

Characterization of L1₀-type FePd Alloy Nanoparticles by Atomic-Resolution HAADF-STEM and Electron Tomography

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Hard magnetic properties of FePd alloy nanoparticles originate from their L1₀-type tetragonal ordered structure with a high magnetocrystalline anisotropy energy. We have studied atomic structures of FePd nanoparticles by C_s-corrected high-resolution transmission electron microscopy (HRTEM) [1] and quantitative electron diffraction [2]. In contrast to HRTEM, atomic number (Z) contrast by high angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM) is sensitive to chemical order in small nanoparticles. In this study, we report on the atomic structure and three-dimensional shapes of FePd nanoparticles using HAADF-STEM and electron tomography.

FePd nanoparticles were fabricated by successive deposition of Pd and Fe onto NaCl(001) substrates at 673 K. After the deposition of Fe, an amorphous Al₂O₃ thin film was deposited to protect the particles from oxidation. Post-deposition annealing at 873 K led to a formation of the L1₀-type ordered structure. Atomic-resolution HAADF-STEM images were obtained by using an FEI Titan 80-300 (S)TEM operating at 300 kV with a field emission gun. HAADF-STEM images were simulated based on the frozen phonon model using the MACTEMPAS software. A single tilt specimen holder (Fischione model 2020) was used for acquiring HAADF-STEM tilt series of FePd nanoparticles. The reconstruction was carried out using the simultaneous iterative reconstruction technique (SIRT). All STEM images were recorded by using a CCD camera attached to the TEM.

Figure 1 shows a HAADF-STEM image of a 10-nm-sized FePd nanoparticle with the c-axis of the L1₀ structure oriented normal to the film plane. The image was acquired with a beam convergence of 10 mrad using a 50 μm condenser aperture and a HAADF detector with an inner angle of 60 mrad. The periodic arrangement of atoms by chemical order can be seen clearly as bright contrast. The {220} atomic planes are also resolved as shown in the inset (top left). Due to the alternate stacking of Fe and Pd in the [001] direction in the L1₀ structure, the {220} atomic planes also possess an alternate stacking sequence of Fe and Pd in the <110> direction. This periodic stacking by chemical order causes intensity modulation at the atomic level (Z contrast). An example of elemental mapping by STEM-EDS is shown in Fig.2. Maps of Pd-L, Fe-K, and Fe-L show particulate structure, while the Al-K and O-K maps show a continuous film. A HAADF-STEM image and the corresponding reconstructed images based on the STEM-tomography are shown in Fig.3. The tilt series were acquired in the angle range between -70 and +70 deg with a 2 deg increment. Thus, three-dimensional shapes have been clearly reconstructed for FePd nanoparticles 10 nm in size.

- [1] K. Sato, T. J. Konno, and Y. Hirotsu, *J. Appl. Phys.* **105**(3) (2009) 034308.
- [2] K. Sato, Y. Hirotsu, H. Mori, Z. Wang, and T. Hirayama, *J. Appl. Phys.* **98**(2) (2005) 024308.
- [3] This study was partially supported by the Grant-in-Aid for Young Scientists (B) (Grant No. 19760459) from the Ministry of Education, Culture, Sports, Science and Technology, Japan.

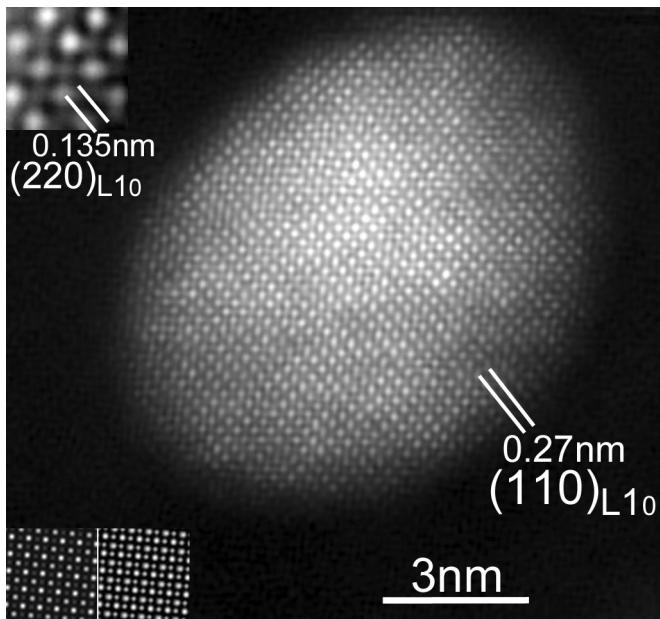


Fig.1 Atomic-resolution HAADF-STEM image of an $L1_0$ -FePd nanoparticle. The beam incidence is [001]. Simulated images are shown in the inset (bottom left: 3nm-thick, right: 7nm-thick).

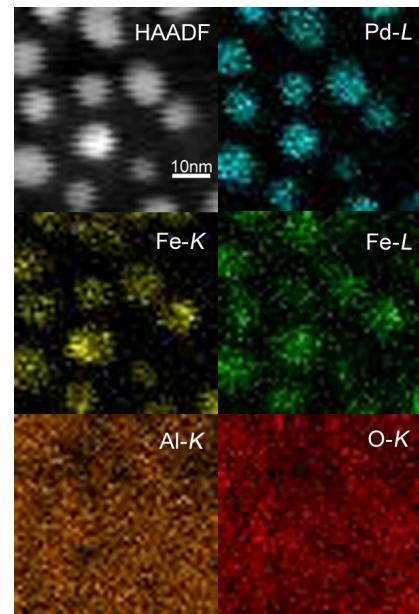


Fig.2 Elemental mapping of nanoparticles by STEM-EDS.

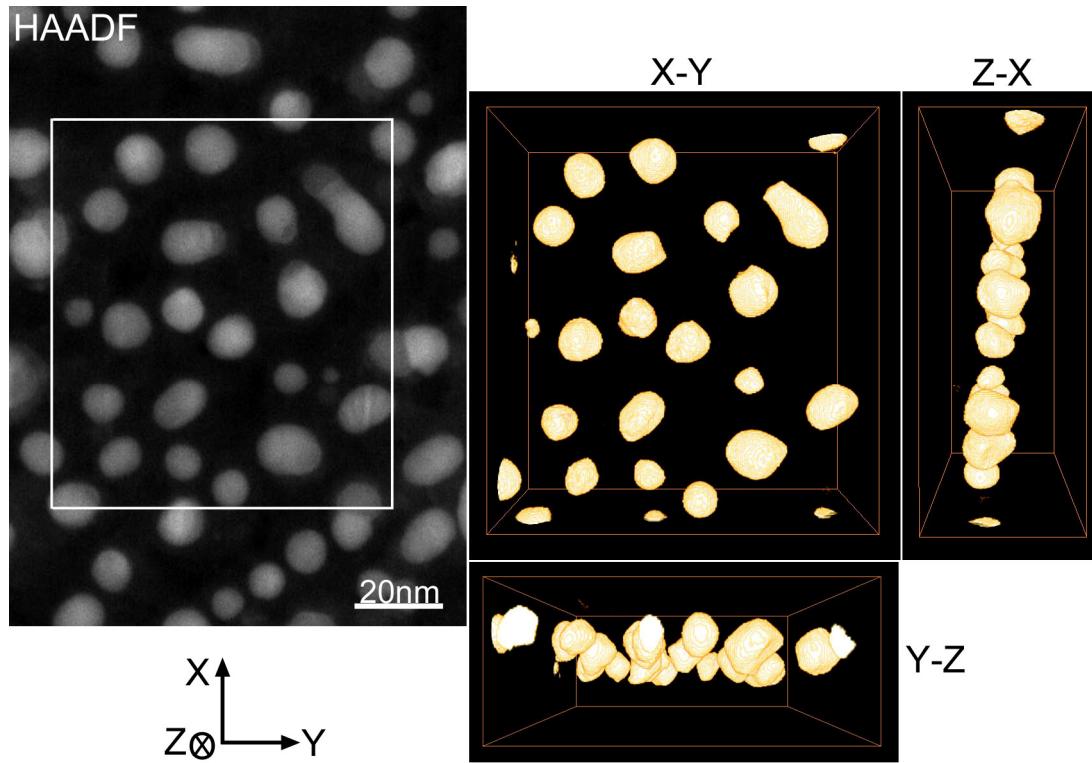


Fig.3 HAADF-STEM image of FePd nanoparticles and the corresponding reconstructed images based on the electron tomography. The tilt axis is along the X-direction.