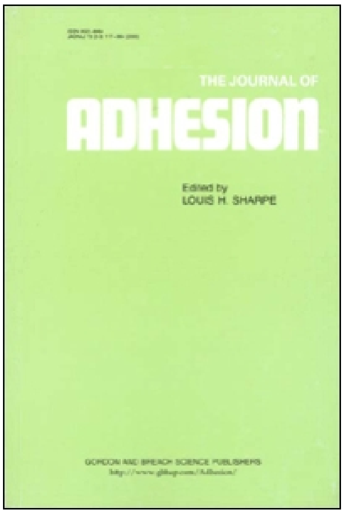

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High Resolution Optical Interference Measurements of Changes in Bond-Line Thickness—A Review

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High Resolution Optical Interference Measurements of Changes in Bond-Line Thickness—A Review†

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A novel moire optical interference method, originally developed[1] to measure the swelling displacement normal to the plane of a simple butt joint during water uptake at the joint edge, was subsequently used to investigate the unexpected occurrence of shrinkage and, at the same time, swelling during exposure to organic solvents. In later experiments, synergistic displacement fields generated during simultaneous exposure to water and organic solvents were studied. The same method has also been used to investigate the origin of adhesion failures at copper plated-through holes in thermally cycled multi-layer circuit boards.

The method employs a thin glass cover slip as one adherend. Changes in bondline thickness flex the cover slip thereby changing the gap between the latter and a nearby optical flat. By using monochromatic light to illuminate the specimen, interference between incident and reflected light is achieved. The small displacements of individual fringes associated with changes in gap size are used to generate moire patterns which bear a 1:1 relationship to the local changes in bondline thickness. Application of thin plate elasticity theory to the flexing of the cover slip yields the stress acting normal to the joint. The magnitude of this normal stress is strongly inhomogeneous, oscillating in sign and reaching compressive values as high as $50\,\mathrm{MN\,m^{-2}}$ for water uptake by a simple butt joint, and exceeding the yield strength of oxygen-free high conductivity copper ($1\,\mathrm{MN\,m^{-2}}$) for the thermal expansion that accompanies simulated soldering of a multi-layer circuit board.

Attention is drawn to the high resolution achievable with the method. Changes in bond-line thickness of the order of $\lambda/10$, where λ is the wavelength of the light giving rise to the interference pattern, are readily resolved. It is pointed out that the method may well be sufficiently sensitive to detect the effects of different physical properties (thermal conductivity, specific heat capacity) of adherend surface on the nature of the cured adhesive and hence on the mechanical behaviour of the bond-line.

KEY WORDS Bondline swelling; moire optical interference method; multilayer circuit boards; organic solvents; water; temperature cycling.

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INTRODUCTION

The experiment described here brings together four topics from classical physics. Firstly the generation of Newton's rings between a polished surface, usually the free surface of a microscope cover slip adhesively bonded to a more massive adherend, and an optical flat. Figure 1 shows such a pattern of Newton's rings, small changes in which are revealed by the second physical phenomenon, namely the formation of moire fringes when two nearly identical periodic patterns are superimposed one on the other. In Figure 1, the moire fringes are the circumferential fringes resulting from small upwards displacement of the rim of the coverslip during swelling associated with water uptake by the underlying adhesive. The third topic from classical physics concerns membrane elasticity theory applied to the cover slip deformation in order to calculate the stresses acting normal to it. These stresses are, of course, generated in the adhesive by the non-uniform changes in bondline thickness. In the holes through circuit boards application of our method, the bondline thickness remains constant. Inhomogeneous changes in circuit board thickness in the vicinity of a hole are faithfully

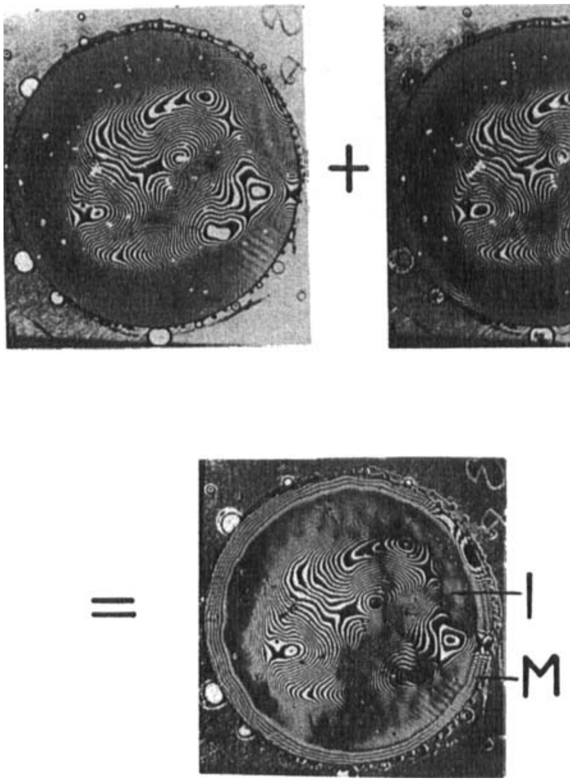


FIGURE 1 Showing the generation of moire fringes by superimposition of successive photographs of the pattern of interference fringes. The moire fringes are the circumferential loops labeled M an Fizeau interference fringes are the thinner lines labeled I.

reproduced by normal displacement of the microscope cover slip. Finally, we make use of a fourth branch of physics, namely the phenomenon of photoelasticity, to investigate the nature of localized swellings (not present in Figure 1) well inside the rim in joints exposed, some to water, some to organic solvents, and others to both water and organic solvent.

EXPERIMENTAL

Figure 2 is a schematic diagram of the optical system used to generate and record optical interference fringes that are formed between the free surface of a thin glass microscope cover slip and a nearby optical flat. The microscope cover slip is adhesively bonded to a massive substrate, *i.e.* it is one adherend in the model joint. Exposure of the joint to hostile envoronments results in expansion or shrinkage of the adhesive and this in turn flexes the cover slip, modifies the gap between cover slip and optical flat and gives rise to displacement of individual fringes in the interference pattern. In the case of thermal cycling of a multi-layer circuit board to which a cover slip has been bonded, the bond line thickness remains constant and it is changes in the circuit board thickness that flex the cover slip and give rise to changes in the pattern of interference fringes.

Figure 1 shows how successive photographs of the interference pattern are used to determine the displacement field normal to the cover slip. By superimposing the interference pattern, photographed after some normal displacement has occurred, on the pattern photographed before that displacement had taken place, a moire pattern is obtained and this faithfully records the geometry of the

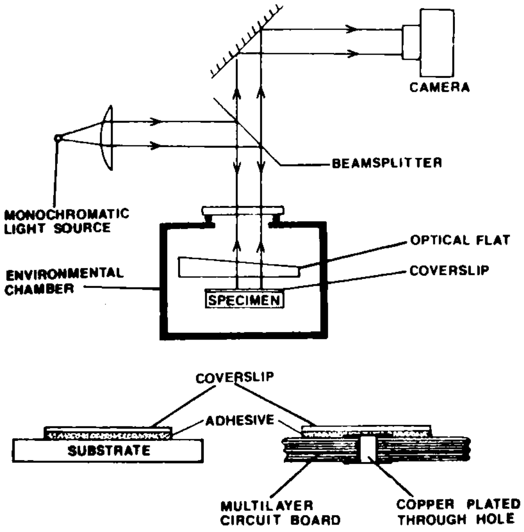


FIGURE 2 Schematic of optical system used to generate and record interference fringes. Also shown, are diagrams depicting the model adhesive joint and the multilayer circuit board specimens.

displacement. In Figure 1, the moire fringes are the circular loops concentric with the edge of the cover slip. Inside the innermost fringe, there has been no normal displacement. Moving outwards to the innermost fringe, a normal displacement of $\lambda/2$ has occurred. Between the first and second fringes, the normal displacement is $3\lambda/2$, and so on.

Using Love's[2] analysis for the displacement field in a thin membrane deformed by a pressure drop across its faces, estimates for normal stress in the adhesive layer, or circuit board, have been calculated.

$$D\nabla^4 w = -p \tag{1}$$

where the flexural rigidity

$$D = \frac{2Eh^3}{3(1-\nu^2)}$$

p = normal pressure
$2h$ = cover slip thickness
E = Young's modulus
ν = Poisson's ratio
w = axial displacement of cover slip

This analysis is valid for normal displacements that are sufficiently small for lateral stretching of the thin plate to be negligible. To a first approximation the deformation in the glass cover slip conforms to this criterion. An example of

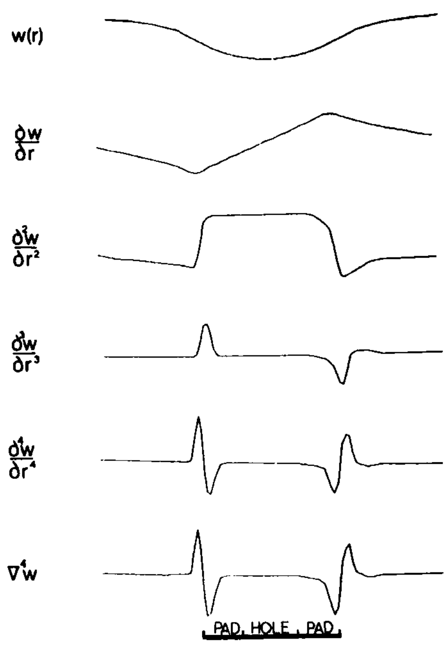


FIGURE 3 Graphical differentiation of the displacement $w(r)$ measured across a plated through hole at 50°C.

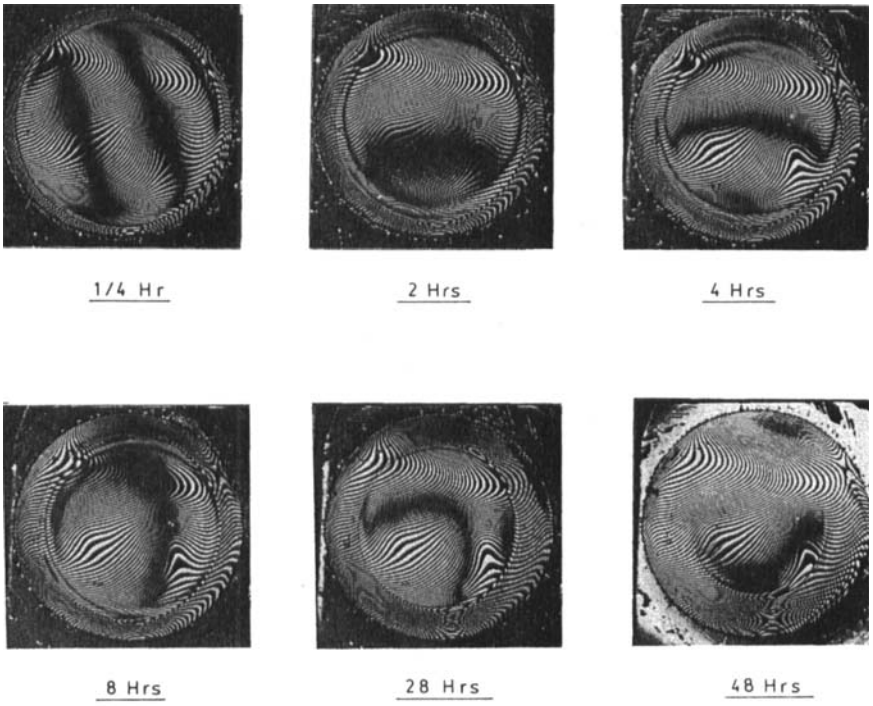
graphical differentiation of the normal displacement, measured across a diameter of a cover slip bonded to the plated through hole region of a multi-layer circuit board, is shown in Figure 3. In this case $w(r)$ is a cross-section of a deformation that has axial symmetry and is best suited to analysis in cylindrical polar coordinates (r, θ, z), thus $\nabla^4 w(r)$ is given by

$$\frac{\partial^4}{\partial r}+\frac{2}{r}\frac{\partial^3}{\partial r}-\frac{1}{r^2}\frac{\partial^2}{\partial r}+\frac{1}{r^3}\frac{\partial}{\partial r}$$

there being no dependence on θ (circular symmetry) or z (thin plate).

Swelling at the edge of a circular adherend[1]

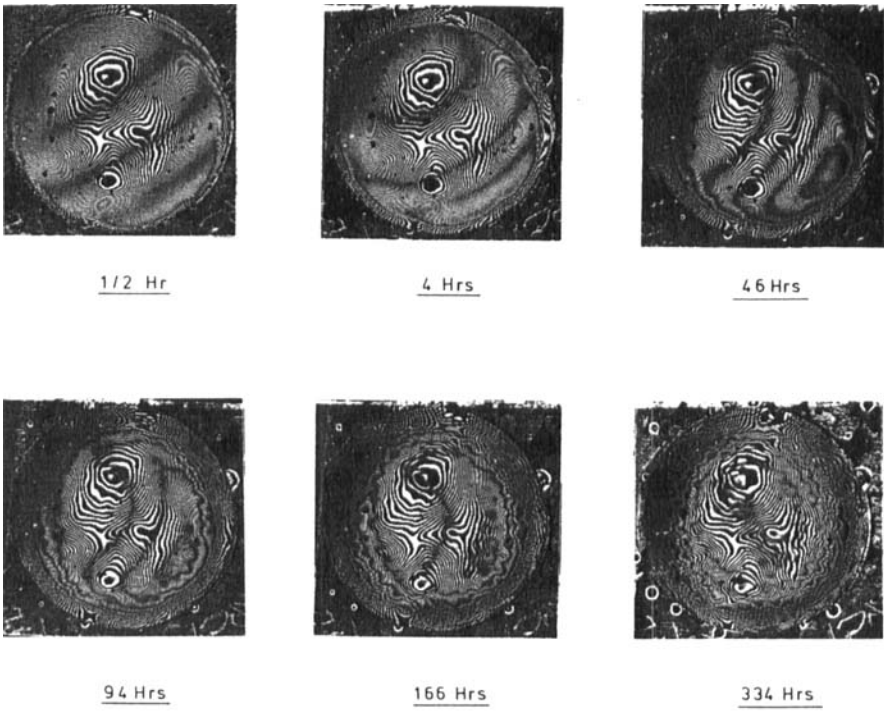
Circular specimens were prepared by bonding 0.3 mm thick, 19 mm diameter cover slips to a rigid block of glass using an epoxy resin adhesive. The sequence of moire pattern development shown in Figure 4 is for a specimen manufactured using a nylon modified epoxy film adhesive (FM 1000, Bloomingdale Rubber Co) cured at 170°C and immersed in water at 62.5°C. The moire fringes in Figure 4 reveal that there exists a circumferential region of swollen resin with a water concentration that decreases from the rim of the specimen towards its centre.



1/4 Hr 2 Hrs 4 Hrs

8 Hrs 28 Hrs 48 Hrs

FIGURE 4 Moire fringes generated by an FM 1000 adhesive joint at various times of immersion in distilled water at 62.5°C.



1/2 Hr

4 Hrs

46 Hrs

94 Hrs

166 Hrs

334 Hrs

FIGURE 5 Development of pertubations in the Moire pattern for a Redux 312/5 joint in distilled water at 62°C.

This behavour is typical of that found for a range of adhesive films.[3] The swelling is non-uniform and leads to considerable self stressing in the joint.

The so-called hygro-coefficient of expansion for FM 1000 is very large (12% linear expansion at saturation) and dominates the observed displacement in Figure 4. That is, the suspected different rate of water uptake and expansion within the material of the carrier cloth is not resolved. Experiments with Redux 312/5 adhesive (CIBA-GEIGY Ltd), which has a much lower hygro-coefficient of expansion, give rise to regular pertubations in the moire fringes that match the mesh size of the nylon carrier cloth in Figure 5. This demonstrates the sensitivity of our technique to detect differences in expansion behavior of multi-component systems exposed to hostile environments.

Effects of re-entrant corners[1]

Figure 6 shows a sequence of a moire fringe pattern generated during immersion in water at 60°C of a model FM 1000 adhesive joint constructed using a square cover slip. The square geometry was chosen as model for exploring the behavior of an adhesively bonded hinge exposed to aqueous environments. The observed behaviour is typical of that found for a range of adhesive joints with one or both adherends having re-entrant corners. The large hygro-coefficient of expansion for

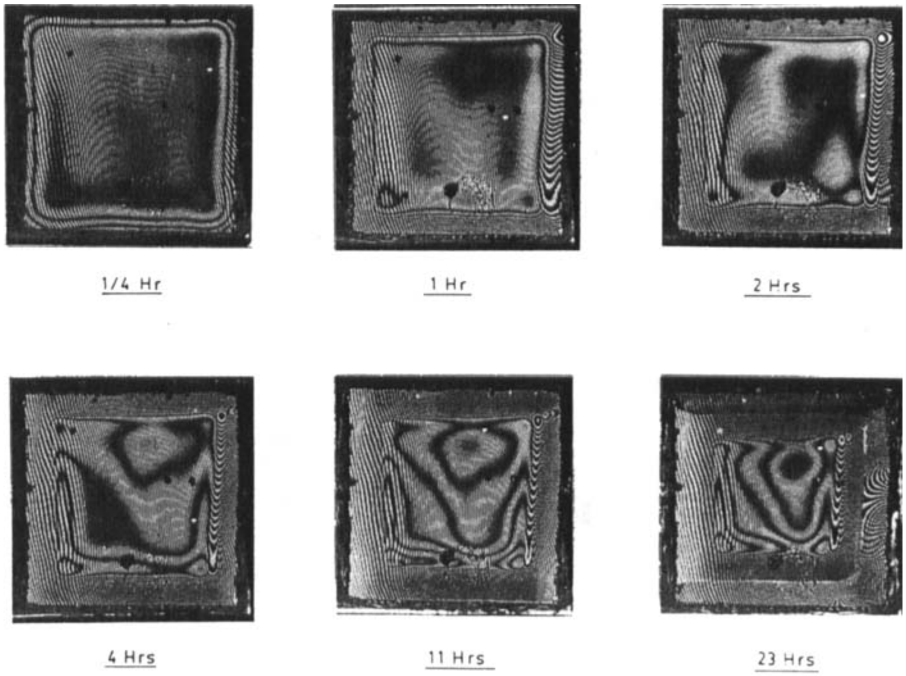


FIGURE 6 Moire pattern for an FM 1000 adhesive joint using a square cover slip immersed in water at 60°C.

FM 1000 gives rise to large tension in the glass in the vicinity of the swelling front and eventually fracture of the cover slip occurred after 23 hours. By taking the fourth differential of the normal displacement measured across the cover slip, the distribution of normal stress was calculated using Eq. (1). The distribution of normal stress is characterized by two regions of large compression, one near the edge of the cover slip ($50\,\mathrm{MN\,m^{-2}}$) and the other just ahead of the swelling front ($60\,\mathrm{MN\,m^{-2}}$). Between these two regions of compression there exists a region of normal tension ($70\,\mathrm{MN\,m^{-2}}$) close to the locus of zero displacement.

Joints involving composite materials[4]

Figure 7 shows a series of moire images for an adhesive joint (Redux 312/5) between a square $150\,\mu\mathrm{m}$ thick glass cover slip and a $0 \pm 63°$ 57-ply laminate manufactured from Modmor type 1 carbon fiber and MY750 epoxy resin. The joint was immersed in distilled water at 60°C. The water uptake in this case was uniform but very rapid in comparison with an identical experiment performed on a similar joint between a cover slip and an undirectional S2 glass fiber reinforced epoxy resin composite. In the latter experiment it was found that water uptake was markedly non-uniform, penetrating the joint more rapidly in the fiber direction than across the fibers.

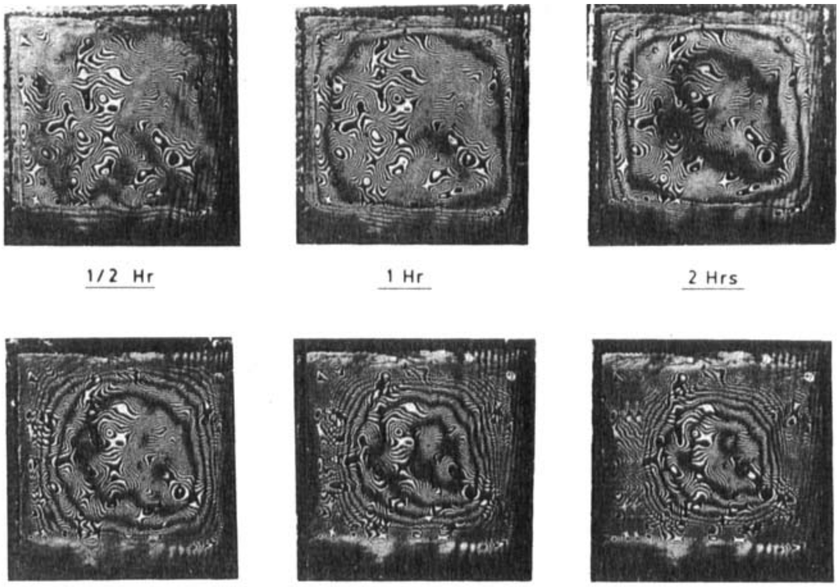
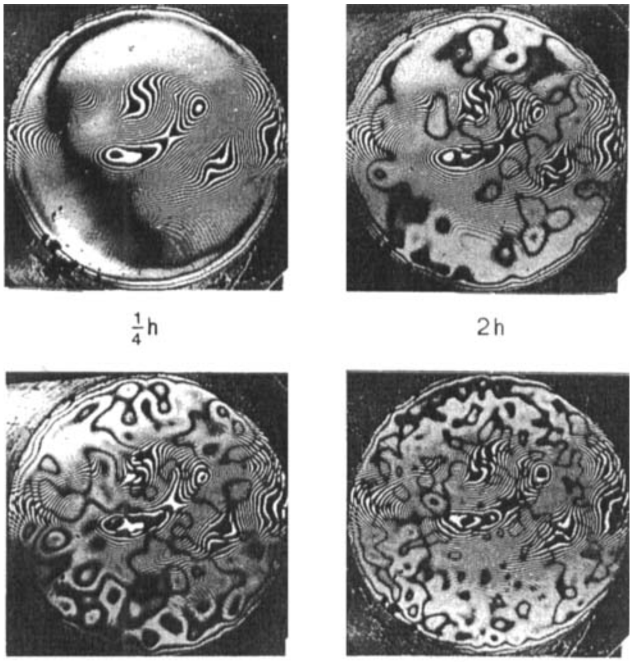

1/2 Hr
1 Hr
2 Hrs
8 Hrs
33 Hrs
49 Hrs

FIGURE 7 Sequence of moire pattern development for a 0 ± 63° 57 ply CFRP specimen immersed in distilled water at 60°C. The cover slip is mounted parallel to the plane of the laminate.

In a further study,[5] direct evidence, from photoelastic images around partially dissolved water soluble inclusions, was presented for concluding that an important source of adhesive swelling is osmosis. The migration of water to soluble impurity inclusions gives rise to pockets of aqueous solution. It is the osmotic pressure associated with these pockets of solution that stresses the surrounding resin and results in optical birefringence. Exposure to saline environments reduces the difference between the chemical potential of water in the pockets of solution and that of water in the aqueous environment outside the joint, and hence lowers the magnitude of the osmotic pressure at the inclusion. In all of this, the adhesive behaves as a semi-permeable membrane.

Changes in bond-line thickness in joints exposed to organic solvents[5]

Many epoxy-based adhesives and composites are exposed not only to water but also to a variety of organic solvents. The use of resin bonded fiberglass underground gasoline storage tanks has, for example, drawn attention to the mechanisms and consequences of migration of petroleum products through resins. Figure 8 shows examples of a moire fringe pattern generated from interference fringe photographs recorded at four different times of an immersion of an FM 1000 joint in dried *n*-octane at 60°C. Circumferential moire fringe loops develop near the edge of the cover slip, and isolated closed loops of moire fringes form throughout the joint. By applying mechanical pressure to the joint, it was



$\frac{1}{4}$h 2h

4h 48h

FIGURE 8 Moire pattern for an FM 1000 specimen undergoing uptake of dried *n*-octane at 60°C.

established that the circumferential loops are due to resin shrinkage and that the isolated loops in the central region are due to resin swelling. That is, the coverslip in Figure 8 conforms to a contorted shape consisting of a dome-shaped deformation all around its edge, plus small isolated dome-shaped deformations well inside the edge.

The adhesive underlying each of the isolated domes contains numerous small pressure pockets. The sign of the optical birefringence seen in a polarising microscope corresponds to that for compression in the resin. Closer examination has shown that the lobes of birefringent contrast are associated with internal impurity inclusions. Osmosis at octane soluble impurity inclusions is believed to to be responsible for the creation of these internal pressure pockets.

The time taken for moire loops to appear at the centre of the joint has been used to estimate the rate of migration of octane through FM 1000 joints. Table I lists, for each of four temperatures, the migration rate so deduced. The slope of migration rate *versus* reciprocal temperature gives as estimate for the activation energy,

$$Q = 14 \text{ kcal mole}^{-1}$$

Assuming Fickean diffusion then D, the diffusion coefficient, is given approximately by

$$D = x^2/t$$

TABLE I

T (°C)	$^1/_T$ (K^{-1})	permeation rate (cm h^{-1})	permeation rate (m s^{-1})
80	2.8×10^{-3}	$\frac{1\,(\text{cm})}{1/4\,(\text{h})} = 4$	1.1×10^{-5}
60	3.0×10^{-3}	$\frac{1\,(\text{cm})}{2\,(\text{h})} = 0.5$	1.4×10^{-6}
44	3.2×10^{-3}	$\frac{1\,(\text{cm})}{54\,(\text{h})} = 0.02$	5.2×10^{-8}
20	3.4×10^{-3}	$\frac{1\,(\text{cm})}{>672\,(\text{h})} = <0.0015$	4.2×10^{-9}

where x is the diffusion distance at time t. Inserting $x = 1$ cm and $t = 900$ s, $D = 10^{-3}\,\text{cm}^2\,\text{s}^{-1}$ at 80°C. This is two orders of magnitude larger than the diffusion coefficient for migration of water in this adhesive at 80°C. It is evident that n-octane is able to migrate through this particular epoxy resin at very high rates.

Figure 9(a) shows moire fringes for a joint that has been immersed half in

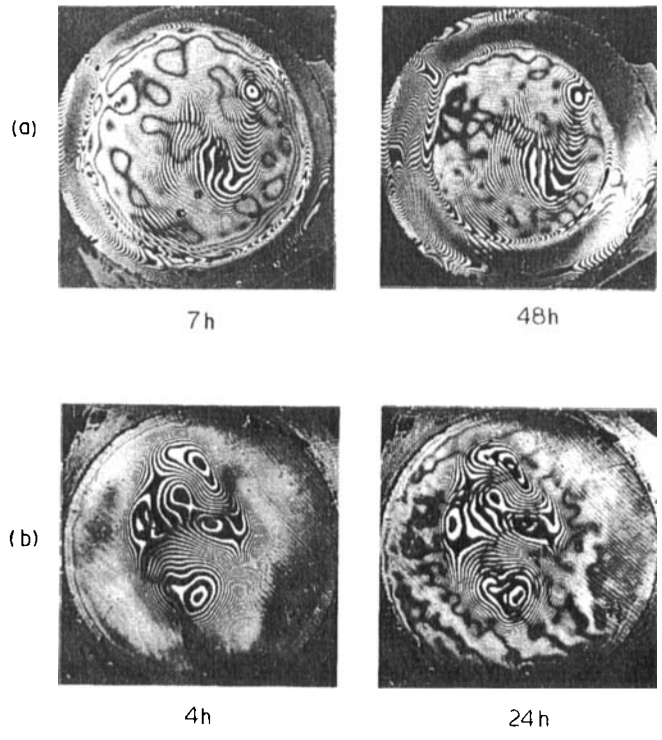


FIGURE 9 (a) Same as Figure 8 for immersion of the lower half of the specimen in water and the upper half in kerosene at 60°C, and (b) for immersion of the entire specimen in kerosene at 61°C.

commercial kerosene and half in water for 48 hours at 60°C; the water/kerosene interface is horizontal and coincides with the horizontal diameter of the vertically mounted joint. For comparison, Figure 9(b) shows a similar joint that has been immersed in kerosene alone. In both cases the time taken for moire loops to appear at the centre of the joint is the same as that for the joint immersed in dried *n*-octane at 61°C. The main difference between the water/kerosene and kerosene experiments is that the circumferential moire fringes for the former case, Figure 9(a), correspond to swelling, not shrinkage.

Application to delamination problems in thick laminates, specifically multi-layer circuit boards[6]

In general, the thermal expansion behaviour of multilayer laminated circuit boards is highly anisotropic. Variations in temperature give rise to expansions and contractions which, because they are not homogeneously distributed, cause the board to become self-stressed. Most laminated circuit boards have large "through the thickness" thermal expansion coefficient. In the vicinity of a copper plated through hole, the fact that the "through the thickness" thermal expansion coefficient of the board may be two or three times larger than that of the copper sleeve deposited inside the hole, gives rise to complex localized stress.

The moire interference technique has been used to measure the local displacement field around a plated through hole during both soldering and subsequent temperature cycling. The substrate in the model adhesive joint described in Figure 2 was a section of 2.5 mm thick 11-ply polyimide-glass fiber multilayer circuit board containing a 0.8 mm diameter copper through-plated hole. The thickness of the copper plating was 30 μm. In this experiment the cover slip bond-line thickness remains constant. The adhesive transmits the underlying deformation to the glass cover slip, which is bonded over the hole. As in earlier experiments, the deformation of cover slip can be measured using the moire interference method and hence used to estimate the through thickness stress acting normal to the plane of the board.

Figure 10 shows the development of moire fringe loops concentric with a plated through hole for a simulated 3 seconds soldering operation. Heat was transmitted to the hole region through a short piece of copper wire located inside and standing proud of the under side of the specimen assembly. This was contacted by a soldering iron for approximately 3 seconds in which time the temperature in the copper wire and its immediate surroundings increased to about 210°C.

Thermal cycling experiments were carried out by mounting the circuit board/specimen assembly on a temperature-controlled brass block. The maximum and minimum temperatures were −40°C and 50°C respectively and the period for one cycle was four hours.

To ensure that the observed relative normal displacement in the locality of the hole is due to the existence of thermal mismatch between the copper and the circuit board and is not, for example, simply an out-of-plane mainfestation of the

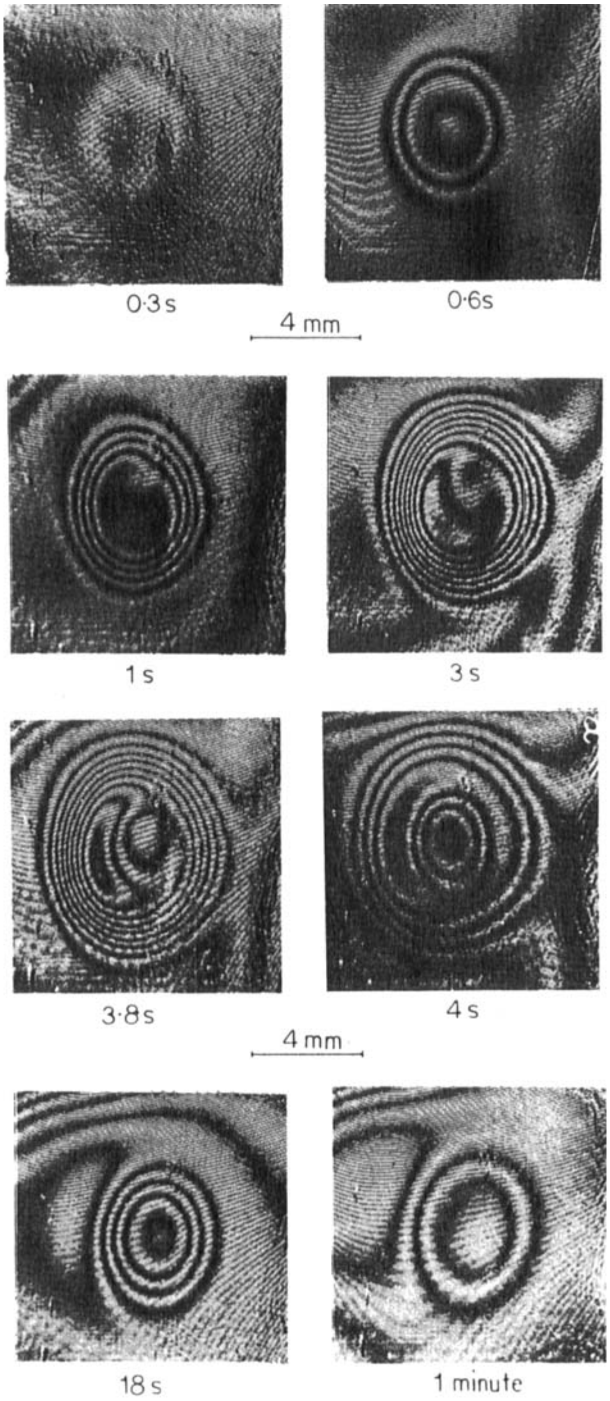


FIGURE 10 Moire pattern generated from photographic images of multi-layer circuit board specimen, recorded for a 3 second soldering operation. The elliptical form of the moire fringes derives from use of a non-parallel sided laser beam splitter. The true fringe diameter corresponds to that of the minor axis.

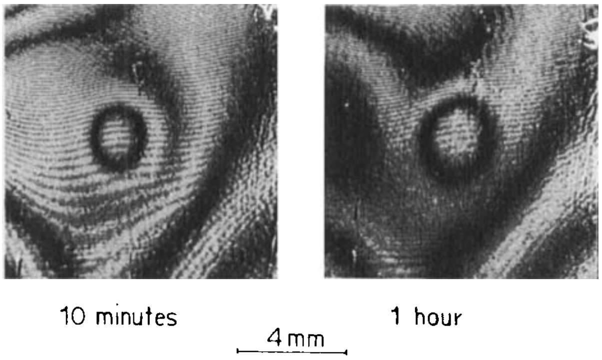


FIGURE 10 (*Contd.*)

highly anisotropic thermal expansion behavior of the circuit board laminate, the thermal cycling experiment was repeated for a cover slip bonded over a hole with no copper plating in place. No relative displacement was observed in the vicinity of the hole at any point in the cycle. This also confirms that the thin adhesive film between cover slip and circuit board does not itself give rise to any significant through thickness displacement.

Surprisingly, results indicate that the plated-through hole region experiences less stress during soldering operations than during thermal cycling. In all cases the normal stress distribution across a diameter was of the form shown in Figure 3. At high temperatures there exists a circular locus of tensile normal stress in the laminate at a radius less than but immediately adjacent to the edge of the pad. This gives way to a locus of normal compression in the laminate alone, at a radius slightly greater than that for the outside edge of the pad. At temperatures below room temperature, these stresses are reversed. The observed stresses at the hole edge are well in excess of the yield strength of OFHC copper (1 MN m^{-2}). It is concluded from this that, during soldering and during in-service temperature cycling, the copper sleeve plastically deforms in order to accommodate expansion of the laminate.

For thermal cycling of unsoldered holes, the state of deformation in the cover slip before and after the temperature cycle is identical. This demonstrates that a single temperature cycle introduces no measurable permanent axial displacement of the plated through hole region; it returns to its strain-free and therefore stress-free state at room temparature. However, the deformation generated during cycling of soldered samples is irreversible; there remains significant displacement around the hole after completion of the cycle. In addition, the maximum stresses experienced at 50°C by soldered samples are about twice that of unsoldered samples.

SUMMARY

A moire optical interference technique has been developed and used to measure small displacements of relevance to adhesive joints and to plated-through hole technologies. The method is ideal for any application involving small changes in shape of a smooth, predominantly planar and optically reflective surface. Attention is drawn to the sensitivity of the technique. Displacements as small as $\lambda/10$ can be determined *via* standard numerical analysis of recorded fringe patterns.

Acknowledgements

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