Fabrication of CeO₂ Buffer Layer Using MOD Process

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Abstract—Biaxially textured Ni was fabricated by electrodeposition process and delaminated from the biaxially textured cathode surface for further buffer layer deposition process. Those electrodeposited Ni substrates showed well-developed biaxial texture and smooth surface. In order to improve the thermal stability of Ni substrates, Mn was alloyed by adding Mn precursor into the electrodeposition bath. Subsequently, CeO₂ buffer layers are deposited by MOD process to prevent interfacial reaction between superconductor and substrates. In particular, Bismuth oxide was added to CeO₂ to realize lower temperature processing of buffer layers. The microstructure and texture development of each layers have been investigated. Preliminary results shows that all electro/chemical process can be a candidate for cost effective route to YBCO coated conductor.

1. INTRODUCTION

There have been many efforts to develop long lengths of high quality superconducting tape in the area of second-generation high temperature superconductor (HTS) wires. Diverse processes have been used to fabricate YBa₂Cu₃Oₓ₋ₓ (YBCO) as well as the biaxially textured template substrate on which the superconducting films are deposited [1-3].

The biaxially textured template substrate for YBCO coated conductors are popularly fabricated with RAbiTS (Rolling-assisted Biaxially Textured Substrate) process followed by subsequent buffer layer deposition with sputtering, pulsed laser deposition, evaporation, etc. The RAbiTS™ process uses industrially scalable rolling and annealing technology. Since, however, metallic substrate prepared by RAbiTS method requires mechanical processing and subsequent annealing process at high temperature, it requires precise control of processing parameters and heavy equipment for mechanical processing. In addition, conventional fabrication method for buffer layers requires expensive vacuum deposition equipment. Then, many efforts to develop low cost fabrication method such as MOD process are going on. In particular, it was shown that a low cost processing of YBCO coated conductors can be possible by combining MOD buffered RAbiTS tape with MOD processing of YBCO films[2].

Recently, present authors showed that a biaxially textured nickel layer can be obtained by electrodeposition process assisted biaxially textured cathode materials [4]. Compared with thermomechanical process such as RABiTS process, the manufacturing cost of electrodeposition process is very small, especially in the production of thin metallic foils. In addition, metallic foils fabricated by electrodeposition process show high tensile strength which may be oriented from the small grain size of metallic foil (a few micrometers)[5].

In this study, a new low cost approach to coated conductor fabrication was developed by combining electrodeposition of biaxially textured Ni tape and MOD processing of buffer layers. In particular, a low temperature processing route for MOD buffer layer was developed to obtain smooth surface and maintain biaxial texture of substrate.

2. EXPERIMENTALS

2.1. Electrodeposition of Ni based foil

Biaxially textured Ni tapes were employed as cathode materials for electrodeposition. The cathode material was electropolished and solution treated for subsequent delamination. The electrodeposition of Ni based alloy tape was performed with nickel sulfamate-based solution. In particular, Mn was added to the solution to fabricate Ni-Mn alloy deposit. After electrodeposition, the deposited Ni alloy tape was delaminated and dried.

2.2. MOD processing of buffer layers

CeO₂ was deposited on the surface of electrodeposited Ni tape by MOD processing method. Cerium acetylacetone was dissolved into 2-methoxyethanol with suitable chelating agent. The bismuth nitrate was added to enhance low temperature processing of buffer layers. The prepared precursor solution for buffer layer processing was dip coated and calcined at 350°C. After calcination, calcined substrates with precursor films were annealed at 850°C under N₂/H₂(4%) mixed gas atmosphere.

3. RESULTS AND DISCUSSIONS

3.1. Electrodeposition of biaxially textured tapes
Ni layer was electrodeposited on biaxially textured cathode which was electropolished for smooth surface. After electrodeposition and delamination of Ni layer, the crystal texture of Ni layer was characterized by XRD. Fig. 1 shows a phi scan data of Ni layer deposited on electropolished cathode or unpolished cathode. The electropolished cathode gives much improved in-plane texture compared with unpolished cathode. Fig. 1 shows the development of biaxial texture of the delaminated Ni layer. The electrodeposits show well-developed biaxial texture and out-of-plane and in-plane texture of 30um-thick Ni layer were estimated to be $\Delta\theta=6.8^\circ$, $\Delta\phi=7.1^\circ$, respectively. This implies that the biaxial texture of cathode was transferred to Ni layer by epitaxial electrodeposition. Thus strongly cube-textured Ni layers were electrodeposited on Ni and delaminated.

Since the conventional MOD processing of buffer layer requires annealing at high temperature, thermally stable substrates are required. It was known that the thermal stability of Ni electrodeposits can be improved by addition of Mn [5]. Fig. 2 presents the change of biaxial texture of Ni electrodeposits after annealing at 850°C for 30min. under $N_2/H_2$ (4%) atmosphere. No significant change in the biaxial texture of Ni alloy layer was observed and this implies the Ni-Mn electrodeposit is thermally stable in this condition.

Thus, biaxially textured Ni alloy tape maintaining biaxial texture after annealing at 850°C was successfully fabricated by electrodeposition on textured cathode with subsequent delamination.

Fig. 1. XRD analysis of biaxial texture of delaminated nickel layers. (a) theta scan, (b) phi scan

![Fig. 1. XRD analysis of biaxial texture of delaminated nickel layers.](image)

Fig. 2. Biaxial texture of Ni-Mn layers. (a) as-electrodeposited state, (b) after annealing at 850°C

Fig. 2. Biaxial texture of Ni-Mn layers.

![Fig. 2. Biaxial texture of Ni-Mn layers.](image)

3.2. MOD processing of buffer layers

CeO$_2$-based buffer layer was fabricated on an electro-deposited Ni-Mn tape by MOD technique. In conventional MOD processing of buffer layers, typical annealing temperature is higher than 1000°C [5]. As a preliminary test, this typical processing condition was employed to form CeO$_2$ layer on electrodeposited metal foil. Fig. 3 shows XRD profile of CeO$_2$ coated Ni-Mn layer after annealing at 1100°C. Only (111) oriented CeO$_2$ was formed and (111) oriented grain in Ni alloy tape were dominantly formed. Hence, the conventional MOD processing condition for buffer layer growth cannot be applied to electrodeposited Ni alloy tapes.

Since the Ni-Mn electrodeposits is stable with respect to annealing at 850°C, a low temperature processing route for CeO$_2$ buffer layer was devised. It is expected that low temperature crystallization of CeO$_2$ buffer layer is possible by applying additives with low melting temperature. In this study, Bi$_2$O$_3$ was employed as an additive for CeO$_2$. Since the melting point of Bi$_2$O$_3$ (820°C) is much lower than that of CeO$_2$ (2600°C), low temperature crystallization of CeO$_2$ is expected by addition of Bi$_2$O$_3$.

Fig. 3. X-ray diffraction profile of CeO$_2$ layer MOD processed on electrodeposited Ni-Mn tape.

![Fig. 3. X-ray diffraction profile of CeO$_2$ layer MOD processed on electrodeposited Ni-Mn tape.](image)
Fig. 4 shows change of phase formation by addition of Bi$_2$O$_3$. Only small trace of CeO$_2$ was detected for specimen fabricated without any addition of Bi$_2$O$_3$. As addition of Bi$_2$O$_3$ increased, the CeO$_2$(200) reflection became higher. This implies that the crystallinity of CeO$_2$ film is improved by addition of Bi$_2$O$_3$. However, CeO$_2$ film prepared by addition of Bi$_2$O$_3$ more than 10% shows poor crystallinity. From the XRD result shown in Fig. 4, it is concluded that CeO$_2$-10%Bi$_2$O$_3$ composition is suitable for low temperature MOD processing of CeO$_2$ films.

The surface of CeO$_2$-10%Bi$_2$O$_3$ film consists of nanocrystalline grains with uniform size distribution as shown in Fig. 5(a). XRD theta scan result for CeO$_2$(200) reflection of CeO$_2$-10%Bi$_2$O$_3$ film (Fig. 5(b)).

![Fig. 4. XRD result of CeO$_2$-xBi$_2$O$_3$ after annealing at 850°C.](image)

The FWHM of theta scan profile was estimated to be about $\Delta \theta = 9.3^\circ$. Since the value of $\Delta \theta$ is still large, improvement in texture is required for application to further YBCO coating. The optimization of texture and thickness of CeO$_2$-10%Bi$_2$O$_3$ film is in progress.

### 4. SUMMARY

Electrodeposition of biaxially textured substrates and subsequent MOD processing of buffer layers were developed. A thermally stable metal substrate was fabricated with electrodeposition of Ni-Mn alloy. The electrodeposited Ni-Mn alloy foil maintains biaxial texture even after annealing at 850°C. MOD processing of CeO$_2$-xBi$_2$O$_3$ film using electrodeposited Ni-Mn alloy foil as a substrate was developed to reduce the annealing temperature. After annealing at 850°C under N$_2$/H$_2$ (4%) atmosphere, c-axis oriented CeO$_2$ film with uniform microstructure was formed with addition of 10% Bi$_2$O$_3$. However, improvement in texture is required for application to further YBCO coating. The optimization of texture and thickness of CeO$_2$-10%Bi$_2$O$_3$ film is in progress.

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### REFERENCES


