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Estimated Uncertainties in Measurements of Molten Metal Surface Tension and Viscosity

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Abstract

Casting manufacturers all over the world increasingly rely upon state-of-the-art computational models of their casting processes to ensure the highest casting quality while maintaining the lowest possible costs. A critical need for the industry is the development of both publicly available as well as proprietary databases of critically evaluated thermophysical property data of industrial alloys in their molten and solidifying states. Auburn University and Space Systems/Loral have formed a teaming relationship to establish reliable, low-gravity measurement techniques for the most critical thermophysical properties. Unfortunately, the standard techniques can not be confidently applied to high temperature, reactive melts due to contamination from the crucibles required to hold the samples. This paper reviews the status of standard and containerless techniques for measuring the surface tension and viscosity of molten alloys. The potential of critical space-based measurements on the Space Station is also discussed.

Introduction

The use of computer simulation techniques in the molten metal processing industries is rapidly growing as manufacturers devise more competitive processes. Unfortunately, simulation results can only be as good as the input thermophysical properties used. Although many techniques exist for determining most of the required properties to accuracies of the order of ±5% or better,[1,2] property measurements on molten alloys are experimentally difficult and considerable errors may be present in the data. For example, Nagashima[3] reviewed the status of thermophysical property data for high temperature semiconductor melts and found considerable uncertainty. Convection effects and crucible contamination in very reactive samples clearly exacerbate the difficulties.

Advanced casting simulation software incorporates mold filling and convection effects and need data on the viscosity and surface tension of the molten alloy. These data are critical when modeling a thin-walled, high-precision casting for application in the aerospace, automotive, or computer industries. Accurate casting models require integrated, self-consistent thermophysical property data sets for reliable simulation of complex solidification processes. This paper reviews the status of standard and containerless techniques for measuring the surface tension and viscosity of molten alloys. In addition, the opportunities for more accurate measurements in the low-g environment of the space station are also discussed, particularly for high temperature, reactive alloys.

The need to understand the relative uncertainties between standard and containerless techniques is obvious. This paper begins addressing the uncertainty estimates. More detailed analyses supported by experiments, particularly with respect to the newly developed electrostatic containerless method, will be presented in future articles.

Surface Tension

Standard Techniques

Surface tensions of molten metals can be measured by many techniques: sessile-drop, maximum bubble pressure, pendant-drop, capillary-rise, drop weight, and oscillating drop methods.[2] The sessile drop technique has been widely utilized because of its many advantages, for example, measurements over a wide range of temperatures. Although the method is inherently straightforward by utilizing a molten drop resting on a horizontal ceramic substrate, surface tension data are particularly susceptible to the deleterious effects of contamination. Thus great experimental care must be exercised to ensure the absence of contaminants.

Bashforth and Adams[4] utilized the fundamental theory of capillarity for the determination of surface tension in 1883 and developed a theoretical description of the contour of a cylindrically symmetrical sessile drop resting on a non-wetting substrate. The equilibrium drop shape is characterized by its surface tension and the interfacial energy between the molten drop and its substrate as shown in Figure 1. The calculations are rather tedious and Butler and Bloom[5] were prompted to develop an iterative computational procedure to automate the curve fitting process by minimizing the error between the theoretical drop shape and the experimental data. Butler and Bloom[5] note that accuracies of 0.1% are possible with extremely careful attention to the parallelism of sessile drop lighting conditions, scrupulous attention to droplet symmetry, microscopic measurement of drop geometry, and avoidance of contamination. The density of molten alloys can also be determined from careful sessile drop experiments.[6]

Reactions between the molten droplet and the substrate are particularly worrisome. Recent research at Auburn University on the surface tension of superalloys

has shown that the wetting angles and apparent surface tension values change with time when utilizing typical sessile drops. [7] Although the system is still being characterized, these changes are presumably due to reactions between the substrate and the molten metal.

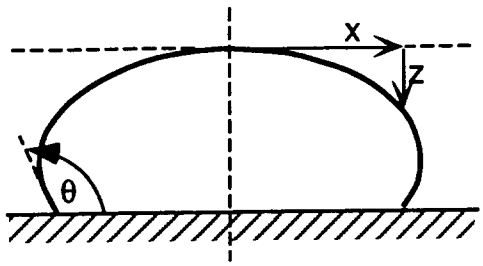


Figure 1. The shape of a typical sessile drop resting on a substrate and the geometrical quantities required to calculate surface tension.

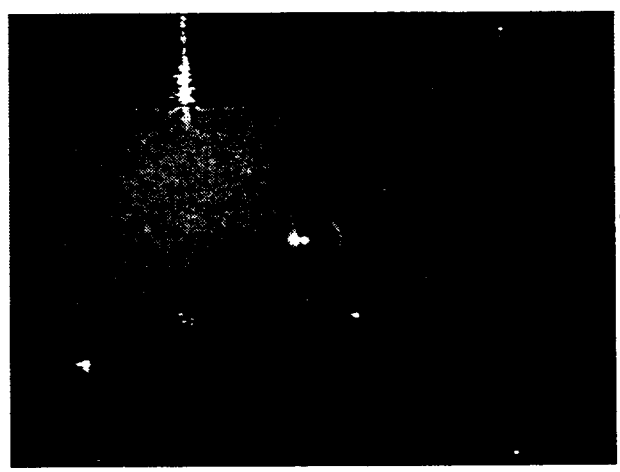
Containerless Techniques

Since many high temperature metals of commercial and scientific interest react with crucibles and substrates, containerless techniques are being developed for high-precision measurements of thermophysical properties. [8-10] These containerless methods are particularly effective in a low gravity environment. Two containerless technologies are under development for manipulating high temperature molten drops and measuring their thermophysical properties: electromagnetic levitation and electrostatic levitation .

Electromagnetic levitation (EML) is a mature technology and has been utilized in a recent series of orbital experiments with the TEMPUS electromagnetic levitator. [8] In EML, eddy currents are induced in an electrically conductive sample subjected to high-frequency, alternating electromagnetic fields. The induced eddy currents provide both Joule heating of the sample and mechanical forces due to coupling of the induced eddy currents with the applied electromagnetic field. The induction coil in Figure 2(a) is of the typical quadrapole design (i.e., upper coil opposing the bottom coil) so that a stable energy well is produced between the coils.

Containerless sample manipulation in electrostatic levitation (ESL) is achieved by application of an electrostatic field to a sample positively charged by thermoelectric and/or thermionic electron emission. [9] Heating of the sample must be accomplished with an appropriate energy beam, e.g., laser or xenon lamp. Since a three-dimensional energy well is not produced by electrostatic levitation, active control of the electrostatic fields is required for stable positioning of samples. ESL has been shown to reliably levitate many molten droplets in the earth's gravitational field, but has not been utilized in a low-g environment. However, unlike the case during electromagnetic levitation, it is believed that only minimal internal fluid flows will develop within a molten droplet undergoing electrostatic levitation. [10] This makes ESL technology particularly attractive for containerless measurements of viscosity (see below).

(a)



(b)

Figure 2. (a) Sample being simultaneously heated and levitated in a terrestrial induction coil.
(b) Sample being levitated in a terrestrial electrostatic device.

The containerless technique for measuring surface tension (γ) is based upon the classical theory of oscillations of a liquid drop. Consider a liquid droplet oscillating along the y-axis as shown in Figure 3. The linearized theory of Reid [11] gives the following relationship:

$$\gamma = \frac{3M\omega^2}{4\pi n\,(n-1)(n+2)} \quad , \qquad (1)$$

where ω_R is the oscillation frequency (typically referred to as the Rayleigh frequency from his original work[12]), M is the mass of the drop, and n counts the normal modes of oscillation. The fundamental mode is n=2 as shown in Figure 3 (b-e). Thus the surface tension is related to the natural frequency of drop oscillation. These quantities can be measured if the drop can be excited and its oscillations detected. Cummings and Blackburn[13] note that the mere presence of the magnetic field causes a slight increase in the "stiffness" of the drop (and which raises the apparent surface tension) which must be corrected for when evaluating the fundamental Rayleigh frequency. The correction factor can be estimated from translational vibration of the drop according to

$$\omega_{Expt}^2 = \omega_R^2 + 2\omega_T^2 \qquad (3)$$

where ω_{Expt} is the experimentally measured oscillation frequency, ω_R is the Rayleigh frequency, and ω_T is the frequency of the drop's translational vibration in the magnetic field.

Measurements of the surface tension of several molten metals have been performed using this technique on earth and these data agree well with data from conventional techniques.[14] The fundamental mode (n=2) is often the only mode excited, as shown by the Fourier-transform spectrum of the oscillation of a 0.464g nickel droplet given in Figure 4 where ω_{Expt} = 63Hz. Small amplitude translational vibrations can be seen at ω_T =10-15Hz. The calculated surface tension is 2.03 Nt/m, similar to the value reported by Fraser *et al*[15], but larger than the reference value given of 1.78 Nt/m in Smithells[16]. Soda *et al*[17] note that large droplet oscillation amplitudes can increase the measured frequencies. This effect is counter to what one would intuitively think and we are currently investigating this phenomenon.

Multiple peaks are also often observed.[18,19] Busse[21] has shown that rotation of a spherical droplet can cause the fundamental peak to split into five separate equally spaced peaks. In addition, Cummings and Blackburn[13] have shown that rotating aspherical droplets can experience splitting of the fundamental frequency into five separate non-equally spaced frequencies. Oscillating, but not rotating, aspherical droplets can exhibit three separate non-equally spaced frequencies.

Samples levitated by the electrostatic method generally exhibit a much more spherical shape which reduces the problem of mode splitting. However, the presence of charge on the sample will influence the measured value of the surface tension. Although the effect of surface charge is taken into account by existing theory, it nonetheless complicates the interpretation of the frequency data.

Viscosity

Standard Techniques

Although measurements of the viscosities of molten metals have been reported using several techniques, the dominant technique at moderate to high temperatures is the oscillating cup technique. In this method, a molten metal is contained within a ceramic vessel suspended by a torsional pendulum. Torsional oscillations are then induced and the resulting motion is damped primarily by viscous dissipation within the molten metal under investigation. The viscosity of a molten metal can be determined by measuring the time period and decay of the oscillations. The principal advantages of this technique are it's mechanical simplicity and the ability to measure the time period and amplitude decay with great precision. Figure 5 shows a schematic of an oscillating vessel viscometer recently developed.[21] The motion of a torsional

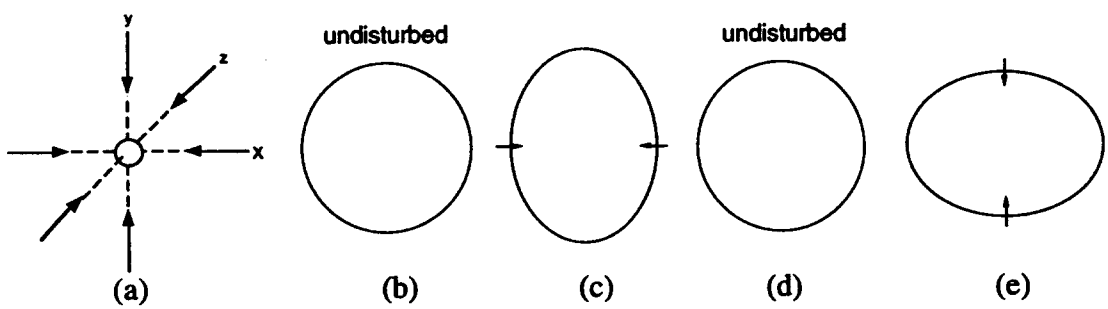


Figure 3. Schematic of Oscillating Drop Measurement of Surface Tension and Viscosity.

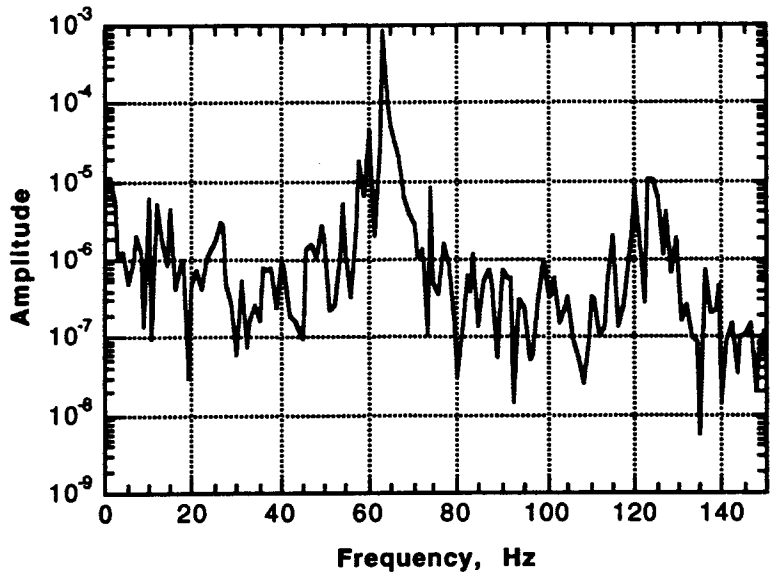


Figure 4. Fourier Spectrum of an oscillating nickel sphere.

pendulum undergoing damped oscillations can be described by

$$\theta(t) = \theta_o \exp\left(\frac{\delta}{\tau}\, t\right) \cos\left(\frac{2\pi}{\tau}\, t + \psi\right) \qquad (3)$$

where $\theta(t)$ is the time-dependent angular displacement, θ_o is the initial angular displacement, δ is the logarithmic decrement of the amplitude of oscillation, τ is the period of oscillation, t is the time, and ψ is the oscillatory phase shift. Only the logarithmic decrement and the time period need to be measured for calculating the viscosity of a molten sample. These can be obtained by a best-fit of Eq. (3) to the observed motion of the torsional suspension system.

A number of analytical equations have been theoretically developed and experimentally tested to relate the observed time period and decrement of the oscillating assembly to the sample's viscosity. Roscoe's equation[22,23] has been widely used and is considered to provide very accurate values of viscosity.[24] In fact, application of Roscoe's formula with a small correction factor has been shown to accurately reproduce calibration quality viscosity data for mercury, lead, tin, bismuth, and indium obtained using the well-accepted capillary technique.[2]

For an oscillating cylindrical vessel, Roscoe's corrected equation is[22,23]

$$\mu = \left(\frac{I\delta}{\pi R^3 H Z \zeta}\right)^2 \frac{1}{\pi \rho \tau} \qquad (4)$$

where

$$Z = \left(1 + \frac{R}{4H}\right) a_o - \left(\frac{3}{2} + \frac{4R}{\pi H}\right) \frac{1}{p} + \left(\frac{3}{8} + \frac{9R}{4H}\right) \frac{a_2}{2p^2}$$

and

$$p = R\left(\frac{\pi\rho}{\mu\tau}\right)^{1/2}$$

$$a_o = 1 - \frac{1}{2}\Delta - \frac{3}{8}\Delta^2$$

$$a_2 = 1 + \frac{1}{2}\Delta + \frac{1}{8}\Delta^2$$

$$\Delta = \frac{\delta}{2\pi}.$$

R is the internal radius of the oscillating vessel, I is the moment of inertia of the torsional assembly including the sample, H is the height of the molten metal, ρ is the density of the molten metal, and ζ is an experimentally determined correction factor dependent upon the construction of the suspension system and the design and materials of the oscillating vessel. The correction factor must be evaluated from experiments with low melting point metals of known viscosity, e.g, mercury, lead, tin, etc. All dimensions and torsional inertias must be corrected for thermal expansion effects. An iterative numerical procedure of successive approximation is required to solve Eq. (4) for the unknown viscosity.

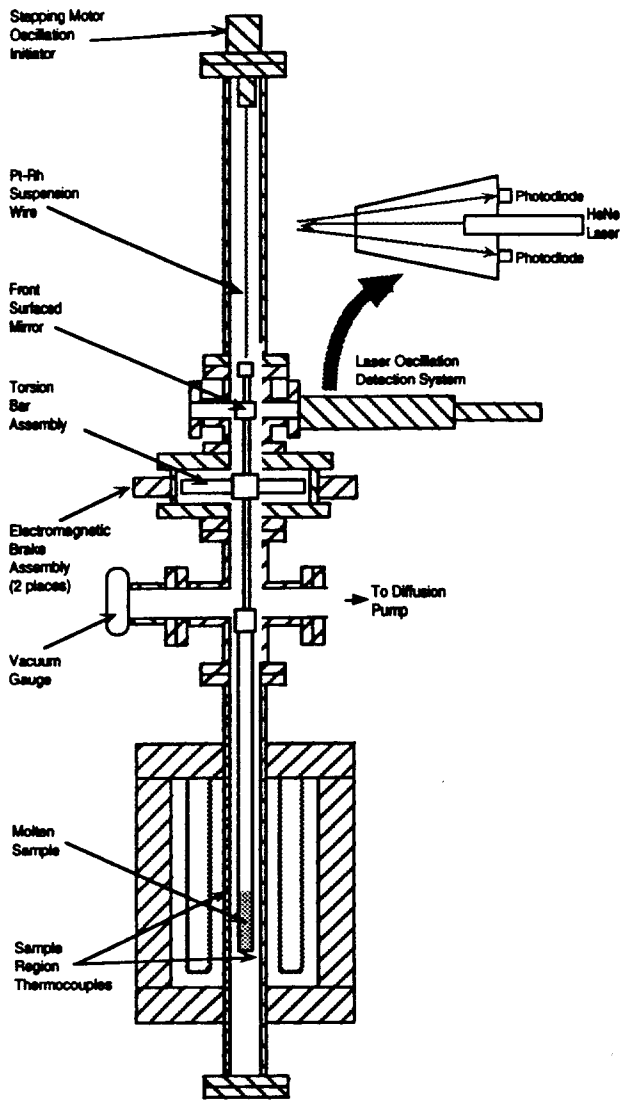


Figure 5. High-temperature oscillating vessel viscometer. [21]

Containerless Techniques

The containerless technique for measuring viscosity is, like that for surface tension, based upon the classical theory of damped oscillations of a freely oscillating liquid drop. Consider a liquid droplet oscillating along the y-axis as shown in Figure 3. The linearized theory of Reid[11] gives the following relationship for the viscosity (μ):

$$\mu = \frac{3M}{4\pi R\, n\,(n-1)(2n+1)}\,\frac{1}{\tau_n} \quad , \qquad (5)$$

where M and n are as before. R is the drop radius and $1/\tau_n$ is the damping. The measurement of viscosity using the damped oscillations of a droplet is analogous to the damped oscillations of an oscillating cup containing the molten metal. Thus the viscosity is related to the damped free decay of the oscillations of the drop. Note that the electromagnetic field must be removed before viscosity measurements can be accomplished. An auxiliary heat source must be supplied to maintain a constant sample temperature.

Measurements of the viscosity of several copper and copper-gold samples were attempted during recent experiments in low-gravity.[10] Unfortunately, the electromagnetically induced eddy currents apparently produced significant internal fluid flows within the samples which caused much more rapid decay of the oscillations than expected. Additional research is planned to confirm the origin of this behavior and minimize its impact on the measurements.

A similar approach to viscosity measurements can be done in electrostatic levitators. As in the measurement of surface tension, the surface charge must be taken into account. Evaluation of this technique is underway.

Uncertainty Considerations

The total uncertainty, ΔG, in any experimental measurement can be estimated using the procedure of Moffat.[25] When j independent variables are utilized in a function G, the individual contributions, ΔX_j, to the total uncertainty, ΔG, can be estimated by the root-sum-square method. Thus

$$\Delta G = \left[\left(\frac{\partial G}{\partial X_1}\Delta X_1\right)^2 + \left(\frac{\partial G}{\partial X_2}\Delta X_2\right)^2 + \cdots + \left(\frac{\partial G}{\partial X_j}\Delta X_j\right)^2 \right]^{1/2} \qquad (6)$$

where the partial derivative of G with respect to X_i is the sensitivity coefficient for the function G with respect to the measurement X_i.

Surface Tension

Sessile Drop: As noted above, Butler and Bloom[5] report that accuracies of 0.1% are possible with extremely careful attention to the parallelism of sessile drop lighting conditions, scrupulous attention to droplet symmetry conditions, microscopic measurement of drop dimensions, and avoidance of contamination.

Oscillating Drop: The uncertainties of the individual terms of Eq. (1) for the surface tension were estimated from actual measurements. If splitting of the fundamental frequency can be avoided so that n=2, then the uncertainty in surface tension is only due to uncertainties in (1) the mass of the drop and (2) the measured oscillation frequency (including sample translational vibration frequency, if present, to correct for magnetic field effects).

The mass of the drop can be very accurately measured before experimentation begins and after the measurements are concluded. Molten metals are typically processed in a vacuum of 10^{-5} torr or better and can lose mass due to evaporation. Thus the mass of the droplet at the actual measurement must be estimated from vaporization kinetics. This can be accomplished to within 0.1% to 2% depending upon the complexity of the alloy system and the length of processing time.

Oscillation frequencies can be detected either by high speed video image processing[18] or by focusing the drop image on high-speed photodetectors. Although video image processing is slow and tedious, all sample image data is retained. The utilization of photodetectors is much faster. A SiTek 2L10SP photodetector with a rise time of 800 ns enables better than 0.1% accuracy in oscillation frequency detection, but image information is lost. This information can be critical if assignment of appropriate degenerate frequencies through digital filtering is required due to a multiplicity of peaks. Very careful attention to (1) coil design, (2) ripple on the RF signal, and (3) external vibrations is required to minimize peak splitting.

Moffat's uncertainty estimation procedure[25] was applied to the linearized Reid[11] equation for the surface tension of nickel [Eq. (1)] and the individual uncertainties are shown in Table I. The total estimated measurement uncertainty (95% confidence limits) is approximately ±0.1 - 2.0% depending upon the certainty in knowing the sample mass. The largest contributor to uncertainty in measuring surface tension by the oscillating drop method is the uncertainty in the exact mass of the sample.

Table I

Uncertainty Estimates Table for Oscillating Drop Measurement of Surface Tension of Nickel

Parameter	Estimated $\pm 2\sigma$ Confidence Limits (%)	Surface Tension Change	Surface Tension Change Squared
Mass of drop, M = 0.894 g	2.0	0.034	0.001156
Normal mode of oscillation, n = 2	NA	NA	NA
Oscillation frequency, ω = 251 rad/sec	0.1	0.0034	$1.13X10^{-5}$
Total Uncertainty in Surface Tension, $[\Sigma(\Delta \gamma_i)^2]^{1/2}$			0.034
Total % Uncertainty in Surface Tension (γ = 1.7 Nt/m)			2.0%

Viscosity

Oscillating Cup: The uncertainties of the individual terms of the corrected Roscoe Equation were estimated from actual measurements and are shown in Table II. The inertia of the torsion assembly and the oscillating vessel radius can both be calculated to within 0.1% by the thermal expansion coefficients of the materials utilized. The logarithmic decrement and the oscillation period are measured to within 0.02% by the extreme sensitivity of the torsional pendulum. The molten metal density is generally known to no better than within 2%.[21] Although the thermal expansion of the alloy is well established up to the mushy zone[21], expansion of the alloy due to the melting transformation and free surface miniscus effects preclude knowing the height of the liquid column to better than about 2%. Finally, although the Roscoe Equation correction factor can be carefully evaluated at room temperature, its application at elevated temperatures is a cause for

concern and its uncertainty must also be of the order of 2%. Moffat's uncertainty estimation procedure[25] was numerically applied to the corrected Roscoe equation [Eq. (4)] and the individual uncertainties are shown in Table II. The total estimated measurement uncertainty (95% confidence limits) from the oscillating cup technique is approximately ± 4.6% for the viscosity of superalloy 718.[21] The largest contributors to the total uncertainty are the uncertainties in molten metal height, molten alloy density, and the Roscoe Equation correction factor.

Oscillating Drop: As noted above, the mass of the drop can be very accurately measured before experimentation begins and after the measurements are concluded. The mass of the droplet at the actual measurement must be estimated from vaporization kinetics. Depending upon the alloy system and the processing time, this can be accomplished to within 0.1% to 2%.

The undeformed drop radius can be measured to within 0.3% from processing the video image from a Sony camera with 420X360 pixels (1 pixel in 360 pixels). The damping constant can be measured to within 0.1% with the SiTek 2L10SP photodetector.

Moffat's uncertainty estimation procedure[25] was next applied to the linearized Reid[11] equation for the viscosity of nickel [Eq. (5)] and the individual uncertainties are shown in Table III. The total estimated measurement uncertainty (95% confidence limits) is approximately ± 0.1 - 2.0%. The largest contributor to uncertainty in measuring the viscosity by the oscillating drop technique is again the uncertainty in the exact mass of the sample.

Summary

Various techniques for the measurement of surface tension and viscosity of molten metals have been developed over the years. Unfortunately, the standard techniques can not be confidently applied to high temperature, reactive melts due to contamination from the crucibles required to hold the samples in earth's gravity. However, a variety of containerless techniques under development by many different research groups offer the potential to make reliable measurements on high temperature, reactive melts. The sample levitation, heating, and characterization technologies required to implement these methods are rapidly maturing.

The primary techniques for measuring surface tension (sessile drop in 1g and oscillating drop in either 1g or low-g) and viscosity (oscillating cup in 1g and oscillating drop in low-g) have been compared with respect to their potential accuracy.

The sessile drop technique has been reported to provide certainties in surface tension of better than ±0.1% when sample contamination is not an issue. The oscillating drop technique is expected to provide certainties in surface tension of ± 0.1 - 2.0% depending upon the certainty in knowing the mass of the sample.

When measuring viscosity by the oscillating cup technique, certainties of about ±4.6% are expected whereas certainties of ± 0.1 - 2.0% are anticipated from low gravity oscillating drop measurements. The largest contributors to uncertainty in the oscillating cup measurements are uncertainties in the molten metal height, molten metal density, and the Roscoe equation correction factor. The largest contributor to uncertainty in the oscillating drop measurements is uncertainty in knowing the exact mass of the sample during the test. Careful characterization of the vaporization behavior of the alloys to be tested can increase the certainty of the technique.

Acknowledgments

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Table II

Uncertainty Estimates Table for Oscillating Cup Measurement of Viscosity of Superalloy 718

Parameter	Estimated $\pm 2\sigma$ Confidence Limits (%)	Viscosity Change	Viscosity Change Squared
Assembly inertia, $I = 495$ g cm^2	0.10	0.02	0.0004
Oscillating vessel radius, $R = 0.483$ cm	0.10	0.02	0.0004
Molten metal height, $H = 5.32$ cm	1.00	0.17	0.0289
Molten metal density, $\rho = 7.35$ g/cc	2.00	0.12	0.0144
Measured decrement, $\delta = 0.00543$	0.02	0.004	0.000016
Measured oscillation period, $\tau = 2.435$ sec	0.02	0.004	0.000016
Correction factor, $\zeta = 1.025$	1.0*	0.17	0.0289
Total Uncertainty in Viscosity, $[\Sigma(\Delta\mu_i)^2]^{1/2}$			0.27
Total % Uncertainty in Viscosity (5.9 mPa sec),			4.6%

Table III

Uncertainty Estimates Table for Oscillating Drop Measurement of Viscosity

Parameter	Estimated $\pm 2\sigma$ Confidence Limits (%)	Viscosity Change	Viscosity Change Squared
Mass of drop, $M = 0.894$ g	2.0	$9.8\text{X}10^{-5}$	$9.6\text{X}10^{-9}$
Undeformed radius of drop, $R = 3$ mm	0.3	$1.5\text{X}10^{-5}$	$2.2\text{X}10^{-10}$
Normal mode of oscillation, $n = 2$	NA	NA	NA
Damping, $\tau_n = 2.906$ sec	0.1	$4.9\text{X}10^{-6}$	$2.4\text{X}10^{-11}$
Total Uncertainty in Viscosity, $[\Sigma(\Delta\gamma_i)^2]^{1/2}$			0.10
Total % Uncertainty in Viscosity ($\mu_{Ni} = 4.9$ mPa sec)			2.0%

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