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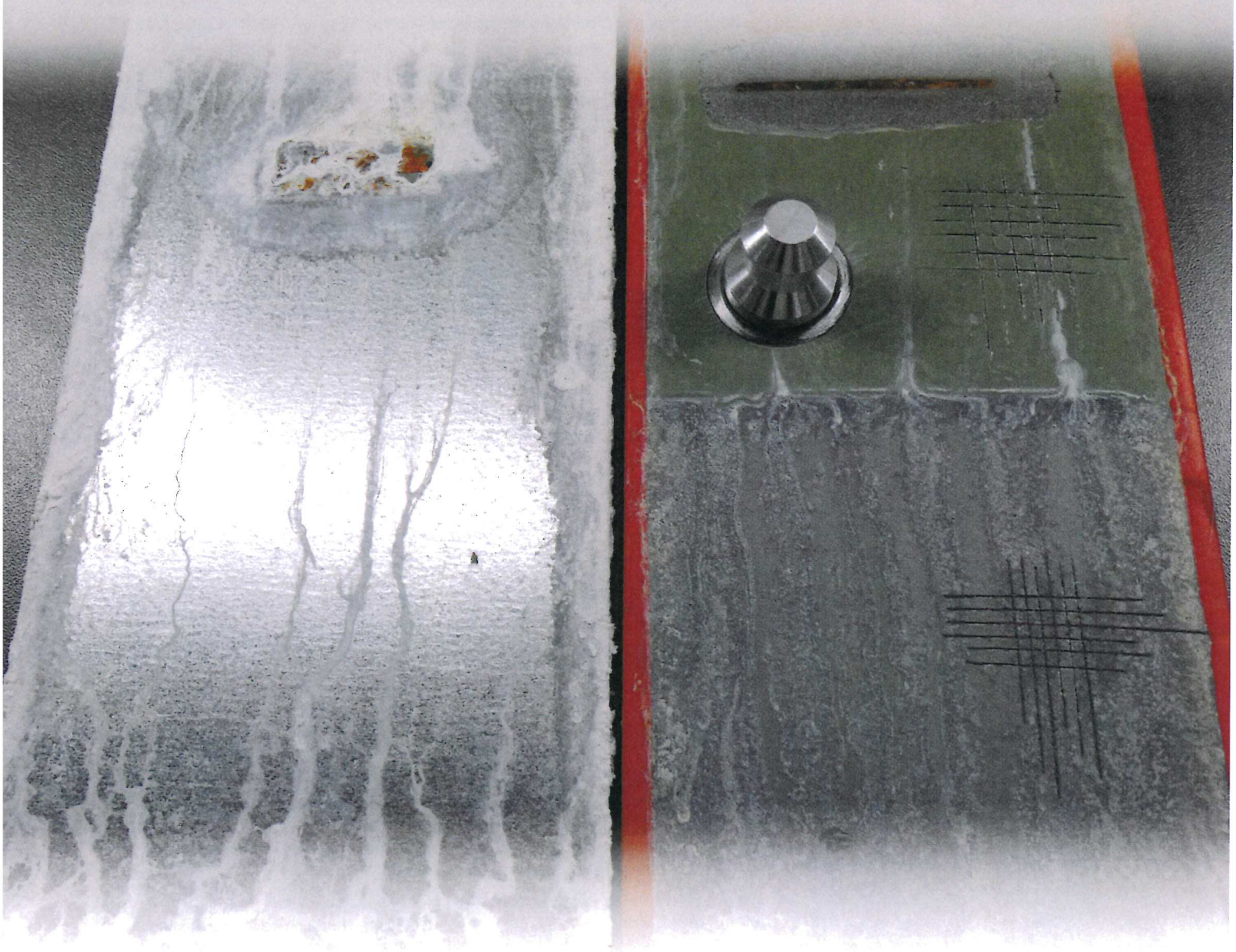

In this issue

- Read about the investigation into the degradation mechanisms of low-voltage instrumentation and control cables used in nuclear power plants
- Learn about the development of zinc flake–based primers as a sustainable alternative to traditional zinc dust coatings and galvanization
- Get to know Qinghua Han, a materials engineer at Rolls-Royce and a specialist in electrical insulation materials for aerospace applications

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Protective Coatings in Highly Corrosive Atmospheres with Focus on Environmental and Resource Conservation

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Key words: zinc flake primer, cathodic protection, zinc reduction, environmentally friendly coatings

Zinc lamellar particles offer a new approach to cathodic steel protection, presenting a more environmentally friendly alternative to hot-dip galvanizing.

Zinc is the most important raw material for the corrosion protection of steel, but it is also environmentally harmful as well as energy intensive to produce. All this considered, we should move on to a more economical use. One possible solution is the use of zinc pigments in organic coatings in spite of galvanization. Corresponding products are already widespread in the form of zinc primers containing spherical particles.

A reduction of zinc in organic coatings can be achieved by altering the shape of the zinc particles from spherical to lamellar. With the testing series shown in this article, it was possible to determine the minimum yet effective amount of lamellar zinc flakes in an epoxy-based coating system. The results showed that a cathodic protection effect comparable to conventional zinc dust paints is achieved with a zinc flake content of at least 13% by volume. Which, coming from a zinc dust primer compliant to ISO 12944 with a zinc content of 50% by volume, is a significant reduction. It has also been determined that a further reduction of the zinc flake content below 13% causes a rapid decrease of protective properties of the coating material. A brief outlook is given in the following on two starting points to solve this problem.

In addition to having a lesser environmental impact, the use of zinc flake pigments has also been shown to improve adhesion on smooth steel as well as intercoat adhesion. Other positive aspects are a greater yield per square meter, better formulation flexibility, additional barrier protection, and reduced affinity to sedimentation. Moreover, various options for the reduction of volatile organic compounds (VOC) are being highlighted.

In comparison with conventional methods (galvanization, zinc dust primer), through the combination of zinc flakes with solvent-free or waterborne binder types, more environmentally friendly and highly efficient coating systems for the greatest demands in terms of corrosion protection are already available, even if they do not appear yet in the standards.

Introduction

The concept of cathodic protection using zinc dates back to the early 19th century. Sir Humphry Davy, a British scientist, found out that more reactive metals sacrifice themselves and so prevent the corrosion of copper on naval ships. Zinc became, because of its widespread availability, the most commonly used cathodic protection for steel and iron structures exposed to corrosive atmospheres. In the mid-20th century hot-dip galvanizing became one of the most widespread applications for long-term protection of steel structures. As industrialization progressed and steel construction expanded, the demand for metallic zinc also increased. However, the production of metallic zinc is associated with high energy and cost expenditure. A total of 16 MJ are required to produce 1 kg of pure zinc from zinc ore (primary zinc). For steel, this value is 9 MJ/kg [1]. The use of an active corrosion-protection mechanism is necessary to protect steel components in extremely corrosive environments (e.g., off shore). Through an electrically conductive contact be-

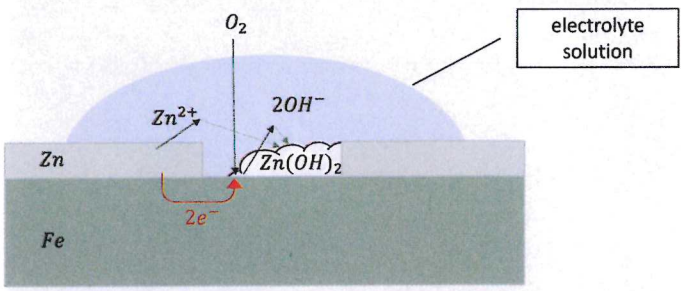

Figure 1. The cathodic corrosion-protection mechanism is based on a galvanic element between zinc and iron as well as a passivation barrier consisting of zinc oxidation products [e.g., $Zn(OH)_2$].

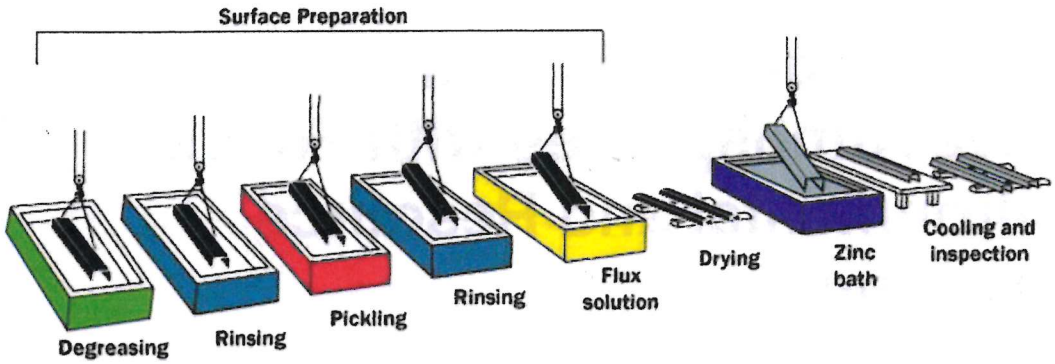

Figure 2. The process for hot-dip galvanizing steel requires a certain number of different bathing steps to ensure sufficient adhesion [3].

tween zinc and iron and under the influence of an electrolyte liquid (e.g., penetration of seawater into a coating defect), a galvanic element is created. This leads to a flow of electrons from zinc (anode), being the less noble metal, to iron (cathode). This way the steel is protected from corrosion. In addition, zinc oxidation products accumulate in the defect and act as a barrier. This principle is shown in Figure 1.

To achieve a sufficiently thick zinc coating of 50 to 150 µm on the steel components, the pure zinc is melted at a temperature of approximately 450°C. After a multistage surface-preparation process, the components are then dipped discontinuously into the molten zinc (hot-dip galvanizing, Figure 2) [2].

The chemicals required for this (e.g., hydrochloric acid, ammonium chloride, alkaline cleaning agents, and so on) must be disposed of as hazardous waste after use. The size of the component is limited by the size of the baths. Application by thermal spraying of liquid zinc is also common (spray galvanizing), but the quality of the zinc coating is less compared with hot dipping. Regardless of the application method, a considerable amount of energy is again used for the coating process, which adds to the already high amount of energy required to produce pure zinc.

In many cases, the galvanized parts are installed without a further coating and exposed to the weather. The reaction of the zinc surface with the environment causes zinc salts (zinc hydroxide, zinc chloride, and so on) to form, which are washed away by rain and, thus, enter the soil and groundwater (Figure 3).

The amount of zinc, that is removed this way, depends on the corrosiveness of the environment. The annual removal rate is only a few micrometers at low corrosion levels; the value increases to up to 25 µm/year in offshore areas [4]. In Germany, around 800 tons of zinc are released into the environment every year [5]. Although the toxic effect on humans is currently not considered a significant problem, the entry into water bodies is associated with serious damages (H400+H410 "very toxic to aquatic life with long lasting effect" [6]). However, these problems can be contained to a certain level by applying an additional organic coating on top of the zinc layer (duplex system).

Another alternative is the use of organic coatings containing zinc pigments (zinc primers). By embedding the zinc in a polymer matrix, it is largely protected from the environment and forms fewer water-soluble zinc salts. The polymer matrix itself consists in most cases of epoxy resin. The combination of good adhesion, moisture tolerance, chemical resistance, and compatibility with various substrates has made it the binder of choice for zinc primers right from their development in the 1950s [7]. At that time zinc primers were still mostly used for

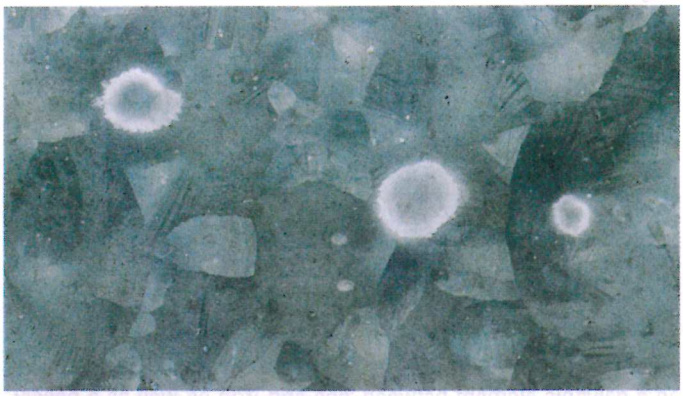

Figure 3. Formation of zinc salts on a hot-dip galvanized component.

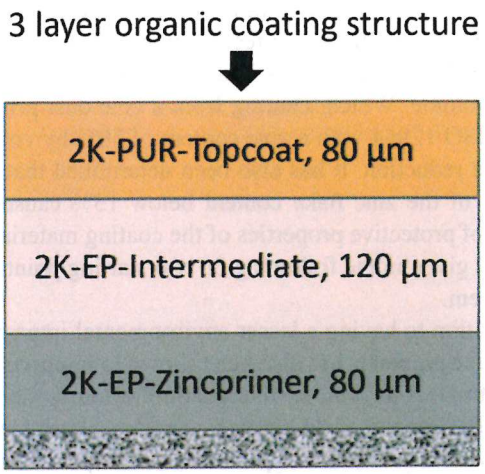

Figure 4. Typical coating structure for heavy-duty corrosion protection of blasted steel with specified overall dry-film thickness of 280 µm. PUR = polyurethane resin coating; EP = epoxy resin coating.

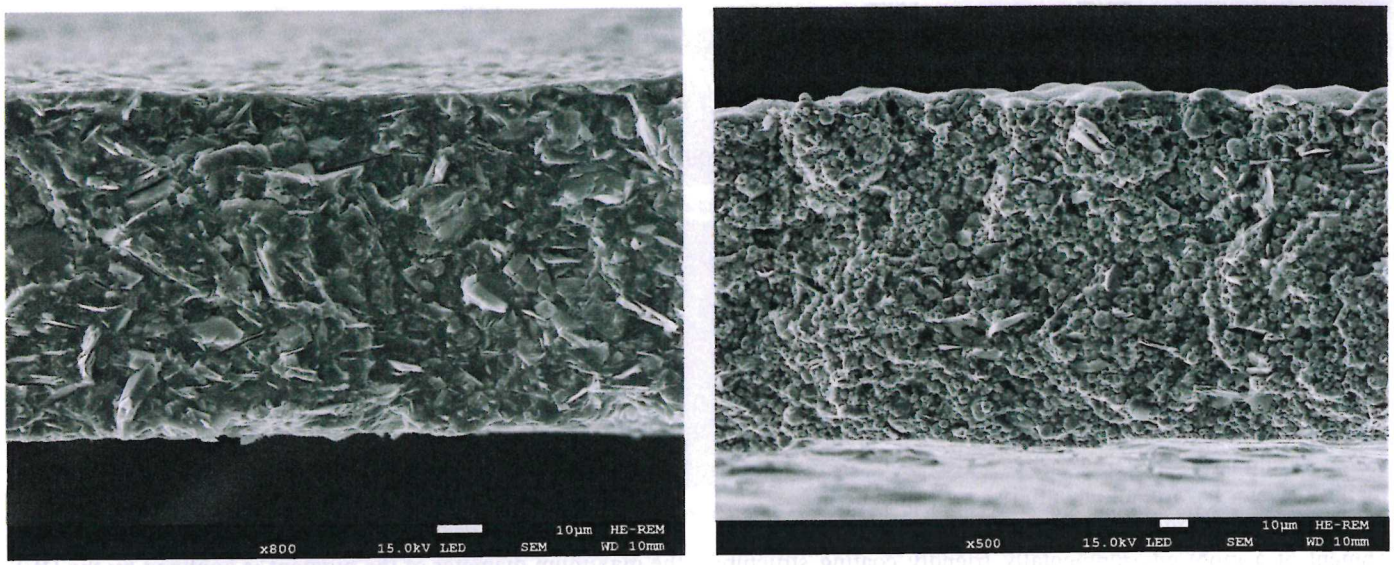

Figure 5. Scanning electron microscope images: (left) zinc flake primer and (right) zinc dust primer.

repairing defects in galvanized components. Over time, they have partially replaced classical galvanizing, because they save energy and labor and make the coating process more flexible. For a zinc primer to achieve a cathodic protection effect comparable to pure galvanizing, it must contain a certain amount of zinc pigment. This is necessary to ensure electrical conductivity (percolation) between the zinc pigments as well as between the zinc pigments and the steel substrate. This is crucial for the formation of the galvanic element. With a conventional zinc dust primer containing spherical zinc particles, the percolation threshold is reached when 80% of the dry paint's weight is zinc.

The application of a second or even a third coating is necessary on top of a zinc primer because of the lesser mechanical resistance compared with metallic zinc coatings. In addition to providing further protection against external influences such as moisture and salts, these subsequent layers also have a decorative function. To guarantee sufficient light and weather resistance, the coating system always ends with a polyurethane resin–based coating layer. Both epoxy and polyurethane binders achieve a high protective function because their drying process is characterized not only by the release of solvents, but also by a chemical curing process. For this purpose, a so-called hardener is stirred in immediately before the coating process (amine binder for epoxy resin, isocyanate binder for polyurethane resin), which starts a chemical reaction that leads to a duromer polymer matrix (two-pack or two-part coating material). The mechanical properties of the coating can be influenced by the mixing ratio between paint and hardener. The molecular weight of the binder molecules, the number of their functional cross-linking groups, and special additives can also influence the hardness or flexibility of the coating. A typical coating structure for heavy-duty corrosion protection is shown in Figure 4.

Even with three layers of paint, considerable energy savings can be achieved compared with 100-µm-thick hot-dip galvanizing. When the same surface is coated with a standard dry-film thickness (DFT) of 80 µm of the zinc primer and a zinc content of 80% by weight, 60% zinc can be saved, which no longer needs to be produced using the energy-intensive methods mentioned previously. In addition, the costly operation of the baths for surface pretreatment and zinc melting is eliminated. The size of the components to be protected is also no longer limited by the size of the zinc bath. It should be mentioned that the production of other paint components is also associated with high energy consumption. For example, the energy required to produce 1 kg of epoxy resin is often greater than that required to produce 1 kg of zinc. However, if the different densities of zinc (7.1 g/cm^3) and epoxy resin (~ 1.2 g/cm^3) are taken into account, it becomes clear that a much larger area can be coated with 1 kg of epoxy resin. The use of high-density materials should, therefore, be avoided to improve the yield of the coating. Another obvious advantage is the weight savings, which is particularly relevant when coating large offshore facilities such as oil rigs or wind farms.

Based on the possibilities already available today with zinc dust primers, several attempts have been made in recent years to further reduce the zinc content. However, a high-performance protective primer is not possible without zinc completely, because the use of active corrosion-protection methods such as cathodic protection is essential in environments with the greatest corrosive loads. The ISO 12944 standard clarifies the minimum requirements that a corrosion-protection coating must fulfil for a specific corrosivity category. The zinc primers [Zn(R)] prescribed for the offshore sector (corrosivity category CX) are defined as "Zinc dust–rich primer coating materials which pro-

Table 1. Comparison of the economic efficiency of zinc flake and zinc dust primers

Item	Zinc flake primer	Zinc dust primer
Yield (70 µm)	4.17 m^2/kg	2.63 m^2/kg
Zinc content	Approximately 197 g/kg	Approximately 750 g/kg
Price per m^2	Comparable	Comparable

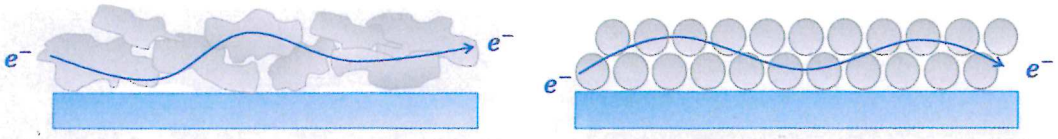

Figure 6. Influence of the particle shape on the required zinc pigment content for electrical conductivity.

vide a coating with a zinc dust content ≥80% (by mass) in the dry film" in Part 5 of the standard. In addition to energy savings through zinc reduction, there is the attempt to minimize the amount of VOC. The reduction of VOC plays an important role in environmental compatibility as well. Such ingredients are found in zinc primers in the form of solvents such as xylene, which should be used as little as possible because of their flammability, water hazard, and health risk when inhaled.

The aim of the work carried out was, therefore, the development of a more environmentally friendly coating structure for highly corrosive areas, with a minimal, yet effective, zinc content.

Reduction of Zinc by Use of Lamellar Particles

A significantly improved property profile can be achieved by using lamellar particle shapes or so-called zinc flakes (Figure 5). The reason for these improvements lies in the optimized space-bridging capacity of lamellar particles. In contrast to spherical pigments, a low zinc content is already sufficient to create an electrically conductive contact between the zinc pigments and the steel substrate. This leads to a lower density of the paint material and therefore a higher yield. Also, the lamellar particles act as a barrier inside the coating layer. Corrosive substances such as electrolytes or acid therefore take longer to diffuse through the coating and reach the steel substrate. The synergy between passive barrier and active cathodic protection is the basis of the excellent corrosion-protection effect of zinc flakes. In addition, the costs to produce zinc flakes are comparable to that of zinc dust (Table 1). The better space-bridging capacity of zinc flakes is also reflected in their larger particle diameter of 11 to 20 μm in comparison with 3 to 5 μm for zinc dust.

In the case of cathodic protection, it is important to consider the volume concentration of zinc, as a weight specification does not consider the space required by the other coating components. The threshold for the volume content of zinc at which a cathodic protective effect can be expected has been found to be at 10% to 15% by volume for zinc flake primers and 50% to 60% by volume for zinc dust primers through studies by the Fraunhofer IPA (Figure 6) [8]. The exact values depend on the actual particle size of the zinc pigments used as well as the homogeneity of their distribution in the polymer matrix. On one hand, bigger pigment diameters lead to better bridging and therefore a lower required zinc load. On the other hand, the maximum diameter of the pigment is confined by the DFT, which should always be twice as high as the diameter of the largest pigment to prevent pores in the coating. If the pigment diameter is too small, the required zinc load increases and the advantages over zinc dust are reduced. In addition, small lamellar pigments have a significant influence on the rheology of the coating material because of their large specific surface area. This leads to a higher viscosity of the coating material and makes a homogeneous distribution of the zinc flakes more difficult.

To determine the lowest possible zinc content, various epoxy zinc primers were produced (solvent based). A conventional zinc dust primer compliant with ISO 12944-5 with a zinc content of 80% by weight (in relation to the dry substance) served as a standard of comparison. In contrast, different zinc flake primers with varying zinc contents were tested. An overview of the different coating materials and the measured values of the average surface resistivity in accordance with EN 63140-2-3 are shown in Table 2. The surface resistivity should provide an indication of sufficient electrically conductive contact between

Table 2. Tested coating materials

Abbreviation	Type of zinc pigment	Zinc volume concentration (%)	Average surface resistivity (MΩ)
ZS49	Zinc dust	49	206
ZF3.4	Zinc flakes	3.4	>40,000
ZF7.5	Zinc flakes	7.5	11,033
ZF10.1	Zinc flakes	10.1	98
ZF13.8	Zinc flakes	13.8	1.3

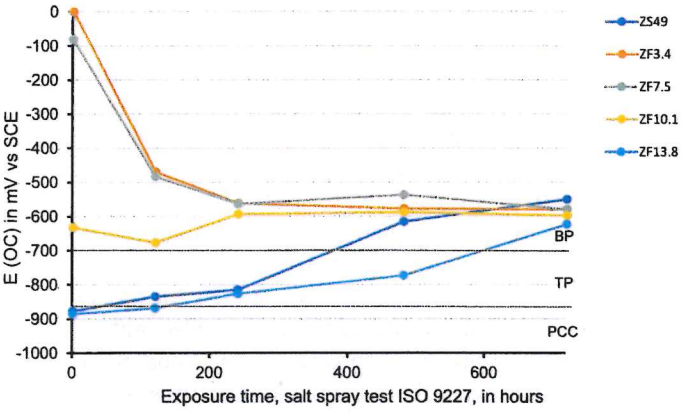

Figure 7. Determination of the open-circuit (OC) potential during exposure to salt spray. SCE = saturated calomel electrode; BP = barrier phase; TP = transition phase; PCC = phase of cathodic corrosion protection. See Table 2 for definitions of the coating-material abbreviations.

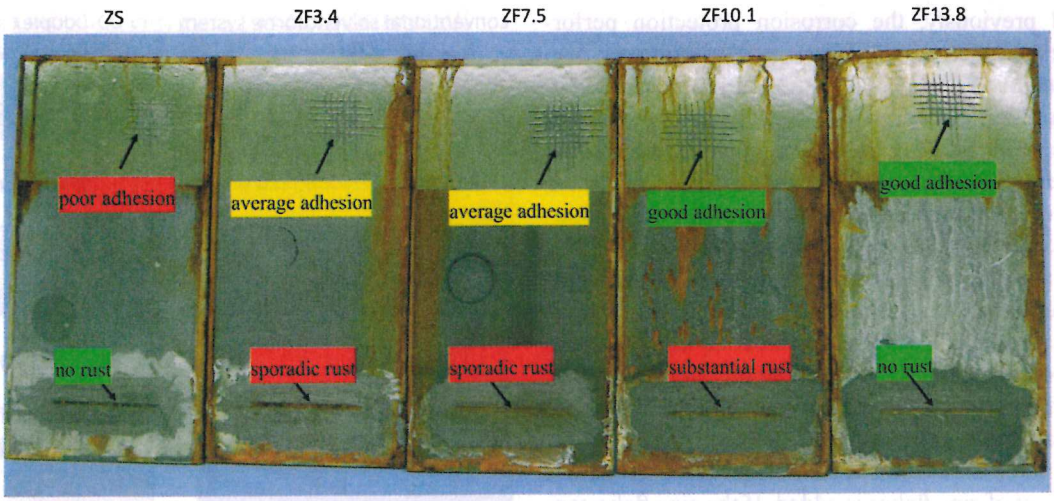

Figure 8. Sample panels after 1,000 hours of neutral salt spray test (ISO 9227). Panels were blasted steel Sa 2 1/2 (ISO 8501-1) 3 mm in thickness; dry-film thickness = 80 µm of primer + 80 µm of topcoat. See Table 2 for definitions of the coating-material abbreviations.

the zinc pigments and thus an effective cathodic protection. The tests showed that the surface resistance of the zinc dust primer (ZS) is already undercut by using a zinc flake primer with 10.1% zinc by volume (ZF10.1).

To prove a cathodic protection effect, the open-circuit potential was determined. The course of the potential was recorded against a saturated calomel electrode immediately after and until 720 hours of exposure to salt spray in accordance with ISO 9227. Above −700 mV, only a passive barrier effect is detectable for the coating (barrier phase). A value below −700 mV is considered an indication of a partial cathodic protective effect (transition phase). Below −860 mV, the protective effect is said to be fully developed (phase of cathodic corrosion protection) [9]. Despite a surface resistance comparable to zinc dust, no cathodic protective effect can be demonstrated for ZF10.1. The potential here is at the border of the transition phase. With a zinc flake content of 13.8%, however, the potential is already lower than that of zinc dust (Figure 7).

The artificial weathering in a salt spray chamber was carried out up to 1,000 hours. Following the weathering test, a cross-cut test (ISO 2409) was carried out to check the adhesion of the coating to the substrate as well as intercoat adhesion to a polyurethane top coat (solventborne), which had been applied to the upper third of the test panel. In addition, the corrosion of the steel substrate was examined in the area of an artificial scratch injury (Figure 8).

According to the measurement of the open-circuit potential, the best protective effect is achieved with the zinc dust primer and the zinc flake primer at 13.8% by volume. In both cases no rust could be detected beneath the coating. A closer look at the adhesion properties, however, reveals noticeable advantages for the zinc flake coating. Because extremely high zinc contents are required when using zinc dust, there is little room for a continuous binder phase. The permeability of the film is increased, and the adhesion properties deteriorate. This aspect highlights the advantages of zinc flake primers, which combine good adhesion with cathodic protection.

Normally, adhesion of zinc primers can only be achieved by anchoring the coating on rough surfaces, which is why blasted steel is prescribed in ISO 12944. Based on the findings of the

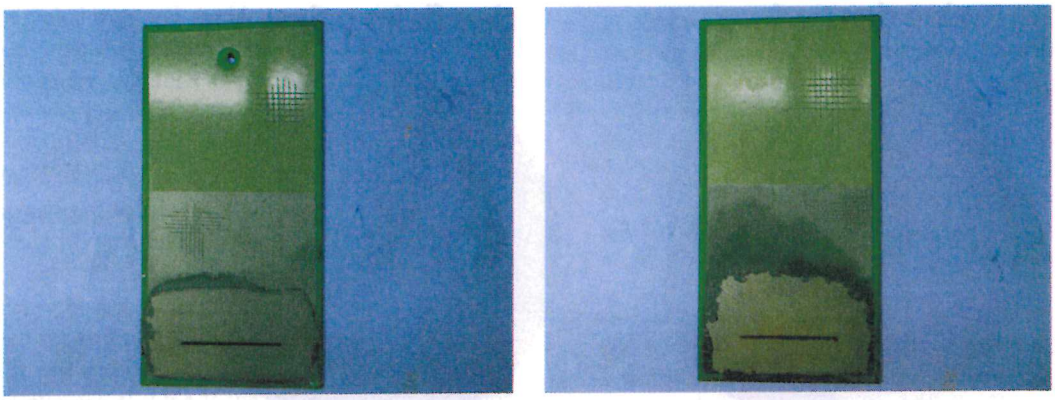

Figure 9. Zinc flake primer (80 µm) on smooth steel, half-coated with polyurethane topcoat (80 µm), (left) after 1,440-hour condensation test (ISO 6270-2) and (right) after 720-hour salt spray test (ISO 9227).

tests described previously, the corrosion protection performance of a zinc flake primer with 14% zinc by volume was also tested on smooth steel. After 720 hours of salt spray and 1,440 hours of condensation testing, neither rusting nor loss of adhesion was recognizable (see Figure 9). The sandblasting process step could therefore be eliminated. Because a certain minimum thickness of steel is required for the blasting process, also, thinner steel walls could be installed, thus saving weight and material costs. Because of the high roughness depth of blasted steel surfaces of 60 µm or more, a minimum DFT of 80 µm is mandatory to sufficiently cover the steel tips of the surface structure. This problem does not apply to smooth substrates with roughness depths up to 25 µm, which is why a DFT <80 µm could theoretically be possible as well.

It must be pointed out that these advantages are only valid if a sufficient amount of zinc flakes is added. If the zinc flake content is too low, the protective properties seem to be worse than those of a conventional zinc dust primer. At least for the tested coating material, the recommended content is above 13% by volume. The use of a different binder or pigment composition could, on the other hand, lead to a different recommended zinc flake content, which is why the range of 10% to 15% by volume recommended by the Fraunhofer IPA acts as a good indicator. However, the exact amount of zinc has to be determined with experiments individually.

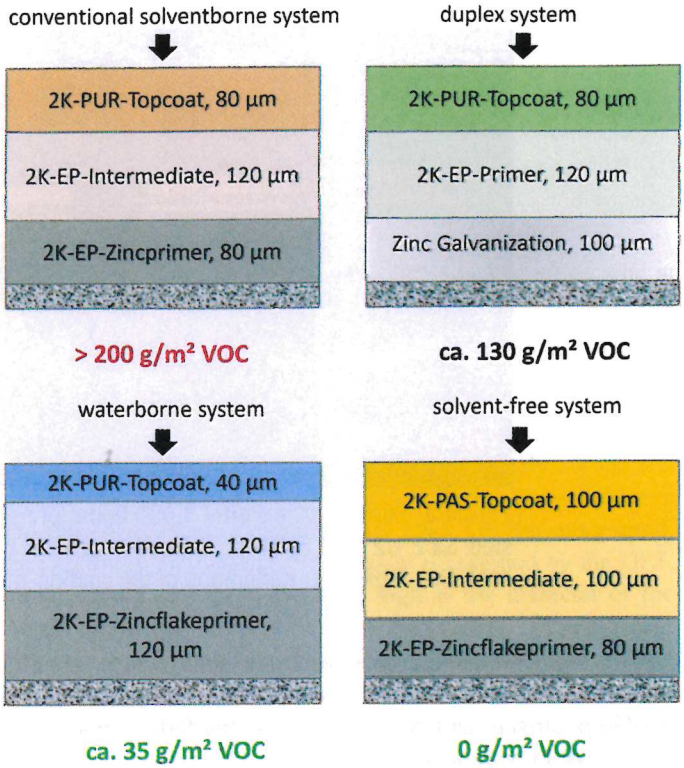

Figure 10. Volatile organic compound (VOC) content of various coating structures for the offshore sector. Individual dry-film thickness for each layer is indicated. PUR = polyurethane resin coating; EP = epoxy resin coating.

Use of Low-VOC Binders

The tested coating materials are already variants with a reduced solvent content (high-solid materials). An equal corrosion protection can be achieved with waterborne epoxy or polyurethane dispersion binders. Although waterborne coating materials also require a proportion of organic solvents to ensure optimum drying properties, the VOC content can be reduced from 15% to 4% by weight compared with solvent-based high-solid materials. In the case of waterborne polyurethane coatings, the disadvantage lies in a maximum achievable DFT of 40 µm. As already shown in Figure 4, the overall DFT of a coating system suitable for the offshore sector consisting of primer, intermediate, and top coat needs to be at least 280 µm. The lesser thickness value therefore must be evened out by the zinc primer or intermediate coating. Also, the DFT of the zinc primer is limited to 80 µm, provided it is based on zinc dust. Because of the low binder content of zinc dust paints, there is otherwise a risk of cracking.

Another interesting type of binder for use in weather-resistant top coats is polyaspartics. Like polyurethanes, these binders must be hardened with isocyanates. Unlike polyurethanes, they are VOC free but inherit comparable protective properties. Another advantage is the short drying time, which allows short-

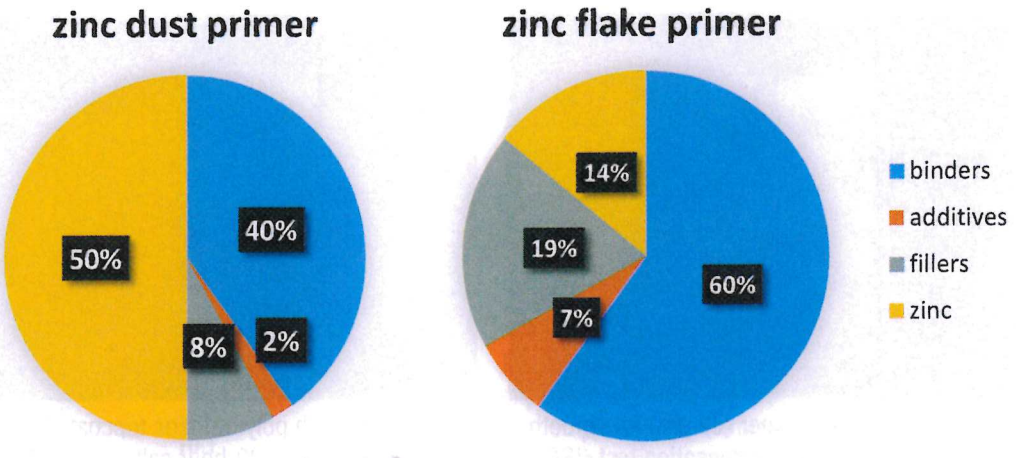

Figure 11. Differences in coating composition by volume.

Embodied energy of paint components [9]

Zinc	→	51 MJ/kg	→	362.1 GJ/m³ (Density 7.1 g/cm³)
Binder/Additives	→	120 MJ/kg	→	144.0 GJ/m³ (Density 1.2 g/cm³)
Fillers	→	7 MJ/kg	→	24.5 GJ/m³ (Density 3.5 g/cm³)

Embodied energy of zinc dust paint

$$(362 \times 0.5 + 144 \times 0.42 + 24.5 \times 0.08)\frac{\text{GJ}}{\text{m}^3} = \sim \mathbf{243}\,\frac{\mathbf{GJ}}{\mathbf{m^3}}$$

Embodied energy of zinc dust paint

$$(362 \times 0.14 + 144 \times 0.67 + 24.5 \times 0.19)\frac{\text{GJ}}{\text{m}^3} = \sim \mathbf{152}\,\frac{\mathbf{GJ}}{\mathbf{m^3}}$$

Figure 12. Comparative calculation of the embodied energy required for the raw materials contained in the respective coating compositions.

er cycle times and therefore a higher production rate. In addition, high layer thicknesses can be applied in a single spraying operation. In the case of epoxy binders, solvent-free product types have been available for quite some time as well. In combination with polyaspartics, a mostly solvent-free coating system can be formulated in which only a small amount of VOC is brought in by certain additives. A comparison of the VOC content of different coating structures for the offshore sector (according to ISO 12944-9) is shown in Figure 10.

Conclusion and Outlook

Considering the possibilities that are available today, the formulation of a zinc primer with noticeably lower zinc content and almost no VOC is already possible. Not only can the environmental impact be significantly reduced, the use of the right amount of zinc flake pigments can also improve protective properties. In Figure 11 the different coating compositions between an exemplary zinc dust and zinc flake primer are shown. Figure 12 shows a comparative calculation of the embodied energy required for the raw materials contained in the respective coating compositions [10]. Embodied energy refers to the energy required for all aspects of the production of a raw material (extraction, processing, transportation, and so on). Because a large number of different raw materials are used in paint formulations, this is only a rough estimate. Also, aspects such as the proportion of recycled material could alter the values.

A switch from zinc dust to zinc flakes can therefore be expected to result in energy savings of approximately 35% to 40% related to the raw materials. A state-of-the-art zinc primer formulation also opens the door for further adjustments. When using zinc dust, 90% of the coating material consists of zinc and binder. This leaves only a little room for additional additives or pigments that could contribute to better corrosion protection. With the use of zinc flakes, it is possible to both increase the amount of binder, which results in better adhesion, and as well leave more space for additional ingredients.

There are different approaches to further reduce the zinc content in zinc flake paints. One is the implementation of electrically conductive pigments (e.g., carbon black, carbon nano tubes) that can act as a bridge between the zinc pigments as well as between zinc and steel. Another approach is the combination of different metals in the form of zinc alloys. The use of less noble magnesium results in a decrease of the open circuit potential and therefore an improved short-term protection. With less noble tin the potential can be raised, which leads to a better long-term protection. Both magnesium and tin do not form any reaction products that are hazardous to the environment. However, the further reduction of zinc is likely limited to a few percentage units. The biggest leap forward in development to date remains the change in particle morphology from zinc dust to zinc flakes. Currently, the definition of a zinc primer in ISO 12944 is still limited to zinc dust–rich primers [Zn(R)]. An amendment to the standard and an extension of the definition to generally include cathodically effective coatings with comparable protective effect would further clear the way for more innovative products and make an important contribution to environmental protection.

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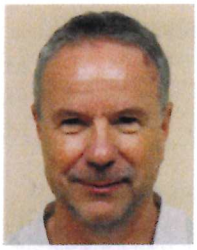

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