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FEPT NANOPARTICLES: SYNTHESIS, CHARACTERIZATION AND MAGNETIC PROPERTIES**НАНОЧАСТИЦЫ FEPT: ПОЛУЧЕНИЕ, ХАРАКТЕРИСТИКА
И МАГНИТНЫЕ СВОЙСТВА****FEPT НАНОБӨЛҮКЧӨЛӨРҮ: ДАЯРДОО, МҮНӨЗДӨМӨ БЕРҮҮ ЖАНА МАГНИТТИК
КАСИЕТТЕРИ**

Abstract. This work presents the synthesis of anisotropic, hard magnetic FePt nanoparticles using pulsed plasma in ethanol. As-synthesized disordered A1-FePt nanoparticles transformed into ordered L10-FePt after annealing at 400°C in vacuum furnace. Structure, morphology, size and magnetic properties were studied using XRD, HR-TEM and VS-Magnetometers.

Key words: FePt, nanoparticles, pulsed plasma in liquid, ferromagnetism

Аннотация. В данной работе представлен синтез анизотропных магнито-жестких наночастиц FePt из импульсной плазмы в этаноле. Разупорядоченные наночастицы FePt типа A1 после синтеза трансформировались в упорядоченные L10-FePt, после отжига при 400 °C в вакуумной печи. Структура, морфология, размер и магнитные свойства исследовались с помощью рентгеновской дифракции, просвечивающего электронного микроскопа высокого разрешения и магнитометра с вертикальным сдвигом.

Ключевые слова: FePt, наночастицы, импульсная плазма в жидкости, ферромагнетизм

Аннотация. Бул иш этанолдо импульстуу плазманы колдонуу менен анизотроптук, катуу магниттик FePt нанобөлүкчөлөрүнүн синтезин сунуштайт. Синтезделген ирээтсиз A1 тибиндеги FePt нанобөлүкчөлөрү вакуумдук меште 400°C температурада күйдүрүүдөн кийин ирээттүү L10-FePtге айланган. Түзүлүшү, морфологиясы, өлчөмү жана магниттик касиеттери XRD, HR-TEM жана VS-магнитометрлердин жардамы менен изилденген.

Негизги сөздөр: FePt, нанобөлүкчөлөр, суюктуктагы импульстук плазма, ферромагнетизм

INTRODUCTION

FePt nanoparticles are notable for their applications in high-density magnetic storage, catalysis, and biomedical fields. FePt nanoparticles can be two types: as synthesized -A1 type with disordered and annealed -L10 type with ordered crystal structures. Thermal annealing after the synthesis induced the Fe and Pt atoms to rearrange into the long-range chemically ordered FCT structure. Sun et al. reported that annealing at 600°C results in an increase of the average particle size and a broadening of the size distribution [1]. Weller et al have calculated that FePt as a recording medium could be thermally stable, even for grain sizes as small as 3 nm [2]. It was also proposed that the coercivity of these ferromagnetic assemblies is tunable by controlling annealing temperature and time, as well as the Fe: Pt ratio and particle size. The FePt nanoparticles shape changes dependence on sintering temperature was reported by Stappert et al. on gas-phase preparation [3]. L1₀-FePt phase exhibits high coercivity (resistance to demagnetization), high magnetocrystalline anisotropy, thermal stability

at nanoscale makes FePt nanoparticles ideal for ultra-high-density data storage (e.g., over 1 Tb/in²).

Here, we present L10 type FePt nanoparticles synthesized from FePt alloy electrodes using pulsed plasma in liquid and thermal treatment. Synthesized A1 and L10 type FePt nanoparticles were analyzed using XRD, TEM and VSM for structural, morphological and magnetic characterization.

EXPERIMENTAL

The schematic of experimental setup for pulsed plasma in liquid method is presented in many of our previous works [4-5]. For this work, FePt rods with diameter of 6 mm and purity of over 99.9% (Tanaka Kikinzoku Kogyo., Ltd. Japan) and ethanol of 99.5% purity (Kanto Chemical Co., Ltd), were used as starting materials. Experimental conditions were as following: power source (200 V, 6 A); the gap between two electrodes is about 1 mm, and electrical current duration between pulsed plasma discharges is equal to 10 microseconds (μs). After 30 minutes experiment, FePt samples were cooled and separated from the liquid

using strong magnets, then dried at 110°C for 2h. Further, the powder-like FePt nano-samples were divided into two: the first half we indexed as "as synthesized" while another half has been thermally treated at 400°C for 1 hour in an Ar gas atmosphere, and indexed as "annealed".

Both "as synthesized" and "annealed" samples were characterized by X-ray diffraction, high-resolution transmission electron microscopy and vibrating sample magnetometer.

RESULTS AND DISCUSSION

Figure 1 shows X-ray diffraction patterns of

FePt nanoparticles synthesized by PPL, before (A1) and after annealing (L10). Broadening of the peaks indicated that the particles were in nanometer scale. As-synthesized nanoparticles possess a chemically disordered FCC crystal structure (PDF#29-0718) showing the broad peaks at 41.05, 47.12, 70.35 and 83.39° 2 θ , which are indexed as (111), (200), (220), and (311) peaks respectively. After annealing at 400°C extra peaks appeared at 23.96 (001) and 32.8° (110). These peaks provide evidence of a chemical ordering phase (L10) transition, matching with FCT FePt (PDF#43-1359).

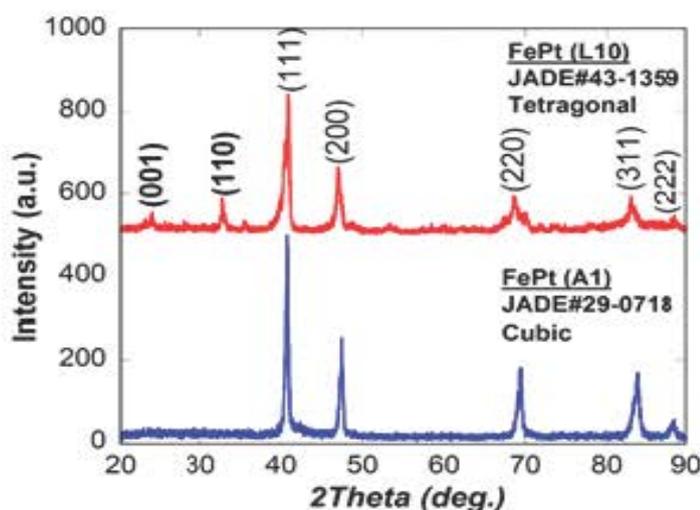


Figure 1. XRD pattern of Fe-Pt nanoparticles synthesized by pulsed plasma in ethanol

Figure 2 presents HR-TEM results of the FePt nanoparticles. As-synthesized FCC- FePt nanoparticles that have spherical shapes and are well isolated (a); a magnified view of HR-TEM image shows that there are larger particles were detected (b); inset shows the d-spacing 0.1914 nm which is well agreement with XRD d-spacing 0.1908 nm of (200) indexed, the second most intense peak at 47.58 2 θ . The as-deposited particles had the chemically disordered FCC structure and could be transformed to FCT only after annealing, which resulted in particle

aggregation. This fact was noticed also in our samples. Aggregated FCT- FePt nanoparticles can be seen at figure 2c), and d) is an HR-TEM image of single FePt particle; inset shows interplanar d- spacing, estimated as $d=0.2187$ nm which is correspond to (111) planes of FCT FePt (d-spacing 0.2197 nm). In high temperatures, nanoparticles have high random mobility, so the probability of their aggregation increases. In addition, after transition to the L10 phase, magnetic interactions also appear in FePt and these elements cause nanoparticles to coalescence.

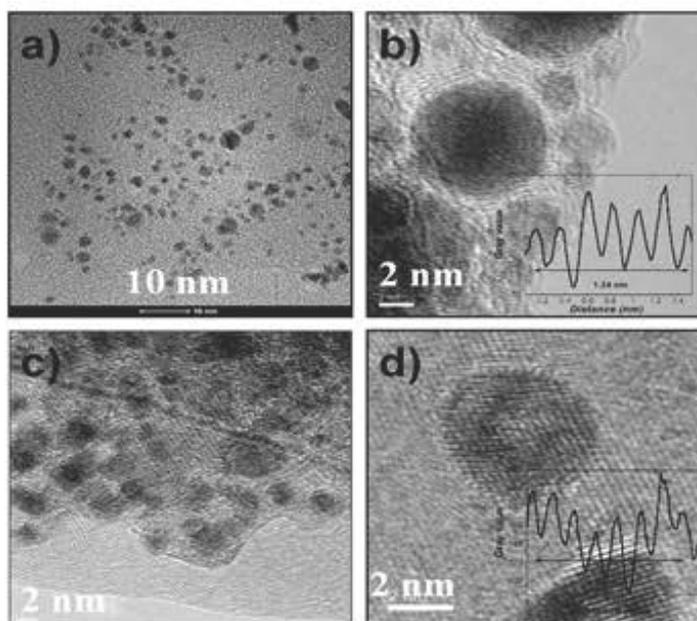


Figure 2. HR-TEM images of Fe-Pt nanoparticles by pulsed plasma in ethanol: a,b – as-synthesized Fe-Pt nanoparticles; c, d – annealed Fe-Pt nanoparticles

Magnetic properties of FePt are composition dependent, and are affected by Fe-Pt interactions within the particles. Also, the structure variations have dramatic effects on the magnetic properties of the alloys. For example, the Fe₃Pt material is paramagnetic, the Pt₃Fe is antiferromagnetic, while the L1₀ structured FePt has a large uniaxial magneto-crystalline anisotropy ($K_u = 7 \times 10^6$ J/m³) and shows strong ferromagnetic properties. Magnetic susceptibilities of FePt samples were measured with a VSM as shown in Figure 3. Blue line is hysteresis curve of as synthesized

FePt with coercivity of $H_c = 750$ Oe. Annealing converts the particles to the high-anisotropy FCT phase and transform them into room temperature nanoscale ferromagnets. After annealing at 400°C, the coercivity of FePt nanoparticles increased up to $H_c > 1100$ Oe as shown in figure (red line). This value is tunable by controlling annealing temperature and time, as well as the Fe:Pt ratio and particle size [6]. In our case, the FePt samples are Pt rich as it was estimated by EDX and XRD, which has weaker ferromagnetic property than those that Fe rich.

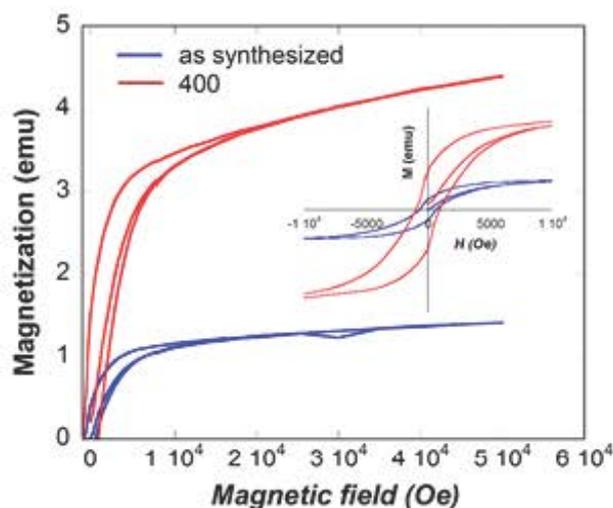


Figure 3. Magnetization curves of Fe-Pt nanoparticles by pulsed plasma in ethanol

CONCLUSIONS

The present synthesis provides a simple procedure for the preparation of A1 type FePt nanoparticles that are transferred to L10 type after annealing at 400°C. X-ray diffraction analysis revealed a good crystallinity of FePt samples and the phase transformation of cubic FCC- FePt to a tetragonal FCT order phase FePt. HR-TEM indicated the spherical shape of FCC nanoparticles with an average size of 3 nm, and semi-aggregated L10 FePt nanoparticles with a mean diameter of 6 nm. Soft magnetic behavior

of FCC- FePt nanoparticles, with coercivity of $H_c=750$ Oe and hard magnetic behavior of FCT- FePt with coercivity of over $H_c > 1100$ Oe were revealed by VS-magnetometer.

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