THE COMPREHENSIVE TECHNIQUE FOR QUANTITATIVE ANALYSIS OF THE STRUCTURE IN COMPOSITE SUPERCONDUCTORS BASED ON Nb₃Sn

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Abstract: The complex technique for study of the structure in Nb₃Sn-based composite superconductors is developed. Technique is based on scanning and transmission electron microscopy on different scale levels. The possibility of using the method of focused ion beam for TEM sample preparation is confirmed.

KEYWORDS: Nb₃Sn, FILAMENT, STRAND, SUPERCONDUCTOR, MICROSTRUCTURE, ITER

1. Introduction

The Nb₃Sn-based superconductors obtained by “bronze process” are complex composite materials, having tens of thousands superconducting filaments in the cross-section of a wire with a diameter of 1 mm [1-7]. Filaments are usually collected in bunches (strands) and distributed evenly in a bronze matrix, surrounded by a niobium diffusion barrier with tantalum inserts and a stabilizing copper shell (Fig. 1.). Each filament of Nb₃Sn superconducting compound (with a diameter of ~2-3 μm) has an ultra-fine grain structure with a grain size from ~50 to 500 nm. Material having such a complex structure demands a comprehensive approach for quantitative analysis of structure characteristics, including studies on different scale levels: macro, micro and nano.

Fig. 1. Macrostructure of superconductors

2. Methods and Results

Images of macrostructure by optical microscopy (magnification x100 - x1000) were obtained for a quantitative analysis of superconductors at the macro level. The following parameters have been measured on the obtained images: diameter of the wire, thickness of copper shell, thickness of diffusion barrier, diameter of strand.

The quantitative analysis of the structure on the micro level was performed using SEM in the electron backscatter mode (magnification x1000 - x5000) (Fig. 2.). For images obtained the following parameters have been measured: diameter of filaments, distance between filaments, a volume fraction of Nb in filaments. Such measurements have been performed including at layer-by-layer polishing with a step of 50 μm.

Fig. 2. Microstructure of superconductors (SEM)

The analysis of structure on the nano level included study of ultra-fine grain structure of superconducting filaments. The analysis of grain structure on the filament fracture surfaces of annealed superconductors was performed using high-resolution field emission scanning electron microscopy in secondary electron mode at magnification x25000. The obtained images of the fracture surfaces were quantitatively analyzed using a specialized software ImageExpertPro. The quantitative analysis included measurement of the diameter of equiaxed and columnar grains, measurement thickness of different morphology layers, measurement of volume fraction of residual niobium (Fig. 3.).

Fig. 3. Image of fracture: a - original of fracture surface; b - image after processing in AdobePhotoshopCS3 (TEM)

The possibility of using the focused ion beam method for wire samples preparation to study the structure of superconductors by transmission electron microscopy (TEM) also has been shown. Foils have been prepared on a scanning ion microscope with gallium liquid metal ion beam. Sample was cut to obtain a thin plate (lamella) (5-15) μm in size and (1-2) μm in thickness. After receiving the lamella it was further thinned and explored by the TEM method (Fig. 4.).
4. Conclusion

The comprehensive technique for quantitative analysis of the structure of composite superconductors based on Nb₃Sn is developed. The developed technique included study of structure by optical microscopy, scanning electron microscopy, transmission electron microscopy on different scale levels.

Comparative study of the structure of Nb₃Sn-based composite superconductors manufactured using different modes of deformation and heat treatment have been performed in the current work. Those superconductors were fabricated at JSC "Chpetsky Mechanical Plant" and verified at A.A. Bochvar VNIIHM.

4. References:


