§3. Depth Profile of Deuterium in Fe<sub>2</sub>O<sub>3</sub> under Low-Energy Deuterium Exposure

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Oxide layers such as  $Fe_2O_3$  will be formed on stainless steel (SUS), one of structure materials widely used for fusion devices [1], during removal process of hydrogen isotopes [2]. Thus, the depth distribution of deuterium (D) and amount of D-retention in the oxide layers are important for designing the devices. We have studied the depth profile of D and D-retention in  $Fe_2O_3$ under low-energy D plasma exposure [3].

Fe<sub>2</sub>O<sub>3</sub> layers were prepared by deposition of Fe on SiO<sub>2</sub>-glass substrates followed by oxidation in air at 400 to 500 °C for more than 5 min. X-ray diffraction (XRD) shows that the oxide is  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, i.e., hematite (hexagonal corundum structure) and the composition is evaluated to be Fe:O=2:3 with an accuracy of 10% by Rutherford backscattering spectroscopy (RBS) of 1.8 MeV He<sup>+</sup>. RBS is also employed to evaluate the film thickness. Nuclear reaction, D(<sup>3</sup>He, $\alpha$ )P, analysis (NRA) with 1 MeV <sup>3</sup>He<sup>+</sup> was employed to obtain the depth profile of D. Here, the incident angle and detection angle of  $\alpha$ -particles are 20° and 70° from the sample surface normal, respectively (NRA angle is 90° measured from the incident beam direction).

Figure 1 shows the depth profile of D in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. One sees that D's are distributed beyond the depth resolution (43 nm) and the range (30 nm) calculated [4] by considering the fractions of  $D_3(60\%)$ ,  $D_2(38\%)$  and D(2%) [5]. It also appears that more than 90 % of D's are located within the film thickness of 0.1 µm, indicating small diffusivity of D in α-Fe<sub>2</sub>O<sub>3</sub> near room temperature. By integrating the D density up to 0.2 µm, the amount of D-retention is obtained to be  $45 \times 10^{15}$  $cm^{-2}$  and corrected to be  $50x10^{15} cm^{-2}$  by taking  ${}^{3}He^{+}$ beam induced desorption (evaluated from measurement of D-retention vs <sup>3</sup>He<sup>+</sup> beam fluence) into account, in reasonable agreement with the reported value [3]. After the D-exposure, the XRD intensity decreased to 1/3 of that before D-exposure and the lattice parameter increased by 0.4 %. These modifications are not likely due to irradiation effects by low energy D (limited within 30 nm) but incorporation of D into Fe<sub>2</sub>O<sub>3</sub> lattice.

In the mixed layers (more than 2  $\mu$ m) of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> (maghemite, cubic spinel structure) and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, which were prepared by oxidation of Fe at 400 to 500 °C, the

depth profile of D appears to be similar to that in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, but D-density is larger than that in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> in regions deeper than 0.1 µm. D-retention is evaluated to be  $73 \times 10^{15}$  cm<sup>-2</sup>, by integrating the D-density up to 0.5 µm and correcting <sup>3</sup>He<sup>+</sup> beam induced desorption, the value being in agreement with the reported value (76x10<sup>15</sup> cm<sup>-2</sup> [3]). D-retention in the mixed layers is larger than that in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. The difference could be largely due to the difference of the D-distribution mentioned above. D-retention in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> is larger than that in Fe (12x10<sup>15</sup> cm<sup>-2</sup>) and SUS316L (36x10<sup>15</sup> cm<sup>-2</sup>). Measurements of D distribution in SUS and Fe, dynamic D-retention and thermal desorption of D are under way.



Fig. 1 Depth distribution of D density in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> (thickness of 100 nm) normalized to Fe density exposed to D-plasma (1.5 kV AC glow-discharge in 0.5 Torr D<sub>2</sub> for 30 min) ( $\bullet$ ). The dotted line indicates the range profile of D calculated using TRIM code [4].

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