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by

W. A. Anderton

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**Phenomena associated with
metal-paint coating interfaces**

By **W. A. Anderton**

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Phenomena associated with metal-paint coating interfaces

By W. A. Anderton

Defence Research Establishment Pacific, Forces Mail Office, Victoria B.C., Canada

Summary

As a result of observations made during studies of ships' bottom paints, several hypotheses are suggested to explain phenomena observed. One of these is the concept of a concentration of water at the steel/coating interface: this has been called the "water rich layer". It is suggested that this layer adversely affects the adhesion of the coatings and provides a relatively conductive path for ionic current between anodic and cathodic areas. This concept is also applied to the explanation of the corrosion of coated steel exposed to atmosphere. Hypotheses are presented to explain the inhibitive effect of aluminium and some other metallic pigments on the adhesion-destroying reactions which affect some coatings on steel protected cathodically in sea water.

Key words

Types and classes of coating

primer
ship bottom paint
vinyl coating

Prime pigments and dyes

aluminium pigment
red lead pigment

Process and methods primarily associated with service or utility
corrosion

Phénomènes associés avec les interfaces métal-peinture

Résumé

On propose de diverses hypothèses pour expliquer les phénomènes observés lors des études sur quelques peintures pour carènes de navires. Une de ces hypothèses comprend le concept d'une certaine concentration de l'eau à l'interface acier-revêtement, que l'on appelle la Couche Riche en Eau. On suggère que cette couche exerce une influence adverse sur l'adhérence des revêtements, et qu'elle fournit, pour le courant ionique, un trajet assez conducteur entre les zones anodiques et les zones cathodiques. On applique également ce concept à l'explication de la corrosion, pendant l'exposition à l'atmosphère, de l'acier revêtu de peinture. On présente des hypothèses pour expliquer l'effet inhibitif de l'aluminium et d'autres pigments métalliques sur les réactions qui détruisent l'adhérence de certains revêtements sur l'acier, protégé cathodiquement, pendant l'immersion en eau de mer.

In Verbindung mit Metall-Anstrichfilm Grenzflächen Stehende Phänomene

Zusammenfassung

Als Ergebnis von Beobachtungen beim Studium von Schiffsbodenfarben werden verschiedene Hypothesen vorgebracht, um die beobachteten Phänomene zu erklären. Davon ist eine die Vorstellung von einer Konzentration von Wasser unter der Stahl-Anstrich Grenzfläche; dies ist die "Wasserreiche Schicht" genannt worden. Es wird nahe gelegt, dass diese Schicht die Haftung der Farben nachträglich beeinflusst und einen verhältnismässig gut leitenden Pfad für jonischen Strom zwischen anodischen und kathodischen Stellen liefert. Dieselbe Vorstellung wird auch auf die Erklärung der Korrosion gestrichenen und der Atmosphäre

ausgesetzten Stahles übertragen. Hypothesen, um die inhibierende Wirkung von Aluminium- und einiger anderer metallischer Pigmente auf die Haftung zerstörenden Reaktionen, denen manche Anstrichmittel auf kathodisch geschütztem Stahl in Seewasser zum Opfer fallen, zu erklären, werden vorgetragen.

Явления связанные с поверхностями раздела красочных покрытий на металле

Резюме

В результате наблюдений при изучении красок предназначенных для днищ судов, предлагается ряд гипотез для выяснения наблюдаемых явлений. Одной из этих гипотез является понятие о концентрации воды на поверхности раздела между сталью и слоем краски, на так называемом «водно-насыщенном» слое. Полагается что этот слой оказывает неблагоприятное влияние на адгезию покрытий и вызывает участок сравнительной проводимости для ионного тока между анодными и катодными областями. Это понятие также применяется в качестве объяснения коррозии покрытой стали подверженной действию атмосферы. Предлагаются гипотезы объясняющие ингибирующее влияние алюминия и некоторых других металлических пигментов на противомалгезионные реакции, которые оказывают влияние на некоторые покрытия для катодно-защищенной стали в морской воде.

Introduction

For some years studies have been carried out on marine coatings at the Defence Research Establishment Pacific* (DREP), which provides scientific advice to the Canadian Armed Forces. In this programme, ships' bottom coatings have been given the greatest priority.

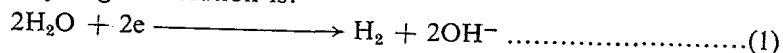
An evaluation procedure has been developed¹ which is in continuous use at this laboratory. Since Canadian Naval vessels are protected cathodically, a test for coatings on cathodically protected steel is incorporated into the procedure. The equipment used for the evaluation procedure has made it possible to conduct experiments, with frequent observations, on a variety of coatings on cathodically protected and unprotected steel in natural sea water.

From these experiments, and the work of others, hypotheses have been suggested, some of which are presented below.

Loss of adhesion with cathodic protection

The tendency for some paint coatings to lose adhesion near a bare area on cathodically protected steel immersed in sea water has been described elsewhere². A strongly alkaline condition develops at the steel-coating interface where adhesion has been lost.

The reaction taking place at a cathodic steel surface at a potential sufficiently negative for hydrogen evolution is:

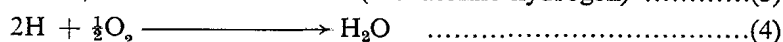
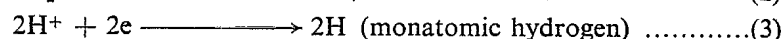


The destruction of the adhesion of coatings near bare areas on steel cathodically protected at potentials negative enough for hydrogen evolution is considerably greater than that of similar coatings on steel at less negative potentials. Reaction (1) may be initially operative only at bare areas and at underfilm areas immediately adjacent to them. As the distance along the

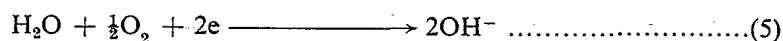
*Formerly known as the Pacific Naval Laboratory.

substrate away from the bare areas increases, the voltage drop due to resistance will reduce the potential between coating and steel until it is no longer in the range required for hydrogen evolution.

If the potential is not sufficiently negative for hydrogen evolution, oxygen is required and the following reactions are applicable:



The overall reaction is:



If water, oxygen and electrons are supplied to an area of steel/coating interface at a potential in the range of reaction (5), hydroxyl ions will be produced there at the same rate as that of the reactant having the lowest rate of supply when all rates are expressed in equivalents per unit time. If the rate of alkali production is changed, it will be because the limiting rate of supply has been changed. The effect of a bare area must be to increase the rate of supply of one or more of the reactants, since it accelerates alkali production at the adjacent coated interfacial area. This implies the lateral transmission through the film to the area of one or more of the reactants. Paints generally have much lower diffusion rates for ions than they do for water or oxygen, and it is assumed that, at least initially, the ion diffusion rate is the controlling factor. To increase significantly the hydroxyl production at the steel/coating interface adjacent to a bare area, there would have to be a considerable increase in the exchange of ionic current between the area and the sea water environment over what would occur with an unflawed coating. The resistance of the film to the ionic current may be reduced with time as adhesion is lost until current supply is no longer the controlling factor. The lateral supply of oxygen and water may also increase with loss of adhesion.

This argument assumes that the alkali is generated at the steel/coating interface by reaction (5) only, and does not diffuse laterally from a site of production at the bare area. Experimental evidence for this is shown below.

Two mild steel panels, 3in \times 4in were grit-blasted and a coat of wash primer applied. Small circular pieces of universal pH paper were fixed at scattered positions on the face of the panel, which was scored with a standard X-cut after four unpigmented vinyl topcoats had been applied.

One of these panels was immersed in a beaker of sea water made alkaline by the addition of sodium hydroxide (pH > 14). In 29 days, there was no indication that alkali had penetrated from the scratch. A band of rust blisters had developed around the score, and a spot on a piece of pH paper near one of these indicated acid.

The other panel was protected cathodically at -0.9 volt (vs Ag/AgCl) in a sea water electrolyte maintained at a pH < 1 with hydrochloric acid. The catholyte was separated by a cotton bridge from the anolyte, which was an aqueous solution about 1 N in sodium hydroxide.

After 3 days it was noted that a piece of pH paper close to the bare area indicated acid adjacent to the score, but strong alkali at points further removed. There was a similar situation with two pH indicators having paper fibres which penetrated the coating. Points on the indicators close to the fibre indicated strong acid; at a greater distance strong alkali was indicated.

The water rich layer

Ions do not usually readily migrate directly through paint coatings, whether they are immersed or not. Why should the resistance to lateral ion migration through the film be so low relatively, with these cathodically protected panels, that there is a considerably larger ionic current along this much longer path than the one directly through the film? If most of the ionic current went directly through the film, it would be expected that the effect would develop at all areas and gradually intensify, rather than starting adjacent to a bare area and spreading out from there as it does. Any modification in the resistance of the affected coating is likely to involve the distribution of water. Good, dry, paint coatings have a low dielectric constant, and ionic materials have little tendency to ionise in them.

It is suggested that water will tend to concentrate in the paint coating near a hydrophilic steel/coating interface in a "water-rich layer" (WRL). Ionic materials can ionise in this layer. If the ionic substances remain in solution the resistance of the WRL will be considerably reduced. Sea water cations, such as Na^+ , migrating through the WRL to cathodic steel would tend to remain in solution in the WRL and lower its resistance. Such cations will be hydrated, and will carry more water into the layer. If the adhesion is destroyed, oxygen may also diffuse along the interface.

Mayne has proposed that water may not always be distributed homogeneously in a paint film, but tends to concentrate around ionogenic sites in the polymer³. He supported this view with the evidence of phase-contrast microscope photographs. The author suggests that the water would also concentrate in water-rich envelopes around hydrophilic pigments, as well as at the hydrophilic metal substrate.

Since first making this suggestion the author has noted from the literature that others have arrived at a similar conclusion. Gay⁴ wrote that his experimental data indicated that water passes through paint films on steel by osmotic or electroendosmotic processes and builds up at the interface between primer and steel. Michaels⁵ stated that "the hydrophilic nature of the particle surfaces renders them capable of adsorbing significant quantities of water: if the particles are very small, their water-adsorptive capacity may exceed by a wide margin the water-absorption of the matrix resin in which they are imbedded". Kumins⁶ concluded that: "Ion transport across these films may involve the initial passage of water vapour and oxygen which concentrate at the metal-film interface". Funke, Zorll and Murthy⁷ have also concluded that water may concentrate at both the pigment/coating and steel/coating interfaces.

It may be useful to review the other evidence that led to this conclusion.

Unprotected panels coated with a nitrocellulose lacquer and scored with an X-cut were also strongly alkaline under the film and had lost adhesion

near the score after a period of sea water immersion. In this case, it may be assumed that the underfilm area is cathodic to corroding bare steel at the scratch.

It is difficult to measure the current passing along the WRL as compared to that which goes directly to bare steel at the scratch on a scored, cathodically protected, panel. However, the cathodic current to such a panel often increases to double or more in 100 days, whereas the cathodic current to a bare steel panel decreases during this period. If the excess current passes along the WRL, this portion of the cathodic current will generally be in the order of 10 μ amp in the case of the red lead or aluminium vinyl ships' bottom systems investigated.

Sometimes severe pitting is experienced at a scratch when an unprotected panel is immersed in sea water or ammonium chloride solution. The only cathodic area other than that on bare steel at the scratch is under the film, yet the extent of corrosion makes it seem unlikely that all the cathodic areas in such cases would be on the very small areas of exposed steel. Also, some types of coats will be accompanied by considerably more severe pitting than others under these conditions. Nitrocellulose coated panels are subject to this pitting, and these readily form an alkaline underfilm area near a scratch.

According to Bullett and Rudram⁸, all the liquid in a blister, in many cases, could not have passed directly through the blister cap but must have migrated laterally under the film from some distance around. It has frequently been observed that blistering of vinyl coatings is much more prevalent near a bare area. It is suggested that some of the blister solution migrated along the WRL. Hydrated cations moving towards a cathodic blister site would contribute to the blister solution, which has invariably been strongly alkaline on the bottoms of the ships investigated.

When scored panels with an unpigmented alkyd coating were immersed in ammonium chloride solution with and without cathodic protection, two layers developed in the film. There was an adherent lower layer with a yellow colour, and an uncoloured upper layer with good cohesive strength. There was a loss of adhesion between the two layers, which were fairly easily separated. IR spectra of the two layers showed them to be similar with minor differences. It is known that ammonia will cause yellowing of alkyd films. If ammonia caused the yellowing it may have been present in the layer near the substrate after having migrated as NH_4^+ along the WRL.

A panel coated with wash primer and then with a scored, unpigmented vinyl coating was exposed for 129 days to a 100 per cent RH atmosphere which was contaminated with ammonia. At the end of this period, adhesion was reduced to a very low value. The colour of the wash primer became grey (similar to the colour observed on scored, cathodically protected panels immersed in sea water). Drops of a green solution were observed at the cut at the end of the first week of exposure. A blister filled with a bright green solution had developed.

In this case, the alkali may have aggravated the loss of adhesion and helped to cause the very low adhesion observed. The adhesion was considerably greater on similar panels exposed to 100 per cent RH without ammonia contamination.

The most likely cause of the green colour is Cr^{3+} which has been produced by reduction of the Cr^{6+} present in the zinc tetroxy-chromate pigment of the wash primer. The solution would have to leach out along a layer near the interface to get to the cut.

Funke⁹, in a paper dealing mainly with the pigment/coating interface, made the remark: "Earlier investigations with unpigmented films showed that the amount of water absorbed by the supported film was in no case less than by the corresponding free film. Moreover, supported films frequently absorbed considerably more water than free ones and tended to blister formation after prolonged immersion. It could be stated that, generally, an increased water absorption of supported films indicated loss of adhesion to the substrate, and finally resulted in blister formation or even film detachment. In other words, a comparison between the water absorption of free and supported films enables one to judge whether the interaction between the film and substrate is strong enough to resist the penetration of water into the interface."

Walker¹⁰ has shown that, on exposure of coatings on stainless steel to an atmosphere of high relative humidity, adhesion was reduced below the initial value. He measured initial adhesion values of various coatings on stainless steel and also the adhesion of these coatings after intervals of exposure under controlled humidity. With all types, there was a reduction of adhesion at relative humidities greater than 90 per cent. At 100 per cent RH the adhesion was, in almost all cases, reduced to less than half the initial value and in some cases to less than 20 per cent. The drop of adhesion, as a percentage, during exposure, varied considerably with various types of coatings. The initial adhesion values were not generally a good indicator of the adhesion after exposure to a humid atmosphere. The loss of adhesion seemed to have been caused by entry of water into the film, with the initial adhesion having no obvious relationship to the adhesion when exposed to a humid atmosphere. This loss of adhesion could be explained by a concentration of water adjacent to the substrate.

This evidence suggests a water concentration near the substrate with a consequent loss of adhesion which will be more severe if the water rich layer is alkaline. The effect of the alkali may be to increase the water concentration by osmotic effects.

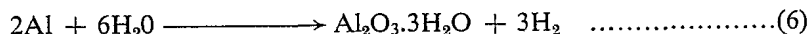
As has been explained elsewhere², unpigmented vinyl coatings will tend to lose adhesion near a bare area when protected cathodically if the electrolyte is an aqueous solution of the chloride of sodium, potassium or ammonium. They will retain it under similar circumstances with the chloride of calcium or magnesium, or with hydrochloric acid. It is suggested that the alkali and ammonium cations tend to remain in solution in the WRL and to lower its electrical resistance. They promote cathodic reactions on coated steel adjacent to bare areas, with the consequent production of alkali and loss of adhesion. The hydroxides of calcium and magnesium are less soluble and tend to precipitate in the WRL. Moreover, dissolved carbon dioxide may convert them to the still less soluble carbonates. With the hydrochloric acid electrolyte, the resultant water would not have much effect on the resistance of the WRL.

The effects of metallic pigments in inhibiting loss of adhesion

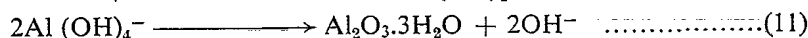
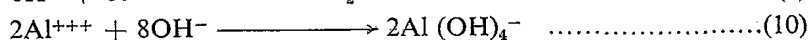
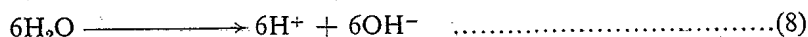
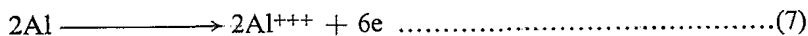
The effect of aluminium pigment in inhibiting loss of adhesion on cathodically protected steel has been previously discussed^{1,2}. The standard Canadian Naval bottom system has consisted of wash primer applied to grit-blasted steel, followed by a red lead pigmented vinyl primer and then a cuprous oxide pigmented vinyl antifouling paint. This system is subject to adhesion loss with cathodic protection. It is being replaced by one in which an aluminium pigmented vinyl primer replaces the red lead primer. The latter system has given very successful performance on our Naval vessels.

If the effect of the aluminium pigment is to increase the transmission of alkali through the film, a lower concentration of adhesion-destroying alkali will remain at the interface. The possibility of a water concentration around hydrophilic pigments has been mentioned. This may occur around the aluminium pigment particles.

Near a bare area on cathodically protected steel the aluminium pigment is converted to a hydrated aluminium oxide and the proposed reaction is:



There is no alkali consumption in this reaction. It is likely, though, that hydroxyl is temporarily combined as the aluminate. The following intermediate reactions are suggested:



Reaction (6) is the resultant of these reactions.

The result of reaction (10) would be a strong affinity of hydroxyl for aluminium. The solution of aluminium may result in a high concentration of water around the pigment particles because of osmosis effects. The water rich envelopes around pigment particles may overlap so that there is a relatively low resistance path for hydroxyl ion through the film to the sea water environment. The hydroxyl ion does not remain concentrated at the substrate but is transmitted through the film. Since hydroxyl ion originates in water at the substrate there may also be a dehydrating effect.

There may be an additional factor involved if reaction (5) is in operation. When alkali dissolves the oxide coating from aluminium pigment particles, the exposed metallic aluminium is very reactive to diffusing oxygen. The oxygen which reacts with aluminium does not reach the substrate to generate hydroxyl ions by reaction (5). The rate of production of hydroxyl ion is lowered, and that which is produced is transmitted through the film. Some hydroxyl ions must be produced at the substrate to dissolve enough of the oxide coating for oxygen scavenging to take place.

It has been confirmed with steel panels protected cathodically at -1.2 volt (*vs* Ag/AgCl) and scored with a standard X-cut that alkali is transmitted through an aluminium vinyl film at a greater rate than through a similar red lead vinyl film. The vinyl system consisted of two coats of either aluminium vinyl or red lead vinyl over wash primer. Pieces of universal pH paper were laid down over these layers, followed by a vinyl topcoat which was the vinyl antifouling paint without pigment. A gradually increasing band of discolouration developed around the scratch in each case, but this was better defined with the aluminium vinyl. Shortly after the boundary of discolouration on the aluminium vinyl panels became tangential to a piece of pH paper, the whole piece indicated strong alkali (Fig. 1). The alkaline blue colour developed more slowly with the red lead vinyl, and only after the pH paper was surrounded by the area of discolouration was there any indication of a lower pH than with aluminium vinyl (Fig. 2).

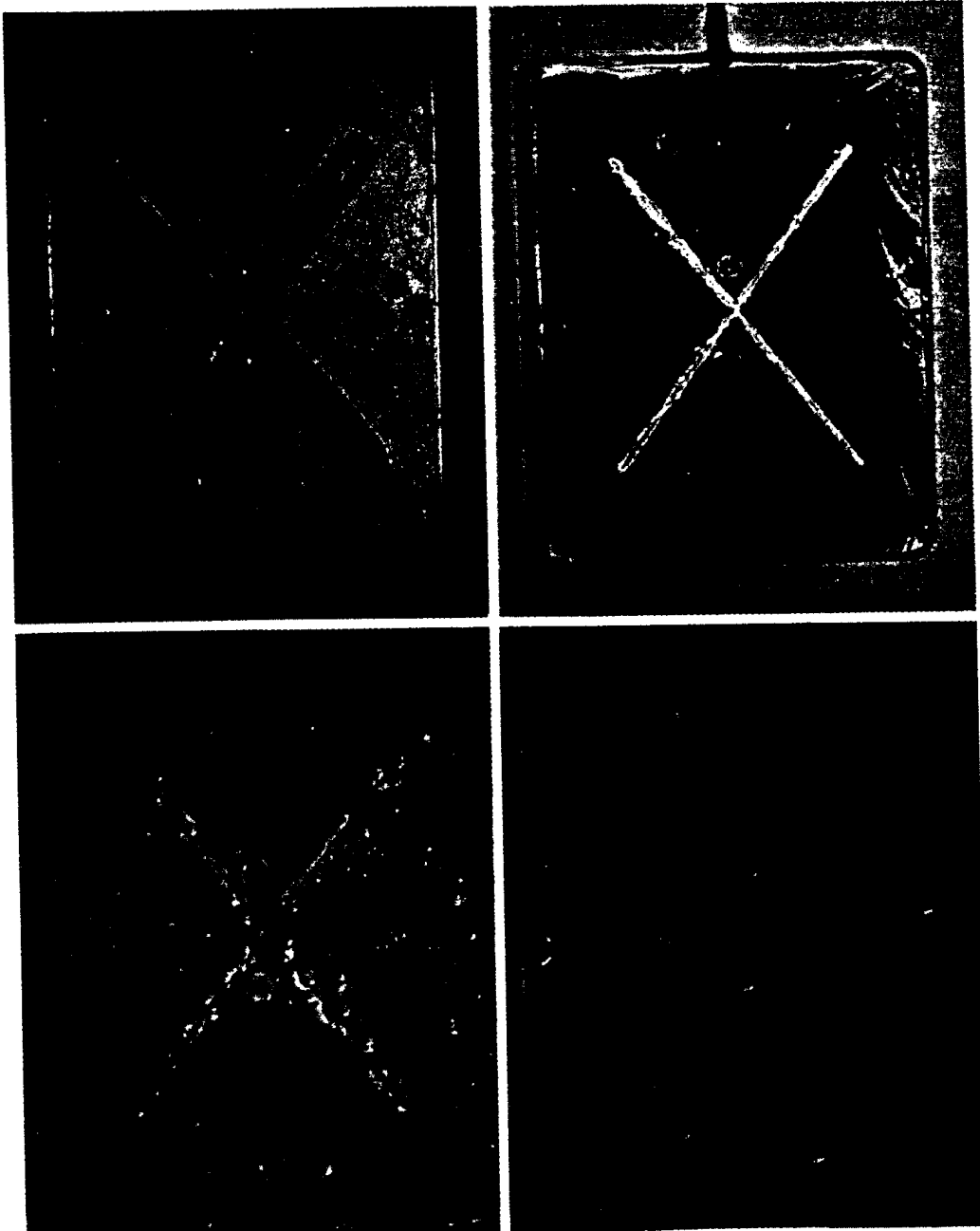
Similar panels were immersed at -1.0 volt. The alkaline colour was slow in developing with the red lead vinyl; no indication of alkali was observed on the aluminium vinyl panels. The lower alkalinity, in the case of aluminium vinyl, could be explained by a lower rate of production of hydroxyl ion due to oxygen scavenging by the aluminium pigment (Figs. 3 and 4).

At potentials more negative than -1.1 volt (i.e. in the hydrogen evolution range, where alkali is produced which must diffuse through the aluminium vinyl coating), it might be expected that there would be an upper limit of thickness at which coatings would resist adhesion loss. The diffusion rate would decrease as thickness increased and, if sufficiently thick, an alkaline condition would again develop near the substrate. It was found that, with 25mil of aluminium vinyl on panels protected cathodically at -1.2 volt and -2.0 volt, adhesion was lost from $\frac{1}{4}$ to $\frac{3}{8}$ in from the scratch in three months, and there was extensive blistering in this area. The blisters were dry, indicating that they may have been caused by hydrogen gas. When cut near the scratch, gas and liquid oozed from the film. The liquid was strongly alkaline.

Vinyl primers pigmented with stainless steel and zinc have also resisted adhesion loss with cathodic protection at -1.0 volt. It is suspected that the stainless steel pigment had become electrically connected to steel and that enough particles were in contact to give the layer appreciable electrical conductivity. This was confirmed by resistance measurements between points on the surface of the layer of stainless steel pigmented vinyl primer. If this had occurred, oxygen would be consumed by reaction (5) at the pigment particles. Although alkali would be produced there, less oxygen would be available at the substrate for production of adhesion-destroying alkali. A similar mechanism might explain the performance of the zinc pigmented primer.

Figs. 1 to 4. Transmission of alkali

1. (top left) Aluminium vinyl, cathodic protection $-1.2v$ (*vs* Ag/AgCl)
2. (top right) Red lead vinyl, cathodic protection $-1.2v$ (*vs* Ag/AgCl)
3. (bottom left) Red lead vinyl, cathodic protection $-1.0v$ (*vs* Ag/AgCl)
4. (bottom right) Aluminium vinyl, cathodic protection $-1.0v$ (*vs* Ag/AgCl)
None detected



Figs. 1 to 4. (see opposite)

In this case, oxygen scavenging by a direct reaction of oxygen with the zinc may also occur.

Effect of the water rich layer during atmospheric exposure

Before underfilm corrosion occurs there must be a concentration of water in the film at the steel/coating interface. If the rate of the corrosion reactions is controlled by electrical resistance, the effect of dissolved substances in the WRL will be important. If there is little in solution, or if the dissolved substances are inhibitive, the corrosion rate will be low. But if corrosion accelerators, such as chlorides, sulphates, or sulphites, are in solution, the corrosion rate may be much greater.

Since the contaminants may have to diffuse through the paint film as ions, the permeability of the film to ion diffusion and hence the electrical resistance, will be a factor in the performance of the paint system. It may be, though, that some atmospheric contaminants, such as ammonia or sulphur dioxide, may penetrate the film in the molecular form and ionise in the WRL. The sulphur dioxide may be converted to the sulphate ion and in this form become locked in the WRL.

Harrison¹¹ has described an atmospheric exposure test using five-foot lengths of six-inch channel girder section, deliberately selected as being as near as possible to the completely millscaled state. These sections are rusted outdoors for six months and wire brushed using a rotary wire brush. Two coats of primer are applied to the rear and two coats of primer and one finishing coat to the front or weather-face. Experience has substantiated a statement made in 1962, to the effect that this girder test is the most reliable accelerated test extant when using the north facing primer coats as a means of prediction. He reports that, when carried out carefully, the onset of breakdown of the primer coat in months when applied in one coat is, by and large, the performance of the system (two coats primer, one coat finish) in years. He now uses two coats of primer rather than the original single coat to eliminate null tests due to over-brushing by applicators of one coat of primer.

To explain this phenomenon, it is suggested that a given primer influences the results not only by its thickness which determines its effectiveness as a barrier to contaminants of the WRL, but also by its inhibitive properties, which are independent of thickness. The barrier effect of the complete paint system delays the onset of corrosion, but the effectiveness of the system is also determined by inhibitors from the primer which become dissolved in the WRL. If corrosion begins when enough corrosion accelerators have become dissolved in the WRL to counteract the effect of the inhibitors, it will be delayed by a system having low permeability to atmospheric contaminants. A system of the same permeability but with less inhibitive properties would be subject to under-rusting at an earlier time.

In addition to diffusion through a paint film, there are two other ways in which impurities may get into the WRL. They may have been on the surface when coatings were applied, or they may have migrated from a flaw or bare area along the WRL.

If corrosion accelerators were present on the surface at the time of paint application, the life of the coating would probably be considerably reduced.

Rust in industrial atmospheres, for example, has been shown to become contaminated with ferrous sulphate and to affect paint coatings adversely.

Ionic contaminants which migrate underfilm from a bare area are likely to be alkali or ammonium cations, since the coated area tends to be cathodic to a bare area. The alkali cations are probable in a marine environment, Harrison and Tickle¹² have pointed out that "the ammonia content of the atmosphere is always positive and is the most consistent measure of atmosphere pollution in a given locality, not only on a national basis, but on a worldwide scale, irrespective of environment, be it industrial or rural." Ammonia, then seems to be a probable contaminant at any location. If the interfacial area is cathodic it will inhibit the entry of anionic accelerators, such as chloride or sulphate.

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