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December 1973

JOCCAB 56(12) 555-602 (1973)

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# OURNAL OF THE IL & OLOUR HEMISTS' SSOCIATION

The characterisation of the mechanical properties of paint films by micro-indentation R. L. J. Morris

Finish first—fabricate later. The continuing story of coil coatings D. S. Newton

Powder coatings.

S. T. Harris

Priory House, 967 Harrow Road, Wembley, Middlesex, England





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Published by	5.5.1.4777574935993999										

#### OIL AND COLOUR CHEMISTS' ASSOCIATION Priory House, 967 Harrow Road, Wembley, Middlesex, England HA0 2SF

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#### **Transactions and Communications-**

## The characterisation of the mechanical properties of paint films by micro-indentation\*

#### By R. L. J. Morris

Materials Quality Assurance Directorate, Ministry of Defence, Royal Arsenal East, London SE18 6TD

#### Summary

Use of the micro-indentation technique is reviewed from the theoretical and phenomenological standpoints. The lack of a universally constant slope in the logarithmic method of presenting load-indentation behaviour of paint films is shown to be due to a deficiency in this form of data presentation. The lack of sensitivity of this method to film thickness variation can also be attributed to this deficiency. The IHV method has an inherent uncertainty associated with the extrapolation of empirically derived curves.

#### Keywords

Process and methods primarily associated with analysis, measurement and testing

indentation test

Another method, which relates to the temperature dependency of indentation depth at a constant load and time under load, produces curves having a characteristic shape during the early period of exposure of the artificial weathering for an acrylic and a tall oil alkyd film but the shape changes during the later periods of exposure. A stoving melamine-epoxide medium shows a different behaviour in this respect.

Properties, characteristics and conditions primarily associated with dried or cured films mechanical property

### La caractérisation des propriétés mécaniques de feuils de peintures au moyen de la technique de micre-indentation

#### Résumé

On passe en revue au point de vue théorique et phénoménelogique, l'emploi de la technique de micro-indentation. On démontre que la manque d'une pente entièrement constante à partir de la méthode logarithmique pour présenter le comportement charge-indentation des feuils de peintures est due à une déficience de la manière de présenter les données. A l'égard de cette méthode, la manque de sensibilite aux variations de l'épaisseur de feuil peut aussi être attribuée à cette déficience. La méthode I.H.V. entraîne une incertitude inhérente associée à l'extrapolation des courbes dérivées

#### Charakteristik der mechanischen Eigenschaften von Lackfilmen durch Mikroindentation

#### Zusammenfassung

Die Anwendung der Mikro-Indentationstechnik wird vom theoretischen und phänomenologischen Standpunkt aus betrachtet. Das Fehlen eines konstanten Gefälles, wenn man das Verhalten von Lackfilmen unter Belastungsindentation auf logarithmische Weise demonstriert, wird als Folge eines Mangels bei dieser Form der Datendarstellung aufgezeigt. Dieser Unzulänglichkeit kann auch die Unempfindlichkeit besagter Methode gegen Variationen in der Filmdicke zugeschrieben werden. Der I.H.V. Methode wohnt eine Ungewissheit inne, die mit der Extrapolierung empirisch

#### Характеризация механических свойств красочных пленок микроиндентацией

#### Резюме

Рассматривается вопрос применения методики микро-индентации с теоретической и феноменологической точек зреняя. Отсутствие универсально постоянного наклона в логаритмическом методе в иллюстрации поведения красочных пленок под нагрузкой, объясняется недостатками в таком воспроизведении данных. Отсутствие чувствительности в этом методе к изменению в толщине пленки может также быть приписано этому недостатку. Метод ничтожной твердости (I.H.V.) обладает присущей ему неопределенностью связанной с

#### Introduction

The physical testing of paints has generally been conducted by attempting to reproduce, in one standard form, the type of behaviour that the paint is required to meet in practice. dans une manière empirique. Une autre méthode, liée à la dépendance thermale de la profondeur d'indentation sous charge constante pour une durée constante, rend des courbes ayant une forme caractéristique, dans le cas des feuils acryliques ou alkydes à l'huile de tall, pendant la première période de l'exposition au vieillissement accéléré, mais la forme se change pendant les dernières périodes de l'exposition. A cet égard un milieu à base de mélamineépoxyde pour séchage au four montre un comportement différent.

abgeleiteter Kurven in Zusammenhang steht. Eine andere Methode, welche mit der Temperaturabhängigkeit der Indentationstiefe bei konstanter Belastung und Zeit in Verbindung steht, ergibt bei einem Akrylat und einem Tallölalkydharzfilm Kurven, die während der frühen Exponierungsperiode eine charakteristische Form haben, welchletztere sich jedoch während der späteren Exponierungsperioden verändert. Ein ofentrocknendes Melamin—Epoxid Bindemittel weist diesbezüglich ein anderes Verhalten auf.

экстраполяцией эмпирически полученных кривых. Другой метод связанный с температурной зависимостью глубины индентации при постоянной нагрузке и временем нагрузки, дает кривые имеющие характерную форму в первоначальном периоде выветривания для акриловой и талловой алкидной пленки, но форма меняется в последующих периодах испытания. Сухая меламино-эпоксидная среда обнаруживает различное поведение в этом отношении.

In recent years a trend has been discernible toward less subjective methods of testing, to obtain more quantification of results and a less ambiguous interpretation in terms of polymer properties. If benefits can be shown to exist then this trend will surely develop.

<sup>\*</sup> Presented at the 1973 Eastbourne Conference.

In the context of mechanical properties, the paint technologist, as distinct from the pure research worker in this field, requires answers, among others, to the following questions.

What are the mechanical properties required of a paint for a particular purpose?

How can the mechanical properties of selected paints best be characterised?

How will these properties change on ageing and at what rate?

One approach to answering these questions is to define appropriate fundamental mechanical properties of the paint film and relate these to film behaviour and performance. Using this approach, it is obviously desirable to relate any particular definition of mechanical property to current methods of its assessment in order to maintain continuity of technical experience. This is likely to be difficult because of the large number of interrelated mechanical parameters associated with the more empirical methods of testing, but that should not be allowed to inhibit the attempt.

Two methods are possible; one relates experimental data to theoretical studies, and the other takes a phenomenological approach by characterising mechanical properties on an empirical basis and relating these to practical events. This paper reviews the author's experience of paint film indentation testing and shows how, in an ideal state, indentation techniques can lead to an understanding of, and characterisation of, mechanical properties.

#### Theoretical aspects of the indentation of polymers

### General consideration of polymer properties in terms of indentation characteristics

The mechanical properties of a material can be determined by examining the deformation of a specimen responding to a defined applied stress. Due to the viscoelastic nature of polymers, the relationship between stress and strain is time and temperature dependent, and this dependency must be taken into account when testing those materials, and when interpreting the results. The response of an ideally elastic material, that is one for which the applied stress is accompanied by an instantaneous time-independent strain, can be analysed by determining either the stiffness or the compliance constants of the material. In the case of an anisotropic material, this would involve the determination of 21 independent material parameters. If the assumption of mechanical isotropy is made, then the number of independent material parameters required is reduced to two, and these are commonly identified as modulus constants.

In order to accommodate the viscoelastic nature of polymers, the time dependency of the physical constants must be considered. To a first approximation, this is accomplished by making the assumption that the material is "linearly viscoelastic", which, in physical terms, is equivalent to stating that if two separate stresses are separately applied to a material, the subsequent strains are in the same ratio as the stresses. That is



Fig. 1. Illustration of linear viscoelasticity for the case where  $\sigma_1 = 2 \sigma_2$ 

In order to be able to test the assumption of linear viscoelasticity with respect to the mechanical behaviour of paint films, a technique is required for applying a known stress to a system and for measuring the strain as a function of time. A number of workers <sup>1-7</sup> have studied the tensile stress-strain creep and dynamic properties of detached paint films. However, it is possible that the paint film and the substrate together form a system and that their separation increases the artificiality of the conditions, and this reduces the validity of the results obtained.

One technique which has recently attracted attention is that of indentation, which has the advantage of being able to examine the mechanical properties of an attached paint coating. Two constant-temperature methods have been proposed, one using a Vickers pyramidal indentor<sup>8</sup> and the other a spherical indentor<sup>9</sup>.

#### The spherical indentor

In the case of a spherical indentor, the relationship between the load applied to the indentor, the radius of the indentor and the depth of the indentation, in terms of the modulus of a perfectly elastic material, as derived by Hertz<sup>10</sup> is

$$\frac{E}{1-v^2} = \frac{3}{4} \cdot \frac{mg}{r^{0.5} h^{1.5}}$$
(1)

where E = Young's modulus of the specimen, v = Poisson's ratio, m = load applied, r = radius of the indentor, h = depth of indentation, and g = gravitational constant.

In the case of those materials whose mode of deformation is principally shear (that is, whose shear modulus G is very much less than the bulk modulus), equation (1) reduces to

$$mg = \frac{16}{3} G r^{0.5} h^{1.5}$$
 (2)

Lee<sup>11</sup> extended the Hertz equation to cover linearly viscoelastic deformation and considered that, for a given step loading, the creep shear compliance J(t) of a material which

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deforms principally by shear is related to the time dependence of the depth of indentation h(t) by

$$J(t) \propto [h(t)]^{1.5}$$
 (3)

In general, for viscoelastic polymers<sup>12</sup>, the effect of temperature on creep shear compliance follows a curve of the for any other indentor free from knife edges or points, which would produce a discontinuous stress distribution. It is possible to test the validity of the expression by examining the logarithmic relationship between load and indentation for a spherical indentor<sup>9, 13</sup> of any given size, since from equation (1)



Fig. 2. Effect of temperature on the creep shear compliance of a polymer

form shown in Fig. 2. The portion AB of the curve describes the glassy response of the polymer  $J_g$  at low temperatures, when the value of  $J_g$  is commonly of the order  $10^{-10}$  cm<sup>2</sup> dyne<sup>-1</sup>. BC describes the transition region of the polymer, and is cross-link density dependent. The region CD, the rubbery response, is dependent upon molecular weight; the higher the molecular weight of the polymer the greater is the length of CD. For linear polymers, an increase in temperature will ultimately result in flow as shown by DE. Thus the total creep shear compliance has been described<sup>12</sup> as being due to the sum of three components of shear

$$J(t) = J_g + J_{D(t)} + \frac{t}{\eta}$$
 (4)

where  $J_g$  is the glassy component of shear compliance;  $J_{D(t)}$  is the viscoelastic compliance, and is time dependent;  $\eta$  is the viscosity, and  $t/\eta$  is the creep due to the viscous flow of linear polymers.

This description of the spectrum of mechanical behaviour of viscoelastic polymers can serve as a basis for the interpretation of results from the micro-indentation technique as applied to paint coatings; it being necessary to test the validity of each step before relating theory to practice.

#### Validity of the Hertz equation

The basis for a quantitative relationship between indentation results and mechanical parameters of elastic materials rests at present, and in the case of a spherical indentor, on some variation of equation (1). This equation can also be applied

$$\log mg = \log \left[ \frac{E}{1 - v^2} \cdot \frac{4}{3} r^{0.5} \right] + \frac{3}{2} \log h$$
 (5)

Thus, at a given temperature and for a fixed time under a loaded spherical indentor, the relationship between the logarithm of the load and the logarithm of the indentation depth should be a straight line of the form

$$y = a + bx \tag{6}$$

where a is given by the first term of equation (5) and b should have the value 1.5.

In an attempt to allow the chlorinated rubber films to become stabilised and free from any solvent effects<sup>9</sup>, they were given 14 days' heating at 40°C followed by seven days' conditioning at 23  $\pm$  2°C, 65  $\pm$  5 per cent relative humidity, whilst the alkyd and acrylic films were conditioned for a period of two years at 23  $\pm$  2°C, 65  $\pm$  5 per cent relative humidity. Results from the stabilised films are given in Table 1.

The data to examine this relationship has been obtained using the ICI pneumatic micro-indentor<sup>14</sup> with a spherical indentor, 200 $\mu$ m radius at a temperature of 23°C.

It is evident, from the last column in Table 1, that in all cases there is a good correlation to a straight line relationship of the form of equation (5). If equation (5) completely defined the situation, then the slope of the straight line relationship should be independent of instrumental and polymer parameters and, in addition, be equal to the value

Medium	Film thickness µm	Time under load seconds	Slope	Intercept	Correlation* coefficient
Chlorinated rubber	33 38 46 46 51	120 120 120 120 120 120	1.20 1.42 1.43 1.30 1.42	1.17 0.90 1.03 0.87 0.61	0.98 0.99 0.99 0.99 0.99
Glyceryl alkyd	30	10 30 60	1.19 1.25 1.29	1.67 1.47 1.32	0.99 0.99 0.99
	52	10 30 60	1.27 1.38 1.31	1.45 1.18 1.04	0.99 1.00 1.00
Solvent based acrylic	30	10 30 60	1.11 1.05 1.08	1.96 1.79 1.68	0.99 0.99 0.99
	52	10 30 60	1.03 1.01 1.15	1.44 1.26 1.12	0.99 0.99 0.99

Table 1 The effect of film thickness and time on the Ln Load/Ln Indentation Relationship (200 µm radius indentor at 23°C)

\*Correlation coefficient of the best linear fit.

of 1.5. That this is not so is seen by comparing the results for the alkyd and acrylic films in Table 1: the slope obtained from the alkyd film is always greater than that from the acrylic and both are also different from the value of 1.5 as is predicted by theory. Since the slope of the line does not appear to be significantly affected by the time under load or the film thickness, one might conclude that the slope is a characteristic of the polymer film studied. Were this so, a major difference could be expected between cross-linked and uncross-linked polymer systems, but this does not seem to be the case, since the data in Table 1 shows the slope of the cross-linked alkyd film to lie between the values of the solvent based chlorinated rubber and acrylic films.

Because the initial term in equation (5), which corresponds to the intercept values quoted in Table 1, contains a modulus value, the time dependence of the intercept values is to be expected, since this reflects the modulus-time dependency of polymers. Additionally, in all cases, a marked dependence of the intercept value on film thickness is observed, in that, as the film thickness increases, a decrease in the intercept value occurs indicating a more compliant response of the paint film. The theoretical effect of film thickness on the indentation characteristics of elastic materials has been considered<sup>15, 16</sup> and a treatment by Finkin<sup>17</sup> indicates that

$$E = \frac{9}{16} \frac{mg}{r^3} \frac{mg}{h^3}$$
 within the limits of  
$$0 \leqslant \frac{9}{16H^3E} \leqslant 0.60$$
(7)

where H is the film thickness. Using this limit relation equation (7), then for a spherical indentor of radius 200 $\mu$ m:

$$H \ge 16.8 h^{\frac{1}{2}}$$

Thus, to make full use of the range of the ICI microindentor with a maximum indentation depth of  $6\mu m$ , a minimum film thickness of  $41\mu m$  is required. At present, there is insufficient experimental evidence concerning the validity of this relationship with respect to paint films, and work is in hand to study this further.

#### Effect of ageing on the logarithmic relationship

Panels, coated with either two unpigmented coats of a stoving melamine/epoxide film or two coats of a solvent based acrylic film, have been examined after different periods of exposure to artificial weathering to BS 3900 Part F3. In both cases it was found that the slopes of the calculated best fit straight lines showed an unexpected scatter in that, in the case of the epoxide films, the slope had an overall mean value of 1.49 with a coefficient of variation of 10 per cent, and for the acrylic, a mean value of 1.30 with a coefficient of variation of 17 per cent. In view of this wide variation, at this time, little reliance could be placed on the interpretation of the effect of weathering on the indentation data presented in the logarithmic form.

#### Determination and culculation of tensile modulus

Since it was found that a variation in slope existed for different films, this necessarily casts doubt upon the extent to which the log load/log indentation approach can currently be theoretically applied, and some ancillary experimental work is desirable in order to test the data arising from the linear fit method. Initially, an obvious method is to use equation (1) to calculate Young's modulus from the indentation data, and to compare this value with that obtained directly from a tensile method. There are obvious difficulties in using such an approach.

- (a) Residual stress in the attached paint film could be relaxed by detaching it from its substrate for the tensile test.
- (b) The modulus measurements are time and strain rate dependent.
- (c) An assumption must be made concerning the value of v, Poisson's ratio.

In order to make a comparison between the two methods, films of unpigmented melamine epoxide were cast on to tin foil and the tin removed by amalgamation. Samples of the free stoved film were used for tensile testing. In an attempt to avoid (a), the indentation specimens were prepared by mounting the free film on to clean glass microscope slides using the Eastman Kodak cyanoacrylate adhesive, EK 910. This adhesive has the advantage that it can be thinly applied and cures at room temperature. The specimens were subsequently conditioned at 23°C and 65 per cent relative humidity. Using, as before, a spherical indentor radius 200 $\mu$ m and a time under load of 120 seconds, the intercept of the Ln load versus Ln indentation plot was found to be 1.025. Using this, and the value for the radius of the indentor  $r = 200 \times 10^{-4}$ m, gives

$$\frac{E}{1-v^2} = 1.4 \times 10^{10} \, \mathrm{dynes} \, \mathrm{cm}^{-2}$$

Since the value of v, Poisson's ratio, can range from 0.3 to 0.5, this indicates a value for *E*, derived from the indentation test, of

$$E = 1.1 \text{ to } 1.3 \times 10^{10} \text{ dyne cm}^{-2}$$
 (8)

The data from the tensile test is shown in Table 2. In all the tests a gauge of length 10cm was used with a sample of width 2.5cm. The testing was conducted using an Instron tensile testing machine with a constant crosshead speed of 0.5mm per minute at a temperature of  $23^{\circ}$ C and 65 per cent relative humidity.

The mean value obtained from Table 2 is 7.1  $\times$  10  $^9$  dynes cm  $^{-2},$  which differs from the indentation value by a factor of two.

Table 2 Tensile testing data on unpigmented melamine epoxide films (23°C, 65 per cent relative humidity)

Sample	Film thickness cms $\times$ 10 <sup>3</sup>	Load dynes $\times$ 10 <sup>-6</sup>	Extension cms	Tensile modulus dyne cms <sup>-2</sup> $\times$ 10 <sup>-9</sup>
A	6.1	0.701	0.0625	7.2
B	6.4	1.00	0.093	6.7
C	6.4	1.00	0.0995	6.3
D	6.1	1.40	0.129	7.0
Ē	6.1	1.40	0.145	6.2
F	6.1	1.60	0.116	8.9

It should be emphasised that this comparison is based on purely elastic considerations whereas it is indicated from the treatment by Lee<sup>11</sup>, equation (3), that the intercept value for some media should be better compared with the creep shear compliance. Further work is required to make such comparisons, for example, by using a pendulum oscillatory method after that of Ladizeski and Ward<sup>18</sup>.

#### Phenomenal aspects of micro-indentation

As indicated earlier, the alternative method of interpretation of micro-indentation data is to collect data on an empirical basis and attempt to interpret measured changes of paint films in the context of measured changes in mechanical properties. Two methods have been used: the infinitesimal hardness behaviour of paints<sup>8</sup>; and that of the three micrometre temperature characteristic<sup>19</sup>. Both methods exemplify the inherent difficulty of the treatment and the reliance that must be placed on empirically determined data.

#### Infinitesimal hardness behaviour (IHV)

The IHV method measures the load-indentation behaviour of a paint film at constant time under load and constant temperature. The IHV of the film is determined by plotting the ratio of the load to the indentation depth versus the load, and extrapolating the curve to zero load conditions. This extrapolated value is claimed to be directly related to the elastic modulus of the surface of the paint film. Most of the work reported<sup>8</sup> has been conducted using a Vickers pyramidal indentor.

Two sets of panels were coated by spray application with either a stoving alkyd or a solvent based acrylic at two film thicknesses. The results of the IHV method using the Wallace micro-indentor with the Vickers pyramid are shown in Figs. 3 and 4. These illustrate the difficulty of the extrapolation of empirically drawn curves from a limited number of data points. In addition, the value of the intercept obtained



Time under load 10 seconds .

30 seconds o 60 seconds +

Fig. 3. IHV curves of a pigmented stoving alkyd (23°C and 65% relative humidity Wallace micro-indentor)

Time under load 10 seconds ● 30 seconds o 60 seconds ◆



Fig. 4. IHV curves of a pigmented solvent acrylic (23°C and 65% relative humidity Wallace micro-indentor)

by the extrapolation is highly dependent on the least accurate of the data points which are derived using the conditions of lowest load.

Using a Vickers pyramidal indentor fitted to a recent simplified model of the ICI micro-indentor<sup>20</sup>, a comparison was attempted between the IHV and linear logarithmic method on pigmented paint films, Table 3. The IHV curves, Fig. 5, were determined using loads in excess of 1g in order to minimise the errors at lower loads, but this necessarily increases the length of the extrapolated curve.

Comparison of the results from the IHV method and the linear logarithmic method on pigmented paint films can be made from Table 3.

#### Table 3

Extrapolated values of pigmented films at 23°C, 65 per cent relative humidity. Film thickness 53µm

Paint	IHV value Vickers Pyramid	Linear logarithmic intercept 200µm radius indentor		
Solvent based air drying acrylic	0.15	0.4		
Medium oil stoving melamine alkyd	0.3	0.5		
Two part air drying polyamide epoxide	0.4	1.8		
Two part air drying aliphatic isocyanate cured polyester	0.6	2.3		

Time under load 120 seconds

Although the two methods have been related to modulus functions<sup>8, 9</sup> and rank the four paints in the same order, the

lack of an exact correlation between the two is not surprising since the IHV value has been related to the root of the elasticity modulus<sup>8</sup>, and the logarithmic intercept to a logarithmic modulus function<sup>9</sup>. In addition, there is also the possibility of different deformation mechanisms associated with the pyramidal and spherical indentors.

Although, in concept, the IHV method can be used with any shape of indentor, practical difficulties arise using, for example, the spherical indentor. The method stipulates that a "zero load" condition be used to locate the surface of the paint film with the tip of the indentor tool. The use of a pyramidal indentor ensures that even at a nominally zero load a much higher stress occurs at the point of the pyramid when it is touching the paint film than for a spherical indentor which would spread the contact load to a much greater extent. Thus, when the spherical indentor is used in this mode a far greater scatter of results is found.

#### Three micro-metre temperature characteristics

This approach characterises a paint film in terms of its indentation-temperature dependency<sup>19</sup> and although it might be expected that a paint film would show progressive hardening on ageing, as realised by a progressive increase in three micro-metre temperatures, this has not always proved to be the case. It has been found that an aliphatic *isocyanate* polyurethane or a polyvinyl acetate, for example, does not seem to show a significant variation in three micro-metre temperature with duration of exposure.

However, additional information can be gained from a study of the shape of the indentation-temperature curve of the particular films. In the case of a solvent based unpigmented acrylic medium, three micro-metre temperatures were recorded (Table 4) from the data plots (Fig. 6).

Exposure to BS 3900 Pt. F3 weeks	Three micro-metre temperature °C
0	34
1	45
2	47
4	54
8	62
14	66

Table 4

Three micro-metre temperatures of a solvent based acrylic on ageing

There is an increase in the three micro-metre temperature as the curves shift to higher temperature with duration of exposure, but in addition there is a quite noticeable change in the shape of the indentation-temperature curve. This latter effect is quite clear if the indentation-temperature curves are superimposed as shown in Fig. 7. There is a marked change in the temperature dependency of the film after ageing in excess of four weeks, which might not be detected by consideration of the three micro-metre temperatures alone.

> > EPOXIDE

1

LOAD

1-3

m4 / 6

LOAD

0.

0

0

In the case of the melamine/epoxide film, the pattern of change of three micro-metre temperature is less clear (Table 5).

7	-a.	Ы	2	- 5
1	u	v	e	-

Three micro-metre temperatures of an unpigmented melamine epoxide on ageing

Exposure to BS 3900 Pt. F3 weeks	Three micro-metre temperature $^{\circ}C$
0	59
Î.	56
4	70
8	66
12	62

The apparent random order of the three micro-metre temperatures did not seem to be due to experimental variation since replicate determinations on different specimens showed good repeatability, and it would appear therefore that some other phenomenon is occurring. Fig. 8 shows the data plots from which the results of Table 5 were taken.

ALKYD



Fig. 5. IHV curves of pigmented films, ignoring 0.5g data points (23°C and 65% relative humidity Simplified ICI micro-indentor)

3

2

9



Fig. 6. The temperature dependence of indentation depth of a solvent based acrylic film exposed to periods of artificial weathering to BS 3900 Pt. F3

(200µm radius sphere, 8g load, 120 seconds under load)



Fig. 7. Superposition of the data from Fig. 6 showing the proposed characteristic shape of the acrylic film affected by the later periods of exposure to artificial weathering



Fig. 8. The temperature dependence of indentation depth of an unpigmented stoving melamine-epoxide film exposed to periods of artificial weathering to BS 3900 Pt. F3 (200μm radius sphere, 8g load, 120 seconds under load)

Again, a marked change in the shape of indentationtemperature curve is apparent during exposure, except that the change is markedly different from that of the acrylic. It would seem that the epoxide system (which had been stoved at 176°C for 30 minutes and in addition been given a poststoving schedule of 40°C for three days and subsequently conditioned at 65  $\pm$  5 per cent relative humidity at a tem-



Fig. 9. Superposition of the indentation temperature data of a pigmented tall oil alkyd film exposed to BS 3900 Pt. F3

perature of 20  $\pm$  2°C) could be considered to be fully cured. It is therefore possible that this early change during exposure is due to the wet period rather than the elevated temperature of the BS 3900 Part F3 artificial weathering cycle.

In the light of the above, some previously recorded data were re-examined with respect to the super-position of indentation-temperature curves and in the case, for example, of a pigmented tall oil alkyd containing 4 per cent rosin, a similar effect to that noted for the acrylic medium was found (Fig. 9).

#### Discussion

The quantitative interpretation of micro-indentation data is obviously far from satisfactory and it would seem that the reasons for the phenomena observed have not yet been established. That there is a connection between microindentation data and other physical parameters of paint films is undisputed. Prosser<sup>21</sup> has reported a correlation between the indentation-temperature characteristics of a number of paints and the temperature at which those paints show a brittle fracture; Osterle<sup>22</sup> has related the IHV behaviour of paint films to wetting and ageing behaviour; and there is some unpublished evidence that links the temperature dependence of the ASTM<sup>26</sup> conical mandrel test with the indentation-temperature characteristics of paints.

However, it must be considered how this type of physical measurement of mechanical property of paint films can or is likely to be developed to the point of increasing our understanding of paint film behaviour. It is known from experimental evidence that polymers not only change due to an

ageing history but also have a memory of previous stress/ strain history, as exemplified by the work of Leaderman on the principle of Boltzmann's super-position of stress12. It is not unreasonable to suppose that a paint film subjected to a natural environment will suffer during its life a large number and variety of stresses due to, for example, temperature and humidity variations and mechanical strains. The ability of the film to cope with these strains without mechanical failure will depend on the mechanism of deformation and stress relaxation. Consideration of these mechanisms should provide a link to a likely method of answering the questions posed in the introduction to this paper. The paint technologist is largely interested in, and practical mechanical testing is largely orientated towards, determining the failure properties of a paint film but the interpretation of these failure properties should not be divorced from the consideration of the mechanisms of paint deformation, and it is towards the linking of the two methods of approach that mechanical testing of paints must evolve. An example of this is the recent work of Zorll<sup>23</sup> who has interpreted the interaction of binder and pigment in terms of the fracture envelope of detached paint films. Here, there is a direct link between an ultimate property (tear) and a viscoelastic-temperature relationship. In considering failure properties, the measurement of crack initiation and propagation is another field of direct interest and, in this context as in others, consideration should be given to the stress history of the film.

Little is known as yet about the effects of mechanical inhomogeneity in the paint film (although two effects have been reported<sup>9, 25</sup>) despite the fact that these effects must profoundly complicate the practical interpretation of data. The obvious difficulties of such investigations are that any method of exploring the nature and extent of inhomogeneities must themselves first be assessed with regard to variation in the test method before any experimental variation can be shown to be due to a significant variation of a mechanical property. It would appear, therefore, that in this field of study, analysis of variance would appear to be essential, and that variability in test data may be of more interest than repeatability.

Since the major discontinuity for a paint film occurs at the film-substrate interface, it is possible that methods like the micro-indentation technique could be modified so as to examine both the mechanical property of the paint film and also that of the paint-substrate interface.

#### Conclusions

This review of the field of micro-indentation testing of paint films has described and examined the different methods of data presentation proposed for the technique. All are shown to be deficient in some respect. The 200µm spherical indentor depends on the film thickness; for the logarithmic presentation, the slope varies; there is practical difficulty in extra-

#### **Discussion at Eastbourne Conference**

DR U. ZÖRLL said that when considering the relationship on which the indentation methods—and similar methods for measuring mechanical film properties—were based, one might be led to the following qualitative observation. As the external loading became more localised and, therefore, the resulting field of stress less homogeneous, the more the relationship between the quantity actually measured and Young's modulus deviated from linearity. Thus, if the bulk properties of the film were to be measured, then polating the IHV curves; and its empirical nature is a disadvantage of the indentation-temperature method.

Nevertheless, some form of data presentation is obviously essential if some meaning is to be obtained from this and allied techniques, which explore the relationship of mechanical properties in terms of experimental parameters. Whether this will ever be satisfactorily achieved is problematical, but the prize is a basic understanding of paint performance and the interpretation of the effects of different weathering situations.

#### Acknowledgments

The work of Mr A. Chalkley in obtaining much of the experimental data is gratefully acknowledged. Thanks are also due to Mr Bray, Research Equipment (London) Ltd., for the loan of the modified ICI micro-indentor.

[Received 24 January 1973

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a system of measurement based on a relation characterised by the least deviation from linearity might give the most meaningful results. Dr Zörll suggested that such a principle could serve as a guide for the paint specialist faced with the task of selecting one of various devices for the measurement of mechanical film properties. Presumably, this would apply to instruments developed in the future as well as to those used at present.

MR R. L. J. MORRIS liked this idea but there was one basic problem which had to be considered: mechanical

inhomogeneity existed in paint media. The problem had been with the paint trade for years, and Dr S. H. Bell had written a paper on the subject some 20 years ago.

It was correct that mechanical inhomogeneity did affect the test data, but Mr Morris felt that it was fundamentally wrong to ignore totally this effect, because not enough was known about its significance with respect to the chemical and other properties of paints. From the point of view of the paint technologist, who would require a reproducible, repeatable and discriminatory method of test, the approach proposed by Dr Zörll was correct. For development of the understanding of paint films, however, it was necessary to use methods which were going to suffer from the fact that work would be directed specifically at mechanical inhomogeneity. Whether this would ultimately prove significant with respect to the properties of the paint could not be determined at present; it was necessary firstly to quantify the effects. Briefly, there were two aspects to this question; depending on which was being looked at, one could either agree or disagree with Dr Zörll's proposal.

DR M. ELLINGER asked whether the tests had involved multicoat systems. She had found that whilst on single coats, the IHB values reflected the material's characteristics (for example weathering properties), in the case of a two-coat stoving system, using various primers but an identical topcoat, the IHB was always the same; that is, a characteristic only of the topcoat.

MR MORRIS replied that all the work which he had spoken of had been on paint films of a single medium. He had been looking at the mechanical properties of particular paint media, and the complication of additional variables had, therefore, been avoided; although he would acknowledge that this was, at present, very far removed from technological practice. He took as a particular example, a red lead primer. Indentation depth was not going to help since most of the deformation would occur within the primer; one had to consider the compliance of each particular phase with the system. The Directorate was conducting some work on the effect of film thickness on indentation depth. Until it was known how far through a paint film the influence of indentation depth extended, it would not be possible to understand the mechanical properties of films containing dissimilar coats.

DR J. E. O. MAYNE said that work in Cambridge indicated that clear varnish films had a very heterogenous structure, which could be detected by hardness measurements. He asked whether the author had found a similar effect in his measurements.

MR MORRIS remarked that he had had a paper published by FATIPEC last year which referred to the results quoted in Dr Mayne's paper. He understood that Dr Mayne had examined indentation depth for films with linseed tung phenolic media\* and had correlated specific small areas of micro-indentation inhomogeneity with D and I areas of ionic conductivity.

He too had observed the discontinuities in mechanical property of paint media with regard to micro-indentation, and had modified the ICI micro-indentor to move the specimen linearly in a known manner beneath the indentor. It was then possible to examine the indentation characteristics of specific areas of the film. He was interested in whether the observed variation in indentation depth was due to the test method or a parameter of the film, so the distribution of hard and soft areas was important. Inhomogeneity was found in a two-coat unpigmented stoving melamine/epoxide film. Soft areas were of the order of Imm across which was in accordance with Dr Mayne's findings.

Mr Morris was examining this two-coat material, when he accidentally scratched the surface with his finger nail and removed the top coat. This lead him to suspect that he was observing local intercoat adhesion failure. In order to examine the effect further, he had used a chlorinated-rubber film which, being a solvent-based material, eliminated the possibility of intercoat adhesion failure. Variations in mechanical properties were again found, but these were almost certainly due to the effect of localised solvent retention. It was not possible to say whether the effect which Dr Mayne had observed was due to different degrees of cross-linking, but Mr Morris believed he had found that intercoat adhesion failure and localised solvent retention might give rise to the same indentation phenomena and that in the case of the chlorinated rubber medium, cross-linking could not be a significant variable.

DR MAYNE thought this was extremely interesting. He could say that the film he had used had a structure, and that this had been interpreted in terms of cross-linking, but if the retention of solvent were localised, it would be plasticised, and this would agree perfectly with the concept. His work had not, in fact, included an examination of solvent retention.

\*The films were made from pentacrythritol linseed alkyd, epoxy polyamide and tung oil phenolformaldehyde media—R. L. J. Morris.

## Finish first—fabricate later. The continuing story of coil-coating\*

#### By D. S. Newton

Head of Organic Coatings Development, British Steel Corporation, Strip Mills Division, Research Centre, Port Talbot, Glamorganshire

#### Summary

A survey of the present status of coil coating is given, indicating differences between the American and European scenes. Recent developments in coating systems, such as oil-free polyesters, fluorocarbons and polyamides are described in some detail.

The progress in pollution reduction with regard to coil coating

#### Keywords

Process and methods primarily associated with application of coatings and allied products coil-coating developments is outlined with reference to pretreatments, high solids dispersions, water-based coatings and plastic films. Finally, looking ahead to the future, the potential of solvent-free coating systems applied by an extrusion technique gives an indication of the direction in which the "finish first-fabricate later" industry can move during the last few decades of the twentieth century.

manufacture or synthesis pollution control

auf die Vorbehandlungsmethoden,

Jahrhunderts bewegen kann.

#### D'abord la finition-la fabrication ensuite. L'histoire en cours de couchage en continu

Résumé

On donne une vue générale de l'état actuel dans le domaine de couchage en continu, et l'on indique les différences qui existent à cet égard entre les Etats-Unis et l'Europe. On décrit en quelque détail certains développements récents en des systèmes de revêtements tels que polyesters exempt d'huile, résines fluorocarbones et polyimides.

On trace les grandes lignes du progrès vers la diminution de la pollution à l'égard des développements dans le domaine de

#### Erst Lackieren-Dann Weiterverarbeiten. Fortgang der Geschichte der Bandlackierung

#### Zusammenfassung

Ein Überblick über den gegenwärtigen Stand der Bandlackierung, in welchem auf die Unterschiede zwischen der amerikanischen und europäischen Szene hingewiesen wird. Neuere Entwicklungen von Anstrichsystemen wie z.B. auf Basis von Polyestern, Fluorkohlenwasserstoffen und Polyimiden werden eingehend besprochen.

Dir Fortschritte auf dem Gebiete der Pollutionsreduzierung im Verlauf der Entwicklung der Bandlackierung werden im Hinblick

#### Сперва отделка —а затем фабрикация. Продолжение вопроса спиральных покрытий

#### Резюме

Дается обзор современного развития спиральных покрытий, ссылаясь на различие между американским и свропейским положениями. Последние достижения в системах покрытий, как например полиэфиры свободные от масла, флуороуглероды и полиамиды, подробно описываются.

Описывается прогресс в понижении загрязнения по отношению к развитию спиральных покрытий, ссылаясь на предварите-

#### Introduction

Perhaps one of the main reasons why "coil-coating" or "prepainting" is less familiar in Europe than many other modern developments in paint technology is due to the way in which the industry has developed. There is a marked difference when compared with the American development, prepainting being first established as an industrial process in

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couchage en continu au point de vue des prétraitements, des dispersions à forte teneur en matières solide, des revêtements à base d'eau et des films en matières plastiques. Enfin, vensant à l'avenir, les possibilités des systèmes de revêtements exempts de solvant et capables à être appliqués par une technique d'extrusion donnent une indication de la direction vers laquelle l'industrie, baptisée "D'abord la finition—la fabrication ensuite" par l'auteur, peut s'incliner pendant les dernières décennies du vingtième siècle.

Festgehalt, wassergetragenen Beschichtungen sowie Kunstoffilme skizziert. In die Zukunft schauend werden schliesslich die Möglich-

keiten lösungsmittelfreier, durch Spritzgusstechnik aufgetragener Beschichtungssysteme als Wegzeichen für die Richtung besprochen, in welcher sich die "Erst Lackieren—Dann Weiterverarbeiten" Industrie während der allerletzten Dekaden des zwanzigsten

Dispersionen mit hohem

льную обработку, высокую дисперсность твердых частиц, покрытия основанные на воде и пластические пленки.

В заключение обращая взор на будущее, потенциал систем покрытий без растворителя, прилагаемых прессовкой, указывает направление в котором промышленность может продвинуться с лозунгом «сперва отделка—а затем фабрикация», в течение последних десятилетий двадцатого века.

the USA some thirty years ago. Beginning in the hands of small private companies, the first operations were slow and consisted of single paint coating applications to narrow width aluminium strip. The end uses of the products made on these lines were in venetian blind slats, and later simulated shiplap board for residential siding. As new markets arrived for coil-coating, coil coaters were able to incorporate these in their operations, so that the concept was born of the

<sup>\*</sup> Presented at the 1973 Eastbourne Conference.

custom or "tollcoater" applying organic finishes to meet the requirement of the fabricator and end-user, and putting them on the substrate metal of his choice.

The National Coil Coaters' Association (NCCA) was formed some twelve years ago with the object of promoting new markets for coil-coating and standardising the quality of the product. At this stage, the prime metal producers began to appreciate the advantages of the process, to see the possibility of new outlets for their products, and a means for reducing the inroads being made, by the plastics industry for example, into their traditional markets. From being rather passive onlookers as suppliers to the coil-coating industry, the steel and aluminium strip producers began to take an active role in the NCCA, to participate jointly with individual custom coaters, and more recently, in putting down coil-coating lines of their own with a custom coater in the background as a consultant. The logical progression of a growth pattern set up years ago has enabled the supplying industries (engineering, pretreatments, paints, etc.) to adjust to it, and sometimes, particularly with the larger paint companies, to direct its course. The existence of the NCCA. desirous of promoting the industry and its image throughout the USA and Canada, and of setting standards, has smoothed the path. Fabricating industries, geared to handling coilcoated material, have grown up around it, familiar with the careful handling techniques required to utilize the product to greatest advantage, both in the fabrication and in the subsequent assembly either in the factory or on site.

The European story has been quite different. Ten years ago, there was a mere handful of lines, each doing different things, making different products, and doing them in isolation. Some coil-coated products were made by prime producers of metal strip, others by private companies. There was no custom-coating industry at all. Suddenly the picture has entirely changed. There are now more than 30 coating lines in Europe and another 15 on the way, mostly wide high-speed operations owned by the prime metal producers. This situation is analogous to the problem of the "late developer;" because these developments have taken place with such rapidity, a rationale has not grown up with it. The European Coil Coaters' Association is a comparatively difficulties attendant on all multi-national and multi-lingual organisations. The paint industry has been undoubtedly surprised by this sudden burgeon, and has not been helped by the predilection in Europe for coating systems which have not found favour across the Atlantic.

#### Properties of existing coil-coatings

Several organic coating systems have become established in coil-coating over the years, and one of the misleading consequences of this is the association of particular types of polymer with particular kinds of application. Since differences in formulation can make the difference between success and failure, this fact has not always helped the process in its advancement. For example, it is not easy to formulate a 250 micron thick pvc coating that will last in a variety of exterior climatic conditions for 15 years before it requires maintenance, and it is quite easy to arrange for a pvc coating of this thickness to erode away in a little over twelve months. Thus "pvc plastisols" may be dubbed excellent, bad or indifferent for exterior architectural cladding in various countries, depending on the major source of supply and the principal environmental conditions encountered in the market supplied; for example, agricultural or industrial buildings, prestige office blocks, schools, hospitals, or power stations

However, with this reservation, there are certain generalities that can be accepted in the classification and grading of organic coatings, and Tables 1 and 2 show those systems which have become the backbone of the industry, and their present applications. The results quoted do not, of course, form a specification but are merely typical. Alkyds are the least expensive, but are limited by their lack of heat resistance. lack of resistance to chemical attack, and except in the case of a few modified formulations, poor weatherability. Thermosetting acrylics, based on acrylamide copolymers have good resistance to heat, and resist breakdown in a corrosive environment. Their shortcoming lies in the poor fabricating performance of the highly cross-linked structure of more resistant formulations, so that the calibre of coil-coatings can never duplicate that of similar post-painted finishes. Epoxy coatings are mostly suitable as thin lacquers that

Ta	ble I
"Typical performance"	chart for coil-coating systems

			the second s			
Property	Alkyd	Appliance acrylic	Architectural acrylic	Solution vinyl	Pvc organosol	Pvc plastisol or film
Dry film thickness ( $\mu$ m) Gloss (60° reflectance) (%)	12—25 15—80	20—25 50—90	20—25 10—80	12—25 10—80	30—75 10—50	150-300 dependent on
Pencil hardness	H—2H	H4H	F—H	Н—3Н	FHB	indeterminable
(inches $\times 10^{-5}$ )	17-20	12-15	15-20	15-18	25-30	200_300
Scratch resistance (BS 3900 F2) (gm)	2 000	3 500	3 000	3 000	3 500	> 2 500
Reverse impact (BS 3900 E2) washers	2,000	5,500	5,000	5,000	- 5,500	23,500
nasced	2 (a)	1 (a)	1 (6)	2 (0)	E (1-)	5 (1-)
Mandral hand (DC 2000 E1) (mandral	5 (a)	1 (a)	1(0)	5 (a)	5 (0)	5 (D)
diameter in inches)	1.7.5	1.45	1.0.5	1.7.5	0	
diameter in inches)	作 (a)	<del>§</del> (a)	₿ (b)	륨 (a)	OT bend (b)	OT bend (b)
Drawing properties (with appropriate						
grade substrate)	good (a)	good (a)	good (b)	very good (a)	excellent (b)	excellent (b)
Salt spray (ASTM-B117-57T) (hr)	100 (a)	500 (a)	1,500 (b)	500 (a)	1.500 (b)	1.500 (b)
Heat resistance (maximum continuous)		1			.,	1,000 (0/
working temperature) (°C)	70	120	70	70	60	50
Stain resistance (household reagents	10	120	10	10	00	50
and grease)	good	excellent	very good	road	warw good	and a second
Nigoting stein registeres	good	excellent	very good	good	very good	good
Weathershiller	lair	excellent	very good	Tair	Tair	poor
weatherability	Tair		tair/good	tair	fair	excellent

(a) Results based on 20 BG CR4 steel substrate.

(b) Results based on 20 BG hot-dipped galvanised steel substrate.

	Applic	ation				Alkyd	Acrylic	Solution vinyl	Pvc organosol	Pvc plastisol or film
Automotive								_		
Underbonnet part	.s	• •	• •	• •				•	•	
Trim	• • •			• •						
Fascia panels		•••	•••	• •	••	•			-	-
Appliance										
Wraps							•			•
Heater cabinets							•			•
Heat resistant par	ts						•		-	
Deep drawn parts								1	•	•
TV cabinets						•	-			•
Light fittings						•	•		•	
Building										
Ceiling tiles								•	•	•
Suspended ceiling	<b>T</b> -bars							•	•	
External cladding										
Curtain walling										•
Infill panels							•			
Skirting boards						•	•		•	•
Partitioning						•	•			•
Cable trunking						•				
Roofing									1	•
Roof decking						•			•	•
Caravans			• •				•			•
Shelving						•			•	•
Steel furniture						•	ļ			•
						-				
Cold storage rooms										
Inside skin							•			•
Outside cladding										•
Garage doors										
Primed					•••	•				
Finished	• • •	• •	• •	• •	••				•	•
Venetian blind boxes		••					•		•	
Skirting board heate	rs						•		•	
Deep drawn parts (g	eneral)						1		•	•

 Table 2

 Applications for typical coil-coatings

need to have very good fabricating properties and chemical resistance. The pvc organosol type system has all the flexibility to be expected from a purely thermoplastic finish, but lacks chemical resistance due to the plasticiser present, and the combination of the latter with high pigmentation makes good weathering formulations difficult to achieve. Pvc plastisols and films have similar properties, but are applied as much thicker coatings which may be embossed or otherwise decoratively finished. A lower pigmentation is needed to hide the substrate metal because the coating is thicker. and, with careful plasticisation and stabilisation, good weather resistance may be obtained. The emboss pattern prevents any scratch marks incurred during site handling from becoming apparent. Dirt pick-up and retention may be reduced by the addition of a mar-resistant lacquer over the top of the pvc. In the case of internal cladding, it may be appropriate to use an acrylic or polyester lacquer to overcome nicotine staining, and various other discolouring influences. For a decorative finish, pvc calendered films are available in a far greater variety of textures and multicolour prints than would be economical with pvc liquid coatings produced on a strip line. The solution vinyl coating is useful in those applications where a mar-resistant surface is required in addition to sufficient flexibility for the more severe deep-drawing applications. The vinyl coating may be applied as a "one-coat" finish of 25 microns thickness

requiring no primer at the interface. The use of this coating, however, is becoming increasingly restricted due to the high cost and low solids content of the paints. Inordinately large quantities of solven have to be removed, which is neither-"cost-effective" nor totherwise acceptable in this pollution conscious age.

Developments at present taking place in coil-coating revolve around three main considerations: reduction in pollution; use of new polymers and of combinations of these with existing systems; and use of new application and curing techniques. Ideally, these developments could all be combined together but, in practice, they have to be considered separately because of the complexities involved in changing anything in such a capital-intensive process as coil-coating.

#### New developments in polymers

#### **Oil-free saturated polyesters**

Oil-free saturated polyesters represent a new type of surface-coating resin designed to satisfy the more stringent requirements of modern paint finishes. This somewhat illdefined group of coatings has emerged over the past five years. The resins may be considered to be special kinds of alkyds, composed of a wide range of polyfunctional acids and alcohols, but without the partially hydrolysed natural oils which the paint industry has used as components of alkyds for many years. Several of the new polyesters are believed to be condensates of monofunctional branched acids denoted by  $R_1R_2R_3 - C - COOH$ , where the *R*s are aliphatic side-chains giving the molecule between nine and eleven carbon atoms, triols and dibasic aromatic acids. In practice, the branched acids (which reduce esterification rates and are virtually insoluble in the resin materials) are present in the form of glycidyl ester which allows rapid esterification via the terminal epoxy group. It is claimed that resins containing these branched chain acids are characterised by good colour and gloss, hardness and good flow.

Many other polyesters are thought to have been developed by using trimellitic anhydride and isophthalic acid as principal esterifying reactants. Such use of an acid for branching the polymer has led to resins of high aromatic content, low excess hydroxyl, and high solubility. By using such combinations, commercial polyesters are now available with excellent stain resistance and very good flexibility.

Generally, the oil-free polyesters have to be cross-linked with an amino-resin, or a resin precursor, such as hexamethoxymethylmelamine, when satisfactory hardness coupled with flexibility and good colour-retentive properties may be achieved. This type of system can be formulated to produce just those requirements needed for paint coatings for domestic and industrial appliances (for example, washing machines and refrigerator wraps and liners) but with the added bonus of being able to withstand intricate fabricating operations without cracking under strain and elongation.

An extension of their properties can be made by incorporating siloxane resins. These have been developed in low molecular weight form to have both maximum solubility and compatibility with organic resins, and unlike the simple silicone blends, it is preferable for condensation to take place between the pre-made polyester and the siloxane resin. By such modification, copolymers are obtained with considerably improved resistance to heat, oxidation, and weathering.

Silicone-modified polymers have achieved some preeminence in coil-coating in North America because of their good resistance to weathering, and much of the prefinished hot-dipped galvanised steel used for fabricating industrial buildings is coated with paint containing thirty to fifty per cent copolymerised siloxane resin. The degree of resistance to oxidative breakdown is proportional to the silicone content, and it is claimed that at high silicone levels, oilfree polyesters have attained eight years outdoor exposure with only slight superficial chalking and fading. A lack of plasticity is an unfortunate characteristic associated with high silicone content and is a limiting factor, generally, in the fabrication of such polymers.

#### The fluorocarbons

Fluorocarbons, or fluoroplastics as they are termed in American nomenclature, comprise a group of polymers of particular interest to coil-coating. The structure of these consists of long vinyl chains, sometimes branched, in which part or all of the hydrogen is replaced by fluorine. As a class, they are characterised by their inertness (which makes for very good resistance to all kinds of chemical attack), good dimensional stability, good temperature resistance without degradation, low coefficient of friction, and good electrical properties. The polymers include materials such as polyvinyl fluoride (pvf), polyvinylidene fluoride ( $pvf_2$ ), and polytetrafluorethylene (ptfe).

On comparing the properties of these polymers, it is found that the thermal stability increases in order of increasing fluorine content (that is,  $pvf < pvf_2 < ptfe)$ , with the chemical inertness following the same order, so that solubility decreases accordingly. Static coefficient of friction and critical surface tension increase with increasing fluorine content, and ptfe is the "non-stick" material par excellence. From the economic aspect, increased fluorine content leads to higher densities with resultant lower coverage per pound of polymer, and at the same time the raw material prices increase with increasing fluorine substitution. The net result is a dramatic rise in the cost of the coating as the number of fluorine atoms increases in the fluoroplastic.

The fluorocarbons have now been tailored to meet the requirements of reverse-roll application and laminating processes for metal strip. Because of the low solubility and the melt flow characteristics of these polymers, it has been necessary to utilize latent solvents in order to achieve suitable dispersions for roll-coating. In the cases of pvf and pvf<sub>2</sub> there are no known room temperature solvents, but certain organic solvents become active at elevated temperatures and exert a solvating effect on the polymers. These "latent" solvents usually have boiling points above 150°C. The basic principle used in processing pvf and pvf<sub>2</sub> is the disruption of the polymer order and subsequent lowering of the minimum film-forming temperature to below the melting point of the polymer. This disruption is effected through latent solvents and processing at an elevated temperature, so that solvation causes the fluorocarbon particles to coalesce into a single phase in the form of a continuous film at a temperature well below their degradation point. Some typical latent solvents with their boiling points are: acetophenone 202°C; n,ndimethylformamide 215°C; isophorone 215°C; dimethyl phthalate 282°C; diethyl phthalate 298°C. In practice, formulations usually contain a mixture of such latent solvents and lower boiling point ketones, used as diluents and given off during the curing of the coat. They have a dispersing effect on the powder at room temperature and a wetting effect on the surface to be coated, thus helping to control the viscosity of the dispersion and allowing proper flow and levelling of an applied coating on a substrate.

The fusion of ptfe takes place at such a high temperature (327°C) that the concept of latent solvents cannot be applied. and ptfe coatings are therefore prepared from true dispersions. These may be obtained in water or in organic solvents. The use of the latter allows scope for modification with solution resins, such as epoxides or polyimides, whilst water-borne ptfe dispersions have a considerable cost advantage. In all cases the requirement of ptfe coatings on metal is the preservation of a low coefficient of friction at the surface, resulting in "non-stick" properties, combined with reliable adhesion at the interface of metal and organic coating. The latter effect can be promoted by oxidative modifications, usually by the incorporation of other ingredients which decompose at the fusion temperature of the ptfe with the liberation of acidic gases. This promotes adhesion by chemically etching the metal surface and degrading slightly the ptfe at the interface. Another method of promoting adhesion is to incorporate with the ptfe a solution containing resins, such as epoxides or polyamides. It is believed that in this type of system a fluorocarbon-rich stratum is formed at the free surface, which provides the easy-clean, low friction properties of the coating, and that a resin-rich stratum forms at the coating/metal interface. Adhesion is thus obtained without a primer.

The applications for fluorocarbons in coil-coating fall into two areas. The first is for very high performance weather and stain-resistant exterior architectural coatings (pvf and  $pvf_2$ ) with a probable life of 20 years, at least, to first maintenance. The second is for non-stick, easy-clean or low friction uses (ptfe and ptfce) as shown in Table 3. heterocyclic rings should be the main recurring units in the structure for promoting heat resistance.

In the case of polyimides these precepts are followed, the structures developing usually in the course of cure. Thermosetting of a soluble resin takes place not by cross-linking

 Table 3

 Examples of "low friction" fluorocarbon dispersions

Dispersion			Type of system	Recommended applications			
Aqueous Fluon MM1			one-coat	Bakeware, steel razor blades, hypodermic needles			
Fluon GP1			one-coat	Mainly aluminium cookware			
Fluon SC3			one-coat	Industrial applications, e.g. chutes, hoppers, domestic irons, spiral feeds			
Fluon SC1			primer				
Fluon Clear Fl			top-coat over Fluon SCI	Industrial applications and cookware, e.g. top-of-the-stove			
Fluon Black Fl	lack FI		top-coat over Fluon SC1 $\int$	ware, ovenware, bakeware			
Organic Teflon 955-100	••		one-coat	Domestic bakeware and cookware			
Teflon 959-203			one-coat	Domestic and industrial bakeware and cookware			
Teflon 954-101			one-coat	Aircraft tooling, kitchen tools, snow shovels			

"Fluon" is a registered trade-mark of Imperial Chemical Industries Ltd. "Teflon" is a registered trade-mark of the Du Pont Corporation.

#### Polyimides

Polyimides are in a category of thermally stable organic polymers, which have been produced, by imaginative synthesis and a greater understanding of structure-property relationships, to meet the demands of new technologies, particularly in relation to the space programmes. From among such polymers as polyphenylenes, polysulphones, polybenzoxazoles, and polythiodiazoles, the polyimides now seem to occupy the favoured position of being commercially the most important of the thermally stable organic polymers in metal-coating applications.

In the synthesis of polymers such as these, an optimum combination of properties is obtained by exploiting three main principles. In the quest for high temperature resistance, the formation of intrinsically rigid polymer chains ("ladder" or "semi-ladder" structures) proves extremely effective. Maximum thermal stability is usually achieved when unreactive rings are incorporated in the chain. Aromatic and to form a network during cure, but by joining up links along the polymer chain to produce the twice-bonded ladder-like formation which gives such good heat resistance and insolubility. A typical example of polyimide formation would be pyromellitic anhydride with 4,4—diaminodiphenyl ether giving a soluble polyamic acid which condenses to an insoluble polyimide. Another possible route to the insoluble polyimide is where the intermediate product has terminal ethylenic unsaturation and undergoes addition polymerisation when heated to a high enough temperature.

The commercially available polymers combine thermal stability (permitting prolonged use at  $250^{\circ}$ —300°C), chemical inertness (being unaffected by organic solvents or strong acids), hardness and abrasion resistance approaching that of vitreous enamels, but with the flexibility of a pvc coating. Their choice as a coil-coating finish for many of the applications in the lower temperature range, where vitreous finishes are mandatory at present, seems marked for the future. An immediate outlet for this type of polymer is as the reverse

Type of coating						Heat stability	Impact strength and flexibility	Detergent resistance	Hardness and wear resistance
Acrylic						110°C—125°C	fair	good	good
Polyester	••					120°C145°C	good	good	good
Epoxy*	• •					160°C—180°C	good	good	good
Anodised	••		•••			215°C-230°C	very good	poor	fair
Silicone	• •			•••		230°C—260°C	fair	good	fair
Polyimide*	••	••	••	••1	•• 1	285°C—300°C	excellent	very good	very good

Table 4
Comparative properties of typical finishes for metal cookware and appliances

\* Not available at present in white or pastel shades because of the colour of the resin.

#### CIBA-GEIGY Pigments for Automotive finishes

Irgazin Yellow 2GLT Irgazin Yellow 3RLTN Irgazin Yellow 3RLTN Irgazin Yellow 2RLT \*Cromophtal Vellow A2R \*Irgalite Orange GR Cromophtal Brown 5R Cromophtal Bordeaux RS Irgazin Violet 6RLT Cromophtal Violet 6RLT Cromophtal Violet B Irgazin Blue 3GT Cromophtal Blue A3R and a comprehensive range of phthalocyanine blues and greens



#### **CIBA-GEIGY**

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side coating for domestic and industrial bakeware. This would be complementary as a reverse coat where "nonstick" fluorocarbons are being applied to the top side of strip for such applications. For other uses where heat resistance is of paramount importance (for example, ovens, electric, gas and oil heaters), this coating can be considered competitive with anodised or porcelain finishes. Table 4 illustrates comparative properties of various finishes for metal cookware and appliances.

#### **Pollution reduction**

Immediate improvements are being effected in the cleaning and pretreatment processes involved in coil-coating. Instead of the necessity for effluent treatment prior to disposal, a regeneration system is available for the cleaner solutions, which effects oil separation and removal. In the case of the spray phosphate treatments, which have long been the standard methods for producing a corrosion-resistant and adhesion-promoting metal surface in coil-coating as in other paint coating processes, these are likely to be supplanted by the metred application of chromate-based treatments which serve the same purpose. Since most of this is applied to the surface there is little or no waste problem. Additional benefits accruing are: the reduction in circulating and heating costs; simplification in the mechanics of the process as coating deposition no longer varies with the speed of the line and the new treatments can be applied regardless to mild steel, zinc-coated steel, and aluminium; and finally the possibility of dispensing with the expensive brushing and linishing section of pretreatment, without which phosphating processes are irregular and unreliable.

With regard to the organic coatings themselves, reduction in pollution may be brought about by increasing the solids content or transferring to water-based systems. The former involves the use of dispersions, as the viscosities of high solids solutions are too great to permit roller-coating application. The "latent" solvent approach is again employed as with fluorocarbons, and the systems which exemplify this principle are pvc organosols and plastisols. Two added bonuses for the dispersion approach are the possibility of thicker coatings and the use of high molecular weight polymers. A good example of the latter is the high molecular weight acrylic type polymer which is suitable in emulsion form. Few other developments for coil application have such a useful performance in exterior durability with maximum flexibility at low applied cost as these particular emulsion polymers. Much of the success of these materials is due to the fact that they are formulated from a combination of thermosetting and thermoplastic acrylics, and this allows adequate scope for incorporating sufficient flexibility, coupled with hardness, into the pigmented film by a moderate degree of cross-linking. Since these systems are water-based a significant departure from the solution coatings, which at present dominate architectural markets for coil-coating, has taken place.

The molecular weight of some of these polymers exceeds 1,000,000 and thus approaches those of equivalent laminated films. If oxidative degradation occurs from the effect of exposure to UV radiation, the degradation products are likely statistically to be of relatively high molecular weight, enabling the film to retain coherence and many of its good physical characteristics. In practice, it has been found that exposure results for this type of coating are closely related to the average molecular weight of the film. It has been reported that it is possible for seven years' exposure results to show no chalking greater than ASTMS standard eight

from films put out at 45° facing south, at the usual Florida testing stations. Another important facet, for keeping film durability to a maximum, is the careful choice of pigment, and attention to pigment volume concentration. In developing acrylic emulsions for coil-coating, it has been found that best results are obtained by maintaining titanium dioxide levels, for example, at the minimum needed to give opacity at the applied film thickness. It then becomes unnecessary to incorporate UV light stabilizers for protection against sunlight during long-term exterior exposure. Such absorbers can have the undesired effects of increasing dirt retention and decreasing colour stability.

The reverse-roll application of emulsions presents problems in any case, and the application conditions for water-based emulsions is particularly critical. The high surface tension of water makes wetting of the metal surface difficult, and the viscosity characteristics of emulsions are pseudoplastic. This means that at low shear, the coating will not flow out, so that a ribbed pattern tends to be imparted to the coating after reverse transfer on to the metal strip. Under conditions of high shear, as in the metering gap between pick-up and applicator rolls, the viscosity is too low to keep the wet coating on the rolls. The result is a tendency for the coating to fall back or be thrown off the roll with resultant "starvation" of the nip. However, the incorporation of suitable additives improves the wetting characteristics and increases the viscosity at high rates of shear without adversely affecting the ability of the coating to flow out and produce an even smooth film under conditions of low shear. It has been found that small additions of a non-ionic surfactant in combination with a wide range of thickening agents, and in the presence of a secondary or tertiary amine or aminoalcohol, are particularly effective in this way, enabling a reasonable measure of linearity to be maintained for the shear rate/shear stress relationship under a wide range of conditions.

No discussion of reduction in pollution with existing technology would be complete without some reference to preformed films. For a long time, film lamination in collcoating has been at something of a discount. The reasons for this have been the high costs of films, poor colour control, limited colour range, and a limited range of laminating adhesives, all of which have required constant careful control to ensure maximum adhesion at the moment of lamination. Now that the cost of proprietory films for lamination has been largely reduced, the colour range of certain high-grade weather-resistant films has been spectacularly increased, and adhesives for lamination have become available which are much less temperature-sensitive for optimum curing conditions.

In the case of pvc films produced by the callendering process, there is considerable freedom of formulation, but this has not been a merit without a suitably rapid test method for evaluating performance in the field. The use of an accelerated weathering test method (which employs an unscreened carbon arc and a thermal shock by spraying refrigerated water on to the reverse side of panels when the arc is off) has proved to be a satisfactory and (to date) reliable means of rating pvc coatings in accordance with their actual performance in sunny environments, unlike the BS 3900 weatherometer test which has proved useless in this respect. Formulations can thus be quickly screened, and modifications made on other than an ad hoc basis. The importance of antioxidants and UV absorbers is being recognised, even for highly pigmented, opaque, thick pvc coatings, to delay the onset of the effect which has now come to be known as "microchecking." This phenomenon produces leprous,

entirely unacceptable appearance to pvc coatings exposed to ultra-violet radiation for a prolonged period of time, and would seem to be due to a catalytic decomposition of pvc at various points on the surface of a coating associated with loss of plasticiser from these areas. The "dew cycle" weatherometer test faithfully reproduces this type of failure. From the foregoing, it might be expected that, far from disappearing from the coil-coating scene, the use of films may well increase, and the absence of solvents, except in the thin layer of adhesive, should encourage this change.

#### New application and curing techniques in coil-coating

Crystal-gazing has never been a particularly rewarding occupation, and there can be few enterprises for which predictions are so likely to go awry as coil-coating because of the very high capital cost of the integrated equipment and the interdependence of raw material supplier, coating producer, (paint company), coil-coater, and ultimate fabricator and customer. The coil-coating industry prides itself on the degree of control exercised in the process in matters like corrosion resistance, coating performance and coating finish. and any "advance" which diminishes these is unlikely to become anything other than an interesting concept. Several ideas put forward during the last five years have been technically ingenious rather than realistic, and it would be discursive to refer to these at length. Nothing can be accomplished in this field without extensive (and expensive) pilot plant equipment, and the problems of scaling up in mechanical and electrical engineering often seem to be entirely unappreciated by those wholly absorbed in the laboratory examination of some intriguing new technique.

Coil-coating has so far progressed by utilising techniques already in existence and proven in other fields. The preparation, printing and coating of paper and fabrics is an industry to which coil-coating owes much, and it is not without significance that equipment suppliers still refer to the metal substrate in coil-coating as the "web." With these thoughts in mind, it will not be surprising that the author's attention is directed towards the process of extrusion as a coating technique of considerable merit in furthering the interests of coil-coating. A long list of advantages can be immediately drawn up for this process:

- 1. Extrusion-coating is an existing industrial process.
- A wide range of polymers can be applied in this way and, if necessary, polymers can be tailor-made for a particular end-use and applied by extrusion coating.
- 3. Solvents are entirely absent, and heating costs are greatly reduced.

- 4. Coatings of "lacquer" thickness can be applied at very high speed.
- 5. If thermosetting systems are not used, application and cure can be regarded as a single operation, not as at present, as two separate parts of the coil-coating process.
- 6. Material fed to the extruder can be in the form of a powder produced from predispersed ingredients, ground or chipped compound, or even a liquid "plastisol."
- 7. An extruder takes up very little line space, the extension of the equipment being at right angles to the direction of strip travel.
- 8. Provided that a little space is left after the main curing oven, an extruder can be inserted at this point on conventional coil-coating lines at no great cost, the laminating and embossing equipment normally placed in this area being utilised for laminating the extrusion coating.
- 9. Adhesion is often obtained to metal under these laminating conditions without the use of a separately applied adhesive, or with polymers that would be difficult to adhere in any case, such as polyethylene and polypropylene.

Even with an established process like extrusion coating, there will be many technical and engineering problems to be overcome before such a coating method can take its place along with the universally accepted technique of reverse-roll application in coil-coating, and for this reason the pilot plant approach to its evaluation is still mandatory. It is appropriate to consider briefly some of the characteristics and variables of the extrusion coating process, as these may apply to coil-coating.

An extruder continuously converts solid or semi-liquid mix, composed of polymer(s), pigments, plasticisers, stabilisers, processing aids, and so forth, from a relatively heterogeneous condition to a homogeneous melt at the required viscosity and temperature. As shown in Fig. 1, it consists basically of a flighted screw rotating in a cylindrical barrel. An alternative design is an extruder with twin intermeshing screws. The compound in the hopper is fed into the barrel and is carried along by the screw. As the material moves along the barrel it is converted into the molten state under the combined influences of external heat and shearing forces. The design of extruder screws is a very specialised field of engineering, but the screw usually consists of three "zones." These are designated, feed, compression, and metering zones. Because of the varied rheological characteristics of different polymers, there is an optimum screw design for a particular polymer. However, general purpose screws for a range of polymers can be used with little loss of efficiency.

Attainment of melt homogeneity is vital for a high quality coating and so the length to diameter ratio (L/D) of the



Fig. 1. Diagrammatic representation of a single-screw extruder

barrel must be high. The exit end of the barrel is connected via an adaptor to the extrusion coating die which must produce a homogeneous web of molten coating of uniform thickness at the required width. The main consideration at this point is to ensure that there are no "dead" spaces within the die where material can accumulate, and degrade or otherwise cause subsequent contamination. Pvc is well-known to be a serious troublemaker in this respect, with the dangerous possibility of auto-catalytic degradation proceeding back through the compound in the extruder barrel.

The conventional method of applying the extrudate is for the web from the extruder die to be drawn on to the substrate in the nip between a rubber-covered pressure roll and a polished stainless steel roll, which is internally water-cooled (Fig. 2). The chill roll, during its very brief contact time



Fig. 2. The extrusion coating process

with the coated substrate, normally has to provide sufficient cooling to enable the strip to be recoiled, but, in the case of a metal backing, there is no reason why the usual watercooling methods as at present operating on coil-coating lines should not be used.

Within the relatively simple process outlined above, there are a number of interacting variables which influence the physical properties of the coating and its adhesion to the substrate. The principal factors are: extrusion temperature of the polymer; die opening; air-gap distance; nip pressure; extruder output; line speed; substrate pretreatment; postheating of product; chill roll temperature; and presence of an

#### **Discussion at Eastbourne Conference**

MR F. ARMITAGE asked to be reminded of Mr Newton's assessment of the paint used in the USA for coil coating application.

MR D. S. NEWTON said the figure was 6 million gallons. This was his own calculation, and he noted that Mr Armitage had quoted in his paper from an American source.

MR ARMITAGE replied that although he had given in his paper the figure of 25 million gallons quoted by Kline, he had subsequently come across another American source which quoted 10.5 million gallons for a year earlier. These two estimates could not be reconciled, and one possibility was that some gallonage applied by spray on to continuous coil had been overlooked in the lower figure, but it might be oxidising or protective atmosphere surrounding the molten web.

Over the last decade much experience has been gained in extrusion techniques. Thin polymer coatings (approximate range 0.0002"-0.0005") have been applied to paper and board substrates as a barrier to moisture and grease and to impart heat stability. Aluminium foil has been similarly treated. Among polymers already used commercially are high and low density polyethylene, polypropylene, ionomer, ethylenevinyl acetate copolymers, phenoxy-resin and nylons. As mentioned above, two of the attractions in considering extrusion for coil-coating are the vast amount of experience gained in extrusion coating technology and the knowledge that grades of polymer have already been specifically developed for the extrusion process.

There are certain markets which would benefit immediately from the use of extrusion coating in coil-coating lines. The first of these is in the high-speed application of lacquers to tinplate, tin-free steel and aluminium. Present line speeds are too slow to produce a competitively priced product, but speeding up with existing technology would involve ovens of inordinate length, and, in the case of tinplate, the temperatures required would melt and thus destroy the carefully prepared tin finish. There has been a certain fixation in the industry for assuming that lacquers must be thermosetting, but consideration of some thermoplastic materials shows that very inert properties are obtainable from such polymers when they are of sufficiently high molecular weight. The problem, of course, has been to obtain satisfactory solutions or dispersions of such polymers.

Another interesting market for extrusion coatings is in the field of vibration-damped laminates such as "sound-deadened steel." Development of this product has been hindered by the lack of satisfactory methods of applying resins in production.

Extrusion coating would not be without its problems, and the difficulties which immediately come to mind are those of effecting rapid change from one coating system to another, colour control, and the means for applying coatings to the reverse side of strip. However, perhaps enough has been said to encourage equipment suppliers to consider engineering modifications to existing machinery, which will help to smooth the path and make extrusion as much a part of the coilcoating scene in the year 2,000 as roller-coating and ovens are today.

[Received 1 January 1973]

reasonable to suppose that a figure in excess of 15 million gallons during 1972 would be acceptable, and one could not, at this stage, dismiss the Kline statistic without further consideration.

Mr Armitage then spoke of extrusion coating and asked whether the energy requirements of the extruder were very much less than the normal heat requirements of a solventbased material.

MR NEWTON said that it was necessary, at present, to heat metal which was passing through a very large oven; moreover, it was heated, incidentally, in order that the coating would be heated and cured, and it was also necessary to remove the solvents and keep the solvent concentration to less than 25 per cent of the lower explosive limit. The actual heating costs for the extrusion in comparison with all this were really very small. Furthermore, it might be possible with the extrusion process to omit the separate stage of applying a primer, which was involved with most of the coating that was carried out at the moment. This was a further bonus as far as heating costs were concerned.

MR ARMITAGE thought that if the powder melt were being extruded on to cold metal (without primer), the improved adhesion was very unexpected.

MR NEWTON answered that polyethylene and polypropylene would be the two materials to consider, because these were the ones which could be applied to metal at the moment from hot melt without carrying out some separate oxidation reaction at the surface. It was presumed, in fact, that the oxidation occurred as the extrudate emerged; it was at quite a high temperature in the case of polyethylene. They were discussing temperatures of nearly 300°C. It would be very difficult to obtain a bond with, for example, a polyethylene film applied at a very much lower temperature, unless there was an additional operation to oxidise the surface. It was Mr Newton's view that, as one could obtain good adhesion in these circumstances, there was much less difficulty with many of the other coatings which would be applied.

MR ARMITAGE asked what other resins could be applied without primer and give acceptable performance. He suggested pvc.

MR NEWTON said that pvc would not work, unless something were being incorporated into it. Pvc was a very awkward compound in this respect, and it was certainly one of the most difficult materials to extrude, because of the ease with which it could be catalytically degraded. If one were going to extrude a pvc either from a compound, powdered compound or from a pvc plastisol, then some sort of priming system would still be applied.

MR ARMITAGE remarked that there were implications that this method was restricted to thermoplastic powders; he presumed that a thermosetting material would have to be heat-cured through a normal oven?

MR NEWTON replied that this was not necessarily true, any more than in the case of the extrusion of rubber, which was a thermosetting material. This was extruded in the thermoplastic state and, subsequently, vulcanised. One was not restricted to the use of thermoplastic materials. It would depend, obviously, upon the temperature at which the compound was extruded, and if the lines of the future could incorporate a further heating stage, then it would be possible to use thermosetting materials.

It was often possible to obtain the required properties from something which was primarily thermoplastic, rather than from a very cross-linked thermosetting structure, and since one was dealing with a material which was going to be fabricated after application of coating anyway, this was a point very much in favour so far as the advancement of the process was concerned.

MR ARMITAGE then asked about the status of the invacuo electron beam system of curing developed several years ago by the British Steel Corporation; this system utilised, perhaps, an extrusion method of application.

MR NEWTON said that electron beam curing was only "half a process". There was still the coating to apply, and a great deal of ingenuity had to be utilised even to do this in the way which Ford and others had done in the US, by curing, not in a vacuum and with a high-energy electron beam. It was certainly more difficult when applying a vacuum in order to utilise the low-energy electron beam source. Extrusion coating was a process already in existence; it was already a familiar type of coating operation in the paper industry, where thin coatings were applied at very high speed.

It might be unwise to look at an entirely new process, with all the difficulties involved in scale-up. The engineering industry was never going to take up anything which would involve vast capital expenditure, until it was reasonably certain that there would be some sort of return on the capital. Certainly, one could present very attractive technical papers about electron beam curing, but as far as the practicalities of the situation were concerned, it did seem that these might very well prove to be insuperable. Even electron beam curing in air was now being abandoned by the few lines using it in USA as part of the pre-painting process or on a pilot-plant basis.

MR TUUKKANEN asked for some figures relating to the various binder systems used for coil coating, generally, and especially for exterior use.

MR NEWTON said that the situation had been different in different places. In the United States, the industry had grown up from fairly small beginnings, and along with an industry which was able to handle coil coating; this was particularly true of the principal market areas of residential siding and pre-engineered buildings. It had been possible, therefore, to utilise thin paint coatings almost from the very beginning in America, because they were handled in the right sort of way, and there was very little physical damage sustained either in the fabricating process or in the subsequent erection of the buildings. The position in Europe, and in the United Kingdom in particular up to the second half of the 1960s, was that those in the building industry were not adept at handling pre-coated material, and there was a tendency towards thick, embossed coatings which could be walked upon with hobnailed boots if necessary, and would not show the damage. This latter tendency posed a problem for the future with regard to coatings which might perform very much better than the thicker coatings but which, since they were thin paint coatings, would not be accepted very well by the people who were doing the fabrication. The best example of this was with the fluorocarbons, such as polyvinylidene fluoride (pvf2), which had very much to commend it as the basis for coil coating for use on buildings. It is difficult to see how this material was going to be used to full advantage, however, until the architects and those who specified materials appreciated that coatings like this, although only 25µm thick instead of 250, had exceptional properties, and it was worthwhile taking a great deal of care with them. He had seen in this country an aluminium smelting plant where pvf2 had been used for cladding, and the building was in a terrible condition. Bitumen had been splashed over the coating, but even this could have been cleaned off, probably, if it had been done soon enough because pvf<sub>2</sub> was very inert. In fact, it had been left, and this was an example of a very high-grade material being completely misused.

MR J. S. BOROKY asked whether the costs of using  $pvf_2$ would be prohibitive, even when set against the lack of maintenance (labour) costs for a period of, say, 10 years. Under extreme weather conditions, such as high ultra-violet radiation in Australia, they had to look for improvements even among the more costly types of polymer.  $Pvf_2$  under



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these conditions could still be economical, but he was not sure whether there were other difficulties in this case.

Secondly, he commented that the adhesion properties of aqueous coatings were sometimes insufficient, and asked whether this could be corrected in the case of reverse roll coating by electrostatically charging the relevant rollers. Correcting by additives might later adversely effect corrosion protection properties.

MR NEWTON answered briefly that pvf<sub>2</sub> was much more expensive, but people who were prepared to pay for it could

expect very good performance. It was possible to completely ruin a  $pvf_s$  coating by improper formulation. Provided it was correctly formulated, however, there was no reason why such a coating should not last for ten years without maintenance, even in conditions of high ultra-violet activity.

With regard to roller coating application, this was a very well-established process, and the particular designs of roller coaters had become very stereotyped. They could be changed, but it was now possible to make the necessary modifications so that water-based systems could be applied with the existing type of roller coating design.

#### Powder coatings\*

#### By S. T. Harris

17 Hall Walk, Coleshill, Warwicks B463ES

#### Summary

Certain physical aspects of powder coatings are discussed with attention paid to the fluidity of granular powders. The essential manufacturing procedures are reviewed and the comments are related to those factors which may affect the rheological behaviour of powder coatings.

Testing methods are recommended to assess the physical character-

#### Keywords

Types and classes of coatings and allied products

Powder coating

#### Revêtements en poudre

#### Résumé

On discute certains aspects physiques des revêtements en poudre et on souligne la quasi-fluidité des poudres granulaires. On passe en revue les procédés essentiels de fabrication et l'on mentionne ces facteurs qui exercent une influence sur le comportement rhéologique des revêtements en poudre.

#### Pulverlacke

#### Zusammenfassung

Besprochen werden gewisse physikalische Gesichtspunkte, wobei der Flüssfähigkeit körniger Pulver besondere Beachtung geschenkt wird. Die wesentlichsten Herstellungsverfahren werden besprochen, und die Kommentare auf solche Faktoren bezogen, welche das rheologische Verhalten von Pulverlacken beeinflussen könnten.

Prüfmethoden werden empfohlen, um die physikalischen Charakteristika von Pulvern zu bewerten, und der Wert dieser verschie-

#### Порошковые покрытия

#### Резюме

Обсуждаются некоторые физические характеристики порошковых покрытий, обращая внимание на текучесть гранулированных порошков. Дается обзор основных промышленных процессов по отношению к тем факторам которые могут повлиять на реологическое поведение порошковых покрытий. Рекомендуются методы испытаний для оценки физических характеристик порошков и значение этих различных испытаний

#### Introduction

In pursuit of the general theme of the Conference, "Towards 2000," there are few aspects of the surface coatings industry which are more logical in development than the concept of powder coatings.

Industrial paint users are currently occupied with costly and troublesome alterations to their production plants to permit the continued use of paints in the medium flash point range. The new legislation on inflammable liquids can be only a first step towards the eventual establishment of painting processes free from fire hazard, and if this aim is coupled with the desire to prevent atmospheric and water pollution then one result could be the extensive use of powder coatings. istics of powders, and the value of these various tests is described in a subjective manner. An important aspect of the production of powder coatings is the consideration of a safe working practice and the essential precautions are outlined.

Possible avenues of powder coating development are summarised.

Properties, characteristics and conditions primarily associated with coatings during application

flow characteristics

On propose des méthodes d'essai pour l'appréciation des caractéristiques physiques de poudres, et l'on décrit dans une manière subjective la valeur de ces divers essais. Un des aspects importants, c'est la considération de la sécurité de la méthode de travail et l'on trace les grandes lignes des précautions essentielles.

denen Prüfmethoden wird von einem persönlichen Standpunkt aus beschrieben. Ein für die Erzeugung von Pulverlacken wichtiger Gesichtspunkt ist die Beobachtung einer sicheren Arbeitsroutine. Die wesentlichsten Vorsichtsmassnahmen werden kurz dargelegt.

Schliesslich werden noch die zur Weiterentwicklung möglichen Wege der Pulverlackierung zusammengefasst.

описывается субъективным образом. Важным соображением в производстве порощковых покрытий является вопрос безопасности рабочих условий и перечисляются основные меры предосторожности.

Суммируются возможные направления в развитии порошковых покрытий.

There are, of course, other alternative methods of coating which would minimise the hazards of the painting process, but the need of industry to increase production rates without excessive demands upon factory space and finances will promote a flurry of activity in powder coating development.

A lot of information has been published already about the techniques and principles of formulating powder coatings, and it is not too difficult for the small paint manufacturer to set up a powder coating production unit with the technical assistance of the machinery suppliers and a modest capital outlay.

Paint chemists are renowned for their ingenuity, diligence and ability to make a "silk purse from a synthetic sow's ear,"

<sup>\*</sup> Presented at the 1973 Eastbourne Conference.
#### 1973 (12) POWDER COATINGS

so that the problems of translating hammer finishes, shrivel lacquers and the whole range of conventional industrial finishes into powder form, is not beyond their inventive capabilities.

More difficult problems, that face the paint manufacturer who seeks to make powder coatings a commercial reality, are discussed under four sub-headings. This paper attempts to recount the present state of the art, and to suggest possible avenues for future examination.

The four sections deal with the fluidity of powder coatings, manufacturing procedures, testing for batch to batch reproducibility, and safety procedures in manufacture.

#### Fluidity

The rheological behaviour of irregularly-shaped particles, ranging from submicron size to one hundred microns in diameter, has proved to be difficult to assess in many industries, because there is no simple test equipment for determining the flow properties of powders.

It is difficult to apply mathematical principles to the flow properties of liquids, but at the solid/gas interface of a powder system it is even more difficult as the gaseous continuous phase does not assist the transmission of forces.

Measurement of the flow characteristics of particles through channels or orifices can help solve storage and recovery problems. However, the only worthwhile observation is whether or not the particles are free-flowing.

The factors which affect the flow properties of powders may be listed as: particle size; particle shape; cohesive forces; presence of moisture; size segregation; electrostatic charge; density; presence of flow aids; and packing.

#### Particle size

It is commonly accepted that particles may be described as being ultra fine (below  $1.0\mu$ ), super fine ( $1.0-10.0\mu$ ) or granular ( $10-100\mu$ ).

Free flowing powders are found to be almost completely granular with an almost complete absence of particles below  $10\mu$ .

Fig. 1 shows the Coulter-Counter graph of a typical epoxy powder particle size distribution. This particular powder gives excellent flow characteristics, good charge acceptance during electrostatic spray application, and good penetration and wrap-round qualities.

In Fig. 2 the same powder, but with a higher proportion of fines, has a tendency to surge through the electrostatic gun and exhibits poor penetration.

The powders shown in the above graphs were classified by air-sieve methods into three distinct regions of particle size:  $10-32\mu$ ,  $32-45\mu$ , and  $45-75\mu$ . Electrostatic spray application trials, using the enclosed box technique (described in the testing section), showed the particle range  $10-32\mu$  to be significantly better than any other for the electrostatic deposition of powder due, no doubt, to the better charge/ density ratio of the smaller particles. There was little to choose between the other two particle ranges, although flow properties were improved by increasing the particle size.



Fig. 1. Coulter-Counter graph of particle size distribution for a typical epoxy powder



Fig. 2. Coulter-Counter graph for a powder having a higher proportion of fine particles

Automated, industrial, powder processes will require the best deposition rates at relatively low film thicknesses and it does seem, therefore, that particles in the range 20-30 $\mu$  may best serve these requirements.

On the graphs shown as Figs. 1 and 2, the smooth curve denotes a uniform distribution of particles within the commercially acceptable range of particle size. A departure from this uniformity, such as that shown in Fig. 3, can result in a powder which agglomerates easily and is therefore difficult to apply.



Fig. 3. Coulter-Counter graph for a powder outside the commercially acceptable range of particle size, and which would agglomerate easily

#### Particle shape

The ideal particle is spherical, but it is difficult to achieve this shape by the present methods of comminution because of the brittle nature of the low molecular weight powder coating resins.

There are three avenues of development which may lead to the uniformly, spherical particle; an increase in molecular weight and complexity of the resins thereby making them less friable; a reduction in the force needed in the comminution of these low molecular weight materials; and the introduction of a carrier or lubricating phase during manufacture. This latter technique has already been highlighted through spray drying processes.

#### **Cohesive** forces

Inter-particle forces are mostly non-specific and are mainly the result of Van der Waals interactions. They will increase as the particle size is reduced and will vary appreciably with relative humidity. Electrostatic charges, even if these are dissipated quickly, may bring particles within the range of Van der Waals forces. A simple screening of the powder prior to application, by the use of a vibratory coarse screen, should be sufficient to annul temporarily the effects of cohesive forces.

#### Presence of moisture

It is a common fallacy that it is necessary at all times to "keep your powder dry," for it can be shown that a moisture content as high as 1 per cent does not adversely affect the flow characteristics of most powder coatings. In fact, a small quantity of water (in the region of 0.4 per cent) can be most beneficial.

Whilst it has already been stated that relative humidity can promote cohesive forces, this is only a minor contribution to poor flow properties. Thin layers of moisture adsorbed on the particle surface can promote free flow by imparting a lubricating effect, although it is equally true that thick films of moisture can bind particles together by surface tension.

The presence of moisture at the correct level will enhance the charge acceptance qualities of a powder and assist the eventual, but necessary, dissipation of charge.

A completely dry powder containing a small quantity of silica gel will show very poor charge acceptance properties.

The essential presence of water is one of the reasons why powder applicators are instructed to mix oversprayed powder with fresh powder prior to use.

#### Size segregation

This occurs mainly during the filling of powders into their containers and results in an excess of fine particles being formed at the centre. If the powder bed is vibrated then large grains will rise to the surface, and this circumstance can occur with most of the feed hoppers attached to powder plants, and also during the transportation of powder in bulk.

In considering the flow of powders from a storage silo there are several interesting properties which may be demonstrated experimentally. The rate of discharge of the powder is independent of the head of material in the vessel, unless the head is close to the point of discharge. Most of the weight of the powder is carried by the walls of the vessel and, unlike fluids, the pressure caused by a powder does not increase linearly with depth but tends exponentially to a limiting value.

#### Electrostatic charge

Powder particles, in general, exhibit electrical resistivities between approximately 10  ${}^{s}\Omega$ cm and 10<sup>11</sup> $\Omega$ cm. It is best to divide this range into three sections to consider the effect of resistivity.

- 1. Resistivity below  $10^4\Omega$ cm. An electrostatic charge formed during the manufacturing process or collected during spray application will be lost readily by the conductive particles. Carbon black has a resistivity as low as  $10^{-3}\Omega$ cm, and products containing a high percentage of carbon black should accept charge and discharge quite easily.
- 2. Resistivity in the range  $10^4\Omega$ cm to  $5 \times 10^{10}\Omega$ cm. Most powder coatings show resistivities in this range, and the particles do not easily lose their charge.
- 3. Resistivity above  $5 \times 10^{10}\Omega$ cm. Certain metallic oxides exhibit high resistivities which can result in back discharge of deposited powder layers and which will not allow the particles to lose their charge. In general, it has been shown that electrostatic charge build-up can reduce the tendency of powders to flow. This is a further reason for advising powder coating users to mix new and reclaimed powders.

#### Density

Powder coatings of high density will flow more readily than those of low density and it is therefore to be expected that a highly pigmented white powder coating will flow more easily than a lowly pigmented black powder coating, if all other factors are equal.

#### Presence of flow aids

Small amounts (up to 0.5 per cent by total weight) of finely divided silicaceous materials can improve the flow characteristics of powder coatings, which normally agglomerate during manufacture.

In the case previously mentioned, where an excessive layer of moisture caused the particles to be bound together by surface tension, the presence of these finely divided, often sub-micron, materials may assist the flow properties by disrupting the cohesive film.

These flow aids must be incorporated by dry blending techniques and, once again, it is advisable to pre-blend new and reclaimed powder.

#### Packing

The manner in which a powder packs into a container will be profoundly affected by the method of pouring the powder, and it is possible to have a range of bulk densities within the same container. If a tightly packed powder is caused to flow, it can be shown that some degree of dilatancy is a necessary first step in order that the mass of particles can change shape and commence motion.

The successive steps for the flow of powder from an unrestricted container have been detailed by Jenike<sup>1</sup> (see Fig. 4). Powder coatings with poor flow characteristics within the hopper will exhibit the effect shown in Fig. 4.g which is commonly referred to as "rat-holing."

Fine grinding equipment already available will allow the selection of four variable factors, and experience of the effect of each variable will enable manufacturers to "tailor-make"



## Manufacturing procedures

The accepted procedures for the manufacture of powder coatings have been outlined in an efficient and concise manner by Levinson<sup>2</sup>.

Powder coating manufacture involves the adaptation of plastics handling machinery to produce the correct chemical homogeneity and physical structure required for the end-use application.

The Z-blade mixers and the variety of extruders must be modified to give improved efficiency of dispersion if the powder coating manufacturer is to ensure the necessary complete homogeneity. Yet, paradoxically, the work expended by the granulating and fine grinding machinery is comparable to the effort of taking a sledge hammer to crack a walnut, and it is, therefore, difficult to control powders to the correct particle size and uniformity of size distribution.

Top quality powder coatings will demand pre-dispersion techniques prior to the use of the extruders, and it is not difficult to foresee an increasing demand for pigment predispersions, and the wider use of Z-blade mixers for the incorporation in a master batch of a variety of additives.

Improved homogeneity of all but one component can be achieved with equipment that is currently available. The exception, at the present time, is the cross linking agent as this is often so reactive that it must be incorporated quickly and at low temperatures. The problems with the present curing agents for epoxy resins are due to their relatively high melting points and poor solubilities in the resin.

A new range of compatible curing agents would be a major step forward in powder coating technology.

The extrudate may possibly benefit from the introduction of shock cooling techniques, which could improve the storage stability and facilitate the eventual comminution to the correct particle size and shape.

The problems of fine grinding will require investigation of the work expended by the grinding mill, which must be reduced to a level at which a minimum of fines are produced. Alternatively, fines produced might be removed continually and then reconstituted. powders to correct particle size and distribution.

Tinting of powder coatings is a field in which there is limited experience, but it may not prove to be one of the major problems of colour matching. Strong colours will have to be master batched to develop maximum colour strength before being added to the extruder pre-mix (on which a simple check can be performed within a period of 30 minutes). There is a genuine need for co-operation between pigment suppliers and powder manufacturers, to develop a range of pigments which will not necessarily be similar to those used in conventional industrial finishes.

For example, is it necessary to supply pigments in micronised form when the powder manufacturer already possesses his own micronising equipment? The experience gained by the pigment supplier in the field of plastics may be a starting point but it should be understood that higher pigment loadings are required for the thin film application of powder coatings.

Finely-divided titanium dioxide pigments are found in powder coatings as a relatively high percentage of the finished product, and the particle size of the pigment may control the proportion of fines due to shearing at the interface of the pigment agglomerates.

Resin suppliers have co-operated wholeheartedly in the development of powder coatings, and it is hoped that pigment suppliers will make their own contribution to this new technology.

#### Testing procedures

#### The assessment of particle flow

It is commonly accepted that the angle of repose of a heaped powder is a direct measure of its ability to flow; powders with angles of repose less than  $40^{\circ}$  are said to flow easily.

Cohesive powders of less than  $100\mu$  diameter have high angles of repose, but the test is easy to apply and may be used as a simple method for the assessment of batch to batch conformity.

The angle of repose can show considerable variation according to the way in which the powder is poured, and it is best to assess this property by a drainage method. A flat, circular, steel plate with a smooth surface of known diameter, is mounted upon a tripod in the middle of a wooden platform which has a central drainage hole. The tripod legs are adjusted so that the surface of the plate is level, and the hole in the platform is closed by a cork. A large tin-plate open-ended cylinder is placed on the platform to surround comple:ely the cylindrical plate and tripod (Fig. 5).



Fig. 5. A method for assessing the drained angle of repose

Powder is carefully poured into this cylinder and it is then allowed to drain through the hole in the wooden platform. The height of the apex of the powder remaining on the cylindrical plate is measured by means of a pointer attached to a retort star.d.

#### The angle of repose, $\alpha = \tan^{-1} 2h/D$

where h is the axial height of the cone of powder and D is the diameter of the cylindrical plate.

Wide variations in the angle of repose may be due to compaction of the powder or to a high moisture content.

Experiments have been made with a series of open-ended glass cylinders with different outlet aperture sizes, but the results have been too inconsistent for meaningful comparison.

A completely practical method is to place the powder into the feed hopper of the electrostatic hand gun, then disconnect the powder feed line from the gun and allow the powder to be transported from the base of the hopper and returned to the top of the hopper. If there is no surging or blocking of the powder during a ten minute cycle, then it is unlikely that difficulties will be encountered on site.



There may be some merit in the future development of a testing apparatus employing the principle of the Jenike shear cell<sup>1</sup> which should enable the angle of internal friction of the powder to be determined, and this is a more relevant measurement of cohesive powders than the assessment of angle of repose.

The test equipment consisted of a shallow cylindrical cell which was split horizontally. A sample of powder to be tested was first compacted by the device shown in Fig. 6. This compacting force must be carefully chosen to avoid the limiting degree of compaction beyond which the side shear force cannot easily be applied. Compaction is necessary to ensure that the volume does not change during the test. The lower half of the cell was fixed and a shear force applied at a constant low rate to the upper portion, which was free to move. A number of different loads were applied to obtain a relationship between normal stress and shear stress, and these plotted to give the yield locus, as shown in Fig. 7, where \delta is the effective angle of friction.



There are several interesting observations to be drawn from this graph.

- The yield locus does not extend indefinitely, but terminates when the ratio of the normal stress to shear stress reaches a critical value beyond which compaction occurs.
- The method by which the powder is consolidated will define the position of the yield locus.
- 3. Extrapolation of the yield locus to the shear axis gives a measure of cohesive strength.
- Extrapolation of the yield locus to the normal stress axis gives a measure of the tensile strength of the powder.

It is also possible by this method to calculate the angle of friction between the powder and the wall of the container.

#### The assessment of particle size

Hand screening of powder through a range of metal or nylon sieves is an unreliable method for the assessment of particle size distribution below  $50\mu m$ .

A reasonably close check on batch to batch compliance with accepted standards can be made by use of an air-jet sieve, which is effective above approximately  $20\mu$ m. Finescreen sieves are costly items for they are made by an electrodeposition process and are not sufficiently robust to withstand frequent cleaning.

The most accurate and speedy method for particle size analysis is, inevitably, the most costly in terms of capital outlay. This is an electrical resistivity method which requires a suspension of particles in an electrolyte to be drawn through a small orifice, the passage of particles between two electrodes being metered electronically.

The electronic counting device can analyse a particle distribution from  $100\mu m$  down to  $1\mu m$  in 20 minutes. A necessary first step in this analysis is the preparation of the dispersion by ultrasonic methods. Typical readings are given in Table 1 and these relate to the graph shown in Fig. 1.

 Table 1

 Typical results of an electrical resistivity method of particle size assessment (see Fig. 1)

Equivalent particle diameter	Number of particles above size D	Cumulative weight per cent above D
D	N	
20.3	0	
16.1	1 1	0.41
12.8	10	2.03
10.2	99	10.6
80.6	395	26.0
64.0	1,087	43.9
50.8	2,321	59.3
40.3	4,031	69.9
32.0	6,784	78.9
25.4	10,940	85.4
20.2	16,761	90.2
16.0	25,670	94.3
12.7	35,364	95.9
10.1	46,553	96.7
8.0	63,631	98.4
6.35	96,308	99.2

#### The assessment of electrostatic attraction properties

The rectangular box shown in Fig. 8 is approximately 30in by 9in square. It has a wooden frame and Perspex side panels. At one end, a hole is cut into the wooden wall to permit the electrostatic hand gun to be inserted to a depth of 6in. A wooden bracket is constructed to hold the gun in place. At the opposite end of the box is an open wooden frame, which is covered by a piece of  $10\mu m$  filter cloth, and this end is easily removed from the frame to permit access

to the interior. A 6in by 4in mild steel panel is suspended from hooks attached to a copper bar on the exterior of the roof panel. This panel is sited nine inches from the detachable end section and is earthed by a lead attached to the copper bar. The panel was weighed and mounted on the supporting hooks. Powder was allowed to flow through the gun before the gun was inserted in the box.

When the gun is inserted, the box is closed, and powder discharged for a period of ten seconds.

The air pressure to the gun and the voltage were recorded, and these were correlated with the weight of powder deposited on the panel. Independent variation of the voltage settings and the air pressure enable an assessment of the optimum conditions for electrostatic deposition of the powder to be made.

The apparatus can also be used to effect a comparative test alongside a sample of a retained, acceptable batch of similar powder coating.

Replacement of the mild steel panel, by a small section of slotted angle iron can afford a visual means of demonstrating the wrap-round qualities of a powder and the degree of penetration into the sharp angle.

## The assessment of specific gravity

A reliable method is outlined in the British Standard<sup>3</sup> method for testing the specific gravity of pigments by the application of a vacuum to remove entrained air. It is important to choose a high boiling point aliphatic solvent to serve as the displacement liquid.

An accurate measurement of specific gravity can be useful to determine the "mileage" of the powder coating, which may be expressed as

Other tests will, no doubt, be applied to maintain standards, and the remarks here on testing methods have been confined



Fig. 8. A single device for assessing the efficiency of powder deposition

to those tests which may not be familiar to the paint manufacturer.

## Safety during manufacture

A code of safe practice, for the application of powder coatings by electrostatic spraying, has been outlined by a technical sub-committee of the Paintmakers Association<sup>4</sup>, and this document covers adequately all safety aspects of powder application. It is essential that a similar code of safe practice should be adopted by the powder manufacturer, and the following comments may help to establish clean and safe working conditions within a paint factory.

Accumulations of organic dusts can present a hazard of explosion if they exceed certain definable concentrations in air and if, at the same time, they are subjected to a source of energy sufficient to initiate combustion.

The building designed to house powder manufacturing equipment should, ideally, be a single storey, brick walled one with a flimsy roof constructed mainly from translucent plastic. Ledges and roof truss sections should be kept to a minimum to avoid unnecessary dust collection, for any primary explosion in the vicinity of the energy source could dislodge this accumulated powder and initiate a more severe secondary explosion.

Dust producing operations occur at the following process sites:

- (a) the weighing station;
- (b) the loading and emptying points of the premixer;
- (c) the charging of the feed hopper of the extruder;
- (d) the granulating of the extrudate;
- (e) fine grinding of the granulated extrudate;
- (f) screening to remove oversized particles;
- (g) filling of containers; and
- (h) cleaning of filter bags and containers used for intermediate stages.

It is a costly operation to install extraction equipment at all of these sites, for the only effective method is the use of separate extraction fans for each dust producing operation.

Transportation of the material through the stages of manufacture is best achieved by the use of bulk closed containers which are top loaded and emptied from the bottom. A relief pipe in the lid of this container should be connected to the local source of extraction during the filling operation, and this will also assist the eventual discharge from the container.

Workmen in powder-manufacturing departments will quickly realise that the easiest way to clean down equipment, filter bags and so forth, is by the use of compressed air. This cleaning method must not be employed unless the whole cleaning operation occurs within a totally enclosed and well ventilated cabinet.

The use of a totally enclosed, walk-in cabinet may be extended to the housing of the fine grinding equipment, for it would serve a dual purpose by considerably reducing the noise level, which can be in excess of 100 decibels. In these compartments, the cleaning operation could be carried out using an industrial vacuum cleaner, with the air line used only to ensure complete cleanliness in difficult cleaning areas.

Electrical fittings do not need to be flameproof, and in fact it is preferable to specify that they should be dust proof.

The most hazardous process at the present time is the fine grinding of powder coatings containing aluminium. Whilst it can be shown that powders containing aluminium do not have appreciably lower explosive limits than standard powders, there is no doubt that the rate of pressure rise during an explosion is considerably higher. If it is necessary to manufacture these products, then it would be wise to examine the use of cryogenic techniques.

As the powder manufacturing industry grows, so will the bulk volumes of powder being stored or transported, and there is a possibility that considerable electrostatic charges could build up within a storage vessel. Not only must the storage vessel be earthed, but the workmen should be equipped with footwear made from conducting material.

## **Future trends**

In conclusion, it may be interesting to predict possible trends in the field of powder coatings towards the year 2000. Some possibilities include:

- 1. The paint chemist giving way to the "powder physicist."
- 2. Particle size distribution being precisely controlled within a narrow band, and the optimum size in the region of 15µm.
- Fine powders are not easily transported by air pressure and it may be necessary to attract the powder to the article.
- 4. An essentially spherical particle shape will permit much lower ionisation charges.
- The electrostatic gun being replaced in large automatic plants by a modified form of electrostatic fluidised bed.
- Cryogenic methods of fine grinding may become widespread in powder manufacture.
- Resins for powders will tend to have higher molecular weights and higher melting points with increased speed of cure at high temperatures.
- 8. Hardener systems may become entirely resinous.

## Acknowledgment

The author wishes to thank the directors of Arthur Holden & Sons Ltd. for their assistance in the preparation of this paper.

[Received 20 February 1973]

#### References

- 1. Jenike, A. W., Bulletin 108 of the Engineering Experimental Station, University of Utah, 1961.
- 2. Levinson, S. B. J., Paint Technol., 1972, 44, 37.
- 3. B.S. Specification 3483, 1962.
- Paintmakers Association of Great Britain, Provisional document, 1973.

## **Discussion at Eastbourne Conference**

MR J. PELGRIMS asked the lecturer to comment on the technical and cconomic advantages and disadvantages of cryogenic grinding.

MR S. T. HARRIS said that he had carried out a brief assessment of cryogenic grinding and had found that it did offer advantages to the powder coating manufacturing process. The low glass transition temperatures of the resins were affected by mechanical heat build-up in the grinding chambers of pin disc mills and melting of the product could result. The term cryogenic grinding implied the use of temperatures as low as - 100°C, and this was a well-known technique for the fine grinding of tough thermoplastic materials like nylon. With the inert brittleness of thermosetting resins, however, it was more likely that freeze grinding would be adopted where the grinding chamber was merely kept cool. Lower temperaturcs would increase the brittle nature of the thermosetting materials, and result in increased amounts of fine particles. If the grinding chamber were kept at an even temperature, this would considerably increase the output, and the additional cost of liquid nitrogen used for this process would easily be offset by the improved efficiency.

MR S. L. DAVIDSON asked why it was necessary to avoid small particle sizes with pigments. Secondly, he asked why moisture was necessary and what part water and water polarity played in the application of powder coatings.

MR HARRIS replied that his experience of finely divided pigments had been mainly confined to the use of titanium dioxide. It was difficult to incorporate large quantities of finely divided pigment in the extruder, because of the short dwell time and the necessity to wet-out the pigment before the coating was compounded correctly. The larger surface area obviously make it more difficult to wet the pigment. In practice, it had been found that titanium dioxide pigments of large particle size dispersed more easily and enhanced the appearance of the coating. Mr Harris thought it was interesting to speculate on the improvements which might result from the use of even coarser grades of titanium dioxide than currently existed. The fine grinding operation was a micronising process, and it seemed certain that the particles broke at the pigment/resin interface because of the brittle nature of the resin. There were considerably more fine particles in a titanium dioxide pigmented coating than in a low-pigmented carbon black powder coating.

The presence of moisture had been found to be essential in the practical application of powder coatings, and the tendency for powder particles to dehydrate during a coating operation was one reason why the powder manufacturer recommended that oversprayed powder should be re-used in conjunction with fresh powder. Even spillages of water, which had occurred accidentally in certain automatic plants, had not been found to adversely affect the performance of the product. Tests carried out with completely dry powder coatings showed that there was poor charge acceptance. Powders which had been collected in a wet spray booth, filter pressed and redispersed, had a moisture content as high as 1 per cent, and demonstrated good application characteristics. The presence of moisture almost certainly acted as a lubricant which overcame the cohesive forces between the powder particles. The well-known catalytic effect of water improved the curing rate of powder coating.

MR J. DUNDERDALE commented that micronising or similar grinding was necessary to develop fully the pigmentary properties of titanium dioxide, but as powder coatings were thicker and contained more pigments, this might compensate for the presence of aggregates, which would normally cause gloss defects and loss of opacity.

The mixing problems referred to seemed to be similar to those experienced by the plastics industry. Particularly, special surface treatments had been developed which rendered plastic grade pigments more readily dispersible in extruders. He suggested that a special surface treatment might be useful for pigments for use in powder coatings.

MR HARRIS replied that Mr Dunderdale's comments were very relevant to the problems of pigmenting powder coatings. They were concerned with much higher levels of pigmentation and did not need, therefore, the fine particle size required by the plastics industry. The pre-treatment of titanium dioxide made this pigment more easily dispersible in conventional paint systems, but the process was not necessarily advantageous in the powder coating process, and he wanted further work to be carried out in this field.

MR D. S. NEWTON said that epoxy resins were the predominant coatings used in powder coating at present. These were of low molecular weight and highly cross-linked, and this was an advantage for comminution of the extrudate to produce a fine powder. He wondered whether there would be a trend away from this in future, towards more flexible materials of higher molecular weight, which had much more to offer in versatility of properties.

MR HARRIS answered that epoxies at the present time held 90-95 per cent of the UK market for powder coatings. Polycsters had now reached the stage of commercial development, but this was not yet true of acrylics. He was certain that the trend with all of these materials would be towards the production of resins of higher molecular weight, so that they would require the minimum amount of cross-linking. In common with the development of conventional industrial finishings, there would always be a need for thermosetting resins to be plasticised by the thermoplastics. At the present time there were no higher molecular weight alternatives to the resins which were currently available, but he could foresee that the paint manufacturer would encroach very soon on areas which were considered to be the province of the plastics manufacturer.

MR J. M. RACKHAM commented on the difficulties referred to in the paper on the incorporation of large amounts of titanium dioxide, and asked whether Mr Harris had any comments to make on the merit of the thin-film evaporation technique of manufacture.

MR HARRIS said that he had little knowledge of the use of this technique, so that it would be unwise for him to comment at this stage.

MR W. A. WILLIAMS asked Mr Harris to expand on his statement in the "Future trends" section of the paper that hardener systems might become entirely resinous.

MR HARRIS replied that at the present stage of development, they were cross-linking resins by the use of crystalline chemicals which were mainly variations of dicyandiamide used in the curing of epoxy resins, although there had already been some interest shown in the use of polyamide curing agents. The paint industry produced thermosetting industrial finishes by reaction between two resinous systems and there was no reason why this should not be extended into powder coatings provided that the resins had an acceptable physical form. There was a limitation in that most of the crosslinking occurred by condensation reactions, and this did depart from the intention to provide coatings at 100 per cent solids content with no volatile by-products. As Mr Newton had suggested, the industry should be seeking to utilise materials of higher molecular weight, and this could apply to the introduction of resinous curing agents to impart the flexibility required for many end-use applications.

# Next month's issue

The Honorary Editor has accepted the following papers for publication and these are expected to appear in the January 1974 issue:

Accelerated testing of durable coatings by E. Oakley and J. J. Marron

A study of microfouling on antifouling coatings using electron microscopy by J. H. Bishop

Ecological considerations in the biodeterioration of interior surface coatings by D. G. McGlown and G. Old

# Addendum

# Sulfate-reducing bacteria, surface coatings and corrosion

The authors of the above paper (*JOCCA*, 1973, **56**, 363-368) have requested the following additions to Table 2 (page 365) to be published:

#### FORMULATION

Zinc chromate (line 2 in the table)

Busan 11-M1 (line 12 in the table)

By simple addition of the pigment to the pitch-epoxy coating.

Zinc chromate (line 13 in the table)

Busan 11-M1 (line 21 in the table)

By addition of the pigment plus sufficient additional pitchepoxy binder to restore the pigment volume concentration to its original value (changes in the PVC exert considerable influence on the porosity, adhesion, blister resistance etc., of a coating).

# Correspondence

# Chromate and phosphate pigments in anti-corrosive primers

SIR—H. F. Clay and J. H. Cox (JOCCA, 1973, 56, 13) state Zinc phosphate provides an alternative pigment to chromates; it has the advantage of being completely non-toxic....

According to our knowledge, zinc phosphate as tested in rats has an oral toxicity  $LD_{50}$  of 10 to 15gm per kgm. In this connection, it is interesting to note that another highly efficient type of anti-corrosive pigment, the coated-core lead silico chromate pigment like ONCOR® M 50®, is even less toxic than zinc phosphate. It is virtually insoluble in water and its  $LD_{50}$  is greater than 20gm per kgm.

Lead silico chromate has a low specific gravity, thus permitting the preparation of highly economical, corrosion inhibiting paints that show outstanding performance, even when applied on manually de-rusted steel.

Yours faithfully,

J. SCHIEFER

Kronos Titan GmbH, D-509 Leverkusen 1, Postfach 80/90, West Germany.

# Information Received-

#### Bowater treble plastic drum production

Following the successful introduction of Bowater Packaging's Mauser plastic drums to the UK market, two additional large blow-moulding machines have been ordered by the company from Mauser-Werke GmbH of Cologne. The new equipment will more than treble the existing production capacity at Bowater's Drum Division plant in Disley, Cheshire.

## Du Pont (UK) Limited

On 1 October 1973, the company name Du Pont Company (United Kingdom) Limited was changed to Du Pont (UK) Limited.

#### J. & William Burt Ltd. moves to new offices

J. & William Burt Ltd., UK distributors of European specialised printing and allied process machinery, has moved its offices to 4 The Street, Ashtead, Surrey. Tel: Ashtead 77116/8.

#### Resicote/Dai Nippon agreement

Resicoat GmbH of Reutlingen, West-Germany, a mutual subsidiary of Akzo Coatings, Holland and Robert Bosch, West-Germany, has finalised a licensing agreement for the production and sales of electrostatic powder coatings to Dai Nippon Ink Co., Tokyo and Toagosei Chemical Industry Co. Ltd., Tokyo.

#### Sonneborn and Rieck to market 3M products

Sonneborn and Rieck has completed negotiations with the 3M Company, and now acts as agents for 3M to market the "Nextel" range of coating materials in UK markets.

# Vinyl Products' agreement with BP Chemicals

Vinyl Products Limited has announced that it has agreed to acquire the range of polyvinyl acetate and vinyl acetate copolymer emulsions currently marketed by BP Chemicals International Limited under the trade name "Epok". Vinyl Products has been handling the sale of these emulsions since 15 October 1973. Manufacture will be continued at BP Chemicals' Barry factory for several months, but the plant and production will ultimately be transferred to Vinyl Product's factories at Carshalton and Warrington.

#### **New products**

#### Aldecon

Jenolite Division of Duckhams Oils has announced a new range of products, under the name "Aldecon," for the composite degreasing, derusting and descaling of steels in one operation. Three types of acid base are available—hydrochloric, hydrochloric/sulfuric mixtures, and phosphoric.

#### Alu-Red pigment powder

Bush Beach & Segner Bailey Ltd. is now marketing "Alu-Red", a new pigment powder formulated for incorporation in anti-corrosive paints. The pigment is manufactured by blending carefully selected bauxite, which is activated at high temperatures and milled to a fine powder. Alu-Red is a Universal pigment, and can be used with practically every binding material, for decorative or protective paints, and is suitable for incorporation in a wide range of formulations.

#### New spectrometer from Spex

A new spectrometer of high optical speed has been introduced by Spex Industries Inc., and is available in the UK from Glen Creston, Stanmore, Middx. The instrument, known as the f/2.5 "Micromate," includes a standard 1,200gr mm<sup>-1</sup> grating and read-out in nm on a 3-digit counter. The makers claim that the Micromate has a surprisingly good performance in terms of scattered light, resolution and lack of optical aberrations for an instrument of rugged construction and compact dimensions.



The new Spex Micromate

#### Multi-Finex MK 11

Russell Finex Limited has announced its new Mark II conversion of the Multi-Finex sieving, straining and grading machine. The model has a completely new base unit embodying the results of development, research and practical experience gained in the application of these machines during the past eight years.

#### BXL hardener for epoxide resins

The Thermosetting Division of Bakelite Xylonite Limited has introduced a new low-cost, 100 per cent reactive hardener for epoxide resins. Known as Q19567, it hardens epoxide resins in thin films even under adverse conditions—such as low ambient temperature and high humidity.

# Solvent resistant disc for Joyce-Loebl centrifuge

A new solvent resistant disc is now available from Joyce-Loebl for the company's particle size disc centrifuge. The new disc made from a transparent thermosetting plastic, is now standard on all new instruments and can be supplied as a direct replacement to owners of existing instruments. The centrifuge can now be used on a quality control basis for routine particle size analysis in the presence of highly reactive organic solvents.

#### Literature

# Spanish translation of "Paint Film Defects"

The universally known book by M. Hess "Paint Film Defects. Their Causes and Cure," has been translated into Spanish and was recently published under the title "Defectos De Las Capas De Pintura (causas y remedios)" by Editorial Blume, Tuset 17, Barcelona 6, Spain.

#### Analysis of epoxy resin

A new bibliography, containing 61 references including 27 foreign language articles, has now been compiled covering the years 1968-72. Full abstracts are given. The price is £3.00 and copies are available from The Librarian, The Paint Research Association, Waldegrave Road, Teddington, Middlessex TW11 & LD.



Multi-Finex Mark II

# Review

Korrosion-Schutz Durch Lacke & Pigmente By J. Ruf

## DIN A 5, pp. 336. Price DM 58

## Verlag W. A Colomb, Stuttgart.

On perusing J. Ruf's book, the title of which means "Corrosion, protection by media and pigments", one is amazed to find how closely he has supported his explanations with drawings, photographs (several coloured), tables and diagrams. One also notices a large literature/author index composed of 409 footnotes. Many well-known English and American workers in the anti-corrosion field are included. The subject index is only of moderate size, but the table of contents very detailed.

Instead of subheadings, short summaries are given in the margins of pages.

There is also an appendix enumerating the German organisations, technical universities, other institutions and advising associations interested in the anti-corrosive field. Listed here are also numbers and headings for specifications by various authorities e.g. Deutsche Bundesbahn, Deutsche

# Section Proceedings-

# London

Whither the London Section?

The first meeting of the session was held on 27 September 1973, at the Great Northern Hotel. Mr R. H. E. Munn presided over the Chairman's Open Forum with the title "Whither the London Section?" Mr Munn said that the purpose of the Forum was to see how the activities of the section could be made more attractive, and better able to satisfy the wishes of members. In 1972, membership of the Section consisted of 616 Ordinary Members, 73 Associate, 4 Honorary Members and 5 Registered Students, vet the average attendance at normal evening meetings was generally only 40 or so. Half-day symposia were considerably more popular, with attendances of up to 100 or more. The recent two-day symposium at Warwick University, held in conjunction with the Organic Coatings Section of the IMF, had attracted over 300. In other words, if meetings were held during normal working hours attendance was good. This problem of poor attendance at evening meetings was common to most societies in the London area, and some societies had abandoned evening meetings altogether.

Another factor was the type of paper presented. Many authors wished to present to London Section papers which would eventually be published in the *Journal*. It was questionable whether the membership wanted to hear this sort of paper or something of a more practical nature. The latter were very scarce, because of the need for commercial secrecy.

In the extensive discussion which then took place, many points of view were expressed. Some sections, it was said, had more of a club atmosphere; meeting at the same place on the same day each month. One speaker said that he had joined Bundeswehr, Ministries, industrial organisations and, in addition, all standardising bodies in the world.

Very useful is a relevant extensive excerpt from "DIN-Normen" and "RAL-Vereinbarungen" with numbers, dates of issue and titles.

The approach to the subject of this book is modern and up to date. While great emphasis has been laid on the theoretical aspects of the corrosion of metals, practical considerations have not been neglected. Areas adjacent to paint technology—metallurgy, electro-chemistry and pure organic chemistry—have been included in quoted theories some of which have been found to be not yet practicable.

The book contains some unnecessary repetitions, and whilst in a few instances, the author seems to expect too little knowledge on the part of the reader, on the whole the level of presumed scientific knowledge for making full use of the book is very high.

The publishers have been generous with space and makeup; everything stands out clearly.

J. Ruf's book "Korrosion-Schutz durch Lacke & Pigmente" is brilliantly written and full of valuable and useful information for everybody, who is entangled in corrosion problems.

M. HESS

the *Association* mainly to get to know people: this had not been successful. It was suggested that a social officer should be appointed, whose task it would be to "host" new members when they attended meetings.

Mr Munn pointed out that finding a suitable rendezvous for meetings had been under discussion for several years, and many places had been tried in successive years. The present venue, the Great Northern Hotel at Kings Cross was central, had good access to the Underground railways and was licensed so that members could linger over a drink after the meetings for further talk and discussion.

In further discussion, it was found that most of those present preferred a fixed meeting place, and there was also a preference for a fixed day of the month for meetings. Asked how long it would take members to get home after the meeting, it was found that most of those present would spend at least an hour, some even two hours, to get home. This was a problem which was special to London. The question of getting younger members (not necessarily students) to come to meetings was discussed. It was said that many would not be allowed time off to attend meetings held during normal working hours.

Summing up, Mr Munn said that he thought the meeting had served its purpose, and the committee would be paying attention to the many points of view which were expressed.

V.T.C.

We regret to record the death recently of Dr V. T. Crowl (the writer of this report). An appreciation will appear in a subsequent issue. 1973 (12) SECTION PROCEEDINGS

# West Riding

## Progress in radiation cured coatings

A meeting was held on Tuesday 11 September 1973, at the Griffin Hotel, Leeds. Mr A. G. North, of Cray Valley Products Limited, presented a paper dealing with radiation cured coatings.

In introducing his subject, Mr North discussed the nature of electromagnetic radiation and identified those areas of interest for the curing of surface coatings. The field of ionising radiation was of particular interest, since by the direct formation of free radicals in the coating, low molecular weight unsaturated polymers, monomers and oligomers could be converted to fully cured coatings in fractions of a second.

Electron beam curing approached the ultimate in that 100 per cent solids coatings could be cured in a fraction of a second on any substrate, without significant increase in temperature, and with very low consumption of energy. Development in this field ought to be accelerated with the recent introduction of the compact laboratory equipment which Mr North described. The types of polymers and monomers most suitable for electron beam curing were briefly discussed. In general, the required unsaturation was normally achieved by the incorporation of acrylic esters into the molecule.

The operational problems of electron beam curing were considered, as well as some of the formulation problems which could arise. A more recent development was electron beam curing in a vacuum, which enabled higher energy levels to be applied to the coating. However, this presented difficulties in view of the high vacuum required, and an interesting alternative was the glow discharge electron beam, which was being employed for welding and for heat treatment of metals. This used a comparatively "soft" vacuum, which had a major effect in reducing equipment costs.

Ultra-violet (UV) curing of coatings had been used commercially for some years, and mercury lamps were the UV source usually chosen. A new source of UV radiation was the enclosed argon plasma, which produced continuous electromagnetic radiation and was basically similar to the radiation emanating from the sun. Very high energy inputs could be achieved in the plasma and, using reflectors, its radiation could be uniformly distributed over any reasonable area. Apart from compactness and simplicity of operation, its main advantage was that since water cooling was employed and the radiation source was very compact, the provision of inert gas was simple, and a good inert atmosphere could be provided cheaply.

A method investigated by the author was the use of open plasmas as a combined source of UV and infra-red radiation, and Mr North summarised his findings so far.

In its present state of development, the open plasma arc was not a viable system for curing surface coatings except on high bulk substrates such as castings, heavy gauge plate, etc., where the energy requirements using conventional stoving techniques could be prohibitive.

Finally, the commercial aspects were considered and suggestions made on how best to get a realistic and accurate indication of the suitability of a given method of curing for any particular application.

The paper prompted a number of questions and Mr P. Bartrum proposed a vote of thanks for a most interesting, well presented lecture.

R.A.C.

# Notes and News-



The Exhibition Committee is pleased to announce that, in addition to strong representation from companies within the European Economic Community, it has welcomed applications for stand space from organisations, many of whom will be showing for the first time from many other countries. The list of countries represented will be as follows: Australia, East Germany, Finland, France, Holland, Hungary, Italy, Poland, Rumania, Spain, Switzerland, USA and West Germany.

#### Venue

As already announced in previous issues of the *Journal*, OCCA 26 will take place at the Empire Hall, Olympia, London, from 23 to 26 April 1974.

#### International character

The Exhibition, which has long been known as the forum for technical display and discussion for the surface coatings industries, in 1973 attracted visitors from more than 50 overseas countries, and there was direct contribution by exhibitors from 14 overseas countries. The motif chosen for this year's Exhibition shows the flags of the enlarged European Economic Community and, by converging on the flag of the UK, symbolises the welcome extended for many years to exhibitors and visitors not only from these countries but from farther afield to the OCCA Exhibitions in London, one of the capital cities of the European Economic Community. To further the aim of a truly international character, the Exhibition is widely adver-tised in technical journals both at home and in 18 technical journals overseas. Furthermore, it has been the practice for many years to issue information cards in six languages (English, French, German, Italian, Spanish and Russian) and these are widely distributed to firms and individuals in many countries. Interpreters are available at the Exhibition without charge to help both exhibitors and visitors alike.

#### Hours of opening

On this occasion the Exhibition Committee has decided to dispense with the Monday opening and to concentrate on four full days as follows:

Tuesday 23 April		09.30	to	18.00hrs
Wednesday 24 April		09.30	to	18.00hrs
Thursday 25 April		09.30	to	18.00hrs
Friday 26 April	• •	09.30	to	16.00hrs

East and West join in OCCA 26 the European Economic Community's forum for technical display and discussion in the surface coatings industries 1974

#### **Exhibition Dinner**

Following the success of the Exhibition Dinner on the opening day in 1973, the Committee has decided to hold a Dinner at the Savoy Hotel, London WC2 on Tuesday 23 April at 19.00 for 19.30hrs. At the request of some exhibitors a cash bar will be made available after the function for those visitors wishing to use this facility. Full details of the Dinner will be announced in this *Journal* from time to time and an application form for tickets will be enclosed in each copy of the Official Guide.

#### **Official Guide**

The Official Guide will be prepared well in advance of the dates of the Exhibition so that copies can be circulated widely, both to members and non-members, thus allowing visitors an opportunity to plan their itineraries. As well as maintaining the Exhibition's unique position as a purely technical display, manned by technical personnel, the Exhibition Committee has for many years followed the successful policy of encouraging visitors in this way, and by making no charge for admission so that the maximum flow of communication between exhibitors and visitors can take place.

Orders for advertising space in the Official Guide to the Exhibition are now being accepted at the Association's offices. Advertising space is not restricted to exhibitors but exhibitors may reserve space opposite their own editorial entries if they so wish. Full details will be supplied on request from the Association's offices.

#### Aims of the Exhibition

The Exhibition Committee wishes the aims of the Exhibition as stated in the Invitation to Exhibit, to be as well known as possible and accordingly these are reproduced below.

The aim of the Exhibition is the presentation of technical advances in those industries supplying the paint, varnish, printing ink, colour, linoleum and other allied industries. The technical advances may relate to: new products, new knowledge relating to existing products and their uses, or in suitable cases, existing knowledge which is not generally available in the consuming industries. The Committee stipulates that exhibitors present a technical theme—that is display in a technical manner the technical developments in raw materials, plant or apparatus illustrated by experimental evidence. It is essential that a technically or scientifically trained person, who has full knowledge of the products displayed, be available on the stand throughout the official hours of opening.

Whilst the Committee naturally encourages the showing of new products, it does not stipulate that a new product has to be shown each year, since it fully appreciates that there are occasions when this is not possible. Accordingly, the Committee draws attention to the fact that new technical data on existing products are regarded as acceptable subject matter.

#### **Charge to Exhibitors**

The OCCA Exhibitions have for many years been planned to keep costs as low as possible for exhibitors. The charge (which includes stand space, floor, walls, muslin ceiling, fascia with coloured felts and pine trims, and painted name plaque), will be £110 (plus Value Added Tax) per unit of 4.32m<sup>2</sup>, and exhibitors receive a free supply of *Official Guides*, six language cards, badges, folders, and so forth. Since there is no charge made for admission to the Exhibition, no "special facility" tickets are issued for distribution by exhibitors. Each visitor is given a copy of the *Official Guide*, together with a folder for technical literature, showing the location of the stands, upon arrival at the hall.

#### **Travel Agents**

The Wayfarers Travel Agency Ltd., Cranfield House, 97/107 Southampton Row, London WCIB 4BQ., will be allocated a stand adjacent to the OCCA Information Centre at the Exhibition and will be prepared to advise on, and arrange, hotel accommodation and travel facilities to the Exhibition. They will also be able to make theatre ticket reservations for the evenings of the Exhibition.

Please address all enquiries to The Wayfarers Travel Agency AT THE AD-DRESS SHOWN ABOVE, and not to the Association.

# **Optional Professional Grade for Ordinary Members**

## List of successful candidates

As laid down in the report of the Working Party on Education, Training and Qualifications which was adopted in the institution of the Professional Grade, a list of all those Members who have entered the grade will be published in the December issue of the Journal each year. The second such list appears below and represents a most successful beginning for this new venture. A total of 360 Members have successfully applied for admission to the grade since the first notice in the Journal in September 1971; of these admissions, 137 were to Fellowship, 215 to Associateship, and is to Licentiateship. This figure represents 14 per cent of the total Ordinary Membership, and is a most gratifying response, particularly when it is considered that applications from a further 40 members are still under consideration, many of these at the Licentiate level, where candidates have to write a dissertation before admittance.

Members are reminded that all those applying must be suitably sponsored as laid down in the regulations. Also, attention is drawn to the certification fees that came into effect from 1 January 1973. These are: Fellows £10.00 + £1.00 VAT, Associates £6.00 + 60p VAT, and Licentiates  $\pounds3.00 + 30p$  VAT. Appropriate remittances must accompany all applications. The regulations for admission to the Professional Grade last appeared in full in the September 1973 issue of the Journal.

#### Fellows

Anderson, George (Scottish) Apperley, Thomas William James (West Riding) Archer, Harold (Manchester) Arnold, Michael Henry Miller (London) Arnold, James Eric (London) Ashworth, Norman (Manchester) Atherton, Donald (Scottish) Austin, Denis Leonard (Bristol) Balbi, Giorgio (General Overseas-Italy) Banfield, Thomas Arthur (London) Bell, Sydney Hector (London) Bennett, Norman Arthur (General Overseas-Malta) Bews, Ian Charles Randall (London) Bishop, Eric Harold Abbott (Thames Valley) Blenkinsop, Arthur Willis (Newcastle) Bohringer, Eberhard (London) Bourne, John Robert (Midlands-Trent Valley Branch) Brooks, Leo James (London) Brown, Arthur Ernest Girdlestone (London) Butcher, George Alfred (Midlands) Butcher, Kenneth William George (Manchester) Butler, Cecil (West Riding) Caldwell, David George (Wellington) Campbell, George Alexander (Manchester) Carter, Eric Victor (Midlands) Ceresa, Raymond John (London)

Chatfield, Herbert Walter (London) Chessman, Clifford Reginald (South African) Clarke, Harry James (Midlands) Colborn, Douglas Charles (Thames Valley) Cole, Derek (General Overseas-Australia) Cole, Reginald Joseph (London) Collier, Claude William (Midlands-Trent Valley Branch) Collings, Arthur Geoffrey (London) Cook, Harold Gilbert (Manchester) Coupe, Raymond Richard (London) Coverdale, Peter Frederic Muir (Midlands) Cutter, John Outram (London) Day, Keith Julyan (London) Duckworth, Samuel (Manchester) Duell, Arthur Albert (Newcastle) Dunkley, Frederick George (Midlands-Trent Valley Branch) Durrant, George Geoffrey (Hull) Ellinger, Marianne Livia (London) Entwistle, Thurston (Newcastle) Fillingham, Thomas Alan (Hull) Finn, Stanley Russell (Hull) Fullard, John Edward (South African) Gate, Peter Atholl Jackson (South African) Gay, Philip James (Hull) Gellay, Victor Peter (London) Gellman, Alexander (London) Gollop, Percy Lionel (Bristol) Gosling, Harry (Manchester) Gray, Denis Roy (West Riding) Grover, Donald Henry (London) Hampton, Horace Arthur (Manchester) Hawkey, John Albert Lawrence (London) Hess, Manfred (London) Hill, Roger Frederick (Midlands) Hipwood, Hubert Allan (London) Hodgson, Kenneth Vickerson (Newcastle) Holbrow, Gordon Leonard (London) Holt, Alfred Gordon (Thames Valley) Hutchinson, Geoffrey Herbert (Scottish-Eastern Branch) Inshaw, John Leslie (Thames Valley) Jacob, Basil (Thames Valley) Jolly, Anthony Charles (Manchester) Keenan, Henry Wilfrid (London) Kekwick, Leslie Oliver (London) Kinsman, Roy Granville (South African) Kut, Siegmund (London) Landmann, Axel Wolfgang (London) Lasser, Howard Gilbert (General Overseas-USA) Lewin, John Buckingham Grey (London) Lewis, Fred (Manchester) Ley, John Barry (London) Long, Denis Terence (Irish) McGowan, Jesse (Manchester) McKelvie, Archibald Neil (London) McLean, Angus (Scottish) McWilliam, Anthony (Manchester) Mell, Cedric Charles (Hull)

Mitchell, John Edmund (Manchester) Mole, Seymour Lloyd (General Overseas-Canada) Moll, Ivor Stuart D'anvers (Manchester) Monk, Cyril James Henry (Thames Valley) Moon, William Robert (Manchester) Morgans, Wilfred Morley (London) Munn, Raymond Henry Edward (London) Newell, George Ashley (London) Newnham, Herbert Alan (London) Newton, Dennis Sydney (Bristol) Newton, Donald Stringer (Bristol) Nutt, William Owen (London) Oostens, Emile Elie Eugène (General Overseas-Belgium) Paifitt, Geoffrey Derek (Newcastle) Penfold, Arthur de Ramon (General Overseas-Australia) Piggott, Kenneth Elliot (South African) Polaine, Sydney Alan (London) Ray, Stanley Arthur (Midlands) Rechmann, Heinz (General Overseas-Germany) Robinson, Ralph Sidney (South African) Roe, David Edwin (London) Rose, Charles (Manchester) Rouse, Robert Earnshaw (South African) Rubin, Wallace (London) Rudram, Arthur Thomas Stephen (London) Saunders, Laurence Frederick (South African) Seymour, Norman Henry (Manchester) Slade, Harold Aitken (West Riding) Slinn, Thomas Walter (Wellington) Smethurst, Jack (Manchester) Smith, Francis Mark (Manchester) Sowerbutts, Frank (London) Stoyle, Francis Wilbert (Irish) Tatton, William Henry (Thames Valley) Tawn, Alec Richard Hornsey (London) Taylor, Cyril James Allan (Midlands) Taylor, John Roberts (Bristol) Taylor, Maurice David (Wellington) Thorpe, William Frederick Albert (Midlands) Tickle, Trevor Cyril Kenneth (Manchester) Tooth, John Henry Collins (London) Touchin, Herbert Roy (Manchester) Tozer, Edwin John (General Overseas-Argentina) Unsworth, Alfred Kenneth (London) Walker, Alan Gordon (Thames Valley) Wall, Dennis Charles (Manchester) Watkinson, Leonard James (West Riding) Westwood, George Ernest (London) Whiteley, Peter (London) Whitfield, Thomas (Auckland) Wilkinson, Thomas William (Hull) Willis, Gervase Hewitson (Manchester) Woodbridge, Richard John (Bristol)

## Optional Professional Grade Successful candidates—cont'd

#### Associates

Acey, John Arthur (London-Southern Branch) Adams, John Charles (Midlands) Adams, Terry Ernest (London) Addenbrooke, Brian John (Midlands) Aitken-Smith, Frank Joseph (Auckland) Allavena, Antonio (General Overseas-Switzerland) Anthony, Alan Sydney (London) Armstrong, Edward (Hull) Arnold, Frank (Manchester) Arnot, William James (Thames Vallev) Assink, Jo (Auckland) Baker, John (London) Baldwin, George William (Manchester) Bannington, Donald Bertram (London) Barnes, Peter James (London) Barton, James Francis (London) Batch, Alan James Edward (London) Beachen, John Frederick (Auckland) Beere, André Jaime (Thames Vallev) Bell, Brian Robert (Midlands) Bentley, Major Gordon (West Riding) Bird, George Donald Chaplyn (Midlands) Blackledge, Amos (Manchester) Bloomfield, Kenneth Vincent (London) Bluck, Ross Steele (Auckland) Bolam, Ion Barrow (Newcastle) Bowerman, David Francis (Manchester) Bowler, Kenneth Ernest (Midlands) Brockman, Andrew Leonard Sloane (General Overseas-Australia) Brooke, Leslie John (Bristol) Brown, Peter Thomas (London) Calder, Robert Malcolm (Auckland) Cartwright, Jeffrey (London) Catherall, Kenneth David (Midlands) Chebsey, Maurice (Manchester) Chellingsworth, Horace Thomas (Midlands) Chippington, Kenneth Alan (Bristol) Clark, Laurence Norman (London-Southern Branch) Clement, Donovan Harry (Midlands) Coates, John Allen (Manchester) Cole, Francis William (Midlands) Constantinides, Erricos (London) Cordwell, Terrence Allan (Midlands-Trent Valley Branch) Cunnington, Robin Roy Carol (London) Daggett, Wilfred Francis (London-Southern Branch) Dalton, Frank (Manchester) Dando, Vivian Charles Owen (London-Southern Branch) Davies, Frank Watkin (London) Davis, Reginald Albert (Bristol) Delorette, Gustav Otto Hans Jurgen (South African) Donkersley, Brian (Newcastle) Dowsing, George Frederick (London) Drew, Harold Henry Lennox (Midlands) Dunn, Paul Alan (London)

Eaton, Michael George (Thames Valley) Ebdon, James William (General Overseas-Rhodesia) Eltringham, James Norman (Auckland) Ernst, Joel (London) Evans, Carey Pearce (Auckland) Fairless, Joseph (London) Fell, Alan William (Manchester) Field, Lawrence Edward (South African) Finlay, Cecil Newton (Newcastle) Fisher, Leslie Alexander (South African) Flood, Geoffrey Terence (Manchester) Ford, Keith Sydney (Manchester) Formanek, Leopold (General Overseas—Czechoslovakia) Frazee, Jerry Daniel (General Overseas-USA) Garratt, Peter Garth (General Overseas-Switzerland) Gay, Alan Stanley (Midlands) Ghosh, Sunil Kumar (General Overseas-India) Gibson, John Carrington (Hull) Green, Brian James (London) Greenall, Brian John (Wellington) Greenfield, Eric (Midlands) Griffiths, Henry James (Midlands) Grime, David (London) Groom, John Robert (London) Gunn, Reginald F. E. (Thames Valley) Harrison, Cyril Geoffrey (Hull) Hasnip, John Anthony (Hull) Hayes, Gerard Francis (Thames Valley) Heald, Desmond (Manchester) Heffer, Victor George (Manchester) Herriott, Charles Edward (London) Hickman, Edwin Peter (Midlands) Hill, Gilbert Victor Geoffrey (Thames Valley) Hill, Raymond Forsyth (Scottish) Hodge, Robert Alexander Paul (Auckland) Holden, William Desmond (Manchester) Holmes, David James (Midlands-Trent Valley Branch) Holt, Clifford (West Riding) Homden, Kenneth James Arthur (London) Honiball, Alan Edward (Manchester) Hopper, Derek Edgar (Midlands) Hossack, James (Scottish) Howard, Eric (Manchester) Howells, Barry John (Hull) Howes, Edward John (London) Hughes, Gilbert William (Manchester) Humphrey, Thomas Lawson Myles (Scottish) Johnstone, James William (Manchester) Jones, Derek Frederick Arthur (London) Kelly, Peter Graeme (General Overseas-Australia) Kenna, Frank William (Manchester) Kerr, Michael Anthony (Manchester) Khan, John Mohammed (London) King, Charles William Henry (Midlands) King, Raymond John (Midlands) Kirlew, Charles Wesley (General Overseas-Jamaica)

Kitchen, John Robert (Midlands-Trent Valley Branch) Knight, Richard Charles (London) Laker, Bernard George (London) Lakshmanan, P. R. (General Overseas-USA) Lang, Robert (Scottish) Langley, Robert (Scottish) Lawton, Cyril Victor (Midlands) Lewis, John David (Thames Valley) Lipscombe, Charles George (Auckland) Low, Charles (South African) Lunt, Walter Richard (West Riding) Macdonald, Alan (Auckland) Macdonald, Arthur Gillings (Newcastle) Mandelson, Jack (Scottish) Maple, Donald Peter (London) Marsden, Chris Eyre (Manchester) Martin, Christian Pierre (General Overseas—France) Maynard, Albert William David (London) McDonald, Kenneth Roy (South African) McCapra, Ronald (Auckland) McKay, Alan Gordon (London) McKay, Robert Bruce (Scottish) McKean, James Newlands (General Overseas-Hong Kong) McMillan, James (Manchester) McQuirk, Peter John (London) Mepham, Brian Edwin (London) Mikucki, Wiktor (London) Miller, James (Scottish) Mitchell, Seward John (Midlands) Moore, Frank Roden (West Riding) Moore, James (Midlands) Moore, Ronald Henry (West Riding) Moore, William Alexander (Auckland) Morpeth, Frederick Johnson (Manchester) Morris, David (West Riding) Munro, Hugh Anderson (Scottish) Murray, Robert Frederick (London) Mynett, Raymond John (Midlands) Ness, Robert Alexander (Auckland) Norton, Douglas Kent (Midlands) Oakley, Ernest (Newcastle) O'Connor, Eugene Daniel (Manchester) Orpwood, John Leonard (London) Pace, Graham (Midlands) Parry, Martin Gerald (London) Pemberton, Joseph James (London) Perry, Leonard C. (Bristol) Pessall, Robert George (Midlands) Poborca, Stefan (Midlands) Proudley, Philip Miles (London) Quorn, Peter James (South African) Rackham, John Michael (Newcastle) Redman, Frank Benson (Manchester) Reeve, Frank Nicholson (South African) Robinson, Arthur Graham (London) Robinson, Francis Derrik (Hull) Rothwell, Gerald William (London) Rout, Peter George (West Riding) Routley, Alan Francis (London-Southern Branch) Scott, Neville (Manchester) Sharp, Peter Frank (Auckland)

# Basic products and auxiliaries for surface protection

Dynamit Nobel CHEMICALS

# provides coatings eminently suitable for stamping and deep-drawing.

High-molecular, linear, saturated polyesters containing terephthalic acid.

DYNAPOL L types are suitable for the production of coatings having high stamping, deep-drawing and weather-resistant properties and which, in addition to having good adhesive strength, are scratch and impact resistant, and neither yellow nor chalk. DYNAPOL L types are best used for coil-coating. Coating surfaces can have a finish ranging from high gloss to matt as desired. Pigmentations of all shades are possible.

# Main fields of use:

- Packaging.
- Packaging for foodstuffs (including sterilizable packaging).
- Fascia sections.
- Appliances

DYNAPOL L types are also suitable for single or double coat roller application on aluminium, steel or galvanized iron.

# Technical data:

Sheen (Gardner)	max. 95 — 100 % approx.
Pencil hardness	max. H
Impact test	180 in. lbs.
T bend	max. depending on type up to $T = O$
Salt spray test	depending on type 400-1000
	hours, unaffected
Kesternich test	Unaffected after 15-20 cycles

These details conform to the test standards of the

ECCA (European Coil-Coating Association, Brussels).



Wherever particularly long-life and outstanding weather resistance at both high and low temperatures are called for, PVF coatings are ideal. For they are corrosion proof, are resistant to chemicals and have excellent shaping properties.

PVF is, among other things, particularly suitable for coil-coating. Surface finishes may range from a silky gloss to matt as desired; they discourage dirt and they neither yellow nor chalk. Many shades are possible.

PVF should be used where the usual coatings afford insufficient protection against corrosion.

# Main fields of use:

- Building units where industrial pollution is severe,
- Facings for industrial plant.
- . Prefabricated building sections.

PVF is suitable for single or double coat roller application on aluminium, steel or galvanized iron.

# Technical data:

Sheen (Gardner) max. 80 % approx. Pencil hardness F --- H Impact test 180 in. lbs. T bend OT Unaffected after more than 1000 hours Salt spray test Unaffected after 30 cycles Kesternich test 0.136 Friction constant

These details conform to the test standards of the ECCA.





Saturated polyesters containing terephthalic acid for the production of powders both for electrostatic powdercoating and for whirl-sintering.

Powders with a DYNAPOL P base are suitable for coating all metals.

By virtue of their special properties (e. g. high resistance to weathering, yellowing and chalking) such coatings are equally suitable for external use.

# Main fields of use:

- Metal furniture,
- Metal window frames,
- Garden furniture,
- Apparatus,
- Metal facings,
- Tubing,
- Household appliances.

### Technical data:

Thickness of single coating  $50 - 70 \mu$ Erichsen test 8 - 10 mm approx. Pendulum test (depending on type) 175 - 185 sec.as per DIN 53157

- Indentation hardness 75 120 approx. (depending on type)
- Sheen as per Lange. Angle of incidence 45°
- 80 115 (depending on type)
- Adhesive strength: very good
- Kesternich test up to 30 cycles (depending on type)

# HARD PVC COATING POWDER

A plasticizer-free polyvinyl chloride compound in powder form for whirl-sintering and powder-coating.

# Advantages:

- Surface toughness
- Weather resistance
- Enduring gloss
- Chemical resistance
- No tendency to brittleness

#### Advantages when processing:

- Pleasant to work with: no offensive odours due to plasticizer vapours.
- High sheen surfaces within seconds,
- No post-treatment necessary.

#### Can be used:

- In the streets: Sign boards, posts for traffic signs, street lighting.
- In industry: Housings, mountings, sectional units, tubing,
- In the building world: Profiles, fittings, fascias,
- In the household: Appliances.

## Technical data:

Coating thickness depending on method of application 80 — 400  $\mu$ 

Erichsen test 8 — 11 mm approx. Pendulum test 170 — 185 sec. approx. Sheen as per Lange. Angle of incidence  $45^{\circ}$  120 — 140 %

Kesternich test. Unaffected after 40 cycles Adhesive strength: with bonding agent, very good



# For protection against corrosion even at high temperatures:

# DYNASIL<sup>®</sup> H 500

A binder having a silicic acid ester base for inorganic zinc dust coatings. When it is a question of protecting iron and steel from corrosion, even at temperatures up to 400° C, zinc dust paints having DYNASIL as a binder serve the purpose admirably.

Zinc dust paints with a DYNASIL base are particularly suitable for industrial processing. They can be welded over; they can be applied either by air or airless spraying, brush or roller, and can be used alone as a one-coat process or serve as a foundation for covering with any of the usual colouring methods.

# Main fields of use:

- Shipbuilding,
- Industrial plant construction,
- Power plant construction,
- Bridge building,
- Large steel structures,
- Mass produced building units.

Test results of DYNASIL based zinc dust coatings: Salt spray test (DIN 50021) Unaffected after 1000 hours. Adhesive strength, grating as per DIN 53151 Grating value 0.

Temperature loading resistance: Permanent load, maximum 400° C. Short term load, maximum 600° C





Dynamit Nobel Aktiengesellschaft Vertrieb Chemikalien M 5 5210 Troisdorf-Oberlai

10 Troisdorf	berstraße 2
D-52	На

Address
Department
Attention

Please send: Detailed information on

DYNAPOL®	L
PVF	
DYNAPOL®	Ρ

Hard PVC coating powder

DYNASIL® H 500



Please send: Detailed information on

DYNAPOL®	L
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PVF

DYNAPOL® P

Hard PVC coating powder

DYNASIL® H 500

# DYNAPOL L





# DYNAPOL P







DYNASIL H 500

samples of:

# Other tried and tested products

# for use in surface protection:

# Paint and varnish resins

## HYDROXYESTER RESINS

Thermo-setting, branched polyester resins containing hydroxyl groups for coil-coating and conventional painting techniques and having excellent stamping and deep-drawing properties.

## **ICDAL®**

Alkyd resins for making high-grade commercial and artists' paints, stove enamels and nitro cellulose combination lacquers.

# Electrically insulating paint resins ICDAL® TE/TI

Ester and esterimide resins, wire lacquer resins for producing insulated leads resistant to permanent temperature loads, resins for insulating impregnating lacquers.

## **Titanic acid esters**

Reactive cross-linking agents for paint and lacquer resins, binders for high temperature lacquers.

## Nitrocellulose

COLLODION COTTON PLASTICIZED NITROCELLULOSE

# Vanadyl alcoholates

Catalysts for polyurethane coatings with variable adjustment of setting times.

## Plasticizers

#### WITAMOL®

for use with various lacquers. Special plasticizer for heat-sealing and non-toxic aluminium coatings.

## Chlorinated paraffins

WITACLOR® For making lacquers flame resistant. For producing fascia paints.

# Chlorophenoles

#### WITOPHEN ®

Oil and dispersion paint additive for preventing fungus formation and decomposition during storage. For the production of fungus-resistant wall paints.

## **Chlorinated hydrocarbons**

DYNATRI® LQ, trichlorethylene. Solvents for paint and varnish resins for hot or cold dip coating processes. For the de-greasing of metals:

DYNAPER® perchloroethylene DYNAPER® perchloroethylene MECLORAN® 1.1.1. tri-ethane chloride MECLORAN® D 1.1.1. tri-ethane chloride (vapour type) For the de-greasing of light metals: DYNATRI® LM trichlorethylene.

#### Special hard aggregates TROCOR®

Electro-corundum as an aggregate for high-stress concrete pavings and for abrasive coatings having a synthetic resin base.

# Blast mediums not conducive to silicosis: REWAGIT®

Standard corundum, economical in use, low in dust, rustproof. For the cleaning or roughening of material surfaces.

DYNAMULLIT  $^{\textcircled{m}}$  — balls for the matting and densifying of material surfaces.

Please contact us if you have problems or require further information. We shall be pleased to give you the benefit of our experience.

Dynamit Nobel Aktiengesellschaft Vertrieb Chemikalien M5 D-5210 Troisdorf-Oberlar, Haberstraße 2, Telephone (02241) 180-1, Telex 889401

® = Registered trade mark



## Optional Professional Grade Successful candidates—cont'd

Sharpe, David (Hull) Shirt, John Michael (West Riding) Shoham, Joseph (General Overseas-Israel) Silsby, Denys John (Midlands) Silverwood, David (Manchester) Smith, Alexander Borland (Scottish) Smith, David Dorman (Scottish) Smith, Harry Bertram (London) Sowerby, Alan Hope (Auckland) Spargo, Robert (Auckland) Speding, George (London-Southern Branch) Springett, Robert Arthur Edward (London) Sreeves, John Ernest (Midlands) Stephenson, Robert Perry (Auckland) Stewart, Donald (Manchester) Stone, James Bryan (London) Stott, Raymond (Manchester) Stubbings, Alec Walter George (London) Suthers, Peter (Manchester) Sutton, Peter Michael (London) Talwalkar, Vinayak Sakharam (Manchester) Tasker, Leonard (General Overseas—Iran) Taylor, Terence (Manchester) Thomas, Anthony (General Overseas—Brazil) Thoms, Hugh Sydney (Manchester) Tillver Richard Brian (London) Topping, George David (London) Trevitt, Edwin William (London) Troparevsky, Alejandro (General Overseas—Argentina) Tye, Terence Thomas (Thames Valley) Unni, Madhavan T. N. (General Overseas-Malaysia) Venus, Norman (South African) Walker, Peter (Thames Valley) Weineck, Terrence Graham (South Africa) Westbrook, Ernest Louis Edward (London) Whalley, James (General Overseas—Kenya) Wheatley, Kenneth Valentyne (Irish) Whetstone, Peter John (London) White, Robert Arthur (Wellington) Williams, Adrian Arthur Owen (London) Williams, Cyril (Manchester) Wood, George (London) Wooll, Frederick James (London) Zissell, Martin John (London)

## Licentiates

Boxall, John (Thames Valley) Canterford, Barry Albert (London) Churchman, Anthony Edward (London) Cox, Garth Anthony (West Riding) Downham, Stephen Airey (Manchester) Hemmens, Anthony John (Bristol) Moss, Noel Sydney (London) Schierbaum, James Helmut (Midlands)

# **Report of Meeting of Council**

A Meeting of the Council was held at 2 p.m. on 17 October 1973 at the Great Northern Hotel, London, when 32 members were present. The President, Mr L. H. Silver took the Chair. The President welcomed Mr T. Entwistle, formerly Honorary Secretary of the South African Section and now attached to the Newcastle Section, who was representing Mr F. Schollick during the latter's absence on leave in South Africa. A report was made on the arrangements for the Annual General Meeting 1974, which it is proposed to hold in London on 26 June at the conclusion of a one day symposium on a techno-commercial subject. Full details of the symposium will be circulated to members in due course and will also appear in the *Journal* for the benefit of non-members. The Council appointed Mr A. R. H. Tawn (Honorary Research and Development Officer) to serve as the Association's representative on the Technical Education Committee of the Institute of Metal Finishing, and discussed the appointments of representatives to various other organisations.

The preliminary details for the Association's Dinner Dance on 31 May 1974 were approved and full details will be circulated to members well in advance of the function.

It was reported that the number attending the Reunion Dinner of past and present members of Council later in the day would be 61 and a report of this function appears elsewhere in this issue of the *Journal*.

The Council considered the half yearly accounts and estimates for the second half of the year which had been previously circulated with explanatory notes.

It was further reported that prior to the meeting of the Finance Committee on 2 October, a meeting had taken place of Section and Branch Honorary Treasurers which had proved most valuable. An encouraging sign reported by some Sections had been the surpluses derived from symposia organised either on their own or jointly with other organisations.

The Honorary Editor reported on the number of papers which had been submitted to him for publication in the Journal, in addition to those arising from the Conference which would be appearing in those issues published during the remainder of 1973. The Professional Grade Committee reported on viva voce examinations which had recently been completed and undertook to review the regulations in the light of the experience of the first two years.

Section reports were tabled by the representatives present and discussed. The Scottish Section were given permission for the Chairman of the Section to endorse certificates of study to be issued by the Reid-Kerr College for those students successfully completing a course entitled "Introduction to Paint Technology". The President reported on his recent visit to the Conference of the Scandinavian Paint and Varnish Technolicans (SLE) held at Sandefjord, Norway earlier in the month when he had been pleased to reply on behalf of the guests partly in Norwegian through the courtesy of one of his acquaintances! He stated that the official OCCA paper presented by Dr G. de W. Anderson had been very well received. The President reminded the Meeting that the Reunion Dinner would commence at 6.30 p.m. later on the same day and there being no further business he thanked members for their attendance and declared the meeting closed at 4.15 p.m.

# **Council Reunion Dinner**

A reunion dinner of past and present Members of Council was held at the Cafe Royal, London, on Wednesday 17 October. There were sixty-one present, including seven Past Presidents (of whom three are currently serving on Council) and twentythree past members of the Council.

As already announced in the Journal, the dinner this year was open to any Members of Council, past or present—the only guests on this occasion being the Past Presidents, two Past Honorary Officers and one Honorary Member. Twenty-four present Members of Council, the Director & Secretary and three senior staff members attended.

The dinner followed a Council meeting held earlier in the afternoon. The President, Mr L. H. Silver, gave the Address of Welcome. He presented a very interesting and informative viewpoint on the state of the paint manufacturing and associated industries, and also reviewed the last two eventful years in the Association's history, since the previous reunion dinner at Wax Chandlers' Hall in October 1971. During that time, the Association had held two more technical Exhibitions at Olympia and a conference at Eastbourne, where more technical papers had been presented than on any previous occasion. These events had been particularly successful; especially so considering that they had been organised at a time when the Association was moving offices, with the result that the Director & Secretary was left with none of the previous staff. It had been possible in addition to publish another Manual, entitled "Works Practice" and to accomplish a change of the Journal size to the international A4 format.

Dr H. W. Keenan (the senior Past President present) made a reply, thanking Mr Silver for his Address of Welcome and the Association for its hospitality.

After the two speeches the company broke into informal groups and enjoyed the opportunity to renew old friendships.

# **Midlands Section**

### Ladies' Evening: 25th Anniversary

The 25th Anniversary Ladies' Evening was held on Friday 21 September, 1973, at the Botanical Gardens, Edgbaston.

The Section's Chairman, Mr A. S. Gay, and his lady welcomed the members and guests, notably the President and Mrs Silver, Mr H. J. Clarke, Chairman of the Midlands Section of the Paintmakers Association, and Mrs Clarke; Mr D. Lovegrove, President of the Birmingham Paint, Varnish and Lacquer Club, and Mrs Lovegrove; the Chairmen of the Bristol, Hull, London, Manchester, Newcastle and Scottish Sections, and the Thames Valley and Trent Valley Branches, together with their partners; and Mr R. H. Hamblin, Director & Secretary.

This special occasion was marked by (in addition to the official guests) the presence of no less than seven past Chairmen. Particularly welcome was Mr R. Calvert, now resident in South Africa, who had fortunately been able to visit Britain at this time.

Following an excellent dinner, the President proposed the toast to the Midlands Section, as his first official function at a Section since becoming President.

After discussing briefly the problems of environmental pollution, and the energetic manner in which these were being dealt with by the paint industry, the President expressed the intention of visiting as many Sections as possible, not forgetting the overseas members.

Referring to the Midlands Section, he said that a Silver Anniversary was an important event. He paid tribute to the Section for the major part it had played in the Association's affairs over many years, mentioning by name several members who have served the Association in various capacities.

In conclusion, two presentations were made. The first a gavel, marked the anniversary of the Section, whilst the second, a baby's outfit, marked the recent achievement of the Chairman and his wife.

In response, Mr Gay paid tribute to his predecessors, many of whom were present. Continuing on the theme of "efficiency", mention was made of that of Council in choosing a President bearing the surname of Silver, presumably especially for this occasion, and of the efficiency of Mrs Gay, in producing a daughter on the date specified.

In proposing the toast to the ladies and guests, the Chairman expressed his appreciation of the honour of holding office for the 25th Anniversary.

A most fitting ladies' gift was provided—a silver bracelet bearing the Birmingham hallmark.

Mr F. Cooper responded to the toast for the ladies and guests. Laying claim to being the instigator of Ladies' Evenings for the Midlands Section, he reminisced over the various venues for the occasion, concluding with praise for the present arrangements. Ladies' Evening was obviously here to stay, and with an excellent record to date, the hope was expressed for an even better future. On behalf of the ladies, Mrs C. H. Morris expressed appreciation of the very appropriate gift.

The evening continued with dancing, a comedian using "Press Gang" tactics during the interval to form a wellequipped, if not highly talented, band! There was little doubt that all who attended the function had a truly memorable evening.

#### F.W.C.



The President, Mr L. H. Silver (right), the Section's Chairman, Mr A. S. Gay, and their ladies



Mr F. Sowerbutts, first Chairman of the Midlands Section (left), Mr A. S. Gay, the present Chairman, Mrs Sowerbutts and Mrs Gay

# Association Notices

#### Applications for membership

It is felt that the members would like to be reminded of the standard of competence for the election of candidates to Ordinary Membership of the Association, as laid down by the Council, when they are sponsoring candidates for election. The qualifications for the granting of Ordinary Membership at the present time are:

1. A degree in a scientific subject or any generally accepted equivalent qualification; or an approved technological qualification in a subject covered by the Association.

2. Or where there is adequate evidence of the technical competence of the candidate other than the obtaining of the qualifications mentioned above, the qualifying period of practice in the industries covered by the Association shall be normally not less than seven years.

Associate Membership is open to those employed in the industries who do not qualify for Ordinary Membership.

The Council has further resolved that Student Membership should be open without restriction to the age of 21 years and may be extended to 25 years of age, where candidates are following courses of technical study to the satisfaction of their employers or technical college lecturers.

#### **Retired members**

Council also wishes it to be known widely that in 1962 it introduced a reduced membership subscription rate for members who have retired from business. This applies to a member who has completed 20 years as an Ordinary or Associate Member and has retired from business, and normally has reached the age of 60, he may apply for his name to be retained on the Register of Members at an annual subscription rate of £1.50 and he will retain the same rights of membership as the class of membership to which he was attached upon retirement.

Members wishing to avail themselves of this concession should write, in confidence, to the Director & Secretary at the address shown on the front cover of this Journal, giving the relevant information under the four headings: (a) name, address and Section, (b) date of election, (c) date of retirement, (d) age.

#### Change of address

Members changing their address are urged to inform the Association's office immediately so as to avoid any misdirection of mail. This is particularly important as far as the *Journal* is concerned.

Will members please note that since membership of the Association is entirely on an individual basis, if notification of the change of an address for a company is sent to the Association's office this will not necessarily guarantee the change of address in the Association's records of the member concerned unless the name of the member is stated on the communication.

#### **Binding of Journal**

Members will be pleased to know that J. S. Wilson & Son Ltd., 14a Union Road, Cambridge CB2 11HE will undertake the binding of back volumes of the Association's Journal sent in by individual Members at a cost of £2.75 per volume.

Members wishing to avail themselves of this facility should send the parts, securely wrapped, direct to J. S. Wilson & Son Limited enclosing a remittance of  $\pounds 2.75$  per volume, and ensuring that notes bearing their names and addresses are enclosed with the parcels. It is particularly important that packets are sufficiently wrapped to negate the possibility of damage in the post.

#### 1974 Members' subscriptions

Members are reminded that 1974 Membership subscriptions to the Association are payable on 1 January 1974. Forms were despatched to all members in October or November, depending upon address.

The Commissioners of Inland Revenue have approved of the Association for the purpose of the Income and Corporation Taxes Act Section 192, so that a Member subject to United Kingdom income tax is entitled to a deduction from the amount of his emuluments assessable to income tax under Schedule E for the whole of the annual subscription to the Association, provided the subscription is defrayed out of the emoluments of his office or employment and that the interests covered by the objects of the Association are relevant to such office or employment.

#### 1974 library subscriptions

The Journal subscription rate to non-Members, including libraries, for 1974 will be £12.00 (\$1) post free by surface mail, home and abroad. Individual copies can be purchased for £1.25. Remittance should be sent with order to the Association's offices.

# Association Dinner Dance 1974

Early in the New Year members attached to the United Kingdom, Irish and General Overseas Sections will be sent the application form for the Association's Biennial Dinner and Dance to be held on Friday 31 May 1974 at the Savoy Hotel, London WC2R 0EU.

The Reception will take place at 7.00 pm for Dinner at 7.30 pm and, on this occasion, there will be two short speeches only—a Welcome by the President and a Reply on behalf of the guests—so that dancing, to the Jerome Orchestra, can commence as soon as possible and continue until 1 am.

The price of single tickets is £7.00 plus 70p VAT each and applications should be made by Members as soon as possible after receipt of the form. Tables will be arranged to accommodate 12 or 10 persons each, although it may be possible to arrange a few tables for eight persons. Non-members wishing to receive forms should contact the Association's offices.

# **Register of Members**

The following elections to membership have been approved by Council. The Section to which new members are attached is given in italics.

#### **Ordinary Members**

- BAECK, G. INGE, Teknikergatan 17, S.25100 Lulea, Sweden. (General Overseas)
- BENNETT, ROSS WILLIAM, MSC, A. C. Hatrick (NZ) Ltd, 66 Main Road, Tawa, Wellington, NZ. (General Overseas)
- BRYANT, CHARLES ANTHONY, BSc, 149A Chichester Road, Cleethorpes, Lincolnshire. (Hull)
- CLARK, MICHAEL DENIS THOMAS, 63 Allington Road, Southville, Bristol BS3 1PT. (Bristol)
- COTTIS, ANTHONY WRIGHT, BSC, 8 Yew Tree Gardens, Harrogate, Yorkshire HG2 9JV. (West Riding)
- CROTHERS, DEREK ANDREW, LRIC, 20 The Ruffetts, Croydon, Surrey CR2 7LQ. (West Riding)
- DENT, MICHAEL JOHN, BSC, Box 38-160 Petone, Wellington. (General Overseas)

DE MEYER, JAN ROBERT, Meiweg, 26 B.8500 Kortrijk, Belgium. (General Overseas)

- GRAY, STUART GEORGE, BSc, Fletcher Industries Ltd, Parry Street, Dunedin. (General Overseas)
- HASLER, DAVID JOHN, BSC, Cassio Photographic Paper Co, Cassio Bridge, Watford, Hertfordshire. (London)
- JALIL, ABDUL, BSc, Room No. E03, University Hall, Bradford University, Bradford 71 DP. (West Riding)
- MYGIND, KAI, MSc, Kemisk Vaerk Koge A/S, 4600 Køge, Denmark. (General Overseas)
- REINGER, JOSEPH R., BSc, Olin Corporation, Designed Products Division, 120 Long Ridge Road, Stamford, Conn. 06904, USA. (General Overseas)
- STEPHENS, MICHAEL WILLIAM, BSC, PT ICI Paints Indonesia, PO Box 2158, K. Kertosono 10, Jakarta, Indonesia. (General Overseas)
- WIMPFHEIMER, LEONARD, BSC, Merck & Co. Inc, Chemical Div, Rahway, NJ07065, USA. (General Overseas)

#### **Registered Student**

WILMOT, PAUL TREVOR, 11 Newland Grove, Newland Grove, Hull HUS 2NG. (Hull)

# Preliminary notice of the Annual General Meeting 1974 and one-day symposium

"OPTIMUM USE OF RESOURCES IN THE SURFACE COATINGS INDUSTRIES"

The Council wishes to announce as early as possible the preliminary arrangements which have been made for the holding of a one-day symposium in conjunction with the Paintmakers Association of Great Britain on the occasion of the Association's Annual General Meeting, 1974.

The Association's Annual General Meeting will take place at 5.30 pm on 26 June 1974 at University College London; a notice allowing for the nomination of the three Elective Members of Council will be sent to Members attached to the United Kingdom, Irish and General Overseas Sections early in the New Year and completed forms must be returned to the Association's offices not later than 1 May 1974. The notice will also give the first information concerning the one-day symposium which is being arranged at the same time on a techno-commercial subject, the general heading of which is "The Optimum Use of Resources in the Surface Coatings Industries". It is envisaged that the first session will take place from 10.00 am to 12.30 pm. Following a Reception, Luncheon will be taken at 1.00 pm and the second session will be arranged from 2.00 pm to 5.00 pm. Dinner will be taken at 7.00 pm following a Reception at 6.30 pm. It is stressed that it is not necessary for those who wish to attend only the Annual General Meeting to register for the symposium. The Agenda, Voting Paper and Annual Report will be circulated to Members in the usual way approximately six weeks before the date of the Annual General Meeting. Council has arranged the inclusive charge for the symposium, which will include both Luncheon and Dinner together with preprints, Conference badges etc., to be£20 plus£2 VAT and forms will be circulated in due course in a forthcoming issue of the *Journal*. Further information, including details of papers to be given will be published in the *Journal*. The Paintmakers Association will be sending application forms to their members. Non-members, who are welcome to attend the symposium, can obtain application forms from the Association's offices in the New Year.

# **Forthcoming Events**

Details are given of meetings in the United Kingdom up to the end of the month following publication, and South Africa and the Commonwealth up to the end of the second month.

#### Monday 3 December

Hull Section: "A Review of the current and future trends in industrial finishing", to be held at the Dorchester Hotel, Beverley Road, Hull at 7.00 p.m. Mr R. McD. Barrett, BIP Chemicals.

#### **Tuesday 4 December**

West Riding Section: "Flame Retardant Coatings" by Mr F. C. Adams, of Building Research Establishment, to be held at the Griffin Hotel, Leeds at 7,30 p.m.

#### Thursday 6 December

Newcastle Section: "Determination of the dispersibility of organic pigments in paints" by Mr Hafner, Hoechst Cassella Dyestuffs Ltd., to be held at the Royal Turks Head Hotel, Grey Street, Newcastle-upon-Tyne.

#### Friday 7 December

Irish Section: Ladies' Night to be held at Clarence Hotel, Dublin at 8.00 p.m. A lecture on "Old Dublin" by Mr F. Dixon.

Manchester Section: "Are we at risk with chemicals?" by Mr K. R. Butterworth, of the British Industrial Biological Research Organisation, to be held at the Manchester Literary and Philosophical Society, 36 George Street, Manchester 1 at 6.30 p.m.

#### **Tuesday 11 December**

London Section: Ladies' Invitation Lecture. "Driving the Police way" by Chief Inspector N. A. M. Ferguson of the Metropolitan Police, at Great Northern Hotel, King's Cross, London N19AN at 7.00 p.m.

#### **Thursday 13 December**

Hull Section: Joint Meeting with the Chemical Engineers—South Humberside Group at 6.30 p.m. at the Haven Inn, Barrow Haven, Lincolnshire. "The Humber Bridge Construction" by Mr J. A. Hyatt of Freeman Fox and Partners.

Scottish Section: "Review of plastics" by Prof. N. Grassie (University of Glasgow). Joint meeting with the British Paper and Board Manufacturers Association, Technical Section at 7.30 p.m.

#### Saturday 15 December

Scottish Section—Student Group: "Marketing techniques" by a lecturer to be advised, to be held at St. Enoch Hotel, Glasgow at 10.15 a.m.

#### Wednesday 19 December

Scottish—Eastern Branch: "Newer modified hydrocarbon resins for paint and printing inks" by Mr A. Laws and Mr K. B. Gilkes, Berger Chemicals Ltd., at Carlton Hotel, North Bridge, Edinburgh at 7.30 p.m.

#### January 1974

#### Thursday 3 January

Newcastle Section: "Masonry coatings" by G. W. Rothwell, Building Research Association.

#### Monday 7 January

Hull Section: "New developments in colour instrumentation" by Mr R. P. Best of Instrumental Colour Systems Ltd., at the Dorchester Hotel, Beverley Road, Hull at 7.00 p.m.

#### **Tuesday 8 January**

West Riding Section: "Epoxy resin curing agents" by Dr C. G. Tilley of Anchor Chemical Co. Ltd. Griffin Hotel, Leeds at 7.30 p.m.

#### Thursday 10 January

Midlands—Trent Valley Branch: "Pollution" by Mr J. B. Davies, Goodlass Wall & Co. Ltd., Speke, Liverpool, to be held at the British Rail School of Transport, London Road, Derby at 6.30 p.m.

#### Friday 11 January

Manchester Section: "Colour difference, the measure of the future" by Mr K. McLaren, ICI Ltd., Organics Division, at the Manchester Literary and Philosophical Society, 36 George Street, Manchester 1 at 6.30 p.m.

Scottish Section: Annual Dinner Dance in the Albany Hotel, Glasgow.

#### Wednesday 16 January

Scottish—Eastern Branch: "Marketing in Europe", speaker to be announced. Carlton Hotel, North Bridge, Edinburgh at 7.30 p.m.

#### Thursday 17 January

Scottish Section: "A trade journalist speaks his mind" by Mr D. E. Eddowes (Polymers Paint and Colour Journal). St Enochs Hotel, Glasgow at 6.00 p.m.

#### Friday 18 January

Irish Section-to be announced.

Midlands Section: Annual Dinner Lecture: "Who needs paint anyway?" by L. H. Silver (SPL Group) in the Birmingham Chamber of Commerce and Industry, PO Box 30, 75 Harborne Road, Birmingham B15 3DH at 6.30 p.m.

#### Saturday 19 January

Scottish—Student Group: Works visit to Continex International-carbon black manufacturers. This will be a joint meeting with the Eastern Branch owing to the intermediate position of Continex International between Glasgow and Edinburgh.

#### Monday 21 January

London Section: "Modern marine coatings and their application" by Mr J. C. Smith of the Central Dockyard Laboratory at East Ham College of Technology, High Street South, London, E6.

#### Thursday 24 January

Thames Valley Section: Paint Research Association's "Inventions" by Mr R. A. Brett. Beech Tree Hotel, Beaconsfield, Bucks at 7.30 p.m.

#### Friday 25 January

Bristol Section: "Printing Ink Developments" by Mr G. H. Hutchinson of Croda Polymers Ltd., Ink Division, to be held at the Royal Hotel, Bristol at 7.15 p.m.

#### Wednesday 30 January

Manchester—Student Lecture: "Organic pigments in paints—automotive finishes" by a Lecturer from CIBA-GEIGY (UK) Ltd., Pigments Division at the Manchester Literary and Philosophical Society at 4.30 p.m.

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