

§39. Measurement of Hydrogen Permeation of Stainless Steel by Electron Stimulated Desorption (ESD) Method

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Hydrogen diffusion in metals has attracted attention in hydrogen embrittlement, hydrogen storage and materials for fusion reactor or ultrahigh vacuum chamber. However, it is difficult to observe directly hydrogen diffusion in metals. The following methods have been reported. One is observations of hydrogen mapping using microprint technique or fracture cross section after tensile test with a scanning electron microscope (SEM) after cathodic charging of hydrogen to specimens. Another is nuclear reaction method for analyzing depth profiling. However, these methods are not available to an observation in real time. In our experiment the spatial distributions of hydrogen on the specimen are visualized sequentially by protons emitted by electron stimulated desorption process with the SEM. This method can not only make clear the diffusion pass of hydrogen by comparing to surface grain structure, but also obtain physical information on diffusion by acquiring the images sequentially in different specimen temperature.

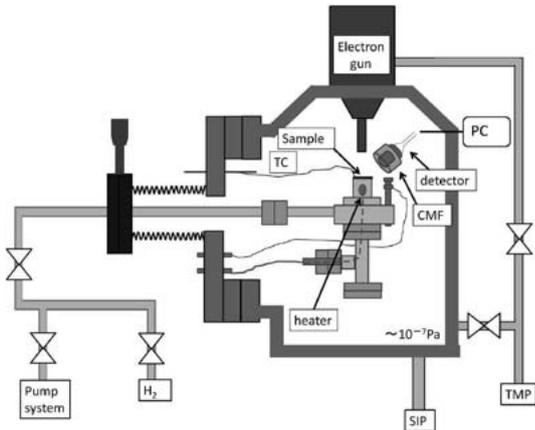


Fig. 1: Experimental setup.

The specimens used in this experiment are two kinds of SUS304, whose thicknesses are 100 (S1) and 200 μm (S2) and each cold working rate is 20 (S1) and 10% (S2), respectively. Therefore, these specimens are characterized by dual phase structure, where a part of austenite phase transfers to martensite. The austenite grain sizes are about 100 μm , which is comparable to the thickness of S1. The schematic diagram of experimental setup is shown in Fig.1. The hydrogen reservoir attached specimen is installed in the sample chamber of the SEM. Hydrogen is supplied to the reservoir up to 5×10^{-4} Pa after evacuation. The specimen is exposed from the back side and the permeated hydrogen to the opposite observation side is observed by desorbed ion images in real time. The specimen is heated in incremental

steps from R.T. to 573 K by the halogen lamp. The specimen temperature is measured by T thermocouple. The residual gas in the sample chamber is analyzed by the quadrupole mass analyzer during the experiment.

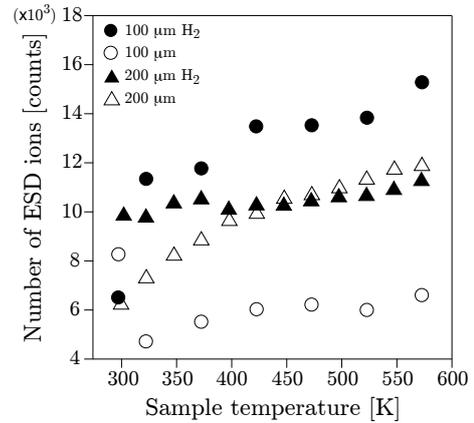


Fig. 2: Dependence of ESD ions on temperature.

Fig.2 shows the number of detected ESD ions at each temperature for specimens of S1 and S2, which are marked by circles and triangles, respectively. Solid marks represent the result with hydrogen gas, and open marks without gas. In S1 the number of ions increases stepwise at 323 and 423 K. Each increase corresponds to contribution of the diffusion through martensite and austenite phases, respectively. The increase at 573 K may be due to unknown diffusion path. S2 is different from S1 in ion yield behavior for heating. In no hydrogen supply the ion yield is more than S1 and in hydrogen the yield is almost constant for heating and less than S1. These features are due to the followings, that is, in S2 residual hydrogen contributes significantly to yield of ESD ions and permeation of hydrogen is suppressed. The difference between two specimens is whether there exists grain boundary between both sides throughout the specimen. The grain boundary suppresses hydrogen diffusion.

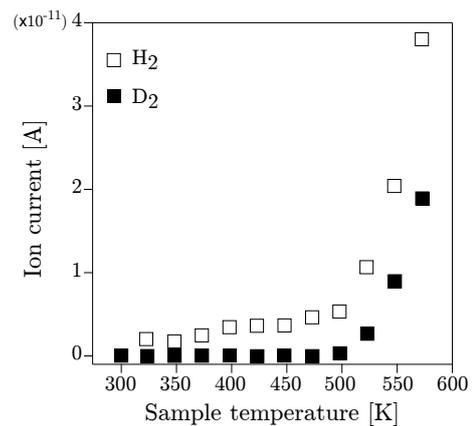


Fig. 3: Dependence of partial pressure on temperature.

Fig.3 is response of hydrogen partial pressure in the sample chamber of the SEM for temperature elevation, solid circles and triangles being the results for H₂ and D₂, respectively. The release to the vacuum occurs from about same temperature for both hydrogens but the amount of D₂ is about half of H₂.