A novel preparation of salt-matrix FePt nanoparticles

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To transfer face-center-cubic (FCC) FePt nanoparticles to the face-center-tetragonal (FCT) phase with high magnetic anisotropy in ultra-high density magnetic recording media heat treatment is necessary. The heat treatment leads to agglomeration and sintering of the nanoparticles. To prevent the particles from sintering, salts are used as the separating media. The salt particles were dispersed by adding hexane to the solution saturated of salt water by ultra-sonic vibration. Through this method, NaCl crystallized particles with uniformity in shape and average size about 1 μm have been obtained to mix with 3.5 nm FePt magnetic nanoparticles. Transmission electron microscopy (TEM) data show well-separated annealed FePt nanoparticles.

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1. Introduction

FePt magnetic nanoparticles prepared by Sun et al.[1], should be self-assembled with high magnetic anisotropy for ultra-high density magnetic recording media. To transfer FePt nanoparticles from FCC phase to FCT phase, heat treatments above 600 ^{o}C are necessary, which undesirably leads to sintering of these nanoparticles [2]. The salt-matrix annealing is a new method which prevents sintering of nanoparticles. Salt acts as a protective agent for prevention of nanoparticles coalescence at high temperature [3]. For this purpose, salt particles sizes should be very small. One way to crumble the salt powder is ball milled method for more than 24 hours. By this approach, the salt particles have an average size about 20 μm which is not suitable for separating media. By using chemical method, pure NaCl powder is solved in de-ionized water to reach saturation and then hexane solution is added to the saturated solution of salt water. Ultrasonic vibration leads to formation and dispersion of the small salt crystallized particles in hexane phase. In fact, the ultra-sonic vibration extracts the Na^+ and Cl^- polar atoms from solution of salt water and form small NaCl crystals as dispersion in nan-polar hexane solution. By this method, we can fabricate NaCl salt particles with an average size about 1 μm. The FePt nanoparticles have been prepared by Sun method [1]. To obtain self-assembled FePt magnetic nanoparticles the as-synthesis FePt particles are combined with salt particles in hexane solution and after annealing, the salts are washed with water in order to remove them from FePt particles**.**

In this paper, we report a new approach to crumble the salt powder by chemical method in order to mix with as-prepared FePt nanoparticles dispersed in hexane. The effects of volume of hexane solution and ultra-sonic duration on NaCl particle size have been investigated.

2. Experimental

Pure sodium chloride 99.5% (NaCl) and hexane solution were purchased from Merck Co. The solution of salt and water was made by adding NaCl powder to de-ionized water until it reached to saturation. Samples were prepared according to table 1 at room temperature. The volume of 10mL hexane solution was added to the saturated solution of salt water in different periods of time and also ultra-sonic process was performed simultaneously. To achieve smaller salt particles the ultra-sonic duration was increased from 30 min to 45 min. The as-synthesis FePt nanoparticles were mixed with the saturated solution of salt water dispersed in hexane solution and stirred until hexane evaporated at room temperature. Then salt-matrix FePt nanoparticles were annealed at 700 ^{o}C for 4 h. The specification of size and shape of nanoparticles, distance between them, and also particles size distribution, were carried out by TEM analysis using a Philips EM 208 TEM (100KV) with resolution 200KX. To determine the nanoparticles structure, the X-ray diffraction (XRD) measurement was prepared after evaporation of hexane on a Silicon wafer using a Seifert with Cu-K_{α} (wavelength = 0.154 nm) radiation.

3. Results

In table 1 the samples prepared in different ultra-sonic duration and volume of hexane were listed. The salts were dispersed in hexane phase. It was observed that the salt particles size is decreased with increasing the ultra-sonic duration. Due to extraction of the NaCl particles from water phase by ultra-sonic vibration and crystallization of them in hexane phase, some water with salt particles enters into hexane phase which was observed after evaporation of hexane. In order to solve this problem increased the volume of hexane solution related to saturated solution of salt water.

Figure 1 shows the microscopic images of the salt particles. Figure 1A shows sample 1 with salt water concentration of 150 mg/ml and an average salt particle size about 20 μm and ultra-sonic duration of 30 min. Figures 1B, C, D, E reveals samples 2,3,4,5 respectively, with salt water concentration of 190 mg/ml and an average salt particle size about 1 μm and ultrasonic duration of 45 min.

Table 1. The list of salt particles prepared in different condition. Ultra-sonic time for sample 1 is 30 min and for samples 2,3,4,5 is 45 min.

Sample	Hexane (mL)	Period of adding hexane per (min)	Salt particles sizes (μm)
1	40	10	20
2	20	10	0.9
3	30	10	1
4	40	8	1.25
5	50	6	1.5

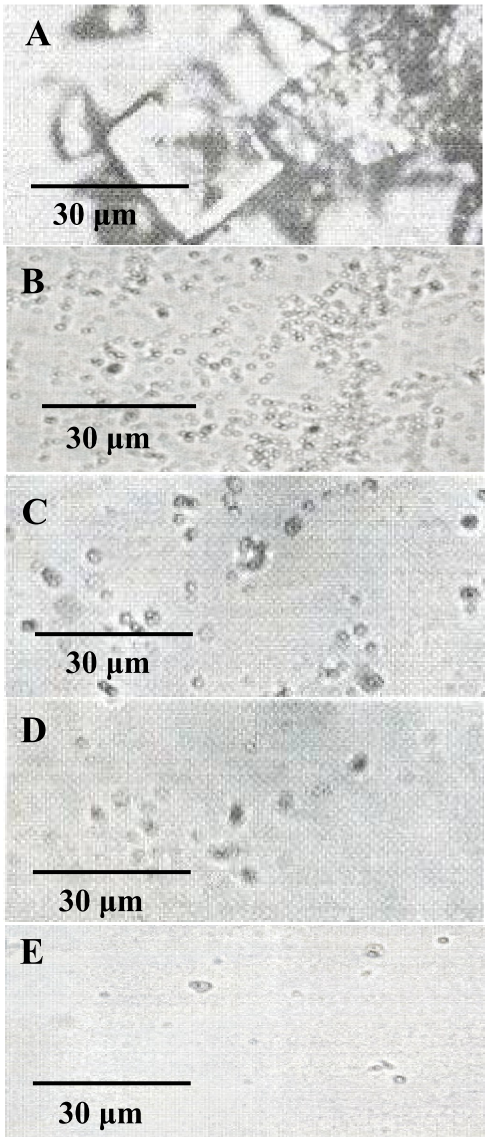

Figure 1. The images of salt particles according to table 1.

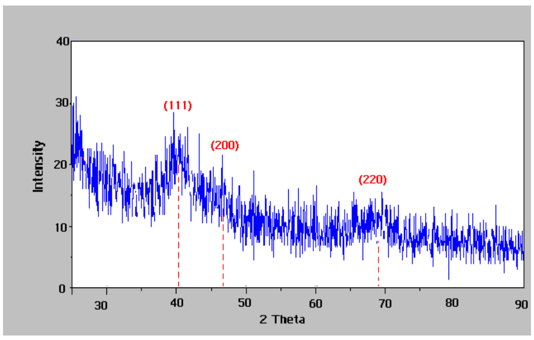

Figure 2. X-ray diffraction (λ=1.54 Å, Cu Kα radiation) pattern of the as-prepared FePt nanoparticles.

Figure 2 shows X-ray diffraction (XRD) pattern for FePt nanoparticles before heat treatment. Some peaks at 40.3°, 46.9°, and 68.5° are seen which reveals an FCC structure with lattice constant 3.871 A° and is consistent with the report made on FePt structures analysis [4].

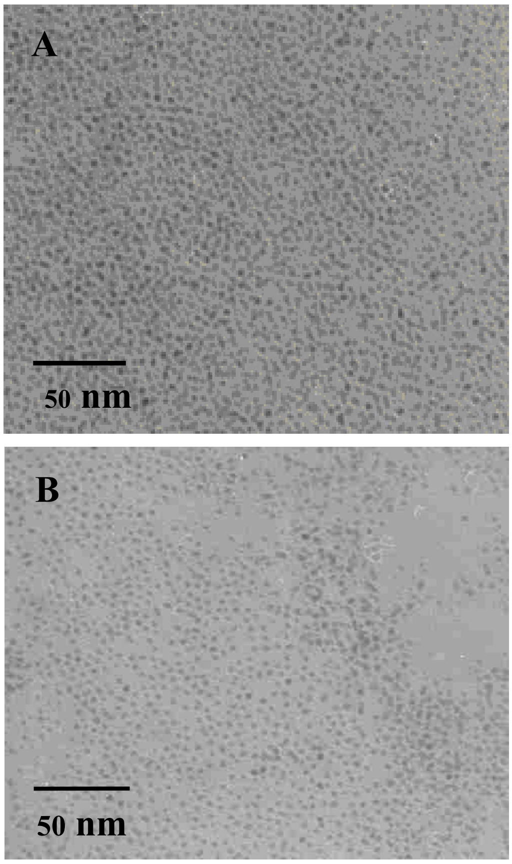

Figure 3. TEM images of the (A) as-synthesis and (B) annealed FePt nanoparticles at 700°C for 4 h.

Figure 3 show the TEM images of FePt nanoparticles prepared by this method before and after annealing. Figure 3A shows as-synthesis nanoparticles with average size about 3.5 nm which particles were stabilized with surfactants [5] and figure 3B shows FePt nanoparticles prepared by salt-matrix process with average size about 4.5 nm annealed at 700 °C for 4 h.

4. Discussion

According to table 1 the volume of hexane is affected to form the NaCl crystal particles without water in hexane phase. As we can see in figure1 by increasing the ultra-sonic duration the mean salt particles sizes is decreased and, also due to increasing the volume of hexane solution simultaneously, the number of crystalline NaCl particles decreases in hexane phase (Fig. 1E). Surprisingly, the salt water solution phase disappeared from hexane solution phase for samples 2, 3 contrary to samples 4, 5. In fact, the small amount of saturated solution of salt water relative to hexane solution, leads the salt crystal particles with water enter into hexane solution by ultra-sonic vibration which is undesirably not appropriate to mix with FePt nanoparticles dispersed in hexane because of oxidation. The large width of the peaks in X-ray pattern indicates that FePt particles are in nanometre scale. The average particle size is about 3.5 nm which is estimated using Scherer's formula on (111) peak. From TEM images we can see the uniformity of nanoparticles in size and distances between FePt nanoparticles after annealing by salt matrix method.

5. Conclusions

The NaCl salt powder was crumbled by chemical method to mix with as-synthesis FePt nanoparticles. The volume and duration of adding hexane were affected to form the NaCl crystal particles without water in hexane phase and also by increasing the ultra-sonic time the salt particles sizes decreased. In order to prepare salt-matrix FePt nanoparticles, the salt particles size was decreased. By this technique, the salt particles were fabricated with uniformity in size and shape and average size about 1 μm that is appropriate to mix with FePt nanoparticles in order to prevent sintering after annealing. By this salt-matrix method the 3.5 nm annealed FePt nanoparticles settle in about equal distances of 3 nm from each other.

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