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Direct observation of nanoscale phase separation in $La_{1-x}Ca_xMnO_3$ using scanning electron nanodiffraction

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Nanoscale phase separation recently found in doped manganites has attracted great attention as a novel phenomenon with a wealth of underlying physics and materials science. Among the competing phases in manganites, a charge ordered (CO) phase characterized by unique superlattice reflections has been widely studied by diffraction techniques, including neutron scattering, x-ray diffraction and electron diffraction [1]. However, observations of the CO nanoscale phase by diffuse scattering in reciprocal space are sometimes difficult to quantify. In particular, the density and the volume fraction of the CO nanoscale phase were estimated to be very small, which is a major obstacle to link the CO structure to the extraordinary properties in these materials, such as colossal magnetoresistance (CMR).

In this talk, we report the direct observations of the CO nanoscale phase using scanning electron nanodiffraction (SEND). This technique employs an electron probe ~ 1.7 nm in size and which is scanned over the manganite crystals. At each location of the scanning area, an electron nanodiffraction (END) pattern was recorded by a CCD camera, showing the CO superlattice reflections (Fig. 1A, corresponding to the electron probe sitting on the CO nanoscale phase) or no CO superlattice reflection (Fig. 1B, corresponding to the electron probe sitting off the CO nanoscale phase). By mapping the intensity of the CO superlattice reflections, the CO phase in the form of nanoclusters was revealed in real space (Fig. 2). The evolution of the CO nanoclusters during the phase transitions at various doping levels was studied using the SEND maps at different temperatures. We estimated the volume fraction of the CO phase as a function of temperature from the SEND maps and the appearance of CO nanoclusters correlates with the CMR effect. Moreover, the mechanism of the formation of the CO nanoclusters and the discovery of an anomalous short range CO in these materials will be discussed. [2]

[1]. P. Dai *et al*., *Phys. Rev. Lett.* **85**, 2553 (2000); C. P. Adams *et al*., *ibid.* **85**, 3954 (2000); J. M. Zuo, J. Tao, *Phys. Rev. B* **63**, 060407 (2001)

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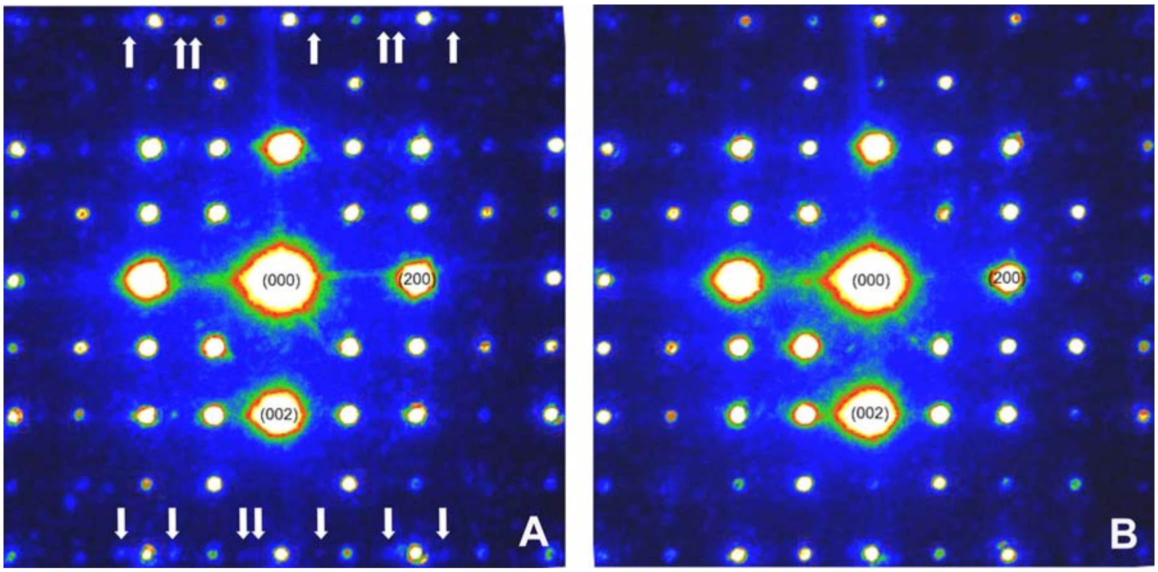

Fig. 1. (A) An END pattern from a single CO nanocluster in the $La_{0.55}Ca_{0.45}MnO_3$ at T = 255 K using the electron probe of ~ 1.7 nm in diameter, showing the superstructure reflections indicated by the arrows. (B) An END pattern from the non-CO area in the $La_{0.55}Ca_{0.45}MnO_3$ at T = 255 K using the same electron probe as that in (A)

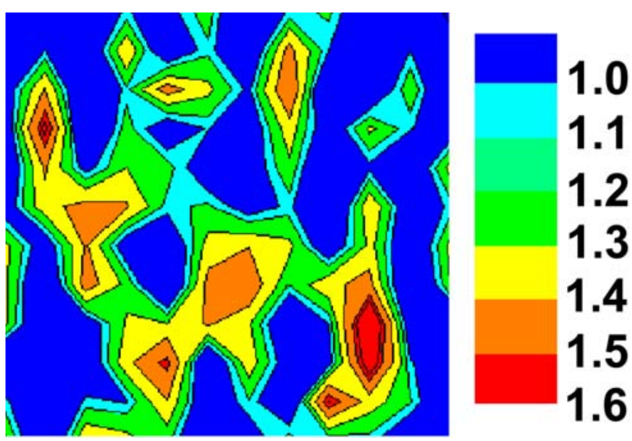

Fig. 2. A SEND map from an area of 12 × 12 nm^2 shows the CO nanoclusters in a single crystal domain in $La_{0.55}Ca_{0.45}MnO_3$ at temperature T = 253 K. A color scale of relative intensity of the CO superlattice reflection in the ENDs is shown on the right.

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