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Gas-driven permeation of deuterium through tungsten and tungsten alloys

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HIGHLIGHTS

- We have designed and performed initial studies on a high temperature gas-driven permeation cell capable of operating at temperatures up to 1150 °C and at pressures between 0.1–1 atm.
- Permeation measurements on ITER grade tungsten compare well with past studies by Frauenfelder and Zahkarov in the temperature range from 500 to 1000 °C.
- First permeation measurements on Ti dispersoid-strengthened ultra-fine grained tungsten show higher permeation at 500 °C, but very similar permeation with ITER tungsten at 1000 °C. Diffusion along grain boundaries may be playing a role for this type of material.

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ABSTRACT

To address the transport and trapping of hydrogen isotopes, several permeation experiments are being pursued at both Sandia National Laboratories (deuterium gas-driven permeation) and Idaho National Laboratories (tritium gas- and plasma-driven tritium permeation). These experiments are in part a collaboration between the US and Japan to study the performance of tungsten at divertor relevant temperatures (PHENIX). Here we report on the development of a high temperature (≤1150 °C) gas-driven permeation cell and initial measurements of deuterium permeation in several types of tungsten: high purity tungsten foil, ITER-grade tungsten (grains oriented through the membrane), and dispersoid-strengthened ultra-fine grain (UFG) tungsten being developed in the US. Experiments were performed at 500–1000 °C and 0.1–1.0 atm D_2 pressure. Permeation through ITER-grade tungsten was similar to earlier W experiments by Frauenfelder (1968–69) and Zaharakov (1973). Data from the UFG alloy indicates marginally higher permeability (< 10×) at lower temperatures, but the permeability converges to that of the ITER tungsten at 1000 °C. The permeation cell uses only ceramic and graphite materials in the hot zone to reduce the possibility for oxidation of the sample membrane. Sealing pressure is applied externally, thereby allowing for elevation of the temperature for brittle membranes above the ductile-to-brittle transition temperature.

1. Introduction

Of particular importance in the design of fusion reactors is the production and mass transport of tritium within blanket, first wall, and divertor structures. Permeation of tritium through plasma facing components (PFCs) can result in excessive in-vessel inventories and possible contamination of the coolant, which can exceed regulatory limits and pose unacceptable safety risks. This concern with in-vessel inventory and predictions for the trapping of tritium in carbon and carbon films has motivated a change to a full tungsten divertor for ITER [1]. The higher temperatures of plasma-facing structures and deep traps caused by neutron interactions, which can occur in future devices, will only increase these concerns.

For the ITER divertor and future fusion devices, tungsten and tungsten alloys are under consideration as a plasma-facing material. Measurements of the solubility and diffusivity of hydrogen in

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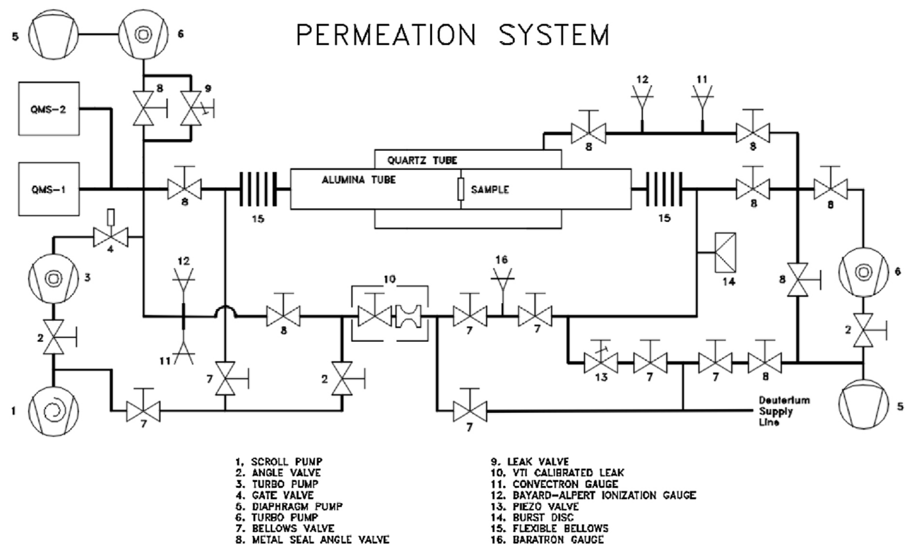

Fig. 1. Schematic layout of the high temperature permeation system used in this study. D_2 gas is introduced from the right side of the sample and the permeating flux is measured by QMS units on the downstream side (left side) of the sample. The alumina tube that contains the sample and the graphoil gaskets that seal it in the hot zone reduce the possibility for oxidation of the sample membrane and the evacuated outer quartz tube prevent spurious D_2 leaking around the seals.

tungsten have been studied since the late 60′s (Frauenfelder: [2,3]) and early 70′s (Zaharakov [4]). Recent reviews describe hydrogen behavior in tungsten and measurements of both gas and ion-driven permeation [5–7]. Investigations of high-flux plasma-driven permeation using tritium [8] are also in progress. However, experiments that use deuterium gas-driven permeation can operate at relevant temperatures and provide useful comparisons of materials under development for plasma facing components. We describe here the design and initial results from a gas-driven permeation cell operating at temperatures up to 1150 °C. This system operates with gas pressures up to ∼1 atm, allowing for measurable permeating fluxes through both blanket and plasma facing materials. Support for the design and construction came from a Phase II Small Business Innovation Research (SBIR) program with Ultramet, funded by the US Department of Energy, and centered on measuring permeation in SiC inserts for the dual coolant lead lithium (DCLL) ITER test blanket design [9]. Measurements made on SiC fabricated by chemical vapor deposition (CVD) were made up to 1100 °C, confirming the very low permeability expected for this material ($<10^{-12}$ mol H_2 m^{-1} s^{-1} $MPa^{-0.5}$).

Subsequent measurements have been directed towards the behavior of tungsten materials at high temperatures that are of interest to the US-Japan Joint Project on Technological Assessment of Plasma Facing Components for DEMO Reactors (PHENIX), and other alloys under development in the US fusion program. The PHENIX project utilizes the HFIR reactor at ORNL for the purpose of examining the thermophysical properties and tritium behavior of neutron irradiated tungsten and tungsten alloys. Further studies to investigate the effects of neutron irradiation on permeation in tungsten materials will be carried out on a tritium gas-driven permeation cell under development at the Idaho National Laboratory [10] in collaboration with Japan. A lower temperature cell (≤900 °C) at Shizuoka University for deuterium gas-permeation is also being used for ion damaged tungsten studies [11].

Section 2 below provides a description of the design and operation of the new Sandia permeation cell, along with data from commercial tungsten foil. Sections 3 and 4 present results from ITER grade tungsten and ultra-fine grained (UFG) tungsten while a comparison of the results and post permeation surface analysis are discussed in Section 5. Section 6 concludes the discussion with a brief summary.

2. Design and operation

The need to measure low fluxes expected for permeation barrier materials was a major consideration in the design of the high temperature cell described here. This cell is to be contrasted with a separate permeation cell at Sandia which operates with metal components and is limited in temperature to ∼500 °C. The lower temperature cell is used for studying permeation in steel and coating of the samples with palladium is required to prevent oxidation. Details of the high temperature cell were based on a design developed at ENEA UTS MAT (Ente per le nuove tecnologie, l'energia e l'ambiente, Rome, Italy [12]) and included the use of ceramic materials within and sealing force application outside of the hot zone. In order to seal to brittle materials (SiC, ITER W), gaskets punched from a Grafoil sheet (760 μm thickness) were used as a soft seal on both sides of the permeation membrane under test. The low permeability of the alumina tubing [13] and thickness of the Grafoil seals (2 × 3.2 mm annular), along with an outer vacuum surrounding the membrane and seals provided for low background D_2 pressures from leaks or permeation through hot zone components. The outer vacuum pressure did not exceed 3×10^{-3} Pa, even at 1000 °C. The effective area of the sample is 2.85 cm^2.

Fig. 1 shows a schematic layout of the major components of the permeation system. The sample is centered within two alumina tubes while a secondary vacuum quartz tube surrounds the sample and seals. Deuterium mass flow is from right to left in the figure; upstream pressure is regulated by a piezoelectric valve with feedback from a temperature stabilized Baratron (MKS model 627D, 10000 Torr, 0.12% accuracy). This Baratron is also used during calibration at pressures up to 8000 Torr to determine the flow through an absolutely calibrated capillary leak (VTI model PSO, rated for 10^{-7} atm-cc/s for gas input of 10340 Torr D_2). The analysis volume downstream is pumped by an oil free turbomolecular pump, with auxiliary pumping to aid in outgassing of the system. Metal valves were used where possible to further limit residual gasses. The downstream flow is measured by 2 MKS MicroVision Plus quadrupole mass spectrometers (QMS). The primary QMS is high sensitivity and has mass resolution to distinguish He from D_2 (limited mass range). Calibration is done for 3 settings of the electron multiplier and also for a Faraday cup. The secondary QMS monitors