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Nonmagnetic to magnetic nanostructures via ion irradiation

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Abstract

A Pt/C multilayer stack (15 layer-pairs) with Fe impurities was prepared on a glass substrate by the ion sputtering technique. Ion irradiation effects on this multilayer were studied following irradiation with 2 MeV Au ions at fluences from 1×10^{14} to 1×10^{15} ions/cm^2. Irradiation induced atomic displacements in such multilayers have been earlier analyzed by a combined X-ray standing wave (XSW) and X-ray reflectivity (XRR) technique with a depth resolution better than 0.2 nm [S.K. Ghose, B.N. Dev, Phys. Rev. B 63 (2001) 245409; S.K. Ghose, D.K. Goswami, B. Rout, B.N. Dev, G. Kuri, G. Materlik, Appl. Phys. Lett. 79 (2001) 467]. Using the combined XSW-XRR technique ion beam induced preferential movement of Fe from C- to Pt-layers has been detected. At the highest ion fluence Pt layers (containing Fe) break into nanoparticles apparently surrounded by C. Grazing incidence X-ray diffraction (GIXRD) measurements indicate the formation of FePt particles in the irradiated multilayer samples. Results of magnetic force microscopy (MFM) and magneto-optical Kerr effect (MOKE) measurements reveal that while the virgin sample hardly shows any magnetism, the irradiated samples show a soft ferromagnetism with an increasing coercive field with increasing ion fluence. Use of focused ion beam to fabricate ferromagnetic nanodots and their possible uses in spin electronics are discussed.

Keywords: Multilayers; Ion irradiation; Ferromagnetism

1. Introduction

In the 1950s, development of computers led to a search for soft-magnetic materials – materials that could be easily made to reverse their magnetization direction – for use in information storage. Also the focus went on thin films. The most promising candidate by mid-50s was permalloy ($Ni_{0.8}Fe_{0.2}$), which had been shown to retain its bulk soft-magnetic properties as a thin film. Given the prospect of an application with a huge financial impact, magnetic thin film research accelerated. Thin film research gradually evolved into magnetic multilayers where alternatively thin films of a magnetic material and a nonmagnetic material are deposited on a substrate. Examples of such multilayers are Co/Cu, Co/Pt multilayers. Magnetic multilayers show new effects like interlayer exchange coupling (IEC), giant magnetoresistance (GMR), tunneling magnetoresistance (TMR) etc., which have given scientists a new capability to construct remarkable devices. GMR effect has turned out to be very useful for sensors, particularly those for read heads in computer hard-disk drives.

New frontiers involving fields like magnetoelectronics, spin electronics (or simply spintronics) are emerging where layered magnetic structures are being used.

The increase of aerial storage density in magnetic hard-disk drives may be limited in the future by the thermal instability of small magnetic domains – a behaviour known as superparamagnetism. The use of nanostructured magnetic storage media instead of continuous recording films

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is a widely discussed measure to achieve thermally stable bit cells for storage densities even beyond 1 Tbit/in^2[1]. Nanopatterned media can be fabricated from continuous magnetic films by means of focused ion beam techniques. Ion irradiation effects on Co/Pt multilayers are extensively studied for this purpose [2,3].

Nonmagnetic multilayers, (such as Pt/C multilayers) where neither material in the multilayer is magnetic, have also many important uses. They are used in X-ray telescope, as filters and monochromators for synchrotron radiation etc. In general, ion irradiation causes atomic displacements in a multilayer leading to change of properties [4,5]. In magnetic multilayers it even destroys magnetism.

In this paper we present a novel phenomenon of transforming a nonmagnetic multilayer to a soft ferromagnetic system by ion irradiation. As focused ion beam of a few nanometer spot size is available it is possible to make nanometer-sized ferromagnetic spots in an otherwise nonmagnetic material with many potential applications. Possible connections of the observed phenomenon to novel devices, such as spin-valve-single-electron-transistor, will be discussed.

2. Experimental

Pt/C periodic multilayers were fabricated on float glass substrates by ion beam sputtering, at a low Argon pressure of 0.1 mbar. Fe impurity was introduced into the multilayer during growth. Post-growth measurement has shown the Fe concentration to be 14 at %. A large multilayer sample $(30 \times 70)\,\mathrm{mm}^2$ with the following specifications was used in the present study. Specifications: $N = 15$ (the number of Pt/C layer-pairs in the multilayer stack), $d = 4.23$ nm (multilayer period, that is the thickness of a single Pt/C layer-pair), $L = 0.38$ (the ratio of the Pt layer thickness to d). The total thickness of the multilayer stack is about 68 nm. Parallel strips of $30 \times 5\,\mathrm{mm}^2$ were irradiated with 2 MeV Au^{2+} ions by rastering the ion beam on them. One strip was kept virgin (unirradiated). The other strips received the following ion fluences (ions/cm^2): V (virgin), B (1×10^{14}), C (3×10^{14}), D (5×10^{14}), E (7×10^{14}), F (1×10^{15}). The projected range of 2 MeV Au ions in such Pt/C multilayers is about 270 nm. Thus the implanted Au ions are buried deep into the glass substrates well past the multilayer stack of 68 nm thickness. The samples were analyzed by a combined X-ray standing wave (XSW) and reflectivity (XRR) technique, grazing incidence X-ray diffraction (GIXRD), transmission electron microscopy (TEM), atomic force microscopy (AFM), magnetic force microscopy (MFM) and magneto-optical Kerr effect (MOKE) measurements.

3. Results and discussions

XSW and XRR experiments were carried out using 14.0 keV incident X-rays. X-ray reflectivity results are shown in Fig. 1. We notice that the periodic multilayer

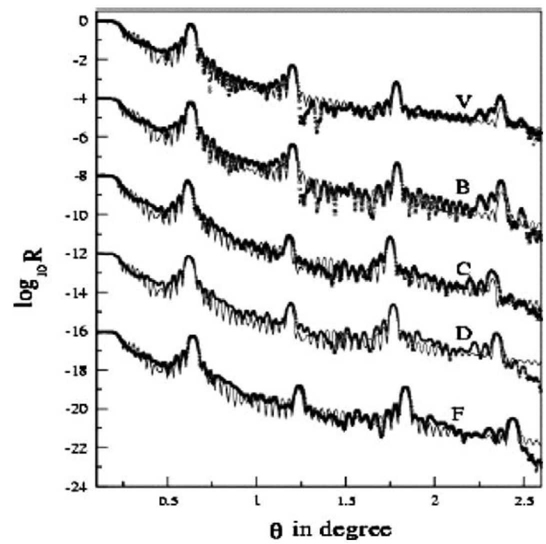

Fig. 1. Measured X-ray reflectivity (R) with fit to theory (solid lines) for the virgin sample (V) and the irradiated samples for various ion fluences. Data for the irradiated samples are shifted vertically down for clarity.

structure gives rise to Bragg peaks. Up to the fourth order Bragg peaks are seen in the reflectivity plots. Multilayer parameters, such as the multilayer period, individual layer thickness, surface and interface roughness, electron densities of layers etc. can be extracted by fitting the data to theory. These aspects will be presented elsewhere [6].

Each Bragg diffraction peak is associated with a standing wave pattern of X-rays within the multilayer. We have used the first order Bragg peak, for which the standing wave field is strong, for the detection of Fe movement within the multilayer. A first order Bragg peak and the associated X-ray standing wave patterns are illustrated in Fig. 2.

The standing wave field has the same period as the period of the multilayer. At the rising edge of Bragg reflection the antinodes are within the C layers. As the angle of incidence increases the antinodal pattern moves inward and eventually the antinodes are within the Pt layers. The effect of this movement of the antinodal pattern within the multilayer can be seen by detecting fluorescence from C or Pt atoms. Fig. 3 shows the fluorescence yield from Pt. Low yield at the rising edge of the Bragg peak (node in Pt layers) and high yield at the falling edge of the Bragg peak (antinode in the Pt layers) corroborate the formation of standing waves of X-rays and the shift of the antinodal position within the periodic multilayer [7].

When Fe impurities are present within a Pt/C periodic multilayer and we detect Fe fluorescence as we increase the angle of incidence over the Bragg peak region, what we will observe is explained in Fig. 4. If all the Fe atoms are in the Pt layers, the shape of the Fe fluorescence yield will follow the shape of the Pt fluorescence yield. If all the Fe atoms are in the C layers, the shape will be opposite, peaking on the low-angle side as the X-ray intensity is intense on the C layers. The shape will be intermediate when 50% of the Fe atoms are in the Pt layers and the other

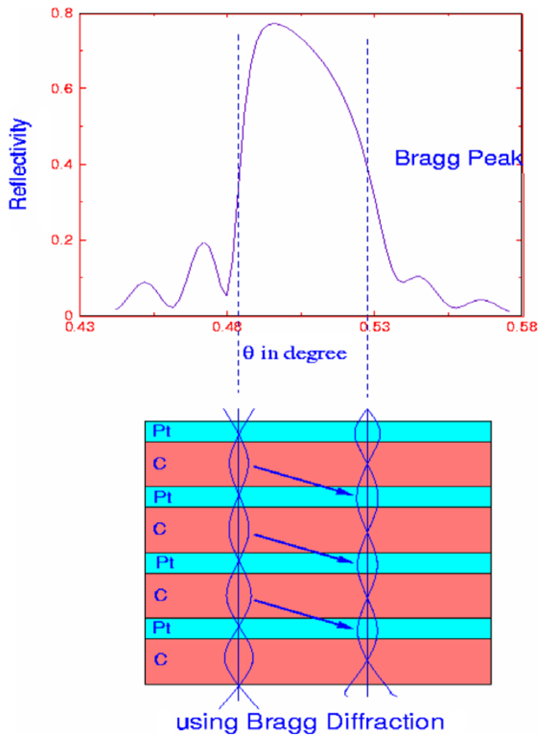

Fig. 2. Illustration of formation of standing waves of X-rays in a periodic multilayer during X-ray diffraction. As the angle of incidence increases through the diffraction peak region (top), antinodes move from the low electron-density medium (C layers) to the high electron-density medium (Pt layers) (bottom).

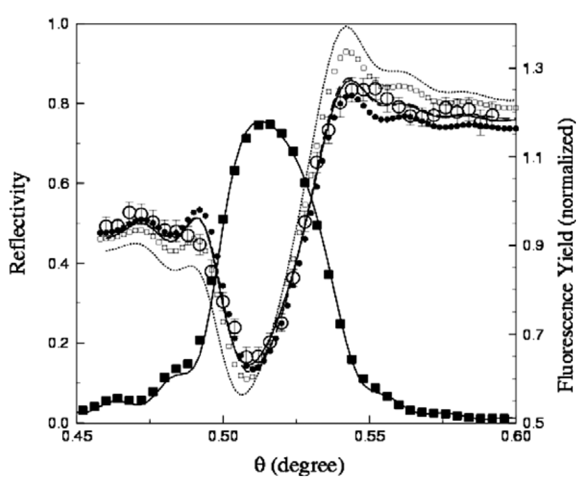

Fig. 3. First order Bragg reflection (square) and Pt L_α fluorescence yield (circle) from a periodic Pt/C multilayer. (From Ref. [7].)

50% in the C layers. In fact any concentration distribution can be extracted by fitting the observed shape of the Fe fluorescence curve to the appropriate theory. In this article we will not discuss the details of the theory. We will only mention the results.

Fig. 5 shows the Pt (L_α) fluorescence yield curves for the virgin sample and the ion-irradiated samples over the first order Bragg peak region. It may be noted that the Bragg peak positions are not the same for all samples (see Fig. 1). While there is variation in the details, the common

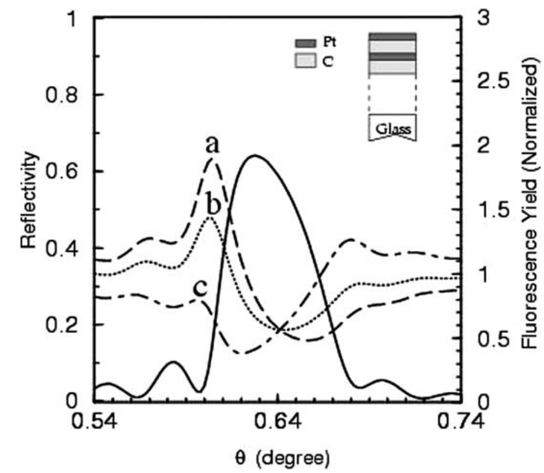

Fig. 4. Expected Fe fluorescence yield curves: (a) (100% Fe in C layers), (b) (50% in C layers, 50% in Pt layers), (c) (100% Fe in Pt layers, the shape resembling Pt fluorescence yield). (From Ref. [8].)

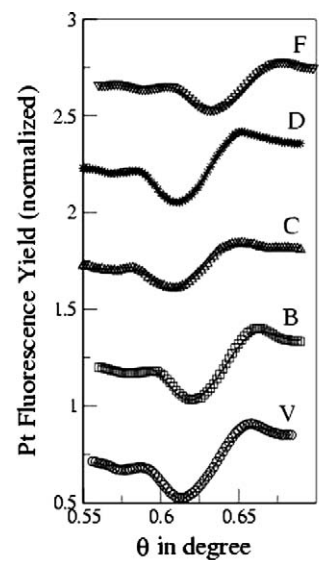

Fig. 5. Pt fluorescence yield variation over the first order Bragg peak.

feature of all the curves is that there is a minimum on the low-angle side and a maximum on the high-angle side. Let us compare this with the Fe (K_α) fluorescence yield curves, shown in Fig. 6. For the virgin sample we notice that the maximum occurs on the low-angle side. The shape is close to the theoretical curve shown in Fig. 4 for 50% Fe in C and the other 50% in Pt layers. Detailed analysis of the fluorescence yield curve yields 55% Fe in the Pt layers [6]. The shape of the Fe fluorescence curve changes when the sample is exposed to higher ion fluences. At the highest fluence studied here, the Fe fluorescence yield curve has nearly the same shape as that of the Pt fluorescence yield curve. This indicates that because of ion irradiation the majority of Fe atoms have migrated from the C layers into the Pt layers. As Fe and Pt can form alloys over a large composition range and there are magnetic phases of FePt alloys, we earlier predicted the possibility of magnetism in the ion-irradiated samples [8]. Indeed we observe the evolution of

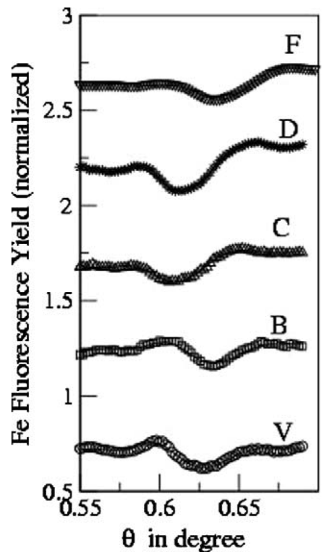

Fig. 6. Fe fluorescence yield variation over the first order Bragg peak.

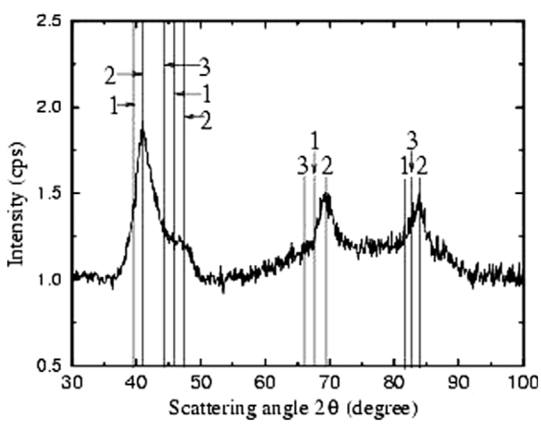

Fig. 7. GIXRD pattern from the irradiated sample F, measured with Cu K_α X-rays. Expected position of diffraction lines from Pt, FePt and bcc Fe are marked '1', '2' and '3' respectively. Peak positions agree with the presence of FePt particles.

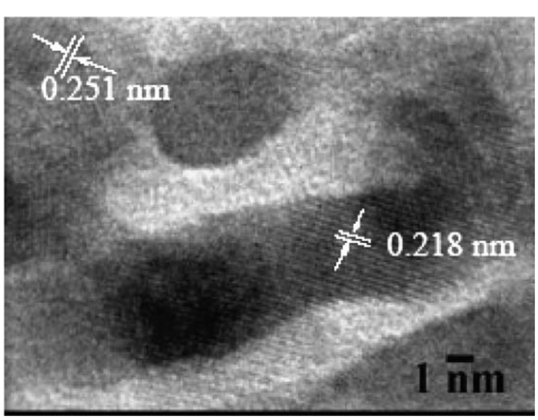

Fig. 8. A cross-sectional TEM micrograph from the irradiated sample F. Disintegration of layer structure and formation of particles are seen. The lattice planar spacing of 0.251 nm belongs neither to Pt nor to Fe. It is rather closer to planar spacing in an FePt alloy.

ferromagnetism with ion irradiation. This will be discussed later. A GIXRD spectrum from the sample irradiated at the highest fluence is shown in Fig. 7. The peak positions are consistent with a 2.5% contracted Pt lattice as well as an FePt alloy. Formation of an $L1_0$ phase of FePt cannot be confirmed from these data with such broad peaks.

The peak positions for pure Pt and pure Fe are marked in Fig. 7. These positions are not in agreement with the observed peak positions, which agree reasonably well with FePt alloy formation. In this sample, because of a phase separation process, FePt particles (also some Pt) surrounded by C are formed. A cross-sectional TEM image is shown in Fig. 8.

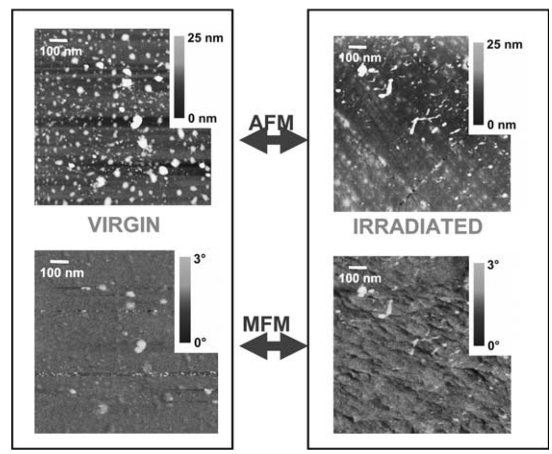

Fig. 9. Topography (AFM results) and magnetic images (MFM results) from the virgin sample and the irradiated sample F.

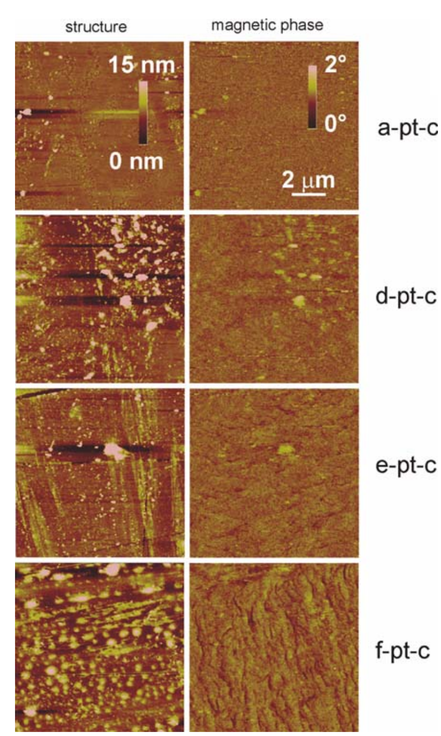

Fig. 10. Topography (left) and evolution of magnetic domains (right) from the virgin to the irradiated samples with increasing fluence.

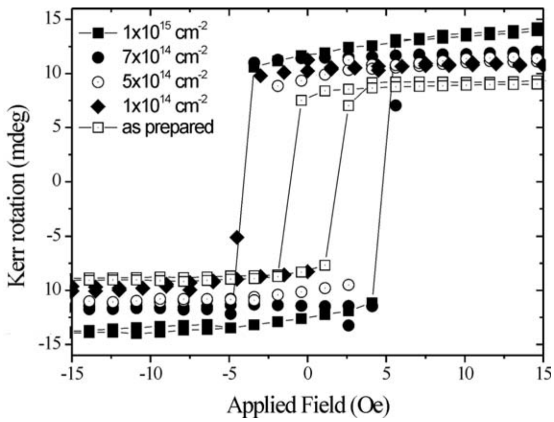

Fig. 11. MOKE results; easy-axis hysteresis loops. Coercive field increases with ion fluence and reaches a value of about 5 Oe.

Results of MFM measurements are shown in Fig. 9 for the virgin sample and the sample irradiated at the highest fluence. While the presence of magnetism is not observed in the virgin sample, the irradiated sample shows the presence of magnetic domains. The gradual evolution of magnetism with increasing ion fluence is seen in Fig. 10. Results of MOKE studies are shown in Fig. 11. An in-plane anisotropy was observed in the hysteresis loop. Fig. 11 shows the easy-axis loops. We notice that the ferromagnetic loop increases in area with a higher coercive field as the ion fluence increases. Although the presence of magnetism in the virgin sample is not clear in the MFM result, here we notice a small loop for the virgin sample as well. The MOKE technique is capable of probing the whole thickness (here 68 nm) of the sample while MFM probes only a few nm thickness of the sample in the surface region. Detailed analysis of the evolution of the shape of Fe fluorescence yield in Fig. 6 provides additional information that Fe migrates from C to Pt layers preferentially outwards. That is why magnetism is more easily detectable by MFM in the irradiated samples.

4. Conclusions and outlook

We have studied the ion irradiation effects on Pt/C multilayers with Fe impurities [Pt(Fe)/C(Fe)]. We found that ion irradiation causes migration of Fe atoms, which preferentially migrate from the C layers to Pt layers leading to formation of FePt particles. This leads to soft ferromagnetism in the irradiated sample, where the coercive field increases with ion fluence.

If a Pt/C multilayer is prepared so that Fe is present only in the C layers, we expect that there would be no magnetism in the virgin sample and the small MOKE loop we observe here for the virgin sample would be absent. Then with ion irradiation it can be converted into a soft ferromagnetic material. Using a focused ion beam with a few or a few tens of nanometer spot size, soft ferromagnetic patterns can be created in an otherwise nonmagnetic material. Such systems are expected to find application in high density storage devices. The smallness of each ferromagnetic spot may allow fabrication of single-electron-spin-valve-transistor.

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