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Thermophysical properties of slags for process control

B. J. Monaghan and R. F. Brooks

Thermophysical properties have been shown to be important in a number of industrial processes. Unfortunately, for a number of properties there are scant data available and/or the data that are available are of dubious quality. In the Thermo-Physical Property Group at the National Physical Laboratory (NPL) the aim has been to develop measurement techniques capable of working in the difficult environments that are typical of industrial processes. This paper presents current developments at NPL in techniques to measure the thermal diffusivity of liquid slags using a laser flash and insights into the copper smelting process using interfacial tension techniques. I&S/1681

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INTRODUCTION

Computer models that simulate industrial processes are increasingly used to reduce costs and improve productivity. The success of such models is highly dependent on the thermophysical property data used in the simulation calculations. There are few data available for these models and the data that are available are often of questionable quality. This lack of reliable thermophysical data is a major impediment to realising the full potential of current process modelling technology. Most of the thermophysical data that are available for liquid oxide systems are contained in the 'Slag atlas' data compendium¹ that was published in 1995.

The fact that there are scant liquid oxide data and that the data often have large uncertainties associated with them is symptomatic of the difficulty experienced in handling liquid oxide systems. The principle difficulties are:

1. Experimentally, liquid oxides are difficult to handle. They are often reactive with both containment vessel and atmosphere. This causes problems with reproducibility of measurement as the oxide changes composition. This is critical in surface tension measurements where small changes in composition can have a large effect on the measurement.
2. Liquid oxide measurements are invariably high temperature measurements. Most traditional thermophysical property measurement techniques were designed for ambient or near ambient temperatures. Modifying these methods for high temperatures often increases the uncertainty in the measurement.
3. Heat transfer in liquid oxides is complex, involving phonon (thermal diffusion) and photon (radiation) conduction. It is difficult to separate the radiation component of heat conduction from the thermal diffusivity measurements.
4. At the high temperatures encountered when dealing with liquid oxides, it is difficult to obtain the thermal

stability in order to avoid convective (mass) flow in a sample. This can lead to apparently high measured thermal diffusivities.

Currently, in the Materials Centre of the National Physical Laboratory (NPL), studies are on-going into the thermal, transport, interfacial, and thermodynamic properties of liquid oxide systems. This paper summarises the developments carried out on the NPL thermal diffusivity laser flash apparatus (LFA) and on using the maximum bubble pressure (MBP) for the measurement of surface tension and density of liquid oxide systems.

THERMAL DIFFUSIVITY

Thermal diffusivity measurement by LFA

Thermal diffusivity is defined as the rate of heat diffusion per unit time, as follows²

$$a = \frac{\lambda}{C_p \rho} \dots \dots \dots (1)$$

where a is the thermal diffusivity ($\text{m}^2 \text{s}^{-1}$), λ is the thermal conductivity ($\text{W m}^{-1} \text{K}^{-1}$), C_p is the specific heat capacity ($\text{J kg}^{-1} \text{K}^{-1}$), and ρ is the density (kg m^{-3}).

A typical laser flash thermal diffusivity measurement involves heating the front face of a disc shaped sample of known thickness using a high intensity laser and monitoring the temperature rise on the back face. From the temperature rise, the thermal diffusivity can be calculated. A furnace is used to heat the sample to the desired temperature.

There are numerous models available to calculate the thermal diffusivity from the temperature transient of a single layer sample.^{3–7} Making a thermal diffusivity measurement of a slag material is more complex, because the liquid oxide is held in a measurement cell. The effects of this measurement cell on the measured temperature transient are not negligible. In this paper, Lee's three layer model is used to account for the cell effects, the details of which have been described elsewhere.^{8,9}

In Lee's model, the unknown thermal diffusivity of the liquid oxide can be determined if the thermal properties (thermal diffusivity, heat capacity, and density) of the cell and the heat capacity and density of the liquid oxide are known. The thermophysical property data used in the three layer model are given in Table 1.

Although density values used may change with temperature, no allowance is made for changes in thickness of any layers with respect to expansion. All values used for thickness are as measured at room temperature.

Experimental

Measurement cell

The measurement cell consists of an alumina cap, platinum lid, platinum crucible, and alumina crucible support (see Fig. 1). The primary function of the alumina cap is to ensure that the platinum lid is seated on the platinum crucible and not floating on the liquid slag surface. This enables definition of the sample thickness, a critical parameter in the measurement of thermal diffusivity.

1 Slag cell (lid and crucible), support, and cap

Materials preparation

The composition of oxides measured in this study are given in Table 2. The oxide material was added to the measurement cell in powder form and melted before measurement. This required more than one filling-melting sequence to ensure the cell was full

Table 1 Thermal diffusivity, heat capacity, and density values of platinum cell and density values of ASK (heat capacity for ASK at 1000–1700°C is 1.44 J g⁻¹ K⁻¹): density and specific heat values of ASE at all temperatures are 2700 kg m⁻³ and 1.35 J g⁻¹ K⁻¹ respectively

Temp., °C	Platinum cell			Density of ASK, kg m ⁻³
	Thermal diffusivity, (× 10 ⁻⁴) m ² s ⁻¹	Heat capacity, J g ⁻¹ K ⁻¹	Density, kg m ⁻³	
20	0.137	0.142	20000	...
100	...	0.146	19960	...
195	0.163	...	...	...
200	...	0.149	19900	...
300	...	0.153	19840	...
392	0.184	...	...	...
400	...	0.157	19780	...
500	...	0.160	19720	...
588	0.197	...	...	...
600	...	0.163	19660	...
700	...	0.166	19600	...
794	0.208	...	...	...
800	...	0.169	19530	...
900	...	0.173	19460	...
996	0.218	...	...	...
1000	...	0.176	19400	2639
1100	...	0.179	19320	2611
1190	0.222	...	...	...
1200	...	0.182	19250	2584
1300	...	0.185	19180	2558
1356	...	...	...	2543
1357	...	...	...	2543
1400	...	0.188	19100	2532
1405	0.229	...	...	...
1500	...	0.190	19020	2507
1600	0.234	0.194	18930	2482
1700	0.236	0.197	18850	2458
1740	...	0.198	...	...
1800	0.237	...	18770	...

2 Effective thermal diffusivity a_e measurements (made by number of laboratories) on slag ASE as function of temperature

In melting, a compromise had to be made between heating to a temperature that ensured the slag material was fully molten, but not so high that significant quantities would be lost to the gas phase. All melting and measurements were carried out in an argon atmosphere.

Results and discussion

The results of measurements made on samples ASE and ASK are shown in Figs. 2 and 3 respectively. Unless otherwise stated, all sample thicknesses were of the order of 1.5 mm. For samples of this order of thickness, the thermal diffusivities of flux ASE and CaO–Al₂O₃–SiO₂ slag ASK are of the order 0.005 × 10⁻⁴ m² s⁻¹. None of the measurements presented in Figs. 2 and 3 has been corrected for a radiation contribution and the values are therefore referred to as effective thermal diffusivity a_e .

On inspection of Fig. 2, it can be seen that, within the scatter in the data, the measurements are in good agreement. Also, the effective thermal diffusivity shows an increase with increasing temperature.

The ASK measurements shown in Fig. 3 were measured at two different thicknesses and on a heating and cooling cycle. In general, the data show a similar trend with temperature to ASE and that measuring on the heating (h) and cooling (c) cycles had no effect on the effective thermal diffusivity. Also, it is apparent from the graph that increasing the thickness increases the effective thermal diffusivity. The increased thickness of the sample was obtained by inserting a platinum ring between the platinum cell lid and crucible.

It is clear from Figs. 2 and 3 that the LFA apparatus can make measurements on slags ASE and ASK. What is not so clear is what these measurements represent. Most slag materials of industrial interest are either transparent or semitransparent to the infrared wavelengths and the laser light wavelength of 1.064 μm used in the LFA.^{1,10,11} As a consequence of this, the heat transfer through the sample has both a phonon (thermal diffusion) and photon (radiation) contribution as shown in the following equation

$$a_e = a + a_{\text{rad}} \dots \dots \dots (2)$$

Table 2 Slag compositions* used in thermal diffusivity measurements by laser flash apparatus, wt-%

Fe (total)	CaO	SiO ₂	Al ₂ O ₃	S	Na ₂ O	F	Other
ASE1 (decarburised mould flux)							
1.06	34.92	38.44	6.72	0.092	12.25	6.66	Bal.
ASK							
...	15.52	64.51	15.05	...	...	...	Bal.

*Data measured by British Steel Teesside Works.

3 Effective thermal diffusivity a_e measurements on slag ASK as function of temperature at two different sample thicknesses: h and c denote heating and cooling cycles respectively

where a_{rad} is a radiation term. The significance of this radiation term will be dependent on the difference in temperature between the adsorbing and emitting surfaces (i.e. platinum surfaces if the slag is transparent, platinum surfaces and the slag if semitransparent), the absorption α /emission ϵ coefficient of the slag, and the effective penetration depth of the radiation.¹² It is a difficult parameter to calculate because:

- there are very few absorption/emission data on slags and most of the data that does exist has usually been measured at ambient temperature¹
- the power of laser pulse is never measured, which makes calculation of the surface temperature differences difficult
- it depends upon the optical thickness αd , where d is the thickness – the radiation conductivity λ_R for optically thick conditions, where $\alpha d > 3$, can be calculated, but for optically thin specimens, as used in this study, where $\alpha d < 3$, it is more difficult to evaluate the radiation conduction contribution.

The matter is further complicated by the fact that to solve for a_e using the three layer model, data for the heat capacity and density of the slag are also required. These data are often not readily available or are of poor quality and as a result may have to be modelled, further increasing the uncertainty in the measured thermal diffusivity.

Most approaches to the radiation problem have involved modelling the data on a grey body^{11,13} or transparent body¹⁰ assumption. According to the authors,¹⁰ the grey body assumption is only required for slags containing FeO or TiO₂.

If radiation conduction is a problem in the data measured at NPL, then the measured (effective) thermal diffusivities would be expected to increase with increasing temperature and increasing size.^{1,10,11,13} Inspection of both Figs. 2 and 3 are consistent with such trends. It is therefore likely that the measured thermal diffusivities do have a radiation component associated with them.

As mentioned previously, Waseda *et al.*¹⁰ stated that a transparent body approximation to radiation could be used for slags that do not contain FeO or TiO₂. They also developed¹⁰ a set of equations that could correct for the effect of radiation conduction under such conditions. Their approach was as follows. They assumed a value for thermal diffusivity of a liquid slag material of $0.004 \times 10^{-4} \text{ m}^2 \text{ s}^{-1}$. Then they modelled the effect that radiation would have on a thermal diffusivity measurement using the LFA by a finite difference method. For an effective thermal diffusivity as defined in

$$a_e = ca \quad (3)$$

4 Thermal diffusivity measurements of slag ASK corrected for radiation contribution as function of temperature: results reported in 'Slag atlas'¹ refer to composition 25CaO-15Al₂O₃-60SiO₂ (wt-%)

where c is the effect of radiation, they proposed the following equations for c , for 0.1, 0.2, and 0.3 mm thick samples respectively

$$c = 2.549 \times 10^{-7} T^2 - 4.504 \times 10^{-4} T + 1.244 \quad (4)$$

$$c = 6.986 \times 10^{-7} T^2 - 1.466 \times 10^{-3} T + 1.883 \quad (5)$$

$$c = 1.253 \times 10^{-6} T^2 - 2.675 \times 10^{-3} T + 2.606 \quad (6)$$

These equations have been derived for sample sizes that are much smaller than those measured by NPL. In order to utilise this approach, the constants in equations (4)–(6) were linearly regressed with respect to sample thickness to obtain the following equations for thicknesses of 1.53 and 2.05 mm respectively

$$c = 7.372 \times 10^{-6} T^2 - 1.628 \times 10^{-2} T + 10.968 \quad (7)$$

$$c = 9.967 \times 10^{-6} T^2 - 2.206 \times 10^{-2} T + 14.510 \quad (8)$$

This is not a rigorous approach as it is always questionable when using relations outside the region that they were derived for.

Ignoring heat loss effects, if there is not a radiation component to a thermal diffusivity measurement by the LFA, then the value obtained should be independent of sample thickness.¹² Also, for highly structured materials (crystalline solids), the phonon contribution would be expected to show a negative dependency of thermal diffusivity with increasing temperature.¹² This is a result of distortion of the thermal energy waves by the vibration of the lattice atoms in a crystalline structure. This vibration increases with increasing temperature. It is not so clear how a liquid structure would behave. If it was highly polymerised perhaps it would follow similar behaviour to a solid lattice with increasing temperature, i.e. the thermal conductivity would decrease. Also, it can be expected that for polymerised melts, the higher the temperature, the smaller the chain length.¹ The smaller the chain length the larger the number of molecules and therefore boundaries between molecules. This increase in the number of boundaries will have a greater resistance to heat flow. Such a process is analogous to grain boundary effects in metal alloys, where the thermal conductivity is inversely proportional to the number of grain boundaries.¹²

To assess the validity of equations (7) and (8), the data shown in Fig. 3 for the two different sample thicknesses should be recalculated using equations (3), (7), and (8). The results are shown in Fig. 4 and compared with values obtained from Ref. 1 for a material of a similar composition to ASK. If the radiation component has been assessed correctly, the thermal diffusivities should be independent

of sample thickness, i.e. reduce to one line. Also, from the foregoing argument on conductivity with temperature, it is to be expected that the diffusivity would decrease with increasing temperature.

On inspection of Fig. 4, it can be seen that the data still persist as two linear groups, although they are closer than in Fig. 3. In Fig. 3, the difference in the diffusivities between the 1.53 and 2.06 mm slag samples is approximately 100%, compared with 25% for the corrected values shown in Fig. 4. Also, the 'curves' do show a negative dependency of thermal diffusivity on increasing temperature. This would suggest that the radiation correction derived for the small sample size may have limited application at the thickness employed in this study. Also, when comparing the values obtained with those of a similar slag composition,¹ it can be seen, particularly for the smaller sample, that the agreement is reasonable.

From the data shown in Fig. 4, it can be concluded that corrections for radiation can be made, but if the capability of the LFA technique for measuring thermal diffusivities of slags is to be fully realised, then the effects of radiation for the particular sample geometry have to be determined. Also, the radiation correction used was for a transparent body approximation. This would not be appropriate for semitransparent materials, and other corrections would have to be determined. This would require data on the optical properties of the slag materials. At present, these data do not exist, or are of poor quality, or are available at unsuitable temperatures.

SURFACE TENSION

Surface tension measurement by MBP

Surface tension γ , defined as the force resulting from the incomplete coordination of atoms residing at an interface between a solid or liquid with a gas, is an important parameter affecting flow and wear patterns in a number of industrial processes.¹² At NPL, a MBP apparatus has been developed to measure the surface tension and density of liquid oxides.

An MBP measurement is made by measuring the pressure required to blow a bubble of gas at the end of a capillary submerged a known distance in the sample liquid. The surface tension can be calculated from¹⁴

$$\gamma = \frac{rP_\gamma}{2} \left[1 - \frac{2}{3} \left(\frac{r\rho g}{P_\gamma} \right) - \frac{1}{6} \left(\frac{r\rho g}{P_\gamma} \right)^2 \right] \quad (9)$$

where P_γ is defined by

$$P_\gamma = P_m - \rho gh \quad (10)$$

and r is the radius of the capillary (m), ρ is the density of the liquid (kg m^{-3}), g is acceleration due to gravity (m s^{-2}), h is the depth of immersion of the capillary (m), and P_m is the maximum pressure measured (Pa).

Density can also be obtained from MBP measurements by blowing bubbles at two different depths using

$$\rho = \frac{(P_{m2} - P_{m1})}{g(h_2 - h_1)} \quad (11)$$

where the subscripts 1 and 2 denote measurements at two distinct depths.

Experimental

The apparatus consisted of an alumina environmental chamber enclosed in a resistance furnace, with an alumina capillary (3 mm i.d.) inserted through an O ring seal at the top of the chamber. The samples were held in an alumina or molybdenum crucible. The depth of immersion was measured relative to the liquid surface by a vernier calliper attached to the capillary lifting mechanism. The surface of the liquid oxide was detected by an increase in the gas

pressure inside the capillary. On detection, the vernier calliper was zeroed and depths measured directly from the calliper thereafter. The calliper had a resolution of 0.01 mm. This was the principle error in the detection of the surface of the liquid oxide. The formation of argon bubbles were controlled by (fine) needle valves to obtain a bubble rate of approximately 6 bubbles per minute. Pressure was measured using pressure transducers, the output of which was read by digital voltmeter and logged to a PC.

The positioning of the capillary in the crucible is critical if end effects resulting from bubble interactions with the wall and the bottom of the crucible are to be avoided.

Materials preparation

Measurements were made on a prefused decarburised mould flux (PMF) and a copper blast furnace slag (CBFS), the compositions of which are given in Table 3. The iron composition given in the table for slag CBFS is primarily oxide. The samples were heated to a maximum temperature, measured, and then cooled for subsequent measurements.

The PMF was chosen as a relatively inert liquid oxide enabling testing of the measurement system. The crucible material used for PMF was molybdenum. The powdered sample was premelted in an induction furnace before measurement in the MBP apparatus. This melting was repeated until the crucible contained approximately 50 mm depth of oxide. Such a depth enabled measurements to be made at 3 or 4 depths, ~10 mm apart.

The CBFS was a more reactive slag, and due to the high metal/iron oxide content, could not be melted into a molybdenum crucible. Therefore, an alumina crucible was used. The high reactivity also meant that the sample could only be melted *in situ* in the MBP apparatus. This limited the amount of material available for measurement, providing only enough to allow two depths (10 and 20 mm) at each temperature. Also, to minimise the risk of damage to the MBP apparatus, measurements were only made over a limited temperature range. Measurements of the viscosity of this material had already been attempted at higher temperatures in the NPL rotating bob viscometer resulting in considerable damage to the equipment.

Results and discussion

Results for PMF and CBFS are given in Figs. 5 and 6 respectively. The different data points at a given temperature represent different measurements depths. In Fig. 5, it can be seen that, within the scatter of the data, the surface tension and density values obtained are independent of measurement depth or temperature, and are 320 mN m^{-1} and 2600 kg m^{-3} respectively. The surface tension and density values for PMF in Fig. 5 are consistent with values given in Ref. 1 for materials of similar composition. The low surface tension of PMF may arise from the significant concentrations of Na_2O and CaF_2 present. Na_2O and CaF_2 are reported to significantly reduce surface tensions in such oxide systems.

In Fig. 6, with the exception of the highest temperature measurement, the surface tension data for the CBFS material

Table 3 Slag compositions* used in surface tension and density measurements by maximum bubble pressure apparatus, wt-%

SiO_2	Al_2O_3	CaO	MgO	Na_2O	F	Fe	Zn	Cu
PMF								
37.9	6.2	35.5	...	9.3	6.4	...	...	...
CBFS								
25.7	12.9	...	3.1	...	...	32.7	4.6	1.5

*The analysis of PMF (prefused decarburised mould flux) and CBFS (copper blast furnace slag) materials came from the suppliers: Carbox, Brazil and IMI Refiners, UK respectively.

5 Surface tension and density measurements for mould flux PMF as function of temperature

are dependent on measurement depth. The surface tension and density of the slag at this high temperature (1375°C) were 550 mN m⁻¹ and 3389 kg m⁻³ respectively. At temperatures lower than 1375°C, the pressures needed to generate the bubbles at the 20 mm depths were very much greater than at the 10 mm, resulting in the different (high) values of surface tension. Also, at the lower depth at ~1305°C the capillary showed signs of blocking and no bubbles could be produced.

The difference in the behaviour of the PMF and CBFS is stark. The PMF measurements were independent of depth, as expected, whereas the measurements of the CBFS were depth dependent. This anomalous behaviour was in part expected. The PMF material was chosen as benign slag that would be fully liquid and single phase over the temperature range measured, thus enabling the performance of the MBP apparatus to be tested. The scatter in the PMF data are considered representative for such a benign material. In contrast, the CBFS material was known to be extremely reactive and therefore a challenging material to perform measurements on.

The CBFS is a high iron oxide slag and unlikely to be stable in the relatively reducing furnace atmosphere of argon. The iron oxide would partially reduce to metallic iron under such conditions. Also, given the composition of CBFS, it is likely to be reacting with both the crucible and the capillary taking more Al₂O₃ into solution. Further uncertainty over the composition of the CBFS material arises from the fact that it was unclear whether the zinc and copper were in their metallic or oxide form in the slag. In summary, the slag composition is changing with time and may be two or three phase. If the slag was more than one phase, it may stratify with composition as a result of the density differences of the different phases or reaction rates.

6 Surface tension measurements for slag CBFS as function of temperature: density at 1375°C is 3389 kg m⁻³

7 Sectioned alumina crucible plus CBFS sample (half tone of original colour photograph)

The cooled crucible containing the CBFS sample used in the measurement was sectioned and visually inspected for stratification (see Fig. 7). The sample was found to contain small metallic particulates throughout the melt. The colour change of the slag from top to bottom indicated that there was composition stratification. Some evidence of reaction between the slag and the Al₂O₃ crucible, which was indicated by the colour change of the Al₂O₃ close to the Al₂O₃/slag interface, was also observed. Given the confirmation of the variable composition and mixed phases of the CBFS material, it is no surprise that the surface tension measurements were anomalous.

In an attempt to understand the behaviour of the sample, effects of changing slag and gas composition on the liquidus temperature were made using the NPL Mtdata thermodynamic software.¹⁵ The Mtdata software uses internationally validated thermodynamic databases.¹⁵ For an initial slag composition based on the composition of CBFS in Table 3 without zinc and copper, calculations of the liquidus temperature of the slag were made for a range of partial pressures p_{O_2} . The reason for not using the full CBFS composition as the base composition was that the thermodynamic data base did not include all the required data.

Liquidus temperatures were then calculated for a similar slag over the same p_{O_2} range, but with an increasing Al₂O₃ content of 10 and 20% respectively, to mimic alumina pickup from the crucible material. On the assumptions of ideal behaviour for copper system interactions with MgO and Al₂O₃ and zinc system interactions with MgO, a further set of liquidus calculations were made using the composition of CBFS given in Table 3. The results of these calculations are shown in Fig. 8.

The values of p_{O_2} in the argon atmosphere of the MBP furnace will be of the order of 10⁻⁵ and 10⁻⁶. Over this p_{O_2} range, the liquidus temperature was relatively insensitive to increasing the Al₂O₃ content from base to base + 20%. Also, over this p_{O_2} range the addition of copper and zinc has little effect on the liquidus temperature. The largest liquidus temperature change being from 1350 to 1370°C. These liquidus temperatures were greater than all but the highest temperatures for the surface tension measurements in Fig. 6. Therefore, if these liquidus temperatures are correct the slag must have been two phase, both liquid and solid, during all but the highest temperatures for the surface tension measurements. This is likely to explain the disparity between the surface tension measurements at different depths. At the lower depths, probably due to some cementation, the bubble was encountering a different slag composition. This slag would have been two phase. The two phase slag at the

8 Mtdata¹⁵ liquidus calculations for slag compositions based on CBFS at different partial pressures of oxygen

bottom of the crucible would have a higher effective viscosity, making the displacement of the liquid by the bubble more difficult. A higher pressure is then required to form the bubble. The same effect can be observed when the bubble approaches a solid surface, such as the bottom of the crucible. Higher pressure is needed to displace the liquid resulting in a higher apparent surface tension.

CONCLUSIONS

1. The laser flash apparatus (LFA) is capable of measuring effective thermal diffusivities, but more research into the optical properties of the slag is required before definitive values of thermal diffusivity can be obtained by this method.

2. Heat capacity and density data are required for the slag to calculate the thermal diffusivities from the three layer model. There is a paucity of such data for liquid slags. If there is a requirement for thermal diffusivity measurements of slags, then issues relating to the limited data sets available for heat capacity and density of slags will have to be addressed.

3. The effective thermal diffusivities of the mould powder flux ASE and CaO-Al₂O₃-SiO₂ slag ASK are of the order $0.005 \times 10^{-4} \text{ m}^2 \text{ s}^{-1}$.

4. The surface tension and density of prefused decarburised mould flux (PMF) are respectively 320 mN m^{-1} and 2600 kg m^{-3} over the temperature range 1200–1400°C.

5. The maximum bubble pressure (MBP) results, and Mtdata calculations, show that the liquid is homogeneous at temperatures above 1370°C and that the surface tension of copper blast furnace slag (CBFS) is approximately 550 mN m^{-1} .

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