Structure and microhardness of the plasma sprayed composite coatings after combined treatment

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Abstract. The principal aim of this study was to evaluate the effect of combination of electromechanical treatment (EMT) and ultrasonic treatment on structure and microhardness of air plasma sprayed composite coatings from Ni–20Cr alloy and R6M5 high speed steel (HSS). The results of the microstructural studies showed fundamental changes of the treated by the EMT plasma sprayed coating with the formation of nanostructured crystalline phases. As a consequence of the coating thus formed, the number of pores in the coating structure reduced from 10.0±1.5% to 2.0±0.5%, the surface microhardness increased from 3100±500 MPa to 7900±400 MPa. Additional ultrasonic treatment on the selected mode decreased surface waviness, which was formed on the surface of the plasma sprayed composite coatings after the EMT. The obtained results revealed the high potential of the combined treatment for post-treatment of the plasma sprayed coatings.

1. Introduction

There are various post-treatment techniques, for example, laser re-melting, surface induction heating, heat treatment, spark plasma sintering and etc., that are applied to enhance cohesive strength and to increase coating's density [1-4].

Electromechanical treatment of coatings is a modern post-treatment technology. The articles [5-11] show that rolling by the roller of the coating with synchronous heating by the electric current improve coating density, cohesive and adhesive strength, while the advantages of the metastable structure stay the same. The important feature of this method is implementation on the usual metalworking machines. The authors of the articles [5-11] use similar terms to describe the post-treatment of the thermal sprayed coatings. Electric contact strengthening (ECS) was accepted as term by the authors of the articles [5-7]. Coating density and adhesive strength were increased after the ESC of the coatings. The authors of the articles [8-11] adopted the term electromechanical treatment (EMT) for method of post-treatment of the coatings. Improvement of the cohesive, adhesive strength, increase of density were determined in the papers [5-11] after the ECS and the EMT of thermal sprayed coatings. In the articles [9, 11] formation of nanostructured crystalline phases was determined after the EMT of plasma sprayed coatings.
Therefore, in the articles [12, 13] mathematical modeling of these processes was performed. Mathematical modeling showed that the cooling rate in the process of the EMT reached the value of $10^4$ K/s, for this reason, the nanostructured crystalline phases were formed in the structure of the coatings.

Ultrasonic treatment (UT) of bulk materials and coatings is widely used in industry [14, 15]. Also, the important feature of this method is implementation on the usual metalworking machines. Ultrasonic treatment is used to increase the surface quality, i.e., to reduce surface roughness, as well as to relieve residual stresses.

Therefore, the combination of these types of post-treatment of the plasma sprayed coating is a topical way to improve the properties of the plasma sprayed coatings.

The purpose of this study is to estimate the effect of combination of the EMT and the UT on morphology, structure and microhardness of plasma sprayed composition coating.

2. Materials and methods

Two types of powders were used for plasma spraying. Commercial feedstock powder of high speed steel R6M5 (chemical composition of 1.00 – 1.04 wt.% C, 4.00 – 4.07 wt.% Cr, 6.70 – 6.71 wt.% W, 5.55 – 5.57 wt.% Mo, 2.10 – 2.12 wt.% V, 0.50 – 0.55 wt.% Mn, ≤ 0.4 wt.% Si, ≤ 0.03 wt.% S, ≤ 0.03 wt.% O, Fe constituted the balance) was chosen for plasma spraying of the main coating layer. The powder was prepared using atomization method (Metsintez OOO, Tula, Russia). The powder particles had spherical form with mean particle size of 50 – 71 μm. Sublayer and protective layer were plasma sprayed with commercial feedstock powder of Ni–20Cr alloy (chemical composition of 0.04-0.06 wt.% C, 20 wt.% Cr, ≤ 0.1 wt.% Si, ≤ 0.06 wt.% S, ≤ 0.03 wt.% O, Ni constituted the balance). The powder was produced by calcium-hydride method (Metsintez OOO, Tula, Russia). The powder particles had irregular shape and size range of 32 – 50 μm.

Substrates from low-carbon steel were used for plasma spraying of coating and subsequent combination treatment. The sample for determining the effect of the ЕМТ modes was a cylinder 20 mm in diameter and 150 mm in length.

| Track | 1 | 2 | 3 | 4 | 5 | 6 |
|-------|---|---|---|---|---|---|
| Current density, A/mm² | 400 | 400 | 700 | 700 | 1000 | 1000 |
| Linear speed, m/min | 3.39 |
| Feed rate, mm/rev | 0.2 |
| Ultrasonic treatment * | + | + | + |

* - 22 kHz, load 3 H, 3,39 m/min, 0.1 mm/rev

Plasma spray equipment was the air plasma spray system UPU-3D (JSCo "Electromechanica", Rzhev, Russia) with the plasma spray gun PP-25. The maximum power of the plasma spray gun PP-25 is 25 kW.

Plasma spraying was carried out according to a standard protocol [11]. Ni–20Cr powder was plasma sprayed on cylindrical samples as a sublayer to reduce residual stresses. The thickness of the sublayer was 25 ± 5 μm.

Then the plasma spraying of R6M5 powder onto the sublayer as the main coating layer of the composition coating was carried out. The thickness of this layer was 250 ± 25 μm.
As the final stage, the Ni–20Cr powder was plasma sprayed on the surface of the R6M5 coating layer as a protective layer to prevent damage from oxidation. The thickness of the protective layer was 25 ± 5 μm.

The plasma sprayed composite coating thickness was 300 ± 50 μm.

![Schematic diagram of the EMT (a) and the UT (b)](image)

**Figure 1.** Schematic diagram of the EMT (a) and the UT (b)

The installation of BUFO (UTINLAB OOO, St. Petersburg, Russia) was used for the ultrasonic treatment of the plasma sprayed coating after the EMT.

Ultrasonic treatment (UT) was performed in the following mode, the frequency of 22 kHz, the load in the contact zone 3 N, the processing speed of 3.39 m/min, longitudinal feed 0.1 mm/rev, the contact area of the indenter with the coating was 2 mm².

Preparing of the test samples was carried out according to a standard protocol [11].

Structures of the plasma sprayed and treated by the EMT coatings were examined by means of the optical microscope (Carl Zeiss Jenavert Interphako) and the scanning electron microscope (SEM), attached with the EDS (Versa 3D). Measurements of porosity and second phases were made with the image analyzer, having software of VideoTest Structure 4.0 (VideoTest OOO, St. Petersburg, Russia). The coating microhardness was determined in cross-section using the Vickers indenter (PMT-3) at the load of 1.96 N (200 gf), for 15 s. Minimum 15 indentations were applied to each sample.
3. Results and discussion

The results of the EMT showed reduction of roughness of free surface of the plasma sprayed coating. During the EMT process, the roller was indented in the coating, compacting the coating in this way. Due to the fact that part of the material was squeezed out along the roller's edges under pressure, the wavy structure was formed on the surface of the coating. Period of the wavy structure was $200 \pm 25 \, \mu m$ because of the constant feed rate ($0.2 \, mm/rev$).

Optical microscopy showed that the splats had different colors: white and gray (Figure 2, 3). SEM analysis detected nanocrystal phases in the structure of the gray splats [6]. They were formed because of breakdown of solid solution. And, perhaps, the nanocrystal phases were carbides of W, Mo, V, Cr.

SEM analysis showed that after the UT the nanocrystal phases in the structure of the gray splats were saved (Figure 4).

![Figure 2. Macrostructure of cross-section of the plasma sprayed composition coating: a – initial coating, b – coating after the EMT, c – coating after combined treatment.](image)

![Figure 3. Macrostructure of the surface of the coating: a – after the EMT, b – after combined treatment.](image)

The relief surface of the coating, which was formed in the process of the EMT, was smoothed after the UT (Figure 3). The waviness of the coating was reduced after the UT (Figure 2c). Reducing the waviness was due to plastic deformation of the surface layer from the alloy Ni–20Cr (Figure 2c, Figure 3b). The plastic deformation of the surface layer was caused by high-frequency deformation by the indenter in the UT process.

The measurement results of the microhardness of the R6M5 layer are presented in Table 2.
Table 2. Microhardness of the R6M5 layer.

| Track | R6M5  | 1    | 2    | 3    | 4    | 5    | 6    |
|-------|-------|------|------|------|------|------|------|
| Microhardness, MPa | 3100  | 5350 | 5400 | 5650 | 5900 | 7900 | 7900 |
| ±S, MPa | 560   | 450  | 500  | 550  | 450  | 400  | 500  |

The microhardness of the R6M5 layer using a load of 200 gf was 3100±500 MPa. The microhardness of the plasma sprayed coating under a 200 gf load increased from 5300 ±500 MPa to 7900 ± 400 MPa after the combined treatment. Such increase of the microhardness (200 gf) is due to the compaction of the coating, the resistive welding of the boundaries of the splats, and the formation of nanostructured crystalline phases in the splats. The microhardness of the coating after the EMT and the combined treatment within measurement error was the same (Table 2). In our opinion this is due to the low energy of the mode of the UT. The force of deformation with frequency of 22 kHz of the plasma sprayed composition coating led to deformation of the surface layer in the UT process, but microhardness of the basic layer was not changed because of low pressure of the indenter.

Figure 4. SEM image (secondary electrons) of nanocrystal phases in the gray splats after the combined treatment
The UT allows reducing roughness and waviness of the coating’s surface, for this reason, subsequent grinding of the coating may be omitted. Important feature of the combined treatment of plasma sprayed coating is realization of the EMT and the UT on the usual metalworking machines in one step.

4. Conclusion
1. After the combined treatment the microhardness of the plasma sprayed coating under a 200 gf load increased from $3100 \pm 500$ MPa to $79\,000 \pm 400$ MPa.
2. The nanocrystal phases in the structure of the gray splats were saved after the UT.
3. In this investigation, the main effect of the UT is to reduce the surface waviness that should improve operational properties of the coating

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