# Microstructure of implanted Fe nanoparticles in silica glass and their effect on magnetic properties Kengo Fukuda<sup>1</sup> Masaaki Ochi<sup>1</sup>, Hiroshi Kojima<sup>1</sup>, Ren Mayumi<sup>1</sup>, Yoshihiro Okamoto<sup>2</sup>,

Satoshi Semboshi<sup>3</sup>, Yuichi Saitoh<sup>4</sup>, Fuminobu Hori<sup>1</sup> and Akihiro Iwase<sup>1,\*</sup>,

# <sup>1</sup> Department of Materials Science, Osaka Prefecture University, Sakai, Osaka 599-8531, Japan <sup>2</sup> Materials Science Research Center, Japan Atomic Energy Agency, Tokai, Ibaraki, 319-1195, Japan <sup>3</sup> Institute for Materials Research, Tohoku University, Sakai, Osaka, 599-8531, Japan

<sup>4</sup> National Institutes for Quantum and Radiological Science and Technology, Takasaki, Gunma, 370-

1292, Japan

### 1 Introduction

Nanocomposite glasses containing metal nanoparticles in matrix have become an object of interest among many researchers because of their promising properties. For example, they can provide new magnetic, optical or dielectric properties and so on. Among them, we focus on magnetic properties. The distribution of metal nanoparticles (Fe, Co, Ni) will lead to the modification of the magnetic properties [1]. There are several well-known methods to prepare metal nanoparticles in solids. Ion implantation is one of the most suitable techniques for the synthesis of metal nanoparticles in solid host matrix. Also, by subsequent heavy ion irradiation, the magnetic property and the state of implanted ions may be changed. In order to understand the oxidation state of implanted elements, the x-ray absorption near edge structure (XANES) is a good tool.

In the present study, we have investigated the effect of Fe ion implantation and the subsequent energetic heavy ion irradiation on the magnetic properties and microstructure of Fe clusters in silica glass by SQUID, TEM and XANES measurements.

## 2 Experiment

Target samples were amorphous SiO<sub>2</sub> sheets with the dimension of 5mmx5mmx1mm. They were implanted with 380keV Fe ions at various fluences at room temperature by using an ion-implanter at Takasaki Advanced Radiation Research Institute, QST. After the ion implantation, some samples were subsequently irradiated with 16MeV Au ions. The effects of the implantation and the subsequent high-energy ion irradiation on magnetic properties were studied by using a SQUID magnetometer. Microstructures of implanted Fe nanoparticles were observed by transmission electron microscope (TEM). In order to investigate the oxidation state of the implanted Fe ions, the x-ray absorption near edge structure (XANES) was measured at a synchrotron radiation facility of High Energy Accelerator Research Organization (KEK-PF).

#### 3 Results and Discussion

In Fig.1, Fe K-edge XANES spectra for some model compounds (Fe, FeO,  $Fe_2O_3$ ) are shown. Depending on

the Fe oxidation state, the XANES spectra vary systematically in the position of the pre-edge peaks  $(7.11 \sim 7.12 \text{keV})$ . The peak position shifts to low energies with decreasing the valence number of Fe ion. From this result, pre-edge peaks for the Fe implanted samples help us estimate Fe oxidation state in SiO<sub>2</sub>.

In Fig.2, Fe K-edge XANES spectra for Fe-implanted silica glass are shown. From this figure, pre-edge peak position shifts to low energies with increasing the amount of implanted Fe ions. This result suggests that the valence number of implanted Fe ions decreases and becomes close to zero (pure Fe) if Fe ion fulence becomes larger. In other words, when focused on one Fe atom implanted in silica glass, more Fe atoms exist around the Fe atom with increasing ion fluence.

In Fig.3, the transmission electron microscope (TEM) images of Fe implanted silica glass are shown. The left figure is for Fe ion fluence of  $1.4 \times 10^{17}$  (/cm<sup>2</sup>). The right one is for  $7 \times 10^{16}$  (/cm<sup>2</sup>). These TEM images show that depending on Fe ion fluence, the size of Fe nanoparticles is different. With increasing the amount of implanted Fe ions, the size of Fe nanoparticles becomes larger.

In Fig.4, the magnetization for the silica glass implanted with Fe ions at 300K as a function of magnetic field is shown. The experimental result shows that the magnetic state of implanted Fe is the ferromagnetism or the super-paramagnetism. Also, the magnetization for one Fe atom implanted in silica glass increases with increasing the amount of implanted Fe ions. From the experimental results of the XANES measurement and the TEM observation (Fig.1, Fig.2), clusters produced from the implanted Fe ions become close to metallic Fe clusters, and their size becomes larger with increasing the amount of implanted Fe ions. This is the reason why the magnetization per one Fe atom increases with increasing the amount of implanted Fe atoms.

In Fig.5, the magnetization for Fe-implanted silica glass before and after the Au ion irradiation is shown. By the subsequent 16MeV Au ion irradiation, the value of magnetization a little decreases. To understand this change by the subsequent Au ion irradiation, XANES measurement and TEM observation will also be useful.

#### Acknowledgement

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#### References

[1] Y. Fujimura et al., J. Alloy. Com. 682 (2016) 805-814

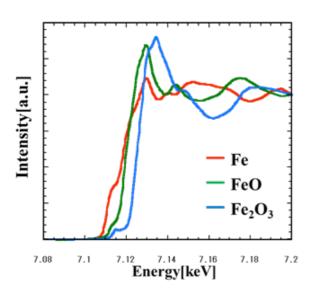


Fig.1 XANES spectra near Fe K-edge for pure Fe, FeO, and  $\mbox{Fe}_2\mbox{O}_3$ 

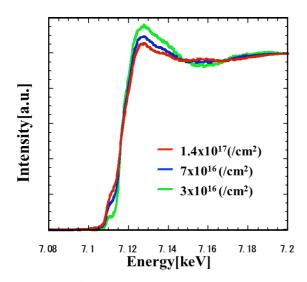


Fig.2 XANES spectra near Fe K-edge for Fe-implanted SiO<sub>2</sub> samples

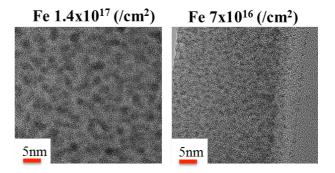


Fig.3 TEM images of Fe nanoparticles implanted in silica glass

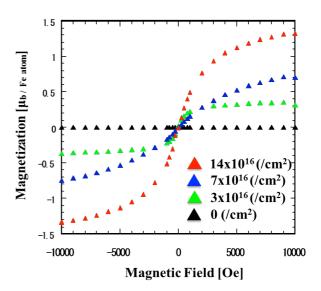


Fig.4 Magnetization for silica glass implanted with Fe ions at 300K as a function of magnetic field.

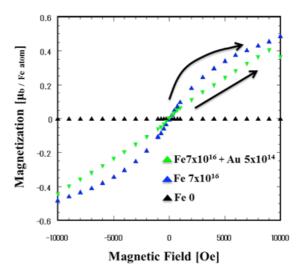


Fig.5 Magnetization for silica glass implanted with Fe ions before and after Au ion irradiation.

\* iwase@mtr.osakafu-u.ac.jp