Methods for evaluating the appearance of hot dip galvanized coatings

S. Sepper¹, P. Peetsalu^{1,2} and M. Saarna¹

¹Department of Materials Engineering, Tallinn University of Technology ²Tartu College, Tallinn University of Technology; e-mail: sirli.sepper@emu.ee

Abstract. Although the science and technology of hot dip galvanizing have improved significantly over the years, it is still a challenge to produce high-quality coatings for decorative and constructional applications. Different applications require specific appearance of galvanized coatings (e.g. dull appearance in roof construction). The appearance of the coatings depends on processing properties, steel content, and substrate surface conditions. The purpose of this study is to work out a technique how to evaluate the appearance of hot dip galvanized coatings. Under observation are substrate steel parameters (chemical composition, surface conditions e.g. roughness), which affect the appearance of hot dip galvanized coatings. Based on this research appearance classifications have been established.

Key words: Hot dip galvanizing, iron-zinc phases, coating appearance, spangle size.

INTRODUCTION

Hot dip galvanizing is used as a very effective steel corrosion protection method, providing a long service-life. The corrosion protection is dependent on the coating thickness and environmental conditions (ASM Handbook, 1994). Zinc coated components are also used to give a good appearance to the constructions. In recent years the interest in hot dip galvanizing for decorative and constructional applications has increased. The difference in the field of use determines the requirements to the coating appearance. Duller coating finish is desired in buildings, because shiny coatings with high reflectivity may cause problems with passing traffic. At the same time most customers prize the bright spangled look for decorative applications. As a result of customer demands the requirements to the coatings and especially to the appearance have increased. Coating appearance is affected by processing properties, steel chemistry, and substrate surface condition.

Coating appearance and zinc consumption mainly depend on the zinc-steel reactivity and on the drainage of zinc from workpieces during their withdrawal (Fratesi et al., 2002). The zinc-steel reactivity is mostly influenced by the silicon and phosphorus content in the steel, but also carbon in excess of about 0.2% and manganese in excess of about 1.3% increase Zn-Fe layer formation (Hornsby, 1995). The zinc drainage is influenced by bath fluidity. The addition of low amounts of certain elements, such as Al, Pb, Ge, V, Ti, Ni, Bi, Cu, Cd, Sn inhibit zinc-steel reactivity and/or increase the bath fluidity (Jalel Ben nasr et al., 2008; Xuping et al., 2010; Pistofidis et al, 2007).

The surface of the galvanized coatings is commonly characterized by spangles (snowflake-like pattern). Zinc coatings with spangles are decorative coatings, and its appearance is closely related to the orientation of zinc crystals and the distribution of alloy elements in the coating. Spangle formation is favoured when Sb, Bi, and especially Pb are added to the zinc bath because all these metals lower the surface tension ahead of the growing dendrites, resulting in larger grains. Grain size is also influenced by the cooling rate and the smoothness of the substrate (Marder, 2000; Pavlidou et al., 2005; Lu Jin-tang et al., 2007; ShuPeng et al., 2010).

The roughness of the steel surface influences the thickness and structure of the coatings. A rough steel surface as obtained by grit blasting and course grinding have a higher surface area and thus generate thicker galvanized coatings. The effect of surface unevenness of the substrate metal generally remains visible after galvanizing. Galvanized coatings are not effective at hiding defects of the steel and indicate steel surface quality problems (defects associated with casting, rolling, and manufacturing processes) (ISO/FDIS 14713-2:2009).

Unfortunately there are no quantitative specifications how to evaluate the appearance of galvanized sheet. International standard ISO 14713-2:2009 divides coating characteristics into two groups relating to steel chemical composition:

1) Coating has a shiny appearance with a finer texture. Coating structure includes outer zinc layer.

2) Coating has a darker appearance with a coarser texture. Iron/Zinc alloys dominate coating structure and often extend to the coating surface, with reduced resistance to handling damage.

The main objective of this study is to work out a technique how to evaluate the appearance of hot dip galvanized coating. Under observation are steel chemical composition and substrate surface condition, not defects arising from the hot dip galvanizing process. The appearance evaluation applies only for fresh zinc coatings, without protective layer zinc corrosion usually causes the appearance of the coated steel to turn a dull grey.

EXPERIMENTAL

Five materials were used in the experiment (with different silicon equivalent). The chemical compositions of the steels are shown in Table 1.

Steel	С	Si	Р	S	Mn	Al	Cr	Мо	Silicon eguivalent
a	0.09	< 0.01	0.012	0.006	0.33	0.034	0.02	0.03	< 0.04
b	< 0.01	< 0.01	0.051	0.008	0.21	0.037	0.03	0.03	< 0.14
c	0.35	0.23	0.009	0.001	0.64	0.030	0.18	0.03	0.25
d	0.79	0.27	0.014	0.005	0.68	0.026	0.09	0.05	0.31
e	0.58	0.05	0.007	< 0.001	0.69	0.012	0.24	0.05	0.07

Table 1. Chemical composition of the substrate steels, wt %.

The steel sheets were degreased for 15min in acid degreasing agent and then pickled for 45min with a 13% HCl containing inhibitor for the protection of metal surfaces. Next the sheets were rinsed in water and then dipped in a flux bath consisting of $242g l^{-1} ZnCl_2$ and $186g l^{-1} NH_4Cl$ which was kept at 40°C.

The fluxed sheets were dried for 15 min at 120°C in drying oven. Then the sheets were dipped in the zinc bath for 6 min at the temperature 460°C. The zinc bath consists of zinc (99.3 wt. % Zn) containing also Al, Bi, Fe, Ni, Sn, Pb.

For the examination of the microstructure, hot dip galvanized specimens were cross sectioned, hot mounted and polished. A nital etchant (nitric acid: 3 wt. %) was used to reveal the microstructures of the specimens and observations were made with optical microscopy.

The thickness of coatings was determined by electromagnetic thickness gauge (Dualscope MP0). The surface roughness of galvanized steels was measured with surface texture measuring system "Perthometer Concept" produced by company MAHR.

RESULTS AND DISCUSSION

Steel chemical composition

The selection of the materials used in this experiment was based on differences in chemical composition of the steels to evaluate the influence of substrate chemical composition to the appearance of hot dip galvanized coatings.

The characteristic photographs of the above mentioned galvanized steels 'a-d' are presented in Fig. 1. Steel 'a' (silicon equivalent <0.04) and 'c' (silicon equivalent 0.25) have shiny spangled appearance. Steel 'b' has dull appearance as a result of the phosphorus present (0.05wt. %) in the substrate steel. Steel 'd' has shiny appearance with no visible spangle. This difference in appearance is a result of the rapid zinc-iron intermetallic growth that consumes all of the bright, pure zinc. The surface roughness of galvanized steels is presented in Table 2. Coatings with spangles have lower surface roughness.

Table 2.	Surface	roughness	of ga	lvanized	steels.
----------	---------	-----------	-------	----------	---------

Steel	R _a , μm	R_z , μm
a	1.4	8.8
b	8.1	55.5
с	3.0	17.8
d	9.5	46.3

The optical micrographs (magnification 200x) of the cross-sections of the coatings are shown in Fig. 2. Microscopic level differences occur due to the amount of silicon and phosphorus in the steel being hot dip galvanized. Steel 'a' has a typical microstructure of the hot dip coating, which is typical for steels with low silicon equivalent. Four layers could be distinguished based on their relief: gamma (Γ) phase, delta (δ) phase, zeta (ζ) phase and eta (η) phase.



Figure 1. Visual appearance of hot dip galvanized steel sheets a–d. Sheet '*a*' and '*c*' with shiny spangled appearance. Sheet '*b*' with dull appearance and '*d*' with shiny appearance with no visible spangle.

From the coating microstructure of steel 'b' and 'd' we can see the excessive growth of the ζ phase, however, ζ layer is not uniform and so the local bursts of the ζ phase layer have occurred. As a result of this the surface roughness is high. Steel 'c' has also increased ζ layer and compose most of the galvanized coating thickness.

Fig. 2 can be used to describe the coating thickness differences due to substrate chemical composition. The amount of silicon and phosphorus (silicon equivalent) in the substrate steel strongly influences the thickness and appearance of the galvanized coating.

Steel chemical composition has a major effect on the appearance of hot dip galvanized coatings. Silicon and phosphorus content influence the reaction rate between zinc and iron and thus control the alloy layer growth and formation during hot dip galvanizing. The visual appearance of the coating depends on Fe/Zn alloys. Local outbursts of the ζ phase influence the coating surface roughness and coating appearance.



Figure 2. The micrographs of the cross-sections of the coatings with following silicon equivalents: sheet 'a' 0.04, 'b' 0.14, 'c' 0.25 and 'd' 0.31.

Effect of article surface condition

The substrate steel roughness and surface defects also affect the appearance of the coating. The roughness of the steel surface has an influence on the thickness, structure, and appearance of the coating. Fig. 3 shows visual appearance differences of steel 'e' due to substrate surface roughness. The steel was ground with 80 grit (Fig. 3.1) and 240 grit sand paper (Fig. 3.2). Ground steel with 80 grit sand paper had zinc coating with peaks and valleys while 240 grit gave smooth, shiny and spangled appearance.

The micrographs of steel with different surface roughness after galvanizing are presented in Fig. 4. The steel 'e' is reactive steel and zeta layer is overgrown and compose most of the galvanized coating thickness (Fig. 3.2). Roughening the reactive steel creates peaks and valleys which interfere with the growth of the zeta intermetallic layer (Fig. 3.1).



Figure 3. Differences in visual appearance due to surface roughness: (1) - 80 grit, (2) - 240 grit.

A rough steel surface gave a thicker coating. The average coating thickness of roughened steel (80 grit) was 105µm and 240 grit gave coating thickness 165µm.



Figure 4. Substrate surface roughness effect on the galvanized coating: (1) - 80 grit, (2) - 240 grit.

The surface unevenness and defects of the substrate metal generally remain visible after hot dip galvanizing. Steel 'c' had a surface defect presented in Fig. 5.1. The reason of the defect was probably contact with a chemical, which caused intergranular corrosion of the steel surface (defect depth 6μ m).

Zinc coating has two different microstructures (Fig. 5.2) which were divided by 'wavy line'. Differences were also in coating thicknesses: $98\mu m$ and $133\mu m$. Surface with intergranular corrosion had thinner zinc coating.



Figure 5. Surface defect before (1) and after (2) hot dip galvanizing.

The chemical composition of steel combined with its surface condition will affect the appearance and the thickness of the galvanized coating. The surface roughening is used to obtain thicker coating but in case of reactive steel the result might be reverse. Rough substrate surface can also affect the spangle formation and coating surface roughness. Surface defects associated with casting, rolling, and manufacturing processes generally remain visible after galvanizing.

Appearance classification

Arising from absence of appearance specification of hot dip galvanized coatings, the three appearance classifications could be composed based on this research.

Class 1. This class is characterized by shiny or mirror-like coating with spangles. The spangle size might be different (regular spangle or minimized spangle), but grain should be visible to the naked eye. Steel reactivity is normal or low. The hot dip galvanized coating consists of four separate layers: gamma (Γ) phase, delta (δ) phase, zeta (ζ) phase, and eta (η) phase. In some cases the pure zinc layer (η) might be absent. The surface roughness of galvanized coating is low.

Class 2. This class is characterized by shiny coating with no spangles visible to the naked eye. The silicon or phosphorus content of the substrate steel is high and therefore the steel reactivity is high. Coating thickness increases with increasing silicon equivalent. ζ layer is not uniform and so local outbursts might occur. This class will provide thick and rough coating.

Class 3. This class is characterized by dull grey appearance with no spangles visible to the naked eye. Excessively thick coating may be formed due to high steel reactivity. This class will provide thick and rough coating with no external top η layer.

Established appearance classification describes only common coating appearances. There might be occasions when classification is not so easy (e.g. half of the coating is dull and the other part is shiny). In that case it is possible to divide steel coatings into classifications by percentage (70% class 1 and 30% class 2).

Galvanized steels, which were used in this study, may be divided as follows (Figs. 1 and 3):

- 1) Class 1 steel 'a', 'c' and 'e' ground with 240 grit sand paper;
- 2) Class 2 steel 'd' and 'e' ground with 80 grit sand paper;
- 3) Class 3 steel b'.

CONCLUSIONS

In the present paper, the influencing factors which affect the hot dip galvanized coatings have been studied. Three appearance classifications have been composed based on visual appearance (spangle size), coating roughness and Fe/Zn alloy layer growth and formation. The following conclusions can be drawn from the present study:

1. Hot dip galvanized coating appearance may be divided into 3 classes.

2. The substrate steel chemical composition has major effect on coating appearance.

3. The substrate roughness and surface defects also have an impact on coating appearance.

ACKNOWLEDGEMENTS: This research was supported by European Social Fund's Doctoral Studies, Internationalisation Programme DoRa, and Estonian Science Foundation targeted financing project SF0140091s08. Cooperation was carried out with AS Paldiski Tsingipada.

REFERENCES

ASM Handbook. Surface engineering, vol 5, ASM Internatinal, 1994.

- Fratesi, R., Ruffini, N., Malavolta, M., Bellezze, T. Contemporary use of Ni and Bi in hot-dip galvanizing. Surface and Coatings Technology, 2002, 157, 34–39.
- Hornsby, M. J. Hot dip galvanizing: A guide to process selection and galvanizing practice. London: Intermediate Technology Publications, 1995.
- Jalel Ben nasr, Ali Snoussi, Chedly Bradai, Foued Halouani. Effect of the withdrawal speed on the thickness of the zinc layer in hot dip pure zinc coatings. Materials Letters, 2008, 62, 2150–2152.
- Xuping Su, Changjun Wu, Daniel Liu, Fucheng Yin, Zhongxi Zhu, Sui Yang. Effect of vanadium on galvanizing Si-containing steels. – Surface & Coatings Technology, 2010, 205, 213–218.
- Pistofidis, N., Vourlias, G., Konidaris, S., Pavlidou, El., Stergiou, A., Stergioudis, G. The effect of bismuth on the structure of zinc hot-dip galvanized coatings. – Materials Letters, 2007, 61, 994–997.
- Marder, A. R. The metallurgy of zinc-coated steel. Progress in Materials Science, 2000, 45, 191–271.
- Pavlidou, E., Pistofidis, N., Vourlias, G., Stergioudis, G. Modification of the growth-direction of the zinc coatings associated with element additions to the galvanizing bath. – Materials Letters, 2005, 59, 1619–1622.
- Lu Jin-tang, Wang Xin-hua, Che Chun-shan, KONG Gang, Chen Jin-hong, Xu Qiao-yu. Crystallographic research of spangle on hot dip galvanized steel sheets. – Trans. Nonferrous Met. SOC. China, 2007, 17, 351–356.
- Shu Peng, Jintang Lu, Chunshan Che, Gang Kong, Qiaoyu Xu. Morphology and antimony segregation of spangles on batch hot-dip galvanized coatings. Applied Surface Science, 2010, 256, 5015–5020.
- Zinc coatings- Guidelines and recommendations for the protection against corrosion of iron and steel in structures. Part 2, Hot dip galvanizing: ISO/FDIS 14713-2:2009.