

Feasibility of Measuring Thermal Diffusivity of Liquid Slags

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ABSTRACT:

A study was carried out to assess the feasibility of measuring the thermal diffusivity of liquid slag using a laser flash apparatus (LFA). Measurements are presented for two slag types; a measurement practice is detailed; measurement issues are discussed and recommendations to develop the slag thermal diffusivity measurement technique further are given. Measurements of an “effective” thermal diffusivity of slags was demonstrated but it is not clear how the radiation and conduction components of the heat transfer could be separated. As such, a study of how radiation effects the thermal diffusivity measurement has been recommended. Further, it was also found that for thermal diffusivity measurements using the LFA to be improved data issues with respect to a slags optical properties, density and heat capacity have to be addressed. The slag types measured were one mould flux and one silica rich, lime-alumino-silicate, both of which had effective thermal diffusivities of the order $0.005 \times 10^{-4} \text{ m}^2\text{s}^{-1}$ at temperatures over 1000°C.

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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 thermo-physical property data used in the simulation calculations. There are little data available for these models and the data that are available are often of questionable quality. This has been recognised by the Department of Trade and Industry who have contracted the National Physical Laboratory (NPL) to develop measurement techniques capable of operating in the exacting environment of liquid metal industrial alloy processing. Specifically the NPL has been contracted to assess the feasibility of measuring the thermal diffusivity of liquid slags using the laser flash apparatus. Knowledge of the thermal conductivity/diffusivity of a material enables the engineer to select or tailor materials to optimise process performance with respect to heat transfer.

Slags used in the metal processing industry are, in general but not exclusively, mixtures of metallic oxides. They serve a number of functions, such as a reactive medium and/or a impurity sink in the refining of liquid metals, an insulation layer on the surface of a metal to prevent heat loss, a physical barrier to prevent pickup of unwanted gases from the atmosphere or a lubricating layer to reduce friction in continuous casting.

There are little data available for the thermal properties of liquid slags [1], much of which is contained in the Slag Atlas data compendium [1] published in 1995. Most thermal diffusivity data which were published since this compendium were measured by a Japanese group [2-5] and some notable others [6-8]. Much of the work has been primarily focused on measuring the thermal diffusivity of mould fluxes, employed in the steel industry, using the laser flash technique. There is a large uncertainty associated with the thermal property data available for slags. The scatter is often so large that only order of magnitude values can be assumed.

There are a number of reasons why there are scant data and that the data available are so error prone. The principle reasons are:

- a. the heat transfer in slags is complex, involving phonon (thermal diffusion) and photon (radiation) conduction. It is difficult separate the radiation component of heat conduction from the thermal diffusivity measurements.
- b. Experimentally slags are difficult to handle. They are often reactive with both the containment vessel and atmosphere. This causes problems with reproducibility of measurement as the slag changes composition. Also the energy associated with the change in composition can in some cases affect the thermal measurement.
- c. Slag measurements are invariably high temperature measurements. Most traditional thermal property measurement techniques were designed for ambient or near ambient temperatures. Modifying these methods for high temperature measurements increases the uncertainty in the measurement.
- d. Also at the high temperatures encountered when dealing with liquid slags, 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.

Within 6MMP05 Thermal Effects During Solidification of Metallic Alloys programme, there was a requirement to purchase a Laser Flash Apparatus (LFA) for measuring liquid metals (milestone 6). As part of the development of the apparatus, there was a milestone to establish the feasibility of measuring the thermal diffusivity of liquid slags.

The German company Netzsch won the contract to supply NPL with the LFA. As part of the purchase of the LFA it was agreed that Netzsch would co-operate with NPL, in the development a cell that was capable containing the slag material and had a geometry compatible with thermal diffusivity measurements using an LFA. Any data generated by Netzsch during this development is annotated as such.

2. Experimental

2.1 Thermal Diffusivity Measurement by the LFA

Thermal diffusivity is defined as the rate of heat diffusion per unit time, as given in Eq. 1 [9]:

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

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 ($\text{J kg}^{-1} \text{K}^{-1}$)
- ρ is the density (kg m^{-3})

A typical laser flash thermal diffusivity measurement involves heating the front face of a disc-shaped sample using a high intensity laser and monitoring the temperature rise on the back face. This is a single layer system, a schematic of which is shown in Figure 1 and a typical temperature transient is shown in Figure 2. From the temperature rise the thermal diffusivity can be calculated.

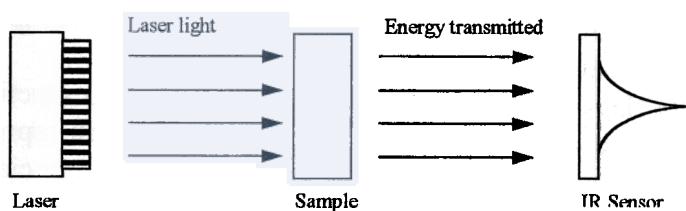


Figure 1 A schematic of the measurement principle for a single layer sample.

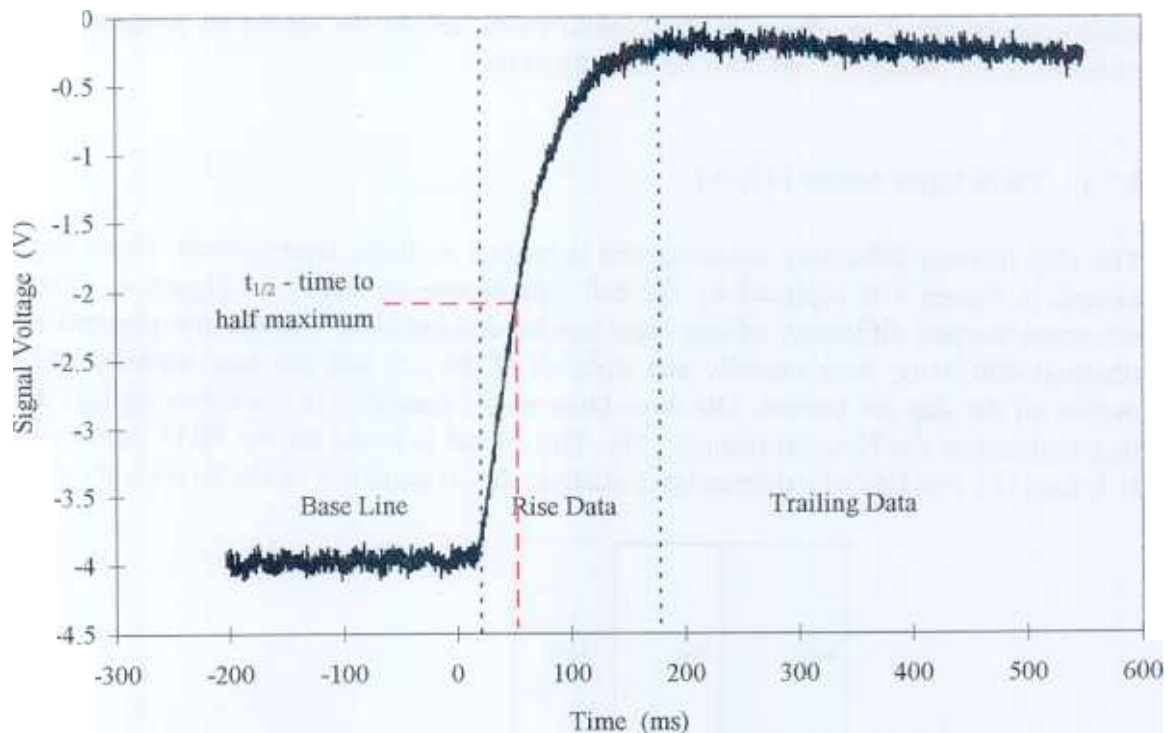


Figure 2. Characteristic thermal diffusivity measurement curve from the Netzsch LFA 427 for a single layer sample.

A furnace is used to heat the sample to the desired temperature. A schematic of the Netzsch LFA thermal diffusivity apparatus as used in this study is given in Figure 3.

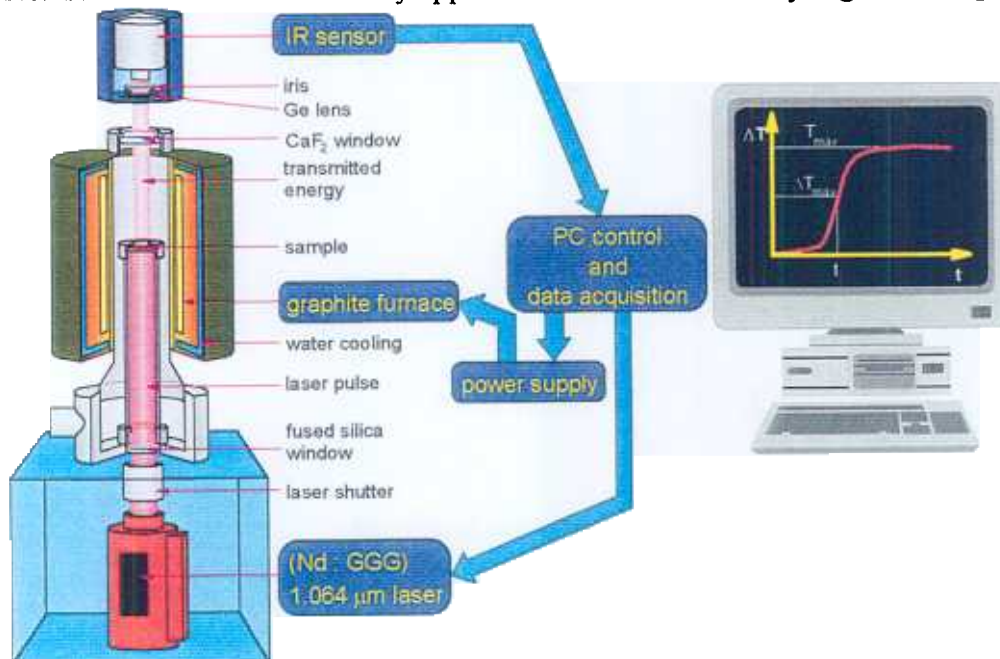


Figure 3. Schematic of the LFA measurement system.

There are numerous models available to calculate the thermal diffusivity from the temperature transient of a single layer sample [10-14]. Making a thermal diffusivity measurement of a slag material is more complex, because the slag is held in a measurement cell. The effects of this measurement cell on the measured temperature transient is not negligible and must be accounted for.

2. 1 Three Layer Model [15,16]

The slag thermal diffusivity measurement is treated as three layer system where the sample in Figure 1 is replaced by the cell plus sample as shown in Figure 4. The unknown thermal diffusivity of one layer can be determined if the thermal properties (thermal diffusivity, heat capacity and density) of the cell and the heat capacity and density of the slag are known. The three layer model described in the following text is that outlined in the Netzsch manual [15]. This model is based on the Ph.D. thesis of H.J. Lee[16]. For Lee's [16] three layer analysis the arrangement of the layers is fixed.

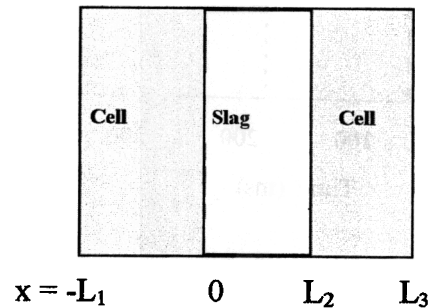


Figure 4. Schematic of a three layer sample.

The Fourier equation [9] is valid for all three layers,

$$(2) \quad \frac{\partial T_1}{\partial t} = a_1 \frac{\partial^2 T_1}{\partial x^2}$$

$$(3) \quad \frac{\partial T_2}{\partial t} = a_2 \frac{\partial^2 T_2}{\partial x^2}$$

$$(4) \quad \frac{\partial T_3}{\partial t} = a_3 \frac{\partial^2 T_3}{\partial x^2}$$

where the subscripts 1 to 3 denote the respective layers and

- a is the thermal diffusivity
- t is time
- T is temperature
- x is the distance through the cell.

The boundary conditions for an exponential pulse form are

$$(5) \quad -\lambda_1 \frac{\partial T_1}{\partial x} = (q_0/t_p^2)t \cdot \exp(-t/t_p)$$

$$(6) \quad \lambda_1 \frac{\partial T_1}{\partial x} = \lambda_2 \frac{\partial T_2}{\partial x}$$

$$(7) \quad T_1 = T_2 \quad x=0$$

$$(8) \quad \lambda_2 \frac{\partial T_2}{\partial x} = \lambda_3 \frac{\partial T_3}{\partial x} \quad x=L_1$$

$$(9) \quad T_2 = T_3 \quad x=L_1$$

$$(10) \quad \lambda_2 \frac{\partial T_2}{\partial x} = 0$$

where

- λ is the thermal conductivity
- L is the length of the layer
- q_0 is the laser energy intensity
- t_p time to maximum laser power of the pulse shot.

The initial conditions are

$$(11) \quad T_1 = T_2 = T_3 = 0 \quad t=0$$

The solution is

$$(12) \quad \frac{T_3}{T_{\max}} = 1 + 2 \sum_{k=1}^{\infty} M \quad t=0$$

where $T_{\max}$ is the maximum temperature at L_3 ,

$$(13) \quad M = \frac{(\omega_1 X_1 + \omega_2 X_2 + \omega_3 X_3 + \omega_4 X_4) Q(\gamma_K, \eta, t)}{\omega_1 X_1 \cos(\omega_1 \gamma_K) + \omega_2 X_2 \cos(\omega_2 \gamma_K) + \omega_3 X_3 \cos(\omega_3 \gamma_K) + \omega_4 X_4 \cos(\omega_4 \gamma_K)}$$

$$(14) \quad Q(\gamma, \eta, t) = \frac{e^{-\frac{\gamma^2 t}{\eta^2}} - e^{-\frac{t}{t_p}} \left[1 + \frac{t}{t_p} \left[1 + \frac{t}{t_p} \left(1 - \frac{\gamma^2 t_p}{\eta^2} \right) \right] \right]}{\left(\frac{\gamma^2 t_p}{\eta^2} - 1 \right)^2}$$

$$\eta_j^2 = L_j^2 / \alpha_j, \quad j=1, 2, 3$$

$$16) \quad H_j = \rho_j C p_j L_j$$

$$H_{i/j} = H_i / H_j$$

$$\eta_{i/j} = \eta_i / \eta_j$$

$$X_1 = H_{1/3} \eta_{3/1} + H_{1/2} \eta_{2/1} + H_{2/3} \eta_{3/2} + 1$$

$$X_2 = H_{1/3} \eta_{3/1} - H_{1/2} \eta_{2/1} + H_{2/3} \eta_{3/2} - 1$$

$$X_3 = H_{1/3} \eta_{3/1} - H_{1/2} \eta_{2/1} + H_{2/3} \eta_{3/2} + 1$$

$$X_4 = H_{1/3} \eta_{3/1} + H_{1/2} \eta_{2/1} - H_{2/3} \eta_{3/2} -$$

$$\omega_1 = \eta_{1/3} + \eta_{2/3} + 1$$

$$\omega_2 = \eta_{1/3} + \eta_{2/3} - 1$$

$$\omega_3 = \eta_{1/3} - \eta_{2/3} +$$

$$\omega_4 = \eta_{1/3} - \eta_{2/3} - 1$$

where

ρ is density

Cp is heat capacity

γ_K is the temporary solution of Eq.1

$$(25) \quad X_1 \sin(\omega_1 \gamma_K) + X_2 \sin(\omega_2 \gamma_K) + X_3 \sin(\omega_3 \gamma_K) + X_4 \sin(\omega_4 \gamma_K)$$

An approximation for the heat loss correction of the three layer mode can be made if [14]

a. mathematically, the layered composite is taken as an homogeneous test piece,

b. the heat loss coefficient of both layers are similar so that the heat loss correction can be carried out for the whole test piece.

2.2 Experimental

As this was a new and very different application for the Netzsch LFA thermal diffusivity apparatus there was little experimental practice to follow prior to the start of this feasibility study. What is outlined in this section is the experimental practice and set-up settled on for the majority of the measurements made and should be viewed as a development in itself. In general, any important issues that were encountered in the development of this practice and set-up will be discussed in Section 3 Instrument Development/Experimental Problems.

The thermal diffusivity measurement and apparatus initiation were made in a similar manner to that outlined in the in-house Procedure for Measurement of Thermal Diffusivity Using the LFA number QPCMMT/B/172 that was developed for liquid metals given in Appendix A. With the exception of how the thermal diffusivity was calculated, outlined Section 2.1.1 The Three Layer Model, the main departures for the existing procedure are outlined below.

2.2.1 The Measurement Cell

The measurement cell comprised of an alumina cap, platinum lid, platinum crucible and an alumina crucible support, as pictured in Figure 5.



Figure 5 Picture of slag cell (lid and crucible), holder and cap.

The alumina cap has two functions. The primary function 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. A schematic showing how the thickness of the liquid slag is defined by the platinum lid and crucible is given in Figure 6.

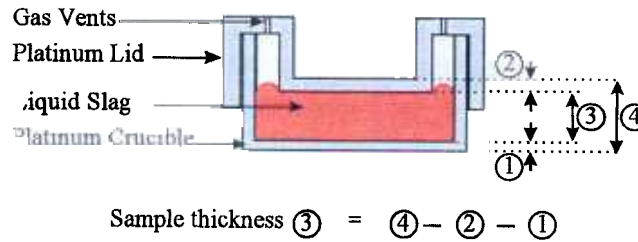


Figure 6. Schematic of platinum cell.

The thickness of the base of the platinum crucible (1), the base of the platinum lid (2) and the distance between the bottom of the base of the crucible and the top of the base of the lid (4) must be measured prior to any melting of the slag. The thickness of the slag (3) can then be calculated. There must be enough liquid slag to fill the gap between the platinum lid and crucible in order for the slag sample thickness to be defined.

The alumina cap also protects the IR detector, blocking the laser light that would otherwise be fired directly onto the IR sensor.

The alumina crucible support, holds and centres the platinum cell in the LFA sample chamber.

2.2.2 Sample Composition

Two sample compositions were chosen to test the measurement apparatus the compositions of which are given in Table 1.

Material	Fe (total)	CaO	SiO ₂	Al ₂ O ₃	S
ASE1 (Decarburised mould flux)	1.06	34.92	38.44	6.72	0.092
	Na ₂ O	F	other	-	-
	12.25	6.66	balance	-	-
Material	CaO	SiO ₂	Al ₂ O ₃	other	-
			15.05	balance	-

Slag compositions in weight %.

The slag compositions were selected for their relative non reactivity and in the case of ASE1 low melting point.

2.2.2.1 ASE

ASE was a continuous casting mould powder supplied by Carbo-ox of Brazil. The material was heated overnight to 900°C in air, on an alumina boat, using a muffle furnace, to decarburise the slag. The composition as given in Table 1 was measured by British Steel Teesside Works after decarburisation.

2.2.2.1 ASK

ASK was prepared in-house from analar grade material supplied by BDH and silica sand from Strathclyde University. The silica sand was washed in HCl prior to fusing with other reactants to remove any FeO. The primary ingredients of ASK were inductively melted in a graphite crucible under argon and cooled to room temperature in air. On cooling the material initially formed a glass, but after a period of time decrepitated. The composition as given in Table 1 was measured by British Steel Teesside Works.

2.2.3 Sample Preparation

There were two primary sample preparation methods.

- Melting the powdered slag in a platinum crucible using platinum resistance furnace, cooling and then machining a slag sample to fit the slag measurement cell.
- Melting the powdered slag in-situ i.e. in the measurement cell, in the LFA.

After much trial and error the in-situ method was adopted. The principle reason for this choice was that it was too difficult to machine a sample of the required dimensions out of the glass.

The in-situ method was not without its problems. The crucible often required repeated filling, melting and milling to ensure that there was enough liquid slag to fill the platinum cell. As a result, it is also a very time consuming method. A schematic of the sample preparation procedure used is given in Figure 7. The schematic also shows estimated time to carry out each preparation step.

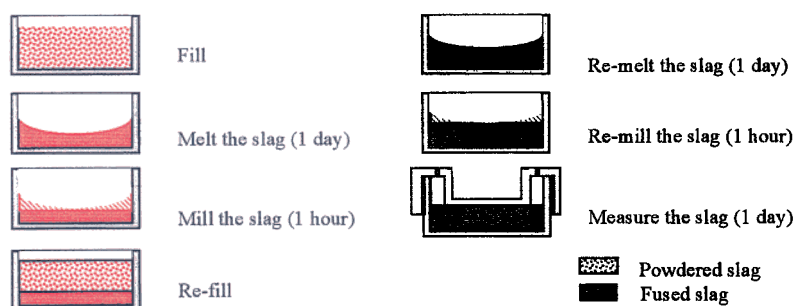


Figure 7. Schematic of the sample preparation procedure.

The milling was required as the slag solidified with a concave surface, as shown in Figure 8. This made refilling and seating of the platinum lid difficult.

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. Also in an attempt to remove bubbles that may form in the liquid, the roughing pump on the furnace would be turned on for 10 seconds prior to cooling.



ASE(2)A Slag Sample (after melting)
19/10/98

Figure 8. Typical picture of a solidified slag after melting

2.2.3.1 Sample Melting Procedure for Sample Preparation

The following procedure was adopted when preparing the sample for measurement. All heating and cooling was carried out in an argon atmosphere.

- Heat from room temperature to 800°C at 12°/minute
- Dwell time at 800°C approximately 10 minutes
- Heat from 800°C to 1500° at 10°/minute
- Hold at 1500°C for 20 minutes
- Turn on roughing pump for approximately 10 seconds
- Hold at 1500°C for a further 10 minutes
- Cool from 1500°C to 800°C at 10°/minute
- Dwell time at 800°C approximately 10 minutes
- Cool from 800°C to room temperature at 12°/minute

2.2.3.2 Sample Melting Procedure for Measurement

All heating and cooling was carried out in an argon atmosphere. In general all thermal diffusivity measurements were made on cooling.

- Heat from room temperature to 800°C at 12°/minute
- Dwell time at 800°C approximately 10 minutes
- Heat from 800°C to maximum temperature required at 10°/minute
- Hold at maximum temperature until temperature stabilises
- Turn on roughing pump for approximately 10 seconds
- If necessary re-stabilise temperature
- Measure thermal diffusivity
- *Cool to required temperature at between 2 and 5°/minute
- *Hold at required temperature until temperature stabilises
- *Measure thermal diffusivity
- For further measurement cool as asterisked above
- If measurement complete shutdown equipment as per

QPCMMT/B/172

2.2.3.3 Calculating the Thermal Diffusivity of the Slag

Much of the Netzsch software associated with the LFA has been developed for single layer sample analysis and not the three layer analysis required for the slag measurement. As such the Netzsch three layer software, while functional, is primitive. None of the automatic functions associated with the single layer software are operative in the three layer model. As a result all three layer calculations require manual data input of the thickness, heat capacity, density and thermal diffusivity for both the platinum crucible (layer 1) and lid (layer 3) for each measurement. For the slag sample (layer 2), inputs of thickness heat capacity and density are needed. The thermo-physical property data used in the three layer model are given in Appendix B.

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 that measured or calculated at room temperature.

3 Instrument Development/Experimental Problems.

3.1 Cell Design

The original cell design for holding the liquid slag was based on the sapphire (the single crystal form of Al_2O_3) cell used in the measurements of liquid metals. The configuration of the sapphire cell was similar to that shown in Figure 6. There were two reasons why sapphire was chosen as the material to hold the liquid metal.

- a. It was transparent to the laser wavelength used in the LFA ($1.064\mu\text{m}$). As a result a relatively simple single layer analysis could be used to calculate the thermal diffusivity.
- b. Sapphire is chemically stable with respect to most industrially used alloys.

Unfortunately sapphire it is not suitable for use with slags as it is readily dissolvable in most oxide slag systems. A number of materials were considered for the cell, but based on their ease of fabrication, limited reactivity with slag and in-house experience of handling them, graphite and platinum were chosen for further investigation.

Both graphite and platinum are opaque to the laser light, as such their heat transport properties could not be ignored. Therefore the three layer analysis outlined previously had to be used to calculate the thermal diffusivity values from the temperature transient.

3.1 Graphite Cell Design

For reasons of cost and ease of manufacture it was decided the initial trial work would be carried out using graphite as the cell material. Cells similar to that given in Figure 6 were machined and used with both ASE and ASK slags. Unfortunately it was found that the slags were reacting with the graphite. The main evidence for this reaction came from observations of slag melted in a platinum crucible under similar conditions and slag melted in the graphite cell crucible. The slags melted in the platinum crucible

solidified to form transparent glasses whereas the graphite crucible melted slags formed dark opaque glasses. It is believed this opaque structure was a result of some carbide formation and that the transparent glasses were a more representative composition of the starting slag. Further evidence in support of graphite reaction with the slag was found on the crucible walls. On occasions when it was possible to remove the slag from the crucible there was a rim at the slag-crucible wall-gas interface.

On some occasions the graphite crucibles cracked on solidification. It is likely this was a result of the different expansion rates of graphite and slag.

Due to the reaction between the slag and the graphite it was decided not to pursue the development of a measurement cell with this material. Although no credible thermal diffusivity measurements were made using the graphite cell useful information was obtained on

- a. slag melting practice,
- b. specific cell geometry,
- c. alignment of the cell in the LFA
- d. and the weight required to ensure the cell lid did not float on the slag. Thereby ensuring the slag thickness would be defined by (3) in Figure 6.

This information was incorporated into the cell design and measurement practice.

3.1.1 Platinum Cell Design

Two platinum cells were made up to the configuration shown in Figure 6 and pictured in Figure 5. The cost of a cell, including alumina parts, was approximately £3000, but successful measurements were made using these cells. Unlike the graphite cell the platinum did not noticeably react with either the ASE or ASK slag. A number of difficulties were experienced using these cells. These had to be overcome before the thermal diffusivity measurements could be made. The details of said problems and solutions to the problems are outlined below.

3.1.1.1 Slag Creep

On the first few attempts at measuring the thermal diffusivity of the ASK slag it was found that the material had crept out of the crucible, through the gas vent holes. The creeping slag covered both the top of the platinum lid and alumina cap. A photograph showing the result of a slag creeping is given in Figure 9.

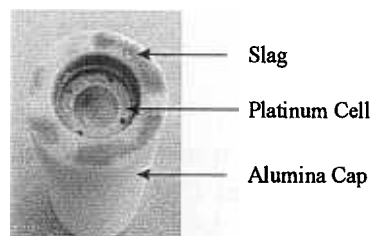


Figure 9. Typical picture of a slag that has shown creep behaviour.

Creep is an interfacial phenomena that can cause liquid material to flow up walls. One theory suggests the mechanism is similar to the classical theory of Thomson's "wine tears" [17]. It proposes that one constituent (A) of the slag is absorbed preferentially on the walls of the crucible and results in an increase in the surface tension (γ) of the remaining melt. That is, the adsorbing species when in solution in the melt lowers the melt's surface tension.

Since the slag is in the form of a thin film, it takes time for A to diffuse from the bulk and replace the absorbed species (i.e. the rate absorption of A onto the metal surface is greater than the diffusion of A from the bulk to the area depleted of A), a surface tension gradient is created and Marangoni flow occurs in the direction shown in Figure 10.

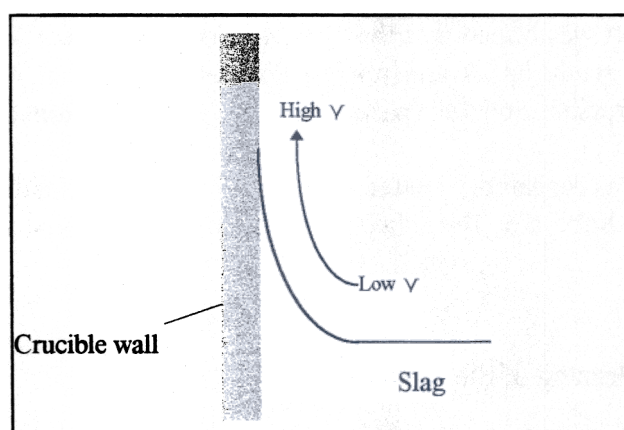


Figure 10. Schematic of creep mechanism.

This drives the liquid further up the crucible where A is again absorbed onto the clean metal surface. This further absorption causes an increase in surface tension of the slag tip and the slag climbs further up the crucible and so on...

Whilst the behaviour is not fully understood it is believed that it occurs more readily on virgin/unused material. With this in mind it was decided to persevere with the platinum cells to see if the creep behaviour would stop. After three attempts to melt and measure the ASK slag in both sample cells the slags stopped creeping out.

3.1.1.2 Cleaning of the Platinum Crucible

It was envisaged that any slag that remained in the platinum crucible would be removed by dissolving in a 70/30 sodium carbonate, sodium tetraborate blend, as used in atomic adsorption techniques. This would involve fusing the slag in the crucible with the borate blend. The resultant fused glass would then be dissolved in nitric acid. Unfortunately the platinum lid could not be removed from the cell. This made it extremely difficult to fuse the borate mixture and the slag as the only entry points for the borate mixture were through the four gas vents on the top of the lid. This approach was abandoned in favour of steeping the platinum cell containing the slag in concentrated hydrofluoric acid (HF) overnight. This procedure degraded the slag to a powder, allowing the lid to be removed.

3. 1.3 Integrity of the Measured Sample

It is good practice in any measurement procedure to inspect the sample after measurement. Unfortunately this was not possible during this study as the platinum lid could not be removed without destroying the sample. The sample would “glue” both the lid and the crucible together. During the measurement the liquid the slag would flow (creep) into the gap between the inside of the lid wall and the outside of the crucible wall. On solidification of the liquid slag, this material locked both cell parts together. A number of methods were employed to remove the lid, some of which are outlined below.

a. Mechanical

1. The cell was suspended by its lid and heated to a temperature of approximately 1500°C. It was hoped that the crucible would fall under gravity.

2. Clamps were made up of appropriate dimensions, so that they could grip both the cell lid and crucible and be twisted (in opposite directions), to break the “glue”.

3. The sample was repeatedly heated and cooled (to approximately 150°C then RT respectively) in the hope that the differential expansion of the slag and platinum would break the “glue”

b. Chemical

As outlined in Cleaning of the Platinum Crucible, section 3.1.1.2.

Only the steeping in HF proved successful. Although it is believed that a new cell design, where the gap between the inside of the lid wall and the outside of the crucible wall is bigger and the cell had flats machined on its side, would be more easily mechanically separated.

The fact that the sample was destroyed and therefore could not be inspected after measurement increases the uncertainty associated with the thermal diffusivity measurement, as there is no way of telling whether the sample was fully dense or contained gas bubbles.

4. Results

The results of measurements made on samples ASE and ASK are shown in Figures 11 and 12. Unless otherwise stated all sample thickness were of the order of 1.5 mm except the Tohoku measurements which were made on samples approximately 0.2 mm thick. Also none of the measurements presented in Figures 11 and 12 have been corrected for a radiation contribution and are therefore referred to as effective thermal diffusivity (a_e).

In Figure 11, NPL denotes measurements made at the National Physical Laboratory, NPL/Netzsch denotes measurements made at the Netzsch Works in Selb, Germany by a NPL staff member, Netzsch denotes measurements made at the Netzsch Works in Selb, Germany by Netzsch staff and Tohoku denotes measurements made at Tohoku University in Sendai Japan by Professor Waseda. The numbers signify the chronology of the measurement.

On inspection of Figure 11 it can be seen that with the exception of the Tohoku2 results all the laboratory measurements are, within the scatter in the data, in good agreement. Also the effective thermal diffusivity shows an increase with increasing temperature.

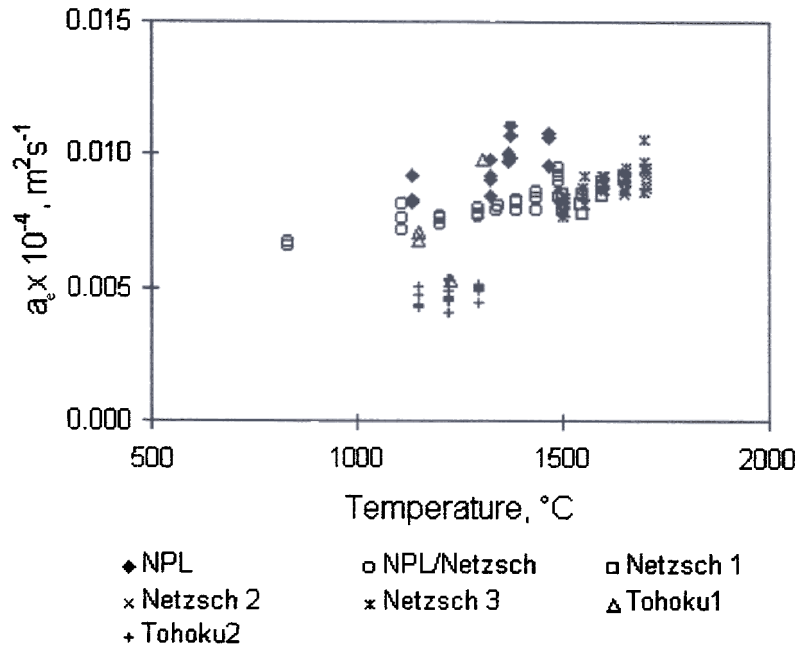


Figure 11. Effective thermal diffusivity (a_e) measurements made by a number of laboratories on slag ASE.

The ASK measurements shown in Figure 12, were measured at two different thickness' and on a heating and cooling cycle. In general the data shows a similar trend

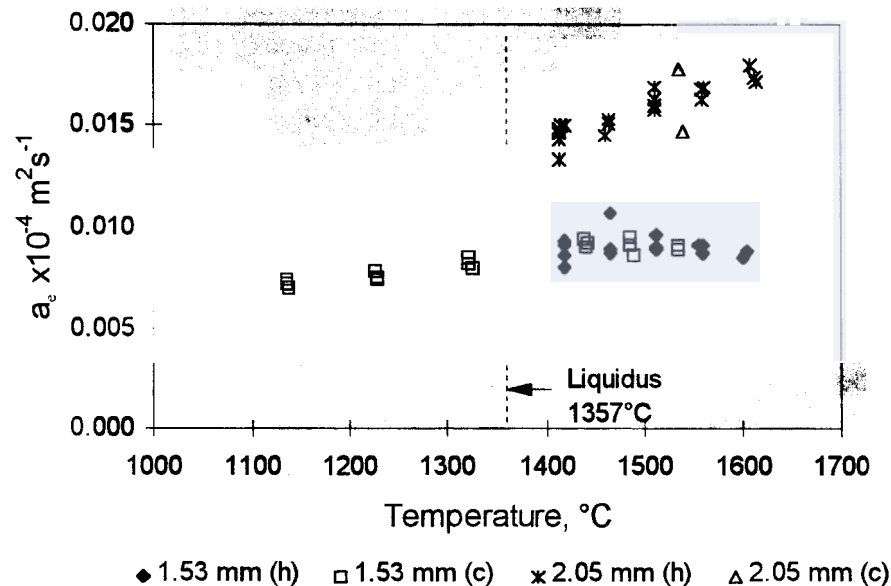


Figure 12. Effective thermal diffusivity (a_e) measurements of slag ASK at two different sample thickness.

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 thicknesses of the sample was obtained by inserting a platinum ring between the platinum cell lid and crucible.

5 Discussion

It is clear from Figures 11 and 12 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 semi-transparent to the infra-red wavelengths and the laser light wavelength of 1.064 μm used in the LFA [1,4,8]. As a consequence of this the heat transfer through the sample has both a phonon (thermal diffusion) and photon (radiation) contribution as shown in Eq. (26).

$$(26) \quad a_e = a + a_{rad}$$

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 semi-transparent), the absorption/emission coefficient of the slag and the effective penetration depth of the radiation [18]. It is a difficult parameter to calculate because,

- a. there are very little absorption/emission data on slags and the data that does exist has usually been measured at ambient temperature [1].
- b. The power of laser pulse is never measured. This makes calculation of the surface temperature differences difficult.

The matter is further complicated by the fact 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 of poor quality and as a result may have to be modelled. Further increasing the uncertainty the measured thermal diffusivity.

Most approaches to the radiation problem have involved modelling the data on a grey body [5,8] or transparent body [4] assumption. According to authors [4] the grey body assumption is only required for slags containing FeO or TiO₂.

If radiation is a problem in our measured data, then the measured (effective) thermal diffusivities would be expected to increase with increasing temperature and increasing size [1,4,5,8]. Inspection of both Figures 11 and 12, ignoring the Tohoku data, are consistent with such trends. It is therefore likely that the measured thermal diffusivities do have a radiation component associated with them.

The Tohoku data is difficult to explain. The group that measured these data probably have more experience with the problems of handling and measuring slag materials than any other laboratory in the world. Therefore their measurements cannot be easily disregarded. They use a thinner sample than the samples measured by both NPL and Netzsch. As such, their values should be lower. This would indicate that the Tohoku2 measurements are consistent with the NPL and Netzsch measurements. There is no

obvious explanation for the difference in the data, it is possible, though unlikely, that one of the Tohoku samples composition had changed. Another possibility is that this is a true representation of the scatter in such a measurement but again this is unlikely given the agreement of the NPL and Netzsch results and other literature data [2-8].

As mentioned previously Waseda et al [4] stated that a transparent body approximation to radiation could be used for slags that contained no FeO or TiO₂. They also developed [4] a set of equations that could correct for the effect of radiation 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 radiation would have on a thermal diffusivity measurement using a LFA by a finite difference.

For an effective thermal diffusivity as defined in Eq. 27,

$$\alpha_e = c \cdot a$$

where c is the effect of radiation, they came up with the following Eq.'s for c , for 0.1, 0.2 and 0.3 mm thick samples respectively.

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

$$c = 6.986 \times 10^{-7} \cdot T^2 - 1.466 \times 10^{-3} \cdot T + 1.883$$

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

These equations have been derived for sample sizes much smaller than the ones measured by NPL and Netzsch. In order to utilise this approach the constants in Eq.'s 28 to 30 were linearly regressed with respect to sample thickness to obtain Eq.'s 31 and 32 for thickness' 1.53 mm and 2.05 mm respectively.

$$c = 7.372 \times 10^{-6} \cdot T^2 - 1.628 \times 10^{-2} \cdot T + 10.968$$

$$c = 9.967 \times 10^{-6} \cdot T^2 - 2.206 \times 10^{-2} \cdot T + 14.510$$

This is not a rigorous approach as it is always questionable 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 values obtained should be independent of sample thickness [18]. Also for highly structured materials (crystalline solids) the phonon contribution would be expected to show a negative dependency of thermal diffusivity with increasing temperature [18]. 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 [18].

To assess the validity of Eq.'s 31 and 32, the data shown in Figure 12 for the two different sample thickness would be recalculated using Eq.'s 27, 31 and 32. The results are shown in Figure 13 and compared with values obtained from the Slags Atlas [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 to be expected that the diffusivity would decrease with increasing temperature.

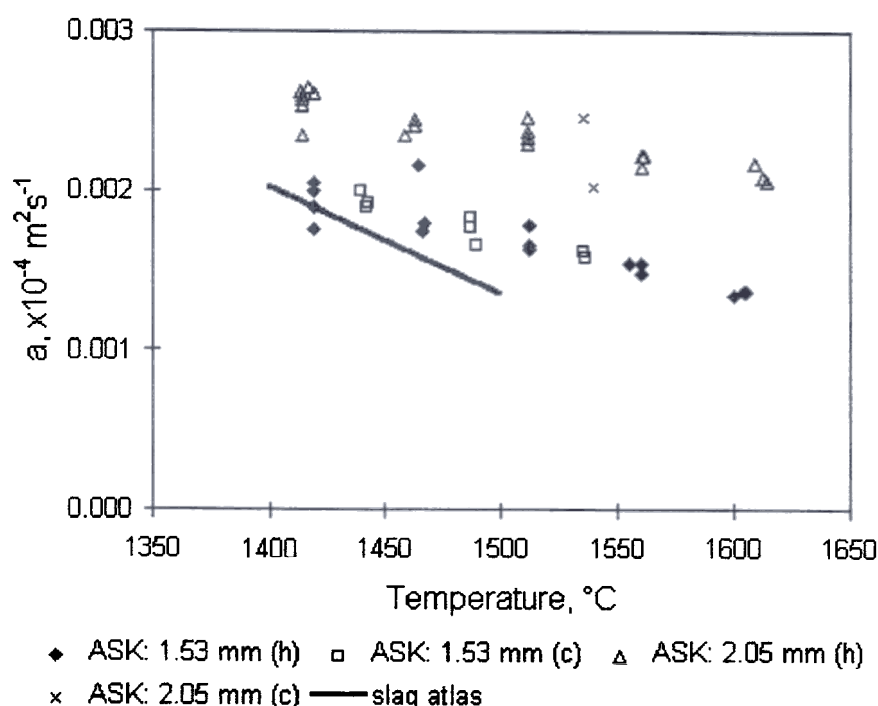


Figure 13. ASK thermal diffusivity measurements corrected for radiation contribution. The slag atlas [1] composition in weight % is 25 % CaO, 15% Al₂O₃, and 60% SiO₂.

On inspection of Figure 13, it can be seen that while the data still persist as two lines although they are closer than in Figure 12 c.f. approximately 100% and 25 % difference in diffusivities from the 1.53 mm to 2.06mm slag samples in Figure 12 and Figure 13 respectively. 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, it can be seen, particularly for the smaller sample, that the agreement is reasonable.

From the data shown in Figure 13, it can be concluded that corrections for radiation can be made, but if the capability of LFA technique for measuring thermal diffusivities of slags is to be fully realised, then the effects of radiation for its particular sample geometry have to be determined. Also the radiation correction used was for a transparent body approximation. This would not be appropriate for semi-transparent 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 either do not exist or are of poor quality or are available at unsuitable temperatures.

It should be noted that liquid slag measurements in general, and thermal diffusivity is no different, are expensive. It requires specialised equipment and experience to handle such materials which are not routinely found in laboratories round the world. The cost of the thermal diffusivity measurement cell, which is unlikely to survive for more than a few measurements, is of the order of £3k. Added to that the preparation and analysis time, approximately seven days, these measurements are very expensive. There is only limited scope for reducing this expense, namely reducing the time for preparation by developing new procedures.

6. Conclusions

- a. The 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.
- b. 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 (in particular) density of slags will have to be addressed.
- c. The measurements are time consuming and expensive and require further development to lower the overall costs.
- d. The effective thermal diffusivities of the mould power ASE and CaO-Al₂O₃-SiO₂ slag ASK are of the order $0.005 \times 10^{-4} \text{ m}^2 \text{ s}^{-1}$.
- e. The user interface of the Netzsch software used to measure and calculate the slag thermal diffusivity from the three layer model needs to be made more user friendly.

7 Recommendations

Data for the optical properties of slags/oxides at high temperatures will have to be measured to fully realise the LFA slag thermal diffusivity measurement capability. Without such data it will be difficult to develop the method. Therefore it is recommended that a measurement programme be embarked upon to address this

problem. The data gained from such a measurement programme will have wider application than just thermal diffusivity measurements. It would prove very useful in a number of industrial process modelling problem such investment casting, BOF steelmaking, continuous casting of steels and numerous other high temperature processes.

It is also recommended that a study into the effects of radiation heat transfer on the thermal diffusivity measurement be undertaken. This study should be carried out with a view to obtaining a relationship that will enable separation of the radiation term from the thermal diffusivity.

Acknowledgements

This work was carried out as part of the Materials Measurement Programme of the Department of Trade and Industry. Thanks is due to Prof. K. C. Mills and Netzsch in Selb, Germany, for helpful discussion and advice during the course of this study.

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Appendix A

An uncontrolled copy of the Procedure for Measurement of Thermal Diffusivity Using the LFA, procedure number QPCMMT/B/172

1

NATIONAL PHYSICAL LABORATORY
CENTRE FOR MATERIALS MEASUREMENT
AND TECHNOLOGY

Procedure
QPCMMT/B/172

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1.

NATIONAL PHYSICAL LABORATORY
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12 **NETZSCH THERMOCOUPLE CALIBRATION**

13 **ERROR ANALYSIS REPORT FOR THE NETZSCH LFA 427 APPARATUS**

14 **APPENDIX**

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1 INTRODUCTION

- 1.1 This document describes the procedures used for the measurement of thermal diffusivity of solid and liquid samples, using the LFA 427 Laser Flash System with the High Temperature Physical Properties Group in the Centre for Materials Measurement and Technology (CMMT)
- 1.2 The aim of this document is to provide the reader with the information required to make basic measurements with the apparatus described.

2 DEFINITION OF THERMAL DIFFUSIVITY

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

$$a = \lambda / (C_p \cdot \rho) \quad \text{m}^2 \text{s}^{-1}$$

λ = Thermal conductivity $\text{Wm}^{-1}\text{K}^{-1}$

C_p = Specific heat capacity $\text{Jkg}^{-1}\text{K}^{-1}$

ρ = Density kg.m^{-3}

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3 SAFETY

The LFA 427 apparatus can only be operated as a CLASS 1 laser if the **Laser Unit** door is closed and all interlocks operating. If the **Laser Unit** door is open and the power to the laser is on then the laser is CLASS 4. To minimise the risk of exposure to class 4 radiation, as even exposure to diffuse radiation can cause permanent eye damage, the **Laser Power Unit** MUST be switched off prior to opening the **Laser Unit** door.

- 3.1 To ensure that the apparatus is always operated under CLASS 1 conditions interlocks have been installed such that:
 - 3.1 If the **laser unit** door is open the power to the laser is shut off.
 - 3.1.2 If the furnace chamber is opened the laser shutter is closed (as indicated by the LED on the LFA controller) blocking any possible viewing of the laser light and should any attempt be made to make a measurement the computer locks up.
 - 3.1.3 If the IR sensor head is removed the laser shutter is closed (as indicated by the LED on the LFA controller) blocking any possible viewing of the laser light and should any attempt be made to make a measurement the computer locks up.
- 3.2 To ensure the interlocks are working properly the following tests must be carried out at least every three months prior to making a measurement:
 - 3.2.1 **Laser Unit** door interlock
 - 3.2.1.1 Start up the equipment as described in section 6 up to the stage at which you are ready to start taking measurements.
 - 3.2.1.2 Obtain the spare **Laser Unit** door lock key (no.3) from keybox in room 40, building 13

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- 3.2.1.3 Unlock the **Laser Unit** door but do **NOT** open the door.
- 3.2.1.4 Disengage the lock by turning the doorhandle but do **NOT** open the door.
- 3.2.1.5 The **Laser Power Unit** interlock should have activated, the power supply should be disabled and the illuminated switches on the **Laser Power Unit** should read **Shutter Closed** and **Interlock Reset**. The display on the Laser Controller should read **IL** and it will be flashing.
- 3.2.1.6 If not, shut down equipment, lock door, return keys and contact the NETSZCH engineer.
- 3.2.1.7 If the **Laser Power Unit** interlock is operating correctly press the interlock reset switch on the **Laser Power Unit** and wait for power supply to reset, then lock the door, return the keys and move onto section 3.2.2.
- 3.2.2 Furnace chamber open interlock.
 - 3.2.2.1 Partially raise the furnace (approximately 15 mm from the bottom stop position) such that the furnace seal obscures the bottom edge of the furnace chamber.
 - 3.2.2.2 Attempt to make a thermal diffusivity measurement of the sample by firing the laser.
 - 3.2.2.3 If the LFA is operating correctly and the interlock is working then the system will lock up at the point of laser firing screen (Tracking red line will appear and a blue dialogue box with "wait for shot data" will also appear) and the LED on the LFA controller should indicate the shutter is closed. This will reset itself after a period of time (approximately 2 min), the furnace can then be closed and the operator can move onto section 3.2.3.
 - 3.2.2.4 If the interlock is malfunctioning and LFA fires the laser then shut down the

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equipment and contact the NETSZCH engineer.

IR sensor interlock.

- 3.2.3.1 After the measurement has timed out the first menu screen will appear again
- 3.2.3.2 Seat the IR sensor in the non-measuring position.
- 3.2.3.3 Place a black rubber cover over the CaF_2 lens. (*Cap is marked LFA*)
- 3.2.3.4 Attempt to make a thermal diffusivity measurement of the sample by firing the laser.
- 3.2.3.5 If the LFA is operating correctly and the interlock is working then the system will lock up at the point of laser firing screen (Tracking red line will appear and a blue dialogue box with "wait for shot data" will also appear) and the LED on the LFA controller should indicate the shutter is closed. This will reset itself after a period of time (approximately 2 min), the IR sensor can then be placed in it's measuring position and the operator can then prepare to use the LFA apparatus.
- 3.2.3.6 If the interlock is malfunctioning and LFA fires the laser, then shut down the equipment and contact the NETSZCH engineer.

4 STAFF REQUIREMENTS

- 4. The apparatus is designed to be operated as simply as possible. The minimum staff requirement is:

Assistant Research Scientist trained to use the LFA to make thermal diffusivity measurements by B Monaghan or Netzsch and has seen the NPL laser safety video on the

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safe use of lasers.

Qualified Staff

Brian J. Monaghan (BJM)

Justin G. J. Neale (JGN)

5 EQUIPMENT LIST

5.1 The equipment is situated in building 13 room 28

Vacuum pump	(Pfeiffer UNO/DUO 004 B)
IR detector	(Indium Antimonide)
Furnace	(Graphite)
Laser	(Neodinium: gallium-gadolinium-garnet)
Electronic controller	(Netzsch LFA 427 controller)
Laser controller	
Laser power unit	
Main power/measuring unit	
Computer	(HP Vectra Pentium)
Monitor	
Printer	

6 MACHINE INITIALISATION

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6 MACHINE INITIALISATION

*****The door on the front of the Laser Unit must NOT be opened when there is power to the Laser Power Unit see section 3 on SAFETY*****

Remove yellow cap from top of IR. detector (See Figure 2 No. 1)

Insert insulated funnel into top of IR. detector

Pour in approximately 20ml of liquid nitrogen

Remove funnel and replace with yellow cap (See Figure 2 No. 1)

Turn on Gas at cylinder (*Outlet pressure should be 10 psi*)

Open valves A & B on furnace control unit fully (See Figure 1 No. 6)

Open inlet and outlet water valves

Close water bypass until inlet water pressure is approximately 2.5 bar

Turn on Plug 1 laser power unit

Turn on Plug 2 laser unit electronics

Turn on 16 Amp feed (*hoist and computer*)

Turn on 32 Amp feed (*furnace and forepump*)

Turn on red power switch on laser power unit (See Figure 1 No. 1)

Insert the keys into the laser power unit (See Figure 1 No. 2)

Turn on key switch on laser power unit (*Ensure this is done at least 10mins before taking a measurement*)

Turn on power switch on front of furnace control unit (See Figure 1 No. 5))

Turn on LFA 427 controller (See Figure 1)

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Turn the control switch to dynamic on the **furnace control unit** (See Figure 1 No. 7)

Open valves E & F on the **furnace** (See Figure 2 No. 4)

Open valve G on the **furnace control unit** until **4 LED's** are illuminated

(See Figure 1 No. 8)

Turn the current limiter on the **furnace control unit** all the way to the left

(See Figure 1 No. 12)

Turn on Monitor

Turn on Printer

Turn on Computer (*HP Vectra*)

Set the laser pulse width and voltage desired on the **laser controller**

(Default values are 400v and 0.8ms)

Replace yellow cap on IR. detector with insulated funnel (See Figure 2 No. 1)

Pour in approximately another **450ml** of liquid nitrogen or until dewer is full

Replace funnel with yellow cap (See Figure 2 No. 1)

Slowly open valve C on the **furnace** (See Figure 2 No. 3)

Very slowly open valve D on the **laser unit** (*Extreme care must be taken when opening this valve as any sample can be dislodged by the gas flow. Do not attempt this unless you have been shown*) (See Figure 4)

Once the **laser unit** pressure gauge reads above 0 bar close valve D (See Figure 4) Open the furnace seal (See Figure 2 No. 5)

Hold the laser unit safety switch in and raise the furnace using the up button (See Figure 4)

Close valve C on the **furnace** (See Figure 2 No. 3)

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Move the furnace out of the way to the left until it hits its stop

Follow the procedure on **loading a sample**

Move the furnace back over the carrier tube

Hold the safety button in and lower the furnace with the down button (See Figure 4)

Tighten the furnace seal (See Figure 2 No. 5)

Turn on the fore pump (See Figure 1 No. 10)

Fully open the vacuum valve

When the pressure indicated on the **furnace control unit** is below **1mbar** turn on the turbo molecular pump (See Figure 1 No. 11& 9)

Close the vacuum valve after the indicated pressure is below **5E⁻⁰⁵ mbar**

Turn off the turbo molecular pump (See Figure 1 No. 9)

Slowly open valve C on the **furnace** (See Figure 2 No.3)

Slowly open valve D on the **laser unit** (See Figure 4)

Allow the pressure to reach above **0 bar** on the **laser unit** pressure gauge
(See Figure 4)

Close valves C & D (See Figure 2 &4)

Fully open the vacuum valve

When the pressure indicated on the **furnace control unit** is below **1mbar** turn on the turbo molecular pump (See Figure 1 No. 11& 9)

Close the vacuum valve after the indicated pressure is below **5E⁻⁰⁵ mbar**

Turn off the turbo molecular pump (See Figure 1 No. 9)

Slowly open valve C on the **furnace** (See Figure 2 No.3)

Slowly open valve D on the **laser unit** (See Figure 4)

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Allow the pressure to reach above 0 bar on the laser unit pressure gauge

(See Figure 4)

Close valves C & D (See Figure 2 & 4)

Fully open the vacuum valve

When the pressure indicated on the furnace control unit is below 1mbar turn on the turbo molecular pump (See Figure 1 No. 11 & 9)

Close the vacuum valve after the indicated pressure is below 5×10^{-5} mbar

Turn off the turbo molecular pump (See Figure 1 No. 9)

Turn off fore pump (See Figure 1 No. 10)

Restrict valves A & B on the furnace control unit so they are only open 2 turns each (See Figure 1 No. 6)

Slowly open valve C on the furnace (See Figure 2 No. 3)

Very slowly open valve D on the laser unit (See Figure 4)

When the pressure indicated on the laser unit gauge reaches just above 0 bar open the furnace seal one half turn (See Figure 2 & 4)

Adjust valves A & B on the furnace control unit until each reads a flow of 75ml (See Figure 1 No. 6)

7 SAMPLE PREPARATION

7.1 Dimensions :

- 7.1.1 Solid samples Diameter $12.5 \text{ mm} \pm 0.05 \text{ mm}$
Preferred length $> 1 \text{ mm}$ but $< 3 \text{ mm}$

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Liquid Samples Diameter $\approx$ 10.8mm

Length $\approx$ 1.45 - 1.48 mm dependant upon expansion

7.2 Coatings

Solid Metallic samples (e.g. copper)

Clean the sample of dirt/grease/oxide (dilute nitric acid used for copper samples)

Rinse sample thoroughly in water

Allow to dry in air

Coat the sample evenly on all surfaces with a graphite spray

Allow enough time before measurement for coating to harden

Thin liquid Metallic samples (e.g. copper)

Clean the sample of dirt/grease/oxide (nitric acid used for copper samples)

Rinse sample thoroughly in water

Allow to dry in air

For sample to seat well in crucible the edges on one face must be filed in

(Note sample will have to be re-weighed) (See Figure 5)

Coat the sample evenly on all surfaces with a graphite spray

Allow coating to harden

Non Metallic (e.g. Graphite)

Ensure sample is clean and that it exhibits good plane parallelism

7.3 Sandblasting

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7.3.1 Substances which may react with carbon (e.g. Iron and Steel)

clean the sample of dirt/grease/oxide

Abrade the sample using a small sandblaster to aid absorption by laser light

Clean any dust from the sample using ethanol

Allow to dry in air

8 MAKING A MEASUREMENT USING THE LFA SOFTWARE

*****The door on the front of the Laser Unit must NOT be opened when there is power to the Laser Power Unit see section 3 on SAFETY*****

Areas in bold are either prompts on screen or indicate the command/button to be pressed

Areas in italics are notes

8.1 At the MsDos start-up menu

select 1: Standard for a windows environment (*not suitable for running LFA 427 software*)

select 2: **LFA427** to enter laser directory (*This ensures optimum memory configuration*)

At laser prompt (C:/Laser>)

Type : **LFA** to enter laser flash measurement software

LFA_DEMO to enter the demonstration software (*Note only the computer must be on to use this software*)

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LFA_RESTART to restart the laser flash measurement software after an aborted measurement

8.1.1 After LFA has been selected

Prompt : Your Data (4 lines available)

Type in NPL address (*Default is NPL, CMMT, Teddington, London, TW11
OLW*)

Prompt : Labor (1 line available)

Enter the company name of the operator (*Default NPL*)

Prompt : Operator

Enter the initials of the person making the measurement

Prompt : Customer (1 line available)

Enter customer (*NPL for internal work*)

Prompt : Run Nr (1 line available)

Enter data in following format

Samples database code/Sample No./Run Letter/Date

Example : For copper sample CGQ 3 run A on 3/10/97 **CGQ3A3-10-97**

After this last entry the first menu screen will load (*Sample input screen*)

Prompt : Material (1 line available)

Enter the sample material e.g. Copper

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Prompt : Sample

(1 line available)

Enter the sample code e.g. CGQ3A

Prompt : Sample Length

Enter length in cm

Prompt : Diameter

Enter Diameter in cm (*Default 1.25cm*)

Prompt : Coating

(1 line available)

Enter coating if any e.g. Graphite

Prompt : Atmosphere

Press Cr to activate Menu (*Default Ar*)

Prompt : Sensor

Press Cr to activate Menu (*Default IR*)

Prompt : Gas Flow

Enter the gas flow through the sample chamber in ml. (*Combined flow through pair of flow metres on furnace control unit*)

Prompt : Emergency Reset Temperature

Enter the maximum sample temperature you are prepared to let the furnace reach (*The furnace and software will turn off at this temperature*)

8.2 Entering A Temperature Program (*Not necessary for room temp measurements*)

There are two methods of entering a temperature program

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- : Enter the temperature program on screen (*Only 19 lines available*)
- 2 : Enter the temperature program on a separate table first (*hundreds of lines available*)

8.2.1 Method 1

Move the cursor over to the first column on the temperature table

Enter the first temperature at which you want to take a measurement (*Do not include room temperature*)

Use the down cursor to select heating rate values in the second column
(*10K/min recommended*)

Use the down key to enter the next temperature and so on (*The number of shots taken at these temperatures is defaulted to 3*)

8.2.2 Method 2

Press **Alt** to activate the drag down menus

Press **H** to pull down the **H**ardware menu

Press **I** to bring up the temperature program table

Enter the values as in **Method 1** (*but in this table it is possible to change the number of shots to be taken at a particular temperature*)

To save this table for future reference press **F3** and give the table a name

To enter this table into your measurement press **F4**

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(Note : If values are entered using method 1 they will appear on the table when brought up by method 2)

LASER HARDWARE SETTINGS

Press **Alt** to activate drag down menus

Press **H** to pull down the **H**ardware menu

Press **P** to select Laser **P**arameters

Press **enter** to edit the Laser Pulse Width *(Ensure the same value is set on the laser controller)*

After editing value press **Cr**

Press the **escape** key twice to return to the main screen

2nd Input Screen

After all values have been entered press **F2** to enter the 2nd input screen

(If this screen will not appear then some information has been missed out)

Prompt : Duration

Enter an approximate value for the duration of the data acquisition. Aim to get approximately 12 half max. *(Default 0.3000s)*

Prompt : Sampl. factor

Press **Cr** to activate Menu *(Default 0)* *(By selecting a value for the sampling*

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factor above 0 the software will automatically write the raw data to an ASCII file. The higher the sampling factor the greater the number of points written to the ASCII file. See LFA manuals for details)

Prompt : Laser Voltage

Enter the desired laser voltage and ensure that this agrees with the value shown on the laser controller

Prompt : Amplification

Press enter to bring up menu

Select the amplification which will give you the minimum of noise and retaining a signal of approximately 2V or greater. *(Default 100)*

Prompt : Preamplifier

Press Cr to select ON or OFF *(Default ON)*

Prompt : Analogfilter

Press Cr to activate menus *(Default 0.01)*

Prompt : Iris Shutter

Press enter to bring up menu

Select a larger value to open the iris further and vice versa. *(Default 7)*

Prompt : Max base drift

This sets the maximum voltage the base line may drift by for a shot to still be taken *(Default 1.0V)*

Prompt : Model

Select the type of model to be applied to the data *(Default Combined Iteration)*

Prompt : Digital filter

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(Default none)

Prompt : Quality control

(Default no)

Prompt : Base line corr

(Default slope)

Prompt : Pause between shots (min)

Sets the minimum time there will be between consecutive shots *(Default 0.0)*

Prompt : Conductivity calculus

By entering values for Cp and density for the sample in question a calculation of thermal conductivity can be made. *(Default no)*

Prompt : Graphics

Select printer if you wish the graphs of each shot to be printed with the report.

(Default none)

Prompt : Results

Select printer if you wish the results to be printed for each shot

(Default printer)

Prompt : Data

Select printer if you wish to have all data printed for each shot

(Default none)

Prompt : Graph.file

Select the directory where you wish graphical files to be stored

(Default none)

Prompt : Resultfile

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Select the directory in which you want result files to be stored

(Default none)

Prompt : Database

Select the directory in which you want all database files to be stored

(Default C:\Laser\)

8.5 Making a measurement (*There are three ways to make a measurement*)

Manual Mode (*The operator must tell the computer when to make a shot, set all measurement parameters and controls when to change to the next temperature on the temperature program*)

Automatic Mode (*When activated the computer will go to the first temperature specified on the table and take the indicated number of shots at that temperature, before moving on to the next temperature. It will try and adjust certain parameters to make the best shots as it proceeds i.e adjusting pulse duration, amplification and iris.*)

8.5.3 Automatic Break (*This is essentially the same as automatic mode but if a problem occurs the computer and furnace will shut down. Ideally to be Used for measurements where the equipment will be left unattended for some time*)

8.6 Manual Mode :

Press **F2** to make the first Laser shot (*a screen with a tracking red line will appear*)

The next screen to appear will show the result of the shot

The ideal result is shown in (Figure 7)

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8.7 Points to check on a result

Duration is approximately 12 half max.

Signal level is greater than 2v but has not flattened out because it was too large

The fit to the data points is good

The diffusivity value is realistic

8.8 Printing Results

Press **Alt** to activate drag down menus

Press **R** to bring down report menu

Press **G** to select graphical output **Cr**

Press **R** to select results output **Cr**

Press **Escape** twice to return to the main screen

Press **F7** to print the results and save them to the database

(N.b. only select the output that you require)

Repeating the Shot

Press **F2** to bring up the shot menu

Change any values as necessary *(Software suggests values in brackets)*

Press **F2** to take the next shot

Repeat the three lines above as needed

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To progress to the next temperature after taking a shot press **F5**

The main menu screen will appear *(There will be a delay until the furnace reaches its next temperature and stabilises)*

Repeat section 7.8.1 until all shots required at all temperatures have been taken

8.8.2 Automatic Mode

Press **F8** to return to the main menu

Select **automatic** at the top right corner of the screen

Press **F5 Heating** to begin operation

2nd Input screen will appear

Check values here are correct and press **F5 Heating** again

Automatic settings box will appear

Prompt : Nr. of repeats (1-bad signal) (Default 8)

Enter no. of times you wish system to repeat a shot due to a bad signal

Prompt : Nr. of repeats (2-non-uniform heating) (Default 3)

Enter no. of times you wish the software to repeat the shot

Prompt : Break/Stop (in case ½) (Default Yes)

Enter Yes if you want the computer to stop measurements in cases 1 or 2 or No to let measurements continue

Prompt : Minimum Signal (Default 1.5V)

Enter the minimum signal level you will accept a shot to be

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Press **F4 Enter** to enter values and start Automatic mode

Aut.Break Mode

Repeat the same steps as for **Automatic** mode *(The difference is that after the measurement the system will shut down. Also in the case of a problem the system will again shut down)*

Saving Data :

8.9.1 There will be two methods to saving data depending upon whether manual or automatic modes have been used.

Manual : Press **F8** to end measurement

Pop up menu will appear *(Sure about exiting?)*

Select **OK** and press **Cr**

Next message will appear *(Sure about exiting?)*

Select **OK** and press **Cr**

Select **Save Data** to save to disk and press **Cr**

Pop up menu showing possible drives and directories to save to

Select drive/directory and press **Cr** *(Default c:\Laser)*

A Pop up menu will appear *(Save Data*

Delete meas. Data

Delete all data)

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Cooling will start

Program will exit to dos

Prompt to back up data Press Y to backup selected file
 Press N to not backup the selected file
 Press A to back up all files

8.9.3 Automatic Mode : Same as for manual mode

Automatic Break Mode : The software will automatically exit and you will be faced with the dos prompt

9 SHUTTING DOWN EQUIPMENT

*****The door on the front of the Laser Unit must NOT be opened when there is power to the Laser Power Unit see section 3 on SAFETY*****

Turn off the Computer

Turn off the Monitor

Turn off the Printer

Turn off the LFA Controller (See Figure 1)

Turn off the Key Switch on the Laser Power Unit (See Figure 1 No.1)

Turn off the Power switch on the Laser Power Unit (See Figure 1 No. 2)

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Close valves on the **C & D on the Laser unit** (See Figure 2 & 4)

Close both valves **E & F** on the **furnace** (See Figure 2 No. 4)

Close valve **G** on the **Furnace Control Unit** (See Figure 1 No. 8)

Set the control switch on the furnace control unit to **off** (See Figure 1 No. 7)

Close valves **A & B** on the **Furnace Control Unit** (See Figure 1 No.6)

Hold the laser unit safety switch in and raise the furnace using the up button
(See Figure 4)

Move the furnace out of the way to the left until it hits its stop

Remove Sample holder from carrier tube

Move the furnace back over the carrier tube

Hold the safety button in and lower the furnace with the down button (See Figure 4)

Tighten the furnace seal (See Figure 2 No. 5)

Turn on the fore pump (See Figure 1 No. 10)

Fully open the vacuum valve

When the pressure indicated on the **furnace control unit** is below **1mbar** turn on the turbo molecular pump (See Figure 1 No. 11& 9)

Close the vacuum valve after the indicated pressure is below **5E⁻⁰⁵ mbar**

Turn off the turbo molecular pump (See Figure 1 No. 9)

Turn off fore pump (See Figure 1 No. 10)

Turn off **Main power switch** on **Furnace Control Unit** (See Figure No. 5)

Turn off **Plugs 1 and 2**

Turn off the **16 Amp Feed**

Turn off the **32 Amp Feed**

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Open the Water Bypass

Close the Inlet Water Valve

Close the Outlet Water Valve

10 **LOADING A SAMPLE**

*****The door on the front of the Laser Unit must NOT be opened when there is power to the Laser Power Unit see section 3 on SAFETY*****

Solid Sample : *(See Figure 3 for Details)*

Using tweezers insert the sample into the test piece support

Insert the solid sample test piece support into the top of the carrier tube

Place the solid sample cover over the sample, taking care not to damage the thermocouple.

Liquid Sample : *(Masking tape, hole cutter and graphite spray are required)*

Ensure sapphire crucible and lid are clean. *(Ethanol and a cotton bud are very effective at removing traces of graphite)*

Cut two circles of masking tape $\approx$ 8mm in diameter

Place one circle centred on the top of the sapphire lid

Place the other circle centred on the base of the crucible

Spray only the top of the lid with graphite

Spray only the bottom and the edges of the crucible with graphite

Allow the graphite spray to harden

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Carefully peel off the two pieces of masking tape

Insert coated sample into crucible with the filed edges down

Place the sapphire crucible into the graphite support

Place the sapphire lid on the crucible

Gently screw the graphite cap onto the graphite support

Ensure sample is not jammed in the crucible by shaking the support gently and feeling for movement

Place the graphite support into the carrier tube

11 EDITING A MEASUREMENT RESULT

11.1 Changing the Limits

11.1.1 Maximum

Press **F4** to bring up the changes menu

Select **New Maximum** and press **Cr**

The top half of the plot alone will appear

Use the cursor keys to position the line where the maximum should be and press **Cr**

Press **F4** to view the new result

Minimum

Follow the steps as for maximum but select **New Minimum**

Adding a Filter : *(Most useful when a noisy signal has been taken)*

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Press **F4** to bring up the changes menu

Move over the filter command and press **Cr**

The filter menu will appear

Select the value of filter required (*i.e. number of points over which the system averages*
Default=0)

Press **Cr** to activate filter

Press **F4** to view result

11.3 Cutting the data : (*Useful when trailing data does not fit the trend*)

Press **Alt** to activate the drag down menus

Press **D** to activate the data menu

Press **A** to bring up plot

Use the cursor keys to put the crosshairs over the point at which you want data cut

Press **Cr** to cut the data

12 NETZSCH THERMOCOUPLE CALIBRATION

*****The door on the front of the Laser Unit must NOT be opened when there is power to the Laser Power Unit see section 3 on SAFETY*****

It is recommended that the sample temperature thermocouple mounted on the side of the carrier tube be calibrated about once a quarter depending upon the amount of use it has seen.

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Removing the Fused Silica Window :

Open the door on the **Laser Unit**

Cover the enlargement optic of the Laser with the black cap

Loosen the Lower & Upper Part by turning the knurled ring

Hold the upper part of the tube support with one hand

Turn the Lower part at a slight angle and draw it out

Turn the Upper Part at a slight angle and draw it out

Remove the Fused Silica Window from the Upper Part

12.2 Fitting the Calibration Thermocouple :

Check that both the O-rings on the Upper Part are clean (Clean in ethanol if necessary)

Pass the thermocouple through the Upper Part until its plug is up against the Upper
Parts base

Fit the Stainless Steel O-ring to the thermocouple as shown in the diagram

Carefully slide the Upper Part with thermocouple back into place

Place the thermocouple plug through the Lower Part

Push the lower part into place

Screw up the lower part until tight

Connecting the Reference Thermocouple :

Collect the two leads marked TC Calib from the top drawer under the HP Vectra PC

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Connect the long one to the calibration thermocouple

Connect the short one to the Ref. thermocouple

Connect both black plug ends together

The two red ends plug into the Fluke 8842A multimeter (Set to the mV scale)

In the back of the LFA Log Book there are calibration details

Follow the same layout and draw up the next calibration table

The calibration T.C. temperatures are calculated using tables

Be sure to take a +ve and -ve reading from the multimeter

12.4 Calibrating the Netzsch Thermocouple :

Carry out section 6 of the procedure (Machine Initialisation) but the Laser Power Unit does not need to be turned on and the dewier does not need to be filled with liquid nitrogen.

Load the temperature program called tccalib (See section 8.2.1)

Enter the temperature program

Load the Setup called Calib

Start heating to the first point

Record necessary details in the Log Book table

13 ERROR ANALYSIS REPORT FOR THE NETZSCH LFA 427 APPARATUS

13.1 A number of experiments have been conducted in order to quantify the errors in measurement.

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Repeatability : A number of runs using the same specimen and the same temperature program were conducted to test this. 95% Confidence levels were carried out at a number of temperatures,

- 13.2 **Iron SRM 8421 (ASU A-D)** $\sim 100^{\circ}\text{C} \pm 2.52\%$
 $\sim 600^{\circ}\text{C} \pm 0.61\%$
 $\sim 1000^{\circ}\text{C} \pm 1.22\%$
- 13.2.2 **Steel SRM 1421 (ASS C-D)** $\sim 500^{\circ}\text{C} \pm 0.66\%$
- 13.2.3 **Copper (CGQ4 A-D) & (CGQ1)** $\sim 465^{\circ}\text{C} \pm 3.65\%$

Affect of length Reproducibility : 6 Graphite discs of different lengths were made up from the same rod. 95% confidence tests were conducted at different temperatures, comparing the results from all samples.

- 13.3.1 **Graphite ASXA (1-6)** $\sim 100^{\circ}\text{C} \pm 2.62\%$
 $\sim 585^{\circ}\text{C} \pm 1.60\%$
 $\sim 1252^{\circ}\text{C} \pm 1.44\%$

14 APPENDIX

The following pages contain the figures indicated throughout the procedure

- 14.2 There are a number of possible furnace settings (PID) dependant upon the temperature

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at which you are operating. These control knobs are found on the **Furnace Control Unit**.

Possible setting are given in tables 1 and 2.

Furnace Temp °C	Xp (P)	Tn (I)	Tv (D)
1000°C	2.5	6	1.5
1500°C	2.0	6	1.5
2000°C	1.5	6	1.5

Table 1. Furnace control settings.

Netzsch supplied some standard settings for the whole temperature range which are

Furnace Temp °C	Xp (P)	Tn (I)	Tv (D)
All	2.5	5	Off

Table 2. Original furnace control settings.

1.

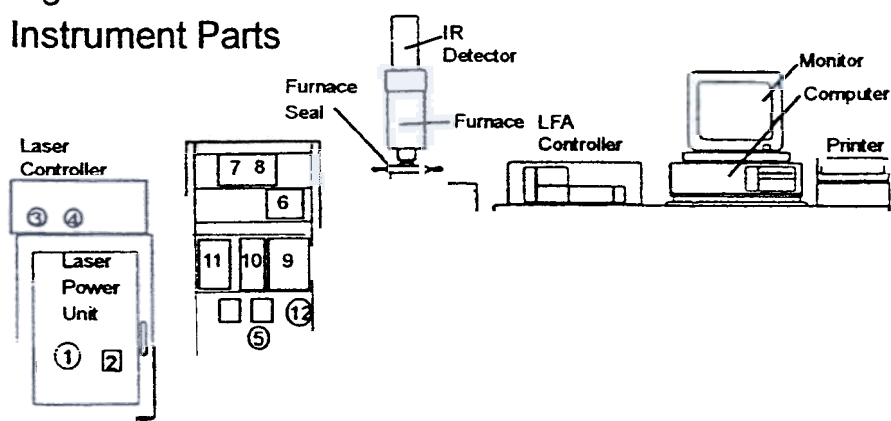
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Figure 1
Instrument Parts

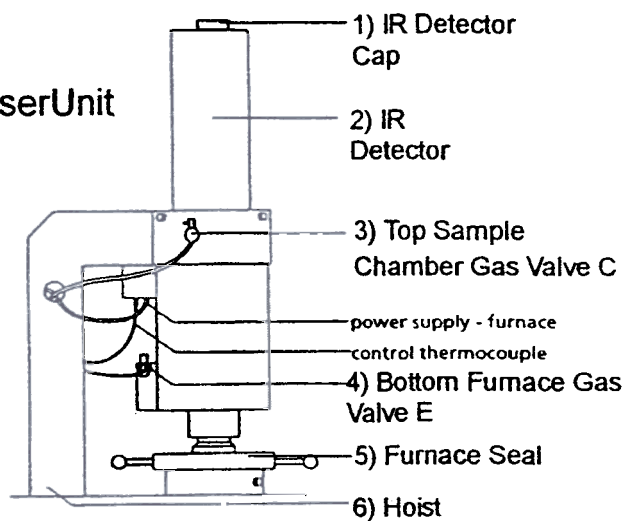


Laser Power Unit
1) Red switch
2) Key switch
3) Laser voltage control
4) Laser pulse width control

Furnace Control Unit
5) Power switch
6) Valves A & B
7) Control switch
8) Valve G
9) Turbo pump control
10) Fore pump switch

11) Pressure gauge
12) Current limiter
13) Pressure gauge & valve D

Figure 2
Top of LaserUnit



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Figure 3
Carrier Tube and Sample Holder

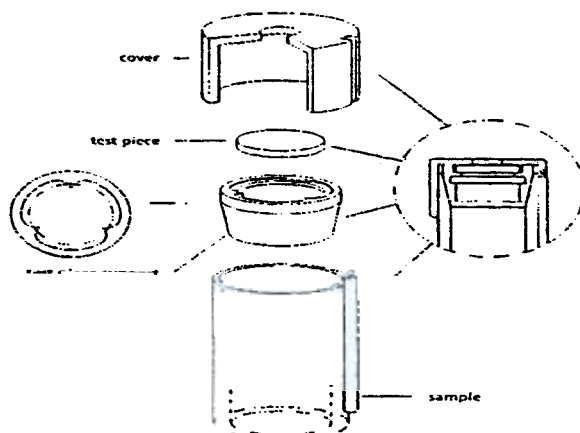
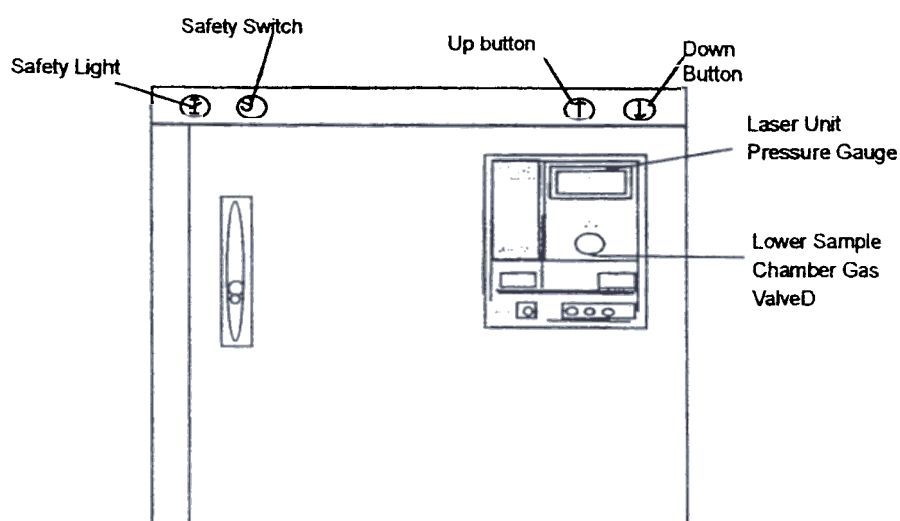


Figure 4
Front of Laser Unit



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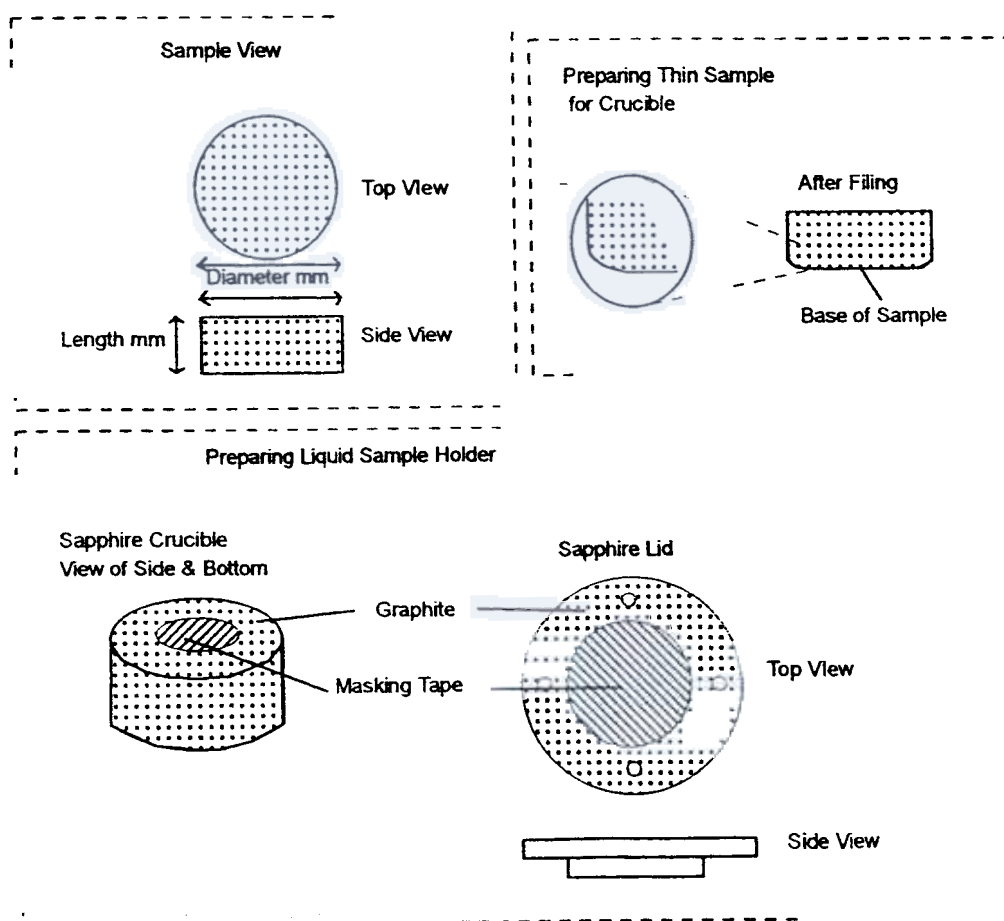
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Figure 5
Samples and Crucible Preparation



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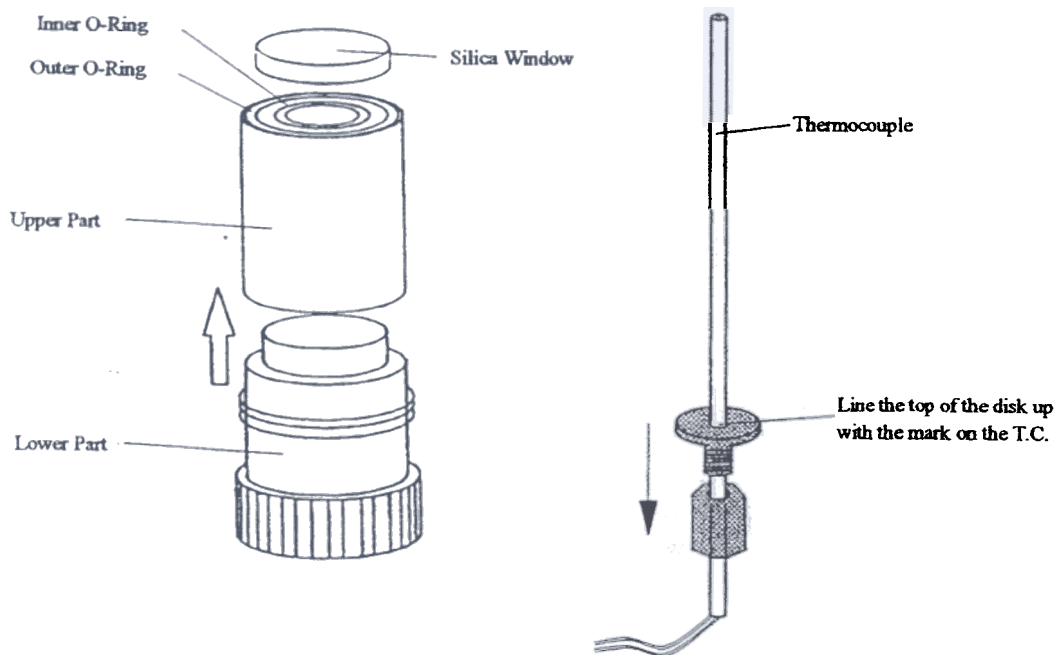
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Figure 6
Netzsch Thermocouple
Calibration



Appendix B

The heat capacity data, density and thermal diffusivity data used in the three layer model to calculate the thermal diffusivity of the slag.

Temperature, °C	Heat Capacity, Jg ⁻¹ K ⁻¹
20	0.1420
100	0.1460
200	0.1490
300	0.1530
400	0.1570
500	0.1600
600	0.1630
700	0.1660
800	0.1690
900	0.1730
1000	0.1760
1100	0.1790
1200	0.1820
1300	0.1850
1400	0.1880
1500	0.1900
1600	0.1940
1700	0.1970
1740	0.1980

Table B1. Heat capacity values of the platinum cell.

Temperature, °C	Density, gcm ⁻³
20	20.000
100	19.956
200	19.900
300	19.842
400	19.783
500	19.722
600	19.660
700	19.597
800	19.532
900	19.464
1000	19.395
1100	19.320
1200	19.252
1300	19.177
1400	19.099
1500	19.017
1600	18.933
1700	18.850
1800	18.767

Table B2. Density values of the platinum cell.

Temperature, °C	Thermal Diffusivity, cm ² s
28	0.1371
195	0.1630
392	0.1840
588	0.1973
794	0.2075
996	0.2175
1190	0.2221
1405	0.2290
1600	0.2338
1700	0.2358
1800	0.2373

Table B3. Thermal diffusivity values of the platinum cell.

Temperature, °C	Heat Capacity, Jg ⁻¹ K ⁻¹
1000	1.4443
1100	1.4443
1200	1.4443
1300	1.4443
1356	1.4443
1357	1.4443
1400	1.4443
1500	1.4443
1600	1.4443
1700	1.4443

Table B4. Heat capacity values of ASK

Temperature, °C	Density, gcm ⁻³
1000	2.6386
1100	2.6111
1200	2.5842
1300	2.5578
1356	2.5433
1357	2.543
1400	2.532
1500	2.5067
1600	2.4819
1700	2.4575

Table B5. Density values of ASK

Temperature, °C	Density, gcm ⁻³	Heat Capacity, Jg ⁻¹ K ⁻¹
All temperatures	2.7	1.35

Table B6. Density and specific heat values of ASE