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Physical Property Measurements for the Mathematical Modeling of Fluid Flow in Solidification Processes[1]

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Measurement methods are being developed to provide values for the density, viscosity, heat capacity, enthalpy, fraction solid, surface tension, and thermal diffusivity and conductivity of commercial alloys in the liquid and mushy states. These data are needed for the mathematical modeling of heat and fluid flow in solidification processes. This paper briefly describes the present state of development of apparatus for the measurement of density by the levitated drop and hydrostatic probe methods and viscosity by the oscillating viscometer in our laboratory.

KEY WORDS: density; levitation; liquid metals; viscosity.

1. INTRODUCTION

There is an urgent need for physical property data for use in the simulation modeling of solidification in industrial processes, and the absence of these data reflects the experimental difficulties encountered in such measurements. Consequently, the UK Department of Trade and Industry is funding programs to develop the measurement techniques for the reliable determination of thermophysical properties of commercial alloys. Such data are relevant to models for the casting and foundry industry, welding, and spray forming production and will encompass a variety of alloys ranging from steels, cast irons, nickel-base superalloys, and alloys of aluminium, magnesium, cobalt and titanium, each displaying its own peculiar difficulties.

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Techniques are being investigated in our laboratory for measurements in the liquid and mushy zone for those properties and their temperature dependence affecting the heat flow and fluid flow, *viz.*, density, viscosity, heat capacity, surface tension, enthalpy, enthalpy of fusion, thermal conductivity, and thermal diffusivity.

This paper describes the measurement of two of the properties which are important when describing fluid flow, *viz.*, density and viscosity.

1.1. Viscosity

Apparatus designs suitable for the measurement of the viscosity of liquid metals are reviewed by Iida and Guthrie [1] and they also report large variation of values for molten iron and aluminium, with values diverging by ± 50 and $\pm 100\%$, respectively, about the mean.

Oscillating viscometers are most suitable for the measurement of the relatively low viscosities associated with liquid metals, and a viscometer of a mechanically similar design to that reported by Ejima et al. [2] has been constructed. Modifications have been made to the method of measuring the time period and the logarithmic decrement. Signals from 39 diodes are used to reconstruct the oscillation curve of the crucible, which in turn allows better fitting of the decay of the sinusoidal oscillation.

1.2. Density

For density, two techniques are being developed: the hydrostatic probe and the levitated drop technique.

In the hydrostatic probe system a cylindrical bob is used to probe the surface and the bulk of a liquid. As the bob is being pushed or withdrawn from the liquid, its apparent mass is monitored as a function of time. Changes in the mass are due to a combination of surface tension and buoyancy forces. The equipment is based upon the work of Minaev et al. [3] and Laurent [4].

The levitated drop technique for the measurement of density was first applied by Ward and co-workers [5, 6]. The apparatus consists of a levitation tube, surrounded by a levitation coil, and prisms for enabling temperature measurement and photographic observation. Provided that the drop has axial symmetry, it is possible to calculate its volume and from the mass of the drop the density.

This paper consists of a brief description of the three sets of equipment and some preliminary results obtained.

2. EXPERIMENTAL PROCEDURE

2.1. Oscillating Viscometer

Figure 1 shows the overall design of the oscillating viscometer. The sample is contained within a cylindrical stainless-steel container (65 mm long and 24 mm in inner diameter) with a screw cap which is supported on a long suspension rod. A boron nitride crucible is available as a liner to protect the sample from contamination. The suspension rod is supported by a Pt–8% W wire which is 450 mm long and 0.2 mm in diameter. The wire was annealed at ~800°C to remove kinks. To set the system oscillating, a rotary solenoid is connected to the wire.

The suspension wire is contained within a water jacket maintained at 30°C. Above the suspension and below the wire is mounted a flat mirror (10×25 mm) and a window within the jacket wall allows a 1-mW laser to be shone directly at the mirror.

The reflected light from the mirror is detected by an array of 39 light-sensitive diodes arranged in an arc of a circle ($\pm 30°$) approximately 350 mm from the mirror. The array needs to be carefully aligned to ensure that the laser falls on the central diode when the mirror is in its rest position. The output voltages from all but the central diode are combined and measured using an A/D card and computer. This output and that from the central diode are separately logged as a function of time. From the position of the diodes and the output as the reflected light passes over them, a waveform is deduced. By use of a downhill simplex algorithm, the

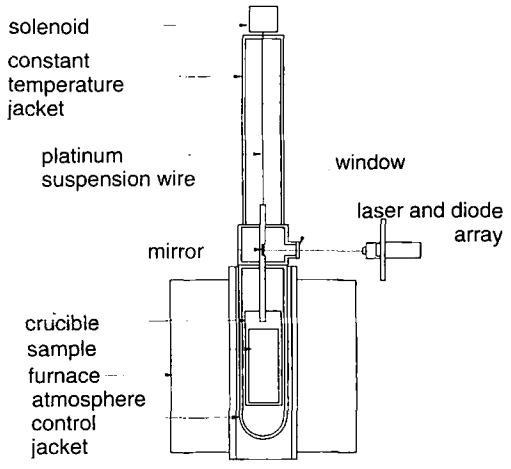

Fig. 1. Schematic diagram of the oscillating viscometer.

Table I. Uncertainties in the Measurement of Viscosity by the Oscillating Cylinder Method

Measurement	Uncertainty	Comment
Temperature	±2.5°C	
Logarithmic decrement	2.5%	Typically 0.0005 in 0.05
Time period	0.05%	
Moment of inertia	0.5%	Temperature dependence for empty system
Radius of crucible	0.1%	Temperature dependence calculated
Density	2.5%	Dependent on alloy
Height of metal	2%	Difficult to define meniscus

logarithmic decrement of the decaying sine wave is obtained over a period of usually 200 s.

The time period of the system is obtained by amplifying the output of the central diode and timing the pulses. The half-time periods from the left- and right-hand oscillations are compared to check that the system is properly aligned.

A three-zone furnace is used to heat the specimen, which is enclosed in a silica tube containing carbon to reduce the oxygen potential in the flowing argon atmosphere. Substitution of the materials to allow viscosity measurement at higher temperatures is under way.

Experiments have been performed on the empty system, ethanol, water, mercury, tin, and the commercial aluminium alloy, LM25. Crucibles were filled to give an aspect ratio (height:radius) of not less than 6:1, which has been found by experimentation to minimize end and surface effects. Roscoe's equations [7, 8], invoking four terms in the expansion, has been used to calculate the viscosity. The equation requires the moment of inertia of the empty system, the dimensions of the crucible, the time period of the empty system, the density of the specimen at the relevant temperature, and the logarithmic decrement for both the alloy and the empty system. Uncertainties in the measurements are shown in Table I.

2.2. Levitation Method For Density

The apparatus is shown in Fig. 2. Power to the coil is provided by a 15-kW, 40-kHz RF generator. The coil is designed to levitate and melt the sample and is able to lift the droplet clear of the lower turns so that the whole of the side view is visible. The levitation tube is modified so that there are four optical-quality flats with the levitation coil wound around the outside. The drop is viewed from the top via a prism and from two directions from the side through the square section. Nikon cameras

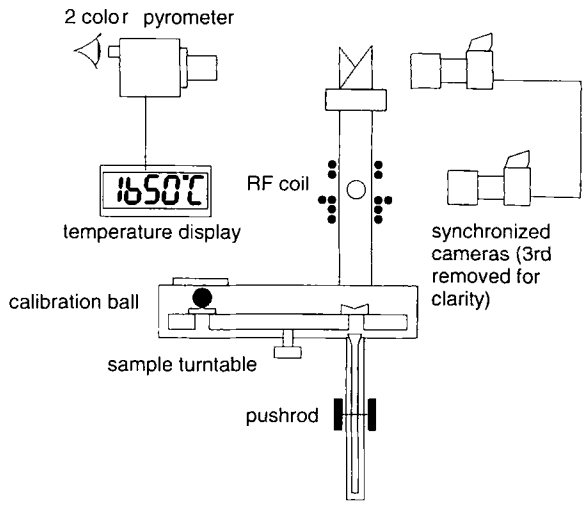

Fig. 2. Schematic diagram of the setup for the determination of density by levitation.

(Model F501) are used to photograph the drops and the shutters are synchronized to open within 1–2 ms of each other to prevent problems associated with changes in shape of the drops [9]. The temperature of the drop is monitored by a two-color pyrometer through a prism at the top of the equipment. A series of exposures is taken of the drop at the required temperature. The negatives are examined, and only sets of photographs which demonstrate axial symmetry are used. The side elevation is divided into a series of equispaced segments and the top and bottom radius of each are measured. The volume of revolution is calculated assuming a frustum of a cone.

Experiments are proceeding with scanned images to improve the repeatability and speed the measurements. Calibration of the negatives is provided with a spherical ball bearing of known dimensions. From the mass and volume the density of the material at temperature is measured.

2.3. Hydrostatic Probe

The hydrostatic probe equipment (Fig. 3) uses a high-temperature, up to 1700°C, tantalum element furnace to melt the specimen in an argon atmosphere. The specimen is placed in a crucible, made of graphite or boron nitride, and probed with a small rod of graphite, boron nitride, or alumina. The bob can be moved up and down under computer control at 0.1 μm steps; the change in mass of the bob and crucible temperature are also recorded.

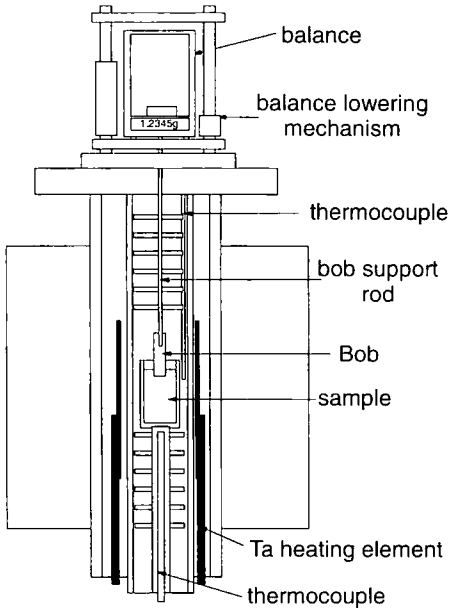

Fig. 3. Schematic diagram of the hydrostat probe.

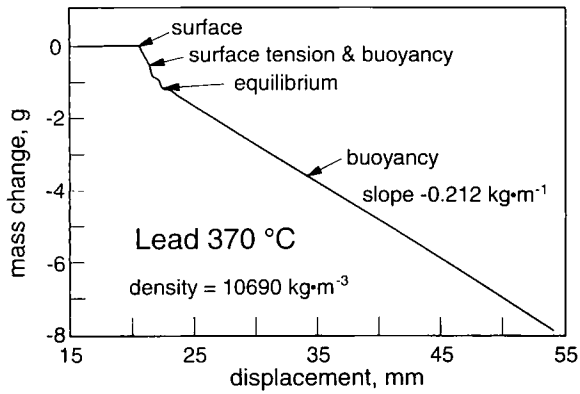

Fig. 4. An example hydrostatic probe mass-versus-distance curve for lead.

It can be seen from Fig. 4 that the change in mass of the bob is zero until the bob touches the liquid surface, when there can be either a mass increase or a decrease, dependent on wetting characteristics. As the bob is pushed into the liquid, buoyancy forces attempt to float the bob, while surface tension forces can either pull the bob down or push it away from the liquid surface. After the bob has moved a certain distance an equilibrium between the buoyancy and surface tension forces is achieved. From this point onward the surface tension effects remain constant and the density can be calculated from mass changes due to buoyancy only, as denoted in Fig. 4.

3. RESULTS

3.1. Oscillating Viscometer

To evaluate the performance of the system the viscosity of mercury, at 20°C, was measured and was found to be within 1% of the literature value [1]. An aluminium Al–Si–Mg alloy, LM25, has also been investigated; two example oscillation curves and viscosity vs temperature data are shown in Figs. 5 and 6.

The measured viscosity values are close to those recommended for pure aluminium [10] and to empirical values from METALS model based on composition. The repeatability of the measurements is high, for example, for LM25 at 700°C the mean time period over nine runs was 4.58947 (SD, 0.00022), and the log decrement 0.04970 (SD, 0.00020).

The other parameters, with typical values in parentheses, needed to calculate viscosity are the liquid radius (0.0075 m), liquid height (0.06 m), empty moment of inertia (2.0E-5 $kg \cdot m^{-2}$), and empty log decrement (0.00288). The density of LM25 at 700°C was taken as 2399 $kg \cdot m^{-3}$, and the temperature dependence as 2595–0.28 (T) $kg \cdot m^{-3} \cdot °C^{-1}$.

Preliminary experiments have shown that there is a dependence on the aspect ratio of the crucibles and the moment of inertia and logarithmic

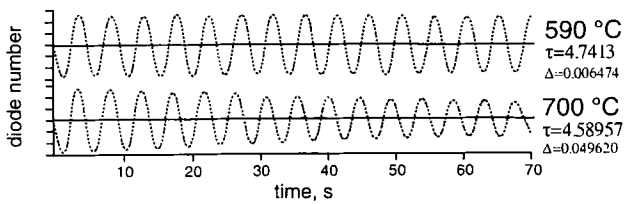

Fig. 5. Two oscillation curves for LM25, in the solid and liquid state. The logarithmic decrements are 0.006474 and 0.049620.

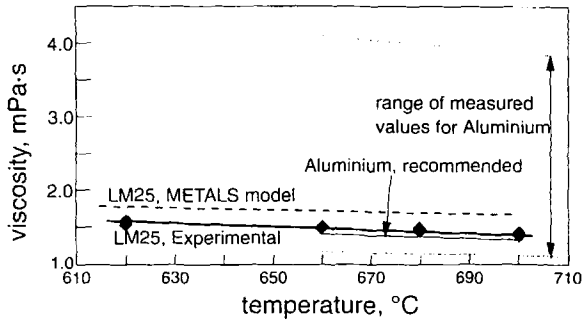

Fig. 6. LM25 viscosity versus temperature: literature values [1] for pure aluminium are shown for comparison.

decrement of the empty system. Further improvements in the system are planned including the use of longer crucibles.

3.2. Density by Levitated Drop

The density of pure iron has been measured as a function of temperature (Fig. 7). The relationship of $d\rho/Dt = 0.901\ \mathrm{kg \cdot m^{-3} \cdot K^{-1}}$ found is similar to that of previous workers, although the absolute values are some 2% lower. This is probably due to a magnification effect caused by the large temperature gradient around the droplet, which changes the refractive index of the surrounding gas. It is planned to investigate this effect.

3.3. Hydrostatic Probe

Mercury, water, copper, lead, an aluminium bronze, and LM25 have been investigated using the hydrostatic probe. There remain, however,

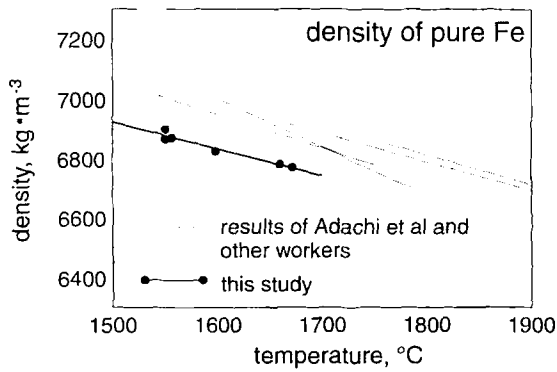

Fig. 7. The density of iron as a function of temperature.

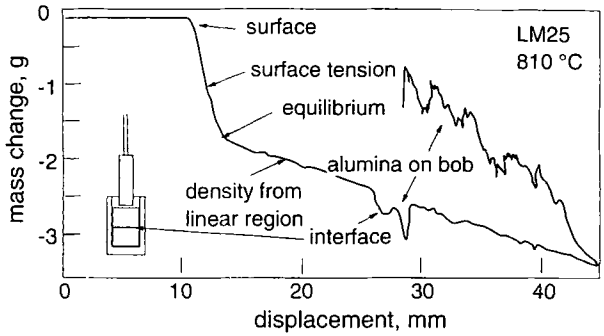

Fig. 8. Hydrostatic probing of LM25 showing the oxide skin problem.

several equipment and materials problems to overcome, the main material problem being minimization of oxide skin thickness.

Figure 8 shows the oxide skin problem. Two machined disks of LM25 were placed in a boron nitride crucible and melted. However the surface oxide skins remained and produced a "blip" in the displacement–mass change curve; from this point on the oxide started to adhere to the probe and upset the measurement, particularly as the probe was being withdrawn.

The measured density of LM25 at 810°C was 2421 $kg \cdot m^{-3}$.

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