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DEVELOPMENT OF TECHNOLOGY OF MICROPLASMA SPRAYING FOR RESTORATION OF LOCAL DAMAGES OF ENAMEL COATING

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Fundamentals of microplasma spraying technology for restoration of local damages in enamel coatings of tank equipment were developed. A double-layer repair coating consisting of a layer of zirconium oxide and tantalum sublayer was proposed. The optimum parameters of microplasma spaying mode were determined in order to obtain a layer of zirconium oxide with dense microstructure (porosity 1.2–1.9 %). It is shown that application of tantalum-based sublayer increases adhesion strength of zirconium oxide coating by 25 % (up to 8.14±2.16 MPa). Evaluation of coating through porosity was carried out. A method was proposed for increase of coating uniformity using epoxy-resin treatment. The technology was approved under real production conditions at CJSC «Kharkovreakhim». 15 Ref., 1 Table, 6 Figures.

Keywords: *microplasma spraying, enamel coatings, restoration, adhesion strength, zirconium oxide, tantalum, coating permeability*

Local damages of the enamel coatings appear under effect of a complex of service conditions and, mostly, at the presence of initial production defect. In chemical industry appearance of the defects of enamel coatings is related with effect of aggressive chemical media (acids, alkalies, salts), high temperatures (as well as rapid changes of temperatures) and mechanical damages due to movement of equipment parts. Thus, the main defects of enameled chemical devices and reservoirs are chips, cracks and defects of corrosion nature [1].

The traditional methods of restoration of such local damages, described in work [2], include additional enameling, putting special seals of gold or tantalum, using of polymer fillings as well as reenameling. However, mentioned methods have a series of disadvantages such as need in reannealing of the whole product or its part, expensive materials, accelerated aging of polymers in the aggressive media and necessity in complete removal of a present enamel coating for new layer deposition.

In recent times, repair of damaged areas of the enamel coatings is mostly carried out using thermal spraying methods (TS), which can provide local repair with high process efficiency in restoration of complex shape surfaces [3–13]. It is noted that combination of electric arc, flame and plasma coatings with enamel surface layers allows 5 times extension of service life of enameled equipment.

However, application of traditional methods of plasma and flame spraying imposes a series of limitations, which do not allow sound, and effective repair of the enameled equipment under production conditions. Thus, for example, repair of inner surfaces of the reservoirs using traditional plasma spraying units is impossible due to their large dimensions.

In this connection it is proposed to use a technology of microplasma spraying (MPS), developed at the E.O. Paton Electric Welding Institute of the NAS of Ukraine for repair of the enamel coatings on equipment of chemical enterprises. It has a series of significant advantages in comparison with traditional plasma spraying [14] for solving set tasks:

- low plasmatron power (to 2 kW) eliminates the possibility of product overheating and damage of enamel coating around area being repaired;
- small size of spraying spot (1–5 mm) provides the possibility of precision repair of small defects with minimum losses of material being sprayed;
- mode of plasma jet outflow from microplasmatron provides a low noise level (less than 50 dB) that allows spraying without special chambers with minimum protective means for operator;
- small dimensions of equipment for microsplasma spraying allow local repair of the enamel coatings on the products without their disassembly as well as inside reservoirs of large dimensions.

Convenience of equipment and geometry accuracy of coating deposition provide a prompt repair of the enamel coatings at early stages of defect formation that is, apparently, a preferable approach.

Determination of requirements to repair microplasma coatings and materials selection. The analysis of existing experience to repair of local defects using TS methods determined the main requirements to repair of the microplasma coatings:

- material of the coating should provide resistance to effect of environment at the level equal to strength of working enamel coating;
- coating should have a layer density which does not allow contact of environment with a base metal surface (product, structure);
- coating strength should be sufficient for adhesion with the base and its keeping on the surface under operation of product or structure.

The following materials, suitable for repair coatings, namely oxide (ZrO_2, Al_2O_3, TiO_2) and metallic (Zr, Ta, Ti) were selected based on considered requirements taking into account available experience of MPS of different materials.

Analysis of the works, dedicated to deposition of repair thermal coatings, allows assuming that the most effective approach for repair of local damages of the enamel coatings will be application of the double-layer metal-ceramic coatings.

The aims of work were generated based on mentioned above:

- determination of parameters for powder microplasma spraying of ZrO_2 coating with minimum porosity for restoration of local damages of the enamel coatings;
- determination of effect of tantalum sublayer on adhesion strength of ceramic coating with the base;
- evaluation of through porosity (evaluation of uniformity) of coatings.

Procedure of experiment and investigations. *Materials.* Zirconium oxide powder (ZrO_2 + 7 % Y_2O_3) of 40 μm granulation was used as a material being sprayed. Figure 1 shows appearance of zirconium oxide powder. The coatings were deposited on the specimens of steel 40 of 10×20×3 mm size subjected to gas-abrasive treatment (corundum).

Equipment. The coatings were produced on microplasma spraying unit MPN-004 with MP-04 plasmatron in powder spraying mode [14].

Procedure of experiment planning. The method of multifactor experiment with half-replication 2^{4-1} was selected in planning. Current (I, A), consumption of plasma-forming gas (V_{pg}, l/h), spraying distance (h, mm) and powder consumption (G_p, g/min) were selected as independent variables (factors). A response function was a value of coating porosity in %.

The values of mode parameters (Table) were taken based on the results of preliminary experiments and accumulated practical experience of plasma spraying of zirconium oxide on MPN-004 unit.

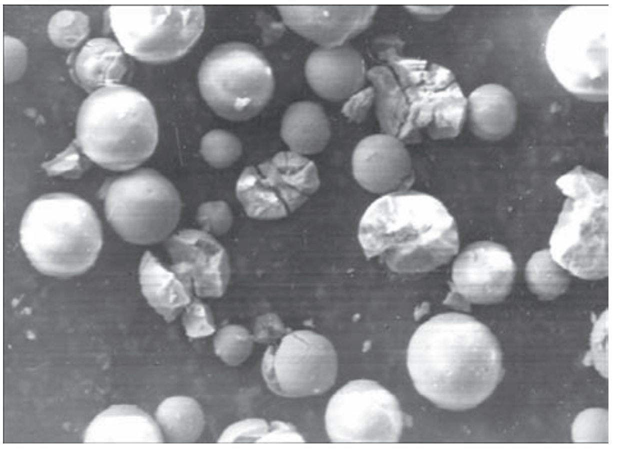

Figure 1. Appearance of zirconium oxide powder (ZrO_2 + 7 % Y_2O_3, size ≈ 40 μm)

Investigation procedures. Preparation of the specimens for metallographic investigations was carried out in accordance with the standard procedures used in metallography. The sections were firstly made on waterproof polish paper SiC with P800, P1200 grain size, after that on the flexible disks containing diamond inclusions of different dispersion (A28/14, A14/10 and A5/3). The final polishing of the sections was carried out on woolen cloth adding diamond suspension with DiaDuo lubricant having diamond particles of 3 μm size. Coating microstructure was examined on «Neophot-32» microscope equipped with add-on device for digital photo. A system of image registration was carried out with a computer program QuickPhoto.

Determination of porosity. Optical methods of porosity determination (method of image analysis) have found the widest distribution for qualitative and quantitative analysis of porosity geometry. They lie in determination of an area covered with found pores relative to whole area of coating section.

Analysis of the sections was carried out using «Neophot-32» and Jenavert devices. A digital image was processed with Atlas program, which allows measuring porosity (outlining inclusions different on color and brightness) on standard ASTM B-276, determine

Parameters of mode of microplasma spraying of zirconium oxide powder

Number of mode	I, A	V_{pg}, l/min	h, mm	G_p, g/min
1	45	2.0	160	2.0
2	45	2.0	80	1.0
3	45	1.0	160	1.0
4	45	1.0	80	2.0
5	35	2.0	160	1.0
6	35	2.0	80	2.0
7	35	1.0	160	2.0
8	35	1.0	80	1.0
9	40	1.5	120	0

dimensions of maximum and minimum pores, number and percent relationship of pores over the area.

Measurement of adhesion strength of coatings with base at tear was carried out on a glue procedure according to GOST-14760–69 and ASTM C 633–76 using versatile servohydraulic machine MTS-318.25 (USA production) with maximum force 250 kN under normal conditions (t = 20 °C). A grip movement rate was 0.17 mm/s. The coating was deposited on edge surface of cylinder specimens of 25 mm diameter and 24 mm height, the specimen with coating was glued to a check specimen and pressed with 0.5 kg/cm^2 force. Epoxypolyurethane glue EPU-TEKhKO with 65 % of epoxy resin and 35 % of hardener was used for gluing.

Evaluation of through porosity(uniformity evaluation) was carried out by means of evaluation of their permeability in a model medium (tap water). The segments of enameled pipes of low-carbon steel with two inches inner diameter were cut for specimens manufacture. A through defect of enamel coating of 10 mm diameter was formed at a face side by the gas-abrasive treatment method. Then the defect was removed using microplasma spraying of zirconium oxide coating of 150 μm thickness. The edges and backsides of the specimen were isolated with multiple application of lacquer. Visual monitoring of appearance of spot exits of corrosion material via through pore channels was performed in course of the experiments.

Results and discussion. Figure 2 shows the microstructures of ZrO_2-coatings produced under MPS conditions. The results of measurement of ZrO_2-coating porosity are given in Figure 3.

The coatings with the densest microstructure and tight contact with the base are formed at I = 40–45 A (experiments 1, 3, 4) values. Reduction of current to 35 A value results in decrease of coating thickness and porosity growth.

Porosity in the experiment rises in the following way, i.e. experiment 3-4-1-9-8-2-6.

Thus, the results of carried analysis of coating structures determined that ZrO_2-coatings with minimum porosity are produced using the mode No.3 (Table), namely I = 45 A, V_{pg} = 1 l/min, h = 160 mm, G_p = 1 g/min.

Coating to base adhesion strength. The investigations of adhesion strength of coatings with base showed that deposition of zirconium oxide coating directly on specimens of steel 40 subjected to gas-abrasive treatment promotes an average adhesion strength 6.11±1.32 MPa during tear testing at coating thickness 150–200 μm. Adhesion strength rises to

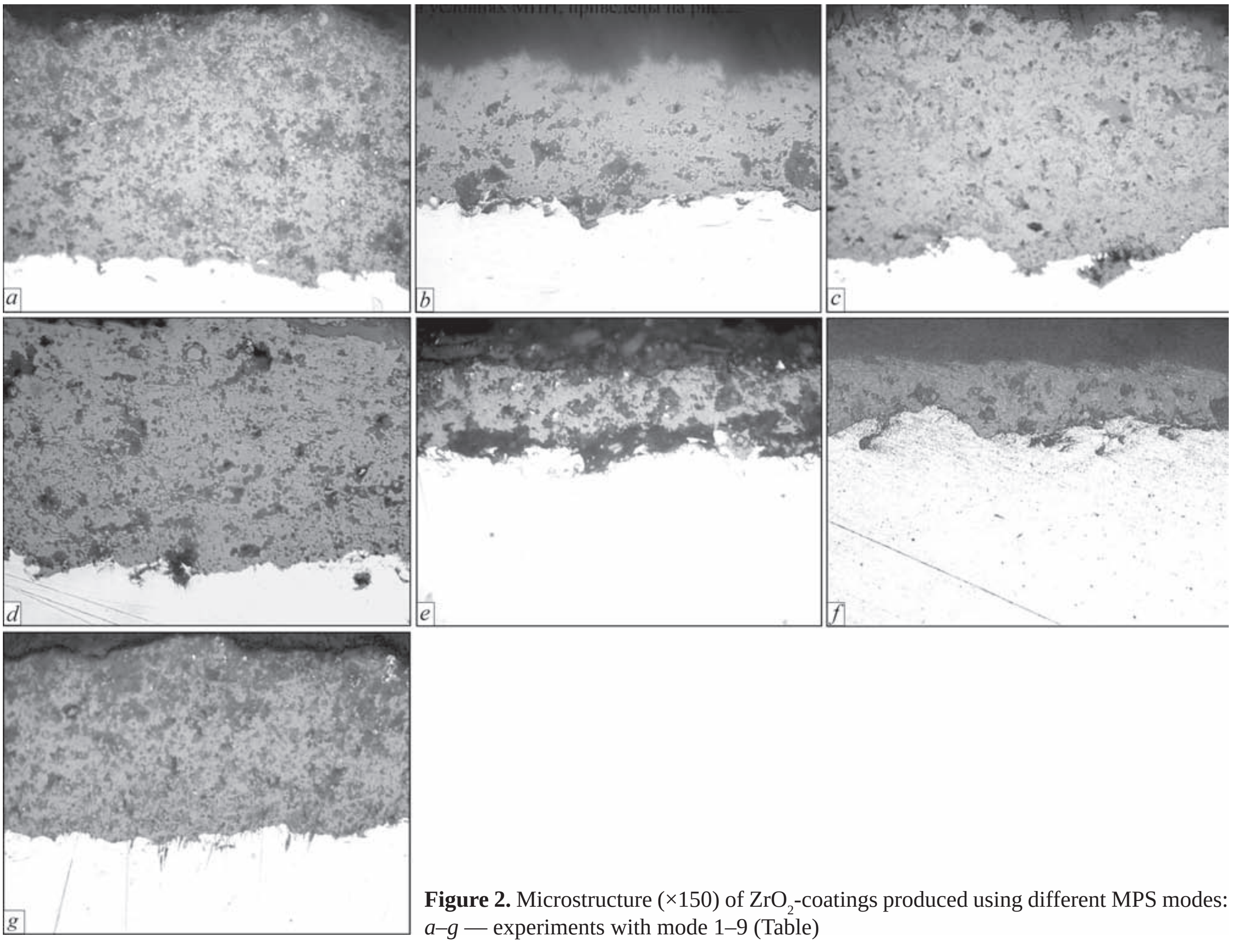

Figure 2. Microstructure (×150) of ZrO_2-coatings produced using different MPS modes: *a*–*g* — experiments with mode 1–9 (Table)

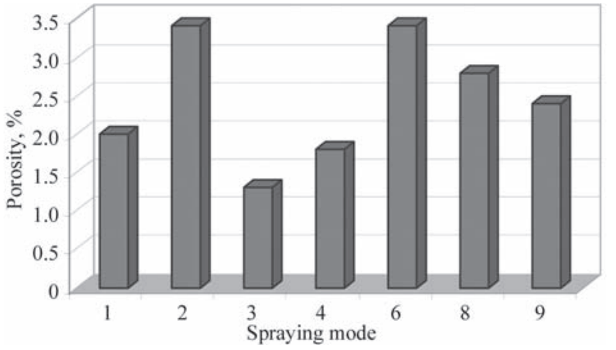

Figure 3. Porosity of microplasma coatings of ZrO_2

8.14±2.16 MPa, i.e. increment of adhesion strength makes 25 %, in deposition of ZrO_2-coating on tantalum sublayer (Figure 4).

Conditions for decrease of surface ripples. In process of coating deposition on the area, which is certainly more than a spraying spot, there is linear movement of the plasmatron in combination with part rotation or transverse displasement of part or plasmatron. At unmovable position of plasmatron and part the material being sprayed forms a bead shape coating on the surface, cross-section of which is described by Gaussian curve:

$$y = y_0 e^{-(r^2/r_0^2)}, \qquad (1)$$

where y_0 is the thickness of the coating on bead axis; r_0 is the scattering radius; r is the distance from bead axis.

It follows from equation (1) that the best uniformity of coating thickness in microplasma spraying is achieved under condition of transverse displacement s of the plasmatron under the next conditions:

$$s = 1.2 r_0. \qquad (2)$$

Spraying of the specimen for measurement of the spraying spot was carried out using the optimum mode for deposition of repair ZrO_2-coating (mode No.3). A scattering radius for given specimen made 2.75 mm on big axis and 2.15 mm on small axis.

It was determined based on the results of analysis of calculation data and conditions of coating uniformity (s/r_0 = 1.2) that transverse displacement of the plasmatron during movement along large diagonal in ZrO_2-coating deposition should not exceed 4 mm and in displacement along smaller diagonal it makes 3 mm. Less than 1 % of ripples is reached under these dimensions of step of plasmatron transverse movement.

Evaluation of coating permeability for outdoor environments. *Impregnation of coatings for sealing.* Carried metallographic examinations of the specimen showed that the coating is tight with the base, including enamel coating (Figure 4).

Appearance of spot exits of rust was noted in the investigated specimens with ZrO_2-coating. In this connection the coating was impregnated with special compound for sealing the coating microdefects. Dichtol (Germany) is a one-component fluid material based on

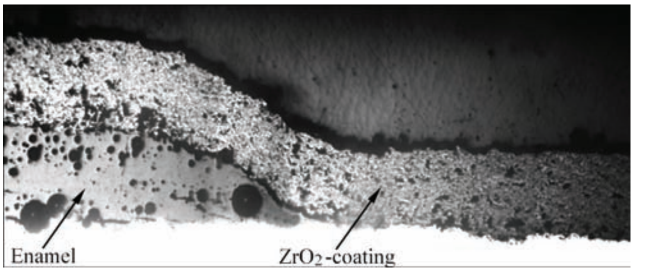

Figure 4. Microstructure (×150) of ZrO_2-coating produced with MPS on specimen simulating enamel coating defect

epoxy-resin, which was used as a compound for impregnation. It is characterized with the possibility to penetrate in the cavities of casting and coating microdefects. After ZrO_2-coating was sprayed on the test specimens a triple impregnation of the coating by Dichtol with intermediate drying was performed. The specimens received after impregnation did not show spot corrosion under the similar test conditions (Figure 5).

Received results allowed determining process parameters and developing the following technological recommendations on restoration of the local damages of enamel coatings using microplasma spraying method:

- preparation of product surface include gas-abrasive treatment (corundum) and degreasing with organic solvents;
- parameters of spraying mode: I = 45 A; V_{pg} = 1 l/min; h = 160 mm; G_p = 1 g/min;
- transverse displacement of the plasmatron in movement along larger diagonal should not exceed 4 mm and 3 mm in movement along smaller diagonal;
- after spraying the coating is subjected to triple impregnation by Dichtol (Germany), one-component fluid material based on epoxy-resin, with intermediate drying during 1.5 h.

Approbation of the developed technology of coating restoration using microplasma spraying method was carried out under production conditions for repair of the local damages of enamel coating on inner surface of V-630 reactor, which is used in produc-

Figure 5. Specimen with defect of enamel coating after restoration using MPS method and uniformity testing

Figure 6. Process of restoration of local damages of enamel coating of V-630 reactor using microplasma spraying method under production conditions

tion of medical products at CJSC «Kharkovreachim» (Kharkov) (Figure 6).

Currently, the reactor restored using MPS method is under testing.

Further realization of the developed technology will be carried out in cooperation with LLC «Emal-service», the leading enterprise in Ukraine on repair and restoration of enameled equipment.

Conclusions

1. The requirements were determined to plasma coatings designed for repair of damaged enamel layer, and selection of the spraying materials for such coatings was performed. Tantalum was taken as a sublayer material and zirconium oxide was a base layer material.

2. It is determined that ZrO_2-coating with the densest microstructure (porosity 1.2–1.9 %) is formed at increased values of current I = 40–45 A. The lowest porosity equal 1.2 % is observed in the coating produced using I = 45 A; V_{pg} = 1 l/min; h = 160 mm; G_p = = 1 g/min mode.

3. It is shown that application of tantalum-based sublayer allows increasing adhesion strength of zirconium based coating by 25 % (in absolute values from 6.11±1.32 MPa to 8.14±2.16 MPa).

4. Evaluation of through porosity of the coatings by testing the permeability for outdoor environment (water was used as a model medium) was carried out. The method was proposed for increase of coating uniformity by exopy-resin impregnation. Received specimens showed water impermeability.

5. Performed approbation of the developed technology under production conditions showed possibility of application of microplasma spraying technology for repair of local damages of enamel coating including on inner surfaces of reservoirs.

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