

Synthesis and Morphology of Face Centered Cubic (FCC) Fe-Pt Nanoparticles

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ABSTRACT

FePt nanoparticles with thermally stable room-temperature ferromagnetism are investigated. The monodisperse nanoparticles are prepared by chemical synthesis and a salt-matrix annealing technique. Structural and magnetic characterizations confirm the phase transition from the disordered face-centered cubic structure. In this paper, 3 nm FePt nanoparticles are first synthesized by superhydride reduction of Fe and Pt. Transmission Electron Microscopy (TEM) images show that the hard magnetic FePt are agglomerated after annealing at 675°C. Scanning Electron Microscopy (SEM) images indicate that the size of FePt nanoparticles increase by increasing of the annealing temperature.

Keyword: FePt nanocrystals; Anisotropy; Hard magnetic nanoparticles; Chemical synthesis; Sintering; Recording media.

1. INTRODUCTION

Development of new high density magnetic media for data recording has become a key issue for data storage industry. In the near future current magnetic media will reach the areal density beyond of 1 Tbit/in² imposed by the phenomenon known as superparamagnetism [1]. Generally, the FePt films deposited at room temperature are face-centered cubic (fcc) and show a soft-magnetic behavior [2]. The fcc FePt phase can be transformed to a hard-magnetic face-centered tetragonal (fct) FePt (L1₀ FePt) ordered phase after annealing at high temperature [3]. In L1₀ alloy, the FePt system

presents highly coercivity, good corrosion resistance, large magnetic energy product but high temperature annealing is required to transform fcc disordered structure to L1₀ ordered FePt phase.

Recently, the areal density of recording media is approaching to 1 Tbits/in². Such a high density would require a grain size of only a few nanometers in diameter [4, 5]. For high density magnetic recording media, the magnetic grains must be small enough to be nanoparticle size and also requires that uniform size and isolated magnetic particles to reduce inter-grain interaction, which leads to lower

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media noise [6-8]. However, small particle size will result in smaller $K_u V / K_B T$ value (where K_u is the uniaxial magneto-crystalline anisotropy, V is the particle volume, K_B is the Boltzmann's constant and T is the absolute temperature), which leads increasing the thermal fluctuation of magnetization [9, 10]. It well know that large K_u can resist thermal fluctuation of magnetization even the particle size is very small. The K_u value of FePt alloy is as high as 7×10^7 erg/cm³ and the saturation magnetization, M_s is about 680 emu/cm³. The large magneto-crystalline anisotropy energy, may be due to the spin-orbit coupling of Pt atom in which Pt atoms can have some induced moment in ferromagnetic state [11].

The chemical synthesis of very small FePt nanoparticles was reported by Sun et al. [12]. Due to the organic coating, particle size and geometry is well preserved even after thermal treatments at high temperatures. As-made particles crystallize in a disordered fcc phase with a relatively small anisotropy. The order-disorder transformation can be obtained by thermal annealing [13]. In stoichiometric FePt complete order of the high anisotropy $L1_0$ tetragonal phase can be only achieved with a temperature annealing higher than 600°C which undesirably lead to the particle agglomeration and sintering [14].

In this paper, the colloid fcc FePt nanoparticles are fabricated with 3 nm diameter. The size and structure of the FePt have been studied by transmission electron microscopy (TEM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and vibrating sample magnetometer (VSM) analyses before and after heat treatment.

2. EXPERIMENTAL DETAILS

Synthesis of the nanoparticles, involves the reduction of $Pt(acac)_2$ and $FeCl_2$, in phenyl ether solvent. The oleic acid and oleylamine surfactants were added to solvent, at 110°C, as a protective agent in order to prevent agglomeration and oxidation. By adding superhydride at 210°C, the FePt nanoparticles were formed. The black reaction

mixture was cooled to room temperature and then combined with methanol to remove the impurity.

The fcc FePt nanoparticles was stirred until all the solvent evaporates at room temperature and then annealed to complete the fcc to fct transition. The annealing temperature fixed at 675°C for 3.5 hours. Morphology of the FePt nanoparticles before and after annealing was observed by TEM analysis using a Philips EM 208 TEM (100kV) with resolution 200 kX. To determine the nanoparticle's structure, XRD measurement was prepared after evaporation of hexane on a Silicon wafer using a Seifert with $Cu-K_\alpha$ (wavelength= 1.54 Å) radiation. SEM analysis was done using VEGA (15 kV) 50 Kx. The magnetization of FePt samples in a variable magnetic field was measured using vibrating sample magnetometer (VSM).

3. RESULTS AND DISCUSSION

Figure 1 indicates the as-synthesized colloidal FePt nanoparticles. It is realized that the color of the samples changes from light to dark with increasing temperature from 110°C to 210°C. It is because that the monomers are released from precursors with increasing temperature and then the number of nanoparticles increases.

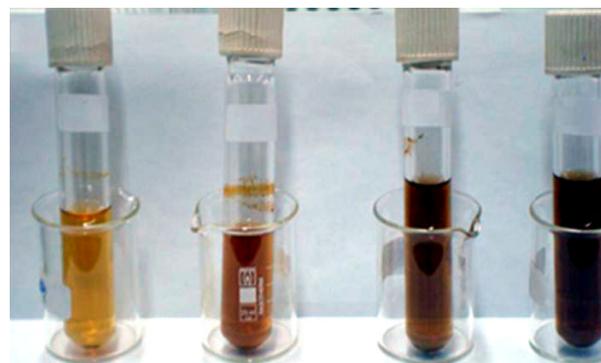


Figure 1: As-synthesized colloidal FePt nanoparticles.

Figure 2a shows (TEM) image of the as-synthesized fcc FePt nanoparticles in presence of oleic acid and oleylamine surfactant stabilizers. The samples were refluxed at 250°C for 20 min. As you

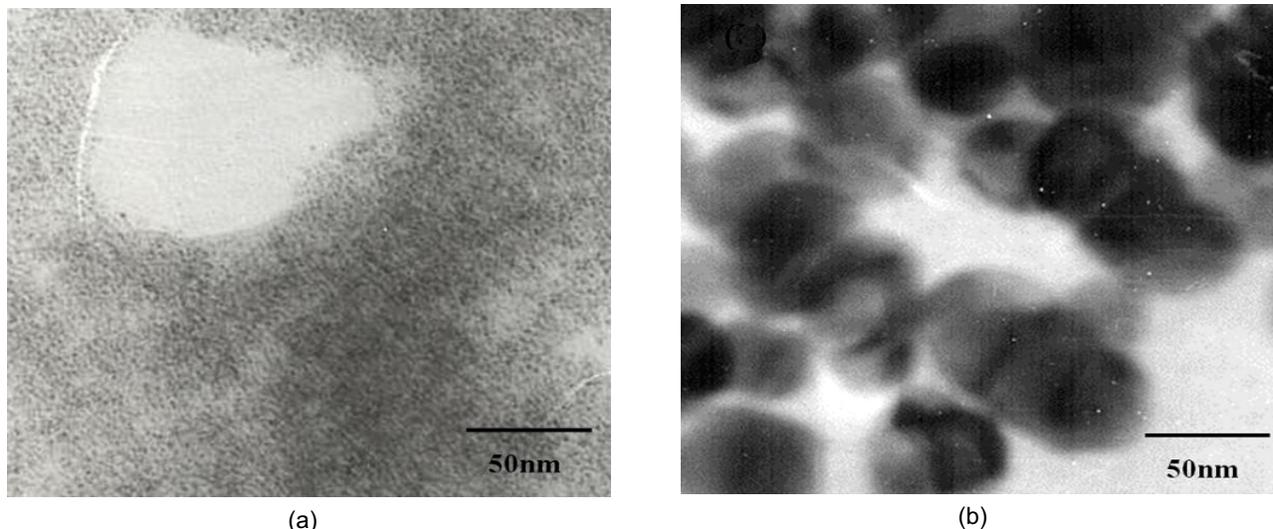


Figure 2: TEM image of the (a) as-synthesis and (b) annealed FePt nanoparticles.

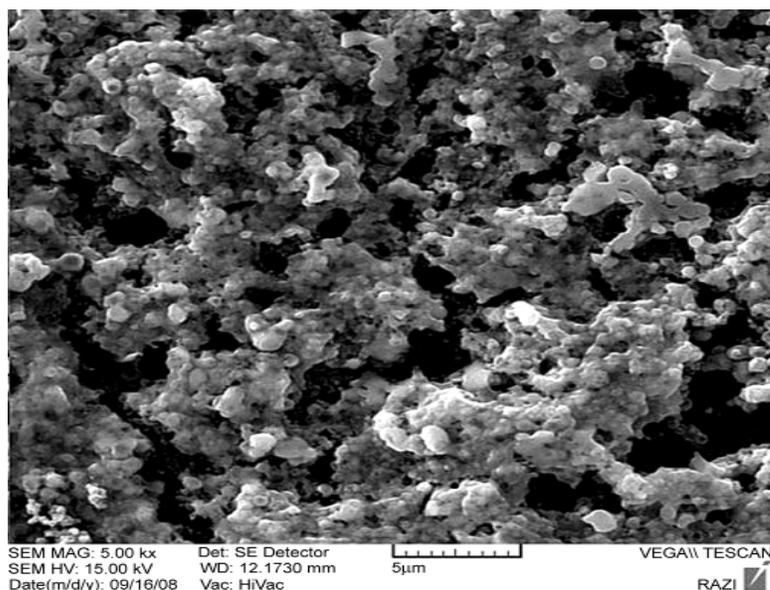


Figure 3: SEM image of the annealed FePt nanoparticles.

can see from the picture, the 3 nm FePt nanoparticles are dispersed on the TEM grid because of surfactant stabilizers. Figure 2b indicates TEM image of the salt-matrix FePt nanoparticles with size of 50 nm in diameter annealed at 675°C for 3.5 hours. It is observed that the nanoparticles agglomerate to each other and the size of them increases [15].

Figure 3 shows the SEM image of the FePt nanoparticles without salt-matrix annealing after

heat treatment. SEM image indicate the size of annealed FePt increases to 1 µm in diameter. In fact, by increasing annealing temperature the surfactants are removed and the FePt agglomerated.

Figure 4 indicates X-ray diffraction (XRD) pattern of the FePt nanoparticles annealed at 675°C for 3.5 hours. The sharp peaks indicate that size and crystallinity of FePt nanoparticles increase after annealing process. These peaks provide evidence of chemical ordering phase fcc to fct transition.

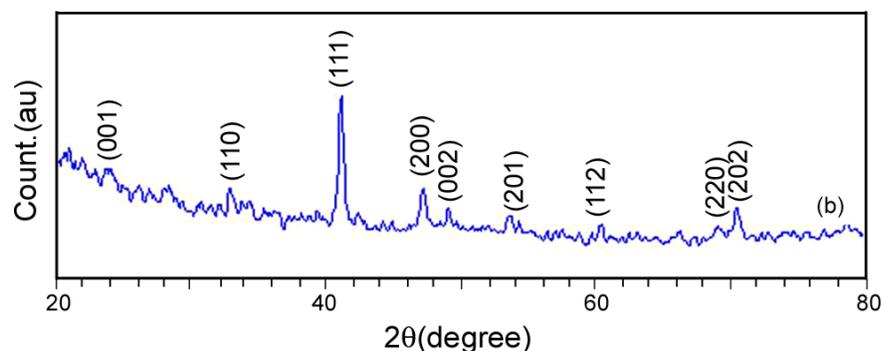


Figure 4: XRD patterns of the annealed FePt nanoparticles.

Figure 5 shows the results of magnetic measurements after heat treatments by vibrating sample magnetometer (VSM). It is understood that after annealing process the saturation magnetization increases to 50 emu/g and the coercivity increases to 6.5 kOe. This high coercivity of fct FePt nanoparticles is because of the high magneto-crystalline anisotropy in $L1_0$ phase.

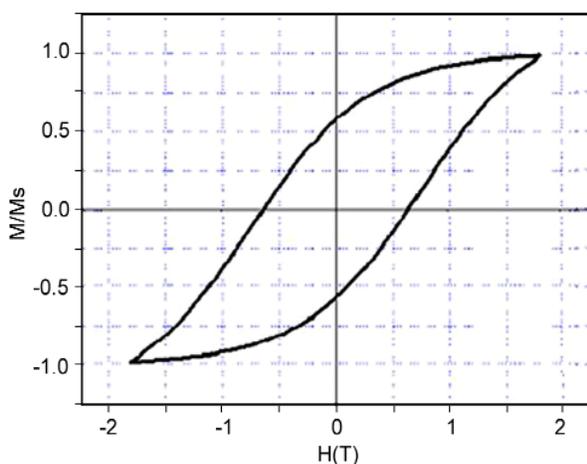


Figure 5: Magnetic hysteresis loops of annealed FePt nanoparticle.

4. CONCLUSIONS

Fcc and Fct FePt nanoparticles were fabricated by chemically route. It was found that by salt matrix annealing the size of FePt nanoparticles increased from 3 nm to 50 nm in diameter after heat

treatment. The size and crystallinity of FePt nanoparticles were increased with increasing annealing temperature. Phase transition from fcc to fct were done after heat treatment.

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