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Substrate and Growth Related Microstructural and Magnetic Properties in $La_{0.67}Sr_{0.33}MnO_3$ Thin Films

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ABSTRACT

Ambient observation of magnetic domain structures by magnetic force microscopy (MFM) in $La_{0.67}Sr_{0.33}MnO_3$ films has not yet been clearly correlated with stresses induced by kinetic or thermodynamic growth processes or the compressive ($LaAlO_3$) or tensile ($SrTiO_3$) nature of the film-substrate lattice mismatch. Although domain-like magnetic structures have been seen in some as-grown films and related to substrate-induced stress and film thickness, no magnetic structure has been seen for other films grown under similar conditions on the same pair of substrates. In this study we have grown films over a range of temperatures by pulsed-laser deposition, using the above substrates, to determine the relationship between growth and stress-induced magnetic structures. Results from scanning tunneling, atomic force, and magnetic force microscopies, measurements of temperature-dependent magnetization and structure-dependent coercivity show the relationship between growth and magnetic properties. Maze-like domain structures, with separations between 150 nm and 200 nm, were only observed for the thicker films grown at the highest temperature, 800°C. Application of an in-plane magnetic field converted these domain structures to stripe-like domains whose spacing and out of plane component decreased as the field was increased.

Keywords: MFM, STM, magnetic imaging, temperature-dependent growth, coercivity, CMR, magnetoresistance, LSMO

1. BACKGROUND

Recent discovery of extremely large negative magnetoresistance[1] in Ca, Ba, and Sr doped lanthanum manganites has sparked wide-spread interest in their potential application in technologically and industrially important magnetic sensors, particularly the next generation of read heads for high-density magnetic data storage. The 33% hole-doped Sr and Ba analogs in thin film form are of particular interest because they possess Curie temperatures (T_c's) above room temperature (~ 380°C for the Sr doped material). Unfortunately, substantial fields are required to take advantage of the entire drop in resistance(50 kOe field decreased ρ by more than 50% for a Ca-doped sample)[2], hindering further development of applications. The interplay between ordered magnetism and electronic transport, which produces the colossal magnetoresistive (CMR) effect, presents an exciting and important challenge to the materials research community. Determining the underlying mechanisms at work in these compounds is an important step towards reducing the field required to observe the CMR effect.

This renewed interest in these particular perovskites has been driven by the need for more sensitive magnetic sensor materials and/or structures to go hand-in-hand with the development of higher density magnetic data storage. The higher bit packing is achieved at the expense of bit size. This translates into weaker stray fields with a finer pitch that need to be detected. Although a great deal of focus has been put on understanding the intrinsic properties of these materials, an equally important area of research is the study of the extrinsic properties induced in thin films through the grow process. That is the focus of the present work

Film processing parameters and supporting substrates play an important role in optimizing properties: ferromagnetic (T_c) and metal-insulator (T_p) transition temperatures, magnetization, transport properties, magnetic hysteretic behavior (remanent magnetization and coercivity field), and the presence or absence of domain structures. The latter impacts on the residual resistivity through spin scattering and on magnetization through coercivity. Although these materials have a cubic symmetry (excluding local Jahn-Teller distortions) and, therefore, no overall magnetocrystalline anisotropy, they have relatively large magnetostriction constants (~10^{-4}). Therefore, stress introduced into the growing film can lead to magnetic domains through the magnetoelastic effect.[3] Such maze-like domains have been observed under ambient conditions by magnetic force microscopy (MFM) for some $La_{0.67}Sr_{0.33}MnO_3$ (LSMO) films grown on $LaAlO_3$ (LAO)[4,5] which produces a compressive lattice-mismatch with these materials but not on films grown on $SrTiO_3$ (STO) which produces a tensile mismatch. The belief is that substrate-induced stress plays an important role in the all the measured properties.

Substrate temperature (T_S) was chosen as a variable because of its well-known relationship to surface diffusion controlled grain size and epitaxial quality. Since these materials are nominally cubic with no magnetic easy axis, no intrinsic magnetic domain structure and little or no coercivity is expected. Also because of shape effects due to the thin film form, no out-of-plane magnetization is expected. However, depending on the sign, magnitude, and distribution of residual stress in the films due to lattice-mismatch with the substrate, spins can be aligned in, canted to, or normal to the film surface. Although domain-like magnetic structures have been seen in some as-grown films and related to substrate-induced stress and film thickness, no magnetic structure has been seen for other films grown under similar on the same pair of substrates.

In addition to the specific applications relevant to these particular manganite materials, the growth issues raised here are important to optimization of properties for all oxide films used in device applications including superconductors. There is a clear and compelling need to understand how growth parameters and template affect extrinsic properties.

2. EXPERIMENT

LSMO films were deposited simultaneously on LAO and STO substrates at T_S = 550°C, 650°C, 700°C, 750°C, and 800°C using a XeCl excimer laser (λ = 308 nm) incident on a stoichiometric target. Pulse repetition rate, width, and energy were 10 Hz, 20 ns, and 2 J/cm^2 respectively. The depositions were carried out in 400 mTorr of oxygen. After deposition, samples were allowed to cool in the chamber under 1 atmosphere oxygen. No thermal anneal was performed on the samples after the deposition. For characterization purposes, multiple samples were grown at the same time. To minimize variations in structure and properties due to differences in substrates, all samples were grown on pieces of the same single substrates of each material that had been broken into enough pieces to accommodate the requirements of the different characterization techniques.

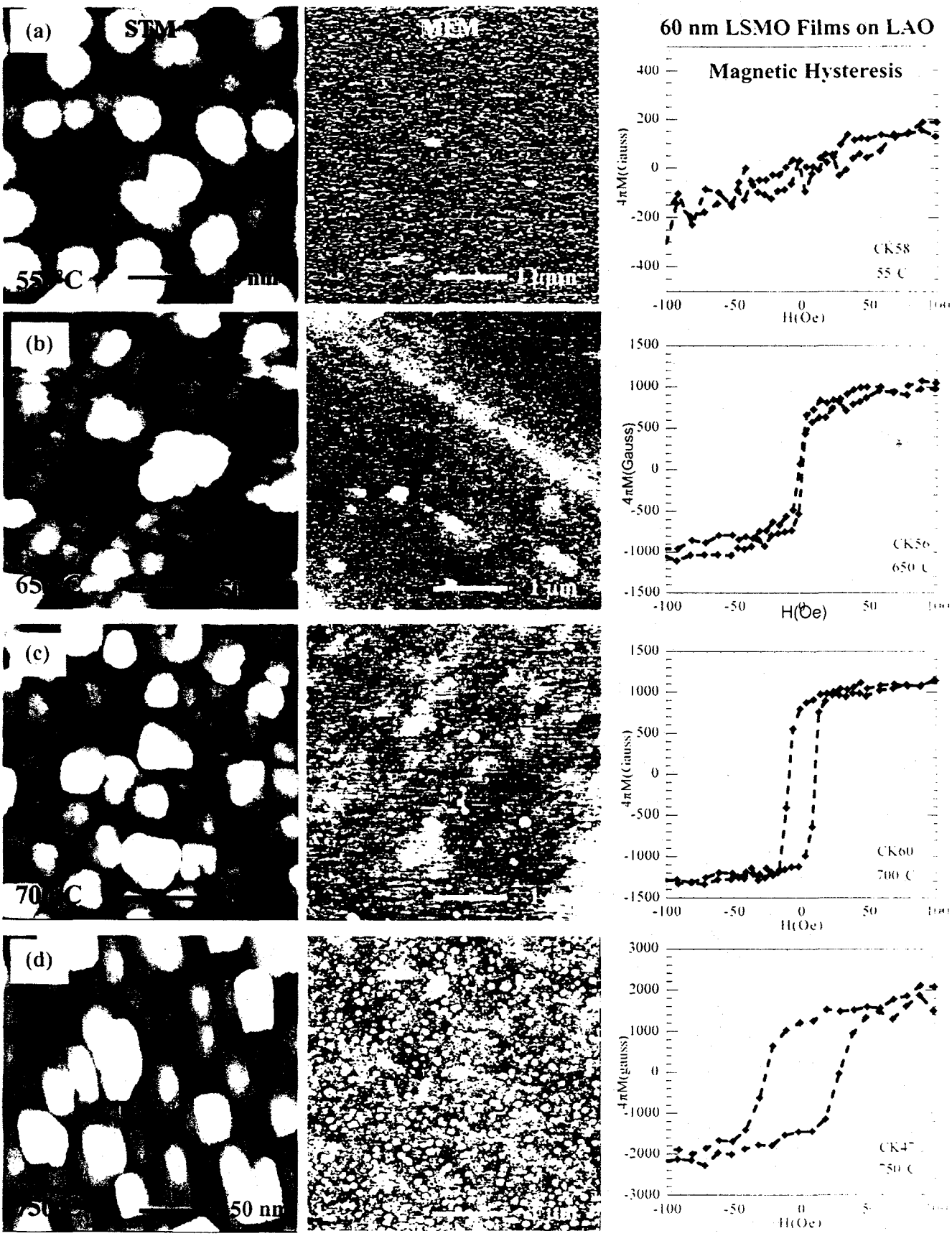
Because variations in deposition rate depended strongly on a number of factors that included the substrate position on the PLD sample holder chuck and changes in the laser output power, film thicknesses were determined after deposition by etching away part of the LSMO with a mixture of HNO_3 and HCl and measuring the step height with a stylus profilometer.

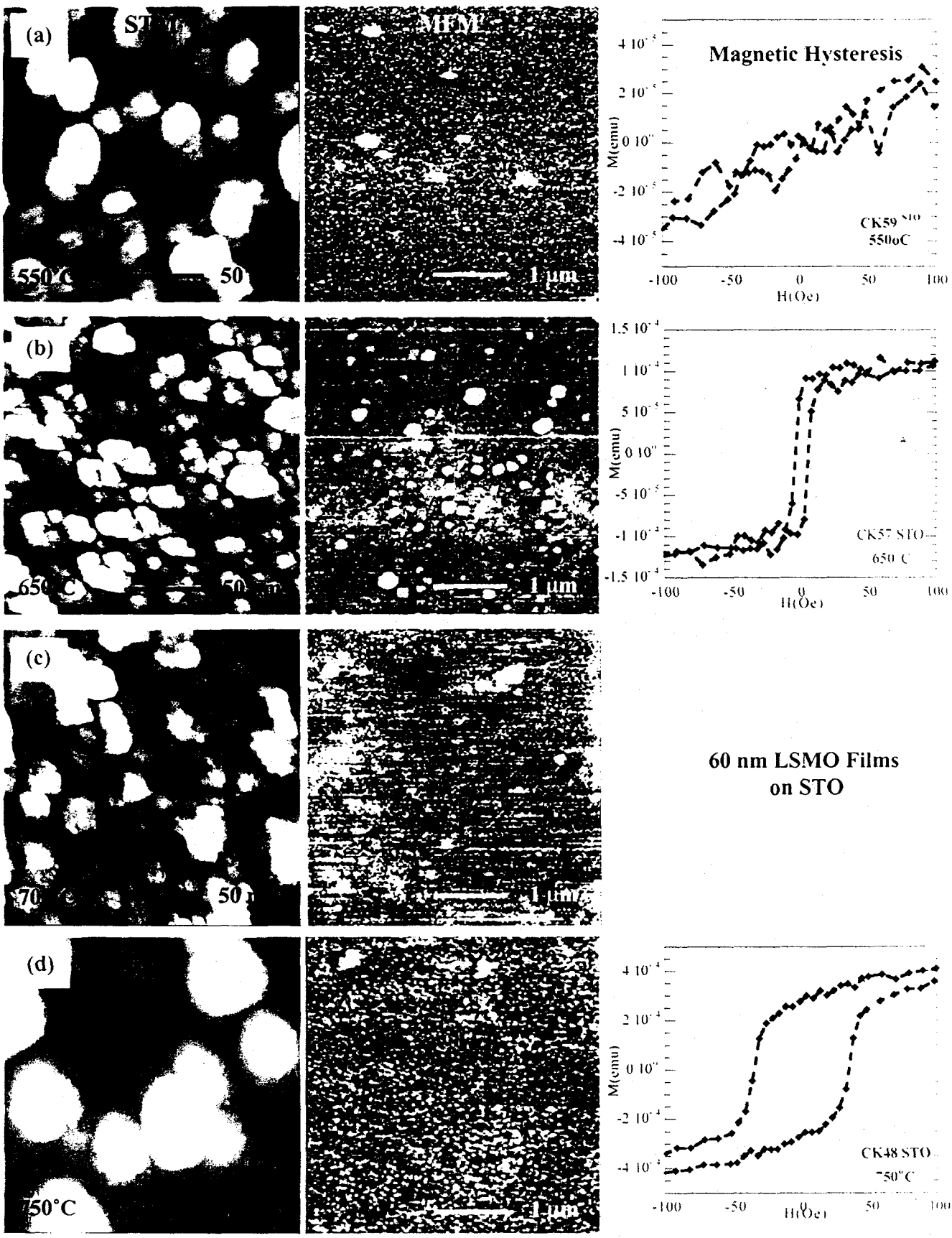
Crystallinity and out-of-plane lattice constants (*c*-axis lattice constant) were characterized with a four circle x-ray diffractometer. Hysteresis loops were obtained at room temperature (RT) using a vibrating sample magnetometer (VSM). These plots provide the film coercivity as a function of growth temperature and substrate. Temperature-dependent magnetization M(T) was measured with a SQUID magnetometer. Magnetization was measured with 100 Oe applied field. For both VSM and SQUID measurements, the field was applied along an in-plane direction of the sample.

The microstructure was characterized by scanning tunneling microscopy (STM) at ~1.4 eV tip bias and 140 pA tunnel current. Magnetic force microscopy (MFM) measurements, which are sensitive to the out-of-plane component of the magnetization, were used to obtain qualitative measurements of magnetic domain structures including their relative strength and orientation[6,7]. The magnetic force gradient was measured using frequency-shift detection with high coercivity Co-Cr coated Si tips. This tapping-mode lift-mode technique, was used to gain the higher sensitivity of the gradient measurement technique. The dark contrast in the MFM images corresponds to regions polarized parallel to the tip polarization (attractive interaction). The light regions are oriented antiparallel to the tip polarization (repulsive interaction). Only qualitative information can be obtained by this technique since the tip's moment and geometry are not precisely known. MFM measurements were also made in the presence of an in-plane external field (160 Oe and 320 Oe) to determine the field-dependence of the domains present in some films.

3. RESULTS

A summary composite of microstructure, MFM data, and magnetic hysteresis measurements for the LAO-based films is shown in Figure 1, and for the STO-based films in Figure 2 for films grown in the temperature range of 550 °C to 750 °C. Note (Table I) that the above films are ~60 nm thick while the 800 °C films are >100 nm thick. The 200 nm × 200 nm STM topographs reveal a similar trend for both sets of films (LAO and STO), i.e. increasing grain size and faceting with increasing temperature, as expected. Although the grains comprising the 550 °C films appear larger than those of the 600°C films, they are actually clusters of smaller grains. This is consistent with a low surface mobility at this temperature, also observed previously for the Ca-doped $LaMnO_3$ films[8]. One exception to the temperature-dependent grain size trend, and unexplained at this time, was the 750 °C STO film which appeared to have 3-D growth on top of the base film.


(a)
STM
MFM
50 nm
(b)
1 μm
(c)
(d)
50 nm
60 nm LSMO Films on LAO
Magnetic Hysteresis
4πM(Gauss)
H(Oe)
CK58
55 C
CK56
650 C
CK60
700 C
CK47
750 C


(a)
STM
MFM
550°C
50
1 µm
Magnetic Hysteresis
M(emu)
H(Oe)
CK59 STO
550oC
(b)
650 C
1 µm
CK57 STO
650 C
(c)
70
50
1 µm
60 nm LSMO Films
on STO
(d)
750°C
1 µm
CK48 STO
750°C

One can see from Table I that the 800 °C LAO films possessed the broadest x-ray peaks, i.e. the largest full-width-at-half-maximum (FWHM). Most of the 800 °C films (both substrates) consisted of incomplete coalesced layers interspersed with shallow voids; in all cases the top layers were partially covered by faceted crystalline-like islands. 200 nm × 200 nm STM images of the three LAO films are shown in Figure 3a. One of these STM images and one from a film grown on STO are shown in Figure 4a and 4b, respectively. This microstructure was very similar to that seen previously for LCMO films grown above 800 °C and for both LSMO and LCMO films grown at 500 °C on the same two substrates and post-annealed for 10 hours at 950 °C in oxygen. The higher temperatures allow greater surface mobility but not enough to obtain completely coalesced layers as was seen for films grown at ≥ 650 °C and annealed as described above. Those annealed films became completely layered without the presence of shallow voids[8].

Although all the lower temperature thin LAO films had elongated c-axis parameters, consistent with in-plane compressive stress, none of these films had significant out-of-plane magnetization. As one can see in Figures 1 and 2, there was a slight increase in out-of-plane magnetization (LAO), along with an increase magnetization, T_c, and coercivity as the growth temperature (grain size) increased. No significant change in out-of-plane magnetization was expected or observed for the STO films which had an in-plane tensile stress. The observed out-of-plane component of the magnetic structure for both STO- and LAO-based films was reproducible, but weak and diffuse. T_c's, shown in Table I, were obtained from T-dependent magnetization and increased with T_S. Even though all the films had T_c's above RT, i.e. they were all at least partially ferromagnetic, the lowest temperature films had weak or broad transitions. Additionally, the T_c's were all below that measured for bulk samples (over 270 °C). There seems to be a clear correlation between grain size and hysteretic behavior: As the grain size increased, the hysteresis loop opened and the coercivity increased, Table I. The largest coercivity was obtained for the 800 °C films which did not have discrete islands or grains as described below.

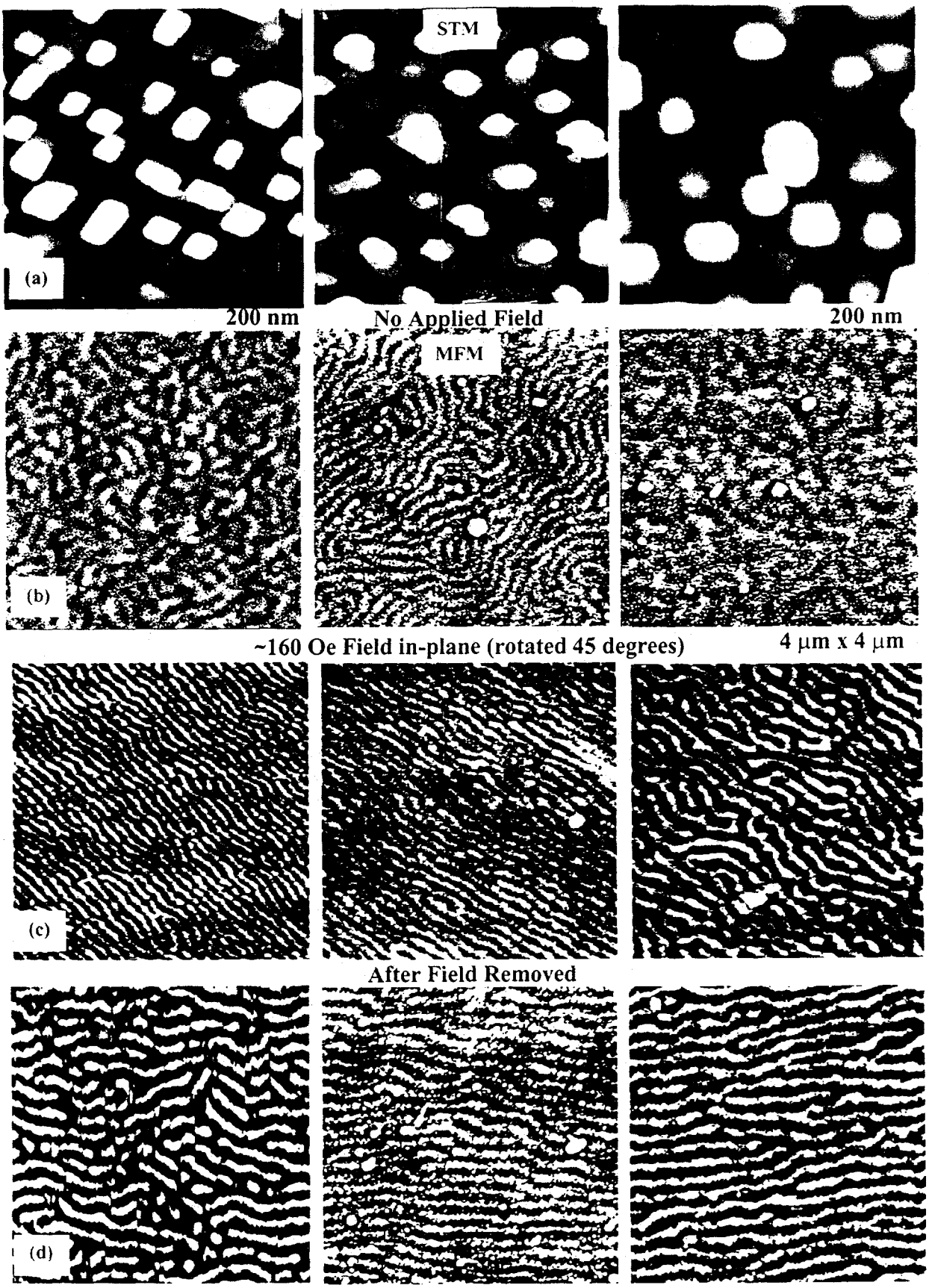
Table I. C-axis parameter, thickness, and T_c for the films grown on LAO single crystal substrates.

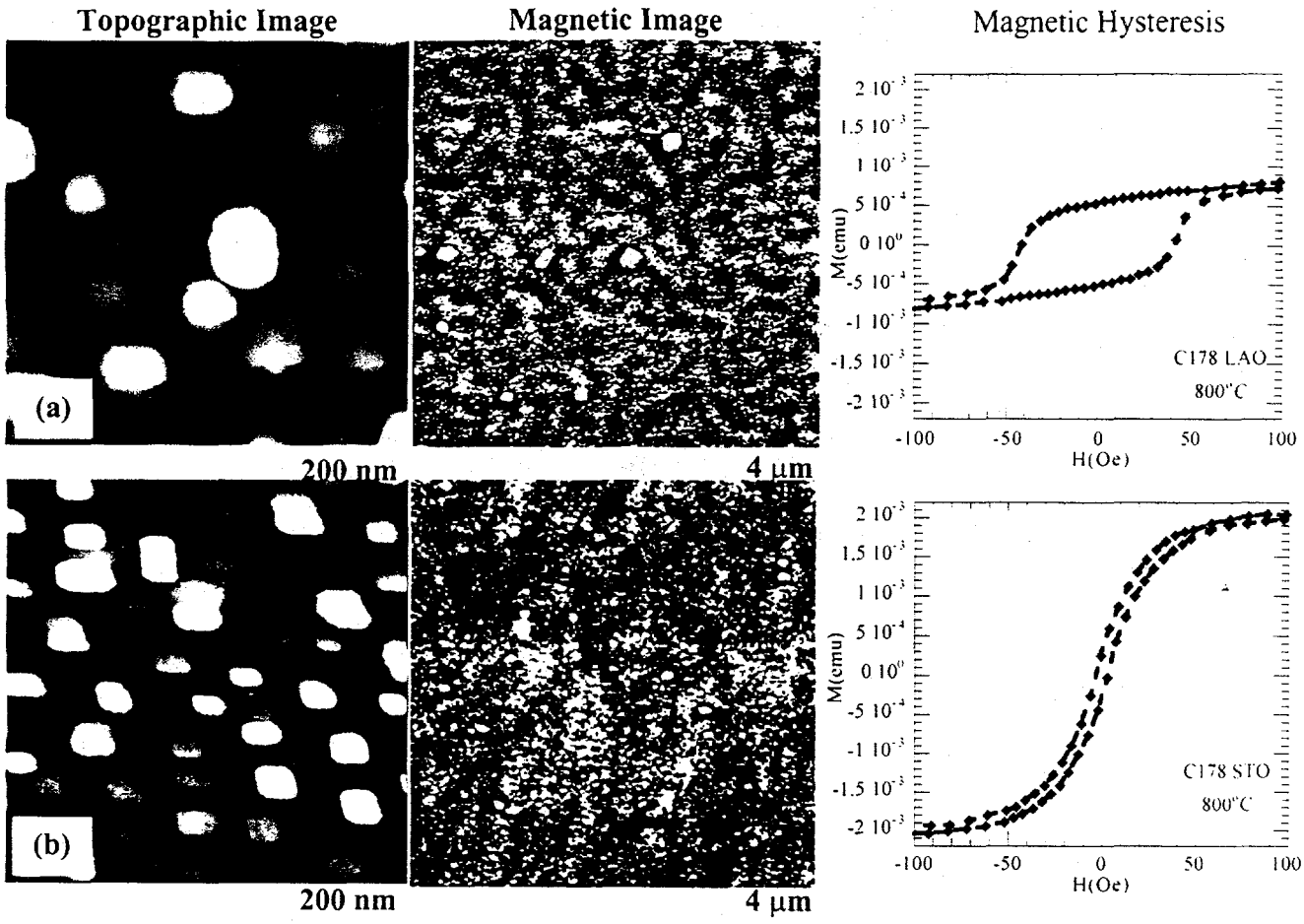
T_S	550 °C	650 °C	700 °C	750 °C	800 °C Deposition Time 3 min.	5 min.	5 min.
c-axis (Å)	3.9426	3.8750	3.8911	3.9136	3.8962	3.9122	3.9110
FWHM (degrees)	0.296	0.3362	0.2986	—	0.6393	0.5983	0.4332
% change	+1.72	-0.03	+0.39	+0.97	+0.52	+0.93	+0.90
Thickness (nm)	~ 60	~ 60	~ 60	~ 60	~130	~170	~170
T_c ±5 (°K)	325*	335**	345	355	355	355	—
H_c (Oe)	0	~2	~10	~30	—	~40	—

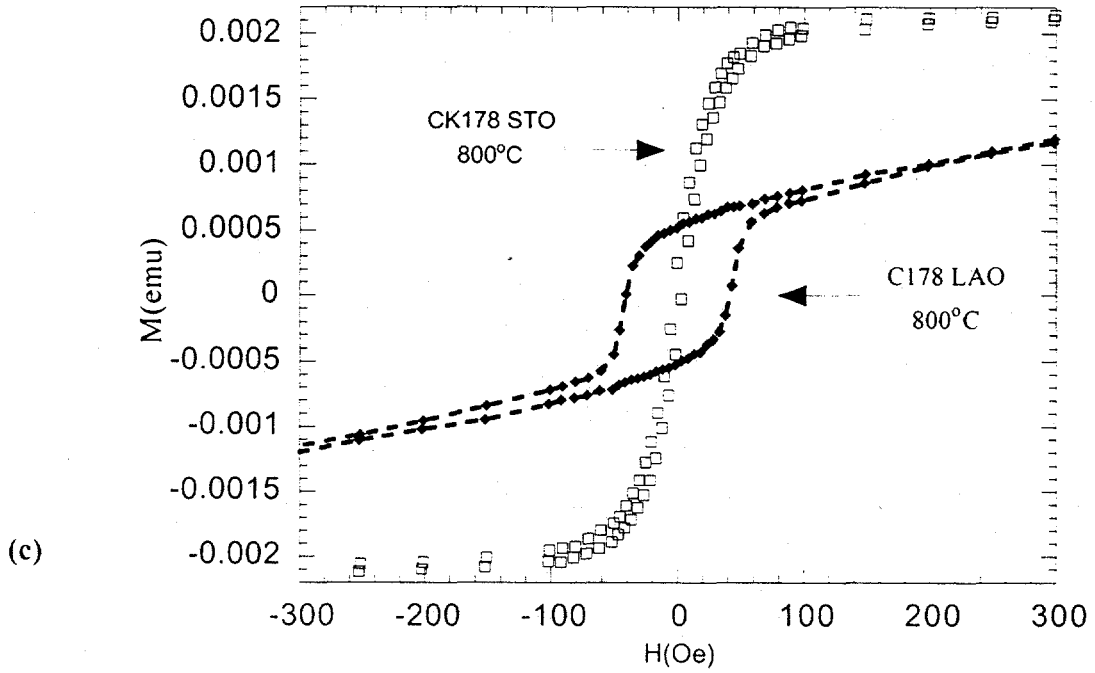
LSMO 3.876, *weak, **broad transition, --- not measured.

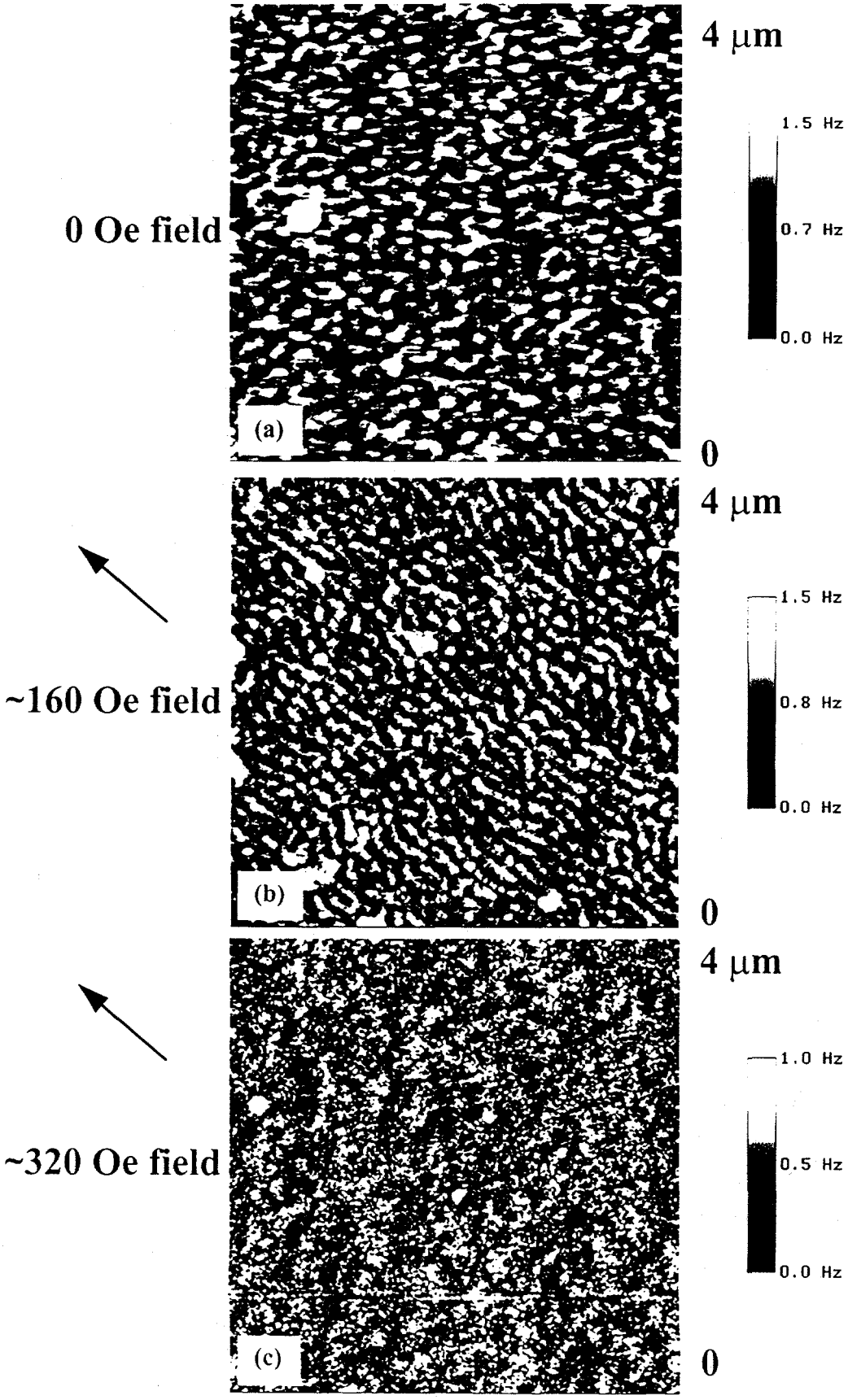
Three of the four 800 °C LAO-based films possessed maze-like domains structures (Figure 3b) which transformed into stripe domains in an in-plane external static field (Figure 3c); the stripe domains were oriented along the field direction. At the same time the spacing between domain walls decreased but remained uniform. The stripe domains in Figure 3b are at angle to the image edges because the 160 Oe field and the samples were oriented approximately 45° to the scan direction. After removal of the external field, the stripe domains remained but the spacing returned to that of the original maze wall spacing (Figure 3d). These images were taken without the sample rotated which is why the stripes are now oriented horizontally. A comparison of the microstructure, magnetic structure, and coercivity and hysteretic behavior between one of these LAO-based LSMO films and one grown simultaneously on STO is shown in Figure 4a, b, and c. While the STO film had a $H_c \sim 0$, the LAO had a H_c ~50 Oe and a considerably smaller total magnetization. Although the microstructures look somewhat different in this figure, the other two LAO films shown in Figure 3a are very similar to the STO film. The magnetic structure is quite different, the LAO film having a maze-like domain structure and STO film a weak diffuse one.

The magnetic structure (Figure 5) for one additional film grown on LAO at 750 °C and capped with 100 Å of STO was also studied. This particular film was intended for a spin tunneling device[10]. A 160 Oe field transformed the maze-like structure (Figure 5a) of this film into stripe domains (Figure 5b) while a 320 Oe field forced the spins almost entirely into the plane of the film (Figure 5c), leaving a weak diffuse magnetic structure reminiscent of that of the thin films and all films grown on


STM
(a)
200 nm
No Applied Field
200 nm
MFM
(b)
~160 Oe Field in-plane (rotated 45 degrees)
4 μm x 4 μm
(c)
After Field Removed
(d)


Topographic Image
Magnetic Image
Magnetic Hysteresis
(a)
200 nm
4 µm
M(emu)
H(Oe)
C178 LAO
800°C
(b)
200 nm
4 µm
M(emu)
H(Oe)
C178 STO
800°C


(c)
CK178 STO
800°C
C178 LAO
800°C
M(emu)
H(Oe)
0.002
0.0015
0.001
0.0005
0
-0.0005
-0.001
-0.0015
-0.002
-300
-200
-100
0
100
200
300


4 μm
1.5 Hz
0 Oe field
0.7 Hz
0.0 Hz
(a)
0
4 μm
1.5 Hz
~160 Oe field
0.8 Hz
0.0 Hz
(b)
0
4 μm
1.0 Hz
~320 Oe field
0.5 Hz
0.0 Hz
(c)
0

STO. This process was completely reversible. Removal of either the 160 Oe or the 320 Oe field led to recovery of the maze-like structure. This reversible behavior was distinctly different from that of the uncapped films.

4. DISCUSSION

The temperature-dependent microstructural evolution from clusters to grains of increasing size, crystal quality and epitaxy were consistent with that expected from increasing surface diffusion with increasing temperature. The x-ray data and measured T_c's indicate that all of the films were crystalline, well oriented, and had close to the correct stoichiometry. Of particular interest in this work is the relationship between the resulting microstructure and the magnetic properties: coercivity, magnetization, and domain structures. A simple direct correlation between grain size and coercivity, unfortunately, cannot be decoupled from issues relating to overall film quality and the unknown impact of this improved epitaxial quality with temperature on all properties. On a superficial level, it appears that there was a grain size effect on the coercivity which may be related to the number of coupled spins per grain that have to be rotated. Whatever the combination of contributing factors, for both LAO and STO films, an increase in both coercivity and magnetization paralleled an increase in T_s.

Compressive stress due to the lattice-mismatch between LSMO and LAO is responsible for the distortion in the film crystal structure (elongation of the c-axis parameter). The crystal distortion creates a crystalline anisotropy and, therefore, a possible magnetic easy axis even though no crystalline anisotropy exists in the bulk cubic material. Since LSMO has a positive magnetostrictive constant, in-plane compressive stress can also lead to an out-of-plane tensile stress tensor created easy axis normal to the plane of the film. One cannot separate these two factors since the in-plane stress was responsible for the crystal distortion. The amount of residual stress in the film after formation of defects, grain boundaries, and other relaxation mechanisms is unknown. The magnetoelastic energy has to be balanced against the magnetostatic energy which minimizes both the stray field escaping the film surface and the overall net magnetization of the material. Shape effects favor in-plane polarization and are particularly important in very thin films. Minimization of the stray field and surface pole energies drives the formation of reverse polarization and closure domains. The observed magnetic domain structure is then a combination of all of these energy considerations. This is certainly the case for films grown on LAO which has a smaller in-plane lattice constant than the film. The c-axis parameter for all of the LAO films grown in this study was, in fact, elongated. The lack of clear domain structure in the MFM data for the thin films, despite the distortion and the high T_c's, means that, although the films were at least partially ferromagnetic, the magnetization was primarily confined to the film plane. Most of the thick films, however, had considerable out-of-plane magnetic structure which consisted of either maze-like or a combination of maze- and bubble-like structures. The large normal component to the magnetization was consistent with the combination of broader x-ray peaks and large c-axis parameters found for these films. For the thin films, magnetostatic energy or shape effect won out over the magnetoelastic energy. However, for the thicker films a critical thickness was reached where the magnetoelastic energy won, that is the magnetoelastic energy has overcome the magnetostatic contribution.

The effect of stress due to the difference in sign of substrate-film lattice mismatch is clearly shown in Figure 4a and b. The magnetic structure for the STO film, which has a tensile mismatch with the LSMO film, is primarily confined to in the plane of the film as would be expected for a positive magnetostrictive film. The easy axis would be the tensile direction. In this case the shape effect and the stress would work together to keep the spins in the plane. Not understood yet is why there must still be some canting of the spins to create the observed diffuse structure. An in-plane magnetic field of either 160 or 320 Oe had little effect on this structure.

The MFM, although a powerful technique for imaging out-of-plane magnetization, cannot, however, directly reveal information about in-plane magnetic structure. However, field-dependent measurements can reveal some information about the in-plane polarization. This is clearly shown in Figure 5 with the field-dependent magnetic structure for the film grown on LAO and capped with a STO film. In this case, the transformation of the maze domain structure into stripe domains with uniform spacing indicates that the in-plane domains are not antialigned. If they were, domains with a primary magnetization component aligned with the field would grow at the expense of those that were not. The narrowing of the domain spacing was accompanied by a reduction in the intensity of the features in the MFM image, i.e. reduction in the polarization cant angle: both phenomena must result from a compromise between the various contributions to the magnetic energy. In addition, since the maze structure did not return on removal of the field for samples without an STO cap layer, the magnetic domain structure must be pinned by the cap layer. Even after several cycles in which the in-plane field was increased and then reduced to zero, the magnetic structure was reversible for the film with the cap layer. Magnetic structure is usually a function of history, many local energy minima present, each separated by an energy barrier. This was true for the three 800 °C films;

the stripe domains remained indefinitely under ambient conditions. The observed magnetic domain behavior for the capped film, before and after application of the applied field, shown in Figure 5, could have implications for the use of this particular combination of materials in a spin tunneling device

Future work will focus on better quantify the field- and in-plane crystal orientation-dependent behavior of the magnetic domains and their impact on room temperature transport properties.

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Figure Captions:

1. Microstructure (200 nm x 200 nm STM scan), out-of-plane magnetic force-gradient stray field structure (4 μm x 4 μm MFM image), and VSM hysteresis data for a 60 nm thick LSMO films grown on LAO at different deposition temperatures: a) 550 °C, 650 °C, 700 °C, and 750 °C. Note the increasing contrast in the MFM images due to the increased canting of the spins relative to the film plane.

2. Microstructure (200 nm x 200 nm STM scan), out-of-plane magnetic force-gradient stray field structure (4 μm x 4 μm MFM image), and VSM hysteresis data for a 60 nm thick LSMO films grown on STO at different deposition temperatures: a) 550 °C, 650 °C, 700 °C (hysteresis not shown), and 750 °C.

3. a) Microstructure (200 nm x 200 nm STM scan), and b), c), and d) out-of-plane magnetic force-gradient stray field structure (4 μm x 4 μm MFM image) for >100 nm thick LSMO films grown at 800 °C on LAO. b) is the maze-like magnetic domain structures for the respective films as grown, c) is the magnetic stripe domain structure with a 160 Oe static field applied in-plane oriented approximately 45° to the scan direction, and d) is the magnetic structure after the field was removed. These images were not taken in exactly the same place.

4. Microstructure (200 nm x200 nm STM scan), out-of-plane magnetic force-gradient stray field structure (4 μm x 4 μm MFM image), and VSM hysteresis data for two LSMO films grown at 800 °C on a) LAO and b) STO. A composite of the VSM data for both films is shown in c) over a wider (300 Oe) +/- field range. Note that the STO film is clearly coalesced into extended layers and that the initial magnetization if in-plane while for LAO film the large surface grains make it difficult to tell whether or how much of this film has coalesced and the domain structure in oriented normal to the surface.

5. a) Out-of-plane magnetic force-gradient stray field structures (4 μm x 4 μm MFM image) for one LSMO film grown at ~750 °C on LAO then capped with approximately 100 Å of STO. a) is the maze-like magnetic domain structures for the film as grown, b) is the magnetic stripe domain structure when a field of ~160 Oe was applied in-plane, oriented approximately 45° to the scan direction, and c) is the magnetic structure when the field was increased to ~320 Oe. The behavior of this film differed from that of the other LAO films with maze-like domain structure. The magnetic structure was reversible, i.e. the maze structure returned when the field was removed.