CHAPTER IV

EXPERIMENTAL METHOD

This Chapter discusses the experimental system, the method of preparation of accretion samples for microstructure investigation and analysis techniques used during the course of the project.

4.1 THE ACCRETION SAMPLE CODE.

The accretion samples in this project were collected from different locations in the Kalgoorlie nickel flash smelter. The G3, G4, H3 and H4 samples were collected from burner ports in the settler of the furnace, at the locations shown in Figure 4.1. The appendage samples (APP) were collected from the appendage roof near the appendage electrodes. The dust samples were collected from the dust recycle stream. The laboratory accretions made from furnace dust have a different code from the furnace accretions. A 7-digit code was used for the laboratory accretions. The experimental temperature is used in the first 3-digits, the 4^{th} digit from the left is the time the sample was held at a constant temperature, the last 3-digits refer to the oxygen potential, for example, the sample code 1353-40 refer to a temperature

of 1350 ° C, a holding time of 3 hours and an oxygen potential of -40 Kcal.

4.2 PREPARATION OF SAMPLES

The accretion samples were mounted in resin and left for 24 hours for curing. The surface of the samples was prepared by grinding with silica carbide paper and polished by 6 μm-diamond paste. The etchants shown in Table 4.1 were used to etch specific components of the accretions.

4.3 RECREATED LABORATORY ACCRETIONS

The objective of these tests was to study the mechanism of formation of the accretions by heating dust samples from the Kalgoorlie flash furnace over the temperature range 1350-1400° C at known oxygen partial pressures. Six gram samples of dust contained in an alumina crucible were heated to 1350 and 1400 °C and 1450° C, at oxygen pressures between 10^{-7} to 10^{-4} atmosphere for a holding time of 3 hours (Table 4.2). The experimental method for recreating the accretions requires generating the gas composition predicted by free energy calculations (see Appendix A). Pure CO and CO_2 gases were used for controlling the oxygen partial pressure in the furnace, which

was calculated using the equation:

$$2CO_{(g)} + O_{2(g)} = 2CO_{2(g)}$$

The CO/CO_2 gas ratio used depends on the system being investigated as shown in Table 4.2. The effect of experimental temperature on the formation of accretion phases was also studied. The samples of laboratory accretions were investigated using an optical microscope and a scanning electron microscope (SEM) after mounting, grinding, polishing and etching.

The experimental tube furnace in Figure 4.2 was designed to allow the reaction gas to enter the furnace at the bottom and pass upward through the reactor tube and out at the top. The main tube (2300 mm. long x ϕ55 mm. O.D. x ϕ50 mm. I.D.) was positioned concentrically inside the furnace, and enclosed with a water cooling cap. The water cooling cap was constructed with a screw thread and O-ring for sealing. The cylinder cap contained the inlet for the reaction gases (CO, CO_2). The temperature of the crucible which contained the dust sample was measured by a type K thermocouple which was positioned directly below the crucible. A Bronkhorst High-Tech B.V. mass flow controller was used to mix and control the ratio of the reaction gas mixture (CO/CO_2).

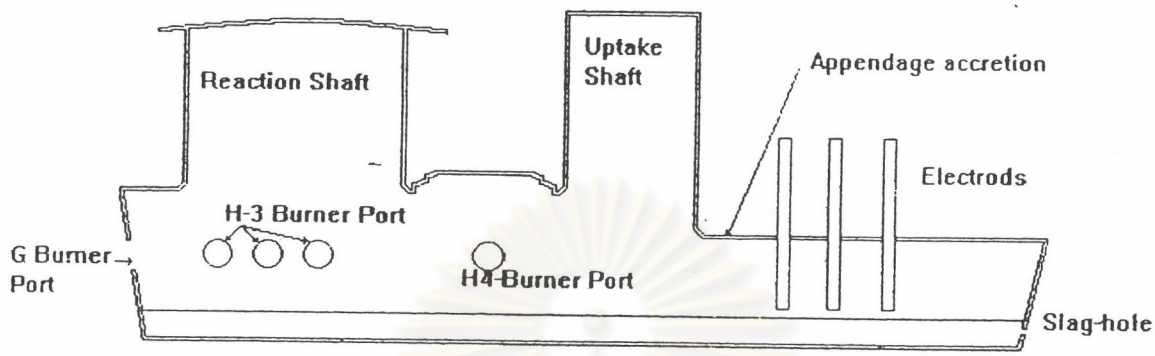

Figure 4.1 Location of Collected Accretion Samples in Flash Furnace.

Table 4.1 The etchants used in microstructure examination.

Etchants	Volume,ml
Methanol (95%)	100
Hydrochloric acid	3
Hydrofluoric acid	0.5
Glycerol	20-40
40% Nitric acid	10
Hydrochloric	20
Methanol	100
Hydrochloric	5

Table 4.2 Temperature and Gas Composition used in Experiments.

Sample Number	Temp °C	CO/CO_2	$\mu(O_2)$Kcal	Time,hours
1353-40	1350	1/99	-40	3
1353-50	1350	7/99	-50	3
1353-60	1350	13/87	-60	3
1403-40	1400	3/97	-40	3
1403-50	1400	13/87	-50	3
1403-60	1400	38/72	-60	3

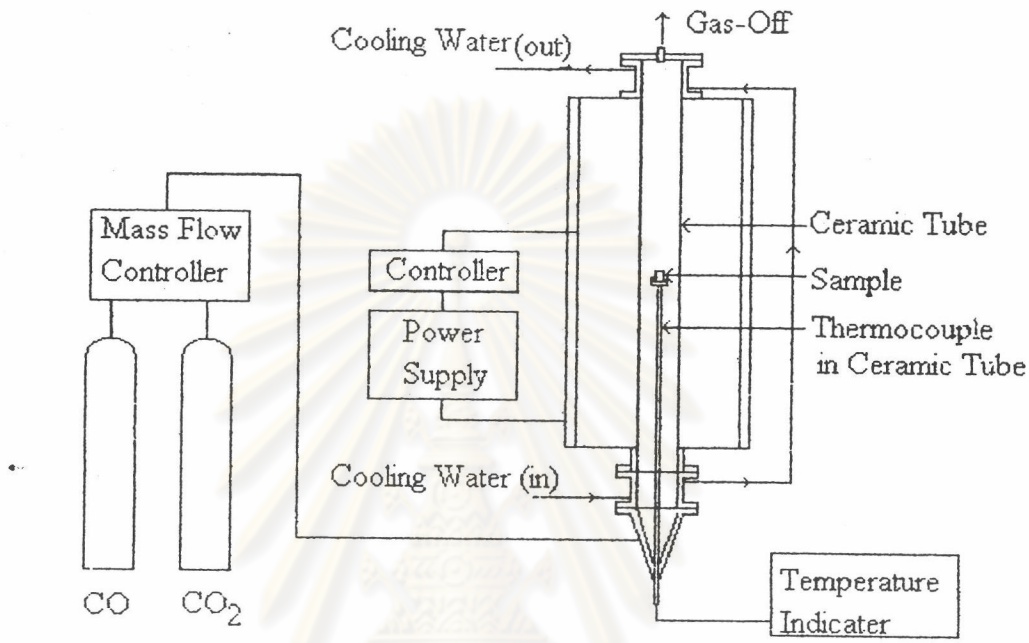

Figure 4.2 Schematic of the Vertical Tube Furnace