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Corrosion resistance of steel coated with different coating systems, containing HAA-polyester powder coating as a top layer

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Abstract

This study aimed to assess the impact of surface geometry, preparation, and coating procedures on the corrosion behavior of HAA-polyester powder coated steel components in a salt spray test. The coating process of the steel parts involved degreasing, Zn spray coating, an epoxy powder primer, and a polyester powder topcoat. Results highlighted the crucial role of surface geometry, with flat surfaces showing superior corrosion resistance compared to welded joints, sharp edges, or hollow cylinders. For standard flat panels, no red rust was observed for over 600 hours in the salt chamber, meeting ISO 12944-6 requirements. However, non-standard panels with complex geometries showed rust much earlier, particularly after 108 hours in welded zones and sharp corners when an epoxy primer was not used. Applying the primer extended corrosion resistance by 2-3 times. Hollow cylinders developed rust after 240 hours due to insufficient coating thickness inside the cylinders. Crevices remained vulnerable, as the coating did not reach inner parts. Applying powder to individual components before assembly, and using brush painting on critical areas, such as welded joints and sharp corners, are strongly suggested to improve protection. Additionally, the evaluated polyester powder coatings exhibited outstanding mechanical strength and camouflage capabilities, making them highly suitable for military applications.

Keywords: HAA (β-hydroxyalkyl-amide) polyester, powder coating, salt spray chamber, steel ball impact test, diffuse reflectivity

1. INTRODUCTION

Powder coating, in the context of surface finishing, refers to a type of finishing process where a fine powder material, composed of resin, pigments, and additives, is applied onto a substrate. Unlike conventional liquid coatings, powder coatings do not require solvents, and yet, employ most of the polymer types used in the conventional solvent borne and waterborne coatings (Du et al. 2016).

The application of powder coatings onto substrates can be achieved through various techniques including electrostatic spraying, fluidized bed, electrostatic fluidized bed, and flame spray. Among these methods, electrostatic spraying is the most prevalent in the realm of metal finishing. It involves the propulsion of dry powder particles using compressed air through a specialized spray gun, resulting in their acquisition of an electrical charge (Ayrilmis 2022). Due to the insulating properties of powder particles, they retain this charge and subsequently adhere to the workpiece. The surplus powder that is not adhered is collected, allowing for the efficient reuse of over 98% of the overall material (Farshchi & Gedan-Smolka 2020).

The extensive advancement of powder coatings originated in the 1960s, driven by the imperative to minimize the use of solvents and to lower energy requirements in organic coatings. This development aimed at fostering environmental friendliness and mitigating the potential for air and water pollution. In addition, powder coatings offer a range of significant benefits, including enhanced resistance to corrosion, chemicals, and UV radiation. Powder coatings have proved to be very environmentally friendly

alternative to conventional liquid coatings. As there are no solvents involved, their application generates almost no emissions (Goldschmidt & Streitberger 2018; Mischke 2019). Furthermore, they exhibit increased flexibility and hardness, rendering them highly desirable for a multitude of industrial applications (Du et al. 2016). In accordance with solvent-borne coating systems, the corrosion resistance of powder coating systems depends upon factors such as powder composition, coating layer thickness, the presence of a primer layer, and the applied pretreatment process. Conventional pretreatment procedures encompass mechanical cleaning methods, such as sand and air blasting, as well as chemical processes, including conversion coating, predominantly involving the application of a phosphate layer (Farshchi & Gedan-Smolka 2020).

The primary objective of this study is to scrutinize the impact of diverse pretreatment processes and various coating systems (with or without primer, for instance) on the behavior of powder coating systems. The sole output parameter assessed in this investigation was corrosion resistance, recognized as a pivotal requirement in the context of military equipment applications. The monitoring of this parameter was conducted within a salt spray chamber. In the course of all conducted experiments, the top layer on all the specimens comprised HAA (β-hydroxyalkyl-amide) - polyester powder coating, while carbon steel was employed as a substrate material. Diverging from prevalent scientific and standard investigations predominantly focused on flat plates, our study encompassed a broad spectrum of sample types. This deliberate variation aimed to evaluate the responsiveness of the coating system within practical applications, encompassing configurations such as sharp and rounded edges, welded joints, and hollow components.

In addition to evaluating their corrosion resistance, the mechanical durability of the polyester powder coating - specifically its resistance to steel ball impacts - and its camouflage effectiveness were also examined. These tests were conducted with the objective of assessing the potential future use of this type of organic coating in military applications.

2. EXPERIMENTAL PART

The substrate for powder coating was carbon steel (EN 10025, S 235 JR). The steel pieces were shaped into one of the following geometries: flat plates, pieces with sharp or rounded edges, pieces with rolled edges and corners, or pieces with welded edges, welded metallic cylinder (hinge) or welded hollow cylinder. Metal active gas (MAG) welding was applied, using pure CO_2 as active shielding gas, and wire electrode containing (C 0.076, S 0.022, Mn 1.49, and Si 0.83 mass. %).

The samples were degreased by spraying industrial degreaser with trade name Supermafrasol (Mafra, Italia) at room temperature, enabling 5-minute contact with the metal surface, subsequent thorough water rinsing and drying of the parts. The weld was cleaned using a Supermafrasol water solution at a 1:50 ratio, followed by thorough rinsing with pure water.

Zinc coating (aerosol, 98% pure Zn with epoxy ester resin as chemical base, Berner, Germany), was applied by spraying 50 – 80 μm thick layer.

Primer powder coating was epoxy based coating with high Zn content (CPC 60 Zn primer, Helios, Slovenia), and the top coat was polyester powder coating with HAA (β-hydroxyalkyl-amide) as a cross-linker. Both coatings were electrostatic sprayed on steel samples, using a Plastamat gun with 100 kV voltage source. The next step was curing the coatings at 180 °C for 15 min, following the coating manufacturer's specifications. The thickness of the primer epoxy coating ranged between 90 and 100 μm, while the thickness of the top coating was between 100 – 140 μm. The top coating was applied after the baking process of the primer coating was finished.

Metal and powder coating thickness was determined according to ISO 2808 using an electromagnetic measuring device (Elcometer 456, Manchester, United Kingdom).

In order to elucidate the effect of each preparation step on the coating system performance, for reference samples some preparation steps were omitted. In this manner, there were samples where degreasing, Zn spray, and/or epoxy primer coating step were not applied prior to the top polyester coating application. The sample designations are listed in Table 1. Each designation is related to different preparation and/or coating process. For each designation, two samples were prepared, in order to ensure reproducibility of the results.

The neutral salt spray corrosion test was carried out according to the ISO 9227:2014 standard in Q-fog CCT 600 chamber (Q-LAB, USA). The test was carried out at a temperature of 35 ± 2 °C in a salt spray environment with a concentration of 50 ± 5 g/L, with continuous moisturization of the samples surface. The pH value of the sprayed solution was within the range of 6.50 – 7.20. The samples were tested over a period of 600 h with intermediate evaluation after every 24 hours.

During the exposure test in salt chamber, the samples were evaluated according to ISO 4628-2, 4628-4, and 4628-5 for the presence of blistering, cracking and flaking of the coating, respectively. None of these three coating deterioration forms were found on any of the parts during the whole testing period. Additionally, the coating adhesion test after corrosion testing was performed at the flat

Table 1. Sample designation, denoting various part geometries and preparation steps for the coating process.

No.	Part geometry	Preparation and coating process
1	plate 150 x 150 mm	polyester coating (PC)
2		degreasing + PC
3		degreasing + rounded edges + PC
4		degreasing + rounded edges + epoxy primer + PC
5	bent with rolled edges and corners	polyester coating (PC)
6		degreasing + PC
7		degreasing + rounded edges + PC
8		degreasing + rounded edges + epoxy primer + PC
9	bent plate with rolled and welded edges and corners	polyester coating (PC)
10		degreasing + PC
11		degreasing + rounded edges + PC
12		degreasing + rounded edges + Zn spray + PC
13		degreasing + rounded edges + + Zn spray + epoxy primer + PC
14	bent plate with rolled and welded edges and corners, and welded hollow cylinders 21.3 mm in diameter and wall thickness of 1.8 mm	polyester coating (PC)
15		degreasing + PC
16		degreasing + rounded edges + PC
17		degreasing + rounded edges + Zn spray + PC
18		degreasing + rounded edges + + Zn spray + epoxy primer + PC
19	bent plate with rolled and welded edges and corners, and welded hinge, 14 mm diameter	polyester coating (PC)
20		degreasing + PC
21		degreasing + rounded edges + PC
22		degreasing + rounded edges + Zn spray + PC
23		degreasing + rounded edges + Zn spray + epoxy primer + PC

surfaces, using the cut grid method in accordance with ISO 2409 (ISO 2020), and the grade "0" was determined for all samples, denoting that the edges of the cuts were completely smooth and none of the squares of the cut lattice was detached.

The impact resistance of the coated samples was evaluated using the Erichsen 273 D device. In each test cycle, 500 steel balls, each with a diameter of 10 mm and a Rockwell hardness of 60–60 HRC, were dropped onto the coated surface from a height of 400 mm. Following each cycle, the coating was inspected for signs of deterioration, such as cracking or delamination. If no visible damage was observed, another cycle of 500 balls was conducted. This procedure was repeated until evidence of coating degradation became apparent.

The spectrophotometric measurements were conducted using the UV/VIS/NIR spectrophotometer Shimadzu UV 3600+, with an integrated sphere ISR-603. The reflectance was measured in the visible and near-infrared area of the electromagnetic spectrum (650-1000 nm). The gloss measurements were conducted using glossmeter Elcometer 480T (United Kingdom).

3. RESULTS AND DISCUSSION

3.1. Evaluation of corrosion behavior of the test panels in the salt spray test

The first criterium for the corrosion resistance of the samples was the exposure time in the salt chamber prior to the appearance of red rust that could be visually observed. Furthermore, the samples were evaluated according to the ASTM D 610 for the degree of rusting. According to the standard, the degree of rusting is evaluated using a zero to ten scale, based on the percentage of visible surface rust. For example, rust grade 10 denotes that ≤ 0.01 % of the surface is covered with rust; grade 5 denotes that 1 – 3 % of the surface is covered by rust, and grade 0 indicates that more than 50 % of the surface is covered with rust. The distribution of the rust is classified as spot rust, general rust, pinpoint rust or hybrid rust (ASTM 2019).

The samples that withstood the shortest time (108 hours) in the salt chamber prior to the appearance of rust, were the ones where the coating system did not contain the Zn epoxy primer, and the sample edges were joint by welding, i.e. the samples 9 – 12 in the Table 1. The red rust appeared in the corners and in the welded zone (Fig. 1). Interestingly, the Zn coating was applied by spraying Zn aerosol on the welded zones on the sample 12, but it did not prevent the occurrence of red rust. On
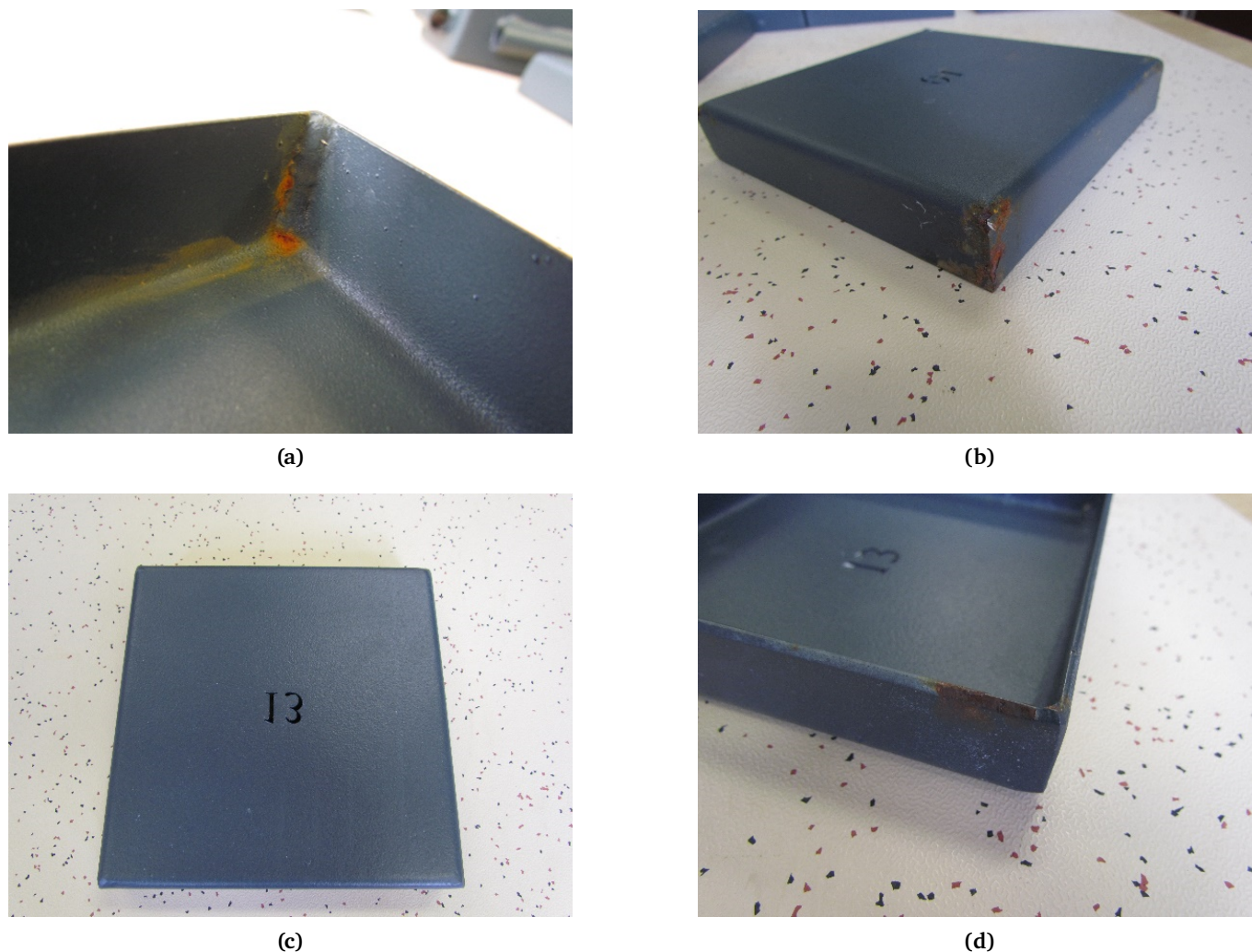
(a)

(b)

(c)

(d)

Figure 1. Two upper figures (a and b): sample No. 12 appearance after 108 hours in the salt chamber. The degree of rust is marked with 3 on the welded zone and 5 at the edge. Two lower figures (c and d): sample No. 13 after 600 hours of exposure.

the contrary, when the Zn primer layer is added to the coating system (sample No. 13), the part may withstand 600 hours in the salt chamber with the rust occurrence only on the part edges, as also presented in Fig. 1. To conclude, this group of samples unambiguously shows that the epoxy primer increases the expected life of the parts for at least 2 – 3 times (Pélissier & Thierry 2020).

The second group of the testing samples consists of the samples 14 – 18, that contain welded edges, but also the two welded hollow cylinder (Fig. 2). The red rust occurrence was spotted after 240 h exposure of these samples to the salt chamber. However, the flat surfaces remained intact, and the corrosion was observed only on the welded zones and at the inner surface of the cylinder. It is clear that the powder coating may not be properly applied at the inner cylinder surface, indicating that longer application time should be prescribed when such parts require coating. The influence of different preparation steps may be easily differentiated in this contingent of samples:

the samples with no preparation and with degreasing only (designation 14, 15) lasted only 156 h in the salt chamber prior to the appearance of visible corrosion, while the samples with Zn spray layer and with epoxy primer layer, lasted until 240 hour of exposure.

The third tested group belongs to the samples that do not contain welded parts, but do contain rolled edges and corners, i.e., the samples No. 5 – 8. The only regions where the rust occurred at these samples, were sharp edges and sharp corners (Fig. 3). The first reason for this occurrence could be the fact that the powder coating did not cover the sharp corners with sufficient thickness. The second reason is the fact that the sharp corners do not dry so easily as the other part surfaces, i.e., always contain higher amount of the electrolyte from the salt chamber, that is in contact with the coating. This corrosion type may be characterized as crevice corrosion (Wang 2004). Although the rust fingerprint is very similar for all the 4 samples, the time after which this corrosion occurs, dif-
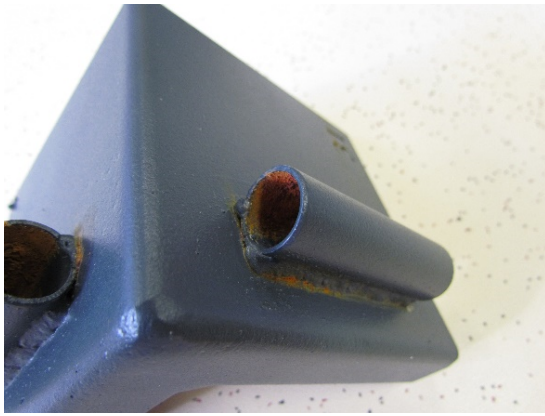
Figure 2. Sample No. 18 appearance after 240 hours in the salt chamber. The degree of rust is marked with 0 in the pipe (more than 50% of total surface corroded), and 6 at the welded zone.
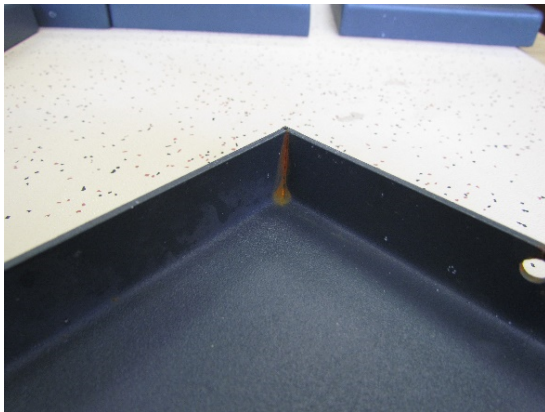

Figure 3. Sample No. 6 appearance after 264 hours in the salt chamber. The degree of rust is evaluated with 9, both in the sharp corners and at the edges.

fers drastically. For example, for the samples 6 and 8 the rust occurred after 264 hours, for the sample 5 after 348 hours, while the sample 7 lasted the full testing period of 600 hours. One could conclude that the crevice corrosion will definitely start, independently on the preparation and coating process applied, and so the best way of avoiding it, is the avoidance of parts with sharp corners similar to those presented in Fig. 3.

The samples with the welded hinge form a very compact group, in sense that all five samples (No. 19 to No. 23) lasted identical time in the salt chamber (348 hours) and that on all of the samples only the crevice corrosion is visible, between the hinge and the flat metal surface (Fig. 4), as well as the corrosion in the welded zone that is of significantly lower intensity. It is important to stress out that the flat surfaces, the welded corners and the rolled edges remained intact and free from rust, as it is also depicted in Fig. 4. It is obvious that the surface geometry plays significantly higher role in the protective action of the coating system, than the preparation process: the powder coating system applied may not protect the parts from crevice corrosion, but on the other side successfully protects the flat and rolled surfaces, independently on the preparation process applied, even independently on the fact whether the epoxy primer is present or not.

The samples that lasted 600 hours in the salt spray chamber, were flat plates (No. 1 – 4). The red rust was not observed at the flat surfaces at any of the sample, however it may be observed at the edges. The visual examination of the samples clearly chows that as the sample number increases from 1 to 4, the rust degree according to the ASTM standard increases from 4 to 7, i.e. each consecutive preparation step (degreasing, rounding of the edges and epoxy primer), improves the corrosion resistance at the edges.

It is interesting to speculate whether the application of the complete preparation and coating process usually applied in the industry (degreasing + rounded edges + Zn spray + epoxy primer + polyester coating) enables significantly higher corrosion resistance in comparison to the cases where some of the steps is omitted. It is important to note that this is true only for the flat plates, as well as for the sample 13 (plate with rolled and welded edges and corners), that also lasted for 600 h in the salt cham-
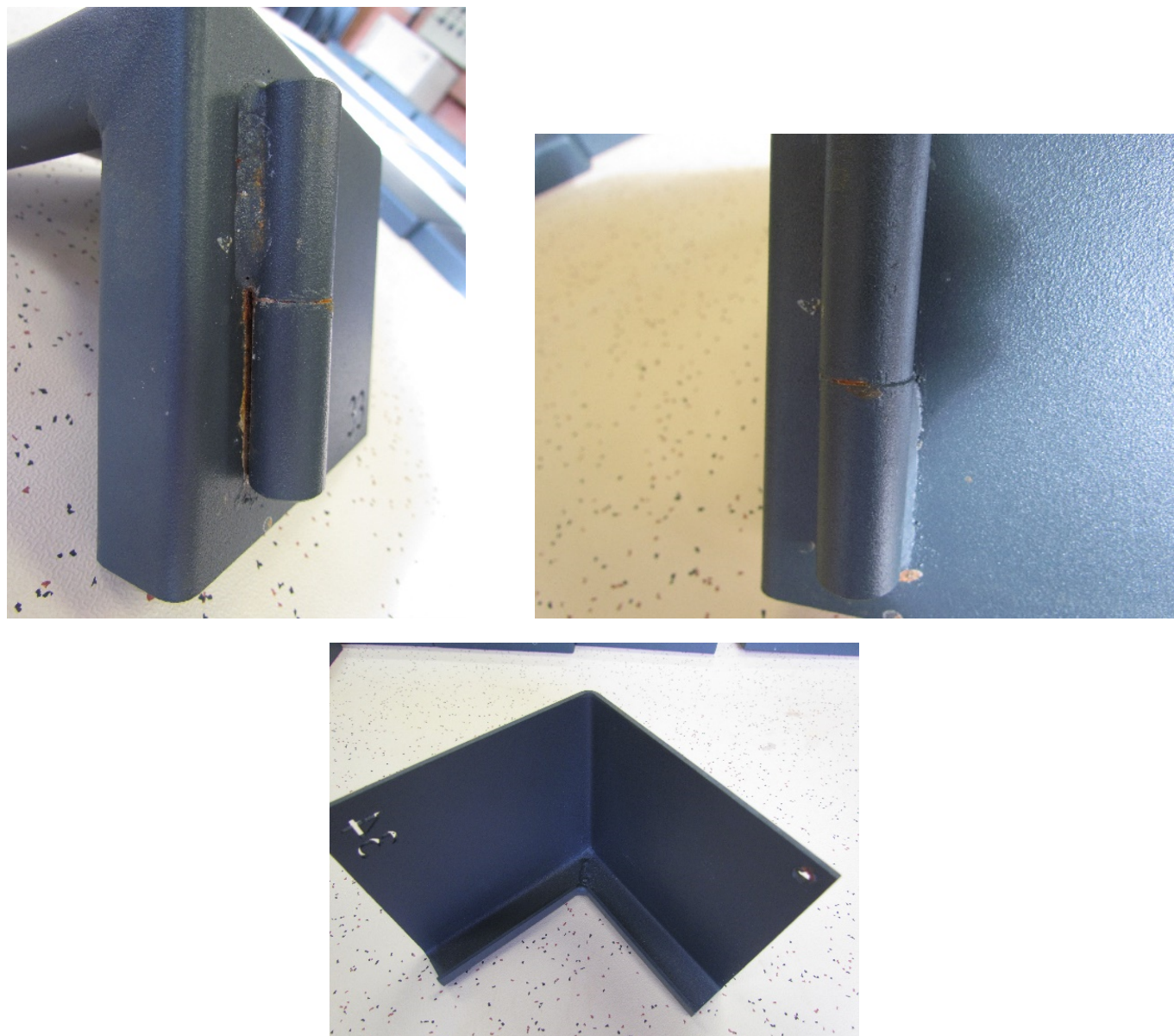
Figure 4. Sample No. 20 appearance after 348 hours in the salt chamber. The degree of rust is evaluated with 8 in the welded zone, and with mark 3 at the hinge (in the crevice).

ber, with minimal corrosion appearance. For all other geometries, the comparison of the salt chamber test results for the samples with and without Zn spray, and with and without epoxy primer, brings to the conclusion that these two intermediate layers do not bring much improvement in the corrosion protection of the examined parts. The ISO standard 12944-6 (ISO 2018) defines the duration of the neutral salt spray test for paint systems applied to carbon steel, for various atmospheric corrosivity categories (C1 to C5) according to the ISO standard ISO 12944 -2 (ISO 2017). If we consider, for example, the corrosivity category C3 (medium category that includes urban and industrial atmospheres, with moderate sulfur dioxide pollution, and coastal areas with low salinity), the prescribed neu-

tral salt test duration ranges from 120 h (for low expected durability), through 480 h (for high expected durability) to 720 h (for very high expected durability of the coating system). The comparison of these required test durations with the test duration applied in this investigation, indicates that the applied coating process (degreasing + Zn spray + epoxy primer + polyester top coat) in this work, falls between the duration limits for high and very high expected durability in moderate atmosphere, since we showed that the red rust does not appear after 600 hours at all flat surfaces of all tested parts. One should bear also in mind that the test was terminated at 600 hours not due to the appearance of rust, but due to the technical limitations of the salt chamber.

However, on the contrary, previous figures clearly show that as soon as the geometry differs from the flat surface, and this includes welded joints, sharp corners and hollow cylinders, the rust appears at significantly shorter exposure times, classifying the coating systems into low durability (120 h) and medium durability (240 h) categories (ISO 2018).

3.2. Resistance to steel balls impact and IR reflection of the test sample

To assess the mechanical properties and reflectivity of the sole powder coating, both attributes were evaluated on ample No. 1 from Table 1, which consists of a steel plate with a polyester coating applied directly, without any intermediary layers. The mechanical impact resistance testing revealed that the polyester powder coating exhibited no signs of damage until the impact of 9000 dropped steel balls. In contrast, a previously tested sample of military-grade polyurethane camouflage coating from our earlier research (Lazić 2016; Lazić, Simić, Samolov, & Jovanović 2017) demonstrated failure after exposure to 5000 steel balls. These findings indicate a substantial enhancement in impact resistance of the polyester powder coating compared to conventional solvent-based polyurethane coatings.
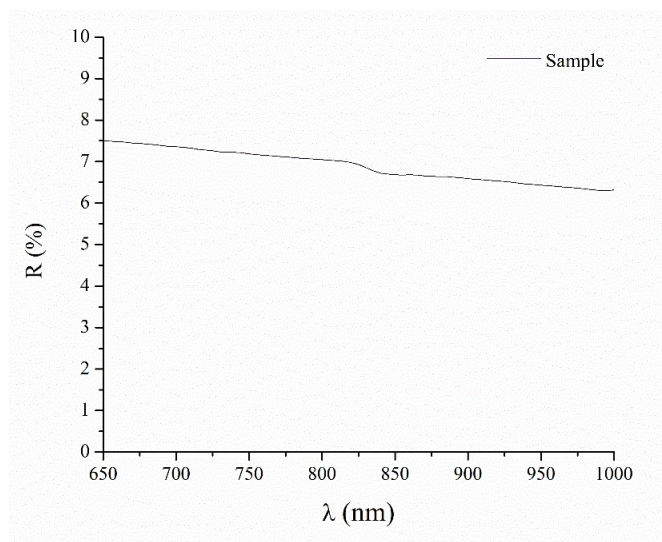

Figure 5. Dependency graph for diffuse reflection in the range between 650 and 1000 nm, for HAA-polyester powder coating.

In addition to the physical and mechanical properties of coatings, the camouflage reflection and adsorption characteristics in the visible and near-infrared regions of the electromagnetic spectrum are of paramount importance for military applications (Simić et al. 2018). It is well known that by suppressing the diffuse reflectivity, the detection and identification of objects in the background becomes difficult. Figure 5 illustrates the results

of diffuse reflectance for the HAA polyester powder coating.

According to Figure 5, the diffuse reflectivity of the powder polyester coating is well balanced across the examined range of visible and near-infrared spectra. The obtained diffuse reflectivity values range from 7.5% to 6.2%, which is comparable to natural diffuse reflection values (for example, the reflectivity of black soil) (Yin et al. 2023). In contrast, S. Bhargava et al. (Bhargava et al. 2015) investigated the use of polyurethane coatings for civil engineering applications, where the coating doped with TiO_2 particles exhibited a significantly higher diffuse reflectivity (R > 90%) in the range from 450 to 750 nm. These high reflectivity values pose a potential risk for the camouflage of vehicles and objects. Therefore, it can be concluded that the coating applied in this work provides superior camouflage properties due to its low diffuse reflectivity, making it more suitable for such applications.

4. CONCLUSION

The goal of this study was to investigate the effect of surface geometry and the preparation and coating procedure, on the corrosion behavior of various steel parts in the salt spray test. The following preparation and coating process was applied: degreasing + Zn spray coating + epoxy powder primer + polyester powder top coating. The results emphasized the critical role of surface geometry in determining the effectiveness of the protective coating system. Samples with flat surfaces exhibited significantly better corrosion resistance compared to those with welded joints, sharp corners, or hollow cylinders.

The ISO 12944-6 standard defines that the carbon panels used for the coating testing should be plates with minimum size 150 mm × 75 mm, with no curves. The results in this work show that, in case the test panels are standard plates, the coating system applied prevents the occurrence of red rust in the salt chamber for a period longer than 600 h.

However, the red rust occurs in significantly shorter time when nonstandard panels are exposed to the salt environment. The rust was observed after 108 h in welded zones and sharp corners, when epoxy primer was not applied prior to the top layer, thus pointing to the inevitable need for usage of the primer layer in all cases. The presence of an epoxy primer in the coating system demonstrated a substantial increase in corrosion resistance, prolonging the time to red rust appearance by at least 2-3 times compared to samples without the primer. Furthermore, the rust was observed after 240 h in hollow cylinders, indicating that the powder did not reach successfully the inner part of the cylinders, and the coating thickness was not sufficient. In order to avoid the corrosion in

the follow parts in the future, the powder should be more carefully directed to the entire surface of the cylinder, and should be sprayed for a longer time. At the rolled edges and corners the red rust occurred in various exposure periods (between 264 and 348 h), but significantly shorter than at the flat surfaces. In order to avoid this issue, the best solution is to avoid such part geometries, where possible.

Finally, the red rust also appears in the crevices. It may be concluded that the powder does not reach the inner part of the crevices, leaving the unprotected steel. One way to prevent this issue is to apply the powder coating at the separate parts, and only after this to complete the assembly containing the crevice.

The additional solution for the issue of corrosion of specific parts of the equipment (welded zones, corners, sharp edges and crevices) would be the application of brush painting of such specific parts. This would lead to higher costs connected with the labor work on one side, but would definitely enable the achievement of paint layer of sufficient thickness on these critical parts.

To assess the suitability of polyester powder coatings for military applications, in addition to evaluating their corrosion resistance, the mechanical durability and camouflage effectiveness were also tested. The impact resistance test, conducted using steel balls, demonstrated that the polyester powder coating can endure a significantly higher number of impacts before any visible defects appear. This highlights the coating's exceptional mechanical robustness. Furthermore, diffuse reflectivity measurements revealed that the coating exhibits excellent camouflage properties, as its low reflectivity makes it highly effective in reducing visibility across various environments. This combination of properties positions polyester powder coatings as a highly promising solution for military applications, where durability and stealth are critical.

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