Influence of oxygen partial pressure on SmBa$_2$Cu$_3$O$_{7-δ}$ film deposited by laser chemical vapor deposition

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**ABSTRACT**

Laser chemical vapor deposition (LCVD) was applied to prepare SmBa$_2$Cu$_3$O$_{7-δ}$ (SmBCO) superconductive films on single-crystalline LaAlO$_3$ (100) substrates. The effect of oxygen partial pressure ($P_{O_2}$) on the crystal orientation, microstructure, and superconducting property of SmBCO film were investigated thoroughly. The results of X-ray diffraction (XRD) and Raman showed that the crystal orientation of SmBCO film was pure ε-axis orientation at the $P_{O_2}$ of 200 Pa, and the a-axis orientation grains gradually emerged in SmBCO film with the increase of $P_{O_2}$. The deposition rate, critical temperature ($T_c$), critical current density ($J_c$) firstly increased and then decreased with the increase of $P_{O_2}$. At the $P_{O_2}$ of 200 Pa, the SmBCO film had smooth and dense surface, the lowest FWHM of φ-scan (1.1°), the highest $T_c$ (89.2 K) and $J_c$ (2.04 MA/cm$^2$), which indicated that SmBCO superconductive films possessed excellent crystalline structure and superconducting performance.

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1. **Introduction**

The second generation of high-temperature superconducting tape REBCO has been attracted much attention in recent years, which has become a hot topic in present research, such as SmBa$_2$Cu$_3$O$_{7-δ}$ (SmBCO) [1,2]. SmBCO superconductor has been applied in different fields (e.g. cable transmission, motor, generator, magnetic energy storage system), which was due to its high critical temperature ($T_c$) and critical current density ($J_c$) [3,4]. The common physical deposition method of SmBCO film, such as pulsed laser deposition (PLD) [5–7], was not suitable for the large-scale commercial application, because of its high vacuum degree requirement and low deposition rate. Compared with PLD, metal organic chemical vapor deposition (MOCVD) is more adapted to industrial production of thin films. The vacuum degree requirement of MOCVD is not high, which is beneficial to reduce the cost of equipment. Moreover, the preparation process is simple and the preparation parameters are easier to control [8–10]. Therefore, the preparation of SmBCO film by MOCVD has a better application prospect. However, the deposition rate of traditional MOCVD method still could not satisfy the large-scale and commercial preparation of SmBCO films. In order to increase the deposition rate, laser has been introduced into the chemical vapor deposition [11–14]. Laser chemical vapor deposition (LCVD) can remarkably improve chemical reaction of precursors on substrates, which is conducive to enhance the deposition rate of film.

In addition, except for the deposition rate, the excellent crystal structure, and superconducting performance are also necessary for the commercial application of SmBCO film. The main impacts of the structure and performance of SmBCO film are deposition temperature, deposition time, oxygen partial pressure and so on. The oxygen partial pressure is particularly important, which directly determines the pure ε-axis orientation structure and oxygen content of SmBCO film [15,16]. In recent years, some researches showed that the suitable oxygen partial pressure contributed to optimize the structure of SmBCO film and improve the superconducting properties [17,18]. Jin et al. found that the low oxygen partial pressure was required to successfully obtain GdBa$_2$Cu$_3$O$_y$ films without impurity phases and a-axis-oriented grains [17]. Li et al. discovered that the morphology, growth rate, and degree of texture formation of YBCO films were affected obviously by oxygen partial pressure deposited by photo-assisted MOCVD [19]. Hence, it is necessary to investigate the effect of the oxygen partial pressure on the structure and superconducting performance of SmBCO films by LCVD, which is beneficial to accelerate the commercial application of SmBCO film.

For this purpose, in this paper, SmBCO film was prepared on single-crystalline LAO (100) substrates by LCVD. The effect of oxygen partial pressure on the phase composition, crystal orientation, microstructure, and critical current density of SmBCO superconducting films were thoroughly investigated.
2. Materials and methods

The vertical cold-wall laser chemical vapor deposition system (LCVD) was applied to prepare SmBCO films. The laser adopted in LCVD was a kind of semiconductor laser, which had continuous wave (wavelength: 808 nm). The substrate was single crystalline LaAlO₃ (LAO) substrates (10 × 5 × 0.5 mm). The precursors were Sm(dpm)₃, Ba(dpm)₂ and Cu(dpm)₂ (dpm: 2,2,6,6-tetramethyl-3,5-heptanedionato, Toshiba Manufacturing Co. Ltd., JP). The precursors were placed in independent vessels and heated in vacuum. The vaporizing temperatures of Sm(dpm)₃, Ba(dpm)₂ and Cu(dpm)₂ were set to 192, 300, and 168°C, respectively. The precursor gases were transported by Ar gas. In order to prevent the precursor gases condensing in the pipelines, the gas pipelines and nozzle were generally heated to higher than 350°C. The precursor gases and O₂ gas were mixed before entering the nozzle. The substrates were placed directly below the vessel and heated by the laser with beam diameter of 15 mm. When the substrate reached 780°C, opening the nozzle to let the reaction gases enter the reaction chamber. The total pressure (P_sub) of the reaction chamber was fixed at 500 Pa. The flow rate of Ar gas (F_Ar) in each line (3 line) was fixed to 100 standard cubic centimeter per minute (sccm). The flow rate of O₂ (F_O₂) was 100, 200, 300, and 450 sccm, the corresponding oxygen partial pressure (P_O₂) are shown in Table 1. The film growth time was set as 10 min. After the deposition, the SmBCO films were heat-treated at laser power of 30 W for 1 h in pure O₂ gas at an atmosphere pressure.

X-ray diffraction (XRD; Ultima-Ь, Rigaku, JP) was used to measure the crystalline phases of the SmBCO films with Cu-Kα radiation. The in-plane orientation of films was estimated by ϕ-scan on the (116) reflection. The Raman scattering spectra were measured in a quasi-backscattering geometry at the wavelength of 633 nm with an Ar ion laser (INVIA, Ger). The surface and cross-sectional micromorphology of the films was characterized by field emission scanning electron microscopy (SEM; FEI Quanta-250, USA). Atomic force microscopy (AFM; Innova, Bruker, USA) working at the tapping mode was used to characterize the surface and to measure the roughness of the films. The superconducting critical temperatures (T_c) of the SmBCO films were tested by standard four-point-probe at the temperature range of 10–300 K by using a physical properties measurement system (PPMS 9; Quantum Design, USA). The critical current (I_c) were obtained at 77 K and self-field, and the voltage criterion was 1 μV/cm [20]. The J_c was calculated from the ratio of critical current (I_c) to the film thickness.

3. Results and discussion

The XRD patterns of SmBCO films deposited on LAO substrate by LCVD at varied P_O₂ were portrayed in Figure 1. It could be observed that only c-axis oriented peaks (00 l) of SmBCO film peaks appeared in Figure 1(a,b), which was corresponding to the P_O₂ of 125 Pa and 200 Pa, respectively. The intensity of c-axis oriented peaks of the SmBCO film enhanced with the increasing of P_O₂. Furthermore, it was noteworthy that the c-axis-oriented peaks intensity of the SmBCO film was the highest at the P_O₂ of 200 Pa, which indicated that the c-axis preferred orientation of SmBCO film was strongest at this P_O₂. When the P_O₂ exceeded 200 Pa (Figure 1(c,d)), a-axis-oriented peaks (h00) appeared in the XRD patterns of SmBCO film, and the intensity of a-axis-oriented peaks was gradually increased with the P_O₂ increasing. The results indicated that the SmBCO film transitioned from pure c-axis to a- and c-axis coorientation with the increase of P_O₂, moreover, the proportion of a-axis orientation particles increased.

The orientation change of SmBCO film was related to the crystal growth mode [21]. The crystal structure of SmBCO was a kind of defect perovskite structure as shown in Figure 1(e). The length of a-axis was similar to that of b-axis, and the length of c-axis was about 3 times the length of a-axis, which resulted in the growth orientation of SmBCO film transforming with the change of P_O₂. When the oxygen partial pressure was low, it was beneficial to the nucleation of SmBCO grains along the vertical direction of small surface area, namely the c-axis direction. When the oxygen partial pressure was relatively high, it would promote the nucleation of SmBCO grains along the vertical direction of large surface area (the a-axis direction). Hence, it could be concluded that the low P_O₂ was conducive to the growth of SmBCO grains along the c-axis direction and restrained the growth along the a-axis direction, especially at the P_O₂ of 200 Pa, the c-axis preferred orientation of SmBCO grains was the strongest.

Figure 2 exhibits XRD ϕ-scan for (116) reflection of SmBCO film deposited at P_O₂ of 125–300 Pa and the effect of P_O₂ on the full width at half maximum (FWHM) of ϕ-scan. At the P_O₂ of 125 and 200 Pa (Figure 2(a,b)), there were four strong peaks appearing in the ϕ-scan for (116) reflection of SmBCO film, the interval between adjacent peak was 90°. The four peaks were attributed to c-axis-oriented SmBCO grain. Compare with the P_O₂ of 125 and 200 Pa, it was obviously observed that extra eight lower peaks appeared in the ϕ-scan for (116) reflection of SmBCO film at the P_O₂ of 250 and 300 Pa (Figure 2(c,d)). The eight peaks were divided into two groups, each group had four peaks and the interval

Table 1. The oxygen partial pressure (P_O₂) of SmBCO films deposited by LCVD.

| F_Ar (sccm) | 100 | 200 | 300 | 450 |
| P_O₂ (Pa)  | 125 | 200 | 250 | 300 |

\[ P_O₂ = \frac{P_{tot} \cdot F_Ar}{F_O₂ + \sum F_{Ar}}. \]
between adjacent peaks was 90°, each group of peaks represented the \( a \)-axis orientation in the SmBCO film. The eight peaks indicated that twinning structure of \( a \)-axis orientation appeared in SmBCO films.

FWHM of \( \varphi \)-scan was utilized to evaluate the in-plane orientation consistency of SmBCO film, the lower the FWHM value, the better the in-plane orientation consistency of SmBCO film, that was, the better the crystallization quality of SmBCO film. The \( \varphi \)-scan on the (116) reflection was measured in all SmBCO films, and the FWHM of \( \varphi \)-scan was the FWHM of average value of FWHM of four \( c \)-axis oriented peaks. In Figure 2(e), it could observe the FWHM of \( \varphi \)-scan of SmBCO film (116) reflection decreased firstly and then increased with the increase of \( P_{O_2} \). The FWHM value was the minimum (1.1°) at the \( P_{O_2} \) of 200 Pa, which implied that the grain-to-grain ordered alignment was relatively excellent. At high \( P_{O_2} \) (250 and 300 Pa), the FWHM value increased to 1.4° and 1.8°, which was due to the appearance of the \( a \)-axis coorientation in SmBCO films. Although the length of \( a \)-axis was similar to three times the length of \( a \)-axis, there was 1.4% lattice mismatch between them (according to JCPDS#89-8900). The lattice mismatch would generate a large number of boundaries, which led to the poor in-plane arrangement of the grains in the film. The formation of \( a \)-axis-orientated grains deteriorated the in-plane alignment of SmBCO film, resulting in that the FWHM of \( \varphi \)-scan for (116) reflection SmBCO film was higher at \( P_{O_2} \) of 250 and 300 Pa.

Figure 3 displays the Raman scattering spectra of SmBCO film deposited at the \( P_{O_2} \) of 125–300 Pa. The peak around at 140 cm\(^{-1}\) was the stretching modes of Cu2-Cu2 in the CuO2 plane. The peak of 220 cm\(^{-1}\) was broken-chain mode or oxygen disorder, which was related to the imperfect grain connectivity and instability of original phases. The peaks located at 317 cm\(^{-1}\) and 445 cm\(^{-1}\) were the out-of-phase bending mode of O(2)/O(3)- and the in-of-phase bending mode of O(2)/O(3)+, respectively [22]. The peak intensity of O(2)/O(3)- should be maximum if the films had a \( a \)-axis orientation in an orthorhombic phase [23,24]. The peak placed at 500 cm\(^{-1}\) was apical O(4) mode, which was connected with the oxygen stoichiometry. The intensity relative ratio of the O(2)/O(3)- peak and O(4) peak in \( c \)-axis-oriented REBCO film was regarded as an indication of tilted grains and \( a \)-axis grains [25]. In Figure 3(a), a broad peak around 530 cm\(^{-1}\) was detected at \( P_{O_2} \) of 125 Pa. There was no enough oxygen for the chemistry reaction during the SmBCO film deposition process, the peak of the apical O(4) mode (the peak around 500 cm\(^{-1}\)) moved to the right [26]. The O(2)/O(3)- peak at around 317 cm\(^{-1}\) was higher than that of 500 cm\(^{-1}\) (Figure 3(b)), indicating the orientation of SmBCO film was mainly \( c \)-axis at \( P_{O_2} \) of 200 Pa, and
the peaks located at 220 cm$^{-1}$ were extremely weak, which manifested that the SmBCO film deposited at the $P_{O_2}$ of 200 Pa had excellent crystal structure. At the $P_{O_2}$ of 250 and 300 Pa (Figure 3(c,d)), it could be observed that the peak at 500 cm$^{-1}$ enhanced obviously, which implied that the $a$-axis-oriented and tilted SmBCO grains formed easily with the increasing $P_{O_2}$. Furthermore, at $P_{O_2}$ of 300 Pa, there

Figure 2. $\varphi$-scan for (116) reflection of SmBCO films with $P_{O_2}$ at (a) 125, (b) 200, (c) 250 and (d) 300 Pa, ($\chi_c = 54^\circ$ for $c$-axis orientation, $\chi_a = 24^\circ$ for $a$-axis), (e) FWHM of $\varphi$-scan for (116) reflection of SmBCO with $P_{O_2}$ at 125–300 Pa.
was stronger peak at 220 cm$^{-1}$, which was due to the poor crystallinity.

The surface morphologies of SmBCO films prepared at different $P_{O_2}$ were displayed in Figure 4. It could be seen that SmBCO film deposited at $P_{O_2}$ of 125 Pa had rough surface with random arranged grains (Figure 4(a)), the irregular grain growth produced large-angle grain boundaries and pores, which resulted in the poor crystallization quality of SmBCO films. The low $P_{O_2}$ was not conducive to the orientation deposition of SmBCO film. When the $P_{O_2}$ increased to 200 Pa, the surface morphology of SmBCO film became smooth and dense (Figure 4(b)), indicating that the SmBCO deposited at $P_{O_2}$ of 200 Pa had relatively excellent orientation and less impure phase. At $P_{O_2}$ of 250 Pa (Figure 4(c)), it could be seen obviously that needlelike grains on a flat surface appeared in the SmBCO film, the needlelike grains was due to the $a$-axis-oriented growth of SmBCO [27]. Moreover, when the $P_{O_2}$ increased to 300 Pa (Figure 4(d)), the quantity and size of needlelike grains noteworthy increased. Under the condition of high $P_{O_2}$, the deposition rate of SmBCO films along the $<100>$ and $<010>$ directions ($a$-axis-oriented SmBCO grains) was faster than that along the $<001>$ direction ($c$-axis oriented SmBCO films), which led to a lot of needlelike grains on SmBCO films [28].

The results of surface morphologies of SmBCO films were consistent with the change of orientation at varied $P_{O_2}$.

Figure 5 exhibits the three-dimensional AFM images of 5 × 5 μm area of the SmBCO films prepared at different $P_{O_2}$. It could be obviously observed that the surface roughness (Ra) of SmBCO films changed remarkably with the $P_{O_2}$ increase. At the $P_{O_2}$ of 125 Pa, the Ra of SmBCO films was 124 nm, when the $P_{O_2}$ increased to 200 Pa, the Ra of SmBCO films decreased to 44.6 nm, which indicated that the surface roughness of the films became smoother with the $P_{O_2}$ increase. However, when the $P_{O_2}$ exceeded 200 pa, the Ra of SmBCO films rapidly increased, and the Ra of SmBCO films at 250 pa and 300 pa were 64.2 nm and 160 nm, respectively, which was due to the $a$-axis-oriented grains growing faster on the surface of SmBCO films than that of $c$-axis oriented grains at higher $P_{O_2}$. The result showed that SmBCO films deposited at the $P_{O_2}$ of 200 Pa possessed the smooth surface and excellent orientation.

The cross-sectional images of SmBCO films and the deposition rate dependence on $P_{O_2}$ were displayed in Figure 6. All of the cross-sections of SmBCO films were dense without gap between the film and substrate, indicating that LCVD was appropriate for preparing SmBCO film on LAO substrates. Noticeably, the upper edge of the cross-sections of the SmBCO film
deposited at the $P_{O_2}$ of 200 Pa (Figure 6(b)) was more smooth, some protrusions could be observed on the cross-sections of the SmBCO film at other $P_{O_2}$ (Figure 6(a,c,d)), especially the $P_{O_2}$ of 300 Pa, the appearance of protrusions on the cross section of SmBCO film were due to the faster-growing $a$-axis-oriented particles [29].

The deposition rate of SmBCO film was the ratio of film thickness and the corresponding deposition time, and the deposition rates at different $P_{O_2}$ are shown in Figure 6(e). It could be seen that the deposition rate of SmBCO film was increased with the $P_{O_2}$ increasing at low $P_{O_2}$ (ranging from 125 to 200 Pa). The deficiency of oxygen led to the slow deposition rate at low $P_{O_2}$, and this situation was noticeably alleviated with the $P_{O_2}$ increasing, the deposition rate reached the maximum (8.76 $\mu$m h$^{-1}$) at the $P_{O_2}$ of 200 Pa. However, when the $P_{O_2}$ exceeded 200 Pa, the deposition rate decreased with the increasing of $P_{O_2}$. The higher $P_{O_2}$ resulted in the oxygen content increase in the reaction chamber, which would surely decrease the other precursor gases content at the constant total pressure (500 Pa). The imbalance stoichiometric ratio of oxygen and precursor ultimately induced the slow deposition rate and the appearance of impure phase. In addition, in Figure 6(e), it could observed that the $R_{\text{dep}}$ of the REBCO films deposited by LCVD was obviously higher than those deposited in other CVD [10,19,30–33]. In the CVD process, high $O_2$ and precursors supply were essential to the high $R_{\text{dep}}$. However, in the thermal CVD, excess $O_2$ and precursors supply would lead to the premature reaction in the gas phase above the substrate and insufficient reaction on the substrate, which would cause the decline of $R_{\text{dep}}$ and the formation of a second phase and degraded orientation [34,35]. Compared with the thermal CVD, the $R_{\text{dep}}$ of REBCO film deposited by LCVD was high at low $P_{O_2}$, which was due to the laser irradiation improving the decomposition of $O_2$ and activating the chemical reaction in the gas phase, giving rise to significantly high $R_{\text{dep}}$ even under low $P_{O_2}$. On the other hand, the heating mode of LCVD (surface heating) could avoid the temperature.
difference in the thickness direction, which was beneficial to improve the orientation consistency [36].

The electrical resistivity to temperature (R-T) curve of the SmBCO film deposited at the $P_{O_2}$ of 200 Pa and the effect of $P_{O_2}$ on the $T_c$ were exhibited in Figure 7. It could be observed that the electrical resistivity of the SmBCO films reduced with the decrease of temperature above 89.2 K (Figure 7(a)), that was, the SmBCO films showed strong metallic temperature dependence above 89.2 K. When the temperature was below 89.2 K, the electrical resistivity of the SmBCO films reduced sharply to zero, which indicated the critical temperature ($T_c$) value of the SmBCO films deposited at the $P_{O_2}$ of 200 Pa was 89.2 K. As showed in Figure 7(b), the $T_c$ of SmBCO film increased from 86.5 to 89.2 K with the increasing of $P_{O_2}$ from 125 to 200 Pa. When the $P_{O_2}$ was over 200 Pa, the $T_c$ of SmBCO film decreased with the increasing of $P_{O_2}$; the $T_c$ dropped to 79.6 K at the $P_{O_2}$ of 300 Pa. Notably, the $T_c$ of SmBCO film was the highest at the $P_{O_2}$ of 200 Pa, which manifested that the superconducting performance of SmBCO film was relatively easy to achieve at the $P_{O_2}$ of 200 Pa.

The critical current density ($J_c$) was utilized to evaluate the current carrying capacity of superconductive film. The higher the $J_c$, the better the current-carrying capacity, The $J_c$ could be calculated from the ratio of critical current ($I_c$) to the thickness. Figure 8 exhibits the current-voltage (I-V) curve (at 77 K, self-field) for SmBCO films deposited at $P_{O_2}$ of 200 Pa and the $J_c$ of the SmBCO films dependence on the $P_{O_2}$. The SmBCO film deposited at the $P_{O_2}$ of 200 Pa was superconducting state at 77 K (as shown in Figure 7), the voltage of SmBCO film should be zero with the increasing of current under this situation. However, in Figure 8(a), it could be observed that the voltage of SmBCO film increased rapidly when the current exceeded a certain value, which indicated that the superconducting performance of SmBCO film was lost. The voltage reaching 1 $\mu$V/cm was considered as the criterion of the critical superconducting performance, and the corresponding current was $I_c$. The $I_c$ of SmBCO film deposited at the
$P_{O_2}$ of 200 Pa was 297 A/cm$^2$ at 77 K and self-field. In the Figure 8(b), the $J_c$ of the SmBCO films gradually increased with the variation of $P_{O_2}$ (from 100 to 200 Pa), and the $J_c$ was the maximum (2.04 MA/cm$^2$) at the $P_{O_2}$ of 200 Pa. It indicated that the increase of $P_{O_2}$ was beneficial to improve the current carrying capacity of the films.
4. Conclusions

SmBCO films were deposited by LCVD on LAO (100) substrates at $P_{O_2}$ of 125–300 Pa. The effect of $P_{O_2}$ on the orientation, crystallinity and superconductivity performance of SmBCO film was investigated. The following conclusions can be drawn from the results:

1. The SmBCO films gradually transformed from $c$-axis to $a$- and $c$-axis coorientation with the increasing of $P_{O_2}$, and the appearance of $a$-axis-oriented grains in SmBCO films led to the poor in-plane arrangement in the film. The SmBCO films deposited at the $P_{O_2}$ of 200 Pa had the strongest $c$-axis preferred orientation and excellent in-plane arrangement (the FWHM value of 1.1°).

2. The deposition rate of SmBCO films first increased and then decreased with the increasing of $P_{O_2}$, which was due to the imbalance stoichiometric ratio of oxygen and precursor at high $P_{O_2}$. The reaction system possessed the optimal stoichiometric ratio of reaction system at the $P_{O_2}$ of 200 Pa, whose deposition rate reached the maximum (8.76 $\mu$m·h$^{-1}$).

3. At the $P_{O_2}$ of 200 Pa, the SmBCO film had the highest $T_c$ (89.2 K) and $J_c$ (2.04 MA/cm$^2$), which indicated that the SmBCO superconductive films deposited at the $P_{O_2}$ of 200 Pa exhibited the best superconducting performance.
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Disclosure statement

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