

**INHOMOGENEITIES IN THE STRUCTURE OF HIGH  
TEMPERATURE SUPERCONDUCTING LAYER**

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**ABSTRACT**

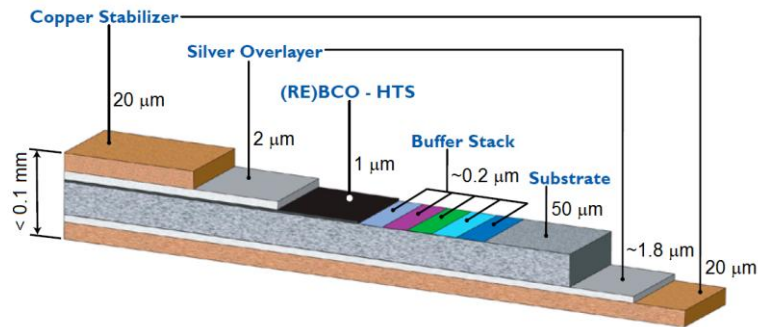
*Superconducting layer in coated conductor was investigated to assess the inhomogeneities present in its structure and to get better insight into the effect of inhomogeneities on degradation of electric properties. In the investigation scanning electron microscopy, energy-dispersive X-ray spectroscopy, electron backscattered diffraction, and laser scanning confocal microscopy were used. The results obtained showed good correlation between the density of inhomogeneities across the tape width and the degradation of current transport properties determined by measurements of the current density*

**KEY WORDS**

*high temperature superconductor, characterization of structure, inhomogeneities*

**INTRODUCTION**

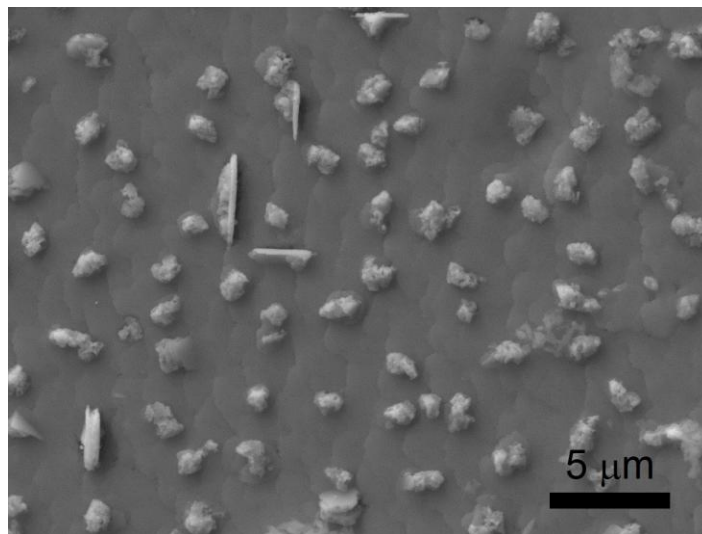
One of the most important issues related to the development of new energy sources is production of electromagnetic coils which will provide strong magnetic fields in a nuclear fusion reactor. The coils will be most probably made of high temperature superconducting tapes enabling the electricity conduction in a micrometer thick ceramic layer. High temperature superconducting coated conductor (*Fig. 1*) is a complex structure consisting of several layers. The most important (functional) layer is the mentioned high temperature superconductor (HTS). Investigated tapes consist of high temperature superconductor containing rare earth elements (RE) in the crystal lattice. This type of superconductor is referred to as (RE)BCO (where (RE)BCO means (RE)Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-y</sub>, RE = Y<sub>x</sub>Gd<sub>1-x</sub>) with orthorhombic crystal lattice.



**Fig. 1** Schematic illustration of coated conductor [1]

## SAMPLE PREPARATION

Prior to planar surface investigation of high temperature superconducting layer, it is necessary to remove the copper and silver overlayers. This was done by selective chemical etching using iodine. The etchant attacks only metal layers (copper and silver overlayer), while the high temperature superconducting layer remains untouched. There are several advantages of this approach. The selective chemical etching provides large uncovered area (the size of uncovered area is basically unlimited) and the localization of uncovered area is controllable. Furthermore, there is no mechanical damage of the uncovered area because the etching stops exactly at the top of the surface layer. By the use of selective chemical etching (*Fig. 2*), surface morphology and chemical composition of the HTS layer is preserved. In *Fig. 2* two kinds of shapes can be identified: needle-like and polygonal structures.



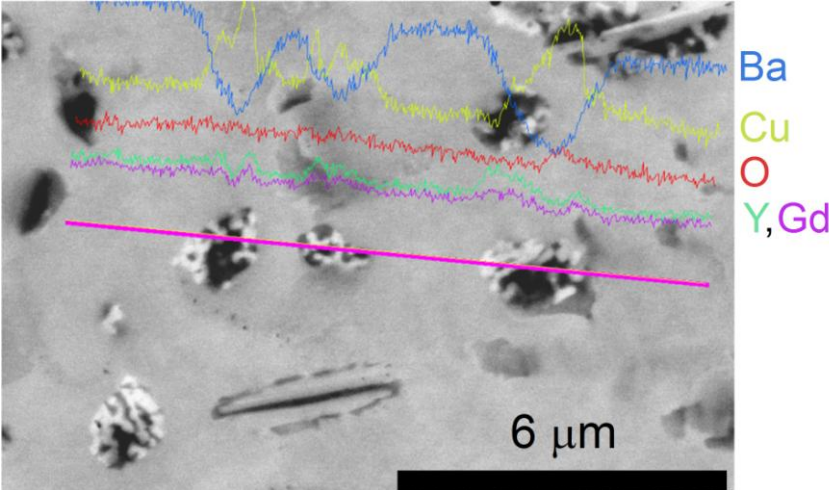
**Fig. 2** Uncovered high temperature superconducting layer prepared by selective chemical etching, secondary electron imaging (SEI) mode

## RESULTS AND DISCUSSION

### *SEM investigation of coated conductor*

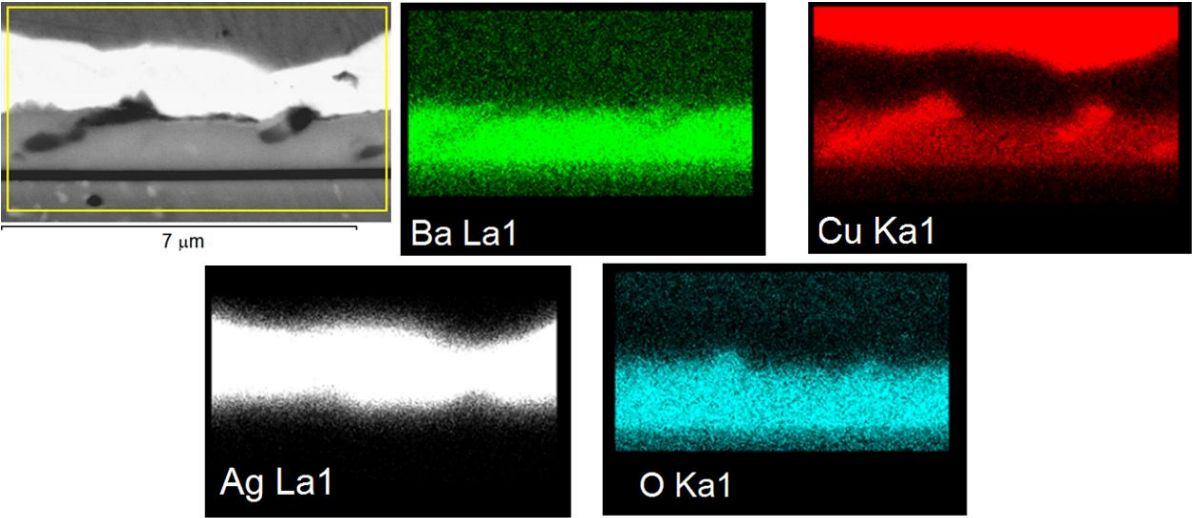
Investigation using scanning electron microscope (SEM) JEOL JSM7600F revealed inhomogeneities in the structure of HTS layer. As it was previously reported in [2], the presence of such inhomogeneities (also known as outgrowths) is typical for good crystalline quality of HTS layer. These particles in HTS layer have generally another orientation than

superconductor matrix and they are classified as a-, or b-axis oriented grains (a-, b-axis is parallel to surface normal) or secondary shaped particles other than (RE)BCO phase [3]. In addition to the size, number density and homogeneity of outgrowths are time and temperature dependent [3]. The energy dispersive X-ray spectroscopy (EDX) line scan (Fig. 3) guided through outgrowths shows the chemical composition of outgrowths is rather complex. RE elements show an increase in the intensity in white parts of outgrowths and the black parts contain predominantly Cu. Minima in the barium, yttrium and gadolinium concentration profiles indicates that the chemical composition of black parts of outgrowths might be as simple as  $Cu_xO$  ( $x = 1$  or  $2$ ). However, to confirm this hypothesis some additional high resolution measurements, for example by TEM, in cross-sectioned samples are necessary.



**Fig. 3** The EDX line scan of outgrowths, SEM backscattered electron imaging (BEI) mode

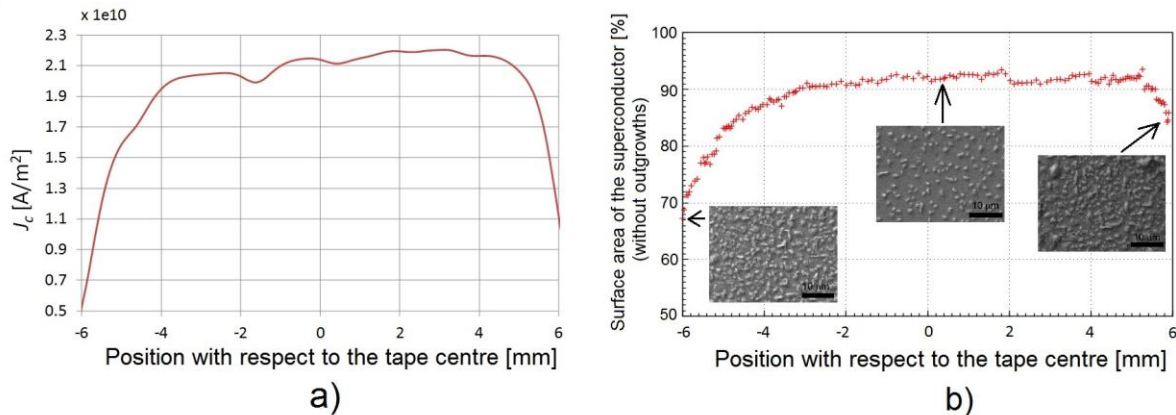
The concentration of elements (Fig. 4) is better observable in cross-section of coated conductor. EDX mapping showed depleted areas of barium in superconducting layer and rich areas of copper corresponding with position of outgrowths in the SEM micrograph.



**Fig. 4** The EDX maps of coated conductor in cross-section, SEM micrograph was obtained in BEI mode

### Correlation between results of electric and structural measurements

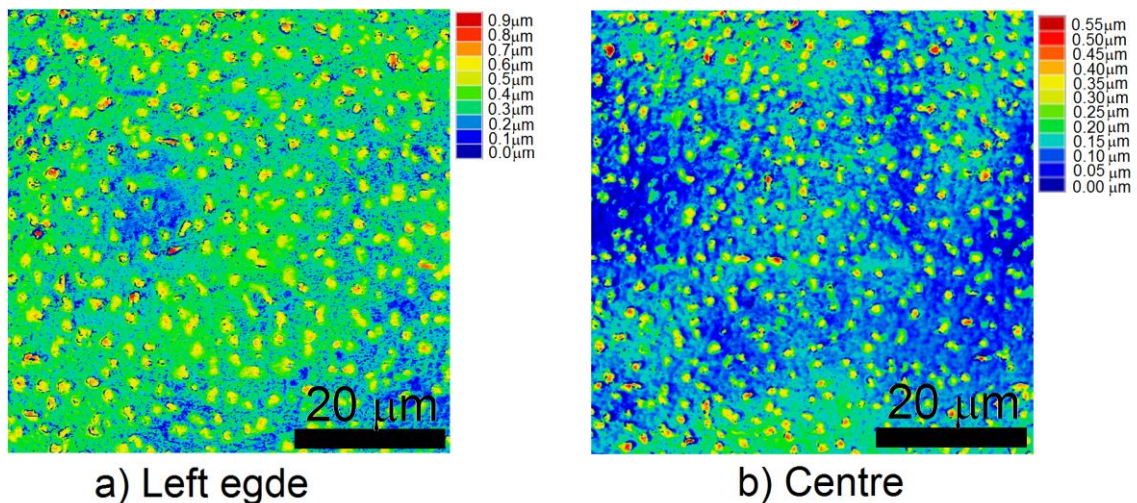
The SEM micrograph in Fig. 4 (top left) shows that outgrowths penetrate deeply in to the superconducting layer but rarely touch the buffer stack. These outgrowths have different chemical composition and crystal orientation as the bulk part of superconductor and may affect the electro-magnetic properties of coated conductor. We suspect that the presence of outgrowths have major influence on the degradation of current transport properties localized mostly at the tape edges (Fig. 5). The profile of critical current density showing a degradation of the current transport properties at the conductor edges (Fig. 5a) is in good agreement with the profile of surface areas (Fig. 5b).



**Fig. 5** a) Profile of a critical current density; b) profile of a surface area of the superconductor (without outgrowths), both as a function of a position across the tape width (0 = center of the tape) [4]

### Laser scanning confocal microscopy

From the point of view of experimental surface characterization, the surface roughness due to the presence of outgrowths is an undesirable feature. Fig. 6 depicts surface micrographs of HTS layer obtained by the use of laser scanning confocal microscope ZEISS LSM 700. These micrographs show that the outgrowths have height about 1 μm and are still too high and unsuitable to allow for crystal structure analysis by EBSD where very smooth surface is required.

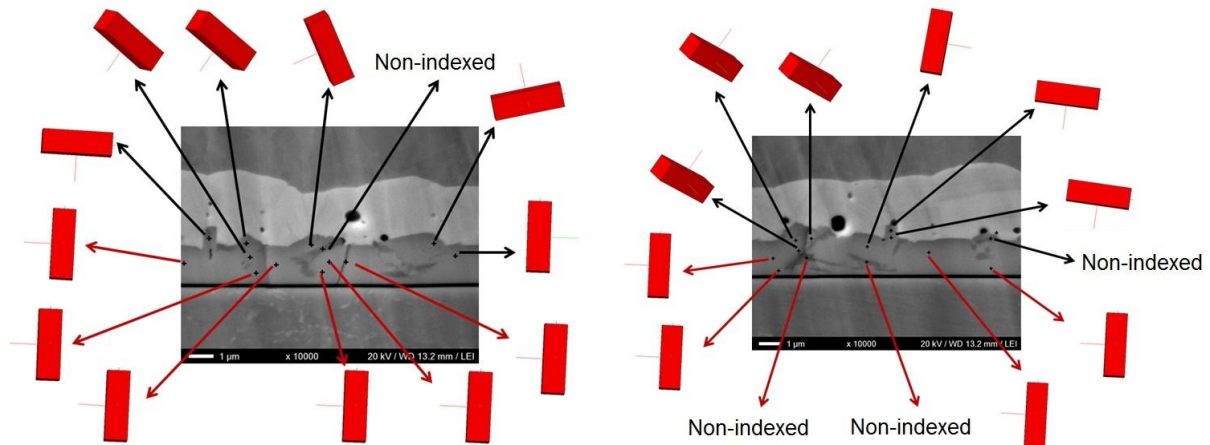


**Fig. 6** Surface height map obtained by means of laser scanning confocal microscope; height of outgrowths are indicated by various colours



## Electron backscattered diffraction of coated conductor

The analysis of cross-section in coated conductor using EBSD (accelerating voltage 20 kV, recording using 4x4 binning on a Nordlys II camera for 30 ms) shows clearly different crystallographic orientation in (RE)BCO outgrowth-free regions (the superconducting layer itself), in outgrowths and in neighbouring regions of outgrowths (*Fig. 7*). The EBSD technique was performed manually as point by point measurement, because automatic EBSD mapping was unable to distinguish between a-, b- and c-axis oriented superconductor.



**Fig. 7** Crystallographic orientation in (RE)BCO superconductor by EBSD

EBSD technique reveals that the (RE)BCO superconductor has almost identical (001) crystallographic orientation (red arrows in *Fig. 7*) as it was expected for good quality of HTS tape. Neighbouring regions of outgrowths indicates different orientation than (RE)BCO superconductor (black arrows in *Fig. 7*). Rotated lattice scheme (in red color) indicates different orientation than it is the expected (001) orientation. It was not possible to index outgrowths, dark areas in superconducting layer, black arrows marked as “Non-indexed” in *Fig. 7*, because of overlapping of two different electron backscattered patterns (EBSP) or obtaining of the EPSP from unknown phase, probably copper (I or II) oxide.

## CONCLUSION

The SEM measurements showed inhomogeneities in the structure of the HTS layer. The observed outgrowths were found to have rather complex chemical composition. They might be formed by simple copper oxide ( $\text{Cu}_x\text{O}$ ; black parts of outgrowths). Moreover, three regions were identified by EBSD: outgrowths-free region (superconductor), neighbouring region of outgrowths and outgrowths. This technique confirmed the expected orientation of superconductor – (001). Neighbouring region of outgrowths (white parts of outgrowths) shows different orientation than the surrounding superconductor. It was shown by EBSD that the latter region is the superconductor with different orientation. It was not possible to index some outgrowths, because they are formed with unknown phase, probably copper oxide.

## ACKNOWLEDGMENT

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