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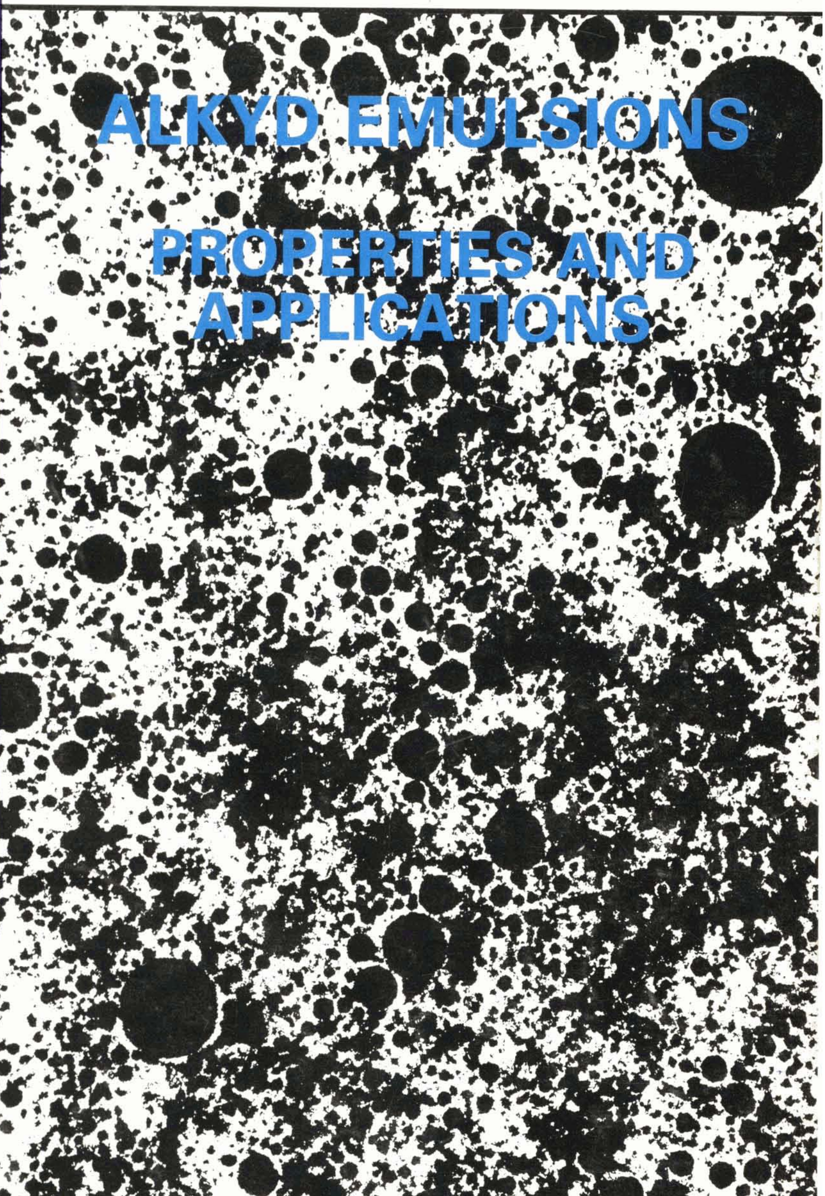

ALKYD EMULSIONS

PROPERTIES AND APPLICATIONS

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- Zinc and zinc alloyed atomised dusts
- Novel polyamide epoxy curing agents

Journal of Oil and Colour Chemists' Association,
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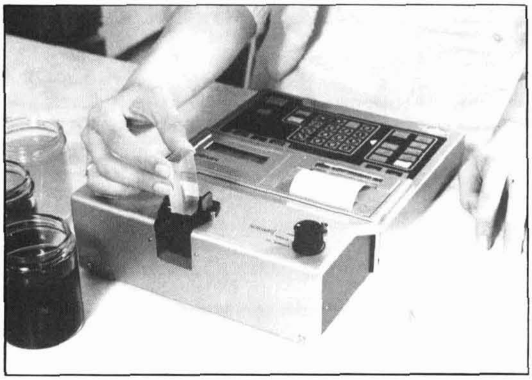

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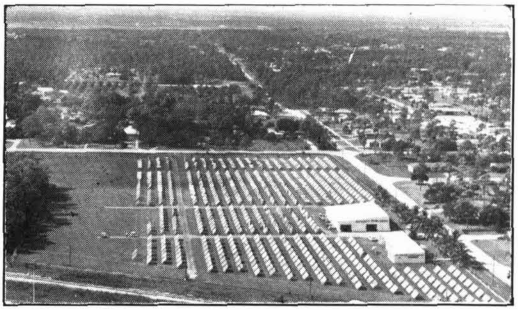

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Alkyd emulsions, properties and applications

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Introduction

With increasing restrictions through legislations on the use of organic solvents, the use of water based paints will increase further in the years to come, replacing mostly white spirit diluted alkyd paints both for interior and exterior use.

Compared with paints based on polymeric dispersions, white spirit diluted alkyd paints have certain technical advantages. In general alkyd paints show better gloss, levelling and adhesion to chalky substrates than emulsion paints. The penetration into porous substrates is good and the oxidative drying prevents thermoplasticity.

A lot of work has been done in recent years trying to improve the properties of dispersion paints in order to make them perform closer to the performance of organic solvent diluted alkyd paints.

As presented in Table 1 there are certain paint properties that can hardly be obtained when using polymeric dispersions as binders.

Table 1

Comparison of properties

	Alkyd resin	Polymeric dispersion
Penetration	+	−
Adhesion to chalky substrates	+	−
Thermoplasticity	+	−
Drying time	−	+
Levelling	+	−
Gloss	+	−
Discolouration	−	+

It is obvious, however, that storage stable alkyd emulsions would provide most of the desired properties.

The idea of incorporating oils or alkyds into dispersion paints is not new. For the last 25 years some dispersion paint formulations have been upgraded by using 5 to 30% of alkyd resin calculated on binder solids. Improvement in properties like penetration, adhesion and flexibility has been achieved.

Table 2 shows how alkyd additions can improve the exterior durability of a semi-transparent wood protective stain, based on acrylic dispersions. As long as only small amounts of alkyd have been incorporated, there are no serious problems with the storage stability of the formulations. If larger amounts of alkyd resin are used, problems with in-can stability are likely to occur. The alkyd resin particles tend to form clusters in the film and the distribution of the alkyd will be uneven as shown in Figure 1. The uneven distribution is likely to reduce the film strength and a better distribution of the alkyd resin could improve the paint performance.

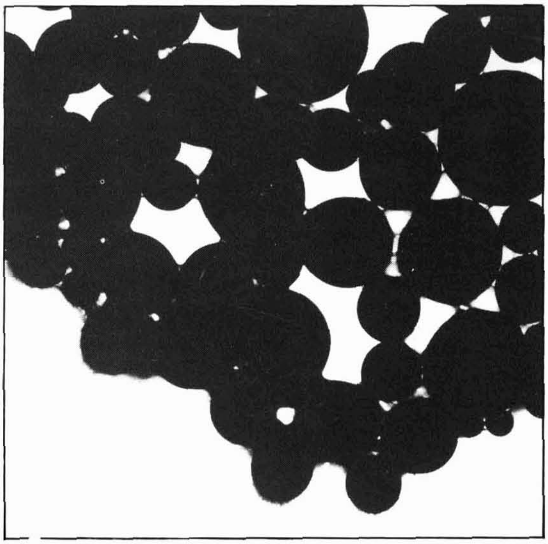


Figure 1. 80/20 ratio of acrylic dispersion to alkyd resin.

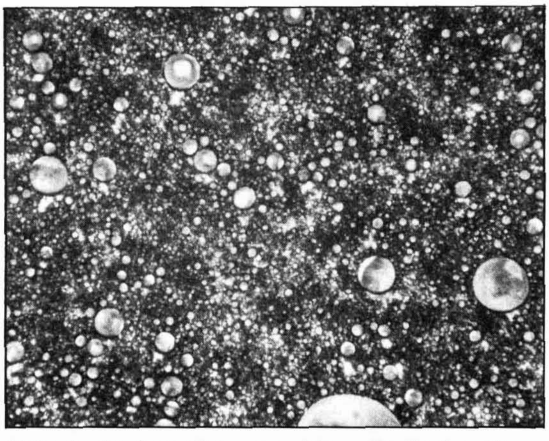


Figure 2. Alkyd emulsion prepared in cowles dissolver (Magn. x20).

Table 2

Exterior exposure of acrylic semi-transparent stains containing alkyd (2½ year 45° South)

Binder	Rating*
Acrylic	3
Acrylic/10% alkyd	4
Acrylic/20% alkyd	4-5
Acrylic/30% alkyd	4-5

*Rating 0-5, 5= best.

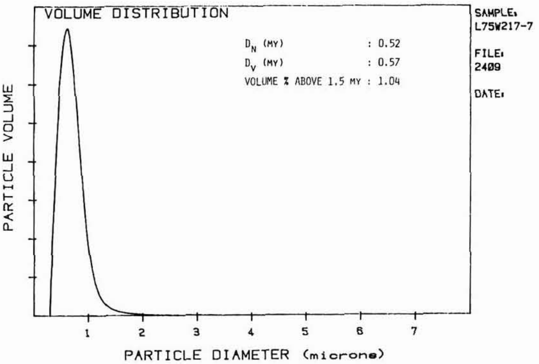


Figure 3. Particle size distribution for an alkyd emulsion.

When an alkyd resin is stirred into a water based formulation the particle size distribution will be very wide. Particles with a diameter of up to 0.5 mm are formed (see Figure 2). Such an alkyd emulsion will start to separate immediately after preparation and a solid alkyd layer will be formed in the bottom of the container after a couple of days. By studying in-can stability in relation to particle size, it was found that a particle diameter of 1.5 microns should not be exceeded in order to prevent separation.

If the particles are very small the amount of emulsifiers needed will increase due to the increase in the surface area that has to be protected. When the amount of emulsifiers used exceeds certain levels the alkyd resin will dry very slowly and the water sensitivity of the dried film will increase. Small particles can also cause a high emulsion viscosity which makes the emulsion difficult to handle, and the high viscosity is also fairly unstable. Figure 3 shows a preferred particle size distribution for an alkyd emulsion. As can be seen the average particle size is 0.6 microns and the volume above 1.5 microns is close to 1%. When the development of the alkyd emulsions started, the aim was to make emulsions from unmodified base resins in order to get paint properties as similar as possible to those obtained when using organic solvent diluted resins.

As the emulsifiers incorporated will lower the water resistance of the film it was of importance to have a hydrophobic base resin in order to counteract the effect of the emulsifiers. Tests with alkyds modified with for example polyethylene glycol have been run. This modification makes the resins easy to emulsify and it is possible to make emulsions with a very narrow particle size distribution. Major problems with these resins are slow drying water sensitive films and rather high and unstable emulsion viscosities.

Other factors affecting the emulsification of the alkyds are molecular weight distribution, oil length and type of oils used. A low molecular weight will make the emulsification of the resin easier. Alkyds with a high oil content are normally easier to emulsify than short oil alkyds. Tall oil resins are preferred to those based on soya or linseed oil.

Some typical data for commercial alkyd emulsions are given in Table 3.

Table 3

Technical data on alkyd emulsions

	T-75-W	L-75-W
Alkyd data:		
Oil type	Tall	Linseed
Acid	Phthalic	Phthalic
Polyol	Penta	Penta
Mol. wt. (GPC)		
Average by wt.	9000	9000
Average by number	2500	2500
Acid value	5-8	5-8
Emulsion data:		
Non volatile	49-51	49-51
Viscosity, mPa.s, v/23°C	10-30	10-30
pH, approximately	6	5
Particle size, microns	0.6	0.6
Sp. gravity, 23°C	1.00	1.01
Freeze/thaw, cycles	0	0
Storage life, at 23°C, min.	6 months	6 months
Tests in exterior stain:		
Touch dry (sand method)	2 h	1½ h
Hardness, Koenig, 1 day	15	17
Hardness, Koenig, 7 days	17	20

Table 4

*Drying of alkyd emulsions compared with white spirit diluted alkyds**

	T-75-M	T-75-M + DBP	T-75-W
Touch dry, hours	2		2
(Falling sand method)			
Hardness			
Koenig. sec.			
Days: 1	15	15	15
7	32	21	20
21	40	25	23
Solids content	50%	50%	50%

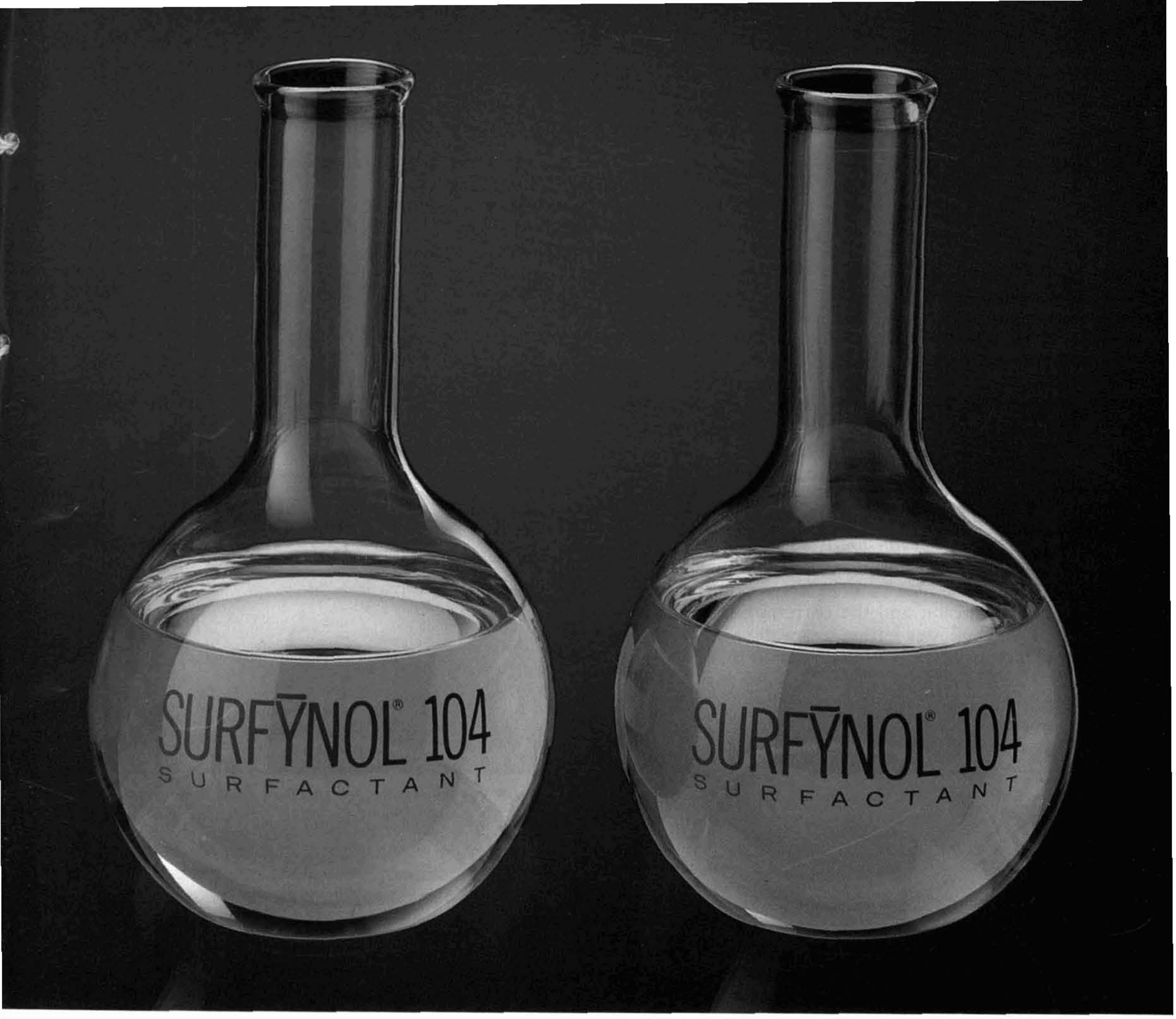
100 microns wet film

*T = tall oil, M = mineral spirit, W = water, DBP = di butyl phthalate, A = acrylic.

The solids content will normally be between 50 and 55%. It is possible to make alkyd emulsions with a solids content up to 60% but the emulsion properties are then not as good as for the 50% emulsions regarding ease of handling and storage stability. With a solids content of 50-55% and an average particle size of approximately 0.6 microns the viscosity is low and the emulsions are easy to handle. If the particles are too small or too large, viscosity increases can be observed. This is caused by interaction between the particles and the effect will differ with storage time and temperature.

Compared with most polymeric dispersions the alkyd emulsions do not possess the same freeze-thaw stability or stability against shear forces, due to the fact that the alkyd polymer is liquid. The storage stability is good. Additional stability can be achieved by adding non-ionic surfactants like nonyl phenol polyethylene oxide with an ethylene oxide content between 16 and 20. The addition should be kept below 0.3% as these surfactants tend to reduce the efficiency especially of associative thickeners.

Table 4 shows the drying properties of a T-75 type of resin. (Tall oil. Oil length 75%). The same base alkyd was used for a white spirit diluted and an alkyd emulsion clear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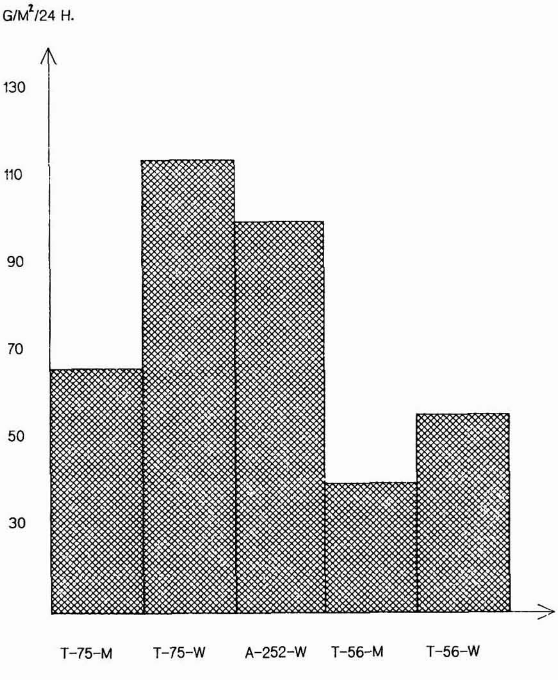
lacquer. The amount of metal drier is the same in both formulations calculated on binder solids. As can be seen there is no difference in sand drying time between the two systems. A study of the solvent evaporation from the two lacquers shows that during the first 20 minutes the water evaporated a little faster than the white spirit. However, after one hour close to a 100% of the white spirit has left the film while 3-4% of the water still remains in the alkyd emulsion film. Obviously this does not affect the sand drying of the alkyd emulsion film, unless a very thick film is applied.

The hardness of an alkyd emulsion film is only approximately half of the hardness obtained when measuring a white spirit diluted alkyd film. When adding an amount of plasticiser to an alkyd resin corresponding to the amount of emulsifiers used in the alkyd emulsion the final film hardness will be the same. The emulsifiers left in the alkyd emulsion film act as plasticisers giving a more flexible film which can be an advantage in exterior wood coatings.

The water vapour permeability of different binders has been measured and the results are shown in Figure 4. There is a significant difference in the water vapour permeability of the alkyd emulsion and the corresponding white spirit diluted alkyd, the latter letting less water vapour through. The permeability of T-75-W is similar to that of an acrylic dispersion. The water vapour transmission will be dependent upon the composition of the alkyd resin used. The T-56 type of alkyd with a lower oil length and a higher molecular weight is less permeable than the T-75. T-56-W has a much lower water vapour permeability than the acrylic dispersion. Thus it is therefore possible to vary the design of the alkyd resin within certain limits in order to obtain alkyd emulsions with different water vapour permeabilities.

Figure 4. Water vapour permeability of different binders. ASTM Cup.



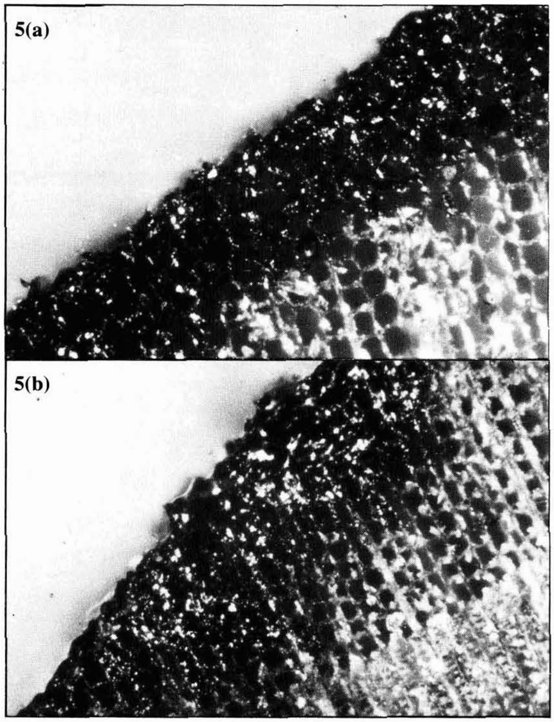


Figure 5. (a) Penetration of an alkyd diluted in mineral spirit into spruce, (b) penetration of an alkyd emulsified in water into spruce.

The water absorbtion and the water vapour permeability of different exterior stains have also been investigated using spruce panels as substrate in order to see if this would confirm the results from the testing of free films. Stains were prepared using the same T-75 base resin in all formulations. Four panels were coated: one with a white spirit diluted stain, two with the same stain containing different levels of non-ionic surface active agents and one with an alkyd emulsion stain. The theoretical film thickness was approximately 50 microns without taking the penetration into the wood into account.

When tests were run at 50 and 100% relative humidity no significant differences were found in the water vapour permeability between the four formulations. The results indicate that alkyd emulsions will provide the same protection against humidity as the corresponding white spirit diluted alkyds. The panels were then soaked in water for 24 hours and the water pickup was measured. The panel coated with the alkyd emulsion stain picked up 15% more water than the one coated with the white spirit diluted alkyd stain. The results indicate also that the water permeability can be related to the amount of surface active agents used in the formulations.

It is well-known that white spirit diluted alkyd resins will penetrate into wood and other porous substrates. Tests have been run in order to establish if the alkyd emulsions would penetrate to the same degree. Tests made on spruce confirms that the penetration of alkyds into the wood is nearly the same for white spirit and water diluted stains. In Figure 5 the penetration depth is approximately 170 microns for both stains.

The penetration depth will vary with factors such as molecular weight and resin concentration in the stain applied. With a molecular weight of 10,000 excellent penetration is achieved but the penetration depth will decrease with increasing molecular weight. At 100,000 the penetration is achieved but the penetration depth will Figure 6 shows the relation between alkyd solids in the stains and the penetration depth. By increasing the amount of alkyd resin applied on the surface a better penetration of alkyd into the wood is achieved.

Tests made on chalky substrates confirm that the alkyd emulsions have a penetration similar to white spirit based alkyd resins. (Figure 7). The differences shown in the test results are related to the slower drying of the alkyd emulsions.

Formulations

Alkyd emulsions have been used as sole binders for exterior paints and stains and experience shows that the exterior durability is somewhat better than for the corresponding white spirit diluted alkyds. A 20% increase in service life has been found.

However, most of the actual formulations used are based upon mixtures of alkyd emulsions and polymeric dispersions. By blending a polymeric dispersion with an alkyd emulsion it is possible to obtain paints possessing the distinctive properties of both vehicles, such as good penetration and fast drying.

The polymers are in general not compatible and will with a very few exceptions not form clear films. This can be seen on drawdowns made on glass panels but is normally not visible on wood substrates. This incompatibility will affect the gloss when formulations are based on a mixture of alkyd emulsions and polymeric dispersions. Such paints will normally show a lower gloss than the paints based on the separate binders. This means that so far it has not been possible to formulate a real high gloss paint using a mixture of alkyd emulsion and polymeric dispersion as the binder.

Alkyd emulsions have a film forming temperature below 0°C while polymeric dispersions have an MFT above 0°C. By measuring the film forming temperature of different blending ratios of alkyd emulsion/polymeric dispersions it is possible to establish at which blending ratios the film is a continuous alkyd or dispersion film. The turning point will normally be in the range of 30 parts of alkyd to 70 parts of polymeric dispersion calculated on resin solids. (Figure 8). The turning point will differ slightly with the particle size of the polymeric dispersion and with the molecular weight of the alkyd resin. An alkyd resin with a molecular weight of 10,000 will have a turning point at 30 parts of alkyd while an alkyd with a molecular weight of 300,000 will have a turning point close to 50 parts of alkyd. More alkyd will be needed to form a continuous film. Thus for formulation work it is necessary to know the changes in the continuous phase. By choosing the correct continuous phase it is possible to decide whether the alkyd resin or the polymeric dispersion properties will be dominant in the ready made paint.

Interior paints

In order to modify a polymeric dispersion based paint without changing the drying time, 30% or more of binder solids could be replaced by an alkyd emulsion. The quantity

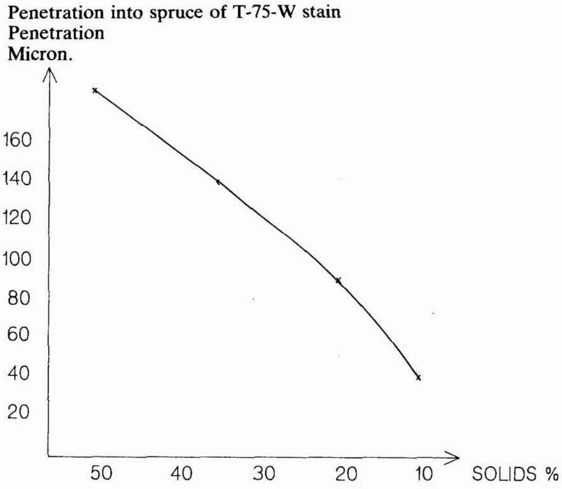


Figure 6. Comparison between penetration depth into spruce and alkyd solids %.

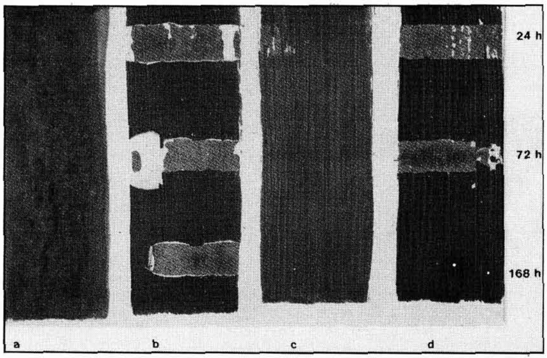


Figure 7. Adhesion of stain onto a chalking surface after 24 hours, 72 hours and 168 hours: a) Alkyd dissolved in mineral spirit, b) Acrylic dispersion, c) Alkyd emulsified in water, d) Alkyd emulsion and acrylic dispersion (50/50).

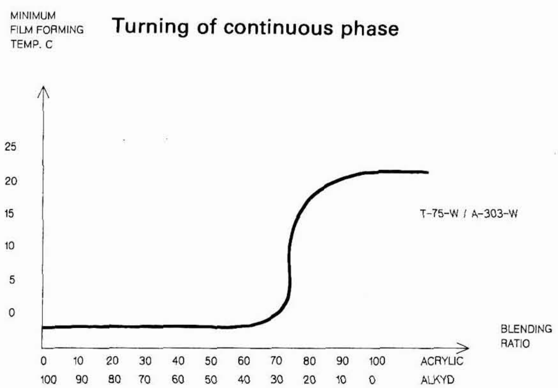


Figure 8. Minimum film forming temperature of different blending ratios of an alkyd emulsion and polymeric dispersion.

will to some extent be dependent upon the molecular weight of the alkyd resin.

An addition of alkyd emulsion should improve properties like:

- * Washability
- * Wet adhesion
- * Adhesion to chalky substrates
- * Thermoplasticity

As can be seen from Table 5, significant improvements in these properties can be achieved when using 20-25% of alkyd emulsion calculated on binder solids. When 30% is added no further improvement in the paint properties can be seen and the drying time is increasing.

Table 5

The effect of the addition of an alkyd emulsion to PVAc paint. Tests were made one week after application on flat interior PVAc paints (45% PVC) containing the alkyd emulsion

Content of alkyd emulsion (Calculated on binder solids)	0%	10%	20%	25%	30%
Drying time, minutes (Sand drying)	15	15	15	15	30
Koenig hardness, seconds					
24 hours	25	45	49	49	52
1 week	29	66	76	73	71
Washability, cycles (500 grams, brush load)	10	15	250	250	250
Wet adhesion (Gloss substrate)	0	1	5	5	5
Adhesion (Chalking substrate)	2	3	3-4	4	4
Stackability (90°C load 0.2 kg/cm^2)	3	3	4	4-5	4-5
Spot test (Lipstick)	4	4-5	4	3-4	3

Where the figures do not refer to actual measurements, an evaluation scale ranging from 0 to 5 had been used (5 = best).

High gloss paints can be made using alkyd emulsions as sole binders but the alkyd emulsions have to be specially designed for these formulations. With a proper formulation initial 60° gloss readings of 95 can be achieved. Compared with paints based on white spirit diluted alkyds there are a couple of problems involved using alkyd emulsions as binders for high gloss paints.

In order to obtain maximum gloss and minimum yellowing a very careful selection of raw materials have to be made and the number of different additives that can be used is somewhat limited. Biocides, surface active agents and antifoamers can all affect gloss and discolouration. Driers have to be selected among the water dilutable grades and metals like calcium and manganese have to be avoided due to stability and discolouration problems.

High gloss paints based on alkyd emulsions will not dry as fast as corresponding white spirit diluted paints if thick films are applied.

Tests were made adding an acrylic dispersion to the paint but as seen in Table 6 no improvements in the drying time were achieved. As mentioned earlier due to incompatibility between the binders a drop in gloss can be expected.

Table 6

Addition of polymeric dispersion to gloss alkyd emulsion paint

Binder	Alkyd emulsion	Ratio on solids Alkyd emulsion/ Acrylic dispersion		
Amount	100	90/10	80/20	70/30
Sand Drying, hours	3	3¾	4	3½
Gloss 60°	92	63	63	65
Discolouration	3	3	3	3

*Evaluation scale ranges from 0 to 5 where 5 = best.

It seems then that for interior paints there is nothing to gain by adding small amounts of a polymeric dispersion to an alkyd emulsion paint while improved properties can be obtained when limited amounts of alkyd emulsions are added to paints based on polymeric dispersions.

Exterior formulations

Exterior paints and stains can be formulated using alkyd emulsions as sole binders and as mentioned previously the exterior durability will be somewhat better compared with the corresponding white spirit diluted alkyds. Alkyd emulsions are also very well suited as primers on wood before the application of dispersion paints. However, the major part of the exterior stains are formulated using alkyd emulsions in conjunction with dispersions and then mainly acrylics. Studies have been undertaken in order to establish the best blending ratio of the two binders.

Panels coated with clear stains with a varying ratio between alkyd and acrylic were run in QUV weatherometer. A cut was made across the lower part of the panels in order to see how the water from the dew cycle would penetrate underneath the stain. The results in Table 7 indicate that the best balance of properties is achieved using a 60/40 ratio of alkyd to acrylic. The same ratio has also shown good results in exterior exposure tests.

Table 7

Weatherometer testing of unpigmented stains (1,100 hours)

Alkyd/Acrylic on solids	Colour*	Film disintergration*	Peeling in cut*
90/10	3	2	8
80/20	4	3	7
70/30	5	5	7
60/40	5	6	7
50/50	5	6	4
40/60	5	7	3
30/70	6	7	4
20/80	7	7	4

*Evaluation from 0 to 10 where 10 = best.

Previously painted surfaces can be regarded as difficult substrates for repainting if the pretreatment of the surface is insufficient. Exposure tests have shown that the best results can be obtained repainting with a pure alkyd emulsion based formulation or by using an alkyd emulsion as primer before repainting.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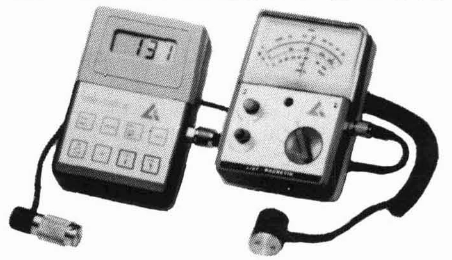

Anti-corrosive primers

A range of alkyd emulsions were tested in primer formulations. The T-56-W type of resin gave the best results after salt spray exposure. This was the resin with the lowest water vapour transmission among those tested.

In conjunction with alkyd emulsions zinc borate seems very well suited as an anticorrosive pigment. The micronized grade is to be preferred. As extender blanc fixe gave a better salt spray resistance than mica or talc. The water sensitivity of the thickening agent used is of importance. Cellulose thickeners are not suited. Best results were obtained using polyurethane thickeners. The surface active agents used in the formulation can also influence the salt spray resistance. A reduction in salt spray resistance has been observed when polymeric dispersions like acrylic and styrene acrylic have been added to the formulation. This is probably due to their higher water vapour transmission.

Primers based on alkyd emulsions have excellent exterior durability even when thin films are applied. Film thicknesses of above 40 microns will be needed if the primer should withstand salt spray testing without any top coat. So far it has not been possible to make alkyd emulsion primers with the same salt spray resistance as corresponding primers based on white spirit diluted alkyds.

Future trends

The consumption of water dilutable paints will increase in the years to come due to new legislation in most European countries. It is also of increasing interest for water based industrial finishes. Alkyd emulsion technology can be applied to these different areas if the base resin is designed for the actual application. The present technology can be improved and new technology has to be developed in order to deal with the requirements from the industrial applications.

The alkyd emulsions designed for exterior wood finishes have been tested for 7-8 years and their performance can be regarded as very good. The consumers are used to water based interior paints with fast drying and no discolouration and they expect the same performance from alkyd emulsion paints. Improved alkyd emulsions have to be developed in order to get interior paints with properties closer to those of polymeric dispersions.

There are no insurmountable difficulties connected to the manufacture of alkyd emulsions based on short oil alkyds and saturated polyesters. Emulsions have been made showing excellent storage stability and a narrow particle size distribution. However, the problems connected with the incorporation of amino resins in these finishes have not been solved. The introduction of amino resin emulsions has resulted in bad gloss and surface defects in the cured film. Using highly water soluble amino resins gives better results but tend to affect the storage stability of the ready-made product.

A general re-design of the resins used will probably be necessary in order to satisfy the requirements of the industry in the future.

Membrane separation for the production of nitrogen enriched inert gas

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Abstract

The GENERON* Air Separation System is described, which utilises membrane technology to separate compressed air into enriched nitrogen and oxygen streams. A full equipment description is given together with operating data and an outline economic analysis. A case history is presented showing how the system has been operated successfully in the paint and chemical processing industries.

Introduction

In early 1985, The Dow Chemical Company commercially launched a new method of inert gas production[1]. A system was introduced which separates compressed air into enriched nitrogen and enriched oxygen streams by use of a semi-permeable membrane. The name given to the system is GENERON*.

This system is the most recent of a series of technology developments by Dow in the membranes field. Beginning with the pioneering work in the 1950's on membranes for artificial kidneys, the company has progressively developed advanced membranes for water purification, analytical applications and gas separations.

**Trademark of The Dow Chemical Company.*

How membrane air separation works

The system utilises membranes formed into extremely fine hollow fibres. Using a unique forming technique, these fibres can be produced in quantity and with a high degree of uniformity.

A typical Generon fibre is shown in Figure 1 and is compared in size to a human hair. It will be seen that the fibre is extremely fine, has a perfectly circular cross section and has a uniform bore through its centre. The wall thickness of each fibre is therefore consistent, which is important for both the physical strength of the membrane and its separation characteristics. Because the fibres are so minute, an extremely large membrane surface area is available in a relatively small space. This results in a compact system.

Gases permeate through the membrane at different rates, depending on their solubility and diffusivity characteristics. The temperature and pressure of the incoming gas stream also affect the rate of permeation.

In the case of air separation, the two major component gases are nitrogen and oxygen. Oxygen has a higher permeability rate compared with nitrogen and passes through the membrane much more rapidly than does the nitrogen. This action affects a separation of the oxygen from the nitrogen.

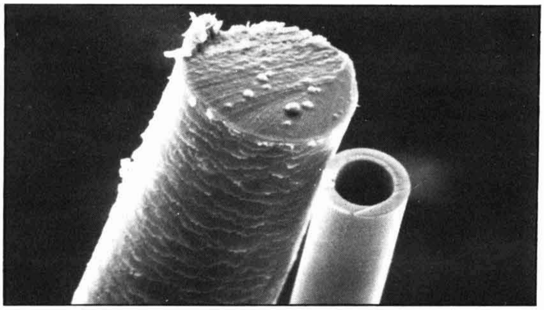


Figure 1. GENERON fibre compared to human hair.

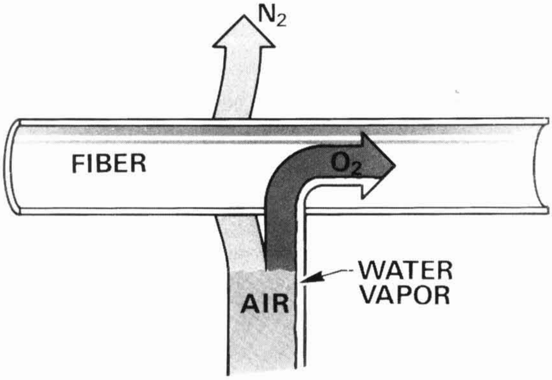


Figure 2. How gases are separated with a GENERON fibre.

All but a small percentage of the oxygen passes through the membrane and into the bore of the hollow fibre, leaving enriched nitrogen on the outside of the fibre. (See Figure 2). Trace gas constituents (e.g. carbon dioxide and water vapour) also have high permeabilities relative to nitrogen and pass through the membrane.

The Generon module

The hollow fibres are assembled parallel to a central perforated feed tube, and the resultant bundle is inserted into a pressure case to form a single air separation module. (See Figure 3). A solid epoxy tube sheet surrounds the open ends of the fibres, forming a barrier between the interior bores of the fibres and the space around them.

Compressed air is introduced via the central feed tube where it flows through the perforations and comes into intimate contact with the membrane fibres. Oxygen, water

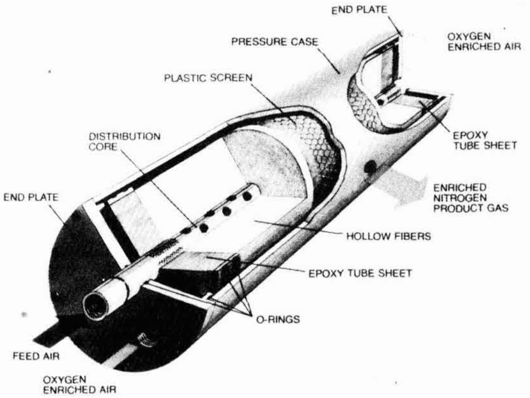


Figure 3. Construction and operation of a GENERON module.

vapour and other 'fast' gases selectively permeate the membrane walls and pass through to the bores of the fibres. The oxygen-rich gas stream flows through these membrane interior bores to the ends of the module where it is discharged.

The remaining gas passes along the outside of the fibres and is discharged at the side of the module as a nitrogen-enriched product stream. This stream is also extremely dry, typically −40 to −55°C pressure dewpoint.

The ability to separate compressed air into nitrogen and oxygen simply by-passing it over semi-permeable membranes holds great technical as well as practical significance. The hollow fibres that accomplish this air separation appear to be quite delicate, yet when they are assembled into an air separation module, they combine to form a tough, durable structure.

Module performance is illustrated in Figure 5. Nitrogen-enriched gas may be produced up to 99% purity. However, at very high purity levels, module product flow is relatively low. For most applications a purity range between 95 and 97% is considered economic. At 95% purity a single module operated at optimum conditions can produce 10.7 Nm^3/hr of gas. Larger flows than this are accommodated by arranging a number of modules in parallel or series.

The Generon System

The system is simply a bank of Generon modules with associated pipework and instrumentation. A typical industrial system is shown in Figure 4; this particular system can deliver 107 Nm^3/hr (approx. 1HCUM/hr) of 95% purity nitrogen enriched gas.

Any source of compressed air is acceptable. The equipment may use air, free of entrained liquids, from an existing plant system or from a dedicated compressor. If a separate compressor is used, an ordinary oil-flooded rotary screw compressor is perfectly adequate.

Generally, an oil-flooded screw air compressor with after-cooler will increase the feed air temperature by about 10°C because of the heat of compression. With approximately 20°C ambient temperature, the system feed air would be approximately 30°C. This is the maximum

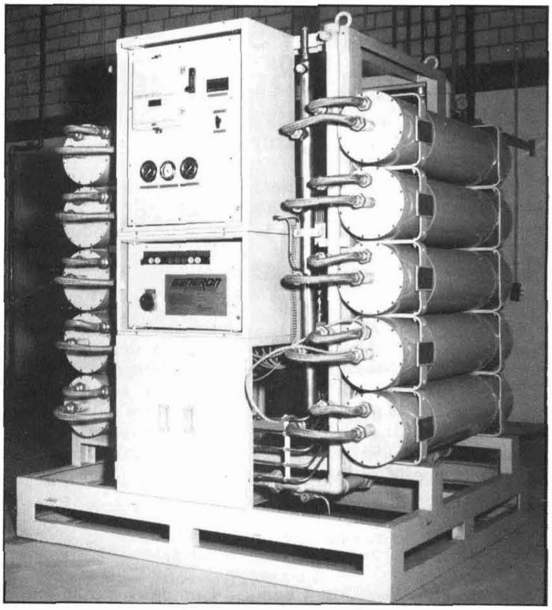
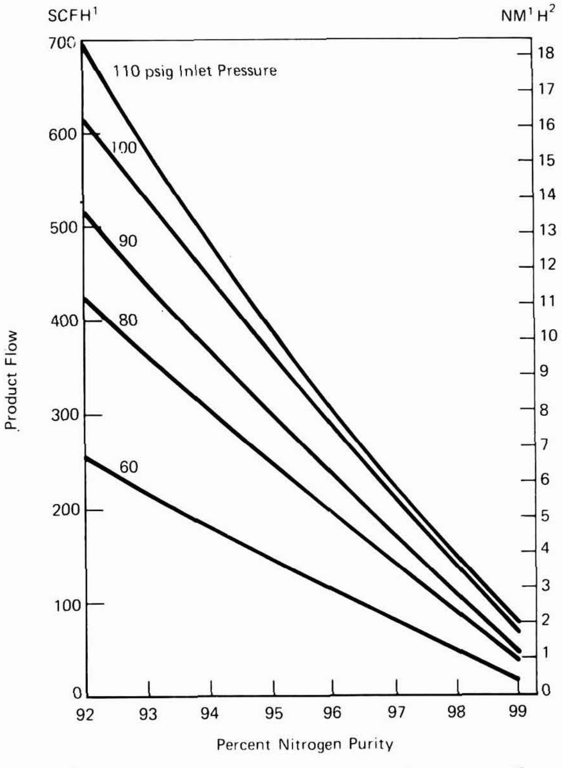


Figure 4. Typical industrial GENERON system.

Purity vs. Product Flow
(Performance of a single GENERON module at 25°C)



^{1}Conditions of SCFH are one atmosphere of pressure at 70°F.

^{2}Conditions of NM^3 are one atmosphere of pressure at 0°C.

Figure 5. GENERON module performance.

continuous temperature to which the membranes should be exposed. If the equipment is to be operated in an environment above 30°C, a chiller is usually recommended. A standard refrigeration-type compressed air dryer/chiller is ideal for this duty.

Coalescing filters are included in the system to protect the membranes from gross fouling by liquid oil or by water. Once again standard compressed air filter units are sufficient.

One major benefit of this system is its simplicity and inherent reliability. The basic process instrumentation required is limited to feed air pressure and temperature gauges, nitrogen product pressure and flow gauges and a nitrogen product oxygen analyser. More complex instrumentation schemes and tie-ins to central process control facilities can be easily accommodated if required.

System economics

The economics of this system depend on a number of factors including power cost, availability of incremental air, degree of plant utilisation and gas purity required. However, for operation in the UK, the following general conclusions can be made:

The primary running cost for the system is the electrical power required for feed air compression.

Equipment maintenance costs are extremely low since the modules themselves are effectively maintenance-free.

At 95% purity, nitrogen-enriched gas will cost approximately £2.00/HCUM to produce. If 97% purity gas is required this cost will rise to approximately £3.50/HCUM.

A system can easily show a payback of under two years when replacing liquid nitrogen installations.

Generon systems operating on already available plant air can have even better economics than complete stand-alone systems.

Typical applications

Nitrogen-enriched gas produced by this system is a suitable medium for many applications where an inert atmosphere is required. Independent data sources, e.g. the US Bureau of Mines[2], show that for many compounds maximum allowable oxygen levels are well above the 3-5% range (i.e. suitable for 95-97% nitrogen-enriched gas).

In the chemical industry, inert gas produced from this system has been used for purging and blanketing operations both at onshore locations and for shipboard installations. Similar applications have been reported for the pharmaceutical industry[3].

In the heat treatment industry, Generon gas has been used as the basis for furnace atmosphere production[4] with excellent results.

In the offshore oil industry, a large purpose-made Generon unit has recently been commissioned for purging duties on North Sea platforms.

Case history: Paint and coatings industry

The application of this new system in the paint and coatings industry was one of the technology's early successes. A report on the application of the system at Sherwin-Williams Chemical Coatings Division in Chicago, USA has recently been published[5].

Inert gas is employed at the Sherwin-Williams plant for a variety of uses including reactor sparging, blanketing of raw materials and finished products, tank purging and line blowing.

The previous system at the plant was based on the production of inert gas by combustion of natural gas (to produce a mixture of carbon dioxide and nitrogen). Following a sharp increase in natural gas pricing, other methods were considered. In addition, colour shifts in resin manufacturing were identified as being associated with production of NO_x in the inert gas.

Collaboration between Dow and Sherwin-Williams began in early 1982. Initially the system was evaluated on a laboratory scale, followed by a one year pilot plant test programme.

Key advantages of the system were identified as being:

Reliability of product gas quality

Flexibility of the plant in coping with variable flow demands

High degree of equipment reliability/low maintenance and operator requirements

Superior operating economics

The original pilot system was eventually replaced with a full-scale commercial unit. Subsequently a further three systems have been commissioned at other company locations.

Conclusions

GENERON Air Separation Systems provide an economic and technically attractive alternative to traditional inert gas and liquid nitrogen systems. The technology has an expanding applications base in a variety of different industries. Early successes in paint and coatings manufacture have been consolidated and will be expanded in the future. A pilot plant evaluation programme has been planned for the UK paint industry during 1987.

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Production of zinc and zinc-alloyed dusts by fine-atomization

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Zinc dust grades complying with the paint industry requirements are now being produced by fine-atomization. The atomized zinc dust produced is quite pure and its size distribution is easily controlled. Its morphology leads to some variation of properties, the consequence of which on ZRP's performance is theoretically discussed.

Long-term outdoor exposure for 2-5 years of test plates coated with paints from various binders, coat thickness, particle shape, size distribution, demonstrates the outstanding protection afforded by atomized zinc dust.

Zinc-alloyed pigments of the ZnAl are not substitutes to pure zinc in ZRP: aluminium addition at the level of a few per cent Al drastically hinders cathodic protection and sealing effect.

Introduction

Anticorrosion performance of zinc-rich paints (ZRP) depends upon formulations that enable the metallic pigment to develop its sacrificial effect. Amongst other parameters of importance in the paint formulation, the selected grade of zinc dust plays a role in protection of a coated steel surface; thus, for a given thickness of paint film, an adequate combination of zinc loading with regard to some physical characteristics of the zinc dust, such as its particle size distribution, will determine the paint's performance.

On the other hand, selection of a given size distribution also depends on practical considerations, such as the associated dispersibility and/or reactivity of the zinc dust, the specified coat thickness, the packing of particles in the dry film after curing. Coating thicknesses commonly used by the paint industry range from about 20 microns for blast-priming up to 80 microns for long-lasting protection with thick coats; this means that grades containing particles with diameters below these limits must be available for each application.

Traditionally, any production process based upon the formation of the zinc vapour phase, permits, by means of an adequate condensation device, the formation of primary dust suitable for the manufacture of these grades. However, direct processes involving reduction of oxidized raw materials are generally excluded, due to the risk of contaminating the final product with harmful impurities, such as arsenic or chloride.

Therefore, distillation from molten zinc was until recently the usual way of obtaining primary dust of a purity complying with paint requirements. Nevertheless, cadmium, which is more volatile than zinc, is not eliminated by distillation, and other impurities like lead can be found at various content levels in the dust, depending on the effectiveness of the separation.

The purest zinc dust can be commercially produced by distillation from SHG electrolytic zinc which contains a maximum impurity level of 50 ppm. This method which is expensive in energy consumption is now no longer necessary with the development of the fine-atomization process. This new process allows the production of very pure zinc dust of a particle size distribution comparable to the one of distilled materials. Air-classification may also be used in a similar way in order to obtain the atomized dust grades suitable for the paint industry.

For more than three years, zinc dust produced by fine-atomization has been successfully used in formulating ZRP's, with a total consumption of several thousand metric tons; again, results after 4-5 years outdoor exposure of test plates have demonstrated the performance of organic formulations based upon epoxy ester and epoxy-polyamide binders. Apart from their use as a substitute to conventional distilled dust, specific properties of atomized zinc dust could introduce some new trends in conceiving ZRP's formulations with a view to optimizing the paint properties from the standpoint of sacrificial protection, long term protection, cohesion and adhesion, etc . . .

1. Characteristics of zinc dust produced by fine-atomization

Zinc dust produced by fine-atomization is very pure, not only with respect to its impurity content but also due to its oxide content; as a result, its minimum metallic zinc content lies between 96.5 and 98.0% for the finest and for the coarsest paint grade, respectively.

1.1 Particle size distribution

The particle size distribution is determined by consideration of the size distribution of primary zinc dust, and by consideration of the cut-off point achieved by air-classification.

As shown in Table 1, available atomized dust grades may be distributed within two main groups, each of them being characterized by their cut-off point in microns. The first group is composed of three grades having their particle diameter between 0 and 10, 0 and 16, and 0 and 32 respectively. The second group contains two grades characterized by their low content in very fine particles, that is, less than 5% w/w below four microns with a maximum diameter of 32 and 64 microns, respectively.

The finest and coarsest grades in the first group are definitely devoted to blast-priming (20 microns coat thickness) and to primers (40 microns coat thickness), respectively; the medium-sized dust in this group may be used either in blast-priming or primers. Zinc dusts in the second group are more especially devoted to the protection by thick coats (±80 microns), but the finest grade in this group may also be used for primers; because of the low amount of the finest particles below four microns in these dusts, both are more easily dispersed, which could be an advantage in two-packs ethyl silicate ZRP's.

1.2 Morphology of atomized zinc dust

Reference to the spherical shape is commonly accepted

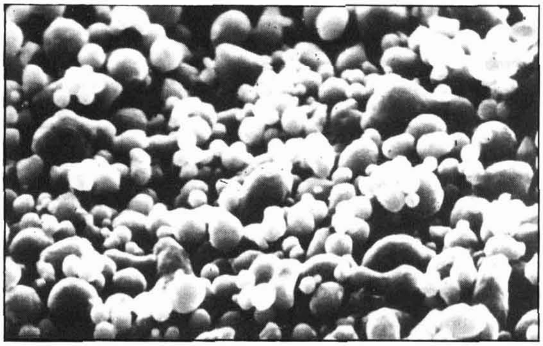

Figure 1. Morphology of particles in the medium-sized atomized dust of the first group of Table 1 (Magn. 4250x).

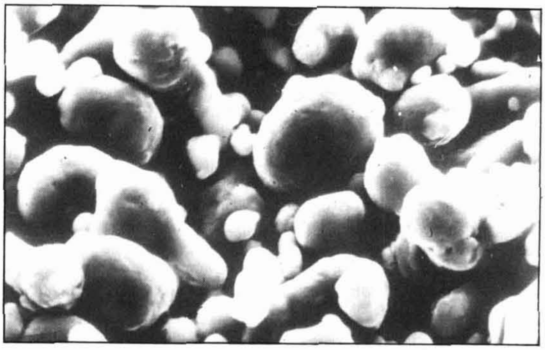

Figure 2. Morphology of particles in the finest atomized dust of the second group of Table 1 (Magn. 4250x).

Table 1

Characteristics of the available paint grades of zinc dust produced by fine-atomization

	First Group			Second Group	
Chemical Composition	1	2	3	1	2
Metallic Zn (% min.)	96.5	97.0	97.5	97.5	98.0
ZnO (% max.)	3.5	3.0	2.5	2.5	2.0
Pb (ppm max.)	50	50	50	50	50
Fe (ppm max.)	30	30	30	30	30
Cd (ppm max.)	5	5	5	5	5
Cl (ppm max.)	10	10	10	10	10
Physical Characteristics					
Particle size distribution under 5 microns (%)	60-70	45-55	25-30	5 max	5 max
5 to 10 microns (%)	30-40	35-45	25-35	25-35	15-25
10 to 25 microns (%)	0	10 max	35-40	55-65	55-65
25 to 50 microns (%)	0	0	5 max	5 max	10-20
Maximal particle size (microns)	10	16	32	32	64
Sieve residues					
125 microns (%)	0.00	0.00	0.00	0.00	0.00
45 microns (%)	0.01	0.01	0.01	0.01	1.00
Mean diameter (microns)	±3.0	±3.5	±5.0	±7.0	±9.0

when speaking about morphology of dust for paint; of course, the actual particle shape is spheroidal, that is, close to spherical. The finest particles in zinc dust produced by fine-atomization are spheroidal just like those in distilled zinc dust. However, the spheroidal shape becomes more and more elongated with increasing particle size.

As shown in Figure 1 for the medium-sized atomized dust in first group, the particles exhibit a shape quite close to the spheroid, very similar to that of distilled zinc dust, for both of the finest grades in this group; on the other hand, particles in the second group are mainly of the elongated type, but are still nodular, as shown in Figure 2 for the finest grade. The coarsest grade in first group consists of both types of particles shape; it is composed of a large number of small spheroidal particles together with a certain amount of more or less elongated particles of a larger size.

1.3 Consequences of morphology on dimensional characteristics

Physical characteristics of zinc dust play a role in the performances of ZRP's, such as permeable surface area and oil absorption, are not only dependent on the size distribution but also on the particle morphology. In the case of distilled dust, particle shape is not variable, and surface area as well as oil absorption mainly vary as a function of the size distribution; consequently, the structure of paint film and its behaviour depend on size distribution.

A variation in morphology gives rise to an increased permeable surface area and oil absorption for a given size distribution; in the case of atomized zinc dust, a decrease in apparent density is also observed in the dry state. These variations are to be considered when devising the paint formulation, and they are likely to influence the anticorrosion behaviour of the coatings.

Permeable surface area measurements are representative of the specific surface area of zinc dust; increased surface area is associated with an increase in reactivity. Consequently, departure from the spherical shape gives rise to increased reactivity of atomized zinc dust for a given size distribution. On the other hand, oil absorption measurements are an indication of the intersticial voids between the closely packed particles; its relation to CPVC, according to the well-known equation is

$$\mathrm{CPVC} = \frac{1}{1 + (\mathrm{OA})\ x\rho\,/93.5} \qquad (1)$$

where OA = oil absorption in grams per 100 grams of dust,
ρ = specific gravity of zinc = 7.14 g/cm^3.

The above equation shows that the CPVC will be lower in the case of atomized zinc dust for a given size distribution. As a result, increased binder loading is allowed in the formulation without a risk of losing porosity and electrical contact between the particles.

The magnitude of these variations was observed for several grades of atomized and distilled dusts, and its consequences upon formulation have been extensively discussed in a recent publication[1].

2. Performance of paint films

Several studies have been devoted to the behaviour of paint films prepared from atomized zinc dust. Accelerated corrosion testing as well as outdoor exposure of coated

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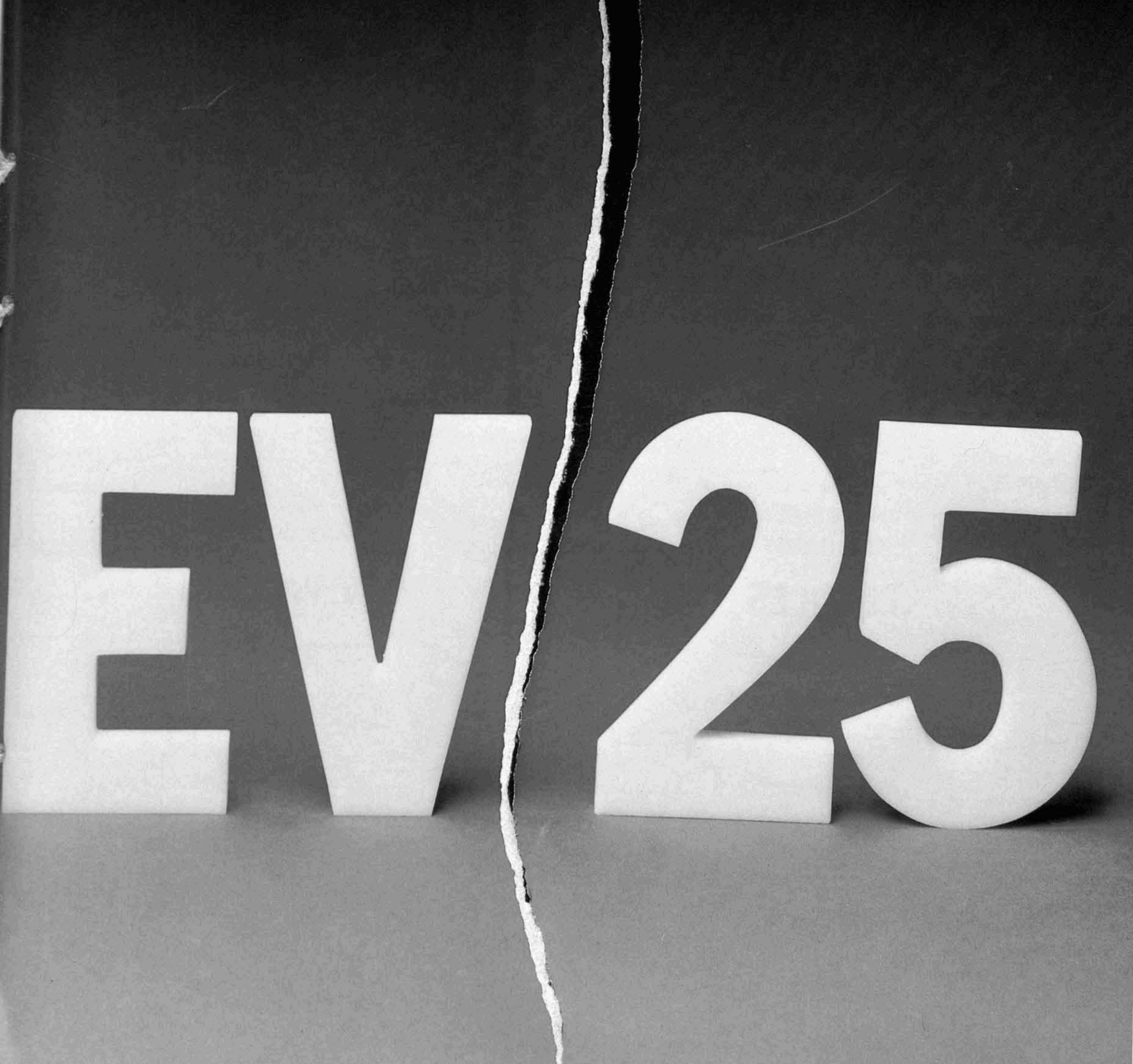

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panels produced results on performance and indicated improved properties of organic formulations.

2.1 ZRP's at nominal zinc loading

Results on test plates are now available after outdoor exposure to an urban-industrial atmosphere in Creil (50 km to the north of Paris), and to an industrial-maritime atmosphere in Calais (on the French coast by the Channel). Two organic formulations have been used with a zinc loading of about 93% w/w, which corresponds to a PVC around 66% v/v; one-pack epoxy ester and two-pack epoxy-polyamide ZRP's pigmented with several grades of dust were studied.

Epoxy ester ZRP's were prepared from the three grades in the first group, as characterized in Section 1.1; shot-blasted steel plates covered with coats of about 40 microns thickness of these paints were examined after four years exposure, at both locations. An epoxy-polyamide ZRP was only prepared from the coarsest grade in the first group; test plates coated with about 50 microns thickness of this paint has been examined after five years exposure. Table 2 gives an evaluation of their corrosion resistance based upon a notation with an arbitrary scale of corrosion (0 = sound coating; 10 = completely destroyed). From this table the anticorrosion performances are quite satisfactory in every case: destruction of the coating starts only after five years in the highly aggressive environmental conditions of the Calais coastal site.

Table 2 also gives useful information on the behaviour of various ZRP formulations prepared from the two finest grades in first group when applied at a 15-20 microns coat thickness such as used in blast-priming. Even though the protective properties of these paints fail earlier in the industrial-maritime atmosphere, they last long enough in every case for complying with the application requirements, that is, for at least one year. Very good results were obtained by using ethyl silicate ZRP's, mainly with the finest grade of zinc dust.

Finally, the outstanding behaviour of epoxy ester paints prepared from atomized dust after four years outdoor exposure is illustrated by SEM-examination of cross sections of the test plates. Figures 3 and 4 show appearance of coats consisting of the finest and coarsest grade in first group, respectively, after exposure in urban-industrial environmental conditions; in both these cases, conservation of the metallic nature of pigment is very good, and severe oxidation affects only a small percentage of the total thickness. Oxidation through the coating appears to be more important in the coastal site (see Figure 5), mainly in the pitted areas of the surface on the edges and in the lower part of test pieces (see Figure 6); nevertheless, metallic pigment is still present at the steel-coating interface even in the less favourable case, thus ensuring long lasting protection by hindering underrusting.

This outstanding behaviour of paint coats may be attributed to the efficient packing conditions of the anticorrosive pigment in the dry film, and to the resulting electrical contact between particles, as shown in the SEM-micrographs of Figures 3 and 4.

2.2. ZRP's at a reduced zinc loading

It is well-known that the effectiveness of electrical contact that gives rise to sacrificial protection by metallic zinc in ZRP's is obtained at rather high zinc loadings corresponding to a PVC of around 65% v/v; this value is close to the CPVC, which itself depends on the particle size distribution. In epoxy-polyamide formulations, the corresponding zinc loading is around 93% w/w, and it is generally recognized that the anti-corrosion performances are depressed at lower zinc loadings, as a result of less electrical contact between the particles.

From the results of oil absorption measurement made on atomized zinc dust, the calculated CPVC according to Equation 1 is lower than that calculated for distilled materials; moreover, lower apparent densities of atomized dust for a given particle size distribution indicate an even

Table 2

Quotations of anticorrosion performances of paints pigmented with atomized zinc dusts

Characteristics of Paint Coat			Parameters of Outdoor Exposure‡							
			Urban-industrial in Creil				Industrial-maritime in Calais			
			Year							
Binder*	Zinc dust†	Thickness (microns)	1	2	3	4	1	2	3	4
EP	1.3	±55	0.5	1	1	1	0.5	0.5	1	1.5
ES	1.1	±40	1	2	2.5	2.5	0.5	1.5	2	2.5
	1.2	±45	1	2	2.5	2.5	0.5	1.5	2	2.5
	1.3	±45	1	2.5	2.5	2.5	0.5	1	1	1.5
EP	1.1	±15	1	2	2.5	6.5	0.5	7	—	—
	1.2	±15	0.5	1.5	2.5	5	0.5	6	—	—
SE	1.1	±20	0	1	1.5	2	0.5	1	—	—
	1.2	±15	0	2.5	5	7	0.5	6.5	—	—
PH	1.1	±20	0.5	2.5	3.5	7.5	1.5	9	—	—
	1.2	±20	0	1	1	2	1.5	7	—	—

*Binders: EP = epoxy-polyamide; ES = epoxy ester; SE = ethyl silicate; PH = phenoxy
†Zinc dusts: grades no. 1, 2 and 3 in first group (see Table 1) ‡Corrosion scale: 0 = sound coating; 10 = completely destroyed

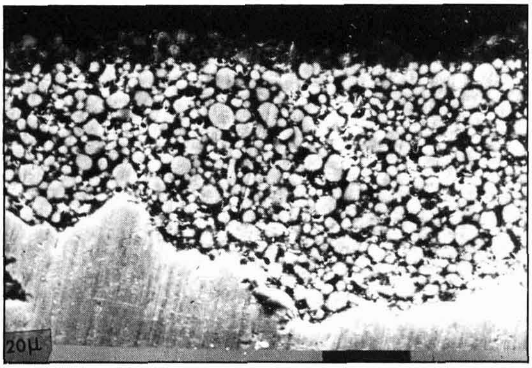

Figure 3. SEM-micrograph of a cross-section of paint pigmented with the finest atomized dust of the first group of Table 1, after four years exposure to an urban-industrial atmosphere (Magn. 950x).

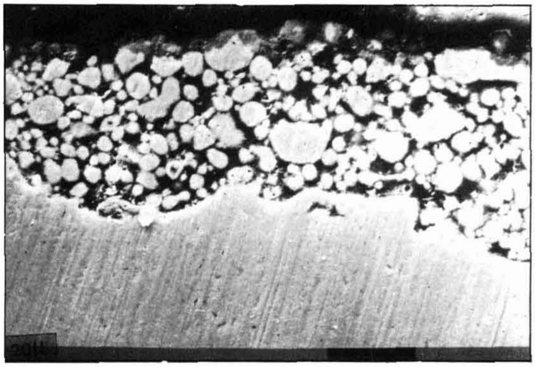

Figure 4. SEM-micrograph of a cross-section of paint pigmented with the coarsest atomized dust of the first group of Table 1, after four years exposure to an urban-industrial atmosphere (Magn. 950x).

Figure 5. SEM-micrograph of a cross-section of paint pigmented with the coarsest atomized dust of the first group of Table 1, after four years exposure to an industrial-marine atmosphere (Magn. 950x).

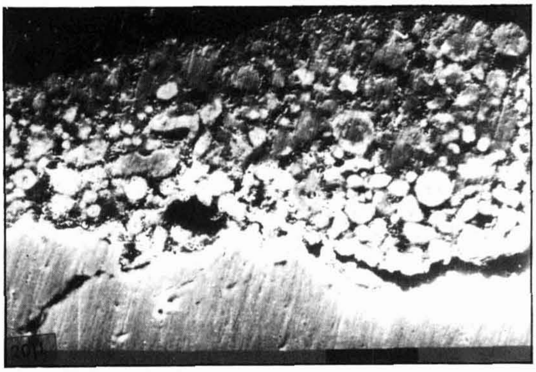

Figure 6. SEM-micrograph similar to Figure 5, but on the pitted area in the lower part of the test plate (Magn. 950x).

larger intersticial void volume between the particles in close contact in the dry state than that indicated by oil absorption measurements. As has been extensively discussed elsewhere[1], this situation leads to enhanced reactivity of paint films formulated at zinc loadings based upon considerations relevant to the distilled zinc dust. This increase in reactivity is a direct consequence of increased porosity and total surface area of the metallic pigment in the paint film.

The use of reduced zinc loadings, as calculated on a basis of oil absorption or apparent density measurements gives:

- improved pigment dispersibility and lower paint viscosity;

- decreased reactivity and optimized anticorrosion properties as determined by accelerated testing on coated plates;

- improved cohesive/adhesive properties of dry films.

Test samples of coated steel were also placed in an urban-industrial atmosphere in Angleur, and their behaviour examined for three years. Results discussed here deal with the observed behaviour of epoxy-polyamide 40 microns thick coats on blasted steel panels.

Nominal and reduced zinc loadings of 92.9% w/w (PVC = 65.8% v/v) and 89.5% w/w (PVC = 55.6% v/v) were considered, respectively; scratches through the coating were performed by turning two intersecting circles by means of a steel tool, in order to check for the cathodic protection by paints on bare steel. As shown in Figures 7 and 8, there is practically no difference in sacrificial effect between both formulations after 2½ years outdoor exposure. From this observation, it may be concluded that the decreased reactivity achieved by reducing the zinc loading has no detrimental effect on anticorrosion performances within the considered range of zinc loadings. This result confirms the conclusion previously drawn from accelerated testing[1].

Outdoor exposure for three years on unscratched coatings indicate that this is only true when atomized zinc dust is used; in the case of distilled dust at the same time reduced zinc loading, as expected, performance is affected; for example, pitting corrosion occurs on edges and in the lower part of test plates where stagnant water stays for a longer time before drying after rain or condensation. In Figure 9 the appearance of pitting corrosion in these areas is compared with the behaviour of the same paint pigmented with atomized dust.

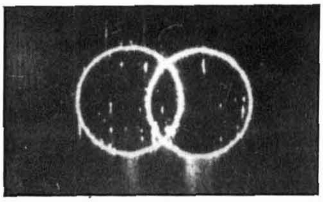

Figure 7. Cathodic protection of bare steel in scratches by a paint formulated at nominal zinc loading, after outdoor exposure for 2½ years (Magn. 0.5x).

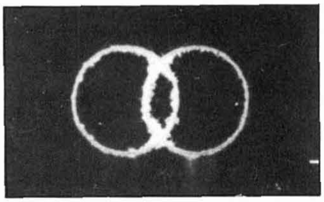

Figure 8. Cathodic protection of bare steel in scratches by a paint formulated at reduced zinc loading, after outdoor exposure for 2½ years (Magn. 0.5x).

These results support the opinion that the way of formulating ZRP's should be reconsidered by accounting for the particular physical characteristics of dusts produced by fine-atomization. Optimal performances are likely to be achieved within a 55-60% PVC range in place of the 65% usually recommended for distilled materials. As increased zinc loading results in higher porosity and higher reactivity giving rise to the formation of larger quantities of oxidation products, this new trend should together with optimized electrochemical behaviour, improve properties such as cohesion/adhesion, mud-cracking, etc . . .

3. Production of zinc-alloyed dust by fine-atomization

Direct production of dust by fine-atomization from a molten bath permits the preparation of alloyed dusts of a wide range of compositions. This possibility was evaluated at pilot scale for producing limited quantities of zinc-aluminium pigments containing quantities of Al from 0.1 to 4% by weight. Some electrochemical studies and accelerated weather testing have been completed on paint coats prepared from this new kind of pigment. Only a small amount of information of practical interest was already available in using such alloyed pigments for actual anticorrosion purposes until this new evaluation was completed.

3.1. Electrochemical properties of paints pigmented with ZnAl dust

Studies of the electrochemical properties of organic ZRP's using these zinc aluminium dusts were undertaken. The particle size distribution and the cut-off point which enable them to be used in both blast-priming (20 microns DFT) and priming (40 microns DFT) were determined (DFT = Dry Film Thickness). The investigated aluminium contents were from 0.05 to 0.15, 0.5 to 1 and 2 to 4% w/w. The following discussion of the results came from research work conducted in the CORI laboratory (Coating Research Institute, B-1342 Limelette, Belgium) sponsored by IRSIA[2] (Institute for Promotion of Scientific Research in Industry and Agriculture of Belgium).

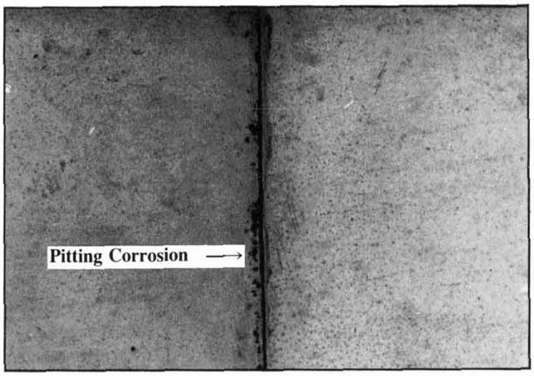

Figure 9. Comparison between paints pigmented with atomized dust on the right and distilled dust on the left, after outdoor exposure for three years (Magn. 2x).

A method derived from the Wolstenholme procedure[3] was employed in order to evaluate the aptitude for cathodic protection of bare steel by paint when immersed in 0.5M NaCl. In this method, a circular bare steel anode is surrounded by a larger surface area of coated steel acting as the cathode, both being electrically insulated; then, the current intensity of sacrificial action can be measured through an external circuit as well as the potential at closed and open circuit. The deposition of inhibiting oxidation products on to the bare steel surface may also be evaluated, as well as the appearance of metal surface rusting.

For coatings of 40 microns thickness of epoxy-polyamide ZRP pigmented with pure zinc dust, open and closed circuit potentials remained at about -1000 mv/SCE for five days, then increased to -700 mv after nine days. For aluminium contents of 0.5% and more, zinc-alloyed dusts exhibited different behaviour from that of the unalloyed zinc, i.e. the initial closed circuit potential is about -800 mv/SCE for 0.5% Al, increasing to -750 mv for higher aluminium contents. Unlike the action of the pure zinc coating where the bare steel surface is covered with inhibiting oxidation products, for the alloyed dusts the bare steel surface undergoes severe rusting after five days when the aluminium content is 2% or more. Whilst rusting is reduced for dusts containing low aluminium contents of 0.5 and 1% Al, the corresponding anodes are not totally intact as found for those protected by pure zinc. The behaviour of zinc-alloyed dusts containing 0.15% Al and less is similar to the performance of unalloyed zinc, with a somewhat decreased quantity of oxidation products deposited onto the bare steel surface.

Impedance measurements have been completed on continuous 32 microns thick coats prepared from the same paints (PVC = 65%), pigmented with zinc and zinc-alloyed dusts containing 0.15, 1 and 2% Al, respectively; these electrochemical experiments were achieved in 0.5M NaCl. The impedance diagrams obtained permit the calculation of the resistances corresponding to the high and low frequency loops, which roughly correspond to the ionic and electrochemical resistance, respectively.

After one hour immersion, the ionic resistance of paints pigmented with zinc-alloyed dusts containing 1 or 2% Al lie in the range 300-400 ohms, while its value is around 50 ohms for zinc and 0.15% Al; this corresponds to a decreased reactivity of zinc-alloyed dusts since it may be

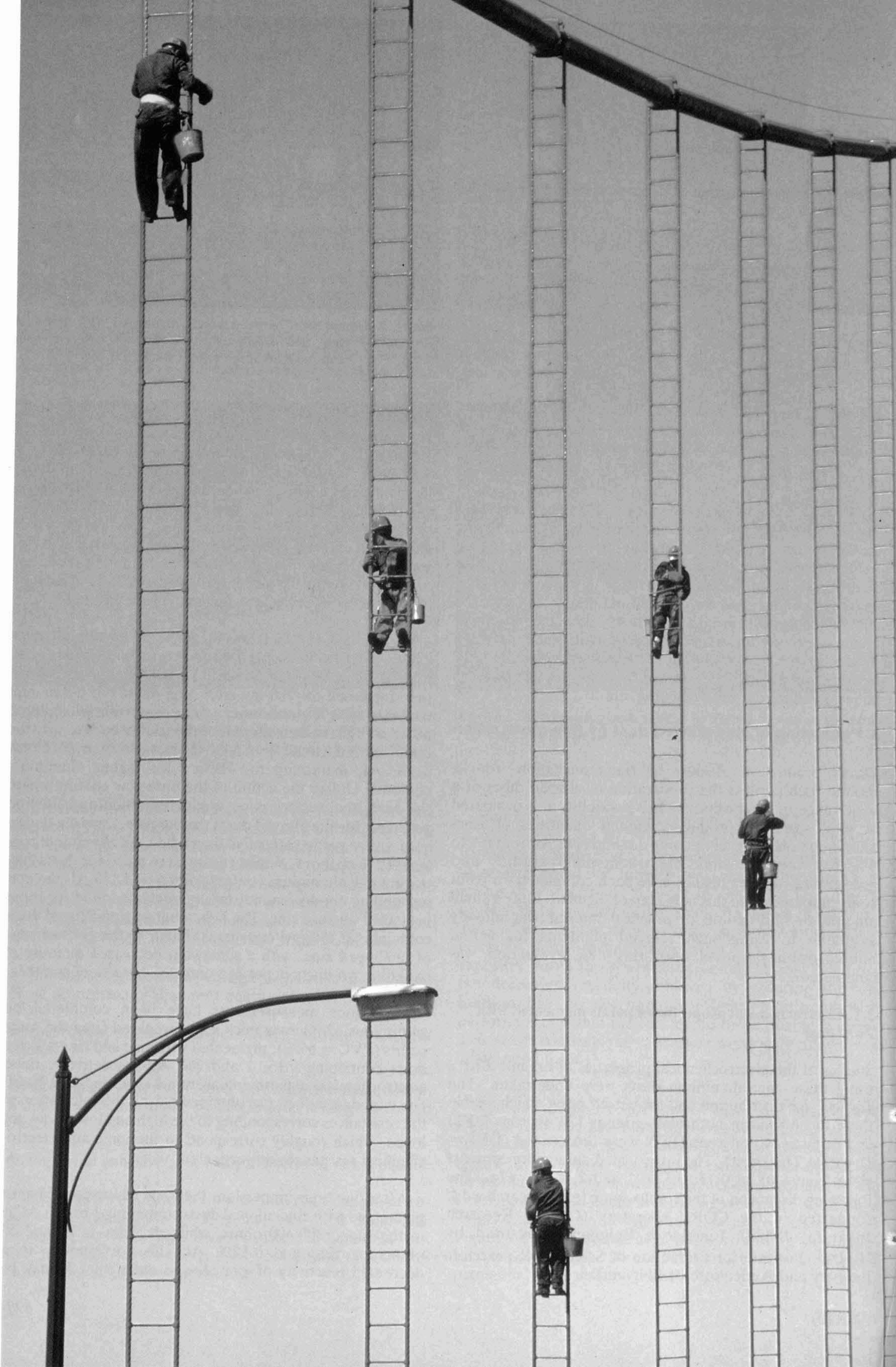

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Table 3

Total coating resistance after increased times of immersion in 0.5M NaCl

Nature of Pigment	Total Resistance in Ohms after:		
	1 hour	24 hours	48 hours
Unalloyed zinc	250	140	150
Zinc + 0.15% Al	300	140	150
Zinc + 1% Al	7000	25000	11000
Zinc + 2% Al	8000	23000	14000

assumed that the weakening of ionic resistance is caused by an increased amount of soluble fraction from the oxidation products present in the coating, which itself results from a higher sacrificial activity. On the other hand, the same difference in the order of magnitude between zinc-alloyed and pure zinc dust is observed for the electrochemical resistance in the low frequency loop; as the electrochemical resistance is inversely proportional to the corrosion rate, it is thus possible to conclude that the oxidation of zinc-alloyed dust is considerably less pronounced than the one of unalloyed zinc.

Table 3 gives the summation of the electrical and electrochemical resistance, that is, the total coating resistance for each type of pigment as a function of immersion time; between one and 24 hours, the corrosion rate of zinc and 0.15% Al increases with the measured decrease in resistance; whilst there is an increase in resistance for the other zinc-alloyed dusts indicating a decrease in corrosion rate during the same time period. These increased corrosion rates occur at the longer immersion times.

The results from the electrochemical studies lead to the conclusion that the addition of aluminium at quantities greater than 0.5% to the zinc dust drastically reduces its reactivity when immersed in NaCl solutions; this decrease in oxidation rate of the pigment could be of great interest in helping to solve problems such as adhesion loss due to the formation of oxidation products which increases the volume at the primer-top coat interface during immersion.

3.2. Corrosion testing on coated panels

Corrosion testing on paints pigmented with zinc-aluminium dusts has been carried out by both accelerated and outdoor exposure testing of the uncoated primers. This permitted an evaluation of the intrinsic performance of the paint coats prepared from the alloyed zinc dusts.

Simulation of industrial atmosphere was achieved in a SO_2—accelerated corrosion test according to a procedure already described in the literature[4]; exposure conditions were 10 ppm SO_2—RH ±94% at 35°C for 21 hours, followed by a washing out of the oxidation products by means of a water spray for one minute, and drying for three hours with interruption of the flow of SO_2 into the cabinet. The appearance of rusting can be observed under these conditions in a shorter time than needed in outdoor exposure; weight loss of test pieces may also be measured in order to differentiate the behaviour of the various pigments.

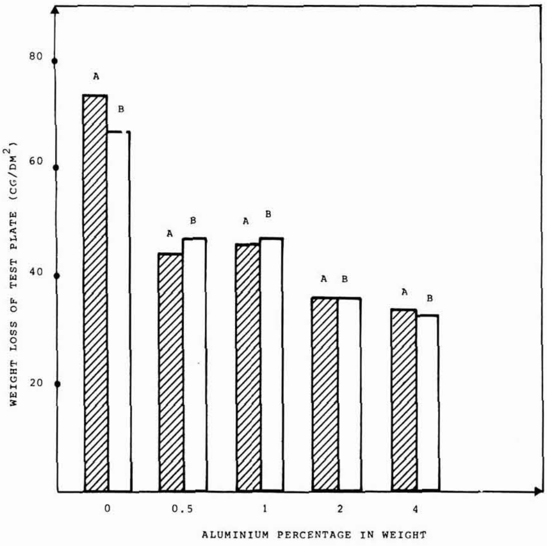

Figure 10. Weight loss of paints after SO_2-accelerated corrosion test as a function of aluminium content in the dust (A and B refer to two separate but identical test pieces).

The decrease in weight loss observed with organic ZRP's as a function of increased aluminium contents in the zinc-alloyed dust is shown in Figure 10; this decrease in reactivity confirms the results of the electrochemical studies. This is associated with higher resistance to rusting of full coatings on steels under these conditions of testing. This favourable behaviour of zinc-aluminium alloys has been verified in various polymer based formulations, such as epoxy-polyamide and isomerized rubber; which due to their sensitivity to alkalies gave problems, for example, premature gelling of the paint could arise in inorganic binder formulations, mainly from the alkaline silicate.

The above accelerated testing results were obtained for coatings of thickness of approximately 40-50 microns; results from outdoor exposure testing were also collected for both blast-priming (15-20 microns) and priming (40-50 microns) thicknesses. The outdoor exposure results are presented in Table 4 employing the same notation for its corrosion scale as in Table 2.

These results indicate the misleading behaviour of paints pigmented with zinc-alloyed dusts. This is especially true for the highest aluminium contents where pitting corrosion appears earlier than in the case of un- or low-alloyed pigments. So far as blast-priming is concerned, the decrease in reactivity has a detrimental effect upon cathodic protection ability of emerging peaks from the blasted steel surface; reduced amounts of oxidation products formed in the coating probably hinder the sealing effect that ensure the long-lasting protection in the case of zinc. Similar conclusions could be drawn from the behaviour of the thicker coats, but these results deserve to be reconsidered in view of top-coating in complex systems of paints. Decreased sacrificial protection to the benefit of reduced amounts of oxidation products formed on the primer surface should be an advantage in aggressive environmental conditions, where problems of adhesion loss could arise under the porous top-coats on highly reactive ZRP's.

Moreover, it must be noted that the corrosion data given

Table 4

Quotations of anticorrosion performances of paints pigmented with zinc-alloyed dusts (ZnAl)

Characteristics of Paint Coat†			Parameters of Outdoor Exposure‡					
			Urban-industrial in Creil			Industrial-maritime in Calais		
			Year					
Binder*	% Al	Thickness (microns)	1	2	3	1	2	3
SE	0	±20	0.5	1	2	0	1.5	8
	0.5	±20	1	2	7	1.5	7.5	9
	4	±30	7	8	9	2	5	7
EP	0	±15	2	3	6	0.5	2.5	8.5
	0.05	±15	1.5	2	6.5	0.5	7.5	9
	0.15	±20	1	1.5	—	0	2.5	9
	0.5	±15	7.5	8	—	0.5	8	9
	4	±15	7	8	—	5	9	—
EP	0	±45	1	1.5	2.5	1.5	3	3.5
	0.5	±35	1	1.5	2	0.5	2	3.5
	1	±40	1	1.5	2.5	0.5	1.5	3.5
	2	±45	1	1.5	3.5	2.5	6.5	8
	4	±40	2	3	5	2	5	6.5
IR	0	±45	0	0	0	0	0.5	1.5
	0.5	±50	1	1.5	2	0	0	1
	1	±60	1	1.5	2	0	0	1
	2	±30	1.5	3	6	0.5	1.5	4.5
	4	±45	1.5	2.5	5	4.5	8	—

*Binders: SE = ethyl silicate; EP = epoxy-polyamide;
IR = isomerized rubber

†Particle size distribution similar to the one of the medium-sized dust in first group (see Table 1 -no. 2)

‡Corrosion scale: 0 = sound coating; 10 = completely destroyed

in Table 4 indicate a satisfactory behaviour for the 40-50 microns coats of paint pigmented with zinc-alloyed dust containing not more than 1% Al, even though they show no marked advantage over the zinc-pigmented paints under the same conditions of testing. The theoretical considerations based upon electrochemical measurements afford a possible interest in searching for specific applications of much less reactive pigments, whatever the resulting paint has to be top-coated with or not over-coated.

Conclusions

Studies so far reported on the behaviour of zinc and alloyed dusts produced by fine-atomization show the following new directions in preparing ZRP's.

1. Atomized zinc dust is a valuable substitute to distilled dust the former characterized by its high purity and high metallic zinc content when produced from SHG-electrolytic zinc.

2. When used only as a substitute for other available grades, atomized zinc dust is also characterized by a slight difference in morphology, the benefit of which can be taken by lowering the zinc loading in formulations.

3. Fine atomization permits zinc-alloyed dusts of specific compositions to be produced within the limits imposed by the manufacturing process.

The use of atomized zinc dust therefore extends the applications of ZRP's as follows:

1. Safe and non-toxic use is ensured together with the maximum sacrificial and sealing effect in the coating.

2. Optimization of electrochemical and mechanical properties of paint films is possible by reducing the zinc loading without losing the essential characteristics of protection by the metallic zinc.

3. Progress in finding improved primers is enhanced through the possible use of more resistant zinc-alloyed pigments in specific applications where failures could occur with unalloyed zinc.

The points cited under (1) above are now well-established; possibilities outlined in (2) above might be easily checked for by measuring oil absorption or apparent density, and by taking it into account in evaluating commercial formulations. With reference to (3) above, the present reported investigations on zinc-alloyed dusts containing aluminium could be confirmed by preparing limited quantities of paint which would be applied onto an area of a steel structure under service conditions.

Finally further research as outlined below could be undertaken:

1. An aluminium content of around 0.25% could be checked in blast-priming applications to evaluate a possible reduction in oxidation products formed on the surface before top-coating.

2. An aluminium content of 1% could be used as a

general purpose pigment for primers in order to reduce sensitivity to oxidation without undue loss of effectiveness of cathodic protection.

3. An aluminium content as high as 4% could eventually be tested in primers for multicoat systems of paints such as those based upon two-pack epoxy-coal tar commonly used for preventing steel corrosion with sea water immersion or in the splash zone.

Pigments produced at pilot scale are available for such investigations; possible development of new coat systems based upon their use is not expected to be less effective than those of similar zinc-aluminium coatings obtained by galvanization or thermal spraying, the known sacrificial activity of which is also somewhat reduced as a function of their aluminium contents. Different alloy compositions could also be prepared on request.

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next month's issue

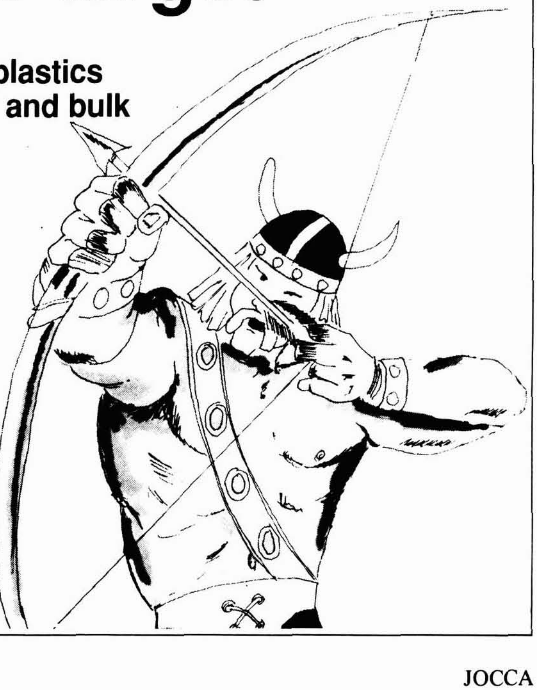
The Honorary Editor has accepted the following papers for publication in the November Issue:

New approach to the effect of binder composition on antifouling efficiency using scanning electron microscopy *By M. M. Abd El-Malek, R. M. Mohsen and M. M. H. Ayoub, Laboratory of Polymers & Pigments, National Research Centre, Dokki, Cairo, Egypt.*

Photopolymerisation and photochemistry of methyl, propoxy substituted thioxanthones *by N. S. Allen, Dept. of Chemistry, Manchester Polytechnic, UK.*

The properties of modified chlorinated rubber in the protection of steel *by D. Caldwell, ICI Chemicals and Polymers Group, Runcorn, UK.*

Colouration and colour enhancement of inks by nacreous pigments *by G. Houseman, E. Merck, Frankfurter, W. Germany.*



Novel polyamide type epoxy curing agents

R. H. E. Munn

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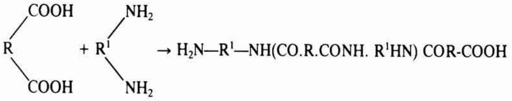
The use of polyamide resins containing free amino groups as curing agents for polyepoxy compounds is well established and such systems are widely used as the basis for high performance coatings. These systems are renowned for their toughness, chemical and corrosion resistance, and excellent adhesion to a wide range of substrates. However, when used with some types of epoxy resins, such polyamides are less than satisfactory. Drying times may be extremely long and surface defects such as residual tackiness, blooming, cissing or rivelling may be apparent especially under adverse conditions such as low temperature and/or high humidity. Another important aspect is the initial incompatibility of this type of polyamide resin with low molecular weight liquid epoxy resins leading again to surface defects which often precludes their use in high solids coatings.

New types of polyamide curing agents are described which overcome the problems associated with commercially available resins but which still retain the essential desirable features of this class of material.

Polyamide resins containing free amino groups which are suitable for use as curing agents for polyepoxide compounds are derived from the polycondensation of polymeric fatty acids and polyamines[1-3].

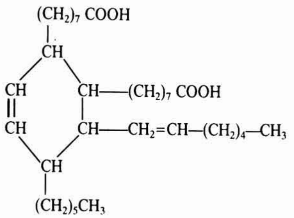
The fundamental reaction leading to the formation of a polyamide may be written as:



$$R(COOH)_2 + R^1(NH_2)_2 \rightarrow H_2N\text{—}R^1\text{—}NH(CO.R.CONH.\ R^1HN)\ COR\text{-}COOH$$

This is a typical linear polycondensation in which the molecular weight is simply related to the degree of reaction. In the polyamides under consideration, the residue R consists mainly of the substituted cyclohexene ring of dilinoleic acid, commonly known as dimer acid.

This may be represented ideally as:

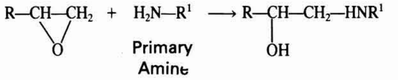


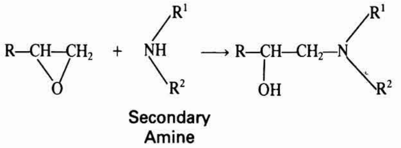
The long aliphatic side chains promote flexibility and reduce cohesive density thus giving improved solubility compared to the nylons.

The idealised linear structure indicated above is typical of "thermoplastic polyamides" although some branching is always present due to the presence of trimer acid in commercially available dimer acid. These resins have low amine values, typically 2-10 mgm KOH/g and are used as binders for liquid printing inks for both flexographic and gravure application, as hot melt adhesives and as coreactants with alkyd resins to produce thixotropic vehicles well-known for their use in dripless paints.

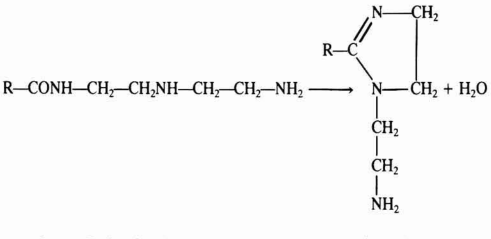
The so called "reactive compounds" which are crosslinkers for polyepoxide compounds are more complex. The amines typically used have functionalities greater than two so that highly branched structures are obtained. The residual free amine groups allow reaction with other materials natably polyepoxide resins, to yield crosslinked products.

The principal reactions occurring in the cure of epoxy materials appear to be:





Other reactions undoubtedly occur, indeed the reactions of polyamide resins are a combination of the very simple and highly complex. As an example, in the formation of a polyamide by the polycondensation of dimer acid with a polyamine, prolonged heating after the initial formation of the amide linkages will result in a ring closing reaction with the formation of imidazoline and the release of water. If heating is continued, more imidazoline if formed:



where R is the fatty acid residue $CH_3(CH_2)_n$—

The level of imidazoline in a polyamide resin has a critical effect on viscosity, reactivity and importantly, compatibility with epoxy resins. Increase in the level will reduce viscosity, decrease reactivity and improve compatibility.

The use of reactive polyamides thus described is well established in high performance two-pack epoxy coatings where their toughness, chemical and corrosion resistance and excellent adhesion to a wide range of substrates is well recognised. Another important factor in this day and age is their relatively safe nature compared to other epoxy curing agents, especially free amines.

However, under certain conditions and with certain epoxy resins, their performance is less than satisfactory. With solid epoxy resins (epoxide equivalent greater than 450), these types of polyamides are initially incompatible and compatibility improves only as crosslinking occurs. The solvents present in the mixture have a solubilising effect on both the epoxy and polyamide resins but when evaporation occurs immediately after application of the two component coatings, the resins separate resulting in blooming, cissing and rivelling of the film, and in extreme cases, the formation of greasy non-drying films. With liquid epoxy resins (epoxide equivalent ca. 200) the effect is aggravated essentially by the greater mobility of the lower viscosity coating after solvent evaporation resulting in complete separation of the two components, and negligible crosslinking. Indeed, certain dimer acid based reactive polyamides would seldom be recommended for use with liquid epoxy resins which does preclude their use in the formulation of higher solids coatings. These deleterious effects are aggravated by adverse conditions such as low temperatures and/or high humidities during application. In practice, these objections can, to a certain extent, be overcome by pre-ageing the two components prior to application. Typically, thorough mixing would be followed by "maturing" for 30 to 60 minutes before application during which reaction commences, compatibility is improved and film defects can be eliminated.

An alternative procedure is to prepare the so called polyamide adducts[4]. Here the reactive polyamide is partially reacted with epoxy resin yielding a resin which still has unreacted amino groups and which has improved compatibility and capable of further crosslinking with epoxy resin. The level of epoxy resin is dependent on the type of polyamide and the level of viscosity which can be tolerated to facilitate handling. With liquid epoxy resins of epoxide equivalent around 200, 5 to 15% is normally sufficient for polyamides having amine values within the range 210 to 400. With solid epoxy resins of epoxide equivalent greater than 450, the level would be 10 to 30%. The adducts would normally be handled at 60 to 75% solids in aromatic hydrocarbons such as xylene and toluene. Reaction of the two components in the presence of the thinning solvent can be carried out at ambient temperature when terminal viscosity is reached in about 10 days at 20°C, overnight at 60°C or one hour at 120°C.

Recent work in the author's laboratories[5] has resulted in the commercialisation of a new type of reactive polyamide which completely overcomes the deficiencies referred to above, but retains the outstanding film performance associated with two pack epoxy coatings based on conventional dimer acid based reactive polyamides. This new product yields defect free coatings even under adverse conditions, is suitable for use with liquid epoxy resins and as such can be used in the formulation of higher solids coatings. In the following discussion the chemistry and performance of this new polyamide will be described.

Chemistry

As already discussed, the reactive polyamides of the prior art are essentially the polycondensation products of dimer acid and a polyamine. It has been found that the described improvements can be obtained by replacing part of the dimer acid by certain aromatic polybasic acids and using such a mixed acids component to carry out a polycondensation reaction with polyamines.

Aromatic acids such as isophthalic acid or terephthalic acid are the favoured acids because of their relatively low cost and availability but other acids such as diphenic acid (diphenyl 2,2′— dicarboxylic acid) or diphenyl ether 4,4′ dicarboxylic acid can be used to give similar results. A 1:1 molar ratio of dimerised fatty acids: aromatic acid results in products having good compatibility, speed of dry and freedom from surface defects. Minor amounts of monocarboxylic acids can be incorporated to control molecular weight and the acid component may contain some aliphatic dicarboxylic acids such as adipic, pimelic, azeleic, sebacic or even minor amounts of anhydride such as maleinised fatty acids, phthalic anhydride, trimellitic anhydride, etc.

The polyamines used in these new crosslinking materials are of the general formulae:

$$H_2NR\ (RNH)_n\ NH_2$$

where R is an alkylene radical, especially a methylene radical and n is an integer from one to six. Examples are diethylene triamine, triethylene tetramine, tetraethylene pentamine, etc. For molecular weight control, minor amounts of mono amines may be incorporated such as fatty amines, propylamine and butylamine. The polyamine component should contain at least 50 mole percent of one or more polyamines containing at least three amino groups per molecule.

Polycondensation of the reactants is carried out using an excess of amino groups over carboxylic acid groups with the object of obtaining in the final resin amine values of 100 to 400 mgm KOH/g and acid values of 0-20 mgm KOH/g.

The reaction products are balsam like materials to hard resinous solids having melting points within the range 60 to 150°C depending on the level of aromatic acid and nature/level of amine employed.

The lower melting point products are soluble in aromatic hydrocarbons e.g. xylene and toluene, whilst the higher melting point resins are soluble in aliphatic alcohols or mixtures thereof with aromatic hydrocarbons.

Properties and performance

As a general indication of the performance of these new polyamides, Table 1 presents the effect of aromatic dicarboxylic acid (isophthalic acid) level on film performance using a liquid epoxy resin (Epikote 828—Shell Chemicals Ltd) as coreactant. No pre-ageing was used before application.

As well as giving improvements in general film appearance without resorting to pre-ageing before application these new polyamides also give improvements in speed of surface dry even at low tempeatures. This is presented in Table 2 where again a liquid epoxy resin, Epikote 828, has been used as coreactant.

Although crosslinking at 0°C is negligible, films based on the new resins are free from surface defects and achieve a surface dry condition which does afford some protection to

Table 1

Effect of aromatic dicarboxylic acid level

	Conventional polyamide*	Example 1	Example 2	Example 3	Example 4
Isophthalic acid (mole %)	0	10	50	80	100
Amine value	220	215	215	350	270
Curing agent/epoxy resin† ratio	57:43	58:42	58:42	46:54	52:48
Film appearance (no pre-age)	Incompatible greasy	Slightly greasy	Good	Good	Slightly greasy
Gloss 20%	—	89	93	90	33
Gloss 60%	—	95	97	96	75
Bend (inches pass)	—	⅛"	⅛"	¼"	¾"
Reverse impact (mm fail)	—	2.5	2.5	1.25	1.25
Slow indentation (mm fail)	—	100	95	95	90
Cross hatch adhesion	—	100	95	95	90

* Versamid 115, Cray Valley Products Ltd.
† Epikote 828, Shell Chemicals Ltd.

Table 2

Effect of dicarboxylic acid type on surface dry and film appearance

	Acid Component	Film Appearance	Surface Dry (hours	
			20°C	0°C
Conventional Polyamide*	Dimer acid	Greasy/cissing	15	24
Example of:	Dimer acid/ aliphatic diacid	Greasy/cissing	15	24
Example of:	Dimer acid/ aromatic diacid	Good	½-3	2-8

* Versamid 115, Cray Valley Products Ltd.

Table 3

Typical properties

	Aromatic Di-acid* Type	Dimer Acid† Type
Non-volatile content %	60	70
Volatile	Xylene	Xylene
Viscosity (stokes at 25°C)	45	15
Amine value (mgm KOH/g on solid resin)	215	210-220
Colour, Gardner	9-10	>11
Specific gravity at 20°C	0.95	0.94
Flash point °C	24	24

* Quickcure 195X † Versamid 115X

Table 4

Formulation of white gloss paints

Part 'A'			
Rutile titanium dioxide	28.20	29.50	31.40
Epikote 1001 (75% in xylene)*	30.50	31.84	—
Epikote 828*	—	—	17.70
n-Butanol	3.70	2.95	2.49
Xylene	10.20	13.62	7.96
Part 'B'			
Quickcure 195X†	20.60	—	36.18
Versamid 115X†	—	18.40	—
n-Butanol	1.60	0.74	1.09
Xylene	5.20	2.95	3.18

* Shell Chemicals Ltd.
† Cray Valley Products Ltd.

the substrate prior to full crosslinking which occurs when ambient temperatures rise above 5°C.

Optimisation of the formulation of these novel reactive polyamides has now given rise to a commercially available product having the properties reported in Table 3. For comparision the properties of a conventional dimer based polyamide of similar amine value are given.

White gloss paints were prepared with both polyamides using a solid epoxy resin (Epikote 1001) as coreactant. Additionally, the aromatic di-acid based polyamide was examined with a liquid epoxy resin (Epikote 828). The paints were pigmented with rutile titanium dioxide at a pigment to binder ratio of 0.8 to 1.0 by weight and thinned to four poises with a 3:1 mixture of xylene and n-butanol. The formulations are given in Table 4.

The paints were then examined for:

1. Film appearance/induction period—100 micron bar gap applied films.
2. Pot-life—viscosity increase with time, 200 gram mass at 20°C.
3. Surface dry time at 20°C—100 micron bar gap applied films.
4. Methyl ethyl ketone swab resistance—100 micron bar gap applied films.
5. Koenig hardening rate at 20°C and 7°C—100 micron bar gap applied films.
6. Mechanical properties—carried out on abraided mild steel panels—dry film thickness of 28 to 33 microns after 14 days curing at 20°C.
7. Chemical resistance of 100 micron bar gap applied films on abraided mild steel using the following reagents: a) 5% acetic acid, b) 30% sulphuric acid, c) 30% sodium hydroxide, d) water.
8. ASTM hot salt spray on single coats applied at 28 to 33 microns dry film thickness and cured for 14 days at 20°C. Substrate abraided mild steel.

Table 5

Evaluation of white gloss finishes

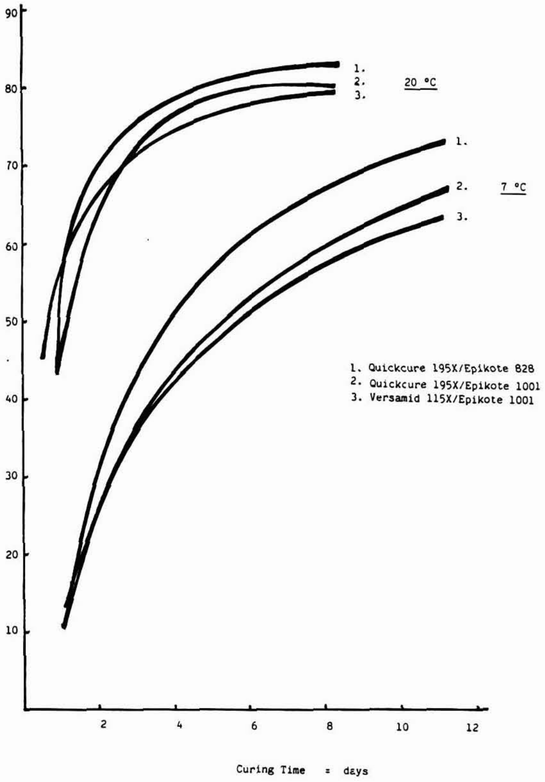
	Quickcure 195X/ Epikote 1001	Quickcure 195X/ Epikote 828	Versamid 115X/ Epikote 1001
Vehicle solids %	47.8	56.6	52.0
Pot-life at 20°C			
Initial viscosity (poise)	4.0	4.0	4.0
1 hour	4.8	4.5	4.8
2 hours	6.0	5.5	6.1
3 hours	6.8	6.4	6.6
4 hours	7.3	7.1	7.2
5 hours	8.3	8.4	8.4
6 hours	10.0	10.1	9.8
Film appearance			
Induction period 0 hours	Satisfactory	Satisfactory	Slight cissing
1 hour	Satisfactory	Satisfactory	Satisfactory
Methyl ethyl ketone swab			
1 day	50	60+	35
2 days	60+	—	50
3 days	—	—	60+
Surface dry time (minute at 20°C)	24	45	60
Bend test (inches pass)	⅛"	⅛"	⅛"
Slow indentation (mm pass)	8	8	8
Reverse impact (mm pass)	1.25	1.25	1.25
Cross hatch adhesion (% intact)	95	95	98

Table 6

Chemical resistance

	Quickcure 195X/ Epikote 1001	Quickcure 195X/ Epikote 828	Versamid 115X/ Epikote 1001
5% Acetic Acid			
1 hour	Slight loss of gloss	Blistered	Moderate loss of gloss
2 hours	Micro blistering	Lifted	Micro blistered
2 days	Dense micro blistering	—	Dense micro blistering
30% Sulphuric Acid			
1 hour	Moderate loss of gloss	Blistered	Moderate loss of gloss
24 hours	Micro blistering	Lifted	Micro blistering
30% Sodium hydroxide			
10 days	No effect	No effect	No effect
Water			
10 days	No effect	Micro blistering	No effect

Figure 1. Rate of hardening of white gloss finishes.



The results of these tests are presented in Tables 5 and 6 and Figures 1 and 2.

As can be seen from the above results, this new class of reactive polyamide retains the desirable properties of dimer based polyamides whilst at the same time giving improvements in speed of drying and hardening, and defect-free films with both solid and liquid epoxy resins without the need to pre-age the two component paints before application. This later property is further illustrated in Figure 3 which compares the appearance of black gloss finishes applied without pre-ageing based on a conventional dimer based polyamide, an adduct with epoxy resin derived therefrom and the commercially available polyamide based on this new chemistry.

Conclusions

Using the chemistry described above, a new reactive polyamide has been developed which is a useful curing agent for epoxy resin based two-pack coatings. Whilst giving film performance equivalent to conventional dimer acid based polyamides of similar amine values this new product exhibits faster drying, improved rate of hardening and vastly improved compatibility with both solid and liquid epoxy resins. This latter property enables defect free coatings to be obtained without resorting to pre-ageing of the two-pack coatings before application and furthermore, allows the formulation of higher solids coatings based on low molecular weight liquid epoxy resins.

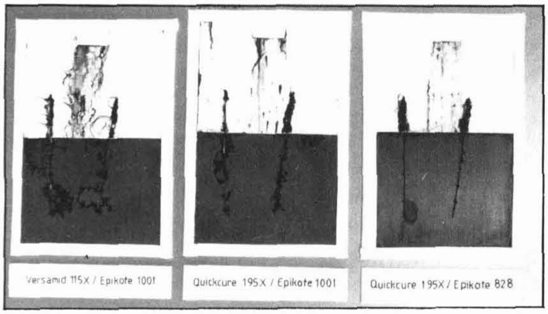


Figure 2. ASTM Salt Spray Resistance of single coat gloss finishes on untreated steel after 90 days.

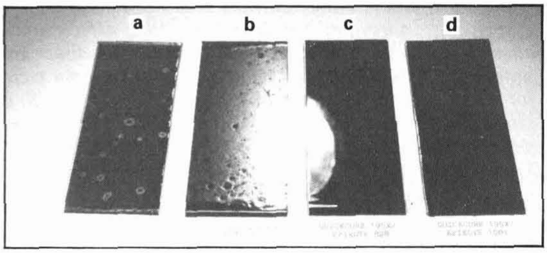


Figure 3. Typical surface appearance of epoxy coatings applied without pre-ageing: (a) Versamid 115x/Epikote 828, (b) Polyamide adduct/Epikote 828, (c) Quickcure 195x/Epikote 828, (d) Quickcure 195x/Epikote 1001.

References

1. United States Patent No. 2430940.

2. United States Patent No. 2705223.

3. British Patent No. 726570.

4. British Patent No. 988484.

5. UK application GB 2 170 813A.

conference discussions

Following the presentation of each paper at the OCCA Conference a period of time was allotted for discussion. Those who put the questions were asked to fill in discussion slips with the details of their question. Edited versions of the discussion that followed three of the papers (authors in bold) published in this issue appear below: questions in ordinary roamn type, answers in italic. Only those questions for which discussion slips were received are published.

T. Fjeldberg

F. B. REDMAN: What comparisons can be made between the minimum film forming temperatures of polymeric and alkyd emulsions?

T. FJELDBERG: In alkyd emulsions the polymer is liquid and they have minimum film forming temperatures below 0°C. Most polymeric dispersions have M.F.T.S. above 0°C.

J. R. BERNIE: Zinc borate is not commonly used in the UK as a corrosion inhibitor, why was it chosen for your tests? Did you try others?

T. FJELDBERG: We have tested seven-eight different anticorrosive pigments and Zinc borate was found to give the best salt spray results.

J. McFETRIDGE: Is the surface active agent used a critical factor in obtaining the properties discussed, e.g. emulsion particle size, flexibility (plasticiser effect as shown), film forming properties, water permeability and water pick up, drying?

T. FJELDBERG: Properties like particle size, Water pick up and drying are dependent upon the surface active agents which have to be selected with care. The type of surface active agents is not that critical for the other properties mentioned but the amount of them can have some influence.

J. R. BOURNE: The benefits of water-borne coatings are well recognised for Health & Safety reasons but as yet are not widely used in UK for exteriors due to poor drying in our climatic conditions. Alkyd emulsions compared with polymeric dispersions usually contain much larger amounts of solvents which partially defeats the original H & S benefits. How much solvent do these alkyd emulsions contain and how well do they dry in poor exterior drying conditions?

T. FJELDBERG: There are small amounts of residual solvents in the alkyd emulsions. The T-75-W for example contains approximately 1.5% white spirit and approximately 1% xylene. On the other hand it is not necessary to use solvents in the paint formulations so in most cases alkyd emulsion paints will contain less solvents than the corresponding polymeric dispersion paints. Water based paints do not dry very well at high relative humidity and low temperatures. Bad weather conditions can influence the drying speed of alkyd emulsions in a negative way.

R. MILLER: I was interested in the results of the water-soaking tests on spruce panels, but would be a little concerned that in a 24 hour test the real differences between formulations might be masked. The principal resistance to water uptake could well be provided by the spruce, rather than by the surface coating. Have you obtained any water absorption results from short times of water contact?

T. FJELDBERG: Measurements have also been made after 8 and 16 hours. These results showed less differences between the different formulations.

D. J. WOODBRIDGE: In permeability studies (Figure 4) what was the humidity gradient across the film?

T. FJELDBERG: The polymer films were fastened to cups containing water. The cups were placed in dessicators so the humidity difference was close to a 100%.

M. Leclercq

D. S. NEWTON: What are the advantages or disadvantages of increasing/decreasing the aluminium content of the atomized powder, in order to approximate to the powder used for metal spraying?

Is there sufficient oxide on the surface of air blown dust to ensure good wetting of the surface, since if not, this could give rise to poor can stability of a primer?

M. LECLERCQ: Regarding the first question, the allowed highest aluminium addition has appeared limited to about 7% by fine-atomization. Thus, it has not been possible to produce zinc-alloyed dust of composition similar to that of other metallic coatings, such as f.i. the Zn15Al alloy used in hot-spraying. But this is not a problem as it has been shown that the electrochemical activity of zinc-alloyed dust in formulation becomes drastically reduced at lower aluminium content than it is in compact metallic coatings. Therefore, the explored concentration range, which covers a wide range of pigment reactivity, seems large enough in order to evaluate the interest of using ZnAl alloys in primers.

Regarding the second question, no information is available about possible problems of can stability; moreover, atomized particles appear to adhere more strongly to the binder in dry films, as opposed to distilled dust, a part of which is dislodged during preparation of cross-sections by polishing before mircrographic examination. Therefore, in this case, wettability giving rise to better adhesion is probably due to particle surface roughness rather than to zinc oxide content.

J. BERNIE: What are the advantages of the low cadmium, lead and iron content of your powders?

M. LECLERCQ: No relation has been evidenced between the high purity and anti-corrosion performances of zinc dust; low cadmium, lead and iron content is due to their low content in the SHG-electrolytic zinc used for their fabrication. Nevertheless, the resulting low concentration of toxic elements present in welding fumes could be an advantage with respect to health and safety regulations in certain countries where they are concerned about this.

P. J. FYNE: What other low level content dopant metals in atomized zinc dust may be worth synthesizing for improved cathodic protection properties?

M. LECLERCQ: At the present time, so many additive elements could be considered alone or together that no further study has been until now achieved on this subject. Anyway, so far as zinc alloys for anti-corrosion purposes are concerned, addition of magnesium, iron or others used in particular metallic coatings could also be involved using the fine-atomization process.

R. H. E. Munn

H. MESNER: How far will the new polyamide resins be accepted for American and other specification paints where previously Versamide resins were recommended?

R. H. E. Munn: Although the new polyamide described will give performance similar in most respects yet superior in drying, hardening and freedom from film defects under adverse conditions compared against its prior-art dimer acid based resin, it will probably be necessary to re-submit paints for approval by the specifying authorities. This will be particularly relevant where binder compositions are closely defined. We are aware of this problem and have taken steps to discuss the matter with various specifying authorities particularly those in the USA.

J. R. TAYLOR: Have you had the products tested by biological laboratories for toxicity? Has any work been carried out regarding the difference between the properties when using aliphatic and aromatic diacids to give resins with increased compatability?

R. H. E. Munn: On the question of toxicity testing, we have not had such tests carried out. The toxicology of dimer acid based polyamides is reasonably well documented and the chemical structure of the new type is very similar and would not give cause to suspect any differences in toxicological activity. Obviously, the presence of free amine can present a hazard but with both resin types this is controlled to a very low and acceptable level. It has been found that only aromatic diacids give the improved compatability and speed of drying with epoxy resins. Aliphatic acids do not give such advantages and this is illustrated in Table 2 of the paper.

NEW

Paint and Surface Coatings

Theory and Practice

Edited by R. LAMBOURNE,
Technical Manager,
INDCOLLAG (Industrial Colloid
Advisory Group), Department of
Physical Chemistry, University of
Bristol.

696 pages; Published April 1987; £79.50/$152.75
(incl. of p+p, overseas orders are sent surface mail)

PAINT AND SURFACE COATINGS comprehensively reviews the polymer chemistry of all common paint binders, bringing together technical information on pigments, enabling the beginner to understand pigment selection. The authors, all acknowledged experts in their field, critically review particle size and its relevance to paints, as well as the colloid chemistry of dispersions, and the rheology of paints and their optical properties. The book offers a totally new approach to building and house paints, looking at the requirements of substrates; and introduces the theory not generally found in paint technology texts.

CONTENTS: Paint Composition and Applications—A General Introduction; Organic Film Formers; Pigments for Paints; Solvents, Thinners and Diluents; Additives for Paint; The Physical Chemistry of Dispersions; Particle Size and Size Measurement; The Industrial Paint Making Process; Coatings for Buildings; Automotive Paints; Automotive Refinish Paints; General Industrial Paints; An Introduction to Rheology; The Rheology of Paints; Mechanical Properties of Paints and Coatings; Appearance Qualities of Paint—Basic Concepts; Specification and Control of Appearance; Durability Testing.

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South African MIO

Dear Sir,

May I be permitted to offer a few comments on Mr E. R. Timm's paper "An evaluation of South African micaceous iron oxide pigment" which appeared in the May 1987 issue 5. I am concerned about Mr Timm's proposal that the quality of MIO pigment can be determined by the extent of lustre development when paints containing it are exposed to artificial weathering.

It has been amply demonstrated by several eminent workers including Bishop & Zobel of British Rail Technical Centre[1,2] and Wiktorek of Australian Iron & Steel Corrosion Research Laboratories[3], that MIO pigment with a flake-like particle shape provides better corrosion resistance than non-lamellar types. This is because the thin platelets can form tightly packed layers within the paint film to impede penetration of corrosion stimulants[4,5].

Recent work at MPLC Laboratories Ltd involving evaluation of synthetic MIO[6] has confirmed that particle shape, flake thickness and size range have a significant effect on protective properties and durability.

I cannot accept Mr Timm's unsupported statement that development of lustre (when MIO paints have been exposed to UV) indicates the lamellar property of the MIO pigmentation and can be related to general performance properties. Granular forms of MIO can develop lustre very rapidly when exposed to weathering but they have demonstrably inferior protective properties. On the other hand, lamellar MIO when incorporated at the correct level in an alkyd binder can be quite slow to develop lustre, due to the low chalking rate, while providing excellent durability.

In the SABS test it was not surprising that "the salt-fog corrosion rating showed no obvious major advantage for either source grades of MIO pigment". (Austrian and South African). The pigment inclusions at 42-52% PVC were well above the optimum level for effective salt-spray resistance in an alkyd binder. Such high PVC levels would also reduce the protection afforded by the lamellar MIO exposed to exterior weathering. I expect there would have been a marked difference in protective properties if both pigment types had been compared at around 35% PVC at the normal DFT for alkyd MIO paint viz 50-70 μm.

Mr Timm makes some rather acid comments about submissions to ISO relating to test methods to identify a suitable particle shape for MIO. The latest draft of the revision of ISO 1248 (to which he objects) includes essentially the same procedure as set out in Australian standard 2855-1986. This consists of a microscopic examination to determine whether the pigment particles consist mainly of thin flakes or not. Viewing under an optical microscope by transmitted light has been the normal method for at least 40 years to my knowledge, to check on MIO pigment. So why do SABS think it is impracticable? The SEM examination is a more recent development but has proved to be very effective in many laboratories in Britain, Europe and Australia. I personally think that either the optical microscope or the SEM methods can be equally effective so there should be no need for the ISO standard to require that both are carried out. There should be no objection to using photographs as comparators. The photographic standards in the document are quite clear and certainly much easier to interpret than photographic standards for surface preparation of steelwork which seem to be universally accepted.

Finally, we surely have to view the whole matter in a realistic context. One must remember that at most only about 5% of the world's annual consumption of MIO pigment is used by the South African paint industry and this mainly is to produce a sparkle in roof-paints. It can be conceded that lustre-development has some importance here and undoubtedly South African MIO should work effectively in such coatings. However the other 95% of MIO supply is mainly incorporated in high performance coatings in a wide range of binders including alkyd, chlorinated rubber, vinyl, epoxy and moisture curing urethane and for these, pigment quality is of paramount importance and the ISO standard is right to insist on a thin flake particle shape.

MPLC Laboratories Ltd,
Mill Hill,
North West Industrial Estate,
Peterlee,
Co Durham SR8 2HR.

Yours faithfully
E. Carte

18 August 198

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2. Bishop, D. M. and Zobel, F. R. G., *JOCCA*, 1983, **66**(3), 67
3. Wiktorek, S. and John, J., *JOCCA*, 1983, **66**(6), 164.
4. Claxton, A. E., *Official Digest*, 1964, **36**(470), 268.
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Durability Testing

Dear Sir,

The recent conference paper by Cutrone and Moulton (JOCCA 1987(8), 219) is one of the most valuable published on the subject of durability testing for many years. After heavy criticism of carbon arcs by the protagonists of xenon and fluorescent mercury lamps over the last 20 years, it is reassuring but a little surprising to find that the source used so widely since the 1920s is still giving the best correlation with natural weathering, at least for rutile pigmented paints. Perhaps the clue is discovered when the rutile spectral absorption curve (Fig. 1 in the paper) is combined with the sunlight energy distribution (Fig. 2). A rough calculation indicates that two thirds of the solar ultraviolet energy absorbed by rutile is in the range 355-415 nm just where the major cyanogen band emission occurs from the carbon arc. The good correlation obtained with the fluorescent lamps peaking at 370 and 410 nm reinforces this explanation.

Two other observations relate to experience at the Paint R.A. and in collaborative testing with other laboratories. First the relation of mass loss to film thickness. Working with freely chalking paints (TiO_2 or 'chalking' zinc oxide in

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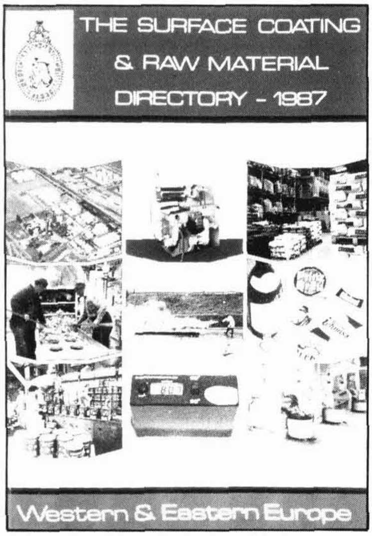

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long oil alkyds) I found that, after the first 100 hours of exposure to carbon arc weathering, the rate of mass loss per unit exposed area became independent of film thickness over the range 20-50 μm. Loss rates were lower for thinner, 10-15 μm, films. With unpigmented alkyd films or films containing UV transparent pigments the rate of loss, although much lower, did increase with film thickness. Incidentally, determinations of volume loss (obtained by weighing panels suspended in water) indicated that only the binder was lost from the film, the apparent density of lost material being close to unity. This was confirmed by the analysis of films that had chalked right through to the substrate and were found to contain at least 90% of the original TiO_2 pigment. Titanium dioxide pigment does not appear to be removed from a film of a well made gloss paint during artificial weathering; it may be scoured from the surface by the greater mechanical stresses during natural exposure to hail, rainstorms and so on. More pigment may also be lost from the clusters in a badly flocculated paint.

With paints more resistant to photodegradation there are complicating factors to consider before isolating the effect of radiation. These include residual solvent, which may amount to over 5% of the initial dried film weight for a thick film and which is gradually released during weathering. There may also be volatile or water-extractable products of curing reactions or subsequent thermal degradation processes. The overall effect of these factors is a loss in mass which is initially quite fast on exposure but decreases with time, rapidly for thin films but more slowly for thick films. Consequently, a higher measured rate of mass loss during exposure for a thick film does not necessarily indicate that photo-degradation has occurred deep in the film. The issue could be resolved, perhaps, by a blank test run with an equivalent heat source replacing the UV lamp in the artificial weathering apparatus.

The second observation relates to silica and deionised water. Paint R.A. and some other laboratories joining in round-robin tests met this problem 20 years ago. Experience then was responsible for the limitation on silica content of water in BS 3900 F3. The difficulty is, as the authors imply, that silicic acid is only weakly held by the anion exhange resin and is easily displaced when the resin approaches saturation by chloride, sulphate or nitrate ions present in most feed waters. Indeed, we found that the last 10% or so of 'deionised' water drawn off before conductivity had risen sharply could contain several times the silica content of the feed water; in effect there was a chromatographic separation. We also noted that films sprayed with this silica-rich water showed enhanced water spread and dried much more rapidly on passing out of the water spray; the apparent improvement in durability was probably due as much to reduction of the water factor as to optical or mechanical screening.

25 Spring Gardens
Dorking,
Surrey

Yours faithfully,
T. R. Bullett
25 August 1987

reviews

Paint and Surface Coatings: Theory and Practice

Editor R Lambourne
Published by Ellis Horwood Ltd
pp 696, £79.50 hardback, ISBN-0470-20809-0

This book should be considered to be a useful addition to any technical library, since it has been written by contributors who clearly show that they have practical knowledge of the science and technology of Surface Coatings. This fact is not surprising since 10 out of the 13 contributors to the book are either active or retired members of ICI Paints Ltd. The Editor is R. Lambourne, Technical Manager of the Industrial Colloid Advisory Group, University of Bristol. The other contributors are also well known experts in their own fields of paint technology and Messrs F. K. Farkas and T. R. Bullett.

The book comprises 19 chapters (696 pages) which have been written to cover most aspects of surface coating technology. The information given is of immediate interest to a paint technologist and excellent references are found at the end of the chapters. The index is easy to use and covers most of the subjects which a reader might require to look up for reference. The format employed in writing each chapter makes for easy reference since the Table of Contents at the commencement of the book gives full details of the descriptive headings of each sub-section contained in the 19 chapters. On a very minor point the reviewer wonders why three organic compounds trimellitic anhydride, hexamethoxy methyl melanine and dimethylol propionic acid are singled out for inclusion in the index, since the chapter on Organic Film Formers, which discusses the compounds, obviously deals with many other organic compounds. Perhaps this is an oversight and these compounds should not be included.

The mass of detailed information given in the book makes it necessary, in fairness to the contributors, to review briefly each chapter in turn.

Chapter 1. "Paint Composition and its Applications" is written in a clear and general way and would be of interest to those who are not necessarily paint technologists and also contains a short history of paint from Cave Paintings to the present day.

Chapter 2. "The Nature of Film Formers" (69 pages). The introduction deals with the reasons for using various types of polymers in paint formulations. The chemical constitution of oils, fatty acids and modified oils are dealt with in a short but adequate manner. Resins of interest in surface coatings such as alkyds, including alkyd reactions and structure and formulation, polyester, amino, epoxy, isocyanates, acrylic, silicone, vinyl, phenol formaldehyde resins and emulsion types are discussed in detail. Special mention is made of resins for electrodeposition, high solids types, radiation curing resins, powder coating resins and water soluble resins. An excellent list of references is given.

Chapter 3. "Pigments for Paints" (82 pages). This chapter includes information on both the chemical structure and types of organic pigment dyestuffs. Full discussion of extenders, inorganic pigments and anticorrosive pigments is included. There is a simplified guide to pigment selection for paint formulation. The various types of pigments are discussed under colour type and Colour Index References are included. A very useful bibliography is appended for further reading as well as an excellent list of references.

Chapter 4. "Solvents, Thinners and Diluents" (17 pages). The physical chemistry of solvents is given in some detail including such items as solvent power and solvency, solubility parameters, three dimensional solubility parameters, evaporation and flash points. Methods of determining flash points are given including the Abel and Penski-Martin apparatus but no mention is made of the Seta Flash Point apparatus. Toxicity and Environmental Pollution are considered briefly and it is regretted that this subject is not given greater space in the chapter as it is now a matter of great importance to the paint industry. There are excellent references provided.

Chapter 5. "Additives for Paint" (13 pages). This is a short but instructive chapter which gives in detail, for example, methods of overcoming problems with antifoams and antisettling agents. In-can corrosion and preservatives to prevent purifaction, dispersion aids, driers, materials with antistatic and electrical properties, UV absorbers and many other additives are discussed. There is a useful list of commercial names and addresses of manufacturers whose products are considered to be of value in the control of these problems. Also included is an extensive bibliography.

Chapter 6. "The Physical Chemistry of Dispersion" (44 pages). The importance of dispersion in paints is discussed in detail from the concept of physical chemistry, and has been written in such a manner as to be easily comprehensible. The range of subjects covered includes immersion and wetting of pigments, deagglomeration and dispersion, (colloid stabilization) and forces between macroscopic bodies, adsorption and rate of flocculation. A very extensive list of references is given at the end of the chapter.

Chapter 7. "Particle size and size measurement" (37 pages). This subject is dealt with in great detail from both the theoretical and pratical stand-points. Methods of the determination of particle size by the use of direct measurement, sedimentation, chromatographic, aperture, optical (light scattering) and surface area methods are clearly demonstrated. Very adequate references are provided.

Chapter 8. "The industrial paint making process" (45 pages). Considers this process from the stand-point of using instrumental methods in the formulation procedure with special reference to the Brabender Plastograph (Torque Rheometer). This method it is stated has been used in the laboratories of ICI Paints Ltd, to formulate optimum mill bases for various dispersion techniques. Practical examples of how to use test data for formulating millbases are given. Machinery used in dispersion together with control techniques for quality assessment in the surface coating industry are discussed. References are included.

Chapter 9. "Coatings for Buildings" (71 pages). This is a most comprehensive and useful survey of the type of surface coating suitable for application to the whole range of substrates found in buildings i.e. wood, masonary, cement, metals and plastic. Consideration of formulation constraints includes pigment, binder and solvent relationships. Important physical characteristics such as porosity and permeability, especially in connection with the painting of concrete surfaces, and the problems associated with tinting methods are discussed. The whole subject of the requirements for coatings suitable for the whole range of situations in the building industry is most expertly surveyed and together with an excellent list of references will be of great value to members of the surface coating industry and it is extremely difficult to do justice to the chapter in a few sentences.

Chapter 10. "Automotive Paints" (58 pages). Gives useful data on the technology, formulation and application of paints suitable for application to motor car bodies on the production line. Information is given on metal protection including electrodeposition, dip and spraying techniques of primers. Paint systems such as Non Aqueous Dispersions and paints containing thermosetting and thermoplastic acrylic resins are discussed in details. Problems associated with the need to paint plastic parts of a motor car are highlighted. Base coat/Clear systems for metallic colours on the motor car body are examined and future developments in the coating of motor car bodies described. This chapter is of interest since few paint technology books have dealt with this subject in such a practical and clear manner. The chapter contains references and a bibliography.

Chapter 11. "Automotive Refinish Paints" (10 pages). This is a short chapter which covers the important subjects of the technology of this type of paint. The physical requirements of the base and top coats are examined. The various types of resins suitable for use in refinishing paints including nitrocellulose, thermosetting acrylic and thermoplastic acrylic resins, alkyd resins and acrylic-urethane resins, are discussed. The development of BaseCoat/Clear systems usually formulated for metallic systems are described and so are details given of the type of pigment used in formulations. Possible future developments are discussed.

Chapter 12. "General Industrial Paints" (27 pages). This chapter gives a concise description of the market distribution of these paints in the UK, Europe, USA and Japan as an introduction into the technical requirements of coatings. The industrial application and curing methods are reviewed and these include for example flow coating, roller coating, vacuum impregnation, and curtain coating. Curing methods such as radio-frequency, infra-red, UV and electron beam are considered. Details of systems suitable for coating such items as the interior of two piece beer cans, exterior cladding for builings and refrigerators for example are discussed. The development and trends in general industrial finishing is considered. A brief list of references is appended.

Chapter 13. "The Painting of Ships" (18 pages). In the introduction the author considers corrosion mechanisms and surface preparation of ferrous metal before giving typical formulations for blast primers etc. This is followed by data on typical topside, and superstructure paints. Information on the use and formulation of antifouling compositions is given, including data on the self polishing types. This information gives a useful insight into marine paint technology. References are as usual appended.

Chapter 14. "An Introduction to Rheology" (26 pages). This chapter written by T. A. Strivens, is of immediate interest to those who require a most useful and easily understood review of the subject. The physical concepts and mathematics of rheology is discussed and definitions are clearly defined. Types of rheological behaviour such as pseudoplastic, dilatant and thixopropic materials are discussed. Measurements of these effects is outlined together with the interpretation of the results. Excellent references are appended.

Chapter 15. "The Rheology of Paints" (23 pages). The same author considers this subject in further detail. Topics include paint application, film formation and flow out, and the desirable rheology for paint application. The whole range of experimental methods for the measurement of rheology for flow out are examined including the flow cup and more accurate methods such as the ICI (relaxation) low shear method, high-frequency (impedance Methods) and rolling ball methods. Excellent references are appended.

Chapter 16. "Mechanical properties of paints and coatings" (23 pages). The same author continues with a discussion of viscoelastic properties of polymers and their ultimate mechanical properties which precedes the methods for their determination and includes the free oscillating torsion pendulum method, forced non-resonance vibration method and ultrasonic impedance method. Technological tests are also described such as hardness tests, scratch hardness and flexibility tests. Excellent references are given.

Chapter 17. "Appearance qualities of paint Basic Concepts" (21 pages). This chapter written by Tom Bullett deals from a theoretical standpoint with the physics of reflection by paint/air interfaces, including scattering of light by white pigments and absorption of light by pigments. A short section deals with fluorescence and phosphorescence. Colour appreciation and the effects of illumination are mentioned in separate sections. Excellent references are provided and recommendations for further reading included.

Chapter 18. "The specification and control of appearance" (39 pages). Written again by Tom Bullett this chapter covers the important physical characteristics of paint films including gloss, opacity, specifications and control of colour. The determination of contrast ratio is discussed from a theoretical standpoint. Visual and instrumental colour control methods are discussed in a manner which is easily assimilated. There are the usual references provided.

Chapter 19. "Durability Testing" (29 pages). This chapter is considered to be a short review of the subject outlining such items as panel preparation, chemical resistance testing to BS3900 and the ASTM methods. Accelerated weathering is described with reference to the Marr Carbon Arc Weathering Apparatus, the Atlas WeathOmeter and the Zenon Arc apparatus, The QUV apparatus is also included in the discussion. Physical tests performed before and after weathering are included. There are references to natural weathering including Florida as a means of accelerating natural weathering.

In conclusion the book contains an immense amount of useful information which has been written by professional practicing paint technologists and physicists for their equivalents in the Surface Coating Industry and therefore should have an immediate appeal to the latter and be a useful addition to technical libraries.

J. R. Taylor

Standing in the Wings

by David Roe
Settle Press (Wigmore House Publishing Ltd), 1987
pp 158, price £7.95 (hardback), ISBN 0907070 44 2

It is a rare privilege to be allowed to review a book by a friend of long standing, particularly one which covers, in such a charming, sympathetic and eminently readable way, so many different aspects of an astonishing career.

I first met David Roe when I became the chief executive officer of this Association in 1951 and learnt that he had been one of the pioneers of the first OCCA Exhibition (held in conjunction with the London Section's A.G.M. in 1949). How apposite it is, therefore, when the London Section is this year celebrating its 50th anniversary, that the memoirs of this distinguished former Chairman of the Section should be published. Many members of the Association will remember David Roe's radio broadcasts in the BBC's "Today" and other programmes but this was just one element in a lifetime's work which started as a paint chemist and encompassed amongst other items the formation of a paint company, property development, the organisation of a television studio, a film of the Philby spy case and a stage musical production. Above all, what the fascinating book (sub titled simply "A 20th Century Chronicle") shows is that when opportunities arise and challenges are accepted with enthusiasm and energy the results in terms of personal satisfaction and sense of achievement are immense. Many OCCA members reading this book will find references to incidents to which they can relate. In one chapter, the author mentions that his engineers had accidentally erased a BBC video tape and he had phoned to explain this to the television facilities manager at BBC Television Centre who spent the last years of her service with the BBC on educational programmes, including one called "On the Move". One day I was called to the entrance of Priory House to witness all sorts of equipment being manoeuvred on our car park to take a scene for "On the Move" involving the sub post office then situated next to Priory House. When the programme was transmitted there was Priory House with 4the actors passing to and from the sub post office. Similarly, many readers will have met some of the interesting people encountered by the author in his different capacities – people such as A. J. Gibson (President of OCCA and father of Dam Buster Guy Gibson VC), Denis Thatcher (with whom he worked in the paint industry), Margaret Thatcher (who was later guest of honour as Secretary of State for Education & Science at this Association's Luncheon at the Savoy Hotel preceding its Exhibition at Olympia in 1971) – but the list of other well known personalities with whom he came into contact, sometimes in the most unusual circumstances, make fascinating reading as the story of his life unfolds.

His earliest memories are of his father's work amongst the poor which, although covering a period which closed just over half a century ago, will seem almost unbelievable to those brought up in the Welfare State.

The author describes how, after an accident in the laboratory at Dulwich College, which damaged his right eye, he decided to enter industry and started as a paint chemist with Atlas Preservative and went on to form a small paint company with his associates. A chance introduction from Neil Fisk of "Paint Technology" to the producer of the BBC's Woman's Hour (who needed someone to talk on emulsion paints which were then in their infancy) led to his services becoming increasingly sought by other radio programmes.

After a spell in property development he and his associates found the ideal opportunity to use his previous experience in broadcasting in the new expanding world of television, where they formed a special facilities studio. His description of the characters who came to the studio and the stories surrounding the events which were covered make fascinating reading.

The book is well illustrated and the story flows smoothly from incident to incident with the editorial technique expected of such an experienced broadcaster.

R. H. Hamblin

news

HPG Industrial Coatings Ltd formed by Hoechst

Berger Britain will be concentrating its marketing efforts on its decorative, wood preservative, aerospace and resin business with Berger Industrial Coatings being transferred to a new management company, HPG Industrial Coatings Ltd. Hoechst UK Ltd has a 75% holding in the new company, with Herberts, Hoechst's industrial paints arm, holding the remaining 25%. The company, trading as Herberts and employing 600 staff will continue to operate from Chadwell Heath.

This is the outcome of a pan-European policy to divide Hoechst's paint interests strategically into decorative and industrial groups. As a result Herberts will be responsible throughout Europe for vehicle refinishing, automotive and industrial sectors, while Berger will continue its decorative coatings, wood preservatives and other non-paint activities.

New European Mearl Office

Mearl International BV, Haarlem, Holland, has opened a new subsidiary office in France at 5, Square Charles Dickens, 75016 Paris.

Thor Chemicals acquisition

Thor Chemicals (SA) Pty Ltd has acquired Chelac Chemicals (pty) Ltd. The Chelac company will continue producing metallic soaps (paint and ink driers) and speciality chemical products in Transvaal.

Sterling ownership change

The Evode Group PLC has purchased the assets of Sterling Technology Ltd of Manchester from Reichhold Chemicals Inc, USA. The company will continue to manufacture and supply surface coatings for the electrical/manufacturing industries from its plant in Trafford.

Borden Japanese joint venture

Borden Inc has signed a joint venture agreement with GunEi Chemical Industry Co Ltd of Takasaki, Gunma, Japan, for the manufacture and marketing of Borden's patented phenolic foundry resins in Japan. Under the agreement, GunEi Chemical will be licensed to manufacture Borden's AlpHaset and Betaset brands of ester-cured phenolic resins.

Rhone-Poulenc (UK) moves

Rhone-Poulenc (UK) Ltd, the UK trading operation of the French Chemical Group has moved to 271 High Street, Uxbridge, Middlesex. Tel: 0895 74080.

Vapocure UK agreement

Vapocure has signed a non-exclusive equipment licence agreement for the UK with AIR Industrial Development Ltd, a Birmingham-based private company. The company designs and installs paint systems to both the industrial and automotive industries. Air has manufactured and installed the Vapocure conveyorised paint facility at Vapocure UK Ltd in Brentford, London, and will promote the Vapocure technology in the UK.

Largest UV roller coater

Probably the largest UV roller coater ever made, a four metre wide Linecure, had been manufactured by Wallace Knight Ltd of Slough for a company in Ireland. The unit, developed in conjunction with and custom built for Tarkett Ltd, of Mullingar, coats and cures cushion vinyl and other floorings. The process of UV cured floorings with polyurethane varnishes is well established but until now it has only been possible up to a width of two metres. The UV curing system has been designed so that it has the ability to vary the gloss levels from matt to mirror finish. For a lower gloss this involves special precuring. Final curing requires high powered lamps able to ensure a completely dry finish prior to rereeling.

products

Fibrous thixotropic additives

Brenntag (UK) Ltd of Kingston upon Thames, Surrey, has available *Armipent,* a fibrous thixotropic reinforcing additive for both water and solvent based surface coatings. During application the fibres tend to orientate themselves in the 'direction of the brush', providing good edge cover – particularly important with corrosion prevention compounds. Armipent's fibrous nature also allows significant coating thicknesses to be deposited in one application with minimal sagging or 'runs'. Armipent is based on polyolefin fibres and for suitable properties between 0.5 and 1.5% by volume is required.

BP's peelable coatings

BP Chemicals Speciality Coatings, Adhesives and Sealants (SCAS) unit of Barry, South Glamorgan has available a new product for offshore called *Peelable Coatings.* This has been specially designed to protect critical welds, wall thickness test points, riser joints and other areas of regular inspection. Once applied to a cleaned and inspected area the coating protects the surface from corrosion and marine fouling, until next inspection. Then it is simply peeled off to reveal an already clean surface. Above water, Peelable Coatings can be used to protect vulnerable areas from adverse weather conditions. The product can be used on oil/gas platforms and on ships in dry dock. The two part coating comprise a base and activator supplied in a co-axial cartridge complete with static mixer. It is applied using an application gun or, for larger quantities, via a dual pumping system. Underwater application is simple, requiring no special skills and makes economical use of the divers time – application to the cleaned surface takes only three minutes.

Anti-slip deck coatings

Mebon Paints Ltd of Sutton in Ashfield has introduced a new anti-slip deck coating called *Epok Treadlite.* Epok Treadlite is a unique anti-slip coating system which can be applied by roller to oily, damp and rusted surfaces, after only minimal surface preparation. This new coating has a design life equivalent to that of conventional coatings on throughly prepared surfaces. Treadlite has been formulated to provide maximum protection and underfoot safety in light duty areas such as walkways, helidecks and plant and storage areas. As such, it finds major application both for offshore platform secondary decking and open deck areas on ships.

New dispersing resins

ICI Colours and Fine Chemicals of Manchester has available two new *'Solsperse' resins.* These products are speciality dispersing resins which are used, together with the appropriate 'Solsperse' hyperdispersants, in the production of multi-media tinters and bases. 'Solsperse' RX50 is supplied as a solution in xylene for use in industrial paint production. 'Solsperse' RW40 is a solution in white spirit and is for use in the production of air-drying paints.

Scratch hardness tester from Sheen

Sheen Instruments now offer the *Wolff Wilborn* pencil scratch *hardness tester* as an addition to its range of surface coating test instruments. It determines the resistance of coating materials to surface scratching and is of particular value for furniture or vehicle lacquers. The Wolff Wilborn pencil test, which is suitable for carrying out scratch hardness testing to ASTM D3363, uses constant pressure and variable hardness of the test tool. Pencils of various degrees of hardness are moved over the surface being tested under a fixed pressure and at a fixed angle to the surface. The degree of hardness of the pencil which damages the surface is taken as a measurement for scratch hardness.

meetings

Painting Plastics

A one day symposium on "Painting Plastics" will be held by the Institute of Metal Finishing on Wednesday 21 October

news

1987. For further information contact: The IMF, Exeter House, 48 Holloway Head, Birmingham B1 1NQ.

OCCA Symposium South African Division 1988: Call For Papers

A Symposium on "Chemistry, Coatings, Auxilliaries – Some Advances" will be held at Sun City, S. Africa, on 14-16 September 1988. The South African Division are requesting that any interested party wishing to present a paper at the Symposium submit a title and synopsis by 15 December 1987 to: The Hon. Secretary, OCCA South African Division, c/o NBRI, P.O. Box 395, Pretoria 0001, South Africa.

Verplast '87

A national conference, Verplast '87 will take place in Torino, Italy on 2 December 1987 about problems and solutions of thermosetting and polyurethanes plastic coating (RIM, RRIM, SMC, BMC, ZMC and other). Companies taking part include Fiat, BASF, Dubois, PPG-IVI, Tecnomax. For further information contact: Verniciatura Materie Plastiche, Via Imbriani, 10-20158 Milan, Italy.

company reports

Ciba-Geigy UK Group

Sales for the Ciba-Geigy UK group were £346.6 million for the first half of this year, 9% ahead of the corresponding figure for 1986. For pigments the increase in volume of sales to £54.7M (+22%) was strongly export-led. The bouyancy of the European printing industry allowed an increase in sales for printing inks. A consultancy agreement with Blythe Burrell Colours has allowed business expansion with their former products. At the end of 1986 pigments in granule form were developed for the lithographic ink industry. The demand for these pigments is expanding due to environmental reasons. Another development is a range of pigment dispersions for aqueous inks. The Unisperse PI range of products was selected for the colouration of water-based flexographic and gravure inks. For the synthetic resins, adhesives and composite products businesses sales amounted to £86.0 million (+4%) with a growing epoxy resins business.

UCB Goup

For the Chemical Sector net sales for 1986 were BF12.113M (−7% on 1985), 90% of which come from W Europe. 1986 profits before tax were BF698M (+107%). The speciality chemicals division increased turnover by 11%. Increased production was recorded in polyester powders for paints, in polyurethanes and curable resins. In the United States, the UCB Group has bought all the shares in Radcure Specialities Inc. The subsdiary UCB Electronics S.A. set up in the course of the year has as its objective to develop new ultra pure chemical products for the electronics industry, such as Plasmask. This is a new chemical resin developed by UCB to increase considerably the memory capacity of integrated circuits.

bsi news

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

British Standards

The following new and revised publications are available:

BS 4164: Specification for coal-tar-based hot-applied coating materials for protecting iron and steel, including a suitable primer.

BS 5666 Part 1: Anaylsis of wood preservatives and treated timber. Guide to sampling and preparation of wood preservatives and treated timber for analysis.

BS 6857: Specification for cadmium pigments for paints.

Amendments

4147: 1980 ECB/34 Specification for bitumen-based hot-applied coating materials for protecting iron and steel, including suitable primers where required.

6044: 1987 PVC/26 Specification for pavement marking paints.

people

Peter Rieck (39) has been appointed Managing Director of Sonneborn & Rieck Ltd – the industrial paints and finishes manufacturer who have their headquarters at Hainault, Essex.

Rob Richards has been appointed Manager of BP Chemicals New Specialities Business. He was previously Vice-President Acetyls in BP Chemicals in America.

Henry Merrell has been appointed Market Manager, Protective Coatings of DeSoto Titanine of Newcastle-upon-Tyne.

occa news

1987 Northern Section's Golf Tournament

The Northern Section's Golf Tournament was held with some 20 participants competing for the Tony McWilliam Trophy, arranged as in 1986, with Manchester Section versus 'The Rest'.

With the Pannel course set amidst beautiful countryside, fair weather and the excellent Clubhouse facilities a good day's golf was had by all, although some of the greens came in for justified criticism.

The best score of the day from Manchester member, Dick Wood, with 36 points, was closely followed by Arthur Baugh who led the guests competition with 35 points. The team prize was narrowly won by Manchester section with Walt Ollet (35) and Norman Seymour (27) and Dick Wood's scores resulting in a total of 98 points. The best three scores from other Sections forced a very tight contest with Phil Jones (34), Roger Wells (32) and John Clarke (31) adding up to a nail-biting 97 points.

After the presentation of trophies and prizes the day finished in good order with drinks and supper in the Clubhouse.

J. Hemmings

News of Members

Mr M. Nixon, Hon. Secretary, Manchester Section, has recently joined Coates Brothers Inks Ltd. in the Liquid Ink Laboratory at Milnrow, Rochdale, Lancashire, and can now be contacted on 0706 881214 or evenings on 061 485 8102.

Obituaries

N. A. Bennett (President 1957-59)

R. H. Hamblin writes:

Just before the Eastbourne Conference, news was received that Norman Bennett (President 1957-59) had died whilst on a visit to the United Kingdom from his home in Malta, where he had lived on retirement for many years. Norman Bennett had been a well-known supporter not only of this Association but also of the Paint Research Association. He was Chairman of the London Section during the dark days of the war 1943-45 and became Hon. Treasurer of the Association in 1951. As President (1957-59) he presided over the largest Conference held by the Association – at Edinburgh in 1959 – and later served as a

The Association welcomes Chris Pacey-Day

On 28 September 1987 Chris Pacey-Day took up his appointment as General Secretary of the Association.

He was educated at Northampton College of Advanced Technology, London, being awarded a degree in Applied Physics and also holds a degree from the Open University.

His career had seen employment within the research divisions of GEC, Lucas and British Steel with an interest in the physical methods of chemical analysis with particular emphasis on surface chemistry. Prior to his appointment with OCCA he has served with the Institution of Chemical Engineers, holding a number of senior positions including his most recent as Director – Membership Services.

Outside employment his main interests are local Government, where he has been an elected member of a local authority for many years and he is Chairman of a national dance organisation.

Chris Pacey-Day.

N. A. Bennett

Vice President of the Association before leaving the United Kingdom. He managed on several occasions during his retirement to arrange his visits from Malta to coincide with Council Reunions and it was always a pleasure to receive a letter from him commenting on Association activities, e.g. I can well recall a letter giving a possible derivation of the word "Dragonara" when the Association returned to Edinburgh for its Conference in 1985. Norman will be remembered by members for his sense of humour and his dedication to any task which he undertook for the Association. Not least of these was the way in which he acted for the Association when it wished to move into offices at Wax Chandlers' Hall. Since the Association was then an unincorporated body, Norman Bennett – as President – had to enter into the lease as tenant, a move which led to the incorporation of the Association as a company limited by guarantee.

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

F. Sowerbutts (President 1967-69)

R. H. Hamblin writes:

It was with very great sadness that I learnt of the death on 29 August of Frank Sowerbutts, during whose term of office as President the Association celebrated its 50th Anniversary. The excellent way in which Frank and his charming wife, Bunny, presided over the celebrations, which were such an important milestone in the Association's development, will long be remembered by all who participated in the many events. So, too, will he be remembered for the visits to the overseas Sections and the newly formed Australian Association which he undertook during his presidency. He and his wife had long planned a private world sea cruise upon his retirement from business and, as this coincided with his term as President, they graciously agreed to amend their itinerary so as to visit the Sections in South Africa and New Zealand as well as the new Australian Association. The warmth of his personality did much to ensure that the new Australian Association felt it could look to the parent Association for guidance and by his statesmanlike approach the co-operation between the two Associations has flourished ever since. I have fond memories of seeing him off on the tour, when he took with him a large portfolio of all the beautiful commemorative addresses presented to the Association at its Golden Jubilee to show the members overseas.

Frank was already a member who had done a tremendous amount for the Association when I first met him. He had helped to found the Manchester Section in 1925, being its Honorary Treasurer for 15 years. On moving to the Midlands he became the Founder Chairman of that Section in 1948. There were some who felt that, because of the existence of the BPVLC (Birmingham Paint Varnish & Lacquer Club), it would not be possible to maintain a section in that area but it is a tribute to Frank and his successors that the Midlands Section not only thrives but the activities of the Section and the club have proved complementary.

Frank was justly proud that he had served on the Council in one capacity or another for a longer continuous span than any other member. Besides being elected to Council as an Elective Council Member on five occasions and serving twice as a Vice President, the post which gave him most satisfaction (other than the Presidency) was his term as Honorary Treasurer of the Association and Chairman of the Exhibition and Finance Committees. He was immensely proud of the Exhibition and long after his Presidency he still took a keen interest in and served on both the Exhibition and Finance Committees. It was with his encouragement, following the Golden Jubilee Celebrations, that the Association took the giant step for a society composed of individual members and moved the Exhibition to Olympia.

F. Sowerbutts

Honorary Membership of the Association was conferred upon him in 1970 and he continued to serve on Council until 1979 (his last appointment being as Representative of the Auckland Section). His long and abiding interest in all aspects of the Association was an inspiration to all who came into contact with him. I last saw him at his home in Eastbourne in June during this year's Conference and his questions about the Association and his suggestions for future activities were as clear and sagacious as they had always been.

He and his wife made friends for the Association wherever they went as representatives of the Association -- to the Conference of the Federation of Scandinavian Paint and Varnish Technologists in Gothenburg in September 1967 and the FATIPEC Congress in Brussels in May 1968 – which strengthened the ties of the International Alliance.

He was a most able Chairman of Committees and Council, getting the work done and the decisions made whilst ensuring that all opinions were carefully heard. His courteous handling of meetings and the way in which he made new members welcome was a reflection of his outgoing personality. His contribution to the Association's welfare and his acts of kindness will be recalled by many members and the Association must be grateful that he was willing and able to devote so much time and effort on its behalf for such a long period.

To his widow and family the Association extends not only our deepest condolences but also our heartfelt thanks for the part which he played in our affairs.

new members

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

Ordinary members

Bryson, I., BSc., Ph.D *(Scottish)*
Dexter, J. W. *(Newcastle)*
Garnett, P. A. *(General Overseas-Zimbabwe)*
Harris, M. G. *(General Overseas-Zimbabwe)*
Jordaan, J. P., BSc *(Cape)*
Maviinga, I., BSc *(General Overseas-Zimbabwe)*
McWilliam, D. A. *(Transvaal)*
Naidoo, D., *(Cape)*
Page, T. P. *(Cape)*
Thomas, G. R., BSc *(General Overseas-Zimbabwe)*
Visser, A. *(Transvaal)*
Wimpory, M. C. *(Midlands)*

Associate members

Botha, A. S. *(Transvaal)*
Marshall, G. A. *(Cape)*
Minnie, K. *(Transvaal)*
Slag, J. B. M. *(General Overseas-Holland)*

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8. **VR Development Chemist,** (27-30) for strong expanding international company in the Midlands. Covers new product development and customer presentations and trials. MIDLANDS **£12,000 p.a.**

9. **Polyester Chemist** (27-30) to lead small development team with International organisation. SOUTH **£13,000 p.a.+car**

10. **Development Technologist** (27-30) for coatings for plastics to cover new product development plant and process development, quality control and customer trials and service. SOUTH **£13,000 p.a.**

11. **Oil Inks Technologist** for product development, process development and service. Also to run small manufacturing unit. NORTH **£15,000+car**

12. **Liquid Inks Technical Manager** (30-35) to lead team for packaging inter development trials and service. SOUTH WEST **£15,000+car**

13. **UV Technologist/Chemist** (27-30) for research and development and customer presentation and trial, within team of major ink company developing advanced UV and EB technology. SOUTH **£14,000 p.a.+car**

14. **Liquid Inks Development Chemist** to cover advanced packaging inks technology within small team of major ink maker. SOUTH **£14,000 p.a.**

15. **Oil Inks Graduate** (c30) as leader for small development team working towards innovative technology for Litho inks. SOUTH **£14,000+car**

16. **Oil Inks Technologist** as Laboratory Manager for formulation and customer development team, together with new product trial and technical service. SOUTH **£15,000+car**

Candidates for these appointments or for others on our expanding list of career jobs are invited to write in confidence to: G. P. Birtles, SITA Selection, 203 Gardiner House, Broomhill Road, London SW18 4JQ or to telephone: 01-871-5011/2.

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TECHNICAL SALES REPRESENTATIVE

Surface Coatings Industry, South of England

A major German manufacturer of raw materials for the Paint, Printing Ink, Adhesives and allied industries seeks a technically orientated representative to cover the South of England.

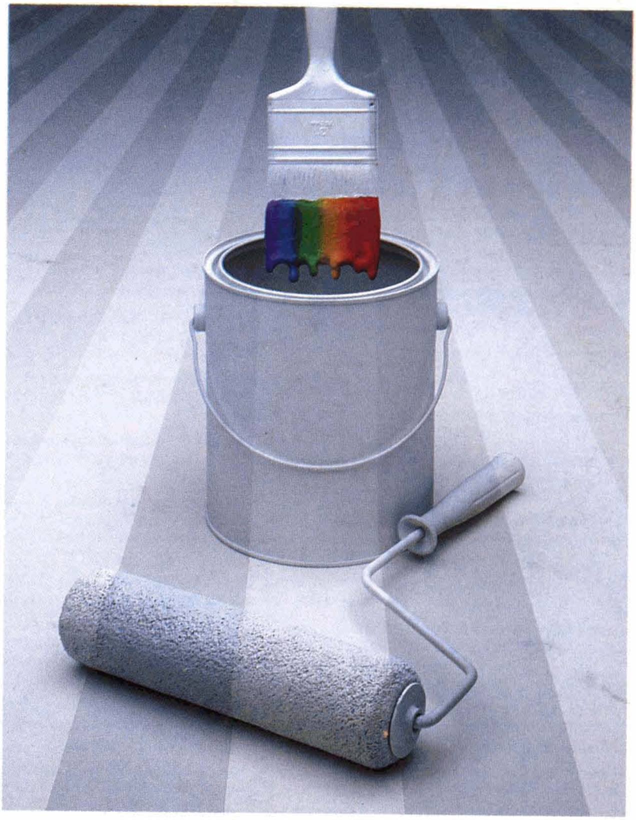
Probably aged over 25 the ideal candidate should be educated to degree level or equivalent, have a technical background in the industrial paint market and possess the ability to communicate technically and commercially at all levels.

Achieving agreed sales targets is essential in this position.

A working knowledge of German is preferable but a willingness to learn acceptable.

This is an excellent opportunity to build a career with a progressive, successful company. The total remuneration package will include attractive basic salary, sales related bonus, contributary pension scheme and company car.

Applicants are requested to initially send a full C.V. to **Box Number 568, JOCCA, 967 Harrow Road, Wembley, Middlesex HA0 2SF, UK.**

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