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Defect Analysis in $La_{0.7}Sr_{0.3}MnO_3$ Epitaxial Thin Films by Electron Channeling Contrast Imaging (ECCI)

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Quantitative analysis of extended defects opens a window to understanding their generation and evolution, as well as their effects on bulk properties. The challenge remains to map and quantify defects with statistical significance, especially over a large scale and nondestructively. Transmission electron microscopy (TEM) is limited to the nanometer scale and is destructive. X-ray diffraction (XRD) provides averages over a large scale and requires significant calibration. Electron channeling contrast imaging (ECCI), however, offers a straightforward and non-destructive way to image extended defects over micron length scales; recent examples include dislocation imaging in semiconductors (GaN) [1] and perovskite oxides ($SrTiO_3$) [2].

In this study, ECCI is employed to investigate defects associated with misfit strains in epitaxial $La_{0.7}Sr_{0.3}MnO_3$ (LSM) thin films. LSM adopts the perovskite structure and possesses many interesting properties, such as ferromagnetism, colossal magnetic resistance (CMR), and excellent oxygen exchange properties for solid oxide fuel cell (SOFC) cathodes. It is of great interest to quantify the extended defect population in LSM [3] thin films to improve our understanding of strain relaxation mechanisms. Here, LSM thin films of thicknesses 50, 150, and 600 nm were deposited on (110)-$SrTiO_3$ substrates by pulsed laser deposition (PLD). As deposited samples exhibit high film-substrate coherency. The lattice mismatch induces 0.8% in-plane tensile strain. Atomic force microscopy (AFM) confirms all sample surfaces have RMS roughness less than 1 nm. No 3D island growth was observed.

We successfully imaged threading dislocations (spots with dark-to-light contrast) for $SrTiO_3$ (110) substrates and LSM films using ECCI (Figure 1). More prominent features in the ECCI images of LSM films were meandering lines that form enclosed loops, which were invisible in secondary electron (SE) mode (Figure 2). In ECCI images, they present a different bright/dark contrast than the rest of the film or the threading dislocations. Furthermore, the line contrast inverts upon inversion of the diffraction vector (Figure 2b,c), which is similar behavior to known crystalline defects (e.g., dislocations). The contrast level also depends on diffraction vectors— being strongest along $\mathbf{g} = \pm[100]$ and almost zero along $\mathbf{g} = \pm[1\text{-}10]$. The meandering line features are observed in LSM films of all thickness (Figure 3).

These line features exhibit unusual morphologies and unexpected contrast behavior with respect to diffraction conditions. They indicate the presence of extended structural defects in LSM, possibly closed dislocation loops (observed in $SrTiO_3$ [2]), anti-phase boundaries of 2D islands, or domain boundaries of twin structures[4]. Coordinated ECCI and high-resolution TEM with chemical composition analysis will be presented along with computational models of ECCI contrast from these defects. Prospects for the spatial mapping of crystallographic dislocations or defect boundaries in LSM will open many avenues for future *in situ* SEM experiments and improved local structure-property relationship determination.

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References
[1] R.J. Kamaladasa, F. Liu, L.M. Porter, R.F. Davis, D.D. Koleske, G. Mulholland, K.S. Jones, and Y.N. Picard, Journal of Microscopy **244,** 311 (2011).
[2] R.J. Kamaladasa, W. Jiang, and Y.N. Picard, Journal of Electronic Material **40,** 2222 (2011).
[3] L. Yan and P.A. Salvador, ACS Applied Materials & Interfaces **4,** 2541 (2012).
[4] J. Santiso, L. Balcells, Z. Konstantinovic, J. Roqueta, P. Ferrer, A. Pomar, B. Martínez,and F. Sandiumenge, CrystEngComm **15,** 3908 (2013).

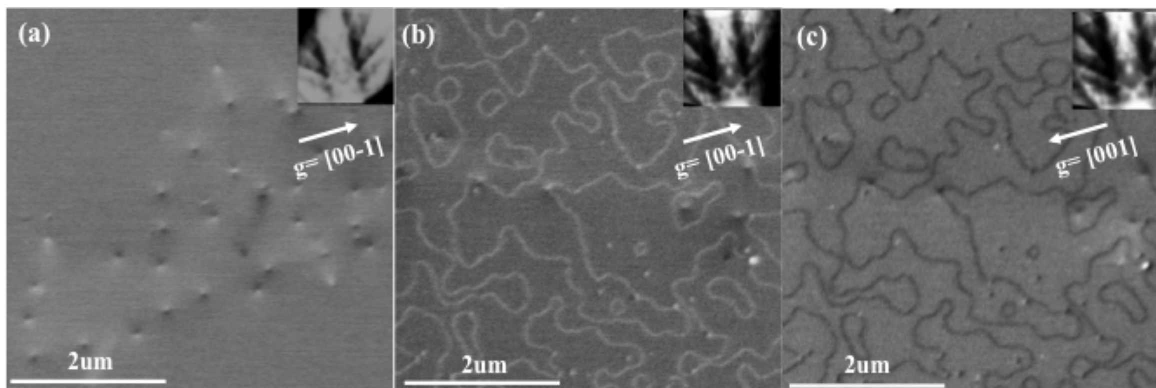

Figure 1. ECCI images of (a) $SrTiO_3$ (110) substrate showing multiple surface penetrating dislocations exhibiting bright-to-dark contrast; (b) and (c) are ECCI images on the same location of 150 nm LSM at opposite channeling conditions (**g** = ±[001]). The meandering lines present either bright (b) or dark (c) contrast, which is inverted upon reversal of **g.** Electron channeling patterns (ECP) are included as inset.

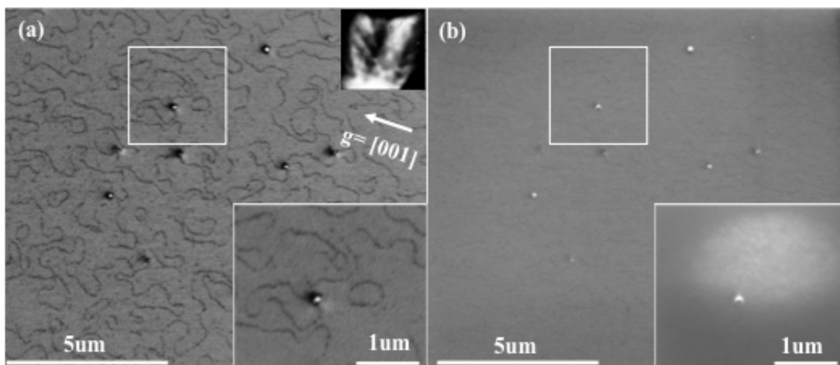

Figure 2. (a) ECCI and (b) SE images at the same location of a 600 nm LSM film. The insets on the bottom right are higher magnification images of areas enclosed by the white squares. ECP is included as top right inset in (a) (**g**=[001]).

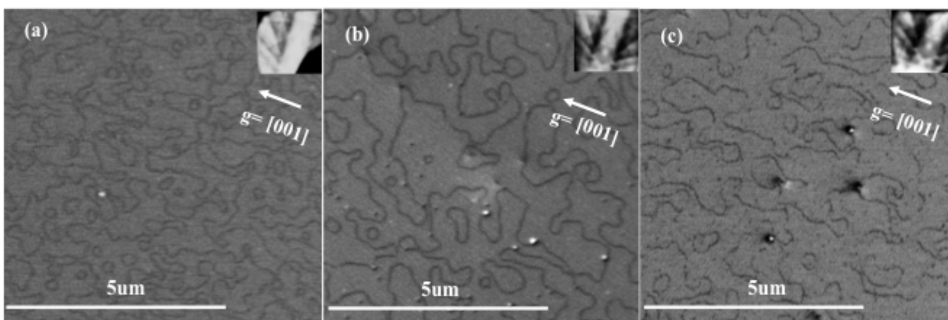

Figure 3. ECCI images of (a) 50 nm (b) 150 nm and (c) 600 nm LSM films and their corresponding ECPs as insets (**g**=[001] for all three images).

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