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Application of Energy-Filtered Imaging and HREM in the Study of Terbium Nanoparticles

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Rare earth elements and compounds form a unique group of materials with diverse properties. Because the magnetic interactions in rare earths, to which Tb belongs, are mediated by the valence electrons, they are very sensitive to the structure and the filling of the conduction bands. Thus small changes in the band structure, originating from nanoscale dimensions, are expected to result in a dramatic effect on the magnetic properties. However, due to difficulties in sample preparation (the rare earths are highly reactive), the studies on nanoparticles are rather scarce [1-3] and no microstructural data have been published yet. The aim of this work was to fabricate Tb nanoparticles and characterize their microstructure, as a first step towards studying the finite size effects on their magnetic properties. Because it is difficult for conventional TEM techniques to reveal microstructure features and elemental distribution at the nanometer scale, energy-filtered image and HREM were used in this study.

The following two procedures were used to produce the precursors, which were further annealed to fabricate Tb nanoparticles with different size: (i) Tb/Cr multilayer precursors were deposited by magnetron sputtering using the tandem mode; (ii) Tb-Cr thin films were deposited on a substrate continuously rotated above the Tb and Cr targets. Here we will report the study on the precursor sample. Both energy-filtered imaging and HREM were carried out on a Philips CM20 TEM at 200kV equipped with a Gatan imaging filter (GIF). In the Tb/Cr multilayer samples, many nanoparticles (<10 nm) with a narrow size distribution are observed in the zero-loss image (figure 1a). Figure 1b and 1c show the corresponding Cr and Tb jump-ratio images, respectively. It is seen that the Tb particles are uniformly distributed within the Cr matrix. HREM confirmed that these Tb nanoparticles are isolated in the multilayer mode sample (figure 2). A series of energy-filtered images in the Tb-Cr sample fabricated by the continuous mode are shown in figure 3. Composition maps show large Cr particles (10~15 nm) (figure 3a and 3b), coexisting with Tb clusters which are distributed in the form of networks (figure 3c). Electron diffraction patterns (EDP) in the continuous mode sample confirm the presence of bcc Cr crystalline phase and an amorphous structure, possibly related to the Tb clusters. Correspondingly, only the crystalline Cr particles are observed in the HREM image of the continuous mode sample (figure 4), implying that for the concentration under study and at the conditions used, the mobility of the Tb atoms is not sufficient to form crystalline particles. Therefore, with the help of energy-filtered imaging and HREM, it is confirmed that magnetic Tb/Cr nanoparticles have been successfully produced via the multilayers precursor technique [4].

References

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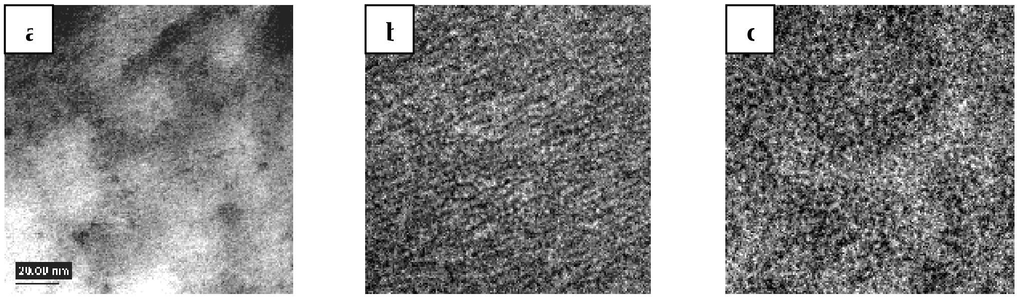


FIG. 1. Energy-filtered images in a Tb/Cr sample fabricated by the multilayer precursor mode: (a) Zero-loss image; (b) Cr jump-ration image; (c) Tb jump-ratio image.

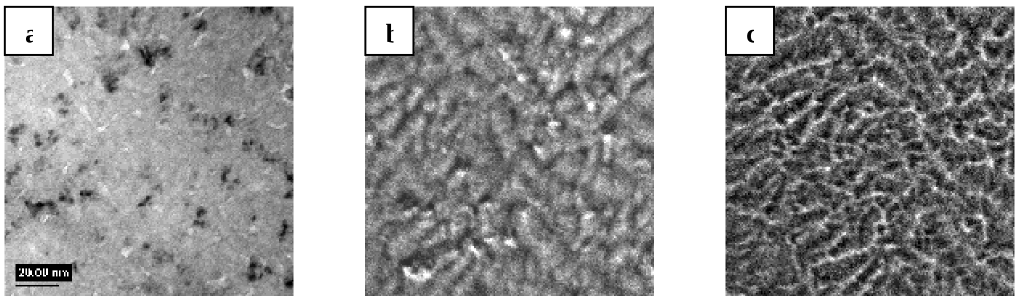
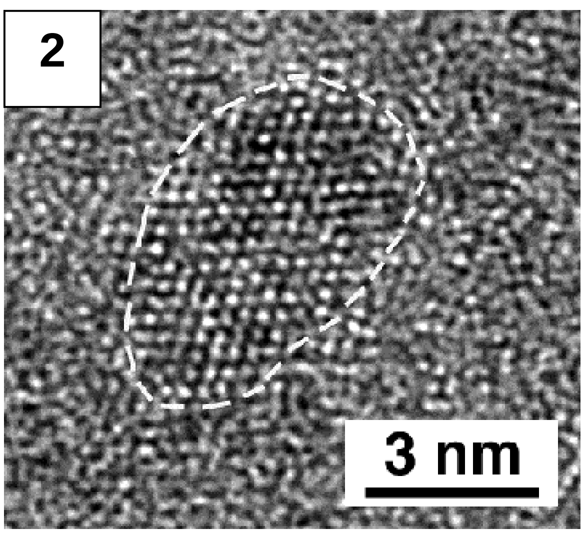


FIG. 3. Energy-filtered images in a Tb/Cr sample fabricated by the continuous mode: (a) Zero-loss image; (b) Cr jump-ration image; (c) Tb jump-ratio image.



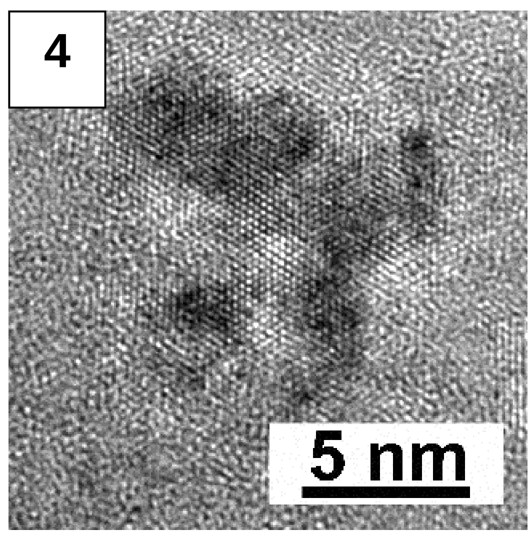


FIG. 2. HREM images showing a Tb nanoparticle in the Tb/Cr sample fabricated by the multilayer precursor mode.

FIG. 4. HREM images showing crystalline Cr particles in the Tb/Cr sample fabricated by the continuous mode.