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## Surface scaling analysis of textured MgO thin films fabricated by energetic particle self-assisted deposition



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#### ABSTRACT

In the fabrication of a high-temperature superconducting coated conductor, the surface roughness and texture of buffer layers can significantly affect the epitaxially grown superconductor layer. A biaxially textured MgO buffer layer fabricated by ion beam assisted deposition (IBAD) is widely used in the coated conductor manufacture due to its low thickness requirement. In our previous study, a new method called energetic particle self-assisted deposition (EPSAD), which employed only a sputtering deposition apparatus without an ion source, was proposed for fabricating biaxially textured MgO films on non-textured substrates. In this study, our aim was to investigate the deposition mechanism of EPSAD-MgO thin films. The behavior of the surface roughness (evaluated by  $R_{\rm q}$ ) was studied using atomic force microscopy (AFM) measurements with three scan scales, while the in-plane and out-of-plane textures were measured using X-ray diffraction (XRD). It was found that the variations of surface roughness and textures along with the increase in the thickness of EPSAD-MgO samples were very similar to those of IBAD-MgO reported in the literature, revealing the similarity of their deposition mechanisms. Moreover, fractal geometry was utilized to conduct the scaling analysis of EPSAD-MgO film's surface. Different scaling behaviors were found in two scale ranges, and the indications of the fractal properties in different scale ranges were discussed.

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#### 1. Introduction

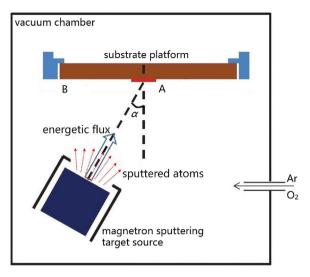
The high-temperature superconducting (HTS) coated conductors could be widely applied in the manufacturing of electrical equipments with high energy density [1]. As the key component of the coated conductor, a YBa $_2$ Cu $_3$ O $_{7-x}$  (YBCO) HTS layer has sensitive weak inter-granular links, thus its critical current decreases sharply with increasing grain boundary angles [2]. Therefore, it is crucial to prepare a buffer layer with a favorable surface and texture qualities as a template for the epitaxial growth of the YBCO layer [3,4]. MgO thin film is regarded as a proper and promising buffer layer for generating the biaxial texture of coated conductors [5]. Ion beam assisted deposition (IBAD) [6,7] has been widely used to fabricate MgO buffer layers on non-textured substrates. Its low thickness requirement (typically 10 nm) to develop biaxial texture [8,9] is a key factor in promoting the industrialization of coated con-

Magnetron sputtering is a popular physical vapor deposition (PVD) method widely used in the fabrication of oxide thin films because of its advantages of low cost, repeatability, and large area fabrication adaptability [11,12]. During the deposition, thin films could be resputtered by the bombardment of energetic particles mainly comprising of oxygen atoms and negative oxygen ions [13–15]. According to our previous study, the energetic particle flux was well collimated [16], and its particle energy was similar to that of the assisting ion beam of IBAD process. Thus in our other study [17], a new method called energetic particle self-assisted deposition (EPSAD) was proposed to fabricate a biaxially textured MgO film on non-textured substrates.

Besides the texture evaluation, surface analysis using atomic force microscopy (AFM) was also used to obtain more understandings of the IBAD-MgO mechanism as reported by Miyata [9]. Surface roughness of thin films was found to be strongly dependent on the scan scale of AFM [18,19], especially for surfaces with a fractal nature [20,21]. For a fractal surface, roughness described by root mean squared value ( $R_0$ , also known as RMS) of a fractal surface

ductor manufacture [10]. However, the ion source is an expensive component of the IBAD system.

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**Fig. 1.** Schematic diagram of experimental apparatus for energetic particle self-assisted deposition (EPSAD).

followed a power law relationship with the scan scale (L) [19,22], i.e. the equation below, and such a relationship indicates an  $R_{\rm q}$ -L linear curve in the log–log coordinates. The Hurst exponent (H) was an effective parameter in describing the  $R_{\rm q}$ -L relationship and the fractal dimension (D, equaling 3-H). Kulesza [22] reported that the scaling analysis of surface roughness was one of the most accurate methods of characterizing fractal dimension. Moreover, fractal properties typically existed in a limited range of scan scale in real world [20].

$$R_{\mathbf{q}} = AL^{H} \tag{1}$$

In this study, surface morphology characteristics were systematically studied to investigate the deposition mechanism of EPSAD-MgO thin films. AFM measurements were performed on the surface of the EPSAD-MgO thin films with various thicknesses. Values of surface roughness were characterized by  $R_{\rm q}$ . X-ray diffraction (XRD) measurements were conducted to evaluate the thin films' textures. The behaviors of surface roughness and textures were discussed to carry out a comparative analysis of EPSAD and IBAD. The deposition mechanism and fractal properties of the EPSAD-MgO thin films were discussed.

#### 2. Experimental details

#### 2.1. EPSAD-MgO film fabrication

As illustrated in Fig. 1, the film deposition apparatus is an off-axis radio-frequency unbalanced magnetron sputtering system, with a magnesium target of 99.99% purity. Hastelloy C276 alloy tapes (5 mm  $\times$  5 mm) coated with amorphous Y<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> seed layers were used as substrates. To get bombarded by the collimated energetic particle flux with the incidence angle  $\alpha$  of 55°, the substrates were placed in the area directly facing the target center, i.e. point A. The sputtering atmosphere consisted of oxygen and argon with a molar ratio of 1/7. The growth rate of the MgO film was about 2 nm/min, and the deposition duration varied from 0.5 to 10 min, which determined the film thickness accordingly from 1 to 20 nm. Other detailed experimental parameters could be found in our previous study [17].

#### 2.2. AFM and XRD measurements

AFM in the tapping mode was utilized to measure the surface morphology of EPSAD-MgO samples using Nanoscope III (Digital Instrument). The as-measured AFM image had  $256 \times 256$  pixels. To analyze the AFM images, flatten modification was a necessary procedure, in which the slope and curvature could be removed by subtracting the fitted polynomials from the as-measured data while the waviness with the higher spatial frequencies could be kept after the flatten modification. Flatten modification with order 2, which is commonly used in coated conductor research, was performed in this study. Our previous study [19] indicated that surface roughness was actually highly correlated to L. Therefore, AFM measurements were carried out with different L of  $0.5 \mu m$ ,  $1 \mu m$  and  $5 \mu m$ . L of 5 µm was one of the most commonly used scales in the research of coated conductors' preparation technologies [23]. L of 1 µm was used in Miyata's study [9] on surface morphology of IBAD-MgO; thus, it was also applied in this study to carry out a comparative analysis. Then, 0.5 µm was applied to perform a further detailed study on the film surface. AFM images were turned into matrix ASCII data, and roughness values at various L values were calculated through MATLAB.

After the surface characterization using AFM, homo-epitaxial MgO layers about 100 nm thick were post-deposited on the EPSAD-MgO samples to conduct XRD measurements using Rigaku SmartLab. The homo-epitaxial deposition was performed at point B as illustrated in Fig. 1, where the MgO films suffered no bombardment by energetic particles as reported in our previous studies [16,17]. XRD  $\phi$ -scan of MgO (002) plane ( $\chi$  angle fixed at 54.7°) and  $\omega$ -scan of MgO (111) plane were conducted, and their full width at half maximum (FWHM) values were calculated to evaluate the in-plane and out-of-plane textures, respectively.

#### 3. Results and discussion

#### 3.1. Surface roughness and texture variations

The AFM images at different L of EPSAD-MgO samples with various thicknesses are provided in Fig. 2. It could be observed that the surface morphology of fine grains changed slightly when film thickness was below 10 nm, whereas the grains became much larger with film thickness increasing to 20 nm. Moreover, the film morphologies indicated in AFM images with L of 0.5  $\mu$ m and 1  $\mu$ m were mainly featured by polycrystalline grains, while the images with L of 5  $\mu$ m were mainly featured by surface waviness of larger scales.

 $R_{\rm q}$  values at L of 0.5  $\mu$ m, 1  $\mu$ m and 5  $\mu$ m were calculated using the original data of AFM images, and their variations against film thickness are illustrated in Fig. 3. The  $R_{\rm q}$  curves remained almost steady when thickness ranged from 1 nm to 10 nm but rose sharply when thickness was 20 nm, which was consistent with the development trend of surface morphology illustrated in Fig. 2.

The FWHM variation curves of  $\phi$ -scan and  $\omega$ -scan were also plotted in Fig. 3. The  $\phi$ -scan FWHM curve firstly dropped when film thickness increased and reached the minimum (about  $10^\circ$ ) at a thickness of 10 nm; thereafter, it increased rapidly. The trend of the  $\omega$ -scan FWHM curve was very similar to that of  $\phi$ -scan FWHM, reaching its minimum (about  $2^\circ$ ) at 5 nm. Based on the trends of both FWHM curves, it could be inferred that the biaxial texture of the EPSAD-MgO thin film developed when its thickness was several nanometers, reaching the optimum at a thickness of 5 to 10 nm, but deteriorated after thickness exceeded 10 nm.

#### 3.2. EPSAD-MgO and IBAD-MgO comparison

The texture development of EPSAD-MgO samples was similar to that reported in IBAD-MgO studies [6,7]. The analysis based on surface morphology development could provide more understanding on the deposition mechanism of MgO thin films, as reported by Miyata [9]. In this study, the AFM images were first processed using

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