High Temperature Surface Tension Measurement

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***Abstract*—Atmospheric radio frequency inductively coupled plasma (RF-ICP) is used as the high temperature heat source in this containerless method to measure the surface tension and viscosity of high melting point materials. Dynamics of melting, droplet formation, and drop detachment of a rod exposed to RF plasma are recorded. Using a three-dimensional model in which flow field, heat transfer, and phase change are solved, the melting process of the rod will be simulated. Comparing simulations and experiments, surface tension, and viscosity of ceramics can be obtained. Some preliminary results from the experiments for copper and alumina are reported in the present study.**

***Index Terms*—Ceramics, high temperature, inductively coupled plasma, surface tension, viscosity.**

ONLY a few of interfacial tension measurement methods [1], [2] can be implemented to melts at high temperatures. At temperatures above 1000 °C, successful application of any method largely depends on the melting dynamics and its effect on factors such as resistance to chemical reaction or volumetric change. It is noteworthy that state of equilibrium is vital, since reactions in progress affect surface tension. Maximum bubble pressure method has been frequently employed in experimental determination of the surface tension of liquid metals and alloys. Maximum drop pressure method has been applied for determining the surface tension of highly reactive, low-melting-point metals, but is limited because of difficulties in the design of equipment for high temperatures. Capillary rise method is not commonly used for liquid metals because the exact knowledge of contact angle is important. This angle is often more difficult to measure than the surface tension [3]. The pendant drop method and drop weight method are applicable to most transition metals. Sessile droplet technique has been used for measuring surface tension of liquid metals and alloys. Levitation methods are generally used for surface tension measurement of melts; however, it is not properly applicable to materials with high electrical resistivity.

In spite of the existence of a variety of methods, measuring surface tension of very high-melting-point materials (e.g., zirconia melts at 2600 °C–2700 °C) is still impossible. The main concern is how to melt such a material and where to keep its melt.

A brief review of the common methods manifests the draw backs in using the methods for high temperature cases. As stated before, maximum bubble pressure method is recommended for measuring surface tension of melts. In the case of high-melting-point ceramics, preparing the appropriate set up is not possible. Drop volume and pendant drop techniques are acceptable if it was possible to melt the material where at least one drop at the end of a vertical rod is formed. Another option could be the sessile drop method, in which, in addition to the symmetry issues, not only stationary droplets should be generated, but also an appropriate container with a melting point at least higher than the material under study is required. Problems such as contaminations, high cost, equipment limitations, and unexpected instabilities are the major reasons that make levitation method [4] deficient for high-melting-point materials. Considering the limitations of the common techniques in addition to industrial need for the physical properties is a consequent motivation for new methods.

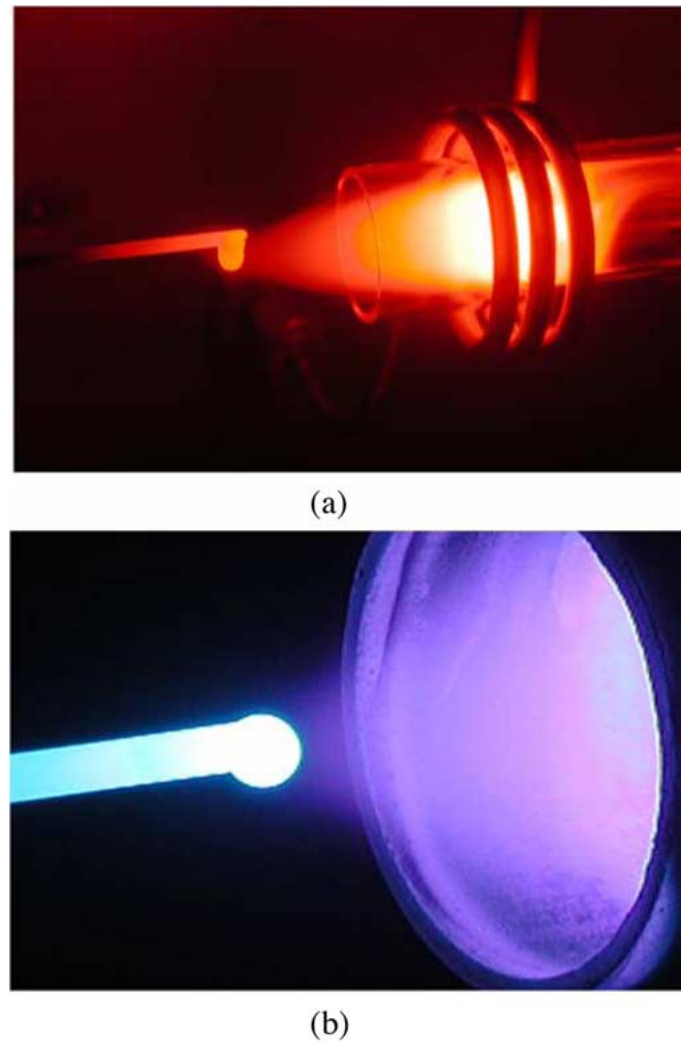

Fig. 1. (a) Melting of a copper wire in argon RF plasma, power: 250 W, (b) alumina bar, power: 650 W.

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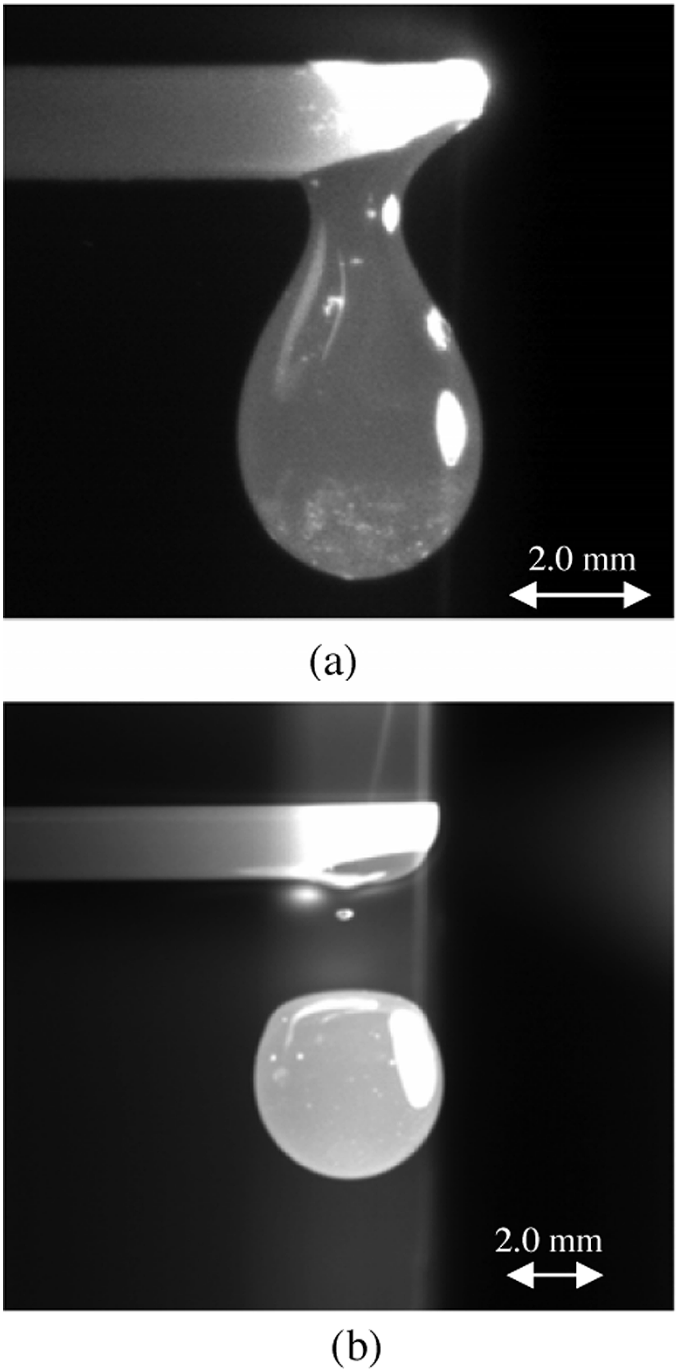

Fig. 2. (a) Symmetric pendant droplet of copper. (b) Detached drop and satellite droplet just after breakup.

A new technique is being developed to measure surface tension and viscosity of high temperature ceramics. In this method, a rod of the desired material is exposed to the high temperature of radio frequency inductively coupled plasma (RF-ICP). Dynamics of the melting, formation of a drop and its detachment are recorded via a high-speed video camera. The size of the droplet along with the measurement of the neck diameter will provide the surface tension. By simulating the process numerically using a three-dimensional (3-D) model [5], one may estimate the viscosity values and surface tension that result in agreement between the experiments and simulations. Such an *inverse engineering* approach can be viewed as a coupled experimental and numerical method. However, the modeling results will not be presented here.

A 40-MHz RF-ICP is employed to melt 1.6-mm-diameter copper wires [Fig. 1(a)] and an alumina bar with 1 mm $\times$ 0.6 mm cross section [Fig. 1(b)]. Quartz torch (Delta Scientific Laboratories, Mississauga) has a diameter of 1.8-cm. RF power supply (Advanced Energy Industries, Inc., Co) can generate up to 1-kW power. A charge coupled device (CCD) camera (Sensicam, Optikon Corporation Ltd., Kitchener, ON) equipped with a 90-mm macrolens with an intensified CCD chip with a resolution of 1280 by 1024 pixels is used. Fig. 2(a) and (b) has been taken with the Sensicam. The camera provides the possibility of single photo capturing at very low exposure times. Triggering can be done either internally or externally. In order to trigger externally, a delay generator is used to trigger the camera at specific frequencies. The exposure time of 100 μs triggered by an external digital time delay generator (DG 535, Stanford Research Systems, Sunnyvale, CA) at 50-Hz frequency. The selection of triggering frequency (50 Hz) was a tradeoff between the memory availability and phenomenon of interest. Time delay between Fig. 2(a) and (b) is 45 mSec.

Dynamics of melting phenomenon recorded by a high-speed video camera (PHOTRON FASTCAM-ultima 1024, Corporation Ltd., Kitchener, ON) with a rate of 500, 1000, or 2000 frames/s will be used compare the experimental observations with numerical simulations.

Fig. 2(b) shows the return of a satellite droplet (one order of magnitude smaller than the diameter of wire) to the wire when the main drop detaches. Considering the necking phenomenon (contrary to Tate's law [6]), the calculated surface tension for copper, 1.375 ± 0.065 (N/m), is in good agreement with the existing data [3], [7]. It should be noted that the plasma and sheath gas with the flow of 1 slpm and 7.6 slpm, respectively, does not affect the symmetry of the hanging drops [Fig. 2(a) and (b)]. Since the plasma is argon, it also protects the wire from oxidation. It is expected that the proposed method is applicable to high-temperature ceramics (e.g., zirconia). However, the power introduced by the plasma is limited by the power generator. Besides, the melting temperature of the plasma torch is another limiting parameter.

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