§2. Deuterium Retention in Iron Oxide (Fe₂O₃) under Low-Energy Deuterium Exposure

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Stainless steel (SUS) is widely employed for the nuclear fusion devices. When O_2 -plasma-discharge is performed to remove tritium [1], layers of iron-oxide such as Fe_2O_3 will be formed on SUS surface [2]. Thus, deuterium (D) retention in the oxide layers is important for designing the devices. In this work, we have studied D-retention in Fe_2O_3 under O_2 plasma exposure [3], as well as in SUS and Fe for comparison.

Fe₂O₃ layers were prepared by oxidation of Fe deposited on SiO₂-glass substrates and Fe sheet in air. According to X-ray diffraction (XRD), the oxide layers on SiO₂ oxidized at 400 to 500 °C for more than 5 min are polycrystalline hematite (hexagonal α-Fe₂O₃) while those on Fe oxidized at 500 °C are mixture of α-Fe₂O₃ and cubic γ-Fe₂O₃ (maghemite). Oxide-layer thickness is evaluated by means of Rutherford backscattering spectroscopy (RBS) of 1.8 MeV He⁺ and H⁺, using the stopping power [4] and density of 3.96x10²² Fe cm⁻³ (5.25 g cm⁻³). For analysis of ion beam data, e.g., RBS, SUS is assumed to consist of Fe only with the density of Fe $(8.48 \times 10^{22} \text{ cm}^{-3} \text{ or } 7.86 \text{ g cm}^{-3})$. The samples were exposed to D₂-plasma under AC discharge of 1.5 kV in D₂ gas pressure of 0.4 Torr for 15 min at room temperature [3]. The amount of D was analyzed by using nuclear reaction analysis (NRA), D(³He,α)H with ³He-energy of 1.0 MeV. The NRA cross section was taken after [5]. The depth resolution of the NRA at very near surface is estimated to be ~0.24 and 0.19 µm for Fe₂O₃ and Fe. Also, the probing depth is roughly estimated to be 1.3 and 1µm for Fe₂O₃ and Fe, based on the calculated path length [4] where the ³He beam slows down to 0.25 MeV so that the NRA cross section is reduced to one-fifth of the maximum value.

The results are summarized in Table 1. Firstly, D-retention in α -Fe₂O₃ appears to be independent of the oxide layer thickness of 0.06 to 0.86 μ m, indicating low diffusivity of D in α -Fe₂O₃ ($<4\times10^{14}$ cm²s⁻¹). Secondly, D-retention in γ -Fe₂O₃ is found to be larger by a factor of at least 1.7 than that in α -Fe₂O₃. An explanation is that in γ -Fe₂O₃, non-negligible fraction of vacancies exists in Fe site (defect-spinel-type-structure, e.g., Fe_{2/3}Fe₂O₄ or one third of vacancies at Mg site in the spinel to keep Fe₂O₃ composition) [6]. Therefore it is suggested that D's occupy the vacancy sites in γ -Fe₂O₃,

enhancing D-retention. Another explanation is that the D-diffusivity in $\gamma\text{-Fe}_2O_3$ is larger than that in $\alpha\text{-Fe}_2O_3$, giving rise to larger D-retention. Measurement of D-depth-distribution, estimation of the ratio of $\gamma\text{-}$ to $\alpha\text{-Fe}_2O_3$ in these mixed layers, and preparation of only $\gamma\text{-Fe}_2O_3$ layers followed by D-retention measurement would be fruitful. In this study, the density of $\gamma\text{-Fe}_2O_3$ is assumed to be the same as that of $\alpha\text{-Fe}_2O_3$. Due to the presence of vacancies as described above, the density of $\gamma\text{-Fe}_2O_3$ is smaller by several % than that of $\alpha\text{-Fe}_2O_3$, which is insignificant in the D-retention evaluation.

D-retention in Fe appears to be the smallest in this study and a half of that in α -Fe₂O₃, in spite of a large diffusivity of H and D in Fe [7]. Surprisingly, D-retention in SUS is larger than that in Fe and α -Fe₂O₃. The amount of oxygen near surface of Fe and SUS is obtained to be ~13x10¹⁶ cm⁻², by using ¹⁶O(d, α)¹⁴N with 1.2 MeV d. This indicates the existence of oxide layers near surface and a contribution of the oxide layers to D-retention.

Table 1 Summary of D-retention in Fe₂O₃, Fe and SUS (an estimated error of 15 %). Thickness of oxide layers, Fe and SUS are given in the parenthesis.

Sample	D-retention (10 ¹⁵ cm ⁻²)
α-Fe ₂ O ₃ /SiO ₂ (0.06, 0.18, 0.86 μm)	21
(α+γ)-Fe ₂ O ₃ /Fe (>2μm)	36
Fe (0.2 mm)	12
SUS (0.25, 2 mm)	27

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