# <sup>06.5</sup> Plasma treatment of cold-sprayed Ni–Al coating

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The results of experiments on plasma jet treatment of cold spray composite coatings Ni–Al are presented. A plasma jet with a heat flux power density in the range of  $8 \cdot 10^7 \le q \le 10^8 \text{ W/m}^2$  is sufficient to heat the coating surface to the melting point of aluminum and nickel over the entire coating depth. The formation of an intermetallic compound of the Ni–Al system occurs due to the diffusion of aluminum atoms into the bulk of nickel and due to the dissolution of nickel in the aluminum melt. The resulting coating is a highly porous intermetallic compound with high hardness.

Keywords: cold spraying, composite coatings, plasma jet, nickel, aluminum, x-ray phase analysis.

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The essential of massive improvement of the quality of materials and articles and enhancement of their durability and performance characteristics becomes especially relevant in the modern context of development of technology and engineering. In most cases, this problem is solved by deposition of multipurpose (e.g., protective and bulkhardened) coatings.

It is known that coatings deposited by cold spraying (CS) form from unmelted particles. Therefore, particles of materials with a high hardness (ceramics and hard alloys) do not deform on impact with a substrate, and surface erosion is observed. The results of studies into the formation of an intermetallic Ni<sub>3</sub>Al coating by CS with subsequent hightemperature treatment in a furnace were reported in [1]. The formation of intermetallic compounds of the Ni-Al system with various stoichiometric proportions (NiAl, Ni<sub>3</sub>Al) was established by XRD and EDS. To achieve a complete transformation of the material into an intermetallic compound, one needs to increase the temperature and the processing time; however, this is not possible in some cases (e.g., due to the use of a specific substrate material). One efficient method for improving the properties of coatings and eliminating their defects via partial or complete surface melting is their processing with concentrated fluxes of energy of a relatively low density  $q \sim 10^7 - 10^9 \, \mathrm{W/m^2}$  (specifically, a quasi-laminar plasma jet  $(q \sim 10^7 - 10^8 \, \mathrm{W/m^2}$  [2]) or a transfer arc  $(q \sim 10^8 - 10^9 \text{ W/m}^2 \text{ [3-5]}))$  that do not induce evaporation of the coating material and excessive heating of the substrate. The aim of the present study is to examine the effect of high-energy post-processing of CS Ni+Al coatings on their phase composition, structure, and performance characteristics.

Coatings deposited onto aluminum alloy (grade AMG2M) substrates from a mixture of aluminum (grade ASD-1) and nickel (grade PNK-UT-1) powders using a high-pressure CS setup (Institute of Theoretical and Applied

Mechanics, Siberian Branch, Russian Academy of Sciences). The conditions of preparation of powder mixtures and coating process conditions were the same as in [1]. The thickness of sprayed CS coatings was  $500 \,\mu$ m.

A plasma torch with an interelectrode insert with a power up to 50 kW was used for processing of the surface of CS coatings. The ranges of power density of a laminar plasma jet  $(5 \cdot 10^7 \le q \le 10^8 \text{ W/m}^2)$  and its duration  $(5 \cdot 10^{-2} \le t_i \le 1 \cdot 10^{-1} \text{ s})$  ensuring heating of the coating surface to the melting point of aluminum and complete melting of aluminum in a Ni–Al coating were determined based on the data from [6,7].

The plasma jet power density was determined at the following values of process parameters: current I = 250 A and flow rate  $G_g = 0.2$  g/s (He)+0.4 g/s (Ar) of the mixture of plasma gases; flow rate  $G_a$  of the anode protection gas (Ar) was varied from 0.14 to 0.42 g/s. The anode diameter was 9 mm, and the nozzle diameter was 11 mm. Distance Z to the substrate was varied from 120 to 20 mm with a pitch of 20 mm. Water-cooled probes 15 and 20 mm in diameter were used to measure heat fluxes (Fig. 1). Since actual diameter  $D_c$  of the heat treatment spot is smaller than the probe diameter (e.g.,  $D_c \sim 8-10$  mm for a distance Z = 20 mm), the maximum heat flux power density used in the processing of coatings was estimated at  $8 \cdot 10^7 \leq q \leq 10^8$  W/m<sup>2</sup>. The traverse speed of the substrate relative to the plasma torch varied from 40 to 100 mm/s.

The phase composition of coatings was examined by X-ray diffraction analysis using a D8 ADVANCE (Bruker, United States) diffractometer and monochromatized  $CuK_{\alpha}$  radiation.

Polished cross-sections of coatings were prepared for structural studies. Presi (France) machines were used in sample preparation.

The microhardness and the Young's modulus were measured in cross sections of coatings using a NanoScan-4D+  $\,$ 

Element	As-deposited coating		Α		В		С		D	
	wt.%	at.%	wt.%	at.%	wt.%	at.%	wt.%	at.%	wt.%	at.%
Al Ni	22.55 77.45	38.79 61.21	22.55 77.45	38.79 61.21	37.01 62.99	56.11 43.89	48.45 51.55	67.16 32.84	0.37 99.63	0.80 99.20

Table 1. Elemental composition of a cross section of a CS coating



Figure 1. Diagram of the setup for determination of heat fluxes.

(TISNUM, Russia) nanomechanical testing complex. No less than ten indentations were performed in order to determine an average value.

The morphology and structure of samples were examined with an Evo MA15 (Carl Zeiss) electron microscope with a detector of back-scattered electrons.

Figure 2 presents polished sections of as-deposited (a) and plasma-processed (b) coatings.

Composite CS coatings had a dense structure with nickel particles distributed uniformly throughout their volume (Fig. 2, a). Three distinct regions may be identified in the cross section of coatings processed with a plasma jet (Fig. 2, b). These regions form in the following way. In region 1, the impact of a plasma jet on the coating surface at the initial time induces heating of the near-surface Ni-Al composite layer to a temperature close to the melting point of aluminum. When a temperature of 773 K is reached, solid-phase diffusion of aluminum atoms into the bulk of nickel particles, which still remain solid, is initiated [8], and NiA1 and Ni<sub>3</sub>A1 intermetallic compounds are synthesized (Fig. 3 and Table 1). The material structure changes, and pores start to form. The volume of pores does not exceed

27%. Similar effects of pore formation were noted in the case of high-temperature post-processing of coatings in a furnace [1]. In region 2, prolonged plasma processing of the coating results in an increase in temperature in the middle material layer (Fig. 2, b), which eventually reaches the nickel melting point (1728 K). The material of region 1 remains solid and acts as a thermal barrier, since the melting point of NiAl is 1911 K. Ultrafine inclusions of intermetallic compounds form due to the dissolution of nickel in molten aluminum and subsequent crystallization of ultrafine NiAl and Ni<sub>3</sub>Al particles (Fig. 3 and Table 1). Since region 3 is in contact with the water-cooled substrate, nickel particles and intermetallic compounds formed due both to the reactive diffusion of aluminum atoms into nickel and to the dissolution of nickel in molten aluminum are found in this region.

The results of determination of the elemental composition at points located in different regions of the processed coating (Fig. 3) are listed in Table 1.

It follows from Table 1 that the Ni+Al composite material undergoes a phase transformation into compounds with various stoichiometric proportions (NiAl, Ni<sub>3</sub>Al) in the process of its layer-by-layer heating. This was verified by the results of XRD analysis, since these compounds were identified correctly: diffraction peaks matched the tabular values for NiAl, Ni<sub>3</sub>Al, and  $\alpha$ -Ni.

The microhardness and the Young's modulus were determined for the as-deposited composite coating and the material in three regions of the processed coating (Fig. 3). The obtained values are listed in Table 2.

The microhardness and the Young's modulus of an aluminum nickelide compound depend on the mechanism of formation of this compound and its stoichiometric composition. The values of microhardness and Young's modulus of the plasma-processed coating material presented in Table 2 correspond to an aluminum nickelide compound (the microhardness is 2.5–4.6 GPa, and the Young's modulus is 150–200 GPa) [9–11].

Thus, we examined the effect of processing of composite CS Ni+Al coatings with a laminar plasma jet for the purpose of synthesis of an intermetallic compound. It was demonstrated that plasma processing of a composite coating deposited by CS from a mechanical mixture of Ni and Al powders at a heat flux power density of  $8 \cdot 10^7 \le q \le 10^8 \text{ W/m}^2$  establishes the conditions for formation of intermetallic compounds of the Ni-Al system. This is attributable to the diffusion of aluminum



**Figure 2.** Images of polished sections of an as-deposited CS coating (a) and a CS coating processed with a plasma jet (b). A — substrate, B — aluminum, and C — nickel. I — region 1, 2 — region 2, and 3 — region 3.



**Figure 3.** Images of polished sections of the coating processed with a plasma jet. a — Regions 1 and 2; b — region 3. A, B, C, and D are points at which the elemental composition was determined.

Material	Average value of microhardness $HV_{0.24}$ /standard deviation SD, GPa	Average value Young's modulus/standard deviation SD, GPa
As-deposited Ni–Al coating Processed coating (region 1)	0.69/0.18 4.77/1.11	76/7.83 154/16.04
Processed coating (region 2)	3.05/0.37	110/23.84
Processed coating (region 3)	0.88/0.18	92/7.50
Aluminum alloy substrate	0.46/0.02	75/2.95

Table 2. Microhardness and Young's modulus of the coating material

atoms into the bulk of nickel and to the dissolution of nickel in molten aluminum with subsequent crystallization of ultrafine particles of intermetallic compounds. The formation of intermetallic compounds of the Ni–Al system with various stoichiometric proportions (NiAl, Ni<sub>3</sub>Al) was established by XRD and EDS. The formation of pores in the processed coating may be attributed to the difference

in density of intermetallic compounds and the initial mixture at the synthesis reaction temperature. It should be noted that thermal processing of the coating provided an opportunity to synthesize a high-porous intermetallic compound from reacting components. The obtained results should help broaden the spectrum of heat-resistant materials.

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## **Conflict of interest**

The authors declare that they have no conflict of interest.

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