Application Note

Fusion™

In Situ FCC to L10 Tetragonal Phase Transition of Magnetic FePt Nanoparticles

Introduction

Current methods of information storage, such as the magnetic hard disk drive, are reaching a fundamental size limit. Many research groups are investigating different materials and methods to increase the amount of information that can be stored per unit area. Magnetic nanoparticles offer an appealing route to high-density information storage.

Iron platinum (FePt) nanoparticles are a promising material for this application. They can be synthesized and dispersed via chemical methods, with uniform diameters. However, FePt nanoparticles usually form in the face centered cubic (fcc) crystal structure or in a partially ordered face center tetragonal (fct) crystal structure. Neither structure possesses strong magnetic anisotropy. To obtain nanoparticles with a fully ordered fct or L10 crystal structure, which possesses very high uniaxial magnetocrystalline anisotropy, an annealing step to 500 °C is required.

To understand the formation of the fcc to L10 phase transition transmission electron microscopy (TEM) was used to visualize the transition of individual particles. TEM can resolve the atomic structure of particles, and when operating in scanning TEM mode, Z contrast can distinguish between Fe and Pt atoms in the crystal lattice. This technique is not possible with bulk methods such as x-ray diffraction (XRD). The fcc to L10 phase transition occurs at around 500 °C, but to obtain meaningful data in the TEM, a heating system capable of maintaining atomic resolution at high temperatures is needed.

When the TEM is combined with an active sample support system, such as the Fusion heating and electrical biasing system, resolution is maintained at temperatures up to 1200 °C. Extremely fast ramp rates (up to 1,000 °C per ms) are also possible making Fusion an exceptionally versatile and powerful platform for nanoparticle analysis.

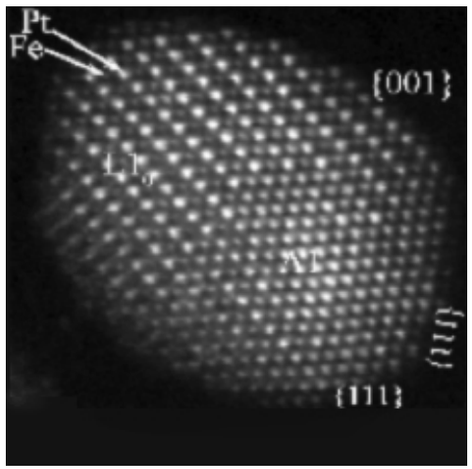

Figure 1: FePt nanoparticles

Experiment

FePt magnetic nanoparticles (shown in Figure 1) used in the experiment described here had a mean size of 4 nm and stabilized with pentadecanenitrile and oleic acid ligands. The nanoparticles were dispersed onto the E-chip™ support membrane from a nanoparticle-hexane suspension and allowed to dry.

The nanoparticles were heated to 500 °C and imaged after 1, 5 and 15 minutes. The electron beam was blanked when possible to minimize beam effects on the nanoparticles. The experiments were done in a JEOL 2200FS in Dr. Larry Allard's lab at Oak Ridge National Laboratory. The TEM is equipped with a CEOS Co. aberration corrector on the probe-forming optics, and was operated in scanning TEM (STEM) mode at 200 kV.

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Reference:
Images courtesy J.E. Wittig, C.M. Lukehart Vanderbilt Univ., and J. Bentley, L. Allard, ORNL

Discussion

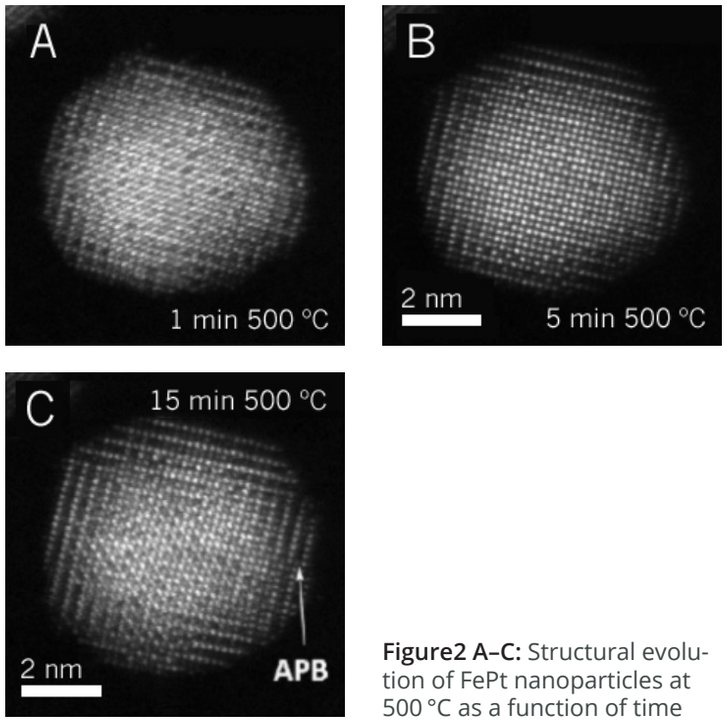

Figure2 A–C: Structural evolution of FePt nanoparticles at 500 °C as a function of time

In Figures 2A–C, a 6.4 nm particle consisting of 48% Fe and 52% Pt is shown. In these high angle annular dark field (HAADF) STEM images the heavier Pt appears brighter than Fe. In Figure 2A, evidence of the fcc to the tetragonal L10 phase transition is seen on the outer layers of the nanoparticle after just 1 minute at 500 °C. After 5 minutes at 500 °C (Figure 2B) the transition to the L10 phase on the outer layers is much more evident and distinct facets have formed.

After 15 minutes (Figure 2C), the phase transition is moving radially inward toward the center of the nanoparticle. An antiphase boundary is also indicated by the arrow. These features were not resolvable with bulk methods such as XRD.

In Figure 2B a FePt nanoparticle is imaged in the process of transforming from fcc to L10. The bottom left of the particle is fcc, and the top left is L10. After annealing, the Fe and Pt form successive planes as seen by this HAADF image, indicative on the L10 crystalline phase.

Applications

This example of FePt magnetic nanoparticle analysis in the TEM is applicable to all nanoparticles that structurally or chemically evolve as a function of temperature. The imaging capability of the TEM coupled with that of the Fusion system enables high resolution *in situ* analysis at temperatures up to 1200 °C. Accurate and precise temperature control, coupled with high-resolution capability, create the quantifiable and actionable data sets required to understand nanoparticle phase transitions with unparalleled clarity. Contact us to discuss the full range of capabilities of Fusion with thermal E-chip specimen supports for your applications. We can be reached at (919) 377-0800 or contact@protochips.com.

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