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MEASUREMENT OF THE PERMEABILITY OF TUNGSTEN TO NITROGEN

Final Report

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P.K. Conn, E.C. Duderstadt, and R.E. Fryxell

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NUCLEAR MATERIALS and PROPULSION OPERATION
NUCLEAR TECHNOLOGY DEPARTMENT
NUCLEAR ENERGY DIVISION

GENERAL  ELECTRIC

Cincinnati, Ohio 45215

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June, 1967

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Technical Management
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Cleveland, Ohio
Materials and Structures Division
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ABSTRACT

Permeation rates of nitrogen through arc-cast tungsten were measured in the temperature range 1800° to 2600°C with nitrogen pressure differentials of 1 and 0.1 atmospheres. Rates were proportional to the difference between the square roots of the nitrogen pressure on each side of the membranes and inversely proportional to membrane thickness, indicating that atomic nitrogen was the diffusing species and that diffusion was the rate limiting step of the process. The activation energy for permeation was found to be 70.0 ± 0.8 kcal/mole of permeating species.

CONTENTS

	Page
SUMMARY	9
INTRODUCTION	11
EXPERIMENTAL PROCEDURES	12
Cell Design and Experimental Apparatus	12
Nitrogen Determination	15
RESULTS AND DISCUSSION	15
CONCLUSIONS	21

FIGURES

	Page
1. Schematic Drawing of Permeation Cells	13
2. Schematic Drawing of Nitrogen Permeation Apparatus	14
3. Nitrogen Content of Helium Sweep Gas as a Function of Time at Test Temperature	16
4. Arrhenius Plot of Permeation Coefficients for Nitrogen through Arc-cast Tungsten	20
5. Comparison of Permeation Coefficients for Nitrogen, Hydrogen, and Oxygen through Arc-cast Tungsten	22

TABLES

	Page
1. Impurity Analyses of Gases	12
2. Summary of Nitrogen Permeation Data from Cell 1	17
3. Summary of Nitrogen Permeation Data from Cell 2	18
4. Diffusion Coefficients for Nitrogen in Tungsten	21
5. Summary of Permeation, Diffusion, and Solubility Data for Nitrogen, Hydrogen, and Oxygen in Tungsten	23

SUMMARY

Permeation rates for nitrogen through arc-cast tungsten were measured in the temperature range 1800° to 2600°C with nitrogen pressure differentials of 1.0 and 0.1 atmospheres. Gas chromatography was used to measure the quantities of nitrogen which permeated through 0.26 and 0.53-mm-thick membranes.

Permeation rates were proportional to the difference between the square roots of the nitrogen pressure on each side of the membrane and inversely proportional to membrane thickness, demonstrating that atomic nitrogen was the diffusing species and that diffusion was the rate limiting step of the process.

Permeation coefficients calculated from the rates may be represented by the equation:

$$P = P_0 \exp(-Q/RT)$$

where:

P = permeation coefficient in cc(STP)-mm/cm²-min-atm^{1/2}

P₀ = permeation constant = 277 cc(STP)-mm/cm²-min-atm^{1/2}

Q = activation energy = 70,000 ± 800 cal/mole of permeating species

R = gas constant cal/mole-°K

T = absolute temperature in °K

The permeabilities were combined with solubilities from the literature to calculate diffusivities for nitrogen in tungsten.

MEASUREMENT OF THE PERMEABILITY OF TUNGSTEN TO NITROGEN

P. K. Conn, E. C. Duderstadt, and R. E. Fryxell

INTRODUCTION

Permeation rates for nitrogen through polycrystalline arc-cast tungsten were measured experimentally in the temperature range 1800° to 2600°C. Nitrogen pressure differentials and membrane thickness were varied in order to identify the rate limiting step of the process. Permeation coefficients were calculated and the activation energy for the process was determined.

For any gas-tight structure (one which does not have channels for molecular flow), permeation should proceed as follows: (1) impact and adsorption of the gas atoms or molecules on one face of the membrane, (2) dissociation of the gas (for diatomic gases), (3) solution of the gas in the metal, (4) bulk diffusion of atoms through the metal, (5) exsolution of the gas at the opposite face (with possible recombination of diatomic gases), and (6) desorption of the gas from the surface. The gradient of the chemical potentials on each side of the membrane provides the driving force for permeation. The rate controlling step may be the reaction steps at either of the interfaces or bulk diffusion.

For diatomic gases which dissociate prior to diffusion, the quantity of gas which permeates through a membrane or wall at an elevated temperature, where bulk diffusion generally is the rate limiting step of the process, is given by the equation:

$$q = P A t (p_1^{1/2} - p_2^{1/2})/d \quad (1)$$

where:

q = total amount of gas permeating a membrane

P = temperature dependent permeation coefficient

A = area of membrane

t = time

p_1 = gas pressure of high pressure side

p_2 = gas pressure of low pressure side

d = thickness of membrane

The permeation coefficient, P, is temperature dependent according to the equation:

$$P = P_0 \exp (-Q/RT) \quad (2)$$

where : P_0 = permeation constant

Q = activation energy

R = gas constant

T = absolute temperature

When bulk diffusion of atoms through the metal is the rate controlling step, the permeation coefficient for a given temperature is equal to the product of diffusivity and solubility at that same temperature, or:

$$P = DS \quad (3)$$

where: D = diffusivity

S = solubility

EXPERIMENTAL PROCEDURES

Cell Design and Experimental Apparatus

Permeation rates of nitrogen through arc-cast tungsten were measured using the two permeation cells shown in Figure 1. The cells had been used previously to measure permeation rates of hydrogen through tungsten under another NASA contract.* Each cell consisted of a cylindrical chamber divided into two parts by either a 0.26- or a 0.53-mm-thick membrane fabricated from high purity (>99.9% W) wrought tungsten sheet made from vacuum arc-cast tungsten. The fabrication of these cells has been described elsewhere.¹

The permeation cells were mounted vertically in a high temperature tungsten resistance furnace and the membranes and the center portions heated to test temperatures. Temperatures were measured by sighting an optical pyrometer on the edge of the membrane through a small hole in a tungsten plate which was fastened to the side of the cell. This arrangement provided an approximate black body condition and eliminated the need for emittance corrections. Appropriate corrections were made for the quartz window through which the temperatures were measured. Argon gas flowed through the 10 liter volume of the furnace at the rate of 4.7 liters/minute. An impurity analysis of the argon is given in Table 1, together with analyses of the other gases used in these measurements.

TABLE 1
IMPURITY ANALYSES OF GASES

Impurity	GASES		
	Argon	Nitrogen ^a	Helium ^b
Nitrogen	N. D. < 4 ppm	-	.5 ppm
Oxygen	N. D. < 4 ppm	0.9 ppm	N. D. < .02 ppm
Hydrogen	N. D. < 4 ppm	-	N. D. < .5 ppm
Hydrocarbons	N. D. < 4 ppm	< 1 ppm	N. D. < .05 ppm

N. D. = not detected

^aUltra-pure carrier grade, >99.999% N₂

^bUltra-pure carrier grade, 99.9995% He

Nitrogen gas flowed through one end of the cell (Figure 2) at rates of 60-100 cc/min (7 to 34 volume changes per minute depending on cell dimensions) entering through the Mo tube having a small W-Re inner extension tube and exhausting to the atmosphere through the other Mo tube. (Short intermediary sections of W-30Re-30Mo tubing facilitated fabrication.) The nitrogen was diluted with 90 percent helium when permeation rates with a 0.1 atmosphere nitrogen pressure differential across the membrane were being measured.

*NAS3-6216.

¹Aitken, E. A., Conn, P. K., Duderstadt, E. C., and Fryxell, R. E., "Measurement of the Permeability of Tungsten to Hydrogen and to Oxygen," NASA CR-54918, May 1966.

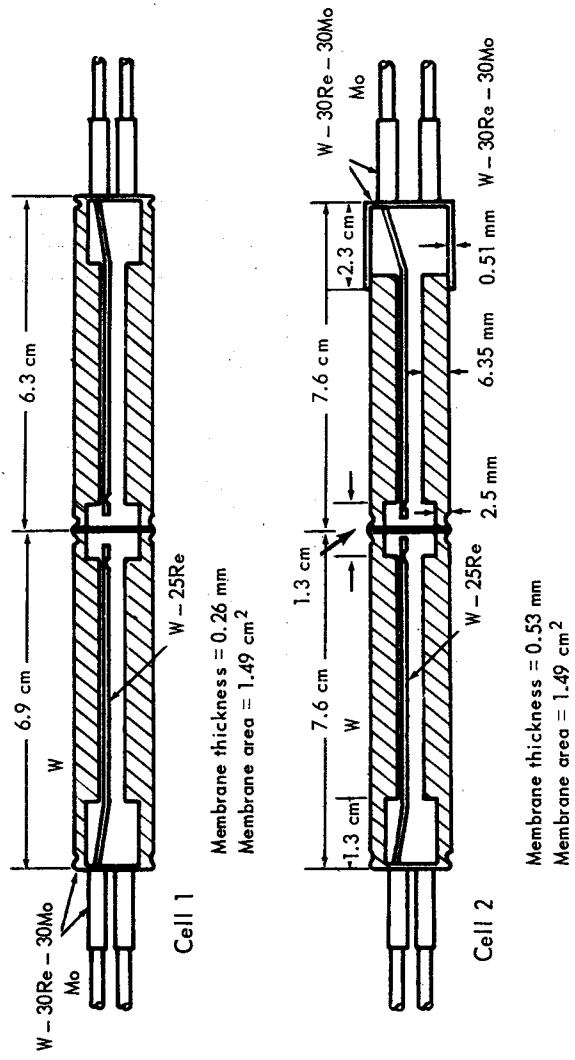


Fig. 1 - Schematic drawing of permeation cells

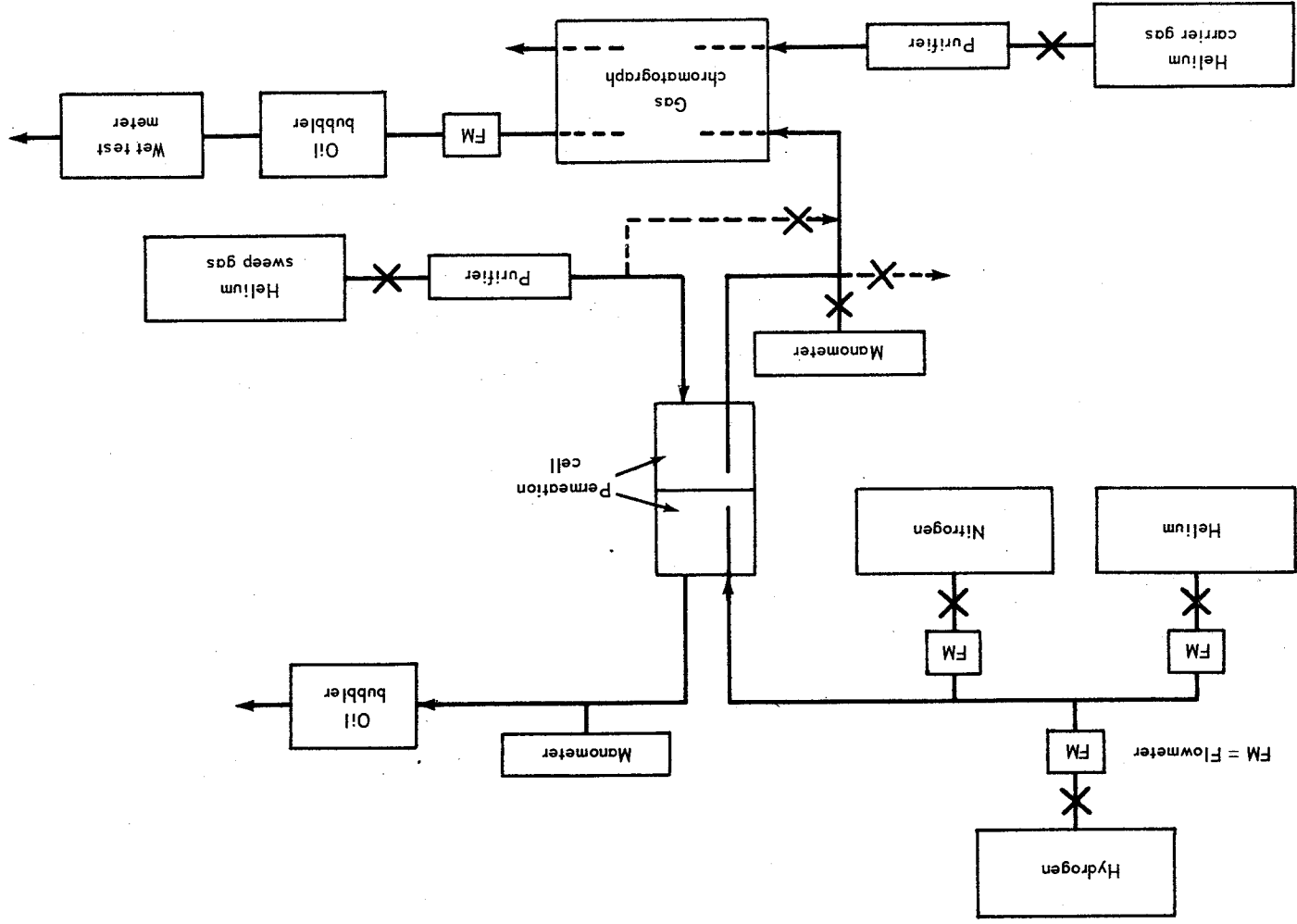


Fig. 2 - Schematic drawing of nitrogen permeation apparatus

A negligible nitrogen pressure was maintained on the opposite side of the membrane by sweeping helium through that end of the cell. Helium flow rates were generally in the range 35 to 100 cc/min but were varied from 20 to 200 cc/min in order to assess the possible effect of nitrogen permeation through the walls of the sweep gas tubes on the permeation rate measurements.

Nitrogen Determination

The helium sweep gas, carrying the nitrogen which permeated the membrane, was analyzed periodically for nitrogen content by gas chromatography. Both the helium sweep gas and the chromatograph carrier gas were an ultra-pure carrier grade gas (see Table 1) and were passed through liquid-nitrogen-cooled molecular-sieve purifiers for additional purification. The chromatograph utilized a molecular sieve column at ambient temperature for separation of gas impurities. Elution times (with a 6.7 meter column length and ~ 11 cc(STP)/min carrier gas flow rate) ranged from about 2 minutes for hydrogen and neon, which eluted simultaneously, to about 25 minutes for carbon monoxide. Oxygen and argon also eluted simultaneously at about 6 minutes; nitrogen, CH_4 , and CO eluted separately at about 9, 19, and 25 minutes, respectively. A thermal conductivity detector was used for analysis. Its output, a linear function of nitrogen content over the 1 to 100 ppm calibration range, was recorded on a strip chart. The detector and associated electronics were capable of measuring a fraction of a part per million nitrogen in the helium sweep gas. It had similar sensitivity to oxygen, CO , CO_2 and CH_4 ; sensitivity to hydrogen was somewhat lower.

Operation of the permeation experiments at atmospheric pressure was necessary to minimize the possibility of deforming the permeation cell. Under such conditions, a trace of air was evidently introduced whenever a gas sample was injected into the chromatograph. This resulted in small oxygen and nitrogen peaks equivalent to about 0.2 and 0.7 to 0.8 ppm respectively. The latter occurred on the chromatograph trace about 0.6 minute later than the peak which resulted from the nitrogen which was present in the sweep gas; this was sufficient separation so that the peaks could be resolved.

At each temperature and nitrogen pressure, a series of nitrogen determinations was made until it was certain that a steady-state permeation rate had been achieved. Steady-state rates were attained rapidly as shown in Figure 3, which also indicates typical precision of the individual determinations. The minimum time between determinations was about 20 minutes.

Permeation coefficients were calculated from the measured permeation rates using a form of equation (1):

$$P = q/t \left[\frac{d}{A} (p_1^{1/2} - p_2^{1/2}) \right] \quad (1A)$$

where: q/t = quantity of nitrogen permeating through the membrane per unit time (ppm nitrogen in helium sweep gas times flow rate of helium sweep gas*)

Based on the extent of bonding observed in examination of a permeation cell having similar thermal history, the inside diameters of the heavy wall tungsten tubes were used to calculate membrane areas.

RESULTS AND DISCUSSION

Steady-state permeation rates of nitrogen through the membrane of each cell were measured at temperatures between 1800° and 2600°C . The data are summarized in Tables 2 and 3 together with permeation coefficients calculated with equation (1A). Coefficients obtained from the two cells are in excellent agreement. Permeation rates were proportional to the difference between the square roots of nitrogen pressure on each side of the membrane

*Flow rate of helium sweep gas was calculated from total flow as measured with a wet test meter in a given time interval.

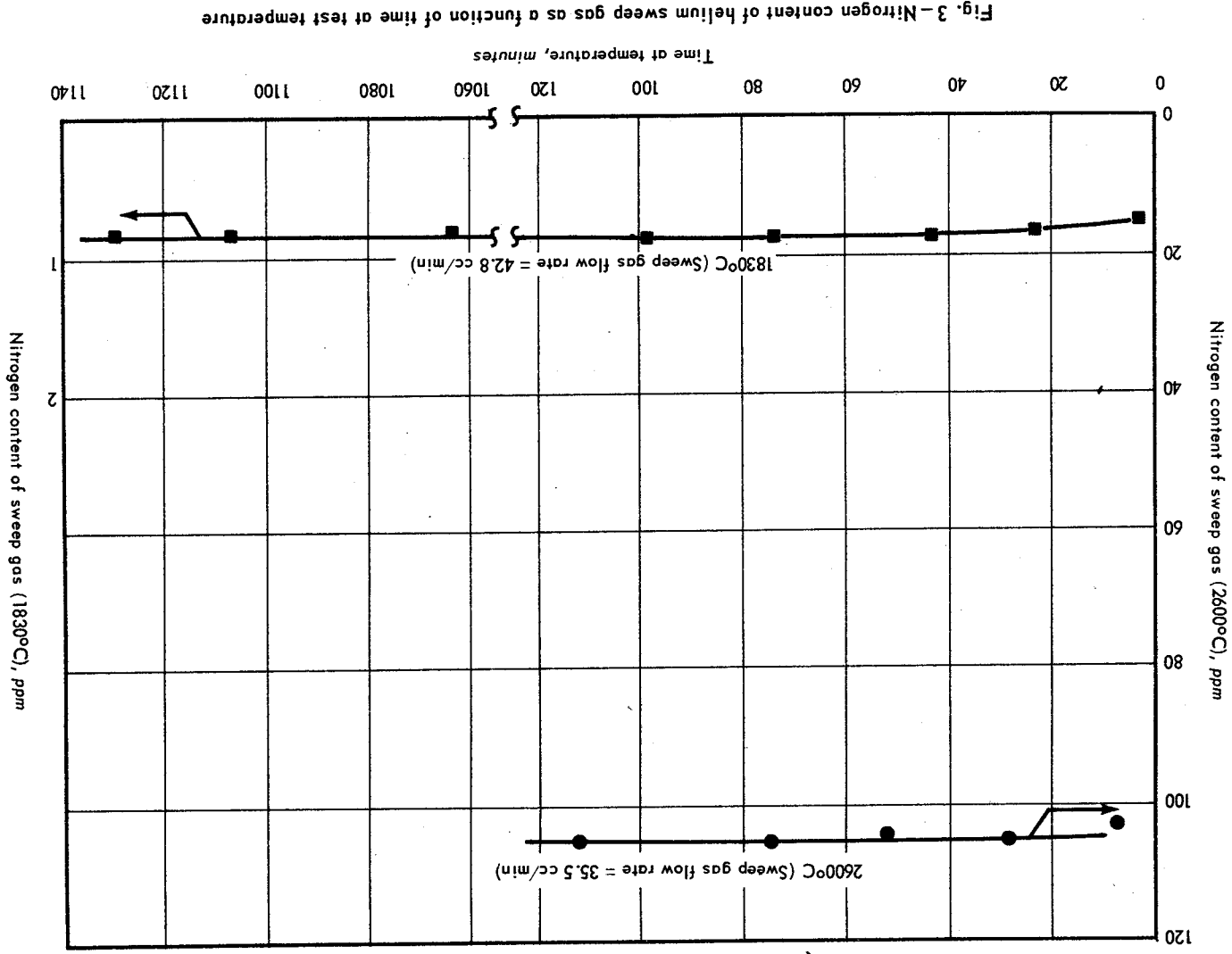


Fig. 3 - Nitrogen content of helium sweep gas as a function of time at test temperature

TABLE 2

SUMMARY OF NITROGEN PERMEATION DATA FROM CELL 1^a

Order of Measurement	Temperature, °C ^b	Nitrogen Pressure, atmos.	Helium Sweep Flow Rate, cc(STP)/min	Nitrogen Content, ppm	Number of Analyses	Permeation Coefficient ^c
34	2610	1	45.2	166	4	1.31×10^{-3}
12	2600	1	97.3	82.1	4	1.39×10^{-3}
13	2590	0.1 ^d	97.3	24.1	6	1.29×10^{-3}
2	2590	1	104	70.4	7	1.27×10^{-3}
35	2580	1	45.2	152	2	1.19×10^{-3}
9	2510	1	101	53.8	7	9.45×10^{-4}
1	2410	1	105	29.6	8	5.43×10^{-4}
30	2340	0.9 ^e	50.0	39.7	1	3.65×10^{-4}
26	2310	1	50.6	33.0	7	2.91×10^{-4}
3	2300	1	103	17.3	2	3.10×10^{-4}
33	2300	1	46.0	38.0	10	3.04×10^{-4}
29	2300	0.9 ^e	50.0	34.7	1	3.19×10^{-4}
22	2300	0.1 ^d	51.2	10.7	7	3.01×10^{-4}
23	2290	1	51.2	34.4	3	3.06×10^{-4}
27	2290	1	50.0	30.7	8	2.67×10^{-4}
36	2290	1	44.6	36.5	4	2.83×10^{-4}
4	2280	1	103	15.0	6	2.69×10^{-4}
28	2230	0.9 ^e	50.0	21.2	2	1.95×10^{-4}
10	2200	1	101	10.2	5	1.79×10^{-4}
11	2170	1	101	8.76	7	1.54×10^{-4}
6	2110	1	103	6.39	3	1.15×10^{-4}
7	2100	1	103	5.63	2	1.01×10^{-4}
8	2090	1	101	5.20	4	9.14×10^{-5}
18	2040	1	53.4	7.26	2	6.75×10^{-5}
20	2020	0.1 ^d	52.8	2.26	2	6.56×10^{-5}
21	2010	0.1 ^d	52.8	2.09	6	6.07×10^{-5}
19	2010	1	53.4	6.27	9	5.83×10^{-5}
5	2000	1	103	3.25	7	5.82×10^{-5}
32	1830	1	46.2	1.75	5	1.41×10^{-5}
25	1810	0.9 ^e	51.9	1.26	6	1.20×10^{-5}
24	1810	1	51.9	1.31	7	1.18×10^{-5}
15	1810	1	55.4	1.29	5	1.24×10^{-5}
31	1800	1	37.8	1.76	4	1.16×10^{-5}
16	1800	1	54.9	1.21	5	1.16×10^{-5}
17	1800	0.1 ^d	54.0	0.37	5	1.10×10^{-5}
14	1800	1	95.5	0.64	4	1.07×10^{-5}

^a Membrane area = 1.49 cm²; membrane thickness = 0.26 mm.

^b Expressed to the nearest 10°C. For statistical evaluation, actual averages were used; measurements corresponding to the number of analyses comprised each average.

^c cc(STP)-mm/cm²-min-atm^{1/2}.

^d 0.1 atm nitrogen; 0.9 atm helium.

^e 0.9 atm nitrogen; 0.1 atm hydrogen.

TABLE 3
SUMMARY OF NITROGEN PERMEATION DATA FROM CELL 2^a

Order of Measurement	Temperature, °C ^b	Nitrogen Pressure, atmos.	Helium Sweep Flow Rate, cc(STP)/min	Nitrogen Content, ppm	Number of Analyses	Permeation Coefficient ^c
3	2610	1	43.3	91.3	6	1.41 × 10 ⁻³
10	2600	0.1 ^d	35.3	31.6	7	1.26 × 10 ⁻³
9	2600	1	35.5	105	6	1.33 × 10 ⁻³
13	2480	1	20.0	102	4	7.30 × 10 ⁻⁴
12	2470	1	212	9.54	7	7.20 × 10 ⁻⁴
4	2290	1	43.5	19.2	6	2.97 × 10 ⁻⁴
8	2270	0.1 ^d	44.7	4.98	7	2.50 × 10 ⁻⁴
15	2150	1	203	1.74	3	1.26 × 10 ⁻⁴
14	2150	1	28.1	12.8	6	1.28 × 10 ⁻⁴
7	2000	0.1 ^d	44.2	1.12	8	5.62 × 10 ⁻⁵
5	1970	1	44.7	2.67	7	4.24 × 10 ⁻⁵
6	1960	0.1 ^d	44.4	0.82	4	4.10 × 10 ⁻⁵
2	1830	1	44.3	0.81	4	1.27 × 10 ⁻⁵
1	1830	1	42.8	0.87	3	1.33 × 10 ⁻⁵
11	1790	0.1 ^d	36.2	0.26	2	1.06 × 10 ⁻⁵

^aMembrane area = 1.49 cm²; membrane thickness = 0.53 mm.

^bExpressed to the nearest 10° C. For statistical evaluation, actual averages were used; measurements corresponding to the number of analyses comprised each average.

^ccc(STP)-mm/cm²-min-atm^{1/2}.

^d0.1 atm nitrogen; 0.9 atm helium.

and inversely proportional to membrane thickness, indicating that atomic nitrogen was the diffusing species and that diffusion was the rate limiting step.

It is possible (see Figures 1 and 2) that the permeation rates could be influenced by (1) nitrogen in the furnace atmosphere and (2) by solution or exsolution of the nitrogen by the cell material and lead tubes on the low N₂ pressure (sweep gas) side of the cell. The possibility that these factors could result in significant errors was investigated as follows.

To determine if nitrogen in the furnace atmosphere was contributing to the permeation rate, the nitrogen pressure differential across the walls of the exit side of the cell and the sweep gas lead tubes was varied by a factor of ~ 10 at 2150 ° and 2500 °C by changing the sweep gas flow rate and/or the furnace flow rate. No significant change in the measured permeation rates occurred.

The nitrogen pressure differential across the lead tubes on the exit side of the cell was quite small as indicated by an N₂ analysis of the furnace argon gas. With the membrane at 2600 °C and a nitrogen differential pressure of one atmosphere, the nitrogen content in the furnace argon was ~ 35 ppm as compared to ~ 100 ppm in the sweep gas.

The potential problem of the exit side of the cell "gettering" the nitrogen (or its opposite, exsolution) was investigated by measuring the time to reach a constant permeation rate after a large increase or decrease in temperature. Essentially the same steady-state rate at 2300 °C was quickly (20-40 minutes) attained after approaching that temperature from 1800 ° or 2600 °C. The maximum amount of nitrogen dissolved in the molybdenum and the other components of the low N₂ pressure side of the cell is considerably less than 1 ppm at the highest test temperature, as can be estimated from the solubility data of Norton and Marshall² assuming the validity of Sievert's law at the low nitrogen partial pressure of the sweep gas. Unpublished data from this laboratory indicate that no precipitated nitride phases should be present in tungsten or molybdenum under the existing conditions.

The possibility also exists that permeation inhibiting surface films on the membranes could influence the permeation rate measurements. Consequently, measurements were made at a few temperatures between 1800 ° and 2400 °C with about 10 percent hydrogen mixed with the nitrogen to assure that the surface of the membrane was free of oxide films. The permeation coefficients obtained were no higher than those obtained with pure nitrogen (see Table 2).

From these evaluations it was concluded that the experimental data were valid measurements of the nitrogen permeation rates through the tungsten membranes.

The logarithms of permeation coefficients are plotted versus reciprocal absolute temperature in Figure 4. Least squares analysis of the data from cells 1 and 2 separately and from both cells combined resulted in values for the activation energy of 69.9 ± 0.9 , 70.4 ± 1.3 , and 70.0 ± 0.8 kcal per gram atom, respectively. The \pm values are the 2σ deviations. The line shown in the figure corresponds to the analysis of the composite data, and is represented by the equation

$$P = 277 \exp (-70,000 \pm 800/RT).$$

A curve obtained by normalizing to atmospheric pressure the preliminary permeation data of Frauenfelder³ is included in Figure 4 for comparison. His measurements were made using non-isothermal membranes in the pressure range 2.5 to 250 Torr at temperatures from 1400 ° to 2225 °C, and resulted in an activation energy for permeation of 52 ± 2 kcal/mole. Frauenfelder also obtained diffusion coefficients for nitrogen in W from the times taken to achieve equilibrium rates of permeation.

²F. J. Norton and A. L. Marshall, "The Degassing of Metals," *Transactions AIME*, 156, 351-370 (1944).

³R. Frauenfelder, Westinghouse Electric Corp., Research Laboratory, Pittsburgh, Pa., Private Communication, May 23, 1967.

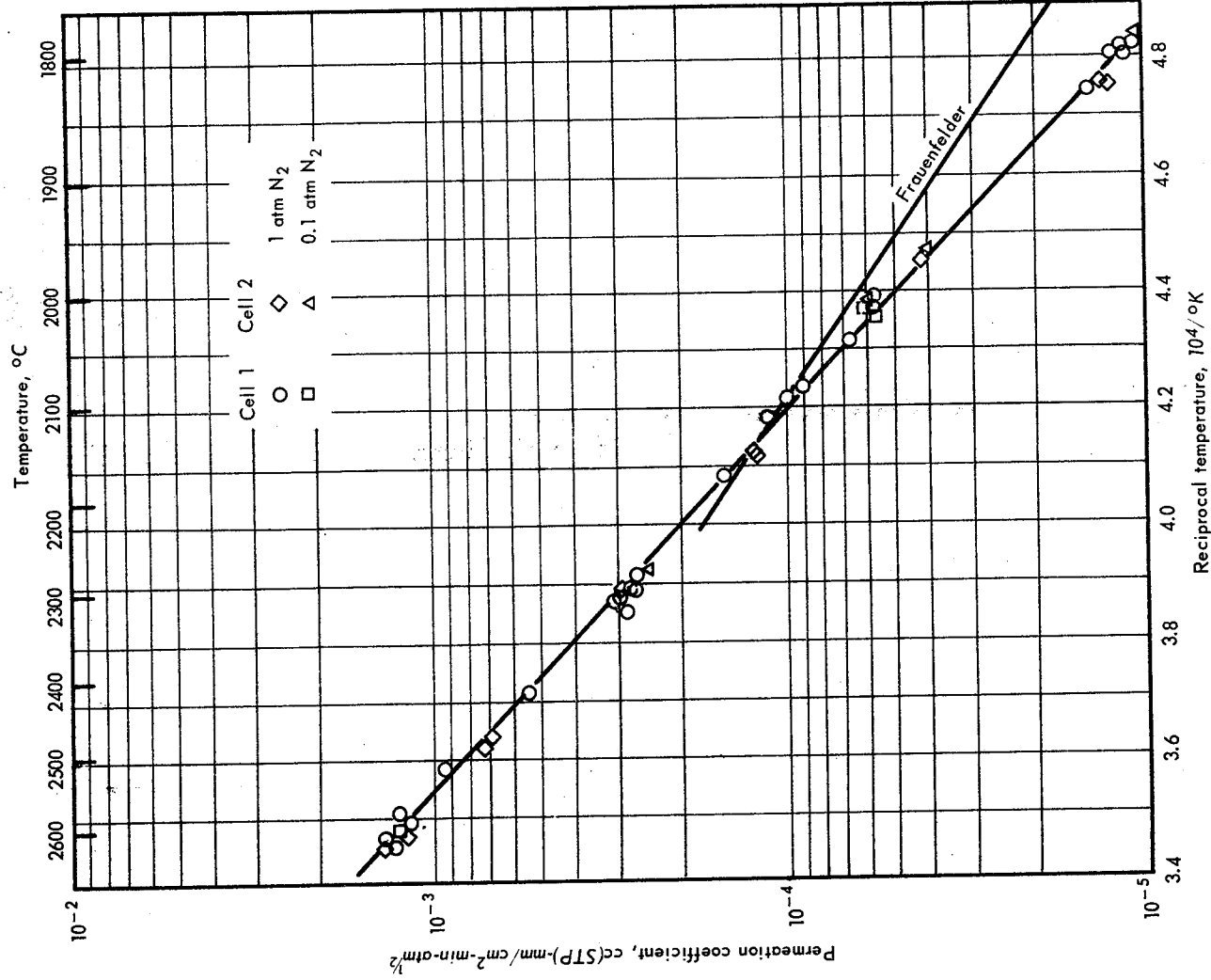


Fig. 4 - Arrhenius plot of permeation coefficients for nitrogen through arc-cast tungsten

Diffusion coefficients were calculated for 2000° and 2400°C from present measured permeation coefficients and the solubility data of Norton and Marshall² using equation (3) and are compared in Table 4 with diffusion coefficient values calculated using Frauenfelder's D_0 of 5.4 cm²/sec and activation energy for diffusion of 62 ± 3 kcal per gram atom. Although the D_0 of 10⁻³ to 10⁻² and activation energy for diffusion of 33 kcal per gram atom derived from the permeation data in this report and the solubility data of Norton and Marshall² differ appreciably from Frauenfelder's reported values, the comparison of D-values is quite good in this temperature range.

TABLE 4
DIFFUSION COEFFICIENTS FOR NITROGEN IN TUNGSTEN

Temperature	Present Work ^a	Frauenfelder ^b
2000	5 x 10 ⁻⁶	5.6 x 10 ⁻⁶
2400	1.5 x 10 ⁻⁵	4.6 x 10 ⁻⁵

^aDerived from the solubilities of Norton and Marshall and measured permeabilities

^bCalculated from $D = 5.4 \exp(-62000/RT)$

The permeation of nitrogen through arc-cast tungsten is compared in Figure 5 to the permeation of hydrogen and oxygen through arc-cast tungsten.¹ The hydrogen results were obtained from the same permeation cells as used in the present work, but using a different detection technique. As is readily evident in the figure, nitrogen permeation coefficients in the temperature range 1800° to 2600°C are lower by two or more decades than the permeation coefficients for hydrogen or oxygen. An interpretation of these results highlights the importance of solubility and diffusion in permeation. Combining the available literature data for nitrogen and oxygen solubility and hydrogen diffusion with the permeation data obtained under this contract and the preceding contract, the summary data in Table 5 were obtained. The deduced diffusivities for oxygen and nitrogen in tungsten shown in the table are in the range expected based on the diffusivity of these interstitials in other bcc metals.⁴ On the basis that these diffusivities are reasonable, the more rapid permeation of oxygen (compared to nitrogen) can be attributed to a relatively high solubility. On the other hand, the much higher diffusivity of hydrogen (compared to nitrogen and oxygen), also observed for other bcc metals,⁵ is the primary cause of its higher permeation.

CONCLUSIONS

Permeation coefficients were determined experimentally for nitrogen permeation through arc-cast tungsten in the temperature range 1800° to 2600°C. Permeation rates were proportional to the difference between the square roots of nitrogen pressure on each side of the membrane and inversely proportional to membrane thickness. Thus, atomic nitrogen is the diffusing species and diffusion is the rate limiting step in this temperature range. Permeation coefficients were dependent on absolute temperature according to the equation:

$$P = 277 \exp(-70,000 \pm 800/RT) \text{ cc(STP)mm/cm}^2\text{-min-atm}^{1/2}$$

ACKNOWLEDGEMENTS

The authors are indebted to H. C. Brassfield for his advice and counsel, to A. F. Rosenberg and G. L. Murphy for their helpful advice and assistance in setting up the gas chromatograph, and to T. M. Welch for experimental assistance.

*Norton and Marshall published an activation energy value of 50.5 kcal/gram-atom for nitrogen diffusion in tungsten but they did not publish a pre-exponential value.

⁴David Lazarus, "Diffusion in Body-Centered Cubic Transition Metals—A Theoretical Critique," in *Diffusion in Body-Centered Cubic Metals*, p. 168, American Society for Metals, Metals Park, Ohio, 1965.

⁵L. N. Ryabchikov, "Mass Spectrometric Investigation of Degassing of Molybdenum, Tungsten, and Niobium on Heating Them in a Vacuum," *Ukr. Fiz. Zhur.*, 9, 293-302 (1964).

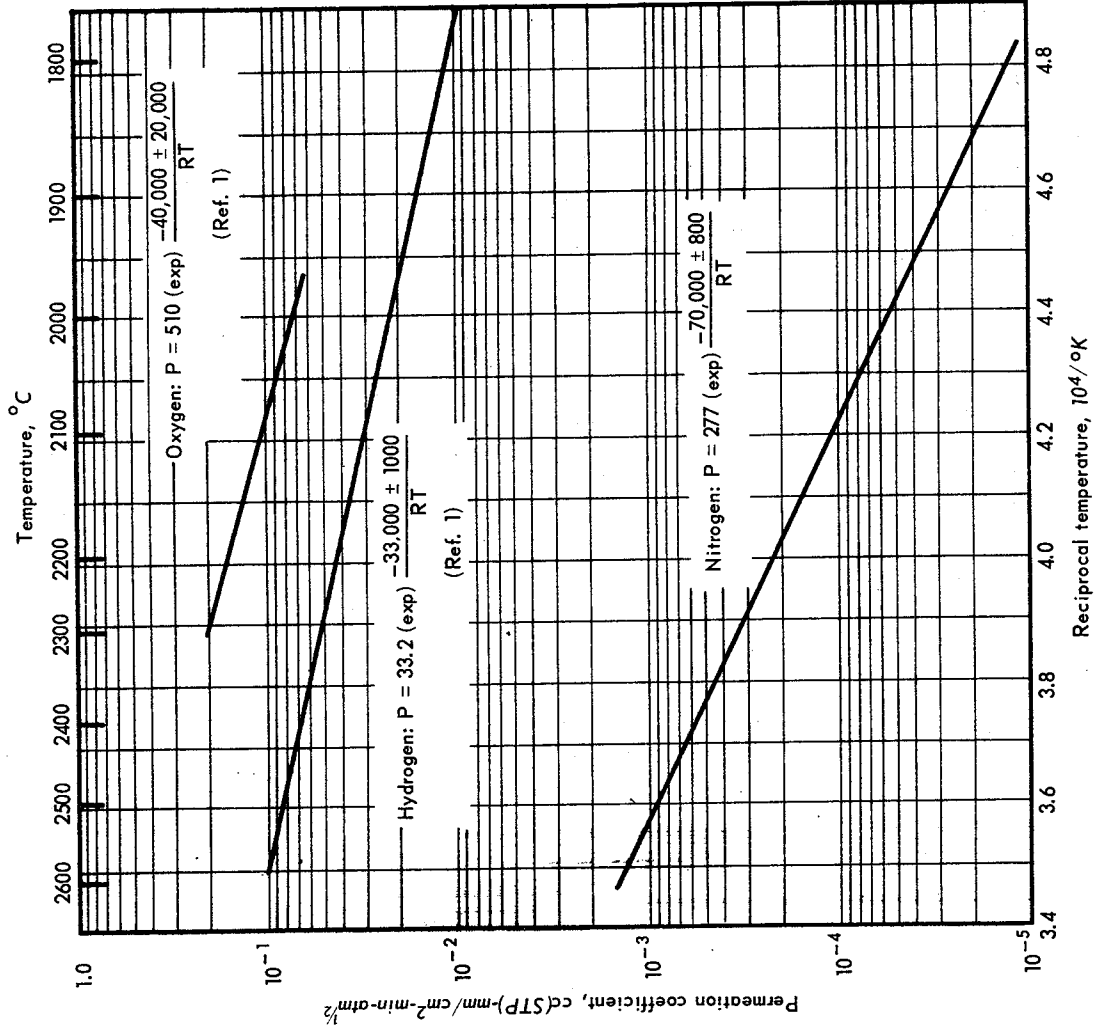


Fig. 5 — Comparison of permeation coefficients for nitrogen, hydrogen, and oxygen through arc-cast tungsten

SUMMARY OF PERMEATION, DIFFUSION, AND SOLUBILITY DATA FOR
NITROGEN, HYDROGEN, AND OXYGEN IN TUNGSTEN

TABLE 5

Permeation		Diffusion			Solubility			
Gas	P, atm and 1700°C	P _a	ΔH _p ^b	D, cm ² /sec at 1700°C	D _o ^c	ΔH _D ^d , 1 atm. and 1700°C	S, at 1700°C	
								Units of cc(STP)-mm/cm ² -min-atm ^{1/2}
Nitrogen	4.2 × 10 ⁻⁶	277	70.0 ± 8	1.6 × 10 ^{-6f}	7 × 10 ^{-3f}	33.1	0.0043	74.7
Hydrogen	7.2 × 10 ⁻³	33.2	33.0 ± 1	5.35 × 10 ⁻⁴	.081 ^g	19.8 ^g	.021 ^f	26 ^f
Oxygen	1.2 × 10 ⁻²	510	40.0 ± 20	3 × 10 ^{-8f}	(h)	(h)	0.54 ^f	(h)

^aUnits of cc(STP)-mm/cm²-min-atm^{1/2}
^bUnits of kcal/g-atom (± values are 2σ limits)
^cUnits of cm²/sec
^dUnits of cc(STP)/cm³ of W
^eUnits of kcal/mole
^fDerived from P = DS
^gRyabchikov, L. N., Ukr. Fiz. Zhur., 9, 293-302 (1964)
^hOnly solubility for oxygen found in literature was at 1700°C
ⁱSolubility at estimated pressure of 7 × 10⁻⁷ atm
^jUnits of kcal/g-atom

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