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APPENDICES**Appendix A CHEMICAL COMPOSITION OF MATERIALS****Table A.1** The weight percentage chemical composition of carbon steel and Hastelloy

	Carbon Steel ASTM A-179	Hastelloy C-276
C	0.91	-
Mn	0.71	-
Fe	97.70	6.32
P	0.37	-
W	-	3.97
Cr	0.31	15.73
Ni	-	56.61
Mo	-	17.38

Appendix B HYDROGEN PERMEATION DATA

Table B.1 Hydrogen permeation data of all test performed

Test No.	Material	Surface condition	Temp (°C)	Initial P (kPa)	Final P (kPa)	Duration (s)	Least Square Eq.	R ²
1	CS	Outer Oxide layer	MTL	964.3	732.2	950,807	$P = 958.7 \exp(-0.001x)$	0.994
2.1	CS	Outer Oxide layer.	150	1017.2	598.3	685,872	$P = 1048 \exp(-0.003x)$	0.996
2.2	CS	Outer Oxide layer	150	1003.6	792.7	600,344	$P = 1013 \exp(-0.001x)$	0.991
3.1	CS	Outer Oxide Covered	250	1087.3	113.3	258,876	$P = 1126 \exp(-0.042t)$	0.999
3.2	CS	Outer Oxide Covered	250	1089.7	165.9	173,623	$P = 1116 \exp(-0.04t)$	0.999
4	CS	Pd on the outside surface	150	1016.4	526.7	274,596	$P = 1035 \exp(-0.009t)$	0.998
5.1	CS	Pd on the outside surface	250	1121.8	159.8	81,304	$P = 1.0603t^2 - 66.942t + 1131.8$	0.999
5.2	CS	Pd on the outside surface	250	1104.4	161.5	81,509	$P = 1.0341t^2 - 65.406t + 1113.8$	1
6	Hastelloy	Outer Oxide Covered	MTL	-	-	-	-	-
7	Hastelloy	Outer Oxide Covered	250	1142.5	772.1	666,218	$P = 0.0014t^2 - 2.239t + 1139$	0.999
8	Hastelloy	Outer Oxide Covered	335	1216.1	408.7	260,770	$P = 0.0447t^2 - 14.455t + 1220.3$	0.999
9	Hastelloy	Pd on the outside surface	250	1148.7	772.2	658,920	$P = 0.0009t^2 - 2.202t + 1142$	0.999
10	Hastelloy	Pd on the outside surface	335	1234.1	394.2	259,234	$P = 0.0528t^2 - 15.342t + 1227.6$	0.999

Table B.2 Calculation Results

Test No.	Material	Surface condition	Temp (°C)	Diffusivity (m ² s ⁻¹)	Resistance (s m ⁻¹)
1	CS	Outer Oxide Covered	MTL	-	-
2.1	CS	Outer Oxide Covered	150	6.65×10^{-13}	1.13×10^9
2.2	CS	Outer Oxide Covered	150	3.37×10^{-13}	2.23×10^9
3.1	CS	Outer Oxide Covered	250	9.32×10^{-12}	8.07×10^7
3.2	CS	Outer Oxide Covered	250	7.50×10^{-12}	1.00×10^8
4	CS	Catalyst coating on the outside surface	150	2.05×10^{-13}	3.65×10^8
5.1	CS	Catalyst coating on the outside surface	250	2.15×10^{-11}	3.49×10^7
5.2	CS	Catalyst coating on the outside surface	250	2.02×10^{-11}	3.71×10^7
6	Hastelloy	Outer Oxide Covered	MTL	-	-
7	Hastelloy	Outer Oxide Covered	250	5.05×10^{-13}	1.49×10^9
8	Hastelloy	Outer Oxide Covered	335	3.54×10^{-12}	2.12×10^8
9	Hastelloy	Catalyst coating on the outside surface	250	5.35×10^{-13}	1.40×10^9
10	Hastelloy	Catalyst coating on the outside surface	335	3.87×10^{-12}	1.94×10^8

Appendix C FLUX CALCULATION PROCEDURE

The measured value of pressure reduction in a sealed tube can be converted into a molar flux of hydrogen J_{H_2} through the tube wall. This conversion is made using the assumption that hydrogen in the system behaves as an ideal gas.

Procedure of calculating hydrogen flux was as follows:

$$J_{H_2} = \frac{1}{A} \cdot \frac{dn}{dt} = \frac{V}{ART} \times \frac{dP_{H_2}}{dt}$$

where	J	=	Permeation flux (mol/hr m ²)
	dn/dt	=	Flow of gas (mol/hr)
	A	=	surface area of the membrane (m ²)
	V	=	Volume of pressurized chamber (m ³)
	R	=	Ideal gas constant (m ³ Pa K ⁻¹ mol ⁻¹)
	T	=	Temperature (K)
	t	=	time of test (hr)
	dp/dt	=	Rate of pressure decrease (Pa/hr)

Calculation Example

Hydrogen permeation data of the test no.3

P	=	1115.9 exp (-0.04t)
dp/dt	=	-44.64 exp (-0.04t)
A	=	0.013578 m ²
V	=	1.552 × 10 ⁻⁵ m ³
R	=	8.314 m ³ Pa K ⁻¹ mol ⁻¹
T	=	523 K
t	=	252000 sec

$$J_{H_2} = \frac{[1.552 \times 10^{-5} m^3]}{[0.013578 m^2][8.314 m^3 Pa K^{-1} mol^{-1}]} \cdot -44.64 \exp(-0.04t)$$

$$= -6.137 \times 10^{-3} \exp(-0.04t)$$

At the time spent 70 hr, the flux of hydrogen would be

$$= 3.732 \times 10^{-4} mol m^{-2} s^{-1}$$

Appendix D SOLUBILITY CONVERSION

Vijay et al. (1979) proposed the relationship of solubility of hydrogen in carbon steel as a function of temperature. The experiment was conducted by gas-phase hydrogen permeation. The tested was performed in the range of temperature of 500 to 900 K.

Table D.1 Elemental composition of Carbon steel SAE 1095 and ASTM A179

	C	Cu	Cr	Mn	P	Si	S	Fe
SAE 1095	0.90	0.6	-	1.65	0.4	0.6	0.05	balance
ASTM-A179	0.91	-	0.31	0.71	0.37	-	-	balance

For Carbon Steel 1095

$$S = 0.941 \times 10^{-3} \exp\left(\frac{-19280}{RT}\right)$$

Where S = Solubility ($\text{cm}^3\text{H}_2 / \text{cm}^3 \text{ metal}$) at STP
 T = Absolute temperature (K)
 R = Gas Constant, 8.3144 J/mol K

At STP condition

$$C = \frac{n}{V} = \frac{P}{RT} = \frac{1 \text{ atm}}{8.205 \times 10^{-5} \text{ m}^3 \text{ atm} \cdot \text{K}^{-1} \cdot \text{gmol}^{-1} \cdot 298 \text{ K}} \frac{10^{-6} \text{ m}^3}{\text{cm}^3}$$

$$= 4.089 \times 10^{-5} \text{ gmolH}_2 / \text{cm}^3 \text{H}_2 @ \text{STP}$$

In case of the atomic hydrogen

$$C = 2(4.089 \times 10^{-5}) \text{ gmolH} / \text{cm}^3 \text{H}_2 @ \text{STP}$$

Thus, Solubility becomes

$$S = 0.941 \times 10^{-3} \exp\left(\frac{-19280}{RT}\right) \frac{\text{cm}^3 \text{H}_2 @ \text{STP}}{\text{cm}^3 \text{ metal} \cdot \text{Pa}^{1/2}} \cdot \frac{8.179 \times 10^{-5}}{\text{cm}^3 \text{H}_2 @ \text{STP}} \text{ gmolH}$$

$$S = 7.697 \times 10^{-8} \exp\left(\frac{-19280}{RT}\right) \frac{\text{gmolH}}{\text{cm}^3 \text{Fe} \cdot \text{Pa}^{1/2}}$$

Calculation Example

$$S = 7.697 \times 10^{-8} \exp\left(\frac{-19280}{8.314 \times 523}\right) \frac{\text{gmolH}}{\text{cm}^3 \text{Fe} \cdot \text{Pa}^{1/2}}$$

$$S = 9.134 \times 10^{-10} \frac{\text{gmolH}}{\text{cm}^3 \text{Fe} \cdot \text{Pa}^{1/2}}$$

$$S = 9.134 \times 10^{-4} \frac{\text{gmolH}}{\text{m}^3 \text{Fe} \cdot \text{Pa}^{1/2}}$$

Appendix E APPARENT DIFFUSIVITY CALCULATION PROCEDURE

Diffusivity Based on Fick's First Law

Fick's law for the mass transfer through a hollow cylinder which is exposed to fluids at different concentrations is given by

$$\frac{1}{r} \frac{d}{dr} \left(rD \frac{dc}{dr} \right) = 0$$

where D is the apparent diffusivity. The concentration of hydrogen in the tube can be determined by solving the equation above and applying the appropriate boundary conditions. The value D is assumed to be constant. The following expression for the mass transfer rate is obtained:

$$m = \frac{dn}{dt} = - \frac{2\pi D_{H_2} \Delta C}{\ln(r_2/r_1)}$$

The ideal gas law is applied to determine the changing moles in hydrogen in the tube as diffusion occurs.

$$\frac{dn}{dt} = \frac{V}{RT} \frac{dP}{dt}$$

and

$$C = \frac{n}{V} = \frac{P}{RT}$$

$$\int_{P_1}^{P_2} \frac{1}{P} dP = \frac{2\pi L D}{V} \cdot \frac{1}{\ln\left(\frac{r_2}{r_1}\right)} \cdot \int_0^t dt$$

Solving for the diffusivity gives

$$D_{H_2} = \frac{V \ln\left(\frac{r_2}{r_1}\right) \ln\left(\frac{P_1}{P_2}\right)}{2\pi L t}$$

Where D_F	=	Diffusivity based on Fick's law (m^2/s)
P_1	=	Initial pressure (kPa)
P_2	=	Final pressure (kPa)
V	=	Volume of the pressurized chamber (m^3)
r_1	=	Inner radius (m)
r_2	=	Outer radius (m)
L	=	Length of the tube (m)
t	=	time spent (s)

Calculation Example

Hydrogen permeation data of the test no.3

P_1	=	1087.3	kPa
P_2	=	113.34	kPa
V	=	0.00001552	m^3
r_1	=	0.002285	m
r_2	=	0.003175	m
L	=	0.945	m
t	=	258876	s

$$D_{H_2} = \frac{(1.552.87 \times 10^{-5} m^3) \ln\left(\frac{0.003175m}{0.002285m}\right) \ln\left(\frac{1087.3kPa}{113.34kPa}\right)}{2(3.1416)(0.945m)(258876s)}$$

$$= 7.55 \times 10^{-12} m^2/s$$

Diffusivity Based on Sievert's Law

The flux through the membrane is given by the following equation:

$$J_{H_2} = -\frac{D_{H_2} \Delta C_{H_2}}{L}$$

The hydrogen within the metal membrane is in the atomic state, while in the gas phase hydrogen exists as a diatomic molecule (H_2). Thus, the absorption of hydrogen by a metal involves the dissociation of diatomic hydrogen molecules into atomic hydrogen. Under equilibrium conditions, the relationship for the concentration of hydrogen in the metal (C) and the partial pressure of hydrogen in the gas phase (P_{H_2}) is given by Sievert's law:

$$C_{H_2} = K\sqrt{P_{H_2}}$$

The hydrogen flux J_{H_2} through the membrane was determined by

$$J_{H_2} = \frac{V}{ART} \cdot \frac{dP_{H_2}}{dt}$$

Assuming equilibrium of atomic hydrogen at the exposed surface, the equation becomes

$$\frac{V}{ART} \cdot \frac{dP_{H_2}}{dt} = \frac{D_{H_2} K \sqrt{P_{H_2}}}{2L}$$

$$\int_{P_1}^{P_2} \frac{1}{\sqrt{P}} dP = \frac{ART}{V} \cdot \frac{D_{H_2} K}{2L} \cdot \int_0^t dt$$

$$D_{H_2} = \frac{(\sqrt{P_2} - \sqrt{P_1})}{RT} \cdot \frac{V4L}{AKt}$$

where	P_1	=	Initial pressure (Pa)
	P_2	=	Final pressure (Pa)
	D	=	Diffusivity ($m^2 s^{-1}$)
	A	=	Area inside the tube (m^2)
	V	=	Volume of the loop holding the pressure (m^3)
	K	=	Solubility coefficient of hydrogen in steel ($mol m^{-3} Pa^{-0.5}$)
	L	=	Length of the tube (m)
	R	=	$8.314 m^3 Pa K^{-1} mol^{-1}$

T = Temperature (K)
t = time spent (s)

Calculation Example

Hydrogen permeation data of the test no.3

P₁ = 1087.3 kPa
P₂ = 113.34 kPa
V = 0.00001552 m³
K = 9.134 × 10⁻⁴ gmol m⁻³metal Pa^{-0.5}
r₁ = 0.002285 m
r₂ = 0.003175 m
L = 0.945 m
t = 258876 s

$$D_{H_2} = \frac{(\sqrt{1087.3 \times 10^3} - \sqrt{113.34 \times 10^3}) Pa^{0.5}}{(8.314 m^3 Pa \cdot K^{-1} mol^{-1})(523K)} \frac{(1.55 \times 10^{-5} m^3)(0.945m)}{(0.01357 m^2)(9.134 \times 10^{-4} mol Pa^{0.5} m^{-3})(258876s)}$$

$$= 7.84 \times 10^{-7} m^2/s$$

The diffusivity derived by Sievert's law indicated in this thesis is consistent with the transport of hydrogen within the metal. It is also useful in assuming the hydrogen transfer when the hydrogen is generated as atomic hydrogen in a corrosion reaction at the steel surface.

Appendix F OVERALL RESISTANCE CALCULATION PROCEDURE

The mass transfer rate:
$$\frac{dn}{dt} = -\frac{A_s \Delta C}{R_s}$$

The ideal gas is applied to determine the moles of hydrogen diffusion within the tube as a function of time..

$$\frac{dn}{dt} = \frac{V}{RT} \frac{dP}{dt}$$

And
$$C = \frac{n}{V} = \frac{P}{RT}$$

Therefore, the equation for overall surface resistance becomes
$$R_s = \frac{A_s t}{V \ln\left(\frac{P_1}{P_2}\right)}$$

Where	P_1	=	Initial pressure (kPa)
	P_2	=	Final pressure (kPa)
	A	=	Inner Area of the tube (m^2)
	V	=	Volume of the loop holding the pressure (m^3)
	L	=	Length of the tube containing hydrogen (m)
	t	=	time spent (s)

Calculation Example

Hydrogen permeation data of the test no.3

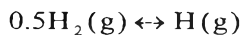
P_1	=	1087.3	kPa
P_2	=	113.34	kPa
V	=	0.00001552	m^3
A	=	0.013578	m^2
t	=	258876	s

$$R_s = \frac{(0.013578 m^2)(258876 s)}{(0.00001552 m^3) \ln\left(\frac{1087.3 kPa}{113.34 kPa}\right)}$$

$$= 1.00 \times 10^8 \text{ s/m}$$

Appendix G EQUILLIBRIUM CONSTANT CALCULATION PROCEDURE

The dissociation reaction of molecular hydrogen to atomic hydrogen is given by:



Thermodynamics properties are shown in Table G.1.

Table G.1 Thermodynamics properties (Perry's Handbook)

Components	ΔG_{298}^f (cals/mol)	ΔH_{298}^f (cals/mol)	C_p (cals/deg/mol)
$\text{H}_2(\text{g})$	0	0	$6.62 + 0.00081T$
$\text{H}(\text{g})$	48585	52100	4.97

$$R \int_{298}^T d(\ln K) = \int_{298}^T \left(\frac{\Delta H_T^0}{T^2} \right) dT$$

$$= \int_{298}^T \left(\frac{\Delta H_T^0}{T^2} + \frac{a}{T} + \frac{b}{2} \right) dT$$

$$R (\ln K_T - \ln K_{298}) = \frac{-\Delta H_T^0}{T} + a \ln T + \frac{b}{2} T \Big|_{298}^T$$

$$R (\ln K_T - \ln \frac{-\Delta G_{298}}{RT}) = \Delta H_T^0 \left(\frac{1}{T_0} - \frac{1}{T} \right) + a \ln \left(\frac{T}{T_0} \right) + \frac{b}{2} (T - T_0)$$

$$\ln K_T = \frac{-\Delta G_{298}}{RT} + \frac{\Delta H_T^0}{R} \left(\frac{1}{T_0} - \frac{1}{T} \right) + \frac{1}{R} \left(a \ln \left(\frac{T}{T_0} \right) + \frac{b}{2} (T - T_0) \right)$$

Apply the thermodynamics data of the dissociation reaction into the equation

$$\Delta H_T^0 = \Delta H_{298}^0 + \int_{298}^T \Delta C_p dT$$

$$\begin{aligned}\Delta C_p &= C_{P(H)} - 0.5C_{P(H_2)} = 4.97 - 0.5(6.62 + 0.00081T) \\ &= 1.66 - 0.000405T\end{aligned}$$

$$\Delta H_T^0 = 52100 - 1.66(298) + \frac{0.000405}{2}(298^2)$$

And

$$\begin{aligned}\sum v_i \Delta G_i^f &= \Delta G_H^f - 0.5\Delta G_{H_2}^f \\ &= 48585 - 0 \\ &= 48585 \text{ cal/mol}\end{aligned}$$

Thus, the relationship between the equilibrium constant (K_T) and temperature is given by the equation:

$$\ln K_T = 0.434 - \frac{26072}{T} + 0.838 \ln T - 2.023 \times 10^{-4} T$$

Appendix H GRAIN STRUCTURE OF CARBON STEEL AND HASTELLOY

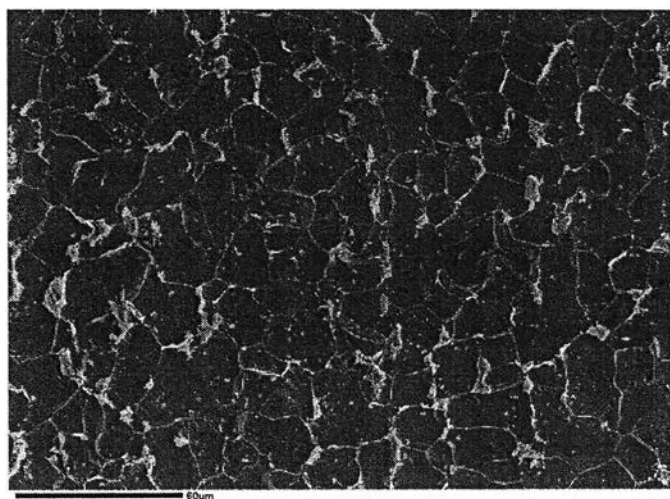


Figure H.1 Grain structure of carbon steel ASTM A-179 (Kongvarhodom,2009).

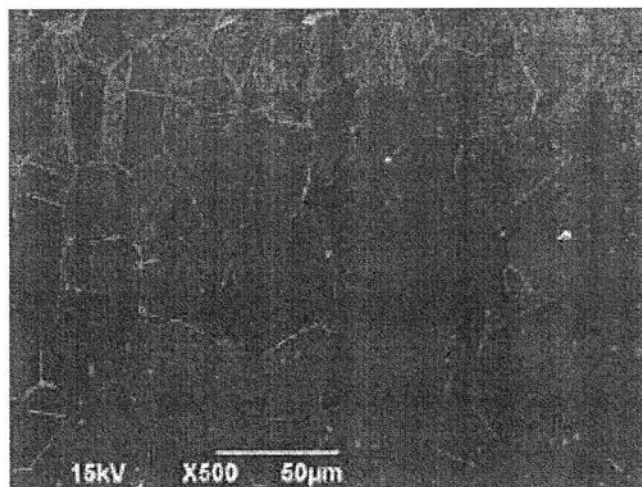


Figure H.2 Grain structure of Hastelloy C-276 (Zhang Q.,2009).

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Presentations:

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