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COMPOSITE POWDERS BASED ON FeMoNiCrB AMORPHIZING ALLOY WITH ADDITIVES OF REFRACTORY COMPOUNDS FOR THERMAL SPRAYING OF COATINGS

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ABSTRACT

The process of producing by mechanical alloying method composite powders based on FeMoNiCrB amorphizing alloy with additives of refractory compounds (ZrB₂, (Ti,Cr)C and FeTiO₃) by processing a mixture of powder components in planetary mill "Activator 2SL" was studied. It was found that after 1.5 h of processing its products are homogeneous conglomerates with average particle size, depending on the composition of composite powders, which was 23 μm in the case of FeMoNiCrB + ZrB₂, 15 μm in the case of FeMoNiCrB+(Ti, Cr)C, and 25 μm in the case of FeMoNiCrB + FeTiO₃. XRD analysis results reveal formation of an amorphous-nanocrystalline structure in the produced composite powders, which consists of Fe(Ni, Cr), Mo₅FeB₂ solid solution and disperse phase of additives of refractory compounds (ZrB₂, (Ti, Cr)C and FeTiO₃) with indications of certain interphase interaction of FeMoNiCrB and (Ti, Cr)C. Here, the amorphous phase appears as a result of the process of mechanical alloying. The values of measured microhardness in the composite powders were as follows: FeMoNiCrB + ZrB₂ – 6.2±1.6 MPa, FeMoNiCrB + (Ti, Cr)C – 5.5±1.1 MPa, FeMoNiCrB–FeTiO₃ – 5.1±0.7 MPa. For application in thermal spraying of coatings with an amorphous structure, the produced composite powders from mechanical alloying products are used to form conglomerate powders with particle size and degree of flowability, meeting the requirements of thermal spray coating technology.

KEY WORDS: amorphizing alloy, refractory compounds, composite powder, mechanical alloying, structure, phase composition, granulometric composition

INTRODUCTION

Progress in creating thermal spray coatings (TSC) with improved functional characteristics is largely determined by development of new types of materials for their deposition, including composite powders (CP).

Promising materials for TSC include alloys with an amorphous structure, which are characterized by a combination of such complexes of properties, as high strength, wear resistance, corrosion resistance, high yield limit, high magnetic permeability and small coercive force, high electric resistance and superconductivity [1, 2].

Formation of material amorphous structure takes place as a result of suppression of the processes of nucleation and crystallization, which is achieved at ultrafast quenching or introducing defects into the metal crystalline structure [2]. Particle cooling at formation of the coating layer under the conditions of thermal spraying at the rate of ~ 10⁶ K/s, enables producing

coatings with a metastable amorphous structure on the part surface [3].

Among amorphous thermal spray coatings, which are promising for commercial application on parts, exposed to wear in operation, we should single out iron-based alloys, which are characterized by high values of mechanical strength and corrosion resistance, and low cost [4]. However, their low values of wear resistance at dry friction and poor impact resistance limit the range of possible applications of such coatings [5].

Improvement of mechanical properties of iron-based amorphous coatings can be achieved by adding dispersed particles to the amorphous matrix. So, application of WC/12Co as inclusions allowed improving the adhesion strength and increasing the friction/wear resistance four times, compared to iron-based coating, having a purely amorphous structure [5]. Increase of amorphous coating wear resistance is also observed at application of dispersed TiN and B₄C inclusions [6, 7].

An important factor, influencing the properties of TS coatings is the uniformity of their structure. In

Table 1. Characteristics of initial powders

Material	Chemical composition, wt. %	Production method	T_m , K	γ , g/cm ³	HV, GPa
FeMoNiCrB	Fe – 36.2; Mo – 29.9; Ni – 23.6; Cr – 7.6; B – 2.7	Melt atomization by nitrogen	1523	7.84	6.2±1.2
ZrB ₂	Zr – 79.5; B – 19.0	Carbon-thermal method of zirconia reduction TU 6-09-03-46-75	3323	6.1	15.0±2.5
(Ti, Cr)C (ICTYX)	Ti – 54; Cr – 25.5; C _{total} – 18.6; C _w – 0.7	Self-propagating high-temperature synthesis TU No.88 Ukr.SSR 147.040-87	~3023	5.35	14.1±2.5
FeTiO ₃	Fe – 36.8; O – 31.6; Ti – 31.6	Crushing and grinding of ilmenite ore	1638	4.72	6.1±0.98

work [8] it is shown that TS coatings produced from a mechanical powder mixture, do not have a homogeneous composition by the coating thickness that affects the strength of the bond between the strengthening phase with the matrix material. At the same time, CP coatings have a layered fine lamellar structure and feature higher degree of density and homogeneity [9].

The main methods of producing CP include cladding and conglomeration, as well as a combination of these methods [10]. Among the modern methods of producing CP those of mechanical alloying (MA) and mechanochemical synthesis (MCS) should be noted.

The process of CP manufacture by MA or MCS methods consists in processing powder components in the high-power ball mills with a high content of grinding balls. During mechanical processing, the balls collide with each other, resulting in energy evolution. Ball collision is of a pulsed nature occurring for a short period of time of 10^{-12} – 10^{-13} s, and at penetration of powder particles into the contact region the energy is partially consumed by their deformation and refinement, and partially leads to increase of their internal energy. Internal energy relaxation occurs in different ways: heat evolution; multiplication and migration of crystalline structure defects; formation of internal interfaces; excitation of chemical reactions. Appearance of juvenile surfaces, as well as linear and point defects coming to the surface promote “cold” welding of the particles to each other with formation of composite particles of a layered structure. With longer processing time, the particles acquire a homogeneous structure, and a balance is achieved between the processes of their refinement and “cold” welding, leading to stabilization of the average particle size [11, 12]. It should be also noted that when conducting the MA process formation of an amorphous structure in the powder particles promotes generation of crystalline structure defects in them during MA.

The objective of the work was producing composite powders for thermal spraying of coatings with an amorphous-crystalline structure by mechanical alloying of a mixture, consisting of an amorphizing

FeMoNiCrB alloy and additive of one of the refractory compounds (ZrB₂, (Ti,Cr)C and FeTiO₃).

INVESTIGATION MATERIALS AND PROCEDURES

PG-N4 analog (FeCrMoB) was selected as the amorphizing alloy, which contains approximately 24 wt.% nickel in its composition. The most widely applied representatives of ceramic refractory compounds: zirconium boride ZrB₂, double titanium-chromium carbide (Ti, Cr)C (solid solution of 24 vol.% Cr₃C₂ in TiC) and FeTiO₃ (ilmenite) were selected as CP alloying phases. Table 1 gives the initial material characteristics.

Particle distribution by sizes, both for initial powders and for MA products, was determined both using Mastersizer 2000 analyzer of Malvern Panalytical Company, Great Britain, and by sieving analysis in compliance with DSTU 2640-94.

CP was produced in planetary mill “Activator 2SL” at the speed of rotation of the central axle of 1000 rpm, and of the drums — of 1500 rpm. The drum body and the balls were made from ShKh15 steel. In order to prevent welding of the mixture to the walls of the drums and crushing bodies during material processing, a surfactant (SA) was added to the mixture: oleic acid (CH₃(CH₂)₇CH = CH(CH₂)₇COOH) in the quantity of 0.5 wt.%. The process was conducted in air with taking of intermediate samples every 0.5 h of processing.

Microdurometric analysis of the initial particles and obtained products was conducted in microhardness meter PMT-3 at 25 g load on the indenter with subsequent plotting of variation curves of microhardness values by the results of 30 measurements [13].

Chemical analysis of the produced CP was performed in electron microscope JSM-6390LV (JEOL, Japan) with energy-dispersive (EDS) attachment INCA ENERGY (Oxford Instrument, Great Britain), in secondary electron mode in low (10^{-4} Pa) vacuum, with 20 kV accelerating voltage.

X-ray structural phase (XRD) analysis of the produced CP was conducted in “DRON-UM-1” unit, in CuK_α radiation, monochromatized.

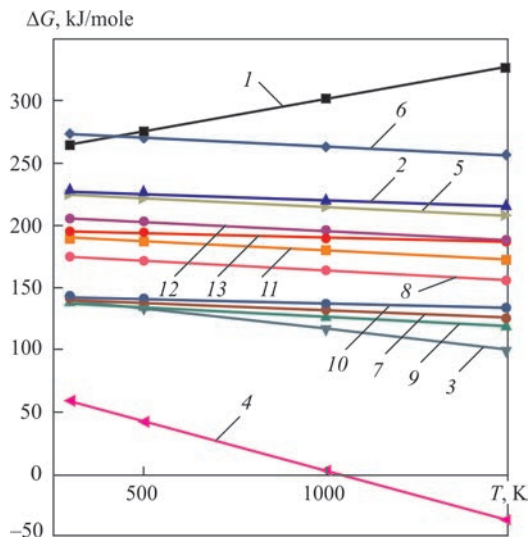


Figure 1. Temperature dependence of the change of Gibb's energy of Me + ZrB₂ reaction, where Me is Fe, Mo, Ni, Cr, B: 1 — 2Fe + ZrB₂ = 2FeB + Zr; 2 — Mo + ZrB₂ = MoB₂ + Zr; 3 — 2Mo + ZrB₂ = 2MoB + Zr; 4 — 4Mo + ZrB₂ = 2Mo₂B + Zr; 5 — Ni + ZrB₂ = NiZr + 2B; 6 — 1/2Ni + ZrB₂ = 1/2NiZr₂ + 2B; 7 — 7/2Ni + ZrB₂ = 1/2Ni₇Zr₂ + 2B; 8 — 5Ni + ZrB₂ = Ni₅Zr + 2B; 9 — 3Ni + ZrB₂ = Ni₃Zr + 2B; 10 — 23/8Ni + ZrB₂ = 1/8Ni₂₃Zr₈ + 2B; 11 — 10/7Ni + ZrB₂ = 1/7Ni₁₀Zr₇ + 2B; 12 — 11/9Ni + ZrB₂ = 1/9Ni₁₁Zr₉ + 2B; 13 — Cr + ZrB₂ = CrB₂ + Zr

THERMODYNAMIC EVALUATION OF THE REACTIONS AT AMORPHIZING ALLOY INTERACTION WITH REFRACTORY COMPOUNDS AND SELECTION OF COMPOSITE POWDER COMPOSITION

Thermodynamic calculations were performed to determine the probability of running of the reactions be-

tween metal matrix elements (Fe, Mo, Ni, Cr and B) and refractory compound inclusions (ZrB₂ (Ti, Cr)C and FeTiO₃) at heating up to 1500 K. In connection with absence of published data on heat of formation and entropy of double titanium-chromium carbide (Ti, Cr)C, thermodynamic data for Cr₃C₂ and TiC carbides were used for calculations. The results of thermodynamic calculations are given in Figures 1–3.

Analysis of the obtained data leads to the following conclusions.

1. Fe, Cr and Ni interaction with ZrB₂ is thermodynamically impossible below 1500 °C, Mo₂B only forms as a result of ZrB₂ interaction with Mo at more than 1000 °C temperature.

2. Thermodynamic calculations for TiC and Cr₃C₂ show that interaction with boron results in formation of titanium and chromium borides (TiB₂ and CrB₂), respectively. More over, in Mo + Cr₃C₂ system a reaction can proceed with formation of molybdenum carbide Mo₂C, and in Ni + Cr₃C₂ — a reaction with formation of nickel carbide (Ni₃C).

3. Thermodynamic calculations for Me + FeTiO₃ system show that oxides, namely TiO₂, Cr₂O₃ and B₂O₃ can form in this system.

RESULTS OF EXPERIMENTAL STUDIES

At selection of the composition of powder mixture to produce composite particles in this study, the authors were guided by recommendations on production of wear-resistant composite materials by powder metal-

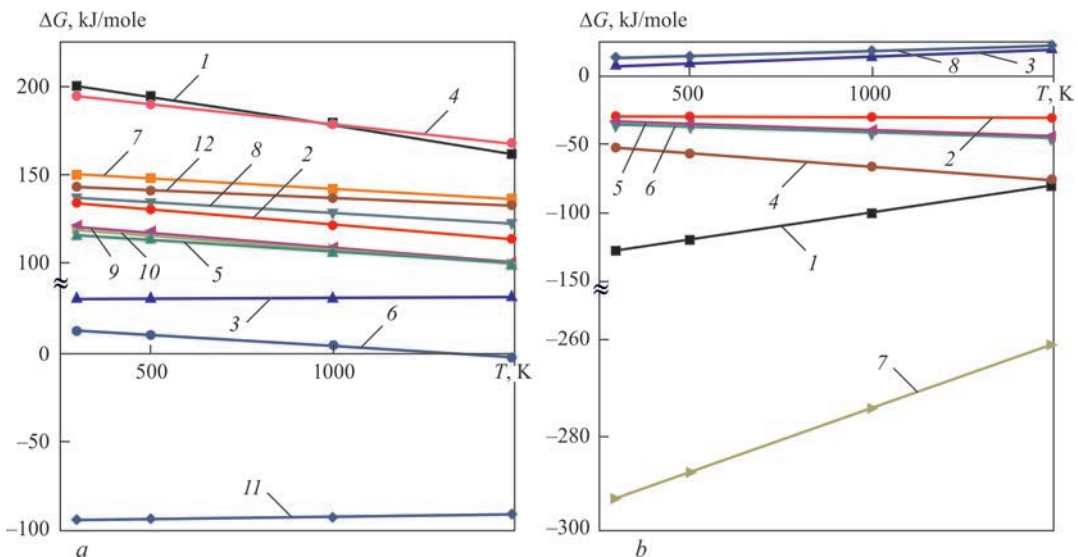


Figure 2. Temperature dependence of the change of Gibb's energy of the following reactions: a – Me + TiC; b – Me + Cr₃C₂, where Me – Fe, Mo, Ni, Cr, B; for a: — 1 – 3Fe + TiC = Fe₃C + Ti; 2 — 2Mo + TiC = Mo₂C + Ti; 3 — 3/2Mo + TiC = 1/2Mo₃C₂ + Ti; 4 — 3Ni + TiC = Ni₃C + Ti; 5 — Ni + TiC = NiTi + C; 6 — 3Ni + TiC = Ni₃Ti + C; 7 — 1/2Ni + TiC = 1/2NiTi₂ + C; 8 — 3/2Cr + TiC = 1/2Cr₃C₂ + Ti; 9 — 7/3Cr + TiC = 1/3Cr₇C₃ + Ti; 10 — 23/6Cr + TiC = 1/6Cr₂₃C₆ + Ti; 11 — 2B + TiC = TiB₂ + C; 12 — 4B + TiC = B₄C + Ti; for b: — 1 – 6Fe + Cr₃C₂ = 2Fe₃C + 3Cr; 2 — 3Mo + Cr₃C₂ = Mo₃C₂ + 3Cr; 3 — 4Mo + Cr₃C₂ = 2Mo₂C + 3Cr; 4 — 6Ni + Cr₃C₂ = 2Ni₃C + 3Cr; 5 — 14/3Cr + Cr₃C₂ = 1/3Cr₁₄C₃; 6 — 5/3Cr + Cr₃C₂ = 2/3Cr₅C₃; 7 — 6B + Cr₃C₂ = 3CrB₂ + 2C; 8 — 8B + Cr₃C₂ = 2B₄C + 3Cr

lurgy methods, according to which the dispersed inclusion content is equal to 20–30 vol.% [14].

Figure 4 shows the appearance and microstructure of initial powder particles, and Figure 5 gives their distribution by size. As once can see, the particles of FeMoNiCrB powder are of a spherical shape, which is characteristic for powders produced by the method of melt atomization with nitrogen. Powders of ceramic compounds are of an angular shape, characteristic for hard and brittle materials. Average size of FeMoNiCrB alloy particles is equal to $\sim 110 \mu\text{m}$, that of ZrB_2 and (Ti, Cr)C refractory compounds is 65 and 55 μm , respectively. Size of 96 % of FeTiO_3 particles is to the range of 40–100 μm .

XRD analysis of initial powders revealed that FeMoNiCrB alloy powder has a multiphase crystalline structure with a low degree of amorphization in the region of iron-based solid solution (Figure 6, *a*). ZrB_2 powder has no admixtures (Figure 6, *b*), a small quantity of TiO was found in (Ti, Cr)C powder (Figure 6, *c*), and Fe_2O_3 can be present in FeTiO_3 (Figure 6, *d*).

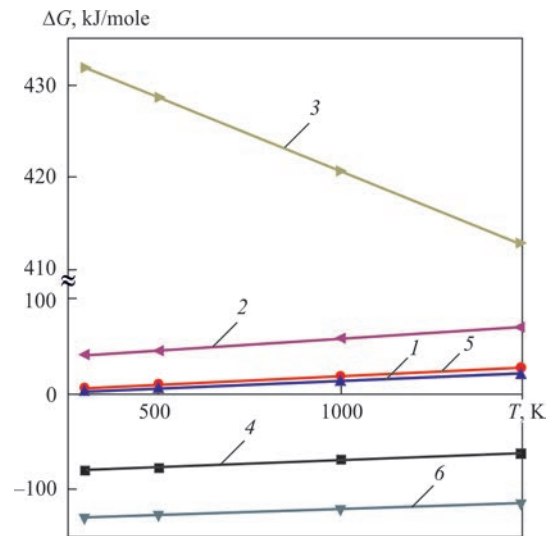


Figure 3. Temperature dependence of the change of Gibb's energy for $\text{Me} + \text{FeTiO}_3$ reaction, where $\text{Me} - \text{Fe, Mo, Ni, Cr, B}$: 1 — $1/2\text{Mo} + \text{FeTiO}_3 = \text{Fe} + 1/2\text{MoO}_2 + \text{TiO}_2$; 2 — $\text{Ni} + \text{FeTiO}_3 = \text{NiTiO}_3 + \text{Fe}$; 3 — $3/4\text{Ni} + \text{FeTiO}_3 = 3/4\text{NiFe}_2\text{O}_4 + 1/2\text{Fe} + \text{Ti}$; 4 — $2/3\text{Cr} + \text{FeTiO}_3 = \text{Fe} + 1/3\text{Cr}_2\text{O}_3 + \text{TiO}_2$; 5 — $1/2\text{Cr} + \text{FeTiO}_3 = \text{Fe} + 1/2\text{CrO}_2 + \text{TiO}_2$; 6 — $2/3\text{B} + \text{FeTiO}_3 = \text{Fe} + 1/3\text{B}_2\text{O}_3 + \text{TiO}_2$

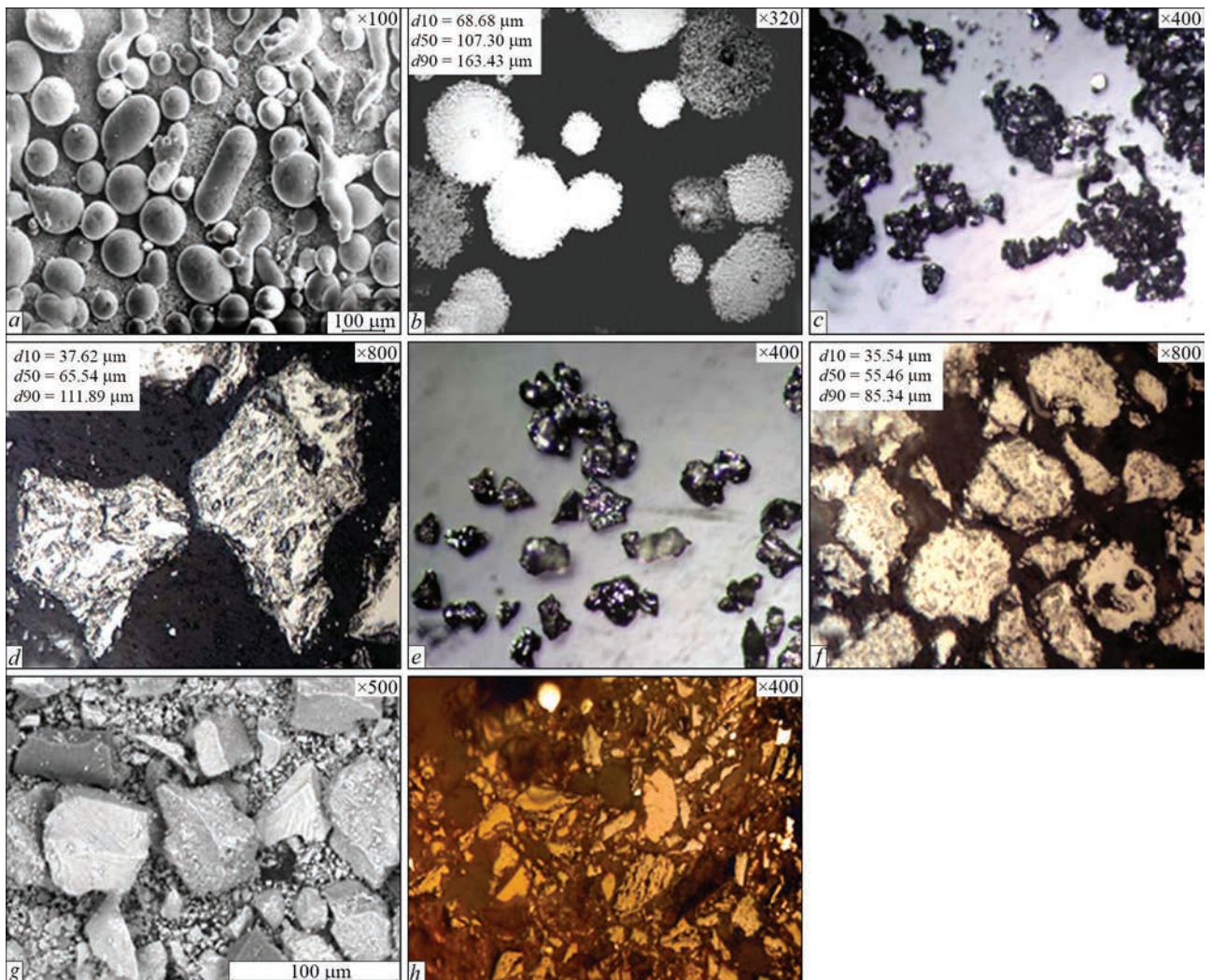


Figure 4. Appearance (*a, c, e, g*) and microstructure (*b, d, f, h*) of the following powders: *a, b* — FeMoNiCrB; *c, d* — ZrB_2 ; *e, f* — (Ti, Cr)C; *g, h* — FeTiO_3

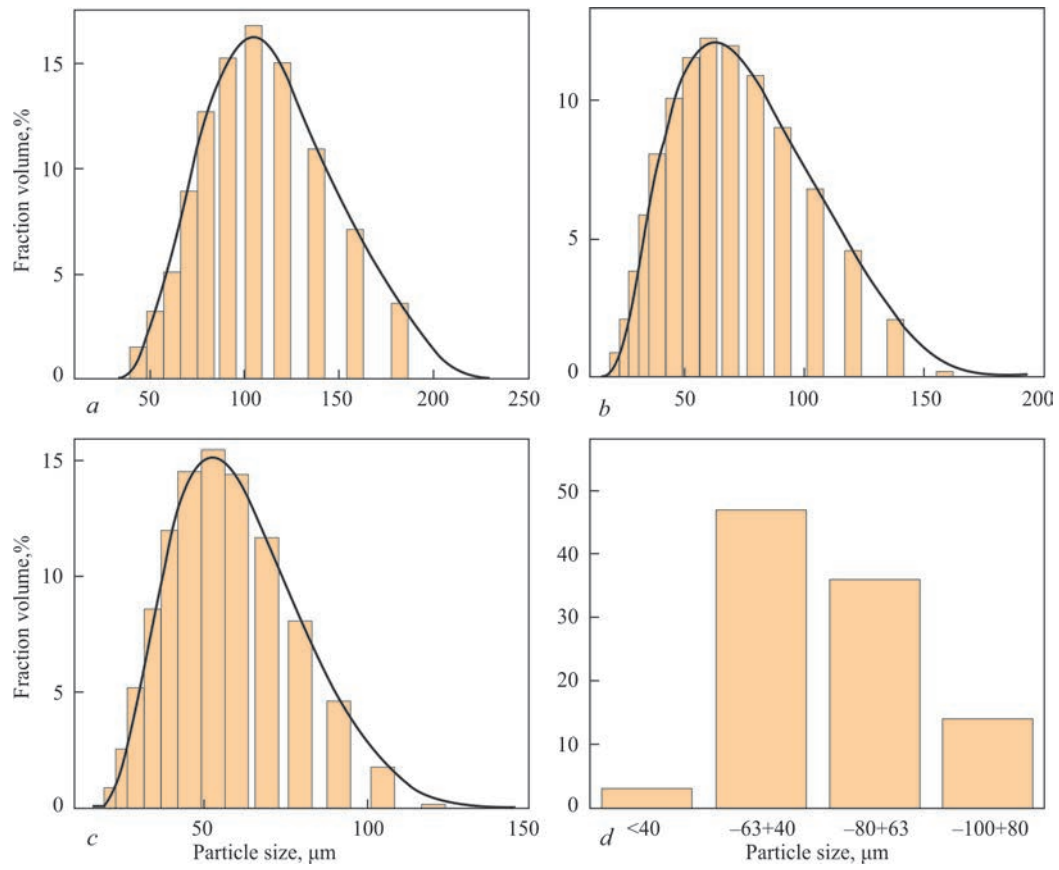


Figure 5. Distribution of initial powder particles by size: *a* — FeMoNiCrB; *b* — ZrB₂; *c* — (Ti, Cr)C; *d* — FeTiO₃ (*a*, *b*, *c* — Master-sizer 2000 analyzer; *d* — sieve analysis)

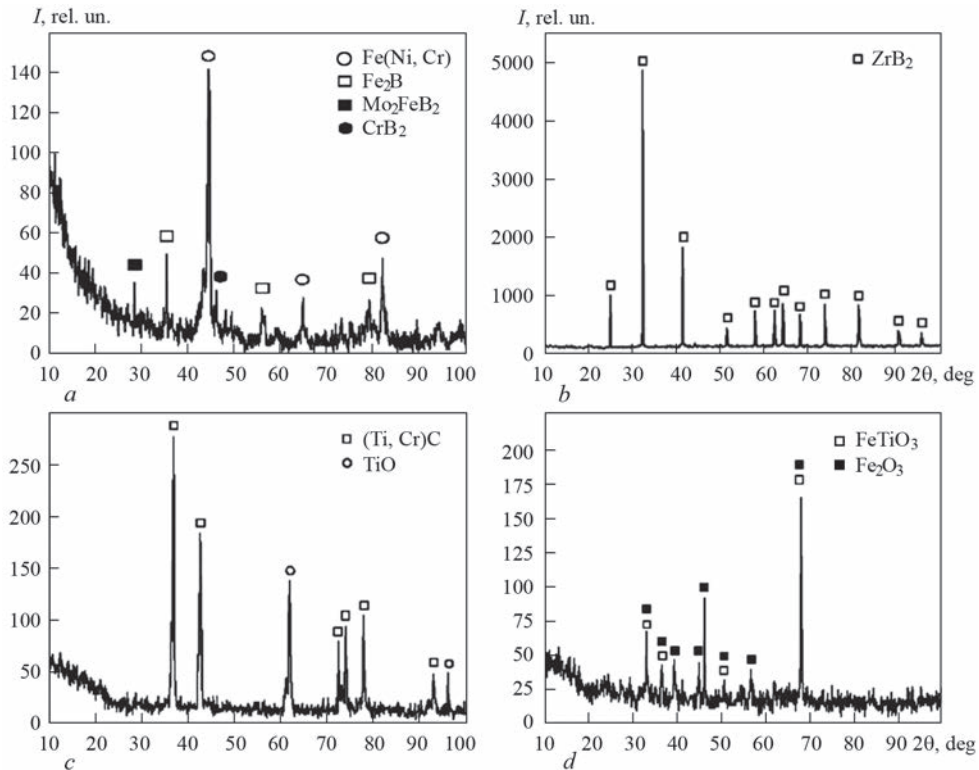


Figure 6. X-ray patterns of initial powders: *a* — FeMoNiCrB; *b* — ZrB₂; *c* — (Ti, Cr)C; *d* — FeTiO₃

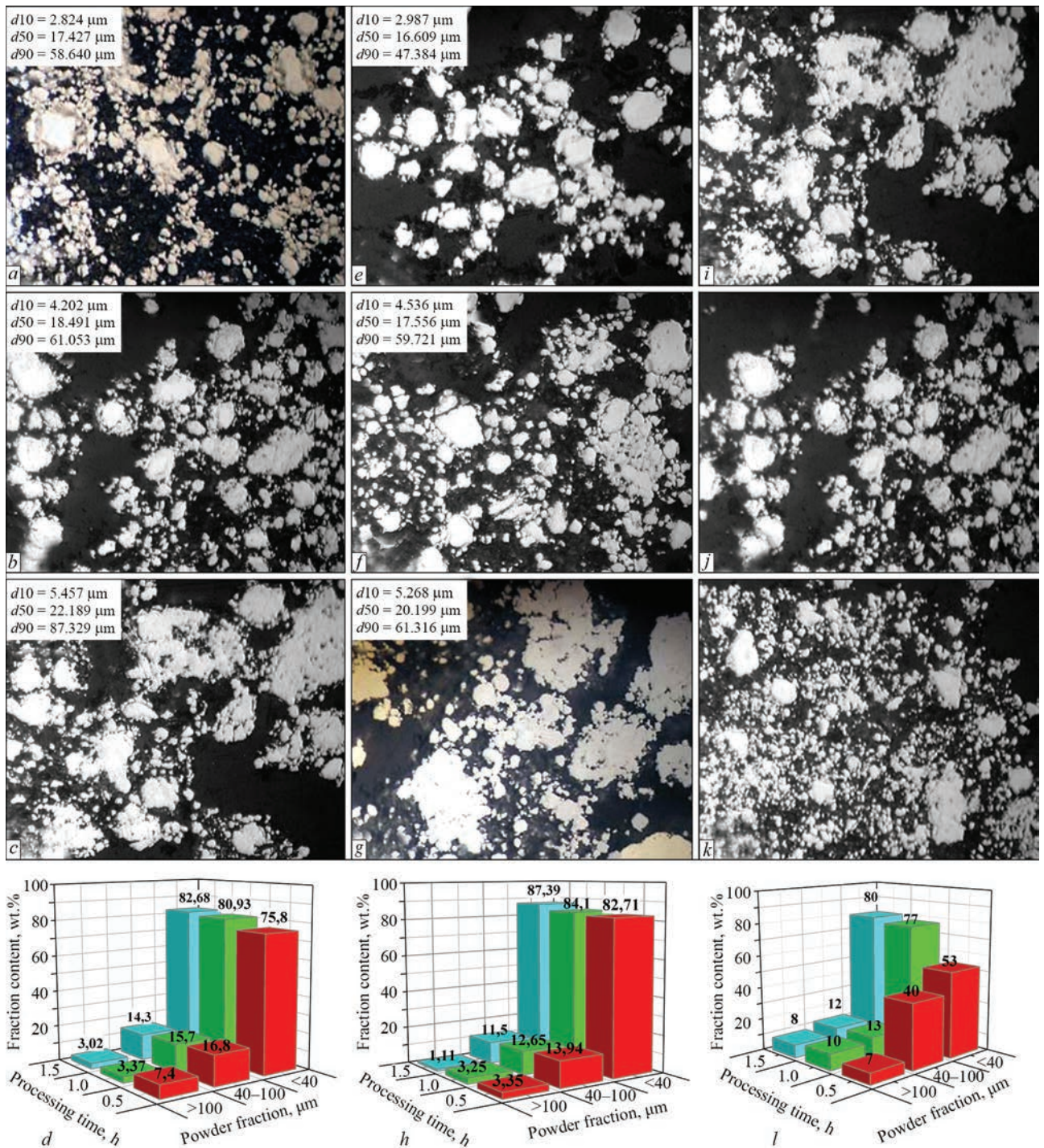


Figure 7. Microstructure ($\times 800$) (a, b, c, e, f, g, h, i, j, k) and histograms of powder particle size distributions (d, h, l) at different MA process duration for the following mixtures: a, b, c, d — FeMoNiCrB + ZrB₂; e, f, g, h — FeMoNiCrB + (Ti, Cr)C; i, j, k, l — FeMoNiCrB + FeTiO₃

Investigation of the structure and size of powder particles during CP formation showed (Figure 7) that at the initial processing stages the refinement processes prevail over those of cold welding. As a result of it, after 0.5 h of processing the size of the main mass of the particles decreases, compared to the initial one to $< 40 \mu\text{m}$, and the average particle size is equal to 16–17 μm . Further processing of the powder mixtures leads to increase of the mass of particles of $< 40 \mu\text{m}$ size (Figure 7, d, h, l). Here, the average particle size

increases up to 20–22 μm (Figure 7, c, g), which is indicative of cold welding of initial powder particles to each other with formation of agglomerates.

Investigation of size distribution of composite powder particles with inclusions of refractory compounds, depending on their processing time (Figure 8), showed that at the initial stages as a result of particle refinement the powder mixtures become bimodal with two most probable modes of 4–5 and 23–26 μm (Figure 8, a). Such a spreading can be re-

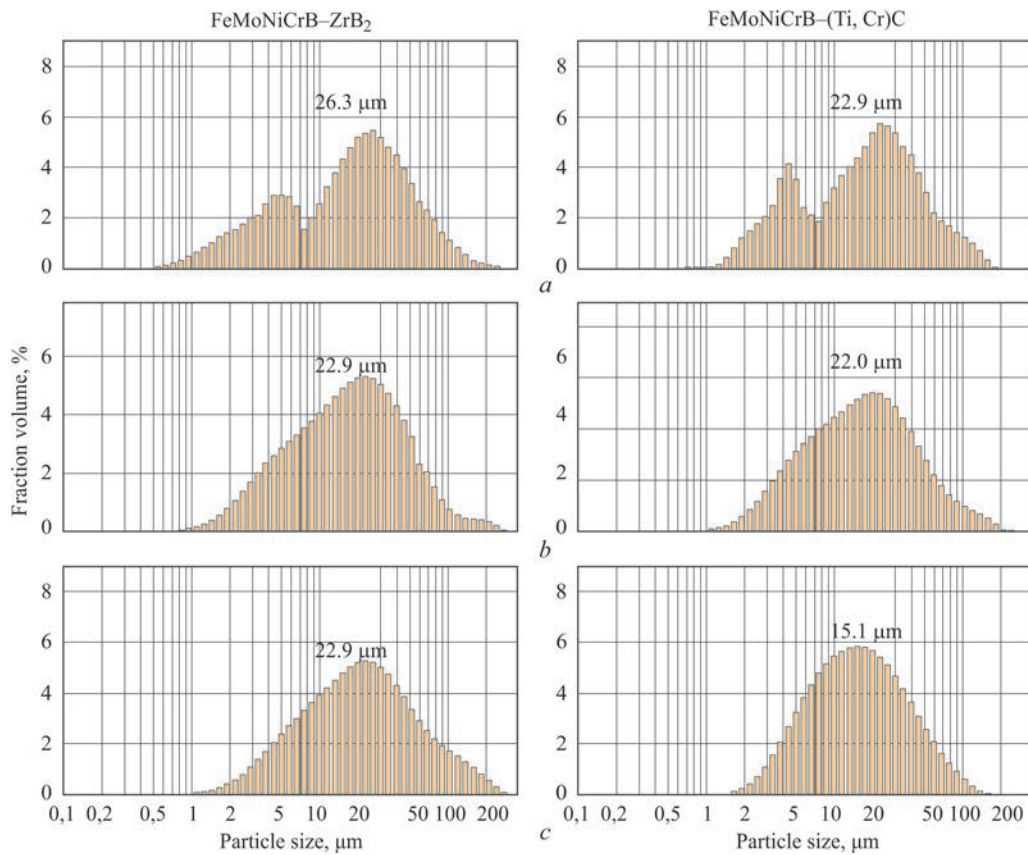


Figure 8. Particle size distribution, depending on mixture processing time: *a* — 0.5; *b* — 1.0; *c* — 1.5 h

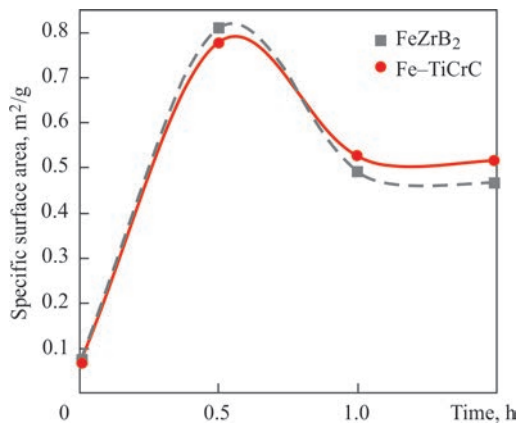


Figure 9. Change of specific surface area of CP particles, depending on their processing time

lated to a more intensive refinement of particles with a higher brittleness value. With the start of agglomerate formation (after 1.0 h of processing) particle size distribution becomes monomodal.

One of the characteristics of powder materials is the specific surface of the particles. Evaluation of the change of the particle specific surface at different stages of powder processing (Figure 9) showed that at the initial stages of processing, when the refinement processes prevail, an increase of the particle specific surface occurs with reaching of the maximum value after 0.5 h. With the start of agglomerate formation, a reduction of the particle specific surface is observed, which goes on until an equilibrium has been reached between the processes of refinement and cold welding of the particles to each other, which is indicated by a

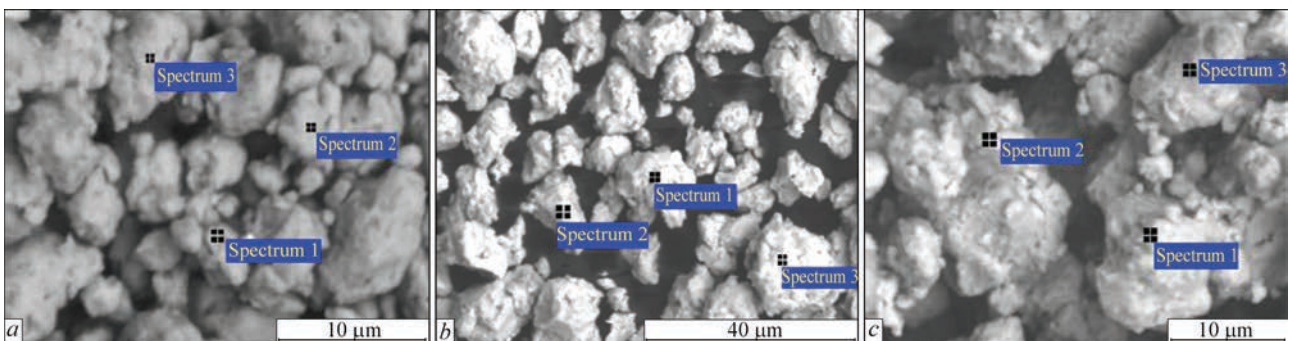


Figure 10. Appearance of particles after processing the following powder mixtures in a planetary mill for 1.5 h: *a* — FeMoNiCrB + ZrB₂; *b* — FeMoNiCrB + (Ti, Cr)C; *c* — FeMoNiCrB + FeTiO₃

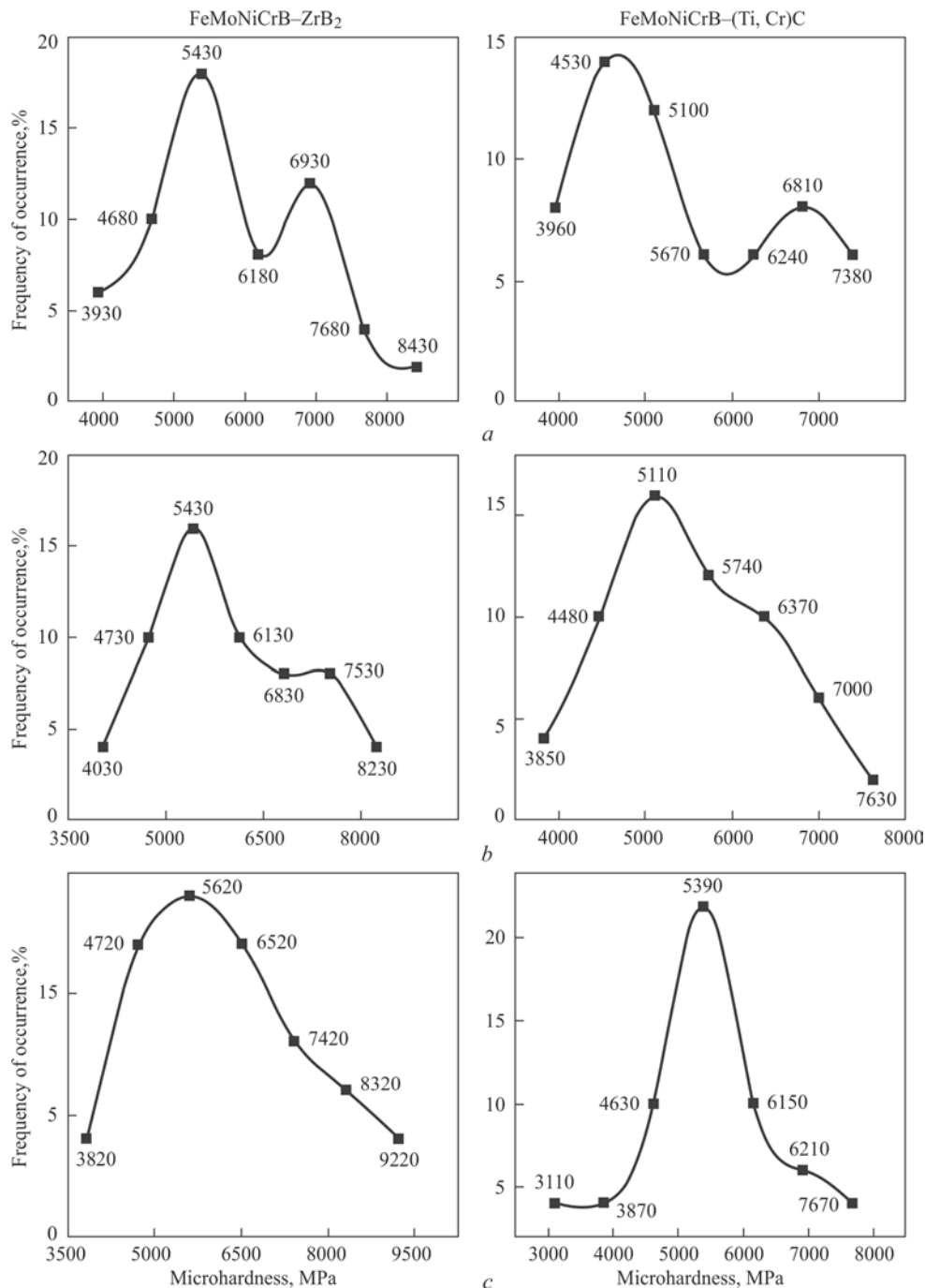
Table 2. Averaged values of particle chemical composition after processing the powder mixture for 1.5 h

FeMoNiCrB–ZrB ₂					
Fe	Mo	Ni	Cr	Zr	O
24.18±0.13	22.82±0.26	22.95±0.11	5.55±0.19	17.47±0.19	7.03±0.09
FeMoNiCrB–(Ti,Cr)C					
Fe	Mo	Ni	Cr	Ti	O
23.02±0.10	23.23±0.31	23.79±0.12	11.52±0.93	10.66±0.2	7.78±0.13
FeMoNiCrB–FeTiO ₃					
Fe	Mo	Ni	Cr	Ti	O
30.98±0.52	23.85±0.24	21.23±0.16	7.05±0.51	4.18±0.37	12.71±0.78

practical absence of changes of the specific surface of the processing products.

After processing of the powder mixtures in the planetary mill for 1.5 h, the particles of all the com-

positions take a rounded shape (Figure 10). Here, as shown by EDS analysis, each of them contains all the elements of the initial components: metal (Fe, Mo, Ni, Cr) and refractory compounds (Ti, Zr), i.e. it is a CP


Figure 11. Variation curves of particle microhardness after mixture processing for *a* — 0.5; *b* — 1.0; *c* — 1.5 h

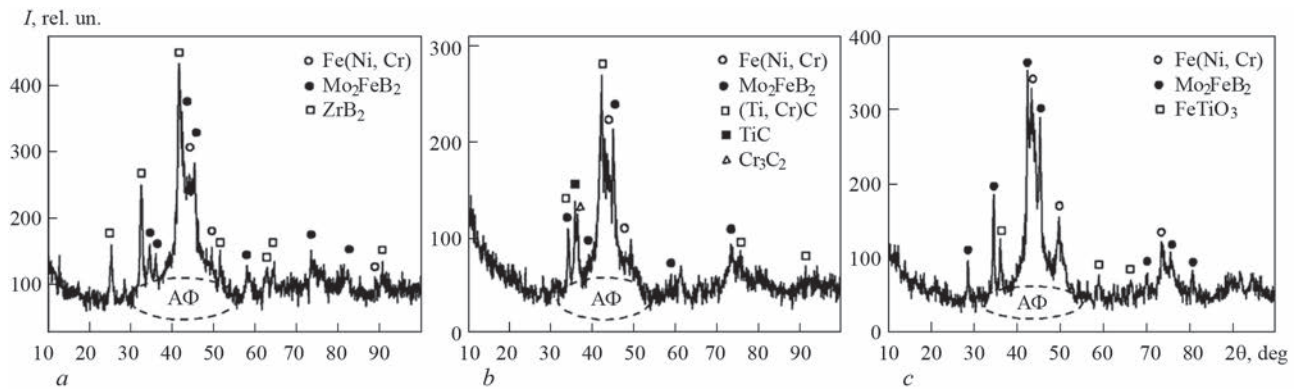


Figure 12. X-ray patterns of the following powders: *a* — FeMoNiCrB–ZrB₂; *b* — FeMoNiCrB–(Ti, Cr)C; *c* — FeMoNiCrB–FeTiO₃

Table 3. Characteristics of MA products, produced after 1.5 h

Composition	Phase composition	Particle size, μm	HV, GPa
FeMoNiCrB–ZrB ₂	Sol. sol Fe(Ni, Cr), Mo ₂ FeB ₂ , ZrB ₂ , amorphous phase	5–87	6.2±1.6
FeMoNiCrB–(Ti, Cr)C	Sol. sol Fe(Ni, Cr), Mo ₂ FeB ₂ , (Ti, Cr)C, TiC, Cr ₃ C ₂ , amorphous phase	5–61	5.5±1.1
FeMoNiCrB–FeTiO ₃	Sol. sol Fe(Ni,Cr), Mo ₂ FeB ₂ , FeTiO ₃ , amorphous phase	<40	5.1±0.7

particle (Table 2). Presence of oxygen in the mixtures with ZrB₂ and (Ti, Cr)C is indicative of oxidation during MA, as the process was conducted in air.

Thus, powder mixture processing for 1.5 h is quite sufficient to produce composite particles with a uniform distribution of the components. Further increase of processing time is pointless, the more so as it will lead only to additional oxidation of the powder and its contamination by the products of destruction of the walls of the drums and grinding bodies.

By the shape of variation curves of powder particle microhardness at different stages of mechanical alloying (Figure 11), the presence of two peaks of microhardness values on the graphs after 30 min of processing can be noted (Figure 11, *a*).

Increase of process duration up to 1.0 h, resulting in formation of agglomerates, consisting from initial ceramic and metal particles, leads to the presence of just one most probable microhardness value being noted on the variation curves (Figure 11, *b, c*). After 1.5 h of processing, the average microhardness is equal to HV = 6.2±1.6 GPa for FeMoNiCrB–ZrB₂, HV = 5.5±1.1 GPa for FeMoNiCrB–(Ti,Cr)C and 5.1±0.7 GPa for FeMoNiCrB–FeTiO₃ compositions.

Method of XRD analysis of composite powder particles obtained after 1.5 h of MA (Figure 12) revealed that all of them had an amorphous-crystalline structure. Broadening of X-ray image lines is indicative of particle refinement to the nanoscale. Evaluation of the coherent scatter regions showed that the size of CSR for ZrB₂ decreased from 128 to 23 nm, for (TiCr)C – from 56 to 17 nm, and for FeMoNiCrB

alloy — from 48 to 12 nm. As regards amorphization of FeMoNiCrB alloy during MA, this is indicated by formation of a “halo” in X-ray patterns in angle ranges of 2θ = 39.9–52.8° and 68.2–84.3°.

No formation of new phases during MA as a result of interaction between FeMoNiCrB alloy elements and refractory compounds was revealed. We can only note merging of the main peaks of X-ray images for Ni, Cr, and Fe solid solutions into one, which may be indicative of formation of an oversaturated iron-based solid solution. More over, a splitting of the diffraction peak is observed at 2θ = 36.8° angle, which corresponds to double titanium-chromium carbide (Ti, Cr)C into two, which correspond to TiC and Cr₃C₂ phases, pointing to decomposition of double titanium-chromium carbide.

Results on phase composition and microhardness of MA products after 1.5 h of processing are shown in Table 3.

Produced during MA amorphous-nanocrystalline cermet powders, which consist of an amorphizing Fe-based matrix with refractory compounds introduced into its volume, can be used in the technology of thermal spraying of composite coatings with an amorphous structure. However, the small particle size (15–22 μm) and high value of specific surface (~0.5 m²/g), resulting in absence of fluidity in powders, complicates their uniform feeding into the flow at TS. In order to increase the particle size of MA products and their fluidity, conglomeration of MA products with 5 % solution of polyvinyl alcohol in water is performed with subsequent drying of the

mixture at the temperature of 80 °C, grinding of the obtained conglomerates and screening of particles of 40–63 µm size.

CONCLUSIONS

1. Studied were the physicochemical processes taking place at formation of composite powders based on FeMoNiCrB under the conditions of mechanical alloying with addition of ZrB₂, (Ti, Cr)C and FeTiO₃ alloying additives.

2. It is found that mechanical alloying results in formation of amorphous-nanocrystalline cermet powders, consisting of an oversaturated Fe(Ni, Cr) solid solution and additives of refractory compounds (ZrB₂, (Ti, Cr)C or FeTiO₃) and having the values of average microhardness of FeMoNiCrB-ZrB₂ cermets of 6.2±1.6 GPa, FeMoNiCrB-(Ti, Cr)C – 5.5±1.1 GPa and FeMoNiCrB-FeTiO₃ – 5.1±0.7 GPa.

3. It was established that stabilization of the size of conglomerate particles during MA of a mixture of FeMoNiCrB + ZrB₂, FeMoNiCrB – (Ti, Cr)C and FeMoNiCrB + FeTiO₃ powders occurs after processing for 1.5 h and the average particle size is as follows: FeMoNiCrB + ZrB₂ – 23 µm; for FeMoNiCrB – (Ti, Cr)C – 15 µm, for FeMoNiCrB – FeTiO₃ – 25 µm.

4. In order to use the obtained MA products for thermal spraying of the composite coatings with an amorphized structure, powders with a conglomerate structure and particle size of 40–63 µm were produced.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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