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**HYDROGEN PERMEATION MEASUREMENT
OF THE REDUCED ACTIVATION FERRITIC STEEL F82H
BY THE VACUUM THERMO-BALANCE METHOD**

March **2005**

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Hydrogen Permeation Measurement of the Reduced Activation Ferritic Steel F82H by the Vacuum Thermo-balance Method

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Hydrogen permeation fluxes of the reduced activation ferritic steel F82H were quantitatively measured by a newly proposed method, vacuum thermo-balance method, for a precise estimation of tritium leakage in a fusion reactor. We prepared sample capsules made of F82H, which enclosed hydrogen gas. The hydrogen in the capsules permeated through the capsule wall, and subsequently desorbed from the capsule surface during isothermal heating. The vacuum thermo-balance method allows simultaneous measurement of the hydrogen permeation flux by two independent methods, namely, the net weight reduction of the sample capsule and exhaust gas analysis. Thus the simultaneous measurements by two independent methods increase the reliability of the permeability measurement.

When the gas pressure of enclosed hydrogen was 0.8 atm at the sample temperature of 673 K, the hydrogen permeation flux of F82H obtained by the net weight reduction and the exhaust gas analysis was 0.75×10^{18} ($\text{H}_2/\text{m}^2\text{s}$) and 2.2×10^{18} ($\text{H}_2/\text{m}^2\text{s}$), respectively. The ratio of the hydrogen permeation fluxes obtained by the net weight reduction to that measured by the exhaust gas analysis was in the range from 1/4 to 1/1 in this experiment. The temperature dependence of the estimated permeation flux was similar in both methods. Taking the uncertainties of both measurements into consideration, both results are supposed to be consistent. The enhancement of hydrogen permeation flux was observed from the sample of which outer surface was mechanically polished. Through the present experiments, it has been demonstrated that the vacuum thermo-balance method is effective for the measurement of hydrogen permeation rate of F82H.

Keywords: Tritium Leakage, Hydrogen Permeation, Ferritic Steel F82H, Vacuum Thermo-balance Method

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真空熱天秤法による低放射化フェライト鋼 F82H の水素透過測定

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核融合炉におけるトリチウム漏洩量見積りの高精度化に資するため、低放射化フェライト鋼の水素透過速度を真空熱天秤法で測定した。真空熱天秤法は、水素透過速度をより精度良く定量的に測定することができる新しい測定方法である。本測定法では、水素ガスを封入した低放射化フェライト鋼 F82H 製の試料カプセルを用意し、試料カプセルを真空中で等温加熱して、試料カプセルを透過しカプセル表面から脱離する水素を以下の 2 つの独立した方法で測定する。すなわち、試料カプセルの正味の重量減少量と、試料カプセルからの水素脱離による排気ガス分析である。このような手法で測定の信頼性を増すことが可能である。

カプセル内の水素ガス圧 0.8 atm、カプセル温度 673 K の時、水素透過速度は、正味の重量減少量から求めた場合 0.75×10^{18} ($\text{H}_2/\text{m}^2 \text{ s}$)、排気ガス分析から求めた場合 2.2×10^{18} ($\text{H}_2/\text{m}^2 \text{ s}$)であった。本実験条件で、正味の重量減少量から求めた水素透過速度と排気ガス分析から求めた値の比は、1/4 から 1/1 であった。カプセル温度の上昇に伴って水素透過速度が増加するという傾向は、両測定法で一致した。両測定法の誤差を考慮すると、得られた水素透過速度はよく一致しているとみなせる。また F82H カプセルの表面を機械研磨した際には、水素透過速度の促進も観測された。本実験を通して、真空熱天秤法が F82H の水素透過速度を評価するために有効な手段であることが示された。

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1. Introduction

A breeding blanket of a fusion reactor is designed to withstand high temperature in order to increase coolant temperature for high thermal efficiency of the power plant. A solid breeder material is to be cooled by supercritical pressure water [1]. In the design, coolant temperature range is about 280 – 510°C and the temperature of coolant tube wall made of a reduced activation ferritic steel, F82H [2], is higher than the coolant temperature. In such conditions, evaluation of tritium permeation through F82H wall is important for both design of a tritium recovery system and a coolant water detritiation system, and then tritium accountability of the fusion power plant. However, number of available data on hydrogen permeation rate of F82H is very much limited. An estimation using the existing data showed that the amount of tritium permeated from the helium sweep gas flowing around the breeder to the water through coolant tube wall was significant so that a half of generated tritium in the blanket permeated in the water, if there is no permeation barrier on the coolant tube wall [3]. Thus, accumulation of the basic data of tritium permeation of F82H and the evaluation of the effect of permeation barriers are very important.

Improvement of the accuracy on the hydrogen permeation is also necessary. Previously, the hydrogen permeation flux has been measured from difference of pressures in two volumes separated by a sample [4-6]. However, this method has some difficulties in quantitative measurement. One of them is the evaluation of the gas pressure, since thermal transpiration causes the pressure in the chamber to be non-uniform. The gas desorption from chamber, for example H₂O, also give rise to the uncertainties of the pressure measurement. The calibration of the gas pressure should also have error.

From the viewpoints stated above, this paper presents the measurement results of hydrogen permeation flux of F82H and the effect of surface treatment on the hydrogen permeation flux. In this study, the authors applied a new method, vacuum thermo-balance method, for the measurement of the hydrogen permeation flux of F82H. For the comparison, hydrogen permeation of 316SS was also measured. To investigate the surface effects on the hydrogen permeation, the surface morphologies and surface compositions of these samples were investigated by scanning electron microscope (SEM) and Auger electron spectroscopy (AES).

2. Experiments

2.1 Sample preparation

The sample capsules made of F82H or 316SS were closed by welding the both ends of each pipe, whose thickness was 0.5 mm and 0.25 mm, respectively. The pipes made of F82H were rolled to be 0.5 mm thickness with annealing at 810°C for 15 min, then thermal treatment was performed at 1050°C for 30 min, and then at 750°C for 60 min. After the thermal treatment, the tube was pickled. Hydrogen and helium gases were enclosed in these sample capsules at 1 atm. Helium gas was introduced to check the leakage due to mechanical pores or cracks. The ratio of enclosed hydrogen gas to helium gas was 8/2. For the background measurement, the sample capsules, which enclosed only He, was also prepared. The measurement of hydrogen permeation flux was performed by using the as-received capsules (no-polishing). A polished F82H sample capsule was also prepared for the

investigation of the surface effect on hydrogen permeation, though it was polished only outer surface. The capsule was polished 50 times, respectively, with emery paper (#320, #600 and #00). The eliminated thickness was estimated to be about 4 μm from weight loss, of which thickness reduction would be negligible. Hydrogen permeation measurement and surface analysis was performed several days after polishing. Prepared sample capsules were summarized in Table 1.

2.2 Vacuum thermo-balance method

The equipment of the vacuum thermo-balance apparatus was schematically shown in Fig. 1. The sample capsules made of F82H and 316SS, which enclosed the hydrogen and/or helium gases, were heated in the vacuum. Residual gas pressure was about 1×10^{-4} Pa. The hydrogen in the capsule permeated through the capsule and subsequently desorbed from the surface. The hydrogen permeation flux was simultaneously measured by two independent methods, namely, the net weight reduction of the sample capsule and the desorption rate of hydrogen gas from the sample capsule. The weight change of a sample was measured by the micro-balance (measurement limit 1×10^{-6} g) in vacuum. The hydrogen desorption rate from the sample was measured by a quadruple mass spectrometer (QMS), which was calibrated using a gas flow controller. Those sample capsules were isothermally heated from 375 to 450°C by furnace. The temperature in the furnace was measured by thermocouple. In the case of the vacuum thermo-balance methods, the hydrogen permeation fluxes can be measured more quantitatively than conventional methods since users can crosscheck the results obtained by the both measurements.

3. Results and discussion

3.1 An example of vacuum thermo-valance measurement

Figure 2 shows the weight change and QMS signal intensities of H_2 and H_2O , which were major desorption species in this experiment. The QMS signals were calibrated by relative sensitivities [7] to compare H_2 with H_2O in this figure. The sample only with He was also shown in this figure. Since no signal of He was observed, it was confirmed that there were no clacks or micro pores in these samples. As shown in Fig.2, the H_2 intensity in QMS from the sample enclosed both H_2 and He gases was clearly larger than that of He gas only. The hydrogen permeation flux is estimated from the QMS signal, as $(2.2 \pm 0.2) \times 10^{18}$ ($\text{H}_2/\text{m}^2\text{s}$).

On the other hand, the weight of the sample enclosed both H_2 and He gases slightly decreased. That only with He increased due to oxidation of sample surface. Therefore, the net weight reduction caused by the hydrogen desorption could be obtained by subtracting the weight change of He only sample from that of H_2 and He gases. The net weight reduction was 2.2×10^{-6} ($\text{g}/\text{m}^2\text{s}$), which corresponded with the permeation flux of 7.5×10^{17} ($\text{H}_2/\text{m}^2\text{s}$). This value is 1/3 of that obtained exhaust gas analysis by the QMS.

3.2 Measurement results of hydrogen permeation flux

The temperature dependence of hydrogen permeation fluxes of F82H and 316SS are

shown in Fig.3 and Fig.4, respectively. The hydrogen permeation fluxes from F82H and 316SS were increased with the temperature. The similarity was observed on the temperature dependences measured by the exhaust gas measurement and those of the net weight reduction.

Figure 5 shows a comparison of hydrogen permeation fluxes from the F82H sample with polished F82H sample. The hydrogen permeation flux from polished F82H sample was larger than that from without polishing. To investigate the reasons why the hydrogen permeation flux of polished F82H was larger than that of un-polished, surface analyses were performed. SEM photographs of un-polished and polished F82H are shown in Fig.6. The surface of polished F82H was rougher than that of un-polished F82H. Depth profiles of atomic composition obtained from Auger electron spectroscopy are shown in Fig.7. No clear difference of surface compositions was observed between un-polished and polished F82H. From these results, it is considered that the difference of surface morphologies rather than that of the surface composition causes the larger hydrogen permeation flux of polished one. One of the explanations is that the specific surface area became larger by polishing, and then the larger specific surface area promotes the hydrogen permeation. The larger specific surface area causes the larger amount of hydrogen desorption, and then the gradient of hydrogen concentration in F82H became larger. Another explanation is that the polishing eliminated the permeation resistive layer, for example processing layer formed during the preparation of F82H pipes. Further experiment is necessary to conclude the mechanism of this surface effect.

The hydrogen permeation fluxes measured in the present experiments are evaluated in Table 2. The ratio of the hydrogen permeation fluxes obtained by the net weight reduction to that measured by the exhaust gas analysis was in the range from 1/4 to 1/1 in this experiment. The uncertainties of exhaust gas analysis are the effects of thermal transpiration, the mixed gas effects of H₂ and H₂O on both the effective pumping speed and the relative sensitivities of QMS. The calibration of QMS by using the gas flow controller also has error. Those of net weight reduction measurements are the superposition effect between hydrogen permeation and oxidation, and noise. Taking the uncertainties of both measurements into consideration, both results are supposed to be consistent.

3.3 Comparison with measurement results of previous method

For a case of steady-state permeation through a single layer barrier, where the rate limiting process is diffusion through the material rather than surface reactions, the permeation rate is given as follows,

$$J = \frac{\Phi}{d} (P_H^{1/2} - P_L^{1/2}) \quad (1)$$

where J is permeation flux (H₂/m²s), d is thickness (m), P_H and P_L is the gas pressure (Pa) of on the high and low pressure sides respectively, and Φ is permeability (H₂/m s Pa^{1/2}). Here we calculate the hydrogen permeability from this equation for the comparison with the results of previous method obtained by other researchers, though the equation is not applicable in a strict sense since the assumptions were not fulfilled.

Figure 8 shows the Arrhenius plots of obtained permeabilities for F82H, polished F82H and 316SS. The error bars in this figure show the influences of decrease of inner pressure in the sample capsules. The measurement results of the previous method were also plotted in the

figure. The results show scatter of almost one order of magnitude, and the measurement at present seems within the range of scatter. The permeability of 316SS obtained by exhaust gas analysis was comparable to that obtained by Forcey et al. [5] and Hashimoto et al. [6]. That of F82H obtained by exhaust gas analysis was about a third of Serra's data [4]. But that of polished (only outer side) F82H was about twice as large as that of unpolished sample. Note that Serra's samples were polished both sides. Considering these effects, permeability of F82H was considered to be comparable of their results.

4. Summary

For accurate quantitative measurements, hydrogen permeation fluxes of F82H and 316SS were measured by vacuum thermo-balance method.

The observed temperature dependences of the hydrogen permeation flux were similar between the results of both the net weight reduction and the exhaust gas analysis. The ratio of the hydrogen permeation fluxes obtained by the net weight reduction to that measured by the exhaust gas analysis was in the range from 1/4 to 1/1 in this work. Taking the uncertainties of both measurements into consideration, both results are regarded to be consistent. The enhancement of hydrogen permeation flux due to the mechanical polishing was also observed. Thus, the effectiveness of the vacuum thermo-balance method was demonstrated to evaluate the hydrogen permeation flux of F82H.

Reference

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Table1 Samples configurations

Sample name	F82H	Polished F82H	316SS
Thickness (mm)	0.5	0.5	0.25
Diameter (mm)	12	12	15
Length (mm)	57.0	57.0	55.5
Surface area (10^{-3} m^2)	2.24	2.24	2.70

Table2 The hydrogen permeation fluxes measured by exhaust gas analysis and net weight reduction

Material	Temperature (K)	Hydrogen permeation flux	
		Exhaust gas analysis ($10^{18} \text{ H}_2/\text{m}^2 \text{ s}$)	Net weight reduction ($10^{18} \text{ H}_2/\text{m}^2 \text{ s}$)
F82H	648	1.4 ± 0.4	0.33
	673	2.2 ± 0.2	0.75
	698	3.0 ± 0.4	1.8
	723	5.0 ± 1.1	4.9
Polished F82H	673	4.0 ± 0.4	1.2
316SS	648	0.49 ± 0.02	0.46
	673	1.7 ± 0.2	0.95
	723	2.9 ± 0.4	1.3

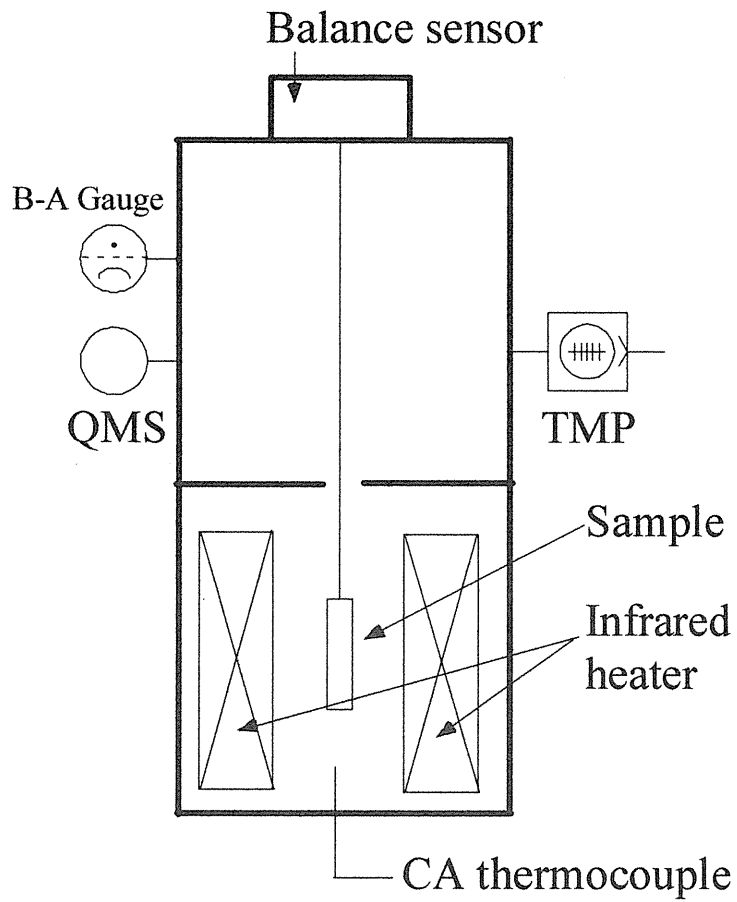


Fig.1 The vacuum thermo-balance apparatus

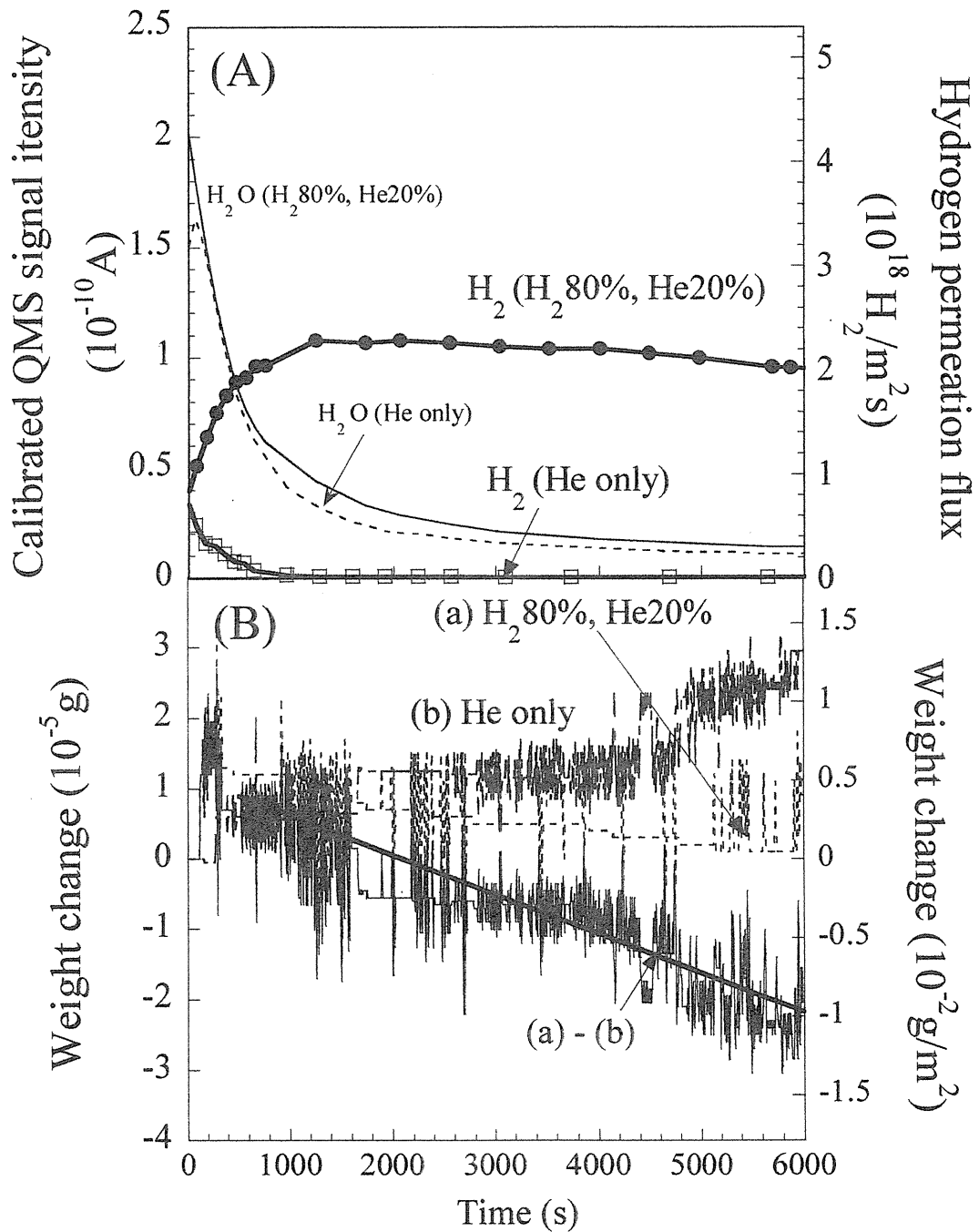


Fig.2 An example of the vacuum thermo-balance measurements. (A) the calibrated QMS signal intensities of H₂ and H₂O, (B) weight variation, F82H sample capsule at 673 K. Total pressure of enclosed gas was 1 atm. The thickness of sample capsules is 0.5 mm.

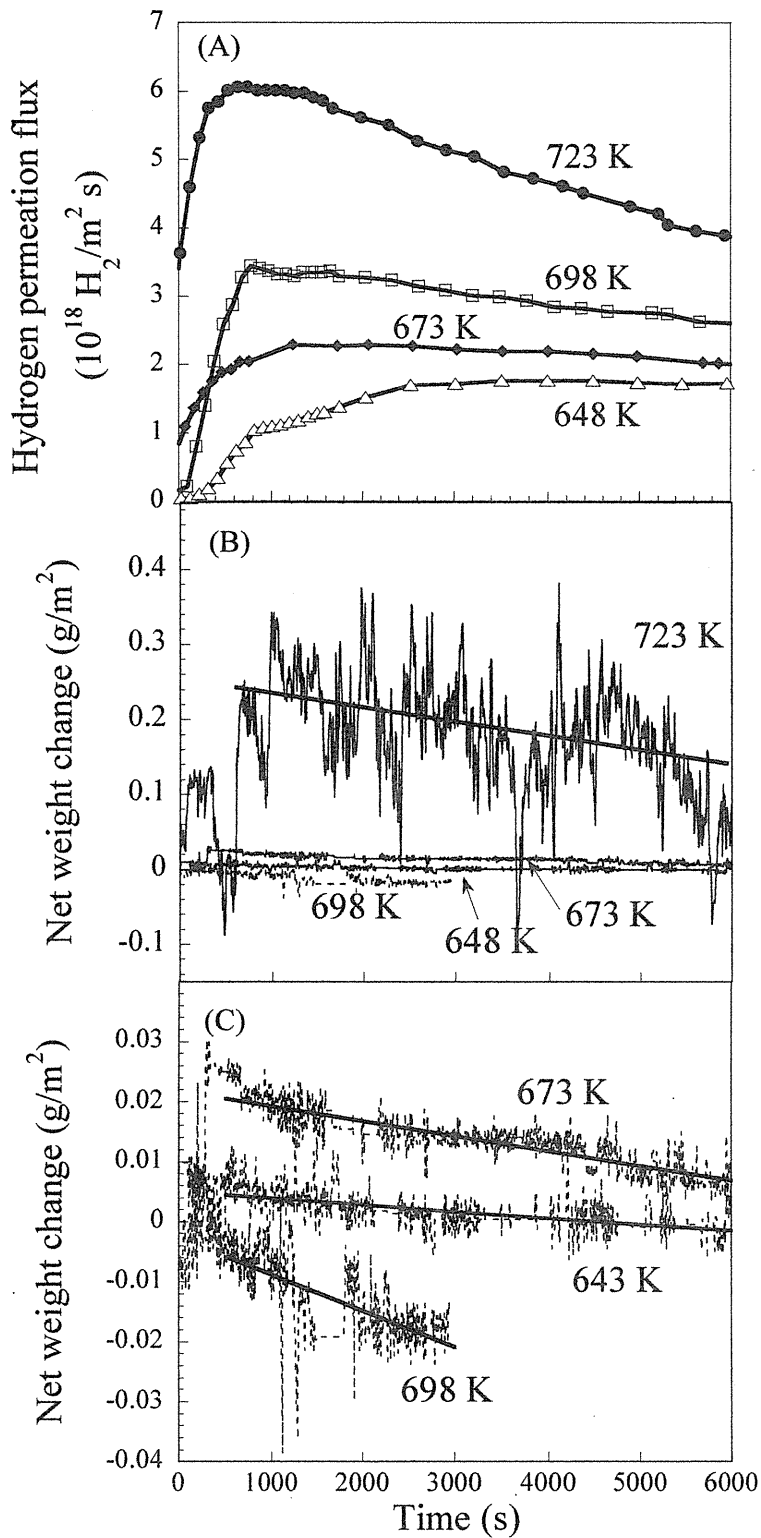


Fig.3 The temperature dependence of hydrogen permeation fluxes of F82H. Total pressure of enclosed gas was 1 atm and H_2/He ratio was 8/2. (A) the QMS signal intensity of H_2 and H_2O . (B) the net weight change. (C) the expansion of (B) for the sample temperature of 673K, 643K, and 698 K. The thickness of sample capsules was 0.5 mm.

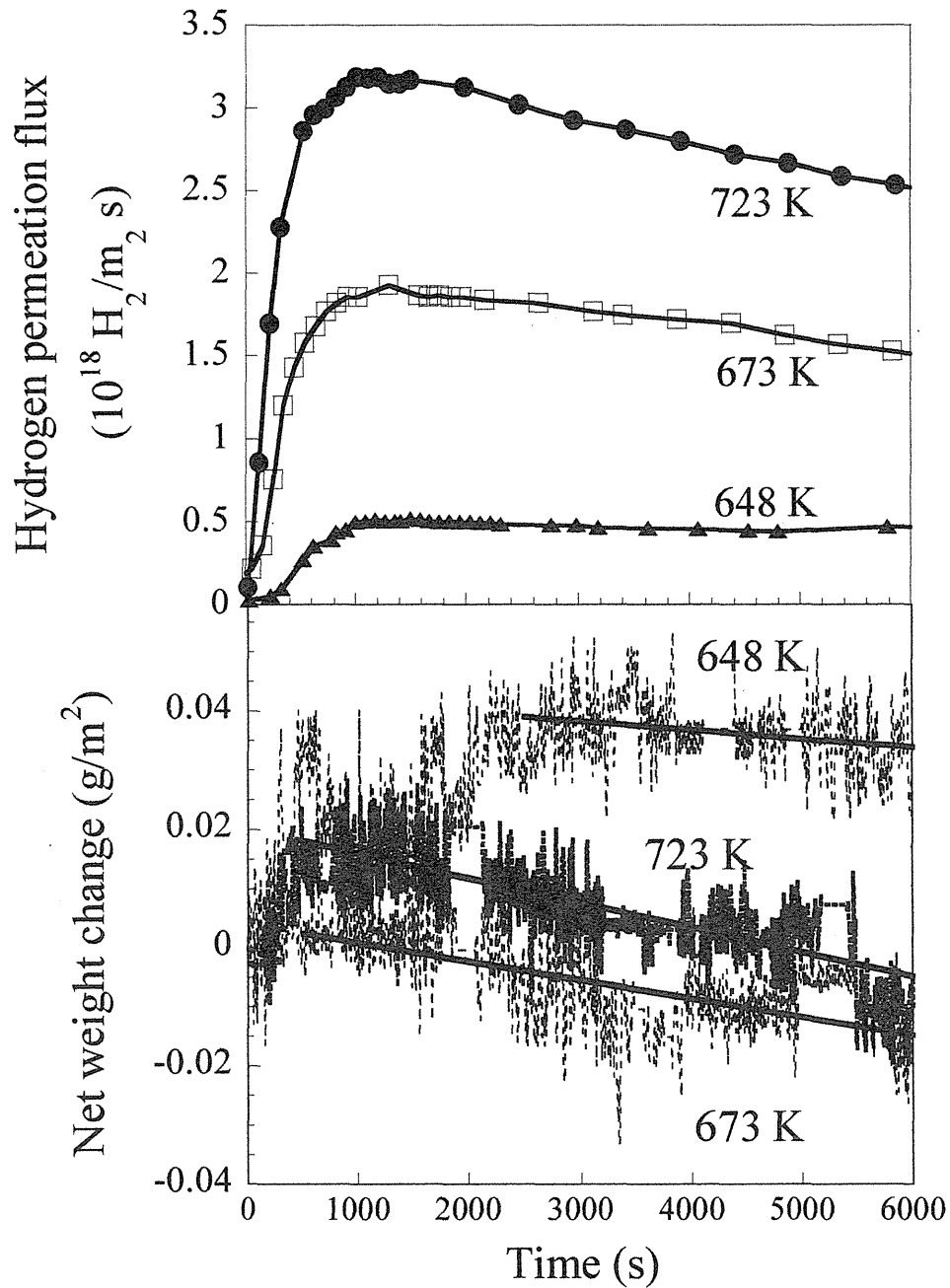


Fig.4 The temperature dependence of hydrogen permeation fluxes of 316SS. Total pressure of enclosed gas was 1 atm and H_2/He ratio was 8/2. (A) is the QMS signal intensity of H_2 and H_2O . (B) is the net weight change. The thickness of sample capsules was 0.25 mm.

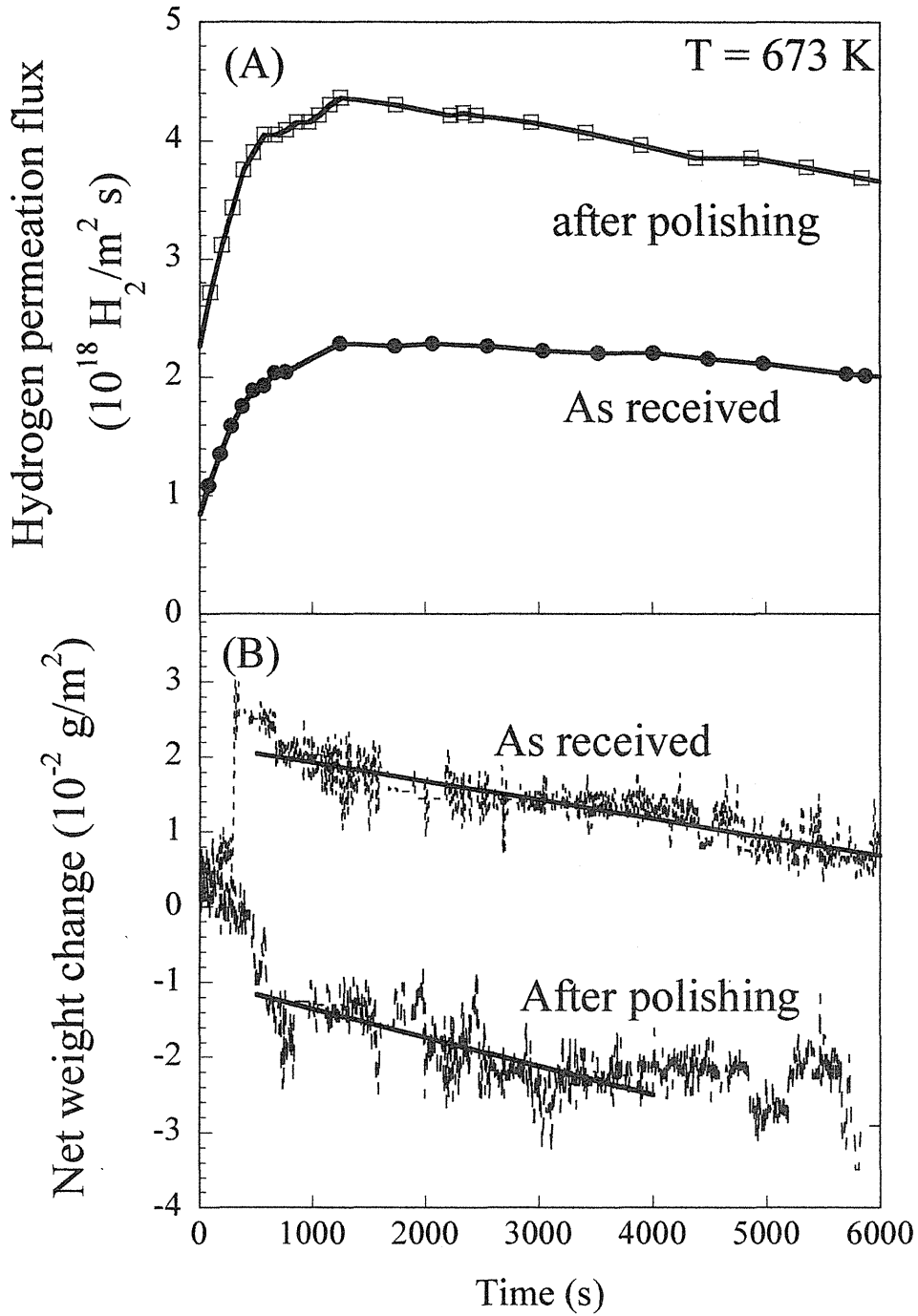


Fig.5 The comparison of hydrogen permeation fluxes between as-received (un-polishing) and polished F82H sample. Enclosed gas pressure was at 1 atm and H_2/He ratio was 8/2. (A) the QMS signal intensity of H_2 and H_2O . (B) the net weight change. The thickness of sample capsules was 0.5 mm.

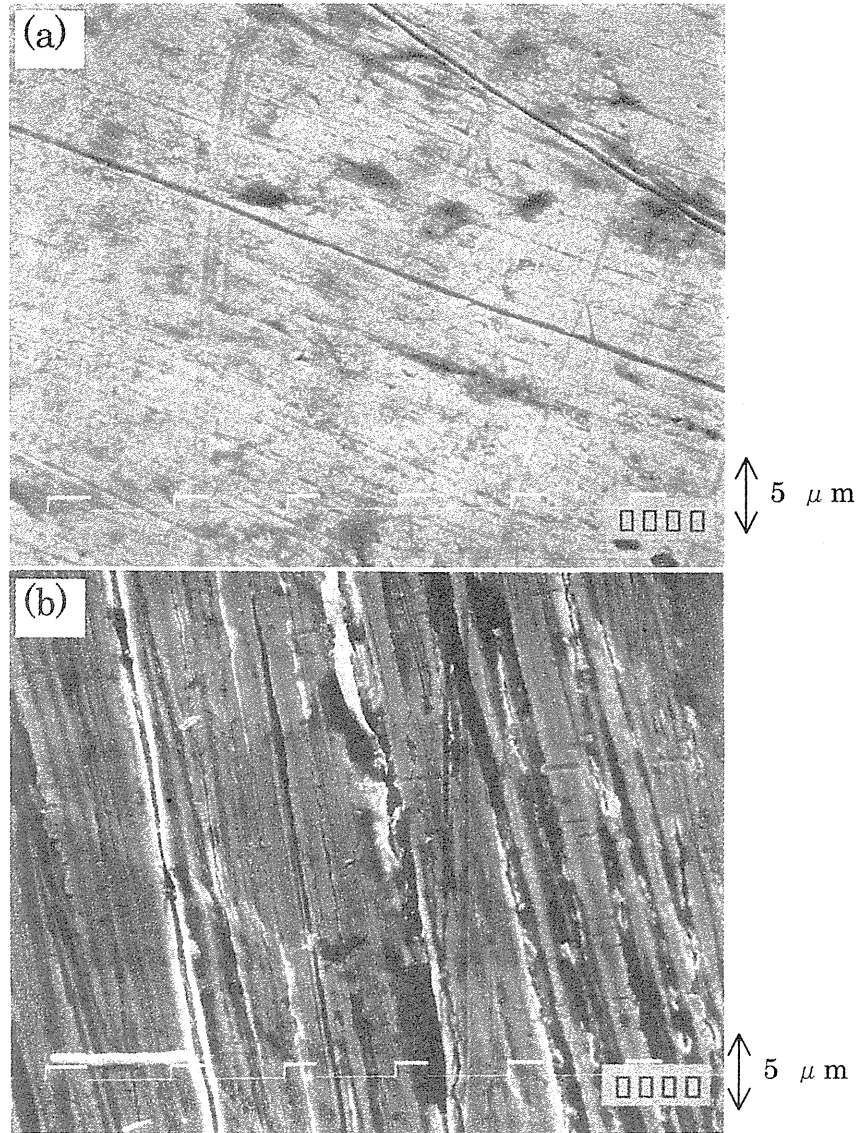


Fig.6 SEM photographs of as-received F82H (a) and polished F82H.(b).

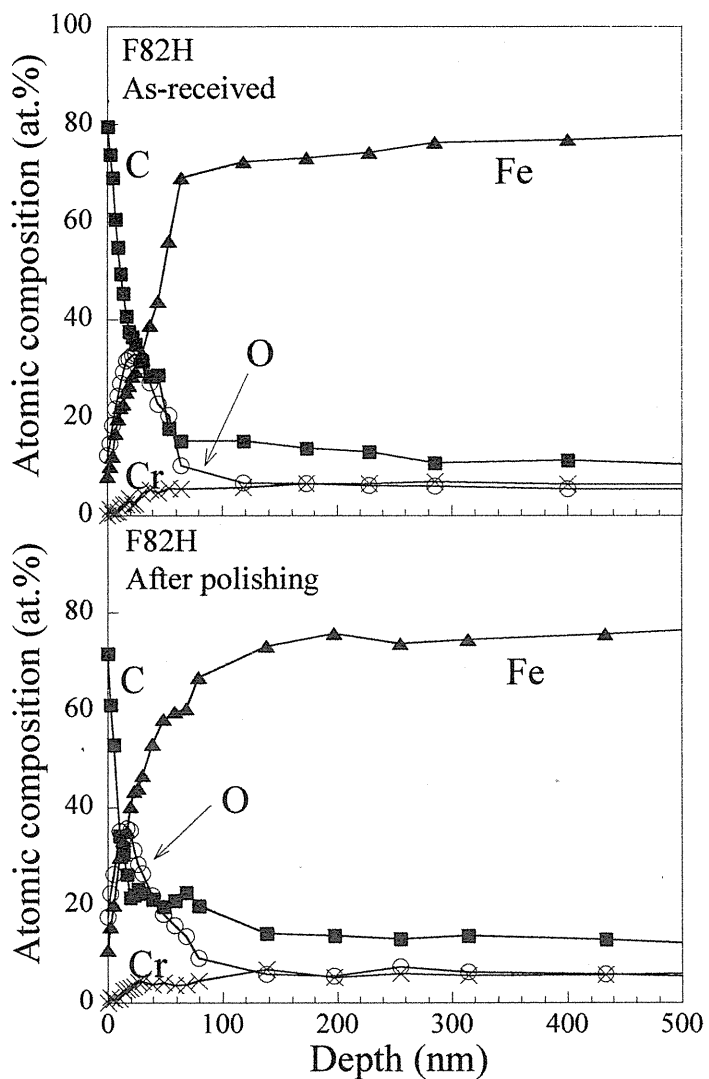


Fig.7 Depth profiles of atomic composition of as-received F82H and polished F82H.

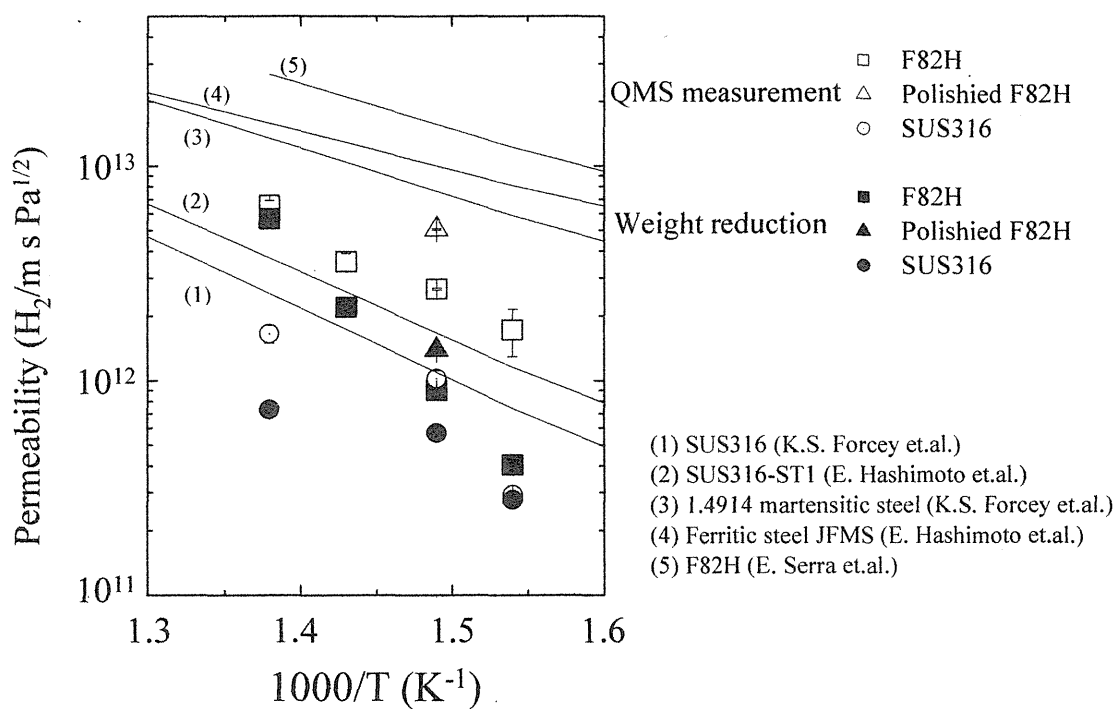


Fig.8 The Arrhenius plots of measured permeabilities for F82H, polished F82H and 316SS.

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国際単位系 (SI) と換算表

表1 SI基本単位および補助単位

量	名称	記号
長さ	メートル	m
質量	キログラム	kg
時間	秒	s
電流	アンペア	A
熱力学温度	ケルビン	K
物質質量	モル	mol
光度	カンデラ	cd
平面角	ラジアン	rad
立体角	ステラジアン	sr

表2 SIと併用される単位

名称	記号
分, 時, 日	min, h, d
度, 分, 秒	°, ', "
リットル	l, L
トン	t
電子ボルト	eV
原子質量単位	u

$1 \text{ eV} = 1.60218 \times 10^{-19} \text{ J}$
 $1 \text{ u} = 1.66054 \times 10^{-27} \text{ kg}$

表5 SI接頭語

倍数	接頭語	記号
10^{18}	エクサ	E
10^{15}	ペタ	P
10^{12}	テラ	T
10^9	ギガ	G
10^6	メガ	M
10^3	キロ	k
10^2	ヘクト	h
10^1	デカ	da
10^{-1}	デシ	d
10^{-2}	センチ	c
10^{-3}	ミリ	m
10^{-6}	マイクロ	μ
10^{-9}	ナノ	n
10^{-12}	ピコ	p
10^{-15}	フェムト	f
10^{-18}	アト	a

表3 固有の名称をもつSI組立単位

量	名称	記号	他のSI単位による表現
周波数	ヘルツ	Hz	s^{-1}
力	ニュートン	N	$\text{m} \cdot \text{kg} / \text{s}^2$
圧力, 応力	パスカル	Pa	N / m^2
エネルギー, 仕事, 熱量	ジュール	J	$\text{N} \cdot \text{m}$
工率, 放射束	ワット	W	J / s
電気量, 電荷	クーロン	C	$\text{A} \cdot \text{s}$
電位, 電圧, 起電力	ボルト	V	W / A
静電容量	ファラド	F	C / V
電気抵抗	オーム	Ω	V / A
コンダクタンス	ジーメンズ	S	A / V
磁束	ウェーバ	Wb	$\text{V} \cdot \text{s}$
磁束密度	テスラ	T	Wb / m^2
インダクタンス	ヘンリー	H	Wb / A
セルシウス温度	セルシウス度	$^{\circ}\text{C}$	
光度	カンデラ	lm	$\text{cd} \cdot \text{sr}$
照射線量	グレイ	Gy	J / kg
線量当量	シーベルト	Sv	J / kg

表4 SIと共に暫定的に維持される単位

名称	記号
オングストローム	\AA
バ	b
バ	bar
ガ	Gal
キュリー	Ci
レントゲン	R
ラド	rad
レム	rem

$1 \text{ \AA} = 0.1 \text{ nm} = 10^{-10} \text{ m}$
 $1 \text{ b} = 100 \text{ fm} = 10^{-28} \text{ m}^2$
 $1 \text{ bar} = 0.1 \text{ MPa} = 10^5 \text{ Pa}$
 $1 \text{ Gal} = 1 \text{ cm} / \text{s}^2 = 10^{-2} \text{ m} / \text{s}^2$
 $1 \text{ Ci} = 3.7 \times 10^{10} \text{ Bq}$
 $1 \text{ R} = 2.58 \times 10^{-4} \text{ C} / \text{kg}$
 $1 \text{ rad} = 1 \text{ cGy} = 10^{-2} \text{ Gy}$
 $1 \text{ rem} = 1 \text{ cSv} = 10^{-2} \text{ Sv}$

(注)

- 表1-5は「国際単位系」第5版, 国際度量衡局 1985年刊行による。ただし, 1 eV および 1 uの値はCODATAの1986年推奨値によった。
- 表4には海里, ノット, アール, ヘクトールも含まれているが日常の単位なのでここでは省略した。
- barは, JISでは流体の圧力を表わす場合に限り表2のカテゴリーに分類されている。
- EC閣僚理事会指令では bar, barn および「血圧の単位」mmHgを表2のカテゴリーに入れている。

換算表

力	N (=10 ⁵ dyn)	kgf	lbf
	1	0.101972	0.224809
	9.80665	1	2.20462
	4.44822	0.453592	1

粘度 $1 \text{ Pa} \cdot \text{s} (\text{N} \cdot \text{s} / \text{m}^2) = 10 \text{ P} (\text{ポアズ}) (\text{g} / (\text{cm} \cdot \text{s}))$

動粘度 $1 \text{ m}^2 / \text{s} = 10^4 \text{ St} (\text{ストークス}) (\text{cm}^2 / \text{s})$

圧	MPa (=10 bar)	kgf/cm ²	atm	mmHg (Torr)	lbf/in ² (psi)
	1	10.1972	9.86923	7.50062×10^3	145.038
力	0.0980665	1	0.967841	735.559	14.2233
	0.101325	1.03323	1	760	14.6959
	1.33322×10^{-4}	1.35951×10^{-3}	1.31579×10^{-3}	1	1.93368×10^{-2}
	6.89476×10^{-3}	7.03070×10^{-2}	6.80460×10^{-2}	51.7149	1

エネルギー・仕事・熱量	J (=10 ⁷ erg)	kgf·m	kW·h	cal (計量法)	Btu	ft·lbf	eV
	1	0.101972	2.77778×10^{-7}	0.238889	9.47813×10^{-4}	0.737562	6.24150×10^{18}
	9.80665	1	2.72407×10^{-6}	2.34270	9.29487×10^{-3}	7.23301	6.12082×10^{19}
	3.6×10^6	3.67098×10^5	1	8.59999×10^5	3412.13	2.65522×10^6	2.24694×10^{25}
	4.18605	0.426858	1.16279×10^{-6}	1	3.96759×10^{-3}	3.08747	2.61272×10^{19}
	1055.06	107.586	2.93072×10^{-4}	252.042	1	778.172	6.58515×10^{21}
	1.35582	0.138255	3.76616×10^{-7}	0.323890	1.28506×10^{-3}	1	8.46233×10^{18}
	1.60218×10^{-19}	1.63377×10^{-20}	4.45050×10^{-26}	3.82743×10^{-20}	1.51857×10^{-22}	1.18171×10^{-19}	1

$1 \text{ cal} = 4.18605 \text{ J} (\text{計量法})$
 $= 4.184 \text{ J} (\text{熱化学})$
 $= 4.1855 \text{ J} (15^{\circ}\text{C})$
 $= 4.1868 \text{ J} (\text{国際蒸気表})$
 仕事率 $1 \text{ PS} (\text{仏馬力})$
 $= 75 \text{ kgf} \cdot \text{m} / \text{s}$
 $= 735.499 \text{ W}$

放射能	Bq	Ci
	1	2.70270×10^{-11}
	3.7×10^{10}	1

吸収線量	Gy	rad
	1	100
	0.01	1

照射線量	C/kg	R
	1	3876
	2.58×10^{-4}	1

線量当量	Sv	rem
	1	100
	0.01	1

Hydrogen Permeation Measurement of the Reduced Activation Ferritic Steel F82H by the Vacuum Thermo-balance Method



古紙配合率100%
白色度70%再生紙を使用しています