

## INFLUENCE OF THE LASER-PROCESSING PARAMETERS ON THE ADHESION STRENGTH OF ADHESIVE Fe–Cr–B–Si-SYSTEM COATINGS

N. N. Dorozhkin,<sup>a</sup> M. A. Kardapolova,<sup>b</sup>  
and O. V. D'yachenko<sup>b</sup>

UDC 621.793.74

*The influence of the laser-processing parameters on the adhesion strength of the Fe–Cr–B–Si-system coating has been investigated. The characteristics of a change in the adhesion strength of coatings, as well as in the amounts of iron borides as functions of the velocity of travel, diameter of a laser beam, and coefficient of overdropping have been studied.*

Alloys of the Fe–Cr–B–Si system possess a high structural sensitivity to the action of power and doping additives [1, 2]. Laser modification allows one to accurately batch the supply of energy and doping substances, whereas the character of the resulting structure accounts for the quality of the strengthened layer. Moreover, due to their composite structure, the alloys possess high wear resistance.

The technique of obtaining wear-resistant coatings by plasma spraying combined with subsequent laser fusion is widely known [1–4]. This technique has been used to advantage in production and restoration of long-dimensional, large-size articles and articles of complex configuration. However, the technology is rather expensive.

After laser fusion, coatings display the needed complex of physicomechanical and service properties. For comparison, some samples were subjected to laser fusion after using the adhesion technique of coating.

The coatings deposited on an article in the process of its service are subjected to the action of mechanical loads, and the adhesion strength limits their applicability. Laser processing of adhesive coatings allows one to increase the strength of adhesion of a coating to the base, preserving to a maximum the initial structure and properties of the powder. To estimate the efficiency, articles are subjected to tear tests according to State Standard 14760-69 "A Method to Determine the Detachment Strength."

The aim of the present work is to investigate the adhesion strength and phase composition of coatings consisting of autoflaxed alloys on an iron base (adhesive ones and those after plasma spraying, fused by laser and burner).

**Investigation Technique.** As a material for applying adhesive coatings and sputtering, we used an autoflaxed alloy of PR-X4G2R4S2F powder of the following chemical composition (in %): Fe (83.1–87.6), B (3.3–4.3), Cr (3.5–4.5), Si (2.0–2.5), Mn (2.0–2.5), C (1.0–1.2), V (0.5–0.9), Al (0.05–0.5), and Cu (0.05–0.5). It was applied by a small brush to a prepared surface of pins by an adhesive technique and also by a method of plasma spraying with the aid of a UPU-3D apparatus with an IPN-160/600 power source and a PP-25 plasmatron at  $I = 250$  A,  $U = 80$  V, and  $P_{sp} = 0.06$  GPa. The thickness of the layer was 0.6 mm.

Investigations of the adhesion strength were carried out on a Riehle tearing machine with a force changing smoothly from 0 to 50,000 N on 40Kh-steel pins put one into the other and lapped. The ends of the cones form concentric rings. The diameter of the smaller cone is 12 mm and that of the larger one 20 mm. Such a construction of the pins allowed us to obtain a more homogeneous detachment.

The method of pin probe used is based on direct determination of the force directed perpendicularly to the deposited and fused surface and detaching the coating from the main material [3, 4]. To fasten the sample in the tearing machine, special mandrels connected with the clamps of the machine with the aid of flexible cables were used.

---

<sup>a</sup>A. V. Luikov Heat and Mass Transfer Institute, National Academy of Sciences of Belarus, 15 P. Brovka Str., Minsk, 220072, Belarus; <sup>b</sup> Belarusian National Technical University, 65 Nezavisimost' Ave., Minsk, 220013, Belarus. Translated from *Inzhenerno-Fizicheskii Zhurnal*, Vol. 79, No. 6, pp. 155–159, November–December, 2006. Original article submitted October 31, 2005; revision submitted April 13, 2006.

TABLE 1. Nature of the Variable Factor

Nature of variable factor	$v$ , mm/min	$d_{las}$ , mm	$k_{ov}$
Basic level	100	2	1
Variation interval	50	1	0.2

TABLE 2. Matrix of Experiment Planning

Test No.	$X_1$	$X_2$	$X_3$
1	—	—	—
2	+	—	—
3	—	+	—
4	+	+	—
5	—	—	+
6	+	—	+
7	—	+	+
8	+	+	+

This provided the perpendicularity of the force that detached the endface surface of the sample. A minimum rate of loading ensuring the statistical character of the latter was used. The force  $P_{det}$  corresponding to the moment of coating detachment from the pin was recorded on a scale graduated in kilograms and was then converted into GPa. The adhesion strength and the endface area of the pin were determined from the formulas

$$\sigma_{ad} = \frac{P_{det}}{A}, \quad A = \frac{\pi d_p^2}{4}. \quad (1)$$

In order to compare the values obtained for  $\sigma_{ad}$  for various samples, the thickness of the sputtered and adhesive coatings was kept constant at 0.6 mm. The time interval between the shot-blasting processing and application of adhesive coating as well as sputtering was kept constant. To ensure statistical reliability, ten samples for each coating applied under identical conditions were used.

The pin surface under adhesive and plasma-sprayed coatings was prepared as follows: first it was degreased by ethyl alcohol and then was shot blasted. Thereafter, the pin was rotated in a gadget to remove errors. A powder alloy was applied by an adhesive technique to the sample surface (3% "AGO" glue in acetone was added to the powder layer) or sputtered [1]. Then, an absorbing coating was applied to the plasma-sprayed layer (yellow water-soluble color). In the case where the adhesive technique was used, the above procedure was replaced by smearing with a doping substance based on boron carbide.

Fusion was effectuated by an LGN-702 continuous laser of power  $N = 800$  W with the laser spot of diameter  $d_{las}$  from  $1.0 \cdot 10^{-3}$  m to  $3.0 \cdot 10^{-3}$  m with velocities of its travel  $v$  of 50, 100, 150, 200, and 300 mm/min and overlapping coefficient  $k_{ov} = 0.8$  and 1.2 to obtain a single-phase composition and specified properties over the entire thickness of the coating.

In the case of laser doping of adhesive coatings of boron carbide, the fusion regimes were selected and the data obtained were investigated by one of the methods of mathematical planning, i.e., by the method of complete factor experiments [5].

Since the number of variable parameters is not large, it appeared possible to realize a total replica in which the number of tests  $Q = 2^n$ , which, in the course of an experiment, makes it possible to simultaneously vary several parameters of different physical nature and obtain separate, independent estimation of coefficients, which is impossible, for example, when a fractional replica is realized. Here, it is necessary that all independent variables that influence the process be variable at two levels: minimal and maximal.

The series consisted of eight basic tests. The model obtained was considered linear, and it took into account the interaction of the following factors:

TABLE 3. Dependence of  $\sigma_{ad}$  on Processing Regimes

Sample No.	Form, mode of processing, and code			$\sigma_{ad}$ , MPa
	$X_1$	$X_2$	$X_3$	
	Adhesive coating fused by laser			
1	50	1	0.8	92
2	150	1	0.8	67
3	50	3	0.8	73
4	150	3	0.8	53
5	50	1	1.2	76
6	150	1	1.2	58
7	50	3	1.2	70
8	150	3	1.2	42
	Plasma-sprayed coating fused by laser			
9	50	1	0.8	156
10	100	1	0.8	129
11	300	1	0.8	113
	Plasma-sprayed coating fused by a burner			
12	—	—	—	142
	Plasma-sprayed coating without fusion			
13	—	—	—	31

$$Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_{12}X_1X_2 + B_{13}X_1X_3 + B_{23}X_2X_3. \quad (2)$$

In order to compile a table of data (planning matrix) limits were placed on the change of the basic inlet parameters, that is, the technological parameters of laser processing (Tables 1–3). The technological factors  $X_1$ ,  $X_2$ , and  $X_3$  were selected proceeding from the possibility of their change, provided their values are accurately measured, and also taking into account their mutual independence (orthogonality). In order to estimate the validity of the results and adequacy of the model, the tests were repeated thrice on the basic level using boron carbide as a strengthening admixture. The strength of coupling between the coating and the base was the optimization parameter  $Y$ .

X-ray patterns were taken on a DRON-3.0 diffractometer at a speed of rotation of a sample of 1 deg/min in monochromatized radiation from copper in the maximum possible range of angles from  $10^\circ$  to  $75^\circ$  for a qualitative and quantitative phase analysis.

**Results of Investigations and Their Discussion.** The factors that limit the strength of adhesion of hot-gas sprayed coatings consisting of self-fluxing, iron-based alloys are the deoxidization of oxidation films between a coating and a substrate and the occurrence of chemical bondings. The time needed to deoxidize iron oxides at  $T = 1300\text{--}1500$  K is 0.75–0.9 sec [6].

In the process of investigation, one could observe an adhesive and adhesive-cohesive character of the rupture of samples when a coating separated entirely or a portion of it remained on the pin, with the former case being typical of higher rates of laser-beam scanning.

A coating begins to break from end faces [7], where the adhesive layer is thinner and defects are present, i.e., there is no satisfactory contact between adhesive and material. Appreciable stresses are concentrated at these places. On increase in loading, microcracks appear in them, and they gradually propagate to the center of the adhesive joint. When the number of microcracks attains a certain level, conditions develop such that they can unite into a large crack, and this leads to the destruction of the coating.

For coatings sputtered by plasma without doping, the maximum value of  $\sigma_{ad}$ , equal to 156 MPa, is observed at a minimum velocity of laser-beam travel of 50 mm/min (see Table 3, sample No. 9); with an increase in  $v$  up to 300 mm/min the value  $\sigma_{ad} = 113$  MPa (sample No. 11) is attained. This is due to the shorter time of residence of the article in the zone of laser heating.

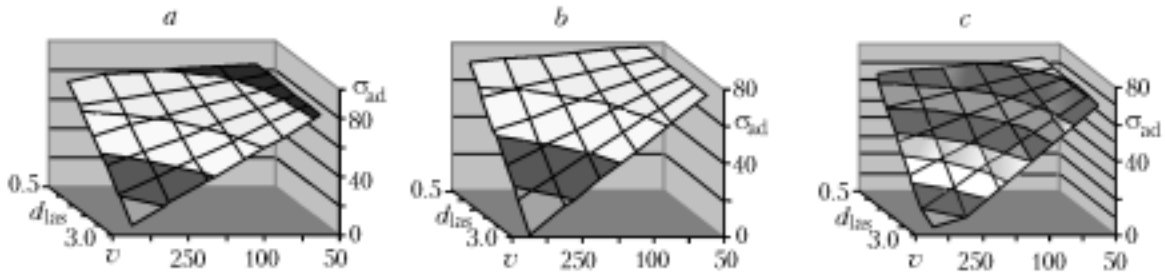


Fig. 1. Strength of coating adhesion after laser doping with B<sub>4</sub>C.

TABLE 4. Regimes of Processing of Adhesive Coatings

Parameters	No. of a sample							
	1	2	3	4	5	6	7	8
$v$ , mm/min	50	150	50	150	50	150	50	150
$d_{las}$ , mm	1.0	1.0	3.0	3.0	1.0	1.0	3.0	3.0
$k_{ov}$	0.8	0.8	0.8	0.8	1.2	1.2	1.2	1.2

TABLE 5. Composition of Phases after Application of Adhesive Coatings and Laser Doping with B<sub>4</sub>C Processed in the Regimes Listed in Table 4

Sample No.	Phase, %					$H$ , MPa
	Fe	Borides of Fe	Carbides of Fe	Carbides	Borides of Cr	
1	27.3	29.5	11.5	23.3	8.4	1136
2	34.4	27.1	8.5	21.9	8.1	1313
3	33.1	27.9	10.1	22.1	6.7	1223
4	37.8	27.1	11.6	19.3	4.2	1210
5	30.6	28.6	8.4	25.8	6.3	1180
6	29.3	25.1	9.7	23.3	12.6	1285
7	30.7	29.1	13.0	23.6	3.7	1243
8	28.3	22.3	9.0	26.3	14.0	1386

TABLE 6. Composition of Phases Depending on the Regimes of Processing of Burner- and Laser-Fused Plasma-Sprayed Coatings without Doping (at  $d_{las} = 1.0$  mm,  $k_{ov} = 0.8$ )

Sample No.	Regime of processing	Phase, %					$H$ , MPa
		Fe	Borides of Fe	Carbides of Fe	Carbides	Borides	
9	$v = 50$ mm/min	56.2	13.9	7.1	12.7	8.6	939
10	$v = 100$ mm/min	55.7	14.9	7.5	14.5	10.4	1050
11	$v = 50$ mm/min	55.8	14.0	7.6	15.6	7.0	1121
12	Deposition and fusion by a burner	27.6	16.8	10.4	27.0	16.7	1015

The minimum value  $\sigma_{ad} = 31$  MPa is observed for the coating deposited by plasma without fusion because of the presence of pores and a large amount of oxide films (sample No. 13).

As to adhesive coatings doped with B<sub>4</sub>C, the best adhesion of a coating to the base  $\sigma_{ad}$  is observed at  $v = 50$  mm/min,  $d_{las} = 1$  mm, and  $k_{ov} = 0.8$ , that is, 92 MPa (sample No. 1). The coating is exposed to a laser-beam long enough and has time to fuse through completely. With increase in the overlapping coefficient up to 1.2, the adhesion strength falls to 76 MPa (sample No. 5). This seems to be due to the beam defocusing.

At  $v = 50$  mm/min,  $d_{las} = 3$  mm, and  $k_{ov} = 0.8$ , the value of  $\sigma_{ad}$  is decreased to 73 MPa (sample No. 3). This seems to be due to the decrease in the power input. At the same velocity and laser-beam diameter, but on in-

crease in the overlapping coefficient to 1.2 (sample No. 7),  $\sigma_{ad}$  decreases to 70 MPa. As the velocity of travel of the laser beam relative to the article is increased up to 150 mm/min at  $d_{las} = 1$  mm and  $k_{ov} = 0.8$  (sample No. 2),  $\sigma_{ad}$  decreases to 67 MPa. This is due to the fact that the time of coating exposure to the laser beam was shorter. At the same velocity and the same beam diameter but on increase in the overlapping coefficient up to 1.2 (sample No. 6),  $\sigma_{ad}$  decreases to 58 MPa. At  $v = 150$  mm/min,  $d_{las} = 3$  mm, and  $k_{ov} = 0.8$  (sample No. 4),  $\sigma_{ad}$  decreases to 53 MPa. At maximum values of the travel velocity and diameter of the beam and of the overlapping coefficient ( $v = 150$  mm/min,  $d_{las} = 3$  mm, and  $k_{ov} = 1.2$  (sample No. 8)), the value of  $\sigma_{ad}$  is the lowest, i.e., 42 MPa.

The resulting adequate model of the response allows one to judge the degree of the influence exerted by the  $X_1 - X_3$  parameters on the adhesion strength  $Y$  in laser doping of adhesive coatings:

$$Y = 67.458 - 9.708X_1 - 6.042X_2 - 5.958X_3 - 5.375X_1X_2. \quad (3)$$

We have constructed the dependences of the strength of adhesion of a coating to the base after laser doping with  $B_4C$  at the overlapping coefficient  $k_{ov}$  that is equal to 0.8 (a), 1.0 (b), and 1.2 (c) (see Fig. 1). One can clearly see the dependence of the adhesion strength for adhesive coatings doped with  $B_4C$ : as the velocity travel of the laser beam increases,  $\sigma_{ad}$  decreases. At  $v = 50$  mm/min, the time of exposure of a coating to the laser beam is longer, the coating is fused more appreciably, and this causes an increase in the adhesion strength. On increase in the laser-beam diameter to 3 mm due to defocusing, the contribution of power is decreased, the coating is less fused, and the value of  $\sigma_{ad}$  is lower than at  $d_{las} = 1$  mm. During processing of coatings with an overlapping coefficient of 0.8, an additional refusion of the coating occurs because of the superposition of laser paths, and this leads to an increase in  $\sigma_{ad}$ . For plasma coating without fusion, the value of  $\sigma_{ad}$  is lowest because of the cracks, large sphere-like cavities, and small pores as well as the large quantity of oxidized films. For plasma coatings fused by laser radiation,  $\sigma_{ad}$  increases as the rate of processing decreases. In the case of tests with plasma sprayed coatings fused by a burner,  $\sigma_{ad}$  depends on the fusion regimes chosen and is somewhat lower than for fusion by a laser beam.

As is known, the properties of coatings are influenced not only by the adhesion strength but also by their phase composition, especially by the amount of the strengthening phase. The data of the x-ray analysis of adhesive and plasma-sprayed coatings after fusion by a laser beam and a burner are presented in Tables 4–6.

It is seen from the tables that with increase in the velocity of travel of the laser beam, the beam diameter, and the overlapping coefficient, the amount of iron borides is decreased as well as the adhesion strength  $\sigma_{ad}$ .

## CONCLUSIONS

1. The maximum value of the adhesion strength  $\sigma_{ad}$  is observed for laser-fused, plasma-sprayed, nondoped coatings at a minimum velocity of laser-beam travel relative to an article.
2. For adhesion laser-fused coatings with an increase in the velocity of laser-beam travel relative to an article from 50 to 150 mm/min, increase in the beam diameter from 1 to 3 mm, and increase in the overlapping coefficient from 0.8 to 1.2, the amount of iron borides decreases, as well as the adhesion strength  $\sigma_{ad}$ .
3. It has been established that an increase in the concentration of boron  $C_B$  leads to an increase in the adhesion strength and to an improvement of the structure of the boundaries of grains.

## NOTATION

$A$ , area of the end-face surface of a pin,  $mm^2$ ;  $B_{ij}$ , coefficients of regression that describe the direction and degree of influence of each factor on optimization parameters;  $d_{las}$ , laser-beam diameter, mm;  $d_p$ , pin diameter, mm;  $H$ , microhardness, MPa;  $I$ , arc current in plasma spraying of coatings, A;  $k_{ov}$ , coefficient of overlapping of laser paths;  $N$ , power of the LGN-702 laser facility, W;  $n$ , number of factors;  $P_{sp}$ , pressure of plasma-forming gases in spraying, GPa;  $P_{det}$ , strength corresponding to the moment of detachment of a coating from the pin, GPa;  $Q$ , number of tests;  $T$ , time needed to deoxidize iron, sec;  $U$ , electric arc voltage in plasma spraying, V;  $v$ , generalized velocity of laser-beam travel relative to an article, mm/min;  $v_1 = 50$ ,  $v_2 = 100$ ,  $v_3 = 150$ ,  $v_4 = 200$ , and  $v_5 = 300$ , velocities of laser-beam travel relative to an article, mm/min;  $X_1$ , velocity of laser-beam travel, mm/min;  $X_2$ , laser-beam diameter at the

time of contact with the surface material, mm;  $X_3$ , coefficient of overlapping of laser paths;  $Y$ , adhesion-strength optimization parameter;  $\sigma_{\max}$ , maximum strength of adhesion between coating and base, MPa;  $\sigma_{\text{ad}}$ , strength of adhesion between coating and base, MPa. Subscripts;  $i, j$ , number of the coefficient of the model (coincides with the ordinal number); las, laser beam; sp, spraying; det, detachment; ov, overlapping; ad, adhesion between coating and base; p, pin.

## REFERENCES

1. V. P. Larionov, N. P. Bolotina, T. V. Argunova, V. D. Tyunin, and N. P. Lebedev, Influence of laser processing on the structure and composition of plasma-sprayed coatings of the Ni–Cr–B–Si–C system, *Fiz. Khim. Obrab. Mater.*, No. 1, 74–78 (1987).
2. I. M. Spiridonova, Structure and properties of iron–boron–carbon alloys, *Metalloved. Term. Obrab. Met.*, No. 2, 58–61 (1984).
3. P. A. Vityaz', V. S. Ivashko, A. F. Il'yushchenko, et al., *Theory and Practice of Applying Protective Coatings* [in Russian], Belaruskaya Navuka, Minsk (1998).
4. V. S. Ivashko, I. A. Kupriyanov, and A. I. Shevtsov, *Electrothermal Technology of Applying Protective Coatings* [in Russian], Navuka i Tékhniká, Minsk (1996).
5. F. S. Novik, *Mathematical Methods of Planning Experiments in Metal Science. Planning of Industrial Experiments* [in Russian], Mashinostroenie, Moscow (1971).
6. L. I. Grechikhin, N. V. Spiridonov, A. G. Vasilenko, M. A. Kardapolova, and O. G. Devoino, Enhancement of the adhesive bond of laser radiation-fused gas thermal coatings, *Fiz. Khim. Obrab. Mater.*, No. 3, 76–81 (1990).
7. M. S. Trezno and E. V. Moskalev, *Adhesives and Gluing* [in Russian], Leningrad, Khimiya (1980).