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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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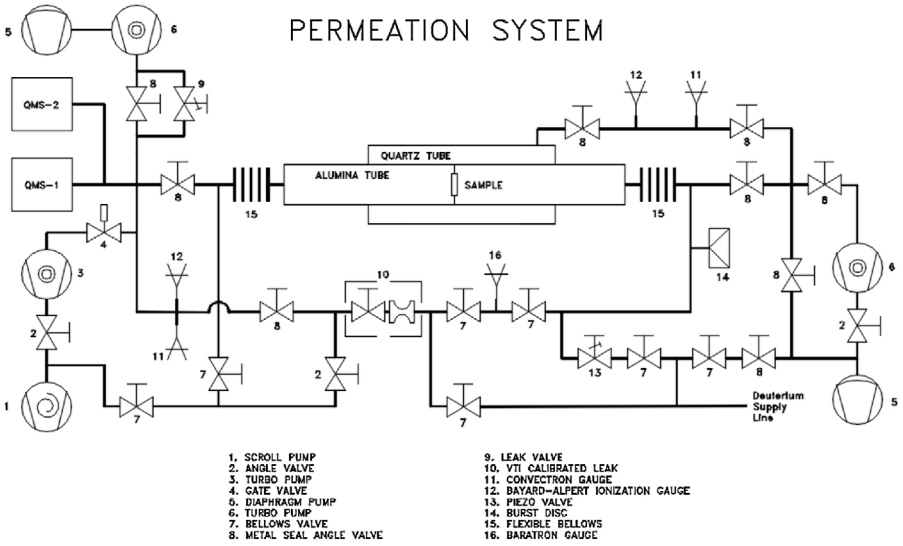

Fig. 1. Schematic layout of the high temperature permeation system used in this study. D_2 gas is introduced from the right side of the sample and the permeating flux is measured by QMS units on the downstream side (left side) of the sample. The alumina tube that contains the sample and the graphoil 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

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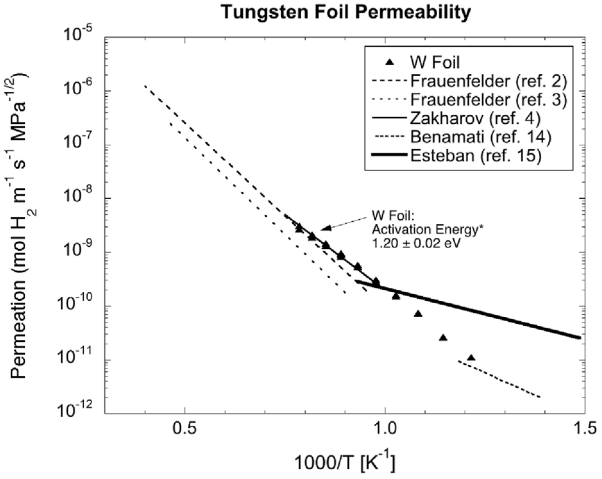

Fig. 2. Permeation data obtained in the cell for commercially available tungsten foil compared to several previous studies. *The activation energy is calculated with data below 850 °C.

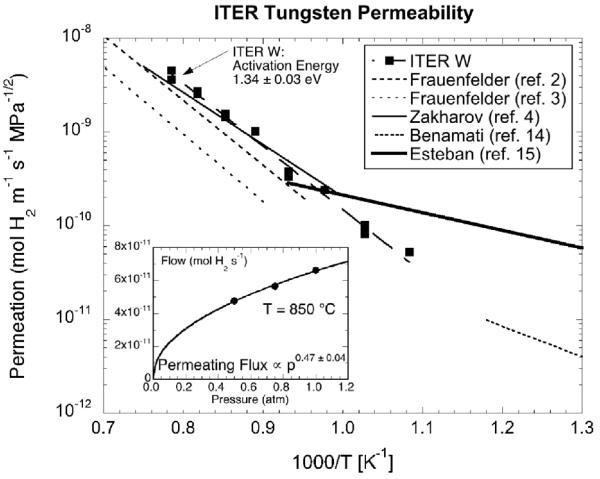

Fig. 3. Permeation data obtained for ITER-grade tungsten, for membrane temperatures up to 1000 °C. The inset plot shows the pressure dependence of the permeating flux at 850 °C, indicating diffusion limited permeation.

the mass range from 1 to 100 AMU. Ion gauges also monitor the downstream and outer vacuum volumes.

During operation, the hot zone is heated by a Lindberg Blue Furnace (<1200 °C, ±1 °C) with ramp rates to steady state of 1 °C/min. The sample temperature was initially measured using a pressure mounted TC on the upstream side of the sample, however, the temperature of the 30 cm long hot zone correlated well with the sample temperature, negating the need for a separate thermocouple. The sealing force is adjusted externally for each measurement due to thermal expansion of materials in the hot zone. Typical experiments with measurements taken at 50 °C intervals between 500 and 1000 °C take about 3 weeks. Measurements are taken at 100 °C intervals as the temperature is increased, and at the in-between temperatures as the temperature is decreased. This provides a check that microstructural changes did not occur that could affect the permeability. Leaks around the seals are checked by examining the downstream pressure when gas is applied, and also by increasing the outer vacuum pressure to about 10 Pa of deuterium.

Fig. 2 shows example data obtained in the permeation cell for commercial tungsten foil (Johnson Matthey, 0.1 mm thick, 99.95% W) and compared with fits to experimental data from the literature [2–4,14,15]. As is customary, permeability of H_2 is indicated and a square root of mass dependence on permeation is assumed. For the range of temperature pertinent to ITER (500–1000 °C, or 0.8–1.3 on the x-axis), a smooth trend comparable to the literature values is obtained. It should be noted that this foil is much thinner than typical test membranes (10× thinner), so some extrapolation beyond the range of calibrated flows was needed (above 800 °C, or below 0.93 on the x-axis). The activation energy is calculated with data below 850 °C.

3. ITER tungsten

The tungsten foil used in Fig. 2 had been fabricated by rolling, which provides a grain structure with elongation in the plane of the membrane. The specification for ITER tungsten, however, is to increase the thermal conductivity in the direction away from the plasma facing surface [16]. Hence, the grain structure for this material is elongated perpendicular to the surface that serves to face the plasma (and incoming tritium). The direction of interest for tritium transport is there therefore along these grains. As a result, sample membranes were prepared with the high density of grain boundaries perpendicular to the surface. This greatly reduced the strength of the material, which led to several failures by membrane fracture when sealing pressure was applied.

To address this, a procedure was adopted in which a modest sealing pressure was used below 500 °C. This prevented large stresses in the membrane until the temperature was raised above the ductile to brittle transition temperature (DBTT: ~300 °C for W). The permeation results for temperatures above 500 °C using a 1 mm thick ITER-grade membrane are shown in Fig. 3. Also shown in the figure are fits to data from the literature. The vertical scatter in the measurements is due to uncertainties in the QMS calibration, likely a result from the high downstream pressures that occurred during numerous membrane failures on prior samples (8 in total). Nevertheless, there is good agreement with both Zakharov's and Frauenfelder's measurement in this temperature range. An activation energy of 1.34 ± 0.03 eV is determined from a fit to this data set and is close to that determined by Frauenfelder (~1.4 eV).

In Fig. 3, an inset figure indicating the pressure dependence of the permeating flux at 850 °C is also shown. Surface limited permeation scales linearly with pressure while diffusion limited permeation should show a square root pressure dependence. The power law fit in the figure ($p^{0.47 \pm 0.04}$) indicates that for these conditions, diffusion limited permeation is evident. More discussion regarding the surface conditions for this membrane is given in Section 5.

4. Ultra-fine grain tungsten

To develop tungsten alloys with greater strength, efforts have been underway in Japan [17] to introduce dispersoids into bulk tungsten. These particles (TiC, TaC) act as grain-boundary growth inhibiters and may also improve resistance to neutron damage by serving as sinks for displacement damage [18]. In the US fusion materials program, the behavior of hydrogen isotopes in such alloys is only now beginning to be studied [19]. Here we will examine the first permeation measurements made on Ti dispersoid-strengthened ultra-fine grained (UFG) tungsten being developed at the University of Utah.

The UFG tungsten is prepared by using high-energy planetary ball milling to generate fine tungsten powder (10–30 nm diameter). This powder is compacted using a uni-axial press and then sintered at a relatively low temperature under a hydrogen atmosphere. The material is then further consolidated by using a rapid heating omni-directional compaction process. Ti is incorporated as a dispersoid at 1% by weight and exists primarily on the grain

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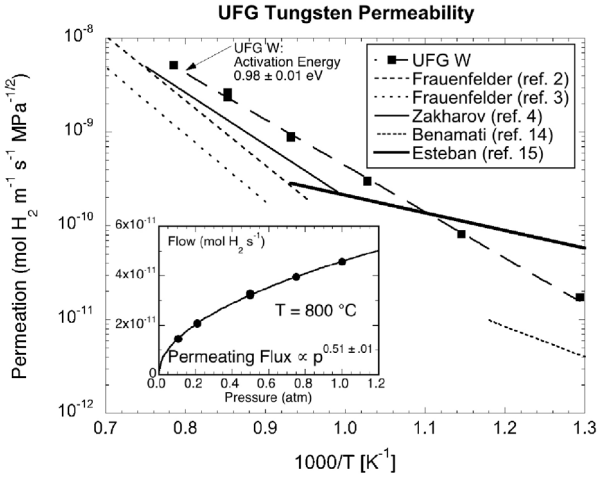

Fig. 4. Permeation data obtained for UFG tungsten, for membrane temperatures up to 1000 °C. The inset plot shows the pressure dependence of the permeating flux at 800 °C, indicating diffusion limited permeation.

boundaries. Auger Electron Spectroscopy has been used to confirm that the Ti is not incorporated within the tungsten, but exists with oxygen as a dispersoid (Auger spectroscopy actually indicates a higher level of oxygen in the dispersoids than Ti) [19]. The grains range between 0.2–2 μm, which is small compared with ITER grade tungsten (10–20 μm in the longest direction).

Fig. 4 shows permeability measurements for a 1.25 mm thick UFG tungsten membrane along with literature values as in the previous figures. A separate membrane run under the same conditions showed permeability nearly identical to these measurements. Note that although the UFG tungsten exhibits higher permeation than the ITER tungsten in Fig. 3, the activation energy is lower (0.98 vs. 1.34 eV), and the two permeation measurements made at 1000 °C have nearly the same value. As in Fig. 3, an insert plot in the lower left corner shows the variation of permeating flux with pressure, indicating a diffusion limited regime ($p^{0.51\pm0.01}$).

5. Discussion

For the data obtained in our permeation cell, we find consistently higher permeability for the case of the UFG tungsten when compared with ITER tungsten (or commercial pure tungsten foil). This also correlates with recent measurements of deuterium retention obtained in the PISCES linear plasma experiment. In that study [19], UFG tungsten exhibited about 3× higher retention when compared with ITER tungsten or rolled tungsten plate. Since the grain structure of the ITER tungsten is significantly different than the UFG material, enhanced diffusion and/or solubility along grain boundaries may be playing a more substantial role in the UFG material. Because there is a high concentration of oxygen within the dispersoids and because the volume fraction of these dispersoids is small, we do not expect large bulk solubility differences between this alloy and pure tungsten.

A study at lower temperatures (−7 to 60 °C) using electrochemical techniques found activation energies much lower than that of Frauenfelder or Zakharov (~0.4 eV as compared with 1.4 eV for Frauenfelder) [20]. The tungsten in this study was deposited by magnetron sputtering, so exhibited a high density of grain boundaries perpendicular to the surface. If the diffusion along grain boundaries is much faster than intergranular diffusion, then the density of grain boundaries can play a role in the UFG material. The activation energy found for UFG tungsten in Fig. 4 is 0.98 ± 0.01 eV, so in between the higher temperature values and the electrochemical study. Also note, in Fig. 3, the activation energy for the ITER tungsten is 1.34 ± 0.03 eV, so very close to Frauenfelder's value. A comparison of the activation energy determined by several studies is found in Table 1.

Table 1
Range of tungsten alloys, temperatures, and activation energies determined from several studies.

Composition	Temperature range (K)	Activation energy (eV)	Reference
W	970–1150	0.95	[22]
99.9% W	1600–2900	1.46	[23]
99.95% W	1050–2400	1.37	[2]
99.9 W	1100–2400	1.43	[3]
W	1000–1330	1.1	[4]
W	673–873		[14]
99.98% W	673–1073	0.38	[15]
99% W	266–333	0.46, 0.86, 0.38	[20]
99.95% W	823–1273	1.20	This study
ITER W	923–1273	1.34	This study
UFG W	773–1273	0.98	This study

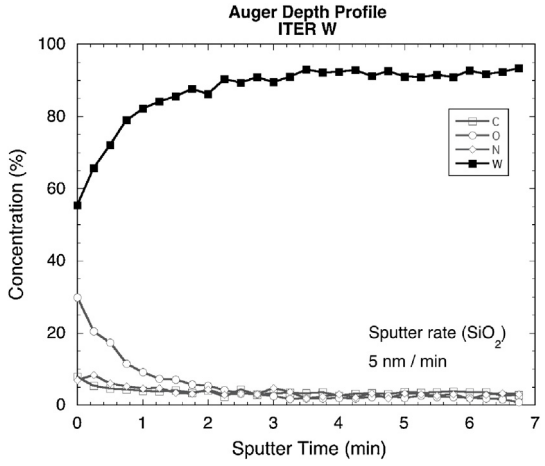

Fig. 5. Depth profile of elements found on the upstream surface of an ITER tungsten membrane.

Surface impurities may also play a role in gas-driven permeation measurements. In the case of the low temperature permeation cell at Sandia, transport of oxygen from oxides covering the metals in the hot zone have been shown to cause oxidation of the permeation membranes and strongly affect permeation. Fig. 5 shows a depth profile of elemental composition as determined by Auger Electron Spectroscopy (AES) for the upstream surface of an ITER tungsten membrane following permeation experiments in the cell. Several locations were profiled, due to a variability of the oxide measured on the surface. The area selected in Fig. 5 had the highest surface oxygen level (30%). Although the depth scale is only approximate, one can see that the surface oxide has a width of <5 nm, while carbon and nitrogen impurities quickly fall to the noise level within the tungsten membrane (several % in this case). This is despite the potential for carbon transport in the upstream region by deuterated methane production at high temperatures. It is likely that the use of alumina surrounding the Grafoil gasket and sample membrane plays a role in limiting carbon contamination, as methane is known to react with Al_2O_3 at elevated temperatures [21]. Of course, the pressure dependence shown in Figs. 3 and 4 also demonstrate that surface impurities are not playing a significant role in these permeation measurements for these samples.

6. Summary

In summary, we have developed a high temperature permeation cell capable of measurements up to 1150 °C, which uses soft seals

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as a way to handle brittle materials. By operating above the DBTT of tungsten, the permeability of commercial tungsten foil, ITER tungsten, and an UFG tungsten under development in the US have been measured in the temperature range between 500 and 1000 °C. The values for ITER tungsten are in good agreement with values from Frauenfelder and Zakharov, while those for the UFG exhibit higher permeability at 500 °C, but nearly the same values at 1000 °C. It is postulated that enhanced diffusion and/or solubility along grain boundaries is playing a more significant role in the UFG alloys, although at the operating temperatures needed for a reactor, the effect is minimal.

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