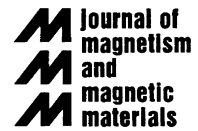




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# Magnetic force microscopy study of $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3/\text{Si}(001)$ around its Curie temperature

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## Abstract

A transform of the surface magnetic domain patterns of a 350-nm-thick LSMO thin film at Curie temperature ( $T_c$ , 340 K) was first observed by magnetic force microscopy (MFM) at its remanent state. This result is in good agreement with that of the SQUID measurements; the in-plane magnetization of this thin film declined with an increase in temperature and the  $T_c$  was measured at 340 K. Therefore, the in situ MFM domain study is found to be a useful tool for thermo-magnetic behavior for thin films of this kind. © 2001 Published by Elsevier Science B.V.

**Keywords:** Sputtering—magnetron; Thin films—sputtered; Curie temperature; SQUID; Hysteresis loop; Coercivity; Domain pattern; Magnetization decay; Magnetic force microscopy; Remanence curves

## 1. Introduction

$\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$  (LSMO) has recently attracted much attention, since perovskite-type manganese oxides exhibit very large magnetoresistance (CMR) and a metal–insulator transition [1]. Curie temperatures ( $T_c$ ) of LSMO thin films are dependent on strain [2,3], granular structures and granular sizes [4]. In this experiment a superconducting quantum interference device (SQUID) magnetometer was used to measure magnetic hysteresis loops of a 350-nm-thick thin film of  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  on Si(001) at temperatures from 300 to 350 K (Fig. 1). Magnetic domain wall scattering may play a leading role in determining  $T_c$  [5]. Transforms of surface magnetic domain patterns of the LSMO thin film can thus be observed by means of comparison between images taken by atomic force microscopy (AFM) and magnetic force microscopy (MFM) at room temperature and elevated temperatures. Seldom in the literature has MFM been applied at elevated temperature measurements. The

purpose of this study is to correlate this observation with the SQUID measured data.

## 2. Experiments

A 350-nm-thick LSMO thin film was deposited on a native  $\text{SiO}_2$  layer of Si(001) from a stoichiometric  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  target, using an off-axis DC magnetron sputtering system. This LSMO thin film was sputtered for 3 h at approximately 750°C in 100 mTorr of a mixture of Ar and  $\text{O}_2$  (5:1), and then post-annealed at 750°C in 100 Torr of  $\text{O}_2$  for 1 h. The temperature of the sample was then gradually reduced at 2°C/min to room temperature. An SQUID magnetometer (MPMS-5S, Quantum Design, San Diego, USA) was used to measure hysteresis loops at various temperatures from 300 to 360 K. The observation of AFM and MFM (Solver-P47 model, NT-MDT, Moscow, Russia) was performed at room temperature and temperature above 340 K ( $= T_c$  of the LSMO thin film). The LSMO thin film was demagnetized and then magnetized by an in-plane magnetic field of 1000 Oe. AFM and MFM images were then obtained in the tapping mode using commercial MFM cantilevers (Pointprobe sensor type,

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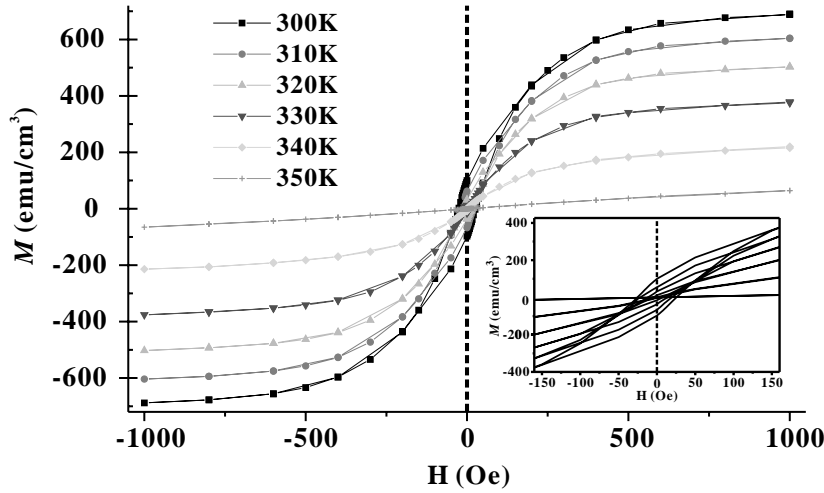


Fig. 1. The hysteresis loops of the LSMO thin film at various temperatures. The inset shows the areas of the hysteresis loops.

Nanosensors, Wetzlar-Blankenfeld, Germany) with the magnetic moment parallel or anti-parallel to the off-plane direction.

### 3. Results and discussion

The 350-nm-thick LSMO thin film on the native  $\text{SiO}_2$  layer was first demagnetized and then magnetized under an in-plane magnetic field for the SQUID measurements. The magnetic hysteresis loops of the LSMO thin film are presented in Fig. 1 at various temperatures, from 300 to 350 K. The hysteresis loops show that the area of the loop decreases with an increase in temperature and is nearly zero above 350 K (see the inset in Fig. 1). The in-plane magnetic remanence ( $M_r$ ) at room temperature is  $98.7 \text{ emu}/\text{cm}^3$  ( $\approx 14.3\%$  of the saturation magnetization,  $M_s = 690.5 \text{ emu}/\text{cm}^3$ ), and then decreased to  $64.9 \text{ emu}/\text{cm}^3$  ( $\approx 10.7\%$  of  $M_s = 604.8 \text{ emu}/\text{cm}^3$ ) at 310 K,  $34.2 \text{ emu}/\text{cm}^3$  ( $\approx 6.8\%$  of  $M_s = 503.6 \text{ emu}/\text{cm}^3$ ) at 320 K,  $13.6 \text{ emu}/\text{cm}^3$  ( $\approx 3.9\%$  of  $M_s = 378.6 \text{ emu}/\text{cm}^3$ ) at 330 K, and 0 ( $M_s = 220.2 \text{ emu}/\text{cm}^3$ ) at 340 K, respectively. In addition, the coercive fields,  $H_c$ , are 24.8 Oe at room temperature, 20.3 Oe at 310 K, 12.8 Oe at 320 K, 6.8 Oe at 330 K and 0 Oe at 340 K, respectively. Two curves in Fig. 2 are presented for the declines in  $M_r$  and  $H_c$  with an increase in temperature. The results show that the  $T_c$  of the LSMO thin film is 340 K, and this LSMO thin film is a soft magnetic material.

The LSMO thin film was demagnetized and then saturation-magnetized under an in-plane magnetic field to support AFM and MFM observations. Fig. 3 shows AFM and MFM images of the LSMO thin film surface, acquired at the remanent state. The AFM images, in Fig. 3(a) and (b), illustrate the surface topography at

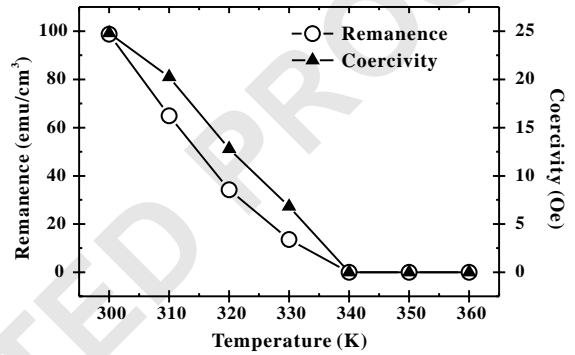


Fig. 2. A graph showing two curves presented for the declines of  $M_r$  and  $H_c$  with an increase in temperature, respectively.

room temperature and 340 K, respectively. These AFM images correspond, one to one, to the MFM images in Fig. 3(c) and (d). A curve in Fig. 4 shows that the surface roughness keeps unchanged ( $\sim 3 \text{ nm}$ ) by increasing the temperature over a small range, from 300 to 340 K; thus, in this study granular structures and sizes are not factors governing the  $T_c$  of the studied LSMO thin film.

However, the MFM images show a transform of the magnetic domain patterns on the surface of the LSMO thin film occurred by comparing that at room temperature with that at 340 K. The feature in Fig. 3(c) depicts the magnetic domain patterns on the LSMO thin film surface at room temperature at its remanent state, where  $M_r$  is  $98.7 \text{ emu}/\text{cm}^3$  ( $\approx 14.3\%$  of  $M_s = 690.5 \text{ emu}/\text{cm}^3$ ). The sizes of the magnetic domain patterns are several microns. Above 340 K, the magnetic domain patterns had disappeared, as shown in Fig. 3(d). Note that the magnetic field lines in the MFM images radiate outward

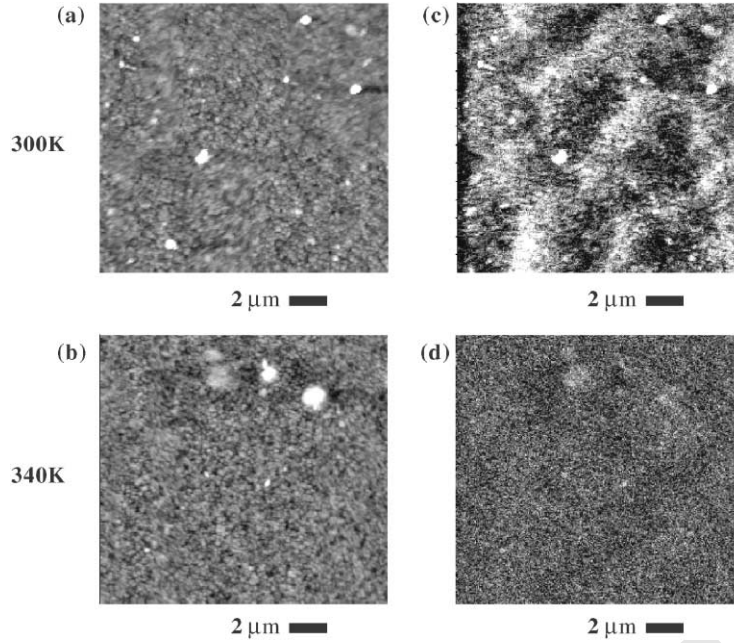


Fig. 3. AFM (a) and (b), and MFM (c) and (d) images ( $15.6 \times 14.6 \mu\text{m}^2$ ) depicting the topography and magnetic domain patterns (at its remanent state) of the LSMO thin film surface at room temperature and at 340 K, respectively.

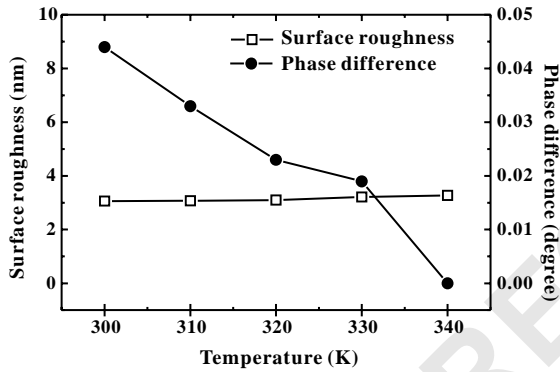


Fig. 4. A graph showing the surface roughness of AFM images and the phase difference of MFM images, respectively, with an increase in temperature from 300 K to 340 K.

from the dark regions and return to the bright regions on the sample's surface (the magnetic moment of the MFM tips is suggested to be parallel to the normal of the sample's surface). Therefore, the surface magnetic contrast is the phase difference of MFM image between the bright and dark regions, corresponding to upward and downward magnetizations, respectively. To understand in more details the relation between the magnetic domain patterns and  $T_c$ , the other curve in Fig. 4 shows the phase difference (= surface magnetic contrast) of the magnetic domain patterns that degrades as the temperature is increased. The phase differences are  $4.4 \times 10^{-2}$

degree at room temperature,  $3.3 \times 10^{-2}$  degree at 310 K,  $2.3 \times 10^{-2}$  degree at 320 K, and  $1.9 \times 10^{-2}$  degree at 330 K, respectively, and entirely faded away as the temperature is 340 K and above. It indicates that no magnetic field line leaves or enters the surface above 340 K, so that the  $T_c$  of the LSMO thin film is determined to be 340 K by this method. This result is consistent with that of Fig. 2. Accordingly, the  $T_c$  of the LSMO thin film is successfully delineated by the in-situ surface magnetic domain patterns.

#### 4. Conclusions

The in-plane magnetization of the 350-nm-thick LSMO thin film was found to decay with an increase in temperature and the  $T_c$  was measured to be at 340 K by the in-situ MFM method. At its remanent state MFM was able to reveal for the first time a transform of the surface magnetic domain patterns at temperature above  $T_c$ . The degradation of the surface magnetic domain patterns was quantitatively related to the temperature-dependant magnetization of the LSMO thin film.

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