

Phase Transformation of FePt Nanoparticles

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To transfer face-centered cubic (fcc) FePt nanoparticles to face-centered tetragonal (fct) structure with high magnetic anisotropy, heat treatments are necessary. The heat treatments, however, often lead to agglomeration and sintering of the nanoparticles. To prevent the particles from sintering, a new method, the salt-matrix annealing, has been adopted in our experiments recently. The fcc nanoparticles produced by chemical synthesis were mixed with NaCl or other salt powders. The mixture was then annealed at temperatures up to 750 °C to complete the fcc to fct phase transition. After the annealing, the salt was washed out by water and monodisperse fct FePt nanoparticles free of non-magnetic impurities were obtained. Magnetic measurements show very high coercivity (up to 30 kOe) of the monodispersed fct nanoparticles. Size dependence of the phase transition and alignment of the ferromagnetic nanoparticles have also been investigated.

Index Terms—Anisotropy, permanent magnets, phase transitions, nanoparticles.

I. INTRODUCTION

FERROMAGNETIC nanoparticles with high magnetic anisotropy are essential for advanced applications like high energy-product nanocomposite magnets and high density magnetic recording media. Due to thermal fluctuation, most available magnetic particles, however, are superparamagnetic at room temperature when the particle size is down to the order of nanometers. FePt nanoparticles are expected to have very high magnetic anisotropy and, therefore, have attracted tremendous attention [1]–[3]. Among currently available techniques for preparing FePt nanoparticles, wet chemical methods produce the nanoparticles with well controlled particle size and size distribution. Unfortunately, as-synthesized FePt nanoparticles always have the face-centered cubic (fcc) structure and have no magnetic ordering at room temperature. Heat treatments to convert the fcc structure to the face-centered tetragonal (fct) structure with high magnetic anisotropy must be performed at elevated temperatures (>600 °C) which undesirably lead to the particle agglomeration and sintering. To solve this dilemma, extensive efforts have been made since 2000 to obtain monodisperse fct FePt nanoparticles. One of the approaches in such efforts is to lower the onset temperature for the fcc to fct phase transformation. Doping by elements such as Ag, Au, and Cu in the FePt phase was tested resulting in the onset of the fct phase at 400 °C [4]–[9]. Direct synthesis of the fct structured particles by the polyol process using high boiling point solvents is another major approach [10]–[13]. In this approach, higher synthesis temperatures can be applied which allows partial formation of the fct phase. Recently, methods have been tested to coat the fcc FePt nanoparticles with high-melting-point materials and then to anneal the mixture to obtain the fct nanoparticles without sintering. Immiscible silica matrix [14], polyethyleneimine linker molecules [15], SiO₂ shells [16], [17] and TiO₂ matrix [18] were used as the high-melt-point materials. The advantage of this coating approach is that high temperature annealing can be performed without causing particle sintering. The coating materials cannot be easily removed after the annealing so that these methods are not suitable for

producing in large quantity monodisperse magnetic nanoparticles free of nonmagnetic impurities.

Other approaches to obtaining monodisperse fct nanoparticles include rapid thermal annealing [19] and pulse laser annealing [20]. To date, however, it is still a very challenging task to obtain both high coercivity and monodisperse morphology simultaneously of fct FePt nanoparticles. Most reported results show a trade-off between the two properties. It is, therefore, highly desirable to find processing techniques to produce completely transformed fct FePt monodisperse nanoparticles.

A new technique has been adopted in our group recently, based on salt-matrix annealing at temperatures up to 750 °C, with which monodisperse fct FePt nanoparticles have been successfully produced [21], [22]. In this invited paper we present our recent research results in producing monodisperse ferromagnetic FePt nanoparticles. We will demonstrate that during the salt-matrix annealing, the fcc-fct phase transition in the nanoparticles can be completed while the particle size and shape are well retained. The annealed fct nanoparticles have huge coercivity values (up to 30 kOe) and other remarkable magnetic properties.

II. EXPERIMENTAL PROCEDURE

Our experimental concept originates from the separation between the FePt nanoparticles during annealing. After testing different organic and inorganic materials as the separation media, we chose salts soluble in water with melting points higher than 700 °C (including chlorides, sulfides and carbonates of Na, K and Ca) as the separating media. The salt powders were ball milled for more than 24 h to reach particle size smaller than 20 microns before they were mixed with the fcc FePt nanoparticles. The fcc FePt nanoparticles with different size (2, 4, 6, 8, and 15 nm) and shape were synthesized by standard airless techniques in an argon atmosphere with adjustable synthesis parameters [1], [23]–[25]. Weight ratios of the salts to the fcc nanoparticles were varied. In case of NaCl, ratios including 1 : 1, 2 : 1, 4 : 1, 8 : 1, 20 : 1, 30 : 1, 40 : 1, 100 : 1, 200 : 10 and 400 : 1 have been tested. [22] The mixtures were then annealed at temperatures from 600 °C–750 °C for 2–8 h under a neutral or reducing atmosphere (e.g., “forming gas” with 95% Ar + 5% H₂). After annealing, the salts were removed from the samples by

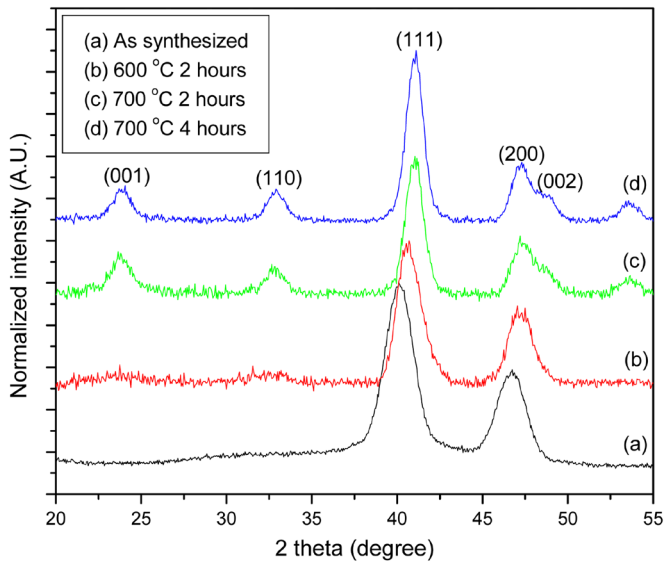


Fig. 1. XRD patterns of the as-synthesized and annealed 6 nm FePt nanoparticles. (Color version available online at <http://ieeexplore.ieee.org>.)

washing the mixtures in deionized water followed by centrifugation. Water was decanted from the centrifuge tube and then the previous step was repeated again to ensure that the salts were completely removed from the samples. The remaining particles were then rinsed with acetone and were centrifuged again. Dispersions of the fct FePt nanoparticles were eventually obtained in an organic solvent such as ethanol.

Among all the tested salts, it was found that NaCl gives the best results because of its high stability at the annealing temperatures and high solubility in water. It is also the most economic choice in view of its availability. NaCl powder from a supermarket was tested and good monodisperse fct nanoparticles were obtained.

Transmission electron microscopy (TEM), powder X-ray diffraction (XRD), inductively coupled plasma (ICP) and magnetization measurements were used to characterize the FePt nanoparticles before and after the salt-matrix heat treatments. A laser particle size analyzer was also used for checking the particle size. The magnetic hysteresis measurements were carried out by using a superconducting quantum interference device (SQUID) magnetometer with field strength up to 7 tesla (T) at different temperatures for the fct particles embedded in hardened epoxy, or in frozen alcohol with and without magnetic field alignment.

III. RESULTS AND DISCUSSIONS

A. The fcc-fct Phase Transition

XRD shows that sufficiently annealed particles in the salt matrix have the fct structure. Fig. 1 shows the XRD patterns of the 6 nm FePt nanoparticles before and after the annealing in different conditions. It was found that the (001) and (110) peaks characteristic for the fct FePt phase started to appear when the nanoparticles were annealed in NaCl matrix at 700 °C for 2 h. With increasing annealing time, the fcc-fct phase transition was more complete, shown by the more pronounced (001) and (110) peaks. From the XRD pattern, no peaks of NaCl was found, indicating that salt was not left after washing the mixture with water. ICP result also confirmed that the NaCl level is very low

(only 0.099% in weight) and the average composition of the annealed particles is $\text{Fe}_{52}\text{Pt}_{48}$.

Magnetization values of the salt-matrix annealed FePt nanoparticles were measured to check if there was any deterioration caused by contamination from the salt matrix during the annealing or from the water exposure during washing. For the recovered particles of 15 nm with salt to particle ratio of 40:1, for instance, the magnetization values at 7 T (which are considered to be close to the saturation magnetization) are listed as follows: annealed at 600 °C for 2 h, 733.5 emu/cc; 700 °C, 2 h, 744 emu/cc; 700 °C, 4 h, 841.5 emu/cc. It is seen that the magnetization values increase with the annealing time and temperature. These magnetization values are comparable to FePt particles annealed on a Si substrate under the same conditions without salt.

Similar phase transition upon annealing was observed for nanoparticles of 4, 6, 8, and 15 nm. In case of 2 nm FePt particles, XRD did not give any peaks before and after the annealing, due to the very small particle size. Therefore, electron diffraction with a TEM has been used to perform the phase identification. It was found that even after being annealed at 700 °C for 8 h, only (111) and (200) fundamental electron diffraction rings can be found and all superlattice rings are missing for all of the 2 nm samples, which indicates that the fcc-to-fct transition did not occur even the nanoparticles were annealed for long time.

B. The fct Particle Morphology

Morphology of the salt-matrix annealed nanoparticles was closely monitored by TEM observations and a laser particle size analyzer. It was found that the annealed particles are easier to be aggregated than for the fcc particles because of the magnetic attractive force between the fct particles. This kind of aggregations can be re-dispersed by vibration. Monodisperse nanoparticles can be observed with proper operations in TEM specimen preparations. It has been confirmed that this methodology has successfully prevented the particles with sizes from 2 to 15 nm from sintering and growth at 700 °C which is high enough for a complete phase transformation from the fcc to the fct structure [22]. It is also important to note that the particle shape can be also retained with this annealing method. The 8 nm fct particles have cubic or rectangular shape, as their precursors before the annealing, indicating that nanoparticle shapes survived the heat treatment. It is, thus, concluded that this technique can be also applied to heat treatments of nanorods, nanowires and other isolated micro- and nanostructures when sintering of the nanostructure is not desirable.

Fig. 2 shows the TEM images of the 4 and 8 nm as-synthesized fcc FePt particles and the annealed fct particles. It is seen that the annealed particles are still monodisperse with unchanged particle size and shape.

It has been found that the salt to particle ratio has to be higher than 20 : 1 [22]. The lower salt ratios gave significant particle sintering and agglomeration. Annealing at higher temperatures and extended time should be accompanied with higher salt to FePt ratio in order to avoid sintering. From our experimental observations, salt particle size has no significant effect on the annealing and the presence of salts has not altered the fcc to fct transition temperature. It was also found that the higher the salt-to-particles ratio, the lesser the sintering. In case of the 15

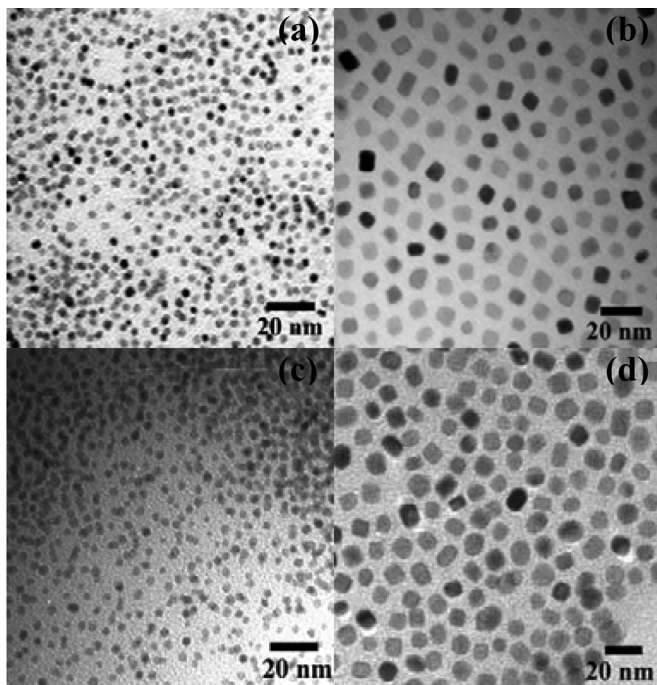


Fig. 2. TEM images of the as-synthesized (a) 4 nm and (b) 8 nm particles. Images (c) and (d) show the particles of the 4 and 8 nm particles after being annealed at 700 °C for 4 h, respectively.

nm FePt particles, when the ratio is lower than 40 : 1, monodisperse fct nanoparticles cannot be obtained from the heat treatment at 700 °C for 2 h. Lower salt ratios give significant particle sintering and agglomeration.

It was also found that smaller particles need more time to complete the phase transition. In our experiments, after being annealed for 2 h at 700 °C, the fcc particles with 4 and 8 nm sizes were not transformed to fct completely. Longer annealing time was required to complete the transformation from fcc to fct phase for these particles compared with the 15 nm particles. When the annealing time was increased, higher NaCl-to-FePt ratio was required to prevent the particles from sintering. For example, when the ratio was increased to 100 : 1 and annealing time was extended to 4 h, monodisperse 4 nm FePt particles were obtained. However, the 100 : 1 salt-to-particles ratio was not sufficient to prevent sintering when the annealing time was increased to 8 h. It was found that the sintering was successfully prevented again when the ratio was increased from 100 : 1 to 400 : 1. Annealing of larger FePt particles at higher temperatures and extended time should be accompanied by higher salt to FePt ratio in order to avoid sintering.

In order to double check if the TEM observations gave a real picture of the particle morphology, XRD analyses were also used to calculate the grain size of the particles. The (111) peaks were chosen to calculate the grain sizes as demonstrated in Fig. 3. The average grain sizes of the FePt nanoparticles annealed at 700 °C for 4 h were obtained as 4.7, 6.8, 8.2, and 13.3 nm for the 4, 6, 8, and 15 nm particles, respectively, showing an excellent agreement to the TEM observations. These grain sizes also represent the particle sizes because all of the particles are single crystalline except the 15 nm particles. The relatively

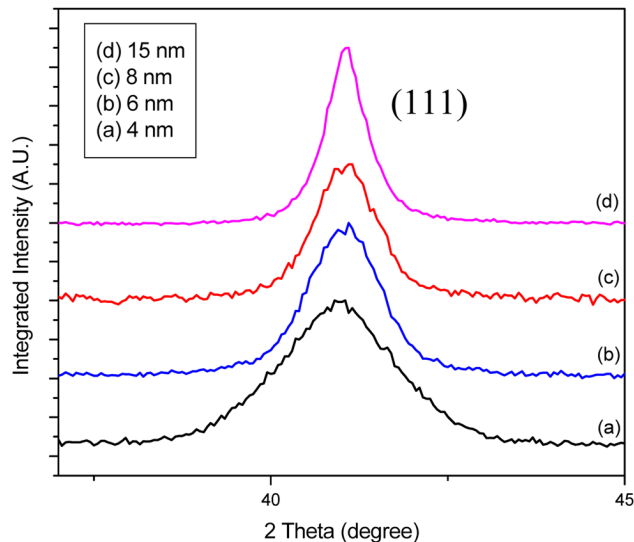


Fig. 3. (111) XRD peaks of 4, 6, 8, and 15 nm FePt nanoparticles annealed at 700 °C for 4 h. (Color version available online at <http://ieeexplore.ieee.org>.)

smaller grain size for the 15 nm particles is obviously related to the polycrystalline structure of the 15 nm fct FePt nanoparticles [21]. Thus, it is also confirmed by XRD that salt-matrix annealing completes the fcc-to-fct phase transition and prevents the grain growth during the heat treatments.

C. Magnetic Hardening

The annealed fct particles were mixed with epoxy to measure their magnetic properties. After the epoxy dried the magnetization loops were measured with a SQUID magnetometer in different directions. Fig. 4 shows the hysteresis loops of the fct nanoparticles annealed in different conditions. Not surprisingly, the particles exhibited giant coercivity up to 30 KOe at room temperature. In all the cases of the annealed particles, when the XRD patterns show the fct structure, coercivity values of the isolated fct particles in epoxy are normally above 15 KOe at room temperature. The 8 nm fct particles have the highest coercivity, which may be related to their faceted shape. It can be seen that the hysteresis loops of the 4 nm particles shows kink even after being annealed for 8 h. This may be attributed to the size dependent phase-transition behavior, as we will discuss in more details. In the relatively small nanoparticles, the transition is not as complete as in large particles which give the two-phase behavior as shown by the loops.

It is also interesting to note that the magnetization of the fct particles is also strongly size dependent. Larger particles have higher magnetizations. This dependence can be related to the reduced magnetization of atoms on surface of the particles.

Fig. 5 shows temperature dependence of coercivity values of the 4, 8, and 15 nm fct nanoparticles. It is interesting to see that the magnetic hardening of 4 nm particles was more temperature dependent than particles with large size. The reason remains to be further studied.

Success in obtaining the ferromagnetic nanoparticles with high coercivity marks the availability of the smallest permanent magnets (down to 4 nm). Since the anisotropic field for fct FePt is very high, even higher coercivity can be expected with a more favorable morphology of the fct nanoparticles.

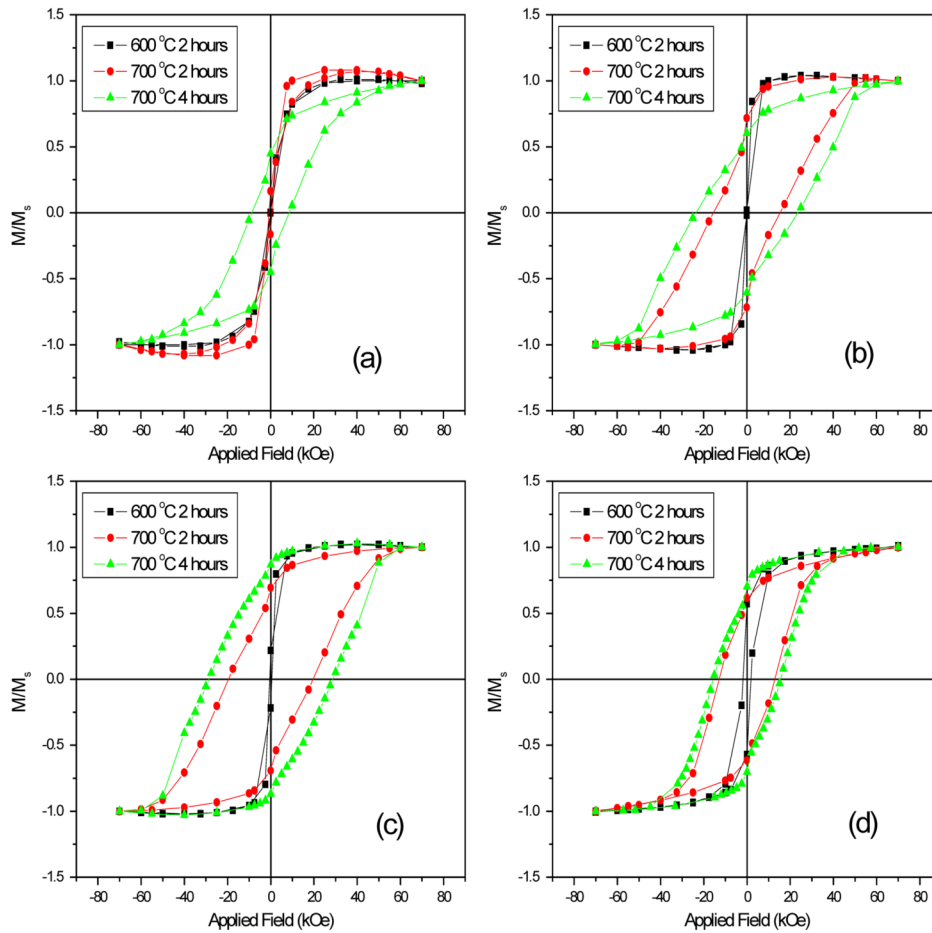


Fig. 4. Hysteresis loops of FePt nanoparticles with sizes of (a) 4, (b) 6, (c) 8, and (d) 15 nm annealed in different conditions. (Color version available online at <http://ieeexplore.ieee.org>.)

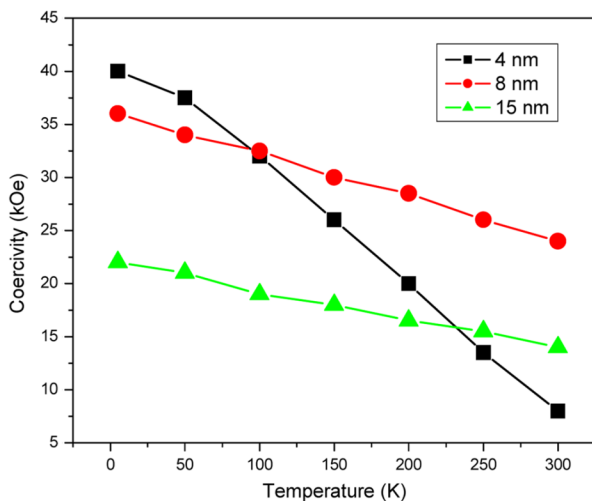


Fig. 5. Coercivity versus temperature for different particle sizes. (Color version available online at <http://ieeexplore.ieee.org>.)

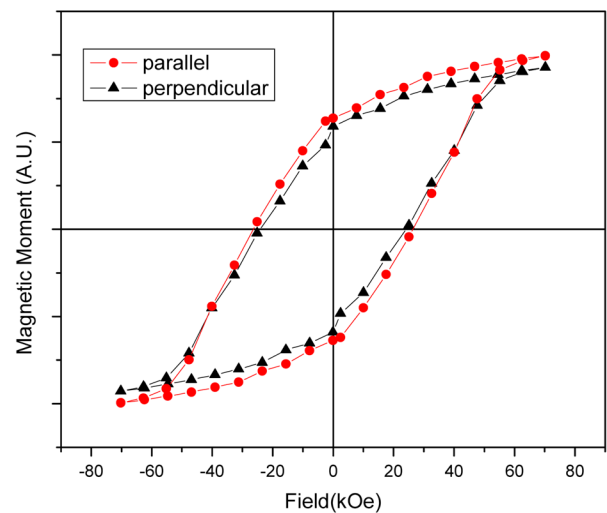


Fig. 6. Hysteresis loops of 8 nm fct FePt nanoparticles aligned under 29 T magnetic field mixed in epoxy. (Color version available online at <http://ieeexplore.ieee.org>.)

D. Alignment of the fct Nanoparticles

It was supposed that ferromagnetic particles with magnetocrystalline anisotropy can be aligned in a magnetic field. However, it has been difficult to observe differences between magnetization curves measured in different directions of the

epoxy specimens made by aligning in a magnetic field. The alignment was performed in a magnetic field up to 29 T.

One reason for this phenomenon is that the fct particles (small permanent magnets) attract each other to form large aggregations, which makes the rotation of individual particles

difficult, or even impossible. We observed the agglomerations in solutions of the fct nanoparticles. It is interesting to note that when the nanoparticle solutions are diluted, monodisperse fct nanoparticles deposited on substrates with patterns can be observed, as we have seen from the TEM images, which is probably related to thermal fluctuation and coherency between the particles and the substrates. In the epoxy specimens for magnetization measurements, the nanoparticle concentration must be high enough in order to obtain magnetic signals, which causes the agglomeration.

Another reason for the difficulty in aligning the fct nanoparticles may be related to the difficulty in rotating the very fine nanoparticles in epoxy because of the high viscosity. The high viscosity of epoxy and the huge surface areas of the nanoparticles can make the friction extremely high, therefore, the rotation can hardly be realized. To reduce the friction, alignment of the particles in alcohol or water was performed in a magnetic field of 7 T. The specimens were cooled down in the magnetic field from room temperature at which alcohol and water are liquids, to temperatures below the ice points for alcohol and water and then the demagnetization curves were measured. These curves were then compared with the curves measured for samples cooled without magnetic field (randomly aligned particles). Significant difference has been found, although we were not able to saturate the magnetization with 7 T magnetic field at 10 K. [21]

The third reason for the difficulty in aligning the nanoparticles is associated with an observation of the polycrystalline feature of the annealed fct particles. HRTEM shows that in case of large size particles (15 nm), polycrystalline particles take the portion of higher than 50% in the annealed particles, from a rough estimation. [21] In case of polycrystalline nanoparticles, the domain-grain relationship can be complicated and the magnetization reversal cannot be described in a simple model. Extended study of this issue is needed.

In case of single-crystal and single-domain nanoparticles, higher magnetic fields should work better for aligning the magnetic nanoparticles. We used a high magnetic field of 29 T at National High Magnetic Field Laboratory at Florida to align the 8 nm fct particles in a liquid PVC binder [26]. After the binder was dry, we took the sample out and measured the hysteresis loops (shown in Fig. 6). Detectable difference between the loops measured in different directions can be found, although no demagnetizing calibration was made.

Recently, alignment of the 8 nm fct particle on a TEM grid was observed with an external magnetic field of <1 T [27], which shows again that the fct particles can be aligned if a proper condition can be created.

E. Size Dependent Chemical Ordering and Magnetic Ordering

As we mentioned in the early discussions, it has been observed in our experiments that smaller fcc nanoparticles need longer annealing time to complete the fcc-fct phase transition. It has been reported that experimental and theoretical studies have shown that the degree of chemical ordering in FePt nanoparticles is related to the particle size. Ordering cannot be achieved when the particle size is below a critical point, which is approximately from 2 nm to 4 nm as stated by previous studies [28]–[31]. We carried out a systematic study on the size-dependent chemical ordering of FePt nanoparticles with the 2, 4, 6, 8, and 15 nm fct FePt particles available in our laboratory.

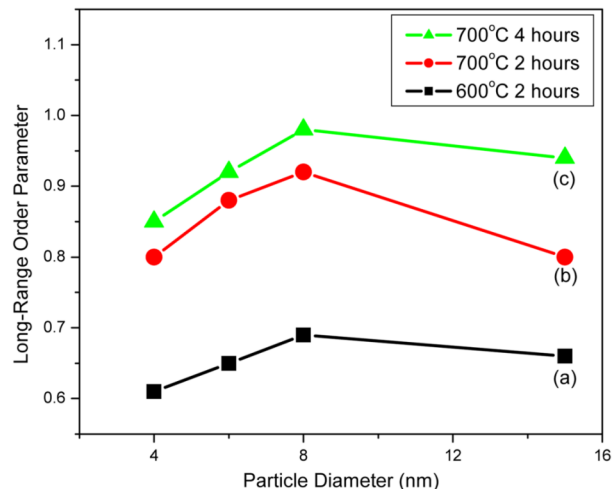


Fig. 7. Long-range order parameter versus particle diameter at the annealing condition of (a) 600 °C for 2 h, (b) 700 °C for 2 h and 700 °C for 4 h. (Color version available online at <http://ieeexplore.ieee.org>.)

The long-range order parameter is determined by the following formula [32]:

$$S \cong 0.85 \left(\frac{I_{001}}{I_{002}} \right)^{1/2} \quad (1)$$

where I is the integrated intensity of [001] and [002] peak from XRD pattern, respectively.

Fig. 7 shows the relation between S and particle diameter at different annealing conditions. As we expect, S increases with increasing annealing temperature and time. It reaches the highest value for different particle sizes when the annealing condition is 700 °C for 4 h. In the case of 2 nm FePt particles, S vanishes because no [001] superlattice structure was found, even after being annealed at 700 °C for 8 h, indicating that no fcc-fct phase transition has occurred in the 2 nm FePt particles.

From Fig. 7, size-dependent ordering behavior of FePt nanoparticles can be found. At each annealing condition, S increases as the particle diameter increases. When the annealing condition is 700 °C for 4 h, S of 4, 6, 8, and 15 nm FePt particles is 0.85, 0.92, 0.98, and 0.94, respectively. This indicates that chemical ordering is more complete for FePt nanoparticles with larger size when the particles are annealed in the same condition. The ordering parameter of 8 nm FePt particles is very close to unity, indicating a nearly complete chemical ordering (which also explains the highest coercivity achieved with the 8 nm particles). The relatively low ordering parameter of 15 nm may be attributed to the polycrystalline morphology. As we saw from Fig. 4, the coercivity follows the same trend as the ordering parameter. Higher coercivity corresponds to higher ordering parameter.

Further studies have found that Curie temperatures of the fct nanoparticles are also strongly size-dependant. The results will be reported elsewhere.

IV. CONCLUSION

Salt-matrix annealing, an easy-handling method has been developed to produce monodisperse fct FePt nanoparticles. Important parameters of this new method, including the salt-to-

particles ratio have been systematically investigated. Monodisperse ferromagnetic FePt nanoparticles with controllable size and shape have been obtained which marks the availability of the smallest ferromagnetic nanoparticles at room temperature with particle size down to 4 nm. This method brings us good opportunity to study size effect of the ferromagnetic nanoparticles on the phase transition, chemical ordering and magnetic ordering. It has been found that FePt particles with diameter of 2 nm cannot be transferred to the ordered fct structure. Under the same annealing conditions, the degree of chemical ordering increases with the particle size. The 8 nm fct particles with faceted shape give the highest coercivity. Magnetic hardening is decreased in the 15 nm fct particles possibly related to their polycrystalline feature.

The processing method is simple and economic which can be used for producing in large quantity the ferromagnetic nanoparticles free of nonmagnetic impurities. These smallest permanent magnets produced with this method will find advanced applications as building blocks of new generation of magnetic materials and devices such as high density recording media and high energy product permanent magnet films or bulks. They may be also used directly in biomedical applications.

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