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Magnetic properties of structurally confined FePt nanoparticles within mesoporous nanotubes

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Abstract

Chemically ordered FePt binary alloys with $L1_0$ face-centered tetragonal (fct) structure have recently attracted considerable attention due to their excellent intrinsic magnetic chemical and mechanical properties. In particular, one-dimensional (1-D) arrays of FePt alloys are prospective materials for ultrahigh density magnetic storage media. We describe a new method to fabricate FePt alloy nanostructures embedded in the nanochannels of anodic alumina templates (AAT) and SBA15 silica through infiltrating porous membranes with FePt nanoparticles. SBA15 silica nanotube is a honeycomb structure with tubular channels of 9 nm in diameter, extending through 10 μm long particles. In contrast AAT have morphology of a membrane with regular array of pores 60 nm in diameter. To transform the FePt alloy phase from chemically disordered face-centered-cubic to chemically ordered $L1_0$ phase the membrane was annealed at 700 °C in a H_2-N_2 gas mixture for 2 h. Transmission electron microscope (TEM) and field emission scanning electron microscope (FESEM) show that nanoparticles transformed into isolated superparamagnetic nanoparticles in SBA15 and elongated nanostructure in AAT due to the annealing treatment. The magnetization was measured by quantum interference device (SQUID). The ordered fct FePt polycrystalline nanostructure in AAT have high magnetic anisotropy and thus large coercivity up to 1.1 T at room temperature.

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High coercivity magnetic nanoparticles organized in regular arrays can potentially be used to fabricate ultra-high density magnetic recording media. First, three-dimensional superlattices of $L1_0$ FePt nanoparticles with face-centered tetragonal (fct) structure were chemically prepared by Murray[1]. Control of the size, shape and arrangement of nanoparticles can be used to tune properties of nanomagnetic materials. The shape of the magnetic nanomaterials can also be reversibly interchanged between nanoparticle and nanowire by using different templates. In the present study, we prepared FePt nanoparticles and embedded them in two different templates. The FePt nanoparticles of 2 nm in diameter have been received by superhydride reduction method. Mesoporous SBA15 is an ideal host material for insertion FePt nanoparticles[2]. The perfect uniform mesoporous SBA15 provides an effective way of controlling uniformity of particle size and prevent agglomeration of the particles[3]. To solve problem of the aggregation of the particles, we incorporated nanocrystals into one-dimensional hexagonal channels with 9 nm in diameter pores. The same particles were used as building blocks to fabricate and array of the FePt nanowires. They were embedded in the nanochannels of anodic alumina templates through infiltrating porous membranes with the nanoparticles. Compared with other templates, AAO template is an ideal template to prepare ordered nanowire

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arrays because of its uniform and nearly parallel pores' structure.

The mesoporous SBA15 host was prepared according to the previous report[4]. To make the silica surface non-polar, the mesoporous SBA15 was functionalized with chlorotrimethylsilane. FePt nanoparticles were synthesized by superhydride reduction of $FeCl_2$ and $Pt(acac)_2$ in the presence of oleic acid, oleylamine, and 1,2-hexadecanediol[5]. The mixture was refluxed while being stirred at 50 °C for 24 h. To transform the particle structure from the chemically disordered fcc phase to the chemically ordered fct phase, the annealing was performed under mixture of H_2 (3%) and Ar in a quartz tube at 600 °C for 2 h. Hexagonally ordered porous AAT used as a template in this work was prepared using a two-step anodization process to oxidize aluminum in acid solution[6]. To prepare FePt alloy nanowires, 10 mL of this FePt nanoparticle solution was infiltrated into the alumina template by vacuum suction assisted by an applied magnetic field. To form nanowire arrays, the sample was annealed at 700 °C for 2 h in a quartz tube filled by the Ar and H_2 (3%).

Recently, Sun et al[5]. have reported on the synthesis of monodisperse ultrafine FePt nanoparticles with controlled size and composition. The problem is a coalescence of FePt particle after annealing for fct ferromagnetic phase transition. To prevent agglomeration of the particles, we utilized the SBA15 ordered channel structure. We used powder X-ray diffraction to check the stability of the SBA15 host as well as the size and structure of the FePt nanoparticles after annealing. The FePt particles show structural transformation from the fcc phase to fct phase, as seen in Fig. 1. The FePt particles size of 2 nm was determined from the TEM images and was verified using Scherrer equation of the XRD peak. The micrographs in Fig. 2 show well separated, spherical 2 nm in diameter particles of FePt within SBA15 host after annealing at 600 °C. Energy dispersive X-ray analysis indicated that the average composition of Fe to Pt is $Fe_{46}Pt_{54}$.

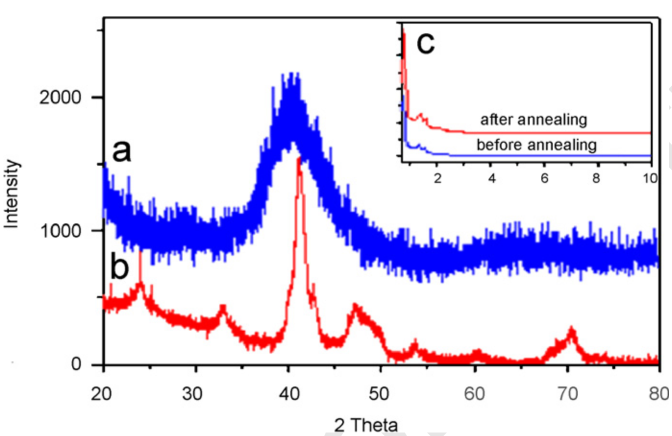

Fig. 1. X-ray diffraction patterns of: (a) the as synthesized FePt nanoparticles; (b) the annealed FePt particles; (c) low angle for spectra in the inset prove the stability of annealed FePt–SBA15.

Magnetic properties of the FePt nanoparticle system are summarized in Fig. 3. The assembly of very small isolated particles inside the SBA host after annealing exhibits superparamagnetic behavior. The temperature dependence of the magnetic susceptibility of the zero-field-cooled (ZFC) sample shows clear maximum at the blocking temperature of about 13 K. The volume V and diameter of the particles can be estimated from the blocking temperature using the formula $T_B = KV/25k_B$, where k_B is the Boltzmann constant and K is the anisotropy constant of bulk FePt. The estimated diameter of the particle of approximately 2 nm is in a good agreement with the TEM observations of the particle size mentioned above. The divergence of the ZFC and field-cooled curves indicates certain distribution of particle's sizes. In contrast to the superparamagnetic behavior of the particles in SBA, the agglomerates of the FePt particles in the form of nanowires (Fig. 2(c)), with average diameter of about 60 nm, display ferromagnetic behavior at room temperature. The large room temperature coercivities of 10350 Oe measured with the field along wires and of 8350 Oe with the field transverse to the wires are characteristic of the hard magnetic phase of the $L1_0$ structure. Slightly different shapes of the curves measured with field parallel and perpendicular to the wires indicate that the assistance of the magnetic field during particle infiltration could give rise to certain grain texture of the polycrystalline wires. In conclusion, this work demonstrates that it is possible to vary magnetic behavior of nanostructures at room temperature from superparamagnetic to hard magnetic properties by arranging ultra-small particles of FePt into

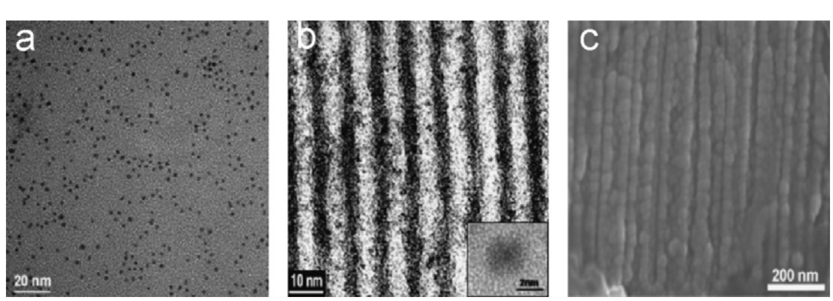

Fig. 2. TEM images of: (a) FePt nanoparticles; (b) FePt–SBA15 after annealing (inset shows HRTEM image); (c) FE-SEM image of FePt–AAO after annealing.

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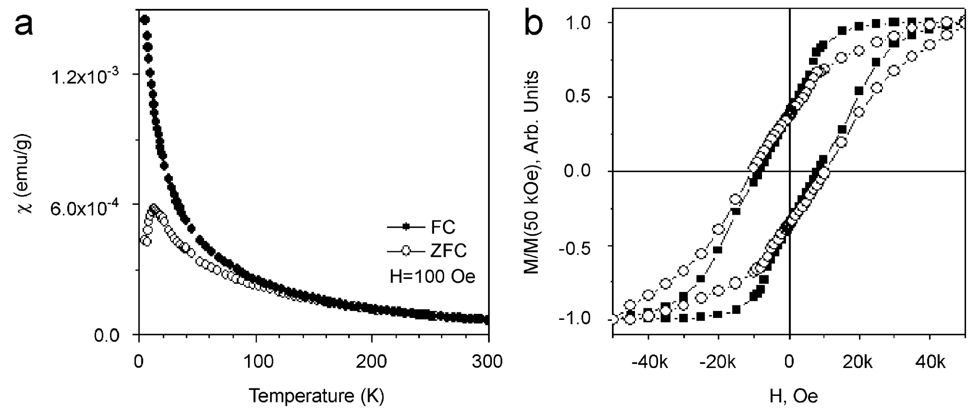

Fig. 3. (a) Temperature dependent FC (filled circle) and ZFC (empty circle) DC magnetic susceptibilities for the FePt–SBA15; (b) hysteresis loops of FePt–AAO measured at 300 K. Solid squares correspond to magnetic field applied in the substrate plane, open circles—perpendicular to the plane.

larger nanowires. This method is very flexible and allows fabrication of arrays of nanoparticles of different sizes and shapes depending on the choice of the template. It can potentially be used for the fabrication of recording media.

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