Research of micro and nano profile surface morphology of vibrational mechanical and chemical zinc coating

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Abstract. There are some results of zinc coating research acquired on the metals' surface as a result of mechanical and chemical synthesis, realized in conditions of vibro wave technological systems. The peculiarities of the surface morphology at the micro / nano level of the zinc coating are shown which are formed under the influence of low-frequency oscillations. The growth interconnection of the micro / nano profile of the applied zinc coating is shown. The determination of changes occurring on the coating layer surface at the micro / nano level. The absence of reliable data of the surface layer of the coating at the nano level has significantly limited its modification background. All of the mentioned above brings up to the following task, the given article is devoted to its solution: establishment of particles dimension, the way of their distribution on the metal surface, besides their maximal and minimal roughness data cause by the alteration of the processing time.

1 Introduction

The mechanism of zinc coatings formation is characterized by the presence of double electric layer with a fixed equal potential between metal and solution. The restoring of zinc ions (Zn^{++}) on a metal is possible in case of shift of potential concerning equal notion to the negative area [1,6,8]. In the conditions of dynamic pressure, activation of metal surface, ions, atoms, molecules is sharply increased, together with all constituents responsible for the coating formation.

Within the activation process molecules and atoms acquire additional energy which enables to overcome potential barrier to influence speed of chemical reactions. The essence of activation process for VMCP (here and after referred to as vibro mechanical and chemical processing) is to provide material surface with catalytic characteristics corresponding the reaction of zinc ions restoring [2, 3, 4, 5]. Zinc ions concentration increase in the boundary layer is facilitated by the working environment circulation, characterized by speed increase of discharging ions to the metal surface.

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VMCZC (here and after referred to as vibro mechanical and chemical zinc coating) spreading occurs during increased activation and concentration of the ingredients in the boundary layer, the electrode potential in comparison with the equal one is shifted to the data that enable zinc ion restoring reaction [11, 12, 13, 17].

2 Research results

Experimental research was implemented on vibration equipment UVG 4-10 with a tank volume 10 liters. Porcelain and glass spheres with diameter of 5-10 mm were used as working environment. The research was implemented under various technological conditions and vibration regimes: the oscillation amplitude varied from 2 to 5 mm, the oscillation frequency varied from 16 to 33 Hz. Steel samples of the following made (St 3, St 20, St 40), surface area 20 cm² were placed into the container with capacity of 10 liters. For zinc coating as the main working solution (250 g / l zinc chloride and zinc powder 100 g / l) is applied [7, 9, 10, 18].

The influence of vibration processing of the working environment thickness on the zinc coating formation was determined by the power of immediate impacts of porcelain balls strike.

The balls collision with the processed parts surface is accompanied by the prints formation of a definite square. These samples of carbon steel with a working surface of 20 cm² were placed into tank with capacity 10 liters. Tank loading with these balls while applying zinc coating comprised 50% and 80% [14,15].

The micro / nano-profile research of VMCZC allows to obtain new data of the coating surface. The acquired research results of the surface coating at macro / micro level with the usage of optical microscopy increase up to 500 times failed to show the overall crystals' size of zinc nucleus, their outlook, inter crystal bonds location on the metal surface. These facts restrained the given direction development. AFM usage provides with the chance of changes evaluation occurring with coating surface so that processing regime might be altered. The initial sample St.3. Two types of coating were studied: made for 30 min. and 60 min. of processing with amplitude $A = 3$ mm and frequency $f = 35$ Hz. During the first stage 30 min. coating surface was studied (square 19x19μm). Within this period light contrast coating patters (zinc crystals) of different size have appeared.

Surface morphology study was carried out on nuclear-powered microscope Nanoeducator of NT-MDT firm [16]. Each sample scanning occupied approximately 45 minutes. The scanning speed for resolution of 19×19 μm where direct and reverse is equal to 1.6 and 0.4 μm / s correspondently. The coating area under research sized 19x19 μm is a surface with forming zinc crystals where two large crystals are clearly visible on 3D-visualization (fig. 1).

Fig. 1. 3D-visualisation of the surface VMCZC square 19x19 μm, time T = 30 min.

The micro profile of the coating front part shown in fig. 2. The light areas of the coating surface show location of 2 large zinc coating crystals and a dent between them. Some of them have an irregular oval shape sized of 5x6 μm and other flat shape with the lengths ranging from 1 μm up to 4 μm .

Fig. 2. Front surface image VMCZC square 19x19 μm , time T = 30 min.

Particular attention is attracted by the dent between two large crystals. That was the reason why the place to make the profilogram was chosen to see the surface profile between the crystals (fig. 3) and determine the nature of the inter crystalline compound. The dent width is 7 μm with 7 crystal peaks at the bottom sized 50-100 nm with width 0,5 μm whilst huge crystals sized Rp max 700 nm min – 600 nm width 11 μm . In such a way it is possible to determine the coating integrity and its formation due to large and small crystals.

Fig. 3. Front image and profile VMCZC, time T = 30 min., square 19 x 19 μm .

As a result of VMCZC coating within 60 minutes the picture changes. The micro configuration of this place is shown in fig. 4. Light areas of the surface indicate crystals distribution of formed coating on the sample surface.

Fig. 4. Surface image of VMCZC square 9x9 μm, time T = 60 min.

On the square 9x9 μm 14 crystals of the formed coating sized from 1 μm to 4 μm are clearly visible. 3D-image of the vibrational mechanical and chemical coating (fig. 5) makes it possible to estimate height and distribution of the zinc coating crystals over the sample surface. It can be seen there are some crystals on the surface almost identical in shape and size, which is completely unlike the coating made within 30 minutes.

Fig. 5. 3D-visualisation of the VMCZC surface square 9x9 μm.

For a further study of the surface locally the definition Rp is stated within the length of 9 μm for estimation. Rp of shattered area (lower left corner in fig. 6) is 0.02 μm while Rp of other area is 0.06 μm. Profile measurement is done in exceptionally narrow range of the evaluation with length of 9 μm. Rp ratio difference is almost to 3: 1 which shows high accuracy of measurement.

Fig. 6. Sample micro profile with VMCZCT = 60 min; a-profile of the horizontal area of the coating surface R_p 660 nm; b – profile of the vertical area of the coating surface. $R_p = 1,1 \mu\text{m}$.

The maximum peak height of the vertical area profile R_p is 0.65 to 1.05 μm . The horizontal area of the surface coating R_p is from 420 to 660 nm. Comparing the areas of the original sample and the coated one, it can be stated that R_p parameters are practically not changed however R_z and R_a have slightly decreased, which is clearly visible on the profile.

Morphology studies and visual surface analysis of VMCZC described above did not provide any explanation concerning coating profile nature and its parameters change. In this connection nano profile study of the coating surface that allows to determine the changes occurring at nano level with samples surface contribute to the general picture of VMCZC formation. Fig. 7 demonstrates the results of study that 3D-visualization of the profile surface of a steel sample with a vibrational mechanical and chemical zinc coating formed for 20; 40; 60 min.

Fig. 7. Nanorelif of VMCZC samples; a – (Tprocess. = 20 min.); b – (Tprocess. = 40 min.); c – (Tprocess. = 60 min.), square 200 x 200 nm.

In the process of comparing 3D visualization of the original surface profile without coating and after VMCZC coating it can be seen that after 20 minutes processing (fig. 7a), coating crystals are formed on the surface that are quite distinct on 3D-visualization shaped as sharp peaks with the size from 2 to 5 nm. The maximum peaks' length is 55.5 nm, minimum one is 35 nm.

Further coating has significantly changed nano profile surface (fig. 7.b) after 40 minutes of processing initial structures gave the way to the formation of larger structures with a height of -39 nm up to -49.2 nm and length up to 50 nm, followed by increased density of the coating. The increase of processing time up to 60 min. (fig. 7c) has led to the formation of periodic regularly repeated structures of the zinc coating sized up to 200 nm on the sample surface. The height has changed from -58.3 nm up to -45.5 nm.

Together with that the following changes are observed: the coating thickness and certain roughness of the surface layer decrease due to tops' deformation and radius of rounding increase of nano inequality peaks. Profile and morphology study of the surface allowed to establish that within mechanical and chemical zinc coating occur a change of both dents depth and the parameters of micro / nano profile of the surface layer. Together with that the following changes are observed: the coating thickness and certain roughness of the surface

layer decrease due to tops' deformation and radius of rounding increase of nano inequality peaks.

Conclusions

1. Zinc coating made under conditions of vibrowave technological systems obtains a number of advantages, such as the absence of a hydrogenated layer, low void structure, high corrosive resistance.
2. The technology of coating does not require special equipment, premises, highly qualified specialists, special equipment for waste purification and recycling.
3. The given method appliance of zinc coatings is reasonable for parts without deep holes at large machine-building enterprises, vehicle enterprises and technical maintenance service.

References

1. A.P. Babichev, *Fundamentals of Vibration Technology. Part I* (DSTU, Rostov-on-Don, 1993)
2. V. Ivanov, A. Babichev, N. Pogorelov, MATEC Web of Conferences, **132**, 01004 (2017)
3. V.V. Ivanov, V. Ju.Valyavin, S.I. Popov, Ju.V. Marchenko, E.V. Marchenko Worldof Galvanics, 1 (29), 34 (2015)
4. V.V. Ivanov, S.I. Popov, A.V. Kirichek, IOP Conference Series: Materials Science and Engineering electronic resource, 032026 (2018)
5. V.A. Lebedev, V.V. Ivanov, V.P. Fedorov, IOP Conf. Series: Materials Science and Engineering (2016)
6. V.V. Ivanov, N.S. Dontsov, A.V. Kirichek, IOP Conference Series: Materials Science and Engineering electronic resource, 032025, 84 (2018)
7. J. Russell, *Mechanical plating* (Bookvika Publishing, 2012)
8. L.K. Gillespie, *The formation and properties of machining burrs* (Utah State University, Logan, Utah, 1973)
9. W. Nebiolo, J. Gardner Business Media, **78**, 26-9 (2014)
10. V.V. Ivanov, *Vibrational mechanical and chemical methods of surface coating (zinc coating)* monograph (DSTU, Rostov-on-don, 2010)
11. E. Holzknecht, J. Metal Finishing, 107, 27 (2009)
12. D. A. Davidson, J. Metal Finishing, 106, 30-4 (2008)
13. V.P. Smolentsev, *Handbook of technological-engineering* (Moscow, 2000)
14. A.P. Babichev, V.V. Ivanov, Ja. S. Bulgakov, Fundamental and applied problems of equipment and technology, 1, 35 (2011)
15. V.V. Ivanov, M.F. Selemenov, Ju.V. Marchenko, Fundamental and applied problems of equipment and technology, 2, 73 (2011)
16. V.V. Ivanov, N.S. Dontsov, A.V. Kirichek, Key Engineering Materials, 736, 105-9 (2017)
17. V. Ivanov, V. Lebedev, I. Davydova, T. Atoyan, MATEC Web Conf, **132**, 01003 (2017)
18. V.V. Ivanov, S.I. Popov, A.V. Kirichek, Engineering Materials, 736, 18 (2017)