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Measurements of the Thermal Effusivity of a Drop-Size Liquid Using the Pulse Transient Hot-Strip Technique[1]

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The thermal effusivity of drop-size liquids was measured by the pulse transient hot-strip technique. A strip sensor, used as a thermometer and heat source, is deposited on a smooth surface of an electrically insulating background material – onto which an insulating liquid sample is applied, completely covering the strip probe. Experiments can be made controlling the thermal penetration depth to within some 10 μm of the liquid sample – here demonstrated by measuring a drop of water at about 1% uncertainty. Measurements were made on water and a series of silicone oils (kinematic viscosity from 5 to 50 cSt; $1\,\text{cSt} = 10^{-6}\,\text{m}^2 \cdot \text{s}^{-1}$) in microgravity conditions using a 10 m drop tower (10^{-3} g, 1.4 s), to investigate if any potential natural convection in the liquid at normal gravity condition is present, influencing the results. However, no such influence was observed.

KEY WORDS: effusivity; hot strip; liquids; microgravity; micro-sized probes.

1. INTRODUCTION

In the pulse transient hot-strip (PTHS) method [1–4], a hot strip acting as both a thermometer and heat source is deposited on the substrate to be studied. The strip is designed with a width comparable to the probing depth of interest – a probing or thermal penetration depth

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which is typically within the thickness of the sample to be measured. A train of square current pulses generates heat in the strip element, and an ac-coupled circuit [1–5] is used to facilitate recording of the periodic temperature variations of the strip. The technique was originally developed for measurements of thermal transport properties of thin samples and insulating layers, down to the order of 1 μm in thickness.

This technique was recently developed to perform single-sided measurements of the thermal effusivity of insulating liquids, using a hot-strip probe deposited on an electrically insulating substrate of known effusivity [5]. Covering the strip with the liquid sample in this way results in a configuration consisting of two half infinite media and a strip in virtually perfect thermal contact [5, 6]. The strip probe can be calibrated by performing pulse experiments against one or two reference media, making it possible to determine a strip constant and/or the influence of the background substrate [5].

Convection may naturally be present and is an important issue to consider. Here, some experiments on insulating liquids performed in normal gravity conditions are compared with a series of corresponding tests performed in microgravity. A drop-tower facility [7] was used for these tests, where 1.4 s of microgravity conditions is obtained in a capsule during a 10 m free-fall drop. The purpose of these tests is to study if any possible convection influence in the experiments performed in normal gravity can be observed.

2. ELECTRICAL CIRCUIT AND THEORY

An electrical circuit with a large blocking capacitor in series with the strip (nonlinear resistance), a pulse generator as a source, and a digital voltmeter as recorder constitute the measurement setup – in a so-called ac-coupled network configuration [1, 4, 5]. The pulse generator produces repeated square heat pulses, selected to result in transient heating of the strip during typically 5% of the pulse cycle period. In the remaining part of the pulse cycle, the voltage output from the pulse generator is set to zero. Although a small back current passes the sensor during this zero voltage output, the strip temperature will decrease to its initial or near initial temperature at the end of each pulse cycle.

The ac-coupled network is designed to operate in the following way: with linear components in the circuit, the time-averaged voltage output over a number of full periods will be zero. However, by introducing a nonlinear resistance in the ac-coupled network – such as the hot-strip element – the output voltage will be non-zero. In the PTHS technique the time-averaged temperature variations of the strip can be determined from the

non-zero voltage reading. By studying the voltage response for different pulse trains, the thermal transport properties can be deduced.

In the original PTHS theory, a general model correlating the average voltage reading over a number of full periods n and a pulse duty cycle F can be expressed as [1, 4, 5]

$$\overline{\Delta U_n} = C\Phi\left(n, F, \frac{P}{\theta}\right). \tag{1}$$

Here, the average voltage is directly related to the thermal conductivity property – included in the constant C – and the thermal diffusivity is included in the dimensionless "time" function Φ. The time to record the transient temperature response is typically of the same order of magnitude as the so-called characteristic time of the strip probe, $\theta = d^2/a$, where a is the thermal diffusivity of the substrate and $2d$ is the strip width. The ratio P/θ relates the period P to the characteristic time of the strip probe, θ.

It has been experimentally observed that the average voltage reading quickly stabilizes around a stationary value independent of n [1, 5]. If conducting an experiment by applying 20–30 different trains of square pulses, using the same pulse height U_p and duty cycle F, the average voltage will only depend on the period P.

A simplified model of Eq. (1), valid for shorter times, can easily be extended to the double-sided case with a liquid sample covering the strip and the solid substrate [5]. For pulse times $FP < \theta/2$,

$$\overline{\Delta U} \cong \frac{AU_p^3}{E_{1,n} + E_{2,n}} g(P), \tag{2}$$

where

$$g(P) = b_1\sqrt{P} + b_2\frac{\sqrt{Fa}}{d}P \tag{3}$$

and $A = \alpha R_s R_0^2 (1-F)^2 F^{3/2}/(R_s + R_0)^4 2\pi^{1/2} hd$ is a constant. Here, $E_{1,n}$ and $E_{2,n}$ are the thermal effusivity of media 1 and 2, respectively, defined in the normal direction with respect to the strip plane, and $b_1(F) = 0.438232$ and $b_2(F) = 0.141047$ for $F = 0.05$. Included in the constant A are the strip properties: the temperature coefficient of resistivity α, the initial strip resistance R_0, and the strip length $2h$, as well as the pulse generator internal resistance R_s.

The strip constant A and the background effusivity can be determined in separate measurements, or by conducting a couple of pulse experiments against one or more well-defined reference media [5].

3. EXPERIMENTS

3.1. Experimental Design

The first design criterion is the selection of the strip dimensions. Typically, the width of the strip is selected to obtain a desired probing depth in the sample material. The probing depth in the present experiments is less than the strip width $2d$. The strip length $2h$ for the original PTHS technique is typically selected much longer than the strip width, to avoid end-contact influence and to obtain an essentially two-dimensional (2D) heat flow around the strip – suitable for the 2D analytical model used in analyzing experimental data [1–4]. The pulse lengths are selected from typically $FP = 0.01\theta$ up to and including $FP = \theta = d^2/a$, in order to obtain maximum sensitivity in the estimation of the thermal diffusivity [1, 4, 8, 9]. The strip width must also be well defined for the original PTHS technique. In the present tests, however, the strip length criteria and the requirement for a well-defined strip width can be somewhat relaxed, as here the technique is used for shorter times than is typical for the ordinary PTHS technique, $(FP)_{\max} < \theta/2$. This is done to obtain a heat flow around the strip in which the 1D heat flow in the normal direction from the strip plane dominates the temperature response [5]. A strip length, at least 10 times longer than the strip width, is sufficient using the present variant of the technique. The strip dimensions are not only dependent on the desired probing depth, but also on limitations of the experimental equipment. For instance, pulse periods P longer than the time constant of an averaging low-pass filter result in unstable voltage readings. The strip material and manufacturing method should be selected making it possible to design a sensor with a strip resistance R_0 similar to the pulse generator internal resistance [4], $R_s = 50\,\Omega$, while the sensor thickness can be kept significantly smaller than the strip width to suppress or preferably eliminate the strip heat capacity influence [4].

A background substrate with a smooth surface is required. For optimal sensitivity, the effusivity of the background substrate should be significantly lower than the liquid sample being measured [5]. Also, the background effusivity property needs to be well-defined. As anisotropy may be present for a solid; this should be considered in estimating the background influence [5]. Likewise, the issue of homogeneity in the background substrate surface should also be taken into account.

The issue of the thermal contact resistance of deposited layers and solid–solid boundary resistance is discussed elsewhere [10, 11], and is negligible in the present tests. If using an evaporation or sputtering deposition technique to produce the strip, it should be noted that the temperature coefficient of resistivity α will not follow tabular data for the bulk material of the strip, due to the effects of annealing [4].

The pulse height U_p is suitably selected in order to give a required temperature response, typically of the order of 1–10 K, for a single pulse.

3.2. Natural Convection

Natural convection may occur in liquid thermal conductivity experiments, particularly if the viscosity of the liquid is low. It is important to consider this phenomenon when exercising experimental design and analyzing experiments. The issue of convection in liquids is addressed by some researchers by making similar tests in microgravity- (μ-g) and normal gravity (1-g) conditions, an approach used here. An analysis that considers influence from natural convection is motivated by the long heating period for which the pulse train is applied to the probe, as the onset of natural convection may occur during the present experiments of low viscosity liquids [5].

3.3. Tests in Normal Gravity

Experiments were recently conducted on a series of insulating liquids such as water and silicone oils of viscosity between 5 and 3000 cSt (1 cSt = $10^{-6}\,\mathrm{m^2 \cdot s^{-1}}$) at room temperature conditions, using this modified technique [5]. In 1-g, water was reproduced within 2.1% in two separate tests, and the silicone oils were reproduced within 3.4% average deviation from available tabular data (tabular results were only available at a temperature approximately 10°C higher than the measurement temperature). No systematic deviation was observed between the results obtained with this technique and bulk reference data.

Two hot-strip probes were used in these tests [5]; probe 1 – an evaporated gold film on a polymethyl methacrylate (PMMA) background (length $2h = 1.27$ mm, average width $2d = 41.9\,\mu$m, initial resistance $R_0 = 73.9\,\Omega$, strip constant $A_1 \cong 0.283\,\mathrm{s^{1/2} \cdot m^{-2} \cdot K^{-1} \cdot \Omega^{-1}}$, and background effusivity $E_{\mathrm{PMMA},n} = 573\,\mathrm{J \cdot m^{-2} \cdot K^{-1} \cdot s^{-1/2}}$), probe 2 – a sputtered gold film on a glass background (length $2h = 2.08$ mm, average width $2d = 67.4\,\mu$m, initial resistance $R_0 = 85.1\,\Omega$, strip constant $A_2 \cong 0.111\,\mathrm{s^{1/2} \cdot m^{-2} \cdot K^{-1} \cdot \Omega^{-1}}$, and a background effusivity $E_{\mathrm{glass},n} \cong 1510\,\mathrm{J \cdot m^{-2} \cdot K^{-1} \cdot s^{-1/2}}$). The surface roughness of PMMA and glass background was of near mirror quality and mirror quality, respectively.

3.4. Tests in Microgravity

Five of the lower viscosity liquids tested with this technique were also investigated in μ-g conditions, obtained using a drop-tower facility (10^{-3} g, 1.4 s) [7]. Any steady natural convection flow that might exist prior to the

drop is suppressed towards a zero-flow condition [7] – here assumed to occur instantly when the capsule is dropped [12].

As we have 1.4 s available in μ-g conditions, an experiment is conducted by initiating the pulse generator some 4–5 s prior to the drop, allowing the average voltage signal to first settle. The voltage recorder is initiated shortly after dropping the capsule, and the first experimental point of a 30-s series of recordings is measured in μ-g where no convection exists. Each voltage point is recorded at approximately 1-s intervals using a 250 ms time-averaging integration time. Figure 1 displays a series of voltage recordings where the first point is obtained in μ-g.

In order to study the influence from convection while avoiding any voltage shifts that may exist from experiment to experiment, the deviation of the first point obtained in μ-g is compared with the average voltage for the same experiment. These deviations are then plotted in the voltage versus $g(P)$ graphs for the experiments performed in 1-g. If the apparent effusivity would be different for the points obtained in μ-g and 1-g, as a result of possible convection at 1-g, the slopes would differ in the graphs. Figures 2–4 show the experiments of water and a couple of different silicone oils, including points obtained in μ-g. It should be noted that a single data point will necessarily have larger scatter than the 30-s aver-

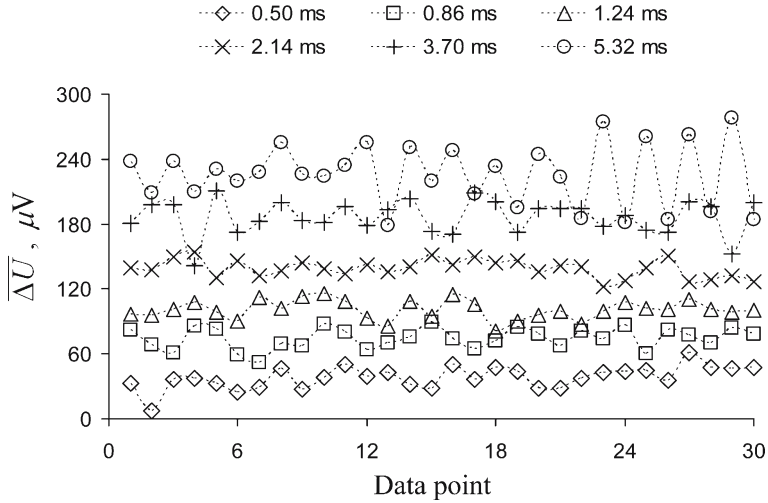

Fig. 1. Voltage recording over approximately 30 s using probe 1, measuring a drop of water. The pulse height is 3.0 V, and the pulse period is indicated in the figure. The first point in the five experiments is measured in μ-g, and they are included in Fig. 2. This recording was made using a digital voltmeter (Keithley DVM2000) preceded by two filters, time-averaging frequency signals above 250 Hz.

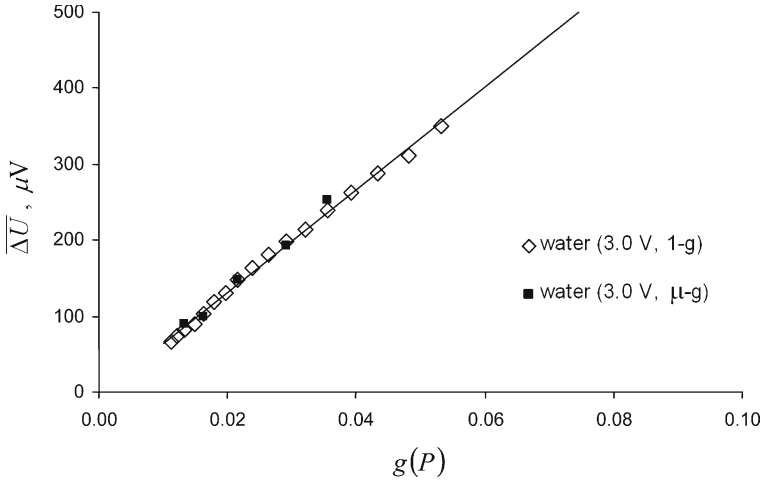

Fig. 2. Average voltage recordings versus $g(P)$, using probe 1 and a 4 ms low-pass filter, measuring a drop of water. The pulse height is 3.0 V, and the marked points represent the corresponding short-time recording in μ-g. The estimated effusivity here is $1597\,\mathrm{J\cdot m^{-2}\cdot K^{-1}\cdot s^{-1/2}}$, a deviation around 1% from tabular reference data [5].

aged voltage value. In particular, as the instability of the voltage signal was found to increase with longer pulse lengths P, it was not meaningful to include experiments of points for pulse lengths longer than the time constant of the filter. On the other hand, the time constant of the filter should not be long, 4 or 10 ms in the present tests, in order to allow a quick response from the instant the capsule is dropped until the first point is measured in μ-g.

In the test on water, it was possible to apply only a single drop to the probe, just covering the sensor. The thermal effusivity of water was reproduced in this test to within 1% compared to tabular reference data at 1-g, using a thermal sensing depth or a probing depth [1] of only $d_{p,\max} \approx 2\sqrt{a_{\text{sample}} F P_{\max}} \cong 17\,\mu\text{m}$, corresponding to a sample weight of less than 1.7 μg, cf. Fig. 2.

4. RESULTS AND DISCUSSION

Often the influence from natural convection in a transient experiment can be observed, typically as a clear deviation when curve fitting a model against experimental data. By studying the measurements performed at 1-g and μ-g in Figs. 2–4, as well as similar experiments on 30 and 50 cSt silicone oils (measured using probes 1 and 2, respectively), it is not

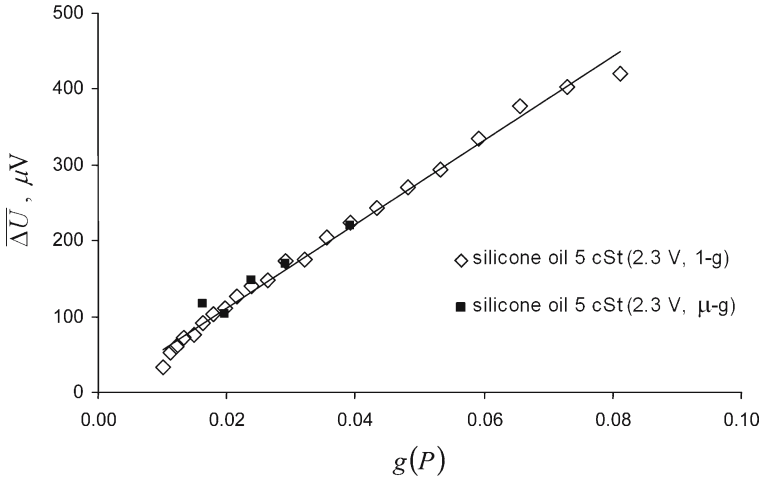

Fig. 3. Average voltage recordings versus $g(P)$, using probe 1 and a 10 ms low-pass filter, measuring silicone oil (5 cSt). The pulse height is 2.3 V, and the marked points represent the corresponding short-time recording in μ-g. The estimated effusivity here is $391\,\mathrm{J \cdot m^{-2} \cdot K^{-1} \cdot s^{-1/2}}$, a deviation of 9.8% from tabular reference data [5].

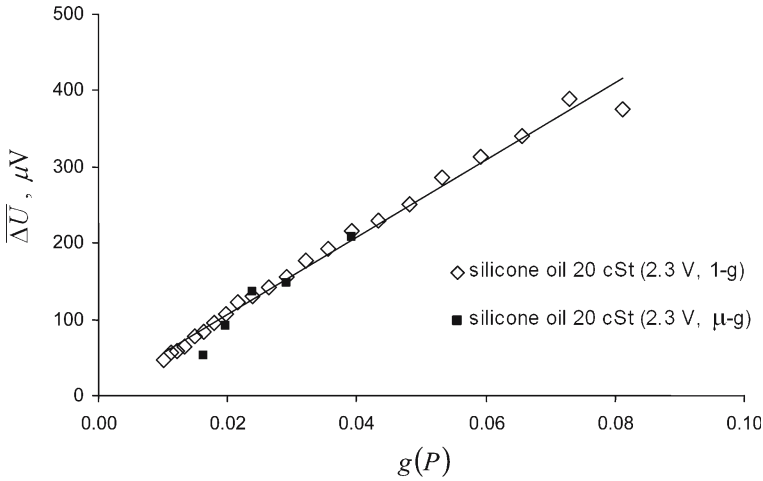

Fig. 4. Average voltage recordings versus $g(P)$, using probe 1 and a 10 ms low-pass filter, measuring silicone oil (20 cSt). The pulse height is 2.3 V, and the marked points represent the corresponding short-time recording in μ-g. The estimated effusivity here is $482\,\mathrm{J \cdot m^{-2} \cdot K^{-1} \cdot s^{-1/2}}$, a deviation of 1.2% from tabular reference data [5].

possible to observe and ascertain any systematic deviations. From this observation, it is tempting to conclude that these results indicate there is no natural convection in the 1-g experiments.

However, in order to make any firmer conclusions, the following three aspects should also be considered:

- *Sensitivity of the recorder.* By measuring over a longer period of time, the average reading becomes stable. For the present μ-g experiments, we do not have this ability of averaging over several data points. A more sensitive recorder might reduce the scatter somewhat and make it easier to detect possible changes in the apparent effusivity. It can be observed in this context, however, that although a certain amount of scatter is observed in Figs. 2–4, there is no indication of any increased scatter in the first point measured in μ-g condition as compared with the following variations observed, cf., e.g., Fig. 1 – an observation that also adheres to the next points.
- *Time response to a sudden change in apparent effusivity.* In order to detect a sudden change in the apparent effusivity, the ac-coupled network circuit and the time-averaging low-pass filters preceding the recorder must be able to settle at the new value within a rather short time. With regard to the electrical circuit and the recorder, there are no time constants too long to give unreasonably long settling times.
- *Will the apparent liquid effusivity change if there is a convective flow?* The question is relevant since it has previously been shown that down-scaling a probe to micro-size reduces the relative errors caused by radiation [6], and recently it was shown that the same reasoning also applies to relative errors caused by convection [5]. Down-scaling a transient experiment by a factor of 100 in geometry typically reduces the experimental time scale by a factor of 10^4. Assuming a convective flow disturbing the experiment with a convective heat loss $\dot{Q}_{\text{conv}}$ during a pulse, the relative error [5] can be expressed as the ratio,

$$\frac{\dot{Q}_{\text{conv}}}{\dot{Q}_{\text{cond}}} \cong \frac{2\beta}{\pi^{1/2}\lambda} f(\tau)\, d, \tag{4}$$

where $\dot{Q}_{\text{cond}}$ is the heating of the liquid by conduction, λ is the thermal conductivity of the liquid, β is the convective heat transfer coefficient, and $f(\tau)$ is a dimensionless time function, varying from 0 up to approximately 0.5 during a pulse. As seen in Eq. (4),

the relative error depends on the strip width $2d$, and this convection error can be suppressed by reducing the strip width. Equation (4) has an equivalent formulation – in terms of the longest pulse time $t_{\max} = (FP)_{\max} \approx \theta/2 = d^2/2a$;

$$\frac{\dot{Q}_{\text{conv}}}{\dot{Q}_{\text{cond}}} \cong \frac{2\beta}{\pi^{1/2}\lambda} f(\tau) \sqrt{2a\,(FP)_{\max}} \tag{5}$$

indicating that a reduction in the time scale by several orders of magnitude will significantly reduce the influence from convection.

From these additional observations, we conclude that we are not able to see any influence from convection in our experiments, yet we cannot rule out the existence of convection at 1-g. In any case, the pulse lengths FP used, typically 25–1000 μs in the present experiments, should according to Eq. (5) effectively suppress any influence from convection to levels undetectable when using this technique.

The thermal effusivity is for the present double-sided experimental configuration simpler to determine accurately than either the thermal conductivity or thermal diffusivity separately. It can be noted that from this technique, it is possible to map the thermal conductivity and thermal diffusivity if the specific heat c_p and the density ρ are determined in separate experiments.

5. CONCLUSIONS

The PTHS technique was recently extended for thermal effusivity testing of insulating liquids, requiring a sample of only a drop size. No systematic deviation in the results of micro-sized samples and bulk reference data was observed when comparing experiments on low-viscosity liquids performed in normal- and microgravity conditions.

The advantages of this technique can be summarized as follows:

- The sample size can be substantially reduced and still maintain results in agreement with macroscopic bulk data.
- Relative errors from radiation and convection are reduced by shortening the experimental time scale by several orders of magnitude.
- A stationary voltage is obtained, which is generally easier to record than a transient temperature response.
- A relatively simple model can be used, simplifying the data analysis.

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