EFFECT OF 1, 2-HEXADECADENIOL AND LiBEt3H SUPERHYDRIDE ON THE SIZE OF FePt NANOPARTICLES

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Monodisperse magnetic FePt nanoparticles have potential applications in high performance high-density magnetic storage media. In this paper, FePt nanoparticles were synthesized by reduction of FeCl$_2$.4H$_2$O and Pt(acac)$_2$ at 250 °C under N$_2$ atmosphere. The effect of 1,2-Hexadecadeniol and LiBEt3H on the size and uniformity of FePt nanoparticles have been studied. TEM observations revealed that 1,2 Hexadecadeniol as reducing reagent, first create the 2 nm Pt core by releasing Pt atoms from Pt(acac)$_2$ in nucleation stage at 190 °C and then intense LiBEt3H superhydride increase the size of FePt nanoparticles to 5 nm by releasing the Fe atoms from FeCl$_2$.4H$_2$O in growth stage at 210 °C, when added to the reaction solution. The mean diameter of FePt nanoparticles decrease to 4 nm by increasing reaction temperature to 250 °C in reflux stage. XRD patterns showed that FePt nanocrystals form L1$_0$ superlattice structure after annealing at 700 °C for 4 hours. The results of EDS analysis indicated that the composition of FePt nanoparticles in nucleation, growth and beginning of the reflux stage give Fe$_{14}$Pt$_{86}$, Fe$_{63}$Pt$_{37}$, and Fe$_{58}$Pt$_{42}$ stoichiometry, respectively.

Key word: Superhydride, FePt Nanoparticles, synthesis, reflux, reducing agent

INTRODUCTION

Research on FePt nanoparticles is attracting a lot of attention because FePt nanoparticles form a good research for physics in nanoscale, [1–4] and also from the chemically ordered L1$_0$ FePt nanoparticles are future candidates of extremely high areal density magnetic recording media [5–7].

In recent years, lots of research works have been devoted to Fe-Pt alloy nanoparticles. Chemical methods have been widely used to produce nanosized materials due to their straightforward nature and their potential to prepare large quantities of the final product [8]. Furthermore, they provide particles that are easily dispersible in liquid media, and therefore give the opportunity to fabricate nanostructured devices through self-assembly [9]. The synthesis of discrete magnetic
Effect of 1, 2-Hexadecanediol and LiBEt3H superhydride on the size and uniformity of FePt nanoparticles during synthesis was studied by TEM and EDS analysis. The crystal structure of FePt nanoparticles, before and after annealing has been investigated by XRD analysis.

**EXPERIMENTAL DETAILS**

The face center cubic (fcc) FePt nanoparticles with size of 3.5 nm were prepared using the synthesis described by Sun et al. [3]. Synthesis of the FePt nanoparticles involves the reduction of Pt(acac)2 (197 mg) and FeCl2·4H2O (139 mg) in phenyl ether solvent (25 mL) in the presence of 1,2-hexadecanediol (250 mg). Oleic acid (0.16 mL) and oleylamin (0.17 mL) surfactants were added to the solvent at 100 °C as a protective agent, in order to prevent agglomeration and oxidation. By adding superhydride (LiBEt3H) (2 mL) under a blanket of N2 at 200 °C, followed by refluxing, the FePt nanoparticles were formed. The refluxing temperature was fixed at 250 °C. The black reaction mixture was cooled to room
temperature and then combined with ethanol to remove the impurity. The product was precipitated and separated by centrifugation (8500 rpm, 10 min). Any undissolved material was removed by centrifugation. Then FePt nanoparticles were dispersed in hexane solution (40 mL) in the presence of surfactants.

To determine the composition of FePt nanoparticles in nucleation and growth stage, energy dispersive spectroscopy (EDS) analysis (15 kV) was carried out. The specification of the size and shape of FePt nanoparticles were examined by transmission electron microscopy (TEM) analysis using a Philips EM 208 TEM (100 kV) with a resolution of 200 kX. The samples were annealed in a 90% Ar + 10% H₂ atmosphere at 700 °C for 4 hours. X-ray diffraction measurements (XRD) were performed to determine the crystalinity of L₁₀ - FePt nanoparticles structure, using a Seifert system with Cu-Kα radiation (λ = 1.54 Å).

RESULTS AND DISCUSSION

Figure 1 shows the XRD spectra for the FePt nanoparticles before and after annealing. The large width of the peaks (Figure 1a) indicates that the as-synthesis FePt nanoparticles are superparamagnetism in nanometer scale with fcc structure. After annealing at 700 °C for 4 hours under a flow of 90% Ar + 10% H₂, extra peaks appeared (Figure 1b). These peaks provide evidence of a chemical ordering phase (L₁₀) transition.

![Fig. 1 – X-ray diffraction (λ = 1.54 Å, Cu-Kα radiation) pattern of a) as-prepared and b) annealed FePt.](image)

Figure 2 show the TEM images of the as-synthesized fcc FePt nanoparticles. In Figure 2a, the Pt core were created with size of 2 nm at 190 °C by 1,2 Hexadecadieniol as reducing reagent. By adding intense LiBEt₃H superhydride to the reaction solution at 210 °C, the Fe atoms were released from FeCl₂·4H₂O and
penetrated to Pt core and the size of FePt nanoparticles increased to 5 nm (Fig 2a.). By increasing temperature reaction to 250 °C at the beginning of the reflux stage some Fe atoms were separated from the shell of FePt nanoparticles and the size of nanoparticles decreased to 4 nm. The uniformity of FePt nanoparticles was improved with increasing temperature reaction in reflux stage. The results of EDS analysis confirm the TEM observations (Fig. 2c).

Figure 2 – TEM images of the as - synthesis FePt nanoparticles after evaporation of hexane. (a) 2 nm at 190 °C; (b) 5 nm at 210 °C and (c) 4 nm at 250 °C.

Figure 3 shows a characteristic spectrum collected by EDS analysis. By comparing the area under each peak to a set of standards with known element concentrations, the concentration of the elements could be quantified, and the compositions gave Fe_{14}Pt_{86}, Fe_{63}Pt_{37}, and Fe_{58}Pt_{42} stoichiometry. It is realized that the Pt atoms are released by 1,2 Hexadecadienol as reducing reagent at 190 °C and the core of FePt constituted by Pt with Fe_{14}Pt_{86} stoichiometry (Fig. 3a). When intense LiBEt3H superhydride are added at 200 °C, the Fe atoms are released from FeCl2·4H2O and penetrate to Pt core quickly and the composition of nanoparticles changes to Fe_{63}Pt_{37} (Fig. 3b). By increasing reaction temperature to 250 °C, some weak connection between Fe atoms on the surface of FePt nanoparticles removed from the shell and the composition of nanoparticles changes to Fe_{58}Pt_{42} (Fig. 3c) that closed to Fe_{50}Pt_{50} stoichiometry.
Fig. 3 – Energy dispersive spectroscopy (EDS) pattern of nanoparticles.
CONCLUSION

Uniform FePt nanoparticles were successfully synthesized with a mean diameter of 4 nm by the polyol method. The effect of 1,2-Hexadecadonol and LiBEt₃H on the size and uniformity of FePt nanoparticles was studied. The TEM and EDS results demonstrated that the first Pt core was formed in the nucleation stage by 1,2-Hexadecadonol as the reducing agent, and then Fe atoms were released by intense LiBEt₃H superhydride and surrounded the Pt core as a shell in the growth stage. FePt nanoparticles were constituted. By increasing temperature in the reflux stage, the uniformity of the FePt nanoparticles was improved, and the stoichiometry of nanoparticles reached Fe₅₈Pt₄₂. The XRD spectra indicated that the FePt nanoparticles form L₁₀ structure after annealing.

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