CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Equipment

This research used a computer laptop model: Intel(R) Core(TM) i7-3537U at CPU 2.00 GHz, RAM: 8 GB and 64-bit operating system to analyze plateau pressure and develop software.

3.1.2 Software

Hydrogen pressure rise inside the devices shaped like cylindrical cups at 5 psig gas pressure inside the carbon steel pipe and 300 °C in the case of hydrogen accumulation with hydrogen transfer around the bottom of the cups(edge effect) and diffusing through the cups wall(material effect), was modeled and solved using a program developed in MATLAB.

3.2 Experimental Procedures

3.2.1 Mathematical Models

A hydrogen effusion probe (HEP) consists of several components: cup, silver tube, valve, pressure transducer; and the method for installing the probe on the pipe is important. Consequently, the experimental set up plan and volume of the components should be considered in this calculation for accurate results.

The cups were assumed to be manufactured from stainless steel and carbon steel, and machined to match the curve of the carbon steel pipe on which they will be installed. Four different dimensions of cups for each material were fabricated and installed on a carbon steel A106-B pipe by welding and silver solder with an edge of 0.5 cm. The schematic diagram of the gas permeation loop is shown in Figure 3.1. The pipe and cup connections are made using Swagelok and VCR fittings, respectively.

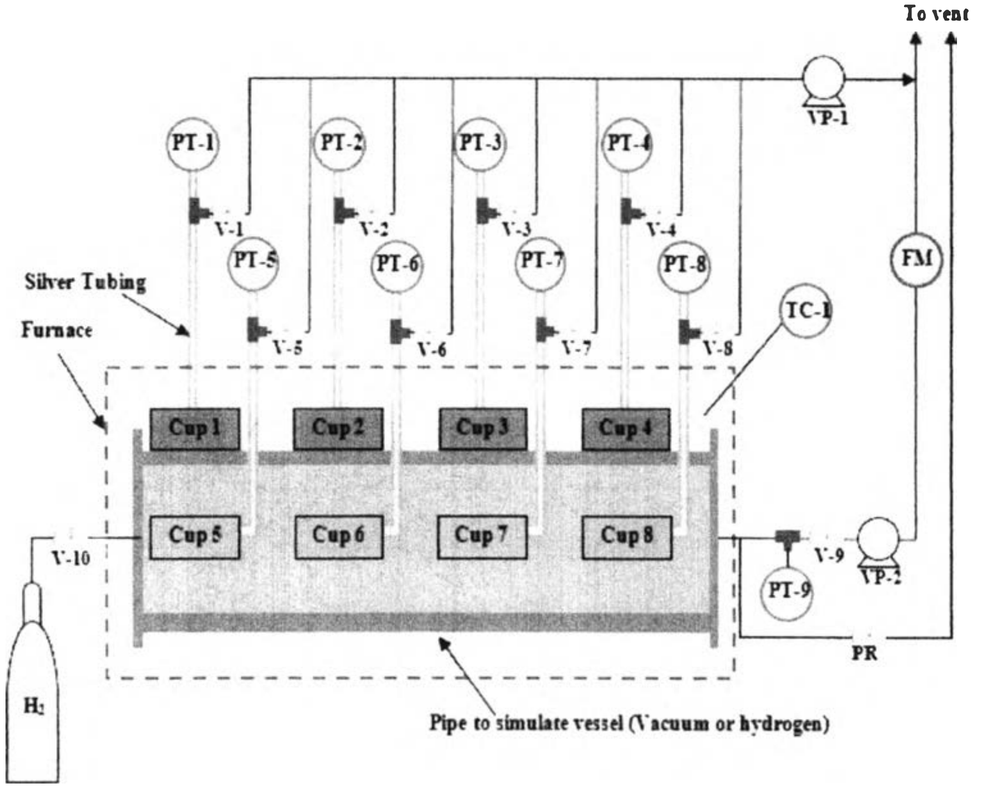

Figure 3.1 Schematic diagram of gas permeation loop.

The pressure transducers were connected to each cup and test pipe via silver tubing and are used to measure the pressure change inside the cups and test pipe labelled as PT-1 to PT-8, which were recorded by a data acquisition system. The temperature inside the furnace was measured by a thermocouple (type K) installed inside the furnace, TC-1. Two vacuum pumps; VP-1 and VP-2, were used for establishing the vacuum in the system. The flow rate of fluid inside the test pipe was controlled by a flow meter, FM, located after the vacuum pump, VP-2. All valves; V-1 to V-10, and transducers were outside the furnace. The system has a poppet check-valve installed with a cracking pressure of 10 psig but a real operating range of 7 to 15 psig. This lower limit (7 psig) is never to be exceeded during experiments to prevent undo venting of hydrogen.

3.2.2 Model Derivation and Model Parameters

The model of hydrogen diffusion and accumulation inside the cups presented in this section is derived from the transport of hydrogen through a pipe based on Sievert's Law:

$$J = \frac{2\pi L\phi(P_{H_{2,f}}^{1/2} - P_{H_{2,p}}^{1/2})}{\ln(r_{p,o}/r_{p,i})\pi D_{p,o} L} \tag{3.1}$$

where:

J – diffusion flux (mol/m^2·s)

L = length of pipe (m)

ϕ = permeability (mol/m·s·Pa$^{1/2}$)

$P_{H_{2,f}}$ = feed side partial pressure of hydrogen (Pa)

$P_{H_{2,p}}$ = permeate side partial pressures of hydrogen (Pa)

$r_{p,o}$ = outer radius of pipe (m)

$r_{p,i}$ = inner radius of pipe (m)

$D_{p,o}$ = outer diameter of pipe (m)

The transport of hydrogen, J, is determined from the Ideal Gas Law based on the increase of hydrogen pressure inside the cups as shown in Equation 3.2 and Equation 3.3:

$$n = \frac{(dP/dt)V}{RT_{mean}} \tag{3.2}$$

$$J = \frac{n}{A} \tag{3.3}$$

where:

n = moles of hydrogen gas inside the cup (mol)

P = pressure rise inside the cup over the test of time t (Pa/s)

t = time (s)

V = total hydrogen gas volume (m^3)

R = ideal gas constant (m^3·Pa/mol·K)

T_{mean} = mean absolute temperature (K)

A = diffusing area which is the outer surface area of pipe under the cup (m^2)

The combination of Equation 3.1 and Equations 3.2-3.3 yields Equation 3.4, which was used to predict the rate of hydrogen pressure rise inside the cups without effect of hydrogen transfer around the bottom and transfer through the cups' wall.

$$\frac{dP}{dt} = \frac{RT_{mean}}{V}\left(\frac{2\pi\phi A(P_{H_{2,f}}^{1/2} - P_{H_{2,p}}^{1/2})}{\ln(r_{p,o}/r_{p,i})\pi D_{p,o}}\right) \tag{3.4}$$

where:

P = pressure rise inside the cup over the test time t (Pa)

t = time (s)

V = total hydrogen gas volume (m^3)

R = ideal gas constant ($m^3 \cdot Pa/mol \cdot K$)

T_{mean} = mean absolute temperature (K)

A = diffusing area which is the outer surface area of pipe under the cup (m^2)

ϕ = permeability of carbon steel A106-B pipe ($mol/m \cdot s \cdot Pa^{1/2}$)

$P_{H_{2,f}}$ = feed side partial pressure of hydrogen which is the hydrogen pressure inside the pipe (Pa)

$P_{H_{2,p}}$ = permeate side partial pressures of hydrogen which is the hydrogen pressure inside the cup (Pa)

$r_{p,o}$ = outer radius of pipe (m)

$r_{p,i}$ = inner radius of pipe (m)

$D_{p,o}$ = outer diameter of pipe (m)

From the literature, the hydrogen transfer around the bottom of the cup was found in the study of Kongvarhodom (2014). Both materials; stainless steel and carbon steel; have moderate hydrogen permeability. Consequently, Equation 3.4 was developed to include the effect of hydrogen transfer around the bottom of the cups and diffusing through the cups wall on the accumulation inside the cups as shown in Equation 3.5. The second and the last terms represent the edge effect and the material effect.

$$\frac{dP}{dt} = \frac{RT_{mean}}{V}\left(\left(\frac{2\pi\phi A(P_{H_2,i}^{1/2} - P_{H_2,o}^{1/2})}{\ln(r_{p,o}/r_{p,i})\pi D_{p,o}}\right) - \frac{p_c l_H \phi P_{H_2,r}^{1/2}}{l_c} - \frac{2\pi\phi_c h_m P_{H_2,p}^{1/2}}{\ln(r_{c,o}/r_{c,i})}\right) \quad (3.5)$$

where:

p_c = perimeter of cup (m)

l_H = width of hydrogen diffusion path leaving the cup (m)

l_c = width of diffusing element which is the thickness of cup wall and silver solder around the cup (m)

ϕ_c = permeability of cup's material (mol/m·s·Pa$^{1/2}$)

$r_{c,i}$ = inner radius of cup (m)

$r_{c,o}$ = outer radius of cup (m)

h_m = inner height of cup (m)

This equation was used to predict the pressure increase of the cups with time and compared with the experimental data.