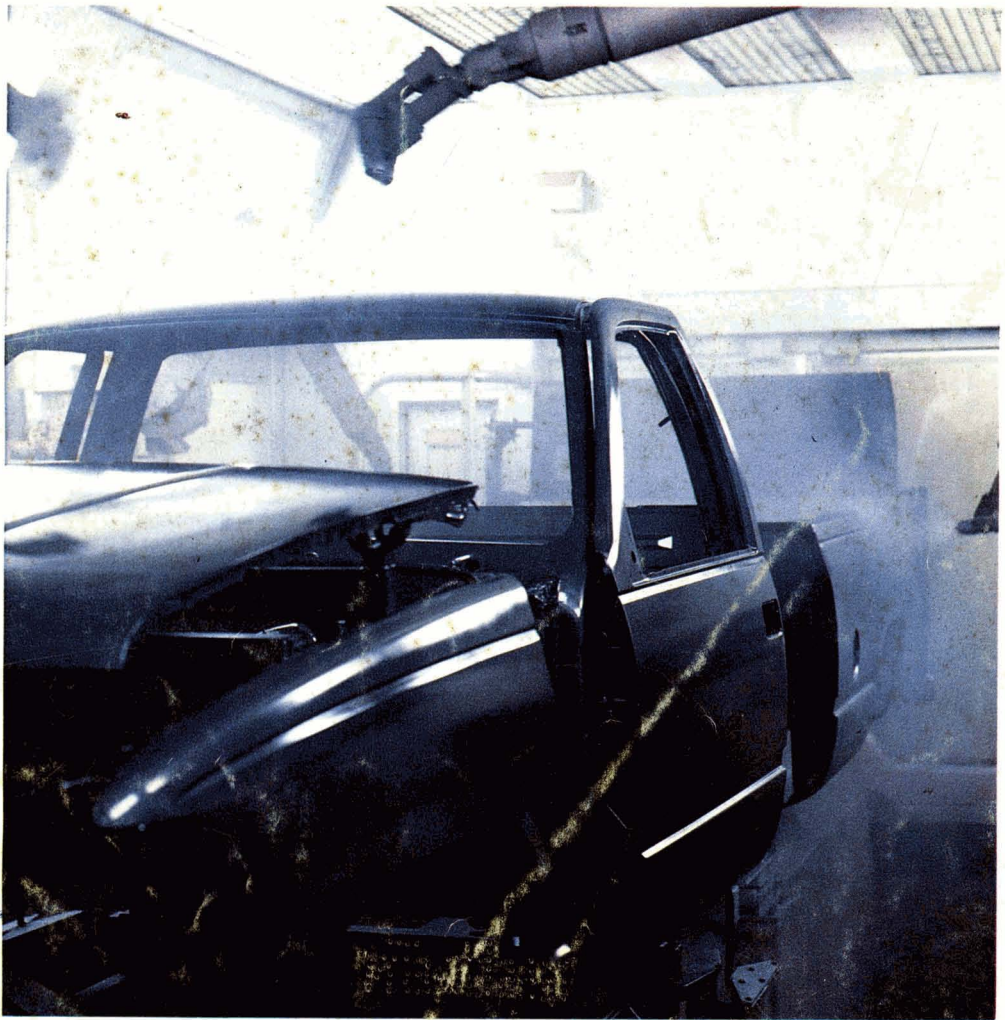




JOURNAL OF THE OIL AND COLOUR CHEMISTS' ASSOCIATION

# JOCCA



■ Automotive/Industrial Coatings

# 1992

1991  
1990

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Tracey Walker

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1992

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JOURNAL OF THE OIL AND COLOUR CHEMISTS' ASSOCIATION

# JOCCA

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Cover: Automotive finishing (Photo by courtesy of ICI Paints) .

Forthcoming Features: May – Fire Retardant Coatings, June – Paint and the Environment, July – Printing Inks. Contributions are welcomed at least five weeks prior to publication date.

**Oil and Colour Chemists' Association**

Priority House, 967 Harrow Road, Wembley, Middlesex HA0 2SF England

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## Chester Conference Column

The response so far in terms of registration for the June event has been very encouraging indeed and it looks at the moment as if we will make the target of 200 delegates.

Much to my surprise the “early” delegates seem to have plumped for the more expensive Chester Grosvenor and this means that there are only a very few rooms still available to us there.

I have just had the opportunity to have a conducted tour of the as yet unopened Chester International Hotel. I have felt duty bound to have personal knowledge of the hotels offered to delegates. There is no doubt that it will be excellent and with confidence I can honestly recommend anyone to opt for it. Indeed it is a pity that it was not built a few years earlier as, if it had been, I would certainly have sited the conference there since, apart from adequate space, it has the most up-to-date sound and visual set-up. The fact that it is only 3 minutes’ walk from the Chester Grosvenor completes the attraction for potential delegates.

Simon Lawrence is in the process of getting the papers into the form for circulation and has the bonus of an extra paper not included in the brochure. Details on the technical side follow this Column.

One of our latest *JOCCA* readers is the Lord Bishop of Chester and so we may well have a cathedral coating appraisal in his proposal of the toast to OCCA on the Friday night! As far as the social programme is concerned it looks as if readers will have to move quickly if they are to get a place on the Lady Diana Thursday evening trip. For those who are unlucky by virtue of being too late with their booking, I will provide a list of alternative ways of enjoying the evening in Chester (tastes will vary I am sure!!).

Way back in 1986 when I started “putting the conference together”, I had visions of an OCCA conference in the style of the fifties/sixties in terms of many delegates and many partners. The social programme was aimed at attracting more of the latter than we have seen at conferences over the last few years. It seems as if my efforts have not been in vain as exactly 50% of the delegates are bringing “their other halves”. I hope this trend keeps up. My Cestrian (the adjective from “Chester”) guide will contain much information supplied by Dorothy in terms of interest to the ladies, whilst my contribution will tend to be to the satisfaction of the males (particularly the “inner man”!).

Finally, I would like to emphasise one point concerning the big “shindig” on the Friday night. It has already been suggested that companies may wish to take complete tables of ten places each. I have fixed a special price for their so-doing—£235. It must be noted that this price pertains ONLY when the complete table is booked. Many companies have already availed themselves of this facility and I hope more will and that no-one will have to be refused due to a “full-house”.

A. C. Jolly  
Hon. Conference Officer ■

## Chester Conference-Technical Column

*“No Manne is an Islande”*  
(John Donne 1573-1631)

Donne certainly wasn’t thinking about the OCCA Chester Conference when he wrote this nor specifically about the impact of scientific developments on our fellow man or the environment.

But in a nutshell this encompasses the idea that no company, no employee can these days work in isolation. They must have an awareness of the likely influence of their work, their products on their surrounds. And in turn their work is influenced by the pressures of society, by legislation, by toxicology concerns, by market needs etc.

Nowhere are these outside pressures more acutely felt than during the research, the design and development of new products. It is within this general theme that OCCA has organised its biennial conference under the title “Profitable Research & Development”.

Eighteen papers have been accepted for presentation during the four technical sessions and these together with the keynote address will seek to elucidate this theme and related topics.

The speakers come from a wide variety of backgrounds—and countries—and they will be discussing philosophy and organisation of companies research departments, the role of independent research organisations, the influence of health and safety legislation, together with specific case histories on the introduction of new products into the market place as well as presenting some more basic research papers aimed at improving our understanding of surface coatings which is so important to the development of new products.

I believe we have been able to get together a set of papers aimed at stimulating “the little grey cells” and have set up a framework which will allow for a good exchange of ideas and concepts.

Naturally not all the discussions are going to take place during the technical sessions. Tony Jolly, the Hon Conference Officer has arranged a splendid social programme where in somewhat more relaxed surrounds the discussions can be continued.

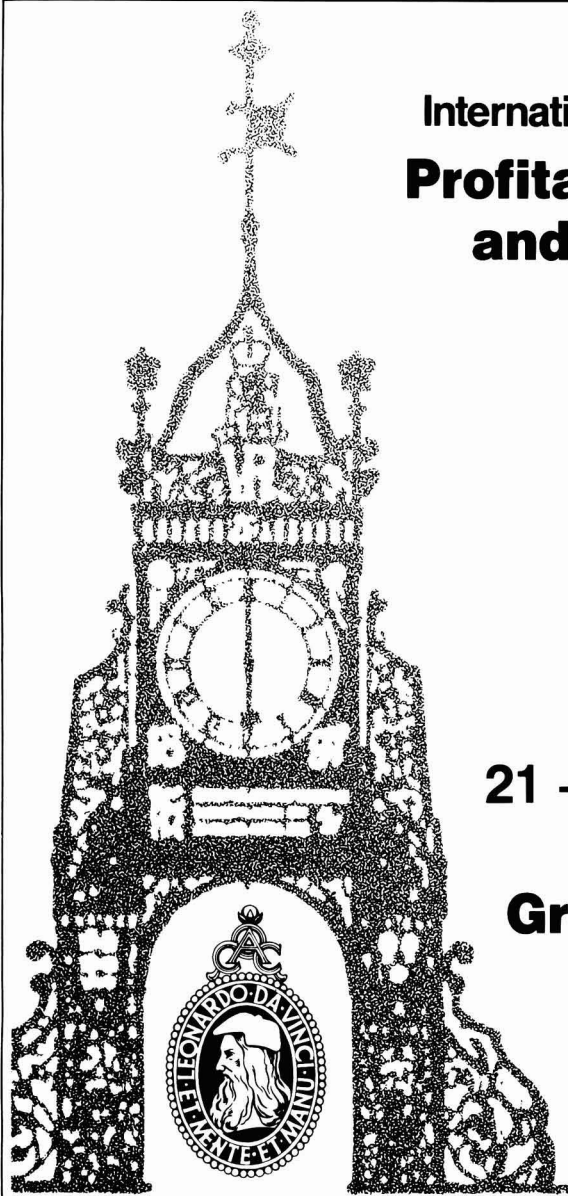
I would ask you all to support the conference. The success of the SURFEX Exhibitions has shown that the

# Oil & Colour Chemists' Association

International Conference on  
**Profitable Research  
and Development**

21 – 24 June 1989

**Chester  
Grosvenor Hotel**



Contact OCCA on 01-908 1086 for Registration Form

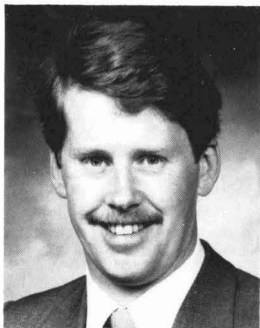
surface coatings industry is progressive and forward looking and needs now a forum for the exchange of technical ideas—the conference.

I look forward to meeting you all in Chester.

S. G. Lawrence

Hon. Research and Development Officer ■

## Keynote Speaker



D. J. Pirret

The Keynote Address at Chester will be presented by **David Pirret**, General Manager of the Additives and Resins Divisions for Shell Chemicals UK Ltd.

Mr Pirret has a BA (Hons) in Marketing from Strathclyde University and in 1974 was a Stewart & Mitchell Prize Winner.

Mr Pirret joined Shell Chemicals UK Ltd in 1975 and has since then held a variety of senior positions for Shell

Chemicals: Development Manager, Elastomers and Urethanes, Shell Chemicals UK Ltd (1978-80); Product Manager, Antifreeze, Shell International Chemicals Co Ltd (1980-81); Product Manager, Ethylene Oxide and Glycols, Shell International Chemical Co Ltd (1981-82); General Manager, Shell Cyprus Trading Co Ltd (1982-85); Planning Manager, Elastomers and Feedstocks, Shell International Chemical Co Ltd (1985-87); General Manager, Resins and Materials Division, Shell Chemicals UK Ltd (1987-88).

## Mechanical properties of crosslinked coating systems (presented on behalf of FATIPEC)

M. Oosterbroek and O. T. de Vries

### Summary

The utility of characterizing the mechanical properties of free films for the development of new coatings will be discussed. Examples of the relation between tensile measurements, dynamic mechanical measurements and impact resistance will be presented.

Mechanical characterization of free

films are not intended to replace traditional tests of coatings on a substrate, but they can be used in a supplementary way to provide insight into fundamental properties. Moreover, these tests open the opportunity for an interpretation based on fracture mechanics and stress distribution calculations.

### Biographies

Dr. ir. M. Oosterbroek received his Applied Physics degree in 1976 at the University of Twente.

He joined the university staff to do research on the rheology of emulsions and obtained his PhD in 1980.

Since 1980 Dr Oosterbroek has worked at Akzo Corporate Research, Holland. He is presently responsible for the basic research into the mechanical and optical properties, and the durability of coatings.

Ir. O. T. de Vries received his university degree in Technical Physics in Delft in 1961. A member of Akzo since 1963 he presently holds responsibility for the research and development of industrial coatings at the Akzo Coatings laboratory of Sikkens, Holland. ■

Continued from p.157

where

$D_1$  = diffusion coefficient for  $CO_2$  in air ( $m^2/s$ )

$c$  = the amount of concentration difference of  $CO_2$  in the air ( $kg./m^3$ )

$J$  = the measured material current density of  $CO_2$  ( $kg./m^2.s$ )

$s$  = thickness of the permeated coat (m)

Step 2: The diffusion resistance of the whole system (including support paper) is reviewed and the individual components diffusion calculated and split, this gives the  $\mu_{CO_2}$  figure for the coating.

Step 3: The diffusion resistance is then calculated in terms of equivalent air thickness layer  $S_D$  from

$$S_D = \mu \cdot s$$

where

$\mu$  = coefficient

$s$  = coating thickness in Metres

Step 4: The  $S_D$  figure for the coating is compared to that of air and must be at least 50 metres equivalent.

The  $CO_2$  diffusion resistance (Taywood) Table 2 is calculated on a similar basis and involves the use of Ficks Law of Diffusion. The calculation

once again allows for removal of the diffusion coefficient for the (ceramic) support plate where a definite value exists. Due to the different methodology with similar calculations, the result appears different but of the same order of magnitude and still greater than the  $S_D$  figure for air ( $\geq 50$  Metres). This method expresses the air equivalent thickness as  $R$  and typically involves the following criteria:

$D_{CO_2}$  = diffusion resistance coefficient for carbon dioxide

$\mu$  = diffusion resistance coefficient for the coating

$D_{CO_2}$  = diffusion resistance coefficient for the ceramic plate

$S_c$  = equivalent thickness of concrete (to the coating)

$S$  = dry film thickness, D.F.T.

Note:  $\mu$  coating x D.F.T. (cms.)

$$S_c = \frac{\mu \text{ (standard concrete)=400}}{\mu \text{ (standard concrete)=400}}$$

From these two comparisons it can be seen that a standardised method is needed to enable meaningful comparisons and it is to be hoped that a British Standard may be forthcoming, which would include a support medium for the coating under test as near to

'concrete' as possible.

## 8 Conclusion

A protective function is possible in conjunction with an aesthetically pleasing coating. It is necessary to select a coating with properties that work in tandem, it is not sufficient to resist decay mechanisms only. A combination of gaseous and liquid resistance, retained elastomeric and coherence properties, long term world-wide weathering resistance, and breathing properties; must be coupled with ease of application and high build character. To this is added definable test parameters and a proven history of use.

## References

1. Kreijger, P. C., "The Skin of Concrete, Composition and Properties", *Materialux and Constructions*, 1984, 17 (100), 275.
2. Technical Note 130, "Protection of reinforced concrete by surface treatments". ISBN 0 86017 280 5, ISSN 0305 1718. © CIRCA 1987. ■

This paper was presented to West Riding Section of OCCA on the 15 November 1988

## JOCCA—One Year On

This issue of the Journal marks the first anniversary following its redesign and relaunch in the spring of 1988. Readers will have noticed that the Journal has been continually developed during these twelve months, in response to editorial and reader feedback, and has now settled down in a format that will be held for some time.

The introduction of editorial feature issues has been one of the most significant changes and it is believed from general reports received that this policy is acceptable to our readers. The policy of obtaining papers which will be of interest to all our readers is always under review and the publication of papers which contribute to the basic scientific and technological knowledge of subjects of interest to the surface coating industries and of which the Association is well respected will continue to be published in the "Transactions and Communications" section of the Journal. In addition, future issues will include a new section called Horizons for papers which may be of immediate technical interest to the industries, but not directly associated with the feature subject matter.

The new Horizons section will include papers presented at OCCA Section meetings, especially papers from overseas Sections' conferences and symposia, and it is believed that this new policy will ensure closer collaboration within the Association and also will be of interest to our readers world-wide. The Section will also include papers submitted from overseas.

It will be noticed that the basic format for technical papers has reverted to the original two column format to give easier reading and clearer settings for equations and tables. This change was brought about after consultation with members of the Publications Committee and Section Committees, resulting in an almost unanimous recommendation to revert to the two column format.

The technical feature chosen for this month's issue of the Journal is "Automotive and Industrial Coatings" an application which often requires much detailed technical knowledge to pass the extremely severe requirements of meeting the customers' specifications.

This is especially true for example in the formulation of automotive and domestic appliance coatings. It is, therefore, of interest to note that a paper on a new water based system (Aquabase) for use in automotive finishing has won the Queen's Award and ICI and its technical staff must be congratulated for their pioneering research and development.

JOCCA, which is read in over 80 countries world-wide, is often the only means by which members may keep in touch with the Association. The Honorary Editor will always be pleased to receive news items, opinions and technical papers from members world-wide. Members can be assured that the Association's editorial staff are continuously discussing methods of improving the quality of the Journal and its value to all its readers. The Association welcomes any comments on the new style Journal and ideas for its future development.

J. R. Taylor ■

## CORROSION PREDICTION AND PREVENTION IN MOTOR VEHICLES

by Hugh McArthur, Faculty of Technology and Construction, Leicester Polytechnic. 438pp, Publ. 1988, Price £59.00/\$115.50 incl. p&p.

The author takes a multi-disciplinary approach to corrosion and materials science of motor vehicles. He explains motor vehicle manufacturing processes, considering the different materials used and their degradation, and gives a simple explanation of the potentials of metals.

Descriptions of materials e.g. paints, plastics, waxes, adhesives and glasses which play a vital role in motor vehicle manufacture are combined with numerous illustrations to show the advances in their technology.

Readership: Technologists with an interest in motor vehicles, their construction materials and degradation. Corrosion science, materials technology, metallurgical science, mechanical and production engineering, applied physics, chemistry, motor vehicle manufacturing, paints, plastics and adhesives and other areas where the lessons of motor vehicle degradation should be applied, e.g. construction and marine engineering.

To: Oil and Colour Chemists Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF, UK.

Please send me.....copy/ies of **Corrosion Prediction and Prevention in Motor Vehicles** at £59.00/\$115.50 (incl. p&p) per copy.

I enclose a cheque for £.....made payable to OCCA.

Name.....

Address.....

.....

..... Telephone .....

## European Colour—The best of both worlds

European Colour plc formed following the merger of Ellis Jones of Stockport and Horace Cory of Plumstead report progress on their organisational merger. Areas where experience and facilities are being combined include: Development, Process Technology, Technical Service, Stock Control, Order Processing, Computerisation and Quality Assurance.

Both sites manufacture similar types of pigment, i.e. azos. An exercise is in hand to obtain information in fine detail on similar products being made at the two sites. It is expected that the majority of the products will continue to be manufactured but that, where one site's version is virtually identical with that of the other site or where one is clearly superior to the other, then, after discussion with the customers involved, the total manufacture will be of one of the identical products or of the superior product.

## Henkel NOPCO formed

The UK business activities of NOPCO, formerly part of Diamond Shamrock Process Chemicals Inc, have been integrated into Henkel Chemicals Ltd. The NOPCO business, with a predicted turnover of some £7m, will form a separate entity within Henkel Chemicals Ltd. Henkel NOPCO, the company's new name, will manufacture and market processing chemicals within the UK and Ireland for the paint, paper, textile and allied industries.

## Sigma Coatings new Amsterdam factory

Sigma Coatings BV, the Dutch sister company of Buckingham based Sigma Coatings Ltd (part of the Petrofina Group), is to build a new hi-tech factory costing £14m in the harbour area of Amsterdam where its main Dutch production facilities will be concentrated. Construction of the new factory will start in September 1989 and is due to be completed by the end of 1990.

## Becker Industrial licences China Paints



John Lyon (centre), Sales Director of Becker Industrial Coatings, with China Paints Executive Director Terry Tsui (left) and Paul Lam, Managing Director.

Goodlass Container Coatings of Liverpool, a division of Becker Industrial Coatings, is to have its container paints manufactured under licence in Hong Kong by China Paints, the colony's biggest paint producer. The complete range of Goodlass freight container coatings will be manufactured and sold by China Paints to outlets in Hong Kong, Taiwan, The Peoples Republic of China and Singapore. This latest licence complements Becker's recent agreements with Sam-Ho Chemicals Ltd in Korea and Techno Quimica in Brazil.

## Croda industrial paint expansion

Croda has agreed to acquire Lusteroid Paints based in Auckland, New Zealand, for £4m. This business complements the successful industrial paint operations Croda has in Australia and the UK. In New Zealand Croda has a printing ink and chemical business based in Auckland.

Croda has also acquired a small industrial paints company, Industrial Paints & Chemicals Ltd, based in Halesowen, Birmingham. This business will be integrated into Croda's existing UK industrial paints business.

## ICI buys 22 paint stores in N. America

ICI Paints are to buy 22 stores from the Texas-based Roach Paint Company. The move is in line with a Glidden strategy of expanding its company-owned distribution nationwide. The Roach stores bring the total of Glidden owned outlets in the United States to 360 with another 30 in Canada and 22 in Puerto Rico.

## Products

### New proofing inks

BASF Coatings + Inks has introduced a series of sheet-fed proofing inks which eliminates the reproduction discrepancy between the original proof and the printed result. The new series, marketed under the Fishburn label, is press ready and offers controlled dot gain plus extended press stability.

*For further information Enter D101*

### Blocked isocyanates

Baxenden has introduced a wide range of blocked isocyanates for various coating applications. Baxenden has also recently developed a room temperature curing blocked polyurethane, which does not cure by the normal urethane route. These blocked products are designed for use in two component systems for such applications as the flexibilisation of epoxy resins.

*For further information Enter D102*



## Novel rust converter

ICI Speciality Chemicals has introduced a novel chelating agent called 'Atrust' that provides the basis for a greatly improved rust converter with significant potential for industrial maintenance and DIY. Trial formulations have shown them to have substantially superior performance and shelf life compared with conventional rust conversion products.

For further information Enter D103

## Croda boosts solvent recovery

Croda Hydrocarbons has announced a further extension of its solvent recovery/toll distillation operations for used solvents or solvent mixtures often considered as industrial wastes following contamination during production processes. Croda can supply a range of recovered solvents, to tight specifications, for use by industry including hydrocarbons, alcohols, halocarbons, ketones and heterocyclics. Croda can also now supply off-site distillation services at Knottingley, W. Yorks. to suit particular processes.

For further information Enter D104

## New waxes

M. Langer & Co has introduced two new Lanco waxes: VP 460 is a superfine micronised PTFE coated polyethylene wax designed for aqueous coating systems. VP 453 is a micronised polyethylene wax which, when high speed stirred into water soluble or water reducible lacquer systems, will impart a high degree of slip and rub resistance.

For further information Enter D105

## Drew foam control agents

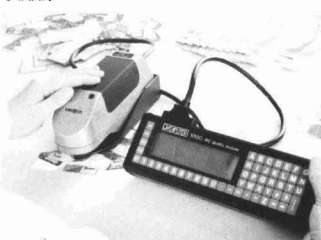
Drew Ameroid UK Ltd has introduced the Drewplus series of foam control agents for the production of water-based paints, lacquers and inks. They are effective in the production of PVA, PVC, SBR and other latices, and are application specific.

For further information Enter D106

## Equipment

### New SPC colour analysis package

Minolta has introduced for the first time a complete package for the Statistical Process Control of colour. Working in conjunction with Sellars Datasystems of Manchester Minolta has developed a means of carrying out SPC Analysis on a wide variety of materials using Minolta Chroma Meters and the Sellars Dataputer 3000.



Minolta simplifying colour SPC

For further information Enter D107

### PRA viscosity services

In response to many requests the Paint Research Association is now offering a new service—the examination of flow cups for compliance with the ASTM D1200 (Ford Cup), BS 3900:A6 (1971) ('B' cups) and BS 3900:A6 (1983—ISO 2431:1980) specifications. All enquiries to Alan Sherwood or Tessa Winter on 01-977-4427.

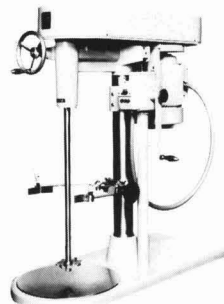
### Viscosity near MP

ChemLab Scientific Products has available a new system for measuring viscosity during the process of solidification. The system is called the Coesfeld Thermorheography System and it is being used for the study of fats and oils. The instrument consists of the standard Rheosyst 5000 rotational viscometer with automatic range switching, which is linked to a calorimeter to control the temperature of the sample very accurately. The temperature of the

calorimeter is controlled by a precision thermostat and precision cryostats.

For further information Enter D108

### New high speed mixer



A new Dispermix high speed mixer specifically for dissolving/dispersing pilot production batches is available from OBS Machines Ltd. Completing the range of Dispermix units from laboratory size to full production models the new DL-Ultra efficiently mixes batches up to 25 litres.

The Dispermix DL-Ultra is equipped with a 5 hp motor driving a high tensile stainless steel mixing shaft with 100 mm diameter turbine at speeds between 1,700 and 4,200 rpm. A telescopic column for raising and lowering the mixing head gives a maximum clearance between the impeller and the solid cast base of 560 mm.

For further information Enter D109

### Industrial scales

Ohaus has introduced a new range of industrial scales. There are 13 different platform models in the Ohaus IS Series, all are portable and are battery powered using the same electronic indicator with a built-in RS232 data interface. Capacities range from 5.5 to 240 kg, each with a stainless steel platform. Six models are fully protected for washdown applications, and all are easily linked to the wall, bench or tower mounted indicator.

For further information Enter D110

## Literature

### SBPIM launches new brochure

The Society of British Printing Ink Manufacturers, aiming to make customers and non-member ink makers alike more aware of its objects and achievements, has recently revised its brochure. The importance of the Society's logo as a symbol of member companies' commitment to technical excellence, after sales service and the Society's recommendations on matters of health and safety is particularly highlighted and amongst its other contents the brochure mentions the Society's initiatives in producing a Quality Assessment Schedule to BS 5750, organising the production of a distance learning scheme in printing ink technology, and the writing and editing of a new edition of The Printing Ink Manual last year. Copies of the brochure are available free of charge from the Director, Society of British Printing Ink Manufacturers, Pira House, Randalls Road, Leatherhead, Surrey, KT22 7RU.

### Miscellaneous

**Eastman Chemical Products Inc** have published a guide to their range of chemical, fibre and plastic products, showing typical properties for each.

*For further information Enter D111*

An 8pp leaflet from **Eckhart-Werke** on their NF-Metal powders and pastes for the paint and surface coating industries, printing and plastics.

*For further information Enter D112*

Speciality Resins, solvents and intermediates are featured in a brochure from **Du Pont** which also offers technical advice, safety programmes and quality control amongst other services.

*For further information Enter D113*

A technical publication from **GAF Chemicals** outlines the physical properties, forms and uses of their solvents, intermediates,

polymer/collids, additives and surfactants/emulsifiers.

*For further information Enter D114*

## Meetings

### Colour

The British Colour Makers' Association will hold a 1 day seminar on Quality on 20 April 1989 at the Crest Hotel, Coventry. For further information call 0737 353253.

### Colourgen colour conference

Colourgen Ltd, the Warrington-based UK arm of colour-matching specialist Colourgen Inc, are holding their second Colour Conference on 26 April 1989 at the Midland Hotel (now the Holiday Inn Crowne Plaza), Manchester. The one day conference starts at 9 am. Admission is by invitation. For further information call 0925 822577.

### Brazilian international paint congress

The 1st International Paint Congress will be held on 2-5 May 1989 at Sao Paulo, Brazil. For further information contact Guazzelli Associados Feiras e Promoções Ltda, Rua Manoel da Nóbrega, 864; 04001-São Paul-SP-Brazil. Tel: (011) 885.3635.

### BPVLC 60th anniversary

The 'Club' starts its celebrations with a 'Jubilee Dinner' on Thursday, 8 June 1989 at the Botanical Gardens, Edgbaston, Birmingham. The special guest will be Mr Jim Geiger, the President of the FSCT, of which the 'Club' is a member. Jim and his wife Lynn will be visiting from the USA with other American business colleagues. The main after dinner speaker will be Mr David Gunson. To book please contact Michael Wright of Holden Surface Coatings Ltd, 021-766-6600, the price will be £15.00 per head.

## People

### New FATIPEC President



**Annik Chauvel** has been elected President of FATIPEC for the next two years 1989-1990. Ms Chauvel is a graduate of Science and Arts. In 1965 she trained at the French CNRS-Laboratory for Macro-Molecular Chemistry and joined the Bourigeaud Company (a manufacturer of additives) which she now heads as President and General Manager.

Ms Chauvel is a founder of EREC Co-Consultants in Colours, Colorimetry and Paints where she is currently Managing Director, and is simultaneously Director and Chief Editor of the leading French Paint magazine "Double Liaison-Chimie des Peintures".

A member of the French Association of Technicians in the field of Paints, Varnishes, Printing Inks, Glues and Adhesives—AFTPV—since 1965, Ms Chauvel was appointed in 1976 to the National Board of this Association.

Ms Chauvel played an important part in the foundation of ICCATCI—the International Committee to Coordinate Activities of Technical Groups in the Coatings Industry—1979 in Paris, and has since 1986 been a member of the FATIPEC Board.

### Manders senior appointments

**Barrie Everitt** becomes managing director of Mander-Domolac Ltd, the industrial coatings division, based at Ruabon and Wrexham, and **Jack Singh** takes over Mr Everitt's former position as financial director of Mander-Kidd (UK) Ltd, the group's fast-growing ink company.

# Convincing.



## Cross-linking for powder coatings.

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# Phosphate pretreatment for coatings

by M. J. Dyett, ICI Paints, Wexham Road, Slough, Berks SL2 5DS, UK

## Abstract

The paper outlines the recent development of zinc phosphate technology aimed especially at the automotive and automotive subcontractor industries. It describes the gradual change from zinc phosphates with a Hopeite structure to those with a Phosphophyllite structure and with the introduction of "Trication" phosphates to treat mixed metal substrates based on zinc, zinc/iron and zinc-aluminium.

## Background

Since first invented by Thomas Coslett at the turn of the century (patent dated 1906) phosphating has come a long way as a means of protecting ferrous metals.

There were several early developments with this process but up until the early 1930's phosphating was aimed mainly at enhancing rust proofing rather than as a means of enhancing paint bonding. This was largely because of the long immersion times required — up to an hour to produce a reasonable coating.

However, with the introduction of certain accelerating agents reaction times were reduced to a few minutes and further refinements led to the development of spray zinc phosphating by the middle 1930's.

Iron phosphates or more correctly "alkali metal phosphates" first appeared just prior to the 2nd World War and while of a thin amorphous nature i.e. not heavy and crystalline as hitherto processes they have yielded coatings with excellent paint bond characteristics.

The following discussion will concentrate on the more widely used zinc phosphate processes and outline recent developments which have been required to:

- a) meet the requirements of improved paint technologies and
- b) meet the requirements of sophisticated metal substrates which have appeared recently.

## Introduction

The two basic reasons for phosphating metals are:

- a) To improve paint adhesion by providing a surface which has many anchor points
  - b) To provide a resistance barrier to the spread of corrosion under the paint film should it be damaged down to bare metal
- As a generalisation an increase in phosphate weight gives an increase in corrosion resistance but a decrease in the mechanical strength of a painted system.

For this reason zinc phosphate coating weights in the region of 1.5-4.5 g/M<sup>2</sup> (160-480 mg/sq ft) are those generally employed under paint systems.

Spray phosphates yield finer coatings at the lower coating weight range while dip phosphates are generally at the higher end although the presence or absence of conditioning (or refining) rinses prior to treatment can complicate this by affecting the reactivity of the metal.

In addition the type of cleaning employed initially can also have a marked effect not only on coating weight but also on the structure obtained.

To sum up, the chemical conversion coating i.e. the zinc phosphate is the all important sandwich between metal and

paint. Paint goes on top of phosphate not metal!

The type of phosphate coating used to provide the best key will depend upon the type of metal which is to be converted e.g. steel, zinc or aluminium and the nature of the applied paint film e.g. anodic or cathodic electropaints, powder or conventional wet spray or dip paints.

## Basic mechanisms

The mechanism by which a zinc phosphate bath works is as follows

### 1) The bath

Additions of concentrated, acidic zinc phosphate feed stock and accelerator are made continuously. Metal enters the reaction zone area where some of the metal is dissolved off triggering the formation of the conversion coating. The dissolved metal is precipitated out as an insoluble phosphate sludge by the action of the accelerator. (Figure 1).

### 2) The reaction

All phosphate conversion coatings have the same mechanism of reaction.

a) Free acid present in the solution attacks the metal dissolving off a thin layer.

b) The metal dissolved is taken up by the surrounding chemical solution — and ultimately is removed as sludge.

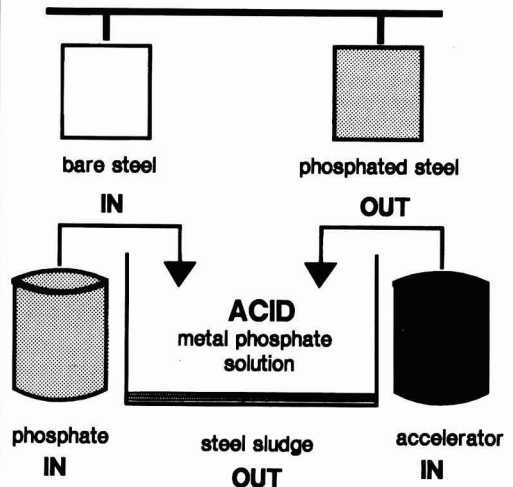
c) The zinc phosphate present in solution undergoes a chemical change and deposits on the metal surface as an insoluble crystalline layer.

d) Replenishment chemicals are added to the bath to maintain the chemical strength. (see Figure 2).

The characteristics of the deposited phosphate coating depends on the formulation of the concentrated chemical feed stock.

Important differences in coating structure can be found

Figure 1



depending on what ions are present in the feed stock and this leads to differences in paint bond characteristics.

An example of the development of zinc phosphates as employed for automotive markets will now be described.

In the early days of slipper dip primers, high zinc feed stocks were used in the pretreatment stage. (Figure 3).

Such processes yielded "heavy" coating weights of around 3g/M<sup>2</sup> and gave pure zinc phosphate Zn<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>·4H<sub>2</sub>O conversion coatings of the hopeite structure (see Figure 4).

With the advent of anodic electropaints in the early 1970's modifications were required to control the coating weight deposited to prevent adhesion failure and the severe problems that could result such as, scab and fillform corrosion. This was achieved by the use of fluoride for example in the formulation. (see Figure 5).

The anodic electroprimers typically yielded 240 hours ASTM B117 Salt Spray performance but increasing demands by the consumer and manufacturer alike for improved corrosion warranties led to the introduction in the late 1970's of cathodic electropaints.

Figure 2

### Acid Phosphate Solution

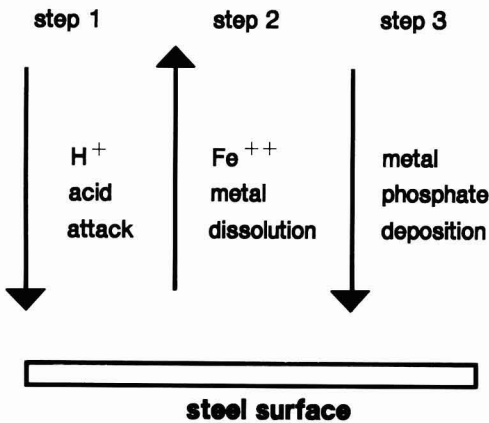


Figure 3

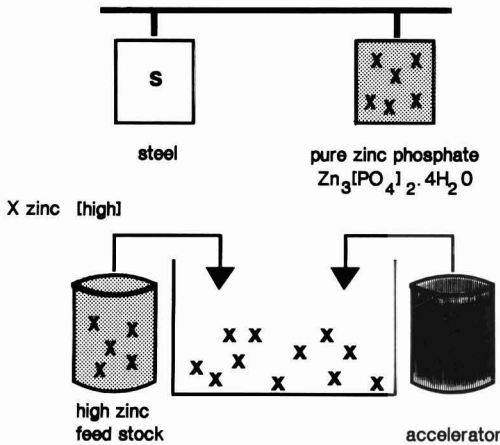


Figure 4

Scanning electron micrograph of "Generation I" zinc phosphate (x2000).

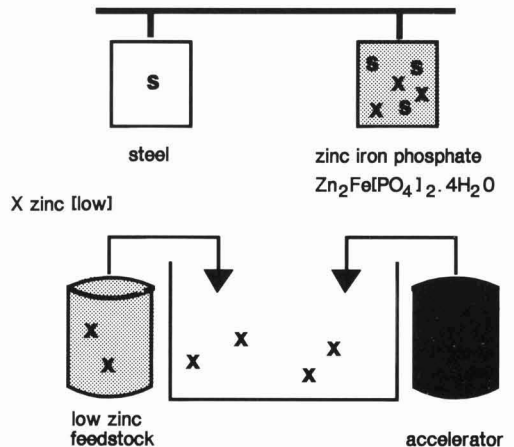


Figure 5

Scanning electron micrograph of "Generation II" zinc phosphate (x2000).



Figure 6



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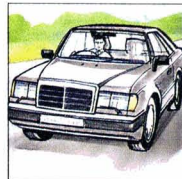
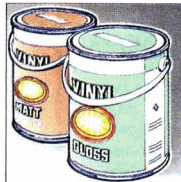
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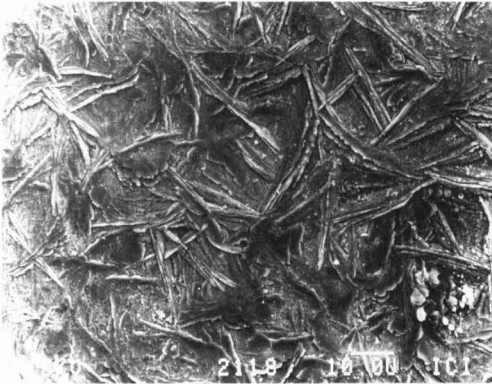
It was very quickly found that the high zinc processes although yielding relatively low coating weights ( $c 2g/M^2$ ) did not perform well under the more demanding cathodic system. Severe adhesion problems could arise leading to large scab blisters etc. This was the result of the relatively weak phosphate coating fracturing under the different stresses set up by this primer and also by the nature of the corrosion mechanism.

Extensive research led to the development of low zinc feed stocks which resulted in the conversion coating having a relatively high content of iron as well as zinc in this composition. The iron content comes about as the result of a lack of zinc (zinc starvation) at the reaction site and as the phosphate film deposited requires a balance of metal ions to complete the conversion, the acid phosphate uses the iron dissolved in the initial reaction to form part of the coating. (Figure 6).

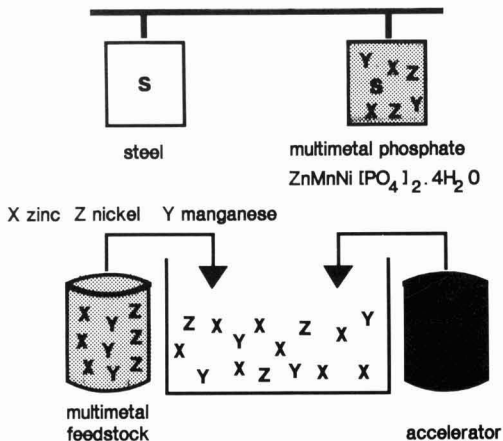
These iron rich coatings have a structure known as phosphophyllite with a composition  $Zn_3Fe(PO_4)_2 \cdot 4H_2O$ . They are characterised by the small needle like structure. (see Figure 7).

The low zinc processes when controlled well gave very good results under cathodic electropaint. However, because the zinc level was kept deliberately low problems sometimes

**Figure 7**  
Scanning electron micrograph of "Generation III" zinc phosphate (x2000).



**Figure 8**



occurred during normal production. Thus

1. Thin and/or incomplete coatings could be given due to zinc starvation at the reaction site.

2. The process depended heavily on an even reactivity of the metal surface.

3. The process was prone to the formation of jelly sludge which was difficult to eradicate without discarding the bath completely.

4. The process invariably contained chlorate as the accelerator which degraded to give chloride which was deleterious to zinc coated substrates producing the "white spotting" defect.

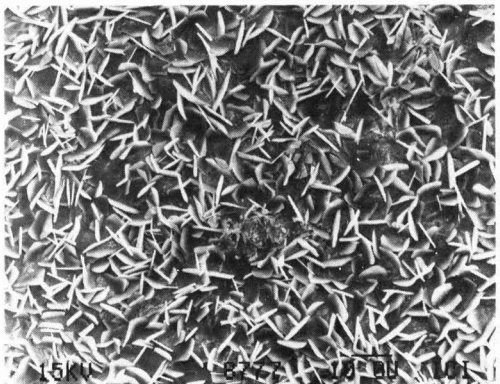
5. The process could not readily cope with mixed metal substrates.

To overcome these problems and more importantly to produce satisfactory conversion coatings on the growing volume of zinc coated steels being used in automotive assemblies multi-metal ion feed stocks have been introduced.

The multi-metal feed stocks whilst maintaining a low zinc solution provides other metal ions which enhance the properties of the resulting conversion coating especially when used under cathodic electropaints. (see Figure 8).

These processes have been variously referred to in the market place as "Tri Cation Phosphates" as the metals in the

**Figure 9**  
Scanning electron micrograph of "Generation IV" zinc phosphate (x2000).



**Figure 10**

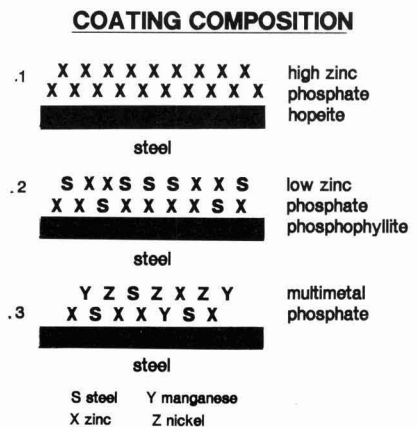
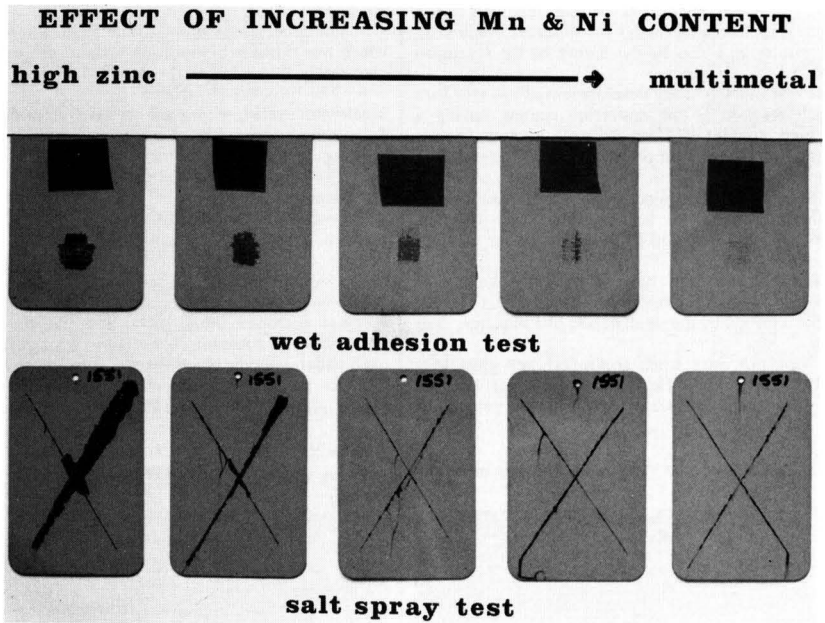
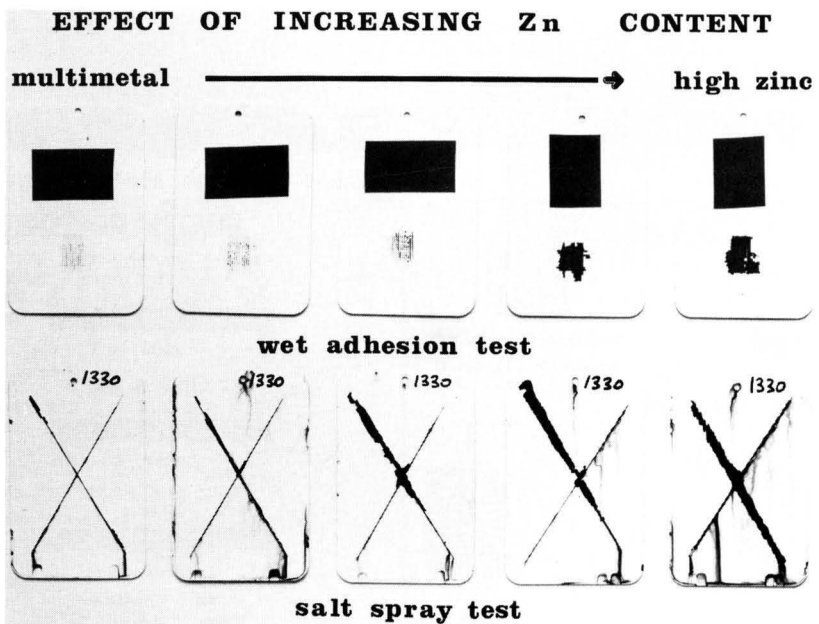


Figure 11

(a)



(b)





feedstock invariably consist of zinc, manganese and nickel. The coatings are composed of multi-metal ions of the type  $Zn Mn Ni (PO_4)_2 \cdot 4H_2O$  and are characterised by a small platelet structure (see Figure 9).

The great advantage of the multi-metal ion technology is that it will produce a uniform conversion coating on all types of metal substrates with a structure which performs well under cathodic electropaints.

Summing up, the changes in coating composition can be illustrated by reference to Figure 10.

It is worth noting that while low zinc processes produce the desirable phosphophyllite structure on steel they can only produce a hopeite type structure on zinc and its alloys. (in Figure 10.2 S then becomes X).

It has been found important that the multi-metal chemical feed stock is of the correct formulation to form the optimum conversion coating from the working bath.

With reference to Figure 11-photo A shows the effect of increasing manganese content and the resulting improvement in performance under the resulting improvement in performance under a full paint system.

Photo B shows the effect of increasing zinc content and the resulting decrease in performance under a full paint system.

### Future trends

Japan has largely led the way in the demand for better salt spray performance. While 480 hours ASTM Salt Spray was the norm until recently, Nissan are specifying 960 hours and Honda 800 hours, and it is only a question of time before European car makers follow suit. To ensure box sections are uniformly coated dip plants rather than spray plants are being introduced and a wide range of zinc, zinc/iron and zinc/aluminium coated steels are being used. (see Appendices 1 and 2). Fortunately the multi-metal ion technology is still the preferred pretreatment to enable these specifications to be reached.

Research is ongoing to understand fully the complex interactions that occur to further enhance bonding and limit the spread of corrosion.

An outline of the corrosion mechanism is given in (Figure 12).

The relevant points to note are:

- (1) An anodic reaction occurs on the damaged portion of the paint film.
- (2) An associated cathodic reaction occurs on the phosphate surface.
- (3) A build up of  $OH^-$  alkali occurs, which attacks the phosphate leading to scab corrosion.
- (4)  $Zn_3 (PO_4)_2$  is a more alkali soluble phosphate than  $Zn_2Fe(PO_4)_2$ . (Ref 1).
- (5) As a result of (4) increased scab corrosion occurs with high zinc (hopeite) processes than low zinc processes (phosphophyllite).
- (6) The alkali solubility is made less with Ni and/or Mn in the phosphate coating. (Ref 2).

### Appendix 1

#### Zinc Coated Steels










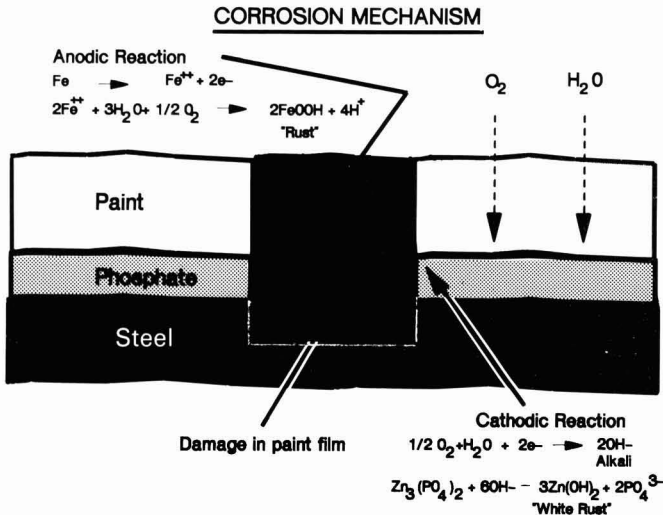
Name	Electrolygyl Composition
ZINTEC	Zinc $\cdot 10\mu$  Steel
MONOZINCAL	Zinc 
ZINCAL	Zinc  Zinc
ZINKLITE	Zn-Ni 2.5-3.0 $\mu$ 
EXCELITE	Zn-Fe 2.7 $\mu$  Zn rich Fe-Zn 0.5 $\mu$  Fe rich
ZINCROX	Zn  Cr  CrOx 

Figure 12



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## Appendix 2

### Zinc Coated Steels

Name	Composition
HDG	Zinc  Zinc
1½ SIDED	Zinc  Zinc
GALVATITE IZ	Zn-Fe  Zn-Fe
GALVALUME	Zn-Al  Zn-Al

### Organic Composite Coatings

ZINCROMETAL	Zincromet  Steel
	Dacromet  Steel
WELCOTE N	Organic Coating 55µ  Zn-Ni
BONAZINC 2000	Zn  Organic Coating 8µ
DURAZINC	Zn-Ni  Organic Coating 0.5µ

### References

1. Davis, J. W., SAE Technical Paper 820336 (1982).
2. International Patent Application WO84/00386.

### Further reading

"Phosphating and Metal Pretreatment" by D. B. Freeman published by Woodhead-Faulkner. ■

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# Low emission waterborne basecoat for mass production automotive finishing

by Z. Vachlas, Automotive Research Manager, ICI Paints, Wexham Road, Slough, Berks SL2 5DS, UK

## Abstract

In the modern painting of a motor car 12-15 litres of organic solvent are emitted to the atmosphere, 40-60% of which comes from the Basecoat in the basecoat/clearcoat finishing systems.

To reduce significantly such organic solvent emission, ICI Paints as early as 1980, pioneered the concept of a waterborne basecoat based on microgel technology by Dr A Backhouse of ICI Paints Research Department<sup>1</sup>. Such basecoats in conjunction with solvent, powder or waterborne clearcoats can meet the present and future anti-pollution demands without sacrificing the performance and aesthetic requirements of the automotive industry.

The commercialisation of such systems over the last few years is clear confirmation of the viability of the concept.

## Introduction

Painting operations in an automotive assembly plant are the largest users of organic solvents in the surface coating industry. It has been estimated that per motor car painted 12-15 litres of organic solvent are emitted into the atmosphere corresponding to 2,000-3,000 tonnes per year for a modest size automotive factory of 150,000-200,000 car/year output.

On a world scale this amounts conservatively to 300,000 tonnes, 80% of which is emitted into the atmosphere.

Such emissions are under increasingly severe scrutiny by occupational health and safety agencies and environmental agencies at government levels in the industrialised countries<sup>2</sup>.

In addition the reliance of the automotive industry on organic solvents and their wasteful use imposes economic and at times supply constraints.

The application of polymeric pigmented basecoats followed by a clearcoat for protection and aesthetic purposes has been universally adopted by the automotive industry and is responsible for up to 40-60% of the total solvent emission in an automotive assembly plant (Figure 1). This is particularly true in metalchrome colours that represent 60-80% of the automotive colour range.

If solvent emission control requirements were to be met

with conventional technologies two possible approaches are open to the industry:

a) Expensive and often complex mechanical, thermal and chemical abatement techniques involving after-burners, scrubbers, carbon absorption units etc.

b) Higher volume solids basecoats and clearcoats with their associated problems on application, aesthetic appearance and often costly plant modification to accommodate them.

In the late 70's ICI Paints scientists pioneered the waterborne basecoat/solvent based clearcoat concept based on patented aqueous microgel technology invented by Dr A. J. Backhouse<sup>3</sup>. This was designed to meet the present and future solvent emission requirements commensurate with the enhancement of the aesthetic appearance and performance of the automotive finish at a minimum investment to the manufacturer and user.

## Waterborne automotive basecoats

### Rheological characteristics

The use of waterbased coatings in the automotive industry is not new. As early as 1970 such coatings were used by GM plants in the USA for anti-pollution reasons at an enormously prohibitive cost as they required application in several successive thin coats under very narrow relative humidity and temperature operating limits and curing conditions. This was chiefly related to the lack of the essential rheology necessary to control application, film formation and pigment orientation at variable relative humidities and temperatures normally encountered in practice.

The ICI waterborne basecoat technology known as "Aquabase" relies on the use of ICI aqueous microgel which confers on the basecoat the essential pseudoplastic rheological characteristics that minimises significantly the dependency of such applied coating on water and relative humidity on application. In addition it imparts to the basecoat film the necessary rigidity to resist disruption (strike back) by the subsequent application of clearcoat.

Figure 2 shows such a viscosity profile on circulation and application. At low shear rates (A) such as in the container or

Figure 1

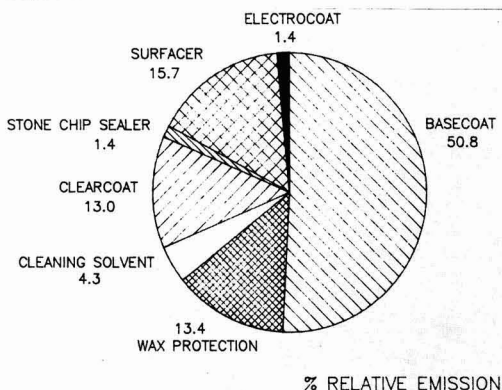
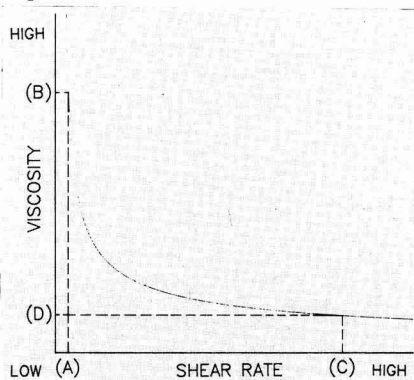


Figure 2



immediately after application the viscosity is very high (B) resulting in:

- a) Excellent anti-settling properties.
- b) Optimum metallic pigment orientation, hence enhanced aesthetic effects.
- c) Excellent sag resistance, independent for all practical purposes of ambient relative humidity and presence of water in the applied coating. (It has been determined that at high (70-80%) ambient relative humidity such applied coating may contain up to 50% water). Hence there is no need for prohibitively expensive humidity and temperature control facilities as would occur in an automotive plant spray booth assembly where large volumes of air are circulated.

The viscosity at high rates of shear (C) typical of spray application, is very low (D) resulting in:

- a) Excellent atomisation of the basecoat.
- b) Excellent metallic pigment orientation.
- c) Good circulation characteristics.

Such basecoats are therefore formulated with a high degree of pseudoplasticity. The degree of pseudoplasticity would be unacceptable in a one-coat finish because it would prevent levelling and lead to low Distinction of Image (DOI). However, in a basecoat, which undergoes considerable film shrinkage after application, this degree of pseudoplasticity is acceptable and even essential for a waterborne basecoat.

Aquabase shows no yield stress (except in some rare colours where a yield stress is induced by the pigmentation).

For the purposes of characterisation we can choose a simple model namely the Ostwald power law:

$$\tau = K\dot{\gamma}^n$$

where  $\tau$  = shear stress

$\dot{\gamma}$  = shear rate

K is a constant

and n = the viscosity index.

Figures 3a, 3b, 3c and 3d illustrate the rheological characteristics of two Aquabase paints.

Figure 3a illustrates the viscosity plotted against shear rate for metallic silver basecoat, while Figure 3b shows the same data but as a plot of the log shear stress against log shear rate. It can be seen that the log/log plot is a good fit to a straight line of slope n.

The value of n will vary between 1 and 0. When n = 1 the rheology is Newtonian and the lower the value of n, the greater the degree of pseudoplasticity. (Note that the data is not a perfect straight line fit). The value of n can therefore vary to some extent with the shear rate range. This variation may be important for calculations of pressure drop in pipe flow.

Figure 3a

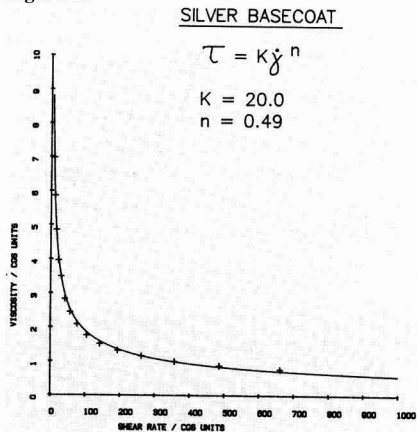


Figure 3b

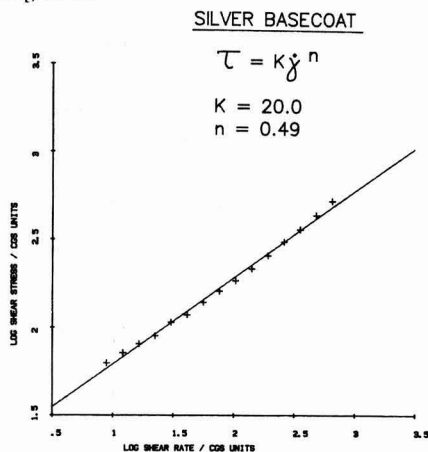


Figure 3c

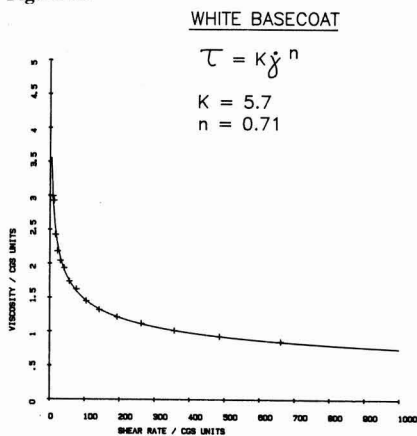
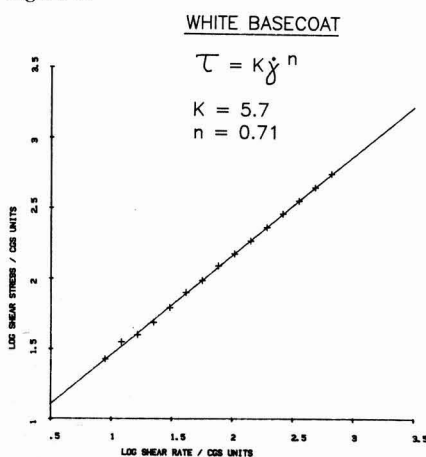


Figure 3d



The data is plotted in CGS units. The value of  $K$  therefore is equal to  $\tau$  when  $\gamma = 1$ ; it is therefore also equal to the extrapolated value of the viscosity of the paint in poise at a shear rate of  $1 \text{ sec}^{-1}$  (what is termed the low shear viscosity, LSV).

Figures 3c and 3d show the same data for a white basecoat. Note that the value of  $K = 5.7$  is lower than in the case of the silver where  $K = 20$ , and the value of  $n = 0.7$  is higher than the silver where  $n = 0.49$ .

The white paint is therefore less viscous at low shear and less pseudoplastic.

In general, high metallics are formulated at higher values of  $K$  and lower values of  $n$  than is the case for solid colours. In the case of metallics, flow within the film must be prevented to a greater degree, in order to prevent aluminium movement, than is the case in solid colours, where it is only necessary to prevent sagging.

In addition, solid colours are often used at higher film thicknesses in order to obtain opacity, and too high a value of  $K$  will prevent air bubble escape and cause "popping."

The data for these curves were obtained from a Rheomat 30 A-cup (Contraves A.G Zurich).

In practice for the purpose of paint characterisation such a rheological profile can be characterised by two single point viscosity measurements. The shear rates at which these viscosities are measured are chosen to represent conditions at

- 1) spray atomisation.
- 2) on the car body (as shown in Table 1.)

**Table 1**  
Viscosities of Aquabase basecoats

	H.S.V. (10,000 $\text{sec}^{-1}$ ) (ICI cone & plate)	L.S.V ( $\text{sec}^{-1}$ )
Metallic basecoats	0.28-0.36	15-30 poise
Solid colour basecoats	0.3-0.4	5-20 poise

## Microgel dispersion

The basis of the original ICI invention is a water dispersible acrylic polymer in the form of a specially designed microgel dispersion.

Each particle of the acrylic co-polymer is about 90 nanometres in diameter and is crosslinked such that it is no longer soluble in the types of solvents normally used in subsequent clearcoats. This particle forms the core and is encapsulated by a mantle layer of polyelectrolyte which contains functional groups for crosslinking. When neutralised with organic bases (amines) the particle forms a structure which is shown schematically in Figure 4.

The particles are produced by a multistage emulsion polymerisation process followed by neutralisation to swell the polyelectrolyte. The polyelectrolyte mantle confers the required rheological properties that enable the paint to be applied over a range of relative humidities while the crosslinked or gelled polymer core packs down in the final film to give a paint film which can be coated with a solventborne clearcoat without an intermediate curing stage. The gelled particles prevent disturbance of the coloured basecoat layer by the solvents from the clearcoat layer.

The monomer composition of the core and mantle and their relative thickness are adjusted to give the desired polymer engineered properties.

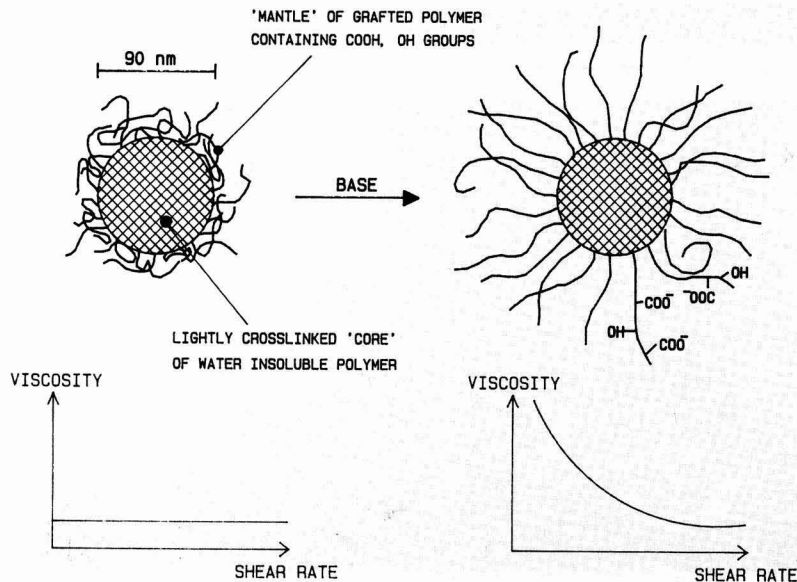
In summary the core provides:

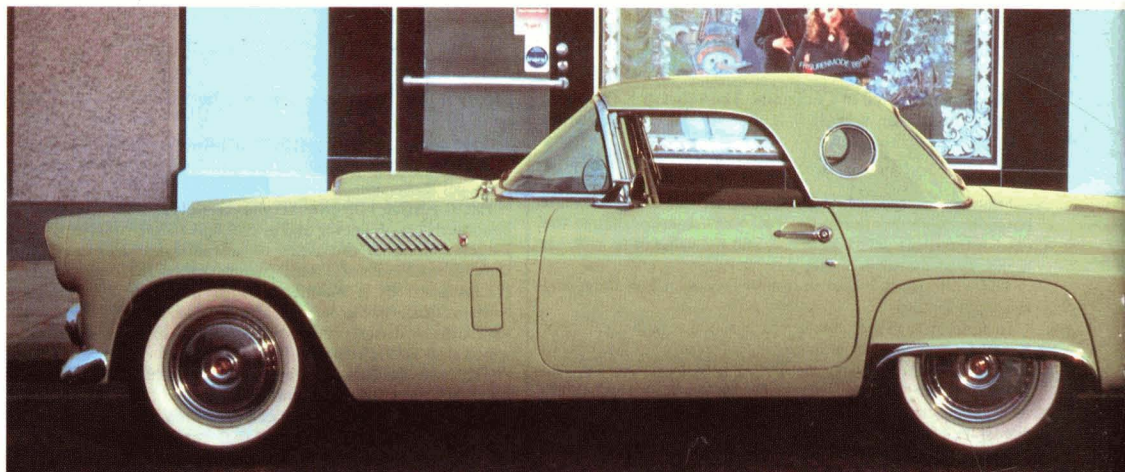
- Resistance to attack (disturbance) from subsequent application of the clearcoat.
- Mechanical toughness and flexibility.
- Durability associated with the chemical nature and high molecular weight of the polymer.
- Chemical resistance associated with high crosslinked density.

The mantle provides:

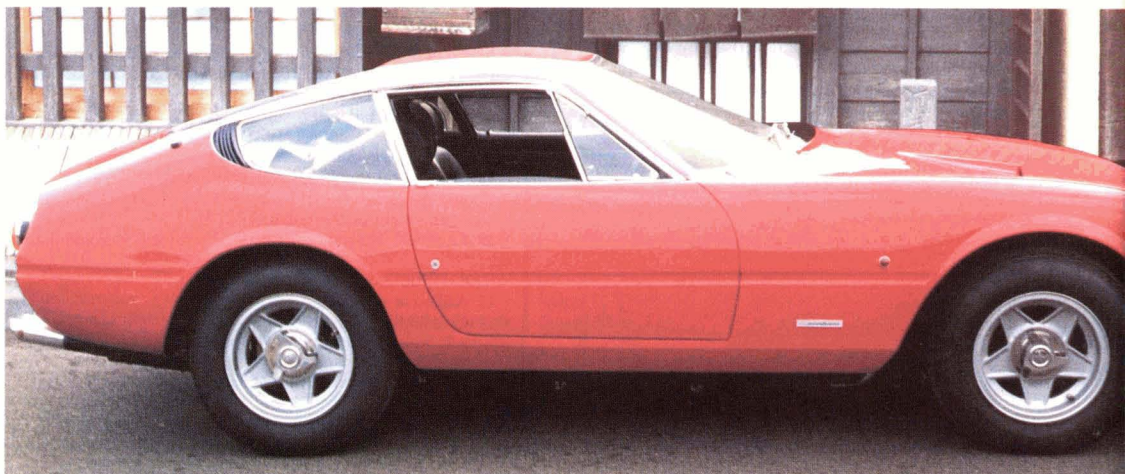
- Adhesive properties.
- Crosslinking potential.

**Figure 4**

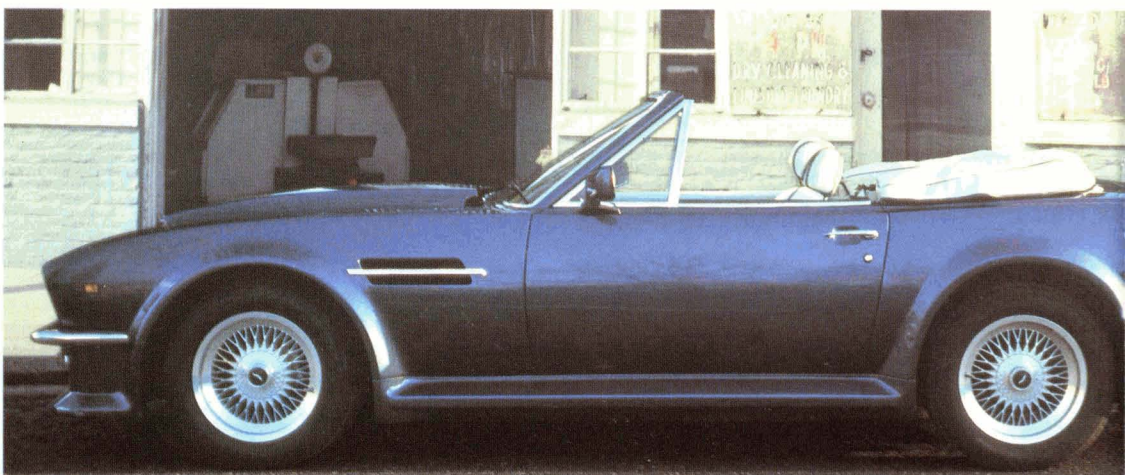




FORD THUNDERBIRD 1955



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# WHAT HELPS MAKE AN AUTOMOBILE A CLASSIC?



Great styling? Quality manufacture? Fine materials? Cardura is now in its third decade as a fine raw material widely used in the manufacture of the highest quality automotive coatings. After 25 years, Cardura has become a classic in its own right.



**Shell Chemicals**

- Rheological characteristics.
  - Stabilising groups static and dynamic stability.
- Extensive production line experience confirms that such basecoat can be applied by a variety of methods common to the mass production automotive industry (Table 2).

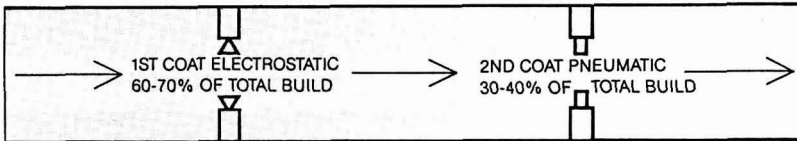
To achieve the best balance of transfer efficiency and aesthetic appearance the application of waterborne basecoats particularly in metallic colour will involve a combination of electrostatic and pneumatic application (Figure 5).

**Table 2**

Line speed	6m/min	8-9m/min
No. of guns/station	2	2
Gun separation	>50% overlap	>50% overlap
Gun off-set	10-12cm	10-12cm
Fluid rate/gun	440ml/min	640ml/min
Atomising air	70psi	80psi
Strokes/min	46	38
Gun-body distance	40cm	36cm
Fan width	50cm	46cm
No. of coats	2	2
Flash off between coats	1-2min	1-2min
Dry film build achieved (for a metallic at 16% solids)	13mm	11-12mm
<b>Electrostatic Application</b>		
Bell	BEHR mini tubo-bell. Dev No EV31/4	
Rotation speed	25,000rpm-30,000rpm	
Shroud air	0.6-1.2bar	
Voltage	80kv	
Nozzle	1.0mm	
Fluid delivery	200cc/min	
Bell-body distance	30cm	

The adoption of this revolutionary concept, conforming to the demanding production line requirements of the automotive industry world-wide, presented a number of engineering challenges. Specifically these were:

**Figure 5**



a) Design of a drying tunnel capable of removing 90-95% of the water from the basecoat in 1½-2½ minutes prior to the application of clearcoat and compatible with the overall painting operations.

b) Design calculations and pumping facilities to cater for pseudoplastic liquids. Equations predicting the flow of such specific materials in various diameter pipes at high flow rates had to be derived.

c) High voltage electrostatic application techniques without the need to isolate the system.

d) Materials of construction to cater for the active components present.

e) Passivation of metallic pigments.

**Summary**

A waterborne basecoat-clearcoat automotive top coat system has been described that offers the best prospect for meeting present and future solvent emission requirements commensurate with expected performance and aesthetic requirements.

The use of such basecoats by the mass production automotive industry necessitated the invention and application of new rheological concepts and the resolution of a number of technical challenges related to the manufacture, application and drying of such systems.

The commercialisation of such basecoats in N. America and Europe and the escalating activity by the leading Paint companies confirms the validity of this concept.

**Acknowledgement**

The author wishes to acknowledge the contribution of Dr A Backhouse in preparing this paper.

**References**

1. U.S.P. 4,403,003.
2. US Federal Register Vol 46 No. 202 October 1981.
3. Backhouse, A. J. "Routes to low pollution Glamour metallic automotive finishes." *J Coat. Tech.*, 1982, 54, 83.

**Introduction to Paint Chemistry and Principles of Paint Technology** by G P A Turner

\* The book is divided into two parts. The first covers the basic science without assuming any prior knowledge on the reader's part. In the second, chapters are devoted to paint composition, formulation and drying mechanisms, paint ingredients such as solvents, pigments and additives, and the different paint groups by chemical type. Throughout the author emphasizes the factors which govern the choice of a particular paint for a particular job.

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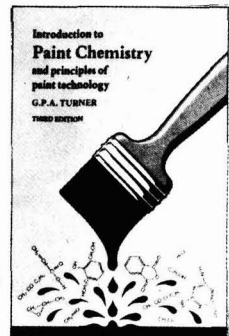
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# Measurement techniques and future trends in powder coating technology

by J. F. Hughes, Department of Electrical Engineering, University of Southampton SO9 5NH, UK

## Introduction

In order to assess the performance of a powder coating installation, measurement of various electrical parameters is an essential requirement. If high or low-voltage corona-charged guns are used, then obviously both the gun voltage and current need to be known. For triboguns, measurement of essential parameters is a little more difficult and requires fundamental knowledge of charge exchange phenomena between the powder used and the gun material. A differential charge-to-mass ratio ( $q/m$ ) measurement technique has now been developed which enables tribo behaviour to be predicted, in addition to assessing the bi-polar charging characteristics of most powders.

The suitability of powders for electrostatic application is usually dictated by the bulk or volume resistivity, while prediction of deposition performance may be assessed from the value of  $q/m$  under simulated coating conditions. Both these parameters may be measured using standard commercial instruments. However, caution must be exercised when attempting to interpret the measurements, and consideration given even to the validity of some measurement methods.

## Measurement techniques

### 1. Powder Resistivity

In all powder coating applications, it must be appreciated that powder resistivity plays an important rôle in system behaviour. It could be argued that no ideal resistivity exists for optimum performance. A contradiction always exists in terms of optimum powder resistivity. At the gun head or charging electrode, the resistivity of the powder should be as low as possible in order to maximise charge deposition on the particles. However, as soon as particle charging has been effected, the resistivity should ideally be as high as possible in order to ensure slow charge relaxation rates, and hence good adhesion, once the particle has alighted on the substrate. In all systems, therefore, the choice of resistivity must always be a compromise. In reality, there is little or no choice of material resistivity. Most powdered paints such as epoxy, polyester, acrylic, nylon and polyethylene are inherently highly resistive (usually  $> 10^{14} \Omega m$ ) and are admirably suitable for displaying excellent adhesive properties.

Efficient charging of such materials, however, is not so easy to achieve. In terms of adhesion, the lower acceptable limit of powder resistivity is about  $10^{11} - 10^{12} \Omega m$ . Below this value, charging will be good but adhesion will be poor. Apart from adhesion problems with low resistivity powder, there is one important advantage in terms of coating finish. There will be no, or very little, back ionization<sup>1</sup>, and therefore an extremely smooth coating is possible. This point is perhaps of relevance to systems using pre-heated workpieces, where electrostatic adhesion is not important, or tribocharged systems where back-ionization does not normally present a problem.

A number of methods have been developed for measuring the bulk or volume resistivity of powder. One of the simplest and most straight-forward is the resistivity cell as illustrated schematically in Figure 1. A sample of powder is simply subjected to a steady d.c. potential ( $V$ ), while the steady state

conduction current ( $I$ ) is measured. Resistance is then calculated from the ratio  $V/I$  and knowledge of the cell electrode dimensions enables easy calculation of powder resistivity.

$$\text{Resistivity } \rho = \frac{R \cdot a}{l} \Omega m$$

where

$$\begin{aligned} a &= \text{electrode area} \\ l &= \text{electrode separation} \end{aligned}$$

Different types of resistivity cells are available commercially, some having incorporated into their design a standard weight on the powder surface in order to ensure uniform packing density. Evaluations of different resistivity cells has indicated that powder compression has minimal effect on resistivity, and a well tried and tested simple method of powder packing requires no more than gently tapping of the test cell base prior to commencement of the measurement.

Commercial resistivity test cells are usually automated to give a direct readout of resistivity in  $\Omega m$ . A typical instrument is illustrated in Figure 2. Unlike the test cells arrangement depicted in Figure 1, this instrument uses a small-capacity-easy-clean cell. The electrodes are mounted horizontally and the cell is made up of two halves, affording easy access to load, unload and cleaning after each sample.

Although volume resistivity is a useful parameter to know when it is necessary to judge the suitability of the powder for adequate adhesion, there is no obvious link between resistivity and charge acceptability. Examination of the Pauthenier equation reveals that particle charging is dictated primarily by permittivity and the electric field which the particle is subjected to.

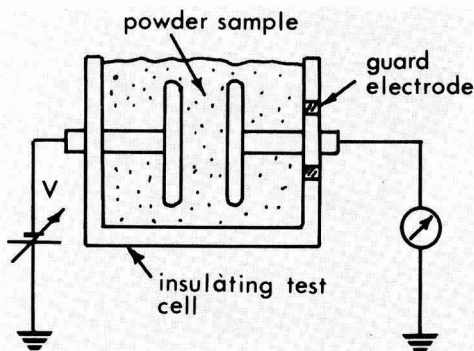
The limiting value of surface charge, the Pauthenier limit, may be expressed mathematically by the following relationship:

$$Q = 4\pi\epsilon_0 a^2 B E \quad (1)$$

where

### Figure 1

Schematic of a simple powder resistivity test cell.



$$B = 1 + 2 \frac{(\epsilon_r - 1)}{(\epsilon_r + 1)}$$

and

$\epsilon_0$  = permittivity of free space =  $8.854 \times 10^{-12}$  F.m<sup>-1</sup>

$\epsilon_r$  = relative permittivity of powder particle

$a$  = particle radius

$E$  = electric field which the particles are subjected to

If a particle is assumed to be spherical, its mass,  $m$ , will be given by:

$$m = \frac{4}{3} \pi a^3 \rho$$

where

$\rho$  = density of the particle

So the maximum value of the charge to mass ratio,  $q/m$ , will be:

$$(q/m)_{\max} = \frac{4\pi\epsilon_0 a^2 B E}{3} = \frac{3\epsilon_0 B E}{\rho a} \quad (2)$$

On substituting typical values into Equation 2, some indication may be gained of the expected  $(q/m)_{\max}$  for any powder under test.

Typical values might be:

$$\epsilon_r = 2, E = 10^6 \text{ v.m}^{-1}, a = 50 \times 10^{-6} \text{ m.}$$

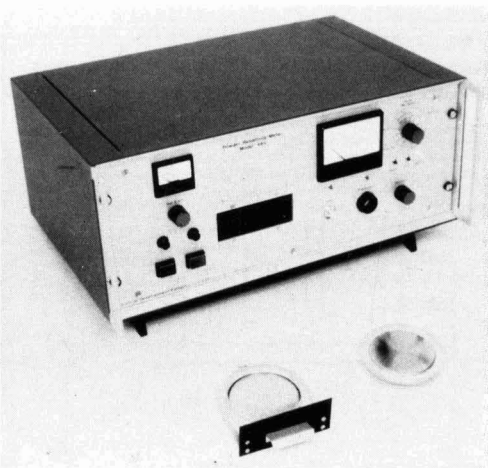
$$\epsilon_0 = 8.8854 \times 10^{-12} \text{ F.m}^{-1}, \rho = 10^3 \text{ kg.m}^{-3}$$

giving  $(q/m)_{\max} = 8.8 \times 10^{-4} \text{ C.kg}^{-1}$

This value for  $(q/m)_{\max}$  is in itself quite interesting in that it is very typical of measured values of  $(q/m)$  in standard commercial equipment. That being the case then the reason for generally low deposition efficiency must be other than purely electrostatic. It is suggested that more attention should be given to the aerodynamics of a coating system. After all, the main forces acting on particles emanating from a typical

**Figure 2**

**Powder resistivity test measurement (photograph courtesy of ID (B) Ltd. UCNW Bangor N. Wales).**



coating gun are aerodynamic – due to the gun air flow characteristics. Only when particles are within a few millimetres of an earthed substrate do they begin to feel the electrostatic attraction of the image charge.

One final note about measurement of volume resistivity.

Although all commercial measurement instruments are all calibrated to give a direct readout in ohm-metres, the units of volume resistivity, it is highly unlikely that the parameter measured is a true indication of the sample's ability to conduct charge through its volume. Because of the inherent insulating properties of most powder paints, any measured charge conduction must surely be preferentially over the surface of the material under test.

The measured parameter is therefore more likely to represent the surface conduction characteristics rather than bulk or volume resistivity. Nevertheless, the measurement can be useful when comparison of conduction characteristics is required between different samples.

## 2. Permittivity

Referring back to Equation 2, the implication is that relative permittivity might be a more important parameter, especially if the charge accepting characteristics of the powder sample are of prime interest.

The measurement of permittivity is quite straightforward and the resistivity cell illustrated in Figure 1 may be used for this test. With the use of a standard capacitance measuring instrument, the relative permittivity may be calculated from just two simple measurements.

Step 1. Connect the empty resistivity test cell to the capacitance bridge. Measure,  $C_0$ , the air capacitance of the cell.

Step 2. Fill the cell with the powder under test. Measure,  $C_r$ , the new value of cell capacitance.

For a simple parallel plate electrode cell, the air capacitance  $C_0$ , will be given by:

$$C_0 = \frac{\epsilon_0 A}{d}$$

where  $A$  = cell plate area

$d$  = cell plate separation

With the test cell filled with the powder under test, then the new value of capacitance,  $C_r$ , will be given by:

$$C_r = \frac{A\epsilon_0\epsilon_r}{d} = \epsilon_r C_0$$

Hence the relative permittivity,  $\epsilon_r$ , will simply be given by the ratio:

$$\epsilon_r = \frac{C_r}{C_0}$$

## 3. Charge to mass ratio $(q/m)$

Perhaps the most important parameter of all to measure when plant assessment is being considered, is the ratio of charge to mass of powder emanating from a gun nozzle. For an electrostatic coating system, imparting electrical charge to the paint particles is of fundamental importance to the entire process. Particles that do not succeed in acquiring charge, or particles that are not sufficiently charged, will probably not contribute to overall deposition efficiency of the coating plant. The gun nozzle and surrounding area is probably the most important region in the entire system. It will be in this region that particles acquire charge, usually having a very short time and one chance of doing so. Acquiring insufficient charge at this station will normally lead to poor deposition

efficiency, with a high percentage of overspray. Measurement of charge-to-mass ratio ( $q/m$ ) is appealingly simple, although certain precautions have to be taken in practice. The measurement usually requires two separate tests. One to measure the net charge on a powder sample, and one to measure the mass or weight of the same sample. The device most commonly used to measure charge is the Faraday cup, coupled to a charge measuring electrometer instrument. Figure 3 illustrates schematically a simple Faraday cup arrangement.

A small metallic cup is enclosed by a larger metallic cup which is grounded. The inner cup is connected to an electrometer instrument by a shielded co-axial cable. Both inner and outer cups have small openings in one end to allow insertion of the sample to be measured.

The normal testing procedure with a powder coating gun would require the Faraday cup to be placed in front of the gun so as to enable a sample of the sprayed powder to enter the inner cup. Any charge associated with the collected sample would be recorded on the electrometer, and weighing the cup before and after the test would lead to calculation of the weight of powder sample collected.

In practice, however, this simple system poses many difficulties, and the validity of measurements must always be suspect. The technique is especially vulnerable to errors when used to assess the performance of high-voltage corona-charged gun applicators. The problem arises primarily from the fact that a very high density of free ions will almost always co-exist with the stream of particles emanating from a gun nozzle. As well as catching powder particles, therefore, the Faraday cup will also allow free, unattached ions, to enter,

thus leading to an erroneously high value of measured charge. The charge associated with the free ions will not contribute to the true charge-to-mass ratio for the powder sample collected. This serious error arises, not surprisingly, from the fact that typically only about 0.5% of all ions generated by a high voltage gun actually end up attached to particles. Some attempt has been made to overcome this problem by attaching a grid screen across the inlet part of the Faraday cup. This can be seen in one of the commercial units illustrated in Figure 4.

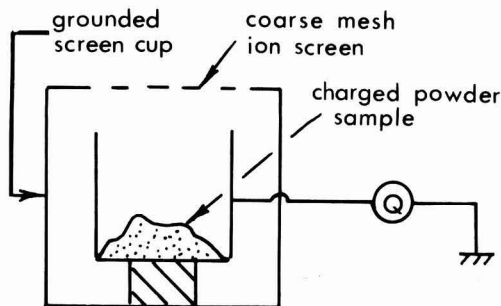
This type of grid relies on the high ionic mobility to trap the ions prior to entry into the Faraday cup. The technique is technically sound, but does not perform satisfactorily in practice. The downfall of this simple grid screen approach is that the grid itself will almost immediately itself become coated with powder. With the first mono-layer of powder deposited, back-ionization will be initiated on the screen, thus discharging further on-coming powder and therefore completely modifying the system under test. A classic example of the presence of a diagnostic instrument interfering with the system under test.

If the Faraday cup technique is to yield sensible results, then the grid ion trap must somehow be prevented from accumulating powder. One system that achieves this is illustrated in Figure 5. This technique is not yet available commercially, but has been demonstrated used extensively as a research instrument for a number of years<sup>2</sup>.

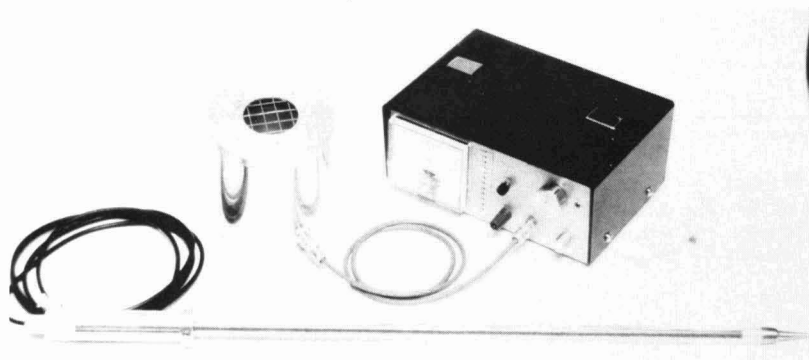
The Faraday cup concept has been completely modified. The cup has been replaced by two flat plates, one mounted vertically and one mounted horizontally. The ion trap grid is a vertically strung parallel array of wires, with the unique additional facility of continuous water washing. Irrigation of the wires ensures that any powder particles that may be collected will not remain on the wire screen, therefore eliminating the possibility of back-ionization. In addition, the L-shaped plate catching area enables four important parameters to be measured from just a single test.

Measuring the charge and mass associated with powder alighting on the vertical plate enables the maximum charge-to-mass ratio to be calculated. Powder particles that are insufficiently charged, or not charged at all will alight predominantly on the horizontal plate. Measurement on the horizontal plate will therefore yield a minimum charge-to-mass ratio. Connecting the vertical and horizontal plates together will enable the overall, or mean,  $q/m$  to be calculated. Provided the powder throughput of the gun under test is known, then conducting collection tests for a known time will enable the deposition efficiency to be assessed. Four useful parameters therefore, from just one test.

**Figure 3**  
Schematic of a simple Faraday cup test cell



**Figure 4**  
Charge-to-mass ratio test instrument (Southampton University).



1. (q/m) max
2. (q/m) min
3. (q/m) mean
4. Deposition efficiency

Irrigation of the ion trap screen will only be necessary when tests are being conducted on high-voltage corona-charged powder guns. Low voltage and triboguns do not generate a high density of free ions, and therefore back-ionization will not occur on the screen during a typical testing period of 5-15 seconds. Likewise, the conventional Faraday cup test system will be quite adequate for q/m tests on low voltage and triboguns.

A parameter which is rapidly gaining importance in powder coating technology is the differential tribo charge-to-mass ratio. It is now widely accepted that some powders will experience considerable tribocharging effects purely as a consequence of being transported from the hopper, through the pipe delivery system and into the gun. In some instances, the level of tribocharging can be of the same order of magnitude as the corona charging. Provided the polarity of

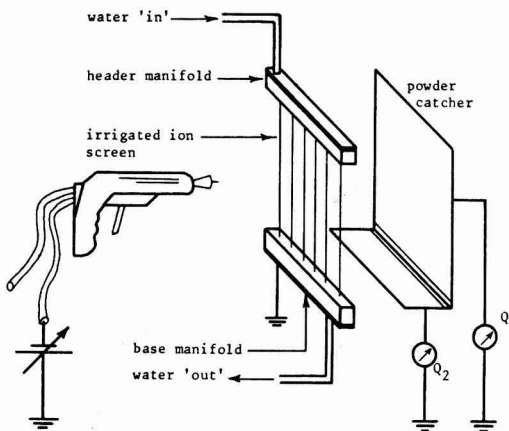
the tribo component of charging is the same as the corona, then good charging will be expected. Tribo polarity can be erratic and unpredictable, and for this reason, prior knowledge of the expected tribo behaviour can be very important. When conducting standard q/m tests, either with a Faraday cup or irrigated grid, it is therefore good policy to first measure the tribo charging characteristics of the gun under test. That is, measure the powder q/m with all external voltage sources switched off. Not only will this enable the tribo component of charging to be measured, but also the all important polarity of charging will be known. The correct choice of gun polarity will then ensure optimum performance for the particular gun/powder combination to be used.

Bi-polar charging of powders is a very common occurrence, which incidentally is still not very well understood. In a typical powder sample transported from a hopper to a gun, it is not unusual to find the larger particles tribo charging to one polarity while the fines may be charged to the opposite polarity. If optimization of gun charging is sought, then it will be necessary to identify the bi-polar tribo characteristic of the powder being used. Neither the standard Faraday cup nor the irrigated grid system will yield this information, and alternative experimental techniques have to be sought. One system that has been used successfully for a number of years is illustrated schematically in Figure 6.

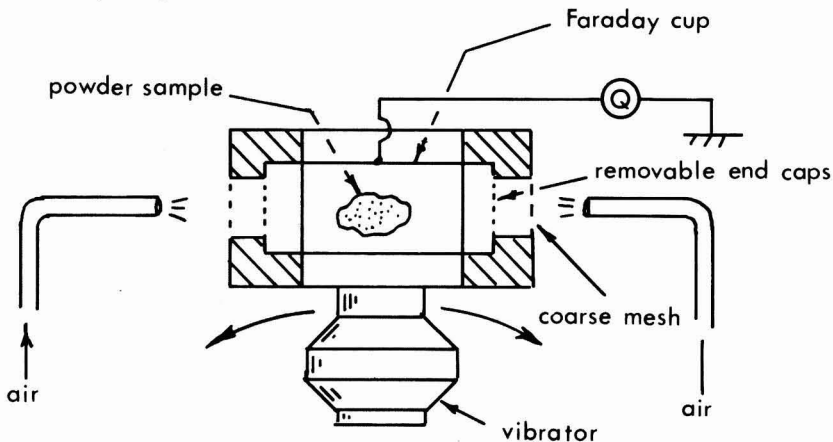
This differential q/m technique is based on the original Faraday cup method illustrated in Figure 4. An inner measuring cup is enclosed by a grounded shielding cup, and the inner cup is connected to an electrometer instrument. This new technique differs from the standard q/m method in that both ends of the cup are open, but may be covered with interchangeable end caps of different size meshes. In addition, the entire cup assembly is vibrated and reciprocating air blasts are directed into and through the inner cup.

By commencing the test with very fine mesh end caps, and gradually progressing to coarser mesh, it is possible to construct a histogram of charge-to-mass ratio as a function of particle size. Since charge on the Faraday cup is measured as the powder exits the cell, then the polarity of charge measured on the electrometer will be opposite to that on the powder itself. This only requires mentally remembering to change the polarity of the measurement if the true polarity of the powder sample is required.

**Figure 5**  
Irrigated grid charge-to-mass ratio test system.



**Figure 6**  
Differential q/m test system.



A typical sequence of running a differential q/m test on a powder sample would be:

1. Insert powder sample into inner Faraday cup
2. Attach finest mesh end caps
3. Weigh cup and powder sample
4. Blow out powder
5. Re-weigh cup and remaining powder sample
6. Repeat steps 1-5 for increasing mesh size end caps
7. Calculate q/m for each size range

It must be appreciated, of course, that this measurement will only indicate the tribocharging characteristics of the powder under test when impacting the surface of whatever material the test cell is made of. Assessment of how the powder might behave in a nylon or teflon gun, for example, would require the inner Faraday cup to be lined with the appropriate material. This will not affect the basic principle of the measurement technique. There may be a component of charging associated with the powder sample impacting the mesh end caps – which must be taken into account for precision measurements. Extensive evaluation of the differential q/m test method has indicated, however, that the contribution of charging by the mesh is usually insignificant.

### Future trends

It is inevitable that if further optimization of powder coating installations is to be achieved, then two important actions must be initiated:

First, there must be much more awareness of the implications of electrostatic measurements, combined with a firm policy of operator instruction in conducting measurements on a routine basis.

Secondly, it is becoming more and more apparent that aerodynamic considerations within the coating environment are as, if not more, important than electrical measurements.

Extensive tests on the charge accepting properties of powders appear to indicate that most corona charged systems are charging the particles quite adequately. It is now almost certain that it is the high free ion density in such systems that contribute to such poor deposition efficiencies and, in some instances, sub-standard coating finish.

Tribocharged systems are rapidly gaining interest. Commercial units have proved very successful, and it is likely that developments in this area will continue. Obvious advantages of tribosystems include the absence of a high-voltage supply, virtual elimination of free ions and superior powder penetration coating characteristics. There may still be a requirement to customise powders for tribo applications, and further developments may be necessary before optimization can be realised.

Perhaps the most crucial requirement for tribo applications is the reliability of a powder's charging characteristics, both on a day-to-day basis and on the predictability of its performance in different equipment. In addition, just as for corona-charged systems, the aerodynamic conditions in the vicinity of the workpiece will be crucially important.

In an ideal coating situation, the forward gun air flow should be just sufficient to carry the charged particles to within about 1 cm of the workpiece surface. At this position, the particles will just begin to be influenced by the electrostatic attraction force due to their image charge on the grounded substrate. In order to maximise the electrostatic deposition process, therefore, all external air flow should be minimised at this boundary. Of course it will be impossible for a mass of air to just suddenly stop moving, so a compromise situation should be sought where at least the air flow will not be the dominant factor in terms of dictating particle trajectories. This will not only necessitate careful adjustment and control of gun air, but also consideration should be given

to the implications of coating performance on the booth air extraction system.

An ideal powder applicator might be one where no external air is used. Experimental airless guns have been evaluated over the years, with some designs showing potential promise. A vibrated inclined plane with a linear sharp corona electrode at its extremity is just one design that has proved quite successful<sup>3</sup>. As with all corona-charged systems, however, a very high free ion density was generated which ultimately leads to an unacceptably high level of back-ionization.

A powder charging technique which has not yet really been seriously considered for powder coating applications, is charging by induction. Induction charging is already used successfully for toner charging in electrocopying machines. It would appear that all the disadvantage associated with both corona and tribocharging may be eliminated with this technique. For example, no free ions are generated, only low voltage is required, there is no dependability on interface characteristic, there is no requirement for intimate contact between each individual particle and the charging electrode, and furthermore the technique naturally lends itself to airless application.

At the present state of powder coating technology, there is of course one very good reason why induction charging is not already widely used. In order to effect efficient charge transfer by induction, the powder resistivity must be relatively low – a requirement that immediately contradicts the ideal characteristic for good adhesion. It is possible, however, that a compromise resistivity may exist, whereby acceptable induction charging may be possible while at the same time retaining good adhesion. An even better approach may be the modification of powder paints such that field dependent characteristics are displayed. This approach is already used commercially in some mono-component developed systems for electrocopying machines. At the toner charging station, where the electric field is relatively high, the volume resistivity is low thus allowing charging by induction. After deposition when the electric field will be relatively low, the volume resistivity reverts to its original higher value, thus retaining the ideal characteristics for acceptable adhesion.

Electrostatic optimization of powders will become more important as higher deposition efficiencies are demanded. It may be unrealistic to strive for 100% deposition efficiency, but a figure of 90% may be quite practical. A deposition efficiency of 90% in a single pass would have extremely interesting practical implications. Re-claim of the 10% overspray could be ignored, thus enabling rapid colour change without the problem of colour cross contamination.

### References

1. "Electrostatic Powder Coating", J. F. Hughes, 1984, Research Studies Press, Letchworth UK.
2. Irrigated Grid q/m measuring device, UK Patent No. 8320654.
3. Experimental airless gun at Southampton University.

Presented at the 1988 DSM Resins Powder Symposium, Nordwijk, The Netherlands.

*Continued from P.160*

the use of Tilcoms in non-aqueous products and the benefits of diluting the complexes before incorporation to improve effectiveness.

The vote of thanks was given by the Chairman, Mr S Lynn, after which the usual excellent buffet was available, with liquid refreshments, all sponsored by Tioxide.

J Bravery ■

# Protective Coatings for Concrete

by R. F. Stanfield, Liquid Plastics Ltd, P.O. Box 7, London Road, Preston, Lancashire PR1 4AJ, UK

## Abstract

This paper is an overview of the causes of decay and corrosion of reinforced concrete. The function of protective coatings in relation to the environment and the position of the concrete is explored. Concrete is defined and micro and macro effects reviewed as are surface and bulk effects. Reactive priming and substrate surface effects are detailed for an ideal system.

Coatings are listed and parameters relative to protection are viewed in the context of experimentation. Conclusions are drawn having shown the need for more than just a protective function.

## 1 Introduction

An owner sees a reinforced concrete structure as a utility with a long life, designed by Engineers to perform a particular function. The Engineer may see the concrete as a uniform composite structure finished in its own right. The non-technical by-stander sees a drab grey structure and may wonder if it can be aesthetically improved. The concrete scientist knows the structure is not uniform, is subject to environmental decay and may not continue to carry out the functionality perceived by the owner or Engineer, short or long term.

The surface coating manufacture attempts to reconcile these viewpoints and is required to be knowledgeable in all aspects thereby advising on Protection.

The emphasis has then moved from simple aesthetics to encompass proven Protection from the causes of decay. These causes can be summarised as:

- Carbonation, leading to re-bar corrosion and spalling/weakening.
- Chloride attack, leading to re-bar corrosion and spalling/weakening.
- Acidic gas attack or solutions of these, leading to re-bar corrosion and spalling/weakening.
- Water permeation, exacerbates carbonation and other attack.
- Alkali aggregate (or silica) reaction (A.S.R.), leading to expansion and cracking.
- Weather erosion, leading to localised problems.
- Chemical or physical abuse or over-

use, diverse surface or bulk stress distress.

These causes can occur in tandem or in isolation and represent a starting point of knowledge for manufacturers of Coatings for Protection.

From this point it is necessary to know the functionality of the coating in relation to the placement of the concrete, to understand the nature of the concrete surface as opposed to the bulk, to work with established or evolving test criteria and parameters, and know the limitations of surface only protection.

The fact that there are hundreds of variations of coatings and even more situations in which concrete is used does not preclude an overview of the following basic type.

## 2 Function of coating

In reviewing functionality it must be stated that differences exist between a surface lying coating film and a surface impregnant, the latter is fully documented elsewhere.

The main function of a protective surface coating is to prevent corrosion of the reinforcement or halt surface attack:

1. Control or arrest carbonation within the concrete.
2. Inhibit or stop water penetration and thereby inhibit –
  - (a) chloride ion penetration from de-icing salts, seawater or coastal air.
  - (b) penetration by airborne gases or solutions of gases from industrial processes.
  - (c) ASR by depriving the reaction of its driving mechanism.
  - (d) other water driven phenomena which lead to surface or bulk cracking, e.g. freeze/thaw.
3. Provide a safeguard from weather erosion to maintain the surface or provide waterproofing to cracked/porous structures.
4. Prevent or minimise chemical attack by industrial processes and maintain the substrate integrity.
5. Prevent or minimise physical attack on floors for example by foot or vehicular traffic.
6. Bind a weather eroded surface.

Other considerations do not

necessarily fall within the function of protection from defects, but may be a priority consideration for the owner:

- graffiti resistance or ease of removal
- hygiene sensitivity internally, i.e. dry and dirt free
- anti-mould growth
- evening out of colour from repaired concrete
- anti-static; nuclear decontamination and other speciality requirements.

From this it can be seen that a coating must always be capable of fulfilling a series of basic protective functions, and provide in each case a speciality role with one not provided at the expense of the other.

## 3 Coating and the environment

Knowing the protective function required of the coating is clearly fundamental, to this must be added a consideration of the placement of the structure in question.

Will a coating for an industrial concrete floor to prevent dusting or chemical attack, suffice for waterproofing a concrete roof in Saudi Arabia or preventing long term chloride attack on a bridge deck or structure? The answer is possibly yes, but will it also suffice for new concrete, weathered concrete, flat concrete subject to water ponding, concrete subject to freezing northern conditions, subject to hot damp tropical conditions – and perhaps most demanding of all – will it resist on all structures cracking in sympathy with existing or new substrate cracking? *'It should be borne in mind that all concrete structures crack as part of their design principle'* – a coating must remain integral in order to carry out its protective role, a cracked coating allows the defect to occur locally that it is supposed to prevent.

If the 'placement' is considered in a defined way on each occasion, then coating system selection is simplified. Accepting that the coating can carry out the protective function as outlined in (1) and (2); then consider these basics into which most jobs will fit:

- (a) *Roof concrete* – new, cured or green, aged, flat or pitched, already cracked, repaired, jointed, pre-cast or poured in place, expansion joints etc.
  - (b) *Wall concrete* – as above but vertical with door and window openings.
  - (c) *Floor concrete* – flat with roof concrete considerations, plus possible chemical attack from processing or use.
  - (d) *Underwater concrete* – via tank, reservoir, basement (wet ground back-filled); contains elements of preceding plus high water pressure and organic growth and bacterial attack.
- (e) *Structural* – e.g. bridge, tunnel –



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contains elements of preceeding plus chemical and pollutant attack, possibly high back pressure.

(f) *Support* – e.g. column – may contain some of preceeding elements and a high stress factor transmitted to the surface.

(g) *Speciality* – e.g. glass reinforced concrete (G.R.C.) often subject to panel construction problems leading to crazing or cracking.

Clearly coatings as a whole will not cope themselves with all these elements and REINFORCEMENT must be considered as an integral part of specification.

### 3a Cohesion v. adhesion

Having defined the protective function and viewed the coating selection against the placement of the concrete, a final consideration (ignoring unit price and application) will be how to ensure retained integrity of coating long term – assuming the protective function is paramount.

One way of viewing this is by comparing the adhesive strength of the system on the concrete in question to the film strength, and selecting the coating system to ensure cohesion exceeds adhesion.

Concrete is known to crack, so any surface can be expected to crack once coated over, i.e. new cracks will appear. *‘These will exert dynamic load on the minimum amount of coating cross sectional area, i.e. cracks start from zero.’* A strongly adhered coating with low tensile strength will crack in sympathy, either immediately or eventually as cyclic movement occurs centred on the crack. *‘Elasticity is not a sufficient parameter, even in excess percentages, to avoid cracking.’*

Experiments carried out by the author, substantiated in practice over many years, suggest the following relationship for high stress concrete, e.g. roofs:

(a) Tensile strength should exceed adhesive strength by at least two times, preferably by a factor of three.

(b) Tensile strength should exceed 6 MPa.

For a coating of adhesive strength of 2 MPa; a tensile strength of 5-6 MPa should ensure coating *disbondment* over a new concrete crack rather than favouring a crack in the coating. To this equation should be added a minimum elongation factor which indicates a non-brittle coating, a figure of 25-50% is sufficient. These figures are achievable by high modulus unreinforced coatings, e.g. some polyurethanes; or by reinforced coatings, e.g. water or solvent based elastomers capable of accepting glass or polymer fabrics

within the specified coating thickness.

This arrangement ensures that well adhered coating systems disbond only to the extent they need to and provide sufficient cross sectional area as a ‘bellows’ to allow absorption of the stress/strain factors involved.

The need for reinforcement in coating specification diminishes as the stability of the concrete increases, and may only apply to existing cracks, day joints, expansion joints etc. where it is equally necessary to carry the coating schedule over to provide the protective function and resist local ingress.

Having considered the cause of decay and the function of the coating relative to the concrete placement in the environment, it is necessary to consider the substrate in more detail and review the more specific parameters for priming, coating and how these can be tested.

## 4 Concrete – the substrate

### 4a Composition

The word “concrete” is derived from a Latin verb and literally means to ‘grow together’. It is a composite material which is produced by using Portland Cement to bind together sand and gravel or a crushed rock such as limestone or granite into a dense coherent mass. The strength is developed primarily by the hydration of the di- and tri-calcium silicates which are the main constituents of Portland Cement. The reaction products formed are calcium hydroxide which gives concrete its high alkalinity and an ill-defined calcium silicate hydrate (cement gel). By controlling the mix design and the amount of water added the more compactly the constituents will grow together thus increasing the matrix density and so producing a more durable concrete.

As concrete is strong only in compression and weak in tension, it is necessary to reinforce the matrix produced with reinforcing bars. The maintenance of integrity of the matrix and the stability of the re-bars is the purpose of protective coating.

### 4b Surface v. bulk

The definition preceeding does not wholly represent the surface to be coated as gradation uniformity does not exist. However, in coating we are concerned only with the *surface* presented or prepared. The bulk imposes secondary but important effects on the subsequent coating performance and is different in make up

from the surface.

The surface is very complex and this complexity varies in both chemical and physical terms. The surface will exhibit differences from the bulk in porosity, cement to aggregate ratio and therefore cement content, density, degree of admixture concentration including polymer additions and of physical additives, e.g. glass or polymer fibres etc.

It is necessary to understand the gradation nature as the initial adhesive bond at this interface is a major factor of coating longevity.

The bonding nature of a primer-coating is a result of:

(a) mechanical adhesion, i.e. interlocking

(b) chemical bond through interaction/valence (see Section 6 ‘Priming’)

Both of these should be used as concrete will present a non-ideal surface, hence preparation is wholly important to maximise on both effects.

Preparation may alter the gradation effects significantly and a coatings manufacturer often seeks to achieve a more ideal surface by removing perceived surface defects, e.g. the cement skin.

A ‘skin’ composition which is definably different from the bulk has been suggested by Researchers and Kreijger<sup>1</sup> proposes the following approximate structure:

Outer surface=cement skin (cement paste) of ca. 0.3%

Next surface=mortar skin (cement paste + sand, fines etc.) of ca. 14.2%

Next surface=concrete skin (ditto + aggregate) of ca. 85.5%

Next surface=concrete

This skin may vary from 5 mms thick and vary with factors such as the casting process and the rate and method of curing.

Blast preparation may remove the cement skin and some of the mortar; whereas wire brushing may only polish the cement skin surface whilst removing extraneous dust – two very different surfaces in terms of mechanical and chemical bonding.

Density and porosity effects are also variable and can necessitate a careful choice in priming or coating systems, these effects can be predominated by surface profile, i.e. roughness, but it would not be wise to assume mechanical adhesion is equally predominant or indeed preferred.

Relaxation may occur with adhesion loss if there is no penetration of the surface matrix by the primer or coating, with subsequent mechanical and chemical anchoring; consideration in this respect should be given to:

(a) is the concrete fresh (>30 days; <6-9 months)



(b) is the concrete aged (>6-9 months)  
(c) how was it placed/finished, e.g. power floated or vibro compacted.

This knowledge could dictate the selection of a more moisture tolerant system and penetrating primers in the case of fresh concrete or more compacted surface; and possibly solution coatings (self priming) or 'reactive' emulsion coatings on aged or less heavily densified surfaces.

#### 4c Micro and macro effects

Micro effects are noticeable on the surface, one such effect is porosity which is almost double that of the bulk due to much larger gradation of pore size. Surface pores can be measured as >10 microns at the surface, but only in hundredths of microns in the bulk. Micro pores resulting from a combination of gel pores and capillary pores are variable and accessible to solution primers or coatings. Capillary pores are some 250 times larger than gel pores.

A range of pore sizes is therefore presented and these are advantageously used in primer/coating penetration and locking. Conversely they are areas where water vapour from the bulk will condense prior to coating and subsequent permeation through the applied coating. A knowledge of the variability of the particular concrete may then dictate the choice of primer and top coat, requiring for example a high water vapour transmission for a fresh concrete with a porous surface in a damp environment, in order to maintain adhesion and equilibrium in use.

Macro effects of surface roughness, surface defects resulting from placement and coarse porosity from air pores (much greater in the top 2 mms); result in a secondary phase of wetting, penetration and adhesion.

An understanding of these combined properties of the surface and near surface allow a coatings manufacturer to apply the effects to good advantage.

In the micro pores a solution or small binder particle size will penetrate by capillary action, especially if water borne, capillarity is a significant driving force particularly in dry or near dry concrete. The number of pores that are penetrated will vary as they offer more or less physical resistance (size), and whether or not they already contain water, e.g. in the gel pores. A further complication is the net electro-negative charge of the walls of the pores, but this can be turned to advantage by reactive priming (see 'Priming').

In the macro pores, penetration is much easier and this is used to advantage by micro emulsion based coatings, the electro chemical barrier is

largely overcome, i.e. the coating binder is absorbed into the near surface.

These properties can be viewed in terms of sorptivity (the rate of absorption), the absorption itself (saturated value), permeability (rate of flow through a pre-saturated specimen) and porosity (weight difference between dry and water saturated concrete)

### 5 Preparation of concrete

#### General

This extremely important but often neglected area of involvement is the first major shift from the scientific understanding of the substrate and coating - via Engineer, Architect and Surface Coating Manufacturer - to the first wholly practical step by the contractor. Preparation will vary as the environmental placement and need to repair varies, but as most concrete falls into the air exposed category then preparation methods are generally definable. Speciality considerations such as the need to retain a dust free environment do dictate changes in method.

Preparation methods are:

(a) Sand or grit blast, bush hammer, needle gun - all of which physically alter the surface and leave a more open structure.

(b) Power washing, this is effective but less physically damaging and may leave behind a more cement paste rich surface depending on original friability.

It is assumed the selected methods will remove dirt which is surface lying or impregnated, algae and mould growth, surface curing agents, friable skin, cement paste in excess, oil/grease and other contaminants.

Methodology is well documented elsewhere, but it is worth adding that a mould contaminated surface should receive a treatment by a residually active Biocidal Wash to deal with deep spores not removed by preparation.

Testing - Testing for Preparation and/or repair should include the following basic diagnostic:

Visual - assessment of spalling, rust staining, cracking, dirt level, dampness, laitance, presence of joints and other details.

Physical - surface strength (scraping), soundness (hammer tapping)

Chemical - carbonation depth (phenolphthalein), chloride level (on or off site)

Electrical - concrete cover depth over re-bars (covermeter)

### 6 Priming

'Priming' and 'Stabilising' are often

interposed but are different perceptions.

Stabilising, as implied, is a penetration priming effect binding an otherwise phased surface to impart substrate stability, thereby offering a firm base for top coat(s). The stabilising solution will rely on as deep a penetration as possible into the skin and the solution binder will form an organic or inorganic polymer network within the matrix to hold the skin phases together; this occurs an evaporation of the carrying solvent. However, stabilising is only part of the requirement.

Priming suggests a surface borne effect with a prime objective of adhesion promotion for the top coat, equally this is not sufficient unless the primer also gains maximum adhesion.

Self priming coatings must rely more on the stabilising effect and they consist of solution or small size emulsion particle binders to gain the penetrative stabilising effect.

It is necessary to achieve the following objectives from priming:

the effect must be penetrative to offer a stabilising function.

the primer must also be surface lying to offer its structure to the top coat for bonding adhesion.

the primer must be able to accommodate pore electro-negativity.

pore water must be accommodated if present.

ideally take advantage of capillarity.

the primer must be able to even out an often diverse surface within a structure, i.e. concrete variations, repairs and existing coatings.

ideally offer adhesion to new or existing sealants and also bond to secondary structures to maintain edge integrity, i.e. window frames.

not restrict water vapour existing from a structure to the detriment of top coat W.V.T.

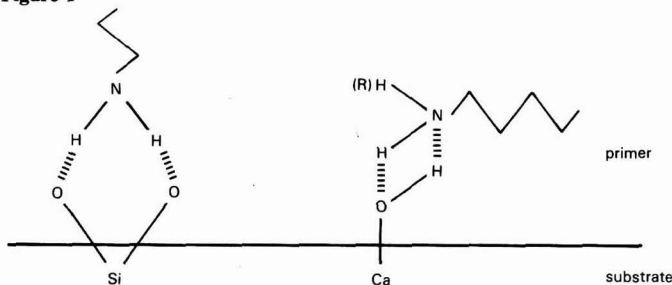
One such primer, fulfilling all these functions, is 'water dispersed epoxy', an outline of its functionality shows what can be achieved:

(a) One of the most important aspects of the 'water borne' epoxy primer is the overall 'cationic' identity which can be used to good advantage.

(b) The complex surface of concrete is electro-negative in character, and affords good opportunity for a water borne epoxy primer to adhere to the surface by electro-chemical effects. This can occur in several ways, e.g.

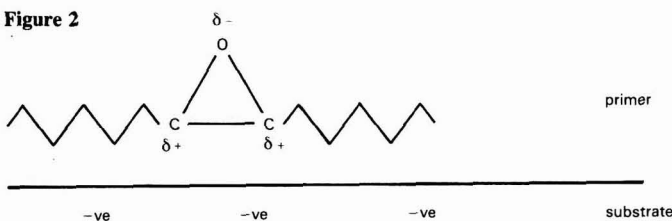
(i) At the interface some of the amine groups which have not reacted with the epoxide will be available for hydrogen bonding through the silicates and hydroxides on and in the near surface (Figure 1).

Figure 1



HYDROGEN BONDING THROUGH (COMPLEX) SUBSTRATE CALCIUM SILICATES.

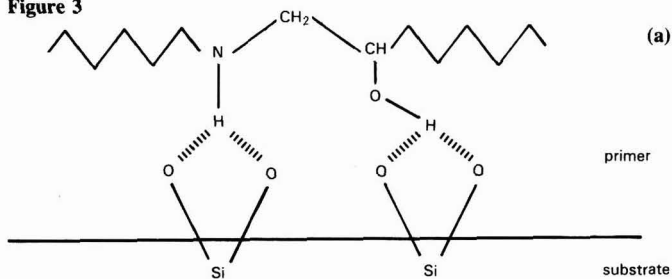
Figure 2



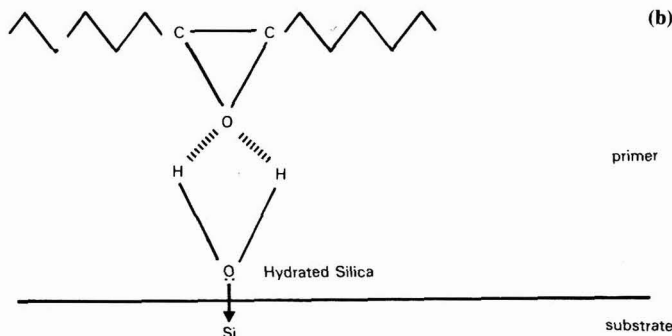
$\delta^-$  small -ve charge  
 $\delta^+$  small +ve charge

WEAK ELECTRO-STATIC BONDING

Figure 3



HYDROGEN BONDING VIA 'REACTING' EPOXY.



HYDROGEN BONDING THROUGH UN-REACTED EPOXIDE (IN EXCESS) ON A HYDRATED SUBSTRATE.

(ii) Electrostatic bonding through the epoxide group which is generally in excess (Figure 2).

(iii) The reacted epoxy can further hydrogen bond (Figure 3). This electrochemical and hydrogen bonding is enhanced by the hydrophilic nature of the substrate which allows thorough wetting and penetration by a water borne primer. The net effect is a physical key and chemical effect at and below the surface.

(c) A further function of such a primer is that it can be prepared with a rheology to allow application by common techniques of roller, brush or spray. A slight thixotropic nature ensures that the primer does not simply soak away but is both 'absorbed and surface lying' fulfilling a stabilising and priming function.

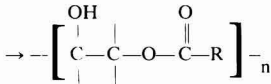
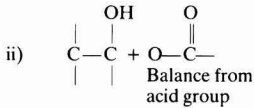
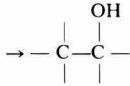
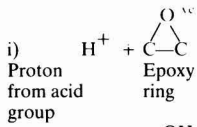
(d) Selective molecular size of the reactants and water base ensures, via low molecular size, a maximising of pore entry - in both capillary pore attraction (micro) and filling of pore defects (macro) to give an even surface. The water base accommodates existing pore water and raises damp substrate moisture accommodation well above solvent solution coating or primer tolerance, hydrophilic attraction is maximised.

Solvent based systems with more polar solvents can accommodate dampness and have the benefit of lower temperature use but may not accommodate subsequent condensation effects from high W.V.T. from within the substrate.

(e) A water based epoxy, being binder only, has no solids to inhibit absorption into the substrate; further they have relatively high W.V.T. rates, usually in excess of the top coats, and therefore do not encourage water blistering behind top coats. The unit with the lower W.V.T. - the top coat usually - is the arbiter of the rate of W.V.T. of the whole system, this is important when discussing meaningful figures. A recent determination shows a W.V.T. rate in excess of 30 grams per square metre per day for such a primer.

(f) Wetting or surfactant effects are present due to the molecular structure of the primer which is a long carbon chain with amine end groups.

(g) Top coat adhesion is gained by allowing an excess of epoxy resin. reactive top coat bonding occurs for example with carboxylic acid groups from some emulsion coatings - acrylics, P.V.A. or co and terpolymers. The epoxy ring opening is catalysed by protons from the carboxylic acid group and a second stage cross link then occurs at the interface:



Cross-linked polymer at interface.

Thus the concrete surface is primed with a system that gives adhesion for a wide variety of top coats.

## 7 Coatings for protection

### 7a Types available

Table 1 lists the 'principal' types available and shows coatings rather than penetrants. The properties listed illustrate the potential for priming (sealer) and top coating.

### 7b Parameters

From this list (plus others not listed), a choice must be made and weighed against the protective functions defined thus far, and the coating properties.

It is then necessary to set parameters for the coating to achieve. Table 2 shows one set of parameters for a coating proven in use both for the

protective function, and which fulfills the criteria of primer adhesion and long term weathering, in this case a modified S.B.R.

The specifier should check these basic parameters, question the method used for testing and if case histories exist.

To these parameters should be added the other important film functions such as permanent elasticity; or sufficient elasticity retention on aging to cope with substrate expansion and contraction overall. Measurable adhesion, direct or with the primer, is essential to allow comparison of film cohesion retention where the structure is expected to continue cracking, e.g. on concrete roofs. An ability to self clean via a small controlled degree of chalking is also useful, as the surface is not prone to a build up of surface contaminants which may otherwise reduce efficiency, i.e. reduced reflectance, increased water contact, provide nutrient for micro-organisms proliferation etc.

Table 1

Functional Type	Physical Forms	Cure Mechanism	Structure Available	
(1) Acrylic	Dry ▷ Sealer ▷ Coating	Wet ▷ Solvent (Sol <sup>N</sup> ) ▷ Solvent-Catalysed ▷ Aqueous	▷ Loss of Solvent ▷ Loss of Solvent + Chemical ▷ Drying	▷ Clear/Pigmented ▷ Hard or Elastomeric
(2) Alkyd	▷ Sealer ▷ Coating	▷ Solvent ▷ Aqueous	▷ Loss of Solvent/ Oxidation	▷ Clear/Pigmented ▷ Hard or Flexibilised
(3) Bituminous	▷ Coating	▷ Solvent ▷ Aqueous	▷ Loss of solvent ▷ Drying	▷ Pigmented ▷ Flexible
(4) Chlorinated Rubber	▷ Sealer ▷ Coating	▷ Solvent ▷ Aqueous	▷ Loss of Solvent ▷ Drying	▷ Pigmented ▷ Flexibilised
(5) Epoxy Resin	▷ Sealer ▷ Coating	▷ Solvent ▷ Solventless ▷ Aqueous	▷ Loss of Solvent/ Chemical ▷ Chemical ▷ Drying/Chemical	▷ Clear/Pigmented ▷ Hard or Flexibilised
(6) Oleoresinous	▷ Coating	▷ Solvent	▷ Loss of Solvent/ Oxidation	▷ Pigmented ▷ Hard or Flexibilised
(7) Polyester	▷ Sealer ▷ Coating	▷ Solvent ▷ Solvent	▷ Loss of Solvent/ Chemical	▷ Clear/Pigmented ▷ Hard or Flexibilised
(8) Polyethylene Copolymers (C.S.P.)	▷ Coating	▷ Solvent ▷ Aqueous (rare)	▷ Loss of Solvent ▷ Drying	▷ Pigmented ▷ Elastomeric
(9) Polyurethane	▷ Sealer ▷ Coating	▷ Solvent ▷ Solventless ▷ Aqueous	▷ Loss of Solvent and Chemical (or Moisture) ▷ Chemical ▷ Drying/Chemical	▷ Clear/Pigmented ▷ Elastomeric or Flexibilised
(10) Styrene Acrylic	▷ Sealer ▷ Coating	▷ Solvent ▷ Aqueous	▷ Loss of Solvent ▷ Drying	▷ Clear/Pigmented ▷ Elastomeric or Flexibilised
(11) Synthetic Rubber (S.B.R.)	▷ Coating	▷ Aqueous	▷ Drying	▷ Pigmented ▷ Elastomeric
(12) Vinyl Acetate Copolymers	▷ Sealer ▷ Coating	▷ Solvent ▷ Aqueous	▷ Loss of Solvent ▷ Drying	▷ Clear/Pigmented ▷ Elastomeric

Resistance to alkali is essential from both primer and top coat, damp concrete will undergo a build up of alkaline salts at the substrate coating interface due to water moving them by a process of condensing (dissolving) and subsequent diffusion to the surface via absorption/desorption.

**7c Testing – general**

Table 2 shows an ideal set of basic parameters. Table 3 (ref. 2) shows the properties that can be measured and the reference standards used. Arguably all these can be considered and Sections A, B, E cover the protective function. Section C may also be predominant in some circumstances. Sections D and F are specific to the placement of the structure, should for example gloss or flash point be the prime considerations.

**7d Testing – specific to protection**

A coating which is resistant to carbonation may also be expected to resist other external gaseous diffusion. A coating which is waterproof will resist penetration by water borne corrosive ionic species, e.g. chloride ions from salt, also acidic solutions of gases from pollution and other industrial chemicals. Halting water ingress will also stop A.S.R. from proceeding.

'It is worth stating that coatings are limited in their effect to further preventing decay which may already be formed, not reversing it in the truest sense.'

One area of continued evolution in testing is in the method(s) used in the evaluation of carbon dioxide diffusion resistance, some consider this property to be the most important in preventing the main corrosion mechanism in reinforced concrete structures – that of carbonation.

This corrosion is the formation of calcium carbonate by the reaction of carbon dioxide with the calcium

**Table 2**

Parameter	Result
○ Film Build (Minimum) D.F.T.	350 Microns
○ Carbon Dioxide Diffusion Resistance (Taywood Method)	R=261 Metres (1)
○ Carbon Dioxide Diffusion Resistance (Engelfried/Klopfer Method)	S <sub>D</sub> =1820 Metres (2)
○ Water Vapour Diffusion Resistance to Din 53122 (Engelfried/Klopfer)	S <sub>D</sub> =0.77 Metres (3)
○ Tensile Strength (ASTM D.412) (Unreinforced)	1.52 M.Pa
○ Tensile Strength (ASTM D.412) (Various reinforcements)	7-14 M.Pa
○ Ultimate Elongation (ASTM D.412) (Unreinforced)	420%
○ Ultimate Elongation (ASTM D.412) (Reinforced)	25-50%
○ Freeze/Thaw Resistance – 50°C to +100°C	No Effect
○ Daylight Reflectance (Diffuse)	86%
○ Sunlight Reflectance	98.6%
○ Bond Strength	1.5 to 2.5 M.Pa

- (1) Carbon dioxide/helium – 'steady state' flow method.
- (2) Carbon dioxide/air – partial pressure desiccant flow method.
- (3) From diffusion resistivity (μH<sub>2</sub>O) for differential R. H. of 100% → 50%.

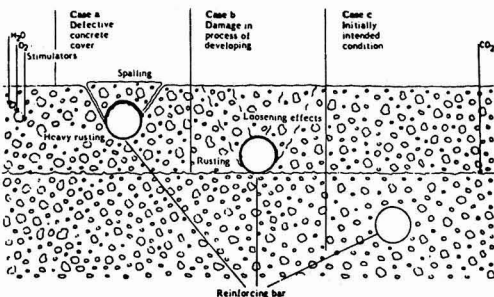
hydroxide contained within the concrete. This gaseous phase attack is exacerbated by dissolved CO<sub>2</sub> in water, especially in porous concrete viz: H<sub>2</sub>O + CO<sub>2</sub>=H<sub>2</sub>CO<sub>3</sub>, the carbonic acid reacts further with calcium carbonate – CaCO<sub>3</sub> + H<sub>2</sub>CO<sub>3</sub>=Ca(HCO<sub>3</sub>)<sub>2</sub>, the calcium hydrogen carbonate is soluble and further water/carbonic acid dissolves it away, leaving the pores open for further carbonation reaction. During a dry phase the Ca(HCO<sub>3</sub>)<sub>2</sub> dissociates back into CaCO<sub>3</sub>, the CO<sub>2</sub> thus released starts the carbonation cycle again, penetrating ever deeper. Eventually alkalinity is lost, the re-bars can corrode in the presence of oxygen

and water at a pH value = 8; the presence of SO<sub>2</sub> and Cl<sup>-</sup>ion furthers the process. The net effect of corrosion is an expansion (x2.5) over the non-rusted bar resulting in internal pressure which cracks and spalls the concrete cover.

Figure 4 illustrates the damage potential. Figure 5 shows how a coating with sufficient resistance will reduce the carbonation depth.

The methods for determining diffusion resistance and their subsequent expression is worth explanation, as much current debate is underway to find a truly practical method which substantiates results found in practice. In the preceding

**Figure 4**  
Damage occurring as a consequence of carbonation (Engelfried)



**Figure 5**  
Effect of coatings on the progress of carbonation

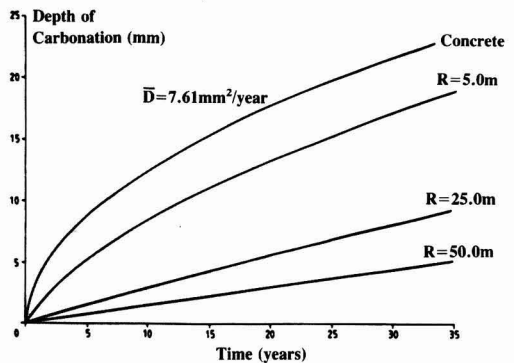


Table 2, the CO<sub>2</sub> diffusion resistance (Engelfried) is expressed as an Air Layer Thickness (Diffusion Resistance); this has been experimentally arrived at and is a comparison method against a known figure for effective resistance to carbon dioxide where a

figure of 50 Metres of Air Layer Thickness is set as the minimum; this is in respect of prevention of corrosion of the reinforcing bar by carbonation. The figure for the coating is arrived at by the following method:  
Step 1: The weight of CO<sub>2</sub> diffused in

unit time is measured and from this the diffusion resistance coefficient ( $\mu$ ) is measured:

$$\mu = \frac{D_L \cdot c'}{J \cdot s}$$

**Table 3**

**Certification and performance testing of coatings for reinforced concrete**

PROPERTY	TEST
<b>A Protective properties</b>	
1. Water vapour transmission	ASTM D1653, ASTM E96-80, BS 4315, BS 3177, DIN 52615, MOAT 24:1983
2. Water penetration resistance	MOAT 24:1983, BS 3424, RILEM CPC 11.3, BS 1881P5 (ISAT), BS 6477, Bundesmann Showerproof Test
3. CO <sub>2</sub> permeability	BRE, PRA, Klopfer, Poly.S.Wales, Taywood
4. Water absorption	MOAT 24:1983
5. Chloride ingress	TRANS. RES. BOARD.244, Whiting
<b>B Durability</b>	
1. Natural weathering	BS 3900/F6, ASTM D4141, DIN 53166
2. Artificial or accelerated weathering	BS 3900/F3, F4, F2, ASTM D1654, G53-77, Defence Spec. DEF/1053
3. Salt spray resistance	Defence Spec. DEF/1053, BS 3900/F4, F12, DIN 53167, ASTM B287, G43, B368, B117
4. Frost-salt resistance	Swiss Nat. Lab., Trans. Research Board
5. Freeze/thaw	Klopfer (Dortmund)
6. Ageing (accelerated heat)	BRS Digest No. 51
7. Temperature cycling	
8. Temperature stability	BS 3900/G7, DIN ISO 3248
9. Resistance to industrial pollutants	BS 3900/F8, G5, DIN 50018
10. Humidity resistance	BS 3900/F2, ASTM D2247
11. Resistance alkali (saponification)	MOAT 24:1983, BS 3900/F1
12. Lightfastness	ASTM D2620, BS 3900/F5
13. Chalking	ASTM D659, DIN 53159, BS 3900/H6
14. Water swelling	BS 3900/F2, ASTM D2247
15. Resistance to wind driven rain	FED. SPEC. TT-C-555B
16. Resistance to hydrostatic pressure	FED. SPEC. TT-P-1411A
<b>C Fire properties</b>	
1. Fire resistance	BS 476 pt 8
2. Surface spread of flame	BS 476 pt 7
3. Ignitability	BS 476 pt 5
4. Fire propagation index	BS 476 pt 6
<b>D Toxicity</b>	
1. Effect on potable water	
2. Bacterial and fungal attack	ASTM D3456-75, ASTM D3274-76
<b>E Performance properties</b>	
1. Elongation/elasticity	TRRL LR636, ASTM D412-68, D1642-70, D522-60, D1737-62, D2370-68, BS 903 Pt.A2
2. Tensile strength	ASTM D412-68, D2370-68, BS 903 Pt.A2
3. Bend Test	BS 3900/E1 & E11, ASTM D1642-70, D522-60
4. Tear strength	ASTM D470-68, BS 2782 Pt.3-360B
5. Adhesion	ASTM D429, D2197, MOAT 24:1983, BS 3900/E6 & E10, BS AU 148/3, DIN 53151, DIN ISO 4624
6. Abrasion resistance	Taber. DIN 53516, ASTM D658, D968, D4060, DIN 65183, BS AU 148/A
7. Scratch resistance	BS 3900/E2
8. Impact resistance	BS 3900/E3, E7, MOAT 24:1983, TRRL LR636
9. Non-slip	TRRL Road Note 27
10. Chemical resistance	BS 3900 GROUP G
11. Crack-bridging ability	MOAT 24:1983, TRRL LR636
<b>F Physical properties</b>	
1. Viscosity	Brookfield R.V.T., BS 3900/A7
2. Hardness	BS AU 148/6, ASTM D1474, D2134, D3363
3. Specific gravity	ASTM D1475
4. Weight % solid	ASTM D1353, BS 3900/A10 & B2
5. Drying time	ASTM D1640, BS 3900/C2 & C3
6. Flash point	Penskey-Martens Closed Cup
7. Gloss-specular reflection	DEF 1053, BS 3900/D5, ASTM D523-80
8. Pot life	
9. Coating thickness	BS 3900/C5, ASTM D4138
10. Cure time	BS 3900/C1, C2, C3



## Have you paid?

One of the first fruits of the Association's computerization programme, apart from more legible address labels, has been that it is now much easier to identify those members in arrears with their membership subscriptions and to make contact with such members, either directly from headquarters or through the good offices of the Sections. As a result a high proportion of late payers were persuaded to maintain their membership in 1988 and in some cases members in financial hardship had been assisted by the granting of concessionary rates of membership.

For the 1989 subscription year and again for the first time, subscription reminder notices were produced on the computer and sent to all members world-wide, during November 1988. These new style subscription notices have met with a good response from members, many of whom paid their subscriptions by the year end or early in 1989.

However, there is still a significant number of members who haven't remitted their membership subscriptions for the year, but who at the same time have been able to benefit from membership, including the receipt of JOCCA. If you are amongst this number, dig your subscription notice out from the back of the drawer, from your briefcase, or behind the clock, and send your membership subscription as soon as possible. The Association depends on the early receipt of subscription income and from the interest earned on the money received to subsidize activities of the Association which otherwise would have been financed through higher subscriptions. The OCCA subscriptions still represent very good value for money and now that VAT has been removed from subscriptions, many members will be paying less than in 1988. A significant number of companies pay membership subscriptions for their employees and it may be that your notice is sitting on

your accountant's desk. If you have lost or mislaid the notice, don't worry, a telephone call to Priory House will ensure that a duplicate notice is sent by return.

Some members may find themselves in financial difficulties following redundancy or illness. If you are in these unfortunate circumstances please write to me. Your letter will be dealt with in strict confidence and I am sure the Association can assist in helping you to meet the cost of continuing membership.

## OCCA and the PRA

The Association and the PRA have combined in the organisation and promotion of a recent symposium on the topical subject of "The Role of Surface Coatings in Fire Protection". The two day symposium attracted an audience of 115, many of whom would not traditionally have attended a surface coating meeting. The majority of the technical papers will be published in special issues of the Journal, commencing with the May issue which will be devoted to the subject of "Fire Retardant Coatings". Other papers will feature in future issues "Paint and the Environment" (June), "Marine and Offshore Coatings" (October) and "Wood Finishes" (November).

The successful collaboration of the two organisations in the joint organisation of the symposium is likely to be mirrored in further OCCA events with other bodies, with Printing Ink and Marine Coatings being the subjects of future symposia. The Association welcomes joint activities with other organisations and will continue to promote such activities.

## Paintmakers Association — Industrial Coatings Conference

London's Hyde Park Hotel was the venue for the third Industrial Coatings conference organised by the Paintmakers Association.

The theme of the Conference was "1992 — The Challenge Before and After". Delegates were treated to a somewhat sober presentation on the challenges and problems presented by the Single European Market in 1992. Papers by Eileen Harding of the DTI and consultant James Dudley described the assistance the Government was providing to enable companies to rise to the challenge of the Single Market and outlined the strategic considerations that companies needed to address in advance of 1992.

A stimulating presentation by Dr

Frank Rose of ICI considered the problems facing companies by increasingly demanding health and safety and environmental legislation coupled with pressure from consumer and Green interests. Dr Rose challenged the Association to regard such legislation not so much as a burden but as an opportunity. The challenge of the legislation was for companies to become involved with environmental matters, to pre-empt the Green lobby and by cutting costs and maximizing efficiency, to minimize the potential impact of new environmental legislation on profitability.

The recurring plea of the presenters was that it was up to companies to get involved in European and environmental matters by representation on appropriate committees and, perhaps more importantly, by ensuring that their representatives were the very best the company could nominate.

The morning session concluded with a report by PA Director Michael Leveit on the organic changes that were proposed for the Association to meet the challenge of Europe.

The afternoon session was devoted to presentations by Ian Hamilton-Fazey of the Financial Times and Martin Simons of Salomon Brothers. The structure of the world paint manufacturing industry was reviewed with particular emphasis on the differences between marketing activities in the USA, Europe and Japan. The financial strengths (and weaknesses) of the UK paint industry were assessed in detail and some interesting suggestions made to improve financial performance.

Although the Conference emphasized the challenge and difficulties presented by 1992, it also stressed the opportunities presented by the Single Market, opportunities that the United Kingdom industry was fit and lean enough to respond to

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## London Section

### Tall oil rosin derivatives for printing inks

The fourth technical meeting for the 1988/89 session was held on Thursday, 16 January 1989, at the Naval Club, Hill Street, Mayfair. Mr Hanu Alalauri of Veitsiluoto Oy gave a paper entitled "Tall Oil Rosin Derivatives for Printing Inks".

The speaker first explained that tall oil fatty acids were a byproduct of the paper making industry. Having outlined their origins, physical and chemical comparisons were then made with Gum and wood rosins. Being of Vegetable origin it was a renewable source of raw material. Whilst much of the 100 to 120 thousand tons produced annually was used in paper manufacture, an increasing amount was now used as a raw material for printing ink resins.

To produce satisfactory printing ink resins the melting point and solubility characteristics of the tall oil acids have to be modified. Reacting with Maleic Anhydride gives a resin of higher melting point with good solubility in polar solvents. Dimerisation or modification with Phenol Formaldehyde and Substituted Phenol Formaldehyde gives resins of high melting point with good solubility and solvent release.

After a lively question and answer section the audience went on to enjoy a buffet and bar sponsored by Cornelius Chemicals.

N. C. Locke ■

## Manchester Section

### Phthalocyanine pigments

The February technical meeting was held at the Mechanics Institute, Manchester, on Monday 6 February. The meeting was attended by 102 members and guests, and a paper entitled "Phthalocyanine Pigments—Recent Developments", was presented by Mr R. J. Cross, the Technical Marketing Manager of ICI.

Phthalocyanine pigments have been commercially available for

about 50 years, technically they are near perfect pigments, inexpensive, and are the principal blue and green pigments for the colouration of paints. Phthalocyanine pigments are made from relatively simple ingredients, the chemistry is straightforward, but the chemical engineering is challenging.

Phthalocyanine blue shows polymorphism and the different crystal forms were described, the two commercially important forms being the reddish blue alpha form, and the greenish blue beta form.

The crude crystalline (nominally alpha form) is tinctorially dull and weak, further processing by either dry salt milling or pasting with concentrated sulphuric acid is used to produce a satisfactory pigment of the alpha form. For the beta form, an active solvent such as glycol is added.

The beta form is the stable form, the alpha form in the presence of heat or strong solvents re-crystallises into the beta form. Phthalocyanine blue pigments are prone to flocculation, and the solution of these historical problems of crystal stability and flocculation has given the familiar range of blue pigments.

The major users of phthalocyanine blue pigments are the Paint, Plastics and Printing Ink Industries, each has its own requirements, which were described, and the different pigments required for each industry outlined.

Other forms of phthalocyanine blue are available, these being the Gamma and Epsilon forms, and also metal-free phthalocyanines with similar polymorphism to copper phthalocyanine.

Crude copper phthalocyanine green is made by the halogenation of crude copper phthalocyanine blue, the level of chlorination and bromination determining the shade. Unlike the blue pigment phthalocyanine greens do not show polymorphism, and are resistant to flocculation. The crude pigment is refined by salt milling or a solvent pigmentation process.

The different requirements for different industries were described, and the various grades of pigment for each outlined.

The talk was concluded with the lecturer outlining the areas where phthalocyanine pigments are unsuitable, and outlining future trends.

After a short question and answer session, the vote of thanks was proposed by Martin Hill, and those present were able to participate of a buffet sponsored by ICI.

M. G. Langdon ■

## Natal Section

### Iron oxide

The most boring questions are . . . What is new in Iron Oxide? Have the innovations for iron oxides in the paint industry been exhausted?

It is very difficult to have something new to say every year but Dr G. von Szakowski of Bayer at an OCCA Natal meeting held on 27 September 1988 in Pinetown, managed to surprise us. He told us that Bayer is improving its range of iron oxides and he commented on these new ideas and developments.

The following are two important ones:

1. some years ago, iron oxides were micronised.
2. flocculation is reduced, ease of dispersion is improved and a better gloss is obtained by after-treatment coating the pigments with alumina and silica. This is similar to the titanium dioxide industry.

Dr von Szakowski illustrated his lecture with numerous slides showing Florida and accelerated exposure results which demonstrated the superior properties of the development products. The lecturer then concluded his talk by answering questions from the floor.

Mr K. M. Engleburt proposed the vote of thanks both to Dr von Szakowski for an excellent presentation and to Bayer who were the host for the evening.

### New wet bead mill

Hans Burri Sales Manager of Stag Bulk Equipment (Pty) Ltd presented an interesting lecture illustrated with slides and a video

tape to OCCA Natal in Durban on 19 October 1988.

The universality of the Fryma CoBall®-Mill is shown by its acceptance in the Paint, Varnish, Chemical, Pharmaceutical and Food Industries.

The Paint Industry is mainly concerned with dispersion of the pigments and therefore the pigments must be of the correct particle size. A thorough pre-mix is first carried out. Dispersion in the mill is created by the gap between the stator and the rotor. The mill is symmetrical in design and has no dead spots. It works well with both low and high viscosity materials including highly concentrated pigment pastes with non-newtonian flow. Wear in the mill is minimal. Change of colour is easily carried out as the small volume of the mill requires very little cleaning solvent. The mill is user-friendly.

A member of the audience confirmed its practical use for a colorant dispersion. The number of passes was reduced from 6 in a sand mill to 2 in the Fryma CoBall®-Mill. Mr Burri said that the price of the mill in spite of duties and surcharges compares favourably with the sandmill.

Mr Richard John proposed the vote of thanks to Mr Burri and also thanked Stag Bulk Equipment (Pty) Ltd for their hospitality.

*E Puterman* ■

## Newcastle Section

### Emulsion paint rheology modifiers

Thirty-five members and visitors attended St Mary's College, University of Durham, on 12 January 1989, for the fourth meeting of the 1988/9 session at which Mr Brian Jolly of Tioxide Chemicals gave a presentation on "Tilcom Products as Rheology Modifiers for Emulsion Paints".

Mr Jolly introduced Tioxide Chemicals as a division of Tioxide International specializing in titanium and zirconium organic complexes which have a variety of uses, such as leather tanning or UV absorbers for sun creams; he proposed to talk about titanium organic complexes as rheology modifiers in emulsion paints.

Their use in emulsion paints produces a good balance of properties, such as attractive in-can appearance and good anti-settle characteristics, together with improved brush loading, easier application with low roller spatter, thicker applied coats with good sag resistance, yet also with good flow and levelling. In practice, the in-can structure produced can vary from very slight to full gel which breaks down readily with stirring or with brush/roller application.

Mr Jolly briefly discussed the chemistry involved, showing a typical diethanolamine titanate structure, pointing out the necessity to stabilize the molecule with covalently bonded alcohol or glycol moieties to prevent rapid hydrolysis in aqueous systems. He showed how the addition of such complexes to colloid-stabilized emulsion resin results in controlled hydrolysis of the stabilizing groups in the complex and the formation of hydrogen bonds between molecules of residual complex and the cellulose thickener/stabilizer incorporated in individual resin particles. Such weak bonding produces obvious rheological structure, characterised by much higher measured gel strength and medium-shear viscosity, but with little effect on high-shear viscosity. This structure develops on standing, unlike that produced by bentonite clays, and can be adversely affected by excessive shear after formation; consequently the recommended method of incorporation is at the end of the manufacturing process, immediately prior to filling. Mr Jolly emphasised that titanium complexes are only effective where the resin used is colloid-stabilized and that they have little effect on the structure produced by hydroxyethyl cellulose added to the paint itself.

With a series of slides he demonstrated differences produced in matt emulsion paints by the most commonly used alkanolamine titanate (Tilcom AT33) in combination with emulsion resins stabilized by various thickener types - hydroxyethyl cellulose, hydrophobically modified hydroxyethyl cellulose, hydrophobically modified ethoxylated urethane and

carboxylated acrylic (alkali soluble). As expected, gel strength and medium shear viscosity were increased by titanium complex addition, with little effect on high shear viscosity for all except the carboxylated acrylic, which showed significant increase. In silk finishes based on the same emulsion/colloid systems, gloss and contrast ratios were unaffected by complex addition, but roller spatter on application was reduced. It is noteworthy that, in all of the slides shown, the superiority of the HME urethane and the carboxy acrylic over the HEC types in application rheology was clear cut, especially with titanium complex.

Mr Jolly then discussed work done for various companies in tropical climates to reduced the level of added cellulose thickener levels in emulsion paints. Laboratory work showed that gel strength and medium shear viscosity could be maintained at half the original HEC level with the use of Tilcom AT33: moreover the washability of the resultant paint film improved, probably as a result of crosslinking of the Tilcom complexed with the resin particle/thickener after dry-out.

He concluded with slides showing the effect on structure development of time, incorporation technique, manufacturing temperature and shear, with three different Tilcom agents. From these it was clear that the level of Tilcom used can lie between 0.1 and 1.0%, depending on the structure needed and that it should be incorporated as quickly as possible, avoiding excessive shear and temperature rise; the product should be filled before structure development necessitates re-shearing, with consequent loss of some structure. Loss of structure on re-shearing is due to breaking of the complex/thickener weak bonding, allowing the complex to react with other materials which do not contribute to structure development in the process.

Questions afterwards concerned the possibility of using titanium complexes to protect colloids against enzymatic breakdown and consequent paint thinning, cost effectiveness with the Tilcoms available, delayed structure development in paint before filling.

*Continued on p.149*



## Scottish Section Annual Dinner Dance



Top table guests at the Scottish Section Annual Dinner Dance held at the Hospitality Inn, Glasgow. Back row (L to R): Mr J Hemmings, Chairman West Riding Section; Mr S Lynn, Chairman Newcastle Section; Mr B Myatt, Chairman Midlands Section, Mr C Pacey-Day, General Secretary and Guest of Honour, Dr S Lawrence, Chairman Scottish Section; Mr C Shaw, Chairman Bristol Section; Mr R Hill, Social Secretary Scottish Section. Front row (L to R): Mrs Hemmings, Mrs Lynn, Mrs Myatt, Miss Lorden, Mrs Lawrence, Mrs Shaw and Mrs Hill.

### Professional Grade

At the meeting of the Professional Grade Committee held on 15 February 1989 the following admissions were made:

#### Admitted to Fellowship

North, Albert Graham (*London*)  
Taylor, Alexander Legg (*Irish*)

#### Upgraded from Associate to Fellowship

Ekeh, Godwin Chukoemeka (*General Overseas - Nigerian Branch*)

#### Admitted to Associateship

Stanfield, Robert Frederick (*Manchester*)  
Swart, Nico Johann (*Cape*)  
Langdon, Michael George (*Manchester*)

#### Admitted to Licentiatehip

Evans, Jess (*Midlands*)

### New Members

The sections to which new members are attached are shown in italics together with the country, where applicable.

### Ordinary members

Bittner, A. (*Manchester*)  
Brooks, S. M. (*Manchester*)  
Chauhan, B., PhD (*Ontario*)  
Hammerson, D. G. (*London*)  
Heathcote, J. E., BSc (*Midland-Trent Valley*)  
Kedia, S. K. (*General Overseas - India*)  
Kingston, E. A. (*London*)  
Solodar, W. (*General Overseas - USA*)  
Von Berg, G. F. (*Natal*)

### Registered students

Denn, M. (*London*)  
Goodwin, R. J. (*Scotland*)  
Livesey, P. A. (*Manchester*)

### Amended December 1988 list of Approved affiliated body—OCCA New Zealand

### Fellows

Aitken-Smith, Frank Joseph  
Caldwell, David George  
Gooch, Colin  
Hackney, Thomas  
McKean, James Newlands  
Sharp, Peter Frank

Slinn, Thomas Walter  
White, Robert Arthur  
Whitfield, Thomas

### Associates

Assink, Jo  
Bluck, Ross Steele  
Clark, Michael Denis Thomas  
Dalzell, Kerry Wayne  
Eltringham, James Norman  
Foster, Alan Howard  
Fry, Jack Ian  
Greenall, Brian John  
Hopkins, Francis Glenn  
Horsfall, Anthony John  
Jenkins, Brian Gordon Allan  
Jones, Geoffrey Peter  
Leathley, George Derek  
McCapra, Ronald  
McFetridge, John Henry  
Ness, Robert Alexander  
Provan, Andrew Wilson  
Ronson, David James  
Rowlands, Michael Peter John  
Sewell, Anthony Robert  
Sheath, Alan  
Spargo, Robert  
Stephenson, Robert Perry  
Thorburn, Alan McGregor  
Vickers, James Edward  
Wagenvoort, Marinus  
Walters, Peter  
Wright, Graham Leslie

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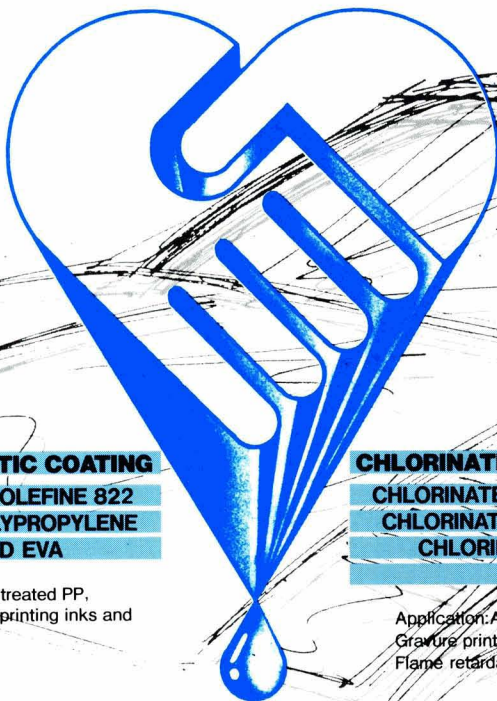
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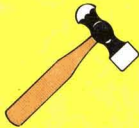
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