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High-resolution imaging and spectroscopy of CoO octahedral nanoparticles

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As the size of a magnetic system decreases, the significance of the surface magnetic moments increases. The finite-size effect is highly relevant for nanoparticles consisting of an antiferromagnetic (AFM) material. Since an antiferromagnet usually has two mutually compensating sublattices, the surface can break the pairing of the sublattices and thus leads to “uncompensated” surface magnetic moments. Néel first discussed this effect in AFM nanoparticles with a net magnetic moment [1]. Later many experimental studies have suggested various scenarios for the unusual magnetic properties, e.g., spin-glass or cluster-glass-like behavior of the surface spins [2–3], core-shell interactions [3-4], or weak ferromagnetism [5]. However, the nature of the surface magnetic contribution has not been fully understood. We have encountered unexpected magnetic phenomena in octahedral-shaped CoO-Co₃O₄ core-shell nanoparticles—exhibiting room temperature ferromagnetic behavior as evidenced by their magnetization hysteresis loop. Hard X-ray magnetic circular dichroism measurements confirmed the absence of metallic Co clusters inside the samples. To investigate possible structural and compositional reasons for the observed magnetic behavior, we conducted structural and spectroscopy characterizations using transmission electron microscopy. Figure 1(a) shows a typical TEM image of CoO-Co₃O₄ core-shell nanocrystals (NCs). In most of these NCs, contrast of voids is present as indicated in the inset. The different 2D projections (FIG.1(b)) are from an octahedral shape, as confirmed by 3D electron tomography (not shown here). Fig.1(c) shows a HRTEM image revealing different structures of the Co₃O₄-shell and the CoO-core. We also performed spot-EELS in STEM mode with a beam diameter of around 1 nm (FIG.1(d)). The spectra further confirm the CoO core and Co₃O₄-like shell structure.

To quantitatively analyse the interfacial as well as the spectroscopic structure concerning the Co-O bond length and electronic states, the atomically-resolved HAADF in combination with EELS analysis was performed. Additional high-resolution imaging and spectroscopic studies of these Co oxides nanoparticles are still in progress and will be reported and discussed in context to temperature and magnetic field dependent magnetic measurements.

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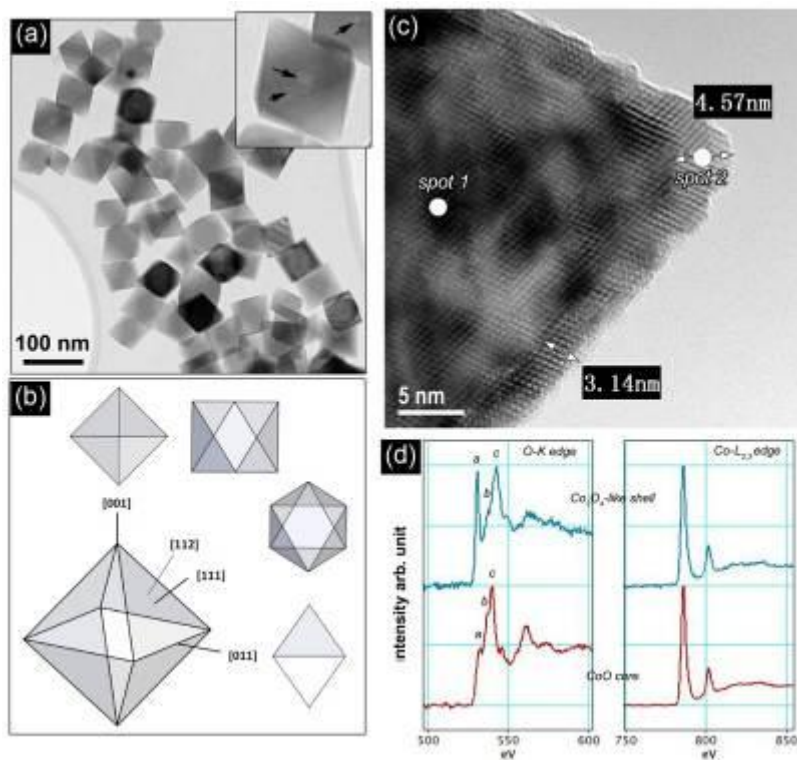


Figure 1. (a) Typical TEM image of octahedral CoO-Co₃O₄ core-shell nanocrystals. In most of these NCs, void contrasts are present as indicated in the inset. (b) The different 2D projections are from an octahedral shape, as confirmed by 3D electron tomography (not shown here). (c) HRTEM image of the Co₃O₄-shell and CoO-core. (d) Spot-EEL spectra were taken at two positions in STEM mode with a beam diameter of about 1 nm. These spectra confirm the CoO core and Co₃O₄-like shell structure.