The advanced setup for synthesis of composite long-length superconducting tape of Nb$_3$Sn

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The description of design of advanced technological setup for the synthesis of composite long-length superconducting tape on the basis of Nb$_3$Sn is presented. The design of setup has allowed us to produce the Nb$_3$Sn tape with the record magnitudes of density of critical current $J_c$ in the magnetic fields $H$ of above 10 T ($H > 10$ T). The high current density conductivity property is reached as a result of both the optimization of technological process of the Nb$_3$Sn tape synthesis and the increase of thickness of superconductor layer, which is characterized by the homogeneous physical properties.

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Introduction

The superconducting composites with the crystal structure $A15$ are characterized by the high magnitudes of critical parameters of superconductor (the critical temperature $T_c$, second critical magnetic field $H_{c2}$, density of critical current $J_c$) [1], allowing their application to develop the laboratorial superconducting solenoids and complex superconducting magnetic systems for the use in the high magnetic fields for various purposes [2].

In the class of the superconductors $A15$, the composite superconductor of Nb$_3$Sn is well researched. Presently, a big number of the liquid-phase and solid-phase technologies for the synthesis of the superconducting tapes and multifilament wires on the base of superconducting composite of Nb$_3$Sn are developed (see, for example, [1, 3, 4]). At NSC KIPT, the composite long-length superconducting tape of Nb$_3$Sn with the record magnitude of critical current density $J_c \sim 3 \cdot 10^2 J_0$, where $J_0 = 1.8 \cdot 10^8 A\cdot cm^{-2}$ is the limiting magnitude of density of critical current, corresponding to the reach of critical velocity by the electrons in superconducting condensate [5], was synthesized. In agreement with the research results in [5], the creation of the current caring microstructure in the superconductor of Nb$_3$Sn is a spontaneous process, connected with the self organization of this structure.

The high magnitudes of critical current density $J_c$ can be reached in the composite long-length superconducting tape on the basis of Nb$_3$Sn, in which the density of critical current doesn’t depend on the thickness of the superconducting tape layer at the high magnitudes of current. The superconducting tape was fabricated with the use of specially designed and developed technological setup, which utilizes the liquid-phase method of phase formation of Nb$_3$Sn.

The layer of Nb$_3$Sn with the improved superconducting characteristics was placed on the thin ribbon of the pure Niobium or Niobium, which was alloyed with ~1.5% Zirconium, using the thermo-diffusion process. During this process, the original ribbon of Nb interacts with the melted Tin or Tin-Copper alloy, undergoing the thermal treatment and oxidation process. The optimal temperature for the superconducting layer synthesis was found, using the diagram of phase state of Niobium-Tin system and going from the precisely measured dependence of the critical current density on the temperature.

The development of setup resulted in the solution of the problem of optimization of synthesis process toward the fabrication of better quality superconducting tape with the thermo-diffusion layer of Nb$_3$Sn with the high critical current density $J_c$ characteristics in high magnetic fields $H$ of 10 T and above. The main challenge was to minimize the origination of accompanying non-superconducting intermetallic phases Nb$_6$Sn$_5$ and NbSn$_2$ in the Niobium-Tin system by finding an appropriate operational temperature range for the plant. These phases of Nb$_6$Sn$_5$ and NbSn$_2$ synthesize at the temperatures of below 906°C and 860°C correspondingly. It was understood that, at long enough exposures of plated tape to these temperatures, the described inter-metals of Nb$_6$Sn$_5$ and NbSn$_2$ synthesize in the form of thin interlayers between the Nb$_3$Sn crystal grains, resulting in the limitation of critical current density. The distribution of temperatures inside the oven was appropriately adjusted with the aim to avoid the Nb$_6$Sn$_5$ and NbSn$_2$ non-superconducting phases origination in the composite long-length superconducting tape on the basis of Nb$_3$Sn.

In this research paper, the detailed description of design of the improved advanced experimental setup for the synthesis of the composite long-length superconducting tape of Nb$_3$Sn is presented.
Principal scheme of advanced technological set up for Nb$_3$Sn tape synthesis

The thin ribbon of pure Niobium with the thickness of 12...14 µm or the thin ribbon of Niobium, which was alloyed with the Zirconium (1,6...1,7 %) with the thickness of 17...20 µm represented the main source material for the synthesis of composite long-length superconducting tape on the basis of Nb$_3$Sn. The fabrication of the thin ribbon of pure Niobium, included a number of functional operations at special setups, devised for the cutting, ultrasonic purification, and ablution. After the preparation stages, the deposition of Tin layer or Tin-Copper alloy layer was conducted at the technological setup, which is shown in Figs. 1, 2.

Fig. 1. Scheme of setup to synthesis composite long-length superconducting tape on basis of Nb$_3$Sn (side appearance): 1 – the cover of top chamber; 2 – the direction guider for descending and lifting of quartz shoot with roll; 3 – the quartz shoot with quartz horn; 4 – the top part of operating chamber; 5 – loading bobbin with pure Niobium ribbon; 6 – the receiving bobbin for Tin plated Nb$_3$Sn tape; 7 – the system of rolls for tape rolling; 8 – the direction guiding rolls; 9 – the system of screens for optimization of thermal losses; 10 – the bottom part of operating chamber; 11 – the heat shielding (top and side) screens; 12 – the Molybdenum heater in oven; 13 – the melted Tin; 14 – the quartz skirt, in which melted Tin is situated; 15 – the oven to melt Tin; 16 – the mechanic vacuum pump BH-2MI; 17 – the pipe to refrigerate setup; 18 – the vacuum pipeline for diffusion pump MM-40 (19) pumping by mechanic vacuum pump BH-2MI (16); 19 – the diffusion oil pump MM-40 with vacuum trap; 20 – the high vacuum absorption pump; 21 – the vacuum tap; 22 – the manometer bulbs JTM-2; 23 – the manometer bulbs JTM-2.

Fig. 1, the setup, which includes the vacuum chamber with the related equipment and pumping system, is shown. It automatically operates and uses of ribbons with the width of 10 mm. The pumping system consists of the mechanical vacuum pump BH-2MI with the limiting vacuum of 3·10$^{-3}$ Torr (16), providing the preliminary pumping of gas up to the first vacuum, and also the pumping by the diffusion oil pump MM-40 (19) and by the absorption pump (20), refrigerated in the liquid Hydrogen, which allows to pump the gas admixtures, including the Hydrogen, exhaling during the process of Tin plating of Niobium or Niobium-Zirconium ribbons.

Fig. 2. Scheme of setup (atop appearance): 1 – the speed reducer; 2 – the electrical motor AOJ/012/4; 3 – the speed reducer’s axle; 4 – the circuit, activating the drawing mechanism; 5 – the bobbin to accumulate plated tape; 6 – the sighting window; 7 – the directing roll; 8 – the side flange; 9 – the shell of upper part of operating chamber; 10 – the handle of shielding screen of sighting window; 11 – the directing roll; 12 – the bolt to close the opening cover in the upper part of operating chamber; 13 – the flange to connect with vacuum system; 14 – the loading bobbin with Niobium ribbon; 15 – the directing roll; 16 – the mechanism of shoot down activation.

The working chamber consists of the upper (4) and lower (10) parts. It is completed in the form of a cylinder made of the stainless steel. On the left side, the flange of working chamber serves to make a connection to the pumping system. The vacuum tap (21), which regulates the inflow of air to the chamber, is situated on the same flange.

The oven (15) to melt the Tin or Tin-Copper alloy is placed in the center of the lower chamber (10). This oven (15) has a system of screens (11) in its upper part, decreasing the thermal radiation by the oven. The heater (12) in the oven is made of Molybdenum wire with the diameter 1,5 mm, which is reeled on the pipe with the cone shape with the decreasing diameter toward the oven’s bottom.
The temperature measurements of the oven and melted Tin are performed by the two Chrome thermo-pairs. The first thermo-pair is situated between the quartz skirt (14) and the oven (15) in a special quartz tube with the curved form, which prevents the deposition of platting allow on the thermo-pair. The second thermo-pair is situated in the quartz tube of shoot (3), allowing the temperature measurement in close proximity to the place of contact between the pure Niobium ribbon and the quartz roll in the melted alloy.

The chamber (10), in which the oven is situated, can be moved down with the help of mechanical jack, and then be moved aside to load the quartz skirt with the alloy, during the retracting operation and other technological operations.

The top part of chamber (4) is covered by the folding cover with sighting window, which is shielded by a screen. The cover can be closed with the help of three adding bolts. There are the loading bobbin with cleaned ribbon (5), receiving bobbin with plated tape (6), drawing rolls (7) and directing rolls (8) in this part of operational chamber.

The top part of operational chamber (see Fig. 2) (top appearance) has the three sighting windows, covered by the screens, making it possible to conduct an observation during the process of tape movement.

The plated ribbon is putted to the melted Tin or Tin-Copper alloy by the shoot (3) (see Fig. 1) with the quartz roll at the end. The upper end of shoot is fixed on the mechanism, sliding toward the direction pointer, situated in the top part of the chamber. The movement of quartz shoot is provided with the help of mechanism with the jagged transmission, having the circular scale, allowing to precisely douse the quartz tube with plated tape down to the needed deepness (see Fig. 2, (16)).

The device to roll and drag the ribbon represents the system described in Fig. 2. At the left part of chamber, there is a loading bobbin with the original pure Niobium ribbon, which is placed on the pillow-block with the aim to decrease the friction. The degree of bobbin’s clench is regulated by the spring, fixed by the two nuts. There is directing roll near to the loading bobbin. This roll rotates on the pillow-block. The ribbon, passing through the roll, upper protective screens, screens of oven, quartz roll submerged in the plating melting pot, comes to the roll (7) of intake part of drawing mechanism. After this, the plated ribbon passes through the system, consisting of four rolls, rotating with the constant velocity and providing the engagement within it. These rolls are activated with the help of speed reducer (3) with the shaft introduced to the chamber through the vacuum packing. The speed-reducer is rotated by the electromotor AO3-012/4. There is a switch on the speed-reducer, allowing to draw the tape with the velocity 5,2; 10 and 15 m per hour.

The ribbon, covered by the alloy, passes the system of four rolls, coming to the receiving bobbin, which is rotated by the chain transmission, which connects this bobbin with the system of drawing rolls. The rotation of receiving bobbin is realized, using the «slipping regime» of operation with the purpose to decrease the stretch of loaded tape. The vacuumed electrodes are used for the power supply to the heater in the oven and the outputs of thermo-pairs from the bottom and top parts of operation chamber. The vacuum packing of all the inputs is made with the help of rubber strips, and the electric isolation is achieved due to the application of the Teflon sockets. The operational chamber has the double walls with the refrigerated water pumping to protect the vacuum packing from a possible overheating.

The power feed with the voltage of 220 V comes to the stabilizer from the distribution dash, and then to the transformer AOCK-10/09, which is connected with the electrical oven in the setup.

The setup is characterized by the following parameters. The volumes of the upper and lower chambers are 154 and 125 dm³ correspondingly. The full preparation time from the switching on the vacuum pumps to the beginning of actual operation is 1.5 hours. The power of oven is 3.5 kW at the temperature 1000 °C. The receiving bobbin can accommodate up to 400 m of the Tin plated composite long-length superconducting tape on the basis of Nb₃Sn with the width of 10 mm and thickness of 40 µm. The removable bobbins for the tapes with the width of 40 and 80 mm are present in the setup.

The subsequent thermal treatment of the Tin plated composite long-length superconducting tape on the basis of Nb₂Sn was done at a separate setup with the special configuration of temperature field, aiming to minimize the time duration of the Nb₂Sn tape exposure to the high temperatures at which the inter-metals of Nb₅Sn₃ and NbSn₁₂, which have a negative influence on the transport current properties, can be originated. These and some other improvements allowed to obtain the homogenous thick layer of Nb₃Sn on the surface of Nb ribbon, resulting in the synthesis of composite long-length superconducting tape on the basis of Nb₃Sn with record magnitudes of the critical current density.

In Fig. 3, the measured dependences of the magnitude of critical current in the Nb₃Sn tape at T=4.2 K in the magnetic field 6 Tesla on the temperature of the Tin melt, measured after its plating, and also after the thermal treatment at T = 905 °C during 20 hours, are shown.

![Fig. 3. Dependences of magnitudes of critical current in superconducting Nb₃Sn tape (related to 1 cm of its width) on temperature of Tin melt: 1 – initial Tin plated Nb ribbon with the layer of Nb₂Sn; 2 – thermally treated Tin plated Nb ribbon with the layer of Nb₃Sn.](image)
It can be seen that that the optimal temperature regime of plating process, which gives an opportunity to synthesize the $\text{Nb}_3\text{Sn}$ composite long-length superconducting tape with best physical characteristics, is in the range of temperatures between $900\ldots920$ °C.

After the completion of optimization of technological process, including the Zirconium oxidation, it was found that the use of the Niobium-Zirconium ribbon, plated by the Tin-Copper alloy, results in a significant increase of the magnitude of critical current density up to the record value $J_c=10^6\ \text{A}\cdot\text{cm}^{-2}$ in the $\text{Nb}_3\text{Sn}$ composite long-length superconducting tape in the magnetic field $H$ of 10 Tesla.

Conclusion

The design of advanced technological setup for the $\text{Nb}_3\text{Sn}$ tape synthesis allowed us to fabricate the composite long-length superconducting tape on the basis of $\text{Nb}_3\text{Sn}$ with the record magnitudes of the critical current density $J_c = 10^6\ \text{A}\cdot\text{cm}^{-2}$ in the magnetic fields above 10 Tesla at the Helium temperatures. The high transport current property in $\text{Nb}_3\text{Sn}$ tape was reached due to an optimization of technological process, including the deposition of Tin or Tin-Copper melting on the surface of original $\text{Nb}_3\text{Sn}$ tape and the application of thermodiffusion synthesis of the $\text{Nb}_3\text{Sn}$ superconductor layers with the thickness up to 6 $\mu\text{m}$ at every side of tape, characterized by the high homogeneous physical properties. The produced $\text{Nb}_3\text{Sn}$ tape was used for the design of series of superconducting solenoids with the high magnetic fields ($H > 10$ Tesla).

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