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Argon Ion Irradiation Effect on the Magnetic Properties of Fe-Al₂O₃ Nano Granular Film

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Abstract

We studied the effect of Argon (Ar) ion irradiation on Fe-Al₂O₃ nanogranular thin film. X-ray diffraction (XRD) patterns show that the ion dose might promote the growth of the Fe₂O₃ phase from an amorphous phase to a crystalline phase. The magnetic and magnetoresistance properties were investigated using a vibrating sample magnetometer (VSM) and a four point probe (FPP). The results suggest that percolation concentration occurred at the 0.55 Fe volume fraction and with a maximum magnetoresistance (MR) ratio of 3%. The present MR ratio was lower than that of previous results, which might be related to the existence of the α -Fe₂O₃ phase promoted by Ar ion irradiation. CEMS spectra show ion irradiation induces changes from superparamagnetic characteristics to ferromagnetic ones, which indicates the spherical growth of Fe particles in the Al₂O₃ matrix.

Abstrak

Pengaruh Iradiasi Ion Argon pada Sifat Magnetik Film Nano Granular Fe-Al₂O₃. Kami telah mempelajari pengaruh iradiasi ion Argon (Ar) pada film tipis Nanogranular Fe-Al₂O₃. Dari pola difraksi sinar X (XRD) diketahui peningkatan dosis ion menyebabkan tumbuhnya fasa amorf Fe₂O₃ menjadi fasa kristalin. Sifat magnetik dari film telah diperiksa dengan alat *vibrating sample magnetometer* (VSM) dan *four point probe* (FPP). Hasil pengukuran menunjukkan bahwa konsentrasi perkolasi terjadi pada fraksi volume 0.55 Fe dimana nisbah Magnetoresistance (MR) bahan bernilai 3%. Pada pengukuran MR kali ini terukur lebih rendah dibandingkan sebelumnya yang dapat dikaitkan dengan keberadaan fasa α -Fe₂O₃ akibat pengaruh iradiasi ion Argon. Spektrum *conversion electron Mossbauer spectroscopy* (CEMS) memperlihatkan bahwa iradiasi ion telah menginduksi perubahan sifat superparamagnetik film menjadi feromagnetik yang diindikasikan oleh tumbuhnya partikel spherical Fe di dalam matrik Al₂O₃.

Keywords: argon ion irradiation, magnetoresistance, nanogranular thin film

1. Introduction

A new type of tunneling magneto resistance (TMR) which appears in nonmetallic materials Co-Al-O granular alloy thin films have discovered by Fujimori et al. [1]. Honda et al. have observed the superparamagnetic nature and the TMR in films having Fe volume fractions smaller than 45% [2]. Different with Zhu et al. works [3] -where they prepared Fe-Al₂O₃ film on well cleaned glass substrates by rf sputtering from a composite target consisting of an Fe plate placed on an Al₂O₃, or Dempsey et al. for Fe-Al₂O₃ film by Laser Deposition method [4]. The physical properties of nanocomposite materials are closely related to the size, shape and distribution of metal particles in insulating

matrix. It has been reported that the film preparation by pulsed laser deposition and the process of ion beam irradiation after film preparation were suggested to control the properties of metal particles in insulating matrix [5,6]. The morphology in conjunction with a conventional anisotropic magnetoresistance effect is the origin of a strongly anisotropic magnetoresistance of the samples [7].

Present research interest is aimed to do development of Tunneling magnetoresistance Fe-Al₂O₃ thin films deposited on Silicon single crystal by using Helicon Plasma Sputtering method [8] and study the effect of Argon (Ar) ion irradiation on the thin film due to the magnetic properties include magnetoresistance.

2. Experiment

Fe-Al₂O₃ thin films with the Fe content 0.55 volume fraction are deposited on Silicon single crystal by Helicon plasma sputtering techniques. We estimate the composition with controlling the alternating time exposure of the target during deposition without break the vacuum. The base pressure of the chamber was lower than 1×10^{-7} Torr. The target size is 50 mm in diameter. The substrate can be rotated during deposition to get uniform layer. The Argon gas pressure was maintained at 6.9×10^{-4} Torr. Deposition rate of materials are $0.6 \text{ \AA}/\text{sec}$ and $0.08 \text{ \AA}/\text{sec}$ for Fe and Al₂O₃ target respectively. The X ray diffraction (XRD) patterns of samples were measured to investigate crystalline structure and estimate the grain size. The magnetic properties were measured with vibrating sample magnetometer (VSM) at room temperature. The magnetoresistance (MR) was obtained by four point probe method in plane field, with the maximum field of 15 kOe. The measurement of conversion electron Mössbauer spectroscopy (CEMS) was done using a Mössbauer spectrometer with 740 MBq ⁵⁷Co γ -ray source (Rh matrix), and conversion electrons were detected with a proportional counter flowed with He + 10% methane mixture gas. Argon ion irradiation were performed to the samples at energy ion 400 keV and at doses range 1×10^{15} ion/cm² until 5×10^{16} ion/cm². All experiment were done at Advanced Industrial Science and Technology (AIST)-Tsukuba Japan.

3. Results and Discussion

X-ray diffraction profile. Figure 1 shows the Fe 110 peak become narrow due to the increasing of Ar ion dose. This indicated that the crystallite size of Fe become larger as ion dose increases. The interesting result was found for the existence of Fe₂O₃ as predicted by CEMS as reported elsewhere [9]. The International Center for Diffraction Data (ICDD) or Joint Committee on Powder Diffraction Standards (JCPDS) number of Fe phase was 06-0696 with system crystal Cubic and Space Group Im3m(229), and Fe₂O₃ phase was 33-0664 with Space Group R3C(169), respectively. The Fe₂O₃ 116 peak appeared after irradiated at dose 1×10^{15} ion/cm² and become larger as ion dose increases. Then at dose 5×10^{16} ion/cm², the another peak of Fe₂O₃ 110 was appeared. We suggest that the ion dose might promote the growth of Fe₂O₃ phase from amorphous phase to become crystalline phase. It is also suggest that Fe₂O₃ phase was surrounded as a coating material at the surface of Fe particle with variation in thickness and depend on ion dose. It is the reason why only at high dose 5×10^{16} ion/cm² Fe₂O₃ 110 and 116 peaks was clearly seen.

Magnetization curve. Figure 2 shows typical magnetization curves measured for Fe-Al₂O₃ sample no#5 post

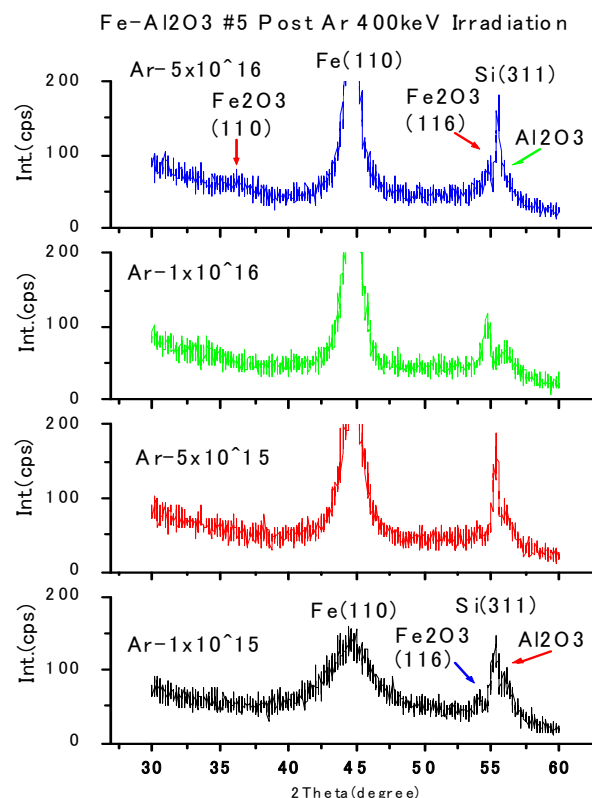


Figure 1. X Ray Diffraction Pattern for Fe-Al₂O₃ Granular Film Sample no#5 Post Irradiation by 400keV Argon at Different Dose

irradiated by 400 keV Argon ion at dose 5×10^{16} ion/cm² (upper), at dose 1×10^{15} ion/cm² (middle) and as prepared sample (lower). In the figure, in plane M-H curve when ion doses increase it is clear that the ferromagnetic component become increase and squareness ratio (M_r/M_s) become larger than zero as for not irradiated sample. This behaviour clearly related to the growing of Fe granular. In case perpendicular M-H curve, it is interest to clarify that the evolution of hysteresis curve strongly related to the growing of Fe₂O₃ ferrimagnetic phase from amorph to crystallie phase as shown in the XRD pattern in Figure 1, in addition to the growth of the Fe core granular indicated by the ferromagnetic component. This characteristic is shown in Figure 2 (upper), especially for the sample post irradiation at ion doses of 5×10^{16} ion/cm².

Conversion electron Mossbauer spectroscopy (CEMS). CEMS spectra show the ion irradiation induces the changes from the superparamagnetic characteristics to the ferromagnetic one and indicate the spherical growth of Fe particles in Al₂O₃ matrix as reported by Sakamoto, et al. [9]. The typical of CEMS spectra for Fe-Al₂O₃ films prepared by on-off Fe deposition, (a) 3s-12s before ion irradiation and (b) 3s-12s after ion irradiation were shown in Figure 3.

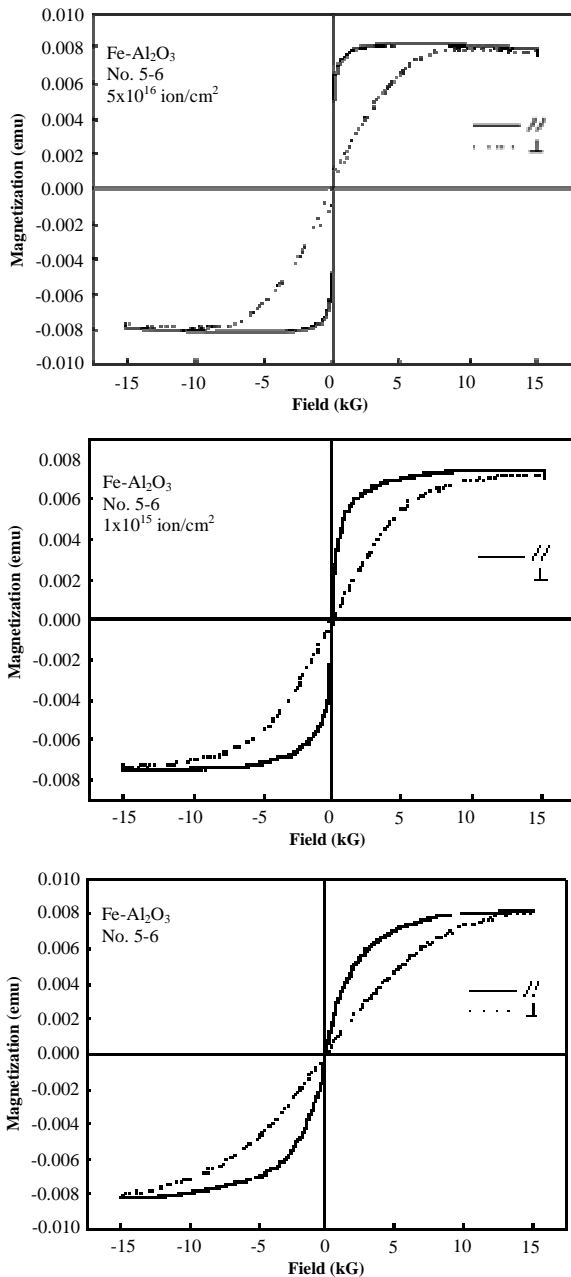


Figure 2. Magnetization Curve for Fe-Al₂O₃ Sample no#5 Post Irradiated by 400keV Argon Ion at Doses as Prepared (Lower), 1x10¹⁵ ion/cm² (Middle) and 5x10¹⁶ ion/cm² (Upper)

Magnetoresistance properties. The magnetoresistance properties of granular film as shown in Figure 4, become lower from almost 3% for as prepared sample to 2% as applied 400 keV Ar ion beam irradiation at dose 1x10¹⁵ ion/cm². This is clearly due to the growth of Fe core granular as indicated by XRD pattern in Figure 1. We suggested that the increasing of ferromagnetic phase and lowering of superparamagnetic phase, as shown in Figure 2, are related to the smaller grains of Fe fine particles in Al₂O₃.

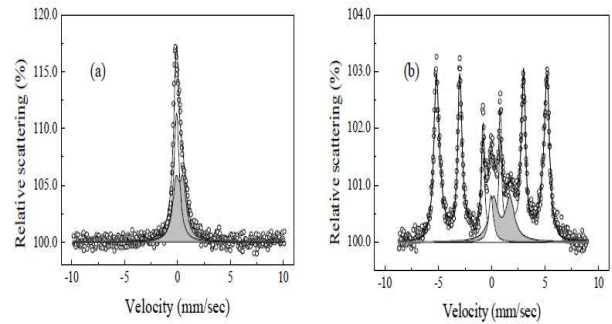


Figure 3. CEMS Spectra of Fe-Al₂O₃ Films Prepared by on-off Fe Deposition (a) 3s-12s Before Ion Irradiation and (b) 3s-12s After Ion Irradiation. The Shaded Peaks are Doublet Peaks

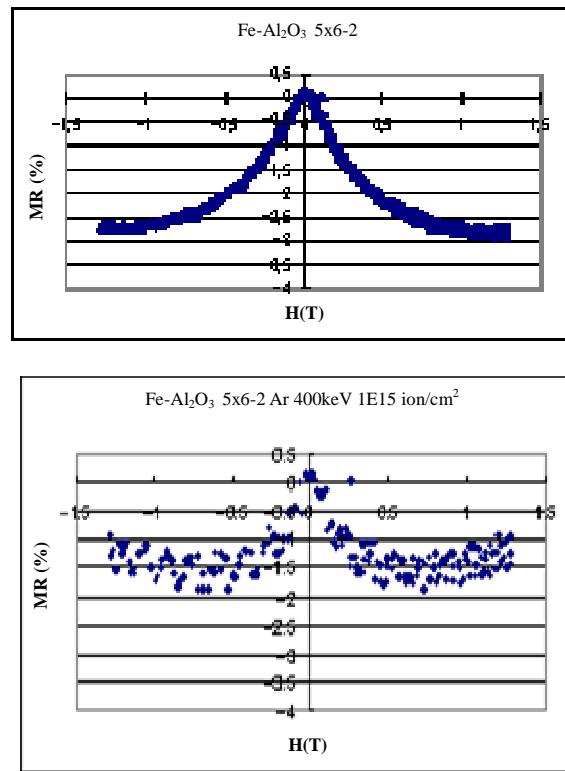


Figure 4. Magnetoresistance Curve for Sample #5-6 as Prepared (Upper) and Post Irradiation at Dose 1x10¹⁵ Ion/cm² (Lower)

4. Conclusions

Argon irradiation at energy 400keV were performed at sample no#5 (Fe volume fraction 0.55) with doses from 1x10¹⁵ until 5x10¹⁶ ion/cm². The effect of ion irradiation were increased the ferromagnetic phase and lower the magnetoresistance ratio. Ion irradiation have promoted growth the Fe₂O₃ phase as indicated by Fe₂O₃ 110 and Fe₂O₃ 116 peak at XRD pattern, beside the growing fast of Fe core granular also CEMS spectra indicate the spherical growth of Fe particles in Al₂O₃ matrix film.

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