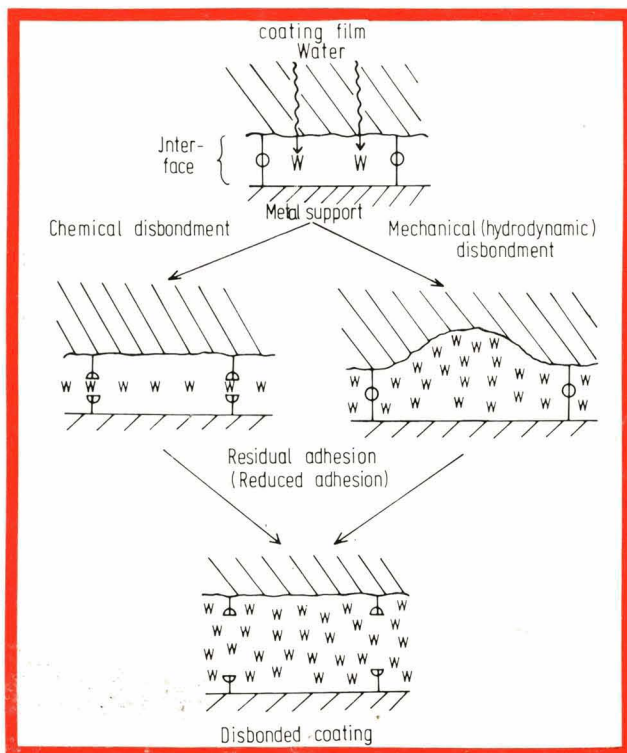
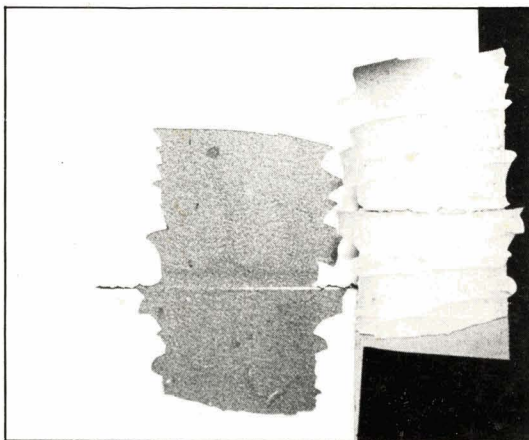




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WATER DISBONDMENT AND WET ADHESION OF ORGANIC COATINGS ON METALS

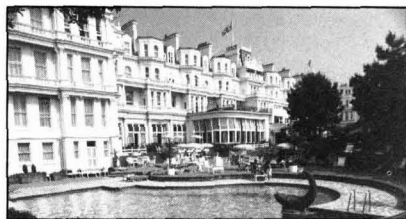


OCCA Conference 1987 Eastbourne



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17 - 20 JUNE, 1987



“ADVANCES AND APPLICATION OF SCIENCE AND TECHNOLOGY IN SURFACE COATINGS”

Lecture Programme

The Technical Sessions will take place in the Devonshire Suite at the Grand Hotel on Thursday and Friday, 18 and 19 June. The Honorary Research and Development Officer (Mr J. R. Taylor) has arranged the following lectures for the Conference:

THURSDAY, 18 JUNE

Session I (9.00 a.m. — 12.15 p.m.)

Chairman: Mr J. R. Taylor (Honorary Research and Development Officer, OCCA)

Speakers: Address of Welcome by the President, F. B. Redman
Keynote Address by Ing. Arja Saloranta (President, SLF)
Dr A. Wilson and Dr J. W. Nicholson (Laboratory of the Government Chemist) — “Preparation of Ionomers for Coatings from Water Soluble Polymers”
Mr R. H. E. Munn (Cray Valley Products) — “Novel Polyamide Type Epoxy Curing Agents”
Mr Thor Fjeldberg (Dyno Industrier AS Norway) — “Alkyd Emulsions, Properties and Applications”
Dr H. Warson (Solihull Chemical Services) — “Recent Advances in Crosslinking and Curing Applications to Surface Coatings”

Session II (2.00 p.m. — 4.30 p.m.)

Chairman: Mr G. W. Fowkes (Vice-President, OCCA)

Speakers: Ilkka Sarvimaki (Tikkurila OY) — “A Novel Water-Based Coating System for Wet Areas” (*Paper presented on behalf of SLF*)
Mr J. Spauwen (Dow Chemical Co Ltd) — “New Propylene Glycol Ethers for Water Borne Coatings”
Mr D. S. W. Dargan and Mr J. Hemmings (Kirklees Chemicals) — “High Binders in Decorative Emulsion Paints”
Mr L. Cutrone and Mr D. V. Moulton (Tioxide Ltd) — “The Reliability of Durability Testing”
Mr A. C. D. Cowley (ICI Organics Division) — “Improving Dispersion of Pigments with Hyperdispersants”
Mrs N. Usman (Paint Research Association) — “Development of Novel Driers for Paint”

FRIDAY, 19 JUNE

Session III (9.00 a.m. — 12.15 p.m.)

Chairman: Mr J. Bernie (Director, Paint Research Association)

Speakers: Keynote Address by Mr G. Phillips (Managing Director, Ault and Wiborg PLC)
Dr W. G. Erskine (Domino Amjet Ltd) — “Advances in Ink Jet Printing and Ink Jet Inks”
Mr P. C. Stievater (NL Chemicals Inc) — “Advances in Environmentally Acceptable Polyurethanes” (*Paper presented on behalf of FSCT*)
Professor Mengies (Loughborough University) — “Rapid Electrodeposition Systems for Metallic Coatings”
Mr P. Fallon (Johnson and Bloy Ltd) — “Advances and Changes of Ink Technology in Cold Seal Packages”

Session IV (2.00 p.m. — 4.15 p.m.)

Chairman: Mr G. J. Gough (President, Birmingham Paint, Varnish and Lacquer Club)

Speakers: Dr H. Robinson (Taylor Woodrow Engineering Ltd) — “Durability of Anti-Carbonation Coatings”
Mr M. Leclercq (Societe des Mines et Fonderies de Zinc de la Vieille Montagne) — “Production of Zinc and Zinc Alloyed Dusts by Fine Atomisation”
Dr B. L. Kaul (Sandoz Huningue SA, France) — “Advances in the Science and Technology of Pigments”
Dr N. Henwood (Dow Chemical Co Ltd) — “Membrane Separation for the Production of Nitrogen Enriched Inert Gas”
Mr I. Tonini (Walter Maeder AG) — “Technical Progress of New Ecologically Safe Paint Systems and Application Technologies” (*Paper presented on behalf of FATIPEC*)

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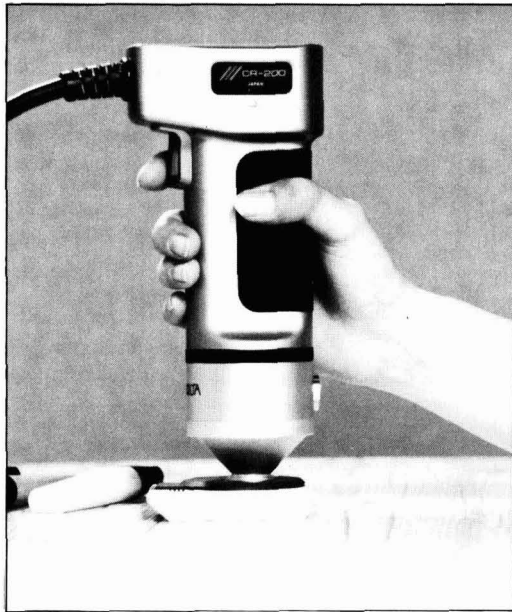
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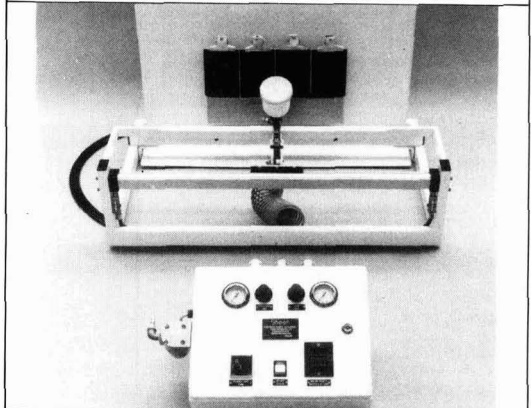
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Water disbondment and wet adhesion of organic coatings on metals: A review and interpretation

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Abstract

The available literature on water disbondment is reviewed and a hypothesis is presented. Water disbondment is a consequence of the formation at the metal/coating interface of a discontinuous or, in some cases, a continuous water film several to many molecular layers in thickness. Water migrates into the coating both through the compact polymer phase and through capillaries or pores in the coating. The driving force for directional water transport through the coating to the interface is diffusion under a concentration gradient. Osmotic force, temperature differences, and chemisorption or physisorption of water on the metal oxide at the interface are among the mechanisms that may lead to accumulation of water at the interface. Disbondment of organic coatings by water is usually considered to be initiated by a direct interaction of water molecules with adhesive bonds at the metal/coating interface. However, it is likely that non-bonded areas exist at this interface where water molecules may accumulate. These water volumes grow laterally along the metal/coating interface because of the continuing condensation of water molecules. Lateral disbondment occurs because of the stress caused by water condensation at the interface. The peeling stress exceeds the adhesive bond strength. If lateral growth of the water layer is prevented by corrosion product formation or by strong adhesive bonds, localized blistering rather than water disbondment occurs. Methods that are used to reduce the severity of water disbondment are summarized. Suggestions for additional experiments to test and refine the hypothesis are made.

Introduction

The title uses two terms to describe the content of this review, "water disbondment" and "wet adhesion". Wet adhesion is understood as the adhesion of an organic coating to a support on exposure to liquid water or high humidity. Wet adhesion was adopted in previous publications^{1, 2} because it was the colloquial phrase most widely used by workers who had to deal with the process of adhesion loss of coatings on metals exposed to an aqueous phase. The term, "water disbondment", to describe the process is attractive because it restricts the behaviour to water and because it emphasizes that it is the loss of adhesion that is of concern. The suggested term has the added advantage that the disbondment process is not limited to that which occurs on exposure of the system to a liquid phase but includes disbondment that results on exposure to a high relative humidity. Water disbondment is thus defined as the separation that occurs between a polymer coating and a solid substrate when a moderate force, insufficient to affect the system when not exposed to water, is applied during or immediately after the coating system is exposed to gaseous or liquid phase water or aqueous solutions. This definition includes effects at polymer/polymer, ceramic/polymer, glass/polymer and metal/polymer interfaces. This review, however, will be limited to metal/organic coating interfaces with the exception of a few statements about other substrates.

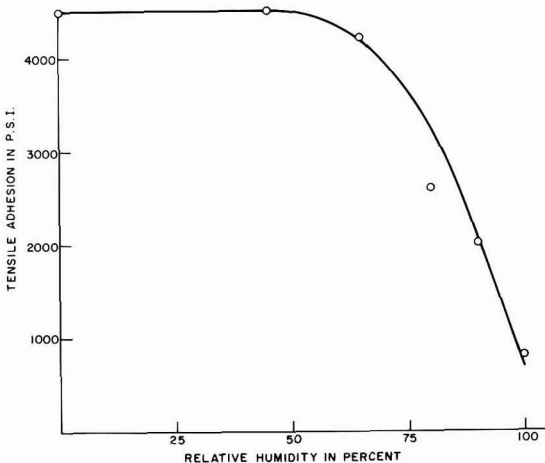


Figure 1. The loss in tensile adhesion of an epoxide ester paint after exposure for 600 hours to high relative humidities. Data taken from Walker³.

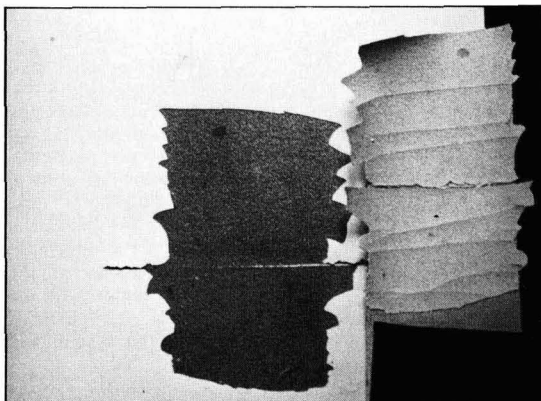


Figure 2. The loss of adhesion of an alkyd-melamine paint, 87 μm thick, after exposure to liquid water for one week at room temperature. Tape was applied to the surface and pulled off.

The phenomenon of water disbondment may be visualized in terms of the reduced adhesion that is observed upon exposure of a coated metal to increasing degrees of relative humidity as shown by the work of Walker³ in Figure 1 and by the poor adherence of a coating on steel when an adhesive tape is pulled from the surface after the coated metal is immersed in water for one week as shown in the example in Figure 2.

Many workers have reported that adhesion is regained when a water-immersed metal/coating system is allowed to dry before testing. Bullett⁴, in answer to a discussion question, stated that after one cycle, adhesion could be as much as 15% lower than the original value, whilst after 20 cycles it could drop by 50%.

Water disbondment also occurs in base-coat/top-coat systems as shown by the work of James⁵. He noted that the effects are very different from those experienced in metal/polymer systems in that the water disbondment did not occur if the base coat was coated after less than four days' drying time. The adhesion behaviour of base coats corresponded moderately well with their wetting characteristics by water, a low receding contact angle of water corresponding with bad adhesion and a high receding contact angle with good adhesion of the top coat. The nature of the top coat was less important than that of the base coat in determining the adhesion of the system. Unlike the results of Walker³ on steel/polymer systems, the water absorptions and water permeabilities of top coats did not correspond with their adhesion properties to the base coat.

The purposes of this review are fourfold: (1) to summarize the available information on water disbondment; (2) to offer a hypothesis to explain the phenomenon; (3) to summarize methods that are used to reduce the severity of water disbondment; and (4) to offer suggestions concerning experiments that may test the hypothesis.

The nature of the metal/polymer interface

It is very difficult at our present level of understanding to give a quantitative characterization of the morphological and chemical nature of the metal/polymer interface. It is particularly difficult to characterize this interface in general because different metals exhibit different surface chemistries and morphologies and different polymers have different types of polar groups, different chain segment lengths, different degrees of cross-linking, different amounts of reactive groups, different elemental compositions, etc. Commercial coating systems have the additional complication that they include components such as liquid components (solvents), wetting agents, pigments, fillers, anticorrosive components, rheological control agents, and other additives for specific purposes. These components may or may not be present in the interfacial region and may or may not play a role in the interfacial chemistry. For convenience in the discussion of the interface in this section, we will focus our remarks on the mild steel/polybutadiene interface since the personal experience of one of us is greatest with this system and work has been published on this system.

A steel surface prepared by pickling or other chemical or mechanical treatment becomes instantly coated with an oxide upon contact with the atmosphere. This oxide is typically 1-3 nm in thickness and both divalent and trivalent iron ions are present in the oxide. Many, if not all, the surface iron ions are in the trivalent state and have surface hydroxyl groups that participate in acid/base interactions with cations in any aqueous environment to which the metal is exposed⁶. Thus, calcium and sodium ions are surface impurities that are often present in non-trivial amounts on a steel surface after contact with an aqueous environment. Other constituents can be detected by X-ray photoelectron spectroscopy on steel exposed to the atmosphere for several

days or more. Fluorine is detected on the surface after immersion of steel in fluorinated water and chlorine is noted after exposure to marine conditions or after surface cleaning with a chlorinated solvent or after immersion in dilute chloride solutions⁷. Nitrogen is often detected. By far the largest environmental impurity on steel, when examined in an Auger spectrometer at 10^{-10} to 10^{-9} torr, is carbonaceous material⁸. Some of this carbon may be present as an alloying agent in the steel and as a carbon monoxide decomposition product or adsorbent from the vacuum system of the spectrometer, but much of the carbon on the surface comes from carbonaceous impurities in the environment to which the steel was previously exposed. This material often includes 20% or more of the elemental composition of the surface. The inability to be removed in a high vacuum suggests that the impurity reacts with the surface oxide and is tightly bound or is embedded mechanically. The importance of this carbon-containing material in the bonding between the metal and the polymer is unclear.

The surface of mild steel after being subjected to many different cleaning processes also contains significant concentrations of manganese and small amounts of sulphur and silicon⁹. These alloying agents at the surface of the steel are not covered with an iron oxide and consequently are detectable as surface constituents by Auger spectroscopy. If the steel is subjected to abrasive blasting, some of the abrasive particles become embedded in the surface and are visible on scanning electron microscopy and their presence is confirmed by a combination of electron and X-ray spectroscopies¹⁰. Furthermore, blasted surfaces are in a compressively stressed condition with surface stresses as high as 45,000 psi as determined by X-ray diffraction analysis¹⁰.

In summary, a conventionally cleaned steel surface contains a surface oxide layer with -OH groups and the surface includes, besides its oxide, a significant fractional area that contains impurities such as carbonaceous material, compounds of calcium, chlorine, fluorine, silicon, sulphur and manganese as well as embedded abrasive. It is to such a surface that an organic coating is normally applied. The alloying constituents, embedded abrasive and grain boundaries provide many minute crevices and fissures which high molecular weight polymers and low surface energy materials are not apt to wet completely on a nm scale. The metal/organic coating interface is thus likely to have non-bonded areas in which, under proper conditions, aqueous phase water can nucleate.

Less information is available about the polybutadiene phase. Our personal knowledge, along with a few other references, is summarized here. The starting resin is dissolved in an organic mixture consisting of various amounts of methyl ethyl ketone, cellosolve acetate, toluene, xylene and butyl acetate. It is applied to the steel by a spin coating technique so as to yield a dry film thickness of 18-25 μm . It is typically cured at 190-200°C in air for 30 minutes. The resulting coating is amber in colour with a glossy surface and is transparent in nature. There is no residual solvent odour and it is felt that only minute amounts of solvent remain in the coating. The curing process occurs by solvent evaporation and then oxidatively by the reaction of oxygen with activated $-\text{CH}_2-$ groups in the neighbourhood of the double bond.

Experiments by Mills¹¹ give an indication of the integrity

of a representative coating. A film, 25 μm in thickness and approximately 13 cm^2 in area on a steel substrate, was immersed in 0.5 M NaCl and cathodically polarized at -1.0 v vs SCE for 24 hr at room temperature. Approximately 25% of the 13 cm^2 square sections exhibited very small delaminated spots. In parallel experiments on similar free films it was observed that three out of four 1 cm^2 sections exhibited a resistance of 10^{11} ohms or greater and one out of four exhibited a resistance of the order of 10^7 ohms. The low resistance sections exhibited D behaviour and the high resistance sections exhibited I behaviour in the terminology of Mayne and Mills¹². The two sets of experiments yielded similar results in that the primary electrical pathways through a 25 μm thick coating are largely limited to a few very small imperfect regions. The cause of this local inhomogeneity is still under investigation.

The polybutadiene coating has slight ion exchange properties¹¹. After several months' exposure to 3.5 M KCl or several days in 0.1 or 1 M NaOH, the resistance of a 1 cm^2 section decreased from 10^{11} to 10^7 ohms. Heating such an exchanged film in 0.1 M H_2SO_4 for 18 hr at 60°C caused the room temperature resistance to increase to 10^9 ohms. The activation energy for conduction in the films having a resistance of 10^7 ohms is 20-40 kJ/mole whereas the activation energy for conduction in the sections of the film having a resistance of 10^{11} ohms is 80-120 kJ/mole. Passage of water and ions through the defect-free regions is probably by occupation of dynamic voids resulting from thermal motion of polymer segments.

Water passes through polybutadiene coatings on steel rapidly as indicated by electrical and gravimetric measurements¹³ and as inferred from delay time measurements during studies of cathodic delamination in aqueous chloride solutions¹⁴.

Oxygen also passes through polybutadiene rapidly when the oxygen is supplied from an aqueous phase as indicated by free film measurements in which the film is exposed to air-saturated alkali metal chloride solution on one side and oxygen-free alkali metal chloride on the other side¹⁵. As a sidelight, an interesting feature of these experiments is the fact that when the oxygen transport is measured simultaneously with a concentration gradient and an applied potential of 1 v that act to drive the alkali metal cation in the same direction as oxygen, the rate of oxygen transport increases in the order $\text{Li}^+ > \text{Na}^+ > \text{K}^+$ ¹⁵. The results are probably best explained in terms of competition of oxygen molecules and hydrated metal cations for sites on the surface or in the body of the polymer.

Now for some comments on the interfacial region where knowledge is insufficient. The adhesion between polybutadiene and steel is very high as shown by scratch and cross-hatch tests. Tensile tests in which a flat bolt is fixed to the polybutadiene surface with an adhesive are not reliable because of the thinness of the film and the adverse effect of the adhesive on the coating. Such measurements also indicate that the adhesive strength is high but we are not willing to assign a numerical value. Many of the conventional analytic tools are not useful for studying the intact interface. Laser Raman spectroscopy is limited by the strong fluorescence of polybutadiene and surface electron and X-ray spectroscopies are not applicable to the intact interface. Dickie et al¹⁶ have used Fourier transform infrared spectroscopy on thin coatings of polybutadiene and

noted that the interfacial region was more oxidized when the substrate was steel or chromium than when it was gold. Recent results by Deck¹⁷ using multiple reflection Fourier transform infrared spectroscopy are interpreted to indicate that carboxyl groups are present at the intact interface. Dickie et al¹⁸ also have observed carboxyl groups on the polymer side of the interface after the coating was delaminated cathodically. Castle and Watts¹⁹ have also utilized surface analysis techniques to study the delaminated interfacial surfaces after contact of the coated metal with an electrolyte but the information that can be extrapolated to the intact interface is limited. Ritter and Kruger²⁰ have utilized ellipsometric methods to study changes in the metal oxide at the interface upon exposure to aqueous solutions. Leidheiser, Musić and Simmons²¹ used Co-57 Mössbauer spectroscopy to conclude that primary bonds exist between polybutadiene and cobalt oxide at the interface after curing in air at 200°C. Many of the Co^{3+} ions in the oxide were reduced to Co^{2+} in spite of the oxidizing atmosphere.

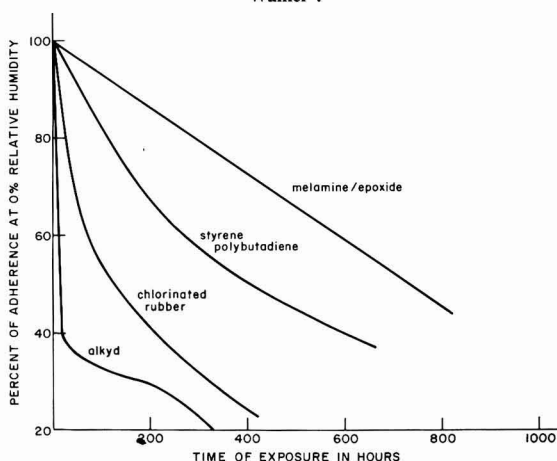
In summary, much is yet to be learned about the interfacial region but it does appear that primary chemical bonds exist between polybutadiene and the oxide on metallic cobalt and, by analogy, presumably between the polybutadiene and the oxide on iron.

It is recognized that the above comments on the iron/polybutadiene system may not necessarily be extrapolated to other metal/polymer systems, but the experimental facts do serve as a reference point and a backdrop for a summary of known facts about water disbondment.

Variables that affect water disbondment

Time of exposure. The rate at which adhesion is lost on exposure of coated steel to 100% relative humidity varies with the nature of the coating as shown in Figure 3³. In some cases, as with the alkyd, the rate of loss of adhesion is very rapid and in other cases it occurs progressively over many hundreds of hours as in the case of the melamine/epoxide copolymer. It is likely, although the available data do not allow a rigorous conclusion, that the rate in these

Figure 3. The rate of loss of adhesion of four coatings of similar thickness exposed to 100% relative humidity. Data taken from Walker³.



experiments was primarily dependent upon the rate at which water penetrated through the coating. Evidence in support of the latter statement is found in the data reported in Table 1 in which it will be noted that the rate at which the coating lost adhesion on immersion in water was related to the water permeability²². In other systems, however, no relationship is observed between the rate at which water disbondment occurred and the permeability of water through the coating^{23, 24}.

Effect of the substrate. The nature of the substrate affects the rate of water transmission through a coating to a small degree as shown by the following works. Katz and Munk²⁵ measured the rate of water vapour transmission through free films of cellulose nitrate and chlorinated rubber coatings formed on different surfaces. Cellulose nitrate films formed on tin foil and removed by mercury treatment were more permeable than those prepared on polyethylene. Holtzmann²⁶, using a capacitance technique, observed that the water permeabilities of adherent films were slightly lower than those of free films. Browne²⁷ noted that films of drying oil paints sorbed less water when bound to a glass substrate than in the free condition. However, if water accumulates at the coating/metal interface, the measured values of water uptake of the supported film may also be higher than those of the corresponding free films²⁸. Murray²⁹ compared the absorption of chloride ions by an epoxy polyamide free film and the same coating on an aluminium substrate. The diffusion coefficient was always higher and the partition coefficient always lower for the film on the aluminium substrate. In a study of water vapour absorption by several hydrophilic polymers it has been found that the steady state concentration of sorbed water was greater in the free film than when attached to glass³⁰. It is suspected, however, that the time of water exposure had an influence on these findings.

The nature of the metal substrate plays an important role in the ability of a coating to maintain adhesion upon contact with an aqueous environment. Two studies are cited to show this effect. Walker^{31,32} studied the adhesion of polyurethane and epoxy polyamide coatings to aluminium, cadmium, copper, mild steel and zinc after 1,000 (aluminium and mild steel) and 1,500 (cadmium, copper and zinc) hours exposure to a water spray and a carbon arc. The data in Table 2 show that the behaviour was a strong function of the metal as well as the coating. The water spray reduced the adhesion of both types of coatings to cadmium, steel and zinc; it had no effect on copper coated with polyurethane but there was a major reduction in adhesion for copper coated with the epoxy paint; and the adherence after water exposure improved in the cases of both coatings on aluminium.

Spadafora³³ studied the length of time required before a coating lost adhesion, as measured by a tape pull, upon exposure to a pH 7 buffer solution at room temperature for four different coatings and five different metal substrates. His data are summarized in Table 3. The adhesion after water exposure was long lived in the cases of copper and all four coatings; it was relatively inferior on nickel in all cases; and the effects were a function of the coating system in the cases of aluminium, mild steel and lead.

There is good agreement between the studies of Walker and those of Spadafora in those cases where the studies overlap. In two cases, polyurethane on aluminium and copper, the results are in agreement in that these systems

Table 1

A comparison of the rate of loss of adhesion of a coating on steel upon immersion in water with the water absorption and water permeability²²

Type of paint	Rate of loss in adhesion	Water absorption in five days (%)	Water permeability (x 10 ⁸)*
Thermosetting acrylic	slow	1.1	1.3
Styrene butadiene	slow	1.6	0.9
Chlorinated rubber	slow	1.0	1.7
Polybutyl methacrylate	slow	1.3	1.2
Polyamide cured epoxide	medium	3.5	3.7
Short oil epoxy ester	medium	3.9	5.4
Long oil alkyd	fast	4.3	40
Melamine/epoxide	fast	1.7	not determined
Polyurethane	very fast	5.7	33

*Units not given by author

Table 2

The effect of water spray in an accelerated atmospheric weathering test on the adherence of paints to five metals^{31, 32}

Metal	Water spray time (hr)	Adherence as measured by vertical pull-off (psi)			
		Polyurethane		Epoxy polyamide	
		Before	After	Before	After
Aluminium	1000	1660	1900	2950	3630
Cadmium	1500	3400	2680	3640	2520
Copper	1500	4050	4120	3970	2930
Mild Steel	1000	2230	2030	2830	2340
Zinc	1500	4280	2120	2970	1690

Note: There is probably a very high degree of uncertainty in the actual values in the table. The important point is the direction of change and the magnitude of the values before and after water exposure.

Table 3

The time required to lose adherence, as measured by a tape pull test, for four coatings on five different metallic substrates³³

Metal	Time to lose adherence (hr)			
	Acrylic	Polybutadiene	Polyurethane	Silicone alkyd
Aluminium	3	>170	>170	>170
Copper	>170	>170	>170	>170
Nickel	3	145	1	6
Mild Steel	3	170	15	25
Lead	>170	>170	50	>170

maintain good adherence under the conditions of the tests. Both studies also showed that the adherence is reduced on water exposure in the case of polyurethane on steel.

Limited useful and quantitative information is available regarding the effect of method of surface preparation

before applying the coating on the water disbondment phenomenon. Walker³¹ has provided some data which are summarized in Table 4 for polyurethane and epoxy polyamide coatings on aluminium and steel. Although the values for the adherence are higher in the case of the grit-blasted surface, the behaviour after exposure to water was similar for the degreased and grit-blasted surfaces. There was very little effect of water exposure for both coatings and both surface preparation methods in the case of aluminium, but both coatings exhibited lower adhesion values after exposure to water for both surface preparation methods in the case of steel.

The application of silanes of the type R-Si(OR')₃ to a steel substrate results in a major improvement in the adhesion of epoxy polyamide and a polyurethane paint on steel and aluminium when subjected to a condensing humidity test³¹ as shown by the data in Table 5 for steel. The silanes have been shown to improve adhesion in two-coat systems after either condensing water conditions or immersion in water³⁴.

Effect of type of coating. It is very difficult to make sweeping generalizations relating the water disbonding phenomenon to the type of coating. One of the difficulties arises from the different behaviour as a function of curing time. For example, Spadafora³³ found that a polyurethane coating cured for a few days exhibited poor adhesion after short times of water immersion, yet the same coating cured for four months exhibited good adhesion after water immersion for many days. Thermosetting acrylics have relatively good resistance to water disbondment whereas an air-dried acrylic exhibited very poor resistance to water disbondment. Table 6 indicates that it is likely that some generalizations will be possible with more work, and probably a lot more information is available in the laboratories of coatings manufacturers, but this information does not reach the public literature. The data in Table 6 indicate that similar results are obtained by two workers using entirely different measurement techniques and probably different coating formulations.

Holubka et al³⁵ have related the humidity-induced adhesion loss of oligoester, oligourethane and epoxy diol coatings to the concentration of -OH groups which interact with the oxide on the surface of the metal. The rate of adhesion loss of the coating to steel on exposure to high relative humidity decreased with increase in -OH group concentration in the polymer.

Effect of temperature. It is generally recognized that the loss of adhesion on immersion of a coated metal in water occurs more rapidly as the temperature is increased. This fact is the basis for the hot water test used by the coil coating industry to determine the efficacy of the pretreatment process. Also, coatings that are normally exposed to a higher temperature as in a dishwasher, for example, are tested in hot water. This general knowledge by the users of coatings is not reflected in quantitative information in the technical literature. Lampe³⁷ found that exposure to water at 24°C for 24 hr of an air-dried and an oven-dried coating resulted in a cross-hatch scratch test rating of five (poorest rating). Similar coatings exposed to water at 8°C for 24 hr resulted in a rating of two (good) for the air-dried coating and 1/2 (very good) for the oven-dried coating.

Firm information does exist, however, on the effect of

Table 4

The change in adherence of two coatings on aluminium and steel after 500 hours in a condensing water environment as a function of the metal pretreatment process³¹

Metal	Coating	Adherence before and after water exposure (psi)			
		Degreased		Grit-blasted	
		Before	After	Before	After
Aluminium	Polyurethane	1660	1680	4000	4090
	Epoxy polyamide	2950	3200	4000	4000
Steel	Polyurethane	2230	500	5200	2210
	Epoxy polyamide	2830	2520	3760	3160

Table 5

The effect of silanization of the steel surface on adhesion after 500 hours of exposure in condensing humidity test³¹

	Silane used	Adhesion after 500 hours condensing humidity (psi)	
		Polyurethane	Epoxy polyamide
	None	2210	3160
γ-methacryloxy propyltrimethylsilane		2580	3830
	β-(3, 4-epoxycyclohexyl) ethyltrimethoxysilane	3700	4140
	γ-glycidoxy propyltrimethoxysilane	3900	3840
	γ-mercaptopropyl trimethoxysilane	3180	3590
	n-beta aminoethyl aminopropyltrimethoxysilane	3130	3830

Note: All steel samples were grit blasted before application of the silane and the coating.

Table 6

A comparison of the rate of loss of adhesion upon immersion in water for generic types of coatings as measured by two investigators

Type of paint	Rate of loss of adhesion as noted by Walker ³²	Number of hours before adhesion was lost as determined by tape pull test (measured by Spadafora ³³)
Thermosetting acrylic	slow	
Air drying acrylic		3
Styrene butadiene	slow	
Polybutadiene		170
Long oil alkyd	fast	
Silicone Alkyd		25
Polyurethane	very fast	15

Note: the discrepancy in the first example is probably a consequence of hydrophilic solvent retained at the metal/coating interface in the case of the air drying acrylic.

temperature on the permeability of coatings for water, the solubility of water in coatings and the diffusion coefficient for 14 different coatings over a temperature range of 11.7°C

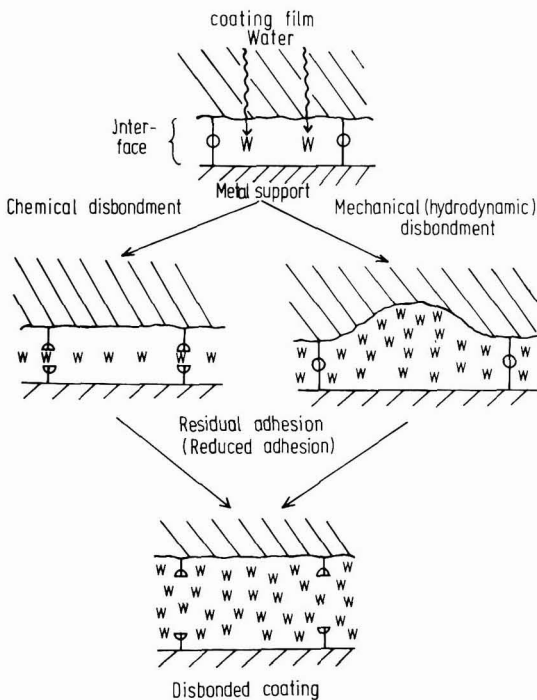


Figure 4. Schematic representation of the mechanisms of adhesion reduction and water disbondment. The mechanism proposed by the authors is shown on the right.

to 40°C³⁸. Such data are given in Tables 7, 8 and 9.

Hypothesis for the water disbondment process

Many workers in the field of adhesion believe that the adhesion of organic coatings to metal substrates may be lost due to the disruption by water molecules of the covalent, hydrogen or polar bonds across the coating/metal interface. The hypothesis offered below proposes a mechanical or hydrodynamic mechanism of disbondment. These two mechanisms are schematically illustrated in Figure 4. The hypothesis is summarized first in the form of several statements and the evidence supporting these statements will then be given:

1 Water disbondment is a consequence of the formation at the metal/coating interface of a discontinuous or, in some cases, a continuous water film several to many molecular layers in thickness. Loss of adhesion is a consequence of splitting of this water layer upon the application of a force.

2 Water moves through the coating by diffusion through the compact polymer phase or through capillaries or pores in the coating.

3 The driving force for directional water transport through the coating to the interface is diffusion under a concentration gradient. Osmotic force, temperature differences and chemisorption or physisorption of water on the metal oxide at the interface are among the mechanisms that may lead to accumulation of water at the interface.

4 The water accumulation at the interface is made possible by the presence of non-bonded areas of sufficient

Table 7

The effect of temperature on the permeability coefficient P for 14 different coatings in a wet cup test, 100 to 50% Relative Humidity³⁸

Coating	Permeability P in $\times 10^{12}$ g/cm ² /sec/mm Hg			Activation energy (kJ/mole)
	(23°)	(30°)	(40°)	
1	8.38	12.06	15.17	26.88
2	19.17	23.31	29.16	18.98
3	45.58	58.88	65.50	16.43
4	42.25	47.83	56.37	13.05
5	22.86	27.86	36.23	20.86
6	26.20	32.76	36.89	15.51
7	8.74	8.47	9.71	4.77
8	10.92	12.93	13.24	8.70
9	14.67	18.77	26.25	26.34
10	11.48	16.59	20.71	26.71
11	11.35	15.51	20.60	26.34
12	137.67	148.67	212.75	19.82
13	26.03	37.05	50.06	29.60
14	8.85	11.86	17.24	30.18

Table 8

The effect of temperature on water absorption by 14 different coatings at 50% Relative Humidity³⁸

Coating	Weight percent water absorbed by coating				
	11.7°	17.2°	23.0°	28.3°	33.9°
1	0.66	0.70	0.71	0.73	0.76
2	.61	.62	.63	.63	.64
3	.60	.62	.64	.65	.67
4	.54	.54	.56	.59	.61
5	.45	.46	.47	.51	.53
6	.28	.28	.30	.34	.36
7	.10	.10	.10	.12	.13
8	.47	.49	.51	.53	.55
9	.49	.49	.51	.53	.55
10	.38	.38	.39	.43	.47
11	.65	.67	.68	.69	.70
12	.65	.67	.69	.70	.71
13	1.11	1.12	1.18	1.22	1.27
14	.96	.97	.99	1.01	1.03

Table 9

The effect of temperature on the diffusion coefficient of water in 14 different coatings at 50% Relative Humidity³⁸

Coating	Diffusion coefficient $\times 10^8$ cm ² /sec		
	(23°)	(30°)	(40°)
1	0.75	1.31	2.51
2	.63	1.06	2.20
3	4.59	7.51	13.18
4	2.59	4.27	7.71
5	2.84	4.46	7.51
6	3.46	5.33	8.40
7	5.63	7.63	12.20
8	0.68	1.00	1.60
9	1.51	2.67	5.00
10	1.27	2.16	3.90
11	0.97	1.90	3.38
12	19.13	30.36	53.48
13	1.40	2.52	4.96
14	0.34	0.58	1.28

dimension to permit formation locally of an aqueous phase.

5 The local water volumes grow laterally along the metal/polymer interface under the driving forces noted in (3) above. The lateral growth occurs because of continuing condensation of water molecules. Lateral growth is permitted because of the stress caused by water condensation and the dynamic nature of metal/coating bonds at the interface.

Statement 1. *Water disbondment is a consequence of the formation at the metal/coating interface of a discontinuous or, in some cases, a continuous water film several to many molecular layers in thickness.* The evidence for the formation of multilayers of water at the interface comes from many sources. First, in some systems, particularly with alkyds on steel, when water disbondment occurs after immersion in water, there is a visible water film that disappears by evaporation very rapidly. Second, water can be seen as interference fringes at the glass/coating interface when an air-dried acrylic coating on glass is immersed in water. The visible water starts at very many small spots which then spread laterally. Third, Funke²⁸ and Funke and Haagen³⁹ have shown, in the case of coatings exhibiting disbondment, that the coated metal has a higher water absorption than the free film. The so-called crossover point, that point in time where the coated metal absorbs more water than the free film, is the point where water begins to condense in significant amounts at the interface. Fourth it has been noted with some coatings^{3,24} that the adherence between the metal and the coating is improved on exposure to water vapour. The improvement of adhesion by exposure to water is difficult to explain but one possibility is the concurrent release of residual or retained solvent. The water in these cases is present at the interface in low concentration and presumably serves to increase the bond strength between the coating and the metal. Fifth, is a well known experiment. When an epoxy is bonded to anodized aluminum, the peel strength is very high when measured in an environment of approximately 50% relative humidity. The coating, on the other hand, is readily removed by peeling when liquid water is in contact with the notch at the coating/anodized aluminum interface. The ready disbondment required liquid water. Sixth, the rate of water disbondment is much greater when the coated metal is immersed in water as compared to exposure to 100% relative humidity as shown by the data of Walker^{3, 24} in Table 10. Seventh, the most convincing evidence comes from water absorption studies on iron oxides. Figure 5 is reproduced from the article by McCafferty, Pravidic and Zettlemoyer⁴⁰ in which it will be noted that a monomolecular layer of adsorbed water occurs on $\alpha\text{-Fe}_2\text{O}_3$ at low relative humidities and that polylayers exist at a relative humidity of 90%. Data are not given for 100% relative humidity but extrapolation of the curve indicates that at 100% relative humidity there will exist many layers of water at the oxide surface. The strong affinity of oxides for water is also shown by effects at oxide pigment/binder interfaces. In many cases, exposure of pigmented organic coatings to water results in the accumulation of water at the pigment/binder interface^{41, 42}. The above data are well in accord with the observations of Vernon⁴³ who noted that rusting of iron begins to occur at an appreciable rate above a relative humidity of 80% when the metal was first exposed to SO_2 . This critical humidity for rusting depends on the partial pressure of water required for liquification of salts on the surface of the iron. The salts serve as electrolytes for the corrosion reaction. Since the corrosion

Table 10

A comparison between the approximate times necessary to lose 50% of original adherence upon exposure of coated stainless steel to 100% relative humidity and immersion in water^{3, 24}

Coating type	Time to lose approximately 50% original adherence (hr)	
	100% RH	Immersion
Alkyd	20	50
Epoxy ester	100	5
Chlorinated rubber	125	10
Styrene butadiene	400	15
Polybutyl methacrylate	300	15
Thermosetting acrylic	greater than 700	10
Melamine/epoxide	700	10
Polyurethane	50	3
Epoxy polyamide	400	1

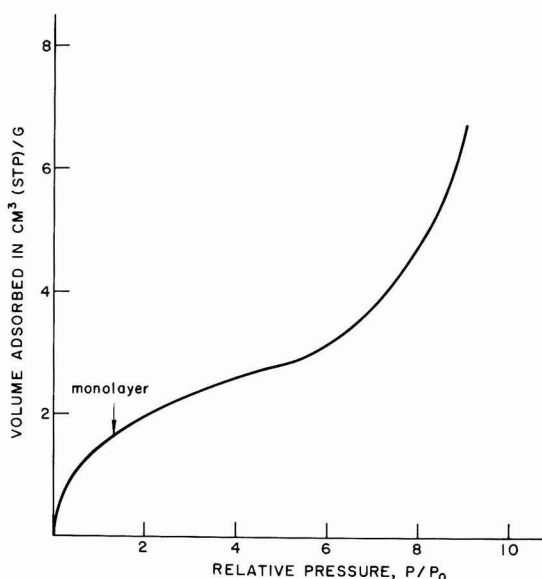


Figure 5. The amount of water adsorbed by powdered $\alpha\text{-Fe}_2\text{O}_3$ as a function of relative humidity. Data taken from McCafferty et al⁴⁰, p/p_0 values x 10.

process requires a conducting film of water at the surface, it is apparent from the work of Vernon that multilayers of water form at relative humidities below 100%. The works of Walker³ and Bullett⁴ show that the decrease in adherence with many coatings begins in the range of 65 to 80% relative humidity and the adherence rapidly degrades at relative humidities in excess of 90%.

Recent work on very thin films of water between mica plates has shown that the bulk viscosity of water is retained to within about 10% of the bulk value in water films as thin as 5 nm or less. The "plane of slip" is within a few tenths of a nm of each surface. Israelachvili⁴⁴ concludes that, at most, one layer of molecules is immobilized at each surface. Extrapolation of these conclusions to water disbondment indicates that poor adhesion after exposure to water may occur in the presence at the interface of a water film only several molecular layers in thickness. The thickness is a minimum value and osmotic driving forces are expected to

increase the thickness of water film rapidly, once a thickness sufficient to have the bulk properties of water is achieved.

The discontinuous nature of the liquid water formation at the interface is confirmed by the experiments cited above for the acrylic coating on glass and by the fact that flexible coatings with continuous water exposure will exhibit small, isolated, water-filled blisters that grow with time. These are explained by the fact that the lateral spread of the water layers in these cases occurs at a lower rate than the combined osmotic forces and corrosion that lead to a visible water phase. (Note: The presence of non-bonded regions on a smooth glass substrate is probably explained by the trapping of solvent at the glass/coating boundary.) The discontinuous nature of the water film at the interface is also seen occasionally in tape pull tests after water exposure in which small sections of the film are separated from the metal, whereas the majority of the coating retains its adherence.

Statement (2). *Water moves through the coating by diffusion through the compact polymer phase or through capillaries or pores in the coating.* The fact that water rapidly moves through most coatings is shown by the routine use of Payne Cup tests in coatings laboratories. The data for coating number 8 in Table 7 may be used to calculate that a 100 μm thick film can pass 10^{-9} g of water per sec under a pressure difference of 1 mm of mercury. If, as an extreme case, the metal/polymer interface can be considered to behave as a water sink in the same manner as the low pressure side of the water gradient across a free film, it would require only 1,000 seconds to build up a water film 10 nm in thickness at the interface under a pressure gradient of 1 mm.

There appear to be two important pathways of water through organic coatings. First, diffusion occurs through the polymer as individual water molecules occupying holes in the polymer network caused by thermal motion of segments of the polymer chain. As these segments oscillate and vibrate there appear free volumes into which water molecules may move and take a random walk through the coating. The driving force for net motion of water molecules in the direction of the metal/coating interface will be discussed in the next section.

Not all migration through the polymer is by the above manner. Some migration is through channels or capillaries or pores that are present in the coating as a consequence of solvent evaporation after hardening of the coating, structural inhomogeneity in curing, impurity particles, solubilization of constituents in the coating, etc. Pigmented coatings also have potential water pathways at the interface of pigments and fillers with the polymer matrix^{42, 45}. These channels may be detected by electrical measurements⁴⁶, by the localized incorporation of radiotracer elements, by the reaction of water with pH indicators under a transparent coating and may be inferred from the highly localized corrosion that often occurs under a coating at an early stage. Boxall, von Fraunhofer and Warren⁴⁷ have interpreted their results on water permeability through free films of an epoxy polyamine, a tung oil phenolic varnish, a vinyl chloride-vinyl acetate copolymer and a chlorinated rubber to indicate that water passes through capillaries or pores in the coating.

Statement (3). *The driving force for directional water*

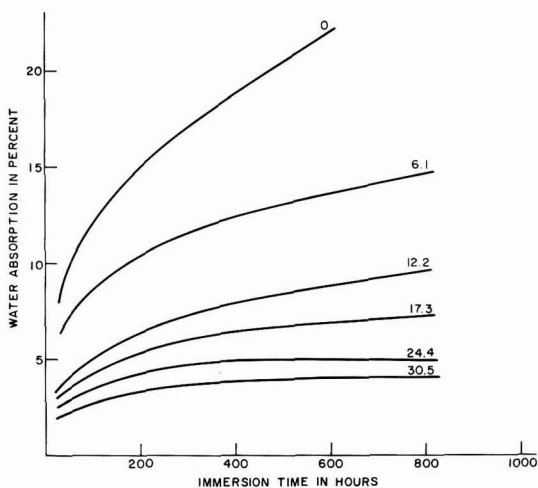


Figure 6. The rate of water absorption of an alkyd coating on different substrates as a function of the osmotic pressure of the solution. The osmotic pressure in atmospheres is shown by each curve. Data taken from Kittelberger and Elm⁴⁸.

transport through the coating to the interface is diffusion under a concentration gradient. Osmotic force, temperature differences and chemisorption or physisorption of water on the metal oxide at the interface are among the mechanisms that may lead to accumulation of water at the interface. The penetration of water through a coating can be driven by osmotic forces as shown many years ago by Kittelberger and Elm⁴⁸. They immersed coatings consisting of a zinc chromate base coat and alkyd top coat on steel, duralumin, zinc and glass in NaCl and sucrose solutions. The rate of water penetration was a function of the osmotic pressure as shown by the data in Figure 6.

The semipermeable nature of coatings composed of a medium oil alkyd, an epoxy resin, a chlorinated rubber and a chlorinated rubber plasticized with a chlorinated paraffin was confirmed by Perera and Heertjes⁴⁹. These workers showed that the coatings were sensitive to osmotic pressure differences and that water diffused through the coating under an osmotic pressure gradient.

Van der Meer-Lerk and Heertjes⁵⁰ studied the rate of blister formation on coatings of polyurethane, epoxy amine and a chlorinated rubber when known amounts of magnesium acetate or sodium sulfite were placed on a clean stainless steel panel before application of coating. A representative result is given in Figure 7. The rate of blister formation was correlated with the measured water permeability and the osmotic force generated by the salt. The results were consistent with the conclusion that the rate of blister growth was not markedly influenced by either the adhesion or rigidity of the film. It was also noted that even if the difference between the thermodynamic activity of the water in the blister and the external medium was small, the blister continued to grow. The implication is that even minute amounts of hydrophilic contaminants present on the surface can cause blistering and thus aqueous concentrations beneath the film.

Thermal gradients also represent an efficient mechanism for water condensation at the metal/polymer interface. The metal is an effective thermal conductor whereas the coating

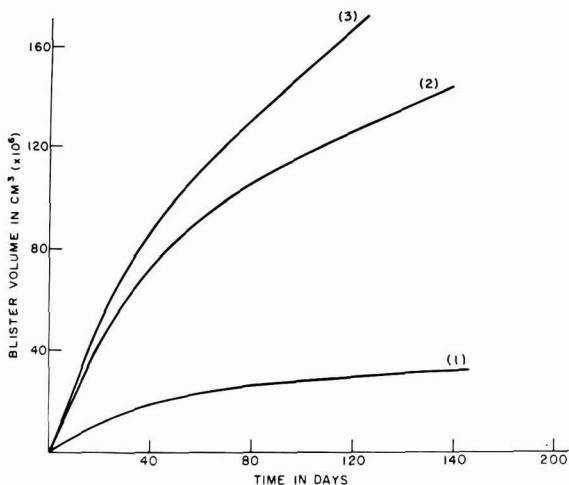


Figure 7. The volume of a blister beneath an 80 μm thick polybutadiene coating as a function of time for three different amounts of magnesium acetate on a stainless steel substrate before application of the coating. (1) 1.89×10^{-6} g; (2) 6.4×10^{-6} g; (3) 8.5×10^{-6} g of $\text{Mg}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$. Data taken from van der Meer-Lerk and Heertjes⁵⁰.

is a poor thermal conductor. Small falls in the environmental temperature after relatively long periods of constant temperature may cause water accumulation in non-bonded areas present at the interface. Apparently small temperature differences result in condensation as seen, for example, by the condensation of water during the night on the top surfaces of an automobile without condensation on the sides. Thermal differences in this case are caused by the more rapid radiation of heat to the black sky relative to the lower rate of radiation to the surroundings. The condensation of water at the interface on the application of a thermal gradient is the basis of an aggressive corrosion test known as the condensing humidity test.

Metal oxides are themselves efficient substrates for the formation of multilayers of water at humidities below 100%. The work of McCafferty, Pravdic and Zettlemyer⁴⁰ on the adsorption of water on ferric oxide has been cited previously. Another work that supports the formation of multilayers of water on nickel oxide is the work of Micalc, Topić, Cronan, Leidheiser and Zettlemyer⁵¹ in which they noted multilayer water formation at relative humidities far below 100%. In practical coating systems containing pigments and fillers, water accumulation also occurs at the pigment/binder interface as stated previously. Competition of these two types of sites, the metal/coating interface and the pigment/coating interface, for water accumulation may be important in some cases in controlling water disbondment.

Statement (4). *The water accumulation at the interface is made possible by the presence of non-bonded areas of sufficient dimension to permit formation locally of an aqueous phase.* In making this statement, two issues must be addressed: (1) whether or not non-bonded areas are present at the metal/coating interface and (2) the size of non-bonded areas necessary to permit condensation of water molecules to a sufficient degree to constitute an aqueous phase with the properties of bulk water.

Non-bonded areas at the interface can come about from

three major sources: voids or fissures in the metal surface; voids resulting from localized lack of wetting of the metal by the polymer; and dynamic voids that result from the making and breaking of surface bonds by segments or polar groups in the polymer. There are no experimental data that unequivocally prove the existence of voids of very small size at the interface. Large voids can, of course, be seen by optical studies of transparent coatings and by microscopic examination of cross sections, but the voids with which we are concerned are not detectable by microscopy. The voids of primary concern are those several nm in diameter that are present because the shape of the void does not permit perfect wetting by the coating. A possible means for answering the question about very small voids will be described later.

Voids primarily caused by the metal come about as a consequence of indentations at grain boundaries or the boundaries between alloy agent precipitates such as manganese oxide or manganese sulfide and the matrix because of etching in a pretreatment step. Imbedded abrasive particles also may provide geometries that cause voids. Blasting processes may also cause rough surface geometries, as seen at high magnification in an electron microscope, that may result in small voids. Voids may also exist in oxide layers, especially in flash-rusted or rusted steel surfaces.

There is some evidence to support the viewpoint that organic coatings do not always completely wet a metal surface. Huntsberger⁵² states that "wetting the smallest interstices with the viscous adhesives may require relatively long times". Also, the activation energy for the bonding process is of the same order of magnitude as the activation energy for the flow of viscous polymers^{53,54}. The room temperature adhesion of coatings often increases with increase in the baking temperature and it is a generalization that baked coatings exhibit better adhesion properties than room temperature cured coatings. The higher temperatures, among other things, lead to better flow of the polymer and better wetting of the surface, higher degree of cross linking and more effective solvent release. This lack of wetting of certain types of surface geometries presumably leads to the formation of nonbonded areas. It should be pointed out that coating technologists design paint systems to have good wetting since these wetting properties may be controlled by paint additives. Wetting properties may be easily controlled and adjusted by surface tension measurements. Serious wetting problems occur only in special cases as with powder coatings. There is little or no problem with water-borne coatings or those using organic solvents. It is, however, not the macroscopic wetting with which Statement (4) is concerned but the lack of wetting on the nm scale.

Kumins⁵⁵ in his Matiello lecture makes a very good case for the fact that the bonds between polymer segments and the surface of the metal are dynamic in nature and that minute voids can be formed at the metal surface for short periods of time. If the voids are sufficiently large to accommodate several water molecules when conditions are appropriate for condensation of water at the interface, recontact of the polymer segment at this location is prevented.

Statement (5). *The local water volumes grow laterally along the metal/polymer interface.* Evidence has been summarized previously for the fact that loss of adhesion starts locally. If

much of the interfacial area is to lose adhesion, there must also be lateral growth. The growth of water volume in the localized regions, after a few molecular layers have formed, is presumably driven by osmotic forces. Impurities on the metal surface, soluble species leached from the polymer and ions formed by the corrosion process all serve to draw water to the surface. The increase in the water volume exerts a peeling force in the contact area between the polymer and the metal. The magnitude of this force is determined by the mechanical properties of the polymer. Flexible polymers will tend to show blisters more readily and rigid, highly crosslinked polymers will tend to distribute the stress more uniformly or crack. As the water spreads from the initial points to form multilayers of water, the bonded region is reduced in area and the adhesion is lost in the manner shown in Figure 3. The rate of adhesion loss is primarily determined by the rate at which water can spread to form multilayers at the interface.

Localized disbonding, in the form of blisters, may also occur in some cases because structural heterogeneities, local differences in coating thickness, and easy diffusional pathways formed by pigment agglomerates allow water molecules to arrive at the interface with locally different rates.

Methods used to reduce the severity of water disbondment

Two methods to reduce the severity of water disbondment, metal surface roughening and the pre-absorption of compounds on the metal prior to the application of the coating, have already been cited and a third method has been implied. The data in Table 4 show that grit-blasted surfaces of aluminium and steel exhibit high values of adhesion, relative to unroughened, degreased surfaces of the same metal both before and after exposure to water. The adhesion values for the grit-blasted surfaces after water exposure are higher in three of the four examples in Table 4 than the values for the untreated surface prior to water exposure. Data from the laboratory of one of us⁵⁶ has shown that adhesion of a powder epoxy coating to a copper surface specially roughened by an electrodeposition method, remained high even after the adhesion was adversely affected by a cathodic delamination process. Thus, surface roughening of the proper character can retain the coating on the metal surface by a "lock and key" effect. This adherence is generally sufficient to maintain the relative geometry of the coating and metal surfaces for the time while the coating is wet. Adhesion may be regained after the coating has dried.

References have been made previously to the fact that silanization of the surface makes the system much less sensitive to water disbondment. De Crosta and Leidheiser⁵⁷ have also shown that steel surfaces treated with silanes are much less sensitive to cathodic delamination, another form of aqueous intrusion at the metal/coating interface. These two citations indicate that modification of the chemistry, or perhaps the topology on a nm scale, of the metal surface is a practical method to improve resistance to water disbondment. More research is needed in this area.

The data in Tables 2 and 3 show that the tendency of coating systems to undergo water disbondment is also a function of the metal. Practical application of this observation is not as yet being made commercially to our knowledge. Very thin electrodeposits of lead on steel are worthy of investigation as good substrates for organic

coatings although the international drive to reduce lead in the environment may make the use of lead impractical. There may be, however, other metals that show the same resistance to water disbondment with specific classes of coatings.

The addition of components to the paint that adsorb at the interface prior to curing is another route to follow. The addition of silanes to paints is practical as judged from the research of Walker³⁴.

Suggestions concerning experiments to test the hypothesis

Additional proof is required that the loss of adhesion is a result of multilayers of water at the interface. The evidence for this conclusion is strong but it would be helpful to know the thickness of the water layer at which the adhesion degrades to a low value. Transmission line impedance studies of coatings on thin films of metals on an insulator substrate may provide useful information. The technique is certainly practical as shown by the work of McIntyre and Leidheiser⁵⁸. The greatest difficulty in such studies is measuring the adhesion simultaneously with a method that determines the water present at the interface. One is forced to utilize duplicate samples, one for the adhesion measurements and one for the water measurement, since there is no suitable method for non-destructive measurement of the adhesion when exposed to high humidity or liquid water. In the dry state, the adhesion of organic coatings may be measured by the vertical pull-off test. This test is difficult to apply to coatings when exposed to water. It is often sufficient, for practical purposes, to decide whether the coating has substantially lost adhesion during increasing times of exposure to water.

Optical microscopic studies of the interfacial region during exposure of the system to water immersion are badly needed. A transparent coating and the use of a thin metal film on a transparent substrate permit observation of the interfacial region from both sides. The ability to use transmission optics with this arrangement extends the number of microscopic techniques that can be applied. Various sensors could also be placed at the interface to determine when the water layer reaches a specific thickness.

The detection of non-bonded areas or voids at the interface and a measurement of their dimensions is required. One possible means of approach to this problem is to prepare the coating in a xenon atmosphere and then to make nuclear magnetic resonance measurements as a function of external pressure⁵⁹. The technique has been applied with great success to zeolites. It will be necessary to use non-ferromagnetic metals as the substrate in such studies.

Additional studies should be made of metal/coating systems in which the coating is kept constant and the substrate changed and other studies in which the metal is kept constant and the coating is changed. Neat resin systems and pure metals should be utilized for ease in interpretation. It is expected that correlations will emerge that allow a more precise explanation of the metal oxide/coating bond.

A better understanding of the kinetics of the disbondment might be assisted if information were

available on the activation energy of the process. Measurements should be made on several metal/coating systems as a function of temperature and a quantitative interpretation of the effect of temperature should be attempted. Care must be taken to select systems in which the glass transition temperature is known, or preferably measured. Account must be taken of the fact that the properties of the system may be different above and below the glass transition temperature.

The greatest need in all research on water disbondment is a technique that permits the adhesion to be estimated without the necessity of destroying the sample. The tensile test, the peel test, the scratch tests and the tape-pull test are all destructive. Each of the techniques has its shortcoming(s) and none looks suitable to be used in conjunction with a measurement of a different type. A non-destructive and reliable adhesion test applicable under corrosion conditions is vitally needed.

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59. We are grateful to Prof. John Larsen, Lehigh University for this suggestion.

next month's issue

The Honorary Editor has accepted the following papers for publication in the June Issue:

The versatile paint resin—'Haloflex' by R. G. Humphries, ICI Chemicals and Polymers Group, Resins and Surface Coatings Intermediates Business, Runcorn, UK.

Heat-cured ionomers prepared from poly (acrylic acid): A new class of waterborne coating material by J. W. Nicholson, R. P. Scott and A. D. Wilson, Materials Development Sub-Division, Laboratory of the Government Chemist, London, UK.

An evaluation of 'Silane' treated concrete by H. L. Robinson, Taylor Woodrow Engineering Ltd, Design and Research Laboratory, Southall, UK.

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An evaluation of South African micaceous iron oxide pigment*

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Summary

There is no intention here to gainsay the excellent properties of the Austrian supply of micaceous iron oxide (MIO) pigment, or the well-documented references and the first-class marketing of that product. However, the simultaneously documented denigration of all other alternative sources of MIO pigment supply was perhaps not fully justified¹⁻⁴.

A series of laboratory environmental and exterior exposure tests were conducted on commercially produced comparative alkyd resin based MIO paints, incorporating Austrian and South African sources of MIO pigment, respectively. From these tests it was established that the South African source of MIO pigment performed favourably compared with the Austrian pigment.

There was an attempt to preclude all other than Austrian sources of MIO pigment supply from the International Organization for Standardization standard, ISO DP1248 Iron oxide pigments for paints—specifications and test methods⁵. One ploy used was an attempt to include the stark extremes of two figures, at “ $\times 250$ magnification viewed under an electron microscope”, of “a typical lamellar structure” and of “a typical granular or amorphous structure”. Being impracticable, this requirement was opposed by the South African representative on the ISO/TC35/SC2 technical committee. An alternative practical laboratory paint environmental test procedure was put forward.

Abbreviations

ASTM = American Society for Testing and Materials
MIO = Micaceous iron oxide
ISO = International Organization for Standardization
QUV = The Q-Panel Company, artificial weathering test equipment complying with ASTM G53
Std dev = Standard deviation
UV = Ultraviolet

Introduction

It was required to determine whether the South African source of micaceous iron oxide was of suitable quality for use in a standard roof paint and in a standard structural steel paint, both based upon a micaceous iron oxide paint with an alkyd resin vehicle.

The formulae parameters for these standard paints were:

1. A long oil length alkyd resin vehicle.
2. Pigment volume concentration: PVC 42-52%.
3. MIO content of the total pigment: 75% (m/m), min.
4. Volatile matter content; 25% (m/m), max.
5. The only permitted extender, fibrous magnesium silicate (asbestine), content of the total pigment; 15% (m/m), max.
6. For tinting, only the permitted pigments as listed in the standard.

Evaluation was to be effected by assessing the comparative performance of pairs of paints manufactured to the standard specifications and incorporating equivalent quantities either of the local South African or of the imported Austrian micaceous iron oxide pigment. Tests were to include comparative laboratory exposure tests, and exterior exposure tests on suitable laboratory prepared, primed and painted panels to be conducted at the South African Bureau of Standards (SABS) Pretoria and Durban test sites.

Five paint companies supplied nine sets of MIO paints for the test series. The sets were in pairs, as indicated in the Table 1(a), incorporating the local or imported grades of MIO respectively.

Experimental

The condition of the paints was assessed after shelf storage of the unopened tins under standard test conditions for nine months. Seven of the paints, as listed in Table 1(a) and which included three of the local/imported sets, showed very hard, dry settlement. These each required separation of the medium, power-stirrer mixing of the base, and gradual re-incorporation of the medium, followed by a 10 minute shaking period on the Red Devil before the paints could be used.

On brush-out, paints V24920 and V24921 both gave a very bitty finish.

Panel preparation

Flat cold-rolled steel panels of thickness 0.60-0.90 mm were degreased with solvent until the surfaces were water-break free.

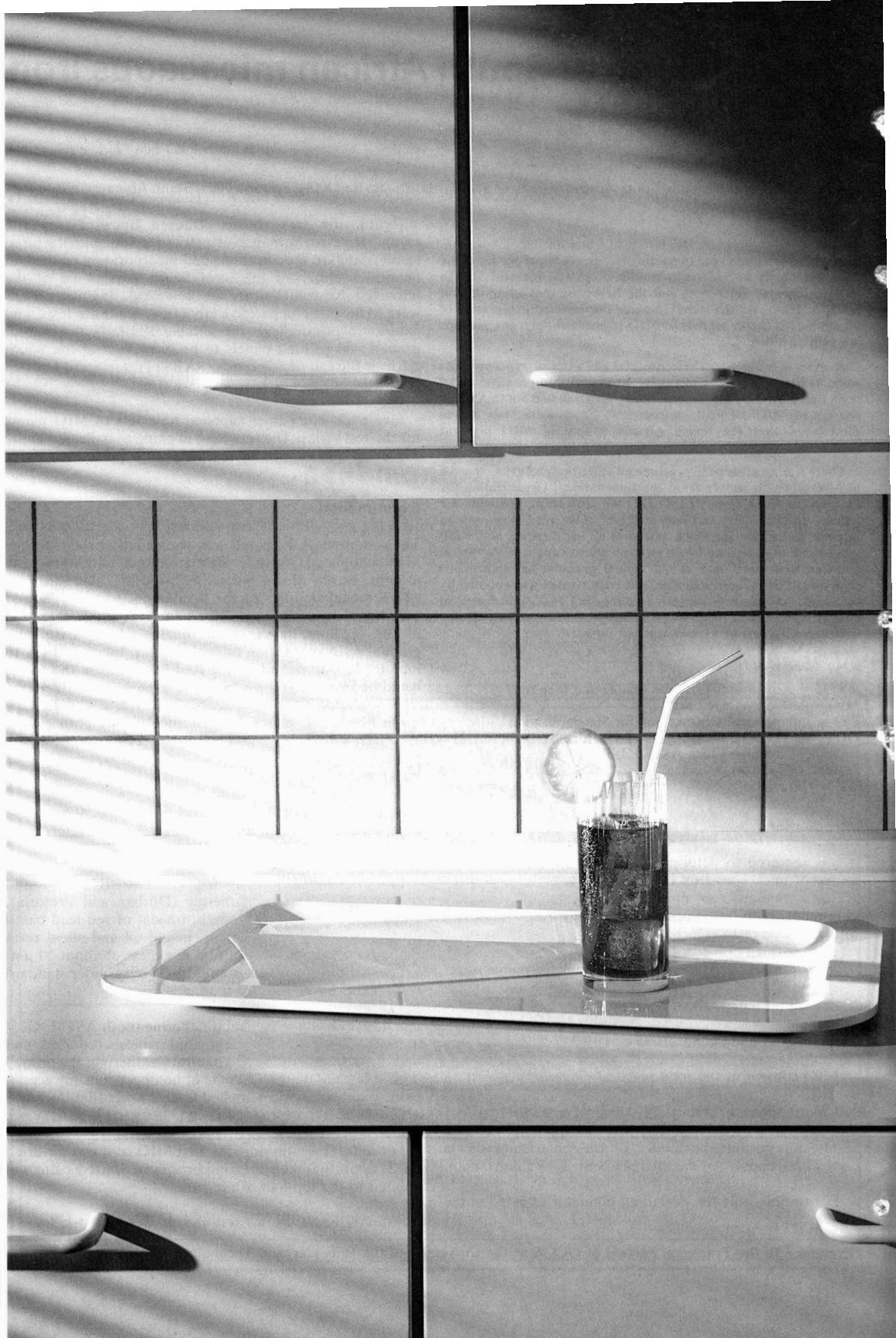
For resistance to salt fog (ASTM: B117-79) and resistance to natural weathering (Durban and Pretoria), panels were primed with one brushcoat of red lead based primer (high red lead content, mixed oil and alkyd resin type) applied to give a wet film thickness of about 50 μm . Primed surfaces were air-dried for 48 hours under standard test conditions before top-coating.

For resistance to artificial weathering (both ASTM: G23-81 and ASTM: G53-84), degreased panels were used, and for resistance to salt fog and natural weathering, the primed panels were used. Single coats of homogeneous micaceous oxide paint were brush-applied to give a wet film thickness of 75-85 μm .

For natural weathering, the first MIO coat was air-dried for 24 hours and then a second brushcoat of MIO paint was applied to the top half of the panels.

After top-coating, all test panels were air-dried for a

*Presented to the Transvaal Section of OCCA at the University of the Witwatersand Technology Centre on 17.9.86



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minimum of seven days before the panels were exposed to the test conditions.

Suitable panel back and edge protection was applied as necessary.

Test procedures

Panels were exposed for resistance to:

1. Salt fog (ASTM: B117-79), for periods of 500 hours and 750 hours.
2. Artificial weathering, ASTM Methods G23-81 and G53-84 (QUV), each for 1,000 hours (ASTM: G53 being based upon consecutive cycles of operation: four hours UV at 60°C, and four hours condensation at 50°C).
3. Natural weathering (Durban, coastal, for 6 and 12 months, 2, 3 and 4 years; and Pretoria, urban high UV incidence, for 2, 3 and 4 years).

Evaluations of the exposed test panels were detailed and are recorded in the Tables 1-3.

Discussion

The incidence of the hard, dry settling of seven of the 18 paint samples would normally be considered to be commercially unacceptable.

The salt fog exposure corrosion ratings showed no obvious major advantage for either source grades of the MIO pigment. The salt fog procedure over the 750 hour test exposure period proved to be too severe for the paint system. A 250 hour exposure period would probably be a more realistic test period for any future salt fog exposure test programme.

The development of lustre and colour fading were readily apparent after the ASTM: G53-84 (QUV) artificial weathering test exposure for 1,000 hours. This lustre development and colour fade were very approximately in the same order as the lustre and colour fade obtained following the two year exterior exposure at Pretoria, this site being well-known for its high intensity of natural UV light.

The development of the lustre and the colour fade after 1,000 hours exposure to the twin carbon arc artificial weathering (ASTM: G23-81) were much less than the effects obtained after the 1,000 hours exposure to the QUV (ASTM: G53-84) artificial weathering. Due to the slight colour fade by the ASTM: G23 test method, it was not practicable to make comparison of the ASTM: G23 results with the Durban exterior exposure test results.

The differences in the development of lustre and of chalking between the four year exterior tests at the Pretoria and the Durban test sites were of much more significance than any of the relatively minor performance differences resultant from the two different source qualities of the MIO pigments.

After four years of exterior exposure at the Pretoria site the maximisation of lustre development corresponded with the minimisation of chalking. The lustre development may be related to general performance properties of the MIO pigmented coatings.

In particular, development of lustre of a paint film indicates the lamellar property of the MIO pigmentation allied to the horizontal orientation of the MIO pigmentation flakes within the paint film.

After four years of exterior exposure at the Pretoria site six out of the nine sets of paints based upon the imported grade of MIO showed only a slight increase in lustre development as compared with the paints based upon the corresponding local grade of MIO.

After the four years exposure period at the Durban site, there was nothing of significance to choose between the performance of the local grade and imported grades of MIO based paints.

Notwithstanding the good performance ratings of all of the Durban exterior exposure panels, the exposure test conditions at the Durban coastal site were such that painted steel test panels without adequate protection of the panel edges and back, corroded to an unserviceable state within nine to 12 months.

MIO protective coating systems, total dry film thicknesses

The total minimum dry film thickness requirements for protective MIO coating systems quoted in the literature range from about 100 µm to 400 µm.

Table 4 details dry film thickness measurements undertaken on the test panels following the four year exterior exposure periods at the Durban and the Pretoria test sites. This table of results had been included in this study, firstly, as the results vary so widely from the expected micrometre thickness ranges of 60-80 µm for two coats and 90-120 µm for three coats of brush-applied paints, and secondly, as accurate coating dry film thickness determinations are not usually detailed in such studies for comment. Finally, the panels have been returned for another four years exterior exposure period at Durban and Pretoria. Following which repeat film thickness determinations are intended.

Mean, maximum and minimum thicknesses in micrometres, and standard deviation results were recorded using an Elcometer type 256FNT2 gauge. The Elcometer electromagnetic induction gauge was calibrated on a degreased 0.9 mm thick flat smooth steel panel using 52 µm and 249 µm thick plastic shims. Not less than 10 readings were taken at random for each panel, on each area of the coat of primer + MIO coat, and the primer + two MIO coats of finishing paint.

Conclusions

The MIO paint films, directly after application and drying, showed nil or negligible lustre.

The main changes in performance noted were the development of lustre and a reduction of the chalking following exposure to high intensities of UV radiation, as experienced after the four year exterior exposure at Pretoria, and to a lesser extent after the 1,000 hours exposure to ASTM: G53 (QUV) artificial weathering.

The effect on the performance of the MIO paint systems arising from the different exterior exposure site conditions,

Table 1(a)

Comparative laboratory test results of roof and structural steel paints incorporating South African versus Austrian imported grades of micaceous iron oxide pigments in alkyl resin base vehicle (See Figure 1)

Paint supplier	Sample number	Colour	MIO	Condition in container ¹	Blank lustre ^{2(b)}	Exposure to salt fog ASTM: B117-79				
						500hr		750hr		
						Blister ³	Rust ⁴	Blister	Rust	SAR% ⁵
A	U18657	Red oxide	Local	Soft settling	0	6-4M	7	4-2MD	1	90
	U21575		Import		0	6-4MD	10	4-2MD	2-3	30
A	U21577	Red oxide	Local	Hard, dry settlement	0	6MD	10	4MD	1	70
	U21576		Import		0	6F	9	6M	1	60
A	U21578	Red oxide	Local	Hard, dry settlement	0	8M	10	6MD	1	60
	U21579		Import		0	6M	9	6MD	1-2	50
B	U22426	Blue	Local	Readily dispersed	0	6-4MD	9	4-2MD	1	70
	U22427	Blue	Import		0-1	8-6F	10	2MD	1	70
B	U22428	Silver	Local	Hard, dry settling	0	6M	7	4-2MD	1	60
	U22429	grey	Import	readily dispersed	0-1	6-4M	6	6-2MD	1	90
B	U22430	Red	Local	Soft settling, readily dispersed	0	6M	10	4-2MD	4	10
	U22431	Red	Import	dispersed	0-1	6-4M	7	2D	1	70
C	U28869	Light	Local	Hard, dry settlement	0	4M	9	4MD	3	60
	U28868	grey	Import		0	8MD	4	8-6MD	1	90
D	U24920	Oxide	Local	Soft settling,	0	8M	10	6MD	7-8	10
	U24921	green	Import	readily dispersed	0	8-6M	10	4MD	5-6	30
E	U26495	Natural	Local	Soft settling,	0	6M	10	4M	7	10
	U26494	grey	Import	readily dispersed	0	4M	10	4-2M	3	60

Legends used in Tables 1 to 3

- The condition of the paint in the container was assessed after shelf storage under standard test conditions for nine months.
- (a). Lustre ratings, that is the reflection from the exposed plane surfaces of the MIO pigment particles, were initially assessed visually using a 12v lamp light source and 45° angles of incidence and reflectance. The two and three year exterior exposure ratings for lustre were assessed using this method of examination and are recorded in Tables 2 and 3a.
- (b). After the four year exterior exposure period, the ratings in Tables 3b and 4 for lustre were assessed using an artificial daylight cabinet source of illumination (ASTM: D1729-82). By the use of this source of illumination, revised values for lustre were also obtained on the test panels for both of the test methods for artificial weathering. These latter revised results are recorded in Table 1. The blank panel lustre assessments were similarly amended.
- (c). 5 = maximum development of lustre; 0 = nil lustre.
- Blistering, ASTM: D714-81.
- Rusting, ASTM: D610-81. 10 = nil rusting; 0 = maximum rusting.
- SAR % = percentage of metal surface area rusted, determined after the removal of the paint coatings.
- Colour fastness rating (ISO R105 Part A02, Grey scale for assessing change in colour). 5 = nil colour change; 0 = maximum colour change; R = redder; Y = yellower, but; D = darker; F = faded; Br = browner, and S = spotted lustre pattern in patches of diameter 4-20 mm, thought to be due to impact from hailstones.
- Chalking (ASTM: D659-80). 10 = nil chalking; 0 = maximum chalking.
- The outline of the magnets of the magnetic chuck panel holder was clearly shown by the MIO pigment orientated during the MIO paint brush-out on panels mounted upon a magnetic panel holder.
- Panel rating, a general visual comparison of the sets of panels, evaluating the general appearance of the paint coatings based upon local versus imported grades of MIO.

Note:

The panels were assessed unwashed, as rubbing of the panel surfaces during panel washing would result in artificial major increases of the lustre of the coatings.

The standard test conditions applicable in South Africa were 25 ± 2°C and relative humidity 50 ± 4%.

Table 1(b)

Comparative laboratory test results of roof and structural steel paints incorporating South African versus Austrian imported grades of micaceous iron oxide pigments in alkyd resin base vehicle* (See Figure 2)

Sample number	1,000 hr, artificial weathering, ASTM: G23-81		1,000 hr, artificial weathering (QUV), ASTM: G53-77		
	Lustre ^{2(b)}	Colour fastness rating ⁶	Chalking ⁷	Lustre ^{2(b)}	Colour fastness rating
U18657	0-1	3-4F	8	3	2F
U21575	0-1	4F	8	3	2F, Y
U21577	0	5	8	1	4-3F
U21576	0-1	5	8	3	4-3F
U21578	0	5	9	2	4-3F
U21579	1	5	8	4	4-3F
U22426	2	5	8	3	3-2F
U22427	2	5	8	4	2F
U22428	2	5	9	3	4F
U22429	2	4Br	9	3	3F
U22430	1-2	4R	8	3	3F
U22431	1-2	5	8	4	2F
U28869	2	5	9	2	4-3F
U28868	2	5	9	3	5-4F
U24920	1	5	8	4	2-3F
U24921	2	4-5Br	8	5	2-3F
U26495	2	4-5F	8	3	3F
U26494	2	4F	8	4	2F

*Legends are defined at the base of Table 1(a)

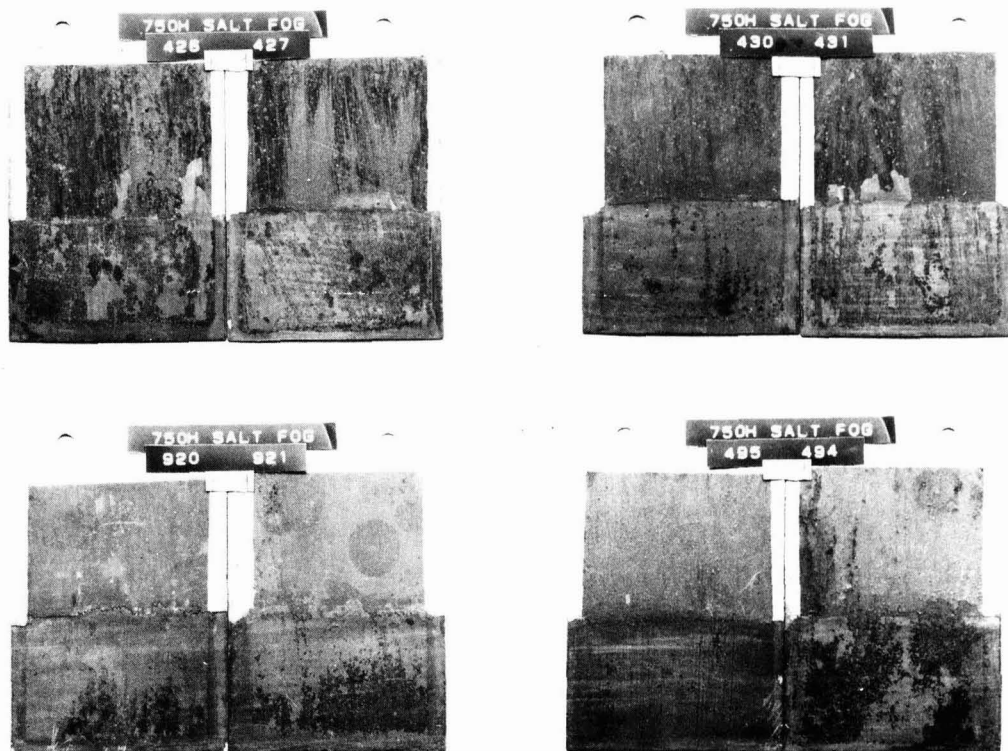


Figure 1. Exposure to 750 hours salt fog (see Table 1(a)) ASTM: B117-79, four sets 426/427, 430/431, 920/921, 495-494. For each set the left-hand side test panel is based upon local South African MIO and the right-hand side test panel is based upon Austrian MIO pigment.

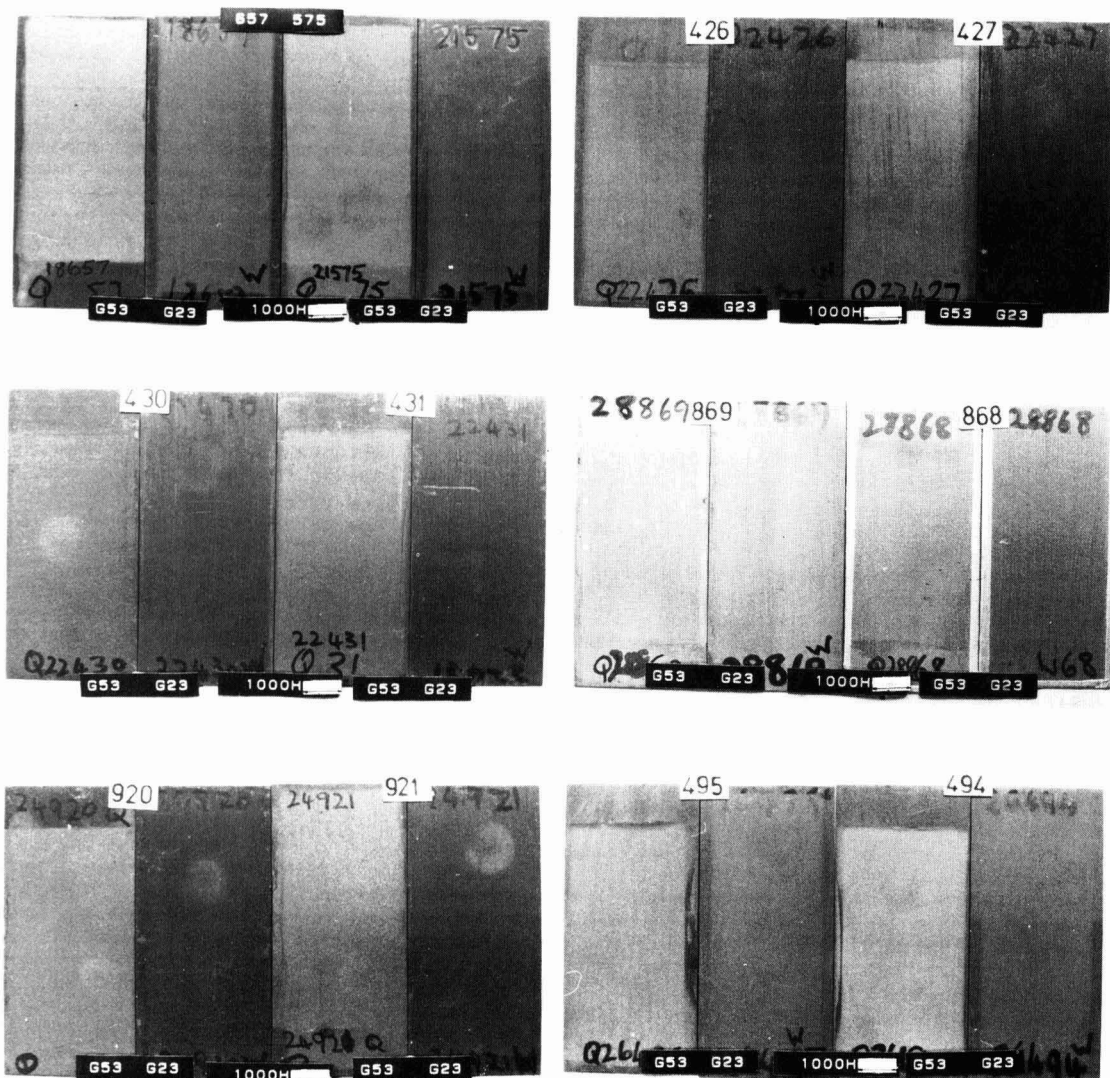


Figure 2. Exposure to 1,000 hours artificial weathering tests (see Table 1(b)), six sets 657/575, 426/427, 430/431, 869/868, 920/921, 495/494. For each pair of a designated set number, the left-hand side panel refers to ASTM: G53 (QUV) exposure and the right-hand side panel refers to ASTM: G23 (twin-arc) exposure.

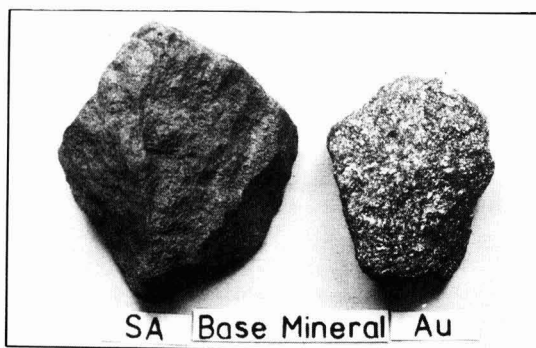


Figure 3. MIO base mineral ores: left, South African; right, Austrian.

Table 2(a)

Exterior exposure—short term*

Sample number	6 months exterior exposure Durban†		12 months exterior exposure Durban†		
	Chalking	Colour fastness rating	Chalking	Colour fastness rating	Panel rating ⁹
U18657	8	3	6	3F	Same
U21575	8	3	6	3F	
U21577	8	5	8	4-5	Same
U21576	8	4-5	8	4-5	
U21578	8	5	8	4-5	Same
U21579	8	5	8	4-5	
U22426	8-10	4-5	8-10	4F	Same
U22427	8-10	4	8-10	4F	
U22428	8-10	3	10	3D	Same
U22429	8-10	3	10	3-4D	
U22430	8	4	8	4-5	Same
U22431	8	4	8	4	
U28869	10	4	10	5-4	Same
U28868	10	4	10	5-4	
U24920	8-10	3-4	8-10	3F	Almost same
U24921	8-10	3	8	3F	
U26495	8-10	4	8-10	4F	Same
U26494	8-10	3	8	3F	

The legend is defined at the base of Table 1(a).

† 6 and 12 month exterior exposure results are not available for Pretoria.

Table 2(b)

Exterior exposure test results—short term—two years

Sample number	Two years exterior exposure Durban				Two years exterior exposure Pretoria			
	Chalking	Lustre ^{2(a)}	Colour fastness rating	Panel rating	Chalking	Lustre ^{2(a)}	Colour fastness rating	Panel rating
U18657	6	3	3F	Same	6	4	3-4F	Same
U21575	6	3	3F		6	3	3F	
U21577	6	0	4F	Same	8	1	4F	Imported, slightly more lustre
U21576	6	0-1	4F		6	2S	4F	
U21578	6-8	0	4F	Same	8	1	3F	Imported, slightly more lustre
U21579	6	0-1	4F		6	2	3F	
U22426	8-10	3	3-4F	Same	8-10	5	3-4F	Same
U22427	8-10	3	3F		8-10	5	3F	
U22428	8-10	2-3	3D	Local lustre slightly better	8-10	5	4	Same
U22429	8-10	2-3	3D		8-10	5	4-5D	
U22430	8-10	3	4Br	Same	8-10	4S	4F	Same
U22431	8-10	3	4Br		8-10	4S	4F	
U28869	8-10	2	3D	Imported lustre slightly more	8-10	4	4-5F	Same
U28868	8-10	3	3D		8-10	4-5	4F	
U24920	8	2-3 ⁸	3F	Same	8	4-5 ⁸	2-3F	Same
U24921	8	2-3 ⁸	3F		8	5 ⁸	2-3F	
U26495	8	2-3	2-3F	Same	8	5	3F	Almost same
U26494	8	2-3	3F		8	5	4F	

*Legends are defined at the base of Table 1(a)

Table 3(a)

Exterior exposure test results—longer term—three years

Sample number	Three years exterior exposure Durban				Three years exterior exposure Pretoria			
	Chalking	Lustre ^{2(a)}	Colour fastness rating	Panel rating	Chalking	Lustre ^{2(a)}	Colour fastness rating	Panel rating
U18657	8	4	3F	Very slightly more lustre	6	4S	3F	Same
U21575	6	4-5	3F		6	3S	3F	
U21577	6	1	3F	Same	6	1S	4F	Very slightly more lustre
U21576	6	1-2	3F	6	2S	4F		
U21578	6	1	3F	Same	6	1S	3F	Very slightly more lustre
U21579	6	1-2	3F	6	2S	3F		
U22426	8	3	3F	Same	8-10	4-5S	3F	Same
U22427	8	3	3F	8-10	4-5S	3F		
U22428	8	3-4	2-3D	Same	8-10	4-5S	3D	Same
U22429	8	3	2-3D	8-10	4-5S	4D		
U22430	6	2-3	3FY	Same	8-10	3-4S	4F	Same
U22431	8	2-3	3FY	8	3-4S	4F		
U28869	8	3	3D	Same	8	4-5S	3F	Same
U28868	8	3-4	3D	8	4-5S	3F		
U24920	6	2-3	3F	Same	8	3S	3F	Very slightly more lustre
U24921	6	3	3F	8	4-5S	3F		
U26495	6	2-3	3F	Same	8	3-4S	3F	Very slightly more lustre
U26494	6	3	3F	8	4-5S	3-4F		

*The legend is defined at the base of Table 1(a)

Table 3(b)

Exterior exposure test results—longer term—four years

Sample number	Four years exterior exposure Durban				Four years exterior exposure Pretoria			
	Chalking	Lustre ^{2(b)}	Colour fastness rating	Panel rating	Chalking	Lustre ^{2(b)}	Colour fastness rating	Panel rating
U18657	7	4	3FD	Same	9	5S	3FD	Same
U21575	7	4	3F		9	5S	3FD	
U21577	8	1	3FYR	Almost similar	9	3-4S	3FD	Imported, slightly more lustre
U21576	8	2	3FYR	Same	9	4S	3FD	
U21578	8	1	3FYR		8	3-4S	3FD	Imported, slightly more lustre
U21579	8	2	3FYR		8	4S	3FD	
U22426	9	2	3FY	Same	9	5S	3F	Same
U22427	8	2	3FY		9	5S	3F	
U22428	8	2	2D	Same	9	4-5S	4D	Same
U22429	8	2	2D		9	5S	4D	
U22430	8	2	4FY	Same	9	4-5S	4F	Imported, slightly more lustre
U22431	8	2	4FY		9	5S	4F	
U28869	8	2	2D	Same	9	4S	4D	Imported, slightly more lustre
U28868	8	2	2D		9	5S	4D	
U24920	8	2	3FY	Same	9	4-5S	3F	Imported, slightly more lustre
U24921	8	2	3FY		9	5S	3F	
U26495	8	2	3D	Almost similar	9	4-5S	4F	Imported, slightly more lustre
U26494	7	2	3DY		9	5S	3F	

*The legend is defined at the base of Table 1(a).

Table 4

Total print film thicknesses, determined after four years exterior exposure, micrometres

Sample number	Durban								Pretoria							
	One coat MIO paint				Two coats MIO paint				One coat MIO paint				Two coats MIO paint			
	Mean	Max	Min	Std dev	Mean	Max	Min	Std dev	Mean	Max	Min	Std dev	Mean	Max	Min	Std dev
U18657	94.8	115	74.2	14.3	137	159	122	11.9	94	111	76.5	11.2	136	163	119	13.0
U21575	104	123	82.1	12.1	147	175	124	13.8	105	133	82.3	15.5	149	185	123	18.8
U21577	79.9	96.5	67.1	9.8	150	183	105	17.2	75.6	90.9	53.8	12.4	135	147	124	9.0
U21576	125	143	112	9.1	163	181	142	11.0	126	155	106	16.1	166	196	143	19.4
U21578	105	118	94.4	8.1	156	172	144	7.5	86.4	98.3	70.6	8.3	160	176	137	12.9
U21579	85.1	100	73.2	8.4	134	147	121	6.5	81.4	105	64.8	13.1	122	137	104	9.2
U22426	131	149	133	12.1	166	187	143	12.9	117	139	103	10.7	154	175	141	11.2
U22427	109	113	81.4	10.3	137	147	112	14.8	107	131	87.6	14.1	142	155	124	11.1
U22428	108	129	83.2	12.8	157	177	120	17.4	107	126	83.5	10.5	151	173	138	11.5
U22429	115	136	104	10.6	147	167	130	11.7	106	123	92.5	9.7	140	176	106	22.2
U22430	111	134	86.8	14.7	162	203	136	18.6	121	142	107	11.2	168	180	147	9.0
U22431	124	146	92.1	14.7	161	193	117	20.5	104	122	70.2	15.4	139	175	92.8	20.4
U28869	119	140	65.5	19.0	161	192	134	14.7	117	134	95.8	11.5	154	185	134	15.0
U28868	144	167	101.6	17.3	242	277	178	30.7	138	170	107	18.7	271	330	202	32.9
U24920	134	178	112	19.8	227	253	212	13.4	163	192	137	14.5	207	237	161	19.8
U24921	120	151	97.7	14.1	200	215	180	12.8	136	149	127	7.5	172	199	144	17.1
U26495	132	162	97.3	20.5	216	235	138	26.9	128	155	112	11.0	177	192	146	13.3
U26494	114	131	96.5	10.9	159	179	149	10.4	112	139	88	16.9	159	183	132	15.9

i.e. Pretoria versus Durban, was considered to have much more significance than any effect due to the variable qualities between the two sources of the MIO pigment.

The MIO paint systems, formulated using oleoresinous vehicles, showed limited resistance to the severe laboratory salt fog test. For severe coastal or polluted environments, high quality resin systems including chlorinated rubber, polyurethane or two-pack epoxy systems may be required.

ISO DP1248, Iron oxide pigments for paints—specifications and test methods

On 21 February 1985, in a letter to the German Secretariat for the above proposed draft specification, from the South African representative on the ISO Committee concerned, it was intimated:

1. That the proposed new electron microscope photographic standard limits for MIO pigments were impracticable and therefore unacceptable; and
2. that their proposed illustrative photographs of back-illuminated microscope translucent ruby-red MIO pigment particles with the inadequate test method detail as provided by the Secretariat were also impracticable and therefore also unacceptable.

It was submitted to the ISO Secretariat that paint formulation variables, and variable exposure test conditions, were of greater importance than any other possible variables existing between the Austrian and South African sources of pigment supply. It was also submitted that the South African source of MIO was acceptable to ISO DP1284.

It must be accepted that the Austrian MIO was based upon superior grade ore and that the Austrian MIO pigment has superior lamellar qualities. However, as shown by this report, the South African MIO pigment grades have

significant lamellar properties, sufficient to develop ample lustre formation, as shown by the four year exterior exposure results at Pretoria, and after the 1,000 hours exposure to ASTM: G53 artificial weathering (QUV).

Whereas there are difficulties in establishing the necessary special performance criteria (other than the general requirements detailed in the relevant tables in ISO DP1248) for the various sources of supply of MIO, the following procedure may form the basis of a practical test for the paint manufacturing industry, especially for paint manufacturers having access to ASTM: G53 test facilities:

Prepare two paints based upon a standard alkyd medium, and a standard paint formulation, and include respectively:

1. a "standard Austrian Grade MIO pigment", and
2. the "alternative MIO pigment".

Apply one coat of each paint, under controlled conditions, to separate aluminium test panels. After due aging, expose the painted panels to 1,000 hours ASTM: G53 artificial weathering, using the test cycle as detailed in the test procedures mentioned previously.

(QUV test panels are of superficial area 150 mm × 75 mm, and have an exposure test area of 123 mm × 63 mm).

Apart from complying with the other relevant requirements given in the tables of ISO DP1248, the "alternative MIO pigment" paint film after the test exposure shall yield a rating of not less than four out of five of the lustre developed by the paint formulated upon the "standard Austrian Grade MIO pigment".

Bibliography

- a Fancutt, F., "Micaceous iron oxide", UK, Paint Manufacture, Dec 1965, p 43.

- b Fancutt, F. and Hudson, J. C., "Micaceous iron oxide as a pigment in paints for protecting steel", UK, Corrosion Prevention and Control, April 1970, p 18.
- c Scotland, N. J., "Micaceous iron oxide—today's best safeguard for the future", UK, Paint Manufacture, Jan 1976, p 22.

References

1. Bishop, D. M., "Micaceous iron oxide pigments", *JOCCA*, 1981, 64, 57.
2. Wiktorek, S. and John, J., "Micaceous iron oxide in protective coatings", *JOCCA*, 1983, 6, 164.

OCCA meetings

Hull Section

The history of Marks & Spencer

Members of Hull OCCA had great pleasure in welcoming the President, Frank Redman and his wife Peggy, who came all the way from Darwen especially to attend the Ladies' Evening on Monday, 2 March.

The occasion was a talk on "The History of Marks & Spencer" given by management trainees Miss Catherwood and Miss Carney attached to the Hull Branch.

The talk covered the early beginnings of Michael Marks' Penny Bazaar up to the present company with a £4 billion turnover and shops in Paris, New York, Canada and Japan.

Among the interesting facts underlining M & S's success are a claimed 40% of all consumer spending and £1 in every £7 spent on clothes in the UK. The Marble Arch store has the highest turnover per area of any store in the world and British food accounts for the largest portion sold at the Paris store.

The trouble taken over quality control was underlined. For example, all food is checked on arrival at each store and random tastings carried out.

Applicants for management positions can amount to 45,000 culminating in a selected few attending a two days final selection process whilst staying in a hotel.

A lively question time followed when all questions were dealt with in an extremely competent and amusing manner.

The Chairman, Michael Gamon, in proposing a vote of thanks congratulated the two lecturers on their presentation.

The evening finished with a buffet supper.

The next social event will be the Treasure Hunt on Friday, 26 June. Details will be announced in the June OCCA Bulletin.

M. Gamon

Eastbourne Conference Details see Inside Front Cover

3. Carter, E., "Micaceous iron oxide, a versatile pigment", *Paint and Resin*, Aug 1985, 30.
4. Schmid, E. V., "The use of micaceous iron oxide in long term corrosion protection", *Pigment and Resin Technology*, Jan 1986, 4.
5. Draft proposal ISO DP1248, Iron oxide pigments for paints—specifications and test methods. International Organization for Standardization technical subcommittee document ISO/TC35/SC2/N367, dd 1984-11-20, NNI Netherlands.

letters

Maleinised bisphenol oils in water-thinnable coatings

Sir,

A recent article¹ demonstrates the use of bisphenol compounds as the oil-yielding polyols in the preparation of maleinised oil type resins, and recommends these as binders in water-thinnable coatings.

Bis(3,5-dihydroxy-phenyl)propane, as a partial replacement of the other bisphenols, seems to offer a means for increasing the functionality and hence the toughness of the film, and it is of interest to see how it compares, in this respect, with pentaerythritol.

The use of higher viscosity and acid value versions of these resins is worth considering as binders in stoving electrodeposition coatings, and where alkali resistance is expected to be still better than in similar films produced by other methods of application, and also to see what kind of corrosion resistance results in these.

24 Conway Street,
London W1

Yours faithfully,
E. Constantinides
18 March 1987

References

1. Ramadan, A. M., Moustafa, M., Darwish, S. I. and Naser, A. M., *JOCCA*, 1987, 70, 75.

London Section

Wood coloration and waxes

The fourth technical meeting of the 1986/87 session was held at the Pearly King, Bromley E3, on Thursday, 12 February, when some 30 members and guests were treated to a double billing on the subject of wood finishing. Adrian Abel, Industrial Manager for Inks and Special Fields of the Colours Division of Hoechst UK talked on 'The Coloration of Wood'.

He described how as a natural product wood has an aesthetic appeal which is largely lost when it is given a coating which obliterates the surface. The many ways of colouring wood which not only allow the surface grain to be seen but can enhance its beauty were demonstrated. Various dyestuffs were described and their effects on different wood surfaces, e.g. open grain, closed grain etc. Problems associated with their application and permanence were also discussed as was the use of pigments where transparency can be difficult to overcome.

The second talk was given by Paul Holifield, Product Manager for Waxes in the Chemicals Department of Hoechst UK on the Application of Polyethylene Waxes in the Wood Finishing Sector.

Different types of waxes such as paraffin, micro-crystalline, montan etc were mentioned with respect to their influence on wood treatments but more specifically the applications of polyethylene waxes were described.

The properties of polyethylene waxes make them eminently suitable for wood finishes where properties such as anti-blocking, sandability, matting etc, are important. Particularly described and illustrated was their use in the new exterior wood stain finishes where their 'water beading effect' would be used to advantage. Additionally the matting of PU and N/C lacquers was discussed and the synergistic effects in combination with silicas.

The vote of thanks to the speakers and especially to Hoechst UK who kindly sponsored the meeting was proposed by Barry Canterford.

D. Bannington

Manchester Section

Software for colour control

The first technical meeting of 1987, held at the Pack Horse Hotel, Bolton, was attended by 35 members and guests and a paper entitled "Software for Colour Control", was presented by consultant Dr D. A. Plant.

Initially the lecturer briefly outlined the history of colour control, shade matching first being introduced into the textile field and then adopted by other industries. With surface coatings, the number of colour combinations are much fewer than in dyeing fabrics, and the most important aspects of colour control are shade passing, and batch correction.

Reflectance spectra can be measured on either a Spectrophotometer which presents the results as a curve or a series of numbers, or on a colourmeter which will give a single reading. Spectrophotometer measurements are absolute, but the illuminants vary considerably. The fundamental colour system is the CIE, but this is not practical for colour measurement in industry, the LAB system being more useful from this respect.

In order to obtain a better correlation between visual and instrumental results various colour systems have been developed. The original, was the Hunterlab system of 1958, this was followed by the ANLAB, and then the CIE Lab systems. From the late 1960's and early 1970's the CIE Lab system has been the best available, however, the later JPC 79 is somewhat better, and its successor, which contains minor improvements, known as CMC (2.1), is being proposed as a new BS Specification.

The lecturer concluded his talk by outlining how a colourist's report on colour difference varies depending on the colour, and hence the Hue/Hue difference system used

to describe colour difference.

Following a question and answer session, a vote of thanks was proposed by Steve Wilkinson. Members were then able to examine two colour systems which were on display.

Water the versatile ink solvent

On the evening of 16 March, 86 members and guests attended the meeting held at the Silver Birch, Birchwood Centre, Warrington, to hear a paper presented by Mr Eric Alston, the Sales Manager, General Industrial Division of Allied Colloids, entitled "Water the Versatile Ink Solvent".

The lecturer commenced with a brief introduction to Alloid Colloids, and the history of water as a non-hazardous solvent. Modern resins are usually copolymers of acrylic type monomers, used either as alkaline solution, or emulsions. The available monomers can be classified into three groups:

1. Backbone—Methacrylate or Styrene derivatives
2. Modifying Monomers—Acrylates
3. Functional Monomers—Wide variety of monomers used as a small proportion of the polymer

Because acrylics are synthetic, it is possible to design polymers for special uses and properties, the optimum being to have a polymer with a wide molecular weight distribution.

The alkali used to neutralise ink formulations can affect the properties of the ink, the best being:

Monoethanolamine
Ammonia
Dimethanolamine

Because they are not true solutions, transfer additives are required to increase the wet tac, and these must be highly water soluble.

The lecturer concluded by outlining the uses of the currently available polymers and the many new developments currently under consideration.

After a brief but interesting question time, a vote of thanks was proposed by Mike Nixon, the section secretary, and those attending were able to participate of a buffet sponsored by Allied Colloids.

A new generation of heterocyclic pigments

Sixty-five members and guests attended an extra meeting of the Manchester Section held at the St James's Club, Manchester, on Monday, 6 April 1987, to hear a paper on "DPP Pigments: 1,4 Diketo-Pyrrolo (3,4-') pyrroles" presented by Dr A. Iqbal of Ciba-Geigy Ltd, Switzerland.

The lecturer commenced by outlining the reasons why the number of new pigment types developed in recent years had been few and far between. The background of this new

discovery was then discussed and the properties of earlier compounds described. Varying the substituents gave a wide range of colours, which were non-bleeding.

The crystal structure of the new pigments was outlined, and it was explained how, by controlling crystal growth, one can get very good hiding, or very good transparency with the same pigment.

The original synthesis did not give yields of commercial value. The experimentation carried out to determine the reaction mechanisms, and develop an economically viable process, was outlined, from the initial discovery in October 1980, to the material's launch in September 1986.

Dr Iqbal concluded his talk by comparing in detail, the performance of two of the new pigments in automotive finishes, with currently available raw materials.

The lecture was followed by a lively question and answer period, a vote of thanks was proposed by Donald Plant, and those present were able to participate in a buffet sponsored by Ciba-Geigy.

M. G. Langdon

Midlands Section

Novel corrosion inhibitors

On 19 March a meeting was held at the Clarendon Suite, Stirling Road, Birmingham. The lecture was given by Mr T. Fletcher of BP Ventures.

BP is a major paint user requiring high standards of protection for pipes, tanks, oil-rigs, etc. Although properly formulated paints on conventional pigments give good performance, worries about lead and chromium from both health and safety and environmental considerations led to a search for a new system of corrosion inhibition. Most newer non-toxic alternatives being relatively unproven. Using their experience of inorganic oxide catalysts BP Ventures developed their "Shieldex" ion exchange pigments.

After a brief but detailed survey of traditional and newer corrosion inhibiting pigments describing mechanisms, advantages and disadvantages, Mr Fletcher went on to describe Shieldex types.

These are based on high surface area hydrous inorganic oxides such as alumina or silica-gel. The hydroxyl groups can be exchanged for a variety of cations or anions in an acid/base reaction. Extensive tests showed calcium/silica to be a clear leader.

Examples of the mechanisms showed how in a zinc chromate primer the level of zinc chromate could be reduced from 15% to only 2% by the incorporation of Shieldex. The new formulations are more responsive, release chromate only on demand and maintain film integrity. Shieldex is not however suitable for acid environments such as etch primers.

Slides then showed the performance of Shieldex

formulations against other pigments in a range of resin systems on various substrates and also in large scale field trials. The results were such as to persuade BP to specify Shieldex for all paintwork, both repaint and new construction.

The lecture period closed with questions and a vote of thanks from chairman Dr G. Lewis.

J. Gascoyne

Midlands Section Trent Valley Branch

Novel corrosion inhibitors

The final technical meeting of the current session, held on 26 March at The British Rail School of Engineering, Derby, was presented by Mr T. E. Fletcher, of BP Research Centre, who, under the title of "Novel Corrosion Inhibitors", described the development of the new product being marketed under the trade name of 'Shieldex'.

Work on ion-exchange pigments began at the BP Research Centre at the beginning of the 80's as a result of increasing concern over the toxicity of the existing lead and chromate based pigments and doubts about the ability of emerging non-toxic replacements to provide equivalent protection. This led to the development of a range of pigments based on ion-exchanging anti-corrosive ions onto amorphous inorganic oxides, such as alumina and silica. Various anions exchanged on alumina were investigated and it was realised that anti-corrosive cations could be exchanged on silica. Anti-corrosive screening by electrochemical tests and in paints indicated that calcium ions exchanged onto silica had great potential as an anti-corrosive pigment and subsequently much effort has been devoted to the development of this non-toxic (lead/chromate free) pigment. Its mode of action is to trap aggressive ions and release anti-corrosive ions.

The use of chromate pigments has largely died out in the maintenance painting sector of the industry, to be replaced by zinc phosphate although it is accepted that the performance of such primers is questionable. Evidence in the form of projected colour slides illustrating accelerated corrosion testing and natural weathering testing on coated panels showed the superior performance of Shieldex paints in comparison with systems based on phosphates and other lead and chromate free pigments. Within the BP Group, trials with calcium ion exchanged silica paints have been conducted since 1982, resulting in a gradual change in specifications from zinc phosphate to Shieldex (calcium/silica).

Further evidence illustrating many accelerated corrosion tests on coated panels showed that calcium/silica, at equal volume loadings to zinc or strontium chromate, is capable of providing equivalent anti-corrosion protection. A wide range of substrates and paint formulations have been investigated and the pigment has performed extremely well.

It was concluded that the simple calcium ion exchanged

silica has demonstrated the ability to produce high performance and cost effective maintenance paint systems and to replace chromates in a wide range of industrial paint systems and there is potential for the further development of the technology which could extend its use even more widely.

After a most interesting and enlightening paper there was a lengthy question and answer session which was concluded by a vote of thanks proposed by Mr Jonathan P. Bourne and heartily endorsed by the large gathering.

J. C. Ellis

Scottish Section

Wood treatment in Scandinavia

On Thursday, 5 February, at the Hospitality Inn, Glasgow, Mr Thor Fjeldberg, Technical Sales Manager for Dyno Industrier, gave a lecture entitled "Exterior Wood Treatment in Scandinavian Countries".

The lecture opened with a brief history of the protection of wooden buildings from the 16th century. Surprisingly, some of the very old techniques are still used today on a limited scale.

In the 19th century with the development of what is now our modern surface coatings industry, Scandinavia extended the range of coatings used for protection of wooden buildings. These include well-known pigmented and varnish systems used throughout the latter part of the 19th and first half of the 20th century.

Today, there are four main types. Two solvent based—a surface coating and a stain. Also two water based types—again a surface coating and a stain.

There is a definite tendency to use more and more water based systems. Norway uses per capita more protective

coatings for wood than any other Scandinavian country and in Norway mostly water based systems are preferred. Research has shown that timber used in buildings, if protected when new, lasts much longer than weathered timber which will have lost resin and essential oils.

A rapid fire question time was ably fielded by Mr Fjeldberg with some rapid direct answers. He was accorded a hearty vote of thanks by an audience of some 35 members and visitors. Messrs Charles Tennant & Co, were thanked for the excellent buffet which they provided to accompany the lecture which was much appreciated by all.

R. L. Barrett

Transvaal Section

The South African Paint Industry 1987

On 28 January, Mr N. D. Peterson, Managing Director of Plascon Paints, talked on "The prospect of the South African Paint Industry in 1987". The current situation in South Africa creates many challenges and opportunities. Import and export problems caused by sanctions or the threat of sanctions force one to be innovative and smarter managers. Mr Peterson felt that the people of South Africa will solve the political deadlock in which they find themselves. He thought steady and positive political reform is an absolute pre-requisite to restoring confidence in the country and economy. Overseas confidence in this country and, relative to that confidence, new investment was vital, if the country was to prosper. Economic considerations included the creation of jobs, a major objective, and also the development of small businesses. Inflation must be controlled and it was essential to all that a stable exchange rate was achieved. There was a dearth of skilled people and management was under constant and ongoing pressure because of the skill shortages. Planning for 1987 and beyond would require a change in management skills to cope with the changes being experienced in a turbulent era.

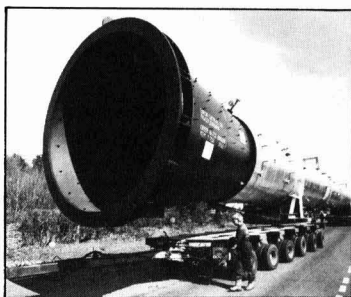
L. F. Saunders

news

EXXON expansion at Fawley and Gravenchon

As part of the current expansion of the Methyl Ethyl Ketone (MEK) plant at Exxon Chemical's complex at Fawley, UK, the company has just taken delivery of a large purification tower with a weight of 74 tonnes and a length of 157 ft. The tower was transported from nearby Calshot to Fawley supported on two trailers each with 48 wheels. The tower is the final part of this expansion project, and its installation will permit the completion and start up of the plant on schedule during May, with a production capacity for MEK increased to 90,000 tpa.

Also Exxon Chemical is expanding its Escorex 1000 catalytic hydrocarbon resin unit at Notre-Dame-de-Gravenchon,



Towering EXXON

France. The multi-million French Franc investment will expand the plant capacity by over 30% to 45,000 tpa. Engineering

work has begun and start-up of the new facilities is expected in the second quarter of 1988.

New production plant at BP Chimie Lavera

BP Chimie is to start production at Lavera of speciality glycol ether acetates. These products are low toxicity solvents intended to join its range of glycol acetates. The production plant costing FF 20 million will have a capacity of 30,000 tpa.

DTI project support for PRA

The Department of Trade and Industry has given approval for 50% support of two major new research projects at the PRA. Both are concerned with mechanisms, one of drier action the other of anticorrosive

paints, at a total cost of nearly £800,000 over a 3-year period commencing February 1987.

Sachtleben's huge dilute acid recycling plant construction given the go-ahead

On the 13th March 1987 the head of local government in Dusseldorf granted the licence for the building and operation of a dilute acid recycling plant (*JOCCA*, July 1986, p.195). The principal is Sachtleben Chemie GmbH, a subsidiary of Metallgesellschaft AG, Frankfurt. This plant will process dilute acid which had previously been dumped and will enable the Federal Republic of Germany to put an end to this type of dumping before the deadline set by the European Commission.

The EC resolution passed in 1983 decreed that dilute acid dumping in the North Sea should not be allowed to continue beyond the end of this decade. Dilute acid is a by-product of titanium dioxide production; the capacity of the new processing plant is sufficient to recycle the residue from three TiO₂ plants. This licence has been granted as a result of intensive negotiations between the Environment Minister of Matthiesen, Duisburg town council and Sachtleben.

Now that the question of the plant's capacity for additional residue has been finally clarified, Metallgesellschaft AG has reaffirmed its support for this 140m DM (approx. £48 m) environmental project.

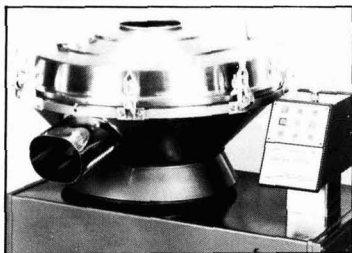
The Duisburg-Homberg recycling plant should be completed within two years. It will process 800,000 tonnes of dilute acid annually, most of the waste from three of the four titanium dioxide plants in Germany. A co-operation agreement has also been signed with Kronos Titan GmbH, Leverkusen. Customers are thus now guaranteed their supply of titanium dioxide well into the future.

products

New generation of impact testing equipment

Englemann of Alton, Hampshire has available a new generation of impact testing equipment called *Dartvis* and *Fractovis*. The *Dartvis* is a bench top falling weight model built to perform impact on standardised plate specimens. The system can be used with the new data acquisition system *Datavis*. The basic *Dartvis* offer a maximum impact velocity of 3.7 m/s. The *Fractovis* model is used for research into biaxial impact testing using a falling dart to achieve its reading. This system offers impact velocities up to 20 m/sec.

Reader Enquiry Service No. 40



Vibrasonic sieve system

New anti-blind sieve system

Russell Finex Ltd of London has introduced the *Vibrasonic* sieve system. This system solves the powder production sieving problem of mesh blinding which occurs when fine meshes become blocked with powder particles and blind it causing production to be halted. The problem is solved by using top grade stainless steel meshes with a high frequency ultrasonic vibration giving effective screening down to 40 microns. This system therefore eliminates the need for scrapers, brush gear etc.

Reader Enquiry Service No. 41

Bayer's PU compound cures underwater

Bayer of Newbury has introduced its *Blendur* polyether polyol and *Baymidur* triol products that combine to make the first polyurethane casting resin compound which is unaffected by moisture with no shrinkage, and cures with no foaming, even under water. The resin can be used in mould construction, as a coating compound, a permanently elastic repair, and for encapsulation. *Bayer* sees applications for these products in the repair and sealing of pipelines in offshore oil rigs or chemical plants.

Reader Enquiry Service No. 42



Bayer underwater PU Curing

OCCA ties at £4.25 each are now available from the Association's offices, with a single Association Insignia on either a blue or maroon background.

meetings

OCCANZ 25th Convention

OCCA New Zealand Ltd will be holding its 25th Jubilee Convention on "Timber—it's protection and decoration" from 22-26 July 1987 at Rotorua, New Zealand. For a registration form contact: OCCANZ Ltd, P.O. Box 5192, Auckland, New Zealand.

UK Corrosion 87

This exhibition of 80 plus stands will be held from the 26-28 October at the Metropole Hotel, Brighton. For further information contact: UK Corrosion '87, Exeter House, 48 Holloway Head, Birmingham B1 1NQ.

International Congress on Metallic Corrosion

The 10th International Congress on Metallic Corrosion will be held between the 7-11 November 1987 in Madras, India. For further information contact: Prof. K. I. Vasu, Chairman Organizing Committee, c/o Central Electrochemical Research Institute, Karaikudi 623 006, Tamil Nadu, India.

literature

South African Paint Digest

A softback book entitled the "South African Paint Digest" 106pp is available (surface mail R45,00, airmail R50,00) from: Keeble Publishing (Pty) Ltd, P.O. Box 3080, Johannesburg, 2000, South Africa.

bsi news

BSI publications are obtainable from: BSI Sales Department, Linford Wood, Milton Keynes MK14 6LE (orders by post only).

British Standards

The following publications are now available:

Test methods for paints: BS3900

Part B15. Rapid method for estimation of lead in liquid paints.

Part J1. Sampling of coating powders.

Part J2. Determination of particle size distribution of coating powders by sieving.

Part J3. Determination of the gelation time of coating powders.

Part J4. Determination of the storage stability of coating powders.

Binders for paints: BS6782

Part 1. Method for determination of volatile and non-volatile content.

company reports

Part 2. Method for determination of saponification value (titrimetric method).
 Part 3. Method for determination of acid value (titrimetric method).
 Part 4. Method for determination of hydroxyl value (titrimetric method).
 Part 5. Method for estimation of colour of clear liquids by the Gardner colour scale.
 Part 6. Method for determination of softening point (ring and ball method).

Hot-applied threomplastic road marking materials: BS3262

Part 1. Specification for constituent materials and mixtures.
 Part 2. Specification for road performance.
 Part 3. Specification for application of material to road surfaces.

BASF AG

BASF reported that in spite of adverse conditions, business in 1986 was successful for BASF. While volume sales were good, almost all currencies continued to weaken, and prices of crude oil and petrochemical products declined substantially. BASF world net sales for the year ending 31 December 1986 was 43,083DM Million (-9.7% on 1985). BASF group pre-tax profit was 2,627DM Million (-13.6%).

Hoechst AG

Hoechst AG reported that 1986 was highly profitable while sales declined. Hoechst 1986 worldwide sales of 38,014DM Million

(-11% on 1985). Hoechst AG pre-tax profit 3,211DM Million (+1.7%).

Hoechst UK Ltd Group

Hoechst UK Ltd Group sales increased marginally to £587 million with exports increasing to £68m. Hoechst UK increased its investment in Berger by £25m. Paint sales for 1986 was £289m (-4.25% on 1985). Berger Britain reported a trading profit growth of £2m to more than £3m in 1986.

Tioxide Group PLC

Tioxide reported that the Group achieved another successful result in 1986 which saw a continuation of the firm trading conditions of the previous year. Group pre-tax profit was £114.35 (+ 48% on 1985) on a turnover of £506m (+ 19%).

OCCA news

Scottish Section Annual Dinner Dance

Despite heavy snow, 260 members and guests braved the elements to attend the annual dinner dance of the Scottish Section.

The function was held on 16 January at the Hospitality Inn, Glasgow, where the principal guests included the OCCA President and Section Chairmen from Newcastle, West Riding and Bristol along with their wives.

As in previous years a feature of the meal was haggis served as a starter and Hugh Munro—Scottish Section, performed the address to the haggis.

The main course of roast baron of beef was ceremoniously carried into the room led by a piper. The OCCA President Mr Redman carved the first slice.

Following an excellent meal Mr Ron Barrett, Chairman—Scottish Section, toasted the health of the guests, Mr Redman replied on behalf of the guests.

The evening continued until 1 am with dancing to the Art Sutter Trio. An enjoyable evening was had by all.

R. A. Hunter

Obituary Thurston Entwistle

J. Clark writes:

It was with disbelief and shock that we learned of the sudden death, on 4 January, at his home in Swainby, N. Yorks., of Thurston Entwistle at the early age of 57.

Thurston had spent all his working life in the paint industry. He joined W. J. Leigh

from Bolton County Grammar School, and continued his studies at Bolton Technical College. Following National Service in the RAF from 1947-49 he joined Firwood Paint and Varnish Co Ltd, as a laboratory Paint Technician and resuming his studies he qualified as a GRIC in 1958, when he joined H. Davidson at Liverpool as Assistant Chief Chemist. His move to Tioxide came in 1964 where his wide experience in the paint industry proved invaluable in the air drying paint and industrial paint sections of the Technical Service Department, and led to his appointment as Technical Service Manager of the South Africa Subsidiary, South African Titan Products (now Tioxide South Africa) in 1969. He remained there until 1973 during which time he was elected a Fellow of the RSC, then he returned to Central Laboratories in Stockton to manage the Industrial Paints Section within the Technical Service Department. In this capacity he travelled widely in Scandinavia, East and West Europe, the Middle and Far East and his unassuming manner and great knowledge and experience gained him friends and respect throughout the world.

He continued to use his experience to the benefit of his employers and their customers when he joined the Manchester Area Sales Office as a Senior Representative in the Spring of 1986, a position he was to enjoy for a tragically short time.

Throughout his career he was a staunch supporter of OCCA, and few members can have given more varied service. Apart from his many excellent lectures at Section Meetings and at Conferences, and his contributions to the pages of the journal he was Secretary of the South African Sections during his time there and a founder Fellow of the Professional Grade. On his return to

England he continued to represent the South African Sections on Council and gave sterling service as a member of Publications, Professional Grade and Finance Committees, the International Working Groups, and as a Vice President from 1984-1986. At section level as well as service on the Newcastle Section Committee he was an honorary auditor at the time of his death.

Always willing to pass on his knowledge and experience, he was a counsellor for the Open Tech. Paint Technology Course.

In his younger days he enjoyed tennis and golf and both he and his wife were active in the Scout and Guide movement. Since moving to Swainby he played his part in the social life of the village and enjoyed walking in the neighbouring countryside, occasionally in pursuit of a somewhat errant golf ball.

He leaves a widow, Maureen, two sons and a daughter, to whom we extend our deepest sympathy.

new members

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

Ordinary members

Bell, C. H. A., BSc (*Thames Valley*)

Bermudez, R. A. (*Transvaal*)

Bradley, R. (*West Riding*)

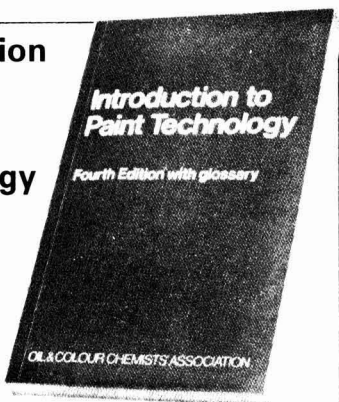
Brussman, O. (*General Overseas — Germany*)

Dooley, D. B. (*London*)

Ellis, R. A., BSc, PhD (*Manchester*)

Frean, A. J., BSc, PhD (*Trent Valley*)

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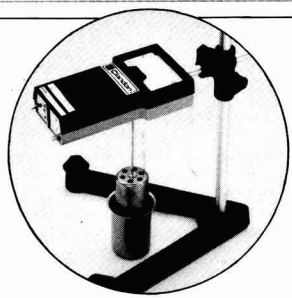
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OCCA NEWS

- Hambling, R. E., MA (*General Overseas –Malta*)
 Matthews, S. T., BSc (*West Riding*)
 O'Neill, M. (*Manchester*)
 Phillips, I. D. (*Manchester*)
 Rees, T. M., BSc, MSc (*Midlands*)
 Robertson, H. (*Newcastle*)
 Rourke, F. (*Manchester*)
 Russell, L. J., BSc (*West Riding*)
 Taylor, P. G. (*West Riding*)
 Ubbey, N. S. (*Thames Valley*)
 Whiteman, P. F. C. (*Thames Valley*)
 Whittle, N. T. (*Manchester*)

Registered students

- Goddard, D. J. (*Manchester*)
 Tennant, I. G. (*Manchester*)

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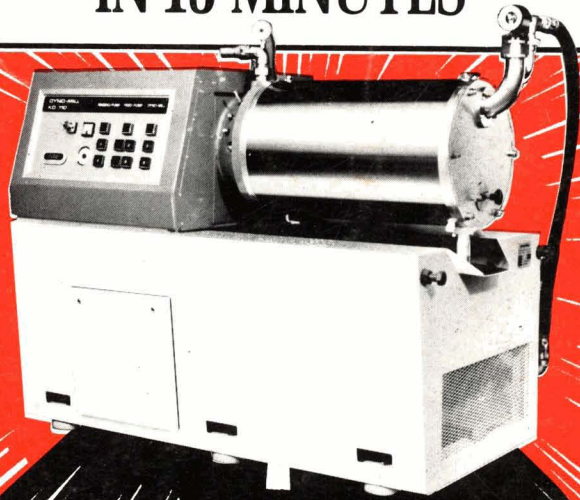
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