§2. Deuterium Retention in Iron Oxide (Fe_2O_3) 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 D_2 plasma exposure [3], as well as in SUS and Fe for comparison.

Fe_2O_3 layers were prepared by oxidation of Fe deposited on SiO_2-glass substrates and Fe sheet in air. According to X-ray diffraction (XRD), the oxide layers on SiO_2 oxidized at 400 to 500 ºC for more than 5 min are polycrystalline hematite (hexagonal α-Fe_2O_3) while those on Fe oxidized at 500 ºC are mixture of α-Fe_2O_3 and cubic γ-Fe_2O_3 (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 3.96×10^{22} 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 (8.48×10^{22} cm^{-3} or 7.86 g cm^{-3}). The samples were exposed to D_2-plasma under AC discharge of 1.5 kV in D_2 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(^{3}He,α)H with ^{3}He-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 Fe_2O_3 and Fe. Also, the probing depth is roughly estimated to be 1.3 and 1µm for Fe_2O_3 and Fe, based on the calculated path length [4] where the ^{3}He 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-Fe_2O_3 appears to be independent of the oxide layer thickness of 0.06 to 0.86 µm, indicating low diffusivity of D in α-Fe_2O_3 ($<4 \times 10^{14}$ cm^2s^{-1}). Secondly, D-retention in γ-Fe_2O_3 is found to be larger by a factor of at least 1.7 than that in α-Fe_2O_3. An explanation is that in γ-Fe_2O_3, non-negligible fraction of vacancies exists in Fe site (defect-spinel-type-structure, e.g., $Fe_{2/3}Fe_2O_4$ or one third of vacancies at Mg site in the spinel to keep Fe_2O_3 composition) [6]. Therefore it is suggested that D's occupy the vacancy sites in γ-Fe_2O_3, enhancing D-retention. Another explanation is that the D-diffusivity in γ-Fe_2O_3 is larger than that in α-Fe_2O_3, giving rise to larger D-retention. Measurement of D-depth-distribution, estimation of the ratio of γ- to α-Fe_2O_3 in these mixed layers, and preparation of only γ-Fe_2O_3 layers followed by D-retention measurement would be fruitful. In this study, the density of γ-Fe_2O_3 is assumed to be the same as that of α-Fe_2O_3. Due to the presence of vacancies as described above, the density of γ-Fe_2O_3 is smaller by several % than that of α-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-Fe_2O_3, 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-Fe_2O_3. The amount of oxygen near surface of Fe and SUS is obtained to be ~13×10^{16} cm^{-2}, by using ^{16}O(d, α)^{14}N 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 Fe_2O_3, 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_2O_3/SiO_2 (0.06, 0.18, 0.86 µm)	21
(α+γ)-Fe_2O_3/Fe (>2µm)	36
Fe (0.2 mm)	12
SUS (0.25, 2 mm)	27

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