

# Thermal wave measurement of wet paint film thickness

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Thermal wave phase measurements are reported on the drying of wet paint films on aluminium substrates. Measurements of the change in thickness as the paint dries have also been obtained using a differential focussing technique on an optical microscope. By including the optical microscope measurements of the drying paint film thickness together with estimates for the density and thermal properties of the drying and cured paint, predictions have been made of the thermal phase/thickness relationships for the wet, dry, curing and cured paints. It is concluded that a phase measurement on the wet paints could be used to predict a final cured paint thickness with an accuracy of approximately  $\pm 2 \mu\text{m}$ . Errors in predicting cured film thickness from a wet film thermal phase measurement arises principally from uncertainty over the solvent content of the wet paint film, the state of cure, and the consequent uncertainty over the paint density and thermal properties. © 2006 Springer Science + Business Media, Inc.

## 1. Introduction

The precise control of paint film thickness is important for aircraft in order to control their weight. Of particular interest here is measurement of wet paint film thickness, rather than dry paint film thickness. This distinction between measurements on wet paint, as opposed to dry paint, arises because in order to give a final dry paint film with the necessary thickness, it is necessary to control spraying parameters in real time and measure paint thickness as the wet paint is being applied, and before the paint dries. To accomplish this, the method used must be both non-contact and rapid, and there must also be an understanding of the relationship between the measured wet paint film thickness and the final thickness after the paint has dried.

Various methods are available for making non-contact measurements of paint film thickness, this includes:

*Laser generated ultrasound.* This is an established method which works by relating the transit time of pulses within the paint layer to the paint layer thickness via the layer velocity. An example implementation may be inspected in the device marketed by Perceptron, Inc, [1]. This device uses lasers for both excitation and detection of ultrasound.

*Gamma ray back scattering.* This method works by measuring the intensity of gamma rays back-scattered from electrons within the paint layer using Compton scattering. The intensity is proportional to the mass density of paint and used to give a direct measurement of paint thickness. For example, the CPC-48 thickness gauge licensed by S&G Technologies of Don Mills, Ontario, is described as being suitable for the control and measurement of wet paint film thickness in car manufacture [2].

*Infra-red spectroscopy.* Fig. 1 shows the transmission spectrum for an epoxy-based paint on a silicon substrate obtained using a spectral radiometer [3]. Inspection of Fig. 1 in the wavelength region between 5 and 6  $\mu\text{m}$  shows an oscillating curve which is characteristic of resonance within the paint layer. A simple resonance/thickness analysis for two adjacent resonance peaks at  $\lambda_1$  and  $\lambda_2$  gives:

$$d := \frac{1}{2 \cdot \eta} \left( \frac{1}{\frac{1}{\lambda_1} - \frac{1}{\lambda_2}} \right)$$

where  $\eta$  = refractive index of paint

As an example, if a paint refractive index ( $\eta \sim 1.5$ ) is assumed and the resonance peaks identified in Fig. 1 are

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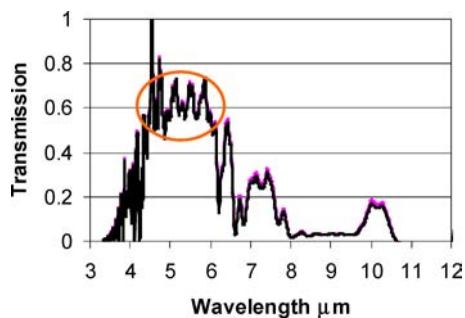


Figure 1 Transmission spectrum for a thin epoxy paint layer on a silicon substrate. The circled area shows adjacent resonance peaks.

used in conjunction with the above equation, this would imply a paint thickness of approximately  $27 \mu\text{m}$ .

*Phase sensitive photothermal methods.* Photothermal thickness measurement of dry films and coatings is an established method [see, for example, references 4 and 5], and works analogously to the use of ultrasound methods, except that layer thickness is deduced from the transit time of thermal waves if the thermal diffusivity is known. Commonly, when used with periodic-harmonic excitation, the transit time is derived from phase measurements of surface temperature with respect to the phase of the excitation source. Typically, for coating/substrate interfaces with good reflectivity, a phase resolution of  $\pm 0.25^\circ$  results in approximately 1% resolution for coating thickness measurement [5].

All the methods described above could, or are, being used for the determination of wet paint film thickness. The particular application sought here was for use with aluminium substrates. It was also deemed necessary that the method should be quick and inexpensive, with the potential for implementation as a compact low weight unit suitable for use with a robotic paint spraying arm. The laser generated ultrasound device is expensive, the gamma ray-back scattering device is quite slow, taking approximately 48 s to achieve suitable counting times with aluminium [6], and the infra-red spectroscopy method used a spectral radiometer, which did not lend itself to the small size and weight desired here. Given the potential for relative simplicity, low weight, compact and inexpensive nature of the thermal wave methods based on phase measurements us-

ing semiconductor lasers and pyroelectric detectors, then a phase sensitive photothermal method was selected as a candidate for wet paint film thickness measurements.

## 2. Experimental method

Initial test of the viability of wet paint thickness measurements were made using an argon/ion based laser system. This was a coherent beam 3 W argon ion laser with a wavelength of 514 nm, full details of which have been described elsewhere [7]. Briefly, laser light was mechanically chopped to generate a periodically fluctuating surface temperature within a frequency range of between 10 and 34 Hz. A specimen was mounted on a translation stage and amplitude and phase of the temperature under the illuminated spot was monitored via a Mullard 825CPY triglycine sulphate pyroelectric detector placed at the focus of an ellipsoidal mirror. The specimen dwelled for a short time (typically between 1 and 3 s), and the phase was measured with reference to the illumination via a phase-locked loop amplifier (using an integration time typically between 0.1 and 3 s) and recorded on a PC. Measurements of laser power at the specimen surface were made using a laser power meter, and were typically in the range 37–90 mW. It was estimated that at 20 Hz, these power levels would give rise to a maximum temperature rise of between approximately 2.5 and  $6^\circ\text{C}$ . Fig. 2 shows in schematic form the elements of the system used here.

Initial testing was first conducted on two dry paint calibration specimen. This was undertaken in order to define the most suitable operating envelope, to estimate the ultimate accuracy of the system, and to measure the thermal properties of the cured paint. Corroborative measurements of thickness on the dry paint calibration specimens was undertaken using an eddy current thickness tester (DeFelsko Positector<sup>®</sup> 6000), and a differential focussing arrangement on an optical microscope (Zeiss Axioplan<sup>®</sup>) using a large magnification objective lens with a small depth of field. This was undertaken by removing a small region of paint such that the substrate was exposed, and then focussing in turn on the paint surface and the paint/aluminium interface, and then noting the traverse of the stage required for focussing. Thickness measurements were undertaken on wet paint specimens using both the thermal wave system and the focussing

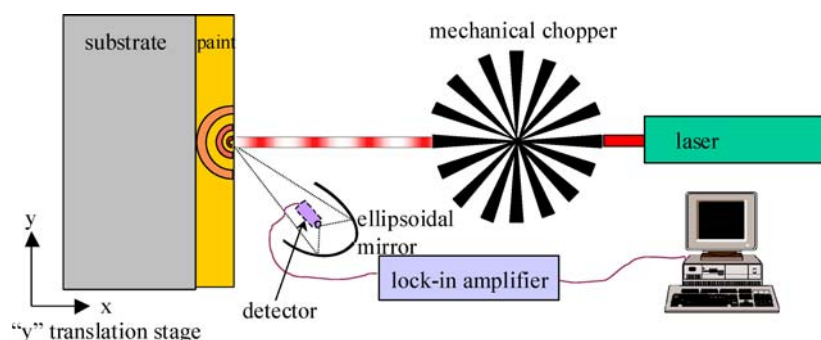


Figure 2 Schematic diagram showing the elements of the photothermal system used for making phase measurements.

arrangement on the optical microscope. These were made as a function of time. The optical microscope focussing method was also undertaken on wet paint specimens in order to provide comparative thickness changes as the paint dried and to calibrate the phase measurements.

### 3. Specimens

All specimens comprised paint layers on an aluminium substrate. Paint type was low VOC chromate free epoxy primer CA7012F30257 (3 parts by vol.) mixed with activator CA7012B (1 part by vol.) manufactured by PRC DeSoto International [8]. Two dry paint calibration test specimens were painted using a professional air spray system (DeVilbiss GTi) to give stepped thickness specimens with regions having a nominal thickness of one, two and three layers across the width of the specimen. This resulted in specimens with nominal paint thickness of 20, 30, 40, 60, 100 and 120  $\mu\text{m}$ . Painted surfaces were light brown in colour. Paint for the wet specimens was applied via a draw bar applicator (Sheen Instruments four sided applicator (Ref. 1107/60)), or wire bar coater (Sheen Instruments wire bar coater (Ref. 1120)). This was performed immediately after addition of the accelerator. The specimens were then allowed to dry and cure at room temperature. Dry paint calibration specimens were dried and cured either in an oven at 60°C or via the use of an infrared radiant heater.

### 4. Results for calibration specimens

The phase difference ( $\phi(L)$ ) between the reference illumination and the sample surface temperature as a function of paint thickness ( $L$ ) is given by Almond and Patel [4] as:

$$\phi(L) := a \tan \left[ -1 \cdot \frac{(1 + R_g) \cdot R_b \cdot e^{-x(L)} \cdot \sin(x(L))}{1 + [(1 - R_g) \cdot R_b \cdot e^{-x(L)} \cdot \cos(x(L))] - (R_b)^2 \cdot R_g \cdot e^{-2x(L)}} \right] \quad (1)$$

where

$L$  = thickness

$R_g$  = reflection coefficient at air/paint interface (approximately = 1)

$R_b$  = reflection coefficient at aluminium/paint interface =  $(1 - \varepsilon_{\text{paint}})/(1 - \varepsilon_{\text{aluminium}})$

$\varepsilon$  = effusivity =  $\sqrt{(k \cdot \rho \cdot c_p)}$

$k$  = thermal conductivity

$\rho$  = density

$c_p$  = specific heat

$x(L) = 2L/\mu$

$\mu = \sqrt{(2\alpha/\omega)}$

$\alpha$  = thermal diffusivity =  $k/\rho \cdot c_p$

$\omega$  = angular frequency

Fig. 3 shows the results of a best fit for Equation 1 to the average measured thickness for each of the paint layers of the paint calibration specimens at 10, 20 and 30 Hz. Using quoted values for the density and

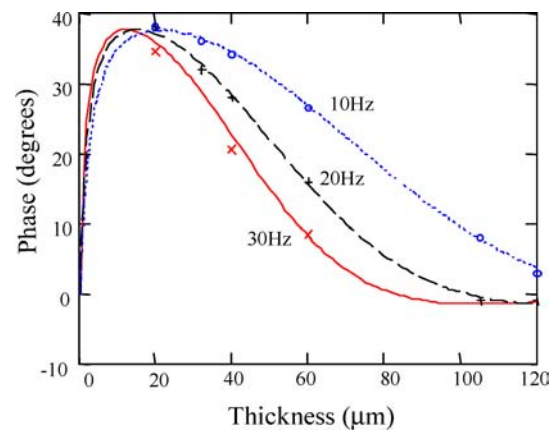


Figure 3 Predicted and measured phase/thickness for the dry paint calibration specimens at 10, 20 and 30 Hz.

thermal properties of aluminium [4], together with a quoted paint density  $\rho = 1620 \text{ kg m}^{-3}$  [8] and an estimate for the paint specific heat  $c_p = 1300 \text{ J kg}^{-1}\text{K}^{-1}$ , this yielded a single value for the paint thermal conductivity  $k = 0.55 \text{ Wm}^{-1}\text{K}^{-1}$  and a thermal diffusivity  $\alpha = 2.6 \times 10^{-7} \text{ m}^2\text{s}^{-1}$  from the fitting procedure. Fig. 4a and b shows results from individual points across the width of each calibration specimen for thickness deduced from phase measurements at 10 Hz and 20 Hz based on a thermal diffusivity  $\alpha = 2.6 \times 10^{-7} \text{ m}^2\text{s}^{-1}$ . This is shown in comparison with thickness measurements derived using the eddy current tester and the optical microscope differential focussing technique. Small local variations in a measured thickness were noted for a single nominal paint thickness using each technique, particularly the thicker layers, and it is believed this variation originated from small local variations in paint thickness that

occurred during drying or curing. Average thickness and standard deviation for the thinnest and most uniform nominal 20  $\mu\text{m}$  paint layer was  $20.9 \mu\text{m} \pm 2.0 \mu\text{m}$  using the eddy current tester,  $21.3 \mu\text{m} \pm 1.9 \mu\text{m}$  for the differential focussing on the optical microscope, and  $20.2 \mu\text{m} \pm 1.1 \mu\text{m}$  for the phase measurements at 20 Hz.

### 5. Results for wet paint thickness measurements

Optical thickness measurements of draw bar applied paint films as they dried are shown in Figs 5a and b for specimens with dry thickness of 20 and 40  $\mu\text{m}$  respectively. It should be noted that there was a minimum time of approximately 20 s between application of a paint film and the time for the first measurement point. Since an accurate measurement of a final cured thickness relies on a

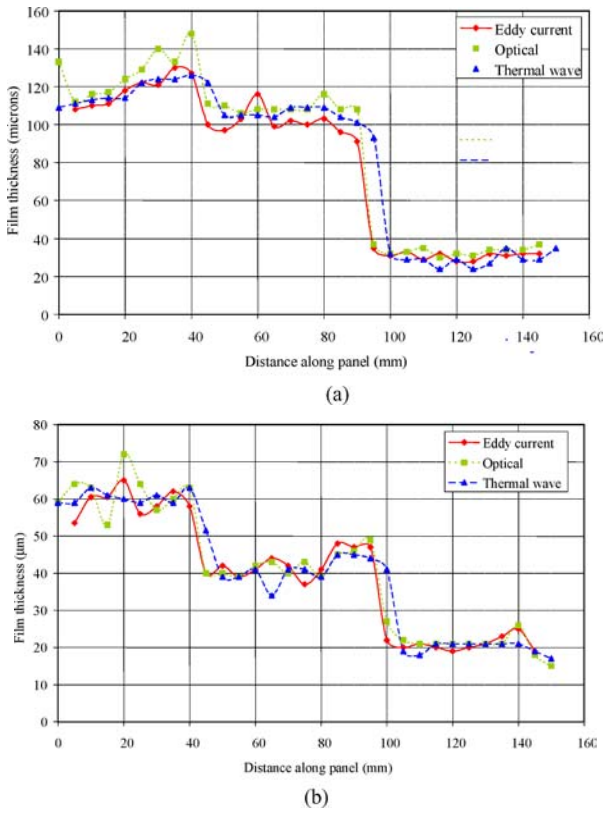


Figure 4 (a) Comparison between thermal phase (at 10 Hz), eddy current and optical microscope measurements for a paint calibration specimen. (b) Comparison between phase (at 20 Hz), eddy current and optical microscope measurements for a paint calibration specimen.

wet paint thermal phase measurement immediately after the paint has been applied, it is important in the calibration that the full thickness drying curve from time  $t=0$  is known. Noting that the drying is primarily controlled by the release of solvent from the paint, a reasonable approximation to this initial drying phase is to model the diffusion within the paint and fit a functional form based on this process.

By assuming that the wet thickness of a paint film containing a fractional content of solvent at time  $t$  may be related to the dry thickness ( $h_0$ ) via an initial solvent fraction at time  $t=0$  ( $C_0$ ) and a fractional mass loss at time  $t$  ( $M_{\text{frac}(t)}$ ), then the thickness  $h(t)$  at time  $t$  may be written as:

$$h(t) = C_0 \cdot M_{\text{frac}(t)} \cdot h_0 + h_0 \quad (2)$$

where  $M_{\text{frac}(t)} = M_t/M_\infty$

Noting that Crank [9] gives an error function (erfc) solution for sorption and desorption from a membrane in terms of the ratio between the mass of diffusing substance at time  $t$  ( $M_t$ ) and at time  $t = \infty$  ( $M_\infty$ ), then the fractional mass loss from a thin film ( $M_{\text{frac}(t)}$ ) due to diffusion is:

$$M_{\text{frac}(t)} := 1 - 2 \cdot \sqrt{\frac{D \cdot t}{L^2}} \left[ \frac{1}{\sqrt{\pi}} + \sum_{n=1}^{\infty} (-1)^n \right]$$

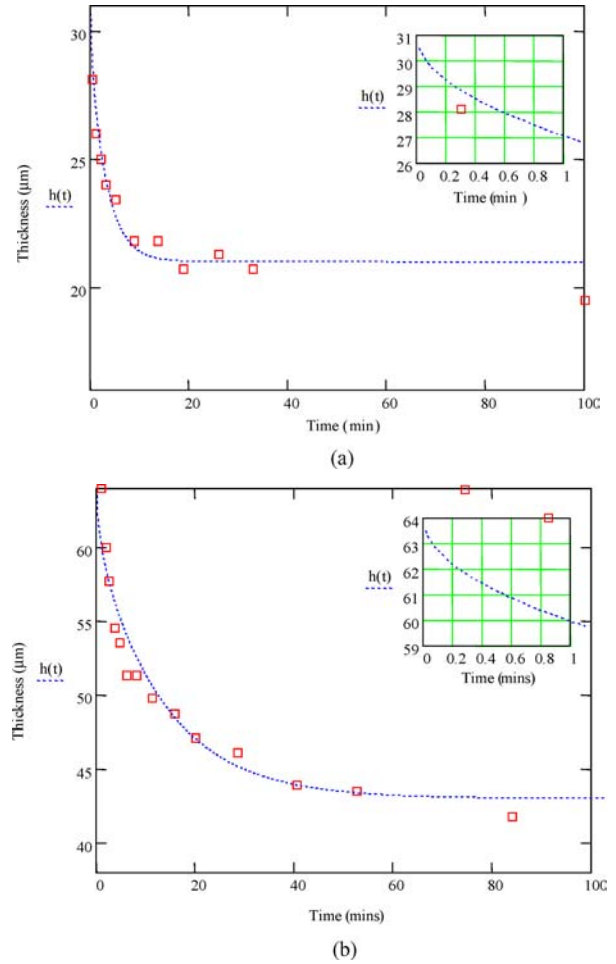


Figure 5 (a) Drying curve for a 20  $\mu\text{m}$  paint layer measured using optical microscope  $\square$ . Predicted thickness using diffusion solution Equation 3 (dashed line). Inset graph shows enlargement of scale up to a time of 1 min. (b) Drying curve for a 40  $\mu\text{m}$  paint layer measured using optical microscope  $\square$ . Predicted thickness using diffusion solution Equation 3 (dashed line). Inset graph shows enlargement of scale up to a time of 1 min.

$$\left[ \text{erfc} \left( \frac{n \cdot L}{\sqrt{D \cdot t}} \right) \right] \quad (3)$$

where:

$D$  = diffusion coefficient and  
 $L$  = thickness of film

Fig. 5a and b shows examples of fitting a curve based on Equations 2 and 3 for specimens with a dry thickness of 20 and 40  $\mu\text{m}$  respectively using a single solvent diffusion coefficient ( $D$ ) of  $9 \times 10^{-13} \text{ m}^2\text{s}^{-1}$ . For four paint films, this indicated an average fractional change in thickness from the wet state at  $t=0$  to its eventual dry film thickness after solvent release of approximately  $49 \pm 1.5\%$ . It may also be noted that the paint film continued to shrink by a small amount ( $\sim 5\%$ ) after the solvent evaporated. It is believed that this was due to the cure shrinkage of the paint in its dry state.

Fig. 6 shows the time evolution for the thermal phase recorded at 20 Hz for the drying of 8 draw-bar painted

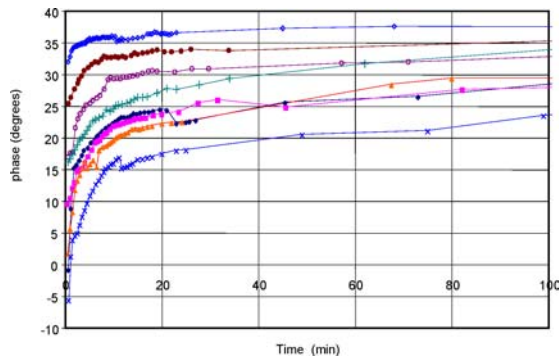


Figure 6 Thermal phase recorded for the drying of wet paint films on an aluminum substrate at 20 Hz. Dry paint thickness between 20 and 40  $\mu\text{m}$ .

specimens, with final cured paint thickness in a range between 21 and 38  $\mu\text{m}$ . It should be noted, however, that a measurement of phase was not made immediately at the time of painting. This was because painting using draw bars was not possible when the specimens were located on the phase measurement rig. This resulted in a minimum time between application of a paint film and the time for the first measurement point.

A derivation of a dry and cured paint film thickness from a measurement of phase for the paint in its wet state at some time  $t_1$  after paint application requires both a knowledge of the thermal conductivity, specific heat and density at this time, and also the shrinkage that then subsequently occurs. The shrinkage may be derived by fitting a measured thickness at time  $t_1$  to a drying curve based on a solution to Equation 3, from which the dry thickness after solvent evaporation may be derived. As an example, this procedure is shown schematically in Fig. 7 for a specimen with a wet paint thickness of 41  $\mu\text{m}$  made at  $t_1 = 0.6$  min, giving a dry paint thickness of 29.5  $\mu\text{m}$ . The cure thickness may then be predicted from the dry thickness by assuming, as noted above, a small additional percentage shrinkage. Direct measurements of thermal conductivity,

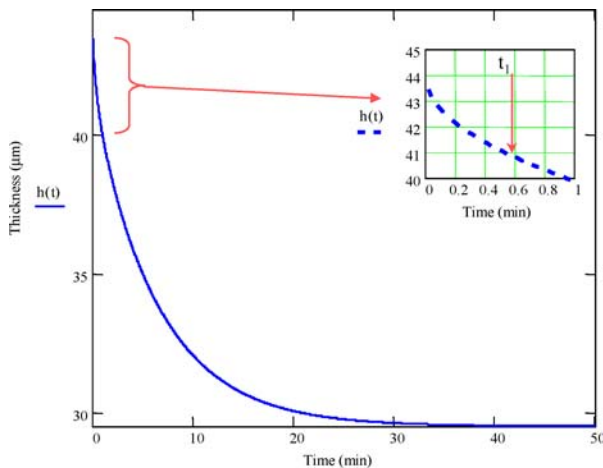


Figure 7 Predicted thickness change based on the solvent released during evaporation from the paint film. Prediction based on a wet film thickness of 41  $\mu\text{m}$  measured at time  $t_1 = 40$  s (indicated by the arrow in the inset graph) and an initial solvent content of 49% of the dry mass.

specific heat and density for this paint system as a function of drying time and cure, were however, not available, and it was necessary to estimate values for these properties.

Wubbenhorst *et al.* [10] has characterised the cure of a DGEBA epoxy system (Epon 828, curing agent DDM) using the inverse photopyroelectric technique. By combining photopyroelectric measurements with results from modulated differential scanning calorimetry they were able to show that the changes in epoxy effusivity (specific heat  $\times$  density  $\times$  thermal conductivity)<sup>1/2</sup> could be related to an initial rise in the product of the density and thermal conductivity before vitrification ( $\sim 40\%$ ), and then a fall in specific heat during vitrification ( $\sim 20\%$ ). By using the estimated values for specific heat ( $C_p = 1300 \text{ J kg}^{-1} \text{ K}^{-1}$ ), density ( $\rho = 1620 \text{ kg m}^{-3}$ ) and thermal conductivity ( $k = 0.55 \text{ W m}^{-1} \text{ K}^{-1}$ ) for the paint used here, it was possible to estimate approximate values for these same properties before cure by using the percentage changes noted above. In a similar vein, given that the wet paint is likely to be composed of 49% solvent at the moment of application, an estimate was made for the wet paint properties by using a rule-of-mixtures combination of paint and solvent at time  $t_1$ . The solvent was assumed to be MEK, which is a common solvent for paints, with density  $\rho = 804 \text{ kg m}^{-3}$ , specific heat  $C_p = 2300 \text{ J kg}^{-1} \text{ K}^{-1}$  [11] and thermal conductivity  $k = 0.15 \text{ W m}^{-1} \text{ K}^{-1}$  [12]. Dry thickness was assumed to be 5% thicker than the cured thickness. Table I summarises the estimated cured, dry and wet paint properties. Fig. 8a–d shows the phase/thickness relationship calculated using Equation 3 based on the estimated paint properties from Table I. Fig. 8a also shows the experimental points measured wet after 30–40 s ( $t_1$ ), (b) after approximately 1.5 min, (c) when dry after solvent evaporation, and (d) also when the paint was fully cured.

Except for three results at the largest thickness in the wet state at 30–40 s, agreement between experiment and predictions at each time is good. Inspection of the final paint surface showed evidence that paint slump had occurred for these three results, and it is believed this resulted in the poorer observed agreement at 30–40 s. Excluding these three results, it was estimated that a phase measurement on the wet paints after 30–40 s could be used to predict a final cured paint thickness with an accuracy of approximately  $\pm 2 \mu\text{m}$ .

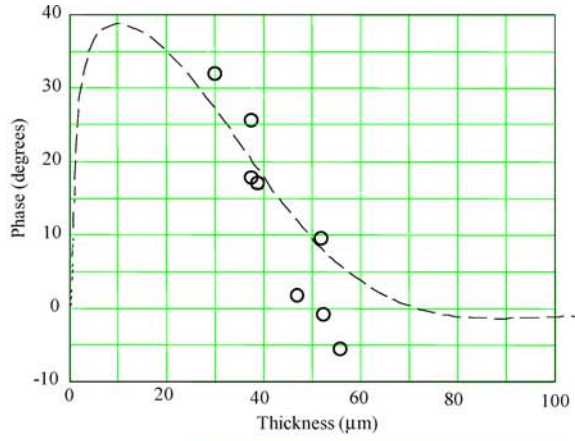
## 6. Discussion

It was estimated that the electrical phase noise when making a measurement of film thickness was approx-

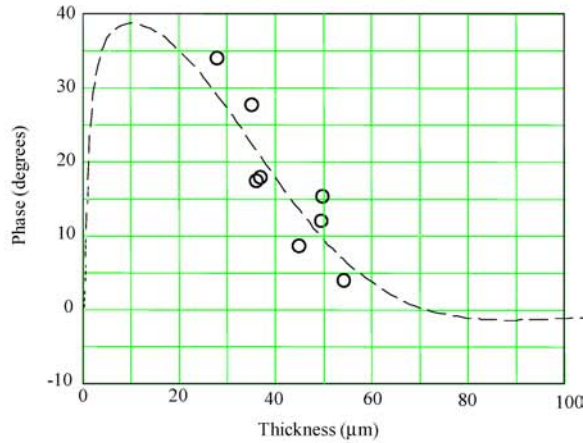
TABLE I Estimated density and thermal properties for CA7012 paint specimens

Paint state	Density ( $\rho$ ) $\text{kg m}^{-3}$	Specific heat ( $C_p$ ) $\text{J kg}^{-1} \text{ K}^{-1}$	Thermal conductivity ( $k$ ) $\text{W m}^{-1} \text{ K}^{-1}$
Cured	1620	1300	0.55
Dry	1543	1625	0.41
Wet (49%MEK)	1296	1848	0.32

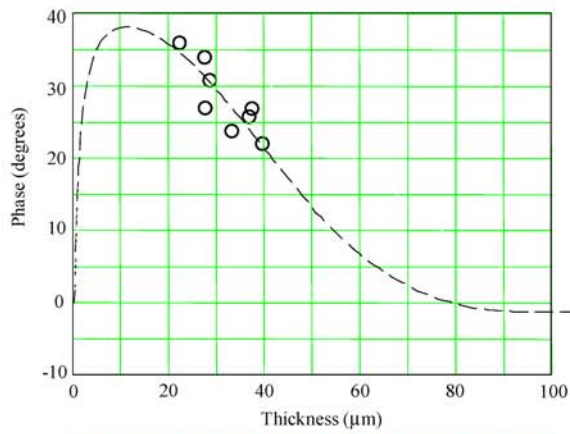
(a) Wet state,  
at  $t_1 \sim 40$  secs  
from  $t=0$ .



(b) Wet state,  
 $\sim 1.5$  mins  
from  $t=0$ .



(c) Dry state



(d) Final  
cured state

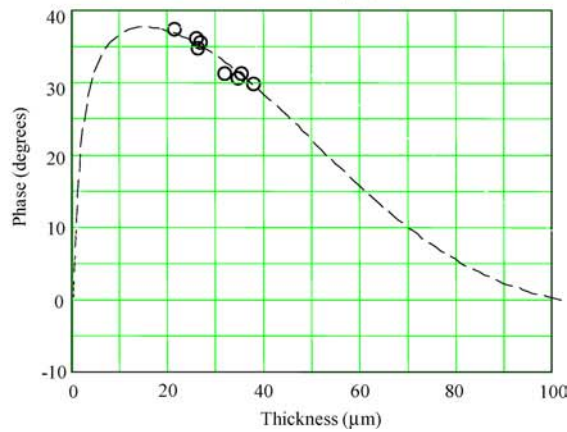


Figure 8 Sequence of phase/thickness predictions and measured thickness for the drying and curing of several painted specimens. Predictions based on thermal data from Table I.

imately  $\pm 0.1^\circ$ . This was equivalent to a thickness measurement error of approximately  $\pm 0.18 \mu\text{m}$  at 20 Hz and approximately  $\pm 0.25 \mu\text{m}$  at 10 Hz. These values were very much less than the roughness variations in dry and cured paint thickness for regions with nominally the same paint thickness. This ultimate accuracy was also degraded when inferring cured paint film thickness from wet paint phase measurements because of the assumption used in the derivation. This included the approximation of a fixed boundary diffusion solution for the evaporation of a known quantity of solvent from the paint, an approximation of the evolution of the paint's thermal properties during drying based on a rule-of-mixtures analysis for paint and solvent, the application of literature values regarding changes in specific heat and thermal conductivity as the paint cured, and an assumed 5% change in thickness when the paint film cured. In addition, final accuracy was probably also determined by paint slumping.

The derivation of the fractional change in thickness as the paint dried used a solution for diffusion from a membrane with a fixed thickness. Given that the paint shrinks and changes thickness as it dries, a more realistic treatment should ideally use a moving boundary solution. However, it is likely that the initial shape of the thickness drying curve near  $t = 0$  will remain unchanged, and the increase in mathematical complexity involved in the solution may not justify the increase in accuracy.

For the paint films applied here using a draw bar method, the solvent content and initial cure state of the wet paint were fairly well controlled. However when using paint which is sprayed from a spray gun, it is likely that care will have to be exercised over variables which influence paint spraying parameters which might influence evaporation of solvent from the paint, this includes spraying pressure, fan size, stand-off distance, gun speed, paint flow rate, etc. In addition, care would clearly also have to be given to controlling the residence time of paint which is held in a pot prior to painting, when any initial curing reactions will affect both the spraying process and the thermal properties of the paint. These will become increasingly important the longer the mixture is kept after addition of the accelerator. It is also important to recognise that if paint fan occurs during application and paint overlap results, then substrate thermal reflectivity may also change. This would result in a consequent change in the apparent phase/thickness relationship.

In spite of these complications, a methodology has been demonstrated which would permit the derivation of cured and dried paint film thickness from thermal wave phase measurements conducted on a wet paint film.

## 7. Conclusions

Thermal wave phase measurements have the potential to form a relatively inexpensive means for a compact, light

weight and quick method for making measurements of wet paint film thickness, from which the dried and cured paint film thickness may be inferred. This could be of significant benefit in paint spraying when accurate layer thickness is required, because it permits rapid feedback which may be used to control paint spraying parameters.

It is important, however, that if the technique is used for the application of paints to give a controlled paint thickness, then a critical awareness of the assumptions used in the analysis should be exercised. This would include the influence of solvent content, thermal properties of the paint during cure and drying, and cure shrinkage on a final predicted thickness.

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