



Processing of High Temperature Superconductors

Edited by
Amit Goyal
Winnie Wong-Ng
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Volume 140

Processing of
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**Ceramic
Transactions**
Volume 140

Processing of High Temperature Superconductors

*Proceedings of the Processing of High Temperature Superconductors
symposium held at the 104th Annual Meeting of The American
Ceramic Society, April 28–May 1, 2002 in St. Louis, Missouri.*

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Preface

Major advances have been made during the last decade in the processing of ceramic, high temperature superconductors (HTS). High temperature superconductor materials are layered oxide compounds, which exhibit complex chemistry, including non-stoichiometry and defects (i.e., dislocations, stacking faults, and intergrowths).

For most large-scale, bulk applications of HTS, long lengths of flexible wires carrying large amounts of supercurrents are required. It has been well established that in order to achieve this long range, biaxial or triaxial crystallographic texturing of the superconductor is necessary. Significant efforts are now directed at attempts to fabricate long lengths of near single crystal-like superconducting wires by epitaxial growth on biaxially textured substrates. Typically these substrates comprise either a biaxially textured metal substrate with epitaxial oxide buffer layers, or an untextured metal substrate and biaxially textured oxide buffer layers. Techniques of substrate fabrication, which have received the most interest, include IBAD (ion-beam assisted deposition), RABiTS (rolling assisted biaxially textured substrates) and ISD (inclined substrate deposition). Epitaxial growth of $\text{YBa}_2\text{Cu}_3\text{O}_x$ (YBCO) and oxides on such substrates can be accomplished by a variety of techniques including pulsed laser ablation (PLD), electron beam evaporation, sputtering, chemical combustion vapor deposition (CCVD), jet vapor deposition, ex-situ BaF_2 process, ex-situ sol-gel techniques, and liquid phase epitaxy (LPE).

For other bulk applications, large, single crystal-like pucks of the superconductor are required. Applications requiring large domain levitators include frictionless bearings for flywheels, contact-less transportation, damping, flux-trap magnets, magnetic shields, and current leads. The superconducting material of choice in this case is melt-processed YBCO.

Lastly, basic information about HTS materials concerning phase diagrams, measurement of physical properties, characterization, and effects of various defects including grain boundaries on supercurrent transmission are of great interest and importance for further developments in this field.

This proceedings volume contains papers given at the Processing of High Temperature Superconductors symposium held during the 104th Annual Meeting of The American Ceramic Society (ACerS), April 28–May 1, 2002, in St. Louis, Missouri. The symposium focused on the above-mentioned issues pertaining to HTS materials as well as the following areas:

Materials Processing for Conductors

Biaxially textured substrates - RABiTS, IBAD, ISD
Other novel approaches to form biaxial texture
Physical vapor deposition techniques for deposition of oxide buffer layers and superconductors
Chemical vapor deposition techniques for deposition of oxide buffer layers and superconductor Novel synthetic methods
Physical properties (transport, flux pinning, field trapping)
Microstructure-property correlations
Issues related to scale-up
Application demonstrations

Materials Processing for Levitators

Fabrication of large area YBCO levitators
Magnetic characterization of levitators
Mechanical characterization of levitators
Issues related to scale-up
Application demonstrations

Basic Issues

Thermodynamics and phase equilibria
Grain boundary doping effects
Fundamental growth studies
Dopants, impurities and stability
Defects and microstructures
Non-stoichiometry
New materials such as MgB_2

The contents of this transaction volume comprise the proceedings of the focused session. A total of 34 scientific papers are featured in this volume. These contributions are divided into two parts: second generation wires and bulk processing. The order in which the papers appear here and the division into which they are organized may be different from that of their presentation at the meeting. It is hoped that this comprehensive volume will be a good summary of the latest developments in high-temperature superconductor research as well as good source material for researchers and managers working in this field.

We acknowledge the service provided by the session chairs and appreciate the valuable assistance from ACerS programming coordinators. We are also in debt to Ms. Mary Cassells and Ms. Sarah Godby for their involvement in editing and producing this book. Special thanks are due to the speakers, authors, manuscript reviewers, and ACerS officials for their contributions.

Amit Goyal

Winnie Wong-Ng

Masato Murakami

Judith Driscoll

Second Generation Wires

SURFACE-OXIDATION EPITAXY METHOD FOR CRITICAL CURRENT CONTROL OF $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ COATED CONDUCTORS

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ABSTRACT

The surface quality of NiO buffer, produced by surface-oxidation epitaxy (SOE) method, was improved by a polishing and a cap layer coating techniques. Critical current density (J_c) of the $\text{YBa}_2\text{Cu}_3\text{O}_x$ (YBCO) film directly deposited on the polished SOE-NiO buffer by pulsed laser deposition (PLD) method was increased up to $0.17\text{MA}/\text{cm}^2$ (77K, 0T) from $0.03\text{-}0.05\text{MA}/\text{cm}^2$ (77K, 0T, on the non-polished buffer).

In addition, a cap layer of perovskite oxide (BaSnO_3) was prepared for promoting the epitaxial growth of YBCO films on SOE-NiO buffer. As a result, J_c of the YBCO film formed on $\text{BaSnO}_3/\text{NiO}/\text{Ni}$ substrate reached $0.45\text{MA}/\text{cm}^2$ (77K, 0T). This is because the number of superconductive weak coupling in the YBCO film was reduced, by the flattening of the NiO surface and the coating of the perovskite oxide cap layer.

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INTRODUCTION

Simple and low cost production methods to form biaxially textured $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) films on long-length metallic substrates are necessary; in order to obtain high critical current density (J_c) YBCO coated conductors. We have proposed a method to produce YBCO coated conductors by using surface oxidized NiO layer grown on long-length nickel tapes. The biaxial orientation of NiO can be realized by considering the epitaxial growth of NiO on the nickel surface. We call this technique surface-oxidation epitaxy (SOE) method.^{1,2} The formation of NiO on cube-textured Ni, or Ni-based alloys has been widely investigated recently.^{3,4,5}

Biaxially textured NiO layer functions as a barrier layer of chemical reaction between YBCO and nickel, and as a template for the epitaxial growth of YBCO film. However, according to the research so far, the J_c of YBCO film directly deposited on NiO was only 0.03-0.05MA/cm² (77K, 0T).⁶ Although the NiO crystal made by SOE is highly in-plane oriented, the surface roughness induced by grooves in the NiO grain boundaries remains large. Such a rough surface may generate the tilt grain boundaries in YBCO films deposited on NiO, and bring about the J_c decrease when the tilt angle is high. Furthermore, a Ni contamination may also bring about the degradation of critical temperature (T_c) of YBCO, since Ni atom from the underlying Ni substrate is easy to diffuse in the grain boundary.

An insertion of MgO cap layer between NiO and YBCO film was very effective in order to solve these problems, and J_c of 0.3 MA/cm² (77K, 0T) was achieved on SOE-NiO.² Nevertheless, the possibility that the local degradation of J_c happens, in the rough surface area, has still remained because the roughness of the NiO surface has not been essentially improved. We have tried to polish SOE-NiO surface mechanically, and also insert a perovskite oxide cap layer between NiO and YBCO film for promoting the epitaxial growth of YBCO films on SOE-NiO buffer. Consequently, high J_c value of 0.45 MA/cm² (77K, 0T) was successfully achieved on SOE-NiO.⁷ In this paper, we describe J_c control of the YBCO films on the Ni tapes through SOE process.

ORIENTATION CONTROL OF NiO LAYER BY SOE METHOD

An oxide of underlying metal is formed on the metal surface by a thermal

oxidation. In an initial stage of the formation of the oxide, the fixed relation exists between crystal orientation of the grown oxide and that of the underlying metal surface. The typical preferred orientations of NiO on Ni(100) face are NiO(100) and NiO(111).⁸ Crystal orientation relationships between thermally oxidized NiO and Ni surfaces, which were observed at 500°C in oxygen atmosphere, are summarized in Table I.

Table I. Orientation relationships between oxidized NiO on several Ni surfaces

Metal	Oxide	Oxidation condition	Crystal plane	
			Ni	NiO
Ni (fcc) a=3.53 Å	NiO (cubic) a=4.17 Å	1atm, O ₂ 500°C 10~120min	{100} // {111}	[110] // [110]
			{100} // {100}	[110] // [110]
			{110} // {110}	[110] // [110]
			{110} // {114}	[110] // [110]
			{111} // {111}	[110] // [110]
			{113} // {110}	[110] // [110]

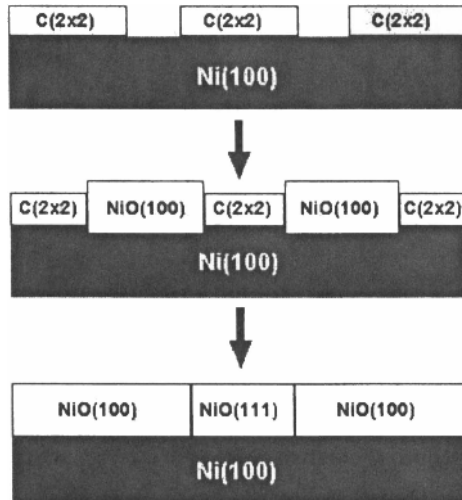


Fig.1. Schematic view of the initial stages of oxidation of Ni(100) surface.

The oxidation of metal begins by adsorbing oxygen molecules on the metallic surface. In the oxidation of (100) face of nickel single crystal, an adsorption of oxygen atoms by a two-dimensional arrangement is generated. A schematic view of the initial stages of Ni oxidation is presented in Fig.1. The layer of the simple cubic lattice in the period equal to nickel is gradually formed, when the adsorption advances, and finally, the isolated NiO nuclei is formed.⁹ The NiO nuclei grow and coalesce, and the Ni surface would be covered with NiO. Then, a fixed crystal orientation relation occurs between NiO and Ni by the regulation of crystal orientation on the surface of underlying metal. Generally, (100)- and (111)-oriented NiO species coexists on the Ni(100) face because lattice misfit between NiO and Ni is large (misfit=18.1%).

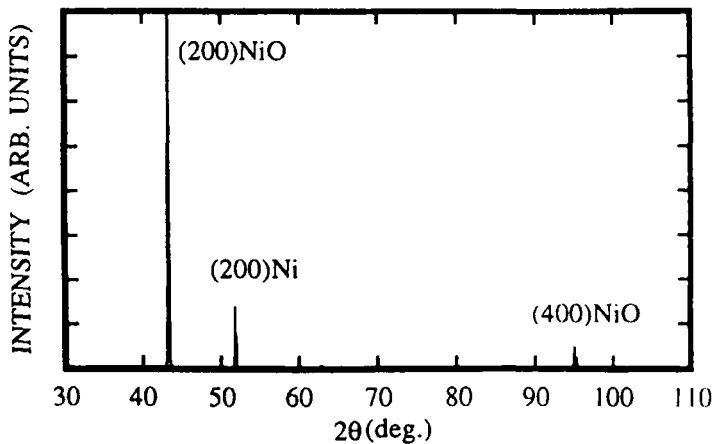


Fig.2. X-ray $\theta-2\theta$ scans data for SOE-NiO on the cube-textured Ni substrate.

We have discovered, however, the NiO(100) face became dominant at the elevated temperature above 1000°C in air. In particular, the (100)-oriented NiO grains grew predominantly on the cube-textured $\{100\}\langle 001\rangle$ nickel tape when it was oxidized under appropriate oxidation condition. Texture degree defined by $I(200)/\{I(200) + I(111)\}$ have reached 0.99 at 1200°C, where $I(200)$ and $I(111)$ mean the X-ray $\theta-2\theta$ intensities for NiO(200) and NiO(111) peaks. Figure 2 shows the typical X-ray $\theta-2\theta$ scan data for the SOE-treated NiO/Ni substrate.

Strong NiO(200) and (400) peaks are observed. This indicates the outermost layer of NiO is almost composed of the (100)-oriented NiO grains. SOE method utilizes these phenomena.

Cube-textured Ni tapes, prepared by the combination of rolling and heat treatment, are used for the SOE processing. The recrystallization heat treatment at 700-800°C is conducted in order to achieve both the flatness and the in-plane orientation of the Ni surface. The SOE heat treatment is carried out subsequently at around 1200°C to form the biaxially textured NiO layer on the Ni surface. Then, crystal orientation relation between NiO and Ni becomes NiO{001}//Ni{001} and NiO<110>//Ni<110>. Highly biaxial texture of SOE-grown NiO layer has been confirmed by the X-ray pole figure measurement. A typical FWHM of in-plane texture, $\Delta\phi$, of NiO(111) peak is 10-14 degrees and FWHM of out of plane texture, $\Delta\theta$, of NiO(200) peak become 6-8 degrees.

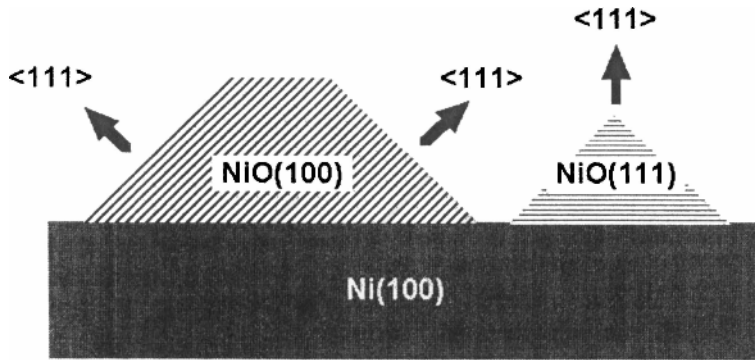


Fig.3. Orientation mechanism of biaxially textured SOE-NiO on Ni substrate.

At the initial stage of SOE process, (100)- and (111)-oriented NiO grains coexist. Why does NiO(100) face dominates on the cube-textured Ni tape after high temperature thermal oxidation? The reason would be speculated as follows: the growth rate of (111) face of cube crystal is larger than that of (100) face because (111) face is the non-singular face. Therefore, the NiO(111) grains grow only in the perpendicular direction, while it is permitted that the NiO(100) grains grow perpendicularly and horizontally, as illustrated in Fig.3. This means that the

NiO(100) grains laterally and quickly grow faster than the NiO(111) grains.

The (100)-oriented NiO grains coalesce and bury (111)-oriented NiO grains, and eventually covers all NiO surfaces. The NiO(100) grains grow by keeping epitaxial relation with Ni(100) surface, so that the biaxial orientation of NiO layer realizes. However, NiO surface becomes rough due to the coalescence of NiO(100) grains. The typical surface roughness of the SOE-NiO is approximately 30-150nm. The defects on the oxide buffer will induce the degradation of J_c of the YBCO films; hence the improvement of surface quality of SOE-NiO is strongly desired.

IMPROVEMENT OF J_c IN YBCO FILM ON SOE-NiO

YBCO films, prepared on SOE-NiO by pulsed laser deposition (PLD) with KrF excimer laser, show cube-on-cube epitaxy. A typical film formation condition is as follows: 700°C-substrate temperature, 200mTorr oxygen pressure and 2 J/cm² energy density. In Fig.4, X-ray pole figures of NiO(111) and YBCO(103) peaks for YBCO/NiO are shown. $\Delta\phi$ values of NiO(111) and YBCO(103) are 12 and 11 degrees, respectively.

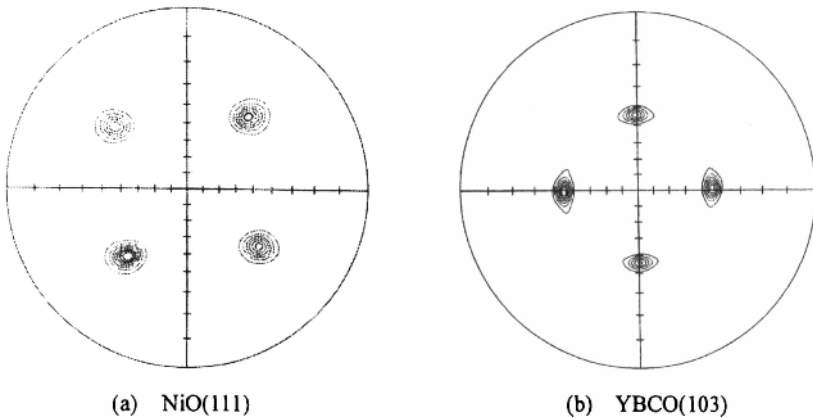


Fig.4. X-ray pole figures of NiO(111) and YBCO(103) peaks for YBCO/NiO.

However J_c of YBCO films deposited on the bare SOE-NiO was only 0.03-0.05 MA/cm² (77K, 0T). This is one order of magnitude lower than that expected

from in-plane texture of YBCO. Recently, the YBCO films with high J_c of 2-3MA/cm² (77K, 0T) have been successfully deposited on NiO(100) single crystal by PLD.¹⁰ Considering the potentiality of NiO(100) surface as a template for epitaxial growth of YBCO, surface roughness of NiO and/or contamination of YBCO film by Ni element through NiO grain boundaries seem to be causes of J_c degradation in YBCO/SOE-NiO system.

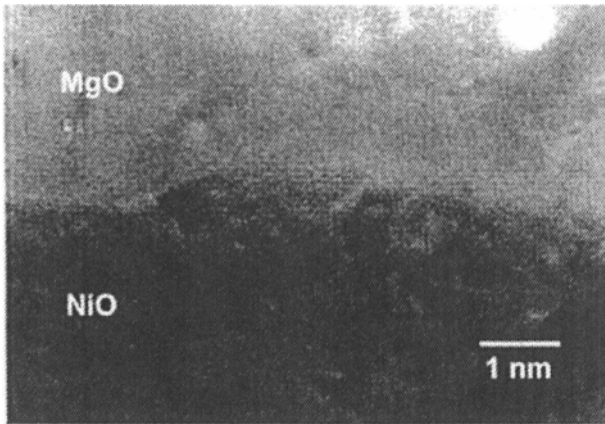


Fig.5. TEM cross-section of the interface between MgO layer and SOE-NiO.

It is effective to form a thin oxide cap layer on NiO in order to solve this problem. Improvement of surface quality of NiO has been attained by using thin MgO cap layer, and J_c of 0.3MA/cm² (77K, 0T) was obtained. The surface roughness of the buffer might be reduced with an excellent wetting property of MgO on NiO. TEM photograph of the interface between NiO and MgO is shown in Fig.5, where MgO was epitaxially and continuously grown on the SOE-NiO surface. MgO and NiO have a same crystal structure (rock-salt type) and they are completely soluble each other in solid state at all compositions. As a result the rough surface of NiO was nicely masked with MgO cap layer. MgO was also very effective in diffusion prevention of the Ni element. According to local EDX analysis, Ni did not diffuse in the MgO cap layer so that the Ni contamination was perfectly suppressed.

Even though MgO grows epitaxially on NiO, a careful control of the exact YBCO film deposition condition is necessary in order to form high J_c YBCO films on MgO. This is because the YBCO grains with cube-on-cube relation and with 45-degrees rotated orientation are easy to coexist on MgO.¹¹ Thus, the perovskite-type materials, which have better chemical compatibility to YBCO than that of MgO, were examined as a new cap layer in the following work.

MECHANICAL POLISHING OF SOE-NiO SURFACE

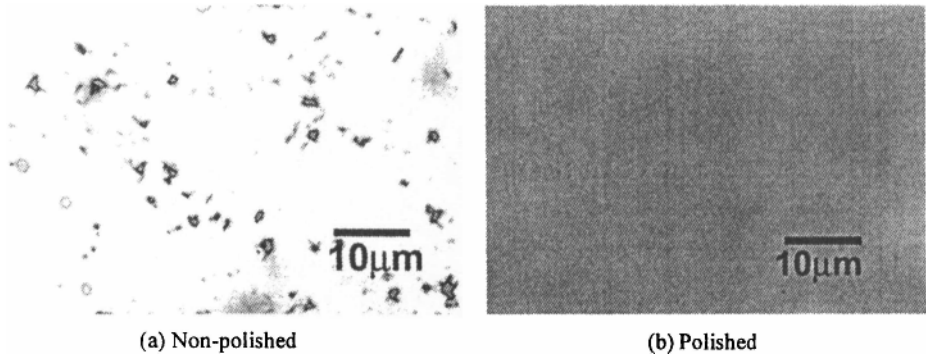


Fig.6. Photographs of Non-polished and polished SOE-NiO surfaces

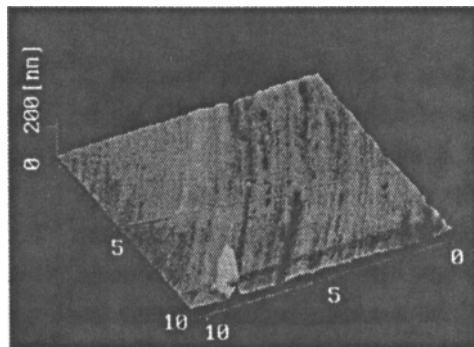


Fig.7. AFM picture of the polished SOE-NiO surface

In order to solve the rough-surface problem, the polishing of the SOE-NiO was investigated by using both 1 μm and 0.25 μm diamond pastes. Photographs of the non-polished and polished NiO surfaces are shown in Fig.6. The highly

smooth and dense NiO surface was realized by this technique. The thickness of polished NiO layer was about 3 μm . No cracking in NiO occurred during polishing. We observed the surface morphology of polished NiO by AFM. Fig.7 shows the AFM image of the surface morphology of smooth NiO. Scan area of NiO was set to $10 \times 10 \mu\text{m}^2$. From the measurement of AFM image, the average surface roughness was 1.1 nm for smooth area and 5.5 nm for whole area.

The YBCO film deposited on the polished NiO surface by PLD showed strong *c*-axis orientation and cube-on-cube epitaxy. In-plane texture $\Delta\phi$ that determined from YBCO(103) peak was also 11 degrees. This value was similar to those of the other texturing technique. We have measured critical current (I_c) of the film at 77K and 0T. The bridge width and length formed in the YBCO film were 1.8 mm and 2.0 mm, and also, YBCO film thickness on NiO was 0.5 μm . Four terminals were attached by silver paste to the YBCO and the specimen was immersed into the liquid nitrogen. I_c of the YBCO film reached 1.52 A; namely, this corresponded to J_c of 0.17 MA/cm² (77K, 0T). These results indicate that the smooth NiO surface is crucial in order to produce the high J_c YBCO films on SOE-NiO.

PEROVSKITE CAP LAYER COATING

On the polished SOE-NiO, the perovskite oxide cap layer and YBCO films were grown by PLD. The perovskite materials, such as BaTiO₃, BaSnO₃ and BaZrO₃ were considered as a candidate since they included BaO layer and the excellent chemical compatibility with YBCO film was expected. As a first step, we chose BaSnO₃ material as a new cap layer because the crystal lattice constant is 0.411 nm.¹² The value is approximate for the lattice constant of NiO (0.416 nm) and misfit between them is -1.2%. Figure 8 shows the new configuration of YBCO /cap layer/SOE-NiO.

Deposition of BaSnO₃ and YBCO on the polished SOE-NiO was carried out by PLD at an energy density of 2J/cm² and repetition rate of 10-25 Hz. The substrate temperature was varied in the temperature range of 700-780°C and oxygen pressure was changed from 30 to 200mTorr to determine the optimal deposition condition. The bridges of 1.0 mm width and 2.0 mm length were patterned in YBCO films for I_c measurement.

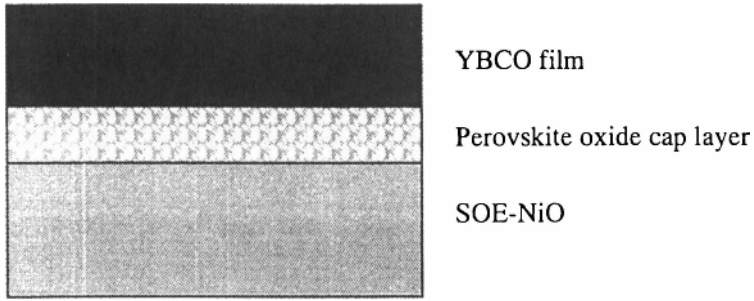


Fig.8. Multi-layer structure of YBCO/cap layer/SOE-NiO.

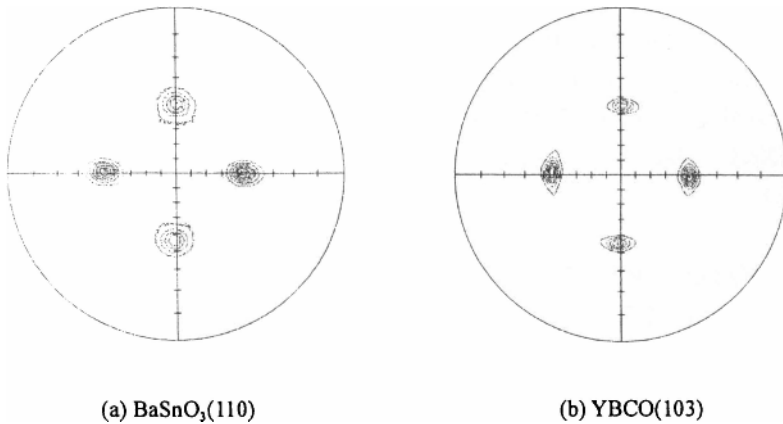


Fig.9. X-ray pole figures of $\text{BaSnO}_3(110)$ and $\text{YBCO}(103)$ peaks for $\text{YBCO}/\text{BaSnO}_3/\text{SOE-NiO}$ configuration.

The BaSnO_3 films deposited on the NiO were *c*-axis oriented and in-plane textured and their thickness were 0.3-1.0 μm . Subsequently, YBCO films were deposited on the BaSnO_3 layer by PLD. The thickness of YBCO films on $\text{BaSnO}_3/\text{NiO}$ was arranged to 0.5 μm . Figure 9 shows the pole figures of $\text{BaSnO}_3(110)$ and $\text{YBCO}(103)$ planes for $\text{YBCO}/\text{BaSnO}_3/\text{SOE-NiO}$ configuration.

It is proven that BaSnO₃ and YBCO films epitaxially grow on the polished NiO(100) surface with cube on cube relation. In-plane texture, $\Delta\phi$ of YBCO(103) X-ray peak, was 10.4 degrees and out of plane texture, $\Delta\theta$, for YBCO(005) X-ray peak was 2.5 degrees. This means that YBCO film on the BaSnO₃/NiO substrate had the advanced biaxial texture.

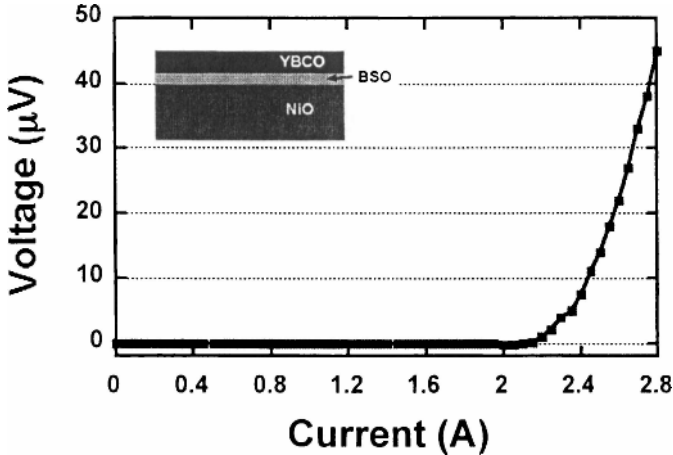


Fig.10. Current-voltage characteristics for YBCO/BaSnO₃/SOE-NiO system.

Current-voltage curve at 77K and 0T for YBCO/BaSnO₃/SOE-NiO specimen is presented in Fig.10. I_c of the patterned specimen was 2.25 A, which corresponded to J_c of 0.45 MA/cm² (77K, 0T). The improvement of J_c is due to the flattening of the NiO surface by the polishing and the good chemical compatibility between YBCO and BaSnO₃. It is considered that the perovskite material, such as BaSnO₃, is one of the candidates of cap layer on the NiO surface to obtain high J_c properties in the YBCO coated conductors by SOE method.

CONCLUSIONS

The surface roughness of NiO buffer by surface-oxidation epitaxy was improved by polishing technique. The surface roughness of NiO was reduced in this manner to 1-5nm, and J_c of 0.17MA/cm² (77K, 0T) was achieved for even YBCO films deposited on the bare NiO surface. In addition, the coating of the perovskite oxide cap layer was also examined instead of the MgO cap layer in

order to promote the epitaxial growth of YBCO. We have obtained J_c of 0.45 AM/cm^2 (77K, 0T) in YBCO/BaSnO₃/NiO/Ni system. The improvement of J_c was due to both the flattening of NiO surface and good epitaxy between YBCO and BaSnO₃. It is considered that the perovskite material, such as BaSnO₃, is one of the candidates of cap layer on the NiO surface to obtain high J_c properties in YBCO coated conductors by SOE method.

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CONTROL OF THE SULFUR $C(2 \times 2)$ SUPERSTRUCTURE ON $\{100\}\langle 100 \rangle$ -TEXTURED METALS FOR RABITS APPLICATIONS.

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ABSTRACT

We investigate the influence of a chemisorbed S template with centered (2×2) structure on the epitaxial growth of commonly used oxide buffer layers on $\{100\}\langle 100 \rangle$ metals for RABiTS fabrication. Our study involves growth of CeO_2 , Y-stabilized Zr_2O_3 , Gd_2O_3 , LaMnO_3 and SrTiO_3 seed layers on biaxially textured Ni and Ni-alloys. We also discuss the effect of an incomplete $c(2 \times 2)$ surface coverage on the seed layer texture and the use of H_2S in a pre-deposition anneal as a mean to control the superstructure coverage and optimize the seed layer texture.

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INTRODUCTION

Recently we found that sulfur impurity atoms present on the Ni (001) surface are determinant for the epitaxial growth of certain oxide films used as seed buffer layers in RABiTS conductors (e.g.: CeO₂ or Y₂O₃-stabilized ZrO₂) [1,2]. Our results showed that chemical and structural properties of the textured metal surface have to be considered in order to obtain the desired texture for the buffer layers. Using reflection high-energy electron spectroscopy (RHEED), and Auger electron spectroscopy (AES), our experiments revealed the existence of a sulfur superstructure on the textured Ni surface that forms after surface segregation of sulfur contained as a common impurity in the metal bulk. During the high-temperature texturing anneal S atoms diffuse to the surface of the tape and arrange in a centered (2 × 2) superstructure. However, depending on the initial S concentration and/or specific annealing conditions, the superstructure layer can exhibit different coverage.

Here we analyze the relationship between S surface concentration and seed layer texture, and present a method that controls and optimizes the superstructure coverage on the metal, providing reproducible quality of the seed layer texture. In addition, we extend our study of the influence of the S superstructure to the case of biaxially textured Ni alloys, such as Ni-3%W, and Ni-13%Cr.

SEED LAYERS ON {100}<100> Ni BY PULSED LASER DEPOSITION

CeO₂ seed layers are successfully deposited by vapor deposition techniques like e-beam evaporation, reactive sputtering, and pulsed laser deposition (PLD). All these techniques use a background H₂O pressure of about 1×10^{-5} Torr and a substrate temperature in the range of 600 to 750 °C. Water dissociation provides enough oxygen in the background to form and stabilize the CeO₂ phase without oxidizing the Ni [3]. The stability of the Ni surface in the presence of water with a partial pressure in the range of 1×10^{-5} Torr was investigated by *in situ* RHEED performed inside the laser molecular beam epitaxy (MBE) chamber used to fabricate films for this study. It was found that such H₂O partial pressure does not cause any structural modification of the sample surface that can be related to growth of NiO islands or adsorbed O. The documented first stage of Ni oxidation consists of a chemisorbed O layer with *p* (2 × 2) structure up to a coverage of 0.25 ML, and with subsequent *c* (2 × 2) structure up to a coverage of 0.5 ML [4] [1 ML ≡ (number of surface adsorbate atoms) / (number of surface substrate atoms)]. None of these structures was observed to form as a consequence of water exposure on a clean {100}<100> Ni surface at a typical deposition temperature of 600 °C. The clean Ni surface was obtained by depositing a Ni film *in situ* on the textured Ni substrate in ultrahigh vacuum conditions at 600 °C by PLD. RHEED