
COMPOSITE MATERIALS

Activated Sintering of Cu-Al₂O₃ powders

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Abstract—A technique for production of high-density composite material (Cu-1 wt % Al₂O₃) by single-fold cold compacting and sintering was developed. It is known that presence of gases in ductile metals, e.g., copper, inhibits consolidation of powder metal products. The technique is able to succeed in degassing of the composite material during heating in a hydrogen atmosphere. The technique is based on addition of cobalt and copper oxalates in a mixture of Cu and Al₂O₃. The efficiency of the technique was estimated by the relative density and hardness values of produced Cu-Al₂O₃ composite materials. The mechanism of impact of activated sintering on compaction of composite materials was investigated. It was found that decomposition of cobalt and copper oxalates at heating in hydrogen atmosphere synchronizes activated reduction of copper oxides and degassing of products, supporting permeability of composite materials. Composition of active admixture 0.5 wt % CoC₂O₄ + (2–2.5) wt % CuC₂O₄ is optimized. It was found experimentally that addition of oxalates produces a positive impact on consolidation processes and properties of composite materials on the basis of copper and Al₂O₃ micro- and nanoparticles irrespective of preparation techniques: electrolysis, evaporation-condensation, salt decomposition, precipitation and coprecipitation of hydroxides with further reducing heat treatment and milling. Application of activated sintering of powders obtained by coprecipitation of hydroxides and compacted at 700 MPa enables the formation of Cu-1% Al₂O₃ composite material with improved properties. As a result, composite material with homogeneous structure (grain size of 2–10 μm), increased values of density (8.4 g/cm³), and Brinell hardness up to 780 MPa was produced.

Keywords: composite materials, dispersion hardening, activated sintering, refining, structure, physical and mechanical properties

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INTRODUCTION

One of the priority classes of new materials is powder composite materials (CM) [1], which are systems consisting of dispersed phases that are specifically distributed in a metallic dispersion medium, e.g., copper. When creating CM for work under conditions of high loads, dispersion media are required which also possess a sufficient level of physical and mechanical properties, which is not characteristic of pure metals. Thus, in order to increase the hardness of copper, dispersion hardening (DH) is most often used, where very small particles of oxides, e.g., Al₂O₃, with high hardness and extreme chemical and thermal stability are introduced into the copper powder. However, the presence of oxide inclusions in the metal powder reduces its compactability during compacting and sintering. Since physical and mechanical properties of powder materials are improved, especially with a decrease in porosity, the production of DH metal materials is currently

carried out using such intensive methods as mechanical alloying [2], hot pressing [3], stamping, extrusion, and rolling [4–7]. In combination with these methods, cladding of oxides with metals [6, 8] and liquid phase or reaction sintering are also applied [8–10]. But such technologies are cumbersome, inefficient, and energy-intensive and require expensive equipment. In this regard, the creation of new alternative technologies of DH composites that reduce the cost of production remains an urgent task.

The aim of this work was to search for an energy-saving method of obtaining powdered DH copper by single-fold cold pressing and sintering. To implement this task, the processes of compaction and structure formation during the sintering of Cu-1% Al₂O₃ CM were studied upon variation of the dispersion, methods for obtaining the components, and conditions for their consolidation. The efficiency of the method for

obtaining Cu-Al₂O₃ was estimated from the density value and hardness of the CM.

MATERIALS AND METHODS

The studies were carried out using three compositions of the initial powders:

C1—a mixture of Cu micropowder and nanodispersed (ND) Al₂O₃;

C2—mixture of ND Cu and ND Al₂O₃;

C3—ND powder of Cu-Al₂O₃.

It was assumed that the uniformity of the distribution of hardening particles in the CM may depend on the particle size of the copper powders, and the size of these particles may depend on the method of introducing a constant particle concentration of Al₂O₃.

In the preparation of the mixture C1, PMS1 copper micropowder (GOST 49-60-75, average particle size ~70 μm) and ND Al₂O₃ powder obtained by vacuum evaporation-condensation (VTU 4-25-90, surface area 15 m²/g, particle size <100 nm) were used. The average particle size d was calculated as $d = 6/Sp$, where S is the surface area and ρ is the density of the compact particle material.

In the C2.1 mixture, the same as in C1, ND Al₂O₃ was used, and for C2.2, ND Al₂O₃ was obtained by decomposition of aluminum nitrate. Powder of ND Cu for the C2 mixture was obtained by reduction of low-water copper hydroxide Cu₂(OH)₂O by hydrogen [11]. The initial materials were copper nitrate Cu(NO₃)₂ · 5H₂O of reagent grade, aluminum nitrate Al(NO₃)₃ · 9H₂O of high-purity grade, and sodium hydroxide NaOH of high-purity grade. During the preparation of low-water hydroxide, solid copper sulfate was treated at room temperature with an aqueous 10 M solution of alkali with the proportion NaOH : CuSO₄ = 10 : 1 mol/mol. The reaction was carried out with constant stirring for 1 h. Using a triple decantation, the precipitate was washed with water to pH 8, then the filtration under vacuum was carried out, and the final dehydration of the hydroxide was carried out by washing the filter with acetone. The copper hydroxide powder was calcined in air at a temperature of 300°C for 1 h to obtain copper oxide CuO, which was reduced with hydrogen for CM 2.1 (400°C, 1 h) and yielded a copper powder with an average particle size of ~100 nm (surface area of 6.5 m²/g). For CM 2.2, CuO was mixed with Al(NO₃)₃ solution in ethanol, dried, and calcined first in air at 500°C for the purpose of decomposition of aluminum nitrate to Al₂O₃ · 2H₂O [12] and then in hydrogen (400°C, 1 h). The surface of the powder obtained from the C2.2 mixture was 7.6 m²/g and the particle size was ~85 nm. The homogenization of the mixtures C1 and C2 was carried out in a ball mill at a ratio of the masses of grinding bodies and powders of 10 : 1 in an ethyl alcohol medium for 10 h.

Powders C3 were prepared by coprecipitation of a 5 M NaOH solution of copper and aluminum hydroxides from a mixture of 0.5 M solutions of Cu(NO₃)₂ and Al(NO₃)₃. The hydroxide precipitate was washed from the ions of the initial reagents by a large amount of hot water with triple decantation. The coprecipitated hydroxides were dehydrated by vacuum filtration and vacuum evacuation of acetone through a filter precipitate. The obtained powder was heated in air at a temperature of 500°C, after which a mixture of copper and aluminum oxides in a layer of ~1 mm was annealed in hydrogen at temperatures of 400, 500, and 650°C, as a result of which copper powders containing, according to the data of micro-X-ray spectral analysis, 1 wt % of Al₂O₃ and having an area of 15.1, 12.5, and 9.2 m²/g (average particle size of 44, 53, and 72 nm), respectively, were obtained.

Samples with the dimensions of 10 × 10 × 55 mm were pressed in a rigid matrix at a pressure of 300–800 MPa. The sintering was carried out in a hydrogen atmosphere (dew point temperature of –30°C) first at 800°C and then at 1000°C with an exposure time of 1 h at each temperature. A cobalt oxalate powder was used as an additive to activate the sintering [16]. Mixing of Cu-Al₂O₃ powders with active additives was carried out for 4 h in the same mill in which the components were mixed.

The density of the pressed and sintered specimens was determined by measuring and weighing them; the relative porosity was determined as unity minus the relative density. The Brinell hardness HB was measured on a TSh 2 M installation in accordance with GOST 9012-59. The microstructure of the samples was studied on an Olympus GX51 inverted microscope and Carl Zeiss EVO 40 electron microscope. The surface area of the powders was determined by the BET method using a Sorptomatik instrument.

RESULTS AND DISCUSSION

The results of compacting the copper powder PMS1 during pressing and sintering at 800°C are shown in Fig. 1. As one can see, when sintering the samples with an initial density of less than 7.8 g/cm³, shrinkage takes place, and in the samples with greater density, growth is observed, that is, “hydrogen sickness.” Consequently, at a single-fold sintering of copper compacts from the PMS1 powder, the maximum attainable relative density is ~0.82. To increase the density in practice, most often copper parts are first compressed with open porosity and sintered at a temperature sufficient to reduce the oxides and then repeatedly pressed at high pressure and sintered at the maximum temperature. There are also known attempts to overcome the “hydrogen sickness” by introducing various additives to the copper powder [13–15]. Previously, this problem was solved by adding 2% Co or Fe oxalates to the copper powder [16].

Cobalt and iron activate the reduction of copper oxides, and the products released during the decomposition of additives (CO , CO_2 , and H_2O) support the gas permeability of the samples, which provides the sintering of copper powder at 800°C to a relative density of 0.95. In this paper, the applicability of the addition of cobalt oxalate was determined to activate the sintering of copper powders containing Al_2O_3 particles. To reduce the possible effect on the composition of CM (which is important, e.g., in the creation of electrotechnical materials), cobalt oxalate was used in conjunction with copper oxalate, which was supposed to provide the same gas evolution when heated instead of CoC_2O_4 . The composition of the additives and their effect on sintering and properties of CM from C1 mixture are given in Table 1.

It follows from the data given that, in order to activate the reduction of copper oxides when sintering CM from C1, an addition of 0.5% CoC_2O_4 is sufficient. In addition, the decrease in the porosity of sintered particles is facilitated by an increase in the pressure of pressing from 500 to 650 MPa, at which the porosity of the copper powder increases in the case of inactivated sintering (Fig. 1). The composite with a minimum porosity (5%) has an increase of 50% as compared with a hardness of 590 MPa of copper sintered without the addition of Al_2O_3 .

Thus, as in the sintering of copper powder, the method of single-fold cold pressing and activated sintering is effective in the production of CM Cu + 1 wt % Al_2O_3 based on copper micropowder.

When creating CM Cu-1% Al_2O_3 from ND powders, C2 mixtures were used, the composition of which is given in Table 2.

Pressing of the $5 \times 10 \times 55$ mm samples from C2 mixtures was carried out once and twice at a pressure of 400–800 MPa (Table 3).

As one can see, the samples sintered at 800°C had a residual porosity of 16.7–25.3%. For samples I and

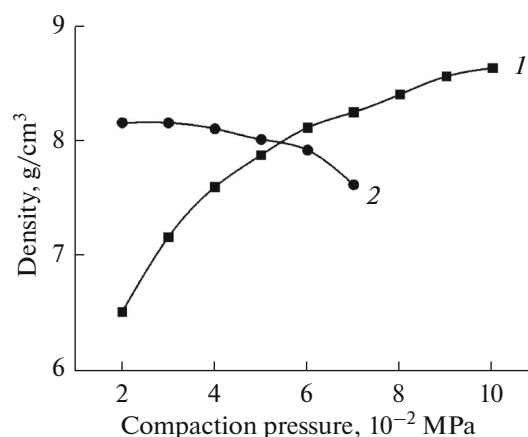


Fig. 1. Dependence of density of (1) compacted and (2) sintered (800°C , 1 h) samples from PMS1 copper powder on compaction pressure.

II compressed for a second time to a porosity of 5–6% after sintering at 1000°C , growth and swelling were detected. To a lesser degree, this is also characteristic of sample III, whereas for sample IV the original form remained and the porosity decreased by half.

The nature of the compaction during the sintering of the materials is illustrated by the microstructure shown in Fig. 2. Figures 2a, 2c, and 2d show the general view of the microstructure, which for sample II consists of a dense outer shell and a central part with large cavities. In the central part of the sample of composition III containing an active additive, there are also large pores. Probably, during the sintering of the sample III at 800°C , the formation of a dense shell outstrips the complete decomposition of oxalates because the concentration of aggregated particles of the ND Al_2O_3 in the C2.1 mixture is not sufficient for the inhibition of sintering of ND Cu. But the gases released from the active additive still somewhat retard the closing of the pores in the sintered material III and

Table 1. Preparation conditions and properties of Cu-1 wt % Al_2O_3 powder composites from C1 mixture sintered at 1000°C

No.	Compaction pressure, MPa	Additive composition, wt %		Porosity, %	Hardness HB , MPa
		CoC_2O_4	CuC_2O_4		
1*	300 + 800	—	—	4.0	390
2	500	—	—	11.2	450
3	500	0.12	1.88	11.8	400
4	500	0.25	1.75	11.4	470
5	500	0.5	1.5	6.5	540
6	650	0.5	1.5	5.0	590
7	500	1.0	1.0	7.6	580
	650	1.0	1.0	5.0	590
8	500	1.5	0.5	9.1	540

*Sample from pure copper.

Table 2. Composition of nanodispersed powder C2 mixtures for preparation of Cu-Al₂O₃ composite material

No.	Mixture	Concentration of the components, wt %	
		base	additive
I	C2.1	100% ND Cu	—
II	C2.1	99% ND Cu + 1% ND Al ₂ O ₃	—
III	C2.1	96.5% ND Cu + 1% ND Al ₂ O ₃	0.5% CoC ₂ O ₄ + 2% CuC ₂ O ₄
IV	C2.2	96.5% ND Cu + 1% ND Al ₂ O ₃	0.5% CoC ₂ O ₄ + 2% CuC ₂ O ₄

Table 3. Preparation conditions and properties of Cu-1% Al₂O₃ composites on the basis of nanodispersed powders of C2 mixture

Mixture no.*	Compaction pressure, MPa	Sintering temperature, °C	Porosity, %	Hardness <i>HB</i> , MPa
I	400	800	16.9	—
	400 + 800	800 + 1000	24.6	240
II	400	800	16.7	—
	400 + 800	800 + 1000	24.6	310
III	700	800	18.9	—
		800 + 1000	21.6	340
IV	700	800	25.3	—
		800 + 1000	12.0	680

* Same as the numeration in Table 2.

reduce the deformation and porosity of the sample as compared to sample II.

In the sintering of samples IV, a macroscopically homogeneous cross-sectional structure consisting of (30–50) × (70–100) μm nonporous regions surrounded by (1–5) × (5–20) μm pores is formed. The formation of such a structure is connected, apparently, with the fact that the closing of pores in the material from the C2.2 mixture is prevented not only by the addition of oxalates but also by very thin films of aluminum oxyhydrate (Al₂O₃ · 2H₂O) coating the particles (and their aggregates), which is present for a longer time than oxalates during the sintering and decomposes on a copper surface onto Al₂O₃ nanocrystals at a temperature higher than 600°C [12].

Despite the residual porosity of 12%, the composite of composition IV has a hardness of 680 MPa, which is ~2 times greater than that of compact copper. The presented results testify to the positive effect of the activated sintering and the use of the hardening component in the form of a solution of its precursor on obtaining Cu-1% Al₂O₃ CM from the powder mixture C2.2.

The compaction data for the sintering of Cu-1% Al₂O₃ powders obtained by coprecipitation of copper and aluminum hydroxides (mixture of C3) and reduced at different temperatures are shown in Fig. 3. Depending on the temperature of the reduction (400, 500, or 650°C), these powders had a specific surface area of 15.1, 12.5, and 9.2 m²/g (average particle size

was 44, 53, and 72 nm), respectively. The samples were pressed at a pressure of 400–700 MPa.

It follows from Figs. 3a and 3b that the sintering of a sample from a powder obtained at 400°C and pressed at 400 and 500 MPa is accompanied by compaction, which is advanced with activated sintering. For CM from the powders obtained at 500 and 650°C without the addition of oxalates pressed at 500 MPa, sintering at 800°C is accompanied by an increase in the density of the samples to 7.8 and 7.6 g/cm³, respectively, and at 1000°C, its reduction to 7.0 and 7.3 g/cm³. When the sintering is activated, both at 800 and at 1000°C, shrinkage of samples from these powders occurs. A similar temperature dependence of the sample density shown in Fig. 3c is also observed in the samples pressed at 600 MPa. In case of pressing at 700 MPa of the powder obtained at 400°C (Fig. 3d), when the sintering is not activated at 1000°C, the CM density decreases significantly, and with activated sintering, a sharp increase occurs. Comparison of the dependences in Fig. 3 shows that the density of both pressed samples and those sintered at 800°C from all powders increases with an increase in the pressing pressure. During subsequent nonactivated sintering at 1000°C, the density of these samples of 7.0 g/cm³ (porosity ~22%) is limiting: at a lower density, the samples continue to thicken, and at higher density they grow.

The decrease in the density of the samples based on copper during sintering at 1000°C is evidently due to the formation of a dense shell on their surface (Figs. 2a, 2b),

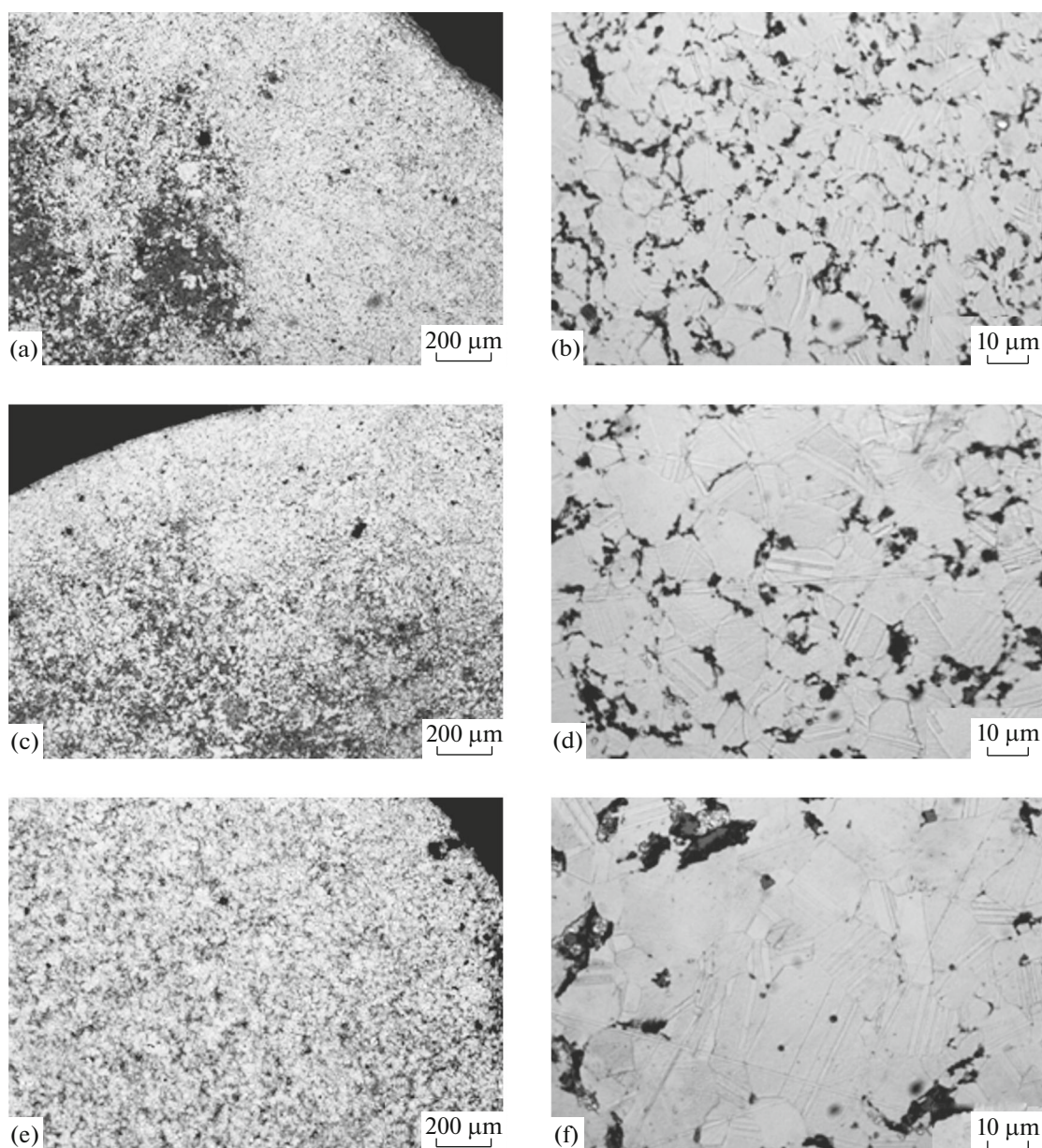


Fig. 2. Microstructure of Cu-Al₂O₃ composite materials based on nanodispersed powders (mixture C2) under different magnification: (a, b) specimen II; (c, d) specimen III; and (e, f) specimen IV. Composition of the materials is indicated in Table 2.

which prevents the escape of gases from the central part. On the basis of this assumption, on the samples pressed at 600 MPa, the change in their relative density and decrease in the mass Δm during sintering was determined (Fig. 4). Figure 4a shows that, in the process of sintering of CM without the addition of oxalates, the main decrease in the mass of the samples takes place during heating to 800°C, and at 1000°C, the change in the mass Δm , apparently related to the dehydration of Al₂O₃ · 2H₂O [6], is 0.26–0.29%. This quantity was sufficient to form large cavities and cracks in the sintering at 1000°C in samples presintered at 800°C to a density of 7.9 and 7.5 g/cm³ from

the powders obtained at 500 and 650°C. Accordingly, their density decreased to 6.35 and 7.1 g/cm³.

Under conditions of activated sintering of CM from the mixture C3, the relative decrease in the mass of the samples changes from 2.35 to 6.88% upon a decrease in the temperature of obtaining the initial powders (Fig. 4b). In addition, for CM from powders obtained at 500 and 650°C, the reduction in mass practically ceases at sintering at 800°C, and for CM from powders obtained at 400°C, it continues even during sintering at 1000°C. Despite this mass reduction regime, samples from all powders after sintering at 800°C continue to compact during sintering at

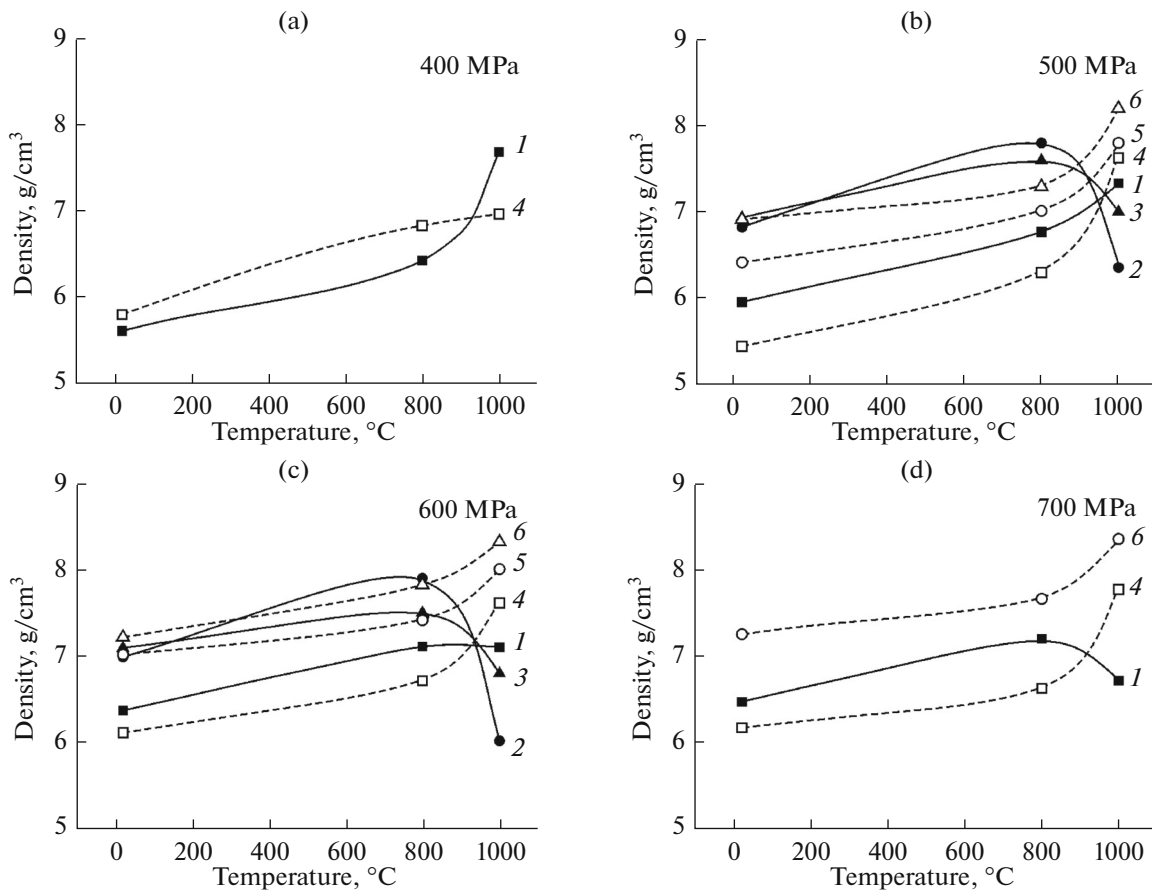


Fig. 3. Dependences of density of Cu-Al₂O₃ composite from C3 mixture produced under different compaction pressure on the sintering temperature. Temperature of the powder production is (1, 4) 400, (2, 5) 500, and (3, 6) 650°C. Dotted line—activated sintering; solid line—nonactivated sintering.

1000°C. This is due to the high porosity (30.1%) of the sample sintered at 800°C from the powder obtained at 400°C and a sufficiently small change in the mass ($\Delta m < 0.1\%$) of the samples sintered at 1000°C based on the powders obtained at 500 and 650°C. The sealing behavior at the final sintering of CM based on powders pressed at 500 and 700 MPa observed in Figs. 3b and 3d is probably related to similar effect of a decrease in mass and intermediate density (after sintering at 800°C).

Thus, the use of activated sintering and an increase in the temperature of obtaining Cu-Al₂O₃ powder from coprecipitated copper and aluminum hydroxides (mixture C3) to 650°C made it possible to create a CM with a density of 8.4 g/cm³ (porosity ~5%) by single-fold pressing and sintering. The microstructure of such CM is homogeneous, fine-grained (grain size 2–10 μm) with a small amount of fine (1–3 μm) pores (Fig. 5), whereas in the structure of CM obtained during nonactivated sintering, grains with size of 5–20 μm and nonuniformly distributed pores with size up to 10 μm are observed. The hardness of *HB* of these CM is 760–780 and 600–650 MPa, respectively.

From the results obtained, it follows that the application of activated sintering improves the properties of CM from all mixtures of powders studied. The best properties were obtained for CM from chemically mixed components (mixture C3). The use of a mixture of industrial powders (C1) is simpler and also makes it possible to obtain CM with a low porosity (5%), but the hardness of CM from C1 (590 MPa) is significantly lower than that of CM from C3.

With a single-fold pressing of mixtures C2 containing the precursors of the ND powders of Cu and Al₂O₃, it was not possible to obtain CM with a porosity below 12%. In addition, the presence in the structure of these CM slit-prominent pores (Fig. 2) is unfavorable for obtaining products with high and stable mechanical properties [17].

CONCLUSIONS

On the basis of micro- and nanopowders of Cu, Al₂O₃, and their precursors, composite materials Cu-1% Al₂O₃ were obtained.

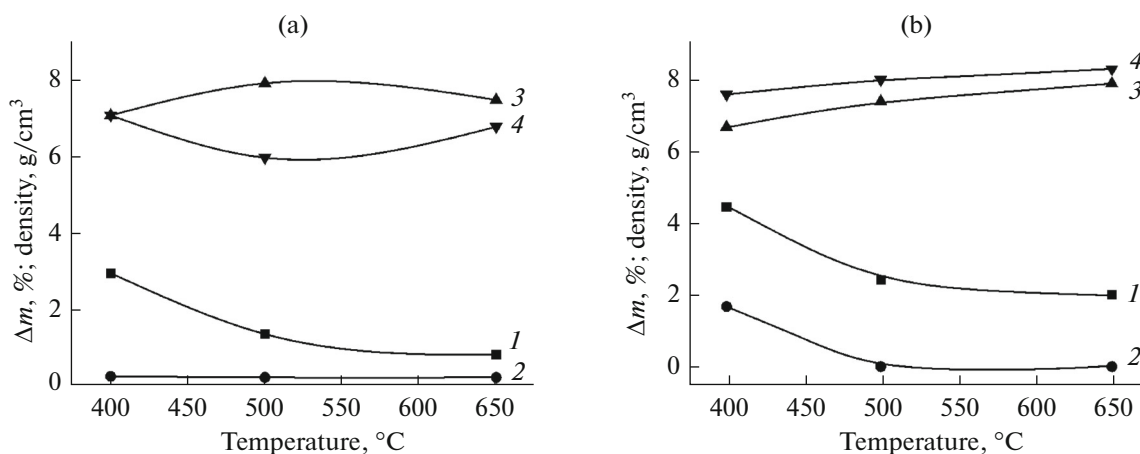


Fig. 4. Dependences of the (1, 2) mass loss Δm and (3, 4) relative density of specimens from C3 mixture on the temperature of the powder preparation. The temperature of sintering is (1, 3) 800°C and (2, 4) 800 + 1000°C: (a) nonactivated sintering; (b) activated sintering.

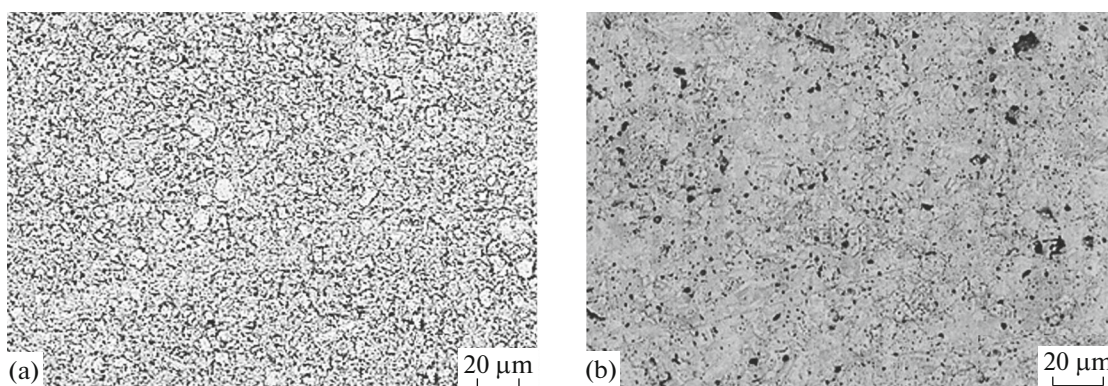


Fig. 5. Microstructure of Cu-1% Al₂O₃ sintered composite material from C3 mixture: (a) activated sintering; (b) nonactivated sintering.

The composition of the addition of a mixture of oxalates of 0.5 wt % CoC₂O₄ + 2–2.5 wt % CuC₂O₄, activating the sintering of Cu-1% Al₂O₃ CM, was proposed and optimized. The mechanism of the effect of active additives on compaction during sintering of CM in hydrogen consisting of synchronization of the activated reduction of copper oxides and the release of gaslike products of decomposition of oxalates that support the permeability of products was established.

The use of activated sintering provides the formation of CM Cu-1% Al₂O₃ with a density up to 8.4 g/cm³ (porosity up to 5%), a hardness *HB* up to 780 MPa (2.5 times higher than the hardness of pure copper), and homogeneous fine-grained (1–3 μm) structure from a powder obtained by coprecipitation of the components.

The results testify to the prospects of using a single-fold cold pressing and activated sintering in the development of technology for the production of dispersed-hardened copper. The established optimal chemical and technological conditions for the production of

powders and methods of consolidation of composite materials based on copper may be used in the production of articles of complex shape with prescribed properties, e.g., bimetallic plain bearings, in which the Cu-1% Al₂O₃ composite may be used as the matrix component of the antifriction coating.

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