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Low temperature ordering of FePt films by in-situ heating deposition plus post deposition annealing

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ABSTRACT

FePt thin films with 40 nm thickness were prepared on thermally oxidized Si (001) substrates by dc magnetron sputtering at the nominal growth temperature 375 °C. The effects of annealing on microstructure and magnetic properties of FePt thin films were investigated. The as-deposited FePt thin films show soft magnetic properties. After the as-deposited FePt thin films were annealed at various temperatures and furnace cooled, it is found that the ordering temperature of $L1_0$ FePt phase could be reduced to 350 °C. For FePt thin films annealed at 350 °C, the in-plane and out-of-plane coercivities of the films increased to 510 and 543 kA/m, respectively, and the films had hard magnetic properties. A highly (001) orientation was obtained, when FePt thin films were annealed at 600 °C. And the hysteresis loops of FePt thin films annealed at 600 °C show out-of-plane magnetic anisotropy.

1. Introduction

Stoichiometric FePt intermetallic alloy with $L1_0$ ordered structure is considered as one of the leading candidate materials for the next generation of the ultrahigh-density magnetic recording media, because of its large magnetocrystalline anisotropy constant (7×10^6 J/m^3) [1], small size (about 3 nm) permitting thermal stability [2], high coercivity [3] and excellent corrosion resistance [4]. However, FePt films deposited at room temperature adopt a disordered face-centered cubic (fcc) structure with soft magnetic properties. Thus, to get hard magnetic $L1_0$ ordered film with a face-centered tetragonal (fct) structure, it is necessary to deposit FePt films on a heated substrate or anneal the disordered films after deposition. In general, an annealing temperature T_a 500 °C is required to transform the disordered fcc structure to the ordered fct structure [5,6]. The high processing temperature results in large grains and increasing media noise, which are the main disadvantages for practical recording applications of the film. Therefore, a technique to lower the ordering temperature of FePt thin films is an urgent problem. Recently, many methods have been adopted to reduce the processing temperature, such as the introduction of underlayer [7–10] or top layer [11], the addition of third elements [12,13], multilayering [14–16], ion irradiation [17,18], *in situ* annealing [19–21], alternate monatomic layer deposition [22], and dynamic stress-induced ordering [23]. But so far, it is still very difficult to reduce the ordering temperature of pure FePt films with the thickness less than 50 nm deposited onto practical substrates, such as thermally oxidized Si or glass. In this paper, we offer a more effective method to lower the ordering temperature of pure FePt films with the thickness 40 nm deposited onto thermally oxidized Si (001) substrates. FePt films were deposited onto preheated thermally oxidized Si (001) substrates at nominal growth temperature 375 °C. After suitable post deposition annealing, the ordering temperature of FePt films could be reduced to 350 °C and in-plane and out-of-plane coercivities of the films were above 500 kA/m.

2. Experiments

FePt (40 nm) thin films were deposited onto thermally oxidized Si (001) substrates by dc magnetron sputtering in an ATC 1800-F magnetron sputtering system. The purities of Fe and Pt targets are 99.99%. The base pressure of the chamber was about 5.3×10^{-5} Pa and a flow of high purity argon at 0.6 Pa was used during sputtering. The substrates were rotated at a speed of 20 rpm during film deposition to obtain uniform film thickness and composition. The typical growth rate of FePt was 0.08 nm/s. The nominal growth temperature measured by thermocouple was 375 °C. Because there is a distance between the back of substrate holder and thermocouple, the actual growth temperature of the substrate surface is lower the nominal

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growth temperature measured by thermocouple. Before sputtering FePt thin films, the substrates were cleaned ultrasonically in acetone, alcohol and de-ionized water consecutively, and then baked in a vacuum chamber at 375 °C for 30 min in order to clean the substrate surface.

The compositions of FePt films were determined to be $Fe_{49.6}Pt_{50.4}$ by X-ray fluorescence. The films were annealed at 300–600 °C in a vacuum furnace with a pressure about 5.1×10^{-4} Pa. The heating rate during annealing was about 20 °C/min, and the annealing time was fixed at 1 h, after which the furnace was cooled in vacuum. The crystal structures of the as-deposited and annealed films were characterized by θ–2θ X-ray diffraction (XRD) on a Rigaku D/max-2500 diffractometer with Cu Kα radiation using a current of 300 mA and voltage of 40 kV. The magnetic properties of the films were measured at room temperature using a vibrating sample magnetometer (VSM) with an applied field up to 1592 kA/m and superconducting quantum interference device (SQUID) in fields up to 4775 kA/m. The magnetic signal from the substrate was subtracted from the multilayer film magnetic signal by fitting a straight line to the high-field region and subtracting the linear portion from the measured signal.

3. Results and discussion

Fig. 1 shows the θ–2θ scan XRD patterns of FePt films which are as-deposited and annealed at various temperatures. In the case of the as-deposited FePt film, diffraction peaks from the (111) and (200) planes of disordered fcc FePt are observed at around 41° and 47° for 2θ, respectively. Even after post annealing up to 300 °C, no significant change can be seen. These results reveal that the fcc FePt phase is dominant when FePt films are as-deposited and post annealed at 300 °C. In the film annealed at 350 °C, weak (001), (200) and (002) superlattice diffraction peaks of the ordered $L1_0$ phase can be observed, indicating that formation of the ordered $L1_0$ phase of the film had begun and the disorder-order transformation from fcc to the $L1_0$ structure started at $T_a = 350$ °C annealing. When the annealing temperature is increased to 400 °C, a stronger (001) peak and more splitting between the (200) and (002) peaks can be seen. The intensities of the superlattice diffraction peaks further increase with increasing annealing temperature. When FePt film was annealed at 600 °C, the (001) superlattice diffraction peak is the strongest peak among all the (001), (111), and (002) diffraction peaks of fct FePt phase and the films show a highly (001) orientation. Thus more $L1_0$ phase with a higher degree of ordering and highly (001) orientation was formed in the film after annealing at higher temperature.

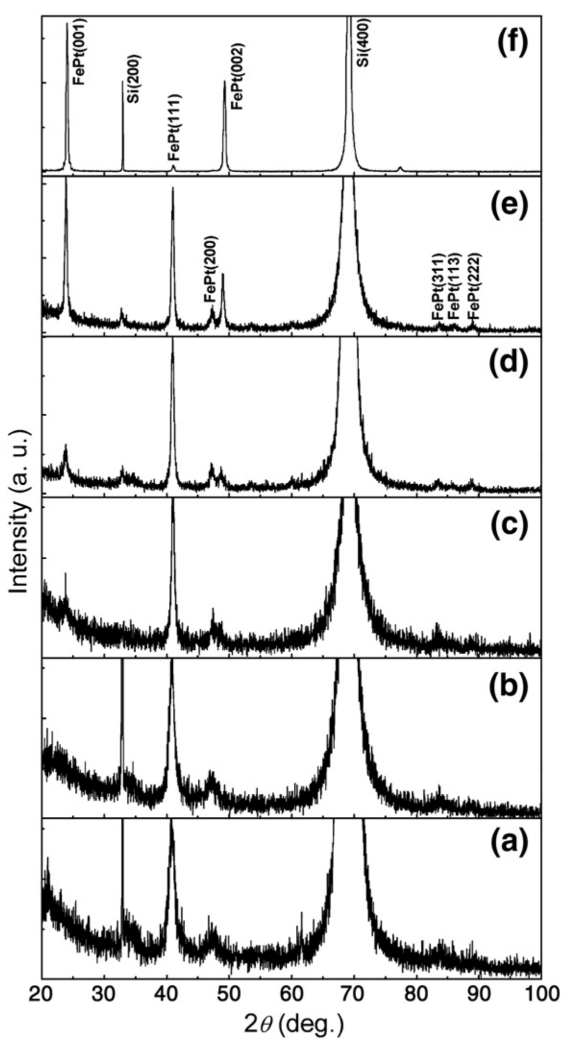

Fig. 1. XRD θ–2θ scans of FePt films: (a) as-deposited and annealed at (b) 300 °C, (c) 350 °C, (d) 400 °C, (e) 500 °C and (f) 600 °C.

Fig. 2 plots the variations in (111) peak position and (111) half-peak breadth for FePt films annealed at various temperatures. It is obvious that there is a critical annealing temperature, about 350 °C, above which both (111) peak position and (111) half-peak breadth show very rapid increase and decrease, respectively.

The disorder-order transformation of FePt phase involves a distortion of the fcc unit cell. The a lattice constant expands approximately 2%, while the c lattice constant contracts approximately 2.5%, resulting in a c/a ratio that is less than 1. From the positions of the (001) and (111) diffaction peaks, the a and c lattice constants, respectively, were calculated for the FePt films annealed at various temperatures. Fig. 3 (a) and (b) show lattice constant and c/a ratio versus annealing temperature. From Fig. 3 (a), the values of a increase and c decrease, with increasing the annealing temperature. When FePt film was annealed at 350 °C, the c/a ratio is 0.977, which reveals that the cubic structure has transformed to the tetragonal structure. In order to evaluate the long-range ordering (LRO) degree of the fct phase for the annealed films in more detail, the order parameter S is employed. An approximate relation between S and c/a can be written as follows [25]:

$$S^2 = \frac{1-(c/a)}{1-(c/a)_{S_f}}$$

where $(c/a)_{S_f}$ is the axial ratio for the fully ordered phase, c/a is for the partially ordered phase. $S = 1$ in this equation corresponds to the fully ordered phase. In the case of FePt film annealed at 350 °C, S is 0.768, which indicates that the fcc disordered phase was mostly transferred

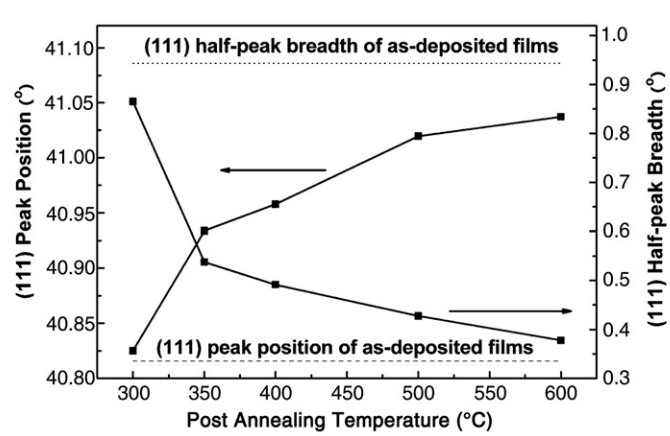

Fig. 2. The variations in (111) peak position and (111) half-peak breadth for FePt films annealed at various temperatures.