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Deuterium Permeation Through a Self-Supported Palladium-Silver Membrane in Helium Gas Mixtures

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Abstract—**Palladium membranes and membrane reactors can separate and purify tritium from impurities in the plasma exhaust processing system for the fusion energy fuel cycle. Membranes can also act as a continuous separation method to remove tritium from helium streams in the breeder blanket tritium extraction system, such as from the purge gas of solid breeders. To investigate the potential of these membranes for these applications, we performed a deuterium permeation campaign with a self-supported palladium-silver (Pd-25Ag wt%) tube of 0.15 m length, 76 µm wall thickness, and 3.0 mm inner diameter. A gas mixture of 3.95% D_2 and a balance of He was delivered to the inside of the tube and D permeated radially outwards through the membrane into a vacuum chamber. Permeation experiments were conducted at 300, 350, 400, and 450°C, from 100 to 1000 sccm total flow rate, and with total pressures of 90, 150, 190, and 250 kPa. Deuterium permeation was consistently lower than predicted from diffusion-limited permeation models, thus we developed a transport model that included gas-phase mass transfer and surface reactions to model experimental results. The dissociation constant was optimized to fit the developed model to experimental data.**

Keywords—palladium, membrane, permeation, deuterium

I. INTRODUCTION

The deuterium (D) and tritium (T) fusion reaction provides the most likely path towards commercial fusion power. However, any DT fusion concept must be coupled with a safe, reliable, and efficient tritium fuel cycle.

Hydrogen permeable membranes are used throughout the fusion fuel cycle and are characterized by high reliability, continuous operations, and, with the use of dense metals, perfect selectivity of molecular hydrogen isotopes (Q_2, where Q is H, D, or T) to other nonhydrogen impurities. Palladium membranes and membrane reactors are used in the fusion fuel cycle to purify deuterium and tritium as well as to remove deuterium and tritium from impurities, such as water, methane, or ammonia [1, 2].

Membrane and membrane reactors could also act as a continuous method of tritium extraction from solid breeder concepts, such as the Helium Cooled Pebble Bed concept developed by EUROfusion [3-5]. In solid breeder concepts, tritium is collected in the purge gas, which is atmospheric pressure He with a small fraction of H_2 dopant (0.1%) to saturate surfaces and facilitate the removal of tritium from the solid Li-containing ceramics. The anticipated purge gas stream consists of predominately He and trace HT and HTO. The primary technology considered for tritium extraction is a combination of reactive and cryogenic molecular sieve beds for HTO and HT recovery, respectively [6]. Membranes are an alternative option where a water gas shift membrane reactor extracts HT from HTO. A proposed zeolite membrane cascade provides a preconcentration stage to increase the concentration of HT in He prior to T recovery by PdAg permeators [4]. However, challenges have persisted in the development of zeolite membranes with sufficient He-HT separation [7]. Thus, understanding and validating the transport phenomena of permeators for hydrogen isotopes in low partial pressures in He gas streams is critical to assessing the applicability of Pd permeators for use as tritium extraction systems for solid breeders.

Of the palladium alloys, the palladium-silver (Pd-25Ag wt%) alloy has received the most attention as a permeator material due to its higher permeability and lower critical temperature for β hydride phase formation compared to pure palladium [8-10].

In this manuscript, the gas-driven permeation of D is measured from both pure D_2 gas as well as a 3.95% D_2 in the He mixture at flow rates from 100–1000 sccm, pressures from 90–250 kPa, and temperatures from 300–450°C. Diffusion-limited permeation models overpredict permeation through the membrane; however, permeation models that also include gas-phase mass transport and surface reactions provide a better fit to experimental data. Due to the uncertainty in the surface reaction rate constants, a rate-constant was optimized to fit experimental data.

II. METHODS

A. Experimental

A self-supported Pd-25Ag tubular membrane was procured from REB Research & Consulting with a 15.2 cm length, 3.18 mm outer diameter, and 76.2 µm wall thickness. The Pd-25Ag tube was brazed on both ends to 316 stainless steel (SS) tubes with 10.2 cm length, 4.76 mm outer diameter, and 889 µm wall thickness. The stubs were connected to 4.76 mm outer diameter 316 SS tubing with Swagelok compression fittings and inserted into a 2.54 cm outer diameter quartz tube with Conflat flanges on either side to form the vacuum boundary. A type K thermocouple inserted through a port touched the center of the membrane. This assembly is referred to as the test section module and is labeled in Fig. 1, a photograph of the test section module and experimental apparatus.

The test section module was installed in a tube furnace with a 30.5-cm-long heated zone, the furnace was controlled with the type K thermocouple touching the membrane. 316 SS heat shields were placed at the end of the heated zone to promote uniform heating as well as to act as membrane supports in the module. Turbomolecular and dry scroll pumps evacuated the annulus of the test section module to a base vacuum of 1.0×10^{-5} Pa measured with a KJLC 392 series wide-range combination gauge. MKS 1259C 1000 sccm mass flow controllers set the flow rate of feed gas that entered inside the tube module. The pressure of the feed gas was set with a Swagelok back pressure regulator and measured with an Omega PX309 pressure transducer. The permeate flow rate was measured with a MKS 1159B 50 sccm mass flow meter.

Two MKS quadrupole mass spectrometers (QMS) monitored gas speciation, an e-Vision 2 with 1–100 amu range and a Microvision 2 with a 1–6 amu range. Prior to each permeation campaign, ultra-high purity (UHP) helium (5N, Norco) was pressurized in the tube side of the membrane to 250 kPa, and no increase in the 4 amu peak was observed by the QMSs in the shell side, thus verifying the module is leak free and that gas species measured during the permeation campaign was D_2. The 4 amu signal during D permeation experiments saturated the detector in the QMS, so an MKS 1159B mass flow meter was the sole permeation measurement device. The membrane was calibrated with UHP D_2 (5N, Advanced Specialty Gases) and tested with a premixed 3.95% D_2 in He cylinder (Norco).

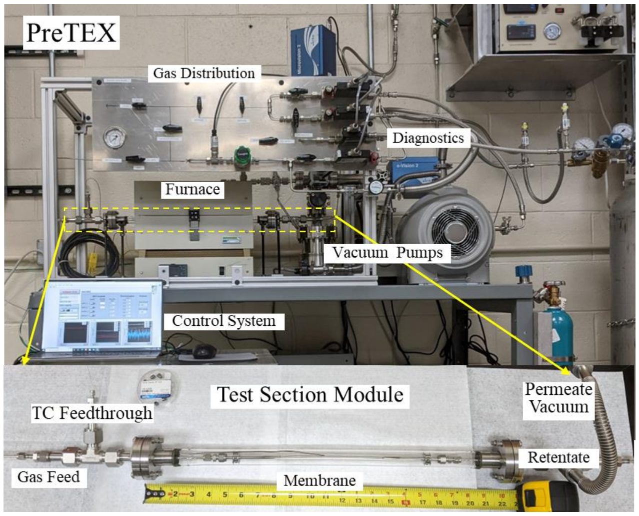

Fig. 1. PreTEX apparatus with an expanded photo of the test section module with the Pd-25Ag membrane installed.

This apparatus was called the Pre–Tritium Extraction eXperiment (PreTEX) as it used diagnostics for the Tritium Extraction eXperiment (TEX), a forced-convection lead-lithium loop under construction at the Safety and Tritium Applied Research facility [11]. A secondary purpose of PreTEX was to validate permeation properties of tubular membranes prior to installation in TEX for PbLi operation.

B. Model

The theoretical model of hydrogen mass transfer through metallic membranes is commonly described by the Richardson equation:

$$J = DK_S\,(P_f^{0.5} - P_p^{0.5})/l \qquad (1)$$

where the hydrogen flux (J, $\mathrm{mol_{Q2}\ m^{-2}\ s^{-1}}$) through the membrane is described by the permeability (Φ, $\mathrm{mol_{Q2}\ m^{-1}\ s^{-1}\ Pa^{-0.5}}$), known as the product of diffusivity (D, $\mathrm{m^2\ s^{-1}}$) and solubility (K_S, $\mathrm{mol_{Q2}\ m^{-3}\ Pa^{-0.5}}$), the membrane thickness (l, m), feed pressure (P_f, Pa), and permeate pressure (P_p, Pa). Solubility is commonly referred to as the Sieverts' constant.

The Richardson equation assumes mass transport is rate limited by the interstitial diffusion of Q atoms through the bulk metal, which is referred to as the *diffusion-limited* model (DM). With low hydrogen partial pressures, fast interstitial diffusion, or contaminated surfaces such as metal oxides, the hydrogen transport can be rate limited by the surface reactions of dissociative adsorption ($H_2 \rightarrow 2H$) on the feed-side surface or recombinative desorption ($2H \rightarrow H_2$) on the permeate-side surface; this is referred to as *surface limited*. In gas mixtures or liquid systems, such as PbLi [12] or FLiBe [13], hydrogen transport can be limited by mass transfer through the boundary layer in the fluid-phase; this is known as *mass transport limited*. Mass transport-limited regime is common in liquid systems [15], but also referred to as concentration-polarization in gas separation membrane systems. Considering these other transport processes is critical when assessing the permeation of hydrogen in a low H partial pressure system.

Thus, the following flux equations were solved in a numerical transport model coded in MATLAB where the permeator was segmented axially into nodes (i). The mass transport of the D_2 in the He gas mixture from the bulk to the feed-side wall surface is described as:

$$J(i) = K_T\,(P_{B,f,i} - P_{S,f,i})/RT_g \qquad (2)$$

where K_T is the mass transport coefficient ($\mathrm{m\ s^{-1}}$), $P_{B,f}$ is the D_2 partial pressure in the bulk feed gas (Pa), $P_{S,f}$ is the D_2 partial pressure at the permeator surface (Pa), R is the ideal gas constant ($\mathrm{J\ mol^{-1}\ K^{-1}}$), and T_g is the gas temperature (K). The surface reactions on the feed side are described by the rate of dissociative absorption on the surface and subtracted by the rate of recombinative desorption back into the gas phase:

$$J(i) = k_d\,P_{S,f,i} - k_r C_{S,f,i}^2 \qquad (3)$$

where k_d is the dissociation constant ($\mathrm{mol_{D2}\ m^{-2}\ s^{-1}\ Pa^{-1}}$), k_r is the recombination constant ($\mathrm{m^4\ mol_{D2}^{-1}\ s^{-1}}$), and $C_{S,f}$ is the concentration of deuterium on the feed-side surface ($\mathrm{mol\ m^{-3}}$). The interstitial diffusion through the membrane is described by Fick's first law in cylindrical coordinates:

$$J(i) = -D(C_{S,p,i} - C_{S,f,i})/r_i ln(r_o/r_i) \qquad (4)$$

where $C_{S,p}$ is the concentration on the permeate surface ($\mathrm{mol\ m^{-3}}$), r_i is the inner radius of the permeator tube (m), and

r_o is the outer radius of the permeator tube. The recombinative desorption from the permeate surface is described by:

$$J(i) = k_r C_{S,p,i}^2 - k_d P_{S,p} \tag{5}$$

where $P_{S,p}$ is the pressure at the permeate surface (Pa). The mass transfer into the vacuum pumping system is assumed to be infinitely fast and is not considered in this transport model, thus the surface pressure is assumed to be equal to the bulk pressure.

The mass transport coefficient for deuterium transport through the mixed gas boundary layer in the feed gas is calculated from the Sherwood number correlation for laminar flow inside a circular tube ($Sh_{Laminar}$) [14]:

$$Sh_{Laminar} = 2r_i K_T / D_{D2\text{-}He} = 1.62(2r_i Re Sc/L)^{1/3} \tag{6}$$

where $D_{D2\text{-}He}$ is the binary diffusion coefficient of D_2 in He (m^2 s^{-1}), Re and Sc are the Reynolds and Schmidt number, respectively, and L is the length of the membrane tube (m). Note that the Re ranged from 5.15 to 143 in our experiments.

The axial advective D_2 transport was solved by the mass conservation equation where the amount of D_2 entering the finite volume in the tube was equal to the amount of D_2 exiting the finite volume in the tube and the amount permeated through the surrounding membrane via the numerically calculated flux from solving equations (2-5). The number of nodes was set to n = 50. This code followed the same logic used in previous work modeling the vacuum permeator for tritium extraction from PbLi [15]. Table 1 reports the parameters used in our model, where T_m is the membrane temperature (K). In this work, the membrane temperature was assumed to be constant in the axial direction due to the 15.2 cm length of membrane placed in the center of a 30.5 cm heated zone and measuring the axial temperature distribution was challenging due to module configuration. However, thermal gradients likely existed due to flowing He thus an uncertainty of 5 °C was assumed in analysis. In contrast to the diffusion-limited Richardson equation, the model including mass transport, surface reactions, and bulk diffusion is referred to as the complete model (CM).

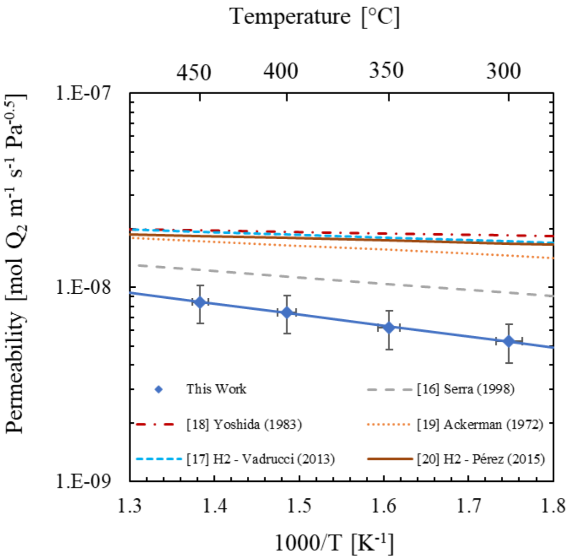

Fig. 2. D_2 permeability measured at 90 kPa feed side and 1 Pa permeate using UHP D_2. The solid blue line is the Arrhenius fit to the data in this work.

TABLE I. VALUES USED IN MEMBRANE MODEL ANALYSIS.

Symbol	***Value/Equation***	***Unit***	***Ref.***
L	0.152	m	—
r_i	1.59×10^{-3}	m	—
r_o	1.51×10^{-3}	m	—
D	$1.87 \times 10^{-7} \exp(-24685/RT_m)$	m^2 s^{-1}	[16]
Ks	$0.184 \exp(18531/RT_m)$	m^2 s^{-1}	[16]
kd^a	$1.30 \times 10^{-2} \exp(-24780/RT_m)$	mol m^{-2} s^{-1} Pa^{-1}	[16]
kr^a	$0.390 \exp(-61842/RT_m)$	mol^{-1} m^4 s^{-1}	[16]

[a.] Optimized in analysis of experimental data.

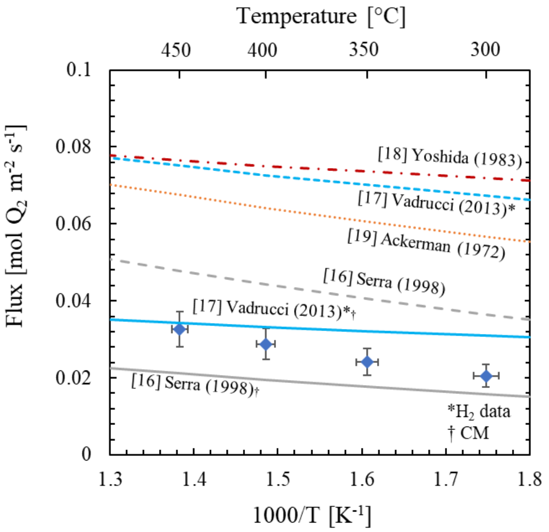

Fig. 3. D_2 flux measured at 90 kPa feed side and 1 Pa permeate pressure with UHP D_2. Solid lines were calculated with the CM and dashed lines were calculated with the DM.

III. RESULTS AND DISCUSSION

A. *UHP deuterium measurement and analysis*

The first experiment measured deuterium permeation using UHP D_2 at 90 kPa with a feed flow rate of 100 sccm and membrane temperatures of 300, 350, 400, and 450°C. The UHP D_2 was delivered to the tube side and vacuum pumped on the shell side. The permeated D_2 was measured with a mass flow meter downstream of the vacuum pumps. The measured D_2 permeability of the Pd-25Ag membrane is compared to literature values in Fig. 2. The measured permeability, calculated using Eq. (1), was less than that reported in literature [16-20]. An Arrhenius fit was applied to the measured data to yield a permeability (Φ) in units of mol_{D2} m^{-1} s^{-1} $Pa^{-0.5}$ and activation energy units of J mol^{-1}:

$$\Phi = 5.03 \pm 0.40 \times 10^{-8} \exp(-10700 \pm 400/RT_m) \quad (7)$$

However, the permeability reported in Fig. 2 and Eq. (7) was calculated using the Richarson equation from the measured flux. Fig. 3 reports the measured flux, which compared well with flux predicted using the CM with values reported by Serra et al. [16] and Vadrucci et al. [17]. The dashed lines in Fig. 3 represent flux predicted using the DM. The values from the work by Vadrucci et al. are for H_2 and use the solubility value for H_2 from Serra et al. [16] This demonstrates the importance of considering the surface reaction transport phenomena and validates the transport code used to analyze data.

B. 3.95% D_2 in He mixed gas results and analysis

The second campaign used the premixed 3.95% D_2 in He as the feed gas at the same membrane temperatures as the UHP D_2 gas test: 300, 350, 400, and 450 °C. The feed gas was held at a fixed pressure, and the flow rate was increased from 100 sccm to a maximum of 1,000 sccm in 100 sccm intervals. A minimum time of 300 seconds was allowed for permeation to stabilize at the set feed flow rate before the flow rate was increased. If no increase in permeation rate was measured after two increases in feed flow rate, the flow rate ramp was ceased and feed flow rate was decreased to 100 sccm. This process was repeated for feed gas pressures of 90, 150, 190, and 250 kPa at each membrane temperature. Fig. 4 displays all experimentally measured data with the premixed gas. The CM predictions reported in Fig. 4 used optimized dissociation constants (CM-O).

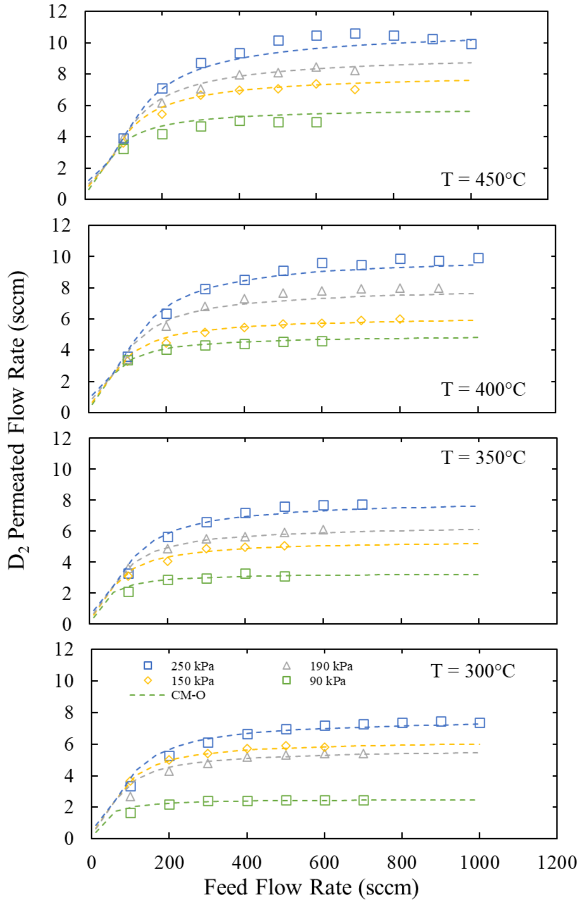

Fig. 4. D_2 permeated flow rate (sccm) as a function of feed flow rate (sccm) and total gas pressure from the 3.95% D_2 in He gas mixture experiments.

When comparing the mixed gas measurements to model predicted results (Fig. 5), a similar observation occurred to that of the UHP D_2 gas. The DM overpredicts the permeated flow rate at the measured conditions. The CM improves the model fit to the experimental data; however, it consistently overpredicted the flux as well. We assumed that the surface reaction rate constants contained the most uncertainty. Therefore, the value of the dissociation constant (k_d) was optimized to fit the CM to the experimental data (CM-O in Fig.4 and Fig. 5). The recombination constant (k_r) was then calculated with Eq. (8), using the solubility of D in Pd-25Ag from Serra et al. [16]. Note that, alternatively, the recombination constant could have been optimized and dissociation constant calculated using the same approach.

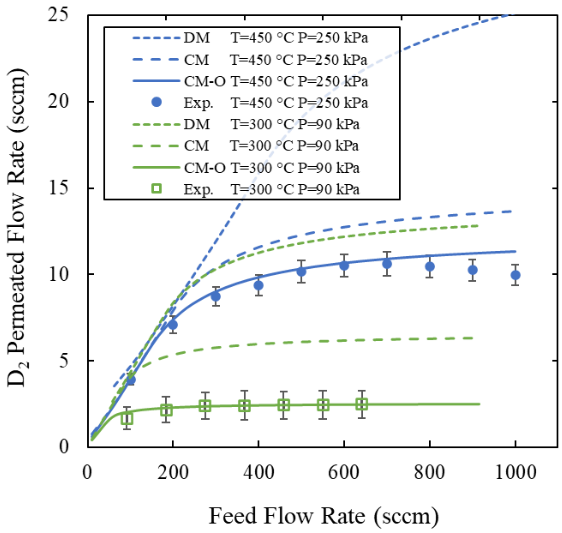

Fig. 5. The D_2 permeated flow rate as a function of feed flow rate for the 300 °C and 90 kPa total pressure data (□) as well as the 450 °C and 250 kPa total pressure data (●). The lines correspond to the various permeation models used in this work.

$$K_S = \sqrt{k_d / k_r} \quad (8)$$

The optimized value of k_d was fit to an Arrhenius dependence, displayed in Fig. 6, and reported in Eq. (9) in units of mol m^{-2} s^{-1} Pa^{-1} and activation energy units of J mol^{-1}. The optimized value of the dissociation constants is approximately a factor of 25 lower than that of Serra et al. [16] and 15 lower than that of Vadrucci et al. [17]. This is likely caused by surface contamination from trace impurities in the gas mixtures and vacuum. In previous work with palladium-coated vanadium samples, the surface reaction rate constants encompassed significant uncertainty based on the cleanliness of the surface [21]. While the “clean” Pd surface was achieved in that work by oxidizing and reducing both surfaces of the membrane, and the dissociation constant compared very well with that of Serra et al. [16]. Prior to cleaning the surfaces, the measured surface reaction rate was reduced by a factor of 10^6. The surfaces in the referenced work [21] were exposed to ultra-high vacuum and UHP D_2 at a similar temperature range to this work. A similar phenomena was also observed by Alimov et al. for Pd-coated V [22]. No oxidation and reduction treatments were performed on the membrane tested in this work; therefore, the surface reaction was determined to be the parameter with the most uncertainty. Thus, the state of the membrane surface plays a key role in the permeation of hydrogen in the limit of low partial pressures and must be evaluated in prototypic conditions.

$$k_d=7.99\pm9.33\times10^{-4}\exp(-27600\pm1100/RT_m) \tag{9}$$

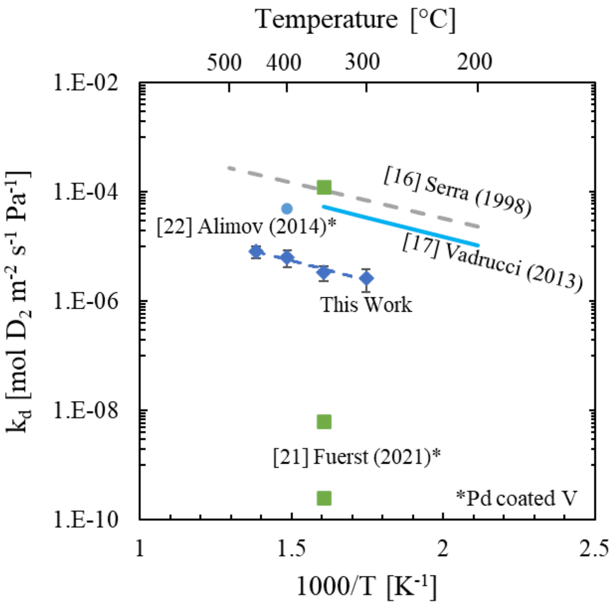

Fig. 6. The optimized dissociation constant based on permeation measurements and comparison to literature values.

IV. CONCLUSIONS

A self-supported Pd-25Ag membrane was examined for deuterium permeation from 300–450°C in both UHP gas as well as a premixed 3.95% D_2 in a He gas mixture. In the experimental configuration of the PreTEX apparatus, the feed gas was supplied inside the tube and a vacuum was pulled on the permeate or shell side. The UHP gas resulted in permeation rates lower than those of literature. However, the experimental measurements compared well to those of literature once surface reactions were considered in the transport model. This effect is more evident with lower partial pressures of hydrogen, such as with the 3.95% D_2 premixed gas mixture. The experimental permeability and flux results were lower than expected from literature values, including mass transport in the gas-phase and surface reactions. Due to the large uncertainty in the dissociation constant, this value was chosen to be optimized to fit experimental data. With this value optimized, excellent agreement between the model and experiment was achieved. Thus, the surface reactions in membrane calculations must be considered when assessing Pd-based membranes for permeation in low partial pressure applications, such as those in the breeder blanket of a fusion reactor.

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