

Atomic Structure and 3D Shapes of FePd Nanoparticles Observed by HRTEM, HAADF-STEM and Electron Tomography

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FePd nanoparticles with the $L1_0$ -type ordered structure is one of the candidate materials suitable for the ultrahigh density magnetic storage media. The hard magnetic properties originate from the tetragonal ordered structure, which gives rise to a high magnetocrystalline anisotropy energy, and thus the atomic ordering is a key issue for improving the magnetic properties of the $L1_0$ -type alloy nanoparticles [1-3]. On the other hand, high-areal density packing of nanoparticles and control of magnetostatic interaction among nanoparticles are considered as the next step to realize ultrahigh density recording media. In this study, we have studied atomic structure, 3D shapes and distribution of FePd nanoparticles by C_s -corrected HRTEM, HAADF-STEM, and electron tomography [4].

FePd nanoparticles were fabricated by sequential electron beam deposition of Pd, Fe, and Al_2O_3 onto NaCl(001) substrates at 673 K, followed by postdeposition annealing at 873 K for 1h. HRTEM and HAADF-STEM images were obtained by using an FEI Titan 80-300 (S)TEM operating at 300 kV with a field emission gun and a C_s -corrector for the objective lens. Focal-series HRTEM images were reconstructed using the IWFR software (HREM Research Inc.). Z-contrast images were acquired by using a HAADF-detector with the inner angle higher than 60mrad. A single-tilt holder was used for the tilt series acquisition with the maximum tilt angle of 65°. Alignment of the tilt axis for the obtained data set (81 Z-contrast images) by an iterative cross-correlation technique and subsequent 3D reconstruction were performed by using the Inspect3D software (FEI Co. Ltd). We employed weighted back-projection (WBP), as well as simultaneous iterative reconstruction technique (SIRT), for 3D reconstruction.

Figure 1(a) shows a C_s -corrected HRTEM image of a 10-nm-sized FePd nanoparticle with the c-axis of the $L1_0$ structure oriented normal to the film plane. The arrangement of atoms due to atomic order can be seen clearly as periodic bright contrast even at the interface between the crystal and amorphous, as well as the inside of the nanoparticle [2]. Figure 1(b) shows a Z-contrast image observed by HAADF-STEM. A magnified image is shown in the inset. Two types of bright dot, strong and weak, arise from Pd and Fe atoms, respectively. Figure 2 compares HRTEM images (a, c) and corresponding phase images reconstructed by focal series HRTEM images (b, d) [$C_s = 8.3\mu\text{m}$, focal step = 4nm]. Successive 5 images were used for the reconstruction. Clear-cut atomic order and faceted interfaces can be seen in the phase images. Particle size, shape and locations were reconstructed by WBP (Fig.3a) and SIRT (Fig.3b). Large discrepancy in particle thickness (height) is apparent between these two images. The results of the two algorithms for reconstruction have been compared quantitatively with those obtained by the extrapolation method, and those independently reported by electron holography (Fig.3c). We demonstrate that WBP yields a better estimation of the particle size in the z direction than SIRT does, within the present experimental conditions [4].

References

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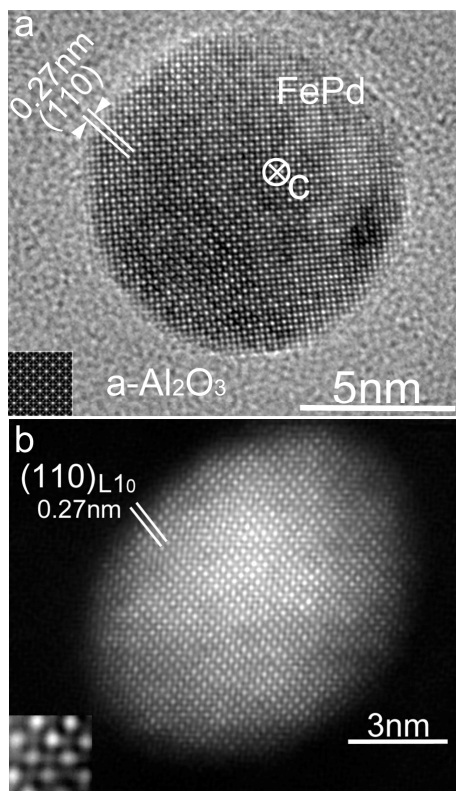


FIG. 1. (a) HRTEM image [$C_s = -340$ nm, $\Delta f = -13$ nm (overfocus)] and (b) HAADF-STEM image of L₁₀-FePd nanoparticles with the c-axis oriented normal to the film plane.

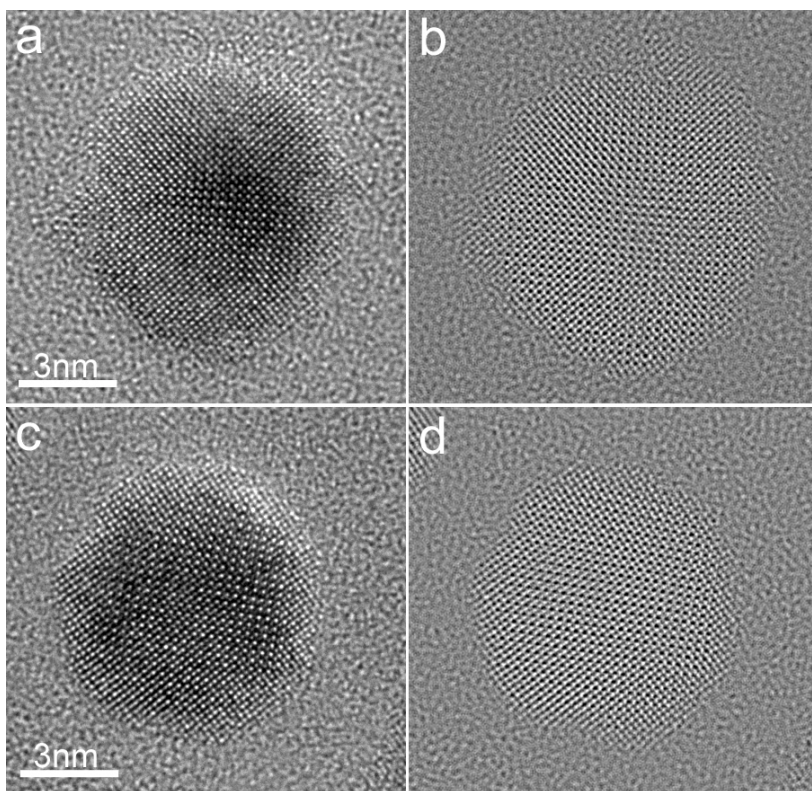


FIG. 2. [(a) and (b)] HRTEM image and the corresponding phase image of exit wave reconstructed by focal series HRTEM images [$C_s = 8.3$ μ m, focal step = 4 nm]. Successive 5 images were used for the reconstruction. The beam incidence is along $[001]_{FePd}$. [(c) and (d)] Another example of a HRTEM image and the reconstructed phase image, respectively.

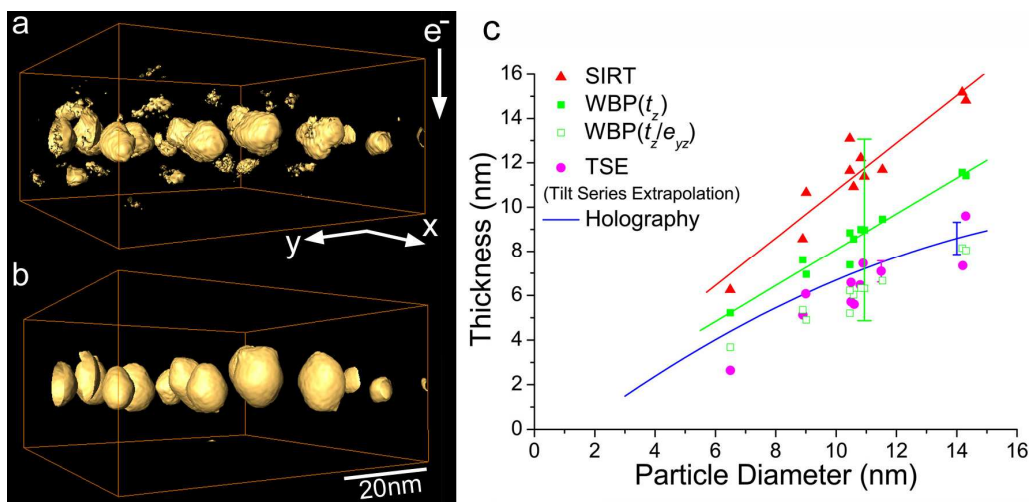


FIG. 3. Oblique-view of the reconstructed volume processed by (a)WBP (upper) and (b) SIRT (lower). The reconstructed volume is $75 \times 75 \times 36$ nm³. (c) The relation between particle diameter and thickness for the FePd nanoparticles estimated by using several techniques.