ON THE JOULE HEATING METHOD OF CALIBRATING
THIN FILM RESISTANCE THERMOMETERS

by

J. R. Busing
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SUMMARY

Measurements have been made of the thickness distribution of single layer thin film resistance thermometers formed from platinum china paint.

The profiles of the films measured were approximately parabolic, and this result has been used in the calculation of the correction factor which must be applied when the films are calibrated by a Joule heating method.
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LIST OF SYMBOLS

\( y \) coordinate along substrate surface
\( z \) coordinate normal to substrate surface
\( \delta \) film maximum thickness
\( h \) film semiwidth
\( R \) electrical resistance
\( E \) voltage
\( \sigma \) electrical resistivity
\( Q \) heat input
\( T \) temperature
\( t \) time
\( k \) thermal conductivity
\( \rho \) density
\( \kappa \) thermal diffusivity = \( \frac{k}{\rho C_p} \)
\( \tau \) penetration depth = \( 2\sqrt{\kappa t} \)
\( \eta \) dummy variables of integration
\( u \)
\( \beta \) thermal penetration parameter = \( \frac{h}{\sqrt{\kappa(t - \tau)}} \)

SUBSCRIPTS

\text{mean} \quad \text{mean value}
\text{10} \quad \text{value corresponding to one-dimensional heat input.}
1.0 Introduction

Thin film resistance thermometers, formed either by metal evaporation in vacuo or by baking platinum china paints on glass, are now standard transducers for shock detection or heat transfer measurements in shock tubes and similar high enthalpy, short flow duration devices. When evaluating heat transfer rates it is necessary to know the thermal properties of the backing material. For films formed from china paint, handbook values are usually not acceptable since the method of formation of the film probably causes some change of composition in the surface layers of the glass substrate. It is common practice therefore to calibrate these films by subjecting them to a known heating pulse, which is conveniently done by passing a square wave of current through the film. The resultant change in resistance is recorded on an oscilloscope and the group of thermal constants required can be calculated. It is assumed, in obtaining this result, that the problem is one dimensional, i.e., the heat input is applied over the whole surface instead of the finite width of the film.

Spence has shown in the case of constant current distribution in the film, i.e., constant heat input across the width of the film, that the mean temperature at the surface differs from the assumed one dimensional temperature by a factor involving the ratio of the penetration depth of the heat pulse to the width of the film. This result is applicable to the evaporated type of film which is generally uniform in thickness, but not necessarily to the china paint type which, even by visual observation, appears thinner at the edges.

In order to get a better assessment of the correction factor applicable to the china paint film several typical samples were carefully examined to determine their thickness distribution. A mathematical approximation to this distribution has been used, following Spence's calculation, to obtain a correction factor.

2.0 Experimental details

2.1 Method of film preparation

All resistances thermometers used in the College of Aeronautics shock tube are made from Hanovia 05X Liquid Bright Platinum applied as a fine line with a ruling pen. It has been found that a film of uniform and reproducible width can best be obtained by this method.

Normally the films are formed on pieces of Pyrex glass tube for shock detection or Pyrex glass models for heat transfer measurements. For the purposes of this investigation films were formed on the backing glass of used Ilford Zenith plates which were readily available. This glass is a soft glass with a softening temperature of about 650°C.

The glass was thoroughly cleaned with acetone and a series of single layer lines of varying widths were drawn with the ruling pen. After a short period of air drying the samples were placed in a cold oven** and to ensure adequate ventilation the door was left open during the initial heating phase. When the temperature reached 500°C.

* Engelhard Industries, Ltd.,
145 - 149, Vauxhall Street,
London, S.E.11.

** Gallenkamp Hot Spot, temperature range 0 - 1100°C.
the door was shut and the temperature continued to rise until 640°C was reached when the oven was immediately turned off. Normally the samples would be left to soak for five minutes, during which time the temperature would rise to 650°C and fall to 620°C, when they would be removed and allowed to cool in the open air. This procedure ensured a bright film adequately bonded to the substrate which could not be removed by scratching with a penknife.

2.2 Thickness measurements

A standard method for measuring the thickness of thin films uses light interference in a microscope. The method is relatively simple for transparent films and can be applied to opaque films if they have a reflecting surface. In addition, by using the multiple interference technique devised by Tolansky high accuracy is possible. Unfortunately, however, suitable equipment was not readily available. It was decided initially, therefore, to make a direct measurement of the film thickness by stripping the platinum from the glass and mounting it so that a cross-section could be obtained. This proved to be simple since it is well known that if the firing temperature is not high enough, and the glass surface does not soften, then although the outward appearance of the film indicates a successful gauge the film can easily be removed by scratching.

Samples were prepared as above but instead of allowing them to soak for five minutes they were removed from the oven when the temperature had reached 630°C. After cooling a lead was carefully attached using a low melting point silver solder, the sample was placed in an acid copper sulphate plating bath, and the current adjusted to 50 mA. As plating progressed the films began to lift from the glass as the copper deposit worked its way in from the edge between the glass and the film. Eventually the films left the glass entirely and plating was continued until the film was embedded in a deposit of copper robust enough to be easily handled.

Standard metallurgical techniques were then used for the mounting and preparation of the specimens. The polishing and etching of the cross-section proved difficult, however, and several processes and etch solutions were tried before the film was successfully isolated and could be easily observed and photographed in the microscope. One such section is shown in figure 1 representing the maximum thickness of the film and showing several defects which are typical of this type of film. Although the technique was eventually successful it proved to be very time consuming and involved many intermediate steps. An alternative method was therefore sought and it was eventually decided that an instrument used for the measurement of the surface finish of materials* would have adequate sensitivity. This proved to be the case and film profiles were readily obtained. A mounting jig was made to ensure that the specimens could be located in the same position relative to the measuring head. This enabled measurements to be made on a particular film which was then removed from the substrate by boiling in aqua regia. By replacing the glass in the same position in the jig a profile of the substrate could then be obtained. A typical profile of film and substrate is shown in figure 2. The discontinuities in the trace are probably

* Talysurf - Taylor Hobson Surface Measuring Instrument,
Max. vertical magnification x 50,000,
Max. horizontal magnification x 100.
caused by holes in the film or dust particles. It is interesting to note that the two indentations A.A in the substrate profile correspond to the edges of the film and confirm that the glass had been replaced in the jig in the correct position. The indentations are probably caused by surface tension effects, arising from higher temperatures in the region of the film. They also indicate that the surface of the glass had reached a softened state.

A series of profiles for films of various widths were taken and selected coordinates plotted as shown in figure 3. Because of the parabolic appearance of the profiles they have been plotted in the form

\[
\frac{z}{\delta} = 1 - \left( \frac{y}{h} \right)^2
\]

where \( \delta \) is the maximum thickness, and \( 2h \) is the film width. The coordinate system is shown in figure 4.

The profile of the films measured approximates reasonably well to equation (1) which has been used in the appendix for the derivation of the correction term.

The shape and size of the fluid paint line must be determined by surface tension effects and the thickness will therefore be affected by ambient temperature, paint viscosity and cleanliness of the glass surface. However, although several films of different widths had the same thickness, there is some correlation between maximum thickness and width, and this can be seen from the table in figure 3.

2.3 Resistivity of thin films

Since measurements had been made of the cross section of the films it was a simple matter to calculate their resistivity. The mean value for the films tested was 90 microhm cm. The value for pure platinum at room temperature is 11 microhm cm. It is unlikely that the high measured value for the films is due to a 'thin film effect' since this may be expected to occur only for thicknesses less than about 0.03\( \mu \). and in addition, the resistivity showed no sign of increasing as the film mean thickness decreased. The order of magnitude difference in resistivity probably arises from the method of formation of the film which may have the character of a sintered material rather than a homogeneous metal.

3.0 The correction factor for parabolic cross section films

If the thickness distribution of a thin electrically conducting material of constant resistivity \( \sigma \) is given by equation (1) above, then the resistance distribution per unit length of film is given by

\[
R(y) = \frac{\sigma}{\delta} \left( 1 - \left( \frac{y}{h} \right)^2 \right)
\]

Hence, the heat input distribution, assuming constant potential across the width of the film is given by

\[
Q(y) = \frac{E^2}{R(y)} = \frac{E^2 \delta}{\sigma} \left( 1 - \left( \frac{y}{h} \right)^2 \right)
\]
If the same heat input is obtained with a constant heat flux, $Q_m$, per unit area over the width of the film, then equation (3), can be written

$$Q(y) = \frac{3}{2} Q_m \left\{ 1 - \left( \frac{y}{h} \right)^2 \right\}$$  \hspace{1cm} (4)

The mean temperature $T_m$ of a film of semi-width $h$ has been obtained in the appendix in the form

$$T_m = T_{1b} \left\{ 1 - \frac{1}{8} \left( \frac{\xi}{h} \right)^2 \right\}$$  \hspace{1cm} (5)

where $T_{1b}$ is the temperature for the one-dimensional heat input problem, and $\xi$ is the "penetration depth" $2\sqrt{k\tau}$ which has a typical value for Pyrex of 53\micro m for a heat pulse of one millisecond duration.

This result can be compared with that obtained by Spence, viz.,

$$T_m = T_{1b} \left\{ 1 - \frac{1}{4\sqrt{n}} \left( \frac{\xi}{h} \right)^2 \right\}$$  \hspace{1cm} (6)

which applies to a film of constant thickness and hence rectangular heat input distribution.

It is physically reasonable that the parabolic heat input should give a temperature closer to the one-dimensional value than the rectangular heat input. This is because the temperature gradients in the transverse direction at the edge will be finite in the former case instead of infinite. The diffusion of heat into the surrounding material of the substrate would consequently be smaller.

The correction for a parabolic film 1 mm. wide using equation (5) amounts to 0.07% whilst the rectangular film result (6) gives 0.47%. In practice, of course, both these errors are extremely small compared with errors arising out of the measurements of heat transfer rates in the shock tube and hence the one-dimensional result is completely adequate for obtaining calibration data.

Conclusions

1. The cross sectional thickness distribution of thin film resistance thermometers has been measured by a surface finish measuring instrument and was found to be parabolic in form.

2. The correction to be applied for distributed finite heating over the width of the film corresponding to the actual thickness distribution, compared to the one-dimensional case, amounts to 0.07% for a typical calibration.

3. The electrical resistivity of the films was measured as 90 microhm cm.
References

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The author is indebted to Mr. J. McEvoy of the Materials Department who succeeded, by his perseverance and enthusiasm, in obtaining successful photo-micrographs of the cross section of the thin film.
Appendix

Assuming a heat input in the form

\[ Q(y) = \frac{3}{2} Q_m \left\{ 1 - \left( \frac{y}{h} \right)^2 \right\} \]  \hspace{1cm} (7)

then the temperature distribution is given by

\[ T(y, z, t) = \frac{1}{2\pi k} \int_0^t \exp \left[ -\frac{z^2}{4\kappa(t-\tau)} \right] \frac{1}{(t-\tau)^{\frac{1}{2}}} d\tau \times \]

\[ \int_0^h Q(\eta, t) \left\{ \exp \left[ -\frac{(y-\eta)^2}{4\kappa(t-\tau)} \right] + \exp \left[ -\frac{(y+\eta)^2}{4\kappa(t-\tau)} \right] \right\} d\eta \]  \hspace{1cm} (8)

Considering the case when \( Q_m \) is independent of time,

\[ T(y, z, t) = \frac{3}{2} Q_m \frac{1}{2\pi k} \int_0^t \exp \left[ -\frac{z^2}{4\kappa(t-\tau)} \right] \frac{1}{(t-\tau)^{\frac{1}{2}}} d\tau \times \]

\[ \int_0^h \left\{ 1 - \left( \frac{\eta}{h} \right)^2 \right\} \left\{ \exp \left[ -\frac{(y-\eta)^2}{4\kappa(t-\tau)} \right] + \exp \left[ -\frac{(y+\eta)^2}{4\kappa(t-\tau)} \right] \right\} d\eta \]

\[ = \frac{3}{2} Q_m \frac{1}{2(\rho \kappa c)^{\frac{3}{2}}} \int_0^t \exp \left[ -\frac{z^2}{4\kappa(t-\tau)} \right] \frac{1}{(t-\tau)^{\frac{1}{2}}} \times \]

\[ \left\{ \frac{\text{erf} \left( \frac{h+y}{2\sqrt{\kappa(t-\tau)}} \right) + \text{erf} \left( \frac{h-y}{2\sqrt{\kappa(t-\tau)}} \right)}{h} \right\} \left[ 1 - \frac{2\kappa(t-\tau)}{h^2} - \frac{y^2}{h^2} \right] + \]

\[ \frac{2}{\sqrt{\pi}} \frac{\sqrt{\kappa(t-\tau)}}{h} \left( \frac{h-y}{h} \right) \exp \left[ -\frac{(h+y)^2}{4\kappa(t-\tau)} \right] + \frac{2}{\sqrt{\pi}} \frac{\sqrt{\kappa(t-\tau)}}{h} \left( \frac{h+y}{h} \right) \times \]

\[ \exp \left[ -\frac{(h+y)^2}{4\kappa(t-\tau)} \right] \right\} d\tau \]  \hspace{1cm} (9)

The mean temperature at the surface is given by

\[ T_m = \frac{1}{2h} \int_{-h}^h T(y, 0, t) dy \]  \hspace{1cm} (10)

Now, putting \( \beta = \frac{h}{\sqrt{\kappa(t-\tau)}} \),
\[
\frac{1}{2h} \int_{-h}^{h} \left\{ \text{erf} \left( \frac{h + y}{2\sqrt{\kappa(t - \tau)}} \right) + \text{erf} \left( \frac{h - y}{2\sqrt{\kappa(t - \tau)}} \right) \right\} \left[ 1 - \frac{2\kappa(t - \tau)}{h^2} \right] + \\
\frac{2}{\sqrt{\kappa}} \frac{\sqrt{\kappa(t - \tau)}}{h} \left( \frac{h - y}{h} \right) \exp \left[ - \frac{(h + y)^2}{4\kappa(t - \tau)} \right] + \frac{2}{\sqrt{\kappa}} \sqrt{\kappa(t - \tau)} \left( \frac{h + y}{h} \right) \exp \left[ - \frac{(h - y)^2}{4\kappa(t - \tau)} \right] \right\} dy
\]

\[
= \frac{4}{\beta} \int_{0}^{\beta} \left( \frac{2}{\sqrt{\kappa\beta^2}} (\beta - u) e^{-u^2} - \left( \frac{1}{\beta^2} - \frac{2}{\beta} u + \frac{2}{\beta^2} u^2 \right) \text{erf} u \right) du
\]

\[
= \left( \frac{4}{3} - \frac{2}{\beta^2} \right) \text{erf} \beta + \frac{8}{3\sqrt{\pi}} \frac{1}{\beta^3} - \frac{8}{3\sqrt{\pi}} \left( \frac{1}{\beta^3} - \frac{1}{2\beta} \right) \exp (-\beta^2)
\]

\[
= \left( \frac{4}{3} - \frac{2}{\beta^2} + \frac{8}{3\sqrt{\pi}} \frac{1}{\beta^3} \right) - \exp (-\beta^2) \left( \frac{1}{\beta^2} + \left( \frac{4}{3} - \frac{2}{\beta} \right) \left( \frac{1.3}{2\beta^2} - \frac{1.35}{2^3\beta^3} \right) + \ldots \right) (11)
\]

For \( \beta > 2 \) the second group of terms on the R.H.S. of (11) is \(< 0.00059 \) and since the minimum practical value of \( \frac{h}{\sqrt{\kappa t}} \) is at least 10 only the first group is retained. Therefore

\[
T_m = \frac{3}{2} \frac{Q_m}{2(\pi\rho kc)^{\frac{1}{2}}} \int_{0}^{t} \left[ \frac{4}{3} \frac{1}{(t - \tau)^{\frac{3}{2}}} - \frac{2\kappa}{h^2} \left( \frac{t - \tau}{h^2} \right)^{\frac{3}{2}} + \frac{8}{3\sqrt{\pi}} \kappa^{\frac{3}{2}} \left( \frac{t - \tau}{h^2} \right) \right] d\tau
\]

\[
= 2Q_m t^{\frac{1}{2}} \left[ 1 - \frac{1}{8} \left( \frac{t}{h} \right)^2 + \frac{1}{16\sqrt{\pi}} \left( \frac{t}{h} \right)^3 \right]
\]

\[
= T_{10} \left[ 1 - \frac{1}{8} \left( \frac{t}{h} \right)^2 + \frac{1}{16\sqrt{\pi}} \left( \frac{t}{h} \right)^3 \right] (12)
\]

\( T_{10} \) is the temperature at the surface if \( Q_m \) is the one-dimensional heating rate and \( h \) is the "penetration depth" \( 2\sqrt{\kappa t} \) in time \( t \). For most practical values of \( \frac{t}{h} \) the cubed term is very much smaller than the squared term and can be neglected.
FIG. 1. PHOTOMICROGRAPH OF CROSS SECTION OF PLATINUM FILM AT X1000 MAGNIFICATION
FIG. 2. TALYSURF RECORD OF PLATINUM FILM ON GLASS. A-A ARE INDENTATIONS CAUSED DURING THE FORMATION OF THE FILM

FIG. 4. CROSS SECTION OF THIN FILM SHOWING COORDINATE SYSTEM