

## Wettability of some refractory materials by molten $\text{SiO}_2\text{--MnO--TiO}_2\text{--FeO}_x$ slag

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### ABSTRACT

The wettability of some refractory materials, including Mo, Pt, W, polycrystalline  $\text{Al}_2\text{O}_3$ , MgO, and graphite, by a molten 38.7 wt.%  $\text{SiO}_2\text{--}44.3$  wt.% MnO–11.4 wt.%  $\text{TiO}_2\text{--}5.6$  wt.%  $\text{FeO}_x$  ( $x = 1$  or 1.5) slag was investigated at temperatures between 1473 and 1673 K in an Ar atmosphere. The slag wets these materials quite well and the work of adhesion is in the range of 0.60–0.75  $\text{J m}^{-2}$ , except for graphite. A strong reaction between the slag and  $\text{Al}_2\text{O}_3$  as well as MgO leads to small apparent contact angles ( $\sim 10 \pm 2^\circ$ ) and good adherence. The wettability of the refractory metals depends not only on temperature but also on metal species. At 1473 K, the wettability is in the order of  $\text{Pt} > \text{W} > \text{Mo}$ . An increase in temperature considerably promotes the wettability of Mo and W while only mildly for Pt. The wettability and adhesion of graphite by the slag is poor. However, a temperature-dependent reaction leads to a considerable decrease in the contact angle as well as a foaming behavior of the sessile drop.

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### 1. Introduction

Knowledge of the wettability of a solid by a liquid is the first-step for a complete understanding of the adherence between them. The wettability is usually characterized by contact angle,  $\theta$ , which is related to the surface or interface free energies according to Young's equation:

$$\sigma_{sv} = \sigma_{sl} + \sigma_{lv} \cos \theta \quad (1)$$

where  $\sigma_{sv}$ ,  $\sigma_{sl}$  and  $\sigma_{lv}$  are the solid–vapor, solid–liquid and liquid–vapor interfacial free energies, respectively. The adherence is determined to a large extent by the work of adhesion,  $W_{ad}$ , defined as the energy needed to separate the liquid from the solid surface, given by the Young–Dupré equation:

$$W_{ad} = \sigma_{sv} + \sigma_{lv} - \sigma_{sl} = \sigma_{lv}(1 + \cos \theta) \quad (2)$$

A low  $\sigma_{sl}$  or a small  $\theta$  is thus normally indicative of a strong adhesion. It should be noted, however, that Eq. (2) does not include a term for strain energy and thus mainly applies to the systems where one of the phases is liquid [1].

There have been a number of studies aimed specifically at the wettability and adhesion of refractory materials by molten slags or glasses. Nakashima and Mori briefly reviewed the contact angles between some refractory materials and liquid slags. They indicated that liquid slags wet most refractories, especially when there

is a tendency to react with each other. Slags with higher FeO contents tend to have relatively lower contact angles [2]. Towers showed that the wettability of platinum by various  $\text{CaO--SiO}_2$  and  $\text{CaO--Al}_2\text{O}_3\text{--SiO}_2$  slags varied only slightly with the slag composition or temperature but could be significantly improved by the presence of MnO in the composition [3]. An almost perfect wetting ( $\theta = 2^\circ$ ) was observed for Pt by MnO–30%  $\text{SiO}_2$  in a nitrogen atmosphere at 1673 K [3]. Pask et al. performed a series of studies on the wetting and adhesion between metals, such as Au, Pt, Ni, Fe, Cu, Ag, Mo, W, Ti, Zr and Ta, and molten sodium silicate ( $\text{Na}_2\text{O--SiO}_2$ ) glasses in different atmospheres [4–7]. They suggested that the wetting of a metal by molten glass depends on lowering of the surface energy of the solid by the liquid. The fundamental requirement for good adherence of glass to metal is the development and maintenance of a strong chemical bond at the interface [1]. Of particular interest, considerable attention was paid to the effect of atmosphere. An enhancement in wetting and adhesion can be achieved in an oxygen-incorporated atmosphere by the formation of a metal–oxygen bond or a metal oxide at both the metal–vapor and metal–glass (or slag) interfaces [4–6,8–11].

It was also found that the mixtures of molten oxides, either slags or glasses, do not wet graphite, and their interfacial bonding is ensured by weak, van der Waals, interactions [12]. The non-wetting of graphite by oxide slags is regarded as one of the advantages of carbon bricks in a blast furnace [3]. However, for some slags, the contact angle appears to decrease with time towards values lower than  $90^\circ$  as a result of interfacial reaction between the slag components and carbon [3,13,14]. Sahajwalla et al. investigated the wettability of graphite and other carbonaceous materials

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by CaO–SiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub>–MgO–FeO<sub>x</sub> slags in the temperature range 1673–1873 K [13–16]. They showed that the wettability could be improved by increasing the iron oxide content and temperature. Because of the reaction-induced gas formation, foaming of the droplet was observed at high temperatures and the apparent contact angles displayed a fluctuation behavior. Moreover, the height of the foam was reported to increase with increasing the iron oxide content in the slag [17,18].

In this study, we investigated the wettability of some refractory materials by a molten SiO<sub>2</sub>–MnO–TiO<sub>2</sub>–FeO<sub>x</sub> slag, which is the by-product of a newly developed welding wire after arc welding (provided by Welding Company, Kobe Steel, Ltd., Japan). The main purpose of this work is to find a suitable material with the least interactions with this slag at high temperatures so as to be used as crucible, mould, or substrate to measure some important thermophysical properties such as surface tension and density of this material.

## 2. Experimental procedure

The slag as-received was 38.7% SiO<sub>2</sub>–44.3% MnO–11.4% TiO<sub>2</sub>–5.6% FeO<sub>x</sub> ( $x = 1$  or 1.5) (in mass%). The composition was determined by both wet chemical analysis and fluoroscopy analysis and the two methods gave the similar values. The melting temperature, as determined by differential thermal analysis, was 1407 K. The refractory materials used in this study were molybdenum (99.95% purity), platinum (99.98% purity), tungsten (99.95% purity), polycrystalline alumina (99.99% purity), magnesia (>99% purity), and graphite (>99% purity). They were in either plate or disc shapes with surface dimensions of 20 mm × 20 mm or  $\phi$ 20 mm and heights of 2–5 mm. Their surfaces were mechanically ground and carefully polished using different sizes of diamond pastes to average roughness (Ra) of less than 40 nm for the metal substrates and less than 100 nm for the ceramic and graphite substrates (Ra was measured by a Dektak 3 surface profilometer, Veeco Instruments, Inc., Woodbury, NY, USA). Both the slag and the substrate were ultrasonically cleaned in acetone for three times before wetting experiment.

The wetting experiment was performed in an Ar atmosphere using a modified sessile-drop method. A detailed description of the experimental apparatus and procedure could be found elsewhere [19]. In all the experiments, the chamber was first evacuated to a vacuum about  $4 \times 10^{-4}$  Pa at room temperature and then heated at a rate of 20 K min<sup>-1</sup>. When the desired testing temperature was reached, the purified Ar gas (99.999% purity) was introduced into the chamber. After the temperature and the pressure in the chamber were stabilized, the slag sample, initially placed in a glass tube outside the chamber, was dropped through an open alumina tube into the chamber and then rested on the substrate surface. The weight of the slag was in the range of 0.1–0.2 g, depending on the substrate material (e.g., a small mass of the slag sample was used for the metal substrates because of excellent wetting and a relatively large mass of the slag was used for graphite because of poor wetting). The pollution of the slag by the alumina dropping tube was avoided by controlling the dropping distance and immediately lowering the slag sample when it contacted the substrate surface. Depending on the testing temperature, it took several seconds to tens of seconds for the slag to melt after it was dropped. As soon as the melting was observed, the drop profile was recorded by a high resolution CCD camera and the time was defined as the start point for the wetting. On the other hand, if the slag was found to adhere to the alumina dropping tube, the results were discarded and the experiment was repeated.

After the wetting experiment, most of the samples were furnace-cooled at a rate of 20 K min<sup>-1</sup> and some were cooled at 10 K min<sup>-1</sup> to examine the effect of cooling rate. Selected samples were immersed in resin with hardener and then sectioned perpendicular to the slag–substrate interface after natural drying. Cross-sections were ground on wet silicon carbide sandpapers and carefully polished by diamond pastes down to 0.25  $\mu$ m to prepare the metallographic specimens. Interfacial microstructures were examined by a confocal scanning laser microscope (Keyence, VK-8550, Osaka, Japan) and an environmental scanning electron microscope (ESEM-2700, Nikon Co., Kanagawa, Japan) with X-ray energy dispersive microanalysis (EDAX) capability.

## 3. Results and discussion

### 3.1. On refractory metals (Mo, Pt and W)

Figs. 1–3 show the time variation of the contact angles for the molten slag on the Mo, Pt and W surfaces at 1473–1673 K, respectively. As indicated, the slag wets these metals quite well. The wettability depends not only on the metal species but also on

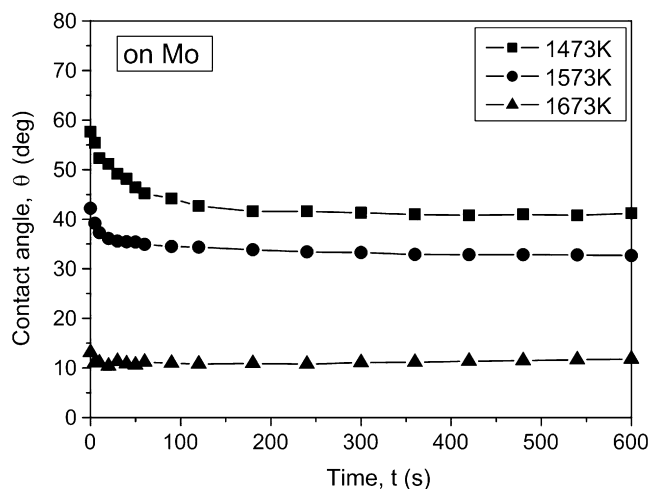


Fig. 1. Variation in the contact angle with time for the slag dropped on the Mo surfaces at 1473–1673 K.

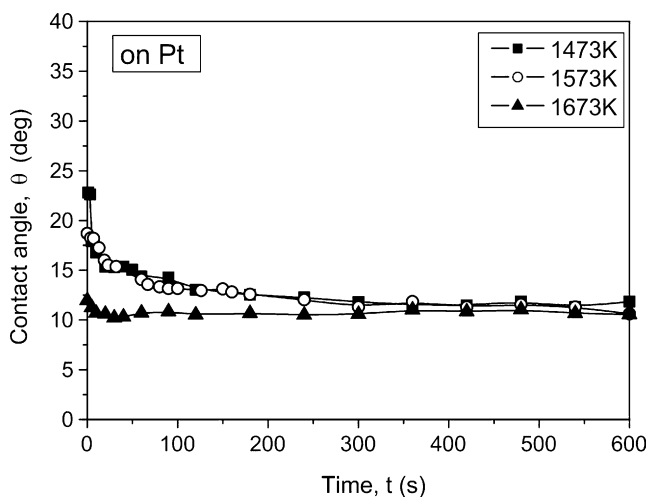


Fig. 2. Variation in the contact angle with time for the slag dropped on the Pt surfaces at 1473–1673 K.

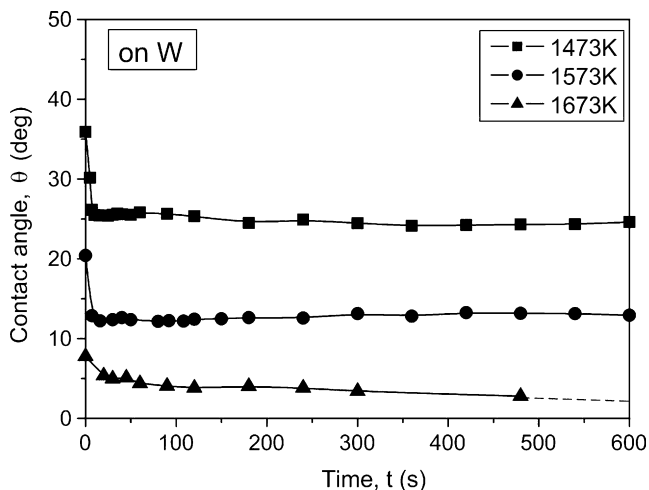
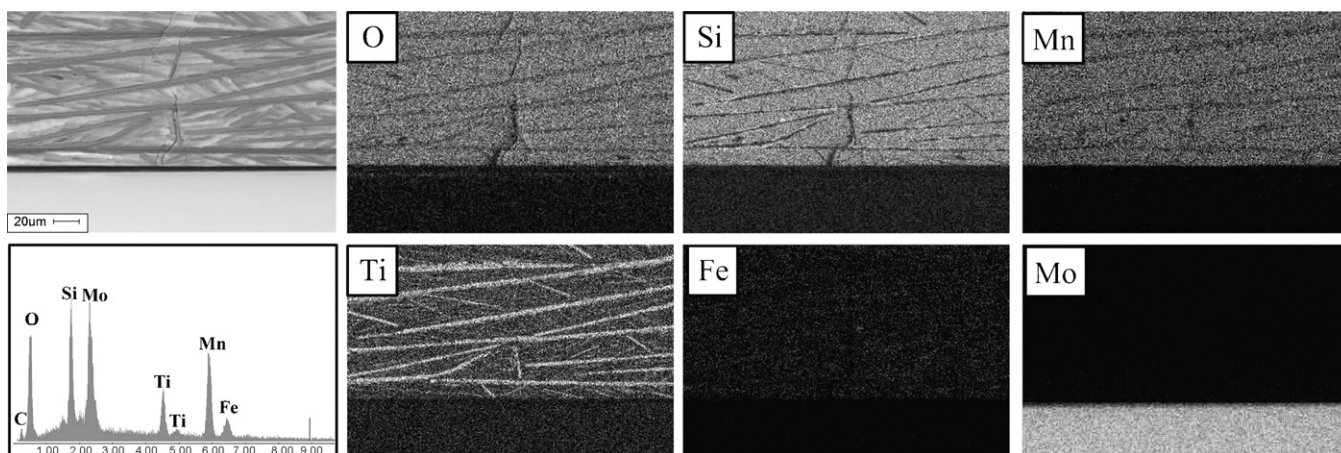


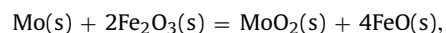
Fig. 3. Variation in the contact angle with time for the slag dropped on the W surfaces at 1473–1673 K.



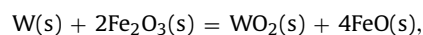
**Fig. 4.** Interfacial microstructure and elemental mapping graphs for the slag–Mo wetting couple tested at 1573 K for 10 min and then cooled at a rate of 10 K min<sup>-1</sup>. Similar microstructures were also observed at the slag–Pt and slag–W interfaces.

temperature. At  $T=1473$  K, the wettability is in the order of  $Pt > W > Mo$ . With an increase in temperature, the wettability of Mo and W by the slag is significantly improved. Perfect or nearly perfect wetting ( $\theta \rightarrow 0^\circ$ ) appears on the W surface at 1673 K. For Pt, however, the improvement is minor.

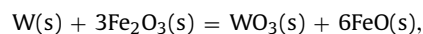
The cooling rate at 10–20 K min<sup>-1</sup> does not seem to greatly affect the detachment behavior of the slag from the substrate surfaces. Most of the solidified slag drops were peeled off from the substrate surface, indicating poor adherence to these metals during furnace-cooling to room temperature. However, it seems that the adhesion was strongest for the slag on the Pt surface since some slag–Pt wetting couples were not separated after cooling. Towers suggested that Pt could react with the manganese silicate slag, which leads to very small contact angles ( $\theta < 10^\circ$ ) [3]. Instead, Bowen and Shairer suggested a direct reaction between Pt and ferrous silicate slag [20]. They argued that Pt reduced the ferrous silicate to metallic Fe, which immediately alloyed with Pt. A study performed by Adams and Pask on the wettability of Pt by molten sodium silicate with various iron oxide additions (ranging from 2.4 to 44.4 mol%), however, indicated that the iron concentration in the sodium silicate glass did not essentially affect either the contact angle or the adherence [21]. Here, we calculated the changes in the standard Gibbs free energies ( $\Delta G^\circ$ ) for all the potential reactions between the metallic substrates (Mo, Pt and W) and the slag components, and suggested that the following reactions might be possible at  $T \geq 1473$  K from the viewpoint of thermodynamics (i.e.,  $\Delta G^\circ < 0$ ) [22]:



$$\Delta G^\circ = -4855 - 77.4T \text{ J mol}^{-1}$$



$$\Delta G^\circ = -7784 - 72.08T \text{ J mol}^{-1}$$



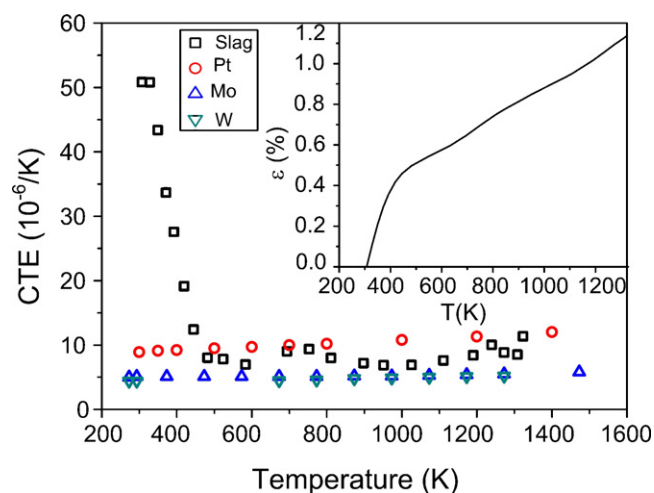
$$\Delta G^\circ = 26,608 - 120.45T \text{ J mol}^{-1}$$

These reactions might account for the temperature and time dependent behaviors of the contact angle, as shown in Figs. 1 and 3. Ringel also suggested that the dissolved  $\text{Fe}^{3+}$  could react with Mo at the interface to form  $\text{MoO}_x$ , which lowered the solid–liquid interfacial energy and resulted in the decrease in the contact angle [23]. Microscopic examinations, however, did not reveal a significant reaction

but only limited interdiffusion between the slag and the metal (Mo, Pt and W) substrates (see, for example, Fig. 4). The comparatively stronger adhesion for the slag on Pt may result, on the one hand, from the effect of surface adsorbed oxygen with regard to great adsorption affinity of Pt for oxygen (it was reported that removal of all chemisorbed oxygen from the Pt surface requires a vacuum of  $\sim 10^{-7}$  Pa at temperatures approaching the melting point of Pt [1,6]), and on the other hand, from the smaller thermal stress caused by the difference in the coefficients of thermal expansion (CTE). The temperature dependence of the CTE values of the slag, Pt, Mo and W [24] is shown in Fig. 5. The relatively smaller difference in the CTE between Pt and the slag, as compared with that for W and Mo, decreases the interfacial stress and thus leads to the stronger adhesion during cooling. In addition, it is worth mentioning that the detachment was found to occur mostly at temperatures below 473 K, where the CTE difference between the slag and the metal substrate is much larger.

### 3.2. On alumina and magnesia

Fig. 6 shows the variations in the contact angle and normalized drop base diameter, which is defined as the proportion of the



**Fig. 5.** The temperature dependence of the CTE values of slag, Pt, Mo and W. The values of Pt, Mo and W are taken from Ref. [24]. The inset figure shows the thermomechanical analysis result of the line expansion strain ( $\epsilon$ ) of the slag at elevated temperatures.

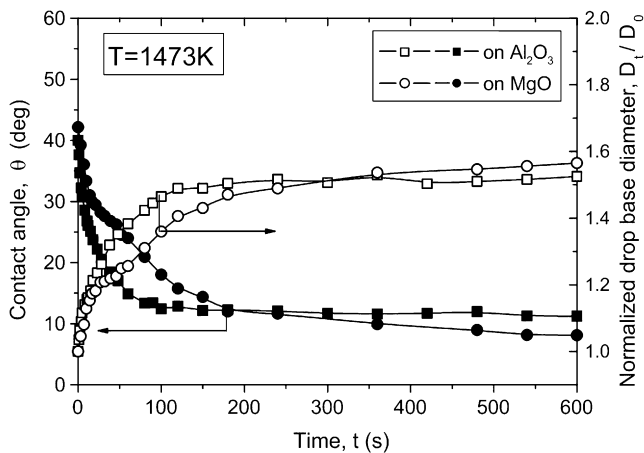


Fig. 6. Variations in contact angle and normalized drop base diameter with time for the slag dropped on the  $\text{Al}_2\text{O}_3$  and  $\text{MgO}$  substrates at 1473 K.

dynamic base diameter ( $D_t$ ) to the initial base diameter ( $D_0$ ), with time for the molten slag on the  $\text{Al}_2\text{O}_3$  and  $\text{MgO}$  substrates at 1473 K. In both cases, the contact angle decreases rapidly in the first 3 min and then gradually reaches a steady value. The initial and the final contact angles are, respectively,  $40^\circ$  and  $11^\circ$  for  $\text{Al}_2\text{O}_3$ , and  $42^\circ$  and  $8^\circ$  for  $\text{MgO}$ .

Fig. 7 shows the interfacial microstructure and elemental mapping graphs for the slag on the alumina surface. Similar microstructure was also observed on the  $\text{MgO}$  surface. As indicated, a significant dissolution of the substrate into the molten slag during the wetting occurred and a concave reaction layer was developed beneath the drop. The spreading front of the slag meets the reaction front in the horizontal direction, implying that the spreading and the reaction proceeded almost at the same speed. In a strict sense, the measured contact angles are not the Young angles since the interface at the triple junction is no longer straight.

Examination of the samples after cooling revealed that the slag spread extensively over the alumina and magnesia surfaces. The periphery of the slag drops, however, did not form a smooth circle, implying a non-uniform spreading. The samples exhibited strong adherence at room temperature.

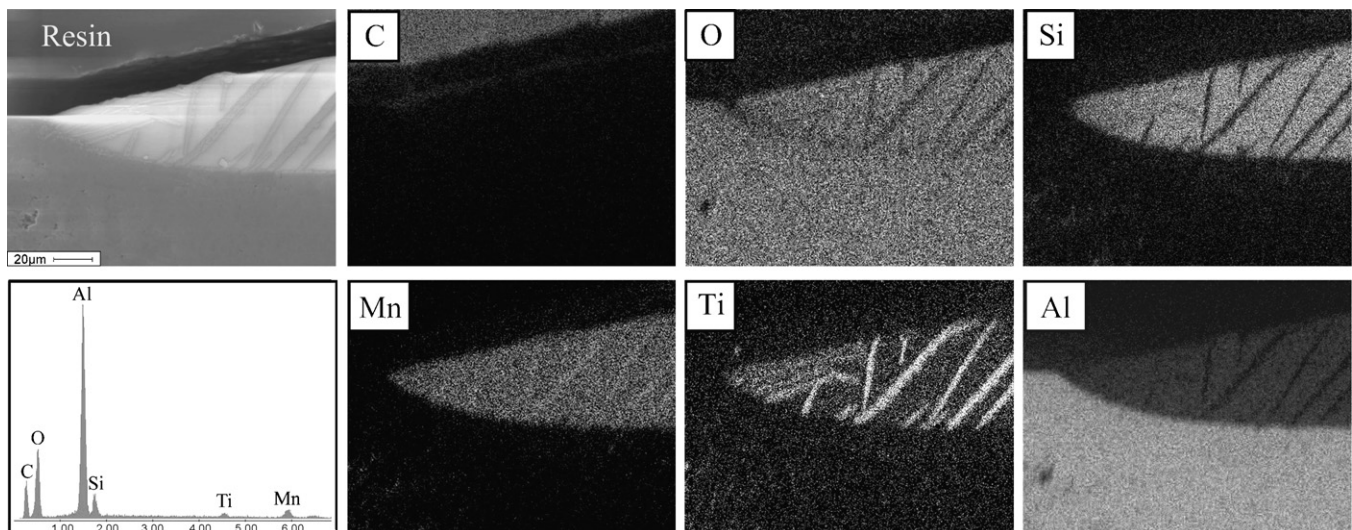


Fig. 7. Interfacial microstructure and elemental mapping graphs for the slag–alumina wetting couple tested at 1473 K for 10 min and then cooled at a rate of  $20 \text{ K min}^{-1}$ .

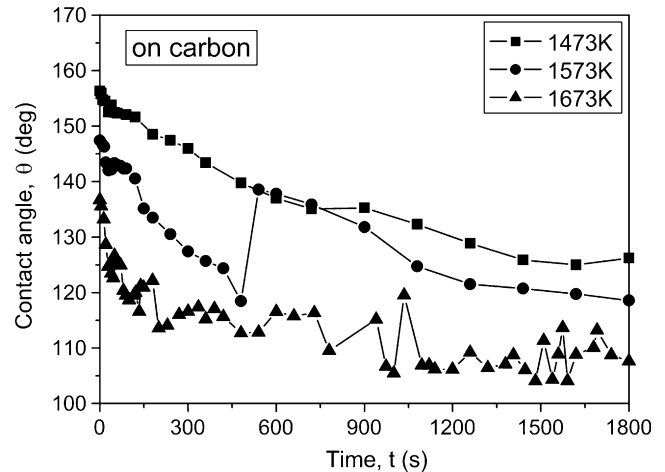


Fig. 8. Variation in the contact angle with time for the slag dropped on the graphite substrates at 1473–1673 K.

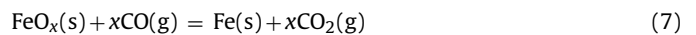
### 3.3. On graphite

Fig. 8 shows the variation in the contact angle with time for the slag on the graphite surfaces at 1473–1673 K. As indicated, the slag does not wet graphite. An increase in temperature considerably promotes the wettability. The contact angles, on the other hand, display a fluctuation behavior at high temperatures. The frequency of the fluctuation increases with increasing temperature. Fig. 9 gives some representative photographs showing this phenomenon.

Fig. 10 shows the interfacial microstructure and elemental mapping graphs for the slag on the graphite substrate at 1573 K after 30 min. The presence of metallic Fe at the slag–graphite interface clearly indicates that the following reaction took place:



The carbon monoxide might further reduce the iron oxide to form carbon dioxide:



The fluctuation is the result of the gas evolution in the reaction process. At low temperatures (e.g.,  $T = 1473 \text{ K}$ ), the reaction

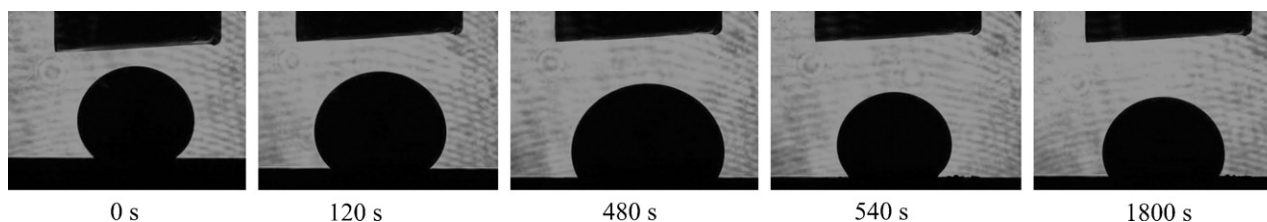


Fig. 9. Representative photographs showing the drop fluctuation phenomenon at 1573 K.

proceeded slowly so that the generated gas had time to escape, leading to a progressive decrease in the contact angle. With the increase in temperature, the reaction and the gas evolution were significantly promoted. As indicated in Fig. 9, when the gas generation rate exceeded its escaping rate, the gas could accumulate in the drop bulk, making the drop inflate and the liquid front advance. Once the gas released, the drop contracted. At higher temperatures (e.g.,  $T = 1673$  K), the reaction was so vigorous that the formation and release of the gas bubbles finished in a short time, and therefore the slag drop displayed a frequent fluctuation behavior rather than a significant volume inflation.

It is not easy to evaluate the extent of the wetting improvement by the chemical reaction between the slag and graphite because of the drop fluctuation. The wetting, however, is indeed improved by the reaction, as witnessed by the decrease in the contact angle and spreading of the triple line at low temperatures, where the influence of the gas evolution is insignificant. Formation of metallic Fe at the slag–graphite interface might contribute to this result, as inferred from the foregoing good wetting of the metals (Mo, Pt and W) by the slag.

Examination of the solidified samples showed that the slag was robustly adhered to the graphite substrates. Reaction traces such as the reduced iron and many pinholes as a result of gas evolution were visible at the periphery of the drops after contraction.

### 3.4. Work of adhesion

Although the surface tension of the slag could be calculated together with the contact angle by using an axisymmetric-drop-shape analysis (ADSA) program adopted here, the values obtained

from the slag resting on the Mo, Pt and W substrates vary considerably due to good wettability, small weight of the slag samples and possible distortion of the drops during spreading, and thus they are not reliable. On the other hand, the results obtained from the molten slag on the graphite substrates could be unfortunately affected by the chemical reaction. Therefore, only the values obtained from the slag–graphite system within a short time (0–10 s) after the formation of the drop were thought as reliable, and the surface tensions of the slag were calculated to be  $385 \pm 9$ ,  $379 \pm 7$  and  $366 \pm 11$   $\text{mJ m}^{-2}$  at 1473, 1573 and 1673 K, respectively.

The work of adhesion,  $W_{ad}$ , in terms of Eq. (2), was then calculated using the available contact angle and surface tension values, and the results are given in Table 1. As indicated, the values of  $W_{ad}$  in the slag–metal (Mo, Pt, and W) and slag–oxide ( $\text{Al}_2\text{O}_3$  and  $\text{MgO}$ ) systems at high temperatures are quite similar (between 0.60 and 0.75  $\text{J m}^{-2}$ ) and are much larger than those in the slag–graphite system. If we take the empirical relationship of  $W_{ad} \approx 0.2W_{c(l)}$  (where  $W_{c(l)}$  is the cohesion of liquid and is equal to  $2\sigma_{lv}$ ) as a typical characteristic of the physical interaction due to van der Waals dispersion forces, while  $W_{ad} > 0.2W_{c(l)}$  as a chemical interaction in nature [12], we can conjecture that the high-temperature adhesion in the slag–metal (Mo, Pt and W) and slag–oxide ( $\text{Al}_2\text{O}_3$  and  $\text{MgO}$ ) systems are ensured by chemical interactions, whereas that in the slag–graphite system by van der Waals (i.e., physical) interactions. With the progress in the chemical reaction, the adhesion between the molten slag and the graphite surface enhanced and a chemical interaction was eventually developed. It is worthwhile to mention again that the work of adhesion discussed here does not include the effect of the strain energy resulting from the difference in the CTE

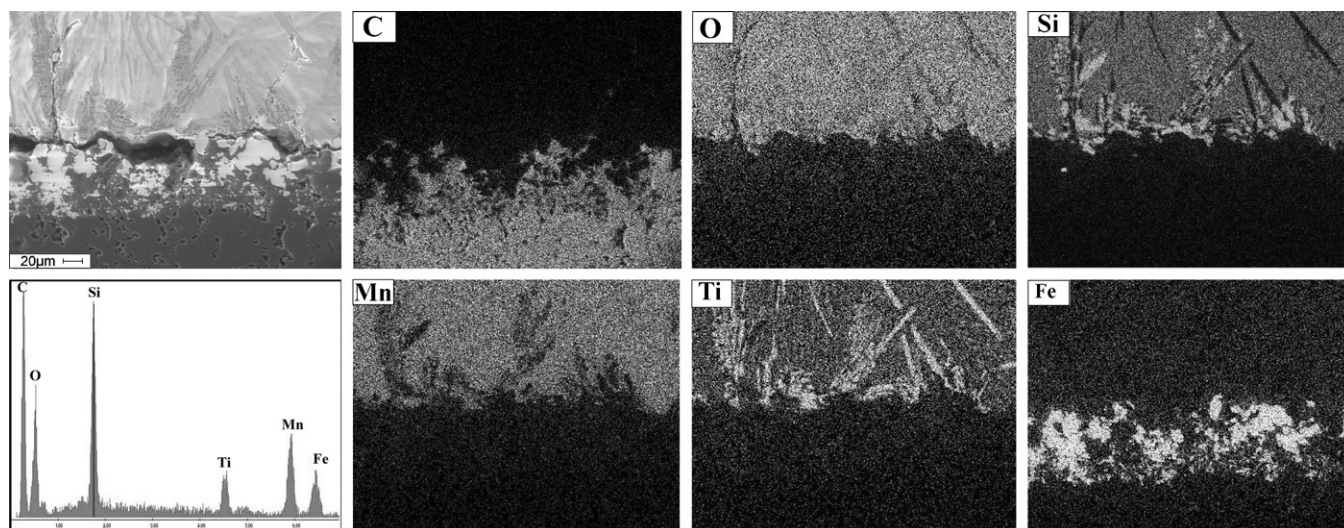


Fig. 10. Interfacial microstructure and elemental mapping graphs for the slag–graphite wetting couple tested at 1573 K for 30 min and then cooled at a rate of 20  $\text{K min}^{-1}$ .

**Table 1**  
Work of adhesion between the slag and various substrates and work of cohesion of the liquid slag.

Temperature (K)	Work of adhesion, $W_{ad}$ ( $\text{mJ m}^{-2}$ ) <sup>a</sup>					Work of cohesion, $W_c$ ( $\text{mJ m}^{-2}$ )	
	Mo	Pt	W	$\text{Al}_2\text{O}_3^b$	$\text{MgO}^b$	Graphite	liquid slag
1473	591 (675)	740 (762)	697 (735)	680	671	32 (158b)	$770 \pm 18$
1573	660 (698)	738 (753)	734 (748)	–	–	60 (198b)	$758 \pm 14$
1673	722 (724)	724 (726)	729 (732a)	–	–	100 (255b)	$732 \pm 22$

<sup>a</sup> If not specifically indicated, the values outside the parentheses are taken from the moment  $t=0$  s, while those inside are from the moment  $t=600$  s. The values with 'a' and 'b' are taken from the moments  $t=480$  and  $1800$  s, respectively.

<sup>b</sup> For alumina and magnesia, no values of  $W_{ad}$  at  $t=600$  s are indicated since the final contact angles measured are no longer the Young angles.

values between the slag and the refractory materials, as we have indicated before.

#### 4. Conclusions

The wettability of Mo, Pt, W,  $\text{Al}_2\text{O}_3$ , MgO and graphite by the 38.7%  $\text{SiO}_2$ –44.3%  $\text{MnO}$ –11.4%  $\text{TiO}_2$ –5.6%  $\text{FeO}_x$  slag at 1473–1673 K was investigated using the sessile drop method. The slag wets Mo, Pt and W quite well. Increasing temperature considerably promotes the wettability of Mo and W but only mildly for Pt. However, because of the large difference in the thermal expansion coefficients between the slag and the metals, the solidified slag readily detaches from the metal surfaces during cooling. For the molten slag on the alumina and magnesia substrates, a strong dissolution/reaction leads to an extensive spreading, small apparent contact angles ( $\sim 10 \pm 2^\circ$ ) and good adherence. On the other hand, the slag does not wet graphite, making it potential for the application as crucible, mould or substrate for measurement of some thermophysical properties (e.g., surface tension and density) of the slag. However, the chemical reaction between the graphite and the slag at high temperatures brings a considerable enhancement in the wettability and adhesion as well as a foaming behavior of the drop, making it also undesirable for such applications.

#### Acknowledgements

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