§23. Measurement of Hydrogen Permeation through Stainless Steel Membrane by Electron Stimulated Desorption Method

Takagi, S., Sakai, Y., Hirata, K. (Dept. Phys. Toho Univ.), Murase, Y., Tosa, M., Itakura, A. (NIMS), Sakaue, H., Kato, D.

Hydrogen behavior in metals is serious problem in fusion reactor as well as hydrogen embrittlement, storage and permeation. There has been considerable research on hydrogen diffusion or permeation in metals, however, permeated hydrogen on metal surfaces has not been dynamically investigated. Permeated hydrogen concentration on the surface of stainless steel and permeated hydrogen pressure in vacuum vessel were measured as a function of time in this experiment.

The surface concentration of hydrogen was observed by electron stimulated desorption (ESD) method. The experiment has been carried out using a scanning electron microscope equipped with a quadrupole mass spectrometer (QMS), ESD ion detection system and a sample holder which can expose the sample to deuterium gas (Fig.1). The sample is SUS304 stainless steel, which contains martensite phase caused by cold working of 20 %. The thickness is 100 μ m of similar size to the grain. After outgassing of hydrogen in the sample (300 °C: 48 hours), temperatures of the sample are set at 200 $^{\circ}$ C or 300 $^{\circ}$ C and then deuterium gas are supplied from backside of the sample. Permeated hydrogen to the other side is observed by ESD method and hydrogen molecules released from the sample surface to vacuum are observed by the QMS. These experiments were carried out separately to prevent each interference.

The hydrogen concentration on the surface (\times) and the hydrogen (m/z=4) pressure in the vacuum (+) are shown as a function of time in Fig.2 and 3, which are the cases of 200 °C and 300 °C, respectively. The intensities of ESD ions and the hydrogen pressure are normalized to compare each increasing tendency. At these temperatures the surface concentration of hydrogen increases immediately after hydrogen exposure. The fast permeation is due to primarily diffusion through martensite phase. However hydrogen outgassing to vacuum causes delay because of second order desorption which contains recombination process of hydrogen atoms. The delay times become shorter an order of magnitude from about 10000 s to about 1000 s as the sample temperature is raised from 250 °C to 300 °C because of increased diffusivity and probably activated surface diffusion. Thereafter desorption rate to vacuum rises with increasing of hydrogen concentration on the surface. At the case of 300 °C, though the surface concentration increases linearly, the desorption rate tends to saturate between 1 and 3 hours. Such result may indicate contribution of a process that competes with recombination of surface atoms.



Fig. 2: Time dependence of concentration (×) and hydrogen pressure (+) at 200 $^{\circ}\mathrm{C}$



Fig. 3: Time dependence of concentration (×) and hydrogen pressure (+) at 300 $^{\circ}\mathrm{C}$