

**STUDY OF MICROSTRUCTURE AND SUPERCONDUCTING PROPERTIES OF Nb₃Al-
TAPES, OBTAINED BY HEAT TREATMENT OF THREE-LAYER AND
MULTILAYER COMPOSITES Nb/AlCu**

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Abstract

The structure and superconductivity of three- and multilayer composite tapes of Nb/AlCu after high-temperature heating at 1750-1850°C for 1-3 seconds were investigated. Three-layer composites of Nb/AlCu/Nb with thickness of 50 microns were obtained by applying a vacuum rolling at 400-450°C and subsequent rolling at room temperature. Multilayer lengthy composites of Nb/AlCu obtained by extrusion with subsequent flattening by rolling of complex blank, collected in a special way. The effect of copper on the structure and properties of three-layer tapes was investigated. Copper increased the critical current of the superconductor. The critical current density in multilayered Nb₃Al-tape reaches the value $8 \cdot 10^4$ A/cm² in magnetic field 14 T. The critical bending radius of the tape is 7 mm.

Key words: *multilayer composite tape, extrusion, rolling, superconducting Nb₃Al-tape, high-temperature heat treatment, critical current density.*

1. INTRODUCTION

Niobium forms with aluminum three intermetallic compounds. The most refractory with a melting point of ~2100°C is a compound Nb₃Al. It is of interest for two reasons.

Recently, alloys of Nb-Al attracted attention due to their possible use as a heat-resistant material. The compound Nb₃Al (β-phase) has a fairly wide homogeneity region, which borders with the region α-solid solution of Al in Nb (Fig. 1). The solubility of Al in Nb decreases with decreasing temperature and, therefore, the alloy located at high (1800-2000°C) temperatures in the single-phase state on cooling is converted into a state with a relatively ductile matrix of α-solid solution hardened β-phase. Ratio of α- and β-phase change management is easy by increasing or decreasing of the original aluminum content in the alloy.

Second, the intermetallic compound of Nb₃Al is a superconductor with a critical temperature ~18 K. In spite of the existence of high-temperature superconducting ceramics, now to him as to the Nb₃Sn, remains of interest as a promising superconductor. Even before the discovery of high temperature superconductivity has been shown [1-4] that the high superconducting parameters of compounds Nb₃Al, formed in the three-layer composite tapes prepared by way of joint deformation by the rolling of niobium and aluminum, were achieved by heat treatment at 1700-1850°C for 1-3 seconds. After the further annealing at 800°C temperature of an onset of the transition to the superconducting state T_c equal to 18.0-18.5 K, the upper critical magnetic field at 4.2 K was ~30 T and critical current density j_c in the layer of β-phase at 4.2 K in a transverse magnetic field of 5 T reached ~2·10⁵ A/cm². An alloying of niobium-aluminum layer with copper increased j_c up to 5.7·10⁵ A/cm² [5].

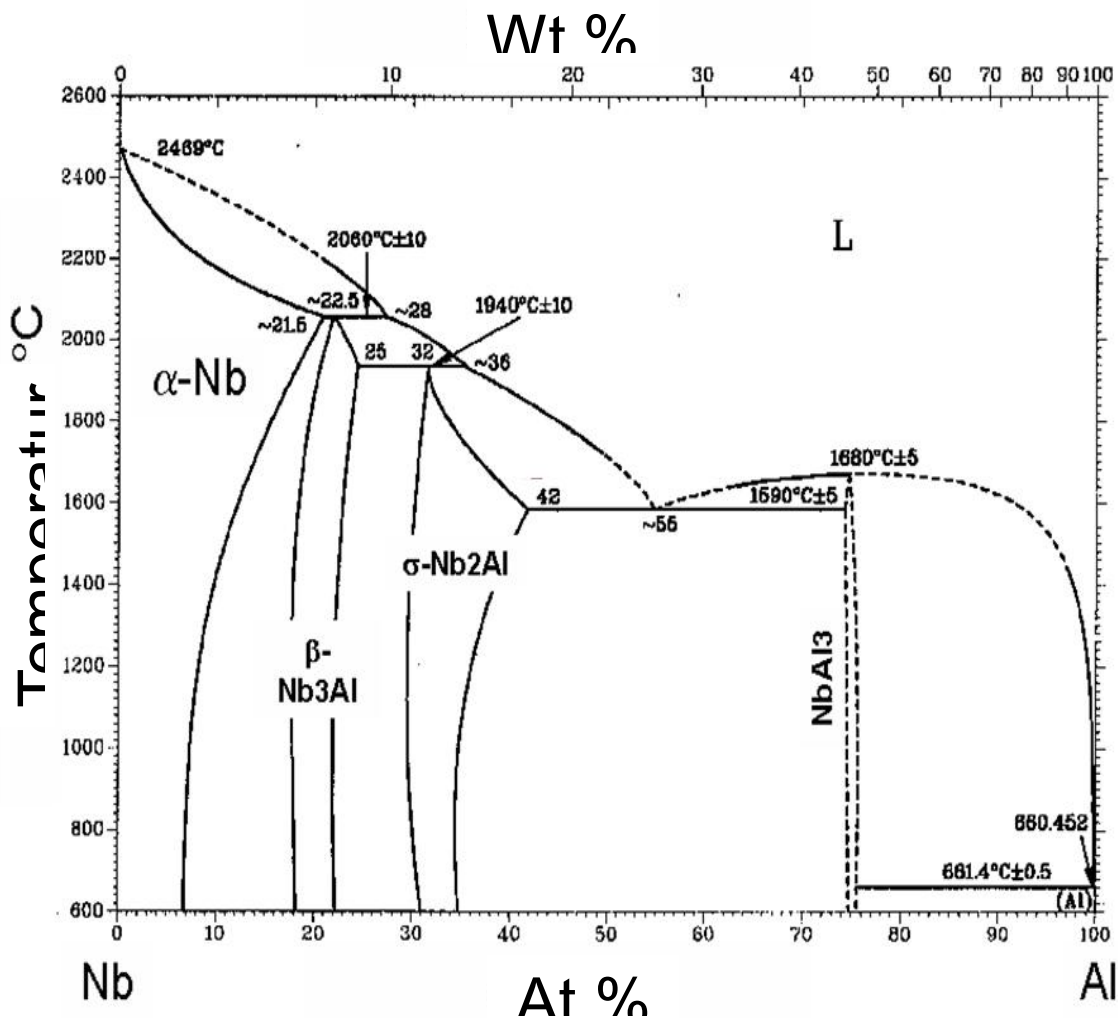


Fig. 1. State diagram Nb-Al

This paper deals with the following questions.

One. The studies of copper effect on T_c of the diffusion layer of Nb₃Al are showed that T_c for the samples with the addition of copper, in the first place, is somewhat higher (18.6-18.8 K) than in samples without copper, and, secondly, the high values of T_c is achieved after heat treatment: 1650°C/20-30 s + 800°C/2 h. The samples without copper after this treatment regime had T_c in the range of 17.0-17.7 K. Based on these data, one could assume that copper lowered for 150-200°C the formation temperature of Nb₃Al of stoichiometric composition. It was therefore interesting to investigate the effect of copper on the superconductivity of Nb₃Al at lower (1000-1300°C) formation temperatures of it in order to clarify the possibility to obtain high values of the critical temperature and the critical magnetic field.

Lowering the temperature of formation of the Nb_3Al compound is of great importance for an achieving of high current-carrying capacity of the intermetallic compound layer. It was shown [6] that the heating of a tape in the range of 1850-1950°C, even for a few seconds, caused a rapid growth of β -phase grain, so that the critical current density decreased with $\sim 10^5$ to $2 \cdot 10^5 \text{ A/cm}^2$.

Two. In terms of diffusion formation of the compounds in case reactive diffusion to reduce the building time of layers of β -phase advantageous to have a small diffusion distances, which should overcome the aluminum atoms to the meeting with the Nb-surface, i. e, small layers of σ -phase (Nb_2Al), which is formed before β -phase. The last is grew on the boundary between σ and Nb. From the solution of the diffusion second equation for finite body [7], we can estimate the thickness of the Al-interlayer in the tape in order to the formation of Nb_3Al was observed at 1000°C for 10 h. Assuming that the thickness of the β -layer will be $\sim 1 \text{ }\mu\text{m}$, the thickness of Al-interlayer must be $\sim 0.6 \text{ }\mu\text{m}$ (the diffusion coefficient $\sim 7 \cdot 10^{-14} \text{ cm}^2/\text{s}$ [8]). The achieving of this thickness of aluminum layers was carried out in a multilayer Nb/Al-tape by means of the rolling. A study of the superconducting properties of tapes was carried out at the annealing temperature in the range of 750-1050°C.

Three. The advantage of multilayer tape compared to the originally investigated three-layer tape is the ability to create a structure of alternating layers of superconducting compounds and niobium. In the multi-tape, due to the large number of layers of superconducting compound the critical current should not be sensitive to changes in thickness or even a local discontinuity of β -layer due to the mutual shunting layers. However, because of the large difference between the mechanical properties of aluminum and niobium and to achieve the correct (laminar) structure along the rolling direction is quite a challenge. Therefore, instead of the pure aluminum for a manufacture of multilayer strips of Nb_3Al , it was decided to try a more durable at room temperature duralumin – an industrial aluminum-based alloy of D16 grade.

2. PRODUCTION OF NIOBIUM-ALUMINIUM TAPE

2.1. Three-layer tape

The influence of copper on the critical temperature and the critical current density is carried out on the tapes after rolling consisting of two outer Nb-layers and one layer of the Al-Cu alloy, located in the middle. The copper content in the alloy was varied from 0.7 to 12 wt.%. The tape got by the rolling of three-layer Nb/AlCu/Nb packet consisted of plates of niobium and Al-Cu alloy at first on a vacuum rolling mill and then by the rolling at the room temperature. Also used the tape with a pure aluminum and duralumin ($\sim 4.5 \text{ wt.\%Al}$) of grade D16.

2.2. Multi-layer tape

In the multilayered tapes to improve the process of joint deformation of metal components of the composite was used instead of pure aluminum duralumin D16.

In the beginning was collected the work piece for an extrusion. Niobium and duralumin foils with a thickness, respectively, of 0.25-0.30 and 0.06-0.09 mm were formed together and with a special device tightly wound on Nb-rod diameter of 10 mm to as long as the outer diameter of a twist does not become equal to 33-35 mm. This twist was placed in a thick-Nb-tube and the whole assembly was laid in the Cu-glass with an outer diameter of 74 mm, which is top-pressed with the copper lid. The height of a fully assembled piece was $\sim 90 \text{ mm}$. Copper at extrusion performed as a lubricant, preventing sticking of niobium on the walls of a tool.

The deformation of twist in time the extrusion occurred so that the stretching of Nb- and duralumin foils comes across to the rolling direction of these foils. Elongation of cold deformed-Nb-foil in a

direction perpendicular to the rolling direction $\delta_{\perp}$ was equal to 0.4% (Table 1). Therefore, to increase plasticity before the winding the Nb-foil was annealed at 1100°C, and then $\delta_{\perp}$ increased to 9-10%. The foil of D16 before the winding annealed at 400°C for 1 h and quenched.

Table 1. The mechanical properties of niobium foil: σ_b – ultimate strength, σ_t – yield strength, $\delta_{\parallel}$ and $\delta_{\perp}$ – elongation of Nb-foil in a direction parallel and perpendicular to the rolling direction respectively

Heat treatment, °C	Along the rolling direction			Across the rolling direction		
	σ_b, MPa	σ_t, MPa	$\delta_{\parallel}, \%$	σ_b, MPa	σ_t, MPa	$\delta_{\perp}, \%$
deformed at 99%	520	340	3.0	390	260	0.4
1100, 1 h	255	180	22.0	265	140	9.0
1100, 5 h	250	190	23.0	260	150	10.0

Extrusion billets was conducted on a hydraulic press with a maximum force of 630 tons in a container with a diameter of 75 mm. Extrusion occurred through the matrix with the channel diameter of 30 mm. Before the extrusion billet was heated to 200-300°C in air. The resulting rod was cut into pieces, which were re-extruded. In our first experiments, they are extruded into a rod with a diameter of 10 mm (macrostructure of the cross-section of one of them is shown in Fig. 2), in the next – in billet $\sim 6 \times 20 \text{ mm}^2$. Then rods or billet rolled at the room temperature to 50-70 microns thick tape without intermediate annealing. The outer Cu-layers before the rolling quite easily removed by mechanical means on one of the initial stages of rolling after the cutting-off of the band edges. The edges were cut using a disc scissors.

For the formation of Nb₃Al-layers samples of the tape were subjected to two types of heat treatment in a vacuum: annealing in the range 750-1050°C for 0.5 to 10 h and high-temperature heating at 1700°C with an exposure of 1 to 30 seconds. In the first case the tapes as with a Cu-coated and without it were used, the second – without Cu-coating. High-temperature heating of the samples was carried out by direct passage through them of an alternating electric current.

3. METHODS OF MEASUREMENT OF THE SUPERCONDUCTING PARAMETERS

3.1. Measurement of the critical temperature

The critical temperature T_c was measured by an inductive method. In Fig. 3 shows a typical experimental curve of the superconducting transition of one of the samples. To compare the measurement results for the value of T_c was convenient to take the temperature of the beginning of the transition to the superconducting state, ΔT – the width of the superconducting transition.

The technique is such that allows you to capture all of the superconducting phase with critical temperature above the temperature of liquid helium. More high-temperature transition at $\sim 160 \text{ mV}$ corresponded formed by heating $\beta(\text{Nb}_3\text{Al})$ -phase the transition at a temperature of about 9 K matched to the niobium, which is always present in the tapes.

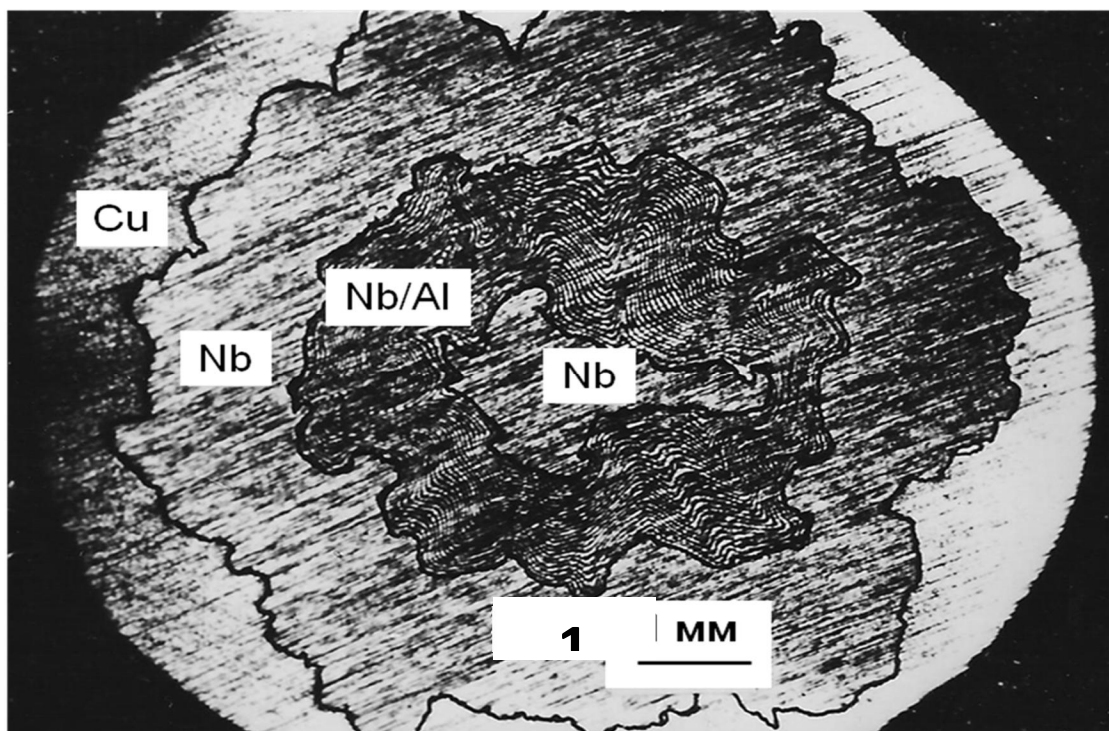


Fig. 2. Macrostructure of the Nb/Al-rod cross-section in Cu-shell diameter of 10 mm

3.2. Measurement of the upper critical magnetic field

From measurements of the upper critical magnetic field H_{c2} near T_c [9] dependence of $H_{c2}(T)$ was constructed and the slope $(-dH_{c2}/dT)_{T=T_c}$ was determined. The experiment consisted of T_c measuring at fixed values of the external magnetic field produced by a superconducting solenoid. The values of H_{c2} at 0 K were calculated by the formula $H_{c2}(0) = 0.69 \cdot T_c \cdot (-dH_{c2}/dT)_{T=T_c}$, where T_c is the intersection of the line $H_{c2}(T)$ with the abscissa. H_{c2} at 4.2 K was equal to $\sim 0.95 \cdot H_{c2}(0)$.

3.3. Measurement of the critical current

The critical current I_c was determined by resistive method at 4.2 K in a transverse magnetic field produced by a superconducting solenoid. For the accepted value of I_c was current at which the voltage drop across the potential contacts of the sample was 0.5-1.0 μV . Relative to the magnetic field the ribbon sample oriented in such a way that a direction of the field was parallel to the rolling plane of it and perpendicular to the transport current flowing through the sample.

Should make a distinction between the critical current density in the layer of Nb_3Al j_c equaled to the ratio of I_c to cross-sectional area of the layer, and a constructive critical current density j_{con} which is equal to the ratio of I_c to the total cross-sectional area of the tape.

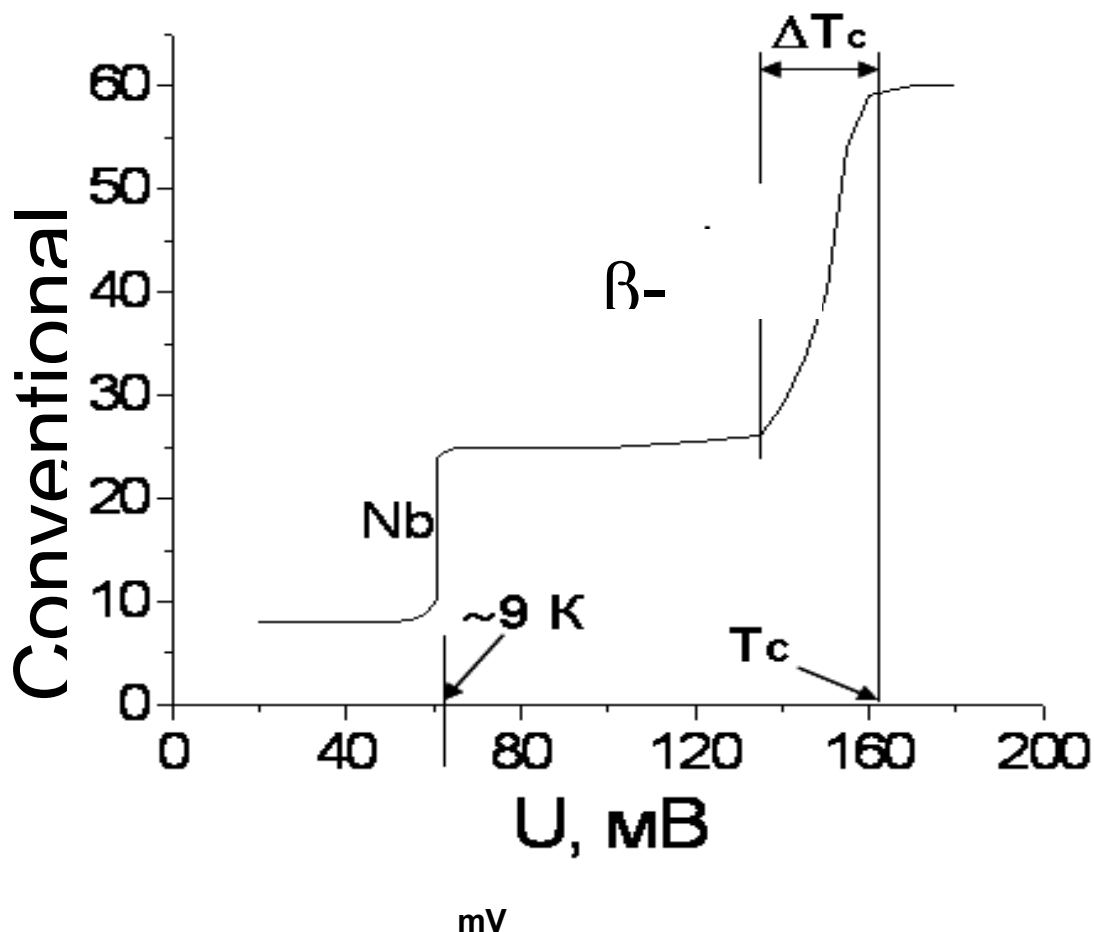


Fig. 3. The experimental curve of the superconducting transition of the Nb/D16-tape sample after heating at 1700°C for 5 s

4. THE INFLUENCE OF COPPER ON THE STRUCTURE AND SUPERCONDUCTING PROPERTIES OF TAPES FROM Nb₃Al

4.1. Samples and methods of investigation of the microstructure

Studies were performed on composite tape that produced by a way of an joint rolling of two plates made of niobium and one plate of an alloy of Al-Cu. As already mentioned above, the copper content in the alloy varied from 0.7 to 12 wt.% (aluminum alloys with more high copper content is bad resisted deformation). The thickness of the tape – 50 mm, the width – 2 mm. For the formation of a diffusion layer of Nb₃Al compound the tape was subjected to heat treatment, which consisted of the high-temperature heating with regimes for 1650°C for 20 and 30 s, 1750°C for 3 and 5 s and 1850°C for 3 s and the low-temperature annealing at 800°C for 2 h.

4.2. Methods of investigation of the structure of the diffusion zone between niobium and the alloy

Studies of the microstructure of the diffusion layer was carried out using metallographic analysis – to monitor the general picture of the cross section of the diffusion layer and to determine the average grain size of Nb₃Al compound (β -phase).

To determine the grain size of β -phase the flat surface of the investigated tape parallel to the rolling plane was subjected to grinding and polishing. Successive polishing and etching is performed several times until did not appear for the first time parts of β -phase layer, which borders with niobium. Etching time was 5-10 seconds. At the same time niobium is not etched and to distinguish β -phase with niobium was easy.

In addition, specifically carried out the studies showed that a microhardness of the sites of appeared β -phase significantly is more than microhardness of niobium. To reveal the grain of β -phase used chemical reagent consisting of equal parts of hydrofluoric and sulfuric acids and water with small amounts of hydrogen peroxide. Grain size was determined by the method of "cutting" [14].

Microstructural studies were carried out also using scanning electron microscopy of electronic scanning devices CamScan MV2300 and Vega 2, equipped with detectors of secondary and reflected electrons and X-ray microanalyzer. The content of elements in the layer in β -phase is determined by local X-ray analysis. A locality of the probe on the sample surface – 2 μm . The accuracy of determining the content of elements in the layers with thickness of 1-2 μm – 10%, in layers thicker than 2 μm – 3-5%.

4.3. Critical current

The best results were obtained in samples heated at 1850°C. The tape samples with 12 wt.%Cu in alloy Al-Cu in the magnetic field of 5 T had a critical current I_c equaled 38 A. Heating at 1650 and 1750°C gave smaller I_c .

Measurements of I_c depending on the alloy composition of Al-Cu can be divided into two regions (Fig. 4). The area 2 illustrates the increase in current-carrying capacity of tapes with increasing concentration of copper in the alloy from 4-8 to 30-38 A. The area 1 is the same, but the values of the critical current are much lower – increased I_c is from 1.5 to 3.6 A. the width of 2 mm.

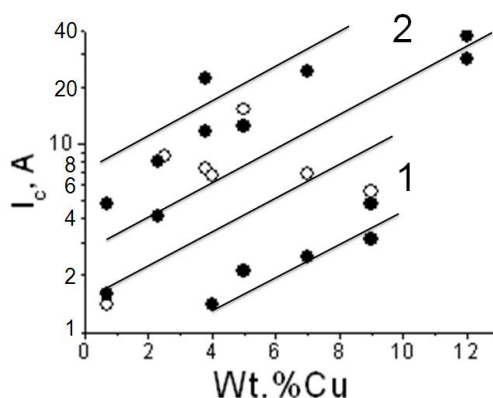


Fig. 4. The critical current I_c of Nb/AlCu/Nb-tapes on the copper content in an external magnetic field of 5 T and at 4.2 K after the heat treatments 1850°C for 3 s (○) and the same as + 800 for 2 h (●):

1 – samples with the diffusion zone of the β -phase, 2 – samples with the diffusion zone of $\beta + \sigma$.

The width of tape – 2 mm

4.4. Discussion of experimental results

The existence of two ranges of values of the critical current due to differences in the microstructure of the diffusion zones in the samples. It was found that the diffusion zone in a samples of the area 1 consists of only a layer of β -phase (Fig. 5a), samples of the area 2 are the diffusion zone of two layers of $\beta(\text{Nb}_3\text{Al})$ -phase and single layer $\sigma(\text{Nb}_2\text{Al})$ -phase (Fig. 5b). Found that copper is present in the layer of β -phase in a number of 0.1-0.2 wt.%, if the diffusion zone – the two-phase, and 0.7 wt.%, if it is single-phase.

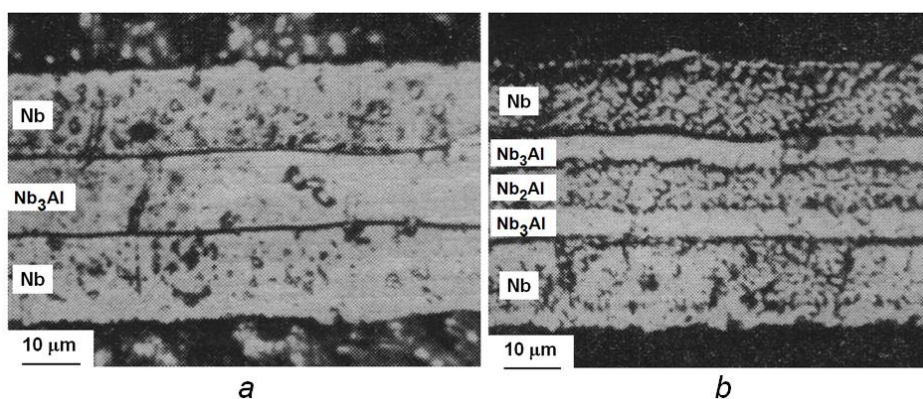


Fig. 5. Microstructure of the cross-sectional Nb/AlCu/Nb-tape after heat treatment: 1850°C for 3 s + 800°C for 2 h. *Wt.%Cu*: *a* – 5, *b* – 12

The diffusion zone is occupied by the third part of volume of the tape. After heating for 3 s, its diffusion zone could consist of as two layers of β -phase with thickness of 5 μm , adjacent to the niobium, and one layer of σ -phase with thickness of 8.7 μm so and only one layer of β -phase with of thickness of 12-14 μm . Such cases can be explained by small deviations of the volume ratio of niobium and alloy on the length of ribbon and slightly an inaccurate compliance of an exposure at high temperature heating. With the second exposures, even a slight deviation of 3 seconds can cause a noticeable deviation in the microstructure of the diffusion zone.

Metallographic studies to determine the grain size of β -phase showed that the average size of grains of intermetallic compound D for the samples of the two areas significantly differ. Samples of the first zone have the grains of β -phase greatly differing in size and many of them are elongated along the rolling direction of the tape (Fig. 6a). The average grain size of β -phase in the direction of the minor axis is equal to 4 μm and in the direction of the major axis can be up to 25-30 microns, and more. In contrast to these results, the grains of β -phase in samples from second zone in the total mass were significantly smaller (Fig. 6b).

It is generally accepted that pinning of superconducting vortices in the compounds with the structure of A15, to what is the Nb_3Al , occurs mainly at the grain boundaries. Therefore, despite the fact that copper increased the critical current of the tapes, the transition from the structure of the diffusion zone

of the two phases ($\beta + \sigma$) to the structure of the diffusion zone, consisting of one β -phase, was sharply reduced I_c , as D became much larger.

The critical current density j_c for the sample of the tape with the alloy of Al-12 wt.%Cu, for which there was a maximum value I_c , equaled to 38 A, is $1.3 \cdot 10^5 \text{ A/cm}^2$ in the layer with thickness of $5.5 \text{ }\mu\text{m}$ (Table 2). In the tape with a pure aluminum in the layer of β -phase of the same thickness $j_c \approx 6 \cdot 10^3 \text{ A/cm}^2$. An increase of j_c in the layer of Nb_3Al compound alloyed with copper, apparently due to the refinement of the grain of compound. Applied the method of determining the grain size of β -phase D has allowed to get a reliable value of $D \sim 3 \text{ }\mu\text{m}$ (Fig. 6b). In samples with pure aluminum D was at least 7-8 μm [4].

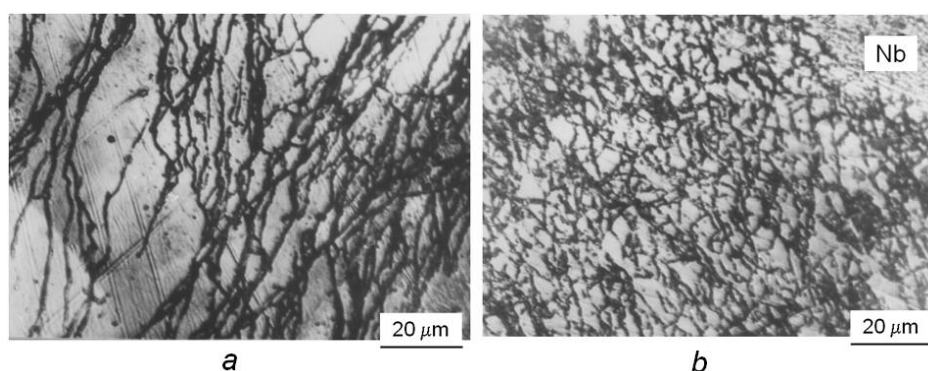


Fig. 6. The microstructure of β -phase layer in a plane parallel to the rolling plane of the tape Nb/AlCu/Nb after the heat treatment: 1850°C for 3 s + 800°C for 2 h. On the photo is visible a part of no etched niobium. Wt.%Cu: a – 5, b – 3.8

Table 2. The influence of the thickness of the β -layer on I_c and j_c

Components of the tape	The thickness of the β -layer, μm	5 T; 4.2 K; $H \perp I$	
		I_c , A/mm	j_c in the layer of β -phase, A/cm^2
Nb/Al/Nb	1.5	5.0	$1.7 \cdot 10^5$
Nb/Al/Nb	5.5	0.7	$6.0 \cdot 10^3$
Nb/AlCu/Nb	1.5	17.0	$5.7 \cdot 10^5$
Nb/AlCu/Nb	5.5	14.5	$1.3 \cdot 10^5$

Notes: 1. I – transport current through the sample.

2. In the calculation should take into account the two layers of β -phase.

An important result of the doping of β -phase layer with copper is the fact that with increasing thickness of the β -layer, the reducing of the critical current and critical current density in β -phase layer for samples with copper is far less than in samples not containing copper. This is clearly shown in

Table 2 – an increase of the thickness of β -phase layer from 1.5 to 5.5 μm causes a decrease of j_c in the samples with pure aluminum at about 30 times but in the samples with copper – at 4-5 times.

4.5. Conclusions to section

One. The effect of copper on the critical current of niobium-aluminum-niobium tape was researched. An adding of copper to aluminum in the investigated concentration range 0.7-12.0 wt.% increased I_c to 19 A/mm, the critical current density in the layer β -phase – up to $5.7 \cdot 10^5 \text{ A/cm}^2$ (the thickness of β -phase layer $\sim 1 \mu\text{m}$). For samples of the tape without copper these parameters, under all conditions being equal, was equalled, respectively, 7.5 A/mm and $2 \cdot 10^5 \text{ A/cm}^2$.

Two. The increase of I_c in the samples with copper due to the smaller grain size β -phase, which in the samples with the alloy Al-12 wt.%Cu was 3 μm (the high temperature heating – 1850°C for 3 s). In the tapes without copper D were not less than 8 microns.

Three. Found that the critical current density in the layer of β -phase, doped with copper, with an increase in the thickness of β -layer is reduced to a much lesser extent than in a β -layer of copper-free.

5. THE CRITICAL TEMPERATURE AND THE UPPER CRITICAL MAGNETIC FIELD OF Nb/AlCu/Nb-TAPES WITH HEAT TREATMENT AT TEMPERATURES OF 1000-1300°C

After annealing of the three-layer Nb/AlCu/Nb-tape at 1300°C during from 1 to 10 hours maximum values of the critical temperature T_c is achieved after exposure for 1 h and then remained practically unchanged. With increasing content of copper in the alloy Al-Cu T_c decreases for all annealing conditions. If, after annealing at 1300°C for 1 h T_c of Nb/Al/Nb-tape was equalled to 15 K, T_c samples with the alloy of Al-12 wt.%Cu was by 1 K less. The critical temperature of the Nb/D16/Nb-tape is 14.2 K.

Reducing the annealing temperature led to a rapid drop in the critical temperature. After the annealing at 1000°C in the experimental curves of the superconducting transition only one transition at around 9 K was observed, corresponding to niobium. Samples after annealing at 1100, 1200 and 1300°C, except for the transition of niobium, had and more high temperature transitions belonging to β -phase of niobium and aluminum, but with T_c , decreasing with decreasing temperature of annealing.

In the measurements of the upper critical magnetic field H_{c2} near T_c dependences of H_{c2} on the temperature $H_{c2}(T)_{T=T_c}$ were straight lines. It turned out that the magnitude of the slope $(-dH_{c2}/dT)_{T=T_c}$ is independent of copper content in the alloy of Al-Cu and equalled 1.6 T/K. However, due to the decrease of T_c , $H_{c2}(0)$ also decreased – after the annealing at 1300°C for 3 h from 17 T for Nb/Al/Nb-tape to 14 T for samples of the tape with alloy Al-12 wt.%Cu. The subsequent the low-temperature annealing at 800°C for 5 h increased the T_c at ~ 0.5 K.

Studies have shown that, contrary to hypothesis, the obtainment of diffusion layers of β -phase alloyed with copper, with high T_c and H_{c2} in the range 1000-1300°C was failed. In contrast to earlier results [5], copper was lowered T_c .

At temperatures of 1200 and 1300°C the 3-layers tape of Nb/AlGe/Nb with the alloy Al-30, and 45 wt.% Ge was also annealed. As expected, the superconducting characteristics of these tapes were higher than those of Nb/Al/Nb-tape. Temperature of beginning of the transition to the superconducting state after 5-hour annealing at 1300°C was equal to 15.8 K. The slope $(-dH_{c2}/dT)_{T=T_c}$ and $H_{c2}(0)$ were respectively 1.8 T/K and 20 T.

Previous [1, 2] and present studies of three-layer tape Nb/Al/Nb possible to trace the change of the critical temperature, depending on the heat treatment temperature in the range from 1100 to 2050°C (Fig. 7). The maximum T_c equaled to ~18.5 K is observed after heating at 1850-1900°C for 1-2 s. Decrease of heat treatment temperature to 1100°C reduced the T_c to ~12 K.

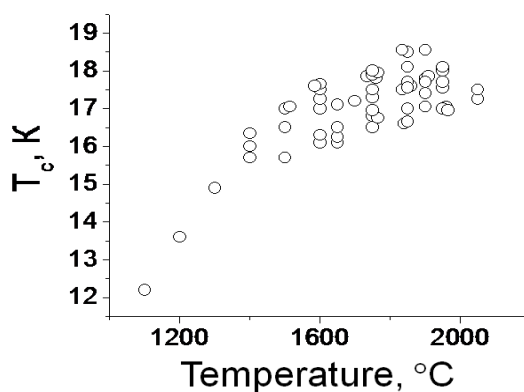


Fig. 7. The T_c dependence of Nb/Al/Nb-tapes of the heat treatment temperature at the optimum time of an exposure

The resulting dependence $T(t^{\circ}\text{C})$ essentially reflects the increase in critical temperature with increasing approximation to the stoichiometric composition of the compound and can be converted into a dependence of T_c on the aluminum content in the β -phase.

6. EFFECT OF ZIRCONIUM ON THE SUPERCONDUCTING PROPERTIES OF THE DIFFUSION LAYER OF Nb_3Al COMPOUND

6.1. Preparation of the tape samples

Influence of zirconium on the properties of Nb_3Al was examined for tape samples of two types. In the first case zirconium was injected into the aluminum (0.1-5.4 wt.%), in the second case – in the Nb (0.7-4.0 wt.%). Alloys of aluminum with zirconium were melted by the without crucible melting in an argon atmosphere. Ingots were solidified in copper molds in the form of plates with section of $3 \times 12 \text{ mm}^2$ and a length of 32-36 mm and were rolled at room temperature until the desired thickness. The alloys of niobium-zirconium were melted in an electron-beam furnace. The ingots with weighing of 0.9-1.2 kg were rolled at room temperature without intermediate annealing.

For the formation of the diffusion layer of Nb_3Al tape was subjected to high temperature heating up to 1650°C for 20 and 30 s and temperatures up to 1750, 1850 and 1950°C during from 2 to 8 s. The heating was carried out by direct passing of an electric current through the sample. After that low-temperature annealing at 800°C for 2 h was followed.

6.2. The tapes of Nb/AlZr/Nb

The critical temperature of the samples without the low-temperature annealing with the increasing of zirconium content in the alloy was varied nonmonotonically. For example, for the samples heated to 1650°C T_c initially was increased by 0.5-1.5 K at 0.4 wt.%Zr and then was fell by 0.5 K at 2.4 wt.%Zr.

The critical temperature of tapes with the heating at 1850°C had a minimum for the tape with alloys containing 0.4-1.6 wt.%Zr. However, all the changes of T_c were occurred in the interval ~ 1 K. Starting with the concentration of zirconium, equal to 2.4 wt.%, a values of the critical temperature for all temperatures of the high-temperature heating are lay by 0.2-0.5 K above the corresponding values of T_c for samples with pure aluminum.

Low-temperature annealing of the samples with heating at 1750 and 1850°C, first, was raised the temperature of the superconducting transition by 1 K, and in some cases by 2 h (samples with the alloy of Al-0.4 and 1.6 wt.%Zr and the heating at 1850°C) and, second, was smoothed the nonmonotonic change of T_c with the change in the composition of the alloy Al-Zr (Fig. 8a). At the same time T_c of about by 0.3-0.5 K higher than for samples with aluminum without zirconium. T_c was reached 18.9-19.0 K for samples with alloys containing 1.6 and 3.3 wt.%Zr, heated at 1750°C for 5 seconds.

Samples without the low-temperature annealing had the slope $(-dH_{c2}/dT)_{T=T_c}$ in the range from 2.4 to 2.7 T/K which practically did not change with increasing of concentration zirconium in the alloy Al-Zr. The upper critical magnetic field $H_{c2}(0)$ for samples with Al-Zr alloys containing up to 2.4 wt.%Zr was equaled 30 T and at further increase of zirconium content was increased to 32-34 T (Fig. 8b).

The values of experimentally defined slope of the dependence $H_{c2}(T)$ near T_c and $H_{c2}(0)$ of Nb/AlZr/Nb-samples were greatly changed after the low-temperature annealing (Fig. 8b). The value of the slope for the sample with the alloy of Al-3,3 wt.%Zr after heating at 1850°C for 3 seconds and the low-temperature annealing was reached 3.5-3.7 T/K. Now, if we calculate the upper critical magnetic field at 0 K, then for Nb₃Al $H_{c2}(0)$ was reached 46-48 T against 32 T for the tape samples with pure aluminum. At 4.2 K $H_{c2} = 44.0-45.5$ T.

Unfortunately, the critical currents of Nb/AlZr/Nb-tapes were small and ranged from 0.4 to 1.4 A in a magnetic field of 5 T at 4.2 K.

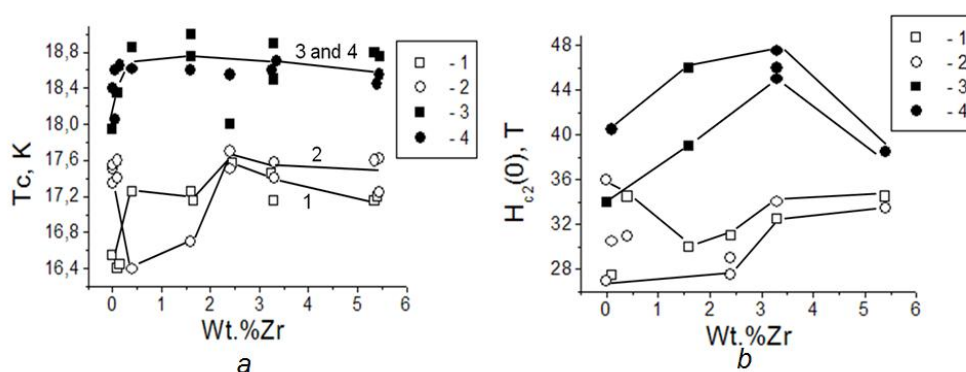


Fig. 8. Dependences of the critical temperature T_c (a) and upper critical magnetic field at 0 K $H_{c2}(0)$ (b) for samples of tape Nb/AlZr/Nb depending on the content of zirconium in the alloy Al-Zr.

Heat treatment, °C: 1 – 1750/5 s; 2 – 1850/3 s, 3 – 1750/5 s + 800/2 h; 4 – 1850/3 s + 800/2 h

6.3. The tapes of NbZr/Al/NbZr

Dependences of the critical temperature on the concentration of zirconium in the alloy Nb-Zr for all regimes of the high-temperature heating had the same look. At 0.7-1.6 wt.%Zr curves were showed a minimum. The values of T_c at a minimum were by 2 K lower than for tapes without zirconium, and equaled 14.0-14.6 K for samples not subjected to the low-temperature annealing. With further increase

of zirconium content T_c was increased, but its value does not exceed the values of T_c for niobium-aluminum tapes. Low-temperature annealing, as expected, increased the T_c of the samples without changing the nature of the curves.

As in the tape of previous design, the critical currents of tapes were small, not exceeding 1.0-1.3 A in the field of 5 T. However, the values of I_c , depending on the concentration of zirconium is completely repeated the same dependences of T_c .

For some samples of tapes H_{c2} near the critical temperature were measured (Table 3). Lowest values of the slope of $(-dH_{c2}/dT)_{T=T_c}$ and H_{c2} again were fell to the concentration of zirconium in the alloy Nb-Zr equaled to 1.6 wt. %.

Table 1. The results of measurements of H_{c2} near T_c for the samples of NbZr/Al/NbZr-tape

Heat treatment, °C	Wt. %Zr	T_c , K	$(-dH_{c2}/dT)_{T=T_c}$, T/K	$H_{c2}(0)$, T	$H_{c2}(4.2\text{ K})$, T
1750/3 s	0.7	15.7	3.3	35	32
1750/3 s	1.6	15.4	3.0	31	29
1750/3 s	2.5	16.7	3.6	40	37
1750/3 s + 800/2 h	2.5	17.6; 17.7	2.8-3.0	34-36	32-34

From the metallographic analysis of these tapes drew attention to the fact that a non-monotonic dependence of the thickness of the layer β -phase on the zirconium content in the alloy. The smallest thickness of the β -layer (0.7-0.9 microns after the heat treatment at 1750°C for 3 s) was observed in the sample with 1.6 wt. %Zr. Tapes of this alloy, as we have seen, had and the lowest values of superconducting parameters.

6.4. Discussion of experimental results

It was expected that the addition of zirconium in aluminum and niobium will enhance the critical current of Nb₃Al. However, this does not give a positive result. The critical current of Nb₃Al-tapes with the addition of zirconium was extremely small, the critical current density in the layer of β -phase was a $(1-2) \cdot 10^5\text{ A/cm}^2$. The data were somewhat unexpected, especially since there are enough works, where the introduction of zirconium in the substrate increased I_c of compounds such as Nb₃Sn and V₃Ga. This fact authors were explained by the presence of particles ZrO₂ which, being located along the grain boundaries of A-15 compound, delayed a growth of them and thus the critical current was increased.

Lack of the increase in the I_c of Nb₃Al can be explained as follows. Apparently the direct introduction of zirconium in aluminum or niobium is ineffective because of oxygen (if it is contained in the alloy) for the second exposure at high-temperature heating did not have time to oxidize zirconium. It should create a two-phase structure in alloys – solid solution of Nb and Zr + ZrO₂ or Al + ZrO₂ – before the high-temperature heating. Perhaps in this way particles of ZrO₂ by brief heating will have time to play a positive role.

Nonmonotonic dependences of the critical temperature on the concentration of dopants, similar to our, were obtained for diffusion layers of Nb₃Sn at alloying of niobium by zirconium and titanium [13], molybdenum and tungsten [14]. Here, in the range 0.1-0.3 wt. %X (X = Zr, Ti, Mo, W), T_c had minimum and further were increased. As we are the authors of these papers do not give a reasonable

explanation for a nonmonotonic change of the superconducting parameters depending on the concentration of alloying elements. Most likely this is due to fundamental reasons for the influence of added elements on the electronic structure of the A15 phase.

Increased upper critical magnetic field H_{c2} of diffusion layers of Nb_3Al in tapes of composition of $NbZr/Al/NbZr$, in comparison with the tapes without zirconium, we connected with the scattering of electrons by impurity atoms. This leads to a decrease in length of the free path of electrons in the normal state l_e , thus increasing the Ginzburg-Landau parameter and H_{c2} .

In another case for tapes of composition $Nb/AlZr/Nb$ in which H_{c2} increased only by low-temperature annealing. The decrease l_e may be caused by beginning stages of a decay of supersaturated (after high-temperature heating) by aluminum β -phase with the preformation of particles of $\sigma(Nb_2Al)$ -phase. Zirconium dissolved in the phase, is able to accelerate its decay. Then in tapes of $Nb/AlZr/Nb$ the last starts at $800^\circ C$ for 2 h but such annealing is not sufficient to initiate the collapse of β -phase in the tapes without zirconium.

7. THE MULTILAYER Nb/Al-TAPE AFTER HEAT TREATMENT AT TEMPERATURES 700-1050°C

For a given temperature interval the maximum values of the critical temperature were obtained after annealing at $900-1000^\circ C$. Top of the superconducting transition T_c occurred at 12.8-13.4 K. The maximum attainable T_c at $950^\circ C$ was obtained after annealing for 1 h and then remained almost constant. At $850^\circ C$ the maximum T_c was achieved after annealing for 7-10 hours, however, the middle of the superconducting transition in all cases did not rise above 10.0-10.8 K.

On the experimental curves of the superconducting transition, the separation of superconducting transitions of niobium (at ~ 9 K) and more high-temperature β -phase, it was possible for the samples annealed at $800^\circ C$ and higher. At lower temperatures and short times of the annealing the transitions of niobium and β -phase is not separated, and the effect of annealing was expressed in the blurring of Nb-transition toward higher temperatures. This situation is observed, for example, for tapes with a Cu-coating after the annealing at $950^\circ C$ for 30 min: $T \approx 13.8$ K, the middle of the transition – 9.5 K.

In experiments measuring H_{c2} near T_c the measurement of temperature of the superconducting transition was measured by the resistance. This measurement technique has always given more abrupt transitions, compared with the measurements of T_c by the inductive method. The tape with the Cu-coating after annealing at $950^\circ C$ for 3 h had $T_c = 14.9$ K with transition width ΔT_c of 1.4 K. The slope $(-dH_{c2}/dT)_{T=T_c} = 1.3$ T/K, $H_{c2}(0) = 12.5$ T and $H_{c2}(4.2$ K) = 1.15 T.

The critical current density j_c , the calculated for cross section of all tape in a magnetic field of 5 T was equaled to $2.5 \cdot 10^3$ A/cm² after annealing at $850^\circ C$ for 3 h and 180 A/cm² after annealing: $1000^\circ C/3$ h.

8. STRUCTURE AND CRITICAL CURRENT OF MULTILAYER SUPERCONDUCTING TAPE FROM COMPOUND Nb_3Al

8.1. Introduction

Laboratory technology using the rolling in a vacuum, developed and successfully used for the manufacture of three-layer tape could not provide the receipt of long (up to 100 m) segments of the superconducting Nb_3Al tapes. Furthermore, it was clear that the high-temperature (up to $1850^\circ C$) heating by direct passing of an electric current through a fixed piece of tape unacceptable for a lengthy

heat treatment of the tape. In this connection there were two tasks: to develop technology to produce long segments of niobium-aluminum tape, and to construct and test a facility for high-temperature heating of the tape in a continuous mode (mean by "to pass").

The technology of manufacture of a multilayer tape more or less fully described above and in [13]. There also the superconducting properties of layered tape are presented, but it is heated by passing an electric current. So here we confine ourselves only to the designation of stages of the technology. The production of tapes included: collection of the work piece, extrusion, and rolling it at room temperature.

The next section will dwell on the hydro-extrusion, tested for the production of the long tape segments of Nb₃Al.

8.2. The use of hydro-extrusion for the production of multilayer superconducting niobium-aluminum tape

The use of high hydrostatic pressure has been widely spread in a metalworking. Hydro-extrusion is an ideal method from the viewpoint of opportunities in the technology of cold working of metals. A favourable scheme of the stressed state of high hydrostatic pressure can reach high degrees of deformation of the material without breaking, even if in the normal state the material is little plastic and hard deformable. Hydro-extrusion may be, in some cases the only possible way to get product from these materials.

In the field of production of superconducting materials is an important issue on the length of pieces of wire or tape superconductor. This is particularly evident for the compounds with the structure of the A-15 with the joint deformation of dissimilar metals. The final tape length is limited by the size of the original piece.

In the usual pressing of the multilayer niobium-aluminum work piece perform was impossible deformation with large compression (for example, from diameter 75 to 10-15 mm). When pressing the work piece is heated to 200-300°C. However, due to the low temperature the resistance of the strain was great and pressing is not performed. And in the case if it was possible to implement, because of the large compressions in focus of the deformation took place additional heating, which led to the melting of aluminum.

We tested the technological cycle using hydro-extrusion, which is allowed to get the long pieces of multilayered Nb/Al-tape. The sequence of operations was as follows: 1) collection of the work piece, 2) the deformation by pressing of it in the rod with a diameter of 25-30 mm, 3) hydro-extrusion and 4) rolling. When assembling the work piece the thickness of the copper cup was calculated so that after the pressing on the rod with a diameter of 25-30 mm and a turning of the rod to a diameter of 18.5-22.5 mm in the sample remained a copper layer with thickness of 0.5-2.0 mm. Extrusion of such billets with length of 200 mm was carried out at the facility of Institute of High Pressure Physics RAS [14]. Pre-lubricated billet cover – ktiol CT-15 or MoS₂. The matrix was the angle $2\alpha = 30^\circ$. The degrees of deformation of billets were 76%, working pressure of the liquid – 12.5-16.0 kbar. The final diameter is 9 mm.

Hydro-extrusion allowed carrying out the deformation of the multilayer Nb/Al-billets without preheating with the high compressions. The length of rods was obtained ~0.8 m. Note that the length of the original piece in this case is limited by the size of the camera, which optionally can be increased. A violation of the continuity of the material and the inner melting of aluminum was not observed. After hydro-extrusion bars were rolled at room temperature in a tape with the thickness of 50 microns. The number of layers of aluminium is ~50.

8.3. Facility for the high-temperature heating of long segments of Nb/Al-tape

In the known domestic and foreign literature does not meet the description of plants intended for the heat treatment sufficiently long and thin conductors at high temperatures for a few seconds. Only A. Müller [15] was described the experimental results of the study of Nb/Al- and Nb/AlGe-tapes, heat-treated at 1700-1900°C in a way that, as you can tell from the brief description of the method of heating, similar to the heating method, implemented in the proposed facility.

An installation for the high-temperature heating consists of a chamber, system of vacuum evacuation, and control panel. General view of the facility is shown in Fig. 9. The camera is a cylindrical tube with an inner diameter of 300 and 800 mm long, horizontally mounted on the frame. The side flanges are made opening the chamber. On the flanges and the camera provides four quartz glass windows that allow observing a passage and heating of the tape. In the middle of the chamber a water jacket was welded to protect the chamber walls from heating due to radiation. In places where the electrical insulation was required from the hull, pyrophyllite was used, details of which before the assembly were subjected to the heat at 1200°C for 2 hours.

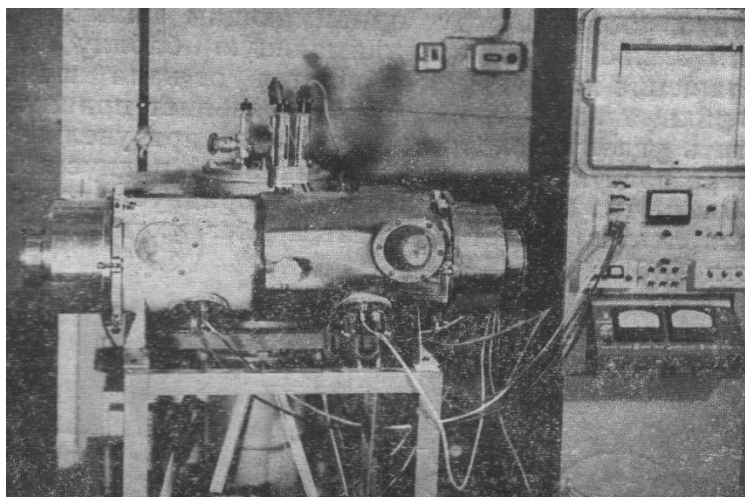


Fig. 9. Facility for the high-temperature heating of long segments of the multilayer Nb/Al-tapes

A vacuum of $1 \cdot 10^{-5}$ mm Hg was achieved within 40 min from the time of the pump. During the high-temperature heating vacuum was reduced, but did not fall lower than $5 \cdot 10^{-5}$ mm Hg.

8.3.1. Heating device

The heating element is a thin-walled (thickness of wall – 0.3 mm) tantalum tube with diameter of 10 and 150 mm long. To its ends by means of copper bars supplied the electric current of industrial frequency. We have adopted the length of the working area – 50 mm. To reach 1800°C a current flowing through the tube is ~200 A. The temperature is controlled by W-Re-thermocouple, which lies in the isolation of beryllium oxide. The signal from the thermocouple is fed to a precision temperature controller. Outside and to the ends of the heating element is surrounded by several W-screens. All heating device for the isolation of the housing is fixed on plate of pyrophyllite 15 mm thick, which in turn is attached to the rack inside the chamber.

8.3.2. Kinematic scheme of passage of the tape

The passage of the tape is realized by two pulleys, the rotation of which is given by the DC servo motors, located on the same roll with pulleys. The scheme of passage includes disc coil filled with the tape and taking disc coil, pinch, stretch, and the guide rollers. With help of reducers you can vary speed movement of the tape in a fairly wide range. Friction device allows the taking disc coil to reduce the angular velocity with increasing radius of the winding as the supply of tape. The initial radius of the winding in the disc coil is 30 transmit *mm*, which exceeds the critical radius at which the tape after the heat treatment can be bent without affecting current-carrying capacity. All pulleys are designed for the transmitting of tape with width of 5 *mm*.

8.3.3. The electrical circuit of the facility

Functional electrical circuit of the automatic speed regulator of tape passage is built on the principle of the tracking system and is included: a sensor, a high frequency generator, an amplifier, a subtracting device, a safety device, a scheme of control of motors speed and power supply. Appearance of an appliance and block-diagram of speed regulator of tape passage are shown in Fig. 10 and 11.



Fig. 10. Appearance of an appliance for a control of speed of tape passage

The sensor is an element that determines the determining the accuracy and the stability of the entire tracking system. For our particular case has been developed non-contact sensor based on the principle of changing of the coupling coefficient and represents itself the two resonant circuits differentially connected and spaced apart from each other at a distance. To protect against induced electromagnetic fields the contours are enclosed in the screens, through which the gap is cut. Between the contours is available freely hanging tape, on which through the brush the current flows from the high-frequency generator. Thus, a piece of the tape is the third circuit, emitting an electromagnetic field of a certain frequency, on which set up two differentially connected receive ring circuits. Mismatch voltage of the two circuits depends on the bias of tape $\pm\Delta y$ (see Fig. 11) relative to the average situation.

The amplifier is designed to amplify signals from the sensor, filtering and transfer these signals to the inputs and subtracting the protective devices.

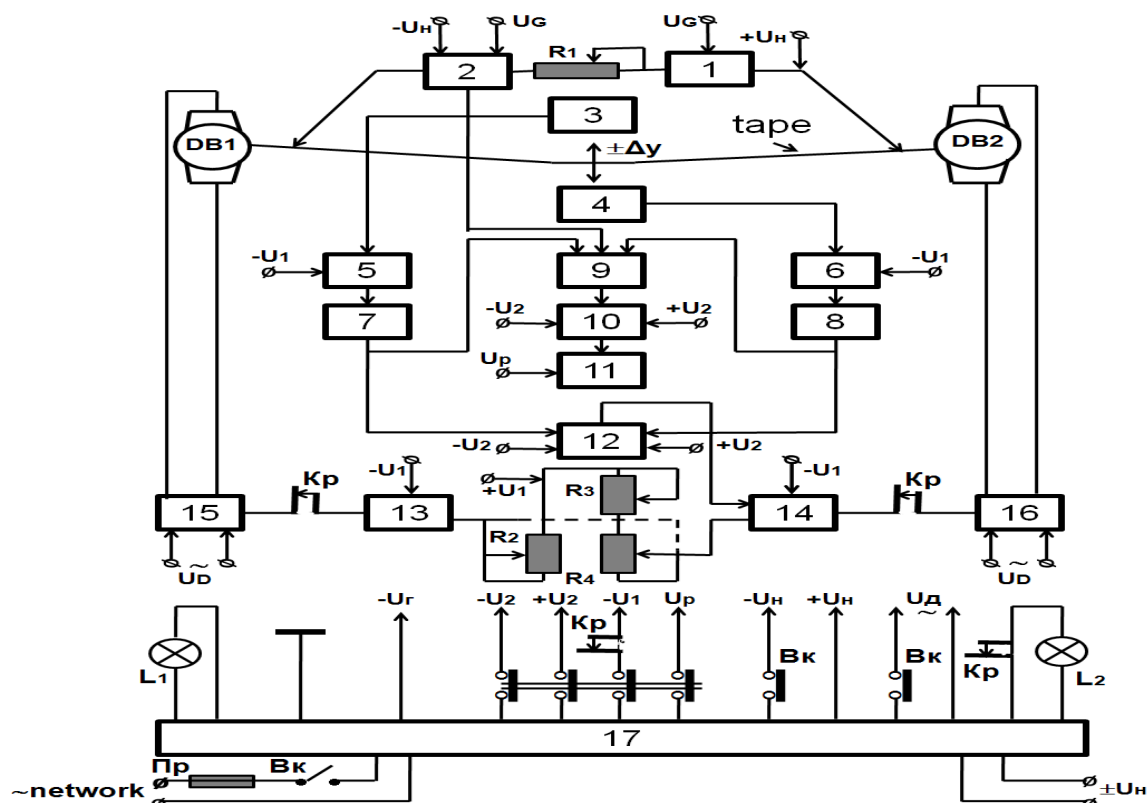


Fig. 11. Electrical block-diagram of speed regulator of tape passage: 1 – generator; 2 – switch of break; 3, 4 – sensors of tape position; 5, 6 – amplifiers; 7, 8 – detectors; 9 – block of outcomes; 10 – the threshold circuit, a comparator; 11 – the executive block with the electromagnetic relay; 12 – operational amplifier; 13, 14 – shaping circuit of pulse of control; 15, 16 – power executing units; 17 – power supply unit; DB1, DB2 – electric motors; R1 – sensitivity control of management system; R2, R4 – controls of the rotational speed of both engines; R3 - control of the rotational speed of engine DB2; Kp – contacts of relay; Bk – switches; L1, L2 – signal lamps (L1 – switch of fault); Пp – safety; U_G and U_r , U_1 , U_2 , U_p , U_d and U_D , and U_H – generator voltage, voltage of automation units, voltage of operational amplifier, voltage of actuating unit, electric motor voltage, and voltage for the tape respectively

Signal proportional to the displacement of the tape from the average position is removed on the input of subtracting device and recorded by means of the needle deflection of the micro ammeter located in the middle of the front panel of an appliance (see Fig. 10).

The protective device is designed to disconnect a power of the amplifier and the subtracting device, to turn off electric motors and to include of light signaling if the heated tape is breakage. Control circuits of the speed of engines are operated on phasepulse "vertical" method [16]. Each engine has its own circuitry of control.

9. TEST RESULTS OF TAPE AFTER HIGH-TEMPERATURE HEATING IN CONTINUOUS MODE

The test results of facility for the high-temperature heating of long segments of the multilayer tapes Nb/D16 (D16 is industrial aluminum-based alloy containing 4.4 wt.% copper) in the continuous mode is shown in the example of the three modes with different parameters of heating (Table 4). The residence time of each section of the tape in the heating zone was set by the speed of tape passage v , which is installed with the help of the receiving pulley. The angular velocity of it in during the all heating process on the selected mode remains constant.

Table 4. Modes of tape passage

Temperature of high-temperature heating, °C	ω , turn./min	v , mm/s	Heating time, s	Performance, m/h
1700	0,2	0,8	62,5	2,9
1800	5,4	25,8	2,0	92,9
1800	8,6	39,0	1,3	140,4

At the first trial limited segments of the tape with a length of ~ 3.5 m. Width of the tape – 2 mm. After high-temperature heating, each piece of tape was cut into several pieces and from each of them the three specimens were taken for measurements of critical current immediately after continuous heating and three specimens – for measurements after continuous heating and followed by low-temperature annealing at 800°C for 3 h. The annealing was performed in a vacuum furnace of resistance.

The values of the critical current along the length of the tape segments, heat-treated in three modes are shown in Fig. 12-14. After heating at 1700°C for 62.5 s the tape had a small value of the critical current I_c , equal ~ 1 A in a magnetic field of 5.5 T. The low-temperature annealing increased I_c to approximately two-fold (see Fig. 12). Based on previous studies [1, 2], we can conclude that at the relatively long heating time (~ 1 min) formed a layer of intermetallic compound of Nb₃Al owing to a diffusion of aluminum around all section of the tape began depleted in last, while not completely turned into a solid solution of aluminum in niobium.

Otherwise was observed in the tapes after the high-temperature heating at 1800°C for 2 s (see Fig. 13). Level of values I_c immediately after the heating is low (4-5 A), but subsequent annealing increased I_c to 70-80 A. Some higher critical currents were obtained after high-temperature heating for 1.3 s (see Fig. 14): 9-10 A – no low-temperature annealing and 100-120 A – after annealing at 800°C. A large number of samples failed to destroy by the current of 115-120 A – the ultimate current of source that we had.

From the analysis of the results shows that the critical current is strong enough varied along the length of the tape. The error in determining of the average value of I_c for tapes without the low-temperature annealing, have undergone the heating in a continuous mode on the second and third embodiment, was reached 37%. The smallest value of I_c was differed from the maximum at about 6.5 times (Table 5). However, it should be noted that of all the samples passed the heating at 1800°C for 2 s, I_c at 2 A and less there was only in two samples. For samples with heating for 1.3 s I_c was less than 6 A – in three. This could be due, even with random kinks of samples in the process of preparing them for the measurements.

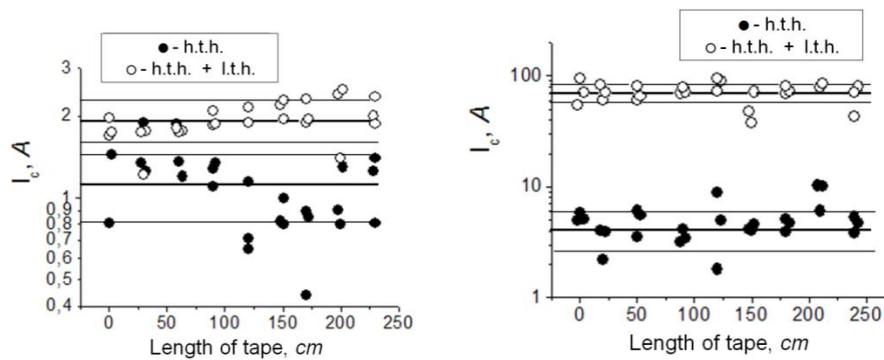


Fig. 12. The values of I_c along the length of the tape after high-temperature heating (h. t. h.) at 1700°C for 62.5 s ($v = 0.8$ mm/s): ● – without the low-temperature heating (l. t. h.) and ○ – after l. t. h. at 800°C for 3 h (5.5 T; 4.2 K)

Fig. 13. The values of I_c along the length of the tape after h. t. h. at 1800°C for 62.5 s ($v = 25.8$ mm/s): ● – without the l. t. h.) and ○ – after the l. t. h. at 800°C for 3 h (5.5 T; 4.2 K)

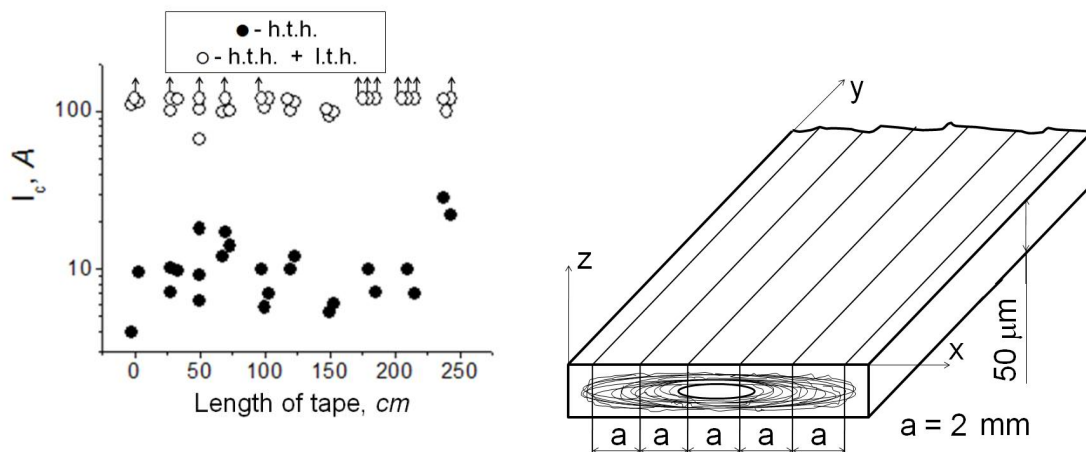


Fig. 14. The values of I_c along the length of the tape after h. t. h. at 1800°C for 1.3 s ($v = 39.0$ mm/s): ● – without the l. t. h.) and ○ – after the l. t. h. at 800°C for 3 h (5.5 T; 4.2 K). The up arrow indicates that the electric current of such value does not transfer the sample to the resistive state

Fig. 15. Schematic representation of the cross-sectional structure of a wide tape, being cut into tapes of 2 mm wide

Table 5. Results analysis of measurements of critical current of tape (5.5 T, 4.2 K, tape width – 2 mm)

Mode of high-temperature heating, °C	The average value of I_c , A		The range of changes of I_c , A	
	without the l. t. h.	800°C for 3 h	without the l. t. h.	800°C for 3 h
1700/62.5 s	1.1 ± 0.2	1.8 ± 0.3	$0.4 \div 1.8$	$1.2 \div 2.6$
1800/ 2.0 s	4.1 ± 1.5	71.0 ± 12.0	$1.8 \div 11.0$	$36.0 \div 96.0$
1800/ 1.3 s	10.8 ± 4.0	$106.0 \pm 8.2^*$	$4.1 \div 27.5$	$92.0 \div >120$

*For samples that have fallen into the resistive state

A more general reason for the change of I_c along length of the tape is associated with varying cross-sectional structure of the tape in the direction of y (Fig. 15), which is a consequence of the method of manufacturing the tape. The tape had some "sickle-shapes", due to which cut out of the broad tape the band with width of 2 mm can have different cross-sectional structure along the length. The most unfavorable turned out to be the case when to come across sections of the outer regions of a wide band, which accounts for the smallest number of layers of D16.

After the low-temperature annealing an error in determining the average value of I_c on the length of tape was significantly decreased: in samples with the high-temperature heating for 2 h – from 37 to 17%. The annealing of the samples after heating at 1800°C was increased the critical current of more than 10 times.

It should be noted that if such a narrow (width of 2 mm) tape we used to facilitate the measurement of critical current at liquid helium temperature. The narrower the tape, the less a current is required to destroy its superconducting state, the less heat up the wires that lead the electrical current to the sample, while it is in a cold area, the less the loss of liquid helium due to evaporation. For quite obvious reasons the wider tape will have a more stable current along its length.

9.1. Conclusions to part

One. High values of the critical current of tapes of Nb₃Al, equaled to 50-60 A/mm in a magnetic field of 5.5 T were produced. This corresponds to the structural current density $(1.0-1.2) \cdot 10^5$ A/cm². The critical temperature and the upper critical magnetic field at 4.2 K are, respectively, ~18.5 K and 30-32 T. If we consider that the cost of industrial alloy D16 is much less than the price of tin, such a set of superconducting parameters of Nb₃Al-tape makes it quite competitive with Nb₃Sn. In addition there are reserves for further the enhancing of the critical current of Nb₃Al: (1) an optimization of modes of heat treatment and the design of composite Nb/Al-tape, (2) the doping of as aluminum [5, 17] so and niobium.

Two. It turned out that the previously selected modes of the high-temperature heating of tape by passing an electric current [5] are coincided with the regimes of continuous heating of the tape means "to pass". In addition, during the continuous heating is not needed to carry out preheating of the tape at 1000°C, which prevents the tape from the so-called "swelling" of its surface. Apparently, the binding of duralumin in the intermetallic compound NbAl₃, containing the largest amount of aluminum, there was still at the stage of temperature increase as an approaching to the work zone of the heating element.

Three. The available results have allowed to verify that the developed technology of the high-temperature heating for some improvement of individual units of the installation can be used to get really long (~100 m or more) segments of the composite tape based on the compound Nb₃Al.

Implementation of the relatively high ($\sim 1800^{\circ}\text{C}$) temperatures in a small volume was not as difficult as it seemed to us at first. And short times are allowed to lead the heating process with high productivity (100-150 meters of tape per hour).

10. THE DISLOCATION STRUCTURE OF THE DIFFUSION LAYER Nb_3Al , THE RESULTING SHORT-TERM HEATING OF A MULTILAYER COMPOSITE Nb/Al -TAPE

Earlier electron microscopic studies of three-layer composite tapes of $\text{Nb}/\text{AlGeCu}/\text{Nb}$ [19] after a brief heating to $1650\text{--}1800^{\circ}\text{C}$ and subsequent annealing at 800°C were founded in a layer of $\text{Nb}_3(\text{AlGe})$ precipitates of the second phase, both inside and along the grain boundaries of compound. This explains the observed increase of the critical current I_c after low-temperature annealing more than an order of magnitude. In the tapes without annealing, precipitates were not observed.

It explored the multi-layer tape of niobium and duralumin with thickness about 50 microns and a width of 5 mm after heating at 1850°C for from 1.2 to 6.4 s. The structure was studied using an electron microscope JEM-150 by method "to the light." Foils for the study was prepared at room temperature in an electrolyte of $\text{H}_2\text{SO}_4:\text{HNO}_3:\text{HF} = 3:2:5$ with a cathode made of stainless steel. The difficulty of preparation of the foils was "hit" on the layers of Nb_3Al , concluded between the Nb -layers (Fig. 16).

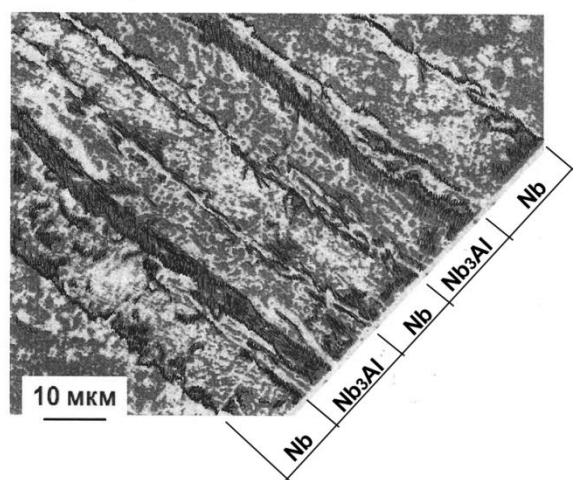


Fig. 16. Microstructure of the cross-sectional of multi-layered strips of Nb_3Al after the high-temperature heating about 1850°C for 1.3 s

After the heating for 1.3 s newly formed superconducting layer was consisted of equiaxed grains of compound Nb_3Al (the period of the crystal lattice of $5.185 \text{ \AA}$) with an average size of $\sim 0.2 \text{ mm}$ (Fig. 17a). With increasing of the heating time up to 4.6 s a polygonization structure with grain boundaries of the sufficiently regular walls and grids of dislocations was formed (Fig. 17b). Block size – of 0.2-0.3 microns. An even greater increase in heating time (6.4 s), accompanied by an increase in the thickness of the intermetallic compound layer with a grain size $>2 \text{ }\mu\text{m}$, was resulted in the formation of three-dimensional grid Franck (Fig. 17c). The nature of the image contrast of dislocations was testified about the environment of their atmospheres of impurities.

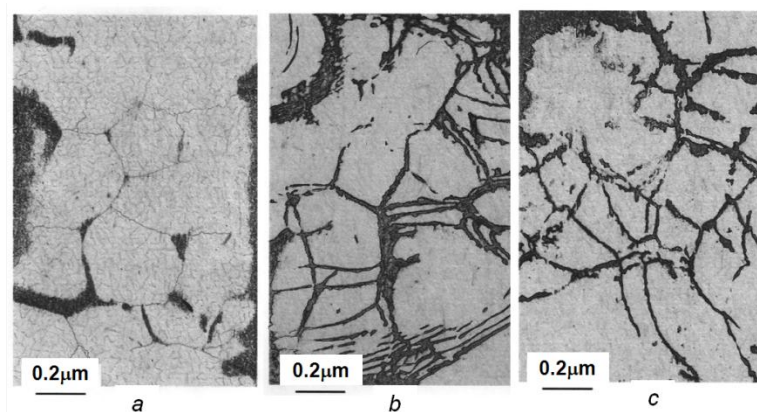


Fig. 17. The microstructure of the β -phase layer in a multi-layered tape Nb/Al after the heating about 1850°C for 1.3 (a), 4.6 (b) and 6.4 s (c)

The highest critical current was observed upon heating for 1.3 s (Fig. 18). Grain growth and formation of dislocation structure were lowered of I_c . This confirmed the concept of the effectiveness of the grain boundaries for pinning of superconducting vortices in superconductors with A-15 structure.

11. CONCLUSIONS

The origins of the present work falls at the end of the 70-s-early 80-s years of last century. The aim was to create a semi-industrial technology of production of the superconducting material on base of the Nb₃Al compound. This material is a justifiable alternative to the well-known compound Nb₃Sn. With equal to Nb₃Sn the superconducting transition temperature, it has a much more critical magnetic field of 32 T, and therefore seems more promising for use in magnetic fields exceeding 10-15 T.

The samples of described here multilayer Nb₃Al tapes were tested in magnetic fields up to 22 T. The values of the critical current density, calculated on the entire cross section of the conductor, at 4.2 K in magnetic fields up to 14 (Fig. 19) and 18-22 T were equated $(7-8) \cdot 10^4$ [4] and $(3.4-4.5) \cdot 10^4$ A/cm² [20] respectively (magnetic fields up to 22 T were created by a combination superconducting solenoid of the Institute of Atomic Energy of name I. V. Kurchatov).

On one of the companies semi-developed technology of production of the long segments of the multilayer Nb/D16 tapes was almost developed. It was included hydro-extrusion of composite rod at the press of horizontal type, then rolling in the profile rolls of 8-cage mill of cold rolling and a fine rolling with four-roll mill, equipped by coilers with precision tension. One of the segments of this tape is shown in Fig. 20.

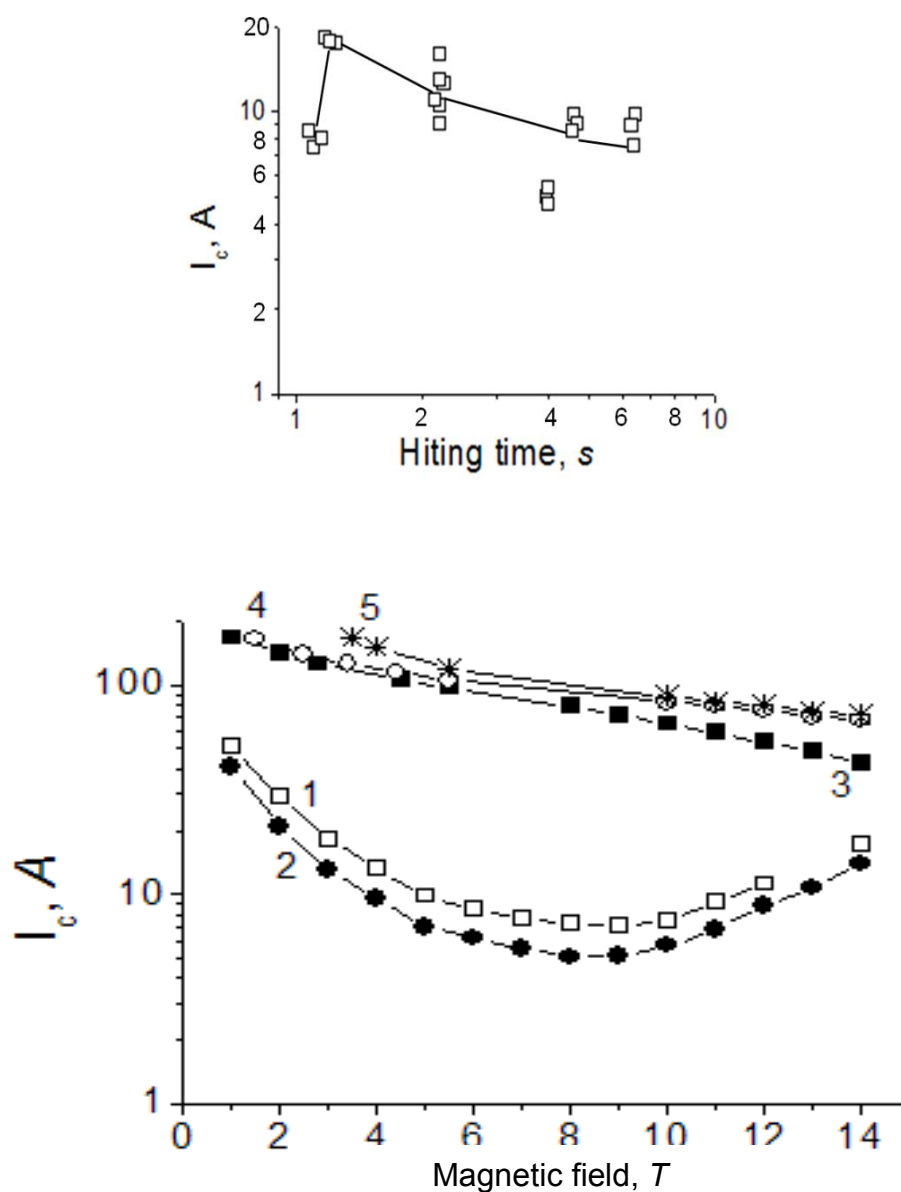


Fig. 18. Critical current I_c of Nb_3Al multi-tape depending on the time of high-temperature heating

Fig. 19. Critical current I_c of multi-layered Nb_3Al tape with width of 2 mm depending on an intensity of the external magnetic field. Heat treatment, C: 1 and 2 – 1000/1 min + 1850/2 s; 3, 4 and 5 – the same + 900/0.5 h, + 800/3 h and + 900/1 hour respectively. Measurements were conducted at the Laboratory of strong magnetic fields and low temperatures (Wroclaw, Poland)



Fig. 20. Segment of multilayer Nb/D16 tape

It seems that even after the discovery of high temperature superconductivity old, so-called now, the low-temperature superconductors should not remain on the side of technological progress. They are used now and in the future to be determined a safe and resistant area of application.

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