Stainless Steel Permeability

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Abstract

An understanding of the behavior of hydrogen isotopes in materials is critical to predicting tritium transport in structural metals (at high pressure), estimating tritium losses during production (fission environment), and predicting in-vessel inventory for future fusion devices (plasma driven permeation). Current models often assume equilibrium diffusivity and solubility for a class of materials (e.g. stainless steels or aluminum alloys), neglecting trapping effects or, at best, considering a single population of trapping sites. Permeation and trapping studies of the particular castings and forgings enable greater confidence and reduced margins in the models. For FY15, we have continued our investigation of the role of ferrite in permeation for steels of interest to GTS, through measurements of the duplex steel 2507. We also initiated an investigation of the permeability in work hardened materials, to follow up on earlier observations of unusual permeability in a particular region of 304L forgings. Samples were prepared and characterized for ferrite content and coated with palladium to prevent oxidation. Issues with the poor reproducibility of measurements at low permeability were overcome, although the techniques in use are tedious. Funding through TPBAR and GTS were secured for a research grade quadrupole mass spectrometer (QMS) and replacement turbo pumps, which should improve the fidelity and throughput of measurements in FY16.

Introduction

Gas transfer systems utilize tritium pressure vessels that are required to operate over long periods with high reliability. Permeation of the tritium and subsequent trapping within material structures occurs, which can lead to increased potential for fracture (hydrogen embrittlement). To address this phenomena, fundamental data on hydrogen isotope permeation and trapping are needed to improve the fidelity of hydrogen assisted fracture models currently in development.

The goals of these studies are to measure the permeation and trapping of materials of interest to GTS. There is a synergistic benefit from similar needs with the fusion program, where data on low activation steels, permeation barrier materials, and plasma facing materials are needed for the design of future fusion power plants. For example, we have measured the permeation of deuterium through SiC, one of the best permeation barrier materials under consideration for fusion blankets.

Approach

Through partnerships with the magnetic fusion energy program, GTS, and TPBAR, we have developed synergistic capabilities for measuring pressure-driven deuterium permeation (upstream pressure < 1.2 atm, temperature < 1150 °C) and plasma driven tritium permeation (upstream ion flux < 2.5×10^{22} D^+ $m^{-2}s^{-1}$, temperature < 1000 °C). The permeation measurements described here were performed in a lower temperature system capable of operating up to 600 °C. The upstream side of the samples must be coated with palladium to inhibit oxidation of the surface during test (due to reduction of oxides in the heated zone). Trapping can be studied using a Thermal Desorption System (TDS) with varying thermal ramps. The diffusion and trapping are modeled using various diffusion codes (DIFFUSE, TMAP) and provide input into continuum models of tritium behavior for GTS. Funding for FY15 provided for the investigation of poor reproducibility in the quadrupole mass spectrometer (QMS) used for the measurements and for several permeation experiments, but not for trapping or modeling studies.

Results and Impacts

Near the end of FY14, we began noticing unusual responses for calibration runs and permeation experiments for low D_2 flows as measured by our SRS QMS. Operating in PvsT mode, partial pressure variations were observed for mass 4 (D_2), which is the permeating gas used in the experiments (see Figure 1a). There were also inconsistencies that seemed to depend on the number of channels being monitored. Through exhaustive discussions with SRS and long term tests, we decided to send the unit in for repair. Upon return, similar behavior continued, including sudden large changes in signal level. Through exhaustive tests in analog scan mode (i.e., performing a full mass scan instead of tracking selected peak values), we determined that the zero level was not being adequately set for the PvsT mode. This behavior was repeated by borrowing a second SRS QMS, although a slightly different model.

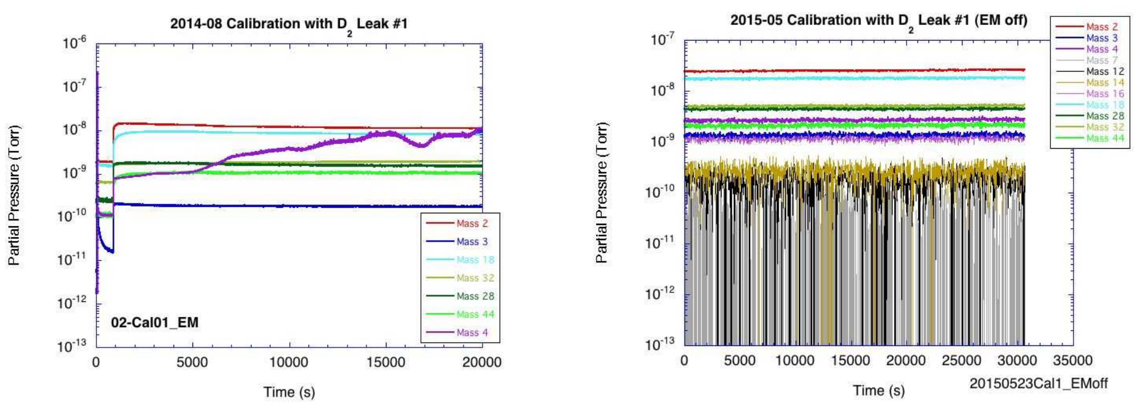

Fig. 1. (a) Behavior of several mass peaks during a calibration run. The D_2 gas is turned on at ~ 1000 s, however, it shows a non-monotonic rise and the variations appear uncorrelated with other masses. (b) Similar masses monitored in analog scan mode.

After much discussion with SRS and observations made in analog scan mode, we decided to operate using this more detailed, although slower mode. This sacrifices time resolution that would be needed to measure transients (useful for diffusion measurements), along with increasing the complexity for reducing the data 10x, however, it proved much more reliable in operation over the weeks needed for a permeation run (see Figure 1b). The system was calibrated in May and this allowed permeation experiments to restart. A new research grade MKS QMS has been ordered, which should allow us to return to the faster data collection mode, and easier analysis of the data. This type of QMS is presently in use on our high temperature permeation cell, which has been in nearly constant use for fusion samples in FY15.

For the first measurements, we decided to return to our investigation of duplex steels, which we had begun in FY14. In earlier studies looking at the role of ferrite in austenitic steels, we found that small amounts of ferrite in 21-6-9 did not increase hydrogen isotope permeation, while weld material did show a modest increase. Figure 1 shows a comparison of hydrogen equivalent permeation for 21-6-9 stainless steel and 21-6-9 weld material. The open squares (weld material) appear systematically above the other base metal values, although the higher levels are well below that for ferritic steel (purple points) and the activation energy is closer to austenitic than ferritic steel. Ferritic steels typically show higher permeation when compared with austenitic steels (typically 1-2 orders of magnitude).

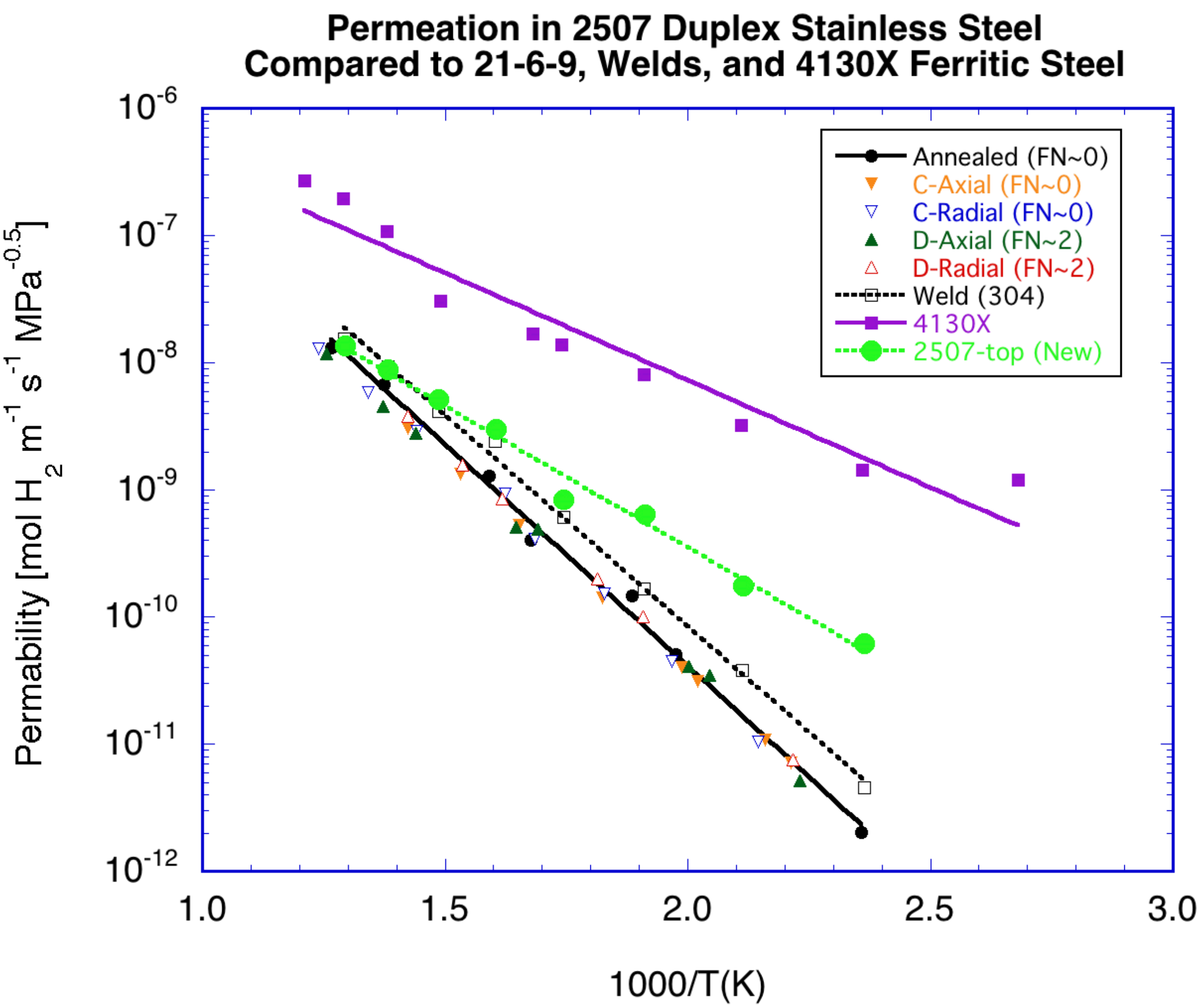

Fig. 2. Hydrogen gas equivalent permeability for 21-6-9 stainless steel (various orientations) compared with 21-6-9 weld material and a 4130X ferritic steel. Also shown in green are new measurements for 2507 stainless steel, with the membrane oriented perpendicular to the ferritic domains.

Also shown in figure 2 are new results for a 2507 duplex steel. In contrast to our earlier measurements which exhibited hysteresis, the points fall nearly along a line in this log-linear plot (as is typical). For this sample, the ferrite domains are elongated through the permeation membrane and the composition of the material is about 50% austenitic / 50% ferrite. Despite the high percentage of ferrite, the permeability of 2507 is closer to austenitic steels, with a divergence increasing as the temperature is lowered. By comparing ferrite content measurements made before and after the permeation experiment, we confirmed that no appreciable conversion to ferrite had occurred. We have additional samples prepared perpendicular to the ferrite domains that will be run in FY16 to continue exploring the role of ferrite in permeation.

Following the measurements of duplex steel made in June, we moved to initial measurements on work hardened material, which was the goal for the FY15 study. The motivation for this work stemmed from earlier measurements on 304L forgings that exhibited systematic deviations from austenitic steel permeation for one region of the forging (position C of forging 11460). Figure 3 shows these permeability curves; the C position along with a replicate both indicate a deviation in permeability from that expected for austenitic steel. Both the level and activation energy showed a difference. Not shown is data from annealing of the C position sample, which recovered the permeability to that of the other curves.

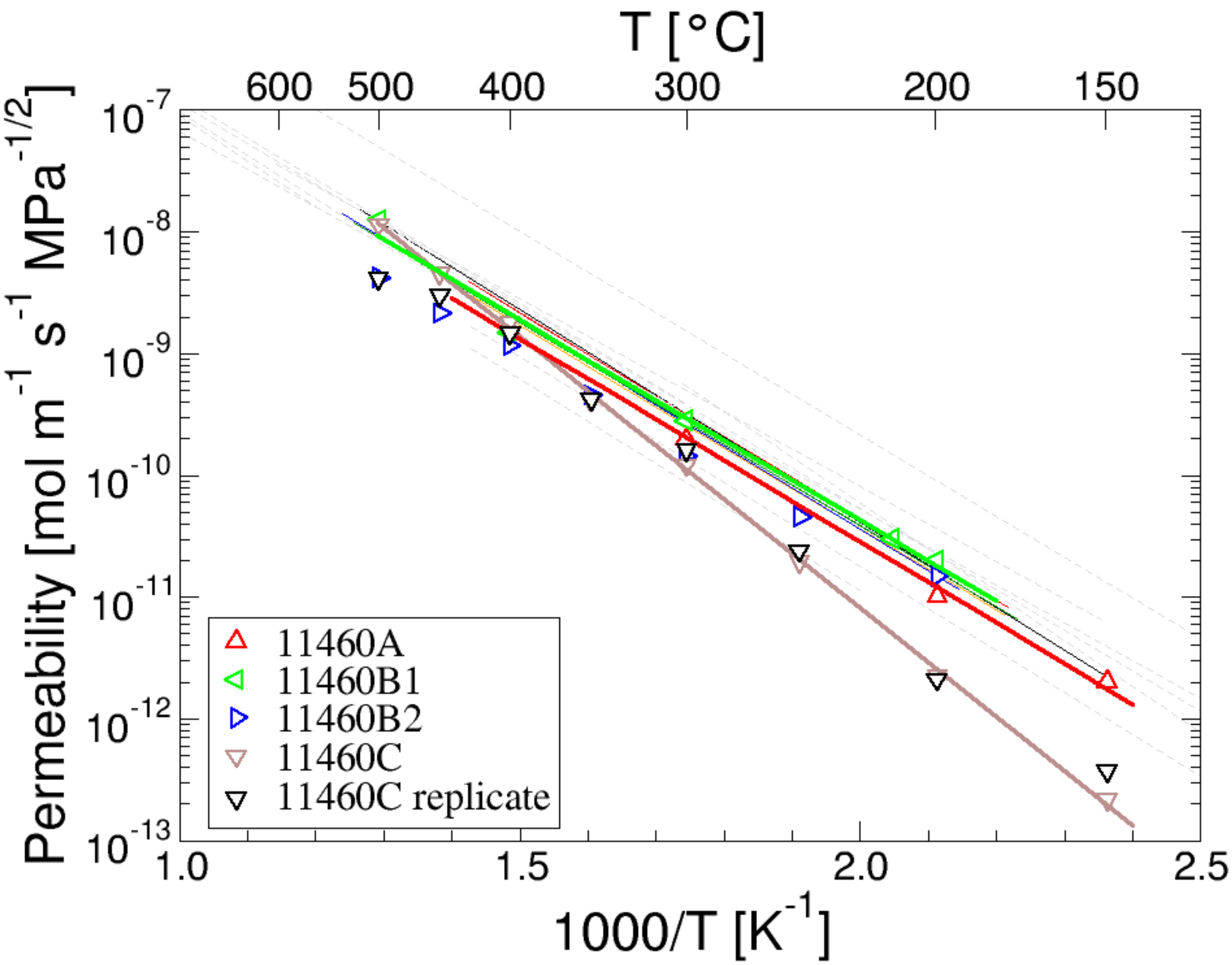

Fig. 3. Hydrogen gas equivalent permeability for 304L forgings. The curves are taken from various locations in the forgings. Position C showed somewhat higher work hardening compared with the other positions (two samples).

To investigate the work hardening hypothesis, highly work hardened 316L stainless steel was obtained from Swagelok, cut into permeation membranes, and coated with Pd to prevent oxidation. Figure 4 shows the first results obtained for this material, in comparison with austenitic and ferritic steels (same comparison data shown in figure 2). Unlike the deviation in permeability and activation energy observed with the C position of forging 11460, the permeability of the Swagelok sample appears to be very similar to that of 21-6-9 stainless steel. Additional samples have been prepared and we plan to repeat these measurements using the new MKS QMS in FY16.

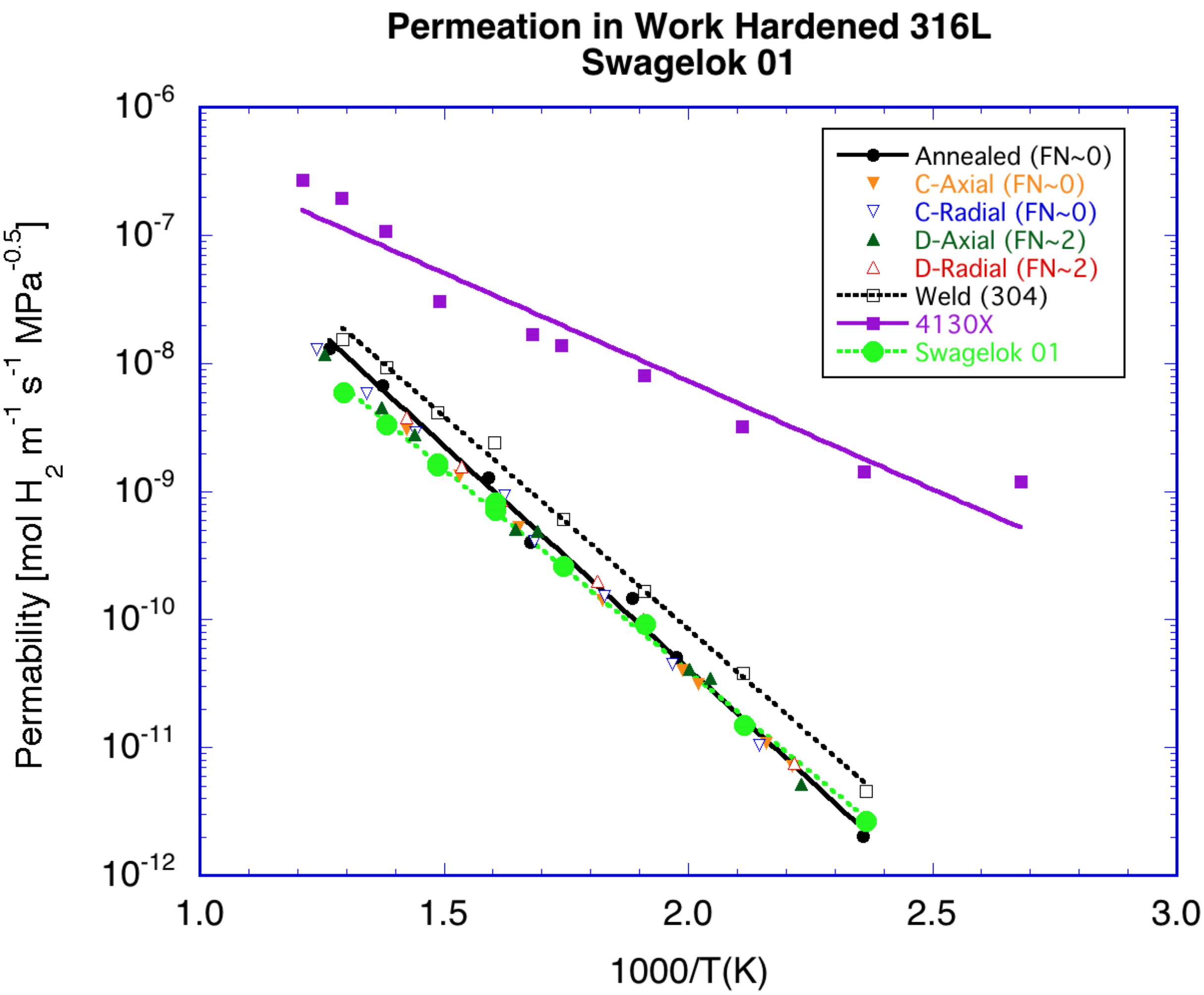

Fig. 4. Hydrogen gas equivalent permeability for 21-6-9 stainless steel (various orientations) compared with 21-6-9 weld material and a 4130X ferritic steel. Also shown in green are new measurements for a work hardened 316L steel from Swagelok.

Conclusions and Future Work

Due to challenges in FY15 associated with our QMS and a hold on purchases in ES/GTS programs, progress has been slow in obtaining reliable permeation data. We expect that the purchase of a new research grade QMS and backup pumps will provide fore more reliable operation in FY16. Samples for both the duplex and work hardened steel will be rerun using the new QMS, as a check on these measurements. Trapping studies on the Swagelok 316L steel can further elucidate if trapping played a role in the 11460C forging permeability.

Summary of Findings and Capabilities Related to Aging

As this was the first full year of work addressing permeation and trapping in GTS materials, further work will be needed to obtain the data needed for improving structural material model fidelity. We expect higher reliability of the instruments in FY16.

No findings relevant to specific component/material aging or capabilities were obtained this year.

References

None

Administrative Addendum

[List any related publications and presentations along with a final status of your FY15 deliverables from your work package agreement.]

- **Related Publications and Presentations:**

[Authors, Title, ……]

- **Milestone Status:**

[Listing and brief status for each of the milestones and final deliverable listed in your FY15 SubTask Work Package Agreement]