

# Effect of water partial pressure on the texture and the morphology of MOD-YBCO films on buffered metal tapes

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**Abstract**-- The influence of water partial pressure in Metal-organic Deposition (MOD) method was investigated on the texture and the morphology of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (YBCO) films grown on the buffered metal tapes. The water partial pressure was varied from 4.2% up to 10.0% with the other process variables, such as annealing temperature and oxygen partial pressure, kept constant. In this work, the fluorine-free Y & Cu precursor solution added with Sm was synthesized and coated by the continuous slot-die coating & calcination step. The next annealing step of the YBCO films was done by the reel-to-reel method with the gas flowed vertically down. From the x-ray diffraction analysis, the un-reacted phase like  $\text{BaF}_2$  peak was found at the water partial pressure of 4.2%, but  $\text{BaF}_2$  peak intensity is much reduced as the water partial pressure is increased. However, the higher water partial pressure of about 10% in this experiment leads to the poor crystallinity of YBCO films. The morphologies of the YBCO films were not different from each other when the water partial pressure was varied in this work. The maximum critical current density of  $3.8\text{MA}/\text{cm}^2$  was obtained at the water partial pressure of 6.2% with the annealing temperature of  $780^\circ\text{C}$  and oxygen partial pressure of 500ppm.

## 1. INTRODUCTION

The fabrication of long length coated conductors (CC) using high temperature superconducting materials has been conducted worldwide [1, 2]. And using Metal-organic Deposition (MOD) method, one of the well known processes to fabricate the superconducting CC, the high performance of the CC has been confirmed with the critical current density over  $\text{MA}/\text{cm}^2$  and long-length over several tens of meters [2, 3].

However, the conversion to superconducting YBCO films from the YBCO precursor films are poorly understood in aspect of microstructural development in MOD method. And the processing variables such as annealing temperature, oxygen partial pressure, and water partial pressure in MOD method essentially control the development of microstructure of the films. Among these variables, the water partial pressure was important factor affecting the growth mechanism of the MOD-YBCO films [3-5].  $\text{H}_2\text{O}$  gas went into the interface between the capping

layer and YBCO precursor films, whereby the chemical reaction occurred and the resulting product HF gas evolved from the growth-front up to the surface of the precursor films. The reaction between the  $\text{BaF}_2$  and  $\text{H}_2\text{O}$  promote the epitaxial nucleation of YBCO films and the following growth of textured YBCO films through the thickness of the films [5].

For the thicker films above the  $1\mu\text{m}$  usually done by the multi-coating technique in MOD method, low water partial pressure, which is lower than one generally used for the single coated film, was mentioned to be more effective to grow highly textured films with high superconductive performance. And it is believed to be related to the driving force for nucleation of grains in the films [6].

Also reported was that no difference in texture development of the MOD-YBCO films on single crystal substrate was observed with the water partial pressure of 2.3, 4.2, and 12.1%, and oxygen partial pressure and annealing temperature was fixed at 1000ppm and  $800^\circ\text{C}$  [7].

The process variables are closely related each other, so it makes it difficult to observe the dependence of the water partial pressure alone on the development of texture and the microstructure of the MOD-YBCO films.

In this paper, the continuous slot-die coating & calcination step and the reel-to-reel annealing step was adopted to fabricate the MOD-YBCO films on the buffered metal tapes. In reel-to-reel annealing step, the water partial pressure was varied to investigate the effect on the texture and the microstructure of the MOD-YBCO films.

## 2. EXPERIMENTAL

The buffer layer architecture  $\text{Y}_2\text{O}_3/\text{YSZ}/\text{CeO}_2$  was deposited on the biaxially aligned Ni-5at%W alloy tape. The three buffer layers were all (100) grown epitaxially to transfer the texture development from the bottom metal substrates to the top superconducting layer.

The fluorine-free Y & Cu precursor solution added with Sm was synthesized and coated on the buffered metal tape using the continuous slot-die coating & calcination step. Here, calcining process was performed at  $400^\circ\text{C}$  in humid oxygen atmosphere in reel-to-reel method.

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The reel-to-reel annealing process using the vertical gas flow system was adopted to convert the YBCO precursor films to superconducting YBCO films. The YBCO precursor films are converted at 780°C with the humid Ar/O<sub>2</sub> gas mixture flowed vertically down onto the moving tape. The oxygen partial pressure of about 500ppm was used. And the water partial pressure was varied from 4.2% up to 10.0% measured with thermocouple in the water reservoir. To prevent the condensation of water, the gas line tube was heated 20°C above the water temperature.

For the phase identification and development, x-ray diffraction (XRD) was used. And the observation of surface and cross section of the YBCO films was conducted with the field emission-scanning electron microscope (FE-SEM). The measurement of critical current was performed using the four-probe method at 77K, self-field with the criterion of 1μV/cm.

### 3. RESULTS AND DISCUSSION

The typical  $\theta$ -2 $\theta$  scan of the buffered metal tapes was shown in Fig. 1. All three buffer layer Y<sub>2</sub>O<sub>3</sub>/YSZ/CeO<sub>2</sub> was grown epitaxially on the biaxially textured metal tapes. And the in-plane alignment of the buffer layers were identified with the measurement of the FWHM values of the  $\phi$ -scans. 7.4°, 7.2°, and 7.3° for Ni-5at%W(111), YSZ(111), and CeO<sub>2</sub>(111) were obtained respectively as shown in Fig. 2.

The water partial pressure was varied from 4.2% to 10.0% by controlling the temperature of water bath. The un-reacted phase like BaF<sub>2</sub> was identified at the water partial pressure of 4.2% as shown in Fig. 3(a) [5]. As the water partial pressure increased BaF<sub>2</sub> peak was much reduced and phase pure c-axis aligned texture was obtained as in Fig. 3(b). At the water partial pressure of 8.1% the crystallinity of the YBCO films shows almost the same as that of 6.2% shown in Fig. 3(c). However, poor texture was developed at the higher water partial pressure of 10.0%. The peak broadening of (00L) peaks of the YBCO films was severe and the intensity of the (00L) peaks became smaller in Fig. 3(d) [6, 7].

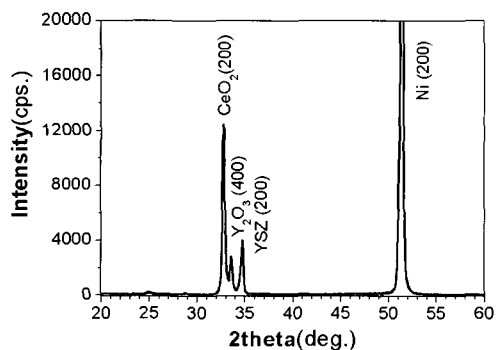


Fig. 1. XRD  $\theta$ -2 $\theta$  scan of the biaxially textured metal substrate buffed with Y<sub>2</sub>O<sub>3</sub>/YSZ/CeO<sub>2</sub> architecture.

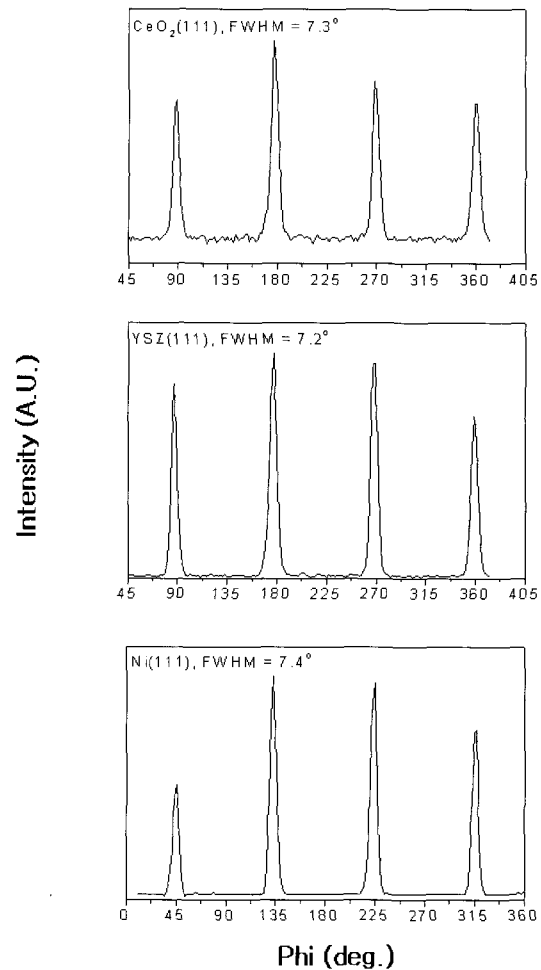


Fig. 2. XRD (111)  $\phi$ -scans of buffer layers Y<sub>2</sub>O<sub>3</sub> /YSZ /CeO<sub>2</sub> on the biaxially textured Ni-5%W showing well textured in-plane alignment.

During the temperature ramp region of annealing step, the nucleation of c-axis oriented grain begins from the interface between the top buffer layer and YBCO layer. The water vapor went through the YBCO precursor films down to the interface and nucleation started from that place. Then the converted YBCO films grew upwards using chemical reaction between the water vapor and YBCO precursor films [7].

At this stage, BaF<sub>2</sub> react with water vapor and release the HF gas outwards. For the higher water partial pressure the nucleation can initiate every part of YBCO precursor films, so nucleation sites above the interface might lose the epitaxial growth. More detail investigation will be performed for these phenomena.

At the water partial pressure lower than the 6.2%, the dense surface morphologies were observed and c-axis matrix was well connected as shown in Fig. 4. As the water partial pressure increased above the 8.1%, the surface became rougher and precipitates were observed by the FE-SEM.

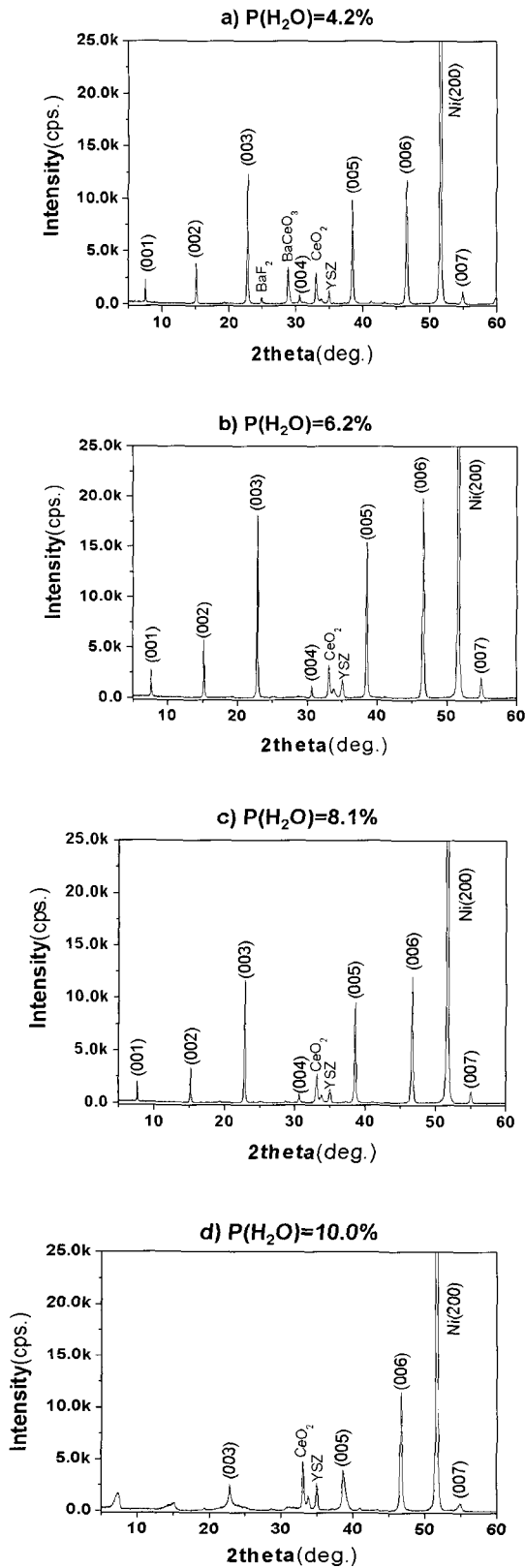


Fig. 3. XRD  $\theta$ - $2\theta$  scans of MOD-YBCO films on buffered metal tapes processed at the different water partial pressure.

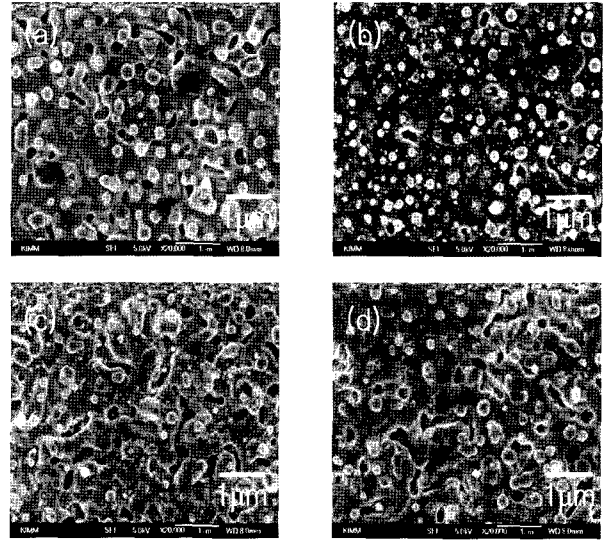


Fig. 4. Surface micrographs of MOD-YBCO films at different water partial pressure of 4.2% (a), 6.2% (b), 8.1% (c), and 10.0% (d) observed by FE-SEM.

We also observed the Ba-rich phase on the surface annealed at the water partial pressure of 8.1%. However, the surface was covered mostly with the matrix of c-axis grains and no a-axis grains were detectable within the variation of the water partial pressure in this experiment.

The critical currents of MOD-YBCO films processed at the water partial pressure of 4.2% and 10.0% were zero possibly due to the  $\text{BaF}_2$  phases and poor crystallinity of the films. However, at the water partial pressure of 6.2% and 8.1% critical current density over  $\text{MA}/\text{cm}^2$  were obtained and the maximum critical current density was  $3.8\text{MA}/\text{cm}^2$  at the water partial pressure of 6.2% with the annealing temperature of  $780^\circ\text{C}$  and oxygen partial pressure of 500ppm.

#### 4. CONCLUSIONS

The effects of the water partial pressure on the texture and the morphology of the MOD-YBCO films were investigated. The water partial pressure was varied from 4.2% up to 10.0% with the other process variables kept constant.  $\text{BaF}_2$  peak was found at 4.2%, which implied the reaction is incomplete due to low water supply to the growth-front of the YBCO films. But  $\text{BaF}_2$  peak intensity was much reduced and disappeared as the water partial pressure was increased. However, the higher water partial pressure of about 10.0% in this experiment lead to the poor crystallinity of YBCO films. In contrast, the morphologies of the YBCO films were not much different from each other when the water partial pressure was varied in the range of this experiment.

### ACKNOWLEDGMENT

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