

5. TESTING PHASE RELATIONSHIPS WITH INDUSTRIAL SLAGS

5.1 Origin of samples

During the investigation samples were obtained from a campaign run at the Iscor pilot smelter (1.5 MW DC furnace). Samples were taken from the launder during tapping with a steel sample spoon (10cm diameter) and air cooled. Previous work (Bessinger et al., 1997) showed that this procedure was sufficient to avoid oxidation of the trivalent titanium in the slag. After being air cooled the samples were put in sample bags, labelled with the tap number and sent for analysis. Samples were chosen to vary in FeO content. The chemical compositions of the slags were obtained through a combination of X-ray fluorescence (for elemental analyses) and wet chemical analysis to determine the titanium speciation. The analyses were performed by Iscor. The results of the analyses are given in Appendix B.

As seen from appendix B, the slags fell into three groups: "high-FeO" slags, with 24-27% FeO, "medium-FeO" slags with 13-14% FeO, and "low-FeO" slags with 10-11% FeO. The different slag compositions were obtained during different periods of the pilot furnace campaign, and resulted from deliberate control actions to change the FeO content of the slag. More detailed investigation focussed on representatives of each of the three groups. These representatives were slags number 7 (high-FeO slag), 36 (medium-FeO slag) and 83 (low-FeO slag) (refer to Appendix B for their compositions.) These samples were further investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM) with energy-dispersive X-ray analysis (EDX). The samples for SEM were mounted in resin and polished. Back-scattered electron imaging was used during SEM to distinguish the different phases through atomic number contrast.

XRD-analysis was used to determine the different phases present in samples with a varying FeO content. The set-up conditions are shown in appendix A; representative XRD diagrams (for samples 7, 36 and 83 with varying FeO content) are shown in Appendices C, D and E respectively. For samples 7 and 10 with the highest FeO content (approximately 25%), two phases were present in the solidified slag namely M_3O_5 and ilmenite. The predominant phase was M_3O_5 . Samples 20 and 83 with the lowest FeO content (approximately 11%) contained M_3O_5 and rutile. Samples 99 and 36 had an FeO content of approximately 14% and showed phases similar to the low FeO content samples. These qualitative XRD results are summarised in the last two columns in Appendix B.

5.2 SEM analysis: slag microstructure

In section 3 FACTSage was used to illustrate the fact that the slag composition is close to M_3O_5 , but slightly on the rutile-rich side. The solidification path of different slags were investigated and phases forming identified. In this section this result is confirmed by SEM analysis.

Figure 25 represents sample 36 with a medium FeO content (13%). From the XRD, two phases are expected namely M_3O_5 and rutile. Rutile was not obvious in the microstructure, but this is not unexpected, given the small rutile content of this slag which is predicted from stoichiometry (Figure 20). As Figure 25 shows, the only second phases observed were a silicate-rich ("glass") phase and some metallic iron particles.

Figure 26 represents sample 7 with a high FeO content (25% FeO). Two phases are observed namely ilmenite (lighter phase in Figure 26) and M_3O_5 (darker phase). The appearance of the ilmenite is consistent with a solidification structure. This is as expected from the predicted phase changes (Figure 22b), for the case where metallic iron is not allowed to form (formation of metallic iron can conceivably be limited by nucleation).

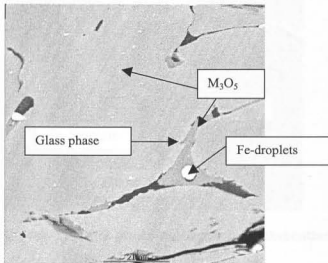


Figure 25: SEM photomicrograph (back-scattered electron imaging) of sample 36 with a medium FeO content

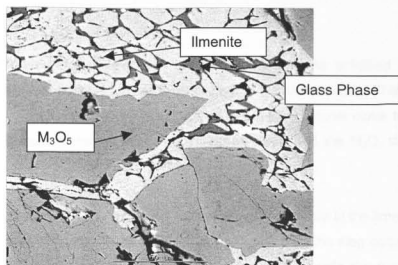


Figure 26: SEM photomicrograph (back-scattered electron imaging) of slag 7 with a high FeO content

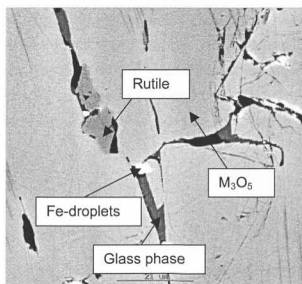


Figure 27: SEM photomicrograph (back-scattered electron imaging) of sample 83 with a low FeO content

Figure 27 represents sample 83 with a low FeO content (10%). Mainly an M_3O_5 phase was observed, with some rutile, and small metallic iron particles in the vicinity of the silicate (glass) phase. The presence of rutile in this structure is in agreement with the prediction that the slag composition should be on the rutile-rich side of pseudobrookite.