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HIGH-RESOLUTION AND LOW TEMPERATURE TEM STUDY OF SUPERCONDUCTING CUPRATES AND CMR-MANGANITES

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After the discovery of high-Tc cuprate in 1986[1] as well as CMR-manganite in 1993[2], we have developed the following different types of TEMs to examine "strongly-correlated transition metal oxides"; (1) 1.3MV high-voltage TEM (H-1500) with 1Å resolution[3], (2) 300kV analytical TEM (HF-3000S) with FEG and GIF, and (3) 300kV Lorentz TEM with FEG and external magnet up to 300G at the specimen. In the field of high-Tc superconductors, we have mainly examined the structures of new compound by HRTEM[4,5]. In the field of magnetic materials, we examined the charge-ordered superstructures and ferromagnetic domain structures formed at low temperature (typically below 150K) in various manganites[6,7]. Here, we present two examples of recent application of our three TEMs to superconductors and magnetic materials.

1. Order/Disorder of CO_3 (partly NO_3) Groups in Oxycarbonitrate Superconductors[5]

A series of new oxycarbonitrate superconductors, $(Cu,C,N)Sr_2Ca_{n-1}Cu_nO_y$ (n=1 to 6), prepared under high-pressure of 5 to 6 GPa, are examined by high-resolution TEM. Ordered arrangements of Cu and C (N) are observed in the compounds with n=1 to 4, while almost random arrangements for those with n=5 and 6. In the first two members, (Cu,C,N)-1201 (n=1) and 1212 (n=2), ordering scheme of -Cu-C(N)-C(N)-C(N)-Cu- with four-times periodicity is observed in the charge-reservoir blocks (CRB), as shown in Fig.1(a) to (d) for n=2 compound. For (Cu,C,N)-1234 (n=4), with highest T_c of 113K in the series, the ordering scheme of Cu-C(N)-Cu-C(N)-Cu- with twice periodicity is observed in the CRB. In the last two members, (Cu,C,N)-1245 (n=5) and 1256 (n=6), no evidence of ordering was observed, suggesting that Cu, C and N are distributed almost randomly in the CRB. Such relations between the order/disorder scheme and n-parameters, are also preserved locally in the crystals which contain plenty of intergrowth defects.

2. Direct Observations of Ferromagnetic Domains in Manganites[8]

Ferromagnetic domains in $Nd_{1/2}Sr_{1/2}MnO_3$, which undergoes the ferromagnetic to charge-order (antiferromagnetic) transitions at around T_{CO}=150K, are examined by Lorentz TEM using mainly the Fresnel-mode. On cooling from paramagnetic state of room temperature, magnetic domain walls started to appear, as black and white lines, below T_C=250 K. The direction of magnetization in each domain is along the long sides of domain, suggesting that the compound has a magnetocrystalline anisotropy. With a further decrease of temperature, the volume of magnetic domains increased with discontinuous domain-wall jumps, and then gradually disappeared below T_{CO}=150K. Clear satellite reflections due to lattice distortion, are observed in the hk0 electron diffraction pattern. On heating process, a characteristic granular contrast with 30-40 nm in size was observed at around 140 K, close to the T_{CO}, as shown in the Lorentz TEM image in Fig. 3(a) and (b), at underfocus and overfocus, respectively. Such a granular contrast was not observed in the cooling process. We consider that the origin of this contrast is the formations of ferromagnetic microclusters in antiferromagnetic matrix.

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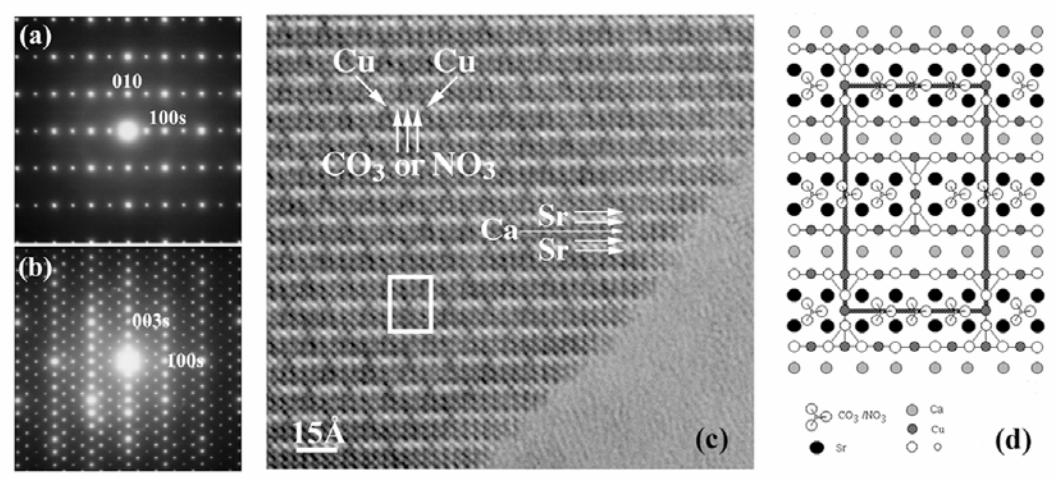

Fig. 1. The (a) *hk0* and (b) *h0l* electron diffraction patterns, (c) HRTEM image projected along *b*-direction and (d) the superstructure model of (Cu,C,N)-1212 type of oxycarbonitrate superconductor. Ordered arrangements of Cu and three CO_3 (partly NO_3 ones) are clearly observed. HRTEM image was taken at 800kV, by H-1500, to prevent electron-beam damage.

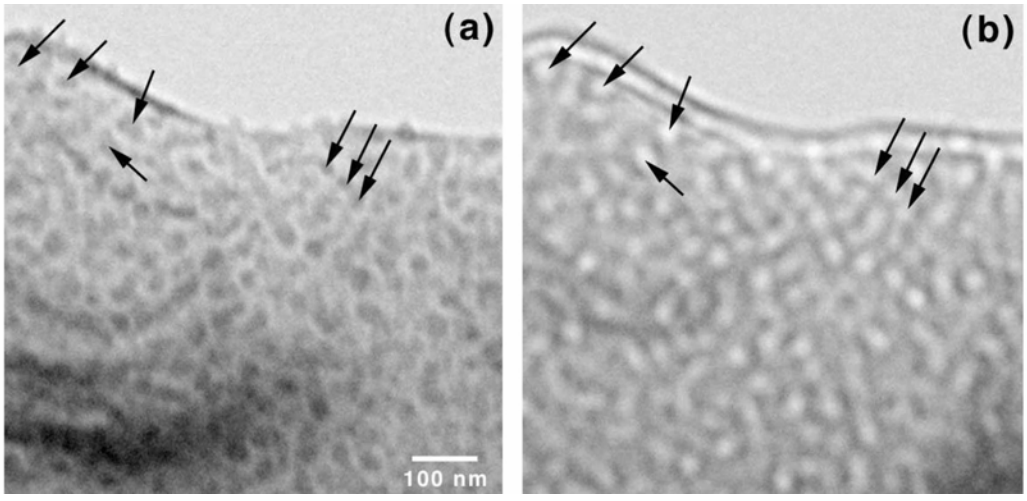

Fig. 2. Lorentz TEM images (300kV) of $Nd_{1/2}Sr_{1/2}MnO_3$ in the (a)underfocused and (b)overfocused conditions at 140K, near T_{CO}. Arrows indicate the reverse in contrast.

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