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Background signal from substrates in characterizations of magnetic thin films



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ABSTRACT

A LSMO thin film was fabricated on SrTiO₃ substrate by radio frequency magnetron sputtering method. The influences of the background signal from SrTiO₃ substrate on magnetic measurements were studied. Wide differences between the cases before and after subtracting the substrate background were observed. The magnetic properties of some commercial substrates, such as Si, *c*-plane Al₂O₃, (100) SrTiO₃, (100) MgO, fused SiO₂ glass and (100) GaAs, were also characterized.

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1. Introduction

Magnetic thin films are important functional materials. Compared to powder and bulk magnetic materials, they can be constructed into various devices, which had been used in wide fields, such as magnetic storage, magnetic sensor, magnetoelectric coupling and microwave devices, etc.[1–4]. In characterizing the magnetic property of thin film, the influences from substrate (including the background magnetic signal, strain and interface state) should be taken into account [5–8]. The measured signal is the superposition of that from the thin film and the substrate. For a weakly magnetic thin film, the magnetic background from substrate is even larger than the signal from the thin film because the thickness of substrate is larger than that of thin film by 1000–10,000 times.

In this work, by taking perovskite $La_{0.85}Sr_{0.15}Mn_{1.04}O_3$ (LSMO) for instance, the influences of background signal from $SrTiO_3$ on magnetic measurements are discussed. Wide difference between the cases without and with subtracting substrate background was observed. Additionally, the magnetic properties of some common commercial substrates, such as c-plane Al_2O_3 , (100) $SrTiO_3$, (100) MgO, fused silica glass (SiO_2), (100) GaAs and (100) Si, were also characterized.

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2. Experiments

The LSMO thin film was fabricated on a (100) SrTiO $_3$ substrate by radio frequency magnetron sputtering method. The thickness of the LSMO thin films is about 100 nm, measured by a step profiler. X-ray diffraction (XRD) was used to determine the structure of the thin film. The magnetization–magnetic field (M–H) curve and zero-field-cooling/field-cooling (ZFC/FC) curve were recorded on a superconducting quantum interference device (SQUID).

3. Results and discussion

Fig. 1(a) shows the $\theta-2\theta$ XRD pattern of the LSMO thin film on (100) SrTiO₃ substrate. The peaks located at 22.8°, 46.7° and 72.7° are assigned to the (100) diffraction peaks of LSMO superposed on the (100) ones of SrTiO₃ because of the very similar lattice constants between La_{1-x}Sr_xMnO₃ and SrTiO₃ [9]. The detail of the superposed (200) peaks is also shown in Fig. 1(a), in which the diffractions from LSMO and SrTiO₃ can be distinguished clearly. The above patterns indicate that the LSMO thin film is crystallized in single perovskite phase with (100) orientation.

Fig. 1(b) shows the magnetization–temperature (M–T) curves under an applied field of 10,000 Oe before and after subtracting the background signal of SrTiO₃, respectively. It can be seen that there appears a marked change in magnetic moment between the two cases. The change is not constant in the whole temperature range. The magnetization value increases from 515 to 651 emu/cc at 2 K, and increases from -91 to 73 emu/cc at 300 K, respectively. Clearly, the background signal from

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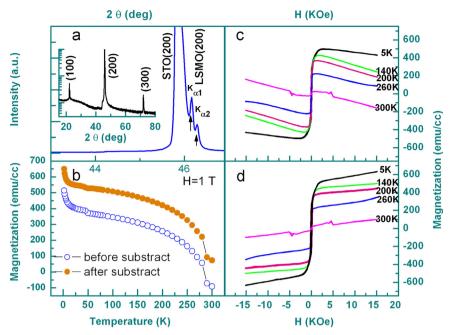


Fig. 1. (a) XRD patterns of the LSMO thin film on SrTiO₃ substrate, (b) magnetization–temperature (M-T) curves under a field of 10,000 Oe before and after the subtraction of SrTiO₃ substrate background, (c) as-measured M-H curves of the LSMO thin film/SrTiO₃ substrate and (d) M-H curves of the LSMO thin film after subtracting the background of SrTiO₃ substrate (in units of 1 Oe = $10^3/4\pi$ A/m and 1 emu = 103 A m²).

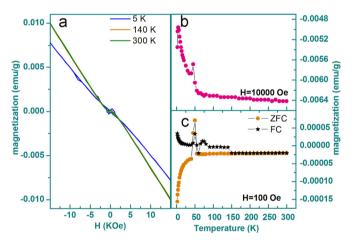


Fig. 2. (a) M–H curves of the (100) SrTiO $_3$ substrate at 5, 140 and 300 K, respectively, (b) M–T curve measured at an applied field of 10,000 Oe and (c) ZFC and FC magnetization as functions of temperature. The applied field is 100 Oe.

substrate influences the precision, or even the trueness of the measurement result.

Fig. 1(c) shows the as-measured M-H curves of the LSMO thin film at 5, 140, 200, 260 and 300 K, respectively, without subtracting the background from the SrTiO₃ substrate. With increasing the field, different behaviors are observed in a wide temperature range. In low field region, the M-H curve is dominated by ferromagnetism and superparamagnetism. When the field exceeds the saturation value, the magnetic moment begins to decrease with increasing the field. Especially near room temperature, the M-H curve extends into the 2nd and 4th quadrants at high field region. The magnetization is up to -161 emu/cc at 1.5 T. Obviously, it is not reasonable that such a large diamagnetic signal at room temperature was observed on a LSMO thin film with a size of $3 \text{ mm} \times 5 \text{ mm} \times 100 \text{ nm}$. After subtracting the SrTiO₃ background, the curves return to the 1st and 3rd quadrants, indicating the normal ferromagnetism or superparamagnetism, as shown in Fig. 1(d).

Fig. 2(a) shows the M–H curves of the (100) SrTiO $_3$ substrate at 5 K, 140 K and 300 K, respectively. The curves are mainly located in the 2nd and 4th quadrants in the figure. With increasing the field, the absolute value of the negative moment increases monotonously, agreeing with the diamagnetism of SrTiO $_3$ crystal. Note that a hysteresis loop with a certain coercive force and remanence is superposed on the M–H curve in the low field region. It indicates that a small ferromagnetic component exists in the SrTiO $_3$ single crystal substrate

Fig. 2(b) shows the M–T curve of SrTiO $_3$ substrate measured at a field of 10,000 Oe. A drastic decrease takes place in the low temperature region below 60 K. At the temperature above 60 K, the magnetization of SrTiO $_3$ almost keeps constant, about -0.0064 emu g $^{-1}$, with increasing the temperature. It is the intrinsic diamagnetic background of SrTiO $_3$ crystal substrate that makes the LSMO (\sim 100 nm)/SrTiO $_3$ sample show an apparent magnetic moment of -91 emu/cc at 10,000 Oe, as shown in Fig. 1(b).

Fig. 2(c) shows the ZFC/FC DC magnetization of (100) SrTiO₃ substrate as functions of temperature. The applied field is 100 Oe. As seen clearly, a bifurcation between the ZFC and FC curves takes place during decreasing the temperature, which is a typical characteristic of a magnetic frustration material. As indicated by the M-H measurements, both ferromagnetic and diamagnetic signals were observed. The diamagnetic signal is originated from the contribution of SrTiO₃. In general, the ferromagnetic component should be originated from the defects or magnetic impurities [10].

As demonstrated above, the influence of SrTiO $_3$ substrate back ground should be taken into account in characterizing some magnetic thin films. Due to the good lattice match, SrTiO $_3$ is one of the most frequently used substrates for perovskite materials growth. In fact, many wafer materials possess considerable magnetic background. For the growth of other thin films, it is also necessary to select appropriate substrate, in order to minimize the lattice mismatch, thermal mismatch and interface diffusion, etc. For instance, GaAs is often used as the substrate for $Ga_{1-x}Mn_xAs$, ZnSe: Mn(Fe, Co) and Fe thin films. In the following, the magnetic properties of some commercial substrates will be characterized, which were usually used as the substrates for functional thin films.

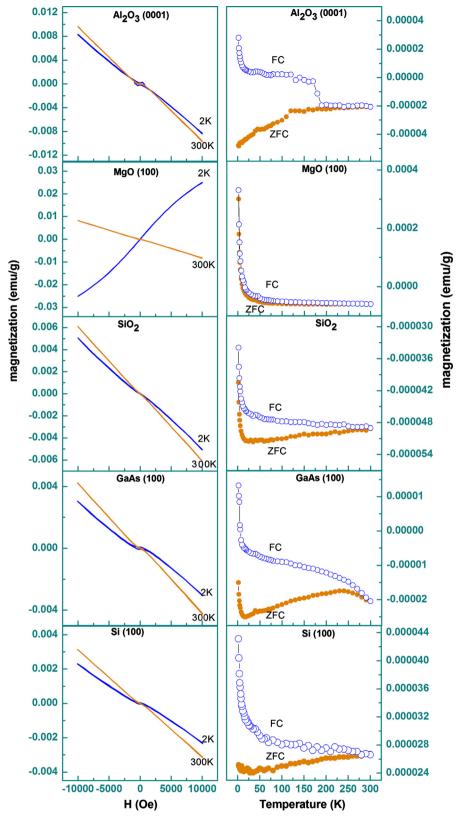


Fig. 3. M-H and ZFC/FC curves of the c-plane Al₂O₃, (100) MgO, fused silica glass (SiO₂), (100) GaAs and (100) Si substrates.

The left side in Fig. 3 shows the M-H curves of c-plane $\mathrm{Al}_2\mathrm{O}_3$, (100)MgO, fused SiO_2 , (100) GaAs and (100) Si commercial substrates, which were measured in the magnetic-field region of 0–10,000 Oe at 2 K and 300 K, respectively. The thickness of these commercial substrates is about 0.5 mm. The ZFC/FC curves of

these substrates are shown on the right side in Fig. 3. The field was applied parallel to the substrate plane. After cooling to $2\,\mathrm{K}$ at the fields of 0– $100\,\mathrm{Oe}$, respectively, the measurements were performed at a 100-0e DC magnetic field with increasing temperature.

Table 1 Magnetization measured at 2 K and 300 K under a field of 10,000 Oe for the c-plane Al_2O_3 , (100) SrTiO₃, fused silica glass (SiO₂), (100) MgO, (100) GaAs and (100) Si substrates.

| | Al ₂ O ₃ (001) | SrTiO ₃ (100) | SiO ₂ | MgO(100) | GaAs (100) | Si(100) |
|-------|--------------------------------------|--------------------------|------------------|----------|------------|---------|
| | emu/g | emu/g | emu/g | emu/g | emu/g | emu/g |
| 2 K | 0.0083 | 0.0054 | 0.0051 | 0.0251 | 0.0031 | 0.0023 |
| 300 K | 0.0098 | 0.0065 | 0.0061 | 0.0082 | 0.0042 | 0.0031 |

Similar with the case of $SrTiO_3$, both diamagnetic and ferromagnetic characteristics were observed in the Al_2O_3 , SiO_2 , GaAs and Si substrate crystals, as depicted by the M-H curves. Correspondingly, a bifurcation between the ZFC and FC curves takes place during decreasing the temperature, as shown in the right side in Fig. 3. It indicates a competition between ferromagnetism and diamagnetism in the materials. This phenomenon is originated from the remanence frozen at low temperature. Note that the ferromagnetic Curie temperature of all the above substrates is higher than 300 K. It means that the ferromagnetism is originated from the impurities or defects.

Being different with the above substrates, MgO crystal shows almost no ferromagnetic signal in the *M*–*H* curve. It shows diamagnetism at 300 K and paramagnetism at 2 K, respectively. In general, the paramagnetic component is mainly caused by the magnetic ions occupying lattice sites with 1–100 ppm concentration [10]. Due to no moment frozen at low temperature, the ZFC and FC curves of MgO almost superpose each other.

Table 1 lists the magnetization for the above six substrates, which were measured at 2 K and 300 K under a field of 10,000 Oe, respectively. There are considerable differences among these magnetization data.

4. Conclusions

A 100-nm-thick LSMO thin film deposited on (100) $SrTiO_3$ substrate was characterized by M-H, M-T and ZFC/FC

measurements. After subtracting the background from the $SrTiO_3$ substrate, the magnetic signals of the LSMO thin film show remarkable changes compared to the as-measured results. Similar with $SrTiO_3$, some commercial substrates, including c-plane Al_2O_3 , (100)MgO, fused silica glass, (100) GaAs and (100) Si, show various magnetic properties with different intensities. Therefore, in characterizing magnetic thin films, it is necessary to take into account the background magnetic signals from the substrate, even to subtract them when the signals from the thin film and substrate are of the same order of magnitude.

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References

- [1] Gary A. Prinz, Science 282 (1998) 1660.
- [2] K. Fujiwara, M. Oogane, S. Yokota, T. Nishikawa, H. Naganuma, Y Ando, Journal of Applied Physics 111 (2012) 07C710.
- [3] Feng Wu, Shigemi Mizukami, Daisuke Watanabe, Hiroshi Naganuma, Mikihiko Oogane, YasuoAndo, Terunobu Miyazaki, Applied Physics Letters 94 (2009) 122503.
- [4] J.D. Adam, S.V. Krishnaswamy, S.H. Talisa, K.C. Yoo, Journal of Magnetism and Magnetic Materials 83 (1990) 419.
- [5] G.T. Thaler, M.E. Overberg, B. Gila, R. Frazier, C.R. Abernathy, S.J. Pearton, J. S. Lee, S.Y. Lee, Y.D. Park, Z.G. Khim, J. Kim, F. Ren, Applied Physics Letters 80 (2002) 3964.
- [6] C. Adamo, X. Ke, H.Q. Wang, H.L. Xin, T. Heeg, M.E. Hawley, W. Zander, J. Schubert, P. Schiffer, D.A. Muller, L. Maritato, D.G. Schlom, Applied Physics Letters 95 (2009) 112504.
- [7] E.M. Kneedler, B.T. Jonker, P.M. Thibado, R.J. Wagner, B.V. Shanabrook, L. J. Whitman, Physical Review B 56 (1997) 8163–8168.
- [8] L.B Steren, M. Sirena, J. Guimpel, Journal of Magnetism and Magnetic Materials 211 (2000) 28–34.
- [9] J.H. Liao, Y.S. Lo, T.B. Wu, Journal of Crystal Growth 310 (2008) 3861.
- [10] M. Khalid, A. Setzer, M. Ziese, P. Esquinazi, D. Spemann, A. Pöppl, E. Goering, Physical Review B 81 (2010) 214414.