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IN-SITU GROWTH AND STRUCTURE OF HIGH-T_c SUPERCONDUCTING THIN FILMS

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Abstract Growing manner of $YBa_2Cu_3O_{7-x}$ (YBCO) thin films on $SrTiO_3$(100) and MgO(100) by reactive evaporation method was investigated by means of in-situ reflection high energy electron diffraction (RHEED) and X-ray diffraction. In-situ RHEED observation showed that the formation of perovskite structure occurred even for initially deposited two atomic planes and the YBCO crystal grew in the layer-by-layer manner. It was demonstrated by RHEED patterns that the in-plane lattice of YBCO on seriously lattice mismatched MgO was initially strained for the matching with MgO and changed abruptly to the lattice parameter of the tetragonal YBCO bulk with increasing thickness. X-ray analysis of the 100Å thick superconducting film on $SrTiO_3$(100) revealed no orthorhombic distortion and the in-plane lattice parameter was close to that of $SrTiO_3$. On the other hand, the 100Å superconducting film on MgO was found to have orthorhombic symmetry.

INTRODUCTION

High quality thin films of high-T_c oxides have an important role in the study of the fundamental physics and device applications. The recent trend in the preparation of high-T_c films has been toward "in-situ" growth of the superconducting phase at relatively low temperatures. The purpose of "in-situ" growth is to attain surface smoothness suitable for fabricating film devices but also to obtain high quality film. We have

grown in-situ high quality superconducting $YBa_2Cu_3O_{7-x}$ (YBCO) thin films by activated reactive evaporation (ARE).[1,2] The investigation of the initial stage of the epitaxial growth and the growth manner in the in-situ growth gives essential information for us to improve the thin film fabrication technique. In-situ RHEED observation is one of the most effective means for the investigation of growth manner. Besides, the X-ray study of the crystal structure of the ultra-thin film (100Å) would give information about the effect of the substrate lattice on the growing film. $SrTiO_3$ has a lattice mismatch of less than 2% with respect to the tetragonal YBCO. MgO has a large mismatch of 9%. The effects of the magnitude of mismatch on the initial growth manner and on the crystal structure of the resultant film were investigated. Generally, an epitaxial film can be stabilized by accommodating the mismatch with the substrate by elastic strain up to a critical layer thickness "h_c". Over h_c, the in-plane lattice spacing of an epitaxial film changes rapidly to its bulk value by the formation of misfit dislocations. In ARE, oxygen is provided to a film during deposition and cooling. We have generated oxygen plasma by RF discharge during deposition. The effect of oxygen plasma on the formation of the YBCO crystal was also investigated by in-situ RHEED observation.

EXPERIMENTAL

The detail of the ARE method was described elsewhere.[1,2] We have sputtered the surface of the substrate by Ar^+ ion beam bombardment at 650℃ for 1 min, followed by deposition of YBCO on the substrate at the same temperature.

X-ray diffraction measurements were performed using a conventional double-axis diffractometer. CuKα radiation (power of 55kV × 250mA) monochromated by a

pyrolytic graphite crystal was employed.

RHEED OBSERVATION[3]

Figure 1 shows in-situ RHEED patterns of a growing YBCO film on $SrTiO_3$(100), where the electron beam is parallel to the [100] direction of $SrTiO_3$. The pattern at the initial growing stage of 3Å is the sharp streaks and is same as that of the substrate. The sharp streaks at the deposition of one or two atomic planes (3Å) suggest that the initial deposition of YBCO occurs in the monolayer overgrowth mode without formation of three dimensional nuclei. The sharp streaks observed at every successive stage reveal that the film surface is atomically smooth and the growth manner is layer by layer.

Figure 2 shows the RHEED patterns of a YBCO crystal deposited on $SrTiO_3$(100) without RF plasma activation. All the patterns exhibited the streaks characteristic of

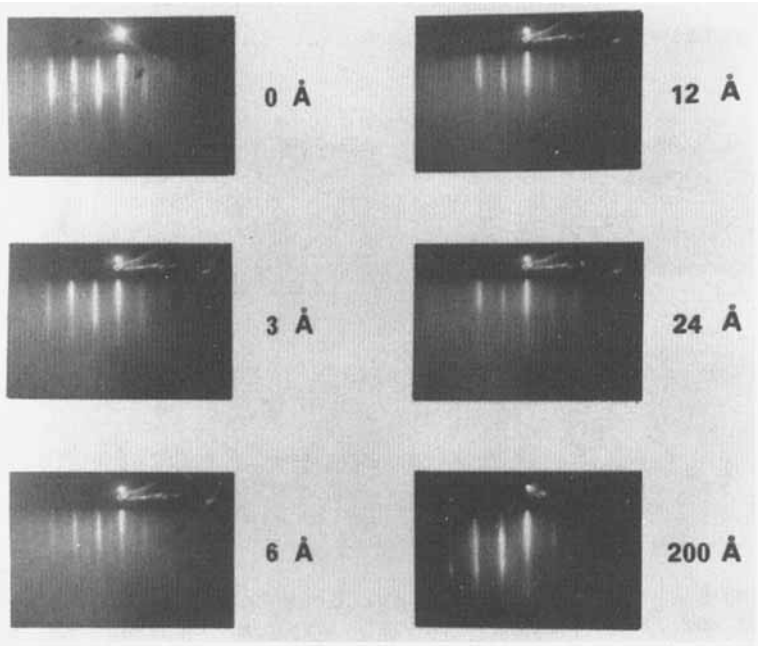

FIGURE 1 In-situ RHEED patterns observed during growth of the YBCO film on a $SrTiO_3$(100).

YBCO. The spotty patterns coming from (110) of Cu_2O appeared in the deposits above 36Å thick. The deposition of Cu_2O must have originated from insufficient oxidation of Cu to Cu^{2+}, because the formation of a YBCO crystal requires the presence of an amount of Cu^{2+} at least more than 67% in copper ions. When the RF plasma activation was applied in the deposition Cu_2O never deposited. The RHEED observation revealed that oxygen plasma assisted the oxidation of Cu to Cu^{2+} and the formation of high quality YBCO films.

In-situ RHEED patterns have also been observed during the growth of YBCO on MgO(100), as shown in Figure 3. The diffraction pattern of the MgO substrate is distinctly different from that of perovskite structure. Even for the deposition of two atomic layers, there appeared new streaks revealing perovskite structure in addition to that of MgO. The streak pattern of perovskite structure at every deposit revealed that the YBCO crystal grew in the layer-by-layer manner. We

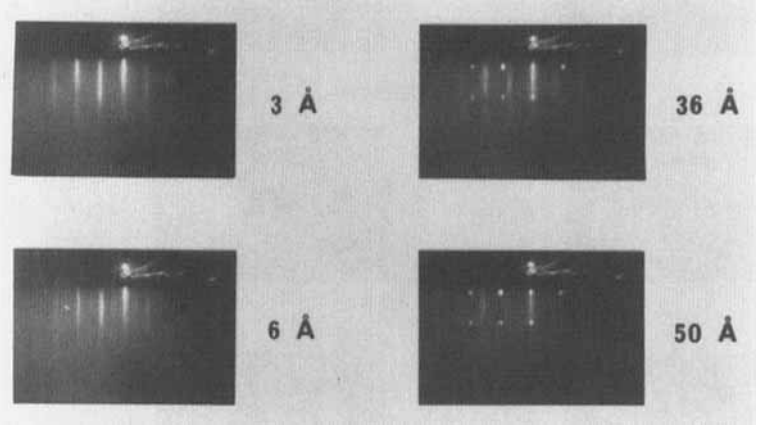

FIGURE 2 In-situ RHEED patterns observed during growth of YBCO film without oxygen plasma on a $SrTiO_3$(100).

estimated the in-plane lattice parameter of YBCO from the spacing between streaks, as shown in Figure 4. The films with thickness from 3Å to 12Å had the same in-plane lattice spacing as MgO. When the thickness became larger than 12Å, the lattice spacing drastically decreased to

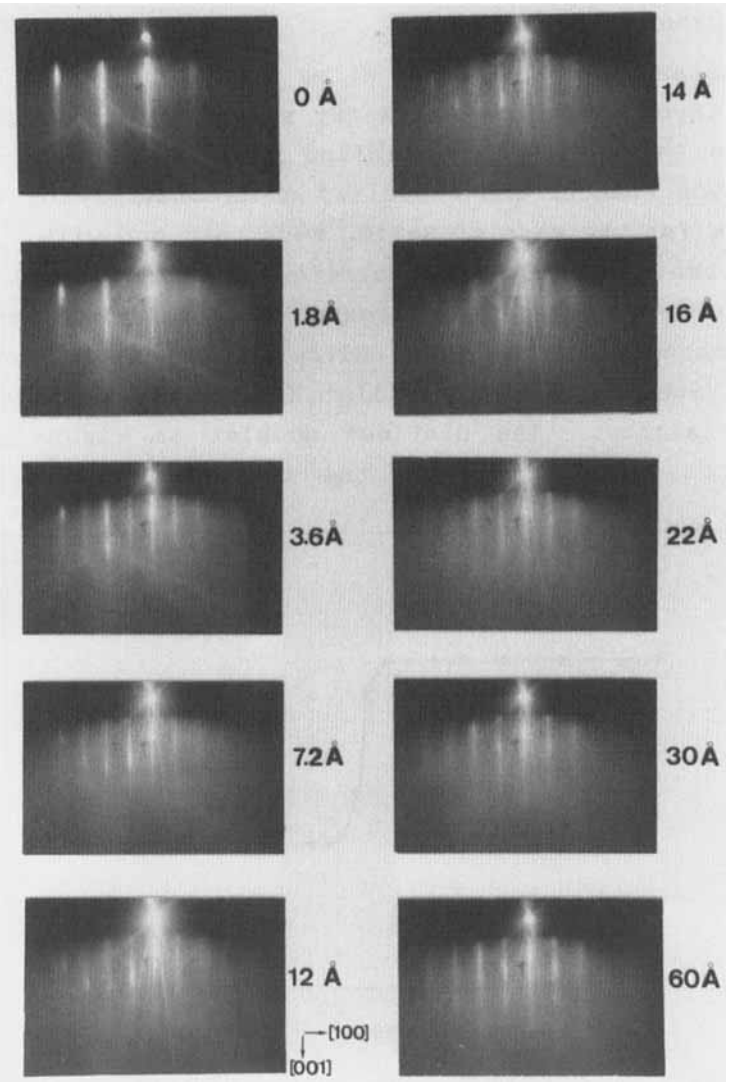

FIGURE 3 In-situ RHEED patterns observed during the growth of the YBCO film on MgO(100).

the bulk value of tetragonal YBCO. The critical thickness h_c of YBCO is about 12Å for the MgO substrate with the large lattice mismatch of 9%. Above h_c, the YBCO film should form misfit dislocations at the interface.

X-RAY DIFFRACTION MEASUREMENT

The structure of 100Å thick films formed on $SrTiO_3$ and MgO was investigated by the X-ray scattering technique.[4] The films were in-situ oxidized below 650°C in the evaporation chamber and exhibited superconductivity with T_c of 80K for the case of $SrTiO_3$ substrate and with T_c of 70K for the case of MgO substrate. Figure 5 shows the X-ray scattering profiles around the (407) reciprocal point scanned along the [100] direction for the films on MgO(100) substrates. The Miller index h is defined by the MgO lattice. The distinct doublet in Figure 5(a) comes from the twinning in the orthorhombic crystal,

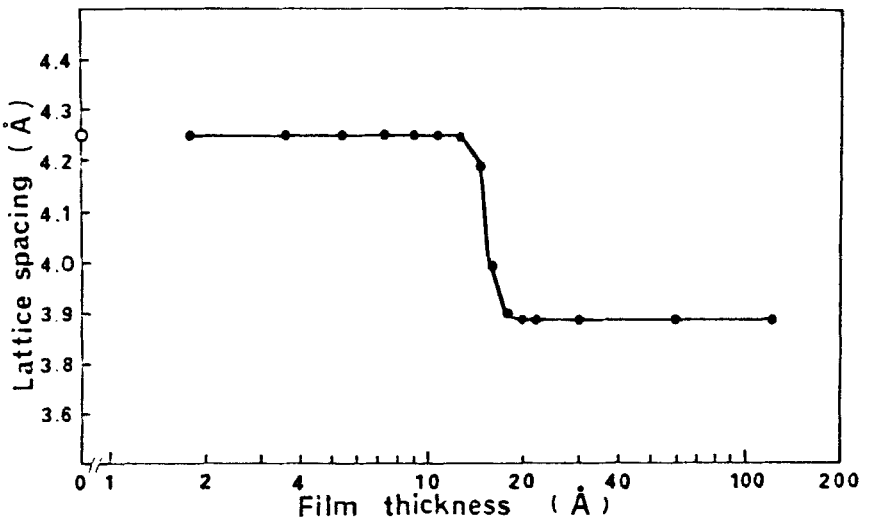

FIGURE 4 Lattice spacing vs thickness of the YBCO film on the MgO(100) calculated from the distance of the streaks.

where the left and the right peaks correspond to the (047) and (407) reflections, respectively. The lattice constants a and b were derived to be 3.827Å and 3.879Å from the peak positions. The peak splitting is also seen in the 100Å thick film in Figure 5(b). From the in-situ RHEED observation the critical thickness h_c of YBCO on MgO was estimated to be 12Å. As the thickness

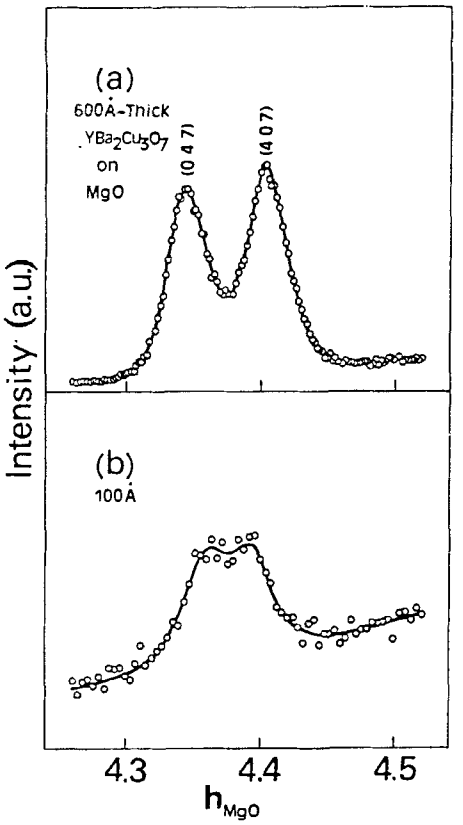

FIGURE 5 X-ray scattering specra for (a) 600Å and (b) 100Å thick superconducting YBCO on MgO(100) scanned along the [100] direction around the (407) reflection.

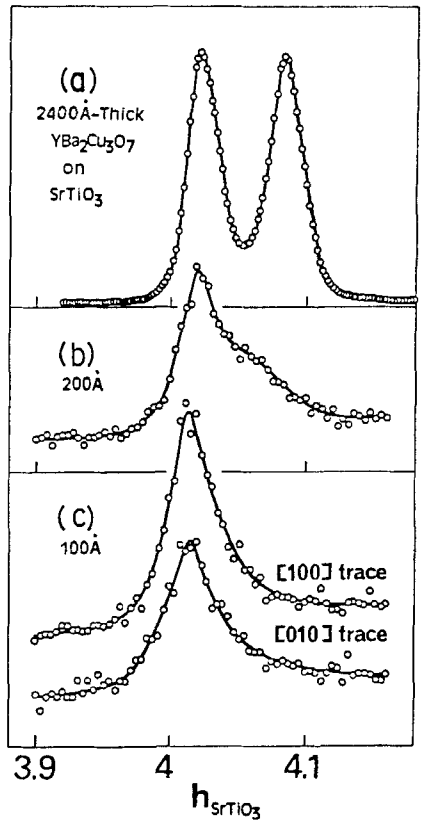

FIGURE 6 X-ray scattering spectra for (a) 2400Å, (b) 200Å thick and (c) 100Å thick superconducting YBCO on $SrTiO_3$ scanned along the [100] direction around the (407) reflection.

is enough larger than h_c, the lattice of the 100Å thick YBCO is assumed to be the unstrained lattice except the strained lattice near the interface.

Figure 6 represents the X-ray scattering profiles around the (407) reciprocal point scanned along the [100] direction for the various thick films on $SrTiO_3$(100). The Miller index h is defined by the $SrTiO_3$ lattice. The spectra in Figure 6(c) show no orthorhombic distortion and the in-plane parameter is close to that of the $SrTiO_3$ lattice. The YBCO lattice epitaxially grown on the $SrTiO_3$ lattice is strained until it becomes about 100Å thick to match the lattice with the substrate; that is, h_c>100Å. The orthorhombic distortion was slightly observed in the X-ray profile for a 200Å thick film, as shown in Figure 6(b). This indicated that the film consisted of the unstrained orthorhombic YBCO lattice for $>h_c$ ($\approx$100Å) and the strained tetragonal lattice for $<h_c$ whose in-plane lattice parameter was close to that of $SrTiO_3$. In Figure 5(b), the YBCO lattice on the substrate with a large mismatch is distorted into the orthorhombic symmetry by oxidation. The presence of misfit dislocations may make it possible to distort the in-plane lattice. On the other hand, the 100Å thick strained YBCO lattice on $SrTiO_3$ could not be distorted in the in-plane by oxidation because of the absence of misfit dislocations, although the film had almost the same lattice parameter c=11.69Å as that of the orthorhombic phase. The resistivity in the film exhibited a metallic temperature dependence and a superconducting transition at about 80K. These facts implied that the orthorhombic distortion was not essential to superconductivity in YBCO.

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