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ABSTRACT

Wide bandgap (E_g ~ 3.1 eV) La–doped BaSnO₃ (LBSO) has attracted increasing attention as one of the transparent oxide semiconductors since its bulk single crystal shows a high carrier mobility (~320 cm² V⁻¹ s⁻¹) with a high carrier concentration (~10¹⁸ cm⁻³). For this reason, many researchers have fabricated LBSO epitaxial films thus far, but the obtainable carrier mobility is substantially low compared to that of single crystals due to the formation of the lattice/structural defects. Here we report that the mobility suppression in LBSO films can be lifted by a simple vacuum annealing process. The oxygen vacancies generated from vacuum annealing reduced the thermal stability of LBSO films on MgO substrates, which increased their carrier concentrations and lateral grain sizes at elevated temperatures. As a result, the carrier mobilities were greatly improved, which does not occur after heat treatment in air. We report a factorial design experiment for the vacuum annealing of LBSO films on MgO substrates and discuss the implications of the results. Our findings expand our current knowledge on the point defect formation in epitaxial LBSO films and show that vacuum annealing is a powerful tool for enhancing the mobility values of LBSO films.

Transparent oxide semiconductors (TOSs) are promising candidates for various future electronic devices such as transistors, solar cells, and display panels. For such applications, high electron mobility (μ) is essential, and this has been a great disadvantage for TOS materials since their mobility values are low compared to classical semiconductors. In this regard, perovskite La–doped BaSnO₃ (LBSO) is gaining significant interest since its bulk single crystal exhibits a wide bandgap (~3.1 eV) and a very high mobility of 320 cm² V⁻¹ s⁻¹, which is comparable to that of doped single crystal Si (~350 cm² V⁻¹ s⁻¹). For this reason, there have been many attempts to utilize LBSO in thin films transistors.

However, crystalline defects prevent the μ in LBSO films from reaching the single crystal value, and many studies were devoted to improving the crystal quality of epitaxial LBSO films. For example, since misfit dislocations occur at the film/substrate interface due to the lattice mismatch, buffer layers are commonly used to reduce the dislocations.

To completely eliminate the film/substrate mismatch, Lee et al. grew LBSO films on single crystal BaSnO₃ substrate. In addition, in our recent study, we fabricated LBSO films under an ozone atmosphere to reduce the amount of point defects. Unfortunately, while these approaches were successful in improving the mobility values in LBSO films (up to ~120 cm² V⁻¹ s⁻¹), they can significantly increase the fabrication cost, which may be a crucial issue in mass production systems at industrial scales.

In large scale production facilities, designing a clever post treating process is often more economical than improving the quality of as-deposited samples. In this regard, very interesting experimental results were released in 2015 and 2018. In one study, N₂ environment at 1000 °C was used to create oxygen vacancies in the LBSO films on SrTiO₃ substrates, and the μ increased from 41 cm² V⁻¹ s⁻¹ to 78 cm² V⁻¹ s⁻¹. In the other study (same research group), oxygen vacancies were generated in the LBSO films on SrTiO₃ substrates using H₂...
forming gas at 950 °C, which further improved the μ up to 122 cm² V⁻¹ s⁻¹. According to these studies, oxygen vacancies can neutralize the negative charges at threading dislocations and induce lateral grain growth at elevated temperatures.

As removing oxygen ions (O²⁻) near threading dislocations decreases their thermal stability, oxygen vacancy doping creates a very strong driving force for lateral grain growths at high temperatures, which significantly increases the free propagation length of the carrier electrons. These results suggest that post treating of LBSO films can be just as effective as modifying the synthesis methods for improving the as-deposited crystal quality of LBSO films. In undoped BaSnO₃ films, vacuum annealing is commonly used to create oxygen vacancies and induce mobile charge carriers. Since vacuum annealing process is much simpler than creating N, C, and O⁻ vacancies in LBSO films and oxygen vacancy in BaSnO₃ films do not have sufficient oxygen vacancies to conduct electricity as all electrons form firmly bound states. The lateral grain sizes were determined from the diffraction spot of the air annealed film was almost the same with that of the as-deposited film, the (204) diffraction spot of the vacuum annealed was two times more intense compared to the other two films (as-deposited and air-annealed). The lateral grain sizes (D) of the as-deposited, air annealed, and vacuum annealed LBSO films were 9.1 nm, 10.2 nm, and 22.3 nm, respectively. The small grain size change after air annealing is not surprising since the film was deposited at 750 °C, and no significant change in the microstructure was expected. On the other hand, the vacuum annealing substantially increased the lateral grain size, which is consistent with the H₂ forming gas experiment. This shows that vacuum annealing can indeed be an alternative method for triggering oxygen vacancy assisted grain growth in LBSO films.

In order to find the optimum vacuum annealing temperature, several 2% LBSO films with similar thicknesses (~41 nm) and electrical properties were fabricated (see Table S1), and they were annealed in vacuum (~10⁻² Pa) at different temperatures ranging from 650 °C to 800 °C for 30 min. With increasing annealing temperature, the D of the films increased gradually from ~5 nm to ~17 nm [Fig. 2(a) and supplementary Fig. S1]. Figures 2(b) and 2(c) summarize the effect of the vacuum annealing on the electron transport properties of the LBSO film. The n increased from ~1.35 × 10¹⁰ cm⁻³ up to ~1.8 × 10¹⁰ cm⁻³ when the sample...
FIG. 1. Lateral grain growth of the LBSO film by the vacuum annealing. [(a)–(c)] RSMs near (204) diffraction peak of 2% La-doped LBSO films (∼64 nm) [(a) as-deposited film, (b) air-annealed at 750 °C for 1 h, and (c) vacuum annealed at 750 °C for 1 h]. The RSMs were shifted using the peak position of (204) MgO \((q_{[100]/2\pi} = 9.50 \text{ nm}^{-1}, q_{[100]/2\pi} = -4.75 \text{ nm}^{-1})\). [(d)–(f)] Diffraction spots of the LBSO films along the \(x\)-axis in the RSM. The lateral grain sizes \(D\) of the films are inversely proportional to the integral width. The \(D\) values extracted from the RSMs were (a) and (d) 9.1 nm, (b) and (e) 10.2 nm, and (c) and (f) 22.3 nm, respectively. The as-deposited film was cut into two pieces. One was annealed in air, while the other was annealed in vacuum. Almost no change in the RSM was observed from air-annealing. On the other hand, significant increase in the lateral grain size and peak intensity can be noticed from the vacuum annealed film. Unfortunately, electrical properties of the annealed samples could not be measured since they were cut from a 1 cm² square sample, and our probe station can only measure 1 cm² square coupons.

was annealed in the vacuum [Fig. 2(b)]. The observed \(n\) was far lower than the [La\(^{3+}\)] (=2.87 \(\times 10^{20}\) cm\(^{-3}\)), indicating the low carrier activation rate of the La\(^{3+}\) ions. The \(\mu_{\text{Hall}}\) of the films greatly increased (up to ∼80 cm\(^2\) V\(^{-1}\) s\(^{-1}\)) after vacuum annealing. The highest \(\mu_{\text{Hall}}\) was observed from the LBSO film vacuum annealed at 725 °C [Fig. 2(c)]. Above 725 °C, the \(\mu_{\text{Hall}}\) started to decrease despite the increase in the \(D\). This is likely attributed to high oxygen vacancy generation rate or chemical reaction at elevated temperatures. According to this result, 725 °C is the optimal vacuum annealing temperature for LBSO films on MgO substrates.

Once the films became thicker than ∼120 nm, both the as-deposited and vacuum annealed mobility values saturated at ∼84 cm\(^2\) V\(^{-1}\) s\(^{-1}\) and ∼100 cm\(^2\) V\(^{-1}\) s\(^{-1}\), respectively. Both as-deposited and vacuum annealed \(D\) exhibited strong thickness dependence (supplementary Fig. S2a). We believe the grain growth is hindered by the lattice strain, which decreases with increasing thickness.\(^{29}\) The highest mobility observed was 101.6 cm\(^2\) V\(^{-1}\) s\(^{-1}\) from vacuum annealed 117 nm LBSO film (as deposited: ∼74 cm\(^2\) V\(^{-1}\) s\(^{-1}\), supplementary Fig. S2d), which is comparable to that observed in LBSO films with buffer layers. This confirms that vacuum annealing is an effective method for enhancing the mobility of LBSO films.

To find the effect of La-dopants on the vacuum annealing process, we annealed LBSO films (average thickness: 43 nm) with varying [La\(^{3+}\)] at 725 °C in vacuum for 30 min. Figure 3 summarizes the electron transport properties of the annealed LBSO films. The \(n\) of the LBSO films always showed an increase
The highest as-deposited $\mu_{\text{Hall}}$ was observed in the 5% LBSO film, the highest $\mu_{\text{Hall}}$ after vacuum annealing was observed in the 2% LBSO film (35.5 cm$^2$ V$^{-1}$ s$^{-1}$ → 72.3 cm$^2$ V$^{-1}$ s$^{-1}$) although the annealed lateral grain size was highest in 7% LBSO film. The decrease in the vacuum annealed mobility from the 5% film is attributed to impurity scatterings from La dopants themselves. This suggests that impurity scatterings start to become significant if the lateral grain size is greater than ~12 nm [Figs. 3(c) and 3(d)]. These results indicate that 2% LBSO films have an optimized balance between oxygen vacancy formation, impurity scattering ($\mu_{\text{Hall}}$), and oxygen vacancy induced grain growth ($\mu_{\text{Hall}}$) for vacuum heat treatments.

It is important to note that mobility improvements were observed from 0.1% to 0.55% LBSO films despite the small changes in their grain sizes. In epitaxial films, threading dislocations often exhibit negative charges and generate energy barriers (mobility edge). Therefore, mobilities in epitaxial films can depend on the Fermi energy ($E_F$), which increases with the carrier concentration. Since vacuum annealing increases both $n$ and $D$, the $\mu_{\text{Hall}}$ improvements observed from 0.1% to 0.55% are mainly attributed to the $E_F$ shift from additional charge carriers because they did not show significant grain size changes. This also implies that mobility improvements observed at other doping levels could have been affected the increase in $n$ (i.e., higher $E_F$). To confirm the effect of $E_F$ shift on $\mu_{\text{Hall}}$, we vacuum annealed a 2% LBSO (45.7 nm) film from 100 °C to 800 °C in sequence in steps of 100 °C (i.e., vac. anneal 100 °C → 200 °C → … → 800 °C) to generate additional charge carriers while minimizing the grain growth. During the sequence annealing, the carrier concentration of the film gradually increased, but its electron mobility remained almost unchanged (supplementary Fig. S5). The drastic drop in $\mu_{\text{Hall}}$ at 800 °C is likely from structural damages due to high $V_O$ generation rate. The sequence annealing shows that the $E_F$ at $n \sim 0.5 \times 10^{20}$ cm$^{-3}$ exceeds the mobility edge of the LBSO films on MgO substrates, and the vacuum annealed mobilities are not strongly affected by the Fermi energy shift if $n > 0.5 \times 10^{20}$ cm$^{-3}$. This number is consistent with the $H_2$ forming gas experiment, where two post-treated LBSO films with the same carrier concentration of $1.1 \times 10^{20}$ cm$^{-3}$ exhibited different mobility values due to structural differences. According to our results, $\mu$ at low doping levels (0.1% and 0.55%) is dominated by the Fermi level. If $n > 0.5 \times 10^{20}$ cm$^{-3}$, the Fermi level seems to be above the mobility edge, and $\mu$ is mainly affected by electron scatterings (supplementary Fig. S5). The sources of electron scattering are point defects (impurity, $V_O$) and grain boundary (threading dislocation). The vacuum annealing effects observed from 2% to 5% LBSO films suggest that grain boundary scattering dominates up to $D > 12$ nm and point defect scattering dominates from $D > 12$ nm [Figs. 3(c) and 3(d)]. Therefore, majority of the mobility enhancement observed in 2% LBSO films (optimized doping) is attributed to the change in $D$ because their $E_F$ ($n > 1.0 \times 10^{20}$ cm$^{-3}$) are likely above the mobility edge.

In order to further investigate the effect of the vacuum annealing on the $\mu$ improvement of the LBSO films,
we performed the X-ray photoelectron spectroscopy (XPS) measurement of the LBSO films [Fig. 4(a): as-deposited, Fig. 4(b): vacuum annealed]. In case of oxygen in perovskite oxides, the lattice oxygen peak (L\textsubscript{O}) is located at \(-529\) eV. If oxygen vacancies are present, this peak shifts to \(~531\) eV (V\textsubscript{O}).\textsuperscript{1,8,31,32} Another oxygen peak around \(532 \sim 533\) eV (A\textsubscript{O}) can emerge from chemically adsorbed oxygen from surface contamination by organic molecules. The source of chemically adsorbed oxygen is unknown, but we believe it is related to the status of the vacuum chambers (annealing, XPS) or storing conditions. For the XPS peak fitting, a convolution between Gaussian (70\%) and Lorentzian (30\%) was used. For each LBSO films, the full width at half maximum (FWHM) was constrained to be the same for all 3 oxygen peaks.

The V\textsubscript{O}/L\textsubscript{O} area ratio from the XPS of the as-deposited LBSO films increased gradually when the [La\textsuperscript{3+}] exceeded 1\% (supplementary Table S2). Upon vacuum annealing, this ratio increased further for all films except for 7\% LBSO film. The largest change in V\textsubscript{O}/L\textsubscript{O} was observed from 2\% doped LBSO film. Since the solubility limit of La in LBSO was reported to be \(~5\%\), the oxygen vacancy reduction in 7\% LBSO is likely attributed to the formation of La\textsubscript{2}Sn\textsubscript{2}O\textsubscript{5}\textsuperscript{33} which could be observed from the ceramic targets used to deposit the films (supplementary Fig. S6). These results show that La-dopants in epitaxial LBSO films affect not only the oxygen stability but also the thermal stability of the film.

The XPS results show that vacuum annealing increases oxygen deficiency in the LBSO films, and oxygen vacancies in oxides normally provide additional conduction electrons. However, associating all changes in \(n\) to additional V\textsubscript{O} from vacuum annealing does not adequately explain our results. For example, according to the thickness dependence, the carrier concentration enhancement was greatly reduced with increasing thickness [Fig. 2(c)] although the oxygen vacancy generated from vacuum annealing would have been similar as the La\textsuperscript{3+} doping levels were the same unless the chemical potential of oxygen depends on the lattice strain. In addition, in the [La\textsuperscript{3+}] dependence, the changes in \(n\) [Fig. 3(a)] do not match the changes in V\textsubscript{O}/L\textsubscript{O} [Fig. 4(c)]. The implications of these phenomena can be very interesting as they suggest strain-dependent oxygen stability or oxygen vacancies not generating additional charge carriers. However, no firm conclusions can be drawn at this moment since the vacuum annealing effect on the activation of [La\textsuperscript{3+}] dopant is unclear.

Although we demonstrated that the mobility boost in the optimally doped films (2\%) after vacuum annealing is strongly related to the lateral grain sizes, we would like to note that there were a couple unusual instances. In supplementary Figs. S2(a) and S2(d), \(\mu_{\text{Hall}}\) of vacuum annealed 280 nm film is slightly smaller than that of vacuum annealed 117 nm film although their grain sizes are both \(>12\) nm and their electron densities are similar \((2.2 \times 10^{20} \text{ cm}^{-3} \text{ and } 2.1 \times 10^{20} \text{ cm}^{-3})\). This is another indicator that suggests strain-dependent oxygen vacancy generation, but this hypothesis requires more experimental evidence. Furthermore, in Fig. 2, the LBSO films
The vacuum annealing approach was very effective for films with small thicknesses. Therefore, it is a very good method for making LBSO film transistors since low thicknesses are desired for reducing the power consumption. We believe these results will be useful for designing low cost fabrication methods for high-mobility LBSO films or can be used to improve the carrier mobility of other perovskite stannates such as SrSnO$_3$.

See supplementary material for detailed XPS characteristics and RSM patterns of the LBSO films.

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