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The microstructure of Nb_3Sn superconductors differing in the number of copper inserts at various stages of heat treatment

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Abstract. Superconducting materials with a high critical current density in magnetic fields up to 16 T at a temperature of 4.2 K are required to create magnetic systems of high-energy physics devices. We have studied the change of the microstructure of model samples of various designs with an internal tin source, differing in the number of copper separators (three, six and without them) at different stages of heat treatment. The formation of the following intermediate phases was studied: Cu_3Sn, Cu_6Sn_5 and triple Cu-Sn-Nb phase after annealing at 370°C/ 100 h, by the use of scanning and transmission electron microscopy. The formation of the Nb_6Sn_5 phase on Nb filaments located close to the tin source and copper separators was studied after heat treatment at a temperature of 665°C from 10 minutes to 25 hours. The results of the analysis led us to understand the kinetics of the Nb_3Sn and Nb_6Sn_5 formation in internal tin superconductors with an increased current-carrying capacity and a low level of hysteresis losses.
Keywords: superconductors, Nb_3Sn, internal tin, heat treatment

1 Introduction.

At present the works are carried out actively to develop a design and technology of Nb_3Sn superconductors fabrication for the construction of high-field FCC accelerator circle of 100 km length. This project requires more than 8,000 tons of superconductors with increased electrophysical properties [1], [2].

One of the most important requirements for such superconductors, in addition to the high critical current density Jc, is the low level of hysteresis losses in coils under a pulsed magnetic field. The generated magnetic moment should be low and well controlled. There are two target parameters: the magnetization $\mu_0\Delta M$ and the effective diameter D_{eff}, connected by the formula 1.

$$D_{eff} = \frac{3\pi\Delta M}{4\gamma J_c} \quad (1)$$

γ— volume fraction of superconducting phase, Jc— critical current density (A/mm^2)

It is necessary to limit the regions with undesirable transverse flow of currents to reduce losses in superconductors. This task is achieved in two ways: by reducing the diameter of composite subelements, or by using separator inserts (tantalum, copper, etc.) inside each subelement [3], [4].

A number of works [5], [6] show a linear relationship between the actual size of individual superconducting filament regions (subelements) and the effective

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diameter of the entire conductor. In addition, a significant influence of tantalum and copper separators on the decrease in the effective diameter has been revealed.

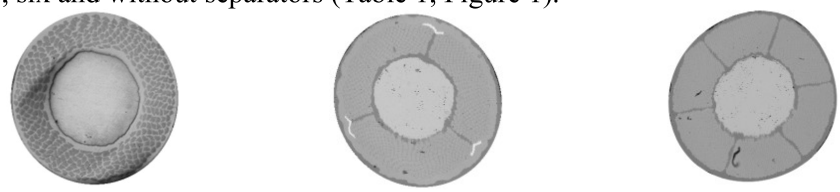
The process of formation of a superconducting compound in subelements without separators was described in [7], [8]. The introduction of copper or tantalum separators into the construction of a subelement affects the formation of high-tin bronze and a superconducting compound. However, there is not enough data in the literature on the kinetics of phase formation in subelements with copper separators during diffusion heat treatment to understand the process in details.

The purpose of this work was to study the microstructure and composition of niobium filaments at various stages of the diffusion heat treatment to determine the main physical features of a superconducting phase formation in subelements with copper separators.

2 Experimental samples and research methods

A number of Nb_3Sn superconductor designs with internal tin have been developed at the Bochvar Institute, differing by the number of copper separators. In finished composite superconductors in diameter of 0,7-1 mm the subelements and correspondingly their structural components have micron sizes. Diffusion processes in them are very fast within a fraction of a minute and it is difficult to fix their features.

Therefore, for a more detailed study of the phase formation processes in composite subelements of different designs, the model samples in a diameter of 1 mm were specially selected, in which the structural elements were an order of magnitude larger. Their designs differed in the number of radial copper separators - three, six and without separators (Table 1, Figure 1).



a b c

Figure 1. Cross-section of sub-elements in diameter of 1 mm with various number of radial copper inserts: a – Sample №1 (without copper inserts); b - sample №2 (three copper inserts); c - sample №3 (six copper inserts)

There were produced three types of 37-subelement conductors using these subelements. The critical current density (4.2 K, 12 T) ranged from 2400 A/mm^2 for design with six inserts to 2800 A/mm^2 for design without inserts.

The samples for the intermediate phases structure and composition study have been taken at the following stages of heat treatment: before heat treatment; 370˚C/100h; 370˚C/100h + 665˚C/10min; 370˚C/100h + 665˚C/20 min; 370˚C/100h + 665˚C/30min; 370˚C/100h + 665˚C/60min; 370˚C/100h + 665˚C/25h.

Table 1. Dimensional characteristics of the structural components of the investigated samples of Nb_3Sn IT-superconductor subelements.

№ ID	*Ø strand's, mm*	*Sn value, %*	*Cu value, %*	*Cu thickness of insert around Sn, µm*	*Cu thickness of radial inserts, µm*	*Ø of filament, µm*	*Number of filaments*
1	1	27	29	53	-	24	388
2	1	21	21,6	23	18	15	957
3	1	18,6	23,3	16	10	13	1632

The microstructure and composition of the samples after all stages of diffusion annealing were examined with the Helios Nanolab 660 scanning electron microscope and the Titan 80-300 scanning electron microscope.

3 Results and discussion

The images of the cross section of samples of three designs after different stages of heat treatment are presented below (Figure 2).

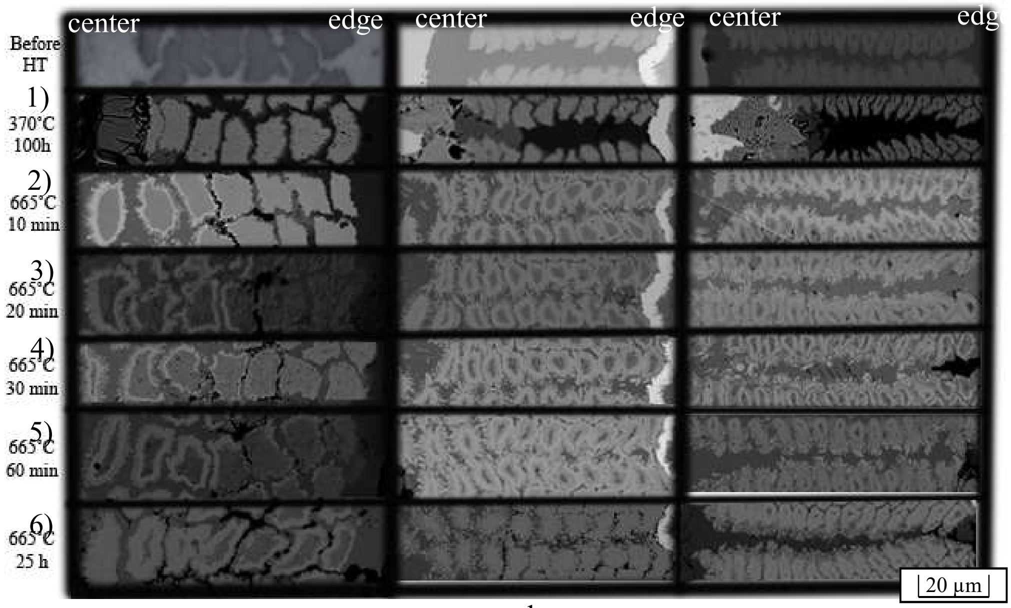
At the first stage of heat treatment on samples 2 and 3 have been revealed the areas of the ε-phase (Cu_3Sn) in the copper interlayer around source of Sn and η-phase (Cu_6Sn_5) in the zone closer to centre. If in inter-fiber space tin extends deep into the distance of 10-20 microns from the first row of filaments, then in the area of copper separators, the penetration depth of tin is about 50 μm. On the sample without copper separators these phases of intermetallic compounds are formed as two layers: more branched Cu_6Sn_5 (4-10 μm) and more uniform Cu_3Sn around the first row of filaments (~7.1 μm thickness). At the boundary of the first row of niobium filaments the layer of triple phase (10 at.% Cu, 18-22 at.% Nb, 68-72 at.% Sn) was found on all three types of samples which composition is rich by tin (Fig. 3).

At the second stage the formation of Nb_6Sn_5 phase in the form of elongated grains on filaments adjacent to the tin source and copper separators was observed on all three samples. Their length was 1-3 μm and the thickness was 0.2-0.8 μm. Presumably this phase is formed from the triple and Cu_6Sn_5 phases produced at 370°C. A thin layer of Nb_3Sn is formed only in filament layers at a distance of more than 30 μm from 1 row of filaments. The average grain size of this phase on all three samples was 60 nm. Presumably the source for the formation of this Nb_3Sn phase is predominantly the two phases of Cu_3Sn and the α phase (less than 9.1 at.% Sn). In addition the displacement of the first rows of niobium filaments was observed due to the fact that in the first stage a low-melting phase Cu_6Sn_5 (with a melting point of 415°C) is formed around them which turns into a liquid when the temperature rises the niobium filaments of the first row shift to the center of the subelement.

At the third stage on all samples the Nb_3Sn phase region grows. The largest one in the first rows of filaments near the source of tin and near copper separates.

From the fourth to the sixth stage a gradual increase in the Nb_3Sn layer thickness (of 2-2.5 μm) and, simultaneously, the growth of the average grain size (from 70 to 100-130 nm) have been observed on all samples. At the same time the unstable phase of Nb_6Sn_5, the formation of which was found at 665˚C / 10 minutes, first grows both in length (up to 2-5 μm) and in width (up to 0.2-2 μm). However, after 60 minutes of exposure, this phase is transformed with almost complete conversion into Nb3Sn with large round grains 0.4-2 μm in size.

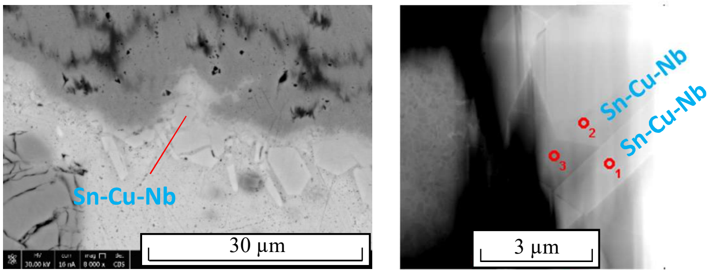
Analysis of the microstructure of the samples after 10, 20, 30, 60 minutes of exposure at 665 °C, revealed a noticeable delay in the phase formation on the peripheral row of filaments in comparison with the first rows of filaments located close to the source of tin. It was noted that the most intensive growth of the layer already occurs in the first 10 minutes (25-30% of the worked filament). About 50-75% of the filament cross-section has been worked into Nb3Sn after 60 minutes of exposure. At the same time, it was noted that the difference between the thickness of the Nb_3Sn layers on the filaments of the first and peripheral rows decreases with increasing the exposure time. So for samples with copper separators after 20 minutes of exposure at 665°C the difference in the thickness of the layer for the first and the last rows of filaments was 50-90%, that after 60 minutes it was 25-50%. But, at the same time, for a sample without copper separators this difference was 80% even after 60 minutes of exposure.



a b c

Figure 2. SEM fragments of the cross section of Nb_3Sn IT-samples (a – 1 sample, b – 2 sample, c – 3 sample) in diameter of 1 mm at different stages of heat treatment.

There are not noticeable differences in the phase formation kinetic in samples 2 and 3 differing in the number of copper separators (three or six). But it is possible to reveal some general trends such as more intensive process of Nb_6Sn_5 phase formation in area of copper separators, the collapse of copper separators due to filament displacements in this zone, and the slowdown of Nb_3Sn phase formation on peripheral filaments.



Element	Atomic %
Point 1	
Cu(K)	10
Nb(K)	22
Sn(K)	68
Point 2	
Cu(K)	10
Nb(K)	18
Sn(K)	72
Point 3	
Cu(K)	0
Nb(K)	0
Sn(K)	100

a b c

Figure 3. The fragment of the cross-section (a), investigated by the composition of the region (b), the composition of the triple phase (c) on the sample 3 after 370 ° C / 100 h.

4 Conclusions

The phase formation processes on the model samples of composite subelements for Nb3Sn superconductors obtained by the internal tin source method with copper separators and without them at intermediate stages of heat treatment have been studied.

It has been revealed that the Nb_6Sn_5 phase forms with radially elongated grains in the filament region (Nb/Cu) which occurs close to the tin source and copper separators. This is due to the formation of tin-rich phases Nausite and Cu_6Sn_5 in the adjacent zone. The formation of Nb_6Sn_5 at the Nb / Sn interface was observed on all types of samples after 10 minutes of exposure at 665°C. It was shown that after heat treatment at 665°C 30 minutes the Nb_6Sn_5 phase decomposition happens and most of it was converted to a coarse-grain Nb_3Sn phase.

It has been established, that there is a shift in the niobium filaments location in this region, which is due to the presence of low-melting point, high-tin phases, when it is converted into a liquid at this stage. In the layers located further from the tin source there is not disturbance in the arrangement of the filaments, because only α phase and Cu3Sn are formed and so the solid-phase interaction occurs between Nb and Sn.

It has been established that in the peripheral layers of the filament zone only the Nb3Sn phase is formed. In samples with copper separators the filament layers close to them are worked out in Nb_3Sn faster than in samples without them, because they are rapidly saturated with tin and serve as additional channels for diffusion.

Thus, when constructing Nb_3Sn superconductors obtained by the IT- method, it has to be taken into account that the use of copper separators on the one hand can intensify the diffusion of tin and help to accelerate the formation of the superconducting phase. But on the other hand their presence can lead to an increase in the fraction of the coarse Nb_3Sn phase, to disturbance of order in the arrangement of niobium filaments and finally lead to a decrease in the critical current density.

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