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Rapid deposition of YBCO films by laser CVD and effect of lattice mismatch on their epitaxial growth and critical temperature

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Abstract

a-Axis- and *c*-axis-oriented $YBa_2Cu_3O_{7-\delta}$ (YBCO) films were grown on (100) $SrTiO_3$ substrate by laser chemical vapour deposition (laser CVD). The effect of lattice mismatch between films and substrates on in-plane and out-of-plane crystallinity and critical temperature (T_C) was investigated. The preferred orientation changed from *a*-axis to *c*-axis as the deposition temperature increased from 928 to 1049 K. The *c*-axis-oriented YBCO showed a minimum of full width at half maximum of 0.5° for the ω-scan and 1.0° for the φ-scan. A smaller mismatch between YBCO films and substrates led a higher crystallinity for in-plane and out-of-plane epitaxial growths. A high T_C of 90 K was obtained for the *c*-axis-oriented YBCO films. The deposition rate of the YBCO films was 58–101 μm h^{-1}, approximately 60–1000 times higher than that of conventional CVD.

Keywords: $YBa_2Cu_3O_{7-\delta}$; Laser chemical vapour deposition; Epitaxial growth; Critical temperature

1. Introduction

Among the various thin-film preparing techniques, metalorganic chemical vapour deposition (MOCVD) for the preparation of $YBa_2Cu_3O_{7-\delta}$ (YBCO) films is promising because it offers uniform coverage, excellent control of composition and can coat large areas of substrates with complex shape. MOCVD YBCO films exhibit high current density of 10^5–10^6 A cm^{-2} at the boiling point of liquid N_2 (77 K) [1–3], indicating great potential in industrial applications. MOCVD is cost effective and suitable for scaling up to continuous preparation by using a reel-to-reel system [4–7]. However, the drawback of MOCVD is the low deposition rate of 0.01 to several micrometer per hour.

In contrast, laser-assisted chemical vapour deposition (laser CVD) can grow various oxide films, such as Al_2O_3 [8], Ba_2TiO_5 [9], at significantly high deposition rates (several tens to hundreds micrometer per hour). We have epitaxially grown YBCO films on (100) MgO [10] and (100) $LaAlO_3$ (LAO) [11] single-crystal substrates by using laser CVD at deposition rates of approximately 60–90 μm h^{-1}. During the film deposition on a single crystal, the film is strained owing to the mismatch between the film and the substrate. Thus, the mismatch affect the crystallinity, microstructure and electrical performance of YBCO films [12,13].

In the present study, we prepared YBCO films on $SrTiO_3$ (STO) by using laser CVD and investigated the effects of deposition temperature and lattice mismatch on the epitaxial growth, crystallinity, microstructure and critical temperature of the YBCO films.

2. Material and methods

The YBCO films were grown on a (100) STO single-crystal substrate (10 mm × 5 mm × 0.5 mm) using $Y(DPM)_3$, $Ba(DPM)_2$/$Ba(TMOD)_2$ and $Cu(DPM)_2$ (DPM; dipivaloylmethanate and TMOD; 2,2,6,6-tetramethyl-3,5-octanedionate) as precursors. The Ba precursor was a mixture of $Ba(DPM)_2$ and $Ba(TMOD)_2$ with a molar ratio of 4:1, which suppressed the decomposition of $Ba(DPM)_2$ at temperatures greater than their eutectic point and vaporization at a constant rate.

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The laser CVD apparatus was described elsewhere [10,11]. A continuous wave Nd:YAG laser (wavelength: 1064 nm) with laser power (P_L) from 0 to 200 W was used. The STO substrate was preheated on a hot stage at 873 K. The flow rates of the Ar and O_2 gases were 0.75 and 0.13 Pa $m^3 s^{-1}$, respectively. The vaporization temperatures of the Y, Ba and Cu precursors were 440, 565 and 445 K, respectively. The composition of YBCO film analyzed by inductively coupled plasma spectrometry was Y:Ba:Cu = 1:2.1:3.6. The deposition temperature (T_{dep}) was measured with a thermocouple at the back side of the substrate. The total pressure was held at 0.6 kPa. The distance between the nozzle and the substrate was 30 mm. The deposition lasted 20 s. After deposition, the YBCO films were heat-treated at 573 K for 14.4 ks, 673 K for 14.4 ks and 773 K for 14.4 ks in pure O_2 atmosphere (100 kPa) to transform the orthorhombic structure of YBCO (*Pmmm*) to tetragonal structure ($P\bar{4}2m$).

The phase of the YBCO films was studied by X-ray diffractometer (XRD; Rigaku RAD-2C). The degree of the *c*-axis preferred orientation (*f*) of the YBCO films was calculated by using the Lotgering formula [14]:

$$f = \frac{P - P_0}{1 - P_0} \quad \text{where} \quad P = \frac{\sum I_{(00l)}}{\sum I_{(hkl)}} \quad \text{and} \quad P_0 = \frac{\sum I^0_{(00l)}}{\sum I^0_{(hkl)}}.$$

P represents the ratio of the sum of the XRD intensity of the *c*-axis-oriented YBCO peaks (00*l*) to the sum of the intensity of all the peaks (*hkl*) of the YBCO films from 10° to 60°. P_0 represents the corresponding ratio of the randomly oriented YBCO bulk (JCPDS #39-0486). The crystallinity of the YBCO films was evaluated by the full width at half maximum (FWHM) of the ω-scan (rocking curve) on the (200) or (005) reflection. The in-plane orientation of the YBCO films was measured by using a pole figure X-ray diffractometer (Rigaku Ultima IV). The schematic of the in-plane epitaxial growth relationship between the YBCO films and STO substrate was drawn by using VESTA [15]. The film microstructure was observed by using a field-emission scanning electron microscope (FESEM; JEOL JSM-7500F). The electrical resistivity was measured from 30 to 300 K by using the dc four-probe method. Au electrodes on the sample surface were prepared by an ion coater and connected to the resistivity measuring equipment with a thin copper wire (0.1 mm diameter).

3. Results and discussion

T_{dep} was increased from 928 to 1049 K by increasing P_L from 69 to 152 W. Fig. 1 shows the XRD patterns of the YBCO films prepared at T_{dep} = 947–1027 K. At $T_{dep} < 928$ K, no film was obtained. *a*-Axis-oriented YBCO films were prepared at T_{dep} = 928–947 K (Fig. 1(a)). YBCO films with a mixture of *a*-axis and *c*-axis orientations were prepared at T_{dep} = 968–988 K (Fig. 1(b)). *c*-Axis-oriented YBCO films were prepared at T_{dep} = 1006–1049 K (Fig. 1(c)). At

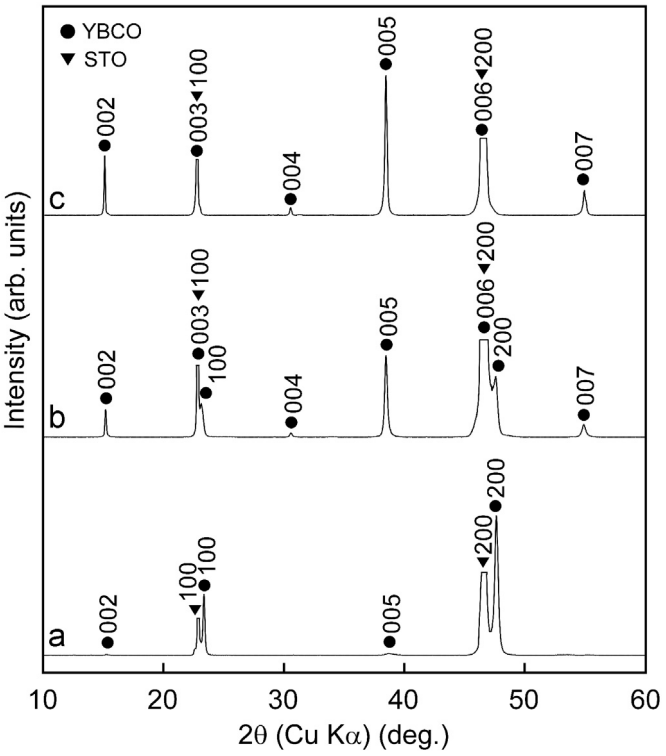

Fig. 1. XRD patterns of YBCO films prepared on (100) STO substrate at T_{dep} = 947 K (a), 968 K (b) and 1027 K (c).

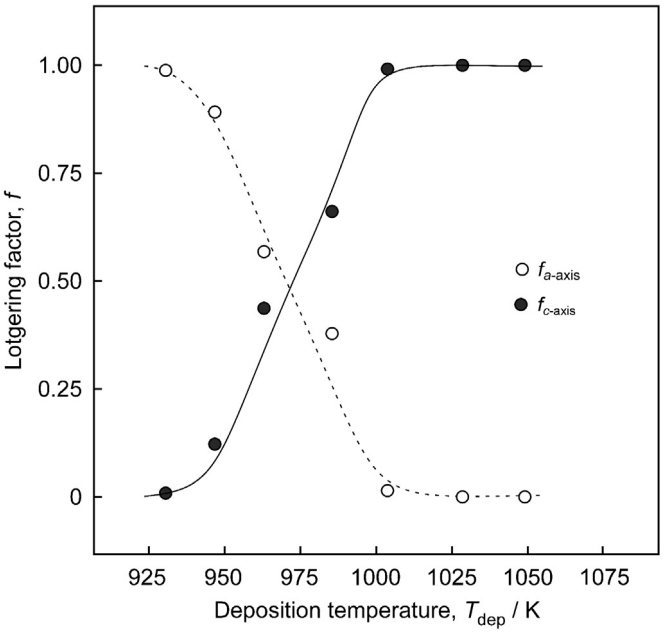

Fig. 2. *a*- and *c*-axis orientation of the YBCO films as a function of T_{dep}.

$T_{dep} > 1049$ K ($P_L > 152$ W), a small amount of Ba–Cu–O phase was present in the YBCO films. The degree of the preferred orientation (*f*) of the *a*-axis and *c*-axis is shown in Fig. 2. At T_{dep} = 928 K, *f* for the *a*-axis was 0.98 and that for *c*-axis was 0.02, indicating an almost fully *a*-axis-oriented YBCO film. With increasing T_{dep}, *f* for the *c*-axis

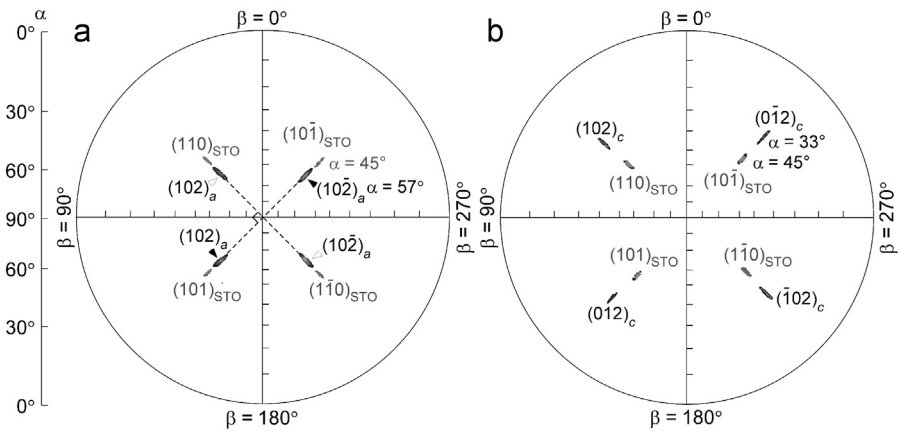

Fig. 3. X-ray pole figures of the (102) reflection in YBCO films grown on (100) STO substrate at T_{dep}=947 K (a) and 1027 K (b) and of the (110) reflection of the STO substrate.

increased and saturated to 1 at T_{dep}=1006 K, whereas f for the a-axis decreased and became 0 at T_{dep}=1006 K, suggesting fully c-axis-oriented YBCO films. The lower T_{dep} favoured the growth of a-axis-oriented grains, whereas the increase in T_{dep} favoured that of c-axis-oriented grains. For the laser CVD of YBCO films on MgO [10] and LAO [11], the dependence of T_{dep} on the preferred orientation is similar to that obtained in the present study.

Fig. 3 shows the X-ray pole figures of the (102) reflection of the YBCO films prepared at T_{dep}=947–1027 K and those of the (110) reflection from the STO substrate. Their in-plain epitaxial relationships are also depicted in this figure. In the a-axis-oriented YBCO films, the pole figure of the YBCO (102) reflection exhibited a two-fold pattern at an elevation angle (α) of around 57°, which was attributed to the YBCO (102) and (10$\bar{2}$) planes with a complementary angle of 33° to the YBCO (100) plane. The four reflections with a repeating angle of 90° were due to the twinning structure or the multi-domain structure of the a-axis-oriented grains [16]. The azimuth angles (β) of the poles of the YBCO (102) reflection appeared at the same angles as the STO (110) reflection (Fig. 3(a)), suggesting the in-plane epitaxial relationship YBCO [001]//STO [001] and YBCO [100]//STO [100] for the a-axis-oriented YBCO grains.

In the c-axis-oriented YBCO films, the strong four-fold pattern originated from the c-axis-oriented YBCO grains and was attributed to the YBCO {102} planes with a complementary angle of 57° to the YBCO (001) plane (Fig. 3(b)). The angle β of the YBCO (102) reflections was at the same angle as the STO (110) reflections, thereby indicating the in-plane epitaxial relationship YBCO [100]// STO [010] and YBCO [001]//STO [100].

Fig. 4 shows the FWHM of the ω-scan and φ-scan of the YBCO films grown on STO substrate. In the a-axis-oriented YBCO films, the ω-scan on the (200) reflection was measured. The FWHM values of the ω- and φ-scans decreased from 0.8° to 0.6° with T_{dep} increasing from 928 to 968 K. The ω-scan on the (005) reflection was measured

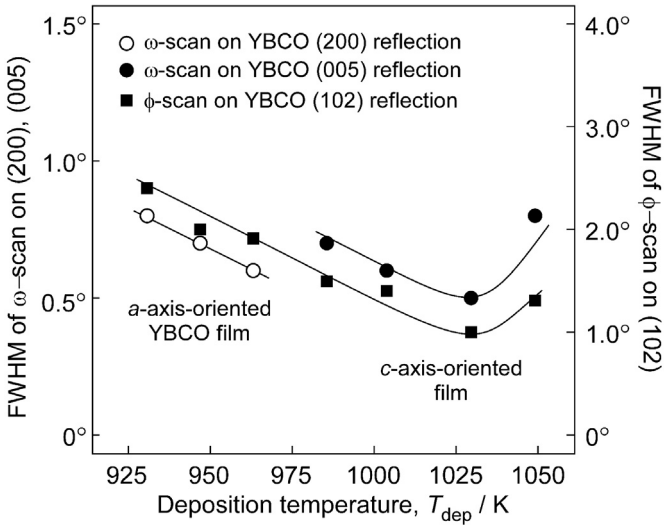

Fig. 4. FWHM of the ω-scan on the (200) and (005) reflections and of the φ-scan on the (102) reflection of the YBCO films on (100) STO substrate as a function of T_{dep}.

in the c-axis-oriented YBCO films. With T_{dep} increasing from 988 to 1049 K, the FWHM values of the ω- and φ-scans first decreased from 0.7° to 0.5° and then increased to 0.7°. A minimum of 0.5° corresponds to T_{dep}=1027 K. The φ-scan on the (102) reflection was measured in all YBCO films. The FWHM values first decreased from 2.4° to 1.0° and then increased to 1.3° with T_{dep} increasing from 928 to 1049 K, with the minimum of 1.0° at T_{dep}=1027 K.

Fig. 5 shows the surface and cross-sectional images of the YBCO films. At lower T_{dep}, *i.e.* 928–947 K, the a-axis-oriented YBCO films consisted of fine crystal grains of approximately 100 nm in size. With increasing the T_{dep} to 988 K, the microstructure of the a-axis- and c-axis-oriented YBCO films comprised needle-like grains and outgrowth particles distributed on a dense flat surface (Fig. 5(a, b)). The needle-like grains were a-axis-oriented YBCO grain, whereas the outgrowths were identified as Cu-rich particles [17–19]. The needle-like grains grew with orthogonal orientation on the film surface, which corresponded to

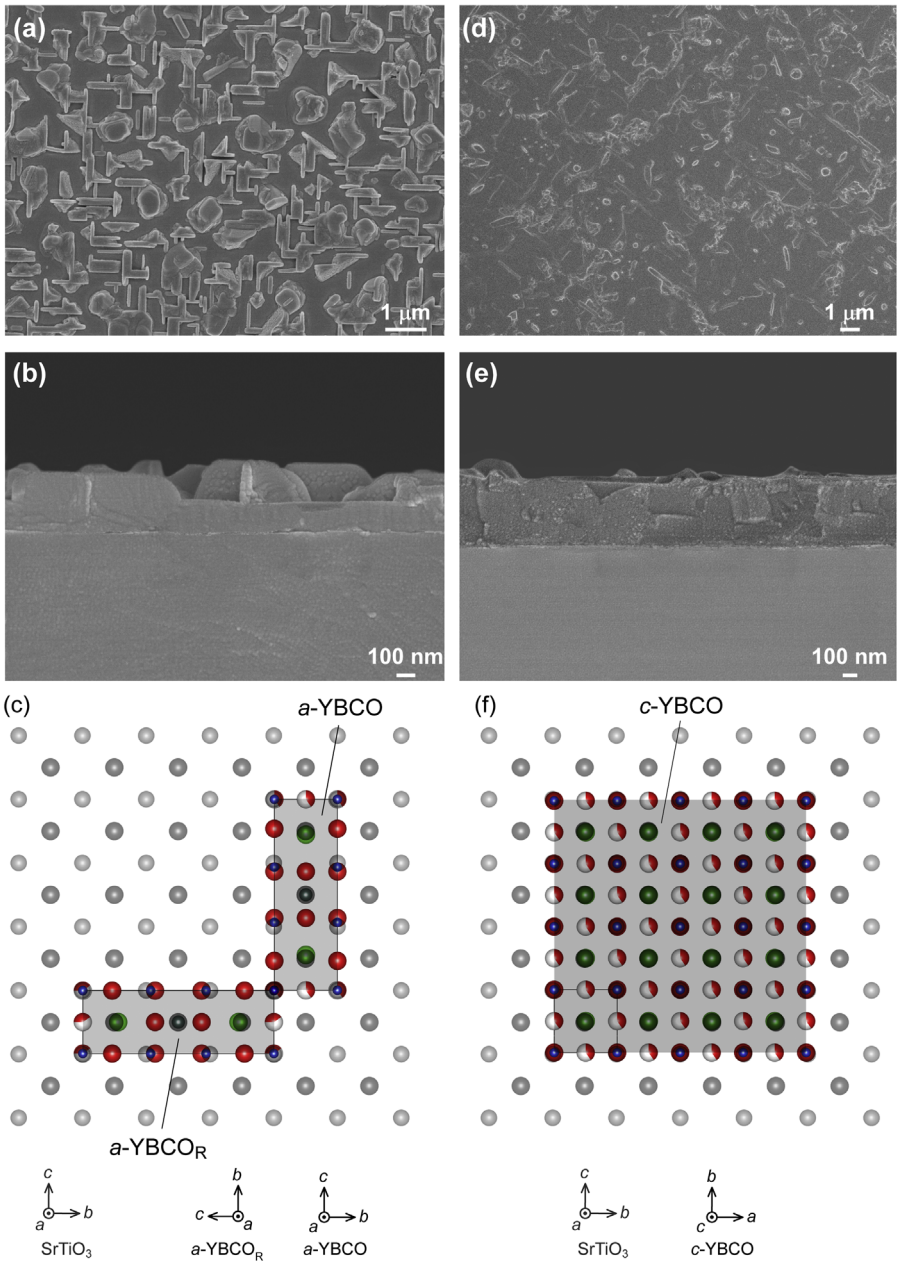

Fig. 5. Surface (a, d) and cross-sectional (b, e) SEM images of YBCO films grown at T_{dep}=968 K (a, b) and 1027 K (d, e). Images (c) and (f) depict the in-plane epitaxial relationships.

the multi-domain structure of *a*-axis oriented grains suggested by the pole figure (Fig. 3(a)) and derived the in-plane epitaxial growth model depicted in Fig. 5(c). As T_{dep} increased to 1006–1049 K, the *c*-axis-oriented YBCO films showed a dense surface and cross section (Fig. 5(d, e)). This was due to the excellent in-plane lattice matching between the *c*-axis oriented YBCO grain and (100) $SrTiO_3$ substrate (Fig. 5(f)). The amount of needle-like grains and outgrowth particles reduced significantly at high T_{dep}.

Fig. 6 shows the temperature dependence of the electrical resistivity of the YBCO films prepared at T_{dep}=947 K (*a*-axis-oriented YBCO film), 968 K (*a*-axis- and *c*-axis-oriented YBCO films) and 1027 K (*c*-axis-oriented YBCO film). The electrical resistivity of the *a*-axis-oriented YBCO films showed semiconducting temperature dependence in the normal state and became zero at transition temperatures in the range of 30–50 K (Fig. 6(a)). The electrical resistivity of the *a*-axis- and *c*-axis-oriented YBCO films showed weak metallic temperature dependence above the superconducting transition and became zero at transition temperatures between 55 and 77 K (Fig. 6(a)). The resistivity of the *c*-axis-oriented YBCO films showed strong metallic temperature dependence in the normal state and sharp zero resistance transition at 78–90 K (Fig. 6(b)). Fig. 7 shows T_C versus T_{dep} of the YBCO films. T_C increased from 30 K at T_{dep}=928 to 90 K at T_{dep}=1027 K

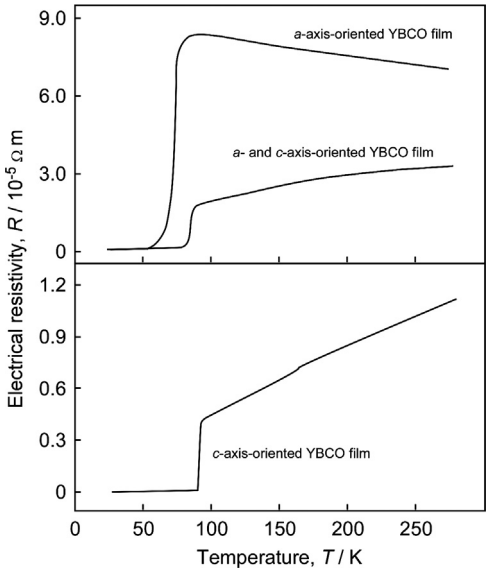

Fig. 6. Electrical resistivity as a function of the deposition temperature of the YBCO films grown on (100) STO substrate at T_{dep}=947 K (*a*-axis-oriented film), at 968 K (*a*- and *c*-axis-oriented film) and at 1027 K (*c*-axis-oriented film).

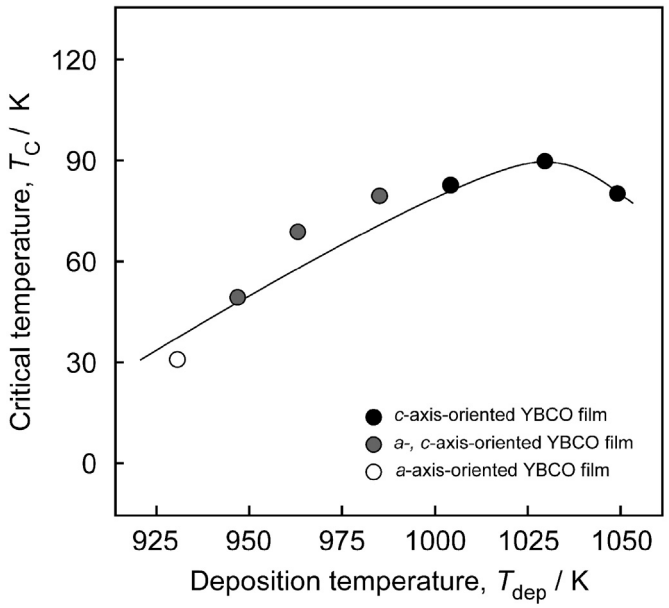

Fig. 7. T_C of the YBCO films grown on STO substrate as a function of T_{dep}.

and then decreased to 78 K at T_{dep}=1049 K. The highest T_C of 90 K was obtained at T_{dep}=1027 K. For the laser CVD YBCO films grown on MgO [10] and LAO [11], T_C as a function of T_{dep} exhibited similar tendencies to that reported in the present study, with the maximum T_C of 89 and 90 K.

Table 1 and Fig. 8 compare the FWHM of the ω-scan and φ-scan, T_C and the lattice mismatch of the YBCO films on the MgO [10], LAO [11] and STO substrates grown by laser CVD and MOCVD [20–23]. Our previous work showed that YBCO films grown on LAO substrate [11] have higher crystallinity and in-plane orientation than films grown on MgO substrate [10]. The reason is the lattice mismatch of 3.0% between LAO and YBCO that is

Table 1
Comparison of the FWHM of the ω-scan, FWHM of the φ-scan, T_C and the lattice mismatch of YBCO films prepared on MgO, LAO and STO substrates by laser CVD and MOCVD.

Method	Substrate	Orientation	ω-Scan (deg)	φ-Scan (deg)	T_C (K)	Mismatch (%)	Ref.
Laser CVD	MgO	*a*-Axis	1.3	4.2	47	22.7	10
		c-Axis	0.6	1.4	89	7.8	
	LAO	*a*-Axis	0.8	2.8	70	8.3	11
		c-Axis	0.6	1.1	90	2.5	
	STO	*a*-Axis	0.7	1.9	67	0.9	Present
		c-Axis	0.5	1.0	90	0.5	study
MOCVD	MgO	*c*-Axis	0.3–0.6	–	89	7.8	20, 21
	LAO	*c*-Axis	0.3	4.3	91	2.5	22
	STO	*c*-Axis	0.2–0.4	–	85–89	0.5	23

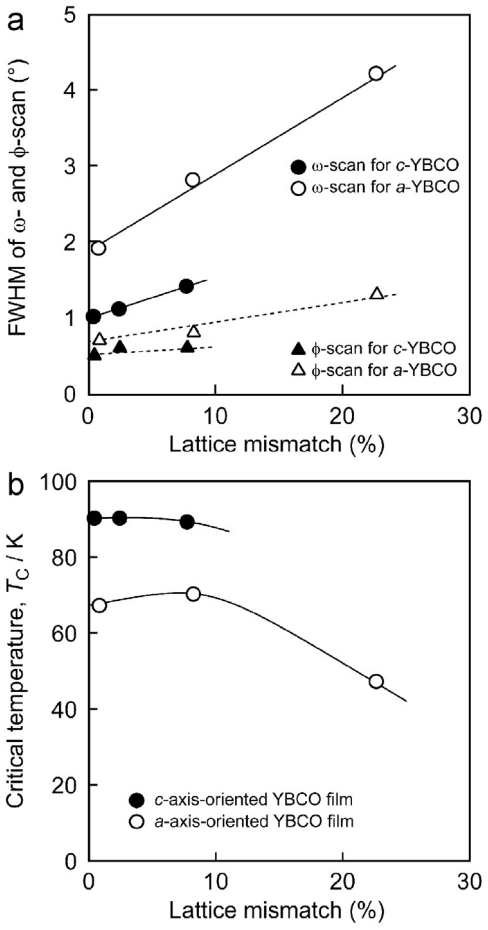

Fig. 8. Effect of lattice mismatch between YBCO films and substrates on the FWHM of ω-scan, φ-scan and T_C grown on MgO, LAO and STO substrates by laser CVD.

smaller than that of 8.6% between MgO and YBCO. On the other hand, the (100) STO single-crystal substrate and YBCO film have a lattice mismatch of 1.2%, which is smaller than that of (100) LAO (3.0%) and that of (100) MgO (8.6%). For the YBCO films on STO in the present study, the FWHM of the ω-scan showed smaller values of 0.5–0.7° than that of 0.6–0.8° on LAO [11] and 0.6–1.3° on MgO [10]. The FWHM of the φ-scan of the YBCO films on STO showed a range of 1.0–1.9° that is smaller than that of 1.1–2.8° on LAO [11] and 1.4–4.2° on MgO [10]. This was caused by the lattice mismatch of 0.5–0.9% between YBCO and STO that is smaller than that of 2.5–8.3% for LAO and of 7.8–22.7% for MgO. In spite of the large lattice mismatch of 0.5–7.8%, the YBCO films have T_C close to zero. For the *c*-axis-oriented YBCO films grown on STO, LAO and MgO by laser CVD, T_C and the FWHM of the ω-scan and φ-scan were comparable to the films grown by MOCVD.

Fig. 9 shows the comparison of the R_{dep} of the YBCO films by MOCVD [24–33] and laser CVD [10,11,34]. For MOCVD, R_{dep} ranged from 0.01 to several micrometer per hour [24–33]. In the CVD process, high R_{dep} requires high T_{dep} and high precursor supply rate. However, the rate-limiting step of the CVD process changes from surface chemical reaction to gas phase diffusion at elevated T_{dep} and thus R_{dep} becomes saturated at high T_{dep} [30,33]. High supply rates increase R_{dep} [25,26,31,32], whereas, in the thermal CVD process, excess precursor supply causes a premature chemical reaction in the gas phase forming powder and an insufficient chemical reaction on the substrate that results in the depression of R_{dep}, the formation of a second phase and degraded orientation. On the other hand, for laser CVD, laser irradiation activates the chemical reaction not only on the substrate surface but also in the gas phase, giving rise to significantly high R_{dep} even under a high precursor supply rate. The highest R_{dep} of the laser CVD YBCO films was up to 101 μm h^{-1}, approximately 60–1000 times higher than that by MOCVD.

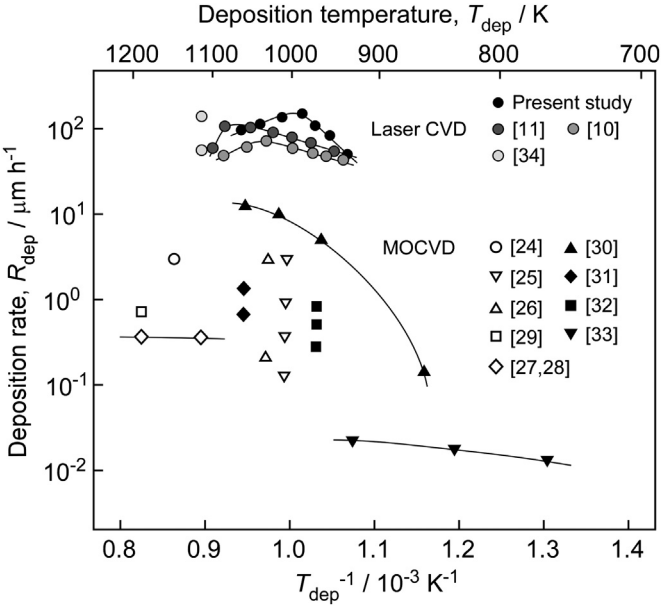

Fig. 9. R_{dep} of the YBCO films prepared by laser CVD and MOCVD.

4. Conclusions

a-Axis- and *c*-axis-oriented YBCO films were grown on (100) STO single-crystal substrate by laser CVD. *a*-Axis-oriented YBCO films were prepared at T_{dep}=928–968 K and *c*-axis-oriented films were prepared at T_{dep}=988–1049 K. For the *c*-axis-oriented YBCO films, the minimum FWHM of the ω-scan and φ-scan was 0.5° and 1.0°, respectively. The YBCO films on STO showed higher crystallinity, in-plane orientation and deposition rate than those on LAO and MgO. In addition, a high critical temperature of 90 K was observed in the *c*-axis-oriented YBCO films. The T_C and the FWHM of the ω-scan and φ-scan of the *c*-axis-oriented YBCO films grown by laser CVD were comparable to the films obtained by MOCVD even at a high-speed epitaxial growth (58–101 μm h^{-1}).

Acknowledgments

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