Structure and properties of powder alloys Fe–(45-15)%Ni–(10-5)%Cu, obtained via mechanical alloying

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The article investigates the structure and properties of Fe–(45-15)%Ni–(10-5)%Cu powder alloys. The production method included low- and high-energy (mechanical alloying, MA) treatment of Fe, Ni, and Cu powders in a planetary ball mills followed by hot-pressing of mixes at 950 °C. After MA, the mixtures consist of composite granules with a lamellar structure and particle size of 10–100 μm.
After low-energy treatment, three phases were detected by XRD in hot-pressed samples: α-Fe-based solid solution BCC-(Fe, Ni, Cu), Ni-based solid solution FCC-(Ni, Fe, Cu), and FCC-FeO. The appearance of FeO is caused by the partial oxidation of iron during mixing and hot pressing. The total oxide content does not exceed 1.3 wt%. For Fe-X% Ni-5% Cu alloys the ultimate bending strength depends on the nickel content (X, %) linearly according to the equation $\sigma^{ben} = -19 \cdot X + 1995$ [MPa], where $15\ \% \le X \le 45\ \%$.
Alloys obtained by MA have a homogeneous structure and depending on the composition can be either two- or three-phase. As a result of MA the hardness of the alloys increased by 20–21 HRB, and the ultimate bending strength increases by 300–540 MPa. The alloy with the composition 80%Fe-15%Ni-5%Cu has the maximum bending strength $\sigma^{ben} = 2135 \pm 60$ MPa. The wear of MA alloys is $(4.1–4.4) \cdot 10^{-5}$ mm³ /(N·m) which is more than two times lower than the wear of alloys obtained by low-energy treatment.
Key words: solid solution, Fe-Ni-Cu alloys, mechanical alloying, hot pressing, strength, wear resistance, XRD, SEM.
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Introduction

It is known that operating properties of metal matrix diamond-bearing composites for cutting are mainly determined by metallic matrix properties [1, 2]. A metal matrix (binding) should provide strong fixing of diamond grains during cutting (from one side) and be wear-resistant for stable operation of a diamond tool in stationary self-sharpening procedure (from other side) [3–6]. Additionally, binding should allow to obtain diamond-bearing segments at relatively low temperatures (usually below 900 °C, to avoid substantial graphitization of diamond grains during sintering) [7–9], as well as to have high heat conductivity for efficient heat removal from the cutting area [10] and also be characterized by sufficient stiffness to prevent incorporation of diamond grains into binding during cutting. Thereby, selection of optimal composition of binding alloy is considered as the complex material science problem because the requirements for such materials are various and often contradictory.

Fe-based alloys are prospective for use as matrix material for cutting diamond tools [11–13]. Pure ferrous powders for manufacture of diamond segments can't be used on the following causes. Due to high melting temperature (T_m=1539 °C), ferrous powders are hardly sintered at the temperatures below 900 °C. Besides that, they are susceptible to oxidation and corrosion, what additionally hampers sintering and has negative effect on integrity of diamond grains. Finally, Fe is a catalyst of "diamond – graphite" phase transition, what increases possibility of degradation of diamond grains during sintering [14, 15]. To decrease the sintering temperature of Fe-based composites, relatively low-melting metals, e. g. copper (T_m=1083 °C) are introduced in their composition. Copper is substantially softened at the typical sintering temperatures of segments 800–900 °C, and the process of hot pressing is conducted at lower temperature. It should be mentioned that copper is a metal with high heat conductivity (397 Wt/(m·K) in comparison with 78.2 Wt/(m·K) for Fe [16]). Therefore, heat removal efficiency from the cutting area will increase in the Fe-Cu alloys. Alloying by nickel leads to solid solution hardening and simultaneously allows to rise oxidation resistance. The known problem of Fe-Ni alloys obtaining from elementary powders is connected with their mutual solubility, which can lead to chemical heterogeneity of a forming solid solution as well as to forming of diffusion porosity. Use of preliminarily molten powders can be the solution of the above-mentioned problem [14, 17–18].

Mechanical alloying (MA) method is another perspective method of obtaining powder alloys with high homogeneity [19].

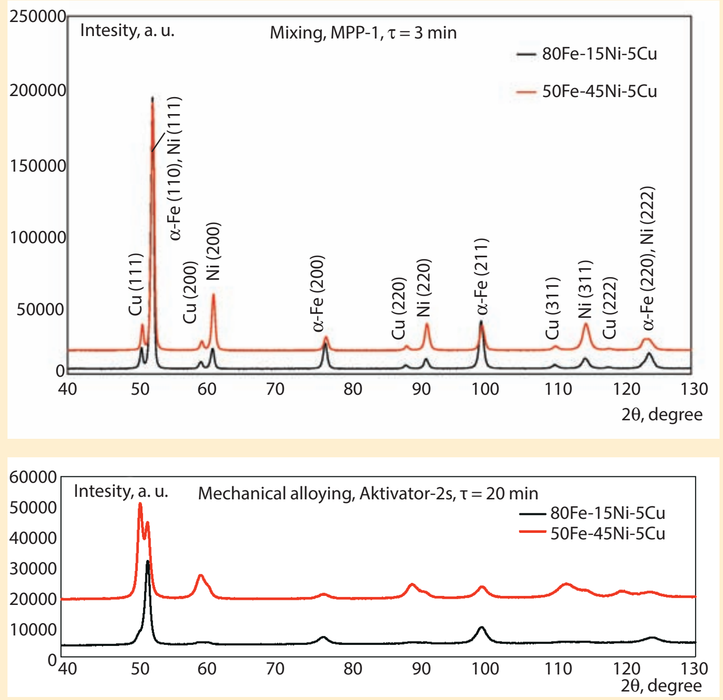


Fig. 1. Diffraction spectra of the mixes with compositions 80%Fe-15%Ni-5%Cu and 50%Fe-45%Ni-5%Cu after low-energy (above) and high-energy (below) treatment in planetary mills

Table 1. **Properties of initial powders**

Powder grade	Producer	Size of particles	Content of the basic component, mass%
VK-3 (TU 2436-045-05807977-98)	"Sintez-PZhK" JSC, Russia	<25 µm — 90 %	Fe > 99.5
PNK-UT3 PM (GOST 9722-97)	"Kola mining and metallurgical company" JSC, Russia	5 µm (FSS)	Ni > 99.9
PMS-1 (GOST 4960-2017)	("Novosverdlovskaya metallur-gical company" JSC, Russia	<100 µm — 100 % <71 µm — 95.5 % <45 µm — 74.7 %	Cu > 99.8

This method allows to obtain non-equilibrium solid solutions even for those elements, which are characterized by minimal mutual solubility, as in Fe-Cu system [20]. The works [21–23] are devoted to investigation of Cu-Fe-Ni alloys obtained from MA powders. It is shown in [22] that MA of Ni and $Cu_{60}Fe_{40}$ mix of powders during 100 hours allows to obtain two solid solutions FCC-Cu(Fe,Ni) and BCC-Fe(Ni) with average crystalline size 13 and 8 nm respectively. It should be noted that preliminarily molten powder mix of Ni and $Cu_{60}Fe_{40}$ was also obtained via MA from elementary powders during 100 hours.

The aim of this research is investigation of structure, mechanical and tribological properties of Fe-based alloys with composition Fe-(45-15)%Ni-(10-5)%Cu, which were subjected to mechanical alloying of elementary powders. These alloys are prospective for further use as bindings of diamond tools.

Technique of investigations

The alloys with composition Fe-(45-15)%Ni-(10-5)%Cu, obtained from elementary powders of carbonyl Fe of VK-3 grade ("Sintez-PZhK" JSC, Russia), from carbonyl nickel of PNK-UT3 PM grade ("Kola mining and metallurgical company" JSC, Russia) and from copper electrolytic powder of PMS-1 grade ("Novosverdlovskaya metallurgical company" JSC, Russia) were investigated in this work (see the **Table 1**).

Initial mixes were subjected to low- and high-energy treatment (MA) in planetary mills. Low-energy treatment was conducted in the planetary ball mill (PBM) of MPP-1 type ("Tekhnika i tekhnologiya dezintegratsii" ("Equipment and technology of disintegration") JSC, Russia), with the controlled relation between masses of balls and mix, and treatment duration 3 min. Gravitation factor of this mill makes about 20g. Mechanical alloying (MA) of powder mixes was conducted in the laboratory PBM "Aktivator-2s" ("Plant of chemical machine-building" JSC, Russia). The ball rotation speed makes 700 min-1, centrifugal factor is 120 g, treatment duration (optimized in [24]) is 20 min. To prevent oxidation before MA of powders, the balls were filled with argon of OSCh grade (99.998 % purity). Granulometric composition of powders after treatment in PBM was examined via the method of laser diffraction at the assembly ANALYSETTE 22 MicroTec plus ("Freetch", Germany).

Compact samples for assessment of mechanical and tribological properties were prepared via hot pressing technology in graphite press forms in the assembly DSP-1 ("Freetch", Germany). The following parameters of hot pressing process were used: temperature 950 °C, pressure on a sample 25 MPa, duration of isothermal holding 3 min (at 950 °C), residual pressure in a working chamber 1000 Pa. Hardness was determined in the Wolpert Rockwell Hardness Tester of Wolpert 600 MRD type by B scale according to the standard GOST 20017-74. Testing for three-point bending were carried out at the universal testing servohydraulic machine LF-100 kN ("Walter + Bai AG", Switzerland), according to the GOST 18228-94. Metallographic investigations were conducted using the scanning electronic microscope S-3400N of the company "Hitachi High-Technologies Corporation", equipped by X-ray power dispersion spectrometer NORAN. Porosity of the samples was evaluated via the metallographic method on the structure micropictures obtained with 500 fold magnification. X-ray phase analysis (XRD) of mixes and compact samples was conducted using automatic diffractometer DRON-3 with monochromatic CoK_{α}-radiation (λ=0.179021 nm) within the angles range 2θ from 0° to 130°. Tribological tests were carried out at the automatic friction machine Tribometer of the company

"CSM Instruments" according to the scheme "stationary ball — rotating disk" for the following conditions: friction counterbody — ball with 6 mm diameter from Al_2O_3; normal load 0.4 N; counterbody speed relating to a sample 5.0 cm/s; distance run 100 m.

Experimental results

Phase composition and structure of Fe–Ni–Cu powders after treatment in a planetary mill

Diffraction spectra of the mixes with compositions 80%Fe-15%Ni-5%Cu and 50%Fe-45%Ni-5%Cu after low- and high-energy treatment are presented on the **Fig. 1**.

The peaks of phases BCC-Fe, FCC-Ni and FCC-Cu with intensities proportional to their content are presented on the diffraction spectrum after low-energy treatment. It should be noted that reflections from the plane (110) α-Fe coincide with the reflection from the plane (111) Ni, and the second order reflections for these planes also coincide after low-energy mixing. After mechanical alloying during 20 min, diffraction peaks widened substantially and moved to the side of smaller angles 2θ (Fig. 1, below), what is connected with increase of density of crystal lattice defects, with decrease of size of coherent dispersion areas and with forming of substitutional solid solutions. Overlapping of the most intensive lines, corresponded to the reflection planes (111), (200), (220) of Ni and Cu phases can be observed on the diffraction pattern of powders after MA; it decreases accuracy of semi-quantitative analysis (**Table 2**).

Table 2. **Parameters of crystal structure and semi-quantitative X-ray structural analysis of powder mixes after mixing in MPP-1 (τ = 3 min) and Aktivator-2s (τ = 20 min)**

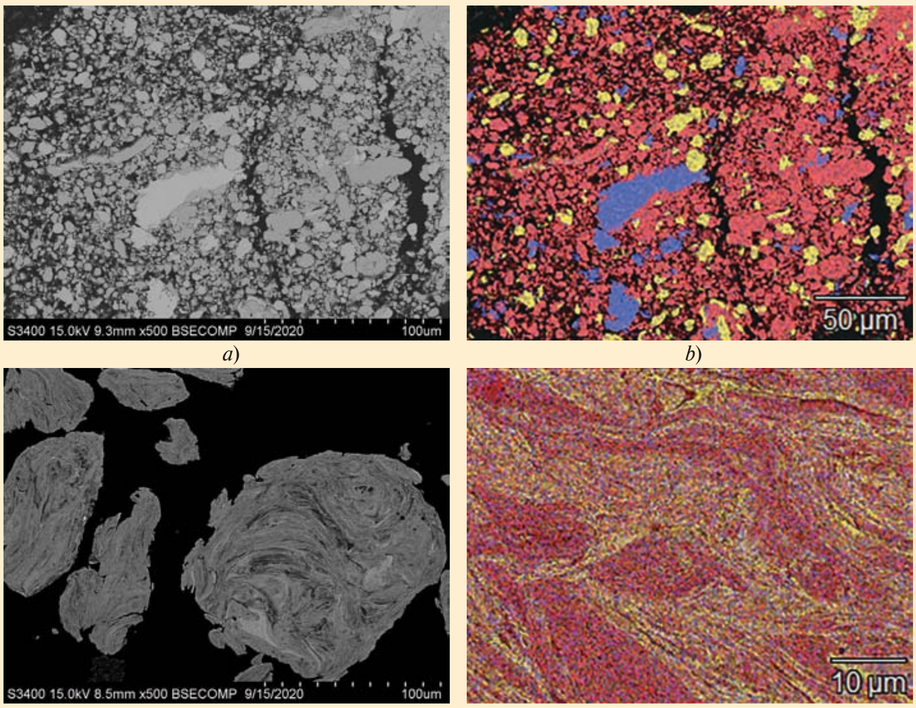
Mix composition	Phase					
	α–Fe cI2/1		Ni cF4/1		Cu cF4/1	
	d, nm	mass %	d, nm	mass %	d, nm	mass %
50% Fe-45% Ni-5% Cu, MPP-1	0.2865	46.6	0.3522	47.0	0.3613	6.4
80% Fe-15% Ni-5% Cu, MPP-1	0.2865	78.5	0.3524	16.4	0.3613	5.0
50% Fe-45% Ni-5% Cu, MA	0.2869	(37.9)*	0.3535	(19.2)*	0.3588	(42.9)*
80% Fe-15% Ni-5% Cu, MA	0.2868	89.2	0.3530	4.7	0.3591	6.0

* — the error of semi-quantitative analysis, connected with overlapping of the lines

After low-energy treatment, the mix presented the combination of low-deformed Fe, Ni and Cu grains with homogenous distribution (**Fig. 2 *a*, *b***). The equiaxial coefficient is located within the interval form 1 to 5, i.e. grain form is varying from equiaxial to strongly extended.

MA of the Fe–Ni–Cu mix led to forming of composite granules with dispersity10–100 μm with lamellar structure (**Fig. 2*c***). Thickness of alternating layers made from 1 to 10 μm, and copper layers with thickness more than 0.5 μm are absent in granules, what means partial forming of solid solutions (**Fig. 2*d***).

Granulometric composition of mixes varies substantially depending on the treatment conditions. So, distribution of particles for the mix 50%Fe-45%Ni-5%Cu after its mixing in MPP-1 is unimodal with the parameter D [4.3] = 6.86 μm (average normalized value for mass of particles); at the same time distribution quantiles of D(10), D(50) and D(90) make 2.9, 6.2 and 11.7 μm respectively. After high-energy treatment, granulometric composition of the mix 50%Fe-45%Ni-5%Cu is characterized by bimodal distribution with the parameter D [4.3] = 38.63 μm, while distribution quantiles of D(10), D(50) and D(90) make 10.8, 20.6 and 83.4 respectively.



a) *b*) *c*) *d*)

Fig. 2. Typical structures and charts of elements distribution in powder mixes 80% Fe-15% Ni-5%Cu, obtained at low-energy (*a*, *b*) and high-energy (*c*, *d*) treatment conditions

Structure and properties of compacted alloys Fe-Ni-Cu

Microstructure and diffraction spectra of the alloys obtained via hot pressing of the mixes with compositions

80%Fe-15%Ni-5%Cu and 50% Fe-45%Ni-5%Cu are presented on the **Fig. 3** and **Fig. 4**. The mixes which were preliminarily subjected to low-energy treatment (Fig. 3 *a*, *b*) have three structural components after hot pressing: (1) solid Fe-based solution (Fe, Ni, Cu) with BCC lattice; (2) solid Ni-based solution (Ni, Fe, Cu) with FCC lattice; (3) FCC-FeO. FeO appearance is stipulated by partial Fe oxidation during mixing and hot pressing, while total oxide content in the alloys does not exceed 1.3 mass%. FeO particles have equiaxial form, size less than 3 μm and are located mainly in the phase BCC-(Fe,Ni,Cu) (see Fig. 3a). Such primary FeO location is explained by most oxygen affinity to α-Fe, than to Ni. FCC-(Ni,Fe,Cu) and BCC-(Fe,Ni,Cu) grains are characterized by gradient structure, i.e. diffusion processes of solid solutions forming don't run completely at the selected hot pressing conditions.

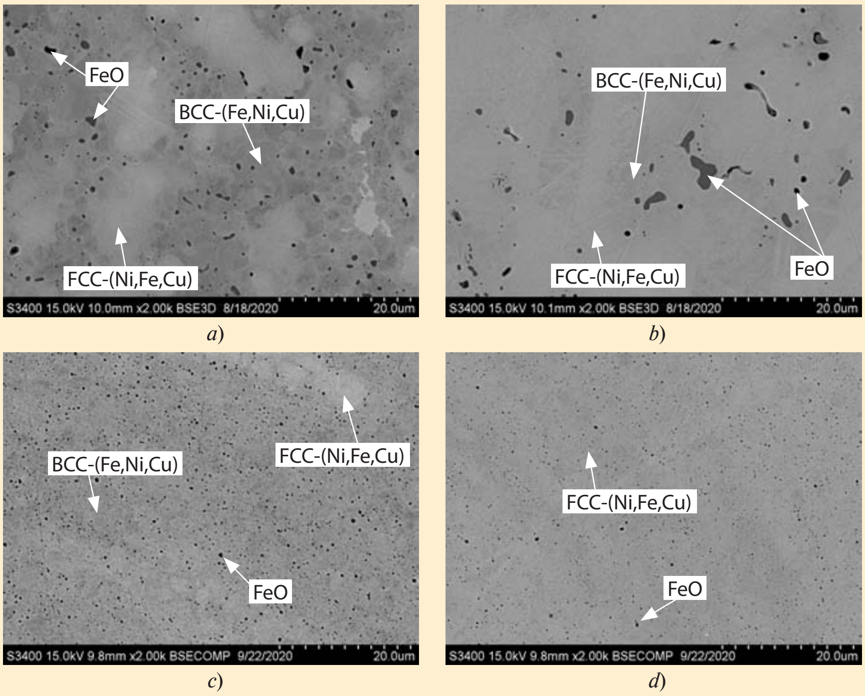


Fig. 3. Structure of hot pressed Fe-Ni-Cu samples: *a* — alloy 80% Fe-15% Ni-5% Cu, low-energy treatment of charge; *b* — alloy 50% Fe-45% Ni-5% Cu, low-energy treatment of charge; *c* — alloy 80% Fe-15% Ni-5% Cu, MA; *d* — alloy 50% Fe-45% Ni-5% Cu, MA

Two or even three structural components can be presented in the compact samples obtained from MA powders, depending on composition (Fig. 3 *c*, *d*). The alloy 80% Fe-15% Ni-5% Cu includes the phases FCC-(Ni,Fe,Cu), BCC-(Fe,Ni,Cu) and FeO, based on the data of X-ray structural analysis. At the same time, the alloy 50% Fe-45% Ni-5% Cu includes only FCC-(Ni,Fe,Cu) and FeO phases. Thereby, MA intensifies substantially diffusion processes of solid solutions forming. It should be noted that average size of FeO particles in the alloys after MA decreased by 5–10 times (to less than 0.5 μm).

Substantial deviation of lattice parameters in solid solutions BCC-(Fe,Ni,Cu) and FCC-(Ni,Fe,Cu) from the reference values for α-Fe (a = 0.2866 nm) and Ni (a = 0.3524 nm) is observed in compact samples. The peaks corresponding to Cu are absent on diffraction patterns, what confirms its complete dissolution during hot pressing (**Table 3**).

The results of investigation of samples hardness and strength after hot pressing are presented in the **Table 4**.

It is seen from the Table 4, lowering of nickel content from 45 % to 15 % in Fe-Ni-Cu alloys, with constant copper content 5 % leads to monotonous lowering of ultimate bending strength and hardness of alloys. Decrease of alloy hardness is explained by smaller microhardness of Ni-based solid solution in comparison with α-Fe-based solid solution [24]. Tensile strength for Fe–X%Ni–5%Cu alloys linearly depends on nickel content X according to the equation $\sigma^{ben} = -19 \cdot X + 1995$ [MPa], where

Table 3. **Phase composition of powder mixes Fe-Ni-Cu after hot pressing**

Powder mix	Phase					
	(Fe,Ni,Cu) cI2/1		FeO cF8/2		(Ni,Fe,Cu) cF4/1	
	d, nm	mass %	d, nm	mass %	d, nm	mass %
50% Fe-45% Ni-5% Cu, MPP-1	0.2872	2.9	0.4298	0.9	0.3590	96.2
80% Fe-15% Ni-5% Cu, MPP-1	0.2869	77.5	0.4291	1.3	0.3590	21.2
50% Fe-45% Ni-5% Cu, MA	–	–	–	–	0.3594	100
80% Fe-15% Ni-5% Cu, MA	0.2872	68.5	0.4298	0.6	0.3592	31.0

Table 4. **Porosity, hardness and tensile strength for compact samples after hot pressing**

Sample composition	Porosity,%	HRB	σ^{ben}, MPa	Absolute deformation, mm
Low-energy treatment in planetary ball mills				
50 % Fe-45 % Ni-5 % Cu	0.1–0.2	86 ± 2	1130 ± 20	6.5
60 % Fe-35 % Ni-5 % Cu	0.1–0.2	85 ± 1	1350 ± 15	6.2
70 % Fe-25 % Ni-5 % Cu	0.1–0.2	84 ± 1	1510 ± 20	3.2
80 % Fe-15 % Ni-5 % Cu	0.1–0.2	80 ± 1	1710 ± 10	2.1
50 % Fe-47 % Ni-3 % Cu	0.1–0.2	84 ± 1	1160 ± 20	7.0
50 % Fe-43 % Ni-7 % Cu	0.1–0.2	87 ± 1	1100 ± 20	7.0
50 % Fe-40 % Ni-10 % Cu	0.1–0.2	80 ± 1	1070 ± 10	9.2
High-energy treatment in planetary ball mills (MA)				
80 % Fe-15 % Ni-5 % Cu	0.1–0.2	101 ± 2	2135 ± 60	1.5
50 % Fe-45 % Ni-5 % Cu	0.1–0.2	106 ± 3	1670 ± 35	2.6
50 % Fe-40 % Ni-10 % Cu	0.1–0.2	101 ± 2	1350 ± 30	4.2

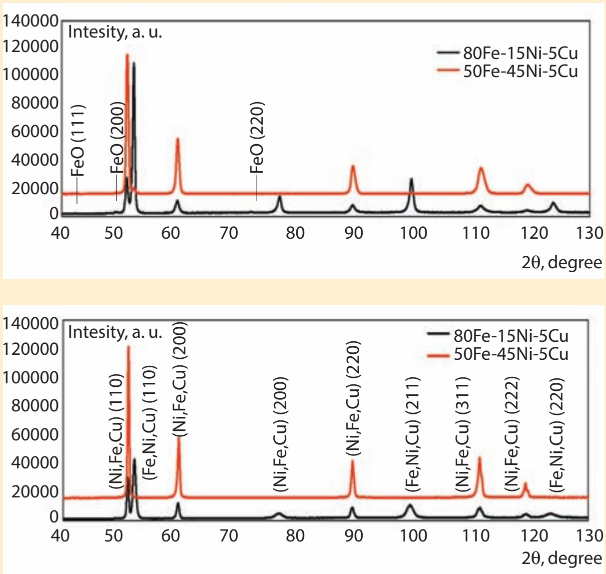


Fig. 4. Diffraction spectra of the compact alloys 80%Fe-15%Ni-5%Cu and 50%Fe-45%Ni-5%Cu, obtained from powders subjected to low-energy (above) and high-energy (below) treatment in planetary mills

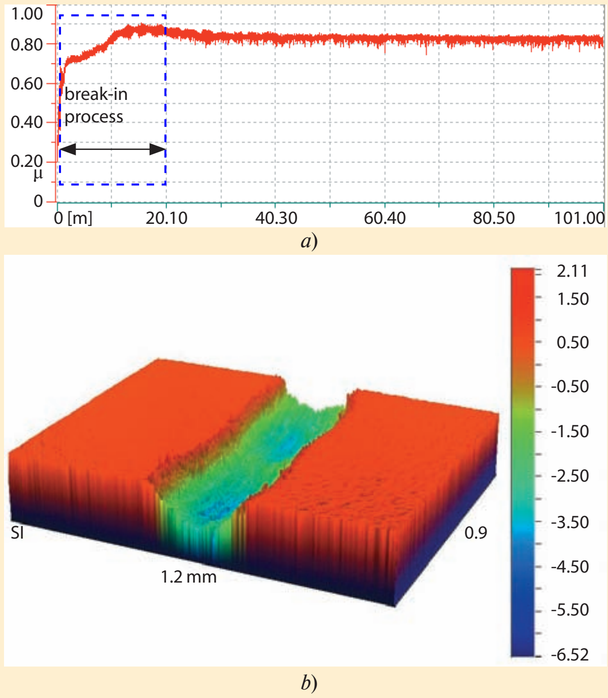


Fig. 5. Friction coefficient dependence on running length in the process of tribological testing (*a*) and 3D-image of wear groove for 80%Fe-15%Ni-5%Cu alloy (*b*)

15%≤X≤45%. As for the alloys with permanent Fe content as large as 50 %, variation of Ni:Cu relation from 47:3 to 40:10 leads to decrease of bending strength from 1160 to 1070 MPa. All samples after hot pressing had porosity less than 0.2 vol%, thereby difference in the mechanical properties of the alloys were stipulated by their composition and treatment conditions for powder mixes, but not by porosity.

Presented wear of samples after low-energy treatment slightly depends on composition and lays within the range $(10.5\text{-}11.8)\cdot10^{-5}$ mm³/(N·m), while the friction coefficient of a tribological pair "corundum ball — Fe-Ni-Cu alloy" made 0.68–0.89 (see **Table 5**). The samples after NA were characterized by specified wear which is lowered more than by 2 times; it is caused by their larger hardness, while the friction coefficient varied slightly. Friction coefficient dependence on running length (**Fig. 5**) has two typical sites for all examined alloys: it increases from 0.2 to appr. 0.8 on the stage of break-in process (running 0–20 m), and after 20 m the friction coefficient does not depend on distanse run.

Table 5. **Tribological characteristics of obtained hot pressed alloys**

Alloy composition	Wear·10^{-5}, mm³/(N·m)	Friction coefficient, k_f
Low-energy treatment in PBM		
50 % Fe-45 % Ni-5 % Cu	10.9	0.79
60 % Fe-35 % Ni-5 % Cu	10.6	0.87
70 % Fe-25 % Ni-5 % Cu	11.9	0.80
80 % Fe-15 % Ni-5 % Cu	10.5	0.82
50 % Fe-47 % Ni-3 % Cu	10.9	0.68
50 % Fe-43 % Ni-7 % Cu	10.8	0.78
50 % Fe-40 % Ni-10 % Cu	11.8	0.89
MA		
80 % Fe-15 % Ni-5 % Cu	4.3	0.79
50 % Fe-45 % Ni-5 % Cu	4.1	0.75
50 % Fe-40 % Ni-10 % Cu	4.4	0.89

As a result of MA, hardness of alloys increased by 20-21 HRB, while ultimate bending strength rises by 300–540 MPa. The alloy 80 % Fe-15 % Ni-5 % Cu had maximal strength σ^{ben}=2135 ± 60 MPa. Increase of hardness of the alloys after MA is connected with refinement of the alloy structural components, as well as with forming of more homogeneous solid solutions. MA leads to strong lowering of plasticity, as it can be seen from the data on absolute deformation of the alloys before destruction (Table 5). We can note high plasticity parameters of a binding phase in diamond-bearing composites are not desirable, because diamond holding is getting worse.

Conclusions

1. Structure forming of powder mixes with compositions Fe-(45-15)%Ni-(10-5)%Cu was investigated at low-energy and high-energy (MA) treatment in planetary mills. It is shown that MA leads to forming of solid solutions based on Ni and α-Fe;

2. Three structural components were revealed after low-energy treatment in hot-pressed samples: solid solution based on Fe with BCC-(Fe,Ni,Cu), solid solution based on Ni with (BCC-(Fe,Ni,Cu) and FCC-FeO. Ultimate bending strength for alloys Fe-X%Ni-5%Cu depends on Ni content X linearly, according to the equation

σ^{ben} = −19·X+1995 [MPa], where 15%≤X≤45%. As for alloys with permanent Fe content 50 %, variation of Ni:Cu relation from 47:3 to 40:10 leads to decrease of bending strength from 1160 to 1070 MPa.

3. Alloys obtained via MA are characterized by more homogeneous structure and can be both dual-phase or three-phase, depending on composition. As a result of MA, hardness of alloys increased to 20-21 HRB, while ultimate bending strength rose by 305–540 MPa. Maximal strength σ^{ben}=2135 ± 60 MPa is observed for the alloy with composition 80% Fe – 15% Ni – 5% Cu.

4. Alloys obtained via MA have specific wear which is lowered by 2 times. It is caused by their larger hardness; at the same time friction coefficient varies slightly comparing with alloys after low-energy treatment.

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