Scale up of Coated Conductor Substrate Process by Reel-to-reel Planarization of Amorphous Oxide Layers

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Abstract-Substrate surface smoothness comparable to electropolished substrate has been demonstrated by planarization of multilayers of amorphous oxide films on as received flexible metal tapes. An in-plane texture of 6.4 degrees was achieved on short samples after the final buffer process. A critical current density (J_c) over 3MA/cm² has been achieved on short samples. The planarization process has been scaled up from a few meters previously done by loop coating to be capable of producing 100 meters. 20m of planarized substrate were processed with standard buffer deposition process and metal organic chemical vapor deposition (MOCVD) manufacturing run. An in-plane texture of eight to nine degrees was obtained on the entire 20m piece after the final buffer LMO process. A uniform critical current (I_c) of 160A was achieved on 15m. The planarization process has the potential to lead to a reduction in buffer layers and alleviate the burden of hazardous waste generated from the electropolishing process. It is capable of for planarization of any substrate alloy, while electropolishing is limited to only a few alloys.

Index Terms— Epitaxial growth, planarization, Superconducting epitaxial layers, Surface treatment, polishing

I. INTRODUCTION

VER the years, the development of commercial viable REBCO (RE = rare earth) superconductors has led to progressively higher currents and longer lengths by utilizing flexible metal substrates and thin film oxide templates and superconductive layers. In order for the superconductor layer to carry a high current, it is desired that a REBCO with low-angle grain boundaries can be formed. Such a structure of REBCO requires buffers and substrates with low-angle grain boundaries. Therefore, researchers had developed ion beam assisted deposition (IBAD) and rolling assisted biaxially textured substrate (RABiTS) technologies to enable the grains of the buffer to be aligned within a few degrees on the metal substrates and the epitaxial growth of

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REBCO grains has been achieved[1-4]. In the RABiTS approach, the texture of the substrate is achieved directly on the metal through cold work and heat treatment [5]. In the IBAD technology, the texture of the template is achieved through ion beam assist during physical vapor deposition. It has been determined that the roughness of the metal substrate should be less than 1nm in order for the crystals of the template to be aligned within a few degrees [1-4].

Over the years, SuperPower, Inc., had collaborated with Los Laboratory National and had developed electropolishing of metal substrate for IBAD process. The electropolished metal substrates had met with the stringent requirements of IBAD texture growth condition and enabled SuperPower to achieve world-record performance in HTS tapes. However, electropolishing imposes a burden since it employs a mixture of sulfuric and phosphoric acids that requiring appropriate handling. Electropolishing is also very selective to alloy candidates. The stringent 1nm roughness requirement by IBAD is difficult to achieve electropolishing in several alloys of interest.

Due to the limitation of electropolishing, new surface finishing technology is preferred for engineering the desired metal alloys to meet the specifications required by IBAD process. One of the promising technologies is called planarization which uses a solution containing hydrolysable metal compound to form a glassy layer of oxide on unpolished alloy substrate surface through dip coating and heat treatment. This process has the potential to reduce the surface roughness of metal substrate as electropolishing and to meet IBAD buffer deposition requirements. It is also possible for the smooth amorphous oxide film thus formed on unpolished metal substrate to act as the diffusion barrier layer. This can replace the barrier layer that is currently deposited by vacuum process which can result in reduction of number of layers in the buffer stack and thus simplifies HTS coated conductor structure.

A loop planarization process was established at Los Alamos National Laboratory (LANL) to planarize a few meters of unpolished metal substrate with multi-layered oxide films. They had demonstrated that a surface roughness less than 1nm by 18 layers of solution deposition planarization on unpolished metal surface with starting substrate roughness of 28nm[5]. LANL had also reported good in-plane texture on IBAD MgO and high Jc of YBCO on the planarized substrate. In a small scale, they able to achieve an in-plane texture of 4.6 degrees in LMO and a Jc of 2.9MA/cm² of REBCO on planarized substrates.

Through collaboration with Los Alamos National laboratory and University of Houston, SuperPower has extended the smooth surface of planarized metal substrate to longer length and has achieved uniform texture of buffer and high Ic of REBCO in SuperPower coated conductor processes.

II. EXPERIMENTAL

A planarization rig was built at SuperPower with capability of processing at least 100m substrate tapes to expend LANL's 5m loop capacity. As received Hastelloy C-276 was used as the target for planarization with solutions made from dissolving organic yttrium, aluminum and other metal precursors into solvent. Additives were used to adjust the viscosity of the solution for the thickness and smoothness desired. The metal substrate was cleaned in a reel-to-reel mode before they were used for planarization.

In the planarization process, a thin solution layer which contains hydrolysable metal compound obtained through a hydrolysis process is applied to unpolished metal surface. As the solution coated metal substrate moves through the coating head, excess solution is blown off by uniformly distributed air across the width of metal tape. The solution coated metal tape is then entered into a heater at 475 - 525C for heat treatment. The tape can be coated repeatedly by moving back and forth or through a series coating bath and heaters and therefore multi layer coatings can be achieved. The planarized substrate was characterized with optical microscope, atomic force microscope (AFM) and scanning electron microscope (SEM) for surface morphology. These substrates were then processed with standard electropolished Hastelloy C-276 tapes through IBAD MgO, homoepi-MgO and LMO buffer processes. The in-plane texture of LMO on planarized substrates was measured by a Bruker Area Diffraction Detector System (GADDS) for short samples. The in-plane texture of the long tapes was measured in reel-to-reel custom built X-ray system. REBCO was then deposited on the LMO buffered planarized tapes by an MOCVD processes. The tapes were sputtered with silver for current contact and oxygenated. The transport Ic of the REBCO tape was measured over the full width of 12mm using four probe method with in 1 µV/cm criteria.

III. RESULTS AND DISCUSSION

The SEM surface morphology of planarized substrates is shown in Figure 1 through 4. The surface smoothness is seen to be improved from 1 layer to 2 layers, 3 layers and 4 layers of Y₂O₃ coatings. It is observed in the planarization process that the liquid/solvent in the metal compound is evaporated and the film becomes denser and denser as it moves out off the coating bath. A smooth surface is maintained by the surface tension of the solution before, during and after the heat treatment. The peaks of the metal substrate surface are smoothened out and the valleys of the metal substrate surface are filled by layers of the metal oxide compound film. Such smoothening effect is illustrated in Figure 5 in AFM scan areas of $20x20\mu m^2$, $5x5\mu m^2$ and $1x1\mu m^2$. The surface roughness of the substrate decreases with more layers of coatings applied. By adjusting the solution composition and process modification, a substrate surface roughness comparable to that obtained with electropolishing has been achieved by planarization. These results are shown in Table I.

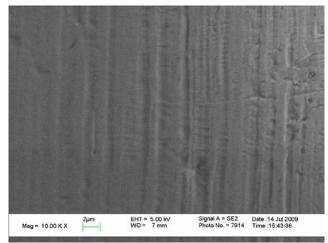


Fig. 1. SEM morphology of 1 layer Y₂O₃ planarized Hastelloy substrate.

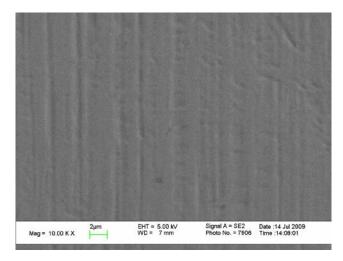


Fig. 2. SEM morphology of 2 layers Y₂O₃ planarized Hastelloy substrate.

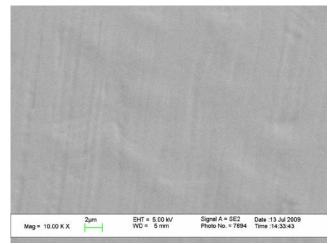


Fig. 3. SEM morphology of 3 layers Y₂O₃ planarized Hastelloy substrate.

Table I summarizes the surface characteristics of asreceived Hastelloy, electropolished and 4 layers Y_2O_3 planarized substrates in scan areas of $20x20\mu m^2$ and $5x5\mu m^2$. These results indicate that the surface quality comparable to electropolished Hastelloy can be obtained using Y_2O_3

planarization process with the same unpolished substrate material.

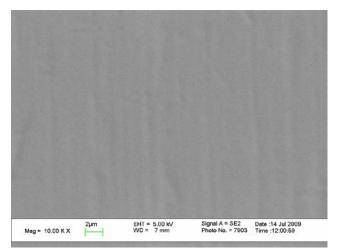


Fig. 4. SEM morphology of 4 layers Y₂O₃ planarized Hastelloy substrate.

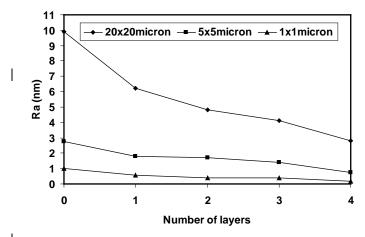


Fig. 5. Surface roughness of Y_2O_3 planarized Hastelloy substrate vs. number of Y_2O_3 layers.

TABLE I AFM CHARACTERISTICS OF SURFACE ROUGHNESS, PEAK TO VALLEY AND SLOPE OF AS RECEIVED, ELECTROPOLISHED AND 4 LAYERS Y₂O₃ PLANARIZED HASTELLOY SUBSTRATE.

Surface characteristics		As received	Electro polished	Planarized 4Layers Y ₂ O ₃
$20x20\mu m^{2}$	Ra (nm)	9.8	2.6	2.2
scan	PV(nm)	41	16	14
	Slope (°)	6	2	0.3
5x5	Ra (nm)	2.5	0.5	0.6
μm² scan	PV(nm)	23	1.2	2
	Slope (°)	5	0.8	0.3

It is understandable that more layers of amorphous coatings are needed if rougher unpolished substrate is used. To meet IBAD deposition surface requirement, the number of layers needed for the planarized surface is a function of starting substrate roughness, solution shrinkage rate and surface tension of the metal compound solution.

Short planarized substrate samples were deposited directly with IBAD MgO, homepi-MgO and LMO. An in-plane texture of 6.4 degrees was achieved on the LMO buffer on planarized metal substrate as shown in Figure 6. A short sample of REBCO was deposited with an MOCVD process.

An I_c of 140A was achieved on $0.4\mu m$ thick film corresponding to a J_c of $3.5 MA/cm^2$. Figure 7 shows that the in-field performance of HTS sample on planarized substrate and is found to be comparable to that of a sample on standard electropolished metal substrate.

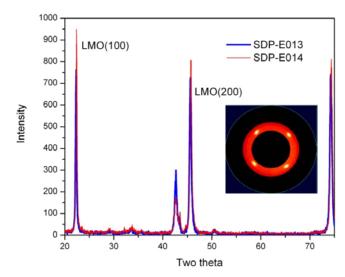


Fig. 6. In-plane texture of LMO on four layers Y_2O_3 _planarized Hastelloy substrate.

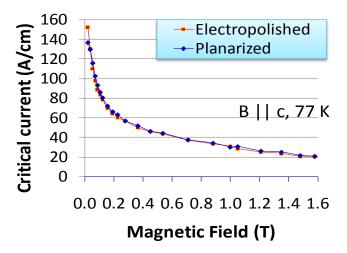


Fig. 7. In-field Ic of HTS tape on planarized substrate compared with standard electropolished substrate.

Long length Y_2O_3 planarized substrate had been process with direct IBAD through LMO processes and REBCO deposition. The LMO texture is shown in Figure 8 in with texture between 8 and 9 degrees measured by reel-to-reel XRD system. This buffer tape was processed in a production MOCVD system. Its I_c profile was measured in 5 m interval through transport tests and the result is shown in Figure 9. A uniform I_c of 160A is achieved on 15m of the 20m with minimum I_c of 125A.

IV. CONCLUSIONS

The substrate planarization process has been scaled up from short lengths to 20 m. It has been shown that good buffer texture and high Jc of REBCO can be achieved on both short sample and long length amorphous oxide planarized Hastelloy

substrate. Our work shows that planarization can be used as an alternative process for metal electropolishing process in HTS conductor manufacturing process. It has the capability to eliminate electropolishing and diffusion barrier vacuum deposition process in one step.

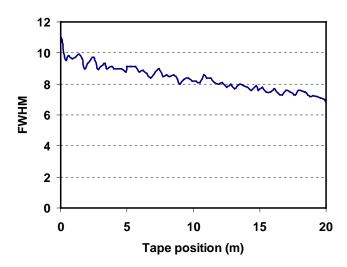


Fig. 8. Texture of LMO along a long length of Y₂O₃ planarized substrate

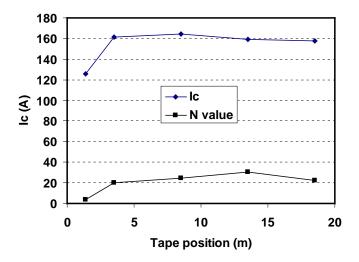


Fig. 9. Ic vs. HTS CC tape position on 20m Y₂O₃ planarized substrate.

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