§43. Preparation for Hydrogen Permeation Measurement through Ceramic Disc Samples

Iida, T., Sato, F. (Osaka Univ.),
Tanaka, T., Muroga, T.

Development of ceramic coatings has been conducted for suppression of hydrogen isotope permeation through high temperature coolant tubes in a reactor. In the present study, examination of hydrogen permeation behaviors through ceramic coatings, mono- and poly-crystalline ceramic discs, etc. are being performed for acquisition of fundamental data and understanding of permeation mechanisms.

For measurements of hydrogen permeation thorough ceramic disc specimens, a similar method to that used in our previous measurements for metal disc and ceramic coated metal disc specimens [1, 2] is used (Fig. 1). A disc shaped specimen with the thickness of 0.3-1.0 mm and diameter of ~20 mm is placed between two vacuum pipes. Hydrogen at 100-200kPa is introduced to one of the pipes and hydrogen permeated through the disc specimen is detected in another tube by using a quadrupole mass spectrometer (QMS). While vacuum pressures in the pipes must be $<10^{-4}$ Pa to detect permeation through disc specimens, it has been difficult to tighten metal gaskets between the pipes and specimen without breaking a fragile ceramic disc. After trying several methods, e.g. using adhesives, to suppress air leakage between the pipes and specimen, it was found that high vacuum is obtained by modifying our previous method using a gasket with a spring. It was confirmed that high vacuum could be kept for a 1 mm thick mono-crystalline sapphire disc and 0.39 mm thick mono-crystalline 3H-SiC plate.

In the fiscal year of 2012, hydrogen permeation through a 1 mm thick low activation ferritic steel (JLF-1) plate coated with a 0.5 μm thick Er_2O_3 coating has been measured. The coating layer was fabricated by the metal organic decomposition method (MOD).[1,2] Since the coating was baked in reduced atmosphere, thick Cr_2O_3 layer was also formed on the JLF-1 disc surface. The result of the measurement is shown in Fig. 2. Our previous measurement showed that the Er_2O_3 coating suppressed the hydrogen permeation through a JLF-1 disc by two orders [3], i.e. the permeation reduction factor (PRF) is 100-200, as plotted with closed triangles in Fig. 2. In the present measurement for the Er_2O_3 coated JLF-1 disc with a Cr_2O_3 layer (closed circle), hydrogen permeation could not detected under 650 ^{o}C due to the background gas. Limitation in the permeation measurement was estimated to ~5 x 10^{-14} mol/m/s/Pa$^{0.5}$. A Cr_2O_3 layer on the JLF-1 disc surface might improve the permeation barrier performance of the present coated specimen. The result indicates that the PRF was >1000 under 650 ^{o}C. Hydrogen permeation could be detected at 700 ^{o}C and the PFR was 850.[4]

Hydrogen permeation measurement for a 1 mm thick mono-crystalline sapphire disc has also been performed up to 650 ^{o}C. While high vacuum could be kept for the ceramic disc and the background level was similar to that shown in Fig. 1, permeation could not be detected.

Since hydrogen permeation through the ceramic coated JLF-1 specimen and ceramic disc specimen was under the background level, measurements using deuterium gas are planned at Osaka Univ. By using ceramic disc specimens, it is expected to examine hydrogen permeation behaviors in ceramic materials without influence of pores and cracks which have been observed in ceramic coating layers on JLF-1 discs. Examinations of influence of electrical field on permeation behaviors etc. are also planned in the study.

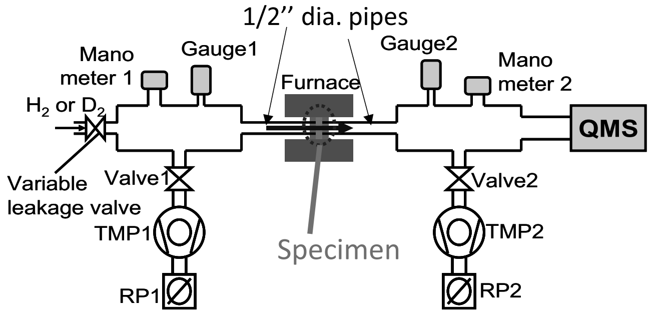

Fig. 1. Schematic drawing of hydrogen permeation measurement system.[1]

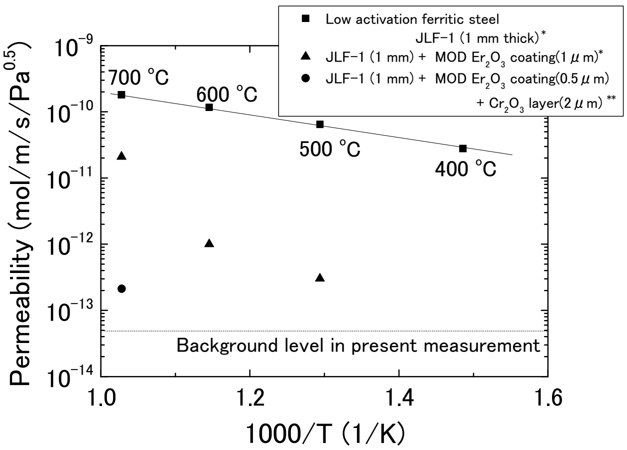

Fig. 2. Results of hydrogen permeation measurements for a low activation ferritic steel JLF-1 disc and Er_2O_3 coated JLF-1 discs (*: Ref. 3, **: Ref. 4). Permeation through 1mm thick mono-crystalline sapphire could not be detected at 600 and 650 ^{o}C due to the background.

1) Zhang, D.: Doctoral thesis, the graduate university for advanced studies, 2011.
2) Zhang, D. et al.: J. Nucl. Mater. **417** (2011) 1249–1252.
3) Zhang, D. et al.: Fus. Sci. Tech. **60** (2011) 1576-1579.
4) Tanaka. T. et al. : Fusion Engineering and Design, in press.