in cable components: the copper core and the intermediate layer of material between the strands within the matrix has grain size ~800 nm; material in the point of rupture and in the intermediate layer between the strands within the matrix has grain size ~850 nm and material of the upper layer has grain size ~1050 nm.
- A diffusion Nb barrier about 250 nm wide is found to occur around the Nb-Ti strands in the copper matrix in a defect-free area; using step-by-step polishing to the depth of 2000 µm, a similar barrier was discovered within a localized deformation zone.
- A change in the chemical composition and shape of Nb-Ti strands in the intermediate layer was observed; in a defect-free area all the Nb-Ti

strands have a round shape and the following chemical composition: 35.66 at.% Nb and 63.07 at.% Ti; in the area of rupture the Nb-Ti strands have a regular shape and the following chemical composition: 35.57 at.% Nb and 63.33 at.% Ti.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

[1] Wilson MN, "NbTi conductors with very low loss: a review", Cryogenics, Vol. 48, 2008, pp. 381-395.

[2] Kozlenkova N, Vedernikov G, Shikov A, Potanina LV, Filatov AV, Vorobieva AE, Pantsyrnyi VI, Gubkin I.N, "Study on Ic (T, B) for the Nb-Ti strand intended for ITER PF insert coil", IEEE Transactions on Applied Superconductivity, Vol. 14, 2004, pp. 1028-1030.

[3] Lu B, Luongo CA, "Parametric estimation study of interstrand conductance in multi-strand superconducting cables", Cryogenics, Vol. 47, 2007, pp. 546-552.

[4] Ochiai S, Oki Y, Sekino F, Hojo M, Tanaka M, Okuda H, Moriai H, Sakai S, Watanabe K, "Fracture of filaments and its influence on critical current and residual strength of fatigued Nb–Ti/Cu superconducting composite", Cryogenics, Vol. 43, 2003, pp. 45-51.

[5] Beloshenko VA, Chishko VV, Matrosov NI, Dyakonov VP, Szymczak R, Pietoza J, Puzniak R, Szymczak H, Gaida D, Zaleski A, "Effect of deformation and heat treatment with equal-channel, multiple-angle pressing on the superconducting properties of NbTi alloy", Low Temperature Physics, Vol. 36, 2010, pp. 1045-1048.

[6] Lee PJ, Larbalestier DC, McKinnel JC, "High titanium Nb-Ti alloys initial microstructural studies", Advances in Cryogenic Engineering, Vol. 34, 1988, pp. 967-974.

[7] Cherny OV, Andrievskaya NF, Ilicheva VO, "The microstructure and critical current density of Nb-48 wt.%Ti superconductor with very high alpha-Ti precipitate volume and very high critical current", A Advances in Cryogenic Engineering, Vol. 48B, 2002, pp. 883-890.

[8] Cherny OV, Storozhilov GE, Andrievskaya NF, Ilichova VO, Starodubov YD, Volchok OI, Chirkina LA, Lazareva MB, Okovit VS, "Structure and properties of differently directed deformed niobium-titanium alloy", IEEE Transactions on Applied Superconductivity, Vol. 15, 2005, pp. 3502-3505.

[9] Zuev LB, Shlyakhova GV, Barannikova SA, Kolosov SV, "Microstructure of elements of a superconducting alloy Nb-Ti cable", Russian Metallurgy (Metally), Vol. 2013, 2013, pp. 229-234.

[10] Shlyakhova GV, Barannikova SA, Zuev LB, "On structure of localization zones of plastic deformation in superconductive cable based on Nb-Ti alloy", Metallofizizika i Noveishie Tekhnologii, Vol. 35, 2013, pp. 453 – 465.

[11] Zuev LB, Barannikova SA, Zavodchikov SYu, "Localization of tensile deformation in a polycrystalline Zr-based alloy", Physics of Metals and Metallography, Vol. 87, 1999, pp. 244-246.

[12] Zuev LB, Danilov VI, Barannikova SA, Gorbatenko VV, "Autowave model of localized plastic flow of solids", Physics of Wave Phenomena, Vol. 17, 2009, pp. 1–10.

[13] Zuev LB, Barannikova SA, "Experimental study of plastic flow macro-scale localization process: pattern, propagation rate, dispersion", International Journal of Mechanical Sciences, Vol. 88, 2014, pp. 1-7.

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Corresponding Author: Svetlana Barannikova