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Production of NbTi/Nb/Cu multilayer sheet composites

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Abstract

The purpose of the joint research between CERN, Wigner Research Centre and the University of Miskolc is the production of mulytilayer (Cu/Nb/NbTi/Nb) sheet compsite material for a superconducting shield application. To produce a multilayer sheet composite OFHC copper (Cu), pure niobium (Nb) and Nb53wt%-Ti niobium-titanium (NbTi) raw materials were used. The Cu/Nb/NbTi/Nb material combination has been used for several decades in the production of superconducting cables. In the composite, copper (Cu) is the thermal conductor, while the niobium-titanium alloy (NbTi) is the superconducting material. Between these two layers, pure niobium (Nb) hinders cross-diffusion.Hot and cold rolling were applied for production. In the mid-nineties, the Japanese Nippon Steel Co. had already produced Cu/Nb/NbTi/Nb multilayer sheet composite but stopped production. In this study the reproduction of the original Japanese technology of making multilayer sheet composite with hot and cold rolling was described.

1 Introduction

CERN is the world's largest particle physics research laboratory, where the most significant scientific and technological challenge after 2025 will be the construction of Future Circular Collider (FCC). In this process, after the collision, the intensity of the beam decreases, and because the beam can become destructive, there are the so-called beam dumpsites in some regions of the ring where the transfer of the beam is performed by special magnets [1]. The SuShi septum magnet is a superconductor magnet where the superconducting shield produces a zero magnetic area in a powerful exterior magnetic field [2]. The key element of the magnet is an approximately ~ 1 mm thick multilayer sheet composite, in which one sequence is made of Cu/Nb/NbTi/Nb layers (Figure 1.).

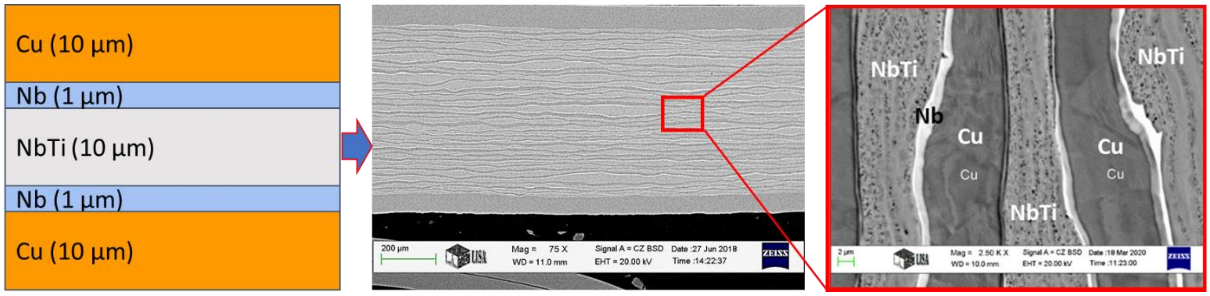


Figure 1. The theoretical representation of the sequence and the layers based on images in 75x and 2500x magnification [3].

The Cu/Nb/NbTi/Nb material combination has been used for several decades in the production of superconducting cables [4]. In the composite, copper (Cu) is the thermal conductor, while the niobium-titanium alloy (NbTi) is the superconducting material. Between these two layers, pure niobium (Nb) hinders cross-diffusion [5]. The NbTi layer is not a homogeneous solid solution. However, in an ideal case, it contains α-Ti precipitations that prevent the magnetic vortexes' movements and create stable superconducting conditions. In the mid-nineties, the Japanese Nippon Steel Co. had already produced Cu/Nb/NbTi/Nb multilayer sheet composite but stopped production. [6].

Our research aims to find a better or cheaper production technology regarding raw materials and production. In our present publication, The reproduction of the original Japanese technology of making multilayer sheet composite with hot and cold rolling was described.

The general purpose of laminar composite rolling is to create a multilayer laminar structure which provides complex properties. The functional sandwich structures have different properties by layers [7]. In the case of laminar composite, there must be a metallic bond between the layers provided by roll bonding. The quality of the bonds is strongly influenced by the rolling parameters and the time and temperature of the post-heat treatment [8]-[9].

In the hot forming process, the bond between the layers is formed very fast, and this bond is stronger due to diffusion - for example solid solution can be formed - which can strengthen the bond [10].

Itoh et al. [11] made Cu/Nb/NbTi/Nb a multilayer superconductor composite which was first hot rolled, then cold rolled (50-76% reduction) at a large scale, and put to intermediate heat treatment. The ultimate thickness of the composite sheet was 0.75 mm. The average thickness of the NbTi and Cu layers in the multilayer composite sheet was 10 µm. Hidetaka Oguma et al. [12] constructed a multilayer sheet composite using thermo-mechanic treatment. After the layering process, the sequences were placed in a copper case, compressed at room temperature, and then hot rolled at 800°C after which the box was cold rolled. In this research area, data about multilayer superconducting sheet composite are limited, therefore, our research was conducted with preexperiments.

2 Results

2.1 Determination of recovery and recrystallization temperature

A large-scale deformation was planned for production, so the knowledge of the recrystallization behaviour of the raw materials of laminar composite is paramount. The recrystallization behaviour of 75% cold rolled Nb and 90% cold rolled Nb53wt%-Ti with DSC (differential scanning calorimetry) method was characterized. The Nb sample was heated to 1100°C, while Nb53wt%-Ti sample was heated to 1000°C using 5°C/min heating speed.

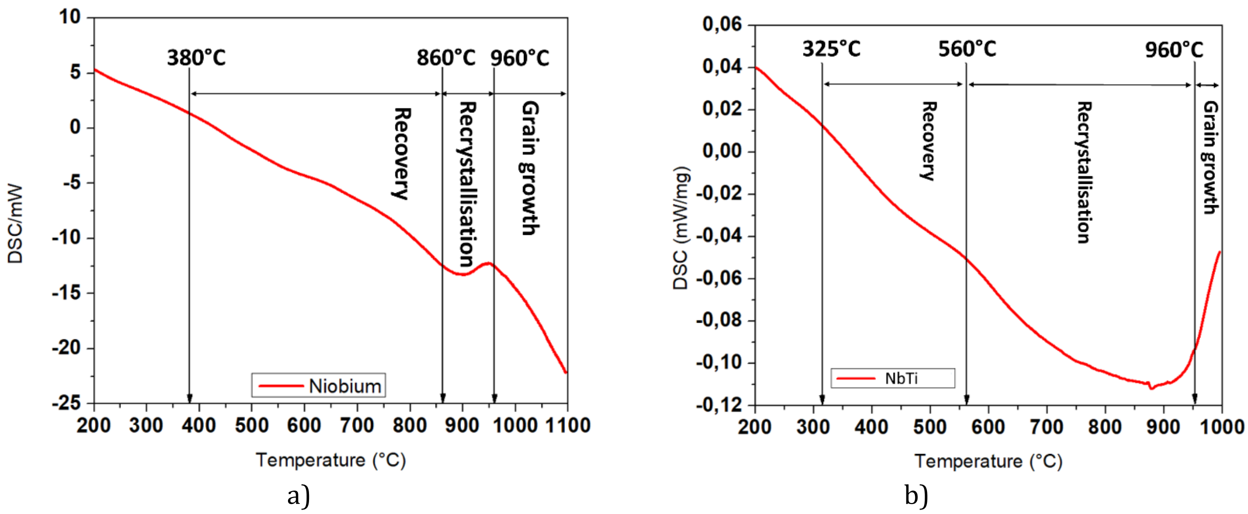


Figure 2. Result of DSC emperiment a) Nb sample and b) Nb53wt%-Ti sample

The DSC curves show (Figure 2.a) that Nb recovery happens between 380 – 860°C, and recrystallization takes place between 860 – 960°C and grain growth starts at about 960°C. Furthermore, (Figure 2.b) Nb53wt%-Ti recovery happens between 350 – 560°C, recrystallization takes place between 560 – 960°C, and grain growth starts at about 960°C. These results are well correlated with the literature data. [13]. Based on this data, the temperatures chosen for the hot-forming process were 960°C and 900°C.

2.2 Production of multilayer sheet composite

To produce a multilayer sheet composite OFHC copper (Cu), pure niobium (Nb) and Nb53wt%-Ti niobium-titanium (NbTi) raw materials were used. The hydrochloric acid etched sheets were placed in OFHC copper box under argon protective gas in Cu/Nb/NbTi/Nb sequence. The cover of these boxes with the sequences was pushed into the box to achieve the necessary anti-oxidation. The boxes were heated in a resistance-heated furnace, and the rolling experiments were carried out with VON-ROLL rolling mill at the Institute of Physical Metallurgy, Metalforming and Nanotechnology in Miskolc. The box was hot rolled from 44 mm to about 15 mm thickness, then cold rolled to a given final thickness. Figure 3. shows the subsequent steps in a single shot. The number of sequences also varied during the tests. The parameters of the experiments are in Table 1.

After the rolling, the sheets were subjected to microscopic tests. A Zeiss Axio Imager M1m optical microscope and Zeiss EVO MA10 scanning electron microscope were used for imagining.

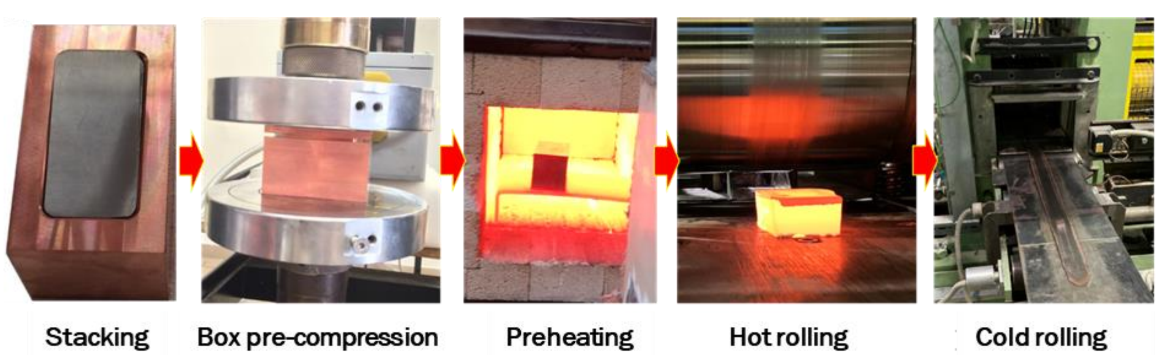


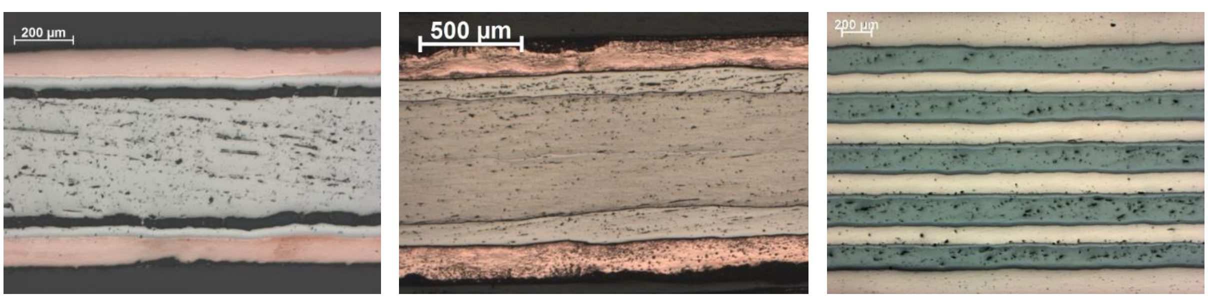
Figure 3. Technologycal phases of the production of multilayer sheet composite

Table 1. The paramters of the experiments

Experiment	*A*	*B*	*C*	*D*
Starting thickness of Nb (mm)	*2*	*0.96*	*0.45*	*0.3*
Starting thickness of NbTi (mm)	*20*	*4*	*2*	*0.96*
Starting thickness of OFHC Cu (mm)	*cassette*	*4*	*2*	*0.96*
Starting thickness of cassette (mm)	*44*	*44*	*44*	*44*
Finish thickness of cassette (mm)	*1*	*1*	*2.4*	*0.5*
Number of sequence (quantity)	*1*	*1*	*5*	*10*
Applide temperature (°C)	*960*	*960*	*900*	*960*
Duration of heating (minute)	*60*	*60*	*120*	*120*

The Figure 4.a shows the optical microscopic image of experiment "A", It is obvious that the Cu and Nb layers streched from the NbTi layer and bond between the layers was not formed on the Nb/NbTi interface. In order to improve the bonding condition, pre-rolled thinner plates were used in the next experiment , "B" The geometry of the cassette was not changed and copper was used to fill the remaining space. All other experimental parameters were unchanged. The optical microscopic image (Figure 4. b) shows that the bonding of layers was successful both on Nb/NbTi and Cu/Nb interfaces.

In the next steps (experiments "C" and "D"), the thickness of the pre-rolled plates was further reduced and the number of sequences was increased to five and ten, respectively. In the preheating parameters, we reduced the temperature in experiment "C" and increased the time in both cases.The optical microscopic image (Figure 4.c) reveals that bonding of layers was formed both on Nb/NbTi and Cu/Nb interfaces in the five sequences.

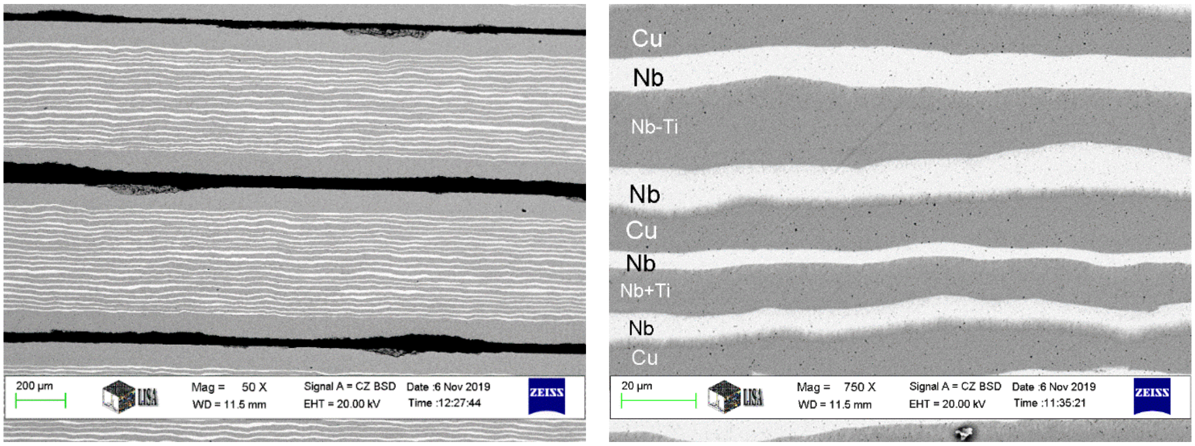


a) b) c)

Figure 4. a) The structure of one sequence composite b) The structure of three sequence composite c) The structure of five sequence composite

The structure of ten-sequences, 40-layer composite (experimet "D") is shown in Figure 5. The bond between the layers was perfect at the different parts of the ribbon. The deformation of the layers is uniform along the entire length and cross-section of the strip. No layer separation or tearing has occurred. The cross-sectional image confirms the formation of a perfect, free-of-impurities bond between the individual layers.

The sheet structure is clearly retained, with each layer of different material being nearly parallel along both the longitudinal and cross-section.



a) b)

Figure 5. a) The structure of ten sequence composite b) The cross-section of the ten sequence composite structure

3 Conclusion

In our research, Cu/Nb/NbTi/Nb multilayer sheet composites was produced using a total of 40 rolling experiments. It was found that the recovery of Nb takes place between 380-860 °C, its recrystallization happens between 850-960 °C, and the grain growth process starts above 960 °C. The recovery of Nb53wt%-Ti takes place between 350-560 °C, its recrystallization happens between 560-960 °C, and the grain growth process starts above 960 °C. In the production of multilayer sheet composites, it is recommended to use prerolled raw materials and large-scale reduction to obtain optimal bonding between Nb/NbTi and Nb/Cu interfaces. In experiment „D" a multilayer composite sheet of 0.5 mm was produced, in which the average thickness of the NbTi layers was 15 µm, and the average thickness of the Cu layers was 12 µm, while the average thickness of the Nb layer was 7 µm. Experiment "D" demonstrates the best correlation with the structure of the Japanese composite sheet. In fact, a more uniform layer thickness with the in-house produced plate was achieved.

4 Acknowledgments

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