Solid State Current-Potential Sweep Sensor for the In-Situ Monitoring of Composition and Transport Properties in High Temperature Metallurgical Slags

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ABSTRACT

An *in situ* electrochemical technique for measuring the concentrations of easily dissociable oxides in slags at temperatures between 1200-1600°C is being developed. The technique consists of using a stabilized-zirconia solid electrolyte, which conducts oxygen ions, to separate a reference-gas compartment from the slag of interest. Using a potentiostat, a direct current potential sweep is applied between the inner and outer compartments of the electrolyte, driving oxygen ions from the slag into a reference gas. With the use of open circuit reference electrodes, the resulting current potential profile reveals the dissociation potential and concentration profile of dissociable oxides such as those of iron, manganese, and chromium. The potential indicates the type of cation present and its thermodynamic activity within the slag, while the current-time profile reveals the relative concentration and transport properties of such cations within the slag. The technique can therefore determine multiple properties of several different cations with only one measurement. The limits imposed by the electronic short-circuit property of the zirconia electrolyte on the sensitivity of the technique are also under investigation.

1. INTRODUCTION

Sensors for the determination of oxygen have been used extensively in the steelmaking process for better control of deoxidation, continuous casting, and ingot-making processes[1-4]. The relative amounts of oxides of transition metals such as FeO_x, CrO_x, MnO_x tend to set the oxygen activity of the slag, thereby controlling oxidation reactions which occur at the slag/metal interface. The importance of such information to the metals processing industry cannot be overestimated. Chemical analyses for FeO_x and MnO_x in slags often require more than 30 minutes, while ladle refining is completed in less than 30 minutes, which does not allow enough time for feedback process control. A sensor which could accurately determine the concentration of transition metals such as FeO_x *in situ* would allow steelmakers to control the slag chemistry by adding suitable fluxes thereby lowering the inclusion content of the steel. Potentiometric sensors based on open circuit techniques utilizing a metal / metal oxide reference are used extensively in the steel industry. These open circuit measurements can give steelmakers an accurate evaluation of the oxygen activity and even the FeO_x activity within a slag[5-6]. However, potentiometric sensors can neither give information on the actual concentration of FeO_x, nor provide information concerning the kinetics associated with the diffusion of oxygen within the slag. Also, slags with drastically different FeO_x concentrations may have identical oxygen potentials, depending upon the structure and properties of the rest of the slag. Meanwhile the diffusion of FeO_x species within the slag will be strongly dependent upon the intrinsic slag structure, basicity, and viscosity - none of which are directly measured in any way by the potentiostatic method. These variables are important because it is often the kinetics and not the thermodynamics which are important in controlling the slag/metal reactions of interest to steelmakers[7]. Models which relate the data provided by the potentiometric sensors with the properties of the slags are available, but they are inherently inaccurate and awkward to implement.

The proposed amperemetric slag sensor described in this paper provides the same information available with conventional potentiometric sensors while concurrently providing data concerning the concentration of the transition metal species and the diffusion kinetics of this species within the slag. Preliminary testing of the amperemetric slag sensor has been completed on Al_2O_3 saturated CaO - SiO_2 - Fe_2O_3 slags. The sensor was successful at differentiating between slags with identical oxygen activities but different concentrations of Fe_2O_3. The electrochemical technique utilized in the amperemetric slag sensor is similar to methods used in aqueous voltammetry. Amperemetric techniques have been used successfully at elevated temperatures for gas sensors[8-10], diffusion coefficient measurements[11], and molten metal refining[12].

2. EXPERIMENTAL

2.1 The Sensor

The electrochemical slag sensor introduced in this paper can be described by the following cell:

Pt / slag / partially stabilized zirconia (PSZ) / Pt / air

The experimental cell is shown in figure 1. A closed-end tube of oxygen-ion-conducting PSZ was used to separate the slag phase of interest from an external reference gas phase (air). The solid electrolyte used in the sensor was a PSZ (type ZDY-4 from Coors Structural Ceramics), containing 9 wt % Y_2O_3. The compartment in direct contact with the slag was continuously flushed with argon (Grade 5 BOC Gases) at a flow rate of 14 ml/min in order to provide an inert environment. The electrode leads were positioned within the cell (figure 1) as follows: a dual lead counter electrode (CE), which also functioned as an S-type Pt/Pt-Rh thermocouple was attached to a Pt gauze which was sintered to the PSZ using platinum ink (Engelhard ink 6926). A reference electrode (RE1) was attached independently 1/4" (0.625 cm) above the CE on the reference gas side of the PSZ using platinum ink. The platinum ink contacts were sintered during the heating up of the cell. The working electrode (WE) and secondary reference electrode (RE2), made from Pt wire were welded together. During the measurement the WE and RE2 are positioned in the slag such that the weld bead touches the inner bottom surface of the PSZ tube. The surface of the WE and RE2 which is exposed to the slag was controlled by shielding the electrodes using an alumina tube, which allowed 1/4 inch (0.625 cm) of each electrode to remain unshielded. The unshielded area was kept constant in all experiments.

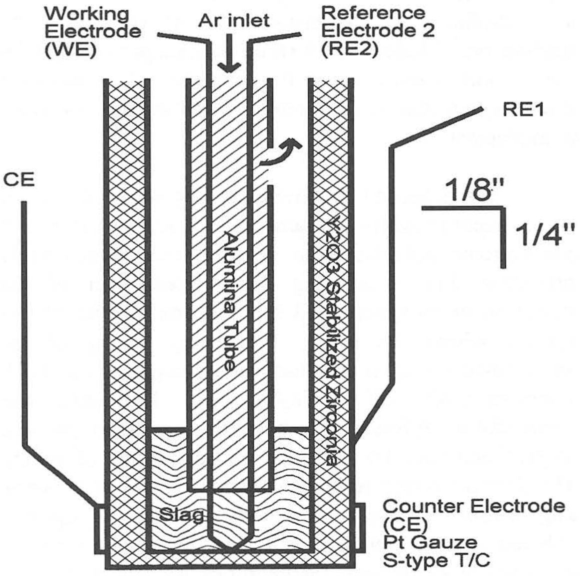

Figure 1 . The cell

The four electrode cell described above was connected to a Solartron 1287 potentiostat which controlled the potential between RE1 and RE2 by passing the required current through WE and CE. An equivalent circuit which may describe the cell is shown in figure 2.

2.2 The Slags

The CaO - SiO_2 - Fe_2O_3 slag system has been extensively studied and hence important parameters such as conductivity, viscosity, and phase diagrams are well known[13-16]. The CaO - SiO_2 - Fe_2O_3 synthetic slags used were saturated with alumina in order to stabilize the geometry of the slag sensor by preventing the dissolution of the alumina protection tube during the experiment. These melts are relatively non-corrosive with respect to the PSZ. We are considering cell designs which will eliminate the need for such precautions. The SiO_2/CaO ratio was held constant at 1.5 for all slag compositions. The Fe_2O_3 concentration was varied between 20 and 40 wt % (calculated in the system CaO - SiO_2 - Fe_2O_3) at 5 wt % intervals.

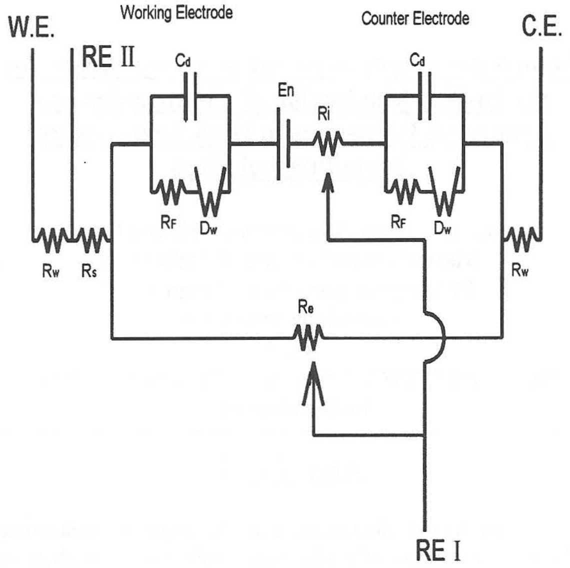

Figure 2. The equivalent circuit of the cell. R_w is wire resistance, R_s is slag resistance, R_e is electronic resistance of PSZ, R_i is ionic resistance of PSZ, C_d is a double layer capacitance, R_F is the charge transfer resistance, and D_w is a diffusional impedance, E_n is the Nerst voltage across the PSZ.

The synthetic slags were prepared by milling $CaCO_3$, SiO_2, Fe_2O_3, and Al_2O_3 powders (Alfa-Aesar) in the appropriate proportions with isopropyl alcohol for 24 hours. The powder mixtures were calcined at 1000°C for 12 hours and pressed into pellets. The pellets were placed into Al_2O_3 crucibles and melted in air at 1500°C for 1.5 hours. The resulting slags were then pulverized and re-melted in the PSZ sensor during the experiment. A similar process was used for preparing a slag in the system CaO - SiO_2 -Al_2O_3 - FeO; instead of Fe_2O_3, FeO was used, and the preliminary melting was done under argon at 1500°C for 1.5 hours.

3. ANALYTICAL PROCEDURE

Each individual experiment consisted of a sequence of measurements which were rigidly standardized as follows: the cell was heated at 4°/min up to 1430°C (1703 K), then the slag was allowed to equilibrate for 40 minutes. During equilibration, the WE and RE2 assembly was located 2.54 cm (1 inch) above the slag. The WE and RE2 were then immersed into the slag and a five minute open circuit measurement (OCV) was taken to determine the oxygen activity. A stable value of the OCV was obtained in all cases. This measurement is analogous to the measurement provided by conventional oxygen sensors.

The oxygen activity of the slag, which should be uniform in the bulk, can be determined from the open circuit potential using the Nernst equation:

$$E_n = RT/nF \ln(P_{O2}/A_{O2})$$

This can be correlated to the relative activities of FeO and Fe_2O_3 via the following equilibrium reaction:

$$2FeO + 1/2\ O_2 = Fe_2O_3$$

If the slag is in equilibrium with pure iron, the activity of FeO is defined by:

$$Fe + 1/2\ O_2 = FeO$$

Some FeO activity sensors make use of the above equation which is an improvement over a conventional oxygen sensor[4-5].

After the OCV measurement, a 5 mV/sec linear potential sweep is run starting at the equilibrium OCV value down to -1.5 V. This is immediately followed by a 15 minute open circuit recovery measurement. All potential measurements are taken with respect to the air reference electrode.

As the potential is swept toward more negative values, the ratio of $[Fe^{3+}] / [Fe^{2+}]$ at the PSZ/slag interface must change in order to maintain the oxygen equilibrium described by the Nernst equation. The removal of oxygen ions from the anions in the melt results in the formation of an oxygen concentration gradient within the slag. The anions may include network formers such as Si^{4+}, as well as amphoteric species as Al^{3+} and Fe^{3+}. This structure will be represented here by the general formula $(Si_aAl_bFe^{III}_cO_d)^{x-}$. The half cell reactions can be written as follows:

- ***cathode*** at less negative potentials:

$$(Si_aAl_bFe_cO_d)^{x-} + e- = (Si_aAl_bFe_{c-1}O_{d-1})^{x-} + 1/2O^{2-} + Fe^{2+}$$

$$(Si_aAl_bFe_cO_d)^{x-} = (Si_aAl_bFe_cO_{d-1})^{(x-2)-} + O^{2-}_{(PSZ)}$$
$$2Fe^{3+} + 2e^- = 2Fe^{2+}$$

- ***cathode*** at more negative potentials (less than -0.84 V) an additional reaction occurs:

$$(Si_aAl_bFe_cO_d)^{x-} = (Si_aAl_bFe_cO_{d-1})^{(x-2)-} + O^{2-}_{(PSZ)}$$
$$Fe^{2+} + 2e^- = Fe$$

- ***anode***:

$$O^{2-}_{(PSZ)} = 1/2\ O_2 + 2e^-$$

Assuming that limited migrational effects are present, a peak in the resulting current would be expected as the dissociation potentials of easily dissociable oxides such as Fe_2O_3 or FeO are reached during the sweep. A thorough mathematical treatment describing the formation of such peaks which involves linear sweep voltammetry under aqueous chemistry conditions is covered extensively by Bard and Faulkner[17].

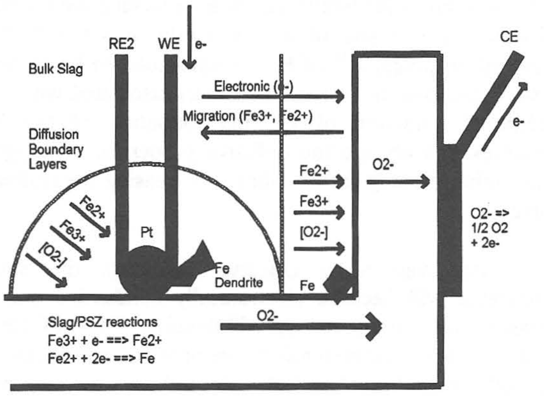

Figure 3. Model of the reactions involving Fe - containing species within the proposed amperemetric sensor.

Figure 3 demonstrates the reactions occurring within the system. Most of the interfacial reactions will tend to occur near the three-phase PSZ/Pt/slag interface, however, some electronic conduction may also occur within the slag via either an electronic (hopping) or a migrational mechanism[14]. This conduction would allow the slag itself to carry a portion of the current, thereby allowing reactions to occur at sites along the PSZ/slag two phase interface away from the three phase interface. A "plateau" may occur in the resulting i-V curves if a boundary layer is formed near the three phase interface. The magnitude of the current passed during such a plateau region would be related to the concentration of Fe^{3+} within the slag and may be approximated by the following equation:

$$I = nFAJ = nFA(C_{bulk}-C_{interface})D_{Fe2O3}/x$$

where J = the flux of Fe_2O_3 - containing species across the boundary layer;
C = the concentration of species Fe_2O_3 in the bulk or at the interface;
D = the steady state diffusion coefficient of Fe_2O_3 across the boundary layer;
x = the distance across the boundary layer;
A = the surface area for the interfacial reaction;
n = the charge transfer number (1 if reaction is $Fe^{3+} + e^- \Rightarrow Fe^{2+}$);

As the dissociation potential associated with Fe_2O_3 is passed, the $C_{interface}$ term for Fe^{3+} becomes essentially zero and J assumes a constant value thus resulting in the aforementioned "plateau".

As more negative potentials are applied, the dissociation potential of FeO will be reached and increased currents associated with the additional reduction of Fe^{2+} to Fe are encountered. The additional flux associated with the reduction of Fe^{2+} may be approximated by:

$$I = nFAJ = nFA(C_{bulk} - C_{interface})D_{"FeO"}/x$$

in a similar way as the flux associated with the ferric reduction. From the Nernst equation, assuming the activity of iron, $a_{Fe} = 1$ and of ferrous oxide, $a_{FeO} = 0.5$, the dissociation potential[18] of FeO is calculated to be around 0.84 V. Besides the increased currents associated with the additional reduction of Fe, the formation of the Fe dendrites will change the diffusive properties of oxygen ions within the slag thereby further increasing the realized current.

At even more negative potentials the solid electrolyte will become electronically conductive and a parasitic short circuit current will develop. This will set a limit upon the maximum achievable potential which can be applied across the solid electrolyte without drowning out the ionic signal. This limit is characterized by the P_θ value of the solid electrolyte, defined as the partial pressure of oxygen at which the ionic transference number equals the electronic transference number[19-23]. Under our experimental conditions the electronic conductivity is expected to exert only a minor influence.

The open circuit recovery measurement indicates the rapidity of equilibration of the PSZ interface with the bulk of the slag. The potential-time relationship also indicates the oxidation state and type of species formed during the i-V sweep of the slag. The recovery curves have three important regions: at extremely negative potentials, below the range investigated in this paper, some O^{2-} ions will be re-introduced through the PSZ because of the high electronic conductivity of PSZ at negative potentials - this will be a rapid process which is important only for very short time periods. At less negative potentials the reoxidation of electrolytically formed iron is indicated by a recovery plateau determined by the equilibrium potential between FeO and Fe. As Fe^{3+} diffuses to the electrolytically formed Fe, the following reaction occurs:

$$Fe + 2\,Fe^{3+} = 3\,Fe^{2+}$$

When all the Fe is reoxidized, the recovery potential then quickly approaches that determined by the ratio of Fe^{3+} / Fe^{2+}. Finally, at longer times (found experimentally to be on the order of 15 minutes), the PSZ interface equilibrates with the bulk slag phase. The new equilibrium OCV has been altered from the original OCV because of oxygen removal from the system.

In a separate experiment, potentiostatic measurements (constant applied voltage) lasting for fifty seconds were conducted at applied voltages varying between - 0.6 and - 1.5 V in order to correlate the shape of the i-V curves with the iron reduction processes described above.

In another experiment, the slag was strongly reduced, such that the final OCV remained near potentials associated with the stability of Fe. The cell was then quenched, cut, and analyzed using under a SEM with an EDS attachment.

4. RESULTS AND DISCUSSION

The experimental i-V curves (figure 4) show the expected dependence of current on the initial concentration of Fe_2O_3 within the slag as described in section 3. For all concentrations of Fe_2O_3 tested with the proposed amperemetric sensor, the initial OCV values were identical; hence, a typical OCV oxygen sensor would not be able to directly determine a difference between these quite different slags. The plateau on the i - V curves represents the reaction Fe^{3+} to Fe^{2+} while the edge represents mainly the reduction of Fe^{2+} to Fe. As stated earlier, the i-V plots shown in figure 4 include an uncompensated solution resistance term in the measurement which tends to shift the i-V curves towards more negative potentials, thereby distorting the i-V curve. This explains why the relative position of the edge appears to be located at more negative potentials than would be expected for FeO dissociation.

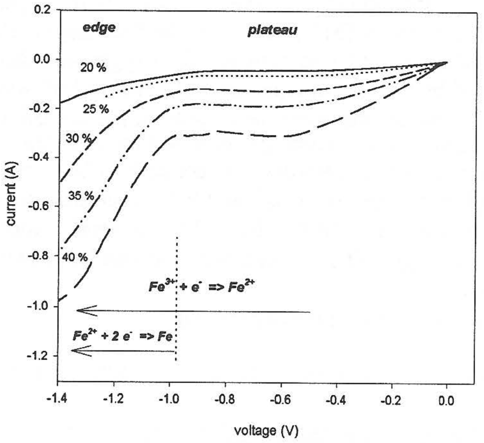

Figure 4. i - V plots at 5 mV/s for Fe_2O_3 containing slags. The amount of Fe_2O_3 varied between 20 - 40 wt %.

The OCV recovery curves (figure 5) for the i-V sweeps in figure 4 reveal more information about the system. The OCV remains approximately the same for several minutes at a potential close to 0.8 V. During this time the Fe formed during the i-V sweep is being reoxidized by the slowly equilibrating slag. When the electrolytically formed iron has been reoxidized, the Fe^{3+}/Fe^{2+} equilibrium will determine the potential, causing a quick relaxation to less negative potentials. Finally, after approximately 15 minutes, the slag acquires a stable potential which describes the new oxygen activity of the entire slag. The slag has changed its oxygen content because oxygen has been removed from the slag through the PSZ. There is a difference in the level of the intermediate plateau on the OCV plots for the recovery of slags of different concentrations of Fe_2O_3. This difference is indicative of a greater activity of FeO near the slag/PSZ interface in the slags with higher Fe_2O_3 initial concentration.

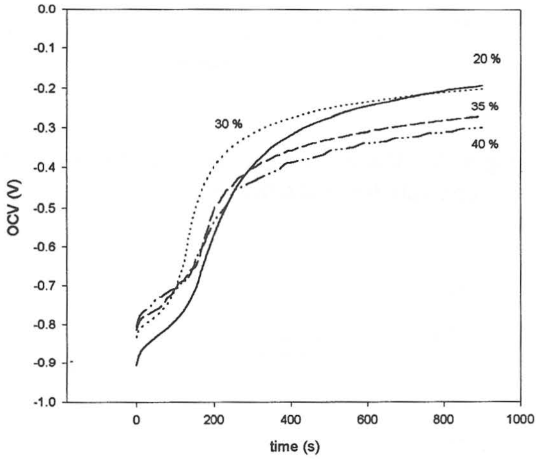

Figure 5. OCV recovery curves for the i-V potential sweeps.

The potentiostatic curves (figure 6) which were run at different potentials along the i-V plot provide further proof for the reactions occurring during the i-V sweep (figure 4). The potentiostatic curves yielded approximately constant currents after long times (50 sec) which corresponded well to the plateau and edge levels generated during the sweep. These constant currents help to support hypothesis on the formation of a boundary layer near the PSZ/slag interface. The potentiostatic curves which are at potentials corresponding to the edge (< -1V) of the i-V sweep demonstrate a flat region corresponding to Fe in their OCV recovery curves (figure 7) and also demonstrate significantly larger currents (figure 6). These details further reinforce the theory that the FeO dissociation is occurring at the edge and Fe_2O_3 dissociation is occurring along the plateau of the i-V curves. The iR compensation technique which is currently under investigation, which will more accurately describe the potential of interest. This technique presently runs into difficulties as the electronic conduction limits of the PSZ are approached. However, preliminary results using iR compensation accurately reposition the location of the FeO dissociation peak to a value closer to the - 0.84 V that is expected from a thermodynamic (Nernstian) standpoint.

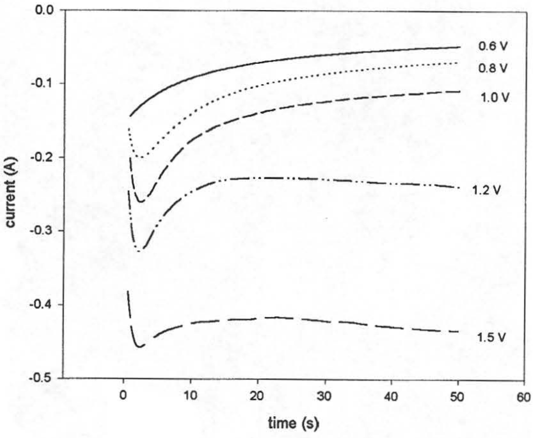

Figure 6. Current-time profiles for potentiostatic measurements.

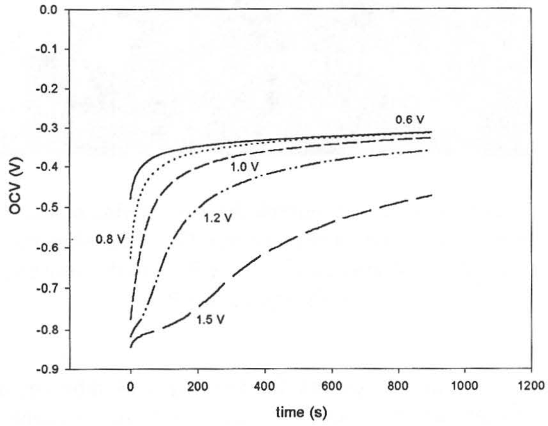

Figure 7. OCV recovery profiles for the potentiostatic measurements in figure 6.

The quenched and sectioned cell analyzed under the SEM helps to confirm the model proposed in the analytical section above. Fe dendrites (figure 8) appear to form near the PSZ/slag/Pt three phase interface. Fe does diffuse into the Pt wire along its entire length, however dendrite formation mainly occurs close to the three phase interface. The interface of a metal phase (e.g., Pt or dendrite) may provide a fast diffusion path for O^{2-} containing species within a less conductive slag phase. Analyzing an analogous system, Pal et al have shown[24] that electronic conductors in an oxide melt may enhance oxidation rates. The Fe dendrites can grow outward from the surface of the PSZ because they enter Fe-rich slag and simultaneously provide a path for the O^{2-} to migrate towards the PSZ. This is potentially an autocatalytic process as the transport properties may be enhanced by the formation of the iron dendrites.

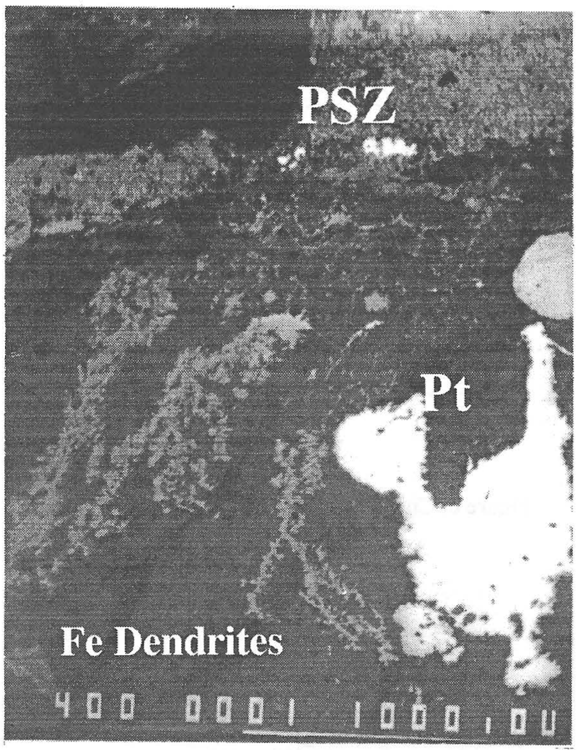

Figure 8. SEM micrograph (back-scattering mode) of an area close to the three phase interface. The white material is Pt, the light gray dendrites are Fe, and the gray material to the right is the PSZ.

The i-V curves for two slags of different initial oxygen activities but the same overall iron concentration are shown in figure 9. The potential difference is determined by the Fe^{3+} / Fe^{2+} ratio in the slag. The reduced oxygen activity in the slag with the higher FeO content can be detected by the initial open circuit value. As expected, the plateau in the i - V curve is lower in comparison with the other slag, because of the lower initial concentration of Fe_2O_3. The OCV recovery curves for these two slags are shown in figure 10. The slag with higher FeO concentration recovers more slowly. This is most likely caused by a less steep concentration gradient across the boundary layer.

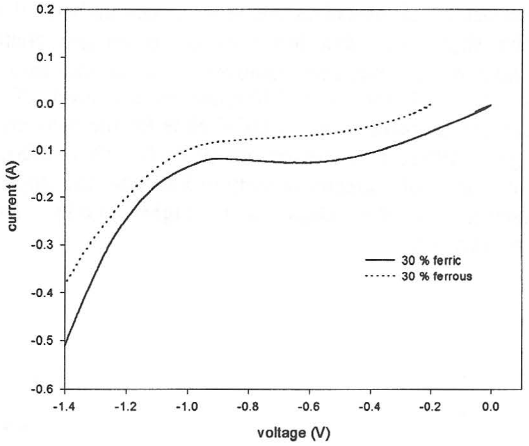

Figure 9. i-V curves for two slags with the same iron concentration and different oxygen activities.

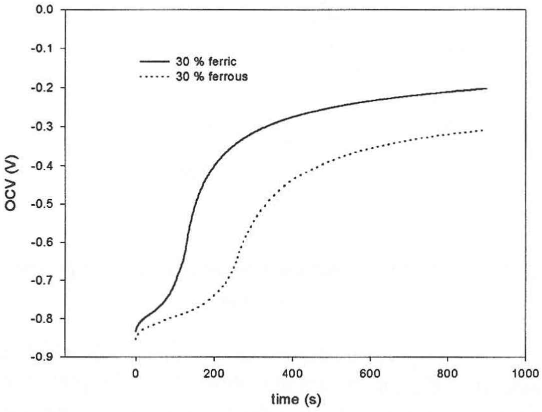

Figure 10. OCV recovery curves for the i - V plots shown in figure 9.

4. Conclusions

The amperemetric slag sensor described in this paper has been shown to be sensitive to the FeO_x concentration of high temperature slags.

Slags with different oxygen activities are differentiated just as with a conventional oxygen sensor.

The amperemetric sensor can also differentiate between slags with identical oxygen activities but different concentrations of FeO_x. This information goes beyond the capability of the conventional (potentiometric) oxygen sensors.

The time scale required for gathering this information for a given slag is expected to be 5 minutes or less.

Additionally, the amperemetric principle of this sensor provides data with kinetic relevance regarding the mobility of FeO_x species within the slag, which is often a rate-controlling step for important reactions occurring between the slag and the metal.

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