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MULTILAYER SUPERCONDUCTING Nb50Ti ALLOY TAPE, OBTAINED FROM THE COMPOSITE Cu/Nb/Ti THROUGH SOLID-PHASE METHOD

Aim: Creation of multilayer superconducting tape made of Nb-Ti alloy.

Methods: Using the methods of diffusion welding and packet rolling, for two cycles the prototypes of a multilayer tape based on a superconducting Nb–50%Ti alloy were made. Copper was used as a stabiliser of the superconducting state of the conductor. At the initial stage, a multilayer Nb-Ti pack was assembled from niobium and titanium foils. Copper stabilising layers were laid in the pack in the 2nd cycle of tape manufacturing. The mutual diffusion between the Nb- and Ti-layers took place generally at the expense of niobium diffusing into the layers of titanium, with the Nb-50%Ti alloy emerging in their place.

Results: Measurements of the critical current I_c with a perpendicular and parallel orientation of the magnetic field relative to the plane of the layers in the composite showed large anisotropy of I_c , which was the result of the superconducting vortices fixing exclusively at the boundaries of the Nb-Ti-alloy and the Nb-solid solution. In general, the composite was capable of carrying large current in magnetic fields of 5-6 T without long-lasting low-temperature annealing for α -phase deposition, which is necessary in the case of Nb-Ti alloy composites produced by the known technology.

Keywords: superconducting tape, multilayer composite, Nb–Ti alloy, solid-phase method, mutual diffusion, batch packet rolling, critical current, superconducting vortex, pinning.

Рубрика 2. НАУЧНЫЕ И ПРАКТИЧЕСКИЕ РАЗРАБОТКИ Направление «Физика конденсированного состояния»

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МНОГОСЛОЙНАЯ СВЕРХПРОВОДЯЩАЯ ЛЕНТА СПЛАВА Nb50Ti, ПОЛУЧЕННАЯ ИЗ КОМПОЗИТА Cu/Nb/Ti ТВЕРДОФАЗНЫМ СПОСОБОМ

Цель: Создание многослойной сверхпроводящей ленты из ниобий-титанового сплава.

Методы: Методами диффузионной сварки и пакетной прокатки за два цикла изготовлены опытные образцы многослойной ленты на основе сверхпроводящего сплава Nb–50%Ti. В качестве стабилизатора сверхпроводящего состояния проводника использовалась медь. На начальном этапе из фольг ниобия и титана собирался многослойный пакет Nb/Ti. Медные стабилизирующие слои закладывались в пакет во 2-м цикле изготовления ленты. Взаимодействие между ниобием и титаном происходило, в основном, за счет диффузии ниобия в слои титана с образованием на их месте сплава Nb–50%Ti.

Результаты: Измерения критического тока I_c при параллельной и перпендикулярной ориентации магнитного поля относительно плоскости слоев в композите показали большую анизотропию I_c , что было результатом закрепления сверхпроводящих вихрей преимущественно на границах NbTi-сплава и Nb-твердого раствора. В целом композит был способен проводить большой электрический ток в магнитных полях 5–6 Тесла без длительного низкотемпературного отжига для выпадения α -фазы, который необходим в случае композитов из ниобий-титановых сплавов, получаемых по известной технологии.

Ключевые слова: сверхпроводящая лента, многослойный композит, сплав Nb–Ti, твердофазный способ, взаимная диффузия, пакетная прокатка, критический ток, сверхпроводящий вихрь, пиннинг.

INTRODUCTION

The intensity of studies of superconducting niobium-titanium alloys falls on the period 1960–1970. In those years, the problem of superconducting alloys in general and NbTi, in particular, was devoted to regular conferences held in IMET them. A. A. Baikova Academy of Sciences. The development of these studies can be traced in the collections of conference articles published in those years, as well as two collections of articles by leading scientific teams of the country, edited by Corresponding Member of the USSR Academy of Sciences E. M. Savitsky and his colleagues [1–7]. In the USSR, these studies are embodied in the development of industrial technology of multi-core

superconducting materials based on Nb–50Ti alloy that has experienced already in the XXIst century, its new renaissance. But at the present time, research works on superconducting NbTi alloys are almost not conducted.

Investigating a number of multilayer materials obtained by packet rolling, for the dependence of their mechanical properties on the thickness of the layers, we obtained composites of niobium with superconducting alloys Nb–31 and 50 wt%Ti – Nb/Nb31Ti and Nb/Nb50Ti [8, 9]. The aim was to measure the critical current density j_c as a function of the thickness of the (Nb–Ti)-alloy layers. It turned out that the dependence of $j_c(t)$, where t is the thickness of the superconductor layer, obeyed the Hall-Petch relation, if the strength of σ is replaced by the critical current density, and the grain size by the thickness of the superconducting layer.

As for the superconductivity of the selected composites, it is known that in superconductors of the second kind the critical current density j_c depends on how effectively the superconducting vortices will be fixed on the structure defects, that is, j_c is the same, and maybe more like hardness or strength, the structure-dependent characteristic of the material. V.V. Schmidt [10], in a theoretical study of the interaction of vortices with a plane surface of a superconductor, showed that even defect-free superconducting plates with a thickness d >> λ (λ – penetration depth of an external magnetic field) in a mixed state can carry a considerable current $\sim 10^5$ A/cm². Now let us imagine that a thick plate is replaced by a set of thin superconducting plates artificially separated from each other by a layer of normal metal. In this case, the current will flow through each of the plates, and a large current will flow throughout the entire section of the multilayer superconductor. It was this situation that was first realized in multi-layer Cu/Nb composites [11], and then Nb/NbTi [8, 9]. In the first of these, layers of superconducting niobium were separated by layers of normal copper. The role of the normal metal in the 2nd composite was carried out by niobium, since measurements of the critical currents of superconducting tapes from (Nb-Ti)-alloys were carried out in magnetic fields many times greater than its second critical magnetic field.

The anisotropy of the critical current density $j_{c\parallel}/j_{c\perp}$, measured with parallel (||) and perpendicular (\perp) orientations of the plane of the layers of the composite and magnetic field of the superconducting solenoid H, was an evidence of the fact that superconducting vortices are effectively pinned over extended boundaries between the layers of niobium and copper or layers of niobium and the (Nb–Ti)-alloy. In the Nb/Cu-composite, $j_{c\parallel}/j_{c\perp} = 410$ in fields of 0.5–0.6 T [11].

In Nb/NbTi in fields of 5–6.5 T the anisotropy increased from 3–5, for composites with layers ~140 nm thick, up to 235 for composite tapes with layer thickness ~3 nm. In some cases, the ratio $j_{c\parallel}/j_{c\perp}$ exceeded 2000 [8, 9].

In this paper, it is proposed to dispense with the use of the melting process for the preparation of the Nb50Ti alloy. On a large scale, the NbTi alloys are produced by the method of skull melting – quite expensive and labor-intensive. The formation of the Nb–50%Ti alloy will occur during two cycles consisting of diffusion welding and packet rolling, using initially Nb/Ti packages with foils of pure metals.

It should be noted that the rolling of packages has a positive effect on the formation of a layer of NbTi-alloy. During rolling, the multilayer composite is stretched several times in length. As a result, atomically clean, so-called, juvenile surfaces of contiguous homogeneous and dissimilar metals are released within the composite [12].

During rolling, these surfaces approach each other to the distances of the action of interatomic forces, and as a result of the formation of metal bonds they "grip". With plastic deformations, this phenomenon is called "gripping". The ability of metals the ability of metals to grasp is a physical property of juvenile surfaces "to grasp" is a physical property of juvenile surfaces. Under ideal conditions of thermodynamics, gripping is an advantageous process and should occur spontaneously, since the energy of the system from the connected metals becomes less due to the elimination of free surfaces.

Ideal conditions are understood as convergence of surfaces that are free of oxide and adsorbed films by a distance equal to the sum of the radii of the atoms of the surfaces being joined. The current rolling is one of the ways of obtaining juvenile surfaces.

In our case, the formation of juvenile surfaces is also useful because through them the mutual diffusion of heterogeneous atoms was made more freely than in comparison with if rolling had not been undertaken.

Methods for obtaining and studying the structure of a conductor

Diffusion welding of packets and packet rolling. To obtain a superconducting tape based on the (Nb–Ti)-alloy, the diffusion welding (DW) of multilayer packets (Fig. 1) was used under pressure in vacuum and subsequent packet rolling (PR) at room temperature.

DW of packets were conducted in a vacuum not lower than 10^{-4} Torr. The welded packets were placed between punches made of high-strength graphite. Between the package and punches were laid foils of thermally split graphite (TSG) with a thickness of 0.3 mm. In the 1st cycle, the Nb/Ti-packets were welded at 1050 °C for 10 min under a pressure of 16–17 MPa.

In the second cycle, packets containing outer and inner gaskets from copper were welded and, therefore, the temperature of the DW decreased to 900–950 °C at a pressure of 17–19 MPa, and the welding time was increased to 1.5-2 h.

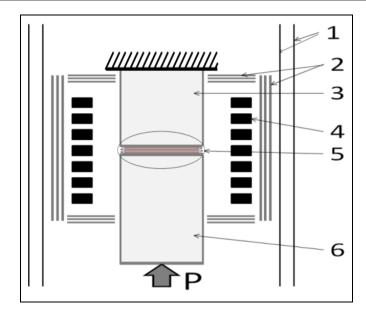


Fig. 1. Mutual arrangement of the main units of the installation for DW:

- 1 water-cooled camera body,
- 2 set of heat shields from pressed graphite wool and Mo-foil,
- 3 fixed punch,
- 4 high-strength graphite heater,
- 5 test package,
- 6 movable punch,
- P force.

The deformation of the welded packs by rolling was carried out at room temperature on a 4-roll rolling mill. Degree of deformation per pass was 2-3 %. The rolling direction of the packages corresponded to the rolling direction of the Nb- and Ti-foils laid in the packages. In the 1st cycle, the tape was rolled to a thickness of 0.2–0.25 mm, cut into lengths of the required length, and a new package of the required design for the second cycle was assembled. After the 2^{nd} cycle, the welded packet was rolled to a tape with a final thickness of -0.1 mm.

Study of the structure of the material. The microstructure of the composites was studied by scanning electron microscopy and X-ray spectral analysis (XSA). Studies involving the imaging of objects in secondary and reflected electrons and XSA were performed on digital electron scanning microscopes Tescan VEGA-II XMU and CamScan MV230 (VEGA TS 5130MM). Both microscopes have W-cathodes, are equipped with YAG-detectors of secondary and reflected electrons and an X-ray microanalyzer.

The studies were performed at an accelerating voltage of 20 kV and the current of the electronic probe 200 pA on a cobalt sample. The size of the electronic probe is 0.16 μ m. The depth of the characteristic X-ray emission region reached 5–6 μ m. On a horizontal section, this corresponded to a circle, and in the volume – a pear-shaped zone with the largest diameter of about 10 μ m.

Structure of multilayer composites

Build of packages. A superconducting multilayer tape made of Nb–50%Ti alloy stabilized with copper was produced in two cycles, each consisting of diffusion welding and packet rolling. The assembly of packets in the 1st cycle was carried out from individual elements of the book form, produced in advance from foils of niobium and titanium, and individual foils of titanium and niobium, respectively [13]. Observing a certain sequence, it was possible to collect relatively quickly and easily packets of ten, twenty or more niobium elements alternating with Ti-foils, or titanium elements alternating with Nb-foils. The package was a single design, so it was possible to produce the necessary technological procedures with it. The components of the original packages are listed in Table. 1.

Notation package	Components of the packages: quantity (pcs.) and thickness (t) of foils	Total number of foils in the package, pcs.
13TiNb1	1. 20 book elements from Ti-foil t = 45 μ m 2. 39 Nb-foils of t = 45–50 μ m	40 39
13NbTi2	1. 20 book elements from Nb-foil t \approx 50 µm 2. 39 Ti-foils of t = 45 µm	40 39
13TiNb3	1. 10 book elements from Nb-foil + 10 Nb-foils t \approx 50 µm 2. 11 book elements from Ti-foil + 9 Ti-foils t = 45 µm	20 + 10 = 30 22 + 9 = 31
13NbTi4	1. 10 book elements from Ti-foil + 10 Ti-foils t = 45 μ m 2. 11 book elements from Nb-foil + 9 Nb-foils t \approx 50 μ m	20 + 10 = 30 22 + 9 = 31

Table 1. Components of Nb/Ti packets in the 1st cycle of DW and PR

In the 1st cycle, the packs contained 31 and 40 niobium layers and one fewer layers of titanium or 31 and 40 layers of titanium and one less layers of niobium. The outer layers of the packets were only Nb- or Ti-layers only. The thickness of the packs was 2.9–3.0 and 3.7–3.8 mm, respectively.

In the second cycle, the packs were assembled in a certain sequence of several pieces of tape obtained after the 1st cycle, two or three plates of copper with a thickness of 0.15 mm or 2 or 4 Nb-foils of thickness 20 μ m, paved between copper plates and segments after the first cycle. These Nb-foils were diffusion barriers against the interaction of copper and titanium located in the Nb–Ti alloy.

If the plates of copper, as a stabilizer, were two, then they were located outside the package, if were three, then one plate was located also in the middle of the package.

The calculated thicknesses of individual layers of a solid solution of titanium in a niobium (Nb) and layers of NbTi alloy in a multilayer ribbon of finite thickness were ~150 nm.

Multilayer structure of composites after the 1st cycle. After the 1st welding, the structure of the Nb/Ti-package consisted of alternating light and dark bands parallel to each other (Fig. 2). Light bands corresponded to niobium, dark bands correspond to titanium.

Precise identification of the layers is established using local XSA. Concentration profiles near the interface between the layers of niobium and titanium (Fig. 3a and b) showed that after 10 minutes at 1050 °C and a relatively small pressure, it was sufficient for 10 minutes to form layers of the niobium-titanium alloy in place of the Ti layers. The concentration of Nb in them in the direction from the boundary to the middle of the layer decreased in the interval from 44 to 27 wt.%. The concentration of Ti increased from 56 to 73% by weight. In addition, the presence of diffuse formations with 75-85 wt.% Nb (spectra 7 and 8, Fig. 3a) was noted at the boundary between the layers.

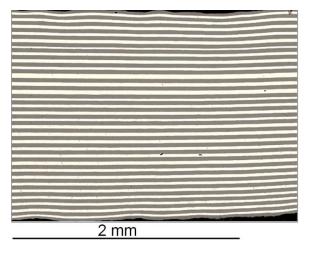


Fig. 2. The macrostructure of the cross-section of the Nb/Ti packet containing 31 Ti-layers and 30 Nb-layers after DW in the 1st cycle (see Table 1, 13TiNb3)

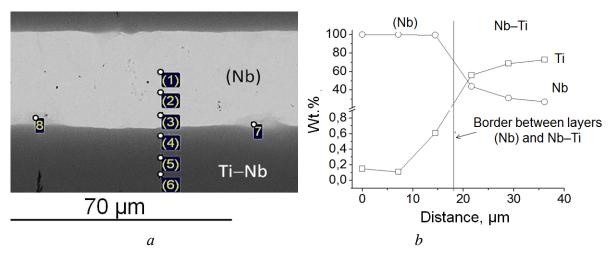


Fig. 3. Results of local X-ray spectral analysis: a fragment of microstructure (*a*) and concentration profiles of titanium and niobium (*b*) at the boundary between layers of a niobium solid solution (Nb) and an NbTi-alloy

Titanium in the layers of niobium was only slightly dissolved: in the middle of the Nb layer it was contained in an amount of less than 0.2 wt.% (see Fig. 3b). This meant that the solid-phase interaction This meant that the solid-phase interaction between the layers was due to the diffusion of niobium into titanium and had a positive effect on the formation of a layered structure in the composite, one of whose layers should be a layer of superconducting Nb–Ti alloy, close to the alloy with 50 wt.% Ti. The Nb–50Ti alloy has the best combination of superconducting characteristics in the Nb–Ti system. Other layers in the composite should be layers of niobium with as little titanium as possible. Then they will be non-superconducting even in small magnetic fields. It is known [14] that non-superconducting defects are more effective centers of pinning superconducting vortices than superconducting ones.

In Fig. 4 shows the cross-sectional structure of a multilayer tape with thickness ~ 0.2 mm after DW and rolling. The initial billet was a 13NbTi2 Nb/Ti composite containing 40 Nb- and 39 Ti-layers (see Table 1). Light layers are Nb-solid solution (Nb) containing titanium in an amount of several tenths of a percent. The dark layers are an alloy of Nb–(20–30) by weight of Ti. It turned out that in the direction across the rolling a liminality of the layered structure is expressed well than along rolling. In the cross section coinciding with the rolling direction (see Fig. 4*b*), a large number of lens-shaped bulges are present in the layers of the NbTi-alloy.

The calculated thicknesses of the layers (Nb) and Nb–Ti in the rolled strip after the first cycle were 2.7 and 2.4 μ m, respectively. But in reality their thickness varied from a few to 10–15 microns (see Fig. 4).

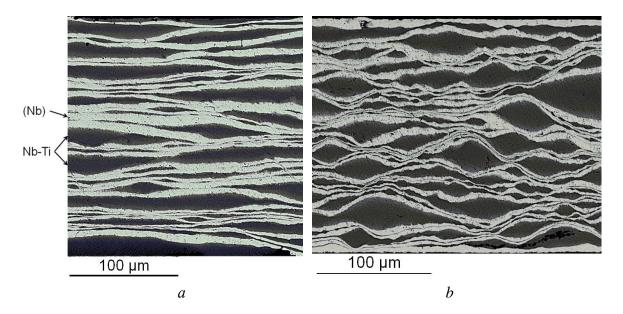


Fig. 4. Microstructure of Nb/Ti-composite tape 0,2 mm thick after DW and PR after 1 cycle across (*a*) and along rolling (*b*)

X-ray spectral analysis of the cross-section of the tape (Nb)/Nb–Ti confirmed the previous results. The dark layers of the niobium-titanium alloy contained ~34.5 wt.% Ti is the average titanium concentration calculated from the four spectra of 2, 3, 4, and 6 on the (Nb–Ti)-layers (Fig. 5).

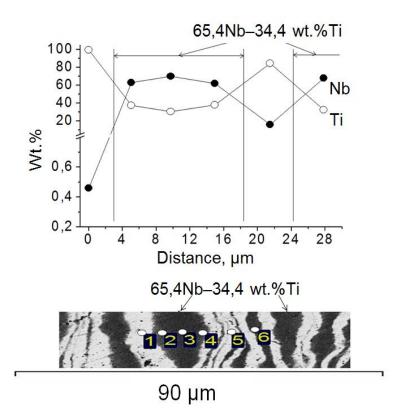


Fig. 5. On the results of local X-ray analysis: the concentration profiles of titanium and niobium and the fragment of the microstructure of the Nb/Ti-composite 13NbTi2 after DW and PR of the 1st cycle

The 2^{nd} cycle. In Fig. 6 shows the macrostructure of the packet collected in the 2nd cycle of 9 pieces of tape after the 1st cycle, and the results of local X-ray spectral analysis carried out at one of its section sections at a large magnification. The section of the packet considered is oriented parallel to the rolling direction of niobium and titanium foils.

When analyzing the given macro- and microstructures of the composite, it is visually seen that its layered character is determined by the light component of the material, in which the content of titanium at best does not exceed 10-15 % by weight.

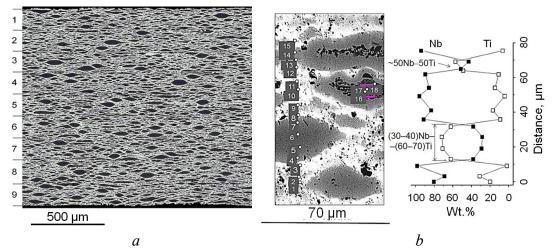


Fig. 6. 2nd cycle: *a* - macrostructure of the cross-section of a package of 9 pieces of tape after the 1st cycle; *b* - the results of XSA. DW: 950 °C at 19 MPa for 2 h

Significantly more often its content in it is in the range from 1 to 3-4 % by weight. This component creates a connected continuous system. A gray component of a niobium alloy with a titanium content of 50-70 % by weight forms elongated inclusions, which often acquire a lenticular shape and, therefore, seem intermittent. This suggests the erroneous conclusion that the niobium alloy responsible for the high superconducting current of the conductor does not have a coherent current path.

In the middle region of some thickening, the presence of inclusions differing from the surrounding volume with their black color was noted (Fig. 6b, spectra 16–18). They are enriched with titanium. Their composition is 21–24 Nb and 79–76 % by weight of Ti.

Critical current measurements

Low-temperature measurements were performed on tapes obtained by rolling composites from segments of a multilayer tape after the 1st cycle, Cubands as a stabilizer and thin Nb-foils, laid between the segments after the 1st cycle and copper. The structures of two such composites, which have not yet been subjected to rolling, are shown in Fig. 7. The first consists of seven multilayered segments (Nb)/Nb–Ti, two outer Cu-plates and two Nb-layers. The second composite, in addition to the two outer Cu-plates, also contains an inner Cu-layer, two packs of four multilayer segments (Nb)/Nb–Ti stacked together and four Nb-foils that are located between Cu and (Nb)/Nb–Ti. The superconducting current carrying volumes with a 2-phase layered structure of (Nb) + (Nb–Ti) in the 1st and 2nd composites are 79.6 and 75.3 % by volume.

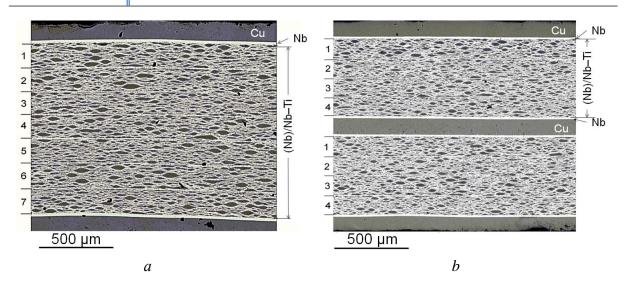


Fig. 7. Structure of composites after DW in the 2nd cycle:
a - 2 Cu-layer, 2 Nb-interlayers and 7 pieces of tape (Nb)/Nb–Ti after the 1st cycle;
b - 3 Cu layers, 4 Nb interlayers and 2 packs of 4 segments (Nb)/Nb–Ti

Concentration profiles of niobium and titanium indicated that there are no pure titanium layers in the (Nb)/Nb–Ti volume. The largest content of titanium, ~70 % by weight, was in the structural thickenings of the lenticular form (spectra 4, 5, 6 and 7, see Fig. 6*b*). Other sections of the Nb–Ti alloy layers look light gray in contrast. This is indirect evidence that the content of niobium in them is greater than in the spectra of 4–7. And, indeed, spectra 13 and 14 show ~50 wt.%Nb and ~50 wt.%Ti.

In the layers of light contrast identified as Nb-solid solution, the titanium concentration at best did not exceed 20, and often remained at the level of several weight %. Summarizing the foregoing, we can state that the solid-phase interaction between niobium and titanium occurs mainly due to the diffusion of niobium into the titanium layers, but not in the opposite direction.

Measurement of critical current. Low-temperature measurements were carried out on samples of the tape obtained by rolling the composites shown in Fig. 7. Their microstructure after rolling is shown in Fig. 8. Unlike the structure of the composite prior to rolling (see Fig. 6, 7), the layered structure appears to consist of uninterrupted layers of the NbTi alloy and a solid solution (Nb), respectively, of gray and light contrasts.

Directly for the measurement of the critical current I_c , the specimens were line segments of a multilayer tape after the 2nd cycle of DW and PR with a thickness of 0.1 mm and a width of 1 mm. The measurements were carried out in a cryostat with liquid helium in a magnetic field H produced by a superconducting solenoid, at perpendicular to H \perp (ab) and parallel to H || (ab) orientations of the direction of the magnetic field H and the rolling plane of the tape (ab) (Fig. 9). The transport current I through the sample in both cases was perpendicular to the magnetic field of the solenoid: I \perp H.

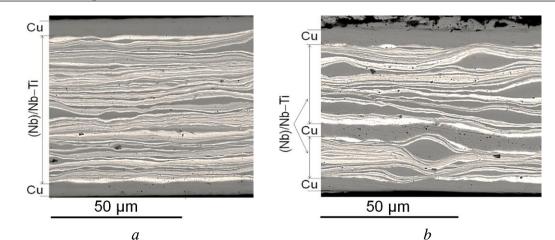


Fig. 8. Microstructure of the cross-section of a multilayer superconducting tape based on the Nb–Ti alloy with two (*a*) and three (*b*) stabilizing copper layers

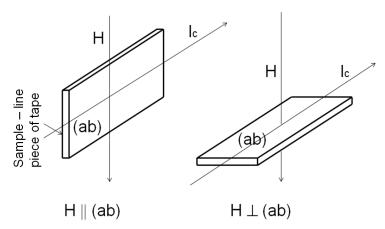
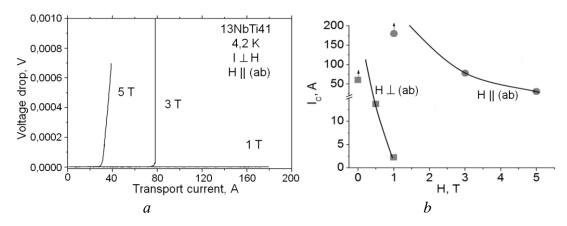
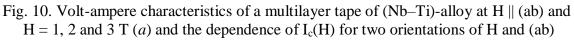


Fig. 9. Two orientations of the sample relative to the field H of the superconducting solenoid

In Fig. 10 shows the volt-ampere characteristics of a multilayer tape in fields 1, 3, and 5 T and H || (ab) and the dependence of the critical current I_c on magnetic field H at H || (ab) and H \perp (ab). Two experimental points with "upwards arrows" indicate that the transport current of such a force did not destroy the superconducting state of the sample.





At $H \perp$ (ab), I_c sharply fell with an increase in the field even in the range from 0 to 1 T (see Fig. 10 *b*). While at the orientation of H || (ab), it gradually decreased, remaining at 5 T at a level of 30 A, which corresponds to an engineering (design) critical current density of $3 \cdot 10^4$ A/cm². The anisotropy I_c , equal to the ratio $I_{c\parallel}/I_{c\perp}$, calculated for H = 1 T, was >82. This indicates the pinning of superconducting vortices localized in the Nb–Ti alloy at the interfaces between the layers of the non-superconducting Nb-solid solution (Nb) and the layers Nb50Ti-alloy, which carry a superconducting current.

It is important to note that such a current density was achieved in tapes without long, about 300–350 hours, low-temperature annealing at 300–350 °C. Such annealing is carried out for widely used multicore and multilayer superconducting (Nb–Ti)/Cu materials for the deposition of an α -phase, on which, in contrast to the above, superconducting vortices are pinned.

Summary and Conclusions

1. Based on the XS-analysis, it can be concluded that in the Nb/Ti multilayer composite as a result of solid-state interaction between foams of niobium and titanium in two cycles consisting of diffusion welding and batch rolling, the layers of the superconducting alloy (NbTi) of the composition Nb50Ti were received.

2. The Nb50Ti alloy layers, having the highest second critical magnetic field, are capable of conducting a large electric current in magnetic fields of 5 or more Tesla. Interlayers of the Nb-solid solution with a titanium content of up to 5–10 wt.% were turned into the normal state even in small magnetic fields and create the (Nb)-NbTi boundaries, which are effective pinning centers for superconducting vortices. Confirmation of this is the large anisotropy of the critical current $I_{c\parallel}/I_{c\perp} > 82$ in H = 1 T.

3. In multicore superconductors of an (Nb–Ti)-alloy of industrial production, the centers of fixation are α -phase particles that are released during long-term low-temperature annealing of already prepared wires. In the composite superconducting material Cu/Nb/NbTi of the solid-phase manufacturing process, in which vortex pinning takes place at the (Nb)-NbTi boundaries, a high critical current density was achieved without annealing.

The authors make it expressly clear that:

- 1. No conflict of interests has taken or make take place;
- 2. The present article does not contain any researches with people involved as the objects of researches.

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