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CoPt/Ag nanocomposites for high density recording media

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Nanocomposite CoPt/Ag films have been successfully fabricated and their microstructural and magnetic properties have been investigated for potential applications in magnetic recording media. This was done by first making sputtered CoPt/Ag multilayers with the face-centered cubic (fcc) structure which is magnetically soft and then annealing the multilayers to transform the fcc phase to the highly anisotropic face-centered tetragonal phase (fct) which is magnetically hard. The final nanocomposite structure consists of CoPt nanoparticles with the hard fct phase embedded in a fcc Ag matrix. Large values of coercivity in the range of 1–17 kOe were achieved with grain sizes in the range of 7–100 nm, respectively. © *1998 American Institute of Physics.* [S0003-6951(98)01549-6]

Over the last few decades dramatic improvements have been made in magnetic information storage systems. Areal densities have increased from 1 Gbit/in.^{2} in 1990 to 5 Gbits/in.^{2} in 1998, doubling every 18 months and are expected to reach 40 Gbits/in.^{2} by the year 2004. The drive for higher magnetic recording density imposes the need for grain sizes below 10 nm. With such small grains, high magnetocrystalline anisotropy is needed[1,2] to avoid thermal fluctuations and demagnetizing fields that tend to destabilize the magnetization of the recorded bits.[3] Furthermore as the size of the particles approaches the superparamagnetic limit, which for existing materials is below 10 nm, the particle magnetization becomes thermally unstable. Recent studies on future high density recording media have been focused on rare earth intermetallic compounds which have high anisotropy and can be prepared with grains in the nanoscale size with H_c higher than 2 kOe.[4,5] An additional important requirement of such future systems is the isolation of magnetic particles which can reduce interparticle exchange interactions and lead to systems with smaller media noise.

We have recently examined the potential of granular CoPt/Ag films for high density media consisting of highly anisotropic CoPt particles embedded in an Ag matrix. In the past CoPt and FePt bulk alloys have been of interest for permanent magnets due to their high anisotropy and coercivity.[6–8] More recently they have been studied for longitudinal and magnetooptical recording media.[9,10] Alloys with equiatomic composition undergo a phase transformation from a disordered face-centered cubic (fcc) structure at high temperatures to an ordered face-centered tetragonal (fct) at temperatures below 835 °C for CoPt.[11] The fct phase is highly anisotropic with $K \sim 5 \times 10^7$ erg/cm^3, $M_s \sim 800$ emu/cm^3, and the easy axis of magnetization along the c axis.[12,13] Most of the studies on CoPt films have been made on samples with high Co concentration.[9,14] The very few studies on equiatomic compositions have been made on uniform single films which showed large coercivities when they had the fct structure.[15–18] The fct FePt phase with $H_c > 20$ kOe has also been prepared in Fe/Pt multilayers after rapid annealing.[19] Very recently nanogranular $Co_{50}Pt_{15}C_{35}$ thin films have been made consisting of Co–Pt particles with the hcp structure in a C matrix having coercivity 1500 Oe.[20] In this study we have successfully prepared granular CoPt/Ag films consisting of fct CoPt phase nanoparticles embedded in an Ag matrix by making first CoPt/Ag multilayers and then annealing the multilayers to obtain the nanocomposite structure.

The films were made with magnetron sputtering deposition using cosputtering from two metallic targets of $Co_{53}Pt_{47}$ and 99.999% Ag with a diameter of 5 cm. A radio frequency (RF) magnetron gun operating at 50 W with deposition rate of 0.3 nm/s was used for $Co_{53}Pt_{47}$ and a direct current (dc) magnetron gun operating at 5 W with deposition rate 0.2 nm/s was used for Ag. The base pressure of the chamber was 3×10^{-7} Torr and high purity Ar (99.999%) was used for deposition at ambient temperature, at a pressure of 3 mTorr with a flow rate 8 sccm. The substrates used were p-type Si(100) 600 μm thick, covered with 750 Å layer of SiN_x to reduce the surface roughness. X-ray diffraction (XRD) spectra were collected with a SIEMENS D500 powder diffractometer using Cu $K\alpha$ radiation. Magnetic hysteresis loops were measured with a Quantum Design MPMSR2 superconducting quantum interference device (SQUID) magnetometer. The microstructure was examined with a Philips CM20 and a Jeol JEM 2000 FX transmission electron microscopy (TEM).

Multilayers of the composition $[Co_{53}Pt_{47}(t_1\ \text{nm})/\text{Ag}\ (t_2\ \text{nm})]_{\times 30}$ with $t_1 = 0.5,1,2$ and $t_2 = 0.3$, 0.5, 1, and 2 were fabricated, using a Ag buffer layer (10 nm) to reduce the influence of the substrate and a Ag capping (10 nm) to protect the structure from oxidation. The chemical composition of CoPt as-deposited films was checked by energy dispersive x-ray analysis and was found to be near $Co_{53}Pt_{47}$. The as-deposited multilayers with larger layer thickness have been found to have the disordered fcc phase with a texture along the ⟨111⟩ direction. Figure 1(a) shows the x-ray diffraction pattern of a $[Co_{53}Pt_{47}(2\ \text{nm})/\text{Ag}\ (2\ \text{nm})]_{30}$ film where the ⟨111⟩ average Bragg peak can be seen, surrounded by satel-

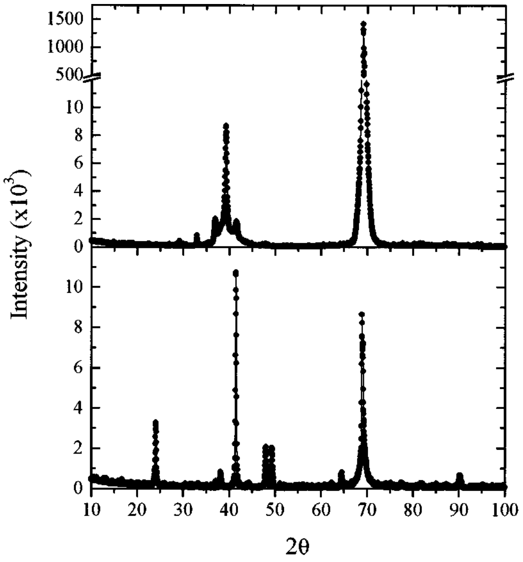

FIG. 1. XRD patterns of a $Co_{53}Pt_{47}$ (2 nm)/Ag (2 nm) film of (a) as-deposited multilayer showing a preferred (111) texture and (b) sample aged at $T = 600\,°C$ where the ordered fct phase appears.

lites indicating the presence of a multilayer structure. In films with smaller layer thickness no satellites were observed; this is attributed to interlayer roughness. The fcc phase was found to transform to fct upon annealing at temperatures higher than 500 °C. Figure 1(b) shows the presence of superlattice peaks in a sample annealed at 600 °C for 120 min, indicating the presence of fct structure.

All the as-deposited films are magnetically soft with $H_c < 100$ Oe. Upon annealing and subsequent transformation to the fct structure the samples become magnetically hard (Fig. 2). In the initial stages of ordering the coercivity is a few hundred Oe but it increases drastically with subsequent annealing reaching values beyond 15 kOe. The maximum value was found to depend on the layer thickness with the highest value (17 kOe) obtained in thicker layer samples ($t_1 = 2$ nm/$t_2 = 2$ nm) (Fig. 2). Also the optimum annealing times are much shorter at higher annealing temperatures as expected. Figure 3 shows typical hysteresis loops obtained on a sample annealed at different temperatures. Prolonged annealing leads to the development of a shoulder in the demagnetization curve. Values of reduced remanence, $m_r = M_r/M_s$ found from the hysteresis loop data (with values of M_s determined from the law of approach to saturation) are in the range 0.62–0.85 with the larger values obtained in underaged samples.

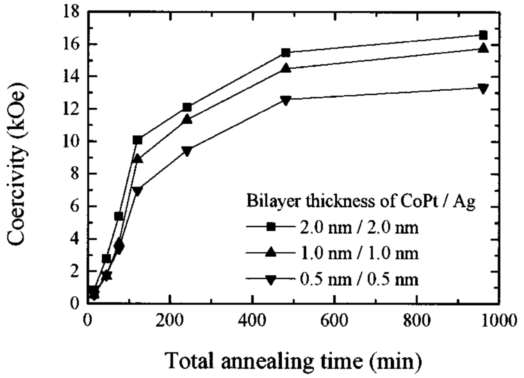

FIG. 2. Development of coercivity with annealing time at $T = 550\,°C$ in different CoPt/Ag samples.

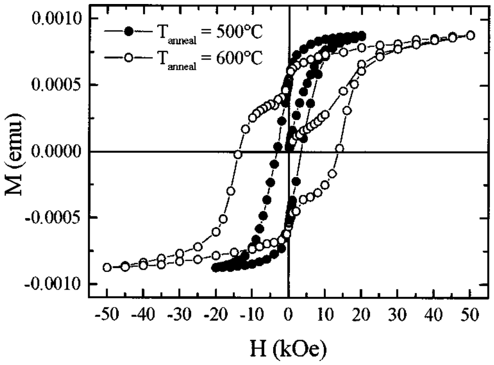

FIG. 3. Typical hysteresis loops for $Co_{53}Pt_{47}$ (2 nm)/Ag (2 nm) films annealed for 2 h at different temperatures.

Another important parameter that is needed for noise reduction in such systems is the magnetic isolation of the particles. In Fig. 4, δM plots[21] for $[Co_{53}Pt_{47}(2\,nm)/Ag(2\,nm)]_{30}$ annealed at 500 °C for 2 h and an fct $Co_{53}Pt_{47}$ single film annealed at 700 °C for 1/2 h prepared under the same conditions are compared. The $Co_{53}Pt_{47}$ single films exhibit strong exchange interactions, while in the nanostructured material only a small amount of dipolar interactions is present, which is a characteristic of isolated single domain particles.

The evolution of microstructure with the ordering transformation to fct (induced by annealing) has been monitored by TEM in order to be correlated with the sample magnetic properties. The results are shown in Figs. 5(a)–5(c), for a $t_1 = 0.5$ nm/$t_2 = 0.3$ nm sample annealed at 600 °C for 5, 10, and 20 min, respectively. Selected area diffraction shows that the sample consists of fct CoPt and fcc Ag. The CoPt is in the form of small grains randomly distributed in the Ag matrix [Fig. 5(a)]. Samples annealed for 5 min having $H_c = 0.8$ kOe consist of very fine grains in the range of 7–20 nm [Fig. 5(a)]. The size of grains and therefore H_c are increased with annealing to 20–50 nm at 10 min ($H_c = 3.8$ kOe) and 20–70 nm at 30 min ($H_c = 11.0$ kOe). The size of CoPt grains is significantly increased with long annealing times reaching values in the range of 20–100 nm [Fig. 5(d)].

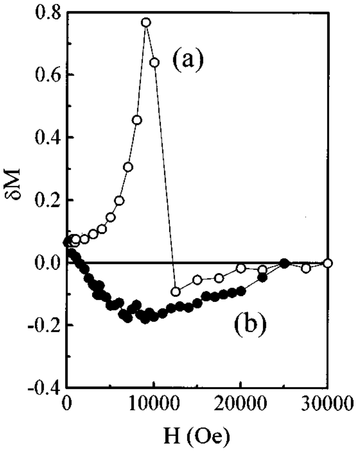

FIG. 4. δM plots for (a) fct CoPt single film (light symbols) annealed at 700 °C for 1/2 h and (b) nanostructure (dark symbols) after annealing at 500 °C for 2 h.

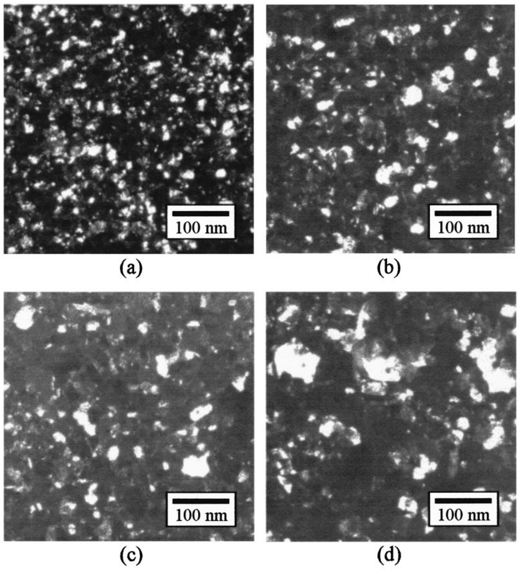

FIG. 5. Evolution of microstructure with annealing time; (a), (b), and (c) refer to a $Co_{53}Pt_{47}$ (0.5 nm)/Ag (0.3 nm) sample annealed at 600 °C for 5, 10, and 20 min, respectively; (d) refers to a $Co_{53}Pt_{47}$ (2.0 nm)/Ag (2.0 nm) sample annealed at 550 °C for 240 min.

The particles sizes determined from the TEM measurements are well below those predicted[22] for the upper limit of the CoPt single domain particle size $D_{sd} = 0.61\ \mu m$ ($D_{sd} = 1.4\gamma/M_s^2$ assuming[13] a domain wall energy density $\gamma = 28\ erg/cm^2$ and $M_s \sim 800\ emu/cm^3$). The values of the maximum coercivities obtained approach those predicted for single domain CoPt particles. For a random distribution of single domain particles with uniaxial anisotropy K, $H_c \sim K/M_s \sim 19$ kOe[22] assuming $K \sim 5\times10^7\ erg/cm^3$.[12,13] The lower values of H_c obtained in the early stages of ordering are possibly due to the smaller particle size and to the expected[22] usual decrease of H_c $(1-D^{-3/2})$ because of thermal effects. The shoulder in the demagnetization curve, observed in all samples annealed for longer times, is possibly related to a nonuniform microstructure with grain sizes below and above the single domain size with the latter showing a low H_c multidomain behavior. Another possibility is the presence of soft $CoPt_3$ but because of texturing and the overlapping of major peaks in electron diffraction patterns, TEM studies are at this point inconclusive. Optimization studies including magnetic annealing are under way to determine the sputtering and processing conditions for the ideal microstructure with the desired magnetic properties. Similar studies are also being made on FePt/Ag.

In summary, we have successfully prepared CoPt/Ag films consisting of highly anisotropic fct CoPt nanoparticles embedded in an Ag matrix. The size of the CoPt particles can be changed from 7 to 100 nm upon annealing. The coercivity of these nanoparticles can easily be tailored from values less than 1 kOe up to several kOe by varying the annealing temperature and time and the relative CoPt/Ag composition. The magnetic and microstructural properties of these systems show that they may have a great potential for application in magnetic recording media, although major improvements are needed for the development of a practical medium.

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