Proceedings of the ICSC-F'96 Jaszowiec '96

GROWTH AND STRUCTURE OF BUFFER LAYERS FOR HIGH TEMPERATURE SUPERCONDUCTING FILMS

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We have studied thin CeO₂ buffer layers prepared by aerosol MOCVD on (1102) Al₂O₃ substrate at high deposition temperature, $T_d = 900^{\circ}$ C. A texture analysis by X-ray diffraction showed a high degree of epitaxial character of CeO₂ films. A study of the microstructure by transmission electron microscopy revealed that the CeO₂ films are in a relaxed state being composed of slightly misoriented blocks surrounded by dislocations. The films are smooth, giving mean square root values of the surface roughness measured by atomic force microscopy up to 1 nm.

PACS numbers: 68.55.Jk

1. Introduction

An intermediate buffer layer is necessary for the growth of high temperature superconducting (HTSC) thin films on technical substrates. R-plane sapphire ((1102) oriented) with CeO₂ buffer layer seems to be an appropriate substrate for the growth of a whole family of HTSC thin films. An epitaxial character and smooth surface of the buffer layer are essential for the subsequent growth of HTSC films with a high critical current density. As an important lattice mismatch between the CeO₂ film and the sapphire substrate (5.7% and 13.7% along the [1101] and [1210] directions, respectively) can give rise to structural imperfections, a careful control of the buffer layer microstructure is necessary. In this paper we present microstructure and surface characterization of (001) oriented CeO₂ films grown on the (1102) Al₂O₃ substrate by the aerosol metal organic chemical vapor deposition (MOCVD) technique.

2. Sample preparation and characterization

CeO₂ films were prepared by the low pressure aerosol MOCVD process, described in details elsewhere [1]. As a precursor we have used heptafluorodimethyl octanodionate of Ce, Ce(fod)₃. Films with a thickness of 20 nm were deposited at the deposition temperature $T_d = 900^{\circ}$ C. The films were then characterized by a standard $\theta - 2\theta$ X-ray diffraction scans. The character of epitaxy of the films was studied by X-ray diffraction using the four circle Philips texture goniometer. A microstructure of the films was investigated by a transmission electron microscopy (TEM) using the JEOL JEM 1200 EX electron microscope and a plane view technique. The atomic force microscopy (AFM) of the films surface was performed using the nanoscope III atomic force microscope in a tapping mode.

3. Results and discussion

The standard θ -2 θ X-ray diffraction reveals that CeO₂ films grow on R-plane cut Al₂O₃ substrate with the preferred orientation CeO₂ (001) || Al₂O₃ (1102). Some films contained a small amount of (111) oriented CeO₂ grains in the (001) oriented CeO₂ matrix. The growth of the (111) oriented grains can be stimulated by the substrate surface imperfections [2] or by instabilities of the deposition process. As it was observed by TEM a mean diameter of 40 nm is typical of the (111) oriented grains. An X-ray diffraction texture analysis of the (001) orient matrix revealed a high degree of the preferred orientation with FWHM value of the 002 reflection ω -scan about 0.3°. The CeO₂ films are in-plane aligned, as can be deduced from the fourfold modulated φ -scan of the 111 CeO₂ reflection, Fig. 1. The FWHM of the φ -scan peak was equal to 1.4°, showing an excellent in-plane alignment of the deposited CeO₂ film.

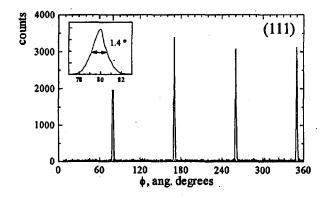


Fig. 1. φ -scan of the (111) CeO₂ X-ray reflection.

Figure 2 shows a plane view TEM image of the CeO₂ layer near the CeO₂/ Al_2O_3 interface and its corresponding electron diffraction pattern. According to the diffraction pattern the layer is epitaxially grown with orientation CeO₂ (001) [100] || Al_2O_3 (0112) [2110]. The electron diffraction exhibits a double diffraction effect and indicates that the CeO₂ film is relaxed.



Fig. 2. TEM micrograph of the CeO_2 film near the CeO_2/Al_2O_3 interface. Selected area diffraction pattern exhibits a double diffraction effect.

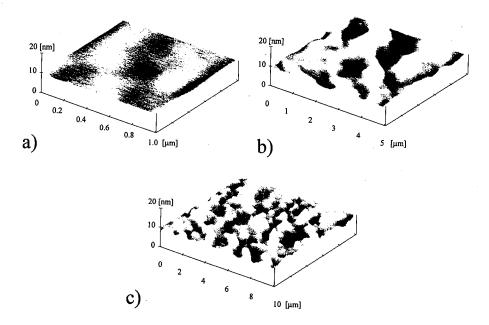


Fig. 3. AFM images of the CeO₂ film with an area of (a) $1 \times 1 \ \mu m^2$, (b) $5 \times 5 \ \mu m^2$, (c) $10 \times 10 \ \mu m^2$.

The epitaxial character of CeO₂ layer is visualized also in TEM image by Moiré fringes which are an image interpretation of double diffraction in TEM (Fig. 2). The (001) oriented CeO₂ layer consists of small blocks with the diameter of about 100 nm, which are slightly rotated each to others around the film surface normal. The Moiré fringes are parallel in the individual blocks and the mutual rotation of blocks is reflected by the magnified rotation of Moiré fringes between the blocks. The mutual misorientation of the blocks deduced from the rotation of Moiré fringes gives values corresponding to the FWHM of 111 φ -scan. The misorientation between blocks is a result of the substrate-film mismatch accommodation and is accompanied by a generation of dislocations parallel to the film surface normal. The (001) oriented matrix contains (111) oriented CeO₂ grains with the mean diameter of 40 nm.

The surface quality of the CeO₂ films was examined by the atomic force microscopy. A set of AFM images with an area from 1 to 100 μ m² is shown in Fig. 3. The surface of the film is smooth, giving mean square root values of the roughness $R_q = 0.27, 0.83$, and 0.95 for the 1, 5 × 5, and 10 × 10 μ m² area, respectively. The value for the 1 μ m² represents the mean value of 5 measurements performed at different places of the sample. The low value of R_q for the 1 μ m² area shows that at these regions the film is atomically smooth.

4. Summary

We have prepared epitaxial CeO₂ films by aerosol MOCVD method on $(1\overline{1}02)$ Al₂O₃ substrate. The films were grown at a high deposition temperature, $T_d = 900^{\circ}$ C. The films exhibit a high degree of epitaxy, as shown by the X-ray diffraction texture analysis. TEM observations reveal that the CeO₂ films consist of small (about 100 nm diameter) slightly misoriented blocks. We suggest that such a microstructure is generated due to the lattice mismatch strain between the film and the substrate. Double electron diffraction indicates relaxation of the CeO₂ layer. The surface of the CeO₂ films is smooth with R_q up to 1 nm. The CeO₂ films prepared by aerosol MOCVD at a high deposition temperature are suitable as an intermediate buffer layer for the growth of epitaxial high- T_c superconducting films.

This work was supported by the Slovak Scientific Grant Agency (grants VEGA 2/1087/95, 95/5305/107).

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