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An organic coating pigmented with strontium aluminium polyphosphate for corrosion protection of zinc alloy coated steel

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ABSTRACT

An organic primer pigmented with strontium aluminium polyphosphate for the corrosion protection of zinc alloy coated steel was investigated by electron microscopy and electrochemical impedance spectroscopy after exposure to sodium chloride solution. The development of defects within the organic coating was found to be related to the solubility of the pigment, which resulted in two conflicting effects. On the one hand, the inhibitive species released from the solid pigment reduced the corrosion rate of the metal substrate by formation of precipitated products containing zinc, strontium, aluminium, phosphorus and oxygen. On the other hand, the dissolution of the inhibitive pigment resulted in porosity in the coating that created easy pathways not only for the inhibitors to reach the substrate but also for the aggressive species to migrate inwards to the metal surface and, importantly, for the transport of corrosion products outwards.

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1. Introduction

Organic coatings are widely used to protect metallic materials [1–3]. Such coatings provide barrier and active protection to the metal substrate mainly through the organic binder and inorganic pigments/fillers. The polymer binder and barrier fillers together contribute to the barrier protection of the metal substrate, while active protection is achieved by incorporation of corrosion inhibitive pigments [4–7]. Phosphate and polyphosphate inhibitive pigments, which are combined with one or more cations of zinc, calcium, aluminium, lithium, magnesium and chromium [8–23], are commonly used to replace traditional chromate-containing pigments that are environmentally hazardous and carcinogenic. These phosphate or polyphosphates pigments are assumed to be sparingly soluble in water but release free ions that protect the metal substrate at locations of coating damage by passivating the exposed metal or by otherwise forming a protective, precipitated, salt film. Hence, the solubility of corrosion inhibitive pigments plays a significant role in the protection of metal substrates. The low water solubility of the zinc phosphate pigment is suggested to be responsible for its relatively

poor corrosion inhibition properties [17] in epoxy/polyamide coatings for protection of steel substrates. Zinc aluminium polyphosphate shows better corrosion inhibition properties compared with conventional zinc phosphate, which is attributed to an increased solubility [17]. Further, strontium aluminium polyphosphate has also revealed greater inhibitive properties again with increased water solubility [21,23]. Strontium aluminium polyphosphates (SAPP) have been studied on steel substrates [16,21,23] and are able to reduce the corrosion rate, assessed by electrochemical impedance and polarisation behaviour, both in solution extracts and in pigmented organic coatings.

Although the use of strontium aluminium polyphosphate has been studied on steel substrates, little information is available to date on applications for zinc and zinc-alloy coated coiled steel. In the present study, a model coil coating primer, comprised of a polyester binder and pigmented with strontium aluminium polyphosphate, applied on zinc alloy coated steel was studied using electrochemical impedance spectroscopy (EIS) and scanning electron microscopy (SEM). The measurement of impedance over a large frequency range provides information about various processes involved, including coating degradation and metal corrosion [24–27]. Such information was obtained by simulation of measured data using an equivalent circuit, in which coating properties are associated with high frequency circuit components, with under-coating corrosion associated with low frequency impedance

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components. Further, electron microscopy together with energy dispersive X-ray analysis provides observation of the physical and chemical changes of the coated system during degradation. Thus, via EIS measurements of the coil coated system, and direct observation of organic primer degradation by SEM, the overall corrosion protection behaviour to the metal substrate by the primer coating pigmented with strontium aluminium polyphosphate inhibitor has been revealed.

2. Experimental

A model binder system was formed using a linear polyester resin (Dynapol LH 820–16 from Evonik) that was cross-linked with an aliphatic poly-isocyanate (Tolonate D2 from Perstorp). The binder was pigmented with a nominal fixed volume of 4.59 v/v% titanium dioxide (from Kronos) + 0.41 v/v% of hydrophilic fumed silica (from Aerosil), and 15 v/v% of strontium aluminium polyphosphate (from Heubach). The model binder and pigments were mixed under high shear in a DAC 150.1 FVZ speedmixer for at least 5 min, and the coating system was then applied to a metal substrate of zinc 5% aluminium alloy coated (average 10 μm) steel (800 μm thick), with a roll bar coater to give a wet-film thickness of 24 μm . The coated steel panels were subsequently cured at a temperature of 300 °C for 1 min and then water quenched at room temperature. After curing, the average thickness of the coating was about 5 μm . The electrochemical impedance measurements were carried out using a three-electrode cell with a working electrode of exposed area 1.8 \times 2 cm, a cylindrical platinum counter electrode and a saturated calomel reference electrode (SCE). The samples were exposed in 0.6 M NaCl solution, made from analytical grade chemicals and deionised water, held at room temperature. Impedance measurements were conducted at the free corrosion potential using a Gamry Reference 600 Potentiostat (Gamry Instrument), with a 10 mV sine amplitude and a frequency range from 0.1 Hz to 10⁵ Hz. Separate samples after immersion for 1, 7, and 57 days were rinsed in deionised water and dried in a cool air stream before SEM examination. Surface and cross sectional examinations of organic primer and zinc alloy coated steel before and after immersion in 0.6 M sodium chloride solution were carried out by scanning electron microscopy (SEM) using a ZEISS Ultra 55 instrument with energy dispersive X-ray analysis (EDX) facilities and Quantum 3D FEGSEM with focused ion beam (FIB) facilities. The cross sections of the zinc alloy coated steel were also examined by transmission electron microscopy (TEM) using a JEOL FX 2000 II instrument operated at 120 kV, with a nominal sample thickness of 15 nm prepared by a Leica EM UC6 ultra-microtome with a diamond knife.

3. Results

3.1. Electron microscopy examination

The SAPP particles used as corrosion inhibitive pigments in the organic coating were examined by scanning electron microscopy (Fig. 1a), which showed them to be of irregular shape and of varied dimensions, from 0.2 to 5 μm across. EDX analysis indicated that the SAPP particles are likely to be a mixture of different chemical compounds, with the majority of the particles containing strontium, aluminium, oxygen, and phosphorus. The strontium to aluminium ratio ranged from 68:1 to about 0.3:1. Representative EDX spectra for a strontium-rich component and an aluminium-rich component are shown in Figs. 1b and c. The zinc alloy coated steel surface before application of the organic coating was also examined by scanning electron microscopy, which revealed the presence of zinc primary phase, and the eutectic phase where the zinc-rich phase and the aluminium-rich phase are present in the

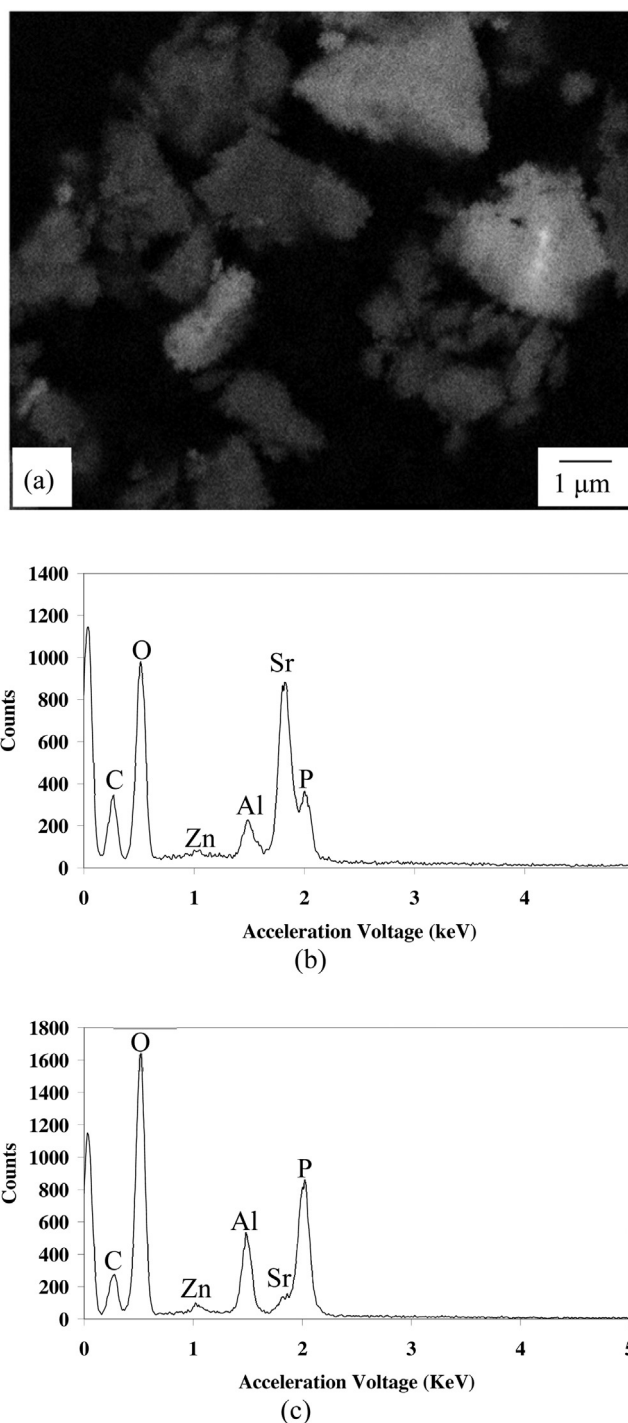


Fig. 1. (a) A scanning electron micrograph of SAPP inhibitive pigment; (b) EDX analysis of strontium-rich SAPP pigment; (c) EDX analysis of aluminium-rich SAPP pigment.

same grain, as indicated in Fig. 2a. A thin conversion oxide film, of about 50 nm thick, containing manganese, silicon, oxygen, and phosphorus was found by cross sectional examination of the zinc alloy employing transmission electron microscopy (Fig. 2b) and EDX analysis.

After organic coating had been applied, the surface of the zinc alloy coated steel did not appear smooth, with random distribution of protruded substances (Fig. 3a). Cross sectional examination showed that these protrusions were associated with large sized SAPP pigment particles, or particle clusters, appearing as lighter

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