



CHAPTER III EXPERIMENTAL

In this chapter, the experimental set-up for the oxide formation apparatus is described. Details of all components including the materials and chemicals, equipment and experimental procedure follow.

According to the constructed test section, it has been calculated that the internal volume is $\sim 13 \text{ cm}^3$ (10.7 cm^3 for tubing, $\sim 0.3 \text{ cm}^3$ for the flange and $\sim 2 \text{ cm}^3$ for the pressure transducer). However, the exposed surface area of the membrane within the internal volume is 3.5 cm^2 . For an average plant installation a footprint of $\sim 20 \text{ cm}^2$ (for a 50 mm cup size) and an internal volume of $\sim 10 \text{ cm}^3$ is expected, thus a ratio of of $\sim 2 \text{ cm}^2/\text{cm}^3$. In order to simulate the constructed test section environment, $\sim 22.5 \text{ cm}^2$ of extra polished surface will be required for each experiment. The required amounts of extra test material to be installed in the test are shown in table 3.2.

3.1 Materials and Equipment

3.1.1 Materials

- Materials for fabrication of test membranes (CS A106B, SS 316 and Ni-Alloy 625)
- CS, SS and Ni-Alloy wire (extra surface)

3.1.2 Equipments

- Polisher with media; abrasive paper (grit 60, 120, 240, 400, 600, 800 and 1200), nylon cloth with 1 and 6 micron diamond solution
- Furnace
- Data acquisition system and computer
- Pressure transducers, range vacuum to 100 psig --PTI-S-NC100-17AQ
- Thermocouple
- Flanges, 1 1/3" OD--F0133X4SWG

- Flange bolt and nut set -- SBS832075
- ¼" Swagelok fittings
- ¼" OD x 0.049" wall x 3' long stainless steel tubing

3.2 Apparatus Set-up

The apparatus set-up is shown in Figure 3.1. The flanges and membrane are placed in the furnace/freezer and connected to a pressure transducer which is installed outside the furnace/freezer. The pressure transducer is connected to a data acquisition system and a computer.

The membrane is installed between two flanges; one side connects to the pressure transducer to measure the pressure change. This side of the membrane will be exposed to the simulated environment of the constructed test section. The other side is exposed to atmospheric air or can be connected to another cavity with a pressure transducer set for further study. Both sides of the membrane will be analyzed.

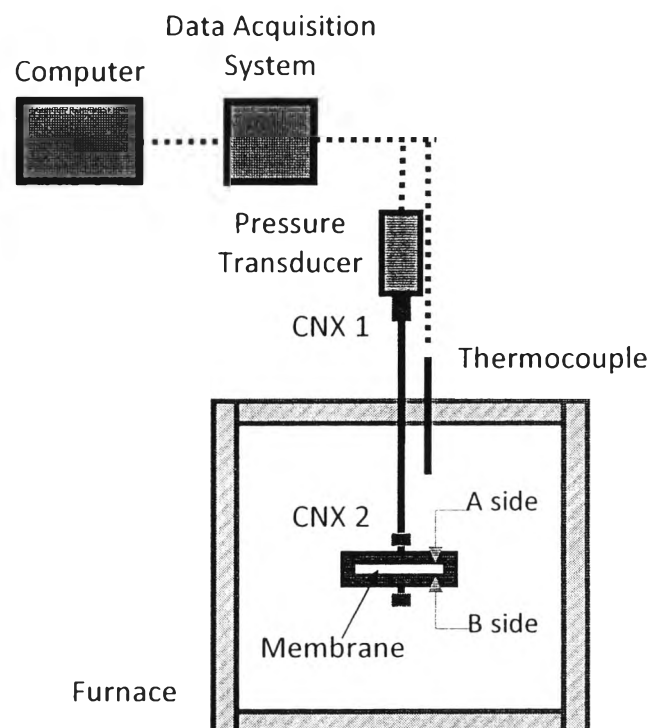


Figure 3.1 Apparatus with one membrane assembly.

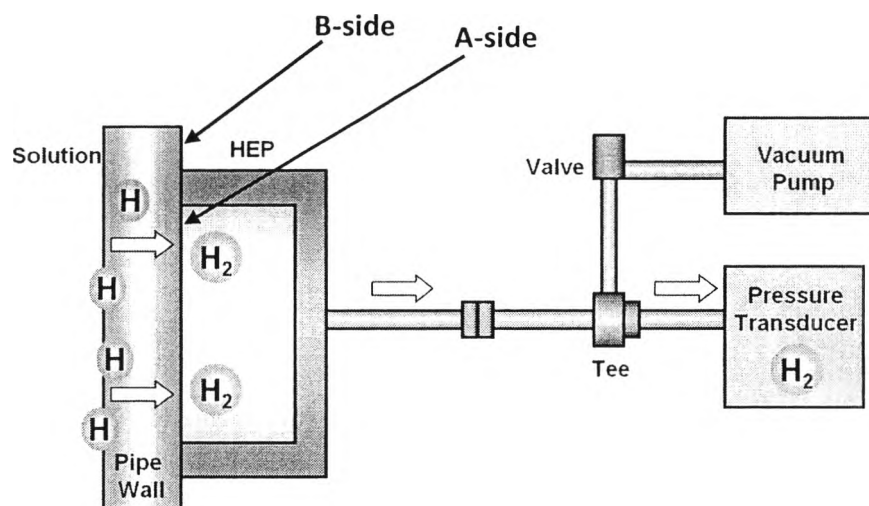


Figure 3.2 Schematic of the constructed HEP instrument.

From Figures 3.1 and 3.2, the A-side of the membrane was exposed to the simulated environment of the metal surface inside the instrument which changes in oxygen concentration during the exposure. The B-side was exposed to the free-air environment without changing in oxygen concentration during the exposure. This side demonstrated the oxidation of metal on the vessel surface outside the cup.

3.3 Experimental Procedure

3.3.1 Surface Preparation

Surface preparation of each material was required in order to eliminate any oxide film that may be present and provide a consistent starting surface condition.

3.3.1.1 *Stamping Membranes*

Each membrane was stamped at approximately half of the radius of the membrane (to avoid the sealing surface and center use for surface analysis) as shown in Figure 3.3, rinsed and dried using degreaser and lint free tissue.

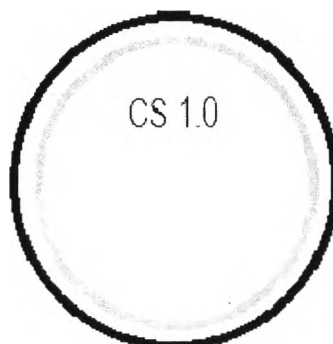


Figure 3.3 Stamp example.

3.3.1.2 Polishing Sample Surfaces

Each surface was polished with abrasive papers; 60, 120, 240, 400, 600, 800 and 1200 grit, then further polished with diamond solution (6 μm and 1 μm) on nylon cloth. After polishing in each step the surface was rinsed using acetone and dried by lint free tissue.

After all of polishing steps were performed, the surfaces were examined for defects or scratches. Surfaces should have a mirror finish with no defects or scratches and stamped labels were legible. If defects or scratches were present, some polishing steps were repeated.

All samples were stored in an argon-purged desiccator between steps and while waiting for the test.

Note: Stamping membranes using the following label;

- CS for low chrome carbon steel (CS A106B)
- SS for super corrosion resistance (SS 316/316L)
- NI for high strength nickel alloy (Ni-Alloy 625)
- 1.0 for run 1, 2.0 for run 2,... and 9.0 for run 9

3.3.2 Test Matrix and Extra Surface Required

The experiment was divided into 2 parts. The second part was performed after the results of the first part showed a change of oxide type on carbon steel surfaces. Also carbon steel and stainless steel which were exposed to 90°C, and nickel-alloy and stainless steel exposed to 400°C showed no significant weight gain

on the metals after the exposure. Therefore, the second part of experiment was focused on the oxide formation of carbon steel at 400°C. The test matrix is shown in Table 3.1 and the required extra surface is shown in Table 3.2.

Table 3.1 Test matrix

	Run	Pressure Transducer	Test Materials		Temp. (°C)	Time
			Membrane	Extra Surface		
Part 1	1	P5	CS A106B	CS wire	400	1 day
		P6	SS 316	SS wire		
		P7	Ni-Alloy625	Ni wire		
	2	P5	CS A106B	CS wire	400	7 days
		P6	SS 316	SS wire		
		P7	Ni-Alloy625	Ni wire		
3	P5	CS A106B	CS wire	400	14 days	
	P6	SS 316	SS wire			
	P7	Ni-Alloy625	Ni wire			
Part 2	4	P5	CS A106B	CS wire	90	1 day
		P6	SS 316	SS wire		
	5	P5	CS A106B	CS wire	90	7 days
		P6	SS 316	SS wire		
	6	P5	CS A106B	CS wire	90	14 days
		P6	SS 316	SS wire		
Part 2	7	P6	CS A106B	CS wire	400	7 days
		P5	-	CS wire		
	8	P6	CS A106B	CS wire	400	1 day
		P5	-	CS wire		
	9	P6	CS A106B	CS wire	400	5 hr
		P5	-	CS wire		

Table 3.2 Extra polished surface requirement

Material	Form	Diameter (cm)	Required Area (cm²)	Required Length (cm)
CS	Wire	0.07	24.4	105
SS	Wire	0.05	24.4	144
Ni-Alloy	Wire	0.04	24.4	190

3.3.3 Preconditioning Components

Three set of apparatus were assembled up to the location of connection (CNX 2 in Figure 3.1) and the ¼” tubing were inserted to the top of the furnace. Both halves of the flange assembly were placed (for all three test sections) in the furnace without a membrane installed.

The furnace was closed and the temperature was set to 400°C for two weeks after tubing and flange were brought to 400°C.

3.3.4 Testing Procedure

The pressure transducers and thermocouples were connected to the data acquisition system to ensure proper measurements were obtained (as show in Figure 3.1). The top half of the flange assemblies were attached to the ¼” tubing for all three test sections.

The required surface was inserted into the tube (ensured that this materials is contained in tubing that will be in the furnace) and then a membrane was carefully installed in each of the three test sections (using the properly labeled membranes from the preparation phase). Bolts were tightened in cross pattern until the flanges met (i.e. the two flange halves engage).

The furnace was closed and the temperature set to the required temperature. The test was terminated at the time given in Table 3.1.

Once the furnace cooled, bolts were removed from the flange assemblies and the membranes were transferred back into the original cloth and envelope and placed in an argon-purged dessicator to await surface analysis.

This procedure was followed for each condition in Table 3.1

3.4 Sample Analysis

Each sample (including the baseline samples) was weighed before and after the exposure to obtain the weight gain on the surface and then sent to surface analysis which included;

Field Emission Scanning Electron Microscopy (FE-SEM) for the surface topography including an image of oxide crystals formed on both sides of the membrane surface and fresh polished surface.

Energy Dispersive Spectroscopy (SEM-EDS) for determining the chemical composition of elements on the membrane surface.

Raman Spectroscopy to indicate the type of oxide formed on the surfaces.