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Properties of coatings obtained from a mechanically treated boron–containing mixture of powders by cold spray method

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Received April 22, 2022

Revised May 18, 2022

Accepted May 19, 2022

For composite coatings obtained by cold spraying from powder mixtures of compositions Al + 50 wt.% B and Al + 50 wt.% B₄C prepared in a V-shaped mixer and by mechanical processing in a planetary mill, a comparison of effect of these methods for preparing mixtures on the boron (boron carbide) content in the coating was carried out. It is shown that, regardless of the method of preparation, the boron and/or boron carbide contents in the coating have close values, which is confirmed by the results of X–ray phase analysis and visual processing of images of the coating structure.

Keywords: cold spray, composite material, high–energy planetary mill, aluminum, boron carbide, X–ray phase analysis.

DOI: 10.21883/TPL.2022.07.54035.19230

The cold spray method is characterized by high particle speeds (up to 1000 m/s) and particle temperatures not higher than 600°C. Thus, deposition of composite particles with a low content of metal binder (below 20 wt.%) results in the substrate erosion without forming a continuous layer [1].

Many papers are devoted to fabricating composite coatings by the cold spray method from a powder mixture of the pre–specified composition (see, e.g., [2–6]). The composite mixtures are conventionally prepared using a vibration machine or V-shaped mixture. The specific feature of the prepared mixture is that its particles inherit the sizes, morphology, structure, microhardness and phase composition of the initial powder particles. Characteristics of the obtained coatings, including the volume fraction of the fixed solid particle component, may be affected only by its volume fraction content in the deposited mixture and its granulometric composition [6].

Papers [7,8] have shown the benefits of using a high–energy planetary mill in preparing the composite powder mixture. In the mixture under treatment there are formed agglomerated particles characterized by a developed surface and sizes of 10 to 100 μm and containing in their volumes ceramic particles 0.1 to 20 μm in size. The goal of this work was to achieve the maximum volume concentration of ceramics in the coating in using different methods for preparing the composite mixture of the Al + 50 wt.% B or Al + 50 wt.% B₄C composition.

In this study, powders of aluminum (ASD-1), polycrystalline boron (SPECS 113-12-11.098–88) and boron carbide (F 220) were used.

The powder mixture was prepared in air using high–power planetary mill „Activator-2SL“ the treatment parameters are listed in Table 1; the weight of loaded balls

was 160 g per drum, weight of the loaded material to be treated was 100 g. As the grinding bodies, steel balls 5 mm in diameter were used.

When the time limit of mechanical treatment in the planetary mill specified in the table was exceeded, the aluminum particles reached a temperature close to the melting point; as shown in [9,10], this resulted in particle adhesion to the grinding body surfaces and cup walls. Fig. 1 demonstrates the appearance of the treated composite mixture; Table 1 presents its granulometric composition.

Phase compositions of the powder mixtures and coatings were studied by X–ray phase analysis at diffractometer D8 ADVANCE (Bruker, USA) using the monochromatic CuK_α-radiation. Analysis of the X–ray diffraction patterns showed that there occurs nothing but intermixing of powders with formation of composite particles without chemical transformations.

The mixtures were deposited at the experimental high–pressure cold–spray setup (ITAM SB RAS, Russia) equipped with an axially symmetric Laval nozzle 150 mm long with critical and outlet cross–section diameters of 2.8 and 6.5 mm, respectively. As the operating gas, compressed air with the brake pressure of 4.0 MPa and brake temperature of 573 K was used. The spray–deposition distance (the distance between the nozzle edge and substrate surface) was 30 mm. To obtain a single continuous coating layer over the entire substrate surface, the nozzle was moved relative to the stainless–steel substrate with the speed of 100 mm/s along a zig–zag trajectory at the shift interval of 3 mm.

Morphology and structure of the samples were studied using electron microscope Evo MA15 (Carl Zeiss) with a backscattered electron detector. The coating porosity was determined by using metallographic microscope OLYMPUS GX-51.

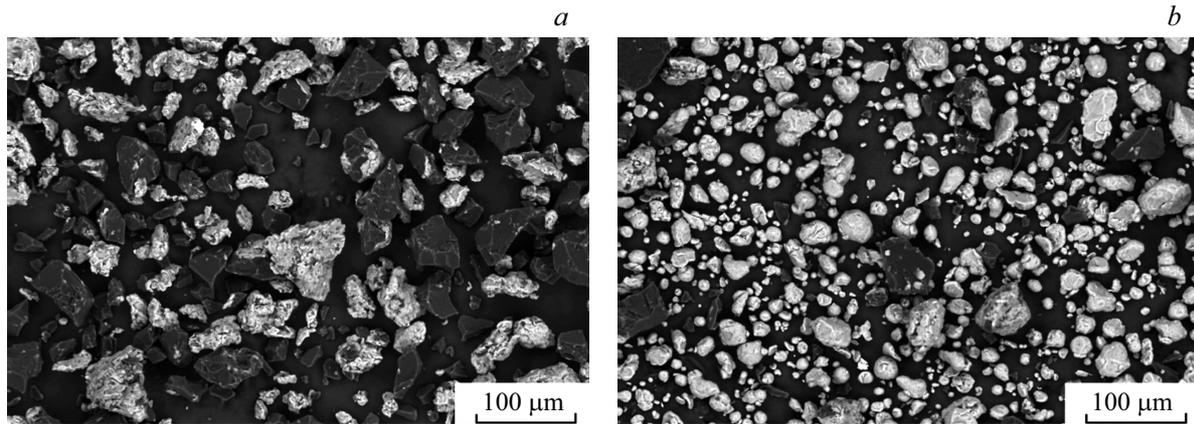


Figure 1. General view of the mechanically treated composite mixture of aluminum and boron carbide (*a*) and aluminum and polycrystalline boron (*b*). The mechanical treatment parameters are: *a* — 3 min, $a_b = 117g$; *b* — 1 min, $a_b = 60g$ (a_b — is the grinding bodies acceleration).

Table 1. The powder mixture composition, type and mechanical treatment parameters for the cold spray method

Mixture No.	Powder mixture composition	Type and parameters of mechanical treatment	Mean size (standard deviation)*, μm
1	Al + 50 wt.% B ₄ C	V-shaped mixer, 30 min	—
2	Al + 50 wt.% B ₄ C	Planetary mill, 3 min, $a_b = 117g$	44.81 (31.74)
3	Al + 50 wt.% B	Planetary mill, 1 min, $a_b = 60g$	49.22 (25.34)

*Histograms of the particle size volume distribution were obtained using laser diffraction analyzer LS 13 320 (Beckman Coulter).

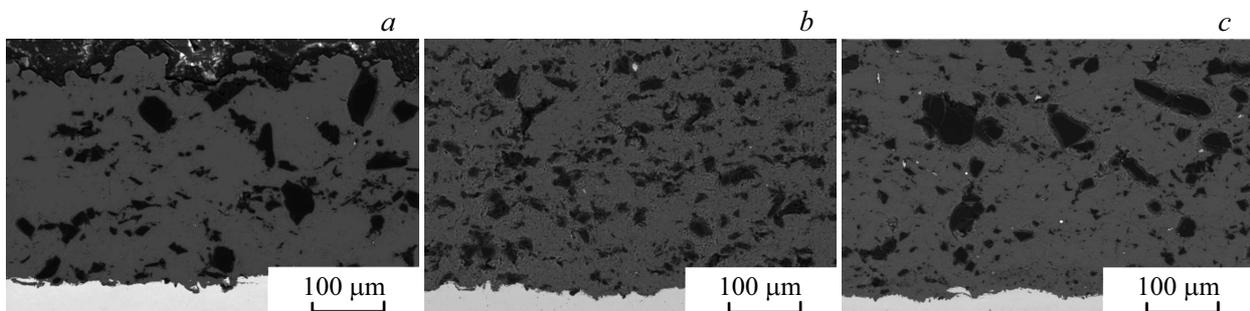


Figure 2. Microphotos of the coating cross-sections: *a* — mixture 1, *b* — mixture 2, *c* — mixture 3.

Fig. 2 demonstrates the coating cross-sections. The deposited coatings had a dense structure with boron carbide (boron) particles distributed over its volume. One can see that coatings fabricated from mixtures 2 and 3 (Table 1) contain a greater number of solid state particles, which is confirmed by the analysis of elemental composition that was determined other things being equal (Table 2).

Table 2 shows that the coating structure contains only basic chemical elements constituting the deposited powder mixture.

To quantitatively characterize the coating components, program package with open source code ImageJ was used.

The total area of boron (boron carbide) particles fixed in the analyzed region was determined for the coatings (Fig. 2 and Table 3). The coating characteristics, such as microhardness and boron carbide (boron) volume fraction, are given in Table 3.

Thus, we have shown that deposition of coatings consisting of mechanically agglomerated composite particles (mixtures 2 and 3) leads to a certain (insignificant) increase in the boron and/or boron carbide volume fraction in the coating material as compared with their fraction in the coating fabricated from powder mixture 1 (Table 1); this is additionally confirmed by the results of the X-ray phase analysis (Table 3).

Table 2. Elemental composition of the coating cross-section

Element	Mixture 1		Mixture 2		Mixture 3	
	at.%	wt.%	at.%	wt.%	at.%	wt.%
Al	15.92	31.68	13.49	27.65	13.42	48.01
B	65.82	52.65	67.72	54.17	86.58	51.99
C	18.26	15.67	18.79	18.18	–	–
Total	100.00		100.00		100.00	

Table 3. Coating characteristics

Mixture No.	Microhardness HV _{0.3}	Volume of boron carbide (boron) particles, %	
		Structure analysis	X-ray phase analysis
1	72 ± 4	15–18	13
2	97 ± 4	15–20	15
3	120 ± 20	15–20	14

Acknowledgements

The authors are grateful to T.M. Vidyuk (Federal State Budget Research Institution Khristianovich Institute of Theoretical and Applied Mechanics, SB RAS) for preparing the samples.

Financial support

The study was supported by the Russian Fundamental Research Foundation in the framework of research project № 20-21-00046; the work was performed at the facilities of CCU „Mechanics“ (Khristianovich Institute of Theoretical and Applied Mechanics, SB RAS).

Conflict of interests

The authors declare that they have no conflict of interests.

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