## Topotactic phase transformation of spinel Fe<sub>3</sub>O<sub>4</sub> to FeO with rock salt structure via ion irradiation

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## Introduction

An interesting topic of recent study is how spinel materials, such as MgAl<sub>2</sub>O<sub>4</sub>, MgGa<sub>2</sub>O<sub>4</sub> and MgIn<sub>2</sub>O<sub>4</sub><sup>1</sup>, are affected by ion implantation, a technique which can change a material's fundamental properties. However, hardly any investigation has been carried out on irradiated spinel ferrite materials. In a previous study, we investigated the effect of Kr ion implantation on the epitaxial growth  $CoFe_2O_4$  thin films, and found that the magnetization decreased due to ion irradiation causing a structural transformation from spinel to rock salt type. However, the mechanism of topotactic reaction in epitaxial films was not well explained. In this study, we focus on prototype spinel Fe<sub>3</sub>O<sub>4</sub> thin films and investigate the structural transformation from spinel to rock salt via Kr ion implantation.

## Experiment

 $Fe_3O_4$  thin films were fabricated on MgO (001) single crystal substrates by reactive RF sputtering. The substrate temperature was kept at 300 °C. Kr ions were accelerated in a conventional ion implantation system in Nanotechnology Platform. The acceleration was set at 30 keV and the ion dosage was controlled at  $5 \times 10^{15}$  ions/cm<sup>2</sup>. An annular 25% <sup>57</sup>Fe enriched tablet placed on a natural Fe sputtering target was custom-made in order to perform room temperature conversion electron Mössbauer spectroscopy (CEMS). The structure transformation was measured by X-ray diffraction at beamline BL-4C of the Photon Factory, KEK, while the magnetization was measured by vibrating sample magnetometer (VSM) at room temperature.

## Results

Figure 1 and Figure 2 show the result of the  $(0 \ 0 \ l)$  scan, which defined with MgO lattice, of sample after  $5 \times 10^{15}$  ions/cm<sup>2</sup> ion irradiation. In Fig.1, the intensity of strongest peak (partial shown) indicates the reflection of the substrate, MgO (0 0 2). The protuberant part on the right side of MgO (0 0 2) peak, shown by the arrow represents the thin film. From the lattice constant value, we estimated that the thin film observed is Fe<sub>3</sub>O<sub>4</sub> (0 0 4). In Fig. 2, the reflection of the MgO (0 0 2) substrate is also observed as the peak of strongest intensity, the arrow indicating the slight swell on the left side of the MgO (0 0 2) peak (shown by the arrow) indicates the presence of FeO (0 0 2) thin film, as estimated from lattice constant.

From these results we understand that after ion irradiation, the spinel structure of  $Fe_3O_4$  changes to the rock salt structure of FeO. However, some  $Fe_3O_4$  remains. In other words, the structural transformation is partial, both  $Fe_3O_4$  and FeO can be observed in the irradiated sample. We consider that this incomplete change is due to ion irradiation not being enough to affect all parts of the thick  $Fe_3O_4$  thin film. To solve this issue, we will fabricate thinner films of about 15 nm thickness.

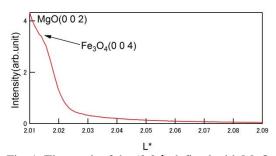


Fig. 1. The result of the  $(0 \ 0 \ l)$  defined with MgO lattice, which shows Fe<sub>3</sub>O<sub>4</sub>  $(0 \ 0 \ 4)$  after  $5 \times 10^{15}$  ions/cm<sup>2</sup> ion irradiation.

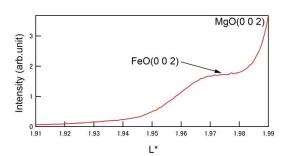


Fig. 2. The result of the reciprocal lattice indexes defined with MgO lattice, which shows FeO (0 0 2) thin film after  $5 \times 10^{15}$  ions/cm<sup>2</sup> ion irradiation.

1) B.P. Uberuga et al., Nat. Commun., 6 (2015)