QUALITY ASSESSMENT OF ZINC COATINGS APPLIED BY SELECTED METHODS

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Purpose: This article presents the problem of corrosion phenomena occurring on steel products, which contributes significantly to the shortening of safe service life. One method of corrosion protection is the application of metal coatings. The most commonly used zinc coatings are described, along with two methods of their application: the galvanizing bath method and the lamellar method.

Design/methodology/approach: Coatings were made on the S195 steel specimens in different process variations. Weight, hardness and thickness, as well as surface defects, were used as criteria for assessing the quality of the coatings.

Findings: It was found that the process in the galvanizing bath can produce untight coatings with defects. On the other hand, the coatings applied by the lamellar method, were characterized by better aesthetics, lower weight and thickness compared to galvanic coatings, higher hardness compared to galvanic coatings, and uniformly covered the material without discontinuities in the structure that could impair durability.

Originality/value: The research clearly indicated the directions of application of the analyzed galvanic and lamellar coatings. The application of coatings is justified in the use of products exposed to corrosive agents. In terms of decorative qualities, more favourable results were obtained on the specimens with lamellar coatings, as these coatings had a silvery colour and an aesthetically pleasing sheen compared to the galvanic coatings. Due to the characteristics of the coatings, galvanic coatings can be used on parts that are operated in harsh environments, while lamellar coatings can be recommended for products that are required to have a low weight change and certain aesthetics

Keywords: galvanizing, galvanic coatings, lamellar coatings, coatings quality.

Category of the paper: research paper.

1. Introduction

The phenomenon of corrosion is the destruction of material as a result of chemical or electrochemical processes in the environment. As losses recorded in industry due to corrosion processes are a real problem, much attention is being paid to protecting materials from corrosion. There is a particular need to protect metals, as the ease with which corrosion can occur on their surface is due to their properties, primarily their good affinity for oxygen (Amin, Ibrahim, 2011; Rezaee, Attar, Ramezanzadeh, 2013). There are many types of corrosion, but due to the nature of the damaged surface, two types in particular stand out: local corrosion and general corrosion. With local corrosion, corrosion foci can be observed with the unaided eye and their distribution is random and does not cover the entire surface of the metal. General corrosion is characterised by corrosion foci distributed uniformly or randomly across the surface, and the corrosion products formed do not have a protective function against further corrosion of the material (Gao, Zhang, Li., Jiang, Zhang, 2018; Le Bozec, Thierry, Rohwerder, Persson, Luckeneder , Luxem 2013; Zhmurkin 2009).

The measures taken to protect against corrosion can be of two types (Surowska 2002):

* measures to reduce the occurrence of corrosion at an early stage, e.g. through appropriate selection of materials or proper planning of the technological process,
* protective coating operations that will separate the product from the damaging effects of corrosive agents.

The primary function of protective coatings is corrosion protection, but developments in coating deposition technology have also made it possible for coatings to perform decorative and technical functions, owing to parameters such as hardness or abrasion resistance.

There are two types of coatings: metallic and non-metallic ones (Gao, Zhang, Li., Jiang , Zhang, 2018; Revie, Uhlig 2008). Metal coatings are produced using pure metal, its properties can vary depending on the type of metal and the application method. Zinc coatings are applied to steel products because, owing to the lower electrochemical potential compared to steel, the corrosive effect only occurs on the surface of the coating. It becomes the anode and forms a barrier on the surface of the product. The zinc coating is therefore an anodic coating. There are also cathodic coatings, in which case the coating is the cathode and the protected product the anode. The coating should be applied evenly over the product, otherwise corrosion foci appear in untight areas causing material deterioration. Non-metallic coatings are produced using ceramics, plastics, enamels and paints. They are aesthetic and have insulating properties in addition to their anti-corrosive properties (Katayama, Kuroda 2013; Prosek, Nazarov, Bexell, Thierry, Serak 2008).

Zinc is a popular material used for protective coatings applied to metals. In industry, galvanisation and lamellar (flake) galvanisation are most commonly used (Lostak, Maljusch, Klink, 2014). The choice of coating depends on the operating conditions, economic aspects and the final properties of the coating (Hulser, Donner, Bauer, Hahn, 2016).

The galvanic method produces a thin coating of zinc in the process of electrolysis. The cathode is the coated product, the anode is the zinc plate. Using an external current source, the zinc ions separate from the anode and dissolve in the electrolyte (aqueous solution). The positively charged ions then move to the cathode, combining with electrons on the outside of the material. They thus form centres of crystallisation, resulting in the formation of a coating (Spathis, Poulios, 1995). In addition to zinc ions Zn^{2+}, there are other additives in the solution to maintain a constant current density. In weakly acidic baths these are Cl^- ions, in alkaline baths NH_4^+ ions, and in both cases also agents that give sheen, colour or other special properties to the coating (Elvins, Spittle, Worsley, 2005). The quality of the galvanic coating is influenced by how the surface is prepared. The substrate should be cleaned of all contaminants such as rust, oils, grease, fat, and oxide layers. Any residue reduces the durability of the coating and impairs adhesion, which may result in the need for frequent renewal of the coating. Before the substrate is cleaned, mechanical treatment (grinding, polishing) is applied to further improve the dimensional tolerances of the product (Gao, Zhang, Li., Jiang, Zhang, 2018; Diler, Rouvellou, Rioual, Lescop, 2014). The substrate is then degreased, rinsed and etched. The most common application is chemical degreasing, which involves immersing the product in hot baths of a suitable chemical composition (Surowska, 2002). Rinsing is an operation that separates the various stages of galvanic coating to prevent the transfer of contaminants or substances between successive surface preparation processes. The products are rinsed in hot (80-90°C) or cold water. It is recommended to rinse the products after each degreasing process first in hot water to wash off the bath components, followed by a rinse in cold water (Surowska, 2002). To remove corrosion products from the surface of the product, etching is used and the product is immersed in a bath of aqueous acid solution, in the case of steel, a 10-20% solution of sulphuric acid H_2SO_4. The bath gradually heats up from ambient temperature to a maximum of 70°C. The uniformity of the etching is ensured by adding inhibitors to the solution (Surowska, 2002). The next step, after surface treatment and preparation, is electrolysis in a suitably prepared bath. It should be able to form a coating with the specified parameters in the shortest possible time, have good opacity and ensure a high dissolution efficiency of the anode when the current density is high. The appropriate pH of the solution is obtained by adding H_2SO_4.

A distinction is made between acidic and weakly acidic, neutral, alkaline, and alkaline-cyanide baths. Acidic (sulphate and fluoroborate) or weakly acidic (chloride and ammonium chloride) baths are used for the galvanisation of iron and steel products with simple shapes of sheet, wire or strip (Surowska, 2002, Revie, Uhlig 2008). The advantages of acid baths are the high rate of coating deposition and the low cost of chemical components compared to other

types of bath, but the surface opacity can be poor. Disadvantages of coatings applied in acid baths can include low penetration, formation of dendrites, brittleness, and de-bonding (Surowska, 2002). In contrast, the opacity of the product in a weak acid bath is very good. The coatings have a mirror-like sheen, due to the shine-enhancing additives contained in the bath. Weak acid baths are also used for products with complex shapes, resilient and hardened components. Disadvantages of coatings deposited in weakly acidic baths include peeling and discolouration. Zinc coatings can be finished using chromate conversion with Cr^{3+} and Co^{2+} ions, which increases corrosion resistance and decorative effects (Szłapa, Jędrzejczyk, Skotnicki, Hajduga, Węgrzynkiewicz, 2014; Schaefer, Miszczyk, 2013).

Galvanic coatings are used in the machinery industry, automotive industry, for coating resilient components, machine parts operating in high humidity, and for decorative purposes. They are checked by measuring the thickness of the layer, hardness, and assessing its uniformity. These are the characteristics on which the durability of the coating and the quality of the corrosion protection of the product depend (Thierry, Prosek, Le Bozec, Diler, 2011; Qian, Li, Jungwirth, Seely, Fang, Shi, 2015).

The lamellar method, otherwise known as the flake method, is a non-electrolytic method of producing anti-corrosion coatings on a metal substrate (Fuarez, Gheno, White, 1993). The coating process involves immersing components or spraying a zinc-aluminium solution onto them. The resulting layer is cured by annealing in a furnace and then cooled by air flow (Giudice, Benftez, Linares, 1997).

As with galvanic coatings, the application of lamellar coatings is preceded by pre-treatment. Particularly important is the removal of impurities, which can be done by alkaline degreasing, shot-blasting or phosphating (21). Degreasing takes place in a hot alkaline bath with a pH of 11-14. The components of the bath produce a solution with very low surface tension, which avoids recontamination with oils. Objects are immersed in a solution of sodium hydroxide, crystalline sodium triphosphate, sodium hexaphosphate and water (in a ratio of: 4-1-0.5-supplementation to 100%) at 85-90°C, which improves the saponification of impurities. After a bath lasting about 10 minutes, the objects are rinsed first in hot and then in cold water. After degreasing, the surface is subjected to shot-blasting or phosphating, and these operations improve the surface adhesion properties. Shot-blasting is used on products with simple, flat surfaces and uncomplicated shapes. Stainless steel shot is commonly used, with a hardness of 450 HV and a particle size of 0.2-0.5 mm (Giudice, Benftez, Linares, 1997, Li, Du, Fan, Zhao, Ma, Wu, 2019).

Phosphating can be used instead of shot-blasting. The phosphating bath is applied by spray or immersion, on complex-shaped components with surfaces that are difficult to access (Hochmannová, 2002).

The proper, anticorrosive coating is applied to the prepared surface. In the lamellar method, these are zinc flakes with aluminium added in a 95:5 ratio. The coating can be applied by hot-dip galvanising with spinning, hot-dip galvanising or spraying. The spinning method uses

a special basket, into which the parts to be coated are placed, immersed in a previously prepared solution. During immersion, the basket rotates, eliminating the formation of air bubbles. The basket then rises and spinning begins, allowing the excess solution to be deposited on the walls of the basket. The immersion method is similar, however, the basket is not subjected to spinning after the immersion process and the removal of excess solution takes place in the furnace during drying (Hulser, Donner, Bauer, Hahn, 2016; Hochmannová, 2002). Spray application, on the other hand, is carried out using a pneumatic or electrostatic gun. In order to accelerate the deposition of the coating, the solution is heated to 20°C and the object to approximately 27-30°C. Compared to immersion, spraying enables a more uniform coating to be achieved, even on parts with complex shapes.

Regardless of how the coating is deposited, the object is then dried in a furnace at 180-320°C to cure the applied pigment. After the coating has cured, the objects are dried with an air stream until the object temperature reaches approx. 25°C (Muller, 2001).

In order to prolong the service life of lamellar coatings, sealing coatings (e.g. water-based lacquers) are additionally used to form an additional corrosion protection barrier. After the sealing coating has been applied, the component dries again.

Lamellar coatings have a number of advantages: resistance to elevated temperatures and to mechanical influences. They are environmentally friendly due to the absence of harmful substances in the coating application process. The replacement of zinc dust with flakes and the absence of acids makes it possible to eliminate hydrogen embrittlement, in-crease adhesion and reduce oxygen permeability. Laminar coatings are used in the auto-motive, aerospace and energy industries, due to their ability to coat customised and mass-produced products with complex shapes with durable coatings in different colours (Hulser, Donner, Bauer, Hahn, 2016; Hochmannová, 2002; Muller, 200; Jędrzejczyk, Szłapa, Skotnicki, 2015).

The thickness of the coating determines its anticorrosive properties. Testing the thickness of the coating is the first step in determining the correctness of the workmanship, and its determination helps to decide the need for further quality tests. If the value obtained is not appropriate, e.g. does not meet the specified standards, then further analysis is discontinued, as it is assumed that the coating will not fulfil its primary, protective function.

The thickness of the coating can be measured by destructive or non-destructive methods by determining the local thickness, measured at a selected location, or the average thickness, which is the averaged result of several measurements taken at different locations on the product (Rodzynkiewicz-Rudzińska, 1985). Due to their less labour-intensive nature, microscopic, weighing, drop or stream methods are most commonly used.

If the thickness of the coating meets the requirements, hardness is measured in the next step. In the case of protective coatings, it has to do with the structure, which can vary due to the possibility of using different baths, ingredients, and temperatures. Hardness is measured on the Brinell, Vickers or Knopp scale, using an indenter of appropriate shape (Łabędź, 2017).

The next step in assessing the quality of the coating is a visual evaluation of the characteristics of the external appearance, e.g. determining the colour, type of defects, degree of sheen, smoothness, etc. This assessment is carried out with the unaided eye or using microscopes (Łabędź, 2017).

2. Materials and Methods

S195 steel plate with the chemical composition given in Table 1, was used in the experimental study, from which 15 x 100 mm specimens were made (Wyrzgoł, 2022).

Table 1.
Chemical composition of S195 steel (Wyrzgoł, 2022)

Ingredient	C	Mn	Si	S	P
Content, %	0.2	1.4	-	0.045	0.045

For galvanic coating, a stand consisting of a galvanic bath and a power supply unit with the following parameters was used: supply voltage 400 V ±10%, frequency 50-60 Hz, short-circuit voltage according to standard (UNE-EN 60204-1:2019), input current max. 9.6 A, number of phases – 3. The function of the anode during the coating application was performed by non-degradable graphite. A zinc sulphate bath from Technologie Galwaniczne Sp. z o.o. was used for galvanising (Wyrzgoł, 2022).

The application of the zinc coating using the lamellar (flake) method was carried out using YATO spray gun YT-82553. A pigment consisting of ZINTEK 400 zinc-aluminium flakes from Atotech was applied with a 2.6 mm diameter nozzle. After coating, the specimens were dried for curing in an FCF 22HP chamber furnace with the following parameters: operating temperature 1.220°C, temperature control range 20-1.300°C, supply voltage 400 V/2N, rated current 12 A, and rated power 4.8 kW (Wyrzgoł, 2022).

All specimens were weighed on an analytical balance before coating, then etched in concentrated H_2SO_4 and later rinsed in distilled water.

Coatings were applied to the prepared specimens using two methods: galvanization in an acid zinc bath and lamellar method.

The bath galvanising process was carried out in the variants given in Table 2.

Table 2.
Parameters of galvanizing in the galvanic bath

Specimen number	Intensity, A	Time, s
1	2	300
2	2	600
3	2	900
4	4	300
5	4	600
6	4	900
7	6	300
8	6	600
9	6	900

On the other hand, flake coating was carried out using a spray gun, in the case of which method the specimens were coated with pigment. All specimens in both coating method variants were then furnace dried to cure according to the scheme given in Table 3.

Table 3.
Variants for drying zinc-coated specimens

Specimen number	Temperature, °C	Time, s
1	210	900
2	210	1500
3	210	2100
4	230	900
5	230	1500
6	230	2100
7	250	900
8	250	1500
9	250	2100

The applied coatings were assessed for macrostructure, thickness and hardness.

Macrostructure testing of the coatings was carried out using an Olympus SZX9 stereoscopic microscope, enabling images to be obtained at 5-30x magnification. Macroscopic images were taken at 12.5x magnification. Markers of the observed coating structures of 19 px for galvanic coatings and 8 px for lamellar coatings were determined.

The thickness of the zinc coating was measured using a TESTAN DT-25 probe thick-ness gauge from ALFA-TECH with the following parameters: measuring range 0-1500 µm, measuring accuracy ±(2%+1) µm, minimum radius of curvature 1.5 mm, minimum diameter of the measuring area 6 mm, minimum substrate thickness 0.5 mm, and operating temperature from 0-60°C.

The hardness of the coatings was measured using an Elcometer 3095 hardness tester from SciTeeX, designed for rapid Brinell hardness assessment using the indenter push method, with a pressure of 500g.

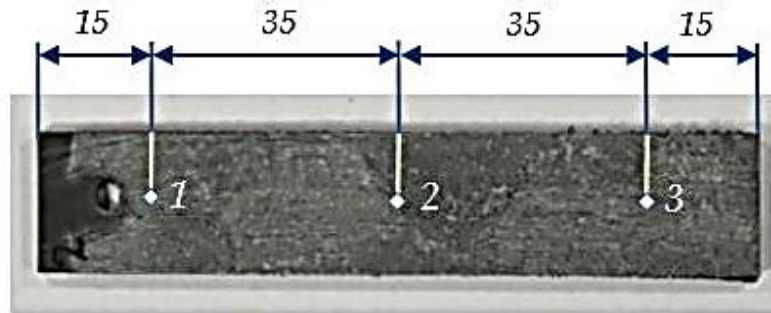The thickness and hardness of the coating was tested at three points, shown in Figure 1.

Figure 1. Coating thickness and hardness measurement locations.

In the hardness tests, the indentation length was determined by gently lowering the indenter onto the test coating over a period of 30 seconds. After raising the indenter, the length of the resulting indentation was read using a microscope after 35 s. Hardness x was determined from the relationship:

x = 100/l   (1)

where:

x – hardness,

HB; l – indentation length, mm.

3. Results

The specimens were weighed before the experiment, and after coating and drying (Figure 2), they were reweighed. The results of the specimen mass measurements are given in Table 4 and 5.
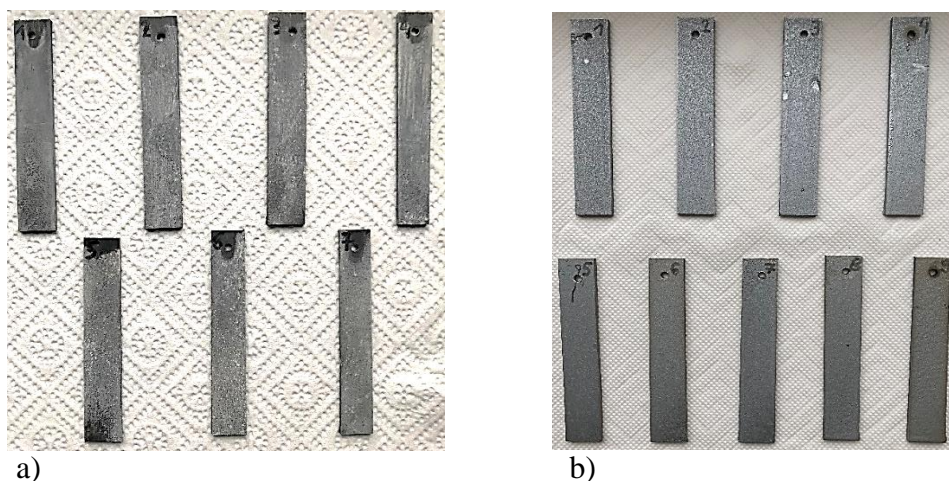

a)   b)

Figure 2. Specimens after zinc coating by: a) galvanic method, b) lamellar method.

Table 4.

Results of weighing the specimens before and after the application of galvanic coating

Specimen number	Weight before, g	Weight after, g	Weight gain, g
1	40.48	40.73	0.25
2	41.14	41.56	0.42
3	41.50	41.74	0.24
4	35.36	35.58	0.22
5	40.92	41.25	0.33
6	38.29	38.67	0.38
7	34.77	34.99	0.22
8	41.12	*	-
9	38.62	*	-

* during the application of coating 7, it was observed that the electrolytic deposition of zinc was not proceeding properly, keeping the specimen in the bath did not result in an improvement of the coating condition. The performance of coatings over a longer period of time (Nos. 8 and 9) was abandoned.

Table 5.

Results of weighing the specimens before and after application of the lamellar coating

Specimen number	Weight before, g	Weight after, g	Weight gain, g
1	40.95	41.08	0.13
2	40.86	41.04	0.18
3	41.21	41.42	0.21
4	41.55	41.76	0.21
5	41.30	41.38	0.08
6	41.16	41.23	0.07
7	41.22	41.32	0.10
8	40.72	40.91	0.19
9	40.86	41.03	0.17

The results of the coating thickness measurements are shown in Tables 6 and 7.

Table 6.

Results of thickness measurements of galvanic coatings

Specimen number	Measurement 1 µm	Measurement 2, µm	Measurement 3 µm	Mean value µm	Variation
1	24.0	22.2	18.8	21.67	6.97
2	25.5	26.2	26.7	26.13	0.36
3	29.3	33.4	25.8	29.50	14.47
4	20.2	17.5	29.7	22.47	41.06
5	34.7	35.0	26.5	32.07	23.26
6	38.4	35.0	26.5	33.30	37.57
7	24.4	22.7	17.8	21.63	11.74
8	-	-	-	-	
9	-	-	-	-	

Table 7.
Results of lamellar coating thickness measurements

Specimen number	Measurement 1 μm	Measurement 2 μm	Measurement 3 μm	Mean value μm	Variation
1	13.3	14.4	15.7	14.47	1.44
2	17.4	17.0	15.9	16.77	0.60
3	17.5	17.4	15.7	16.87	1.02
4	17.2	16.8	16.4	16.80	0.16
5	13.9	13.6	13.0	13.50	0.21
6	12.7	12.6	12.5	12.60	0.01
7	12.4	12.3	12.1	12.27	0.02
8	11.9	12.1	12.2	12.07	0.02
9	14.7	14.0	15.6	14.77	0.64

The results of the hardness test for coatings applied in the galvanic bath are given in Table 8 and those applied by the lamellar method in Table 9.

Table 8.
Hardness of galvanic coatings

Specimen number	Measurement 1 HB	Measurement 2 HB	Measurement 3 HB	Mean value HB
1	52	50	56	52.7
2	40	42	48	43.3
3	53	50	48	50.3
4	55	58	52	55.0
5	47	43	43	44.3
6	43	55	50	49.3
7	58	66	63	62.3
8	-	-	-	-
9	-	-	-	-

Table 9.
Hardness of lamellar coatings

Specimen number	Measurement 1 HB	Measurement 2 HB	Measurement 3 HB	Mean value HB
1	83	77	71	77.0
2	77	83	77	79.0
3	83	91	100	91.3
4	100	111	100	103.7
5	111	111	91	104.3
6	100	125	111	112.0
7	91	91	100	94.0
8	100	83	91	91.3
9	67	77	83	75.7

The results of the macroscopic examination of the coatings are shown in Figures 3 and 4.
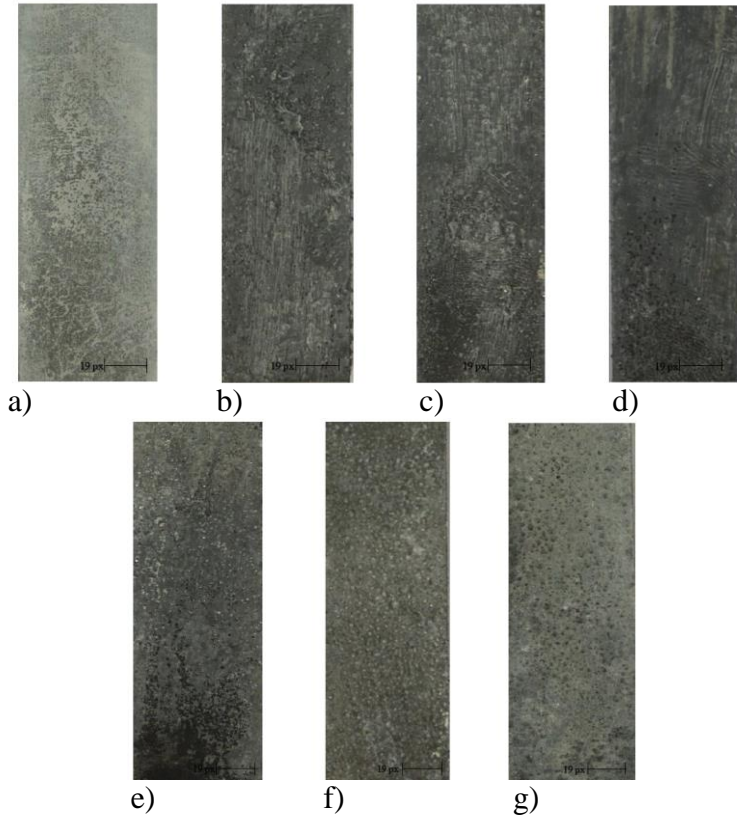
a) variant 1, b) variant 2, c) variant 3, d) variant 4, e) variant 5, f) variant 6, g) variant 7.

Figure 3. Macrostructure of galvanic coatings on specimens.
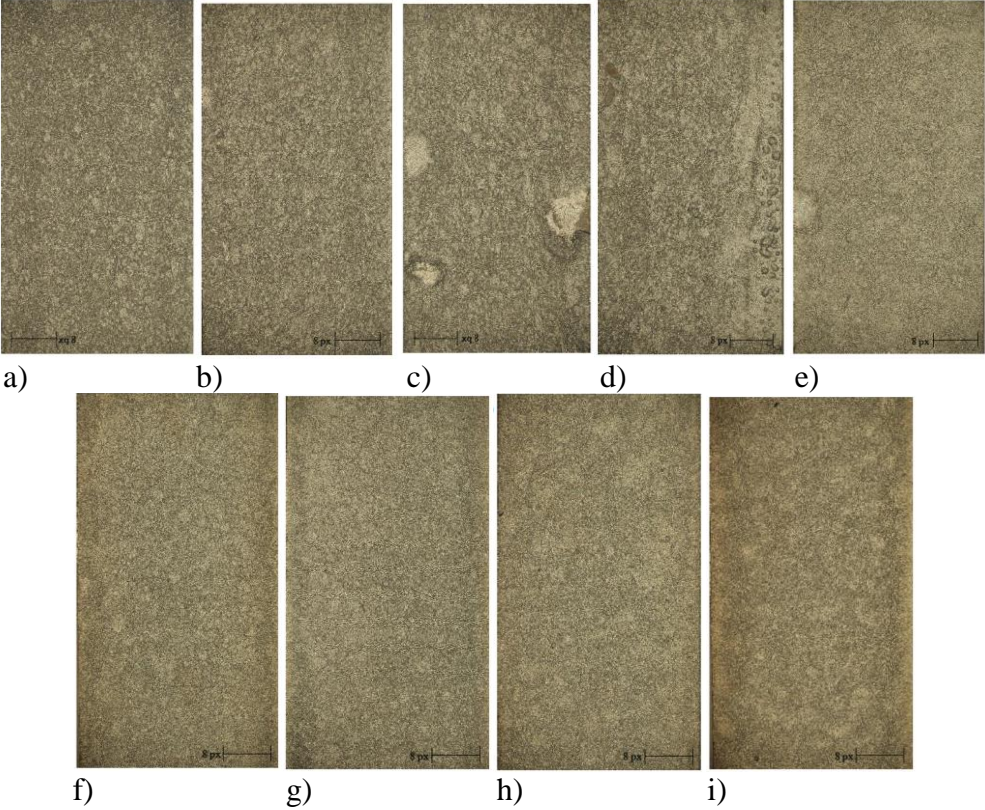
a) variant 1, b) variant 2, c) variant 3, d) variant 4, e) variant 5, f) variant 6, g) variant 7, h) variant 8, i) variant 9.

Figure 4. Macrostructure of lamellar coatings on specimens.

4. Discussion

In the experiment carried out, the application of the coating by the galvanic method in variants 8 and 9 proved to be unjustified. Observing the specimen during bath No. 7, it was observed that the electrolytic deposition at 6A for 300 s did not proceed properly. Separations in the form of zinc dendrites appeared on the surface of the coating, indicating an abnormal process and thus a lack of tight coating of the specimen surface. Therefore, extending the process to 600 and 900 s made no sense. The lamellar coating process was not affected with such difficulties.

Galvanic coatings were heavier than lamellar coatings (Figure 5). Their weight ranged between 0.22 and 0.42 g, while that of lamellar coatings ranged between 0.07 and 0.21 g.

The largest weight increase, i.e. by 0.42 g, was recorded for the plating on specimen 2 (2A, 600 s). In contrast, the coating on lamellar-coated specimen No. 6 was the lightest, 0.07 g (dried at 230°C).
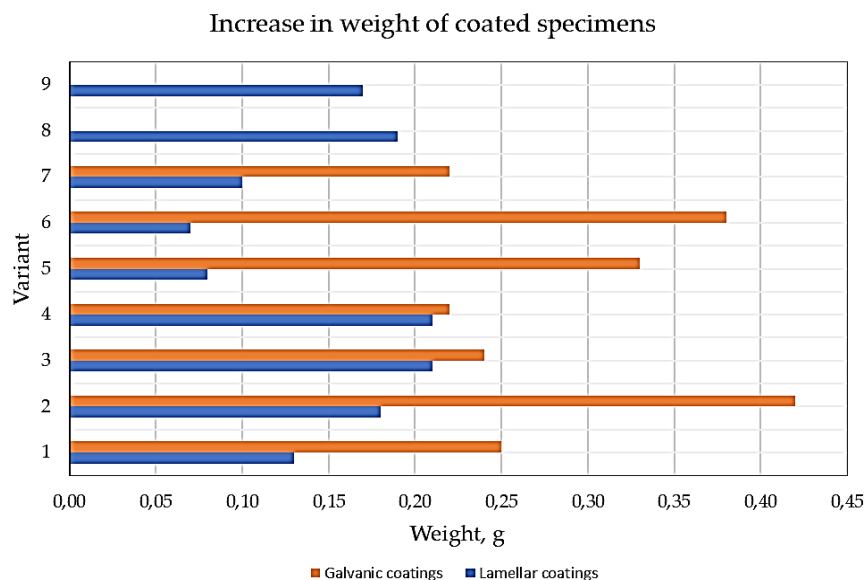

Figure 5. Differences in weight gain of specimens with coatings.

However, despite the implementation difficulties, all galvanic coatings, due to their thickness, meet the requirements of the industry (Rodzynkiewicz-Rudzińska, 1985). It is expected that such a coating should have a thickness in the range of 5 to 40 μm, and the coatings obtained in the experiment had an average thickness in the range of 21.63-33.30 μm, so both the thinnest (in variants 1 and 7) and the thickest in variant 6 are coatings that meet the quality requirements.

For lamellar coatings, a thickness of 5 to 15 μm is required (ISO 10683:2014:2014-05). In the study, the average thickness of the lamellar coatings was found to be between 12.07 and 16.87 μm. Only the coatings on specimens 2, 3 and 4 meet the thickness requirements.

The variance calculated in Tables 6 and 7 shows that lamellar coatings have a higher uniformity (variance in the range of 0.01-1.44), while galvanic coatings differed significantly (variance in the range of 0.36-41.06).

The average hardness of the galvanic coatings was in the range of 43.4-62.3 HB (Figure 6). The hardest galvanic coating was obtained in a bath with a current flow of 6 A (variant 7). Zinc coatings applied using the lamellar method had a significantly higher hardness, with hardness in the range of 75.7-112.0 HB. For these coatings, as the curing temperature increased, the hardness increased, but only up to 230°C. At 250°C, the hardness decreased.

Macroscopic observations of the galvanic coatings indicate that a uniform and tight coating was obtained for specimen No. 1 (2 A, 300 s). No dendritic crystals were observed. As the current increased, the quality of the coatings deteriorated. On specimen No. 5, thickenings of random location were formed. This phenomenon may have been the reason for obtaining different coating thickness measurement results. Coatings of uneven thickness were also visibly rougher. A zinc bath with a current flow of 6 A for 300 s (specimen No. 7) produced the worst coating; excessive hydrogen release caused a spongy structure to appear, which was evident in pitting, sometimes reaching the substrate of the specimen. In order to improve the quality of the coating in this treatment variant, it may be proposed to apply a conversion coating in an additional operation, such as chromating.
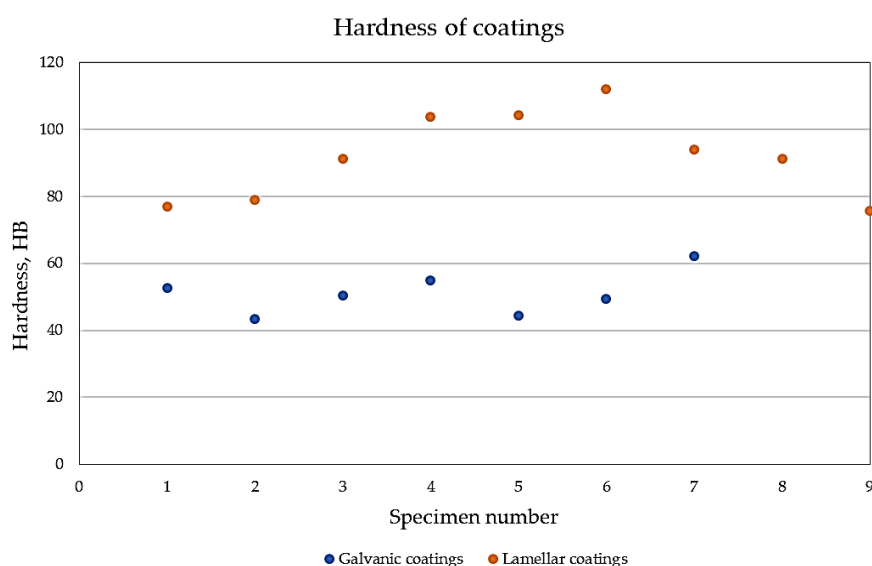

Figure 6. Comparison of the hardness of the applied coatings.

In contrast, in the case of lamellar coatings, the higher the temperature and longer the curing time, the worse the quality. The best structure was the coating on specimen No. 1 (210°C and cured for 900 s) and specimen No. 2 (210°C and cured for 1500 s) fulfilled the thickness requirement at the same time. Both coatings were uniform and tight, with no visible cavities or cracks. In the case of specimen No. 4, coarsening and fine pitting (230°C, 1500 s) were observed at the edge. In addition, scorch marks appeared on specimens 7, 8 and 9 as a result of excessive heat, particularly evident on specimen 9 (250°C, 2100 s). These may have affected the previously described results of the hardness test of the coatings.

5. Conclusions

The application of coatings is justified in the use of products exposed to corrosive agents. The application of coatings can take place in different ways, in which case the correct choice of process parameters is important. Based on the experiment, it can be concluded that the best zinc coating applied in the galvanic bath can be obtained in the galvanizing process at a current of 2 A in 300 s, and when applying the lamellar coating using furnace curing at 210°C in 900 s. However, the lamellar coatings had more favourable characteristics. Lamellar coatings were more uniform than galvanic coatings. Macroscopic examination revealed that the lamellar coatings were continuous, while the galvanic coatings had untightnesses. Lamellar coatings were also harder than galvanic coatings, influenced by the temperature of the furnace during curing.

In terms of decorative qualities, more favourable results were obtained on the specimens with lamellar coatings, as these coatings had a silvery colour and an aesthetically pleasing sheen compared to the galvanic coatings.

Due to the aforementioned characteristics of the coatings, galvanic coatings can be used on parts that are operated in harsh environments, while lamellar coatings can be recommended for products that are required to have a low weight change and certain aesthetics.

Acknowledgements

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