CHAPTER III
EXPERIMENTAL

3.1 Materials

To study the effect of flow velocity on oxide film characteristics, oxide film was formed on A106B carbon steel probe under four different velocities 5, 10, 20 m/s respectively. The chemical composition of A106B steel is shown in Table 3.1.

Table 3.1 Chemical compositions of A106B carbon steel

Chemical compositions	%weight
C	0.30
Mn	0.29
Si	0.10
P	0.035
S	0.035
Cr	0.03

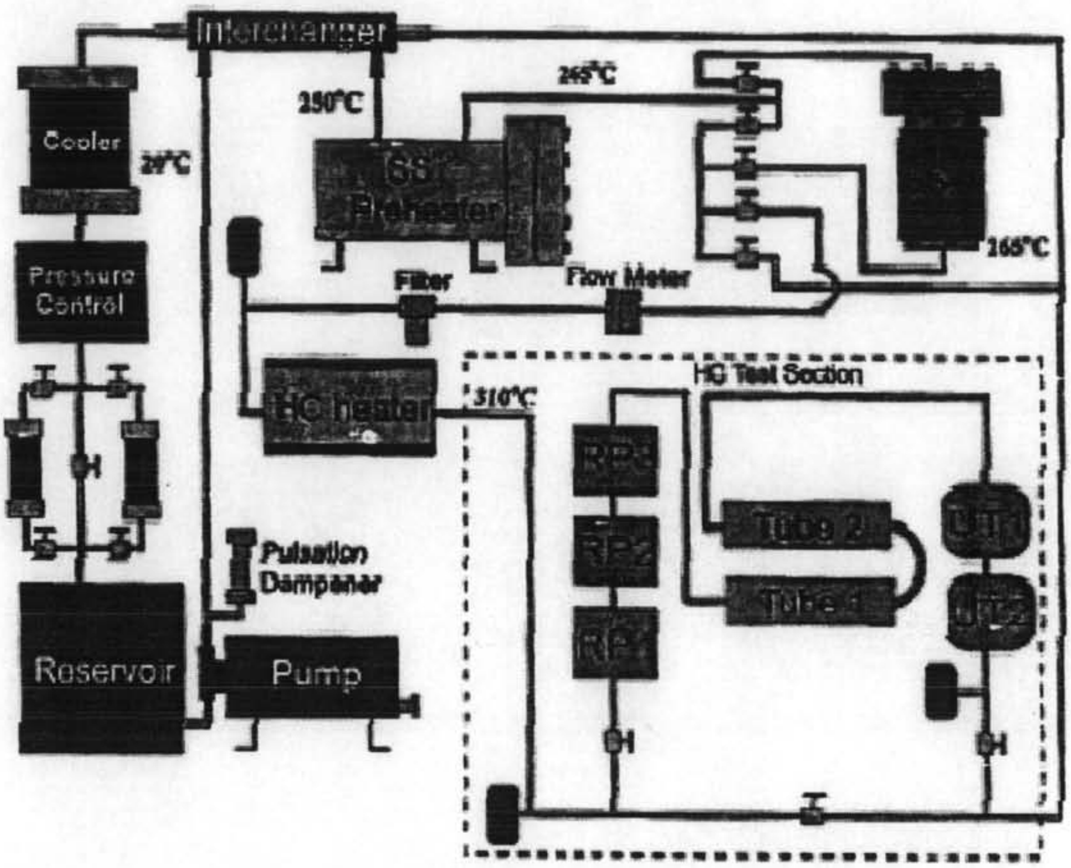

Figure 3.1 The schematic diagram of simulated primary coolant system of CANDU reactor (Cook, 2000).

Different flow rates of coolant at 310^{o}C flowed through the probes at pH 9.9 in the simulated high-temperature test loop as shown in Figure 3.1. LiOH solution is added to maintain the pH in the loop. This loop is 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 water solution was pumped from the reservoir through the SS pre-heater to pre-heat the water to 225^{o}C. It then flowed through the autoclave, filled with iron fillings which represents the inlet feeder pipe so that the coolant is saturated in iron at 265^{o}C. The coolant passes through an inert heat exchanger, made by zirconium and nickel and is heated to

310°C to ensure that no addition iron is added during the heating. The water coolant then flows to test section which is at outlet feeder pipe conditions.

Surface analysis techniques were used to characterize the oxide film formed under different coolant velocities. In order to prepare the samples for analysis, all pre-filmed tube probes were cut by a special cutting method to avoid moisture content and heat generated during the cutting.

3.2 Equipment

Surface analysis is provided by EDX coupled with SEM. An EDX Pheonix X-ray microanalyser with a Si (Li) detector and Genesis microanalysis software was used to analyze the chemical content in the oxide film at operating conditions for EDS X-ray analysis, 15 kV and 1.5 nA. JEOL-6400 SEM collected the oxide images with an accelerating voltage of 15 kV and the beam current was 150 pA. To obtain cross section images of oxide thickness, TEM was carried out in JEOL 2010 STEM operated at 200 keV. The spectra were corrected by using an EDX Genesis 4000 system. A "Lift Out" technique was used for polishing the metal surface by an ion-beam of gallium (Ga^{+}) until the desired oxide film perforation (120-500 nm thick), was obtain when preparing a sample for TEM.

3.3 Methodology

SEM couple with EDX were use to study the morphology, thickness and chemical composition of oxide films. SEM gives images on the oxide surface and the cross section. Special cutting is required to provide samples, at approximately 1,000X and 10,000X magnifications. EDX provides chemical information on the oxide film. For TEM analysis, sample preparation was required to investigate the cross section and morphology images. Moreover, TEM couples with electron diffraction patterns were also conducted to investigate the microstructure of oxide films.