3.3 Experimental set-up

This section will give a description of the equipment used in the production of the slags investigated in this work.

3.3.1 Cold crucible induction furnace

The cold crucible high frequency induction furnace consists of a power supply from Farfield Electronics PTY Ltd and a water cooled copper crucible from ANSTO (Australian Nuclear Science and Technology Organization). The high frequency generator operates at 750 kHz and has a power capacity of 75 kVA. The power control on the furnace consists of a radio tube where an acceleration current is applied. This results in a current running through the induction coil. The power control for the experiments is the measured current running through the induction coil. All slags have been kept at equal power setting, thus exposing the slags to equal conditions. This is also done because it is not known how properties like the electrical conductivity of the slag or the liquidus temperature is influenced by the composition of the slags. Changes in these properties will probably influence the slag's ability to 'pick' the field. Thus, at equal power setting one controllable parameter is kept constant. A picture of the furnace is shown in figure 3.3 and a sketch of the crucible and furnace chamber is given in figure 3.4. The coil and furnace chamber is located within a Faraday cage as can be seen in figure 3.3.

The water cooled copper crucible is vertically segmented in order to prevent induction in the crucible. The gaps between the segments are filled with boron nitride to prevent charge materials falling out during charging, and melt to flow into the gaps. The inside of the crucible has also been treated with a boron nitride coating. This coating will limit the wear and tear on the crucible as well as making the extraction of the sample from the crucible easier. The coating is assumed not to

Figure 3.3: Picture showing the cold crucible induction furnace ©Melinda Gaal

contaminate the samples. The crucible has one water inlet and one outlet connection (8), thus the cooling is internal.

The furnace chamber is constructed by fitting a pyrex cylinder (1) between rubber gaskets on the crucible rim (9) and the top connection hub (3). On the top connection the part in contact with the pyrex glass is also water cooled as it also needs to withstand the radiation from the molten material. The top hub features a connection to a vacuum pump that will evacuate the furnace chamber to below $5*10^{-1}$ mbar prior to melting. The gas in- and outlet (4) are also located in the top hub. A filter is connected to the gas outlet to minimize particles escaping into the atmosphere. At the top of the hub there is a window(5) that enables the operator to visually observe the molten material through a mirror placed over the window at a suitable angle. The temperature measurement during operation is conducted by using a spectropyrometer (6) (see section 3.3.2) mounted over the top hub also viewing through the window on the hub.

A mica sheet (2) is placed between the induction coil (7) and the furnace chamber pyrex glass and a second one is placed between the glass and the crucible to prevent arcing between the coil and crucible. Finally two pyrex cylinders are placed on top of the crucible (not included in the sketch) to act as radiation and splashing shields for the outer pyrex cylinder. The crucible, furnace chamber, top hub and filter is encapsulated in a Faraday cage to lower safety risks.

3.3.2 Spectropyrometer

The equipment for measuring the temperature during the experiments in the cold crucible induction furnace is a FAR Associates spectropyrometer of the type FMP 2. This spectropyrometer measures on 500 wavelengths and thus calculates and corrects for the emissivity of the specimen. From the spectropyrometer the central temperature is logged along with the tolerance (standard deviation) of the measure-

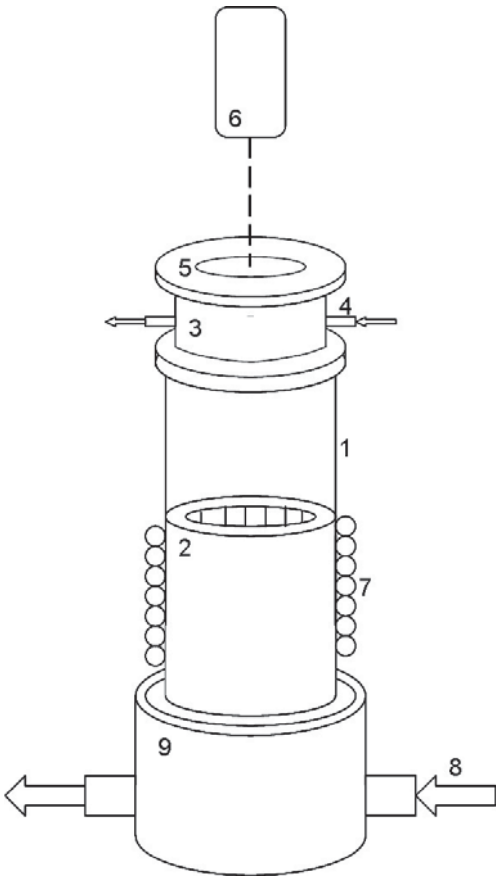

Figure 3.4: Sketch of the cold crucible furnace chamber

ment and the signal strength. The tolerance is the standard deviation calculated from the 500 measured temperatures. The signal strength is equal to the emissivity under ideal conditions, i.e. the area in focus must be filled, there must be no attenuation and clean optics. The range of the pyrometer is from 800 to 2500 °C with an accuracy of ± 0.25 to 0.75 % on non grey targets[1]. Automatic compensation for absorbtion or emission from the off-gas is also included. The lograte of the pyrometer can be set at given intervals or whenever the pyrometer has enough information for an accurate reading. It is this last lograte which is used in this work and this corresponds to a loginterval of about 5 readings per second during the holding time. The loginterval will decrease as the temperature is lowered due to less thermal radiation. It should also be noted that the temperature measurement is not coupled to the furnace power setting. For further information about the spectropyrometer the reader is refered to the FAR Associates web-site[2] or publications such as 'Pyrometry for Liquid Metals' by Felice[63].

During cooling in the crucible, a sudden steep drop is observed in the temperature curve. The temperature is at first steadily decreasing in the melt before this drop. The drop start corresponds to the initial solidification on the top of the sample which is also visually observed. All the way up to this solidification a stirring action is observed in the slag. It is thus believed that the slag's liquidus temperature is located at the start of this drop. This type of liquidus temperature measurements is generally done for slower cooling and a thermal arrest is observed (as used by Grau [21]). Hals [64] used the same equipment as that used in this work to melt a titanium alloy and equivalent observations were made on the cooling curve where the point of origin

[1]For a non grey surface the emissivity is a function of the wavelength, whereas a grey surface has a constant emissivity of less than unity and for a black surface the emissivity is unity.

[2]www.pyrometry.com

for the drop corresponded well to the melting temperature of the alloy. It is believed that this is a promising method to determine the liquidus temperature of the slag. This will be discussed further in section 6.5.

3.4 Characterization

This section will give a brief description of the different methods and analysis equipment which have been used to characterize and further investigate the samples. Some of the analysis needs to be further treated/corrected before plotted and utilized further. Such procedures are also given in this section.

3.4.1 X-ray fluorescence (XRF)

The XRF analysis is performed at the laboratory of Eramet Titanium & Iron AS in Tyssedal, Norway. The XRF machine is of the type Philips PW2404. From the XRF analysis the total amount of TiO_2, FeO, MnO, CaO, MgO, SiO_2, Cr_2O_3, V_2O_5, Al_2O_3 and Nb are obtained. This analysis method is based on bombarding the sample with high energy X-rays which will lead to changes in the electrons in the various elements. When the initial state is restored for the elements secondary X-rays are emitted from the sample and may be detected. Lighter elements such as oxygen are not detected using this procedure, thus, when analysing oxide mixtures it is the cations that are analysed and the oxide content is calculated for a given phase. When a sample contains more than one oxidation state of an element additional analyses must be made. In the case of high titania slags the amounts of TiO_2 and FeO will need to be corrected as some of the titanium will be trivalent and some of the iron will be in its metallic state. The additional analyses are explained in section 3.4.2 and the correction procedure is explained more in detail in section 3.4.6.