Efficiency of surface cleaning by a glow discharge for plasma spraying coating

A M Kadyrmetov[1], N F Kashapov[2], S N Sharifullin[2], A I Saifutdinov[2] and S A Fadeev[2]
[1] Voronezh State Academy of Forestry and Technologies
[2] Kazan Federal University

E-mail: kadyrmetov.a@mail.ru

Abstract. The article presents the results of experimental studies of the quality of cleaning steel surfaces by a glow discharge for plasma spraying. Shows the results of measurements of the angle of surface wetting and bond strength of the plasma coating to the surface treated. The dependence of the influence of the glow discharge power, chamber pressure, distance between the electrodes and the processing time of the surface on cleaning efficiency. Optimal fields of factors is found. It is shown increase joint strength coating and base by 30-80% as a result of cleaning the substrate surface by a glow discharge plasma spraying.

Key words. Glow discharge, cleaning, activation, activation, surface, substrate, experiment, plasma spraying, plasma torch, plasma coating, optimization.

1. Introduction

Preparation of the substrate surface prior to application of plasma coating is one of the most important groups of factors that influence the formation of the coating [1]. The presence of the oxide film on the surface of steel reduces its surface energy and thus prevents the formation of the most complete atomic bonds with the applied coating [2]. For plasma coatings deposited plasma generators in an open and controlled atmosphere, it creates one of the most urgent problems of increasing the strength of the connection of plasma coating to the substrate. Prior to application of such coating a surface prepared in various ways, of which the most commonly used are the chemical surface treatment and sandblasting of the surface. These methods have disadvantages. The chemical surface treatment method is associated with environmental problems. The disadvantage of sandblasting associated with the presence of residues of ultrafine dust embedded in the surface layer. Analysis of available data shows the possibility of overcome these deficiencies and significant increase in bond strength coating to the substrate when it is processed glow discharge [3, 4]. However, presently unknown mechanisms of the processes explained such influence on the strength of the coating to the substrate. Therefore urgent is the definition of the main factors and their impact on the effectiveness of the surface cleaning.

The study aims, firstly, to determine the influence of factors of surface treatment by a glow discharge on the effectiveness of cleaning. Second evaluation of the impact of the quality of cleaning on the strength of the plasma coating to the metal substrate.

2. The experimental technique

The quality of treatment was evaluated by the contact angle of wetting the surface. The angle is measured at the sample surface after its cleaning, filling with argon vacuum chamber to normal pressure, and cooling the sample. For measurements distilled water was used, the droplet size was 2-2.5 mm in diameter. The drop was deposited to the surface in stages: the drop squeezed out of the capillary, and then it is brought to the surface to the ground, then the capillary taps. The angle was measured after 10 minutes.

For surface treatment used glow discharge mode of abnormal combustion. This allows a wide range to change the power and, therefore, energy of the bombarding ions at different gas pressures in the chamber. For ion cleaning, etching diode scheme used, the cathode was treated samples of steel 45 with a working surface of 14 mm diameter.

The working medium is argon. Therefore, the main processing mechanism served as a natural spray that is characterized by the coefficient of dispersion. The coefficient of dispersion essentially depends on the pressure in the treatment chamber and at pressures greater than 13.3 Pa decreases. This is due to the formation on the treated surface dense adsorbed layers, delaying and preventing the dispersal of the material of the surface [5]. The lower limit of the pressure is determined by the conditions of existence of a glow discharge at a given diode circuit. For this reason, in this work the gas pressure is set within the range 1...14 Pa.

The main factors of the process is the pressure, the discharge power, the distance between the electrodes and the processing time. Discharge power was fixed at a predetermined level by controlling the current-voltage characteristics of the power supply. Voltage and current values determined by the power and voltage-current characteristics of the discharge. The voltage-current characteristics of the discharge depends on the pressure in the chamber and the distance between electrodes.

In the abnormal discharge mode power value is selected from the conditions of effective surface treatment. The discharge voltage has changed in the range of 500-5000 V, current densities of 0.2-1.5 mA/cm^2 [6]. At voltages less than 500 V significantly reduces the efficiency of cathode sputtering. And at voltages over 5000 micro arcs appear in the electrode gap. These intervals changes of current density and discharge voltage correspond to change of the specific discharge power in the range of 1-5 W/cm^2. The distance between electrodes was varied in the range of from 5 to 15 mm, the processing time – in the range from 30 to 600 seconds.

On the basis of these data and preliminary experiments to find the empirical dependence of the angle of wetting the surface of the above factors was built of three-factor experimental design [7]. Values of the factor levels and their variation ranges are shown in Table 1 and Fig. 1.

Table 1. Factors their levels and ranges of variation.

Name factor	Unit of measure ment	The code mark	Intervals of variation	Levels factors				
				*+	upper (+)	main (0)	lower (-)	*-
Specific power, W	W/cm^2	X_1	1,34	4,604	3,69	2,35	1,01	0,096
The distance between the electrodes, L	mm	X_2	2	11,36	10	8	6	4,636
Chamber pressure, P	Pa	X_3	3	11,55	9,5	6,5	3,5	1,455

As the plan of the experiment was taken a central rotatable composite plan of the second order with the kernel as a complete three-factor plan, six experiments in star points and six in the center of the plan.

The quality of the surface cleaning was determined by the strength criterion compound coating to the substrate. Plasma spraying samples was performed through certain time after treatment. Coating on the part of the samples was carried out immediately after the treatment. In this case, to protect the surface from oxidation unused portable container. The remaining samples were in the air for a while before coating deposition.

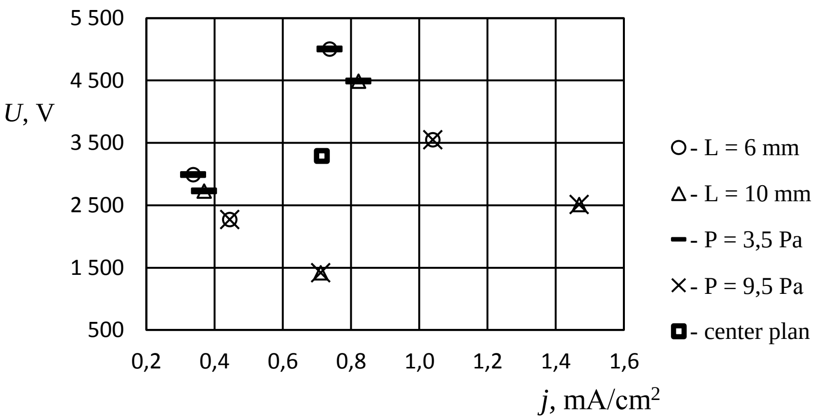

Figure 1. The values of the factors in the core of the plan of the experiment.

Plasma spraying was carried out using an arc plasma torch EDP-104 in the mode: current is 110 A, voltage is 250 V, plasma-forming gas is nitrogen, with the addition of propane, transporting gas is nitrogen, spraying distance is 160 mm. Spraying material is powders self-fluxing material nickel-based PG-HN80SR4, nickel-titanium and nickel-aluminum intermetallic PN55T45 and PN85YU15 dispersion of 40 and 63 microns, respectively. In all cases, deposition time is selected so that the coating thickness was 0.6 ... 0.7 mm, which meets the criterion of correctness [8]: $r / \delta \leq 2$, where r is radius end of the pin; δ is coating thickness. Spraying time was ~ 8-10 seconds.

To quantify the strength of the bond coating to the base method is used stretching taper pin [6] in the centering device on the tensile testing machine MP-05.

3. The results of experimental studies the quality of cleaning steel surface contact angle criterion

Processing the results gave regression equation of the contact angle θ of the factors which is a dimensionless normalized relationship

$$\theta = 22{,}5486 + 1{,}6875X_1 + 1{,}1875X_3 + 3{,}5547X_2X_3 + \\ + 5{,}43634X_1^2 + 1{,}24363X_2^2 + 5{,}07176X_3^2, \qquad (1)$$

where $X_1 = \frac{(W-2{,}35)}{1{,}34}$; $X_2 = \frac{(L-8)}{2}$; $X_3 = \frac{(P-6{,}5)}{3}$.

Verification by the Fisher test showed the adequacy of the regression equation (1): $F_{\text{р}} < F_{\text{т}}$ where the estimated value of the Fisher criterion $F_{\text{р}} = 4{,}6$ and the table - $F_{\text{т}} = 5{,}1$ at 5% significance level. Calculated according to the equation (1) the value of θ differ from the experimental to the value not exceeding the experimental error of 10%, which is also confirmed by additional experiments. Therefore, the equation in this region of the factor space can be used as an interpolation formula for calculating the value of the contact angle with an accuracy of 10%.

Optimal values of the contact angle θ, defined by equation (1) is the area of factors: specific power discharge 1,88-2,4 W/cm^2 (~3100-3400 V discharge voltage, the current density ~ 0,60-0,72 mA/cm^2). The residual pressure in the vacuum chamber is 5,5-6,0 Pa; the distance between the electrodes is 8-9 mm. Given the experimental error contact angle at an optimal processing mode was θ = 22±2 degrees.

Wetting angle θ depending on factors surface treatment process constructed according to the regression equation (1), plus additional experimental values of θ in the optimum factors are presented in Figures 2-4. These results show the correspondence between the experimental data and the values calculated by the regression equation (1), with an error of not more than 10%.

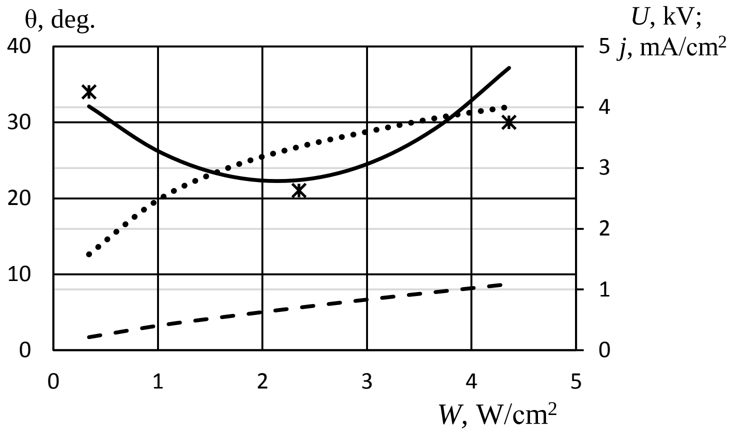

Figure 2. The dependence of the contact angle θ of the specific power W glow discharge: ✳ – experiment (P = 6 Pa, t = 300, L = 9 mm); —— – equation (1); - - - - j; ⋯⋯ – U.

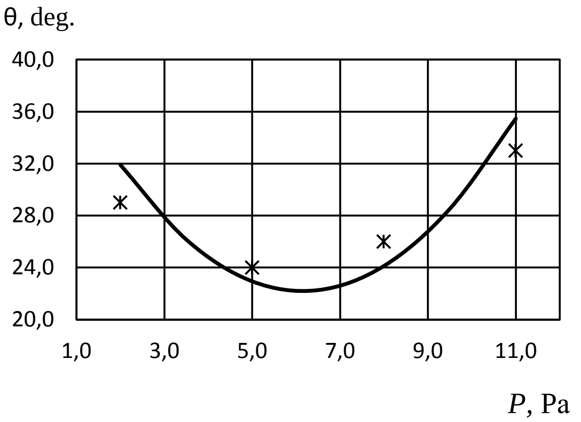

Figure 3. The dependence of the contact angle θ of the pressure P: ✳ – experiment; W = 2,15 W/cm^2 (j = 0,66 mA / cm2; U = 3300); L = 9 mm, t = 300 s.

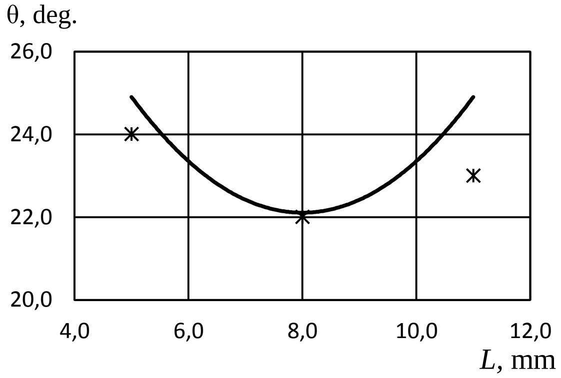

Figure 4. The dependence of the contact angle θ of the distance between the electrodes L: ✳ – experiment; W = 2,15 W/cm^2 (j = 0,66 mA/cm^2; U = 3300 V); P = 6 Pa, t = 300 s.

The increase of the discharge power density W to a value of 2.35 W/cm^2, respectively, a discharge current density j to values of 0.7 mA/cm^2 (Fig. 2) reduces the contact angle to 22 degrees. The subsequent increase in the values of W and j leads to growth of the contact angle. This is due to the heating surface. Therefore, visually detectable formation of the oxide film, and the growth rate of its oxidation due to the presence of oxygen in the chamber as part of the residual gas and is letting argon. For the same reason there is stabilization intensity of the treatment surface with the consequent formation of the oxide film at voltages above 2500 V. n this case, the processing efficiency deteriorates, apparently due to the fact that the rate of oxidation of the surface increases and exceeds its velocity spraying.

Since ion surface cleaning process is characterized in that the discharge current density j is determined by the pressure of residual gases in a square P^2, in the pressure range of P = 5-6 Pa also has a minimum contact angle θ = 22-24 ° (Fig. 3).

The distance between the electrodes L in the region considered the factors has a similar extreme nature. When L is less than 6 mm intensified thermal processes on the treated surface, leading to increased speed of its oxidation state (Fig. 4). The increase in the contact angle with the distances L more than 10 mm is probably due to the weakening of the frequency of ion bombardment work piece.

In these experiments discharge treatment was performed for a fixed time of 300 seconds. To determine the optimal treatment time t test was performed in the range of 30-600 s at previously determined optimal mode (W = 2,15 W/cm^2 (j = 0,66 mA/cm^2; U = 3300 V); L = 9 mm; P = 6 Pa). The results show that the minimum processing time t, ensuring optimum contact angle θ, is 180 (Fig. 5).

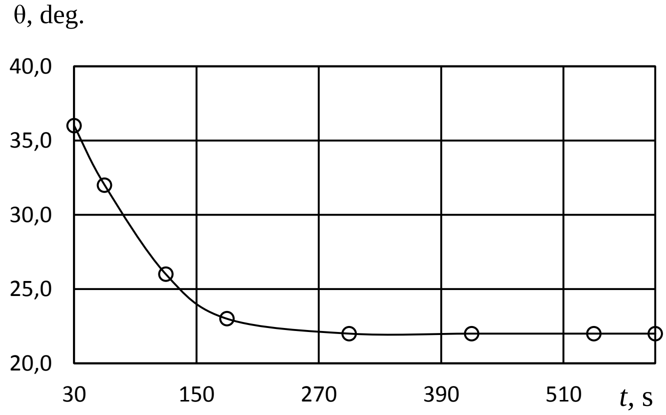

Figure 5. The dependence of the contact angle θ on the processing time t: W = 2,15 W/cm^2 (j = 0,66 mA/cm^2; U = 3300 V); L = 9 mm, P = 6 Pa.

4. Results of the study quality of cleaning the steel surface by the criterion of the bond strength of the plasma coating to the base

Effect surface treatment by a glow discharge before plasma spraying on the strength of the coating to the surface shown in Fig. 6. The results show an increase of strength between the overlay material GHG HN80SR3, PN55T45 PN85YU15 and with a core of 45 steel by 60-80% if prior to the deposition surface of the substrate treated by a glow discharge at an optimal mode. One of the classic methods of surface preparation prior to plasma spraying using an arc plasma torch is sandblasting. Therefore, the effect of treatment by a glow discharge was evaluated on the strength of the coating to the substrate after blasting. The results show for this case an additional increase in the bond strength by up to 55%.

Under real conditions, after cleaning the surface prior to the deposition of coatings in the open atmosphere, there is always a period of time during which the treated surface is contacted with atmospheric air. To assess the effect of this time on the strength of the coating to the substrate were tested, the results of which are shown in Fig. 7.

Samples were kept before spraying in air for 1, 2, 4, 10, 20, 40, 70 and 120 minutes. The nature of the changes to the bond strength of coatings on the basis of the residence time of the substrate in the air indicates the intense decreasing of chemical activity of the surface in the first minute followed by a decrease of the intensity to constant values after 40-60 minutes.

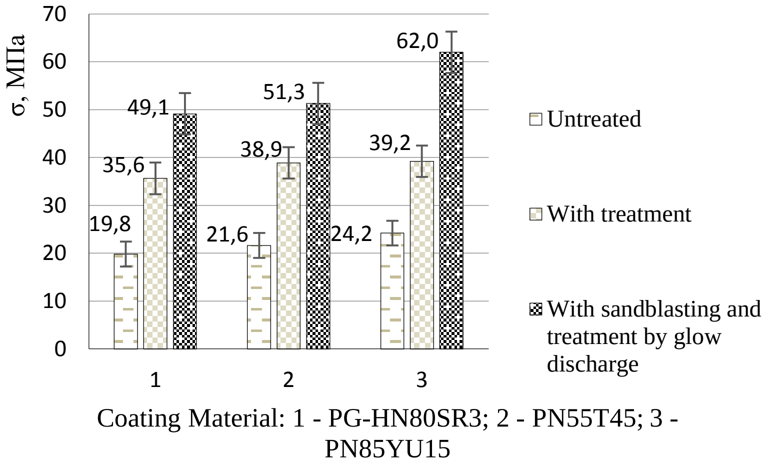

Figure 6. Effect of pre-treatment of the surface of steel 45 on the strength σ it covers

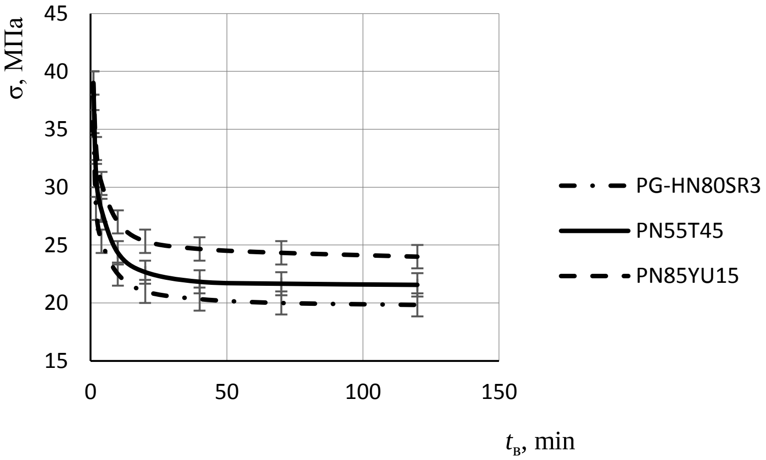

Figure 7. The dependence of the bond strength of the coating σ to the substrate of the length of the substrate in the air finding t_B between the treatment by a glow discharge and plasma spraying.

5. Conclusions

Experimental studies of the quality of cleaning the steel surface by a glow discharge have shown that it is advisable to use an abnormal discharge regime in the parameters. Specific power discharge 1,88-2,4 W/cm^2 (~ 3100-3400 V discharge voltage, the current density ~ 0,60-0,72 mA/cm^2), the residual pressure of argon in the vacuum chamber was 5,5-6,0 Pa; the distance between the electrodes was 8-9 mm; processing time – 180 s. The dependence of the criterion of quality of steel surface (contact angle) by a glow discharge from the factors of the process. This allows you to quantify the impact.

Surface treatment by a glow discharge before spraying metal and intermetallic coating enhances the strength of their connection to the base by 60% to 80% in the case of ground samples and 30-55% in the case of samples with sandblasting. Increase strength characteristics of the coatings that have passed before spraying pretreatment by a glow discharge, allows you to use the method of preparation of surfaces for the cases to provide the necessary high bond strength of coatings to the substrate. This can be actual for applications in the production of coatings, working in environments with high contact and shear loads. Obtained optimal modes treatment of steel surfaces by a glow discharge are technological recommendations of such processing.

Evaluation results of cleaning steel substrates compared with chemical etching provides the required strength quality compound at high process ecology. In comparison with the cleaning by the arc discharge – surface uniformity of treatment of simple and profiled surfaces. Further development of research to improve the efficiency of cleaning of surfaces in the direction seems dynamization mode glow discharge and the protection of the activated surfaces before applying coatings on them.

Acknowledgments
The reported study was funded by RFBR, according to the research project No. 15-38-51042 mol_nr.

References

[1] Anciferov V N, Bobrov G V, Druginin L K and etc 1987 *Powder metallurgy and sprayed coatings* (Moscow, Metallurgy) p 792
[2] Rikalin N N, Shorohov M H and Kudinov V V 1969 *On the mechanism of formation kinetics of a firm connection between the coating and the substrate during the deposition* (L, Science) P 5-28
[3] Saifutdinov A I, Saifutdinova A A, Kashapov N F and Fadeev S A 2016 *J. Phys.: Conf. Ser.* **669** 012045
[4] Saifutdinov A I, Fadeev S A, Saifutdinova A A and Kashapov N F 2015 *JETP Lett.* **102** 637–642
[5] Gabovich M D, Pleshivcev N V and Semashko N N 1986 *The beams of ions and atoms for controlled thermonuclear fusion and technological objectives* (Moscow, Energoatomizdat) p 249
[6] Bolotov G P, Bolotov M G and Rigov R N 2009 *Vestnik NTU* **(57)** 124–127
[7] Spiridonov A A 1981 *Planning experiment in technological processes* (Moscow, Mechanical engineering) p 184
[8] Tushinskii L I, Plohov A V, Tokarev A O and Sindeeb V I *Methods for Materials Research: Structure, properties and processes applying inorganic coatings* (Moscow, World) p 384