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MEASUREMENTS OF THE HYDROGENIC RECOMBINATION COEFFICIENT FOR THE TFTR VACUUM VESSEL

By

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MEASUREMENTS OF THE HYDROGENIC RECOMBINATION COEFFICIENT FOR THE TFTR VACUUM VESSEL

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ABSTRACT

Characteristic values of the recombination rate coefficient for hydrogen and deuterium in stainless steel have been measured for the inner wall of the TFTR vacuum vessel for vessel temperatures of 25-100 C. In situ measurements of k_r are important for predicting the hydrogen isotope retention in the wall as a function of time, temperature, and discharge exposure, particularly because existing laboratory measurements of k_r for stainless steel span a range of four orders of magnitude. The measurement technique involved the observation of the decrease in hydrogen pressure during a glow discharge in the TFTR vacuum vessel with an initial static gas fill. The resulting values of k_r at 25 C are in the range of $(0.4\text{-}4) \times 10^{-27} \mathrm{cm}^4\text{-}\mathrm{s}^{-1}$ assuming a value of the hydrogenic diffusivity of $2 \times 10^{-12} \mathrm{cm}^2\text{-}\mathrm{s}^{-1}$ at room temperature. No significant isotopic dependence was observed and the temperature dependence of k_r is consistent with the literature value (0.5 eV) of the activation energy. The implications of this range of values of k_r, for the estimation of the in-vessel tritium inventory following D-T operation in TFTR are discussed.

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I. INTRODUCTION

We report on the first measurements of the characteristic value of the hydrogenic recombination rate constant k_r, for the inner wall of the TFTR vacuum vessel. For austenitic stainless steels near or above room temperature, the rate-limiting process for hydrogen re-emission is surface recombination.[1,2] The value of k_r characteristic of the stainless steels commonly used for first wall construction in fusion devices is an important parameter since it will determine: (1) hydrogen isotope recycling rates; (2) isotopic replacement rates; and (3) wall retention. The latter topic is of particular importance for a device like TFTR which is required to operate with a minimal tritium wall inventory during its first phase of D-T fusion experiments. The need for in situ measurements of k_r within TFTR became apparent when literature values of k_r were examined. Laboratory measurements[1] of k_r showed a wide variation depending on the surface conditions of the stainless steel test samples. In situ measurements generate the most appropriate value of k_r, averaged over the vessel surface area and surface condition, for estimating hydrogen re-emission and retention.

In this paper measurements are presented of the quantity k_r/D, (where D is the hydrogenic diffusivity), for the 304LN stainless steel wall of TFTR. The first measurements were performed after the initial vacuum vessel conditioning, and subsequent measurements were obtained during the first operational phase with ohmically heated deuterium plasmas. Measurements of k_r/D were obtained for vessel temperatures of 25, 50, and 100 C.

The results of in situ TFTR measurements yield values of k_r/D ($\approx 10^{-15} cm^2$ at 300 K) which are consistent with the laboratory measurements of Waelbroeck et al.[3] for glow discharge-cleaned stainless steel. This is not surprising since glow discharge cleaning was the primary cleaning technique used to condition the TFTR vacuum vessel prior to first plasma operation.[4] The TFTR values of k_r are also consistent with the recent *in situ* measurements for the 304 stainless steel vessel of ISX[5] and the Inconel liner of TEXTOR.[6]

Details of the measurement technique and the results are presented in the next two sections. In the final section of the paper, the implications of the obtained k_r/D values for TFTR operation are briefly examined. Of primary importance is the conclusion that the planned operating scenarios will result in a moderate (~ kCi) in-vessel tritium inventory.

II. EXPERIMENTAL METHOD

The technique for measuring a value of k_r characteristic of the surface conditions of TFTR involved the observation of the decrease in pressure during the application of glow discharge. The technique was first described by Waelbroeck *et al.*,[3] and this group has recently applied the methodology to *in situ* measurements in TEXTOR.[6] The TFTR measurements employ a variation of the technique described by Howe and Langley,[5] that was used to obtain *in situ* measurements of k_r in the ISX device.

In the method of Howe and Langley (HL), the pressure decrease during the application of a constant current glow discharge is monitored following an initial static fill (p_o) of hydrogen. As molecular hydrogen is ionized in the glow discharge and accelerated into the vessel surface (with a flux ϕ_g) the resulting pressure decrease can be related to the change in hydrogen concentration in the wall.

$$p = p_o - \frac{\Delta N_w}{2} kT \tag{1}$$

where ΔN_w is the depth integral of the hydrogen concentration $c(y,t)$:

$$\Delta N_w = \int_o^\infty c(y,t)\, dy \quad . \tag{2}$$

$c(y,t)$ is a solution of the diffusion equation which is consistent with the boundary conditions at the surface ($y = 0$) during the glow discharge experiment. HL show that for stainless steel, at temperatures at or above 300 K, the implanted glow discharge (atomic) flux ϕ_d and the desorbed molecular flux, ϕ_m which is related to the near surface concentration of hydrogen, c_o, by the definition

$$\phi_m = k_r c_o^2 \tag{3}$$

are rapidly equilibrated, and thus:

$$2\phi_d = \phi_m \tag{4}$$

In addition, if the initial hydrogen wall loading is assumed to be zero, the details of the function c(y,t) are unimportant for these experiments and c(y,t) takes the simple form:

$$c(y,t) = c_o \operatorname{erfc}\left[\frac{y}{\sqrt{4Dt}}\right] \tag{5}$$

Thus,

$$\Delta N_w(t) = A \int_0^\infty c(y,t)dy = \frac{2A}{\pi^{1/2}} c_o(Dt)^{1/2}$$

$$= A\left(\frac{2}{\pi} \frac{D\phi_d}{k_r}\right)^{1/2} t^{1/2} \quad , \tag{6}$$

and substituting this result back into Eq. (1),

$$p = p_o - \frac{A}{2V} kT \left(\frac{2D\phi_d}{\pi\ k_r}\right)^{1/2} t^{1/2} = p_o - \beta t^{1/2} \tag{7}$$

yields the relation described by HL for the observed proportionality of the pressure drop Δp and t, to the characteristic value of k_r/D:

$$\frac{\Delta P}{t^{1/2}} = \frac{p_o - p}{t^{1/2}} = \beta = \frac{AkT}{2V} \left(\frac{2D\phi_d}{\pi\ k_r}\right)^{1/2} \quad . \tag{8}$$

For the TFTR measurements, the analysis leading up to Eq. (7) required modification to account for several experimental complications which occurred with the higher temperature measurements. The first problem concerned the nonuniformity of vessel temperature during vessel bakeout. At the time of these measurements, only the TFTR torus, and not the relatively large vacuum pumping ducts, could be elevated in temperature. The pumping ducts enclose an area approximately equal to 40% to the total vessel area. Thus, Eq. (8) was modified to include the effects of the two surfaces: the pumping duct of surface area A_1, characterized by room temperature (T_1) values of $k_r^{(1)}$ and $D^{(1)}$; and the torus surface area, A_2, characterized by high temperature (T_2) values of $k_r^{(2)}$ and $D^{(2)}$.

$$p = p_o - \beta_1 t^{1/2} - \beta_2 t^{1/2}$$

$$= p_o - \left[\frac{A_1}{2V} kT_1\left(\frac{2D^{(1)}\phi_d}{\pi\, k_r^{(1)}}\right) + \frac{A_2 kT_2}{2V}\left(\frac{2D^{(2)}\phi_d}{\pi\, k_r^{(2)}}\right)\right] t^{1/2} \quad . \qquad (9)$$

The experimental quantity that is measured in the high temperature measurements is the proportionality factor $\beta_m = \beta_1 + \beta_2$. The value of $k_r^{(2)}/D^{(2)}$ is easily extracted assuming a reliable measurement of the proportionality factor at room temperature β^{RT} for the total torus area, $A_T = A_1 + A_2$, and assuming that β scales linearly with area, $\beta_1 = (A_1/A_T)\beta^{RT}$. Thus,

$$\beta_2 = \left(\beta_m - \frac{A_1}{A_T}\beta^{RT}\right) = \frac{A_2}{2V} kT_2\left(\frac{2D^{(2)}\phi_d}{\pi\, k_r^{(2)}}\right)^{1/2} \qquad (10)$$

is the expression to be used for relating the measured pressure transients at temperature T_2 with the characteristic value of $k_r^{(2)}/D^{(2)}$.

A second experimental problem with the TFTR measurements occurred with the attempt to make measurements at T_2 = 100 C. Since these measurements followed the cessation of plasma operations by only two days, a significant level of hydrogen outgassing was evident which invalidates the basic assumption in the analysis of HL that the initial wall loading is negligible.

In this case a constant source c_w is assumed to be responsible for the outgassing:

$$c(y,o) = c_w, \text{ when } y > o$$

$$= c_o, \text{ when } y = o \quad . \qquad (11)$$

The appropriate solution of the diffusion equation which meets these boundary conditions is:

$$c(y,t) = c_o + (c_w - c_o) \text{ erfc } \left[\frac{y}{2\sqrt{kT}}\right] \quad . \qquad (12)$$

Equation (6) for the change in wall loading $\Delta N_w(t)$ is now modified according to:

$$\Delta N_w(t) = A \int_o^\infty \left[c(y,t) - c_w\right] dy$$

$$= \frac{2A}{\pi^{1/2}} \left(c_o - c_w\right) \left(Dt\right)^{1/2} \quad . \tag{13}$$

In the previous case of negligible initial wall loading, the near surface concentration c_o was related to the glow discharge flux by detailed balance at the surface, thus $c_o = (\phi_d/2\ k_r)^{1/2}$. Similarly, c_w can be equated to the observed hydrogen pressure rise in the vessel in the absence of a glow discharge:

$$c_w = \left(\frac{2V}{A\ kT} \frac{dp}{dt}\right)^{1/2} \quad . \tag{14}$$

Thus, Eq. (7) is modified to:

$$P(t) = p_o - \frac{A\ kT}{2V} \left[\left(\frac{2D}{\pi\ k_r}\right)^{1/2} \left(\phi_d^{\ 1/2} - \left\{\frac{2V}{A} \frac{dp}{dt}\right\}^{1/2}\right)\right] \quad . \tag{15}$$

As a final comment on the method of analysis, it should be noted that the derived quantity from Eq. (7) [or Eq. (15)] is the ratio k_r/D. To extract a value of k_r, an independent measurement of the hydrogenic diffusivity must be assumed. For the results presented in the next section, a fit to the measurements of Tison and Fidelle[7] was used to derive k_r. This fit yields the expression:

$$D = \left(2 \cdot 10^{-2}\right) \exp(-\ 0.535\ \mathrm{eV}/kT) \tag{16}$$

for the diffusivity.

The TFTR measurements were made using instrumentation that comprise the TFTR glow discharge cleaning system.[4] The glow discharge is formed by biasing one or two stainless steel anodes which are positioned within the torus on moveable probe mechanisms. The DC bias supplies are capable of supplying 20 A per anode and are current regulated. The voltage that appears on the anodes during the glow discharge is 380 V, which is the expected potential drop for a hydrogen glow discharge with Fe electrodes.[8]

The decrease in torus pressure during the course of the glow discharge is monitored with a capacitance manometer that spans the pressure range of 10^{-5}-1 Torr. Table I is a listing of the various parameters for the TFTR measurements that are necessary for the derivation of k_r/D from Eq. (7) or Eq. (15).

III. RESULTS

A total of six experimental runs are presented in this paper. Run 1 and 2 were performed with H_2 with the vessel at 25 C. Those runs were made after the TFTR vacuum vessel had been conditioned with 80 hours of glow discharge cleaning, but previous to any significant exposure to high power tokamak discharges. Runs 3 and 4 were performed with H_2 with the vessel at 46 C, Run 5 with D_2 with the vessel at 25 C, and Run 6 with D_2 with the vessel at 100 C. For the latter runs, 3-6, the vessel had been exposed to an additional 200 hours of glow discharge cleaning interspersed with several hundred ohmically heated H_2 and D_2 tokamak discharges.

Previous to each experimental run, the stability of the static gas fill was measured prior to the initiation of the glow discharge to gauge the influence of possible wall-pumping effects, leaks, and vessel outgassing. Only in the run at a vessel temperature of 100 C was a significant correction necessary. For Run 6, the vessel was observed to have a hydrogen pressure rise of 2.2×10^{-7} Torr/sec., which necessitated the correction described in the previous section leading to the derivation of Eq. (15). In all experimental runs the purity of static gas fill was checked with the TFTR Residual Gas Analyzer before and after the glow discharge measurement. In all cases, the nonhydrogenic components remained below 0.1% of the total pressure.

Figures 1 and 2 show the measured pressure curves during the six separate Runs. For convenience and comparison the curves are plotted in the same units that were used by HL in the ISX measurements.[5] Thus, a straight line on these Δp-$t^{1/2}$ plots demonstrate the $t^{1/2}$ dependence predicted by Eq. (8) and (15). For some of the TFTR measurements, particularly the first two Runs, the initial pressure measurements lie above the $t^{1/2}$ curve. We surmise that this effect may be due to local outgassing in the vicinity of the anodes since (1) the anodes reach a temperature ~ 200 C at a discharge current of 2.5 A; (2) the effect is less evident with sequential measurements.

Table II summarizes the measured values of the slopes, β_m, of the pressure curves from Figs. 1 and 2, the corrected value $\beta_2 = (\beta_m - (A_1/A_T)\beta^{RT})$ that is necessary for the non-room-temperature measurements, the derived values of k_r/D, and the derived values of k_r for the listed value of $D(T)$.

Figure 3 shows the TFTR values of k_r derived from these measurements compared with the literature values of laboratory measurements that were recently compiled by Wilson.[1] Plotted in such form, the TFTR values are seen to occupy the portion of the graph which is midway between values that are characteristic of atomically clean stainless steel (sticking factor $\alpha = 0.5$), and values characteristic of heavily oxidized stainless steel

($\alpha = 5 \times 10^{-5}$). The TFTR values are consistent with the laboratory measurements obtained by Waelbroeck et al.[3] for glow discharge treated stainless steel, and the k_r values at 25 C are similar to the ISX measurements.[5] Although, the number of data points at higher temperatures is sparse, the trend of the data is consistent with the calculated Arrhenius factors (≈ 0.5 eV) of Baskes[9] or Ali-Khan.[10]

The TFTR k_r measurements show no significant isotopic dependence, although a subtle difference would be hidden by the relatively large estimated error for these measurements. No obvious mechanism for an isotopic dependence is evident in the model calculations of k_r, nor have isotopic dependencies been detected in measurements of D that are significant compared to experimental variations.[11]

The errors bars shown in Fig. 3 for the TFTR data points are derived from the estimated error in input parameters: the implantation area A, the implanted flux ϕ_d, and the variation about the mean fit of D to the data of Tison and Fidelle. Considering only these variations, the estimated accuracy of k_r is (+ 100%/- 50%).

IV. CONCLUSIONS

The values of k_r obtained for TFTR have important implications for device operation in terms of the hydrogen recycling rates and hydrogen retention properties of the vessel wall. For a given incident charge-exchange neutral flux, ϕ, a characteristic recycling time can be defined[3] which is:

$$\tau_r \simeq \frac{D}{k_r \alpha \phi} \qquad (17)$$

where α is the effective sticking coefficient. Thus for an estimated typical flux of ~ 10^{16} $cm^2 \cdot s^{-1}$, the recycling time is ~ 0.1 s for the TFTR vessel at 300 K and ~ 10^{-3}s at 373 K. For comparison, expected particle confinement times for TFTR are ~ 0.3 s and discharge durations are ~ 3 s. Since the power loading of the wall during high power plasma operation is expected to raise the temperature to the vicinity of 100 C, we conclude that recycling phenomena will equilibrate rapidly during the course of a single discharge. Therefore, the recycling flux from the stainless steel wall should be relatively constant and hydrogen isotope replacement times should be short. (However, this simple estimate does not address the contribution to recycling from the high flux first-wall hardware such as limiters, which are not made of stainless steel.)

The net retention of hydrogen isotopes is a particularly important quantity when TFTR begins D-T operation. It is desirable to maintain in-vessel tritium inventories as low as possible following operational periods. Estimates of the expected tritium inventory in TFTR have been calculated by Baskes and Wilson [12] following a proposed D-T operational sequence of 25 full-power D-T shots per day. With the assumption that the charge-exchange flux is uniform on the torus area, the calculated tritium wall inventory after three months of such operation is ≈ 1 kCi, using the measured values of k_r at 373 K. The more realistic case of a locally high charge-exchange flux at limiter positions will tend to decrease this inventory estimate since the re-emission is proportional to the square of the near-surface concentration.

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Table I: Glow Discharge and Wall Parameters for TFTR k_r Measurement

Parameter		Value	Comments
Torus Volume	$V(cm^3)$	8.65×10^7	measured
Surface Area	$A(cm^2)$	2.1×10^6	calculated[a]
Wall Temperature	$T(\ K)$	300	
Glow Discharge Current	$I_g(A)$	5(15)	measured
Implanted Atomic (H) Flux	$\phi(cm^{-2}s^{-1})$	$1.5(4.5) \times 10^{13}$	calculated[b]
Diffusion Coef. pre-exponential	$D_o(cm^2s^{-1})$	2.0×10^{-3}	measured[c]
Diffusion Coef. activation energy	$Q_D(eV)$	0.535	measured[c]
Diffusion Coef. at 300 K	$D(cm^2s^{-1})$	$2.0 \cdot 10^{-12}$	measured[c]

Notes

a) Estimated contributions of $1.3 \times 10^6\ cm^2$ from torus and $0.8 \times 10^6\ cm^2$ from the pumping ducts.

b) Calculated from $\phi = I_g/eA\ (1 - r)f$, where the value of the reflection coefficient is estimated to be 0.5 for the predominant incident ion, H_2^+, which has a charge fraction of $f = 2$ (See Ref. 8).

c) From measurements of P. Tison and J. Fidelle (Ref. 7)

Table II: Results of k_r Measurements in TFTR

Run	Gas	T(K)	β_m $(mT \cdot m^{-1/2})$	$\beta_m - (A_1/A_T)\beta^{RT}$ $(mT \cdot m^{-1/2})$	$D(cm^2 \cdot s^{-1})$	$D/k_r (cm^{-2})$	$k_r (cm^4 \cdot s^{-1})$
1	H_2	298	0.33	---	$1.8 \cdot 10^{-12}$	$1.4 \cdot 10^{15}$	$1.3 \cdot 10^{-27}$
2	H_2	298	0.25	---	$1.8 \cdot 10^{-12}$	$7.8 \cdot 10^{14}$	$2.3 \cdot 10^{-27}$
3	H_2	319	0.20	0.07	$6.9 \cdot 10^{-12}$	$1.4 \cdot 10^{14}$	$5.0 \cdot 10^{-26}$
4	H_2	319	0.27	0.14	$6.9 \cdot 10^{-12}$	$5.8 \cdot 10^{14}$	$1.2 \cdot 10^{-26}$
5	D_2	298	0.43	---	$1.8 \cdot 10^{-12}$	$2.3 \cdot 10^{15}$	$7.9 \cdot 10^{-28}$
6	D_2	373	0.22	0.09	$1.1 \cdot 10^{-10}$	$7.9 \cdot 10^{13}$	$1.4 \cdot 10^{-24}$

FIGURE CAPTIONS

FIG. 1 Pressure curves for the first two k_r measurements performed on TFTR after the initial vessel conditioning.

FIG. 2 Pressure curves for k_r measurements in TFTR performed after the first operational period with ohmically heated plasmas.

FIG. 3 Summary of the laboratory measurements of k_r for stainless steel as given by Wilson in Ref. 1, and the k_r values obtained for TFTR. The spread of data points is bounded by theoretical calculation of Baskes (Ref. 9), which includes the effective molecular sticking coefficient α, as a parameter.

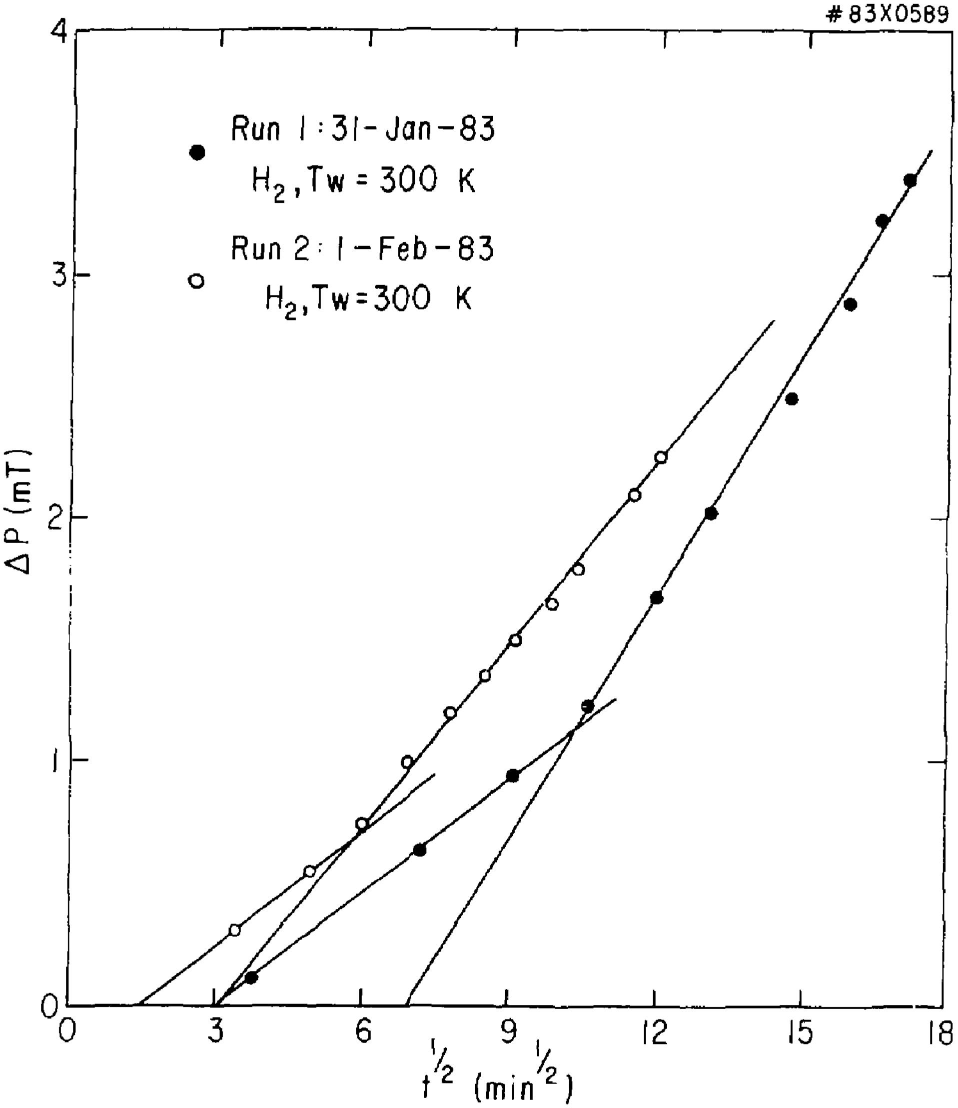
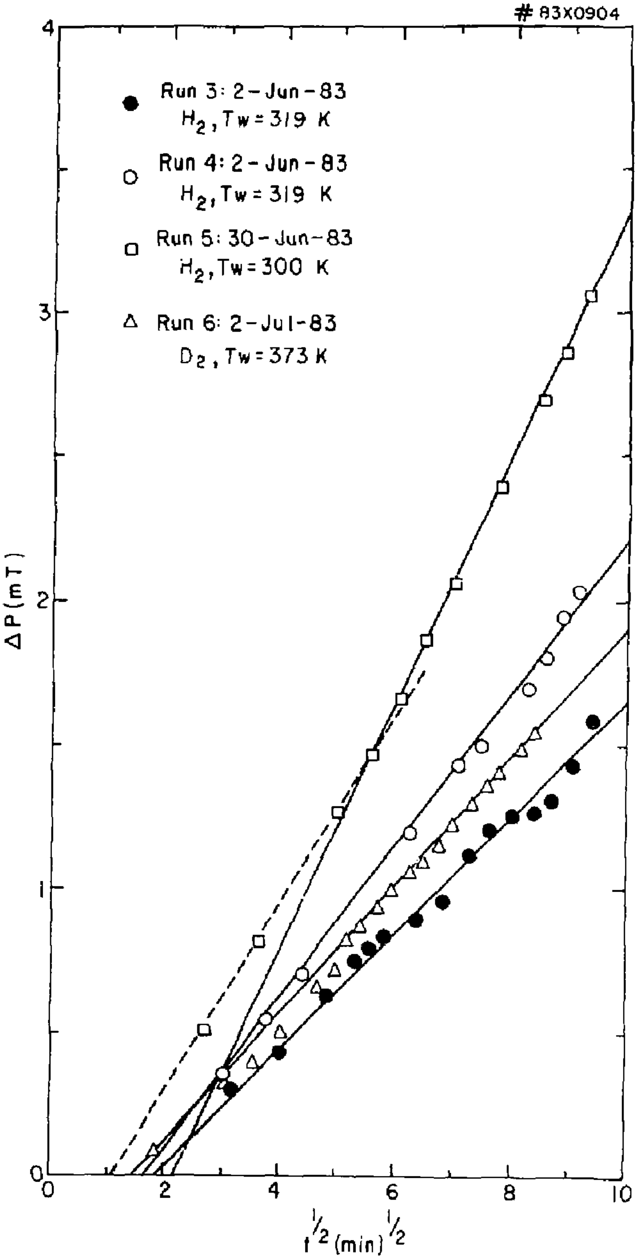

#83X0589
Run 1 : 31-Jan-83
H_2, Tw = 300 K
Run 2 : 1-Feb-83
H_2, Tw = 300 K
ΔP (mT)
0
1
2
3
4
0
3
6
9
12
15
18
$t^{1/2}$ ($min^{1/2}$)

Fig. 1



83X0904
Run 3: 2-Jun-83 H_2, Tw=319 K
Run 4: 2-Jun-83 H_2, Tw=319 K
Run 5: 30-Jun-83 H_2, Tw=300 K
Run 6: 2-Jul-83 D_2, Tw=373 K
ΔP (mT)
0
1
2
3
4
0
2
4
6
8
10
$t^{1/2}$ (min)$^{1/2}$

Fig. 2

#83X0626

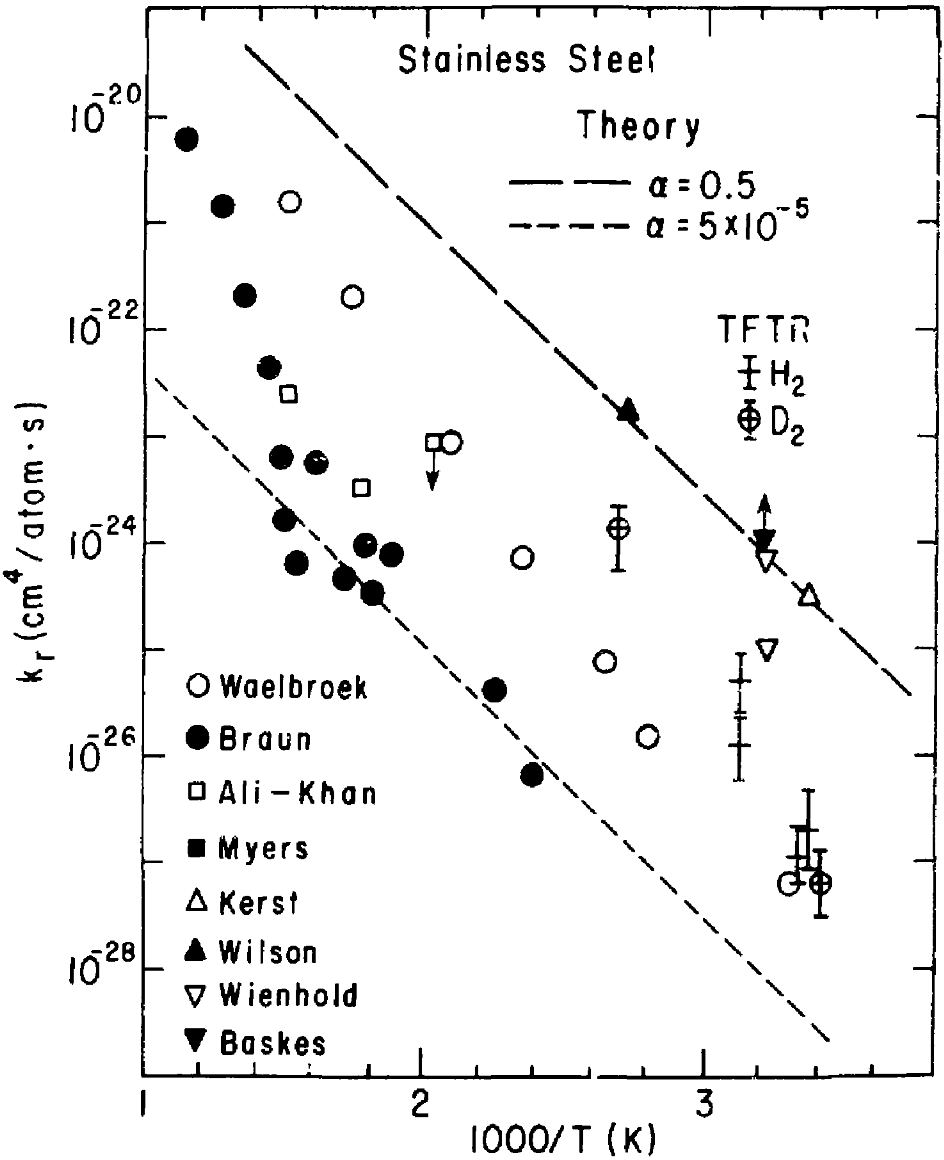


Fig. 3

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