# ANALYSIS OF THE INFLUENCE OF THE HEAT TREATMENT ON FUNCTIONAL PROPERTIES OF HOT-DIP ZINC COATED FASTENERS

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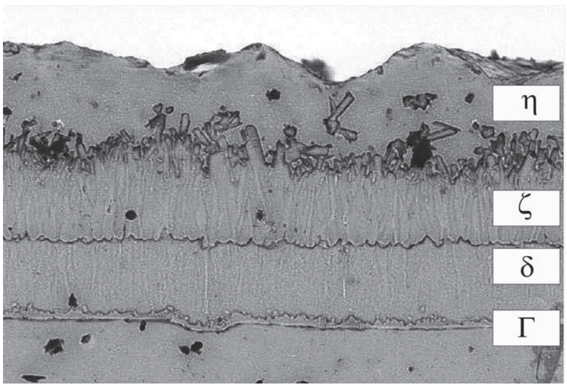
**Abstract**

The presented work constitutes the continuation of the previous research concerning the measurement of the tribological properties of different zinc coatings deposited to fasteners. The applied heat treatment was mainly focused on increasing the hardness of the outer layer of the zinc coating and increasing wear resistance. Fasteners (M12-60 bolts) were galvanized in industrial conditions (according to PN-EN ISO 10684), and next were subjected to the heat treatment in an electric chamber furnace. Bolt sets were subjected to analyses using the Schatz ® Analyse device. It lets for determining the quality of fasteners in realistic working conditions (e.g. assembly) and according to international standards. Results of attempts allowed for determination the rate of the friction coefficient of bolts and nuts on the thread and under the head and verification of the coating heat treatment influence on measured parameters - microstructure changes, micro-hardness distribution and coating topography of the zinc coating before and after the heat treatment.

**Keywords:** Hot-dip zinc coating, fasteners, Schatz Analyse

## 1. INTRODUCTION

The hot dip zinc galvanizing process has been studied for many years and still cause a lot of difficulties. Large majority of problems regard the ready made surface where defects are observed on the coating surface just after installation or after relatively short time of operation. Hot-dip galvanized surfaces are exposed to different kind of abrasive wear: mechanical friction, dynamic impacts, interaction with environment containing sands and other agressive powders. The surface hardness is one of the fundamental parameters both in the contact mechanics as well as in theory frictions, and also the basic parameter in forecasting the exploitation permanence of detachable connections, applied in machines [1]




| Hot-dip zinc galvanizing coating | |
|---|---|
| Alloyed layer | Hardness |
| 100 % Zn - eta (η) | 70 HB |
| $FeZn_{13}$ - zeta (ζ) | 220 HB |
| $FeZn_7$ - delta (δ) | 270 HB |
| $Fe_3Zn_{10}$ - gamma (Γ) | - |

**Figure 1** Microstructure of hot-dip zinc coating and its hardness [2]

The hardness is also the significant parameter of the quality assessment of protective coatings. The most frequently used zinc coatings differs essentially in hardness values. The hardness of the coating indirectly influences the anticorrosion properties. The wear resistance is an important criterion of the selection of the coating for the given application. Zinc-plated coatings deposited in the galvanic technology demonstrate the hardness ab. 50 HV. It is possible to increase this hardness level up to 500 HV through introducing the alloyed elements, i.e. Ni (14%) [3,4]. The hot-dip zinc coatings shows layers structure: it was proved, that in Fe-Zn diagram occurs three phases, arising as a result of the peritectic reaction: $\Gamma$ - $Fe_3Zn_{10}$, $\delta$ - $FeZn_7$, $\zeta$ - $FeZn_{13}$ and iron solid solution in zinc - $\eta$ - **Figure 1** [5]. Hardness of outside layer (constituting about 10% of the whole) is about 50 HV, diffusion layer Zn-Fe (90% of the whole of the coating) is characterized by a hardness level 150 HV [6]. The lamellar coatings show an increased hardness of outside surface that reaches even 500 HV because of application the thermosetting surface varnish as the last layer. The second important parameters that could be used for coating quality evaluation is the friction coefficient. Normally the dynamic friction coefficient is measured using a traditional pin-on-disk tribometer, where a pin is pressed against a reciprocating counterface [7]. During the tests the following parameters are measured: friction force, the total linear wear of test specimens, chamber temperature, rotational speed, and time and the number of disk revolutions (sliding distance). Using the mentioned equipment only the flat samples can be tested. In the fastener case the more proper is to verify the properties of the coating deposited on the threat or under the bolt's head. For this application more suitable is Schatz Analyse system that allows the determination of torque, clamping force and rotating angle as well as thread friction and head friction in bolted joints. The parameters are measured under realistic conditions. The system calculates friction coefficients and enables determination of the correlation between torque and clamping force. The test are conducted according to ISO 16047 standard and simulates real conditions of screws assembling. To improve the hot-dip zinc coating properties also the heat treatment is proposed [8,9]. In the literature only a few works regarding this topic is presented but unfortunately the given heat treatment parameters are incomplete. So, the aim of presented investigation was to determine the influence of the heat treatment on the microstructure, hardness level and friction coefficient of the hot-dip zinc galvanized bolts M12-60 to size up the range of HT parameters for further research work.

## 2. EXPERIMENTAL

### 2.1. Methods of investigation

The assessment of the heat treatment impact on the properties of steel elements - M12-60 bolts, covered with the hot-dip zinc coating were determined on the basis of an microstructure analysis of samples conducted using the optical microscope (Axiovert 100 A), the measurement of the microhardness changes at the cross section of both: coating and subsurface layer of steel (Vicker's HV 0.02, Mitutoyo Micro-Vickers HM-210A device 810-401 D; Brinell's HBW method, Innovatest universal tester 700M) and the examinations of tribological properties (vertical Schatz Analyse M12 system, type 5413-2777-03 C - laboratory of S.F. BISPOL S.A, enabling to perform inspections of threads with size diameter from M5 to M12).

### 2.2. Sample preparation technique

During the experiment M12 - 60 bolts and flat samples made of 23MnB4 steel (0.2-0.25 %C; max 0.3 %Si; 0.9-1.2 %Mn; max 0.025 %P; max 0.025 %S; max 0.3 %Cr; max 0.25 %Cu; max 0.005 %Ni) and dimensions ∅25x4 mm, made in the 8.8 strength class were used. Samples were hot-dip galvanized according to PN-EN ISO 10684 [10] - etching in 12% HCl, fluxing, dipping in Zn bath with Al, Bi, Ni; in temp. 460 °C, within time 1.5 min, cooling in water. Next samples were subjected to the controlled heat treatment, in temperature range: 200 ÷ 530 ºC (in addition screws were subjected to processing in temperature 300 and 430 °C). This process was carried out in an electric chamber furnace. The time of treatment was 7 minutes for samples and 11 minutes for bolts. After the heat treatment samples were taken out of the furnace chamber and were cooled in the air, to the environmental temperature. Results achieved for ∅25x4 mm samples will be analyzed separately. Before tribological and metallographic examinations the zinc coating thickness was measured using electronic PosiTector 6000 tester (method of the magnetic induction, head with the diameter Ø = 9 mm). The thickness of the coating on the bolts head was every time included in range 80 ÷ 110 µm. Next samples were tested tribologically using the Schatz Analyse M12 testing machine system, type 5413-2777-03 C, the friction coefficient was measured according to ISO 16047 standard. Next, the samples from bolt head were cut for metallographic observations.

## 3. ANALYSIS OF RESULTS

### 3.1. Metallographic observations and micro-hardness distribution

Observation of the microstructure at the cross section of the coating and the subsurface steel layer was conducted using the magnifications: 200 ÷ 1000x. Three samples were being compared: crude (0), after processing in temperature 300 °C (1) and after processing in temperature 430 °C (2). **Figure 2** is presenting an example of microstructure observed at the steel bolt cross section of the head (0, 2).During microscopic analysis, at magnification 1000x also a thickness of the coating was verified.

a b

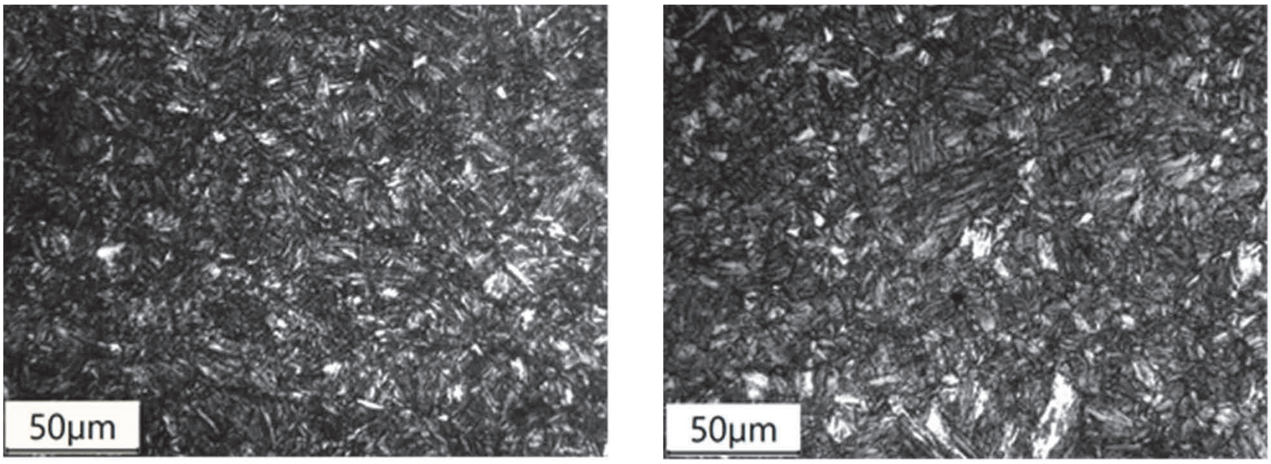



**Figure 2** Microstructure observed in the bolt head, etched with 4%$HNO_3$; a - sample without heat treatment, b - sample treated in 430 °C

a b

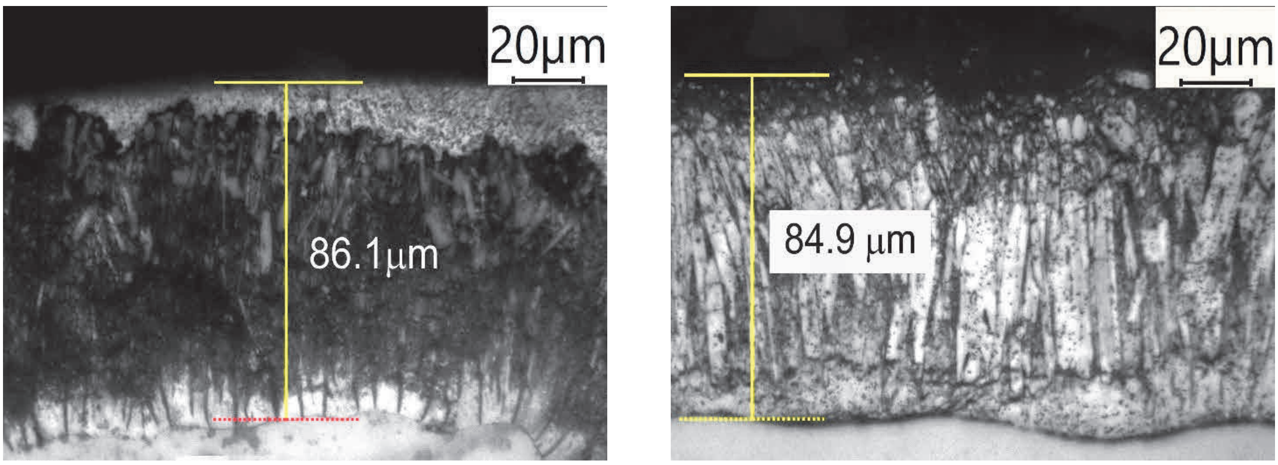



**Figure 3** Microstructure observed at the cross section of zinc coating deposited on the bolt head, etched with 4%$HNO_3$; a - sample without heat treatment, b - sample treated in 300 °C

Additionally the microstructure observed at the zinc coating cross section is presented in **Figure 3.** Tested M12-60 bolts are made in the 8.8 strength class. So, to achieve such high mechanical properties the heat treatment of steel is necessary. Bolts were hold in temperature of 900 °C, cooled dawn in oil and tempered. The structure achieved in crude bolt without heat treatment is typical feather-like, where the length of precipitations is in the range of 10-14 μm - **Figure 2a**. After the heat treatment the structure admittedly is keeping its character, but dimensions of precipitations after processing in temperature 430 °C increased to 20 μm. There are also visible change in zinc coating structure. The coating without heat treatment shows structure composed of four phases, whereas the structure after heat treatment reveals practically two phases.

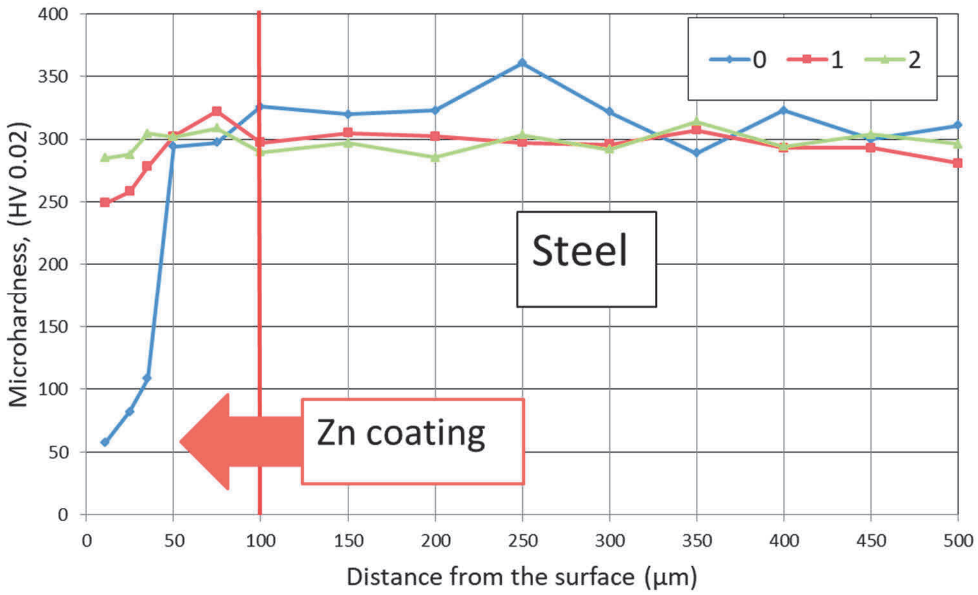



**Figure 4** Microhardness distribution at the cross section of zinc coating and subsurface layer of steel; 0 - without heat treatment, 1 - HT in 300 °C; 2 - HT in 430 °C

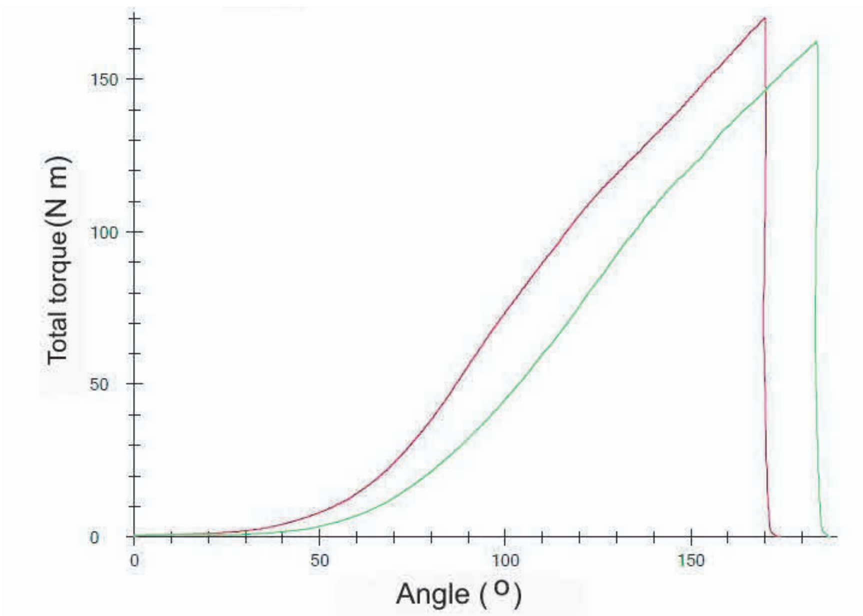



**Figure 5** An example of the graph registered during the friction coefficient measurement using Schatz Analyse M12 testing machine - sample no. 2 - HT in 300 °C

The observed structure corresponds well with measured micro-hardness distribution at the coating and subsurface steel layer cross section - **Figure 4.** It is clear that as a result of heat treatment the outside coating hardness essentially increases from 58 (sample 0) to 250 (sample 1) and 285 HV 0.02 (sample 2). Unfortunately, hardness of subsurface steel layer slightly decreases from 320 to 300 and 290 HV 0.02. The measurement of HBW hardness in depth of 2 mm confirmed hardness decreasing correspondingly from 271 to 255 and 245 HBW. There is no delamination, cracks and surface degradation visible as the result of conducted heat treatment. Considering that the target hardness increase is on the level of 100 HV 0.02 the applied parameters, both temperature and time looks to be too high. The achieved relatively high hardness level can results in too high brittleness of zinc coating. This problem will be further investigated during typical pin-on-disk tribological tests.

### 3.2. Friction coefficient measurements

Typical graph registered during friction coefficient measurements using Schatz Analyse M12 testing machine system, type 5413-2777-03 C is presented in **Figure 5**, whereas the achieved results are listed in **Table 1**. During the test the following parameters were used: abutting diameter - 15.55 mm, rate - 10 per minute, sensor T/Ang : T - 200 N m - 1033595, sensor F/Tth : F - 100 kN - 150 N m. The following coefficients were measured:$\mu_b$ - head friction coefficient, $\mu_{th}$ - thread friction coefficient, $\mu_{tot}$ - overall friction coefficient.

**Table 1** Results achieved during friction coefficient measurements

| Sample No. | F (kN) | T (N m) | $T_b$ (N m) | $T_{th}$ (N m) | $\mu_b$ | $\mu_{th}$ | $\mu_{tot}$ |
|---|---|---|---|---|---|---|---|
| 1.1 | 36.69 | 169.54 | 122.93 | 46.61 | 0.43 | 0.16 | 0.31 |
| 1.2 | 36.70 | 161.57 | 102.12 | 59.45 | 0.36 | 0.21 | 0.29 |
| 1 average | 36.69 | 165.55 | 112.52 | 53.03 | 0.39 | 0.19 | 0.30 |
| 2.1 | 36.69 | 130.87 | 78.31 | 52.56 | 0.27 | 0.18 | 0.23 |
| 2.1 | 36.69 | 139.53 | 91.78 | 47.74 | 0.32 | 0.16 | 0.25 |
| 2 average | 36.69 | 135.20 | 85.05 | 50.15 | 0.30 | 0.17 | 0.24 |

The achieved results - **Table 1**, confirm that the heat treatment influences the friction coefficient of zinc coating. Both values $\mu_b$ and $\mu_{th}$ are lower after the heat treatment in 430 °C than after heat treatment in 300 °C. So, it gives the total friction coefficient correspondingly at the level: $\mu_{tot}$ = 0.30 (300 °C) and $\mu_{tot}$ = 0.24 (430 °C). This changes tendency can follow from coating hardness increase as well as from surface quality changes (smoothness, degree of surface development, etc.). Nevertheless, the measured values of friction coefficient are similar to results achieved using the traditional method - pin on disk - **Table 2**, where measurements were being conducted on the head of zinc galvanized screws [11].

**Table 2** Friction coefficient measured by authors using pin-on-disk method [11]

| Kind of process | | Layer thickness (µm) | | Friction coefficient, µ | |
|---|---|---|---|---|---|
| | | steel | cast iron | steel | cast iron |
| 1 | Galvanic | 18.9 | 20.2 | 0.201 | 0.212 |
| 2 | Hot-dip | 87.8 | 82.1 | 0.223 | 0.285 |
| 3 | Lamellar | 10.5 | 9.8 | 0.122 | 0.131 |

It is necessary to emphasize the fact that the zinc coating thickness deposited in the thread area is much lower than this one put on the bolts head. Considering coating properties diversification (head, thread) to proper bolt properties evaluation very useful is two tests application - standard pin-on-disk tribometer that enables also wear mechanism analysis and Schatz Analyse system that allows bolts characterize under realistic conditions.

## 4. CONCLUSIONS

The conducted examinations enabled to express the following conclusions:

1) The conducted heat treatment (300 - 430 °C, t = 11 min), beside influence on the structure and properties of hot-dip zinc coating also exerts the impact on structure and properties of bolt's material. So, considering the proper heat treatment parameters (time and temperature) also the change of steel structure should be taken into consideration.
2) The observed zinc coating structure changes correspond well with measured micro-hardness distribution at the coating and subsurface steel layer cross section. It is clear that as a result of heat treatment the outside coating hardness essentially increases from 58 (sample 0) to 250 (sample 1) and 285 HV 0.02 (sample 2). Whereas hardness of subsurface steel layer slightly decrease from 320 to 300 and 290 HV 0.02.
3) Using the Schatz Analyse system it is quite easy to evaluate the heat treatment influences on the friction coefficient of zinc coating. Both values $\mu_b$ and $\mu_{th}$ are lower after the heat treatment in 430 °C than after heat treatment in 300 °C. This parameter value knowledge is especially important during automatic assembly.
4) To proper bolt properties evaluation very useful is two tests application - standard pin-on-disk tribometer that enables also wear mechanism analysis and Schatz Analyse system that allows bolts technical description characterize under realistic conditions.

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