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RESEARCH OF SURFACE-OXIDATION EPITAXY OF NiO FILMS ON CUBE TEXTURED Ni TAPES

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ABSTRACT

Thin films of NiO were fabricated in air by surface-oxidation epitaxy (SOE) on biaxially-textured Ni substrate, for use as buffer layers for YBCO coated conductors. The surface-oxidation conditions such as the temperature and the reaction time, as well as the cooling rate, play a determining role in the process of epitaxial growth of the NiO layer. A continuous, biaxially-aligned textured NiO layer, was obtained for a 10 min oxidation in air at 1120° C, and a cooling rate of 150 K/h. The roughness of the NiO layer is mostly controlled by a coarsening of some NiO grains.

1. INTRODUCTION

The deposition of YBCO films on biaxially-textured metallic substrate is a promising technology for the fabrication of high field superconducting tapes. Currently there are several approaches to achieve this goal. One of these is the use of biaxially-textured ribbon substrates obtained by rolling and re-crystallization, upon which one or several buffer layers are deposited before the deposition of YBCO film. This process is called deposition on rolling-assisted biaxially textured substrate (RABiTS) [1]. Usually the substrate is a cubic metal, such as Ni, in which a strong {100}<001> cube-texture is developed by cold rolling, followed by a re-crystallization process. Under certain conditions, the buffer layer/layers deposited on such a highly textured substrate can grow epitaxially, and the subsequent deposition of the YBCO film may result not only in a highly c-axis oriented film, but in a high degree of alignment of the (a,b) plane as well.

Critical current densities in excess of 10^6 Acm⁻² were obtained for short length (centimetres) of composite YBCO/YSZ/CeO₂/Ni conductors, at 77K and zero applied magnetic field [2]. The drawbacks of this approach are that the CeO₂ is subject to cracking, which degrades the effect of the buffer layer, and that the both of CeO₂ and YSZ have to be deposited using PLD, same as the YBCO, which makes the process sequential and lengthy.

Recently a new approach has been reported [3,4,5], which uses a scalable method to produce in-plane aligned NiO buffer layers on biaxially-textured Ni substrates, suitable for YBCO deposition. The characteristic of this method is that a cube-textured NiO layer is made by controlled surface oxidation (SOE) of a cube-textured nickel or nickel alloy ribbon, in an electric furnace. A NiO buffer layer is suitable for YBCO film growth. It has a FCC unit cell with a lattice parameter of 4.17 Å, similar to the lattice parameter of MgO, which is a well-known substrate for YBCO film growth. Also, a NiO layer provides a suitable barrier for the protection of the YBCO film from adverse chemical interactions with the nickel substrate.

It has been reported that the critical current density of an YBCO composite tape produced by pulsed laser deposition (PLD) of YBCO film on a cube-textured NiO, previously fabricated by SOE of a cube-textured Ni, was between $4 \times 10^4 \text{ Acm}^{-2}$ and $6 \times 10^4 \text{ Acm}^{-2}$ (at 77K, and 0T) [6]. This value is approximately two orders of magnitude lower than the critical current density of an YBCO/CeO₂/Ni tape where the CeO₂ buffer layer is produced by PLD using ion beam assisted deposition (IBAD) on randomly-oriented Ni, or by PLD on RABiTS Ni [1,7].

A possible explanation for this difference in J_c is the existence of macroscopic defects in the NiO film. The growth of the NiO film through an oxidation process is difficult to control compared to PLD or other vapour deposition techniques such as magnetron sputtering, and as a consequence, their surface roughness is relatively high. This increased roughness is reflected in the two-fold increase of the full width at half maximum (FWHM) of X-ray rocking curve of NiO films, as compared to CeO₂ film deposited by IBAD and PLD or by RABiTS and PLD [8].

Although the growth of NiO by SOE is an appealing option, the above-mentioned problems indicate the importance of improving the quality of the NiO buffer layer. In this paper we report results on the quality of NiO film obtained by SOE.

2. EXPERIMENTAL

All of the NiO film samples used in this investigation were grown in air on {100}<001> cube-textured Ni substrate. The Ni substrate was 0.1mm thick, fabricated by a standard procedure, using a large reduction cold rolling and re-crystallization method [1].

The average roughness of the as rolled Ni substrates determined by atomic force microscopy (AFM) on $(40 \times 40) \mu\text{m}^2$ scans was 480nm Rms. This value was reduced by electrolyte polishing, down to 24nm, which is comparable with the average roughness of a polished ceramic substrate.

The oxidation of the Ni substrates was carried out in an electric furnace in air. The oxidation temperatures ranged from 900°C to 1200°C, for between 10 s and 90 min. In order to prevent damage to the NiO film by thermal shock, the reaction time was controlled by flushing the furnace with Ar, and allowing the samples to cool down to room temperature inside the furnace.

3. RESULTS AND DISCUSSION

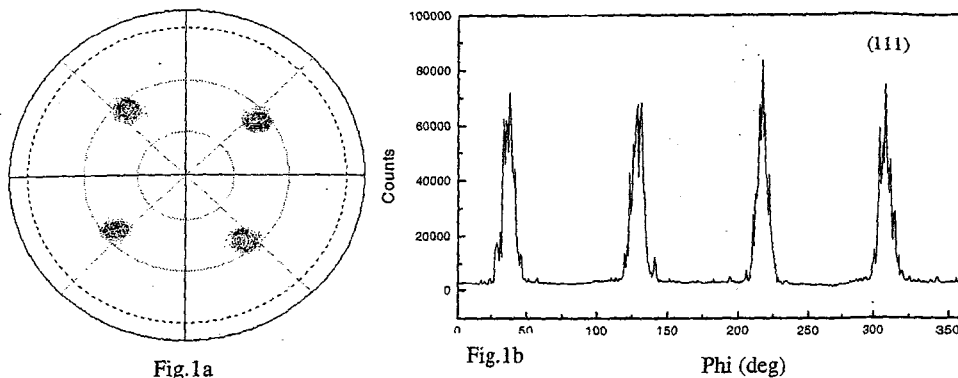


FIGURE 1a. Pole figure of (111) plane of cubic textured Ni sheet. FIGURE 1b. ϕ -scan for (111) plane of Ni substrate, FWHM is 8° .

In FIGURE 1a is presented the (111) pole figure for a typical Ni substrate, which shows a sharp $\{100\}\langle 001\rangle$ cube-texture. For the same sample, a x-ray ϕ -scan was obtained for (111) crystallographic plane, shown in FIGURE 1b. All four (111) equivalent reflection peaks have approximately the same intensity, with a $FWHM_{(111)} = 8^\circ$. The results presented in FIG. 1a and FIG. 1b, are indicative of a sharp in-plane alignment of the crystallographic axes of the Ni substrates. A similar (111) pole figure and ϕ -scan was performed on a Ni substrate after electrolyte polishing, and the results indicated that the cube-texture was not affected by this process.

The texture of the NiO layer grown on the electrolyte polished Ni substrate is strongly influenced by the oxidation temperature and the oxidation time. In FIGURE 2a it is presented the dependence of the c-axis orientation on the oxidation temperature, for a 10 min reaction time, assessed by θ - 2θ X-ray scans. It is evident that between 1110°C and 1130°C , there is a minimum of the NiO (111) reflection. This minimum is located between 1115°C and 1125°C .

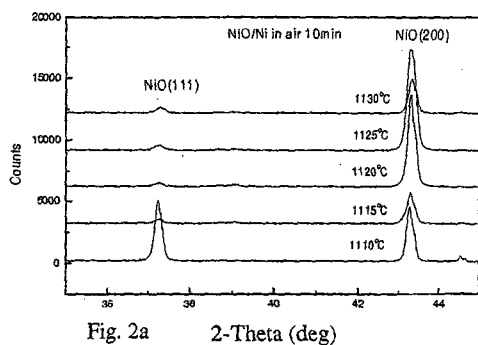


Fig. 2a 2-Theta (deg)

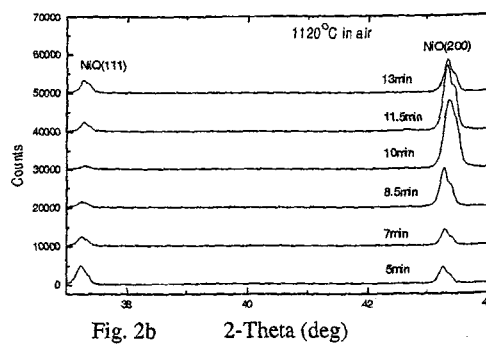


Fig. 2b 2-Theta (deg)

FIGURE 2a. X-ray θ - 2θ scan for NiO/Ni at different oxidation temperature and for oxidation time 10min. FIGURE 2b. X-ray θ - 2θ scan for NiO/Ni at oxidation temperature 1120.0°C and for different oxidation time

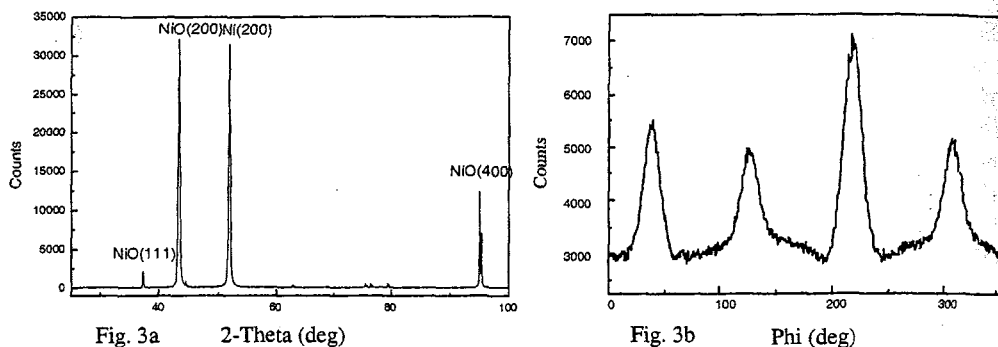


FIGURE 3a. X-ray θ - 2θ scan for NiO/Ni, $I_c=I(200)/(I(200)+I(111))=95.2\%$.

FIGURE 3b. Phi-scan for (111) plane of NiO film, FWHM is 20° .

In FIGURE 2b is presented the dependence of the c-axis orientation on the oxidation time, at 1120°C , assessed by θ - 2θ X-ray scans. This result indicates that between 10sec and reaction time, there is a maxima in the intensities of both (111) and (200) reflections of the NiO layer, located around a reaction time of 10 min.

The X-ray θ - 2θ scan for NiO/Ni sample obtained at 1120°C for a reaction time of 10 min, shown in FIGURE 3a, reveals that the NiO layer has a strong c-axis orientation 15 min. Relative to the broad face of the Ni substrate. A quantitative assessment of the c-axis orientation can be defined by the intensity ratio of (200) and (111) reflections of NiO layer, $I_{(200)}$ and $I_{(111)}$ respectively. For the sample scan presented in FIGURE 3a, this ratio $I_{(200)}/[I_{(200)}+I_{(111)}]=95.2\%$, which represents indeed a strong c-axis orientation.

The in-plane orientation of the NiO layer obtained at a temperature of 1120°C for 10 min was investigated using X-ray ϕ -scan of the (111) reflection, and the result is shown in FIGURE 3b. A quantitative measure of the in-plane orientation is given by the FWHM of the ϕ -scan of the (111) reflection, which is 20° for this sample. It was concluded that the best texture of a NiO buffer layer grown on cube-textured Ni in air, can be obtained at 1120°C for 10 min, and it has a strong c-axis texture and a partial in-plane texture.

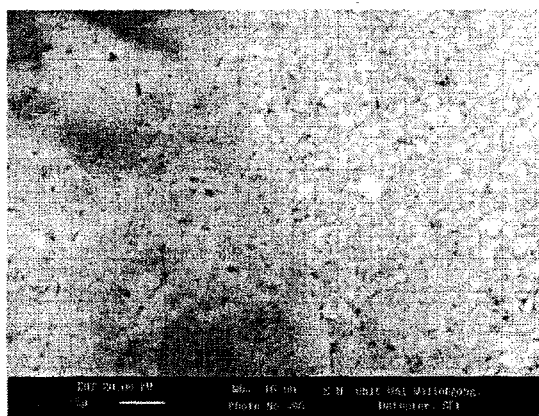


FIGURE 4. SEM image of NiO film on Ni. There are two different parts of film.

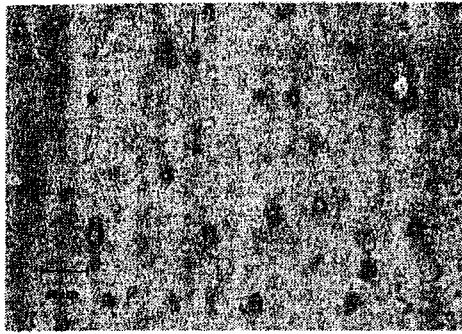


Fig.5

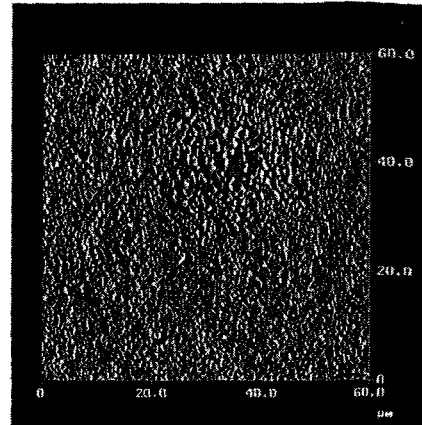


Fig.6

FIGURE 5. Optical microscope image of NiO film. There are a lot of spots.

FIGURE 6. AFM deflection scan image of NiO film. It is shown the spot is a group of large size grains

Apart from the crystallographic orientation of the buffer layer, other properties such as surface roughness, cracks and continuous coverage of the substrate are also important in preserving the properties of the YBCO layer. In the case of a fast cooling rate from the oxidation reaction temperature, cracks often appear in the NiO film. For very fast cooling rates, the NiO layer may exfoliate from the Ni substrate. Such a catastrophic breakdown of the NiO layer takes place when the cooling rate is higher than approximately 100K/min.

In FIGURE 4 is presented a typical SEM image of NiO surface, viewed along the normal to the broad face of the substrate, for a sample cooled at such fast cooling rates. Two distinctive parts of the NiO layer are clearly identifiable in this image: on the left-hand side there is a smooth and continuous area, and on the right-hand side there is a rough area, which appears to be formed by fractured grains. A fractured area of the NiO layer loses its crystallographic orientation relative to the Ni substrate, and it is very likely that a continuous YBCO film cannot be deposited on such a surface.

In our work the oxidation time was controlled by introducing Ar in the furnace, which makes the reaction time independent of the time the sample was kept in the furnace. A continuous, non-fractured NiO layer was obtained for a sample-cooling rate of approximately 150° K/h.

The surface roughness of a continuous NiO layer was examined by optical microscopy and AFM. The optical microscopy image shown in FIGURE 5 suggests the presence of discontinuities on the surface of the NiO layer, of approximately 10µm in diameter. A more accurate examination around these discontinuities was carried out by AFM on a (40x40) µm² scan, and the result is presented in FIGURE 6. It is evident that the discontinuities represent clusters of larger than average grains of NiO. This grain coarsening takes place not only in the (x,y) plane, which corresponds to the (a,b) crystallographic plane of NiO layer, but in the z-direction as well, leading to an increase in the average roughness of the NiO layer to 240nm Rms, as compared to the electrolyte polished Ni substrate. The grain coarsening may be caused by a localised tension in the NiO layer, originating from the difference in volume of the NiO and Ni unit cells.

4. CONCLUSIONS

A continuous and epitaxial NiO layer can be grown on a cube-textured Ni substrate by oxidation in air at 1120° C for 10 min, and a cooling rate of the sample of approximately 150K/h. For larger cooling rates, the NiO layer may be discontinuous, with large areas of the film fractured into small grains which are not attached to the Ni substrate. The increase roughness of the continuous NiO layer is caused by clusters of relatively large NiO grains, which may in turn be a cause for the lower J_c of YBCO films deposited directly onto NiO buffer layers.

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