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Formation of an intermetallic Ni₃Al coating by cold spraying of a Ni–Al powder mixture mechanically processed in a planetary mill

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The features of the formation of an intermetallic Ni₃Al coating by cold spraying of a Ni–Al powder mixture mechanically processed in a planetary mill and prepared in a V-shaped mixer are presented. It is shown that the powder deposition efficiency is 1.5 times higher when coatings are obtained from a mechanically treated powder mixture of Ni and Al components. Subsequent high-temperature treatment in the furnace chamber creates a condition for the formation of an intermetallic compound of the Ni–Al system of various stoichiometric proportions.

Keywords: cold spray, composite material, high-energy planetary mill, nickel, aluminium, titanium nitride, X-ray analysis.

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Mechanical assemblies and individual parts with complex surface shapes are used in modern industry. Since materials used in the fabrication of such parts are not always resistant to high temperatures, surface reconditioning and passivation by hot spraying or laser cladding may be infeasible. In the process of cold spraying (CS), coatings are formed from unmelted particles that should be made of a material with a sufficiently high plasticity. Particles of materials with a high hardness (ceramics and hard alloys) do not deform on impact with a barrier; the process of surface erosion is observed [1].

Special attention is paid at present to heat-resistant high-hardness intermetallic alloys [2–4]. Since CS is ill-suited for deposition of hard particles, one of the ways to solve the mentioned problem and form composite coatings (e.g., Ni₃Al coatings) consists in spraying a powder mixture of nickel and aluminum metals with subsequent *in situ* synthesis in the process of heat treatment. The mixture for spraying may be prepared both by traditional mixing on a vibration stand or in a V-shaped mixer and with the use of high-energy attritors.

It should be noted that the physical and strength properties of alloys depend to a large extent on the material structure (specifically, the grain size). The potential to control crystallization processes by introducing nanosized refractory particles, which give rise to a finer grain structure, has been demonstrated in [5,6].

The aim of the present study is to produce intermetallic coatings by cold spraying from Ni–Al powder mixtures (including a mixture with added ultrafine titanium nitride particles).

Aluminum (ASD-1) and nickel (PNK) powders and ultrafine titanium nitride particles were used in experiments.

Powder mixtures were prepared in a V-shaped mixer within 8 h and in an „Aktivator-2SL“ high-energy planetary mill in air with the following process parameters: the mass of loaded balls was 160 g, the mass of material processed was 100 g for each vial, the acceleration of grinding bodies was $a = 117g$, and the duration of mechanical processing was 30 s. Steel balls 5 mm in diameter were used as grinding bodies. The characteristics of particles of powder mixtures are listed in Table 1.

If a powder mixture is processed mechanically for more than 30 s, the temperature of aluminum particles reaches a near-melting point. In view of this, they stick to the surface of grinding bodies and the walls of cylinders (as in [7]) and induce the formation of layered mechanical composites, which are characterized by low plasticity values. When such materials are sprayed, it is advisable to use nitrogen as an accelerating and carrier gas and raise the temperature of its preliminary heating. However, this is not feasible in certain coating deposition scenarios (e.g., due to the characteristics of the substrate material).

The overall view of the mechanically processed composite mixture is shown in Fig. 1.

It can be seen from Fig. 1, *b* that the mechanically processed mixture contains agglomerates of a large number of loose-packed micrometer-sized nickel and aluminum particles.

The phase composition of mixtures prepared for spraying and coatings produced from them was examined by X-ray diffraction analysis using a D8 ADVANCE (Bruker, United States) diffractometer and monochromatized CuK α radiation. X-ray diffraction patterns of mechanically processed powder mixtures revealed no chemical transformations: components only mix with each other, forming agglomerated particles.

Table 1. Composition and type of mechanical treatment of a powder mixture for cold spraying

Mixture number	Powder mixture composition	Mechanical process type	Mean size (standard deviation)*, μm
1	3Ni+Al	V-shaped mixer	22.56 (17.01)
2	3Ni+Al	Planetary mill	38.84 (30.87)
3	3Ni+Al+1 wt.% TiN	Planetary mill	27.43 (26.48)

*Histograms of the volume distribution of particles over their size were determined using an LS 13 320 (Beckman Coulter) laser diffraction analyzer.

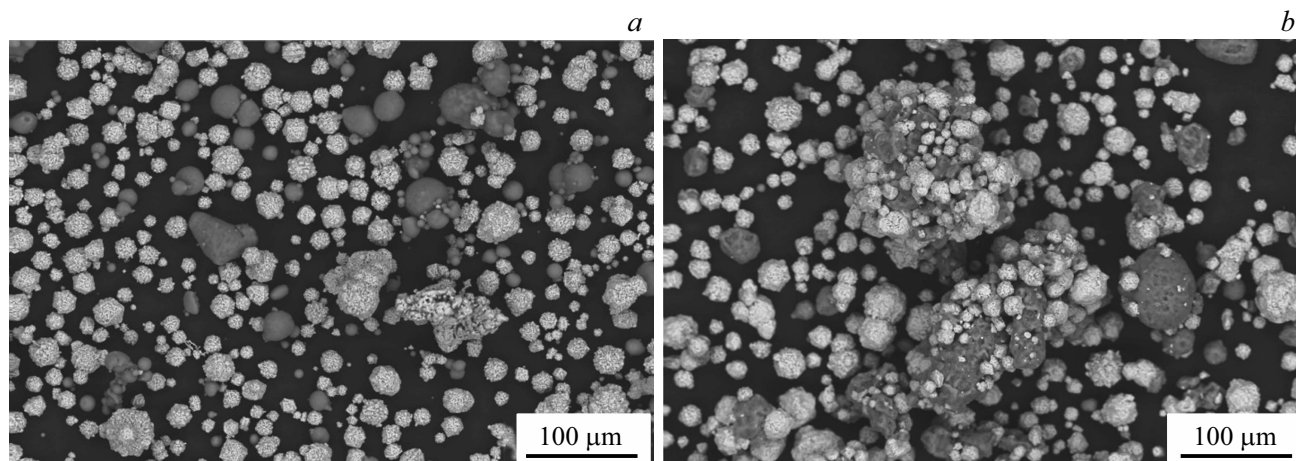


Figure 1. Overall view of the powder mixture of nickel and aluminum after mixing in a V-shaped mixer (a) and mechanical processed (b). Light and dark particles correspond to nickel and aluminum, respectively.

Just as in [8], the mixtures were deposited onto a titanium (VT20) substrate using a high-pressure experimental CS setup (Khristianovich Institute of Theoretical and Applied Mechanics, Siberian Branch, Russian Academy of Sciences).

The morphology and the structure of samples were examined with an Evo MA15 (Carl Zeiss) electron microscope with a detector of back-scattered electrons. Open-source software (ImageJ) was used to characterize the coating components quantitatively. The net area of nickel particles in the analyzed region was determined by analyzing ten microphotographic images of cross sections of coatings; the obtained value was then used to estimate the weight fraction of nickel in the material.

Samples were processed thermally in an RM-1700AV (LLC „Rusuniversal“, Chelyabinsk, Russia). The working chamber was evacuated to residual pressure $p = 10^{-3}$ MPa in preparation to this procedure. Samples with coatings were held for 1 h at a temperature of 500°C in the working chamber.

The powder use factor is one of the key parameters characterizing the process of coating deposition. The specific masses of coatings and the relative powder use factors, which were determined as the ratio of masses of coatings produced from a mechanically processed powder and the initial particles, are listed in Table 2.

Figure 2 presents the overall view of coatings. These coatings have a close structure with nickel particles distributed uniformly over the volume.

It can be seen that the coating produced from the powder mixture processed mechanically in the planetary mill contains a greater number of nickel particles. This conclusion is verified by the results of examination of the elemental composition and graphical analysis of images of cross sections (Table 2).

It follows from Table 2 that the use of the powder mixture processed mechanically in the planetary mill prior to spraying is advantageous: the powder use factor increases ~ 1.5 -fold, since coatings are formed from agglomerated particles with a residual porosity. Similar results were obtained in [9]. The weight percentage of nickel in the coating material increases relative to the one determined for the coating produced from powder mixture No. 1 (Table 2). However, the weight percentage of nickel in the coating material is not sufficient for all mixture components to react fully.

Figure 3, a presents cross-section images of thermally treated coatings with porosity (attributable to the diffusion of aluminum into the bulk of nickel particles) distributed over the material volume. The process of *in situ* synthesis in microvolumes is accompanied by the emergence of new chemical compounds, such as NiAl , Ni_3Al , and Al_2O_3 . This was verified by the results of X-ray diffraction analysis.

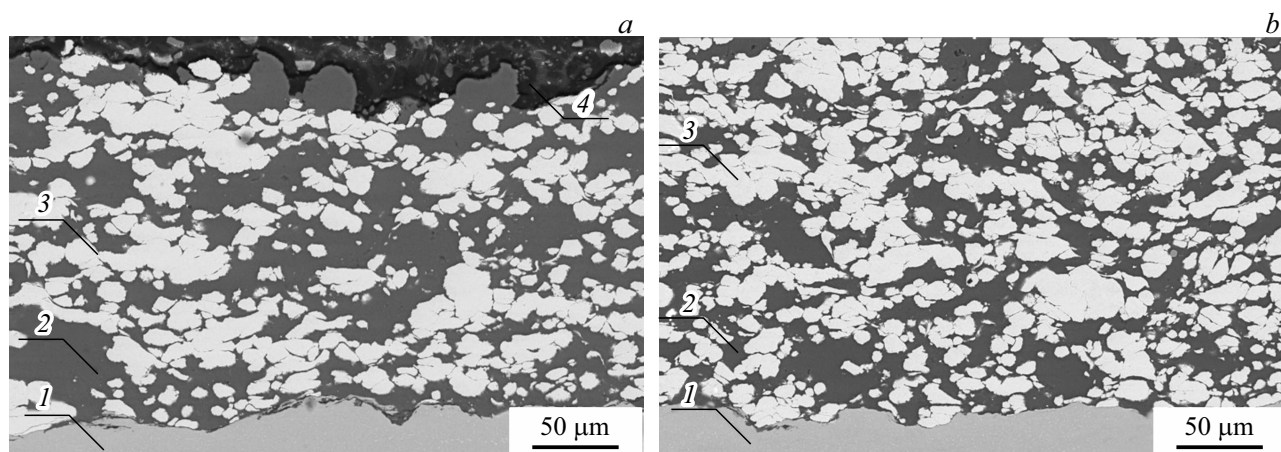


Figure 2. Microphotographic images of cross sections of coatings. *a* — Mixture № 1, *b* — mixture № 2. 1 — Substrate, 2 — aluminum, 3 — nickel, 4 — resin.

Table 2. Characteristics of coatings

Mixture number	Specific mass of the coating, kg/m ²	Powder use factor	Nickel fraction, wt.%	
			Analysis of images of cross sections	Elemental analysis
1	1.56	1	70.1	68.8
2	2.55	1.63	76.8	75.5
3	2.37	1.52	77.2	75.9

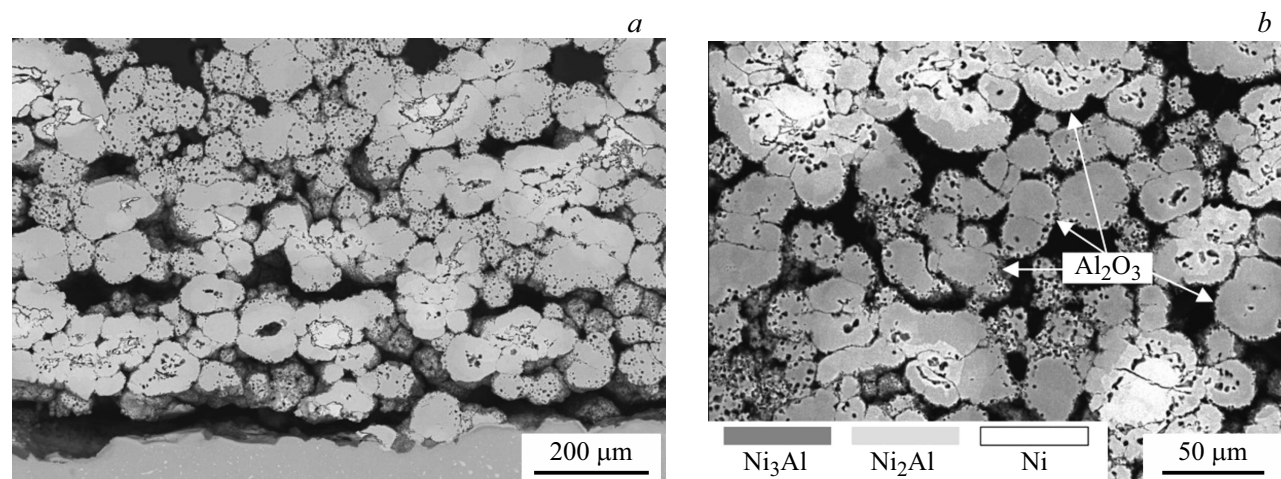


Figure 3. Microphotographic images of cross sections of thermally treated coatings under various magnifications. *a* — Mixture № 1, *b* — mixture № 2.

According to the energy-dispersive analysis data, particles with a gradient phase composition are present in the coating. Particles may contain large Ni lamellae, which are normally located at the center. NiAl forms (due to an insufficient aluminum concentration) in the middle section of a particle, and intermetallic compound Ni₃Al is synthesized in the near-surface layer. The particle surface is coated by a thin aluminum oxide film, which has a higher melting point and is a diffusion barrier for aluminum (Fig. 3, *b*).

Thus, the procedure of mechanical processing of a Ni–Al powder mixture in a planetary mill performed prior to spraying allows for a ~ 1.5-fold enhancement of the powder use factor and makes it possible to increase the weight percentage of nickel in the coating material. In order to make the coating material completely intermetallic, one needs to process samples thermally in the working chamber of a furnace in an inert gas (argon) and raise the temperature and the holding time.

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Conflict of interest

The authors declare that they have no conflict of interest.

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