The Measurement of Surface Temperatures With Platinum Films During Nucleate Boiling of Water

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This report describes a simple and reliable method of constructing small platinum film thermometers on Pyrex glass. The thermometers were used to measure surface temperature during nucleate boiling of water at pressures between 1 and 4 lb./sq.in. abs. The thermometers showed that as superheated microlayers of water evaporated beneath vapor bubbles, the surface temperature often dropped 40° to 75°F. before ultimately reaching the saturation temperature. The thermometers proved to be accurate ($\pm 1^{\circ}$ F.) and capable of fast response (145°F./msec.).

Many recent experiments in nucleate boiling have focused upon surface temperature fluctuations beneath bubbles (3, 4, 7, 8, 12, 13 to 15). A very interesting result of the experiments is that when a bubble grows on a heated surface, a film of liquid forms under the bubble. In at least some instances, bubble growth results primarily from the evaporation of the liquid film or microlayer (3). High-speed photographs have shown that a heated surface cools during bubble growth and recovers during bubble departure (4, 8, 14). As the microlayer evaporates, the surface temperature beneath a vapor bubble decreases very rapidly. Yet, the magnitudes and durations of the decreases vary considerably among experiments. For example, Moore and Mesler (12) boiled water at atmospheric pressure; they observed temperature drops of 20-30°F. in 2 msec. using a Chromel-Alumel thermocouple. Bonnet et al. (3) measured temperature drops of 15° to 20°F. that lasted for 4 to 6 msec. Hendricks and Sharp (8) boiled water on a metal ribbon at reduced pressures, and they observed drops of 50° to 60°F. in 10 to 20 msec.

Almost all experiments have utilized extremely small thermocouples as temperature sensors. Each thermocouple was usually mounted in another metal such that the thermocouple was very close to the boiling surface. As a result, information is still lacking about nucleate boiling on nonmetals, except for one recent paper. Cooper and Lloyd (4) mounted four semiconductors on glass, 0.01 to 0.06 in. apart, and recorded surface temperature fluctuation of 25° to 50°F. under toluene bubbles. They showed that the surface temperature fluctuations depend upon the position of the surface relative to the bubbles. The temperatures fell and recovered in 5 to 15 msec. under the centers of the bubbles and in 50 to 70 msec. at the outer edges of the bubbles. They offered credible evidence that their liquid films completely evaporated in many instances. However, contrary to what one would expect, not one of their temperature recordings reached the saturation temperature; the minimum surface temperatures were 2° to 20°F. above the saturation temperature. Others (3, 8, 11) report similar findings using metal surfaces. This is disturbing because elementary considerations would suggest that the surface temperature drop to the saturation temperature as the last bit of liquid microlayer evaporated.

The purpose of this experiment was to determine the

minimum surface temperatures during nucleate boiling of water. Water was chosen as the boiling liquid because of its predominance in other experimental work. It was felt that a nonmetal surface such as Pyrex glass would be the best boiling surface because of its low thermal conductivity. The use of a surface with a very low thermal conductivity provided more time to observe the temperature drops by throttling the removal of heat from the solid (15). The working pressures were subatmospheric in order to promote large, single bubbles that were large in relation to the size of the temperature sensors (4, 8, 15) vs. (9, 13).

An auxiliary purpose of this study was to develop an easy method of fabricating an accurate, small, fast-response sensor for use with water. It became evident that some sensor other than a thermocouple would have to be used with a Pyrex glass. A thin-film resistance thermometer was a likely alternative if it could be appropriately mounted on Pyrex. Cooper and Lloyd (4) attached multiple semiconductor sensors to glass by vapor deposition through a mask with an intricate technique requiring extensive equipment. One of the sensors was used to initiate bubble growth. They used a two lead resistance measuring technique in which they had to maximize the resistance of the sensor and minimize the resistance of their leads. The fluid boiled had to be a poor electrical conductor which limited them to several organic liquids, and they used

For this study a four lead technique was chosen. This technique allows a lower sensor resistance and relaxes the restrictions on electrical resistivity of the fluid boiled permitting water to be boiled. Lower resistance films can be easily made by applying platinum paint to glass. Such films have found wide use in temperature gauges for shock tunnels but have not been used previously in nucleate boiling.

THEORY IN THE USE OF RESISTANCE THERMOMETERS

Metal films can be used profitably to measure surface temperature because they can be very sensitive to local temperature changes. Figure 1 depicts a thin metal film ABĈD with film leads BE and CF mounted on a Pyrex glass. The length of film BC is the temperature-sensing site (TSS) of the film: Temperature fluctuations produce changes in electrical resistance that cause voltage fluctuations across BC. The high-impedance oscilloscope can monitor the voltage fluctuations across the TSS. The offset

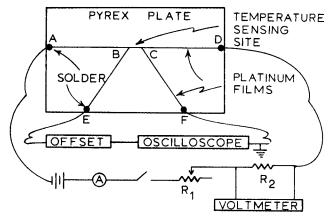


Fig. 1. Schematic diagram of a circuit with a metal film as a temperature sensor.

is a constant-voltage source that opposes the TSS reference voltage (voltage at a particular reference temperature) so that the oscilloscope will amplify voltages about the reference voltage. The voltage source V can be considered constant for large resistive loads, and the external carbon resistor R_1 is very large compared with the film resistance. Under these conditions, the current measured by the ammeter A or with the voltmeter, which measures the voltage across the constant resistance R_2 , will be essentially constant for temperature changes of less than a few hundred degrees near the TSS. Grounding the negative lead of the oscilloscope helps reduce electrical noise. The objective is to have a bubble grow over the TSS so that the oscilloscope can indirectly record the temperatures at the solid-fluid interface.

With a constant current through the TSS, a first-order approximation to the change in voltage across the TSS is

$$\Delta E = i\Delta R \tag{1}$$

The resistance of most metals, including thin films, is approximated by (2, 17)

$$R = R_o[1 + \alpha(T - T_o)] \tag{2}$$

The temperature coefficient of resistance (TCR) is a property of the metal, and it depends upon the composition and thickness of the metal among other things. By using Equation (2), Equation (1) becomes

$$\Delta E = i\alpha R_o \Delta T \tag{3}$$

Since the voltage can be displayed on an oscilloscope as a function of time, Equation (3) can be rearranged to express the temperature as a function of time t:

$$T(t) = T_o + [\Delta E(t)]/(i\alpha R_o)$$
 (3a)

Equation (3a) gives the temperature of the sensor; it also gives an accurate indication of the true surface temperature, provided the sensor is very thin. This is implicit when one considers that the temperature transient to be measured should be slow compared with the time of diffusion of heat through the film. The theoretical work of Kurzrock (10) relates the response time of a thin film to the film thickness for the case of a constant heat flux. When Pyrex and platinum are used, the platinum should be less than 1μ thick for a response time of approximately 1 msec. That is, the temperature rise at the interface between a 1µ thick platinum film and a Pyrex mounting is 95% or more of a Pyrex surface temperature with no film after 1 msec. has elapsed. After 1 msec., the temperature of the platinum film and the surface are essentially the same. One can surmise the relative importance of the sensor thickness for the varying heat-flux case. In this study the surface coverage of the TSS is also important. When the surface area is small, the TSS will act as a point (as opposed to an area) sensor; when the surface coverage is large, the site will indicate an average of the point temperatures.

PROCEDURE TO CONSTRUCT THE SENSORS

The procedure for fabricating a sensor was similar to that outlined by Vidal (17). The general technique was to clean a Pyrex plate, to apply a solution of Hanovia liquid platinum with a Leroy lettering pen, to bake the plate and film, and to attach leads to the platinum films. For convenience, the Pyrex plates were 5 in. sq., and thicknesses of 1/8, 3/16, and 1/4 in. were used. After each plate was washed, platinum films were drawn on one side with the aid of Leroy pens. The use of pens, sizes 000, 0, 2, 3, and 4, was just a variation in the fabrication technique of thin film thermometers (6, 16, 17). Most of the TSS's were drawn with one stroke of a pen, while others were overstroked two to four times. Overstroking was necessary at times to insure electrical continuity.

The plates were then baked in a furnace with the door open to provide good ventilation (17). At 900°F. the door was closed; when the temperature was between 1,100° and 1,300°F., the plates were removed from the furnace so they would not warp. After the furnace cooled a few minutes, the plates were returned to anneal at 500° to 800°F. for 2 to 3 hr. so that the resistance would stabilize (1, 2). They were then air cooled at room temperature. Copper leads were then bonded to the films. Of the many circuit and brazing paints, solders, and fluxes that were tried, only one combination gave satisfactory results: a 50-50 solid solder with a flux solution of 18 g. of ammonium chloride and 42 g. of zinc chloride in 100 g. of water. The TSS's of the films were calibrated by determining their resistances as functions of temperature; the circuit was like that in Figure 1 with a differential voltmeter in place of the oscilloscope and offset.

PROCEDURE TO MEASURE THE SURFACE TEMPERATURES

Figure 2 is a schematic representation of the experimental apparatus. The heart of the assembly is the Pyrex plate with the $3.0 \times 3.8 \times 6.0$ in. Plexiglass tank (TK). A silicone gasket separates the tank and Pyrex plate. The burner (B) beneath the Pyrex is merely a glass capillary tube with an air and gas distributer. The pressure in the tank is controlled by the train of vacuum equipment: vacuum pump (P), manostat (MS), pressure release valve (PR), manometer (MM), drying tower (DT), and condenser (C). The potentiometer (PO) and the calibrated thermocouple (T) indicate the saturation conditions of the water more accurately than does the manometer. A 45 v. battery (V) provides a d.c. voltage source. The voltage drop across R2, measured with a Keithley 660A differential voltmeter (DV), can give an accurate value of the current. The oscilloscope (OS), a 531 Tektronix base with a 1A7 differential amplifier, senses voltage fluctuations across the TSS and displays these fluctuations. The offset (OF) matches the reference voltage across the TSS (voltage at the reference temperature) so that the oscilloscope actually displays voltage changes about the reference voltage. It is possible to synchronize the growth of bubbles with the oscilloscope displays by using a 16 mm. Fastex WF-17 camera (CA). The time-mark generator (TG) gives a pulse of light every millisecond, and

| TSS | Pen size | Film width, in. | TSS resistance, R_0 , ohm | $\alpha = \text{TCR}$ $^{\circ}\text{F.}^{-1}$ | α R ₀ , ohm/°F. | Minimum TSS length, in. | Maximum TSS length, in. |
|----------------|----------------|--------------------|-----------------------------|--|-------------------------------|-------------------------|-------------------------|
| 1 | 2 | 0.023 | 20.726 | 0.00124 | 0.0257 | 0.112 | 0.154 |
| $\overline{2}$ | $\bar{2}$ | 0.023 | 17.174 | 0.00138 | 0.0237 | 0.080 | 0.134 |
| 3 | $\overline{2}$ | 0.024 | 14.737 | 0.00114 | 0.0168 | 0.195 | 0.281 |
| 4 | 0 | 0.018 | 7.742 | 0.00139 | 0.0108 | 0.029 | 0.080 |
| 5 | 0 | 0.018 | 11.683 | 0.00131 | 0.0153 | 0.058 | 0.115 |
| 6 | 000 | 0.013 | 135.88 | 0.00137 | 0.186 | 0.202 | 0.225 |
| 7 | 000 | 0.013 | 39.35 | 0.00141 | 0.0553 | 0.066 | 0.084 |
| 8 | 0 | 0.018 | 8.752 | 0.00141 | 0.0121 | 0.021 | 0.084 |
| 9 | 0 | 0.018 | 7.042 | 0.00142 | 0.0100 | 0.039 | 0.091 |
| 10 | 0 | 0.018 | 5.251 | 0.00140 | 0.00733 | 0.150 | 0.084 |
| | | 0.018 | aritl | hmetic average | | 0.095 | 0.133 |

the variac (VA) controls the camera speed at less than 5,000 frames/sec. Two 750 w. lamps (L) illuminate the bubbles.

The procedure for determining the surface temperatures was to fill the tank with demineralized water, to close the circuit and determine the current, to establish the reference voltage, to degas the water for 30 min. by heating in a vacuum, to adjust the instruments, and to take films of the growing bubbles and the corresponding voltage fluctuations. The thermocouple voltage and the voltage across R_2 were recorded before and after each run, while the offset voltage and oscilloscope drift were checked frequently. The process was repeated at other pressures and then at the original pressure again. Several Polaroid photographs of the oscilloscope traces were also taken to supplement the information recorded on the movie films.

Heat was applied near the center of the glass plate so that bubbles grew over the TSS and appeared often enough to permit photography. Thinner plates were advantageous in minimizing a natural tendency for bubbles to nucleate at the gasket. Sometimes nucleation occurred near the platinum film, but this did not seem to make a difference in the temperature behavior. The natural nucleation sites used in this study contrast with the artificial initiation of bubbles as practiced by Cooper and Lloyd (4).

EXPERIMENTAL RESULTS AND DISCUSSION

The calibration results for the ten TSS's that were later used in boiling are in Figure 3 and Table 1. Varying the current from 1 to 8 ma. did not affect the resistances, and the resistances did not change during a 2 mo.

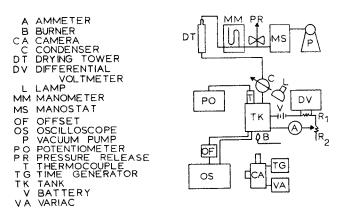


Fig. 2. Schematic diagram of the experimental apparatus.

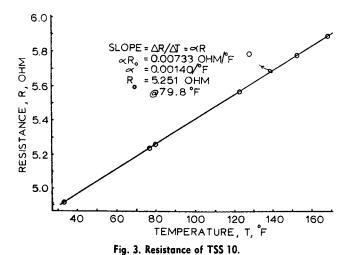
testing period. The slopes of the resistance vs. temperature curves were constant for each TSS. Some of the TSS's covered relatively large surface areas, but others still met the needs of this experiment. Films with large resistances were preferred because they were generally thinner and produced larger voltage changes for a given temperature change. The TCR's were nearly constant, and they apparently did not depend on the pen size. Others report TCR's of 0.000927 to 0.001758/°F. (6) and 0.00125/°F. (17) for Hanovia No. 05-X platinum solutions. Variances in the TCR were probably due to different film thicknesses and differences in the amounts of metal oxides in the films (2).

Pyrex plates that were heated beyond 1,175°F. were dull and did not adhere well to the Pyrex.

The films were not uniform in thickness as was evident by the amount of light that different sections transmitted (2). In order to determine the thicknesses of the gauges, attempts were made to crack the gauges and obtain crosssectional views of the film with a compound microscope. The attempts were not successful because of diffraction at the plate edges and problems in mounting the specimen. By using a large Bausch and Lomb metallograph, it was possible to alternately focus on the center of the film and then on its edges. It was evident that the films were definitely less than a few microns thick. Finally, after cross sections of a plate and film were cut with a diamond pointed saw, two test specimen were mounted in a Bakelite cast and polished. The maximum thickness of both films, which were drawn with five and eight overstrokes of a No. 2 pen, was less than 1μ. Therefore, most of the TSS's should have been less than 1μ thick because they were drawn with fewer strokes and normally with smaller pens. Vidal (17) suggests that films made from the No. 05-X platinum liquid can be less than 0.1μ thick.

The movie films and photographs were first culled. Of the fourteen films and sixty-three photographs that were taken, eight films and twenty-seven photographs were considered useful and reliable, and they were examined in detail. Figures 4 and 5 are representative temperature histories. The first temperature drop in Figure 4 commenced when a bubble nucleated; the surface temperature continued to drop as the microlayer of trapped water evaporated.

Another important observation can be made from Figure 4. Immediately after the first bubble lifted from the surface, and 80 msec. after the previous minimum temperature, a second bubble appeared. The surface temperature again dropped, though much faster (145°F./msec.) than the first time. A second temperature drop, significantly faster than the first, was frequent at 1 and 2



lb./sq. in. abs. but not at higher pressures. The minimum temperatures of the second drops were never less than the corresponding minimum temperatures of the initial drops. The photographs showed that the abrupt halt in the second temperature drop was possibly due to an increase in the microlayer thickness as a result of considerable turbulence at the bubble base. Further examination revealed that the second bubble did not grow from beneath the first bubble's tether. (Each bubble formed a tether as it rose from the Pyrex surface.) Conversely, much smaller temperature drops were observed when second and subsequent bubbles grew from beneath the first bubble's tether.

Temperature histories of two successive bubbles are shown in Figure 5; the time between bubbles was 1.9 sec. The reference time was arbitrary, and it was selected in order to show the similarities in the curves. At the time of nucleation, the surface temperatures at the TSS were about 195°F. in both cases. The initial temperature drop of the first bubble lasted 40 msec. At that time the minimum surface temperature was 149°F; the saturation temperature was 148.9°F. An error analysis indicated that the experimental temperatures were accurate within ±1°F. Thus, within the experimental error, the minimum surface temperature did reach the saturation temperature for these two bubbles. Similar results were obtained over the experimental pressure range of 1.0 to 3.8 lb./sq. in. abs. but more frequently for slower temperature drops than for fast ones. The initial temperature drops of 40° to 75°F. that lasted 9 to 80 msec. were observed at these pressures. When the surface temperature did drop to the saturation temperature, the photographs normally showed that the temperatures started to rise while the bubbles were still covering the TSS (the outlines of the bubbles at the surface were not always well defined during the last stages of growth.) With the bubble covering the TSS, the only explanation for an increase in the surface temperature is that the microlayer evaporated completely. All of the bubbles were initially hemispherical, and they often grew to be so large that they filled the tank.

Not all of the minimum temperatures reached the saturation temperature. Only when all of the bubble covered the TSS and, simultaneously, when the microlayer completely evaporated should the two temperatures have been the same. It is emphasized that the platinum films were small and thin enough to record the saturation temperatures when these two simultaneous conditions occurred.

The current and offset voltage were essentially constant during all of the runs; they never changed more than 0.38 and 0.02%, respectively. These two factors and

others were incorporated in the error analysis. There was no indication that the water conducted enough electricity between the platinum films to introduce a significant error. The resistivity of the water was determined before and after each series of runs, and it was always greater than 5.4×10^5 ohm-cm. at 75° F. It should be noted the approach to saturation temperature was consistently shown by several different thermometers with varying values of αR_0 . This gives added confidence in the validity of Equation (3a) and the whole technique of temperature measurement.

A theoretical model was used to predict the surface temperature during the cooling produced by the evaporating microlayer. The model assumed the evaporation of a uniformly thick microlayer on a thick solid base all at constant initial temperature. The unsteady state heat conduction equation was applied to both the liquid microlayer and solid and solved on a GE 625 computer using finite-difference methods. The evaporating face of the microlayer was assumed to drop to the saturation temperature and remain there until vaporization of the microlayer was complete. By starting with an assumed microlayer thickness and initial temperature, the surface temperature history and time for complete evaporation was calculated.

Three methods can be used to estimate microlayer thickness. Two methods depend upon estimating the heat transferred and equating this to the thickness of a microlayer of the same latent heat content. One estimate of the heat transferred can be obtained by calculating the temperature change in the solid and the corresponding reduction in enthalpy. Another method of estimating the heat transferred is to calculate the heat flux across the microlayer by assuming that the evaporating face of the microlayer is at saturation and the other is at the surface temperature. Cooper and Lloyd (4) used both of these methods and report the two methods gave comparable results. Others (3, 8, 12, 13) have used only the former method.

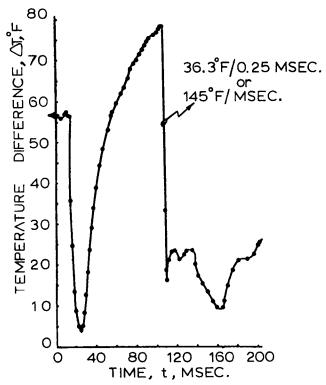


Fig. 4. Temperature history of film 5 (TSS 8) at 1.08 lbs./sq.in.

TABLE 2. THEORETICAL MICROLAYER THICKNESSES

| | | | r Saturation pressure, lb./ sq. in. abs. | | Initial surface temp., °F |
|---|----|-----|--|-------|---------------------------------|
| 1 | 26 | 160 | 3.63 | 149.0 | 201 |
| 2 | 14 | 130 | 1.04 | 103.0 | 163 |
| 3 | 19 | 140 | 2.06 | 127.3 | 177 |
| 4 | 41 | 160 | 1.02 | 102.4 | 175 |
| 5 | 75 | 370 | 1.04 | 103.0 | 171 |
| 6 | 16 | 130 | 2.34 | 131.9 | 190 |
| 7 | 40 | 170 | 3.62 | 148.9 | 195 |
| 8 | 67 | 350 | 1.98 | 125.8 | 175 |
| 9 | 30 | 240 | 3.67 | 149.5 | 186 |
| | | | | | |

Still another method is to use the above model and take as the microlayer thickness the thickness which the model predicts will evaporate in the observed time. This method depends primarily on the observed initial surface temperature and time for evaporation and does not depend on the exact temperature history. This seems to be a significant advantage, so this last method was adopted here. Comparisons between this last method and the others showed differences of from 3 to 19%.

One of the theoretical histories is shown in Figure 5 for a microlayer thickness of 170 μin. An evaporation time of 40.5 msec. was predicted. Estimates of microlayer thickness by the other two methods were 150 and 170 µin., respectively. The theoretical temperature dropped faster than the observed temperatures, possibly because the sensors were measuring a temperature averaged over an area where evaporation did not begin at the same time and perhaps microlayer thickness varied. This disagreement early in the temperature history influenced the selection of the third method of computing microlayer thickness.

Table 2 gives results of analyzing nine bubbles. Evaporation times ranged from 14 to 75 msec. Initial superheat varied from 37° to 73°F. The microlayer thickness varied from 120 to 370 μ in. Six of the nine thicknesses fall between 120 and 170 μ in. There is no noticeable trend between microlayer thickness and the other variables.

The values for microlayer thickness found here may be compared with the following ranges reported by others: Cooper and Lloyd, toluene, 1 to 2 lb./sq. in. abs., 200 to $800 \mu in.$; Moore and Mesler, water, atmospheric 78 to 89 μin.; and this report, water, 1 to 4 lb./sq. in. abs., 130 to 370 μ in. At the lower pressures in this study, bubble growth is much faster than at atmospheric. This faster growth may account for the thicker microlayer for water at reduced pressure as compared to atmospheric.

The theoretical temperature histories in this study con-

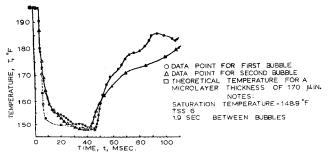


Fig. 5. Temperature histories of film 14 at 3.62 lbs./sq.in.

trast sharply with the theoretical temperature histories predicted by Cooper and Lloyd (4) in their Figure 7 for toluene on glass and steel. The typical behavior of the theoretical temperature histories in this study is seen in Figure 5 for the temperature to drop sharply at first and then asymptotically approach the saturation temperature. The theoretical temperature histories of Cooper and Lloyd show, instead, a slower initial fall which becomes sharper at the end of the temperature drop.

CONCLUSIONS

The following conclusions can be drawn from this study:

- 1. The surface temperature under a bubble often reaches the saturation temperature in boiling water at 1 to 4 lb/sq. in. abs.
- 2. Small platinum films can be drawn on pyrex glass to give accurate surface temperatures beneath bubbles during nucleate boiling of water.
- 3. The films were capable of indicating temperature changes of as fast as 145°F./msec.
- 4. The factor αR_o was constant for each TSS for the temperature range of interest.

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HOTATION

E= d.c. voltage, mv. = d.c. current, ma. i

R = resistance, ohm T= temperature, °F.

t= time, msec.

= TRC, temperature coefficient of resistance, ohm/

Subscript

= reference state

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