Techniques for Measurements of Electrical Resistivity and Surface Tension through Sample Rotation in the High Temperature Electrostatic Levitator

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ABSTRACT
New non-contact techniques of measuring electrical conductivity and surface tension are introduced. These techniques are based on the sample rotation which was induced by applying a rotating magnetic field to a levitated drop. Experimental verifications of these techniques were made using a molten aluminum and a tin drop which were levitated in a high vacuum by an electrostatic levitator. Also demonstrated in this paper is the influence of drop rotation on the drop oscillation.

INTRODUCTION

Research on Metastable phases of materials is one of the most important research area in materials science and many related experiments will be conducted on board the International Space Station (ISS). A metastable solid phase can often be obtained from a deeply undercooled liquid phase. Such a deeply undercooled liquid state is often realized when the liquid is cooled in isolation of any potent nuclei which may trigger the liquid to solid phase transformation. For this reason, the National Space Development Agency of Japan (NASDA) is developing an Electrostatic Levitation Furnace (ELF)[1] which will be flown on board the ISS.

The ground-base electrostatic sample levitation technique and various non-contact techniques for thermophysical properties have been developed mainly at the Jet Propulsion Laboratory (JPL). At the present time, the high temperature electrostatic levitator (HTESL) at JPL [2] can measure the density, the thermal expansion coefficient, the ratio between the specific heat and the hemispherical total emissivity, the surface tension and the viscosity [3-5]. However, the techniques for the surface tension and the viscosity are based on the resonant drop oscillations of levitated drops, therefore, they are applicable only to relatively low viscosity liquids. For higher viscosity liquids, a different technique was needed. Also needed for the HTESL was a non-contact technique which can measure the electrical resistivity from a levitated melt. It is generally known that, in the ground laboratories, thermal conductivity data are more susceptible to the convective flows in the liquid than those of electrical resistivity. However, for those liquids where the Wiedemann-Franz-Lorenz law can be applied, more accurate thermal conductivity data may be obtained from measured electrical resistivity.

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In this paper, we introduce two new non-contact techniques: one for the measurement of electrical resistivity, and the other for the measurement of surface tension of liquids including high viscosity liquids. Both of these techniques are based on the sample rotation induced by applying a rotating magnetic field to a drop levitated by the HTESL.

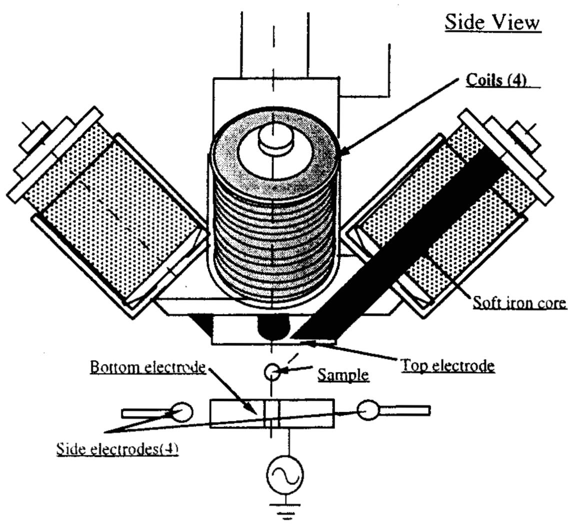

Fig.1 A schematic of the electrode assembly used for the experiment

EXPERIMENTALAPPARATUSANDAPPROACH

The principle of the sample rotation is based on the application of a constant torque to a molten metallic drop that is levitated in a high vacuum by the HTESL. The HTESL levitates a sample about 3mm in diameter between a pair of parallel disk electrodes that are spaced about 12mm (Fig. 1). The electric field between these two electrodes produces a strong electrostatic force on the charged sample to cancel the gravitational force. The four small side electrodes located around the bottom electrode control the sample position along the horizontal directions. The main position control voltage is connected to the upper electrode, while the bottom electrode is electrically grounded through an AC voltage source. This AC voltage source produces an oscillating electric field on the drop. When the frequency of the AC field matches a resonant oscillation frequency of the drop, a resonant oscillation is induced. To induce a sample rotation, a four-coil system was mounted on the top electrode. These coils produce a horizontal magnetic field that rotates at an appropriate frequency (400Hz in the present experiment). The details of the sample rotation and the detection methods are described in an earlier publication [6]. The electrode assembly was housed by a stainless steel chamber that was typically evacuated to 10^{-8} torr. The chamber was surrounded by various non-contact diagnostic instruments for the measurement of the sample temperatures, the drop rotation rates, and the drop oscillation frequencies. Samples were heated using a xenon arc lamp. A detailed description of the HTESL can be found elsewhere [2].

ELECTRICAL RESISTIVITY MEASUREMENT

The basic principle of the sample rotation mechanism is essentially same as the asynchronous induction motor. The four-coil assembly works as the stator while the levitated sample acts as the rotor. According to the principle of the induction motor [7], [8], if an ac current I_1 at frequency ω_s is applied to a stator, the torque τ experienced by the rotor (having its own resistance R_2 and inductance L_2) which is rotating at an instantaneous frequency ω is given by

$$\tau = I_1^2 \omega_s \left(\frac{sR_2}{R_2^2 + s^2 L_2^2} \right) \qquad (1)$$

where

$$s \equiv \frac{\omega_s - \omega}{\omega_s} \qquad (2)$$

Eq. (1) was tested by levitating an aluminum sphere and measuring torque as a function of instantaneous sample rotation frequency. It can be seen in Fig.2 that the torque is a linear function of the sample rotation frequency. This means that the relationship $R_2^2 >> s^2 L_2^2$ is well satisfied in Eq. (1), transforming the equation to a simpler form;

$$\tau = I_1^2 \omega_s \frac{s}{R_2} \qquad (3)$$

Eq. (3) means that torque τ is inversely proportional to the rotor resistance at a fixed rotor frequency.By using this principle, electrical resistivity of a sample as a function of temperature can be measured from a levitated sample in the HTESL. An aluminum sample, which was levitated and heated at different temperatures, was spun by fixed stator current. Time intervals for spinning up the sample from 20Hz to 30Hz and spinning down form 30Hz to 20Hz were measured, and average torque was calculated. Fig.3 shows the result of the experiment. A literature value[13] of resistivity of molten aluminum at the melting point was used as a single calibration point to obtain this result. Our temperature dependence of resistivity in liquid phase is

$R_{e,liq} = 24.9+1.306 \times 10^{-2}(T-T_m)$ $\mu\Omega$cm (4)

which are approximately 10% smaller than that of reference. Detailed of this experiment is found in earlier publication [6].

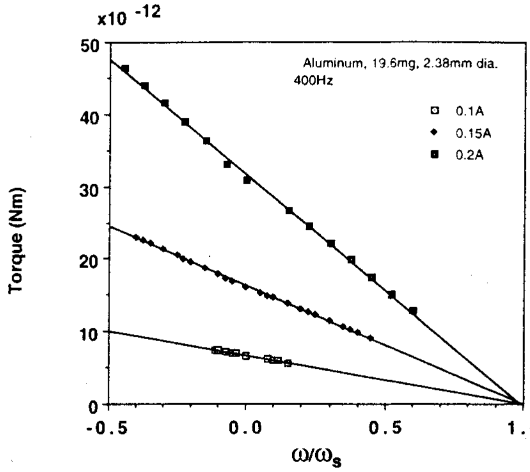

Fig.2 Linear relationship between the measured torque and the instantaneous rotation frequency of a levitated aluminum sphere for three different coil current

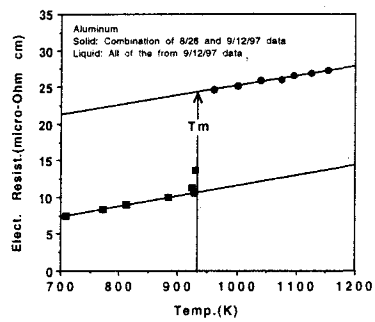

Fig.3 Electrical resistivity of a pure aluminum sample measured as a function of temperature. The squares are for solid phase and the circles are for the molten phase.

SURFACE TENSION MEASUREMENT

SHAPE EVOLUTION OF ROTATING DROPS

We chose a high purity aluminum and a tin sample and conducted the rotating drop experiments. All parameters relevant to these samples and experimental conditions are shown in Table I. The experiment was initiated by applying a rotating magnetic field to the sample, and video-recording the side view of the drop. The drop rotation frequency was regularly measured and overlaid on the video images. As the drops started rotating, the nearly spherical initial shape progressively turned into oblate spheroidal shapes, and this trend continued until they reached a critical point, the bifurcation point, at which the axisymmetric shape became unstable and the transition to the triaxial ellipsoidal branch (or the two-lobed branch) took place. As the drop gained angular momentum, the drop rotation frequency started to decrease as a result of rapidly increasing moment of inertia. Fig.4 shows the side views of the rotating aluminum drop in the order of increasing angular momentum. In the figure, **a**, b, c, and d correspond to the axisymmetric shapes. At d, a quick transition took place from the axisymmetric to the two-lobed branch. As the drop gained more angular momentum, the shape changed to the point f passing though e.

Table I Parameters relevant to the samples

Samples	Aluminum	Tin
Weight(mg)	26.5	27.1
Melting Point (℃)	660.5	232
Sample temp. (℃)	705	320
Density at Tm (g/cm3)	2.38	6.98
Surf. Tension at Tm(mN/m)	914	560
Viscosity at Tm(mPa s)	2	1.81
Net Elect. Charge (nC)	0.445	0.3451
Rayleigh limit (nC)	1.843	0.851
$Q/Q_{Rayleigh}$ (%)	24.16	41.2

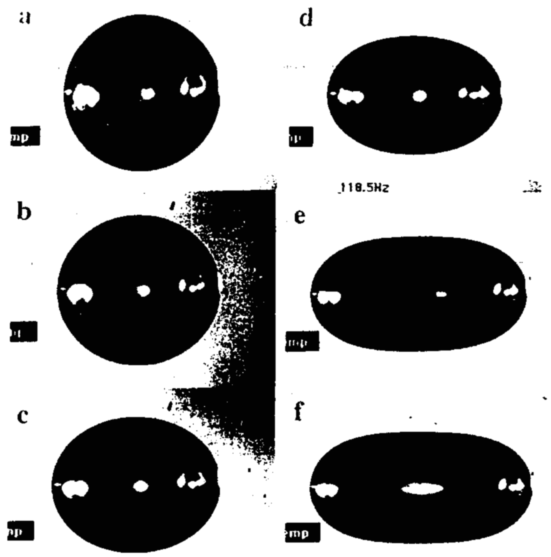

Fig.4 Side view of the rotating aluminum drop in the order of increased angular momentum.

Fig.5 shows the evolution of a normalized drop dimension, R_{max}/R_0, versus the normalized rotation frequency, $\omega_{rot}/\omega_{osc}$, where R_{max} and R_0 are respectively the maximum of the rotating drop at a given rotational rate and the radius of the drop when it was spherical. The drop rotation frequency ω_{rot} is normalized by the oscillation frequency ω_{osc} of the drop at $P_{2,0}(\cos\theta)$-mode in the non-rotating state. Also shown in Fig.5 is the theoretical result obtained by Brown and Scriven [9], showing the initially axisymmetric shape branching out to a two-lobed, a three-lobed, or a four-lobed drop shape at the respective critical frequencies. Transition between axisymmetric and two-lobed branch was reversible as long as the applied torque level was kept sufficiently low to ensure the solid body rotation.

The experimental points of both the aluminum and tin drops followed the theoretical curve within 2%. The data points along the axisymmetric branch are slightly below the theoretical curve due to the gravity effect. The observed bifurcation points also agreed with the predicted value, $\omega_{rot}/\omega_{osc}$=0.559, within 2%.

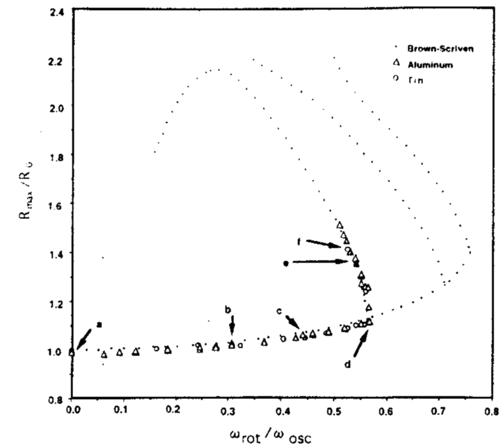

Fig.5 Experimental data of normalized drop size versus normalized drop rotation frequency of both the molten tin and aluminum drops are compared with the theoretical curves by Brown & Scriven.

SURFACE TENSION MEASUREMENT BY DROP ROTATION

For low viscosity liquids, the drop oscillation technique is a commonly used non-contact method of measuring the surface tension of a levitated drop. In this technique, the $P_{2,0}(\cos\theta)$-mode of the drop is exited by some means, and the surface tension is obtained from the oscillation frequency ω_{osc}. However, for highly viscous liquids where induction of $P_{2,0}(\cos\theta)$-mode oscillation is difficult, a different method is needed. If a drop rotates following the curves in Fig.5, the oscillation frequency of a drop in its non-rotating state, ω_{osc}, can be determined by the shape parameters R_{max}/R_0 and the rotation frequency ω_{rot}. We note that the liquid viscosity doesn't play any role for theory other than helping the drop to obtain solid-body rotation condition. This proposal of measuring the surface tension based on the drop rotation was first made by Elleman et al [10].

A molten tin drop was used to verify this technique. When the drop reached a steady state temperature, the $P_{2,0}(\cos\theta)$-mode of the oscillation was induced in its non-rotating state and the frequency (99.3Hz) was measured. Then, this frequency was compared in Fig.6 with those that were determined from the tin data in Fig.5. At lower rotation frequency, the effective oscillation frequencies scattered widely, then, it converged to actual oscillation frequency as the drop rotation frequency approached the bifurcation point. This can be understood since R_{max}/R_0 increases rapidly as the rotation frequency approaches the bifurcation point. From Fig.6 one can see that the effective oscillation frequency agreed with the actual frequency within ±2% (which corresponded to ±2Hz). At present, this accuracy is lower than that of the oscillation technique that has reproducibility better than 0.5Hz. However, the accuracy is expected to improve as a more sophisticated drop shape analysis technique is applied. Once the effective oscillation frequency is determined, the surface tension can be calculated following the recipe given in an earlier publication [5]. It is important to emphasize that this new technique can be useful for the surface tension measurement of conducting liquids where the drop oscillation technique cannot be applied.

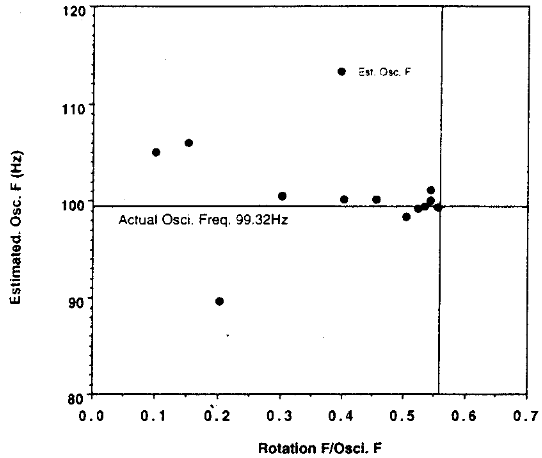

Fig.6 The effective oscillation frequencies of a tin drop obtained at different rotation rate are compared with the directly measured oscillation frequency of the same drop.

OSCILLATION OF ROTATING DROP

Utilizing the systematic drop rotation capability, an attempt was made to verify the Busse's prediction [11], using a molten metallic drop levitated in a high vacuum. Busse investigated the effects of the frequencies of the shape oscillations of a drop immersed in a fluid of a certain density that is moving at the same angular velocity. He considered the linear, incompressible, inviscid liquids. Under the assumptions that the axisymmetric rotation-induced shape deformation and the amplitude of the oscillations were small, and host fluid is replaced by a high vacuum as it was in our experiments, his analytical expression for the effect of rotation on the axisymmetric resonance frequency (i.e. the $P_{2,0}(\cos\theta)$-mode) can be expressed by

$$\frac{\Delta\omega_2}{\omega_2^{(0)}} = \frac{\omega_2 - \omega_2^{(0)}}{\omega_2^{(0)}} = \frac{19}{21}\left(\frac{\Omega}{\omega_2^{(0)}}\right)^2 \qquad (5)$$

Where ω_2 is the resonance frequency at a drop rotation frequency Ω and $\omega_2^{(0)}$ is the resonance frequency when Ω =0.In an earlier experiment, Annamalai et al. [12] used an oil drop that was acoustically suspended in a rotating water tank, and demonstrated a good agreement with the theory. In the present experiment, a molten aluminum drop levitated in a high vacuum was rotated along the vertical direction and the axisymmetric mode of oscillations, i.e. $P_{2,0}(\cos\theta)$-mode, was induced at various rotation frequencies. The mass of the aluminum drop was 26.2mg and the temperature was 998K. In Fig.7, the experimental results are compared with Busse's theoretical prediction expressed by Eq. (5). The agreements are excellent throughout the entire axisymmetric branch.

This result also demonstrates the effect of the rotation on the drop oscillation frequency if the surface tension of a liquid is to be determined from the drop oscillation frequency. Surface tension measurement with oscillation method may result in a wrong measurement if the sample's rotating condition is not well controlled.

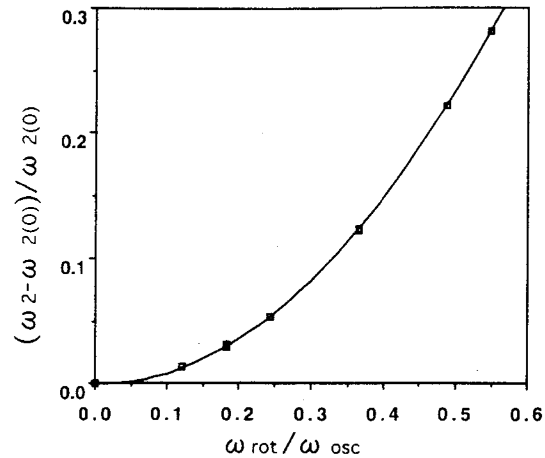

Fig.7 Normalized oscillation frequency of an aluminum drop as a function of normalized rotation rate.

CONCLUSIONS

In this paper, we described two non-contact techniques:one for the measurement of electrical resistivity of conducting liquids, and the other for the measurement of surface tension of highly viscous liquids. We also demonstrated the importance of controlling the sample rotational state when the surface tension is to be measured by the drop oscillation method. We plan to implement these techniques to the Electrostatic Levitation Furnace that will be flown on board the ISS.

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