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On the growth and magnetic properties of flower-like nanostructures formed on diffusion of FePt with Si substrate

Parmanand Sharma [a,c,*], Neelam Kaushik [b], Masayoshi Esashi [b], Masahiko Nishijima [c], Akihiro Makino [a,c]

[a] Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan
[b] World Premier Initiative (WPI) Center, Advanced Institute for Materials Research (AIMR), Tohoku University, Sendai 980-8577, Japan
[c] Research and Development Center for Ultra High Efficiency Nano-Crystalline Soft Magnetic Materials, Institute for Materials Research, Tohoku University,Sendai 980-8577, Japan

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ABSTRACT

Nearly equi-atomic FePt films were deposited on a silicon substrate with a native SiO_2 layer by co-sputtering technique. Structural and magnetic properties of the films are influenced by the growth temperature. The native SiO_2 layer acts as a diffusion barrier for the FePt film on Si. In the absence of diffusion of Fe and Pt with Si substrate, a high coercivity (~8.2 kOe) $L1_0$ $Fe_{50}Pt_{50}$ phase is formed. Depending on the growth temperature and the thickness/quality (continuously or containing some pinholes) of SiO_2 layer, diffusion of FePt film with Si substrate is observed. Diffusion results in the change in film composition, and formation of various Fe- and Pt-silicide phases along with the flower-like surface morphology. Growth of flower like nano-structures are shown to be governed by the accelerated diffusion of Fe on a dimpled surface of Si substrate. The detailed structural and magnetic characterization revealed that the flower-like patterns are composed of chemically ordered antiferromagnetic $FePt_3$ phase (Q_2-type). The Neel's temperature for these flowers-like nano-structures is ~80 K and they are surrounded by a ferromagnetic matrix. Growth mechanism of flower-like patterns is identified. Site specific growth of antiferromagnetic nano-structures in a ferromagnetic matrix is also demonstrated.

1. Introduction

In addition to oxides and semiconductor nano-structures, magnetic nano-structures have also attracted much attention in recent years [1–3]. In particular FePt alloys have become the focus of intensive research because of their chemical stability and potential applications in high-density data storage and high-performance permanent magnets [4]. Various experimental reports (bulk and thin films deposited on single crystal or amorphous substrates) on Fe_xPt_{1-x} alloys have shown the formation of both chemically ordered ($L1_0$) and disordered phases (fcc-FePt) of FePt in the compositional range of x ~0.35–0.60 [5–8]. Island like $L1_0$ FePt films epitaxially grown on MgO single crystal substrates exhibit high coercivity (H_c) whereas a continuous film grown on the same substrate possesses a low H_c [8]. These observations encouraged some of the researchers to develop magnetic nano-structures (such as nanowires, nanorods and nano-cubes/crystals) of FePt [8–16].

In FePt system, besides $L1_0$ phase (FePt), formation of $L1_2$ phases ($FePt_3$ and Fe_3Pt) are also possible. The Fe_3Pt phase is soft ferromagnetic (saturation magnetization, M_s~1.8 T at room temperatures), and it is useful for the development of exchange-coupled type nano-composite magnets in a mixture with $L1_0$ FePt [17–21]. The $FePt_3$ is ferromagnetic in the chemically disordered state (Curie temperature, T_c~360–400 K), and antiferromagnetic (AF) in ordered state. The physics of chemically ordered $FePt_3$ phase is interesting because of two coexisting AF states. The stoichiometric composition, where the Fe atom at the corners of each cubic cell and Pt atom in the middle of each face is AF with Neel's temperature T_{N1}~160 K [19]. This is also known as Q_1 phase. For slightly Fe-rich alloys, a second AF phase transition occur at $T_{N2} < 100$ K. This phase is known as Q_2 and in this excess Fe atom occupy the face centers and couple ferro-magnetically with the nearest Fe atom in the (100) sheet [19]. In Fe-rich samples, two AF phases may coexist; however, the phase fraction of Q1 diminishes with a further increase in Fe content. In terms of nanostructures, only chemically disordered $FePt_3$ nano-rods have been reported [22]. However, to our knowledge, chemically ordered $FePt_3$ phase is obtained in bulk crystals, nano-particles and in thin films epitaxially grown on single crystal substrates. There is no report on the fabrication of AF $FePt_3$ based nanos-tructures such as nano-wires, nano-tubes, spirals, flowers etc.

* Corresponding author at: Tohoku University, Institute for Materials Research, 2-1-1, Katahira, Aoba-Ku, Sendai, Miyagi, 980-8577 Japan. Tel.: +81 22 215 2158.
E-mail address: sharmap@imr.tohoku.ac.jp (P. Sharma).

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For high density perpendicular magnetic recording media, very thin films (less than 50 nm) of *c*-axis/(001) oriented $L1_0$ FePt are required to grow on a Si or SiO_2 substrate. The interfacial strain between FePt and Si substrate caused by a thin layer of SiO_2 is known to be effective in promoting the growth of *c*-axis oriented growth of $L1_0$ FePt [6,23–29]. High temperature processing (to obtain ordered $L1_0$ phase), and large negative heats of mixing between Fe and Pt with Si, make FePt films vulnerable to diffuse with Si substrate. Formation of various silicide phases and the changes in the stoichiometry greatly influence the magnetic properties of $L1_0$ FePt [6,23–29]. During the growth of $L1_0$ FePt on Si substrate with a native SiO_2 layer, we have observed the formation of submicron-sized flower-like patterns. In this paper, we report on the observation, growth mechanism and magnetic properties of submicron-sized FePt-based flowers. Diffusion of FePt thin film with Si substrate plays an important role in the formation of flower-like patterns. The site specific growth of flowers is also possible. Detailed structural and magnetic characterization revealed that the flowers are surrounded by a ferromagnetic matrix and are mainly composed of chemically ordered Q_2 phase of $FePt_3$.

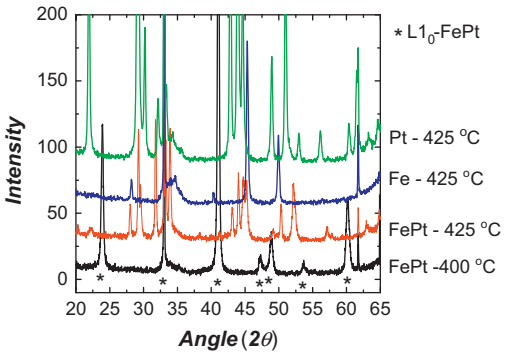

Fig. 1. X-ray diffraction (XRD) patterns of $Fe_{50}Pt_{50}$, Fe and Pt thin films deposited at different T_s=400 and 425 °C on Si substrate with a native layer of SiO_2. Formation of $L1_0$ FePt at T_s ~400 °C can be noticed. The peaks observed in XRD for FePt thin films deposited at 425 °C are different from the film deposited at 400 °C, and matches with the diffraction peaks of pure Fe and Pt deposited under similar conditions. XRD data revealed formation of various Fe- and Pt-silicides, indicating diffusion of film with silicon at T_s~425 °C.

2. Experimental details

$Fe_{50}Pt_{50}$ films (thickness ~50 nm) were deposited on silicon (100) substrates by using a dc-magnetron co-sputtering technique. High purity Fe and Pt targets were used. After initial cleaning by using acetone and alcohol, the substrates were plasma cleaned before deposition of FePt thin films. The base vacuum of sputtering chamber was $<10^{-5}$ Pa. A high purity argon gas at a pressure of ~0.2 Pa was used as a sputtering gas. Target to substrate distance was ~150 mm, and the substrate was rotated at ~10 rpm during deposition. Films were deposited at different substrate temperatures (T_s~ room temperature–450 °C). The deposition rate of FePt was ~2.8 nm/min. Macroscopic and microscopic compositional analysis of films were carried out by using an electron probe micro-analysis (EPMA) technique, and energy dispersive spectroscopy (EDS) in combination with transmission electron microscopy (TEM) at five different locations of each sample. Diffusion of FePt film with Si substrate was understood by compositional depth profiling using Auger electron spectroscopy (AES). Focused ion beam (FIB) technique (with Ga ion source) was used to understand the growth mechanism of flowers like nanostructures. The crystallographic information of the films was obtained by using a Rigaku (model: RINT – Ultima III) X-ray diffractometer (XRD). Magnetic measurements (temperature range ~5–330 K) were performed using a Quantum design superconducting magnetometer (MPMS 5S). Commercially available magnetic cantilevers, and a standard magnetic force microscopy (MFM) imaging procedure were used to image the magnetic domains at room temperature. At first MFM experiments were done on known samples.

3. Results and discussion

3.1. Growth and structural characterization

3.1.1. Effect of substrate temperature

X-ray diffraction (XRD) patterns of FePt thin films deposited at a substrate temperature (T_s) of 400 and 425 °C are shown in Fig. 1. Diffraction peaks match with $L1_0$ FePt for films deposited at T_s=400 °C, but for T_s=425 °C, many peaks were observed and none of them matches with $L1_0$ FePt. XRD result shows that the $L1_0$ FePt films are polycrystalline and magnetic easy axis of each grain is randomly oriented, i.e. some of the grains have easy axis in out of plane [(001) oriented], and some of them in inplane [(110) oriented] or in some other directions. In view of strong demagnetization effects associated with thin film geometry (strongest in out of plane and weakest or negligible in inplane), magnetic measurements were performed in inplane direction. A large inplane coercivity (H_c) of ~8.2 kOe was obtained for the films deposited at T_s~400 °C (Fig. 2). It is consistent with our XRD results, that showed formation of hard magnetic $L1_0$ FePt. A drastic decrease in H_c (~0.2 kOe) was noticed for the films deposited at T_s=425 °C (Fig. 2). This could be due to diffusion of Fe and Pt into the silicon substrate or vice-versa. To get more information about the structure, pure Fe and Pt thin films were deposited under similar conditions (T_s=425 °C). XRD patterns (Fig. 1) exhibit diffraction peaks corresponding to various Fe- and Pt-silicide phases, indicating diffusion of Fe and Pt in silicon substrate or vice-versa. From Fig. 1, it is also noticiable that the XRD peaks of FePt thin film deposited at T_s=425 °C matched with pure Fe and Pt films deposited under similar conditions.

Auger electron spectroscopy (AES) combined with Ar-ion beam sputtering was used to understand the diffusion behavior of FePt thin films on a silicon substrate. Fig. 3 shows the compositional depth profiles i.e. the relative variation of Fe, Pt, Si and O contents with Ar-ion beam sputtering time for films deposited at T_s=400 and 425 °C. It should be noted that the vertical axis i.e. intensity is not calibrated to atomic percentage. The profiles show almost the same concentration (constant intensity) of Fe and Pt up to the sputtering time of ~8.5 min for film deposited at T_s=400 °C [exhibiting $L1_0$ FePt phase (Fig. 3(a))]. Moreover, up to this sputtering time, the concentration (intensity close to the background) of Si is almost zero. Beyond this, concentration of Fe and Pt decreases very rapidly up to zero level and at the same time concentration of Si increases very rapidly and attains a saturation level. This behavior indicates negligible diffusion of Fe and Pt in silicon substrate or vice-versa for films deposited at T_s=400 °C. On the other-hand, concentration depth profiles are very different for films deposited at T_s=425 °C (Fig. 3(b)). The presence of Si along with Fe and Pt can be noticed from the very beginning. The concentration of Fe and Pt do not remain constant with film thickness. From the depth profile, it appears that Fe is enriched on the film surface and below the film-substrate interface whereas Pt enrichment is in between the film surface and film-substrate interface (Fig. 3(b)). The possible explanation for this kind of compositional profile shall be given in Section 3.1.3. This behavior suggests that diffusion of Fe on Si surface is faster as compared to Pt. The presence of silicon in the film indicates formation of various Fe- and Pt-silicide phases. Relatively fast diffusion of Fe in silicon compared to Pt at same the temperature (T_s=425 °C) is