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The effect of pre-processing with various holding times on hydrogen permeation experiments

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1. Introduction

Permeation of hydrogen isotopes inevitably occurs due to high-temperature operation in a fusion power reactor. Many researchers have already studied the permeation phenomenon in structural materials [1,2]. The permeation test is very sensitively affected by experimental procedures and environmental conditions. It is often the case that differences in the measurements are seen among the studies. So, it is critical to properly evaluate permeation properties. In particular, it is well known that the oxidization layer or existence of hydroxyl on the samples impacts the permeability and the pre-processing is required to remove them.

In this work, a surface pre-processing condition is introduced using 316L stainless steel samples to establish the standard procedure for proper permeation experiments at HYPER.

2. Experimental description

A series of permeation experiments have been conducted by pressure rise method and Fick's law with single layer permeation [3]. HYPER (HYdrogen PERmeation experimental facility) is a permeation apparatus to investigate permeation properties of metals for fusion reactor in the Korea Institute of Fusion Energy (KFE). This is especially aimed to investigate permeation properties of Advanced Reduced-Activation Alloy (ARAA) that is under development [4]. Schematic diagram of HYPER is shown in figure 1.

The pre-processing is to remove oxidation layer on samples before permeation test. A simple pre-processing was adopted that the both sides of surface were exposed to hydrogen with 100 kPa for several hours at 500°C. To find a proper duration, various times from 0 to 12 hours were considered and applied. After the pre-processing was achieved, both sides of vessel were evacuated to ~10^{-6} Pa by each turbo molecular pump (TMP). And then, permeation experiments were conducted continuously as follows. Feed side was filled with hydrogen up to 100 kPa, and permeation side was set to high vacuum to ~10^{-6} Pa as the minimum pressure in the system using a TMP. Samples were prepared with diameter of 19.8 mm and thickness of 0.7 mm from commercial austenitic stainless steel 316L. Temperature was maintained at 500°C during experiments. Pressure rise data were recorded by two Capacitance Diaphragm Gauges (CDG, 0.1&10) for cross-check.

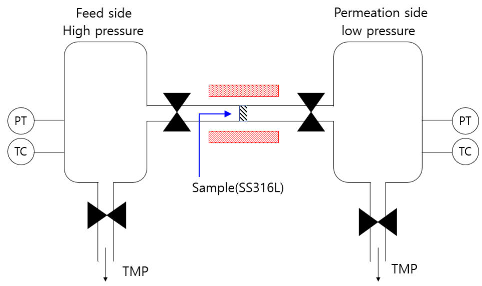


Figure 1. Schematic diagram of HYPER

3. Results

Permeabilities were calculated from the slope in steady state region from pressure rise data. Figure 2 shows pressure rise data with various holding times, there are significantly differences among 0 hour with no pre-processing and other results. It is noted that the bare metal without the pre-processing could contain an error on permeation value. Calculated results of each slope and holding time are represented in Figure 3(a) and 3(b). The slopes, indicating permeation rates, increase as the holding time increase up to 3 hours and then they are saturated. It is recommended that the pre-processing duration is required approximately 3 hours. On the other hand, the time lag which is defined as a x-intercept, decreases according to increasing holding time. The discrepancy of the time lags at each holding time tends to become small, which strongly suggest that the pre-processing more than 2 hours is necessary to remove oxidized layer on the samples and to obtain consistent permeation data.

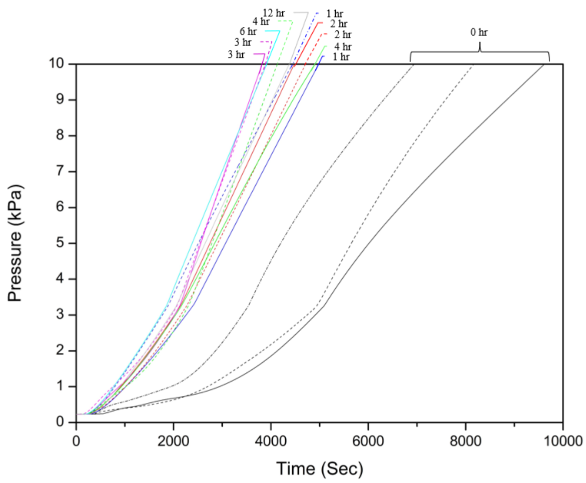
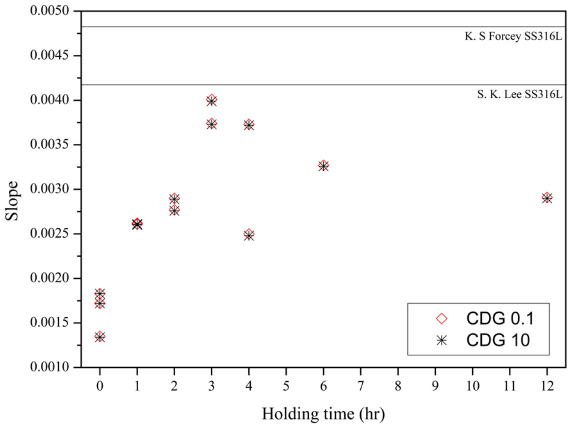
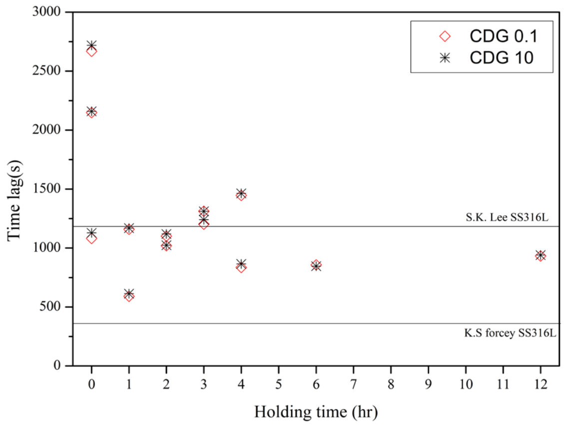


Figure 2. Pressure rise data with various holding times



(a) Slopes with various holding times



(b) Time lags with various holding times

Figure. 3 Slope and time lag data with various holding times and comparison with previous studies [2,3]

4. Conclusions

In this study, the pre-processing was tested to improve consistency of permeation results on SS316L samples. The experiments show that a sufficient holding time is necessary to reduce discrepancy in results, and it was decided to apply 3 hours for a holding time as the pre-processing for future experiments. This pre-processing will be tested on ARAA to find proper holding time for further works. Also, it is expected to contribute on repeatability and reliability of permeation properties on the HYPER.

Acknowledgments

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