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## STRUCTURAL INVESTIGATIONS AND DETERMINATION OF CURRENT AND VOLTAGE CHARACTERISTICS IN HTSC 2G SF12050 TAPES

### BADANIA STRUKTURALNE ORAZ WYZNACZANIE CHARAKTERYSTYK PRĄDOWO-NAPIĘCIOWYCH DLA TAŚM HTSC 2G SF12050

Investigations of a state-of-the art high-temperature ceramic superconducting tape of SF 12050 series are presented. The study focuses on current and voltage characteristics, examination of microstructure and analysis of chemical composition in superconducting tapes of SF series. In order to measure current parameters, a measuring system was designed to investigate sections of the tapes at the temperature of liquid nitrogen. The system is powered with direct current with parameters  $I = 0 \div 580\text{A}$  and  $U = 0 \div 8\text{V}$ . Measurements of critical current were taken by means of determination of the decline in voltage along the measurement section for the tape with accuracy of  $1\text{nV}$ . Analysis of chemical composition was carried out using scanning microscope which features chemical composition microanalyser EDX. The paper presents microscope images which are a result of examination of the structures by means of light microscope and scanning microscope. The method of preparation of superconducting tapes for soldering and the method of selection of solder were also presented.

**Keywords:** high-temperature superconductors, current and voltage characteristics, critical current, analysis of chemical composition, microstructure

W pracy zaprezentowano wyniki badań ceramicznej wysokotemperaturowej taśmy nadprzewodzącej najnowszej generacji z serii SF 12050. W artykule przedstawiono badania dotyczące wyznaczania charakterystyk prądowo-napięciowych, obserwacji mikrostruktury oraz analizy składu chemicznego taśm nadprzewodzących z serii SF. Do badań parametrów prądowych został zaprojektowany i zbudowany układ pomiarowy umożliwiający badanie odcinków taśm w temperaturze ciekłego azotu. Układ ten jest zasilany źródłem prądu stałego o parametrach  $I = 0 \div 580\text{A}$  oraz  $U = 0 \div 8\text{V}$ . Pomiar prądu krytycznego są wykonywane przez określenie spadku napięcia na odcinku pomiarowym taśmy z dokładnością do  $1\text{nV}$  na odcinku pomiarowym nadprzewodnika. Analizę składu chemicznego przeprowadzono przy zastosowaniu mikroskopu skaningowego z mikro-analizatorem składu chemicznego EDX. W pracy zamieszczone zostały obrazy mikroskopowe będące wynikiem obserwacji struktur za pomocą mikroskopu światłnego oraz skaningowego. Przedstawiona została również metoda preparatyki taśmy nadprzewodzącej do lutowania oraz sposób doboru lutowni.

## 1. Introduction

First precursor in the domain of superconductivity was Heinke Kamerlingh-Onnes, who was the first to obtain liquid helium. The discovery allowed for finding relationships between electrical current in metals and temperature. While testing the purest from the then available elements i.e. mercury, he found that, instead of a smooth decline in electrical resistance with reduction in temperature, at the temperature of ca.  $4\text{K}$ , resistance rapidly decreases to zero level and, below this temperature, mercury does not show electrical resistance [1].

The scientists worldwide have investigated a variety of different materials, searching for superconductors with even better functional parameters such as: critical current  $I_C$ , critical field  $H_C$  or critical temperature  $T_C$  [19]. Density of critical currents is dependent on: i.a. fabrication method, type of superconducting phase, presence or absence of external magnetic field and several other conditions. These densities of critical current reaches the values of a few  $\text{GA}/\text{m}^2$  [2÷9]. The highest critical temperature, equal to  $291\text{K}$  was reported for  $(\text{Ti}_5\text{Pb}_2)\text{Ba}_2\text{MgCu}_{10}\text{O}_{17+}$  superconductor, these results weren't confirmed by other scientists [10].

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Currently, the superconducting materials in the form of tapes are produced using several methods of production. Superconductors BSCCO typically are produced using powder metallurgy method by oxidation of the powder in the tube (OPIT), but it is also possible to obtain these materials by electrophoretic deposition on flat surfaces [11÷13]. The YBCO tapes are formed using deposition type method (i.e. three most often applied technologies on the global scale: RABiTS, IBAD and ISD), by deposition of superconducting layer on previously obtained substrates with buffer layer [14÷16]. Superconducting materials with comparable properties are also produced in the form of rings, tapes, tubes [1, 17÷18]. Nowadays all over the world, there are being conducted an advanced investigations about superconductivity.

## 2. Research methods and material

The investigations were carried out for high-temperature superconducting tape 2G HTSC (2nd Generation High Temperature Superconductors) of SF 12050 series (SF means stabilized free), whereas 12050 means width 12 mm and thickness of 0.5 mm). The types are delivered with manufacturer's specifications, which contain information about the following parameters:

- critical current  $I_C$  at the temperature of liquid nitrogen (77 K, in a uniform field), minimal value  $240 \text{ A} \pm 10\%$ ,
- tape width 12 mm,
- tape thickness 0,055 mm,
- thickness of silver coating  $2 \mu\text{m} \pm 0,5 \mu\text{m}$ ,
- base material: alloy of Ni and Mo and alloying elements, commercially sold as Hastelloy,
- base material magnetic properties: non-magnetic,
- resistivity of base material:  $125 [\mu\Omega\text{cm}]$ .

Commercial sections of the tape are up to 1000m. In order to carry out the investigations, a 10-metre long tape section was purchased

A measurement system was developed in order to determine the values of critical currents. Exceeding critical current causes the material to switch back from superconductivity state to normal state. In this article, the values of critical current were determined from the current-voltage characteristics by application four points method. It was measured voltage drop on the measuring section of the tested materials. Measuring section where voltage value drop was equal to 3 mm. As a criterion of critical current value was accepted appearance of voltage value equal to  $1 \mu\text{V/cm}$ .

The system allows for long-term loading the tape with current exceeding critical levels, which leads to destroying superconducting materials. Further investiga-

tions will attempt to characterize a mechanism of destroying HTSC. The current circuit was composed of three-phase current supply HP 6681A with maximal direct output current intensity of 580 A (Readback accuracy for investigated critical currents, using current supply HP 6681A in the range from 0 A to 580 A is 15 mA to 311 mA), and output voltage within the range of 0÷8 V. Voltage measurement circuit was equipped in a very sensitive digital nanovoltmeter Keithley 182 with measurement accuracy of 1nV. Current-based investigations of superconducting tapes were performed at the temperature of liquid nitrogen in a cryostat. In order to solder clamps and terminals, a mixture of fusible tin, Wood's alloy (with melting point of 66-72°C), rosin and flux was used. Soldering temperature doesn't exceed 100°C.

Figure 1 presents a stand for tape preparation as well as preparation of current and voltage circuit.

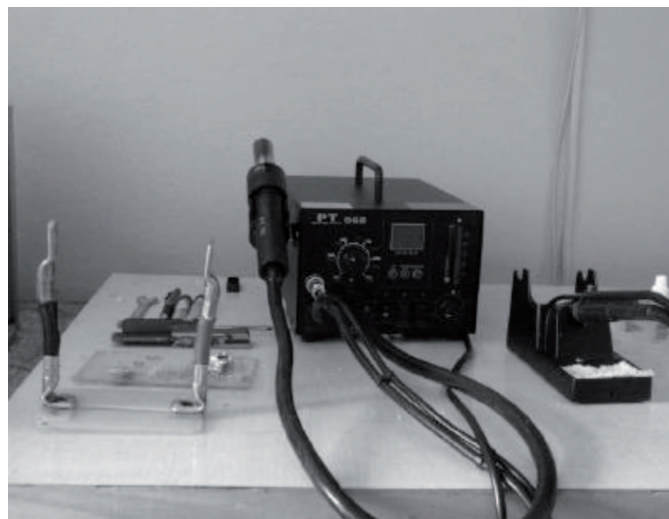


Fig. 1. Stand for preparation of current circuits

Except for determination of current and voltage characteristics, investigations of chemical composition in tape profile were also carried out using scanning electron microscope SEM JSM – 5400 manufactured by JEOL, with EDX micro-analyzer of chemical composition.

During the investigations for the listed bismuth tapes and tapes of SF series, a documentation of microstructures was prepared by means of light microscope with 100x and 500x magnifications.

## 3. Results and discussion

One of the most important parameters which characterize materials which demonstrate superconducting properties is critical current  $I_C$ . Exceeding critical value of current causes return of a superconductor to normal state although the temperature in the circuit is maintained

within the range of superconductivity. If the current exceeding the value of  $I_c$  passes through the circuit for a certain time, permanent damage to superconducting tape occurs. Figure 2 presents current-voltage characteristic for superconducting tape of second generation, SF 12050 series.

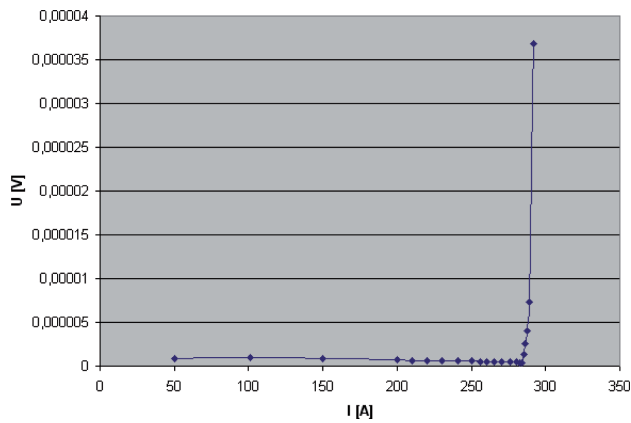


Fig. 2. Current-voltage characteristics for superconducting tape of SF 12050 series determined at the temperature of liquid nitrogen

In order to compare differences which occur in current-voltage characteristics and critical current  $I_c$ , current-voltage characteristics were determined for two additionally selected tapes which differed with their chemical composition and structure. Tapes SF 12050 and SF 4050 differed with width (SF 12050: 12 mm; SF 4050: 4 mm). Bismuth high-temperature superconducting tape was also subject to investigations: it was marked as Bi2223 (BSCCO;  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_9$ ) and differed with its structure and composition. Figures 3 and 4 present current and voltage characteristics for bismuth tape and the tape of SF 4050 series.

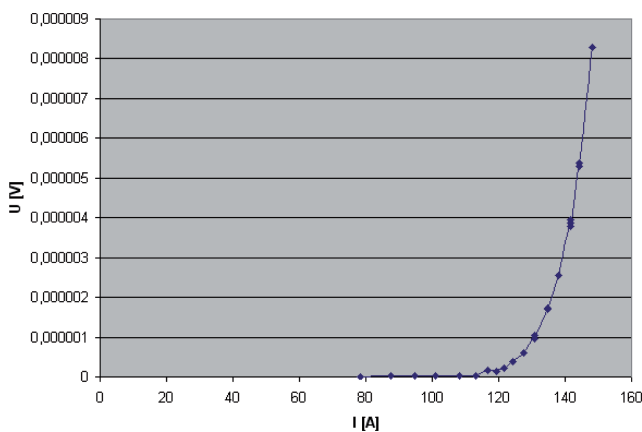


Fig. 3. Current and voltage characteristic for bismuth superconducting tape Bi2223 at the temperature of liquid nitrogen

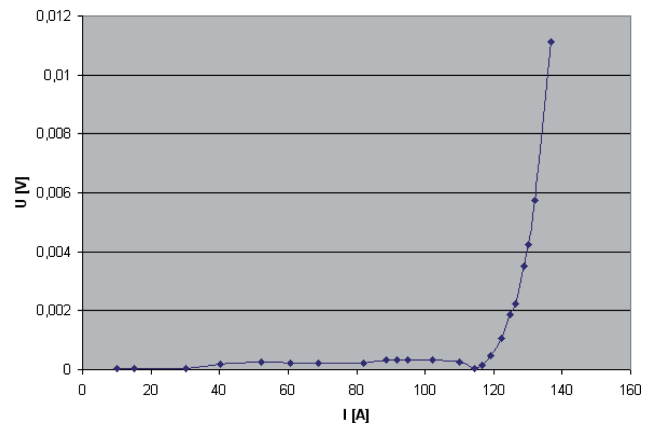


Fig. 4. Current and voltage characteristic for superconducting tape of SF 4050 series at the temperature of liquid nitrogen

As results from characteristics presented in Fig. 2 to 4, highest critical current of  $I_c=272$  A among the investigated tapes can be observed for the tape of SF 12050 series. Critical currents for bismuth tape and SF 4050 tape were similar and amounted to nearly 120 A.

Figure 5 and 6 present microstructures registered by means of AXIOVERT optical microscope.

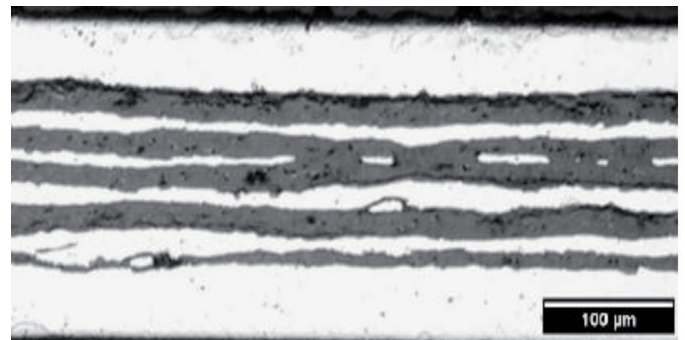


Fig. 5. High-temperature superconducting tape Bi2223, magnification 100x



Fig. 6. High-temperature superconducting tape of SF series, magnification 500x

Photographs of microstructures for superconducting bismuth tapes and tapes of SF series were taken for non-etched longitudinal metallographic specimens for magnifications of 100x and 500x.

Bismuth tapes have a laminated structure. The layers of the bismuth-based phase, which show superconducting properties are placed in metallic matrix.

Tapes from SF group are multilayer materials covered with protective coating, in this case silver, which is aimed, among other things, at protecting against the effect of the environment and other factors which could deteriorate current parameters or strength properties of superconducting tape.

In order to determine chemical composition of a superconducting tape of second generation, SF 12050, a section of the tape was randomly selected for the analyses of chemical composition (JEOL SEM JSM – 5400 scanning electron microscope which features EDX chemical composition microanalyser was employed). The examinations were aimed at determination of local differences in chemical composition in relation to average chemical composition claimed by the manufacturer. Chemical composition of SF 12050 superconducting tape expressed in weight percentage (%wt.) is presented in Table 1.

TABLE 1  
Chemical composition of HTS 2G SF 12050 tape

Chemical composition in %wt.											
Co	Cr	Cu	Fe	Mn	Mo	Ni	Si	Ag	W	V	*
2.3	15	0.5	5	1	15	52	0.1	6.3	4	0.3	2

\*REBCO; \*RE – rare earth elements: Eu, Dy, Gd, Y, Sm;  
BCO denotes barium, cuprum and oxygen.

Fig. 7 presents the places where chemical composition measurements were taken. It is represented by a continuous line on the photograph of longitudinal microsection in SF series.

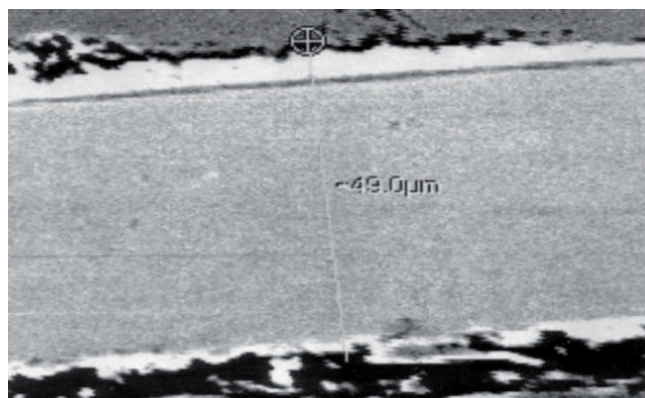


Fig. 7. Randomly selected location of measurements of chemical composition

Measurement of chemical composition were taken for the coating deposited on the material which shows superconducting properties and for the superconducting

material included in composition of the tape of SF series. The results of measurements are presented in Tab. 2÷3.

TABLE 2  
Chemical composition of the coating deposited on superconducting material of SF series in %wt.

Chemical composition in %wt.			
C	Al	Fe	Ag
3.28	0.58	3.83	92.32

From the table above, it can be concluded, that in protective coating, next to Ag elements were also detected the presence of Fe, Al and C, which enter into material during the preparation of cross-sections for investigated samples.

TABLE 3  
Chemical composition of the area of superconducting material for the tape of SF series in %wt.

Chemical composition in %wt.						
Cr	Mn	Fe	Co	Ni	Mo	W
14.79	0.47	4.98	1.24	56.80	16.91	4.80

Figure 8 presents a linear distribution of the concentration of elements along the line shown in Fig. 7.

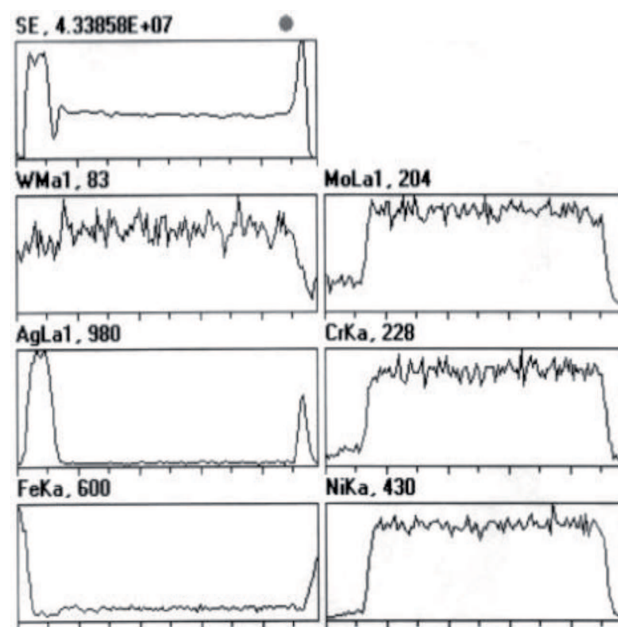


Fig. 8. Linear distribution of the concentration of elements for HTS 2G tape of SF series

As results from this figure, elements such as Ag and Fe are distributed in surface area, which constitutes a protective coating. In the deeper layers of the material are located W, Mo, Cr and Ni elements which are part of substrate.



#### 4. Conclusions

- Structural investigations of the selected superconducting tapes revealed that they show different structure. In HTS 1G tapes, layers of superconducting phase are separated with the materials of matrix (Bi2223). HTS 2G tapes are multilayers materials covered with protective coating. They are represented by the tapes of SF series.
- Chemical composition and structure of high-temperature superconducting tapes considerably affects critical current  $I_C$ . Measurement for critical current for the tape of SF12050 series amounted to 272 A, whereas this value for bismuth tape was ca. 120 A.
- Tape width considerably affects critical current values. The studied tapes SF12050 and SF 4050 show the same chemical composition but they differ with width. The difference in critical currents is considerable, but it is not directly proportional to the tape width. For SF 12050 tape,  $I_C$  amounts to 272 A, whereas in the case of the tape SF 4050, the level of  $I_C$  was 120 A.
- As results from the comparison of chemical composition given by the manufacturer with chemical composition determined by means of JOEL scanning electron microscope, segregation of elements occurs.
- Knowing current-voltage characteristics of high-temperature tapes will allow for application of suitable current load which does not exceed the value of critical current.

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