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# Argon Ion Irradiation Effect on the Magnetic Properties of $Fe\text{-}Al_2O_3$ Nano Granular Film

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## Abstract

We studied the effect of Argon (Ar) ion irradiation on $Fe\text{-}Al_2O_3$ nanogranular thin film. X-ray diffraction (XRD) patterns show that the ion dose might promote the growth of the $Fe_2O_3$ 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 $\alpha\text{-}Fe_2O_3$ 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 $Al_2O_3$ matrix.

## Abstrak

**Pengaruh Iradiasi Ion Argon pada Sifat Magnetik Film Nano Granular $Fe\text{-}Al_2O_3$.** Kami telah mempelajari pengaruh iradiasi ion Argon (Ar) pada film tipis Nanogranular $Fe\text{-}Al_2O_3$. Dari pola difraksi sinar X (XRD) diketahui peningkatan dosis ion menyebabkan tumbuhnya fasa amorf $Fe_2O_3$ 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 $\alpha\text{-}Fe_2O_3$ 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 $Al_2O_3$.

*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 superpara-magnetic nature and the TMR in films having Fe volume fractions smallers than 45% [2]. Different with Zhu et al. works [3] -where they prepared $Fe\text{-}Al_2O_3$ film on well cleaned glass substrates by rf sputtering from a composite target consisting of an Fe plate placed on an $Al_2O_3$, or Dempsey et al. for $Fe\text{-}Al_2O_3$ 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 $Fe\text{-}Al_2O_3$ 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

$Fe-Al_2O_3$ 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 $1x10^{-7}$ 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 $6.9x10^{-4}$ Torr. Deposition rate of materials are 0.6Å/sec and 0.08Å/sec for Fe and $Al_2O_3$ 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 $^{57}$Co 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 $1x10^{15}$ ion/cm$^2$ until $5x10^{16}$ ion/cm$^2$. All experiment were done at Advanced Industrial Science and Technology (AIST)-Tsukuba Japan.

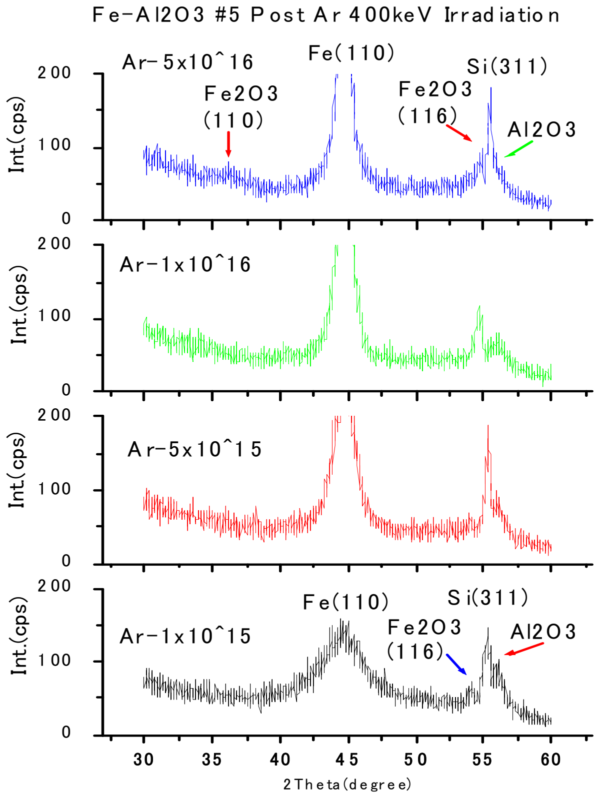


**Figure 1. X Ray Diffraction Pattern for $Fe-Al_2O_3$ Granular Film Sample no#5 Post Irradiation by 400keV Argon at Different Dose**

## 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 $Fe_2O_3$ as predicted by CEMS as reported elsewhere [9]. The International Center for Difraction 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 $Fe_2O_3$ phase was 33-0664 with Space Group R3C(169), respectively. The $Fe_2O_3$ 116 peak appeared after irradiated at dose $1x10^{15}$ ion/cm$^2$ and become larger as ion dose increases. Then at dose $5x10^{16}$ ion/cm$^2$, the another peak of $Fe_2O_3$ 110 was appeared. We suggest that the ion dose might promote the growth of $Fe_2O_3$ phase from amorphous phase to become crystalline phase. It is also suggest that $Fe_2O_3$ 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 $5x10^{16}$ ion/cm$^2$ $Fe_2O_3$ 110 and 116 peaks was clearly seen.

**Magnetization curve.** Figure 2 shows typical magnetization curves measured for $Fe-Al_2O_3$ sample no#5 post irradiated by 400 keV Argon ion at dose $5x10^{16}$ ion/cm$^2$ (upper), at dose $1x10^{15}$ ion/cm$^2$ (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 (Mr/Ms ) 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 hysterisis curve strongly related to the growing of $Fe_2O_3$ 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 $5x10^{16}$ ion/cm$^2$.

**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 $Al_2O_3$ matrix as reported by Sakamoto, et al. [9]. The typical of CEMS spectra for $Fe-Al_2O_3$ 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.

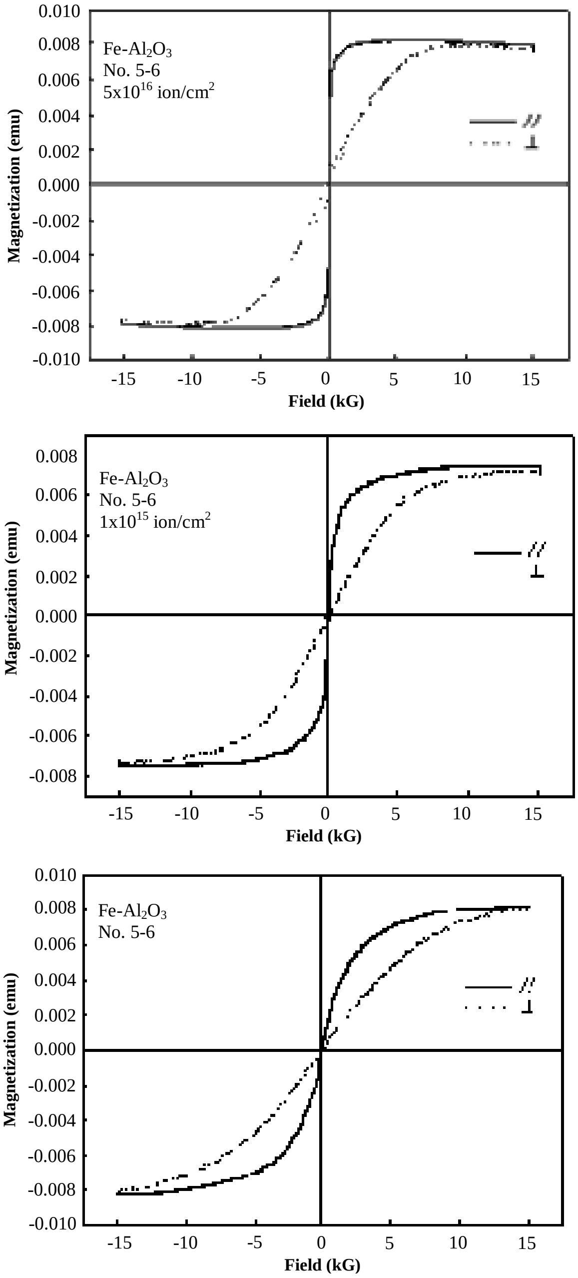


**Figure 2. Magnetization Curve for Fe-$Al_2O_3$ Sample no#5 Post Irradiated by 400keV Argon Ion at Doses as Prepared (Lower), $1x10^{15}$ ion/$cm^2$ (Middle) and $5x10^{16}$ ion/$cm^2$ (Upper)**

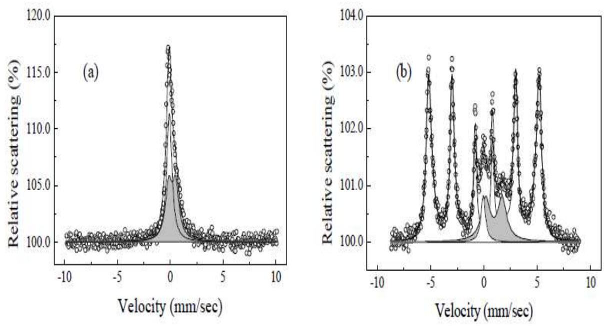


**Figure 3. CEMS Spectra of Fe-$Al_2O_3$ Films Prepared by on-off Fe Deposition (a) 3s-12s Before Ion Irradiation and (b) 3s-12s After Ion Irradiation. The Shaded Peaks are Doublet Peaks**

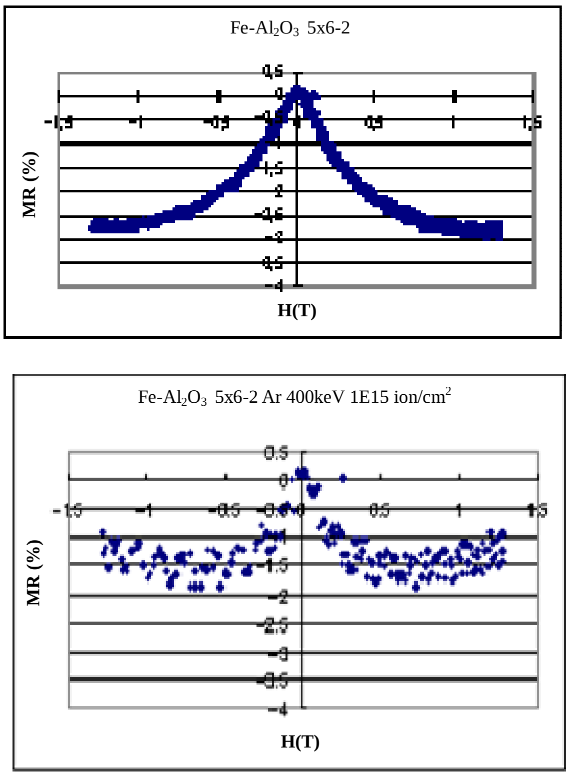


**Figure 4. Magnetoresistance Curve for Sample #5-6 as Prepared (Upper) and Post Irradiation at Dose $1x10^{15}$ Ion/$cm^2$ (Lower)**

**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 $1x10^{15}$ ion/$cm^2$. 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 $Al_2O_3$.

## 4. Conclusions

Argon irradiation at energy 400keV were performed at sample no#5 (Fe volume fraction 0.55) with doses from $1x10^{15}$ until $5x10^{16}$ ion/$cm^2$. The effect of ion irradiation were increased the ferromagnetic phase and lower the magnetoresistance ratio. Ion irradiation have promoted growth the $Fe_2O_3$ phase as indicated by $Fe_2O_3$ 110 and $Fe_2O_3$ 116 peak at XRD pattern, beside the growing fast of Fe core granular also CEMS spectra indicate the spherical growth of Fe particles in $Al_2O_3$ matrix film.

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