Measurements of the Hg Fill Level in Low-Pressure Hg Discharge Lamps via Macrophotography

Kaitlin A. Fundell, Charles M. Klimcak, James C. Camparo
Photonics Technology Department
The Aerospace Corporation
El Segundo, CA, USA
kaitlin.a.fundell@aero.org

Abstract - Using a simple macro-photographic method and standard image analysis software we determined the dimensions and subsequently the mass of Hg droplets contained in small, cylindrical, rf-excited discharge lamps like those used in Hg-ion atomic clocks. The mass derived from an analysis of these photographs was compared to that determined from a measurement of heat required to melt condensed Hg in the lamp using the technique of Differential Scanning Calorimetry (DSC). These two mass determination methods were found to agree within ≈ 25 % in an investigated Hg mass fill range that varied from 0.2 to 17.3 mg.

Keywords—Hg+ clock; Deep Space Atomic Clock (DSAC); Differential Scanning Calorimetry (DSC); Hg lamp fill

I. INTRODUCTION

Some atomic clocks (e.g., rubidium and mercury-ion frequency standards) rely on a metal-vapor rf-discharge lamp to generate the device's atomic signal. Briefly, rubidium (Rb) or mercury (Hg) atoms in the lamp are excited by the discharge, creating Rb or Hg resonance light that is employed for optical pumping. Surprisingly, though rf-discharge lamps have a nearly 100-year history, understanding the subtleties of their operation and their life-limiting mechanisms is only now beginning. In part, this renewed interest in atomic clock rf-discharge lamps derives from the fact that their performance bears directly on the capabilities of global navigation satellite systems, and the satellites for those systems are expected to have decade (or longer) lifetimes. Crucial to studying rf-excited discharge lamps and their long-term operation is a non-destructive assessment of the mass of Rb or Hg that has been placed into the lamp bulb [1-3]. While calorimetric measurement methods that deduce mass from the change in heat that accompanies a phase transition have been exploited for this purpose, they require acquisition of a costly differential scanning calorimeter capable of accommodating the lamps and the maintenance of trained technical staff to perform the measurements. Here, we show that the metal fill in a Hg rf-excited discharge lamp can be determined using a simple photographic method that requires only a visible CCD camera, extension bellows and a standard camera lens. Since Hg is liquid at room temperature, and has appreciable surface tension, it can be condensed into the base of the lamp to form a single, high contact angle, contiguous droplet exhibiting a spherical cap geometry that can be easily photographed. Analysis of the photograph via standard image processing software permits its dimensions to be determined from which one can derive its volume and mass. Although imaging through a small diameter cylindrical lamp creates significant optical distortion, we have demonstrated an image analysis method that results in a maximum metal mass error of less than ≈ 25%. While not as accurate as the calorimetric mass measurement procedure that is currently used to determine rf-discharge lamp fills for Rb and Hg consumption investigations, the simplicity of our photographic method will allow researchers to easily assess mass fills in their rf-discharge lamps studies.

II. EXPERIMENTAL PROCEDURES

Six low pressure cylindrical lamp bulbs approximately 7 mm in diameter and 20 mm in height fabricated from Heraeus Suprasil Quartz by Precision Glassblowing Inc. (PGB) [4], that were filled with 10-15 mg Hg and in some cases 2 torr and in others 5 torr Ar buffer gas were used in this investigation as well as a lower fill Hg lamp (200 micrograms) that was 5 mm in diameter and 20 mm in height fabricated and provided to us by a private party. Three of the 7 mm diameter lamps had a round, dome-shaped base while the other three lamps had a flattened base which was designed to facilitate DSC measurements. The base on the 5 mm diameter low-fill lamp was also dome-shaped.

The lamps were photographed using a reverse-mounted, Nikon 50 mm f/1.4 lens in conjunction with a bellows system to permit macrophotography. We used a bellows extension that provided a magnification of approximately 4.7X for all photographs. A visible CCD camera manufactured by Imaging Source (Model DMK41BU02) having a 1280 x 960, 1.2 Mp, ½" diagonal CCD was used to record images of the droplets using manufacturer provided software. A photograph of the camera/bellows/lens system is shown in Figure 1.

978-1-6654-3935-0/21/$31.00

Fig. 1. Photography setup with reverse-mounted lens, bellows and camera aimed toward a post-mounted lamp.

Diffuse backlighting was used for all photographs to reduce strong reflections from the highly reflective drop and the lamp envelope. The dimensions of all recorded images were determined using ImageJ image processing software [5]. No additional image enhancement or post-processing was used.

The masses of Hg in the lamps that were used to assess the fidelity of the photographic technique were determined using the technique of Differential Scanning Calorimetry (DSC) using a Perkin-Elmer Diamond DSC and the resulting scans analyzed using their provided Pyris software. Details of the calorimetric measurement procedure are provided in References 1-3. Analysis of the scans provides the amount of heat energy required to melt Hg that had been previously crystallized in the lamp, a quantity that is directly proportional to the mass of free metallic Hg contained in the lamp. Typically, DSC measurements are performed with samples that are encapsulated in flat aluminum pans that are confined to thermally isolated ovens that minimize thermal losses, a measurement configuration that is not possible to achieve with a conventional commercial calorimeter that has been adapted to accommodate an oblong lamp that obviously cannot be encapsulated. Consequently, DSC measurements of lamps typically yield a mass that is slightly lower than the mass of metal contained in the lamp due to the presence of additional heat losses. This necessitates the use of a multiplicative mass correction factor typically in the range of 1.02 – 1.20 to determine the true mass. The lamp correction factor is separately determined from a calorimetric measurement of a known, weighed mass of Indium metal that has been added to an identical lamp envelope. After determining this correction factor the masses of the Hg droplets in the lamps were calculated from our DSC measurements of the energy required to melt the Hg droplet in each lamp and the calorimeter calibration factor that we had separately determined that compensates for heat loss in this unconventional sample geometry.

III. Results and Interpretation

Typical side-on photographic images of Hg droplets in low and high mass lamps are shown in Figure 2. Calibration of the dimensions in these images was obtained by comparison to images of a reticle having a finely divided rule that was placed at the location of the drop after removing the lamp.

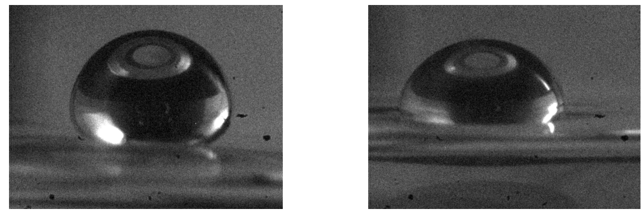

Fig. 2. Sample round-bottomed lamp Hg droplet image (left). Sample flat-bottomed lamp Hg droplet image (right).

The volume of a sessile drop small enough to be considered a spherical segment is given by [6]:

$$Volume = \frac{\pi}{6}\, h\,(3a^2 + h^2) \qquad (1)$$

where h is the drop's height and $2a$ its base diameter (Figure 3). This equation is valid for drops that are not significantly affected by gravity. It has been shown previously [3] that gravity begins to flatten drops larger than ≈ 640 mg, approximately 38 times larger than the mass of the largest drop we photographed. Consequently, the assumption of a spherical cap geometry is very reasonable. The height and base diameter of the drop were then determined by analysis of the photographs using ImageJ software. After computing its volume, the masses of the drops were determined from the known density of Hg at room temperature (13.53 g/cm^3).

Calorimetric mass measurements on flat bottom lamps were performed by placing sample and empty reference lamps in the ovens of the sample and reference calorimeters as shown in Figure 4.

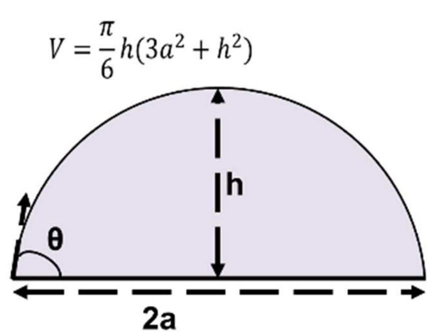

Fig. 3. Spherical cap geometry used to determine the volume of Hg droplets in the rf-discharge lamps

Fig. 4. Flat-bottomed discharge lamps in DSC

Round bottom lamps were first placed in a standard Perkin-Elmer aluminum calorimeter sample pan contain 25 mg of powdered alumina to facilitate heat transfer to the round bottom lamp and provide support before placing them into the sample and reference ovens. Additional support was provided by an aluminum disk containing holes through which the lamps protruded, thus securing them in place. The ovens were then covered with a cap shown in Figure 4 that contained a milled-out section to accommodate the lamps. The lamps were maintained at -70°C for a duration sufficient to solidify the Hg (MP -39.8°C). Once solidified, DSC scans were then obtained at a scan rates of 5 °C/min for the high mass lamps and 20 °C/min for the low mass lamp over a temperature range of -70°C to -20°C. The areas under the melting transitions were determined using the Pyris software peak integration feature (Figures 5-6). Hg masses in the lamps were computed from these areas and the enthalpy of fusion of Hg (ΔH_{FUS} = 11.47 J/g) and the previously determined multiplicative lamp correction factor that compensates for thermal loss in this unconventional DSC measurement.

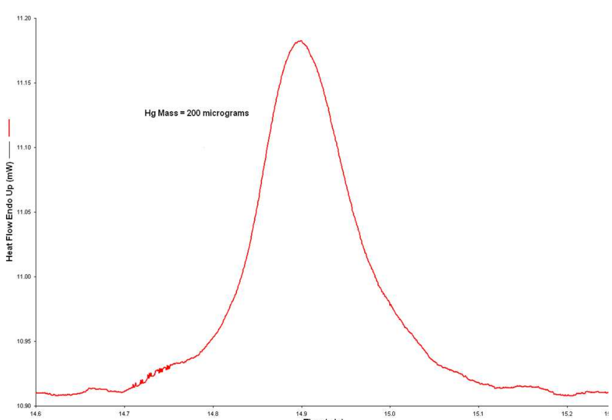

Fig. 5. DSC Measurement of 200 microgram Hg drop

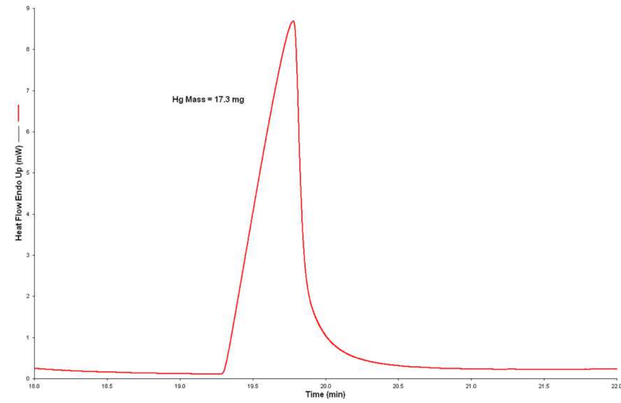

Fig. 6. DSC Measurement of 17.3 mg Hg drop

A comparison of our photographic and calorimetrically determined masses is shown in Table I:

TABLE I. COMPARISON OF PHOTOGRAPHIC AND CALORIMETRICALLY DETERMINED MASSES

Lamp ID	Comparison of Hg Droplet Masses		
	DSC Mass, mg	***Photographic Mass, mg***	***% Difference***
RB1	7.14	5.32	-25.5
RB2	7.20	5.38	-25.3
RB3	9.56	7.74	-19.0
RB4	0.20	0.17	-15.0
FB1	17.15	16.27	-5.1
FB2	6.29	6.28	-0.2
FB3	8.33	7.78	-6.6

[a.] Table of results comparing the Hg droplet masses determined using the photographic method vs. DSC melting scans. The photographic technique consistently indicates a lower mass, suggesting that this method would yield a lower-bound estimate of Hg mass in a lamp production process.

IV. Conclusions

Although imaging through a small diameter cylindrical lamp creates significant optical distortion, we have demonstrated an image analysis method that results in a Hg mass that is within 25% of that derived from a DSC measurement. Lamp curvature produces image distortion that results in dimensional measurement uncertainties that lead to mass uncertainties for the flat bottom lams that are considerably smaller than those obtained for round bottom lamps. We are currently investigating mass measurement discrepancies by photographing ball bearings in lamp ampoules to identify the optimum conditions for measurement. While photographic mass determinations are not as accurate as the calorimetric mass measurement procedure that is currently used to determine rf-discharge lamp fills for Rb and Hg consumption investigations, the simplicity of this photographic method will allow researchers to easily assess Hg mass fills in their rf-discharge lamps studies.

V. Acknowledgement

This work was funded by U.S. Air Force Space and Missile Systems Center under Contract No. FA8802-09-C-0001. The authors thank Drs. Lin Yi and Eric Burt of JPL for many helpful discussions.

References

[1] C.H. Volk; R. P. Frueholz; T. C. English; T. J. Lynch; and W. J. Riley, Lifetime and reliability of rubidium discharge lamps for use in atomic frequency standards, in Proc. 38th Annual Frequency Control Symposium (IEEE Press, Piscataway, NJ, 1984) pp. 387–400.

[2] C.M. Klimcak, M. Huang, and J.C. Camparo, “Alkali Metal Consumption by Discharge Lamps Fabricated from GE-180 Aluminosilicate Glass,” 2015 International Frequency Control Symposium.

[3] C. M. Klimcak, M. Huang, and J. C. Camparo, “Measurement of Mercury Mass in the Deep Space Atomic Clock’s (DSAC’s) rf-Discharge Lamp”, September 1, 2019, Aerospace Report No.. TOR-2019-02557

[4] Precision Glassblowing, 14775 E. Hinsdale Ave., Centennial, CO 80112

[5] ImageJ website: https://imagej.nih.gov/ij/

[6] C.W. Extrand and S.I. Moon (2010) “When Sessile Drops Are No Longer Small: Transitions from Spherical to Fully Flattened”. *Langmuir*, *26*(14), 11815–11822. https://doi.org/10.1021/la1005133