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STRUCTURAL CHARACTERIZATION OF (RE)BCO LAYER DEPOSITED BY PLD AND MOCVD TECHNIQUES

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ABSTRACT

(RE)BCO thin films prepared by PLD and MOCVD techniques were investigated to characterize structural defects - outgrowths in thin film. For this purpose SEM, EDX analysis and LSCM were used. Outgrowths are often penetrating into the thin films. Evident differences in chemical heterogeneity, outgrowth morphology and outgrowths density between PLD and MOCVD thin films were proven in this study.

KEY WORDS

(RE)BCO thin film, HTS tape, outgrowths, PLD, MOCVD

INTRODUCTION

The high-temperature superconductor (HTS) (RE)$Ba_2Cu_3O_{7-x}$ ((RE)BCO) exhibits high superconducting transition temperature (T_c), low surface resistance (R_s), high superconducting critical current density (J_c), and superior superconducting capability under magnetic field. As such, it is of high interest in both low and high current applications (1).

This paper is focused on structural characterization of $YBa_2Cu_3O_{7-x}$ (YBCO) and $GdBa_2Cu_3O_{7-x}$ (GdBCO) thin films of the second generation (2G) HTS tapes used for high current application, such as electrical power transmission (2), superconducting fault current limiters (3), superconducting magnetic energy storage (4), etc. The GdBCO and YBCO thin film was deposited by pulsed laser deposition (PLD) and metal-organic chemical vapour deposition (MOCVD), respectively. Both thin films were grown on Ni-based alloy covered with buffer stack, where the Ni alloy provides main mechanical strength of the tape. It is frequently observed that (RE)BCO superconducting thin films made by in-situ deposition methods have particles on the film surface sized from a few hundred to more than one thousand nm (5).

These particles (outgrowths) can act as defects in superconducting film, which degrade functional characteristics of HTS tapes. For production of tapes with high J_c, thin films of high crystalline quality, with as less as possible microscopic defects are important (1, 6).

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EXPERIMENTAL

The investigated tapes consist of several layers and arrangement of which is shown in Figure 1. Thickness of the (RE)BCO layer is 2.1 µm in case of PLD thin film and 1.6 µm in case of MOCVD thin film. The (RE)BCO layer was deposited on several layers of buffers. In order to observe (RE)BCO surface, top layers were removed by selective etching based on aqueous solution of KI (potassium iodide). The **cross-section samples were prepared by ion milling in cross-section polisher.**

For this study, a scanning electron microscopy (SEM) and a laser scanning confocal microscopy (LSCM) were used to do microstructure and morphology characterization. EDX measurements were performed to show chemical heterogeneity in thin films.

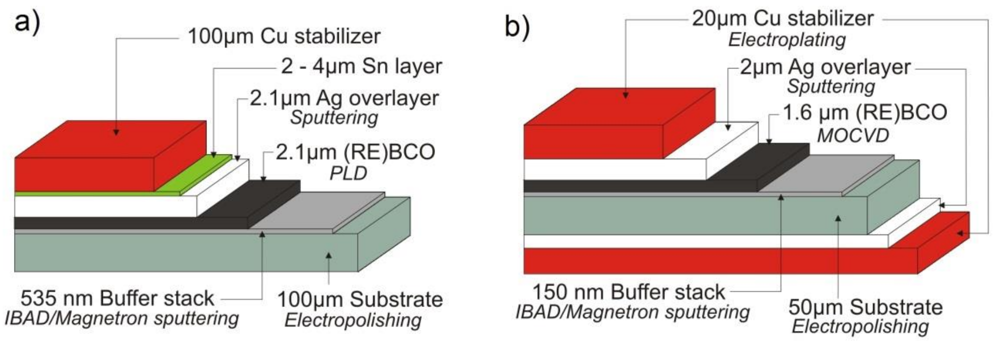

Fig. 1 *Arrangement of layers in investigated HTS tapes prepared by a) PLD and b) MOCVD techniques*

RESULTS AND DISCUSSION

Outgrowths characterization

Since sizes of outgrowths in (RE)BCO layer range from few hundred nm to few µm, electron microscopy can be advantageously used to characterize them. SEM images of superconducting surface and cross-section were taken to see the surface and under surface morphology.

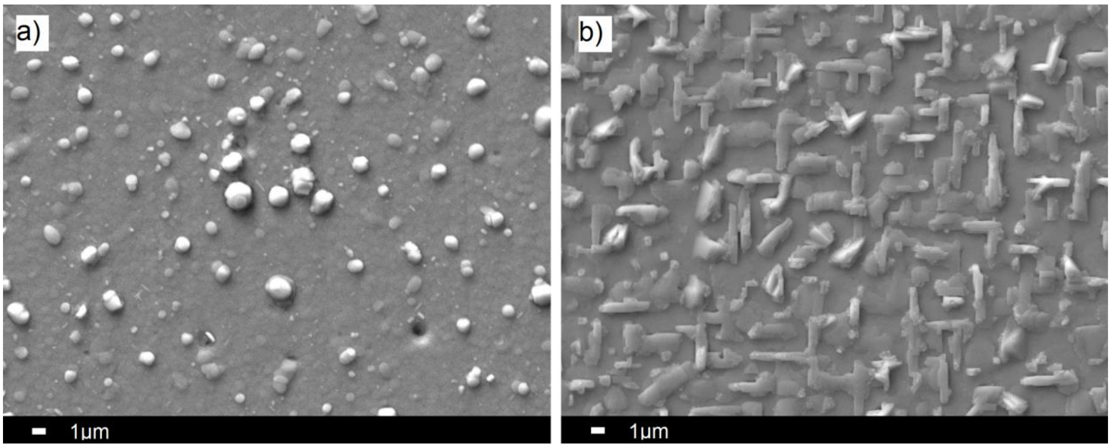

Fig. 2 *SEM images of (RE)BCO surface: thin film prepared a) by PLD and b) by MOCVD techniques*

Evident difference in the morphology of outgrowths formed in films prepared by different deposition techniques is shown in Figure 2. Three types of outgrowths can be recognized: rounded, angular and needle like. Thin film deposited by PLD (Figure 2a) contains a lot of rounded outgrowths, which can also be dissected and a small amount of tiny needle-like particles. Figure 2b shows surface of thin film deposited by MOCVD. Almost the half of this surface is covered by needle-like and angular outgrowths. They are often touching each other. The cross-sections of investigated tapes are shown in Figure 3. It is possible to recognize particular layers: Ni alloy, buffers, (RE)BCO layer, silver layer and copper layer (in case of MOCVD thin film). While the silver layer is porous with non-uniform thickness, buffer stack is compact and homogeneous. This applies to both types of tapes. The outgrowths are outgrowing up to 1 μm above the (RE)BCO surface in PLD as well as in MOCVD thin film. In terms of outgrowth penetration into superconducting layer, the outgrowths in the MOCVD thin film grow more deeply into (RE)BCO layer. If compared with the outgrowths in PLD films they are better observable due to dark appearance. It can be assumed, that in MOCVD thin film the outgrowths are covering larger area in (RE)BCO surface, as well as in cross-section. These outgrowths are more seriously affecting the effective cross-section (i.e. cross-section which is involved in leading of electric current).

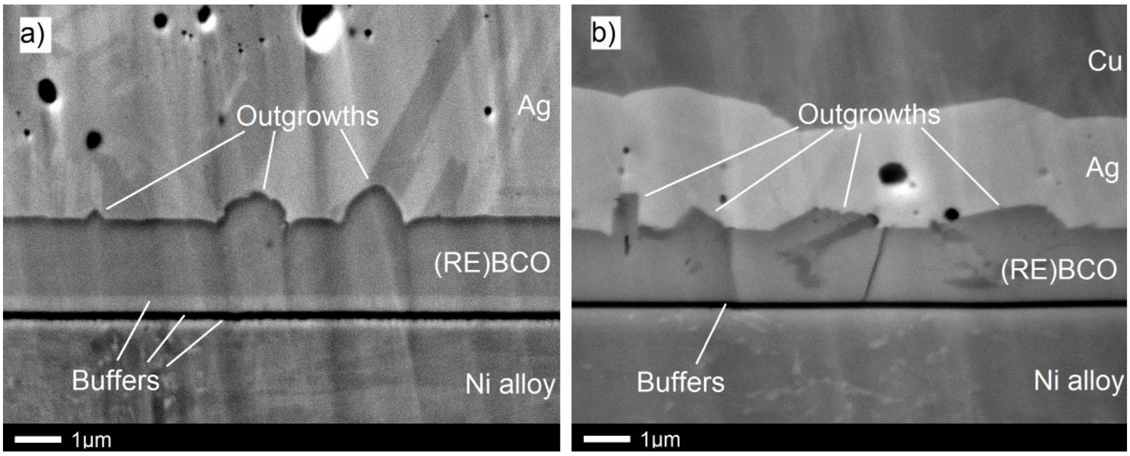

Fig. 3 *SEM images of (RE)BCO cross-section with outgrowths in superconducting films deposited by a) PLD and b) MOCVD techniques*

Topography of (RE)BCO surface

The information about surface topography was obtained by laser scanning confocal microscopy. Scans of areas in the middle of the tape width and near to the tape edges were performed in order to see onset of the outgrowths from sides and to compare the surface roughness in the centre and at the edges of the tape.

Considerable difference in surface roughness (Sa) measured in the middle of tape width (PLD: Sa = 0.053 μm, height of outgrowths up to 1.4 μm; MOCVD: Sa = 0.210 μm, height of outgrowths up to 2.5 μm) was observed between PLD and MOCVD thin films (Figure 4a, b). The MOCVD thin film was rougher (Sa = 0.242 μm) also near to the edges of tape in comparison with the PLD thin film (Sa = 0.104 μm) as indicated Figures 4c and 4d, respectively. The comparison of roughness values obtained from areas middle and edges of the same tape shows that the PLD thin film was more protruding on the edges than in the middle. The MOCVD thin film possessed smaller differences of surface roughness in the compared areas. However, overall roughness values were much higher for the MOCVD film than for the PLD film.

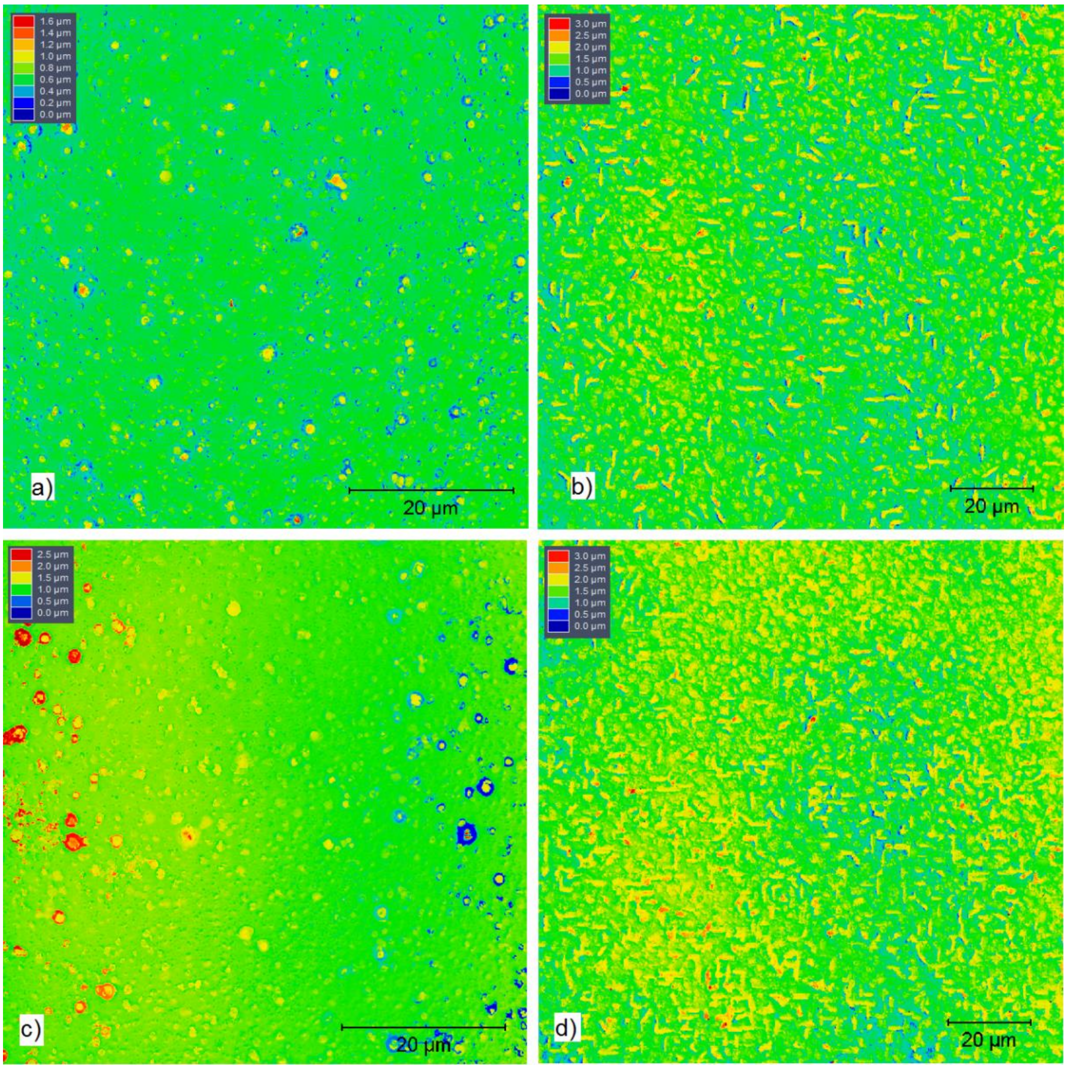

Fig. 4 *Surface roughness measured by LSCM in the middle of the tape a) for PLD thin film: Sa = 0.053 µm, b) for MOCVD thin film: Sa = 0.210 µm and near to the edges of tape c) for PLD thin film: Sa = 0.104 µm, d) for MOCVD thin film: Sa = 0.242 µm*

EDX MEASUREMENTS

EDX mapping of the surface was performed to proof chemical homogeneity of both thin films. The distribution of crucial elements in (RE)BCO thin PLD and MOCVD thin films is shown in Figures 5 and 6, respectively. The PLD thin film contains prevalently Gadolinium prior to Yttrium and the MOCVD thin film contains mainly Yttrium with a small amount of Gadolinium. These elements are well substitutable and have not any impact on functional properties of thin films. Electron image in Figure 5 illustrates the PLD thin film surface with frequently observed defect in the middle of the image. This defect was taken purposely to see distribution of elements in this defect. We could see relatively good homogeneity of all elements except for oxygen. In the map there were only small spots of Barium, Yttrium and Gadolinium related to several outgrowth positions.

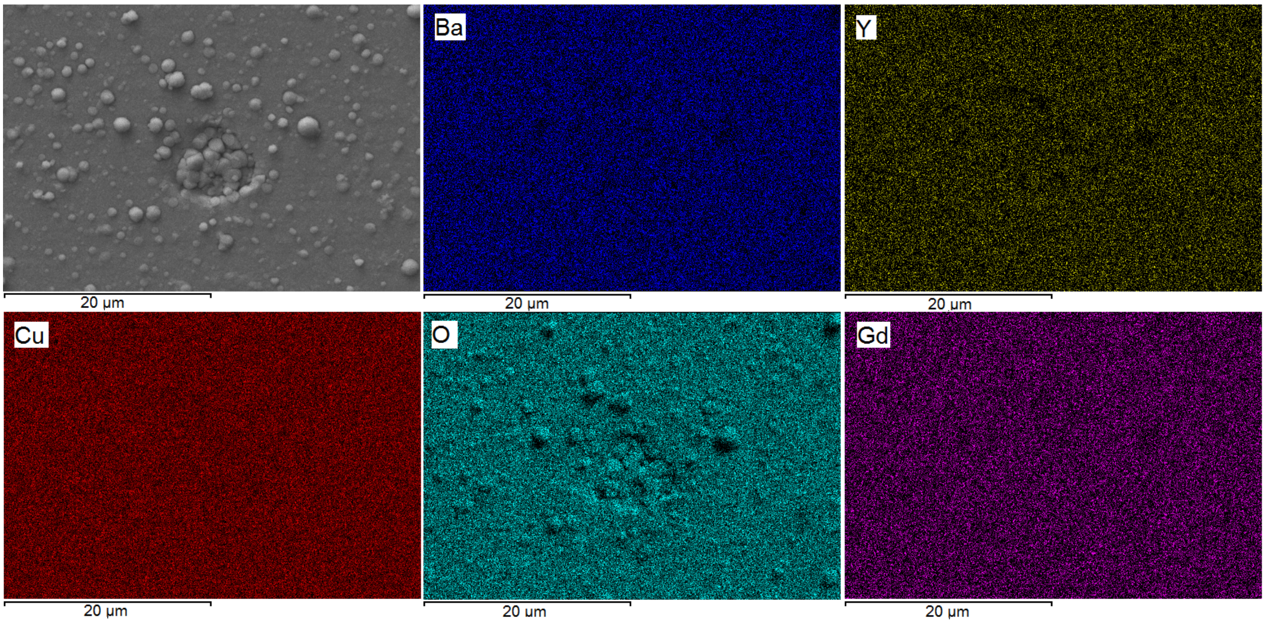

Fig. 5 *EDX maps obtained from surface of PLD thin film*

There were also concentration fluctuations of oxygen around outgrowths. Uneven distribution of oxygen predicts the variation in the stoichiometry, which could result in loss of superconducting properties. Elemental maps of the MOCVD thin film surface in Figure 6 illustrate uneven distribution of the majority of elements in the MOCVD thin film. Relatively strong partitioning of copper between the matrix and outgrowths is observable; on the other hand the depletion of Barium was observable in copper enriched areas. It could be assumed that the outgrowths are of type Cu-O (1), so they are definitely out of superconducting material. The Oxygen fluctuation in both the investigated thin films is comparable.

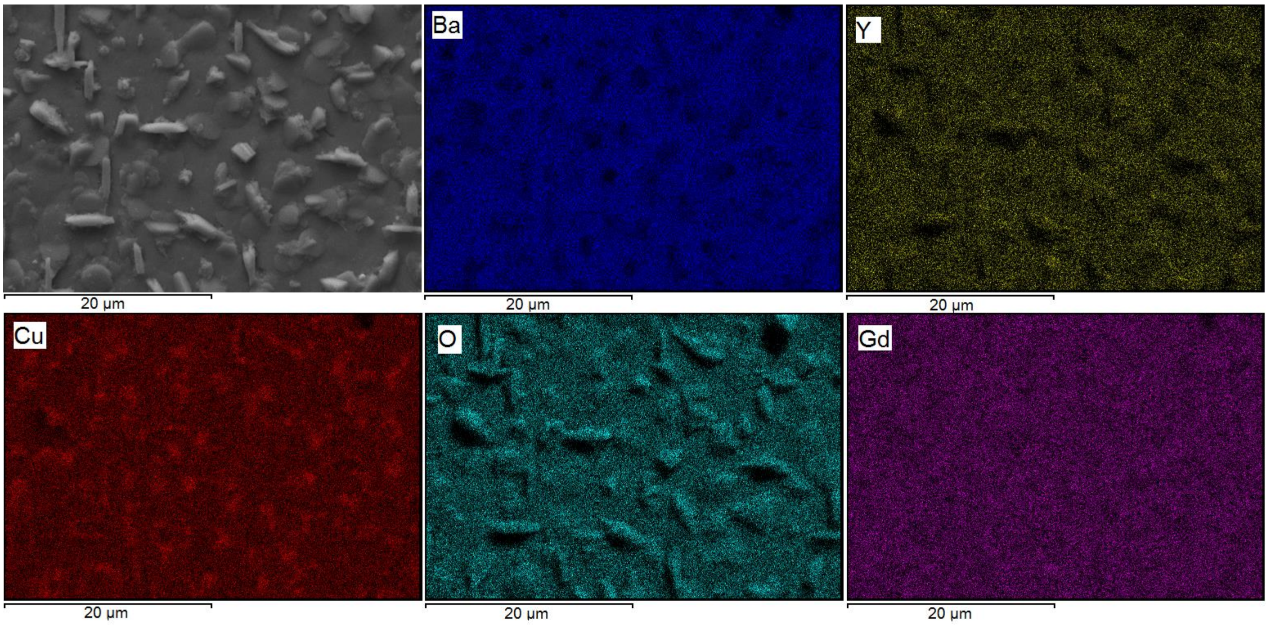

Fig. 6 *EDX maps obtained from surface of MOCVD thin film*

CONCLUSIONS

The main aim of this study was to characterize and compare (RE)BCO thin films deposited by PLD and MOCVD techniques. The occurrence of outgrowths was observed in both the thin films. They exhibited however different morphologies. The thin film deposited by PLD contained mainly outgrowths of rounded shape. The MOCVD thin film contains predominantly needle-like outgrowths. These outgrowths were penetrating more into the (RE)BCO layer and had higher density in comparison with the outgrowths from the PLD film. Thus, they can affect significantly the effective cross-section. EDX measurements show a good chemical homogeneity of the PLD thin film and a partial chemical heterogeneity of the MOCVD thin film. The PLD seem to be more usable technique for preparing films for both low current applications where thin interconnecting lines are needed and high current applications where the outgrowths cause obvious difficulty for orderly sequential thin film growths (1).

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