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DETERMINATION OF TRITIUM PERMEATION RATE THROUGH T-22 IN GCFR HELIUM ENVIRONMENT

by
L. YANG and W. A. BAUGH

Prepared under
Contract DE-AT03-76SF71023
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ABSTRACT

Measurements are made on tritium permeation rates through T-22 tubular samples in the temperature range of 300° to 550°C. The tritium source consists of tritium in helium containing hydrogen at 6 vol % and sufficient water vapor to maintain a hydrogen-to-water-pressure ratio of 10, the total pressure being 1.01×10^5 Pa (1 atm). Two tritium sources at specific activities of 2.6×10^{-3} and 3.1×10^{-2} $\mu\text{Ci}/\text{std cc}$, respectively, are used for determining how the permeation rate varies with tritium concentration. The T-22 tubular samples have a wall thickness of 0.437×10^{-2} m (0.172 in.), and two samples are used for checking the reproducibility of the results. During the measurements, the tritium diffusing through the wall of the sample is swept out with a helium-steam mixture by bubbling helium at atmospheric pressure through a water reservoir maintained at 90°C.

Examination of the surface of the sample after the measurements shows the presence of a thin dark film (less than a few microns thickness) on the wet helium-hydrogen-tritium side, which has higher chromium, molybdenum, and silicon concentrations than that in the bulk of the T-22 substrate. Under the dark film there is a decarburized layer about 75 μm thick. Examination of the steam side shows the presence of an oxide layer 15 μm thick, which is mainly Fe_3O_4 , and no appreciable decarburization in the metal.

The permeation results, when plotted in the form of an Arrhenius plot, do not fall on straight lines, indicating changes in the controlling factors on the permeation process as the temperature changes. This is attributed to the instability of Fe_3O_4 on the surface of the sample at the wet helium-hydrogen-tritium side at high temperatures, as shown by thermodynamic analysis.

For the temperature range studied, the permeation rates for the two tritium sources differ by a factor of 4 to 6, while the specific activities of the two tritium sources differ by a factor of 12. This implies that the increase of permeation rate with tritium activity follows neither the linear nor the square-root relationship.

1. INTRODUCTION

In the selection of steam generator material for gas-cooled fast breeder reactor (GCFR) application, both T-22 and Incoloy 800 are under consideration. While T-22 is favored from the point of view of stress corrosion and cost, its higher tritium permeability represents a major concern. During the past few years, the permeation behaviors of hydrogen isotopes through T-22 and Incoloy 800 have been studied by various authors (Refs. 1-4). It was observed that the presence of surface films, such as that formed by interaction with steam, could have a profound effect on the tritium permeation rates through these materials. Reduction of permeation rates by two orders of magnitude has been reported.

Since the GCFR helium environment has low hydrogen-to-water-pressure ratio (<10), and the inside surface of the steam generator tubes is exposed to steam at high temperatures, the presence of oxide films on both inside and outside diameters is anticipated, and the tritium permeation rate is therefore expected to be lower than that for unoxidized T-22 metal. Thus, quantitative information on the tritium permeation rates through such oxidized T-22 in a helium environment of GCFR composition is needed for assessing whether T-22 can meet the environmental safety requirements. Such information, however, cannot be deduced accurately from existing literature data obtained from samples and tritium sources different from GCFR design. This is due to the fact that the laws governing the change of tritium permeation rate with these parameters are still not very well established, since they are strongly dependent on the surface condition of the sample and the tritium concentration and impurity contents of the tritium source. To obtain useful design data, it is therefore necessary to make direct measurements with tritium sources and sample dimensions similar to those of GCFR design.

2. EXPERIMENTAL

2.1. TEST SAMPLES, TRITIUM SOURCES, AND TEST TEMPERATURES

Measurements of the tritium permeation rates were made on two T-22 tubular samples obtained from the same T-22 tube containing the following concentrations (in weight %) of major alloying elements: carbon, 0.10; manganese, 0.52; phosphorus, 0.01; sulfur, 0.01; silicon, 0.36; molybdenum, 0.95; and chromium, 2.19. Each sample has the following dimensions: length, 15.24×10^{-2} m (6 in.); o.d., 2.515×10^{-2} m (0.990 in.); i.d., 1.641×10^{-2} m (0.646 in.); and wall thickness, 0.437×10^{-2} m (0.172 in.). The surfaces of these samples were polished with fine (4-zero grade) emery paper and degreased with xylene prior to installation in the permeation apparatus.

The tritium source used consists of helium containing hydrogen (6 vol %) and tritium at concentrations of GCFR interest. Two tritium sources were prepared for this work, which have specific activities of 2.6×10^{-3} and $3.1 \times 10^{-2} \mu\text{Ci}/\text{std cc}$, respectively, to provide information on variations in permeation rate with tritium concentration. Prior to entering the test chambers, each tritium source passes through an ice-cooled water saturator to attain a hydrogen-to-water-pressure ratio of 10. This hydrogen-to-water-pressure ratio is selected because it represents the upper bound of the hydrogen-to-water-pressure ratios of GCFR interest and therefore the least oxidizing GCFR helium environment. It is believed that if the tritium permeation rates obtained for such a hydrogen-to-water-pressure ratio are acceptable, there should not be any problem with lower (more oxidizing) ratios.

Measurements were carried out at 300° , 375° , 450° , and 550°C , with the tritium source flowing past the o.d. of the T-22 tubular sample at atmospheric pressure and a flow rate of 100 cc/min. The tritium diffusing

through the wall of the sample was swept out with a helium-steam mixture prepared by bubbling 99.999% pure helium at atmospheric pressure and a rate of 100 cc helium/min through a water reservoir maintained at 90°C. The experimental arrangement and the procedures for determining the tritium content of the helium-steam purge gas are described below.

2.2. EXPERIMENTAL ARRANGEMENT

The experimental arrangements for measuring the tritium permeation rates are shown in Fig. 1. The sample tube is welded to an Incoloy 800 end cap on one end and to an Incoloy 800 sample pedestal on the other end. The sample pedestal is then welded to an Incoloy 800 inner purge pedestal, which is, in turn, welded to an Incoloy 800 outer purge pedestal. The inlet and the outlet of the inner purge tubes, which carry the helium-steam mixture used for sweeping out the tritium that diffuses through the sample tube wall, are welded to the top of the inner purge pedestal. To insure thorough sweeping, the outlet of the inner purge tube is extended to the top of the sample tube, while the inlet stays at the bottom. The outer purge line isolates the tritium source from the inner purge stream. Any tritium diffusing from the tritium source through the outer purge tube is swept out with helium. Without such an outer purge line, cross contamination from the tritium source is possible as a result of diffusion through the inner purge tube wall, which would thus introduce errors into the tritium permeation measurements. Specially designed purge flanges and copper gaskets join the outer purge line to a helium source and the inner purge line to the helium-steam source. Any possible leaks in the valve systems between the tritium source and the inner purge line are isolated with helium purge.

The whole sample assembly is situated in a high-purity recrystallized-alumina (Morganite) permeation chamber inside a nichrome resistance furnace. By adjusting the power inputs in the four separate windings of the furnace, the temperature of the sample, as indicated by three Chromel-Alumel thermocouples (tc_1 , tc_2 , tc_3), can be kept uniform to within $\pm 2^\circ\text{C}$ over its entire length.

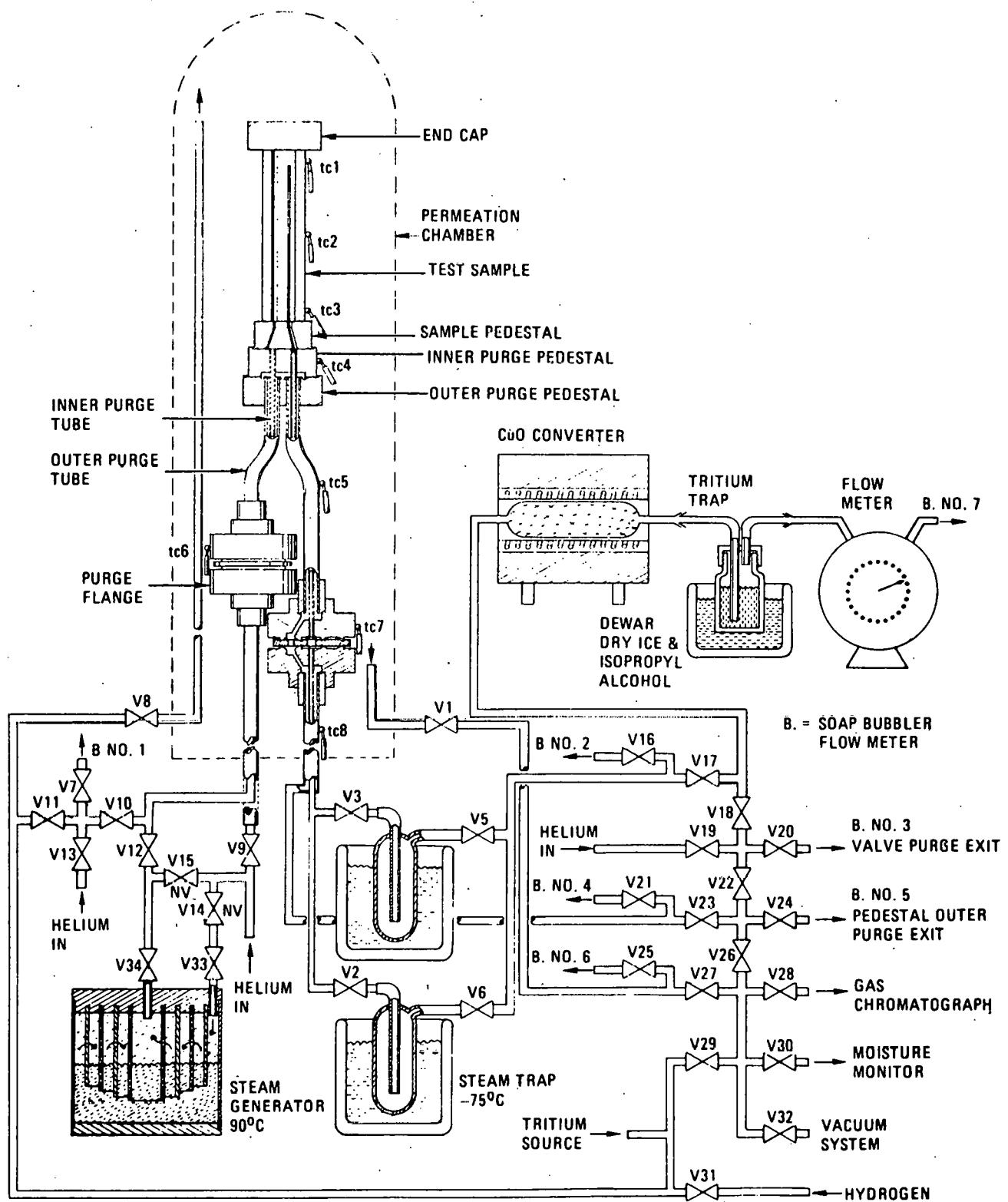


Fig. 1. Experimental arrangements for the study of tritium permeation through T-22 tubular samples

Manifolds at the bottom of the permeation chamber connect the system to (1) inlet and outlet of the tritium source, (2) inlet and outlet of the helium outer purge line, and (3) inlet and outlet of the helium-steam inner purge line. The impurity contents in the tritium source can be measured with a gas chromatograph prior to entering the ice-cooled water saturator (not shown in Fig. 1). A dew point moisture monitor is used to determine the water content in the tritium source both before and after it has passed through the permeation chamber.

During the run, the tritium source flows continuously through the chamber at a rate of about 100 cc/min. Any tritium diffusing through the wall of the sample is swept out with a helium-steam purge at atmospheric pressure, prepared by passing 99.999% pure helium through a water reservoir maintained at 90°C. After leaving the permeation chamber, the helium-steam inner purge stream passes through a cold trap to freeze the steam, which now contains both water and tritiated water formed by the exchange of tritium with water. The helium leaving the cold trap flows through a CuO converter to oxidize the remaining tritium into tritiated water, which is then collected in a liquid scintillation medium that has been cooled with an isopropyl-alcohol - dry-ice mixture. The total volume and the flow rate of the exit helium gas during a run are measured with a rotary gas meter and a soap-bubble flow meter, respectively.

A vacuum system consisting of a liquid-nitrogen-trapped oil diffusion pump backed with a rotary mechanical pump is connected to the system for evacuating the gas lines and outgassing the sample prior to each run. A hydrogen source is also connected to the system, which is used to remove the oxide films that have formed on the sample surface during mechanical polishing.

2.3. EXPERIMENTAL PROCEDURES

After the sample assembly was installed in the permeation chamber, the Chromel-Alumel thermocouples were spot-welded to the sample surface and the pedestals (see Fig. 1). The Al_2O_3 envelope was then lowered over the sample

assembly and joined to the base of the permeation chamber by an O-ring Cajon fitting. The permeation chamber and all the gas lines (except the steam generator) were evacuated to a residual gas pressure of less than 1.33×10^{-3} Pa (10^{-5} torr). The steam generator and the outer purge line were then purged with helium, and the nichrome resistance furnace (not shown in Fig. 1) was lowered over the Al_2O_3 envelope. The temperature was raised to the highest value for which the tritium permeation rate was to be measured, and the sample was heated in flowing hydrogen for 4 hours to remove any oxide film on the outside surface of the tubular sample. At the same time, the helium-steam mixture was admitted into the inner purge line, passing through the inside of the sample tube, the cold trap, the CuO converter, and the flowmeter.

At the completion of the hydrogen treatment the sample was outgassed for 4 hours. Afterwards it was cooled to room temperature in vacuum, and the tritium source was admitted into the permeation chamber. The inner purge helium-steam mixture was allowed to flow for 4 hours. The condensate in the cold trap and the cooled liquid scintillation fluid in the tritium trap were checked for tritium activity. The absence of tritium activity above background indicated little or no leakage in the sample, the welds, and the copper gasket of the purge flanges, and showed the effectiveness of the outer purge system.

The temperature of the sample was then raised to the desired value. The system was allowed to equilibrate for at least 24 hours before trapping of the tritium in the helium-steam purge stream began. Usually two or three successive trappings were made, with each lasting from 2 to 4 hours.

After the completion of each trapping, both the steam condensate in the cold trap and the liquid scintillation fluid in the tritium trap were collected and each was counted for tritium activity by liquid scintillation techniques. The amount of tritium present (in microcuries) was calculated

from the counting rate (in counts per minute) by the equation

$$\text{amount of tritium in microcuries} = \frac{(\text{dpm})}{2.22 \times 10^6} = \frac{(\text{cpm})}{y \times 2.22 \times 10^6}$$

where 2.22×10^6 = conversion factor from disintegrations per minute (dpm) to microcuries of tritium,

y = counting efficiency = 0.357, as determined with a tritium standard for the counting geometry used.

The tritium permeation rate of the sample was calculated by the equation

$$\text{tritium permeation rate} = \frac{(\mu\text{Ci})_1 + (\mu\text{Ci})_2}{At}$$
$$(\mu\text{Ci}/\text{m}^2 \cdot \text{h})$$

where $(\mu\text{Ci})_1$ = tritium in steam condensate in microcuries,

$(\mu\text{Ci})_2$ = tritium in cooled liquid scintillation fluid in tritium trap in microcuries,

A = effective area of the sample, $93.1 \times 10^{-4} \text{ m}^2$, which is calculated from the formula

$$A = \frac{2 \ell (R_2 - R_1)}{\ln R_2 - \ln R_1}$$

where ℓ = length, R_2 = o.d./2, and R_1 = i.d./2,

t = time of trapping in hours.

In all cases studied, more than 90% of the tritium activity was in the steam condensate, indicating rapid exchange between tritium and water at the test temperatures.

3. RESULTS AND DISCUSSION

3.1. TRITIUM PERMEATION RATES

The measured tritium permeation rates are listed in Table 1. For each tritium source, the mean values of the observed permeation rates obtained for these two samples are plotted in Fig. 2 versus the reciprocals of the absolute temperatures under which these measurements were made. The plots are not straight lines as typical Arrhenius plots should be, indicating changes in the controlling factors of the permeation rates as the temperature changes. For the same temperature, the observed permeation rates for the two tritium sources differ by a factor of 4 to 6 for the temperature range studied. Since the specific activities of these two tritium sources differ by a factor of 12, this implies that the increase of permeation rate with tritium activity follows neither the linear nor the square-root relationship.

3.2. EXAMINATIONS OF TEST SAMPLES AFTER MEASUREMENTS OF PERMEATION RATES

Figure 3 shows the appearance of test sample 1 after the measurements of the permeation rates. The external surface of the sample, including that of the Incoloy 800 cup and the Incoloy 800 pedestal, is covered uniformly with a dark film. The post-test appearance of sample 2 is similar to that of sample 1.

Figure 4 shows the typical appearance of a cross section of the steam-side surface of the test sample. An oxide layer of about $15 \mu\text{m}$ thickness has been formed and no appreciable decarburization can be detected. At 1000x magnification, a thin white layer is present on the top of the oxide layer. X-ray study indicates that the oxide layer has the Fe_3O_4 magnetite structure. Electron microprobe analysis shows that while the oxide layer

TABLE 1
TRITIUM PERMEATION RATE THROUGH T-22

Tritium Source Specific Activity (μ Ci/std cc)	Temperature ($^{\circ}$ C)	Tritium Permeation Rate (μ Ci/ m^2 . h)			
		Sample 1	Sample 2	Mean	Standard Deviation
2.6×10^{-3}	550	2.22, 4.80,	2.32, 4.95,	3.57	1.50
	450	0.57, 0.45, 0.83,	0.53, 0.77, 0.40	0.59	0.17
	375	0.12, 0.16, 0.20,	0.10, 0.16, 0.27,	0.17	0.06
	300	0.04, 0.07	0.04, 0.08	0.06	0.02
3.1×10^{-2}	550	15.9, 17.8	14.6, 13.8	16.0	2.70
	450	3.44, 2.50, 2.80,	5.01, 4.04, 3.00,	3.46	0.93
	375	0.57, 0.69, 1.03,	0.82, 0.95, 1.08,	0.86	0.20
	300	0.26, 0.27	0.29, 0.18	0.25	0.05

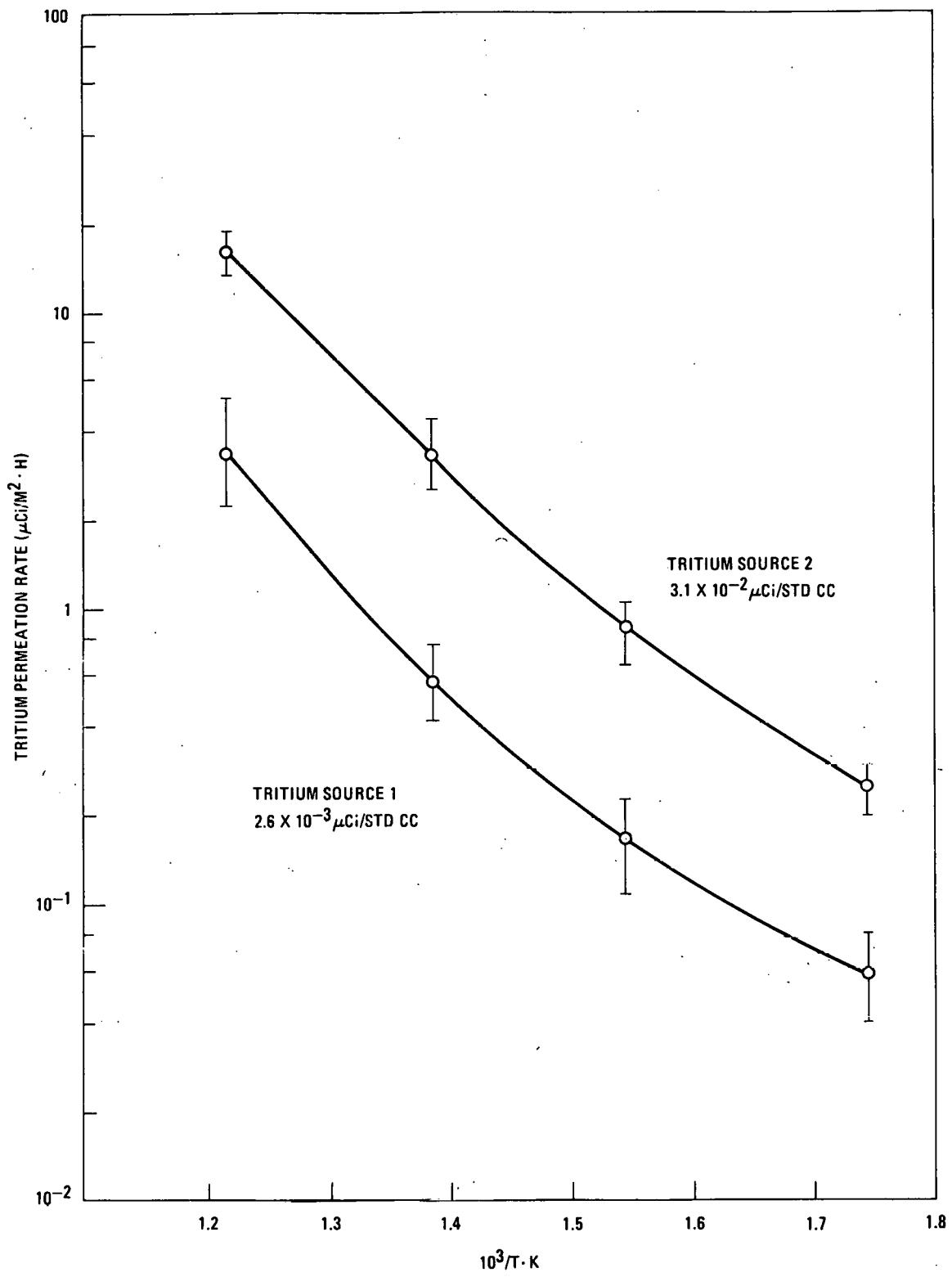


Fig. 2. Tritium permeation rate through T-22. The ranges shown are standard deviations.

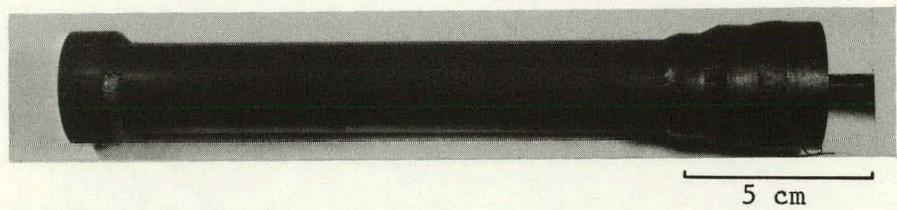
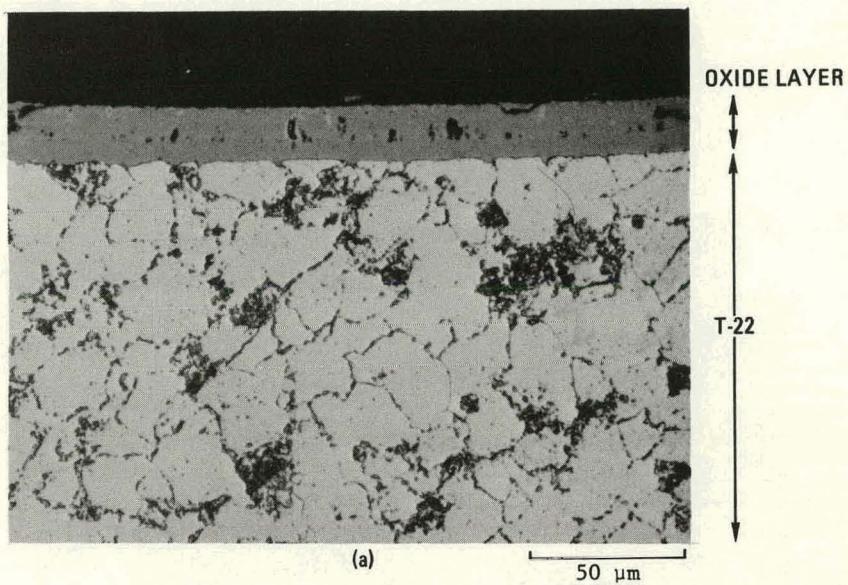
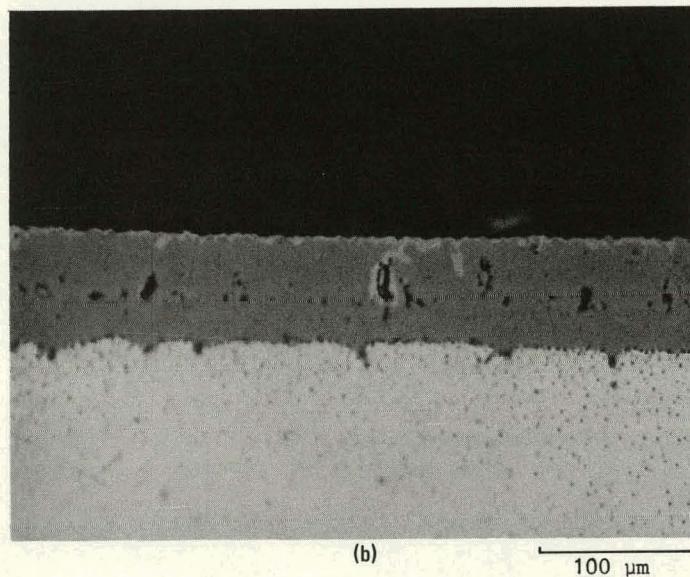


Fig. 3. Appearance of the sample after test. Note the dark film on the surface.



Etched. Note the oxide layer (15 μm thick) and no appreciable decarburization.



Unetched. Note the white thin layer on top of the oxide layer.

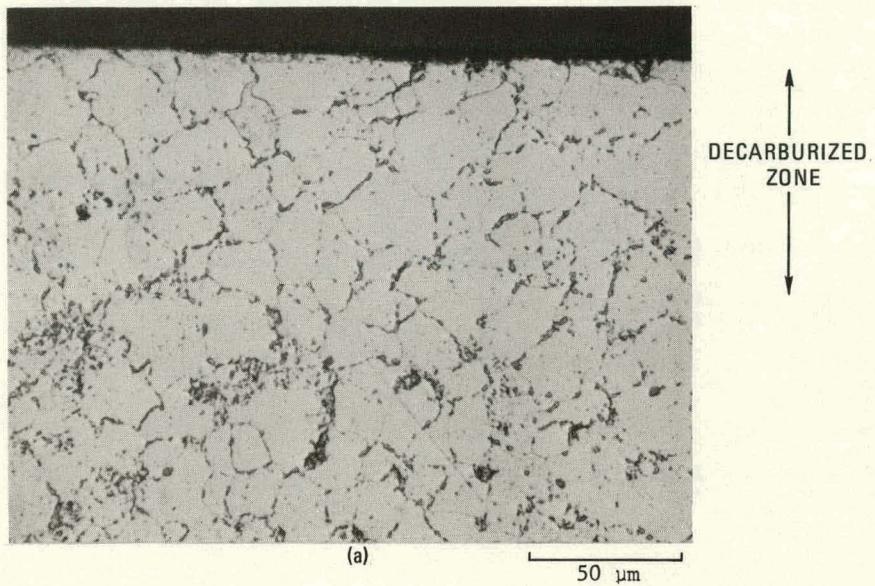
Fig. 4. Typical appearance of a cross section of the steam side surface of the test samples

adjacent to the metal surface contains higher chromium (5% versus 2.2%) and molybdenum (2% versus 1%) concentrations than the bulk metal, the major part of the oxide layer consists of Fe_3O_4 . The thin white layer on top of the oxide layer has high silicon (3.5%) and aluminum (0.8%) contents.

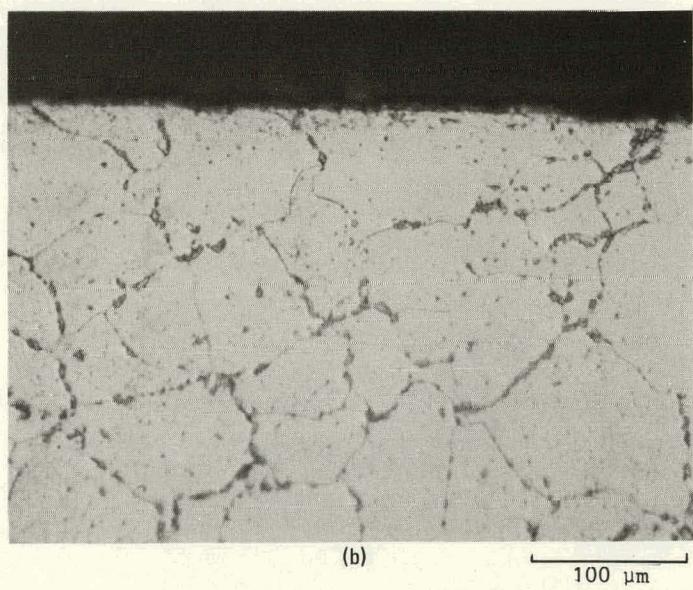
Figure 5 shows the typical appearance of the surface of the test sample on the wet helium-hydrogen-tritium side. A decarburized layer extends into the metal for a distance of about 75 μm . The dark surface film layer is barely visible at 1000x magnification. To gain information on the composition of this thin dark film formed in wet helium-hydrogen-tritium mixture, four samples were taken from different locations of sample 1 and examined by EDAX techniques, using low-energy (~ 10 keV) electron beam to minimize interference by the metal substrate. The results, together with that obtained on the metal substrate, are shown in Table 2.

It is clear that the dark surface film formed in the wet helium-hydrogen-tritium mixture has higher chromium, molybdenum, and silicon concentrations than that in the metal substrates. The test sample has been exposed to temperatures ranging from 300° to 550°C. It is possible that the composition of this film changes with temperature. Detailed studies of the change of the surface film composition with temperature, however, are beyond the scope of this work. Nevertheless, since iron is the major component of this film, it would be of interest to deduce the stability of various iron oxides in the testing environment from known thermodynamic data (Ref. 5).

Table 3 lists the calculated critical $\text{pH}_2/\text{H}_2\text{O}$ ratios in the testing environment above which the various iron oxides are unstable. The most stable oxide is Fe_3O_4 . For $\text{pH}_2/\text{pH}_2\text{O} = 10$, which is the ratio in the testing environment for this work, Fe_3O_4 is stable only at 300°C and 375°C, but not at 450°C and 550°C. This may be the reason why the tritium permeation rate increases more than that called for by the Arrhenius equation as the temperature increases (see Fig. 2).



Etched. Note the decarburized zone. The dark surface film is too thin to show in this picture.



Etched. The dark surface film is barely visible.

Fig. 5. Typical appearance of a cross section of the test sample surface on the wet helium-hydrogen-tritium side

TABLE 2
 CONCENTRATIONS OF METALLIC ELEMENTS IN THIN DARK FILM FORMED
 ON SURFACE OF TEST SAMPLE ON WET HELIUM-HYDROGEN-TRITIUM SIDE
 (Concentration in wt %)

Sample No.	Cr	Mo	Si	Al	Fe
1	5.2	1.8	6.1	0.4	86.4
2	6.3	1.8	7.3	0.4	84.3
3	5.4	1.7	7.4	0.3	85.1
4	6.6	2.0	7.3	0.5	83.6
Metal substrate	2.0	0.7	0.7	0.9	95.7

TABLE 3
 CRITICAL p_{H_2}/p_{H_2O} RATIOS ABOVE WHICH VARIOUS IRON OXIDES
 BECOME UNSTABLE AT 300° , 375° , 450° , and $550^\circ C$, RESPECTIVELY^(a)

Temp ($^\circ C$)	Fe_3O_4	FeO	Fe_2O_3
300	36.1 (S)	14.0 (S)	6.4 (N)
375	16.7 (S)	9.0 (N)	3.4 (N)
450	9.1 (N)	6.4 (N)	2.0 (N)
550	4.8 (N)	4.4 (N)	1.2 (N)

(a) S signifies oxide is stable at $p_{H_2}/p_{H_2O} = 10$; N signifies oxide is not stable at $p_{H_2}/p_{H_2O} = 10$.

While the thin oxide film on the wet helium-hydrogen-tritium side may change with temperature, the thick Fe_3O_4 layer on the steam side should remain stable in the testing temperature range since the p_{H_2}/p_{H_2} value in the helium-steam inner purge gas should be very low, although cracking of the Fe_3O_4 layer may occur upon thermal cycling. Both the surface film on the tritium side and the Fe_3O_4 layer on the steam side should contribute to the reduction of tritium permeation, but individual contribution from each cannot be estimated from the results of this work.

3.3. EFFECT OF OXIDES FORMED IN TESTING ENVIRONMENT ON TRITIUM PERMEATION RATE

The effect of the oxides formed in the testing environment on tritium permeation rate can be estimated by comparing the results shown in Fig. 2 with the tritium permeation rates through clean T-22. The latter can be calculated from hydrogen permeability through clean T-22 on the basis of thermodynamic and kinetic considerations of the permeation process. Figure 6 shows the measured hydrogen permeability through clean T-22 at various temperatures and hydrogen pressures (Ref. 1). The tritium permeation rates through clean T-22 for the tritium activities and sample geometries used on this work can be calculated as follows:

$$\frac{\text{tritium permeability}}{\text{hydrogen permeability}} = \frac{C_T}{C_H} \cdot \frac{1}{\sqrt{3}} \quad (1)$$

where C_T and C_H are tritium and hydrogen atom concentrations, respectively, and $\sqrt{3}$ represents the correction due to the difference between the tritium atom mass and the hydrogen atom mass. Also,



$$K_1 = \frac{C_H^2}{p_{H_2}} \quad , \quad (4)$$

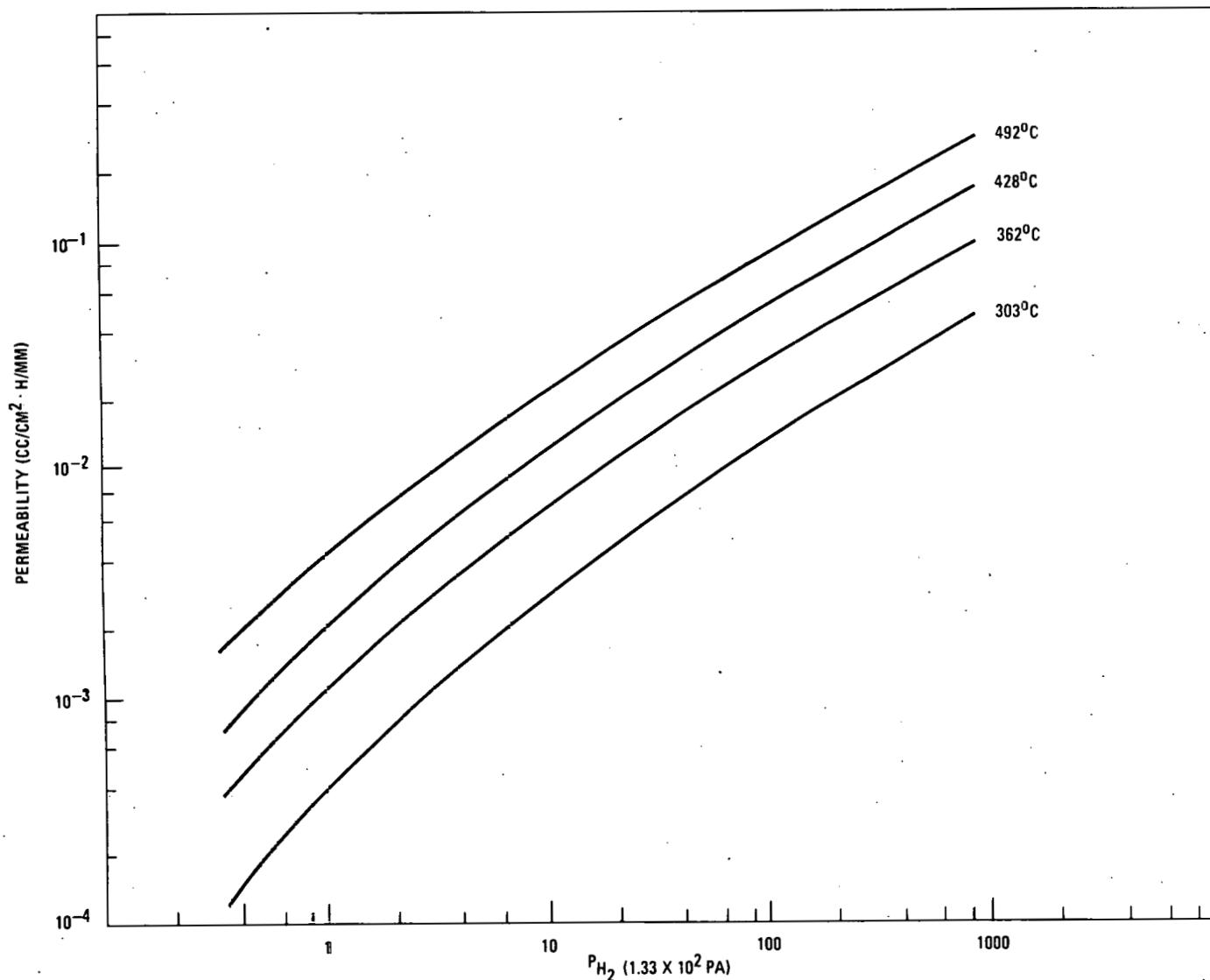


Fig. 6. Hydrogen permeability through T-22 at various hydrogen pressures and temperatures

$$K_2 = \frac{C_H C_T}{P_{HT}} \quad (5)$$

One can combine 1, 4, and 5, and since $K_1 = 2 K_2$,

$$\text{tritium permeability} = \text{hydrogen permeability } P_{HT} / (2\sqrt{3} P_{H_2}) \quad (6)$$

where P_{HT} and P_{H_2} are the partial pressures of H_2 and HT in the helium environment.

For the testing environment in this work, $P_{H_2} = 6.08 \times 10^3$ Pa (45.6 torr) (6 vol %), $P_{HT} = 2.05 \times 10^{-4}$ Pa (1.54×10^{-6} torr) for tritium source 1 (2.6×10^{-3} μ Ci/std cc), and $P_{HT} = 2.45 \times 10^{-3}$ Pa (1.84×10^{-5} torr) for tritium source 2 (3.1×10^{-2} μ Ci/std cc). The tritium permeabilities are calculated using Eq. 6 and hydrogen permeabilities from Fig. 6 for the temperatures listed in that figure. These tritium permeabilities are then converted to tritium permeation rates in the unit shown in Fig. 2 for the sample wall thickness (4.37 mm) used in this work, assuming that the permeation rate varies inversely with the wall thickness. Such an assumption is probably true for clean metals. The results are given in Table 4.

The results in Table 4 show the effect of the oxide films formed under the testing conditions on the tritium permeation rates. The reason for the higher reduction factor for the tritium source of higher activity is that according to Eq. 6, which was used to calculate tritium permeability through clean T-22, the tritium permeability varies linearly with tritium activity (i.e., P_{HT}), while experimentally the measured tritium permeation rate does not vary linearly with tritium activity (see Fig. 2). The reason for this difference is a matter requiring further investigations. Caution should therefore be exercised in using these reduction factors. A more direct way to determine the reaction factor is to measure the tritium permeation rate through the samples used in this work under clean conditions, i.e., no moisture present on the tritium source side and no steam on the tritium

TABLE 4
CALCULATED PERMEATION RATES OF TRITIUM THROUGH CLEAN T-22 FOR TESTING
SAMPLE WALL THICKNESS (4.37×10^{-3} m) AT VARIOUS TEMPERATURES

Temperature (°C)	Hydrogen Permeabilities (From Fig. 6) (cc/cm ² ·h/mm)	Calculated Tritium Permeation Rates ($\mu\text{Ci}/\text{m}^2 \cdot \text{h}$)		Measured Tritium Permeation Rates Through Oxidized T-22 in Testing Environment of This Work (Interpolated from Fig. 2) ($\mu\text{Ci}/\text{m}^2 \cdot \text{h}$)		Factor of Reduction of Tritium Permeation Rate by Oxide Films	
		Tritium Source 1 (a)	Tritium Source 2 (b)	Tritium Source 1 (a)	Tritium Source 2 (b)	Tritium Source 1 (a)	Tritium Source 2 (b)
303	8.0×10^{-3}	0.47	5.56	0.06	0.26	7.8	21.4
362	1.9×10^{-2}	1.11	13.2	0.13	0.64	8.5	20.6
428	3.4×10^{-2}	1.98	23.6	0.39	2.3	5.1	10.3
492	5.6×10^{-2}	3.27	38.9	1.25	6.6	2.6	5.9

(a) Tritium source 1 = 2.6×10^{-3} $\mu\text{Ci}/\text{std cc}$.

(b) Tritium source 2 = 3.1×10^{-2} $\mu\text{Ci}/\text{std cc}$.

exit side. This work should be included in any future study on tritium permeation behaviors.

The data in Fig. 2 can be used to calculate the total tritium release rate per year from a GCFR on the basis of the area of the steam generator tubes and the temperature distribution. The results should indicate whether the release rate meets the environmental requirements.

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