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Temperature dependence of hard magnetic properties of FePd nanoparticles prepared by sonoelectrochemistry

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Abstract: Hard magnetic properties of magnetic nanoparticles FePd were investigated in dependence of temperature. Magnetic nanoparticles FePd were prepared from palladium acetate and iron acetate by sonoelectrochemistry, an useful technique to make metallic nanoparticles using ultrasound. Upon the annealing at 450°C to 650°C samples have ordered $L1_0$ structure and show hard magnetic properties with high coercivity up to 2.1 kOe at room temperature and increases to 2.43 kOe with decreasing temperature down to 2 K.

1. Introduction

In magnetic recording applications, the higher density requires smaller the magnetic nanoparticles. However, it is limited by the critical grain sizes due to thermal fluctuation. FePd alloy nanoparticles with ordered structure of the type $L1_0$ have large uniaxial magnetocrystalline anisotropy of $K_u \sim 1.8 \times 10^7$ erg cm^{-3}, which can reduce the critical grain size. This chemically stable phase of FePd nanoparticles can be achieved by annealing at high temperature, making FePd as one of the potential materials applicable for the ultrahigh density magnetic storage media [1-11]. In not large number of previous studies, FePd were prepared by various methods, however the ordered $L1_0$ phase transition varies with preparing methods [4-11]. In this work, we prepared FePd nanoparticles by sonoelectrochemical method, which was developed to make nanoparticles [12] and successfully used in preparation of FePt nanoparticles [13]. This method combined the advantages of sonochemistry and electrodeposition. Upon the annealing at 450-600°C they have $L1_0$ order phase. Then, their hard magnetic properties were investigated in dependence of temperatures.

2. Experimental

The synthesis of FePd nanoparticles was conducted by sonochemical reaction using a Sonic VCX 750 ultrasound emitter within 90 minutes and described elsewhere [12]. The mixture

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of palladium (II) acetate [Pd($C_2H_3O_2$)$_2$] and iron (II) acetate [Fe($C_2H_3O_2$)$_2$] with distilled water were prepared in a 150 ml flask and was ultrasonicated with power of 375 W, frequency of 20 kHz. The FePd nanoparticles were collected from the solution by using a centrifuge with alcohol at 9000 rpm for 30 minutes and then dried at 70^{o}C-75^{o}C. Collected powders then were annealed at various temperatures from 450^{o}C to 600^{o}C under continuous flow of (N_2 + Ar) gas at heating rate of 5^{o}C/min for 1 h.

The annealed FePd samples at various temperatures were studied by X-ray analysis in Solid State Physics Department, Eotvos Lorand University, Budapest, Hungary. Magnetic properties of samples were studied by using a Vibrating Sample Magnetometer (VSM) module in Physical Property Measurement System (PPMS, Quantum Design) Evercool II in Nano and Energy Center, Vietnam National University, Hanoi, Vietnam.

3. Results and discussion

Fig. 1 shows the X-ray patterns of annealed FePd alloy nanoparticles at 550°C for 2 hours. Samples show the tetragonal order phase of FePd alloy (PDF 02-1440). The diffraction peaks are shifted to higher position with increasing annealing temperature (data not shown). These peaks are fundamental and superlattice reflections of the $L1_0$ ordered phase of FePd. In this tetragonal superlattice structure, Fe atoms can substitute Pd atoms if they have larger amount than Pd. From the half-width of the diffration peaks, the particle size is calculated to 48 ± 5 nm. The lattice parameters of the ordered phase is estimated to a = 3.868 Å and c = 3.690 Å for the sample annealed at 550°C. From these values and the ion radius of Fe and Pd, the average ratio of Fe:Pd can be estimated as 1.47:1. The degree of the order S can be estimated by the area ratio of the peaks (200) and (002) [14]. It increases with increasing annealing temperature and reaches maximum of 0.6 at 550°C then decreases when annealing temperature increases to 600°C. The low value of maximum S indicates the chemical composition as well as the degree of the order may change from particle to particle.

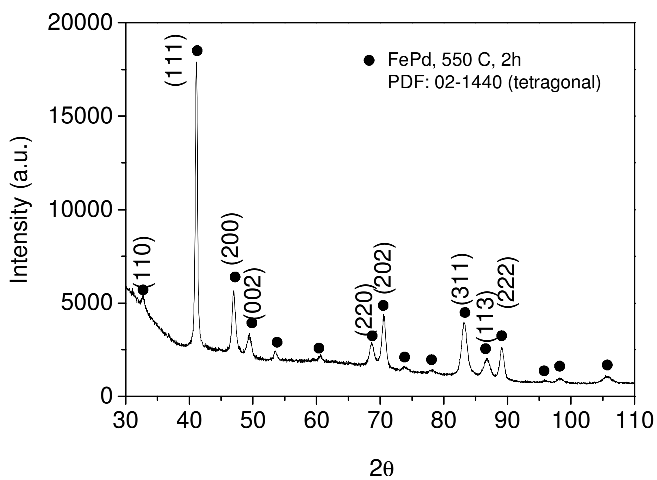

Figure 1. X-ray patterns of FePd nanoparticles prepared by sonoelectrochemistry annealed at 550°C for 2 hours.

Magnetic properties of the annealed samples were investigated in dependence of annealing temperature. At room temperature, annealed samples show hard ferromagnetic properties as shown in hysteresis curves in Fig. 2. As can be seen in this figure, sample annealed at 450°C have smallest coercivity H_C. The coercivity increases when increasing annealing temperature and has maximum value of 2.1 kOe at annealing temperature of 550°C. The coercivity then decreases when annealing temperature increases to 600°C. At magnetic field of 1.35 T, magnetization is almost saturated and continuously decreases when increasing annealing temperature. From these results, it can be recognized that sample annealed at 550°C has largest $(BH)_{max}$ as well as largest coercivity H_C. The hard magnetic properties of annealed sample were then studied in dependence of reduced temperature from room temperature down to 2 K. Fig. 3 and Fig. 4 show the hysteresis curves of samples at various annealing temperature measured at 50 K and 2 K, respectively. At all the measured temperatures, the coercivity shows similar behavior as at room temperature, which have maximum value at annealing temperature of 550°C. The saturated magnetization at 1.35 T also decreases when increasing annealing temperature. Fig. 5 shows the full range temperature dependence of the coercivity H_C at various annealing temperature. The coercivity monotonically increases when decreasing temperature at all annealing temperature and have the highest value of 2.43 kOe measured at 2 K when sample annealed at 550°C. It can be clearly seen that the sample annealed at 550°C has largest coercivity at all measured temperatures. The degree of the order S of this sample also has highest value compared to that of samples annealed at different temperatures, indicating that the hard magnetic properties strongly depend on the order phase of the $L1_0$ of FePd nanoparticles.

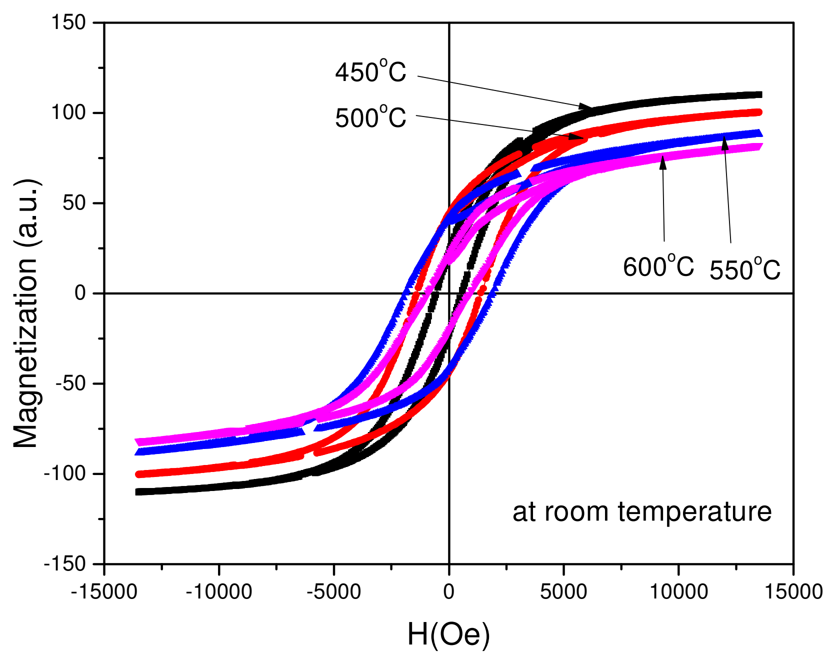

Figure 2. Hysteresis curves measured at room temperature of FePd nanoparticles at various annealing temperatures.

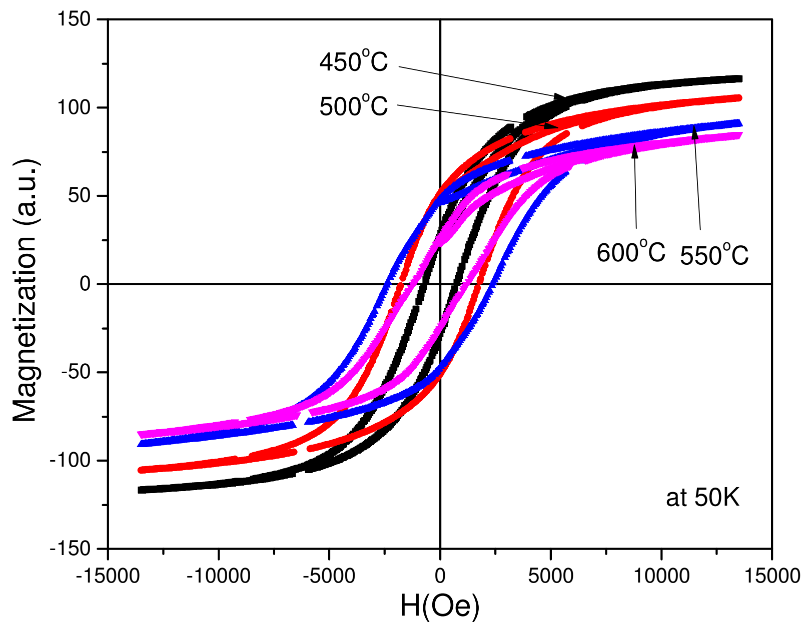

Figure 3. Hysteresis curves measured at 50 K of FePd nanoparticles at various annealing temperatures.

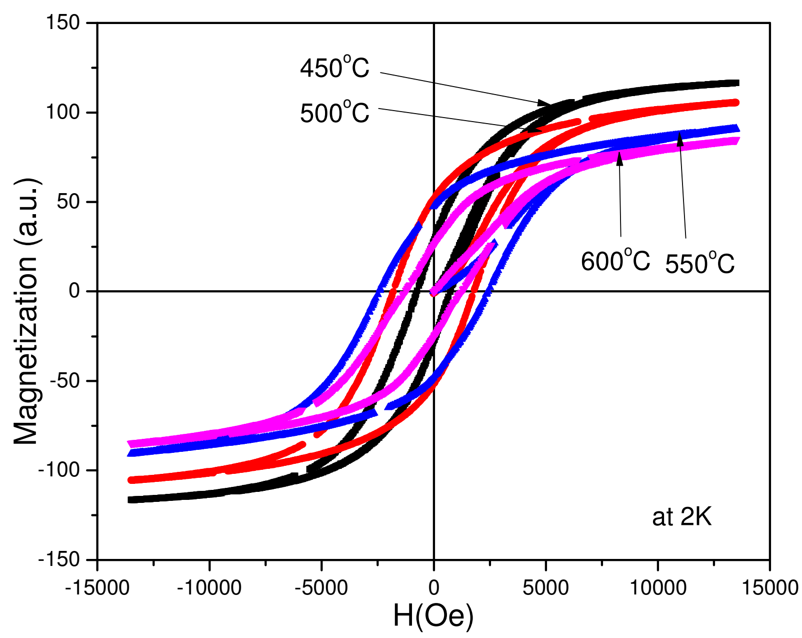

Figure 4. Hysteresis curves measured at 2 K of FePd nanoparticles at various annealing temperatures.

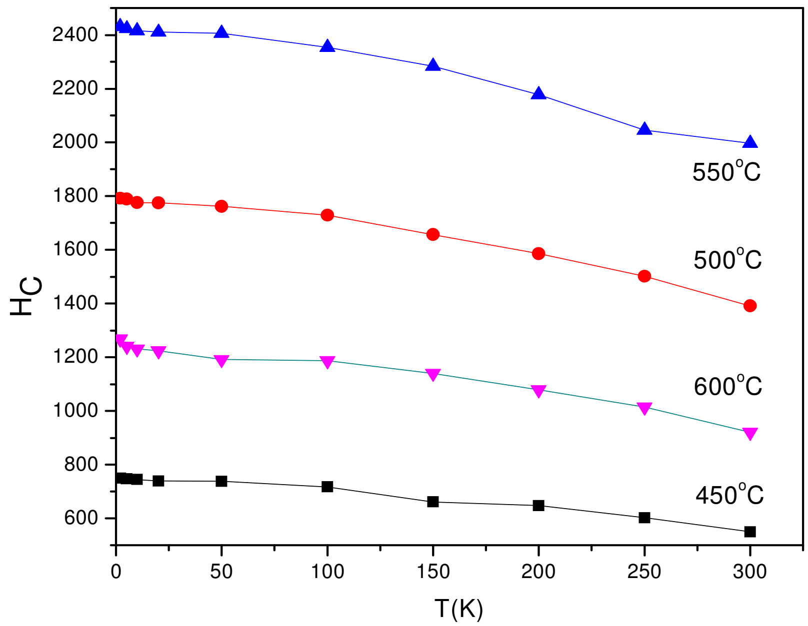

Figure 5. The temperature dependence of the magnetic coercivity of the FePd nanoparticles annealed at various temperatures.

4. Conclusion

Temperature dependence of hard magnetic properties of FePd nanoparticles prepared by sonoelectrochemistry were systematically studied and show strong dependence on annealed temperature from 450°C to 600°C. The coercivity H_C of sample annealed at 550°C shows highest value of 2.1 kOe at room temperature. The coercivity increases when decreasing temperature down to 2 K for all sample annealed at temperature from 450°C to 600°C. The chemical order degree of sample annealed at 550°C also shows highest value, indicating that the hard magnetic properties strongly depend on the formation of the $L1_0$ of FePd nanoparticles.

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