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National Research Council of Canada
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An overhanging beam method for measuring internal stress in coatings

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Summary

The internal stress in polystyrene and polyisobutyl methacrylate lacquers was measured by coating a thin steel substrate on one side and measuring its bending as the coating dried. The substrate was supported simply at two points, so calculated that the deflection at the centre of its span was independent of weight changes in the coating, where the bending displacement was due only to stress in the coating. Internal stress proved to be large and independent of dried coating thickness and initial solution concentration and the results obtained agreed well with internal strain data obtained previously. The simply supported overhanging beam configuration used here seems to be more accurate than a cantilever arrangement, where the constraints imposed by the end clamp result in values for internal stress that are too low.

Keywords

Equipment primarily associated with analysis, measurement or testing

overhanging beam balance

Miscellaneous terms

stress

Properties, characteristics and conditions primarily associated with

dried or cured films

shrinkage

Types and classes of coatings and allied products

clear coating
lacquer

Raw materials for coatings binders (resins etc)

methacrylate resin
polystyrene resin

solvents

toluene

Une méthode utilisant une lâme en portique pour mesurer la tension au sein des revêtements

Résumé

On a déterminé la tension au sein des vernis à base de polystyrène ou de méthacrylate de polyisobutyle en les appliquant à une face de minces supports en acier et en mesurant la déformation du support lors du séchage du revêtement. Le support était soutenu simplement à deux endroits, dont l'emplacement avait été calculé de façon que la flêche au centre de la portée était indépendante des changements de poids du revêtement, et où la déflexion était due seulement aux contraintes au sein du revêtement. La tension se montrait importante et indépendante de l'épaisseur du revêtement sec et aussi de la concentration initiale de la solution. Les résultats étaient en accord avec les données sur las tension que l'on a obtenues antérieurement. Le système d'une lâme en portique simplement soutenue, que l'on a utilisé au cours de cette étude paraît plus précis qu'une configuration en cantilever, où les contraintes imposées par la crampon au bout rendent les trop faibles valeurs de la tension.

Eine Oberbalkensmethode zur Bestimmung der Innenspannung in Beschichtungen

Zusammenfassung

Bei der Ausbiegungmessung eines dünnen Stahlsubstrats während des Trockens einer Beschichtung, wurde die Innenspannung von Polysyrol und Polyisobutylmethakrylatlacken bestimmnt. Das Substrat war an zwei Stellen unterstützt, damit die Ausbiegung an dem mittelpunkt der Spannbreite unabhängig von der Gewichtsänderungen der Beschichtung war, und der Ausbiegungsgrad kam ausschliesslich von der Beanspruchung her. Die Innerspannung zeigte sich wichtig und unabhängig von der Dicke der trockenen Beschichtungen und von der Anfangskonzentation. Die erhaltenen Ergebnisse standen in guten Ubereinstimmung mit den Innenspannungsdaten, die fruher erhalten sein worden. Das einfach unterstützte Oberbalkensgebilde, das man hier benutzte, scheint genager zu sein, als eine freitragende Einrichtung, wo die von einer Endklammer geschaften Zwänge, Innenspannungswerte zur Folge haben, die zu schwach sind.

Introduction

Refs. 1-7

Internal stress, or strain, has been measured for a variety of coatings and, in many cases, has proven to be large[1,2]. High stresses or strains can reduce the adhesion of a coating considerably and even promote spontaneous peeling or flaking[3]. In some instances the coatings may crack under the influence of internal stress[1]. Thus, it is desirable to determine the presence, magnitude and origin of such stresses.

Measurement of internal stress is usually made by observing the bending of a thin metallic plate coated on one side only. As the film dries and tries to shrink, it bends the plate. The plate can be either clamped at one end, like a cantilever, or supported on knife edges. Use of the knife-edge support method has one intrinsic advantage over the cantilever clamping arrangement[4]. Clamping is thought to

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influence the way in which the cantilever bends. One end of the substrate plate is kept flat, whereas the coating attempts to shrink in all directions and should bend the substrate plate both ways, across and along its length. Supporting the plate on knife-edges does not impose any constraint on the bending.

In addition, most films are applied either from solution or suspension; in both cases there is considerable weight loss during drying. If the substrate plate is oriented so that its plane is horizontal, the loss in weight will produce a deflection due to the action of gravity, which cannot be separated continuously from the deflection due to the increasing internal stress. One can allow for it by weighing the coating wet and dry and making appropriate corrections. An alternative is to use the cantilever arrangement with the plane of the plate and coating vertical. The wet coating would be prone to sagging however, and produce an uneven thickness which renders analysis of the data very difficult. This paper presents a configuration of the knife-edge support method that is completely insensitive to weight changes in the coating so that deflection of the plate is entirely due to the bending produced by the internal stress in the coating. Thus, analysis of the data is simpler and it is not necessary to weigh the film to determine solvent loss.

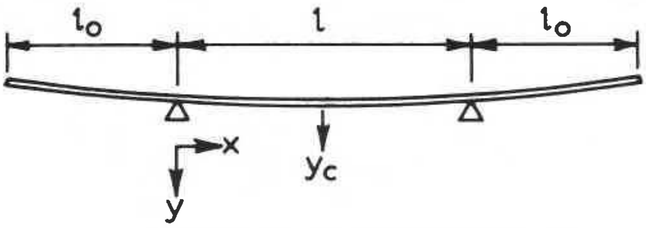

Figure 1. Diagram for overhanging beam analysis

Theory

The principle behind the configuration suggested here (Figure 1) is that the weight loss from the overhanging parts of the beam should provide a moment that opposes the upward deflection of the beam in the centre span due to weight loss there. Lengths l and l_o can be chosen so that the deflection in the centre of the span, due to weight loss, is zero.

Weight change on a simply supported overhanging beam

The coating on top of the plate is responsible for a downward force/length, corresponding to its weight, constant along the length of the beam ($2l_o + l$). If that force is $\omega(\mathrm{Nm}^{-1})$ then the bending moment, M, at the supports as given by Morley[5] is:

$$M = \frac{\omega l_o^2}{2} \qquad (1)$$

For a simply supported beam, e.g., on knife-edges, with a uniformly distributed load:

$$\omega = EI\frac{d^4y}{dx^4} \qquad (2)$$

where E = modulus of elasticity of the beam material
I = moment of inertia of the area of the cross-section of the beam about its neutral axis.
y, x = Cartesian co-ordinates, as in Figure 1 (the origin lies at the left-hand support).

In practical situations the coating is much thinner than the substrate and its modulus is about two orders of magnitude less. Its effect on the bending of the composite beam, therefore, is assumed negligible for this calculation. The deflection, y, of the beam can be found by integrating Equation (2):

$$EI\frac{d^2y}{dx^2} = \frac{\omega x^2}{2} + Ax + B \qquad (3)$$

A and B are constants of integration which can be determined at this stage by applying the boundary conditions[5]. When $x = 0$ and l:

$$EI\frac{d^2y}{dx^2} = M = \left(\frac{\omega l_o^2}{2}\right) \qquad (4)$$

Thus $B = M$ and $A = -\frac{\omega l}{2}$.

Integrating Equation (3) further, the expression for the deflection is given by:

$$EIy = \frac{\omega x^4}{24} - \frac{\omega l}{2}\cdot\frac{x^3}{6} + \frac{Mx^2}{2} + Cx + D \qquad (5)$$

Now the integration constants C and D must be determined. At $x = 0$ the deflection $y = 0$, as at $x = l$.

Thus $D = 0$ and

$$C = \frac{\omega l^3}{24} - \frac{Ml}{2} \qquad (6)$$

The deflection at the centre of the span, y_c, can now be written out substituting Equations (6) and (1) in (5), i.e., at $x = l/2$:

$$EIy_c = \frac{\omega l^2}{16}\left(\frac{5l^2}{24} - l_o^2\right) \qquad (7)$$

If this deflection is to be insensitive to changes in weight, i.e., $dy_c/d\omega = 0$, then it follows from Equation (7) that:

$$l_o^2 = \frac{5l^2}{24} \qquad (8a)$$

or

$$l_o = 0.4564l \qquad (8b)$$

Therefore, if the ratio of the overhanging length, l_o, to the span, l, is chosen according to Equation (8b), the deflection of the beam at the middle of the span caused by internal stress will not change if the weight changes. A similar arrangement has been used before[6] but the appropriate overhang, l_o, was assumed to be half the span length and not 45.6 per cent as indicated by the full analysis presented here.

Calculation of internal stress

The displacement of the substrate, d, as it bends allows the equivalent bending radius, r, to be calculated:

$$r = \frac{l^2}{8y_c} \quad \text{.......(9)}$$

where l = length of the span, as in Figure 1
y_c = deflection measured at the centre of the span.
Internal stress, σ, can then be calculated[7] using:

$$\sigma = \frac{Et^3}{6t_c r(t+t_c)\ (1-\nu)} \quad \text{.......(10)}$$

where E = Young's Modulus of the substrate.
t = thickness of the substrate.
t_c = coating thickness.
ν = Poisson's ratio for the substrate.

The expression for σ does not require knowledge of the mechanical properties of the coating. A full analysis of this experiment shows that there should be additional terms to Equation (10) involving the properties of the coating. In general, the extra terms are negligible[7] under normal conditions where $t \gg t_c$ and the modulus of the coating is two or more orders of magnitude less than that of the substrate (usually steel). The bending of the substrate is caused by the stress in the coating but the response of the beam is dominated by the metallic substrate.

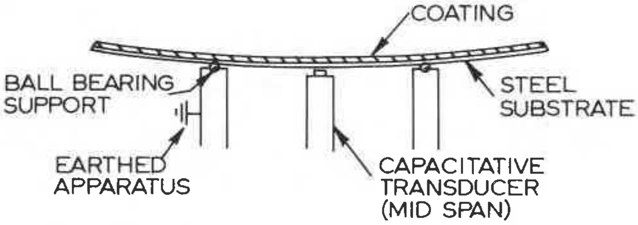

Figure 2. Schematic of the apparatus for measuring internal stress

Experimental

Refs. 3, 5

All the experiments were conducted in a room maintained at 23°C (±1°C) and 50 per cent RH (±2 per cent). The coatings investigated were polystyrene, PS (Dow Styron 685), and polyisobutyl methacrylate, PIBM (Elvacite 2045), cast from solutions in reagent grade toluene. Results were obtained using a variety of coating solution concentrations.

Internal stress

The apparatus used to measure the internal stress is drawn schematically in Figure 2. A non-contacting capacitive transducer system (Wayne Kerr Dimeq TE200, fsd 200 μm, accuracy 0.5 per cent) was used to measure the displacement at the centre of the span. These transducers are very sensitive and permit the use of comparatively thick substrates that do not bend much and are easier to handle. The substrate (steel feeler gauge stock, 12.7 mm wide) was earthed (to form the other electrode of the capacitor) through the 1/16 in. ball bearings used as supports. Three ball bearings were used, two at one end of the span, in a tripod support system.

The central span length used was 50 mm; the total length of the substrate was 95.64 mm (overhang calculated by Equation 8). Use of a continuously recording strip chart to record the output from the transducers proved necessary because deflection caused by internal stress began almost immediately.

Coating thickness was measured using an Elcometer Minitector FN.

Substrate Properties

Substrate thickness was measured by micrometer. The modulus of the steel substrates varied enough to warrant measuring for each one used. If one end of the strip is clamped in place and a weight, F, is attached to the other end then the deflection, y, along the cantilever is given by Morley[5]:

$$y = \frac{12F}{Et^3b}\left(\frac{Lx^2}{2} - \frac{x^3}{6}\right) \quad \text{.......(11)}$$

b = width of the cantilever
t = thickness of the cantilever
L = length of the cantilever
x = position co-ordinate along the cantilever, zero at the clamp, $x = L$ at the end with the weight.

All the quantities in Equation (11) can be measured, permitting E to be determined. The value of E was typically 1.9 GPa. Poisson's ratio, ν, of the steel was taken to be 0.29.

Results

The final, equilibrium, value of internal stress is plotted as a function of dried coating thickness in Figure 3(a) for the PIBM coatings and 3(b) for the PS coatings. There is no discernible trend with coating thickness for either coating, over the range of thicknesses employed.

Mean value of residual stress in PIBM =	5.6 MPa
standard deviation =	0.3 MPa
Mean value of residual stress in PS =	17.28 MPa
standard deviation =	0.67 MPa

PS coatings thicker than 18 μm spontaneously detached[3] from the substrate and so could not be used to measure internal stress. Thinner coatings of PS adhered, but frequently crazed under stress, a well-known phenomenon.

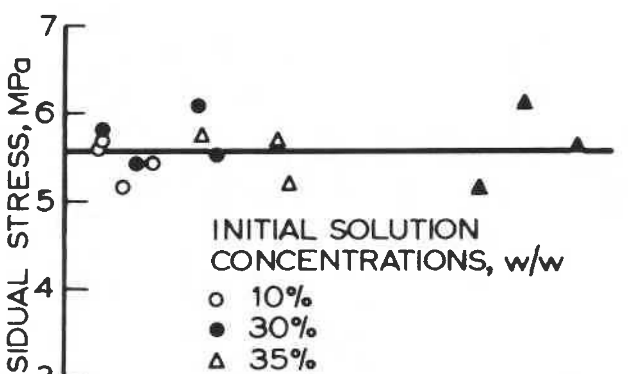

Figure 3(a). Dependence of residual internal stress on coating thickness and solution concentration in PIBM coatings

Before a coating is completely dried, the solvent content will vary with depth and thus internal stress will also vary through the coating. Consequently, deflection of the beam at intermediate times gives a value of internal stress averaged through the thickness of the coating; examples are given in Figures 4(a) and 4(b).

Discussion

Refs. 3, 8

In both lacquers the residual stress is independent of dried coating thickness and initial solution concentration over the ranges tested. The same features were present in residual strain measurements published previously[8].

Additionally, the magnitudes of the residual strain (ϵ) and stress can be compared. Stress-strain curves obtained for both PIBM and PS coating films were used in a previous study[3] and are reproduced here as Figures 5(a) and 5(b). Values obtained for the residual strains[8] are:

PIBM, $\epsilon = 5.8 \times 10^{-3}$ ($\pm 4.4 \times 10^{-4}$)
PS, $\epsilon = 1.75 \times 10^{-2}$ ($\pm 1 \times 10^{-3}$)

Using Figures 5(a) and 5(b), these correspond to stresses (σ') of:

PIBM, $\sigma' = 3.4$ MPa
PS, $\sigma' = 10.9$ MPa

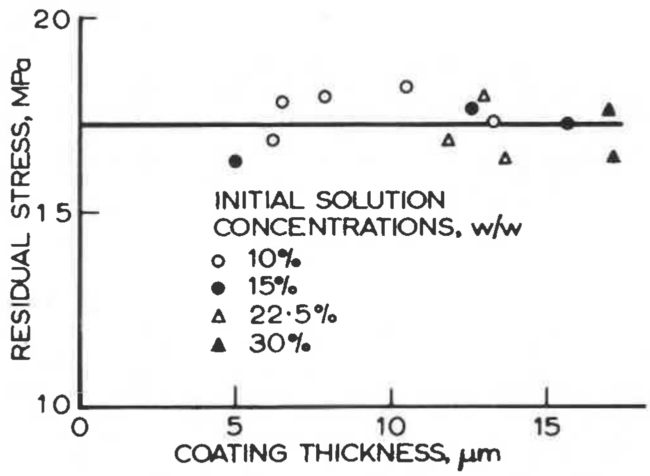

Figure 3(b). Dependence of residual internal stress on coating thickness and solution concentration in PS coatings

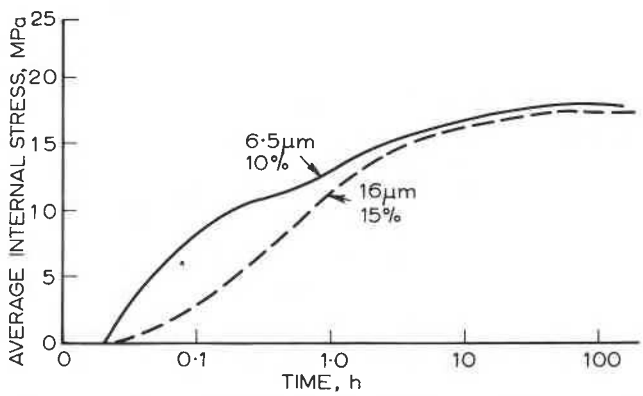

Figure 4(a). Development of internal stress in PIBM lacquers, showing initial solution concentration (% w/w) and dried coating thickness (μm)

Now the shrinkage in the coating is isotropic, so the film has two equal stresses or strains at right angles in the plane of the coating. In this plane stress situation

$$\sigma = \frac{E(\epsilon).\epsilon}{1-\nu} \quad \text{..........(12a)}$$

or

$$\sigma = \frac{\sigma'}{1-\nu} \quad \text{..........(12b)}$$

This adjustment is necessary because the residual strain and stress-strain curves are measured uniaxially whereas the bending plate determination of residual stress measures the combined effect of the stresses in the plane of the coating.

Poisson's ratio for the coatings has been given before[8] as 0.4 for PIBM and 0.39 for PS. Equation 12(b) predicts that the residual stress measured should be 5.67MPa for the PIBM and 17.9 MPa for PS. The agreement is very good between values predicted from the residual strain and the values for residual internal stress measured. Crazing in PS does not influence the agreement between experiments.

Previously, the residual stress was measured for the same two lacquers using a cantilever substrate[8]. Values obtained then were 4.5 MPa for PIBM and 14.3 MPa for PS, about 20 per cent low in both cases. The discrepancy is probably due to the clamping of one end of the substrate, which restricts its bending. Presumably it restricts the bending arising from the component of stress across the width of the beam.

Internal stress can be followed better as it increases with time in this type of experiment where changes in coating weight have no effect on the beam deflection. However, the experiment gives only the average value of internal stress because the coating dries at a rate that varies according to depth. This particular method seems to be more accurate and rather simpler than other, similar, methods. The coating can be horizontal, keeping its thickness even, and there is no need to weigh it to determine the deflection caused by weight change.

As in investigations carried out previously on solvent cast coatings, the residual internal stresses or strains have been shown to be considerable. For PS coatings the internal stress causes adhesive loss even in very thin coatings and is close to the maximum stress the films can bear (Figure 5), and which, in fact, often craze. Although the

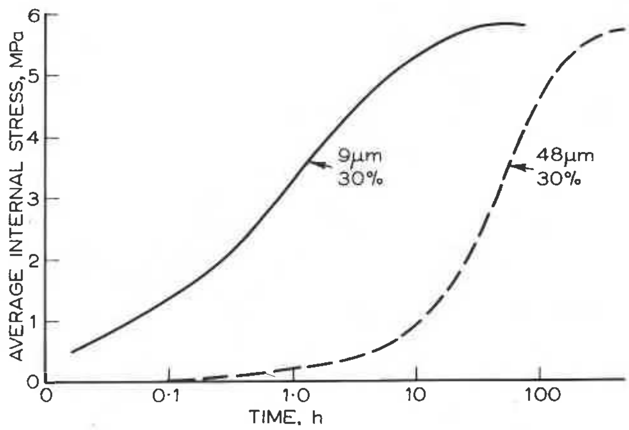

Figure 4(b). Development of internal stress in PS lacquers, showing initial solution concentration (% w/w) and dried coating thickness (μm)

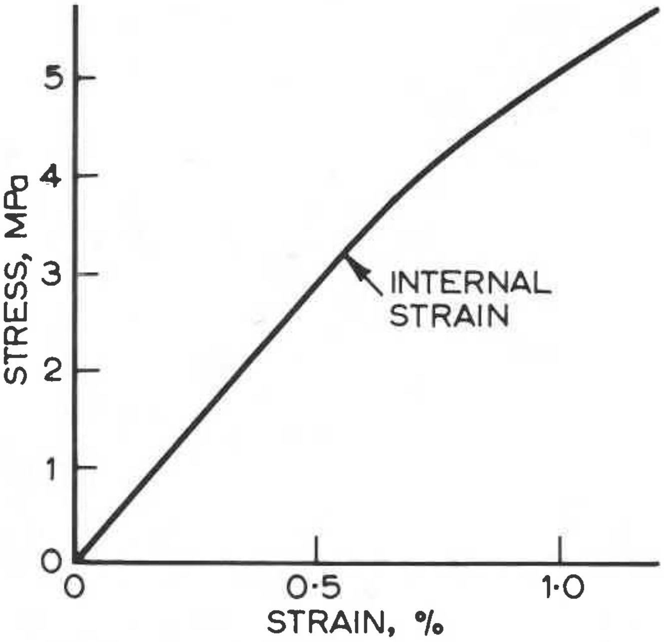

Figure 5(a). Uniaxial, relaxed stress–strain relationship for PIBM films

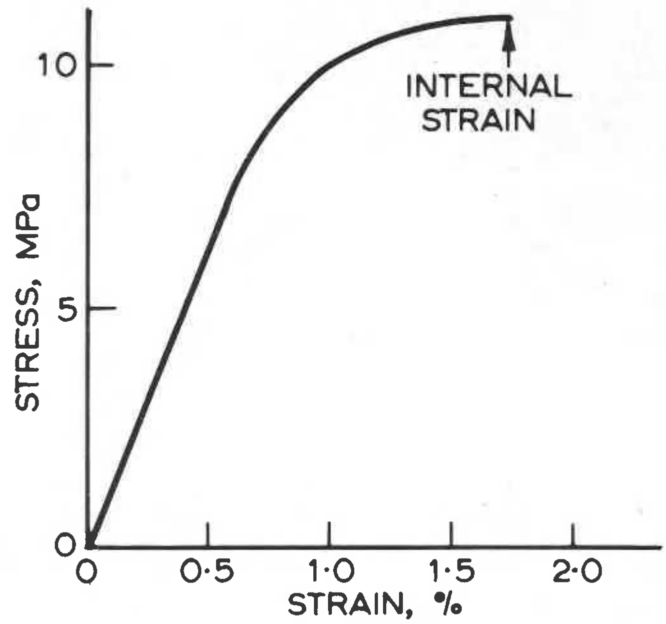

Figure 5(b). Uniaxial, relaxed stress–strain relationship for PS films

adhesion of PIBM coatings is good, and the stress much lower than for PS, the residual stress is still a significant fraction of the maximum stress the films can withstand.

Conclusions

The overhanging beam configuration adopted here proved to be a simple and accurate apparatus for measuring internal stress in coatings. The length of the centre span and overhang can be adjusted so that there is no deflection at the centre of the span due to weight loss from the coating. Displacement of the beam substrate at that position is due solely to internal stress in the coating. The change of average internal stress with time can be monitored well because the changing weight of the coating has no influence on the measurement.

As in the case of internal strain measurements, the residual stress proved to be independent of dried coating thickness and initial solution concentration. The magnitude of the residual stress predicted from the residual strain data was in close agreement with the value obtained from the overhanging beam configuration. The two measures of the same phenomenon proved to be consistent.

Employing the substrate as a cantilever yields values for internal stress that seem about 20 per cent lower. Clamping one end probably restricts the bending deflection produced by a given stress in the coating, and hence the cantilever arrangement should be used with caution.

It can be seen, particularly with the PS lacquer, that residual stress poses a considerable threat to the cohesive and adhesive properties of such coatings.

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