Sol-gel Synthesis of GdAlO$_3$ Buffer Layers on SrTiO$_3$ (100) Substrates

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Abstract. To identify the possibility of using GdAlO$_3$ (GAO) as a buffer layer for YBa$_2$Cu$_3$O$_{7-\delta}$ (YBCO) coated superconductor wire, we report the result of GAO deposition on SrTiO$_3$ (STO) single crystal substrates by sol-gel processing. Precursor solution was prepared by dissolving stoichiometric quantities of gadolinium hexahydrate and aluminum isoproxide in a mixed binary solvent (2-4 pentanedione:methyl alcohol=1:1). The solutions were spin-coated on STO (100) single crystal substrates and heated at 500°C−1100°C for 2 h in wet Ar-5% H$_2$ atmosphere. A FESEM observation of the surface morphology of the GAO has shown that it has a very smooth surface with a faceted morphology indicating epitaxy. It was shown in x-ray diffraction characterization that epitaxial GAO films with epitaxial orientation relationship of (001)$_{\text{GAO}}$|(100)$_{\text{STO}}$ have been grown on STO (100) substrates.

Introduction

During the last 5 years, greater effort has been placed on the ‘2nd generation’ wire, known as coated conductors based on YBCO [1-3]. YBCO has superior current carrying capabilities in high magnetic fields compared to Bi$_2$Sr$_2$CaCu$_2$O$_8$. Deposition of YBCO on oxide-buffered, rolling-assisted biaxially textured (RABiT) Ni substrates is one promising approach to these issues. High-$J_c$ YBCO cannot be directly deposited onto RABiT substrates due to oxidation and poisoning of the substrates. As a result, a buffer layer of an intermediate material is generally required to preserve the biaxial texture of the substrate for subsequent deposition of YBCO, and to prevent oxidation and diffusion of the substrate material during the high-temperature used during YBCO crystallization and oxygenation anneals. For YBCO-coated conductors, many potential buffer layer materials have been studied, including MgO, La$_2$Zr$_2$O$_7$, BaZrO$_3$, LaAlO$_3$, CeO$_2$, Y$_2$O$_3$-ZrO$_2$, and SrTiO$_3$. Considerations for the candidate buffer layers include lattice matching and chemical compatibility with substrate and YBCO, and development of biaxial orientation (good out-of-plane and in-plane alignment). GAO has an orthorhombic perovskite structure with a close lattice match with YBCO (~3.6%). GAO offers excellent chemical stability and is possibly an excellent diffusion barrier. Films of GAO have been previously grown on R-plane sapphire single crystals via r.f. sputtering [4].

It has been known that sol-gel process represents a low-cost, non-vacuum process, and also presents a convenient way of coating long-length substrates [5,6]. Therefore, sol-gel processing is a very attractive method for the fabrication of superconducting and buffer layers when compared to other vacuum methods. However, no sol-gel processing of GAO buffer layer has been reported yet. Therefore, in this paper, we report the result of GAO deposition on STO (100) single crystal substrates produced by sol-gel processing to identify the possibility of using GAO as a buffer layer for YBCO-coated conductor.
Experimental Procedure

The GAO precursor solution was prepared by dissolving stoichiometric quantities of gadolinium nitrate hexahydrate (STREM) and aluminum isoproxide (STREM) in a mixed binary solvent of 2-4 pentanediene and methyl alcohol with 1:1 volume ratio. The final concentration was adjusted to produce 0.3 M solution and the solutions were stirred with a magnetic stirrer for 2 h. The solutions were filtered through a 0.22 µm filter to eliminate particulates from the solution. Thin films were then prepared via spin coating onto STO (100) single crystal substrates at 3000 rpm for 30 s. Prior to spin coating, the substrates were cleaned by rinsing in acetone and isopropanol for 30 min, heated at 950°C for 30 min and cooled to room temperature in a furnace with a stream of O₂ gas. Heat treatments were carried out between 500°C and 1100°C for 2 h in an environment of Ar-5%H₂ + 0.2~0.5 ml/h of H₂O.

The wet Ar-5%H₂ atmosphere was used because the synthesis of GAO requires either a low partial pressure of oxygen or water vapor in lieu of oxygen. Heat treatment in water vapor will be necessary in the actual process of YBCO-coated superconductor wire because RABiT Ni substrates will not oxidize as readily in water vapor relative to oxygen. Powders were also prepared to determine the parameters needed to synthesis GAO in a water environment.

Phase identification and film orientation was determined by x-ray diffraction (XRD) (Philips Materials Research Diffractometer, Philips, Mahwah, NJ) using θ-2θ scan, ω-scan, and φ-scan. Microstructural characterization of the thin films was performed with a FESEM(6300F, JEOL Ltd., Tokyo, Japan).

Results and Discussion

Studies were initiated by synthesizing powder in an alumina crucible. These studies showed, as expected, that the synthesis of GAO in Ar-5%H₂ forming gas environment required an oxygen partial pressure sufficient to stabilize GAO, but insufficient to oxidize the RABiT Ni substrates. Namely, GAO is not stable in a reducing environment imposed by the reducing gas and but requires the introduction of a small amount of oxygen into the furnace. The oxygen partial pressure required to form GAO, yet avoid the oxidation of Ni, was achieved with the introduction of water vapor. At a given temperature, water vapor partially decomposes to H₂ and O₂ gases. At a particular temperature, which controls the equilibrium constant for this decomposition reaction, the partial pressure of O₂ is controlled by amount of water vapor in the environment, which, in turn, is controlled by the amount of water vapor introduced into the furnace tube. Thus, the oxygen partial pressure within the furnace can be controlled by controlling both the flow rate of water vapor and the temperature. Since Ni requires a high oxygen partial pressure to form NiO, relative to other metals, the oxygen partial pressure needed to synthesize GAO and prevent the formation of NiO could be achieved with modest flow rates of water vapor. Powder studies showed that between 0.2 ml/h and 0.5 ml/h were required to optimize the formation of orthorhombic GAO.

Fig. 1a shows the XRD 0-2θ scan of the GAO powders heated at 1100°C for 2 h in Ar-5%H₂ forming gas with a flow rate of water of 0.5 ml/h, indicating that GAO with orthorhombic perovskite phase is formed. Small GdO peaks detected might be caused by weighing error. The GAO powders heated under Ar-5%H₂ without water vapor (Fig. 1b), however, show poor crystallinity which should be due to the restriction on the removal of the carbon. X-ray diffraction of GAO powders processed between 500°C and 1100°C showed the formation of GAO occurred at approximately 650°C. The degree of GAO formation increased with increasing temperature until approximately 1000°C. XRD peak intensities remained stagnant with further increases in temperature.
Fig. 1. XRD 0-20 scans of the GAO powders heated at 1100°C for 2 h in (a) Ar-5%H₂ + 0.5 ml/h of H₂O and (b) Ar-5%H₂.

Since powder studies have shown that water vapor was required to optimize the formation of orthorhombic GAO, experiments to produce GAO epitaxy on STO (100) single crystal substrates were carried out in the wet forming gas atmosphere. Fig. 2 shows an XRD 0-20 scan of the GAO thin film grown on STO (100) substrate in the Ar-5%H₂ + 0.5 ml/h of H₂O at 1100°C for 2 h. The only GAO peak present is assigned to (002), indicating that the film exhibits c-axis preferred orientation on the STO (100) substrate.

ω-scan of the (002) GAO reflection verifies the good c-axis alignment with full-width-at-half-maximum (FWHM) determined to be 0.37º (Fig. 3a). φ-scans for the {112}GAO and {110}STO planes in Fig. 3b show that the GAO film has good in-plane orientation relationship with respect to the STO substrate. The FWHM values of 1.05º for the {112}GAO and 0.91º {110}STO indicate that the film has a good in-plane alignment.

Fig. 2. XRD 0-20 scan of the GAO thin film grown on STO (100) substrate in the Ar-5%H₂ + 0.5 ml/h at 1100°C for 2 h.

Fig. 3. XRD ω and φ scans of the GAO thin films grown on STO (100) substrate in the Ar-5%H₂ + 0.5 ml/h at 1100°C for 2 h: (a) ω-scan for (002) reflection of GAO, (b) φ-scans for (112) reflection of GAO and (110) reflection of STO.
Fig. 4 shows FESEM micrograph of the GAO film grown on STO (100) substrate in the Ar-5%H₂ forming gas with a flow rate of water of 0.5 ml/h at 1100°C for 2 h. It shows the development of a good epitaxial film with a very smooth and faceted surface morphology.

Conclusions

GAO thin films were prepared by a sol-gel processing. The GAO films on STO (100) single crystal substrates heated at 1100°C for 2 h in the Ar-5%H₂ + 0.5 ml/h showed a c-axis preferred orientation with both good out-of-plane (FWHM = 0.37º for the (002) reflection) and in-plane (FWHM = 1.05º for the {112} reflection) alignment. The results showed that buffer layer architecture for a coated superconductor wire can be potentially simplified using sol-gel processed GAO films.

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