REACTIONS BETWEEN SOLID METALS AND MOLTEN SLAGS

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ABSTRACT

Reactions between solid metals-iron, nickel and platinum, and molten $MnO\text{-}SiO_2$ (MS) and $CaO\text{-}Al_2O_3\text{-}SiO_2$ (CAS) slags were studied by the sessile drop method in the temperature interval 1350-1450°C in reducing atmosphere. The terminal contact angles (after 240 min) for $MnO\text{-}SiO_2$ slag were: for iron substrates – 5±2° at 1350°C, 9±2° at 1390°C, 6±2° at 1450°C; and for platinum – 15±2° at 1350 and 1390°C, and 12±2° at 1450°C. The contact angle for the Ni- MS system was close to zero – 3±2° at 1350 and 1390°C. Contact angles with CAS slag were: for iron – 55±2° (1350°C), 60±2° (1390°C), 44±2° (1450°C); for nickel – 59±2° (1350°C), 60±2° (1390°C); and for platinum – 15±2° (1350, 1390 and 1450°C). The work of adhesion for all three substrates with the MS slag changed in a very narrow range from 910 to 930 mJ/m^2. Interfacial tension with this slag was 1,480 mN/m for Ni at 1350 to 1390°C, and 1,880 to 1,890 mN/m for Pt in the temperature range 1,350 to 1,450°C. For iron, interfacial tension was 1,720 mN/m at 1350°C; it decreased to 1,590-1,580 mN/m with increasing temperature to 1390 and 1450°C. Lower work of adhesion and higher interfacial tension was found for metals with CAS slag. Dissolution of manganese in nickel and platinum substrates at elevated temperatures modified the interface chemistry, causing formation of a liquid alloy phase. The degree of silica reduction from MS slag was much smaller in comparison with MnO reduction (negligible for Pt); it was very minor from CAS slag. The reduction of oxides and adsorption of oxygen modify the metal-oxide interface, making wetting dynamic. They have a profound effect on interfacial properties.

INTRODUCTION

In the temperature range of steel solidification (1450-1200°C) many oxide/oxy-sulfide segregates remain liquid and spread on inter-dendrite surfaces, depending on the local interfacial conditions. Metal-oxide interfacial properties affect inclusion morphology and shape.

Wetting of solid metals by molten oxides is also of interest in strip casting [14]. Reported that heat transfer between the molten steel and mould was affected by oxides in the FeO-MnO-SiO_2-MnS system present at the interface. When these oxides were molten, heat transfer between the steel and mould encountered very little resistance. Good wetting of the Cr-coated mould and steel interfaces by the low-melting oxide (similar to the MnO-SiO_2 eutectic) enhances heat transfer, as the molten oxide will form a film across the interface. Recently, Nolli and Cramb [9] have also shown that manganese silicate mould coatings can improve heat transfer rates and productivity in strip casting.

Molten oxide-solid metal systems are expected to present contrasting *wetting* behavior to the *non-wetting* character of molten metal-solid oxide systems. It can be demonstrated by considering balance of surface energies given by Young's equation. [1]

$$\gamma_{LV} \cos\theta = \gamma_{SV} - \gamma_{SL} \tag{1}$$

where γ_{LV} is surface tension of a liquid, γ_{SV} is surface tension of a solid, γ_{SL} is the interfacial tension of the actual liquid-solid contact. Given the relatively high surface tensions of metals compared to oxides, the solid metal–liquid oxide configuration is expected to produce good wetting (low contact angle) while liquid metal–solid oxide systems favor a non-wetting state (high contact angle).

In metallurgical literature, the majority of work is devoted to molten metal-solid oxide or molten metal-molten slag interfaces, while data on the contact angles for molten slag-solid metal system are limited. This work studied wettability and reaction of solid iron, nickel and platinum substrates with liquid MnO-SiO_2 and CaO-Al_2O_3-SiO_2 systems, with the aim to contribute to further understanding of solid metal–molten oxide interaction.

METHODOLOGY

The study is based upon in-situ observation of high-temperature wettability of a solid metal by low-melting non-metallic oxides (slag) by the sessile drop method. In these experiments, the change in behavior with time and temperature of a liquid drop of slag upon a solid metal substrate was recorded.

The experiments were conducted in a graphite furnace as schematically shown elsewhere [13]. The furnace was powered by a welding power supply with a peak current of 400 A. This high current supply permitted heating rates up to 1000°C/min.

A Sony DCR-TRV18E digital video camera with a resolution of 720 x 576 pixels was used to record image sequences during the wettability experiments. A pair of Vivitar zoom lenses (one 4 x, one 1 x) was screwed onto the camera lens to provide the correct focal length.

The MnO-SiO_2 (MS) slag was prepared from the MnO and silica powder. The CaO-SiO_2-Al_2O_3 slag was prepared from silica (99.995 wt%), alumina (99.8 wt%) and CaO, which was produced by calcining $CaCO_3$ powder (99+ wt%) in a muffle furnace. The MnO-SiO_2 slag contained 58.5 wt % MnO and 38.5 wt % SiO_2, which is close to the eutectic composition of 61.7 wt % MnO and 38.3 wt % SiO_2. CaO-Al_2O_3-SiO_2 slag, containing 63.1 wt% SiO_2, 22.1 wt% CaO and 14.2 wt% Al_2O_3, was also near-eutectic composition.

The chemical analysis of iron, nickel and platinum is given in other work [13]. The weight of a substrate was ~1 g and of slag ~0.02 g. Wetting experiments were conducted in atmospheres of Varigon (95% Argon, 5% Hydrogen) and argon of purity 99.999%.

The inlet gases were passed through copper turnings at 550 °C to remove oxygen. A stabilized zirconia oxygen probe sensor measured oxygen partial pressure at the outlet of the furnace.

A sample was placed on the sample stage within the furnace, which slides in and out of the hot zone. The base for the stage was aligned using a spirit level on the stage. Before experiments, the furnace was evacuated by a mechanical vacuum pump for 15 min, and then the furnace was backfilled with Varigon or argon, which passed through the furnace at a low flow rate.

Each experiment began with heating to 1000°C at a rapid heating rate (200-300°C/min), where it was held for 30 minutes. Following this pre-heating, the temperature controller was then set to attain experimental temperature at 100°C/min.

The video camera captured footage at a resolution of 720 x 576 pixels. Still images were selected and stored individually using the VirtualDub program (version 1.4.10, developed by Avery Lee, sourced from www.virtualdub.org).

Measurement of surface tensions from low angle curves observed in this work was meaningless; however, the contact angle was measured accurately. A graphical analysis program was used to convert the images to pixel co-ordinates. The drop profile was marked by the user in a series of x and y pixel co-ordinates. The droplet profile was usually described by manual selection of 13 – 25 points along the edge. Each point was accurate to within ±1 pixel (±0.02 mm given the zoom lenses used on the camera). These selected co-ordinates were then subject to simple linear regression to obtain a theoretical curve matching the experimental points. The polynomial equation describing this line was used to find the value for the derivative at the limits of the curve. Taking the arc tan of these evaluations gave the contact angle for the drop. Marking the drop profile showed an error in contact angle calculation of ±2°.

After experiments, samples were analyzed visually and chemically by microscopic methods. Initially, the surface appearance of the samples was recorded by the digital video camera and optical microscope.

In preparation for analysis by electron microscopy, the resin-mounted samples were carbon coated in a vacuum chamber. Samples were then used for scanning electron microscope (SEM) analysis with an electron-dispersive spectroscopy (EDS) attachment.

The Cameca SX50 electron microprobe analyzer (EPMA) was used for quantitative composition analysis of the sample cross-sections. Profiles across the centre of the slag-metal interface and parallel to the interface outwards from the drop edge (as near as practical to the surface) were made with a step size of between 2 and 10 μm across and 100 μm parallel to the interface.

RESULTS AND DISCUSSION

Isothermal wetting of iron and platinum substrates by MS and CAS slags was studied at temperatures of 1350, 1390 and 1450°C; reactions with nickel substrate were examined at 1350 and 1390°C ; holding time was 30 and 240 min for Fe and Ni substrates and 30 min for Pt substrate. The change in contact angle with time as measured for Fe, Ni and Pt substrates at different temperatures is shown in Figures 1-3.

The contact angles for iron and nickel substrates with MS slag were much smaller than in reactions with CAS slag. Very good wetting and dynamic behavior of the contact angle for the MS slag were attributed to the interface modification as a result of MnO reduction from the slag, dissolution of manganese into the substrate and oxygen adsorption on the metal surface.

Figure 4 shows the manganese concentration profile in reaction of the metallic substrates with MnO-SiO_2 slag. The concentration and depth of manganese dissolution in the nickel are far more profound than in iron and platinum. Platinum shows slightly higher levels of manganese near the interface than does iron, but the transfer occurs to a similar depth in both; the difference in manganese contents in iron and platinum is within the experimental error.

Dissolution of manganese in nickel decreased the melting temperature of the substrate, causing its partial melting and radical change of the interface.

Reduction of silica from the MnO-SiO_2 slag and dissolution of silicon into the metal substrate was much smaller than that of MnO. Relatively low levels of silicon dissolved in metals were also observed in reactions with the CAS slag. This is seen in Figure 5 which compares silicon concentration profiles in nickel obtained in reactions with CAS and MS slags in 30-min experiments at 1390°C. In the platinum and iron substrates, no difference in silicon dissolution from MS and CAS slags was seen.

The effect of oxygen, modifying chemistry of the interface and interfacial energies, is well-recognized in wetting and other surface phenomena. A reactive or non-reactive regime of wetting depends on the oxygen partial pressure in the furnace, oxide stability, oxygen adsorption and solubility of oxygen and oxide-forming elements in the substrate.

The solubility of oxygen in solid nickel, iron and platinum is very low. Solubility of oxygen in solid nickel was found in the range of 0.01-0.05 at% [18], and rises to 1.5 at% upon melting [16]. Iron has similarly low levels of solubility in the solid state: -0.0008 at% in γ-Fe; 0.002 at% in δ-Fe, [Kitchener *et al.,* 1953; 15] while liquid iron at temperature close to melting point dissolves 0.06 at% [16]. According to [18], solubility of oxygen in platinum is negligible. [16]

The intake of oxygen in the wetting experiments depends on the bulk diffusion of oxygen in the metal substrates and the permeability (product of diffusion coefficient and solubility). At temperatures near those in the experiments, iron and nickel have similar levels of oxygen diffusion coefficient and permeability. The platinum substrate is expected to have almost no permeability for oxygen, with the diffusion coefficient being several orders of magnitude lower at the same temperature.

However, adsorption capacity of solid platinum, nickel and iron for oxygen is known to be high. The surface activity of adsorbed oxygen on the metal substrates is highly important in determining surface tensions. Hondros [6] estimated the surface excess of oxygen on iron at 1410°C to be $\Gamma_O = 4x10^{14}$ atoms/cm^2. The effect of oxygen on iron surface tension was estimated at -10^7 ergs/cm^2/at% O. Comparable data for the other substrates were not found.

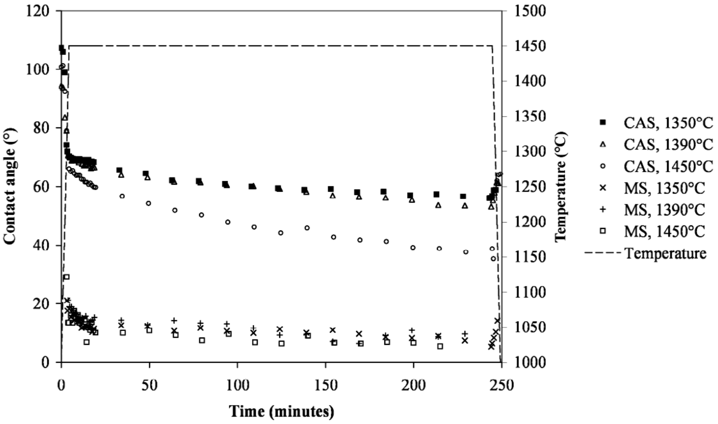

Figure 1: Contact angle for Fe substrate, 240 min exposure at various temperatures

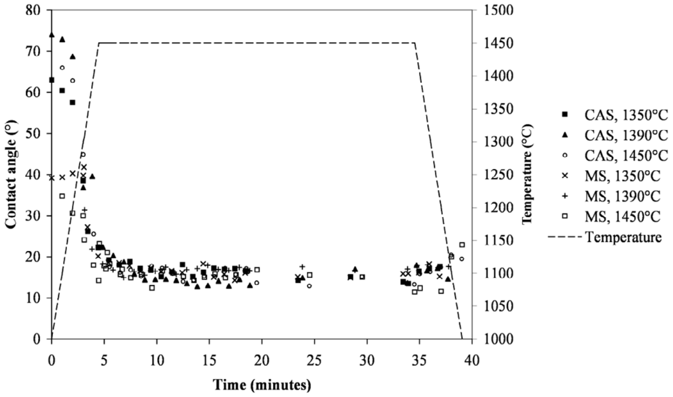

Figure 2: Contact angle for Ni substrate, 240 min exposure at various temperatures

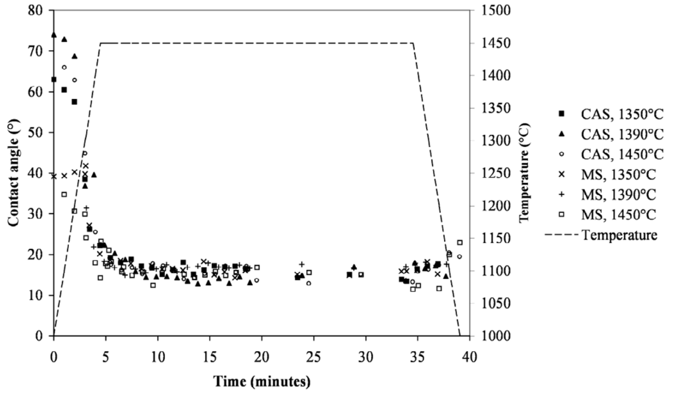

Figure 3: Contact angle for Pt substrate, 30 min exposure at various temperatures

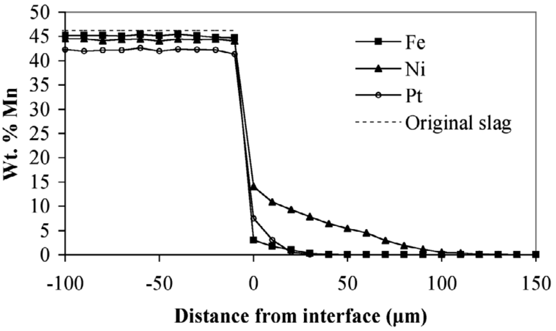

Figure 4: Mn concentration profile across substrate - MS slag interface after 30 min reaction at 1350°C

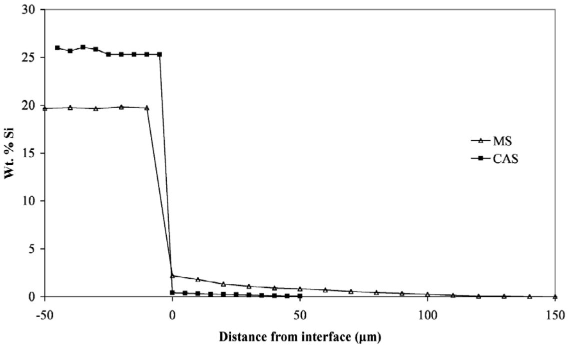

Figure 5: Si concentration profile across nickel-slag interface after 30 min reaction with MS and CAS slags at 1390°C

With the use of the 5 %H_2-Ar gas in the graphite furnace, the partial pressure of oxygen was measured below 10^{-20} atm, although during the preheating at 1000°C it was slightly higher at 10^{-19} atm. Therefore, experimental conditions are reducing for MnO and SiO_2 in the MS slag and for SiO_2 in the CAS slag. Oxygen released in the reduction reactions may form oxides with metals of substrates, be dissolved in the metal phase and adsorbed on the interface or transfer to the gas phase. Oxygen solubility in solid iron, nickel and particularly platinum is very low; it is adsorbed on the metal surface with high surface coverage but in small quantity. Therefore, oxygen can report to the gas phase.

Low activity coefficients of manganese and silicon stimulate reduction of MnO and SiO_2 oxides, with transfer of Mn and Si into the metal substrate. In accordance with the activity coefficients, degree of reduction of manganese oxide is expected to increase from Fe to Pt and further to Ni. The highest degree of silicon reduction is also expected in the reaction with nickel, followed by iron and platinum.

Visual confirmation of reaction taking place was seen in the changing color of slag phases for most systems. The original color of MS slag was black, but appeared brown-orange in most samples of iron and nickel substrates, and purple-orange in platinum samples. The original CAS slag was colorless and transparent, but converted to a black, opaque phase after reaction with iron or nickel. No color change was evident upon reacting CAS with platinum substrates. Trace levels of reaction product phases (metallic iron, nickel or platinum) are known to alter color of oxides, even on the ppm level [5].

The occurrence of chemical reaction, such as reduction of MnO and SiO_2, at the interface is known to provide an additional driving force for wetting, above the balance of surface tensions [3]. The reduction of oxides, particularly MnO, in wetting experiments causes change in slag and substrate chemistry, and makes wetting dynamic.

Immediately after melting, the slag spread over a substrate and took a shape that changed only gradually. The change of contact angle in the initial period of a few seconds duration was of a non-reactive character [3]. Subsequently, the reactive wetting between the slag and substrate, with dissociation of MnO or SiO_2 on the interface, adsorption of oxygen on the metal surface and dissolution of Mn and Si into the metal took place. The reaction modifies the interface and improves wetting, which was reflected in the lower contact angle. The chemical reaction at the interface progresses towards equilibrium, however the transport of reaction products away from the interface (desorption of oxygen and diffusion of Mn (Si) into metal ensures continued reduction. The reduction reaction and mass transfer of Mn (Si) from the interface continuously decreased MnO (SiO_2) content in the slag, altering equilibrium conditions and reaction kinetics. The equilibrium was not achieved under experimental conditions described in the paper.

The rate of reaction is elevated near the triple point, where the slag encounters *fresh* substrate material to react with. This creates a localized depression of interfacial tension, which the system responds to by increasing interfacial area locally. Observations of this for each substrate were made as *necking* of soft, alloyed metal at the interface occurred, transferring metal across the interface.

The platinum substrate behaved differently than Fe and Ni substrates, showing similar reaction with both CAS and MnO-SiO_2 slags [2], suggested formation of the unstable platinum oxide by adopting non-bridging oxygen ions from the slag phase. Oxygen adsorption at the interface of the platinum substrate, and formation of a lower viscosity surface layer on the slag favors spreading of slags on Pt under reducing conditions.

Under experimental conditions in this work, metal-slag equilibrium was not achieved; however, the rate of the slag-metal reaction in isothermal experiments was low. Considering that a response of interfacial forces to the changing chemistry of the interface was much faster than the change itself, we can operate with a quasi-equilibrium state and estimate interfacial tension and work of adhesion, defined as $W_{ad} = \gamma_{Me} + \gamma_{slag} - \gamma_{IT} = \gamma_{slag} (1 + \cos \theta)$. The surface tensions of metals and oxides used in the calculations are given in Table 1.

Calculated interfacial tension and work of adhesion, using angle measurements after 240 min exposure for Fe and Ni and 30 min for Pt substrate, are summarized in Tables 2 and 3. Work of adhesion for the MS slag changes in a very narrow range, 910 - 930 mJ/m^2. This is a reflection of good wettability of all three metal substrates by the MS slag; the contact angle between the MS slag and different substrates is in a range of 2 - 15°. The trend in interfacial tensions of the MS slag with different metals ($\gamma_{IT} = \gamma_{Me} - \gamma_{slag} \cos \theta$) follows the trend in the surface tensions of these metals.

For iron and nickel substrates with CAS slag, the work of adhesion is lower and interfacial tension is higher than for these metals with MS slag, reflecting the difference in wetting. Contact angle between platinum and CAS slag is 15°, about the same as between platinum and MS slag. The difference in the work of adhesion and interfacial tension between the platinum - CAS and platinum - MS systems is due to the 40 mN/m difference in the surface tensions of CAS and MS slag.

Table 1: Surface tension of metals and oxides

Phase	Surface tension (mN/m)	Temperature (°C)	Reference
γ-Fe	2170	1360 – 1400	Jones (1971)
δ-Fe	2040	1440	Jones (1971)
Ni	1940	1357 - 1437	Jones (1971)
Pt	2340	1310	Jones (1971)

$MnO-SiO_2$	465	1350 – 1450	Mills (1995)
$CaO-Al_2O_3-SiO_2$	425	1350 – 1450	Mills (1995)

High work of adhesion is expected in reactive systems with strong chemical bonds. The work of adhesion for the platinum – $MnO-SiO_2$ system, reported by Towers (1954), is in the range 800 – 1000 mJ/m^2, matching the present results. Interfacial tension for the platinum – $MnO-SiO_2$ system measured by [17], was in the range 1300 – 1400 mN/m, based upon assumed surface tension for Pt of 1800 mN/m. Having recalculated for γ_{Pt} = 2340 mN/m, interfacial tension would be in the range 1840 - 1940 mN/m, being very close to data obtained in this work. For other solid metal-liquid oxide systems, the work of adhesion and interfacial tension were not found in literature. However, it was reported for the solid oxide – liquid metal systems. The work of adhesion for these systems is strongly dependent on the oxygen dissolved into the metal. [11] reported the increase in W_{ad} from 500 to 1000 mJ/m^2 between a Fe-Co alloy and Al_2O_3 upon dissolution of 0.12 wt% [O]. [12], found interfacial tension of liquid iron on solid MnO to be 1450 mN/m. Literature values for interfacial tension of liquid iron on solid oxides (Al_2O_3, ZrO_2, MgO, BeO, TiO_2 and ThO_2) indicate a range of 1500 – 2300 mN/m, [4]. The main determinant was oxygen adsorption; [10] reported a decrease of the interfacial tension to about zero, with visible emulsification when dissolved oxygen in iron reached 600 ppm.

The interfacial tension of liquid nickel on a variety of solid oxides fell in a range of 1500 – 2200 mN/m. Experimental data for liquid iron and nickel – solid oxide systems [4, 12] are reasonably close to the data for solid iron and nickel – liquid oxide systems obtained in this work. Reports of work of adhesion for liquid iron on solid Al_2O_3 substrates are in the range of 500 – 685 mJ/m^2 [4]. This encompasses the values seen for iron and nickel substrates at most temperatures, indicating that a similar balance of interfacial energies takes place.

The interfacial tension and work of adhesion for metal – oxide systems, either solid metal-liquid oxide or liquid metal-solid oxide, are governed by similar factors, the major of these being metal-oxide reaction and modification of the interface. Chemical bonds at the interface are also of a similar nature. Oxygen is a surface-active element in both liquid and solid metals, and although solubility of oxygen in liquid metal is orders of magnitude higher than in solid, the oxygen concentration on a metal surface is close to saturation in solid as in liquid metal. Strong chemical bonds between this oxygen and metals of substrate and oxide provide high work of adhesion and interfacial tension. The increased oxygen adsorption with MnO and SiO_2 reduction will lower interfacial tension, and enhance wetting behavior.

Table 2: Interfacial tensions for MS and CAS slags (mN/m)

Substrate	**MS slag Temperature**			**CAS slag Temperature**		
	1350°C	1390°C	1450°C	1350°C	1390°C	1450°C
Fe	1720 (γ-Fe)	1590 (δ-Fe)	1580 (δ-Fe)	1980 (γ-*Fe*)	1780 (δ-*Fe*)	1700 (δ-*Fe*)
Ni	1480	1480	-	1770	1750	-
Pt	1890	1890	1880	1930	1930	1930

Table 3: Work of adhesion for MS and CAS slags (mJ/m^2)

Substrate Temperature	**MS slag**	**CAS slag**				**Temperature**
	1350°C	1390°C	1450°C	1350°C	1390°C	1450°C

Fe	930	930	930	670	640	730
Ni	930	930	-	640	640	-
Pt	910	910	920	840	840	840

CONCLUSIONS

In contrast to poor wetting of oxides by molten transition metals, solid metals – iron, nickel and platinum, are wetted by molten oxides very well. The measured terminal contact angles (after 240 min) for the MnO-SiO_2 slag under reducing conditions were: for iron substrates – 5±2° (1350°C), 9±2° (1390°C), 6±2 (1450°C), and platinum – 15±2° (1350 and 1390°C), and 12±2° (1450°C). Contact angle for the Ni- (MnO-SiO_2) system was close to zero – 3±2° at 1350 and 1390°C. *Terminal* contact angles after 240 minutes of reacting CaO-SiO_2-Al_2O_3 slag with metal substrates were: for iron – 55±2° (1350°C), 60±2° (1390°C), 44±2° (1450°C); for nickel – 59±2° (1350°C), 60±2° (1390°C); and for platinum – 15±2° (1350, 1390 and 1450°C).

The substrates reacted with slags, with reduction of oxides and interface modification. Reaction at the interface aided wetting in metal – slag systems, above the balance of surface tensions, leading to dynamic wetting behavior. Significant MnO reduction and manganese dissolution into substrate was observed in reactions with all three substrates. Silica also was reduced in reaction with Fe and Ni, although the extent of reduction was much smaller in comparison with MnO, and was not observed in reaction with Pt. Concentration of dissolved manganese and silicon in iron and nickel substrates increased with increasing reaction time. No silicon was detected in the platinum substrate.

The oxygen solubility in the substrate metals was below the detectable level, with a greater tendency to adsorb at the metal-slag interface, since the reducing conditions made oxide formation on the substrates unfeasible. Adsorbed oxygen is known to be strongly surface active, lowering the surface tension of the metal substrates and favoring wetting conditions. Visual observation of oxygen desorbing as gas bubbles from the interface was made with platinum substrates.

The interfacial tension of the substrates with the MS slag was relatively high: for iron – 1580-1720 mN/m; for nickel – 1480 mN/m; and for platinum – 1880-1890 mN/m. The values with the CAS slag broadly reflected the less wetting configuration: for iron – 1700-1980 mN/m; for nickel – 1750-1770 mN/m; and for platinum - 1930 mN/m. These values compared reasonably with literature values determined for comparable molten oxide-solid metal systems, and also similar molten metal-solid oxide systems.

The work of adhesion was determined for all the substrates with MS slag as being in the range of 910-930 mJ/m^2. Values varied more widely between substrates with the CAS slag: for iron – 640-730 mJ/m^2; for nickel – 640 mJ/m^2; and for platinum – 840 mJ/m^2. Greater work of adhesion reflected better bonding at the interface when strong wetting (low contact angle) took place.

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