§29. Measurement of Hydrogen Permeation of Stainless Steel by ESD Method

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Hydrogen behavior in metals is an important problem in order to understand mechanisms of hydrogen embrittlement, storage and permeation. In also fusion device, where hydrogen is used as fuel, it is necessary to make clear this problem. We have sequentially visualized the spatial distributions of permeated hydrogen on the stainless steel membrane using ions emitted by electron stimulated desorption(ESD) with the scanning electron microscope(SEM). This method can not only make clear the diffusion pass of hydrogen by comparing with the surface grain structure, but also obtain physical information on permeation by conjunction with a measurement on time dependence of hydrogen pressure in the vacuum. Hydrogen permeation contains the processes of solution, diffusion, adsorption and desorption. The information on these processes can be obtained from time dependence of hydrogen concentration on the surface and hydrogen pressure in the vacuum. In the present study an isotope effect for hydrogen permeation was investigated by these two kind of experiment using H_2 and D_2.

The experimental setup, which equips the sample holder with hydrogen supply system, the ESD ion detector and the quadrupole mass analyzer (QMS) in the SEM, is shown in Fig.1. The electrode for collecting ESD ions to the ion detector is attached at the sample holder. The QMS is used for measuring the pressure of permeated hydrogen. The used sample is SUS304 stainless steel, which contains martensite phase caused by cold working of 20 %. The grain size of austenite phase is about 100μm. The thickness of membrane is 200μm. After outgassing of hydrogen in the sample (573K:48hours) the following experiments were performed at the same temperature. The back side of SUS membrane was exposed to hydrogen (2.7×10^5Pa) and the permeated hydrogen on the opposite observation side is observed by ESD method. In addition, the desorbed hydrogen in the vacuum was measured by the QMS. The ESD ion image and hydrogen pressure were measured sequentially immediately just after the hydrogen exposure at different temperatures. These experiments were carried out separately in order to prevent each interference. The vacuum chamber was evacuated by the sputter ion pump (100ℓ/s) under the experiments.

Fig.2 and 3 are the time dependences of hydrogen pressure and the number of ESD ion at 573 K, respectively. The isotope effect is not obvious in the hydrogen pressure curve (Fig.2). But each ESD ion curve for H_2 and D_2 is distinguished from one another at even initial stage to about 7 hours. The isotope effect is obviously demonstrated in the time dependence of ESD ion (Fig.3), although the data is not corrected for each escape factor of ESD H^+ and D^+ ions and an isotope effect for the pumping speed of ion pump. Permeation is a complex process containing solution, diffusion, adsorption and desorption. The results suggest that it is necessary to take into account an effect of each process separately.

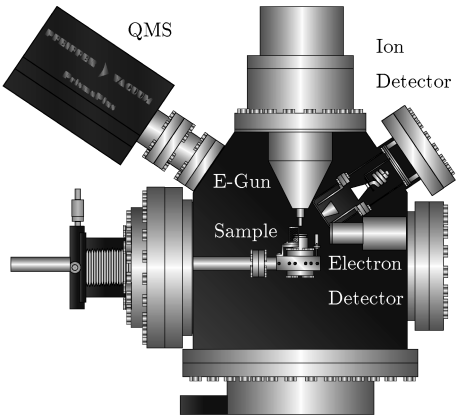

Fig. 1: Experimental setip.

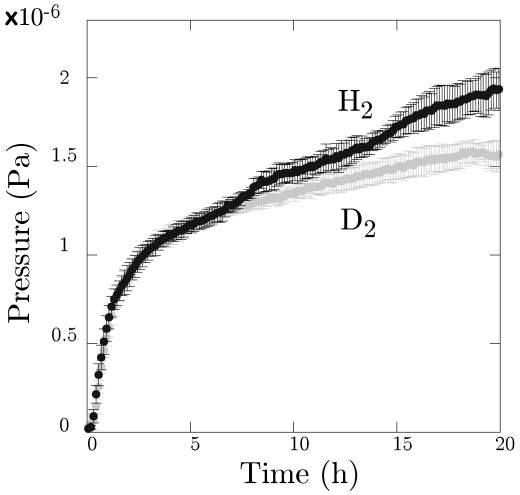

Fig. 2: The time dependence of hydrogen pressure.

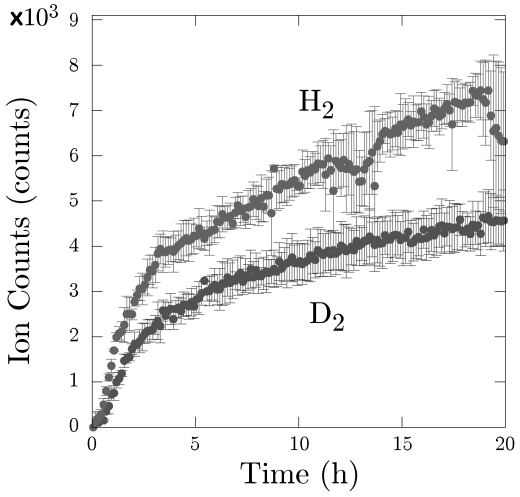

Fig. 3: The time dependence of ESD ions.