CHAPTER III
EXPERIMENTAL

3.1 Materials

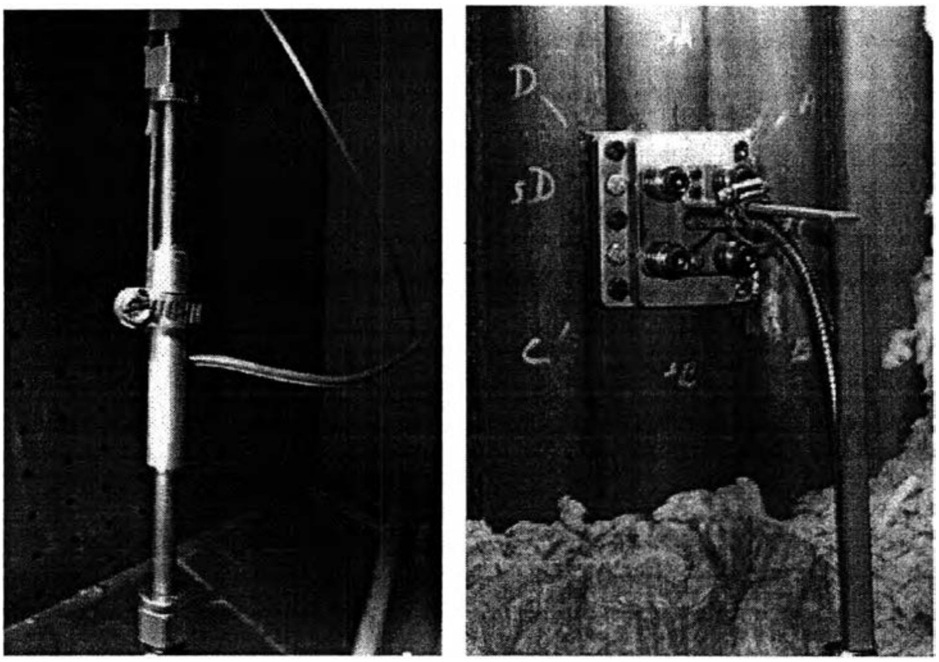

Figure 3.1 Configurations of the Hydrogen Effusion Probe, HEP; the HEP installed on the test section of Loop1 at CNER (left), and the HEP installed on the feeder tube walls at Coleson Cove generating station (right).

The experiment was carried out on the lab scale HEP as shown in Figure 3.1. The HEP consisted of a carbon steel ASME SA106 Grade B tube covered by a silver annulus to trap the hydrogen that diffuses through the probe wall from the corroding inner surface. Silver was used because of its low permeability to hydrogen gas at high temperature. The silver annulus was connected via silver capillary tubing to the instrumentation cabinet containing a valve, pressure transducer, vacuum pump and controller unit.

In order to determine the ability of the HEP to monitor the corrosion rate in the field applications, HEPs were installed on a feeder pipe at the Point Lepreau Generating Station (PLGS), and on a boiler wall in the Coleson Cove Generating

Station (CC). The assembly of the HEPs used in the field is different from the one used in the experimental loop as can be seen in Figure 3.1. The hydrogen accumulation section was made of a silver cup inside a carbon steel clamp instead of a silver annulus covering the pipe as used in the lab. The silver cup is connected by silver tubing to stainless steel tubing which runs to the instrumentation cabinet.

Table 3.1 Chemical composition of carbon steel ASME SA106

Element	Mass fraction (%)
Fe	Balance
C	0.30
Mn	0.29
Si	0.10
P	0.04
S	0.04
Cr	0.03

3.2 Equipment

The experiment was conducted in the CNER Test Loop1 as shown in Figure 3.2. This loop was designed to simulate the operating conditions of the primary heat transport system in a CANDU reactor. However, light water was used as testing coolant instead of heavy water. The experiment was performed under the same flow velocity and solution chemistry, 4.5 m/s coolant velocity, low oxygen concentration (<1 ppb) and 9.9 pH_{25} of solution as in the station. Loop chemistry and operating conditions are outlined in Table 3.2. The HEP was placed in the test section of the loop. During normal operation, the controller unit automatically switches on the vacuum pump and opens the valve once the measured pressure reaches 1000 Pa.

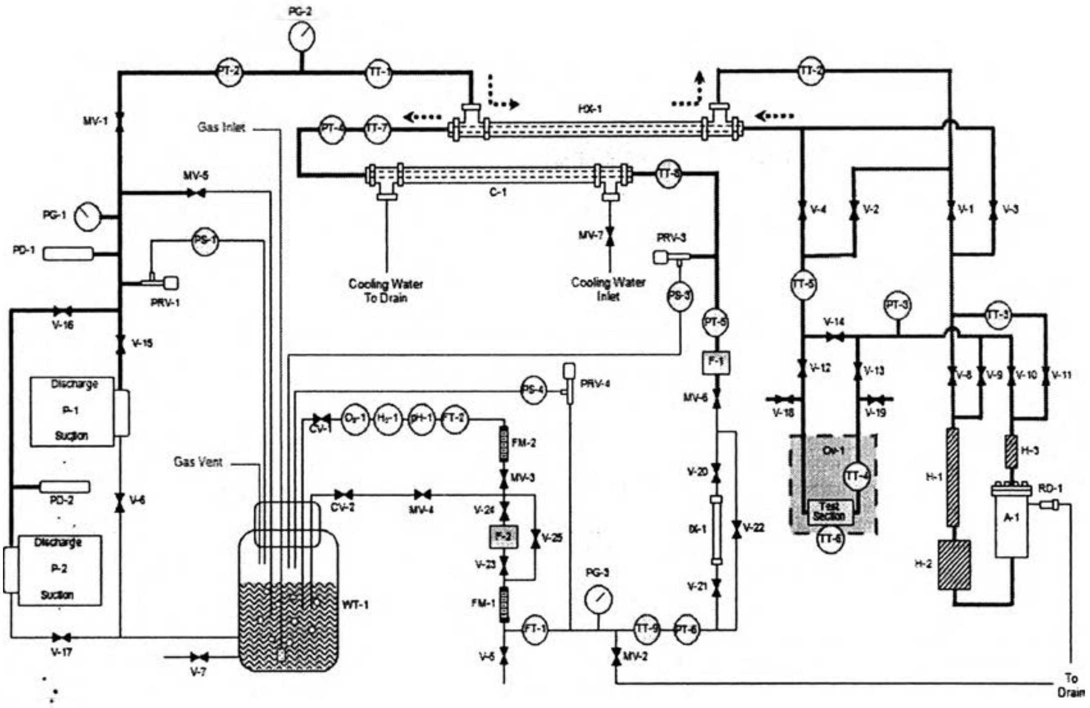

Figure 3.2 Schematic diagram of the CNER Test Loop1.

Table 3.2 Loop 1 process parameters

Parameter	Value	Allowable Deviation
Oxygen	<1ppb	+ 1 ppb
Hydrogen	3-10 cm^3/kg	6.5 ± 3.5 cm^3/kg
pH	9.9	± 0.15
Chlorides	<detectable	-
Flow Rate	840 mL/min	± 100 mL/min
Pressure	1400 psi	± 50 psi

3.3 Experimental Procedures

3.3.1 Study of the Effect of Temperature and Hydrogen Transport

First, the inside of the HEP was cleaned using 10% by volume HCl solution. The cleaned probe was placed in an oven at 300 °C to degas all soluble gases within the metal. The pressure of the HEP was monitored until there was no pressure increase which indicated that all gases had been removed from the steel.

The experiment started with no flow of solution in the test section at 80 °C. Then the test section valves (V-12 and V-13) were opened quickly to let the solution flow into the test section. The hydrogen pressure was measured at this temperature for 2 days. The loop operating temperature was then increased to the normal operating temperature of the test loop, 300 °C.

To study the effect of temperature on the corrosion rate and the performance of the HEP, the experiment was started at a temperature of 300 °C and was operated at constant temperature for 7 days. Next the solution temperature was reduced to temperatures of 260 °C and 220 °C. The temperature of the system was maintained constant for 7 days at each of these temperatures. Temperatures lower than 200 °C were not studied, since the fluid flow rate is normally changed substantially in the plant when the operating temperature is less than 200 °C. The HEP monitored the change in hydrogen pressure increase with time.

The test section was returned to operation at a temperature of 300 °C for 15 days. Then the temperature was reduced to 80°C and 1 ppm of oxygen was dissolved into the solution. It was expected that oxygen would replace hydrogen as the oxidizing species during FAC. Therefore the hydrogen pressure was expected to stop increasing.

3.3.2 Theoretical Studies and Plant Data Analysis

An HEP has been installed at the plants after the pipe surface was cleaned by removing the existing exterior surface oxide layer. The integrity of the seal is verified by determining the air ingress leak rate at all junction points. During normal operation, the controller unit automatically switches on the vacuum pump and opens the valve once the measured pressure reaches 2000 Pa. The data of temperature and pressure are continuously recorded by the data acquisition system. These data was analyzed and compared to the experimental data. Besides the fundamental principle of hydrogen transport through steel was reviewed and summarized in this work.

3.4 Assumptions for Thinning Rate Measurement by the HEP

The requirements for an HEP to be used for the quantitative measurement of FAC rates are as follows (CNER, 2006):

1) The stoichiometric factor relating the rate of atomic hydrogen production to the rate of iron atom oxidation is fixed, and is defined by the reaction,

$$Fe + 2H^{+} \rightarrow Fe^{2+} + 2H\,.$$

This means that for every one mole of iron lost, one mole of H_2 is produced.

2) All the atomic hydrogen produced by the FAC process is absorbed locally by the carbon steel experiencing FAC and recombines into molecular hydrogen.

3) All the absorbed hydrogen diffuses through the wall of the carbon steel pipe experiencing FAC, and recombines to molecular hydrogen at the external interface, and passes into the gas phase in contact with the external surface.

4) Molecular hydrogen, once formed within the collection volume does not diffuse back through the pipe wall.

5) Any molecular hydrogen dissolved in the electrolyte coolant does not contribute to the hydrogen flux through the wall of the pipe. All of the hydrogen that effuses through the steel comes from the corrosion reaction.

6) .A very low concentration of oxidizing species is present in the coolant

7) Atomic hydrogen does not accumulate at the grain boundaries or at defects

8) The area over which colleted hydrogen is generated is assumed to be thinning at a uniform rate

9) The molar mass and density of the carbon steel are assumed to be equal to those of pure iron.