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Effect of mechanical activation of tungsten powder on the structure and properties of the sintered Sn-Cu-Co-W material

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ABSTRACT

Introduction. One of the methods for improving the properties of sintered materials is mechanical activation of powders. It ensures milling the powders, changing its energy state, intensifying the sintering of powder materials, and forming a fine-grained structure in it. When tungsten powders are mechanically activated in planetary centrifugal mills, nanoparticles can be formed, which have a high reactive power. **The objective of the paper** is to study the effect of mechanical activation of tungsten particles on the structure and properties of the sintered *Sn-Cu-Co-W* powder material. **Research technique:** Mechanical activation of *W16,5* grade tungsten powder is carried out in a planetary centrifugal ball mill *AGO-2U* for 5…120 minutes with carrier speeds of 400…1,000 rpm. The mixture of tungsten, tin, copper, and cobalt powders are compacted by static pressing in molds and then sintered in vacuum at 820 °C. The morphology and size of powder particles, as well as the structure of the sintered samples, are studied by scanning electronic microscopy, X-ray microanalysis, and optical metallography. Porosity of the sintered samples is identified by the gravimetric method. Microhardness of the structural constituents and macrohardness of the sintered materials are measured, too. **Results:** in the modes under study, mechanical activation is accompanied by the formation of tungsten nanoparticles with the minimum size of 25 nm. Alongside this, the powder is exposed to cold working, which hinders further milling. Tungsten nanoparticles, characterized by high surface energy, have a significant effect on the dissolution-precipitation of cobalt during liquid-phase sintering of *Sn-Cu-Co-W* powder material. Addition of nanodispersed tungsten into the material slows down the growth of cobalt particles during sintering and contributes to the formation of a fine-grained structure. The sintered *Sn-Cu-Co-W* material, containing mechanically activated tungsten, features higher hardness of 105…107 HRB, which is explained by cold working of tungsten particles and dispersion hardening. The results can be applied for improving mechanical properties of *Sn-Cu-Co-W* alloys used as metallic binders in diamond abrasive tools.

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Introduction

Sn-Cu-Co and *Sn-Cu-Co-W* alloys are used as metallic binders in diamond abrasive tools manufactured by the powder metallurgy process [1–3]. Binders of the sintered diamond tools have to be physically and chemically compatible with diamond, strong, and highly resistant to abrasion wear.

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One of the methods for enhancing the properties of sintered materials is mechanical activation of powders. It provides milling of the powders, changing its energy state, intensified sintering of the powder materials, and forming a fine-grained structure in it [4–7]. When certain powders are mechanically activated in planetary centrifugal mills, nanoparticles having a high reactivity can be formed [8].

Works [9–13] demonstrate that addition of nanoparticles into metallic binders ensures dispersion hardening of the binders and helps enhance operational properties of the diamond tools considerably. For this purpose, nanopowders of carbon-based materials, boron nitride, and high-melting oxides and carbides (ZrO_2, *WC*) are used. The melting temperature of nanoparticles is known to be lower than that of micropowders [14]. So, to ensure dispersion hardening, nanoparticles have to be preserved in the structure of the material after sintering.

An important characteristic of metallic binders is its adhesion activity to diamond, which provides strong retention of diamond grains in the binders. Nanoparticles located on the matrix-filler interface are known to be able to produce a considerable effect on mechanical properties of the composite material [15–17]. With regard to this, one can suppose that addition of nanodispersed particles of carbide-forming metals to the binder will allow enhancing its adhesion to diamond essentially. One more factor contributing to adhesion activity of binders can be the changed energy state and higher reactivity of the powders after its mechanical activation.

Tungsten is one of the most refractory metals. Annealed tungsten of high purity has the hardness of 225–300 HB, ultimate strength of 800–1,200 MPa, and its relative elongation is close to zero [18]. Such properties make it possible to mill tungsten mechanically to nanosized particles [8, 19]. The authors of this work have conducted preliminary experiments [20] demonstrating the possibility of obtaining 25–90 nm sized particles of tungsten in milling the *PVT* and *W16*,5 grade powders with a planetary centrifugal mill.

With tungsten being a carbide-forming element, adding it into the powder material enhances diamond adhesion activity of the binder. However, under certain conditions, the additive can prevent the binder from sintering, which leads to increase in its porosity while also reducing its hardness and strength [21].

The objective of this work is to study the effect of mechanical activation of tungsten particles on the structure and properties of the sintered *Sn-Cu-Co-W* powder material.

Research technique

For the experiments, the following powders were used: *PO1* tin powder (up to GOST 9723-73), *PMS-1* copper powder (up to GOST 4960-75), and *Diacob-1600* cobalt powder with the particle size of 1–2 μm (by Dr. Fritsch Kg., Germany). It was the *W16,5* special tungsten powder (by Pobedit JSC) containing not less than 99.9% *W* with particles sized 19–24 μm (technical specifications TU 48-19-417-8) that was exposed to mechanical activation. Mechanical activation was performed in the *AGO-2U* planetary centrifugal mill for 5, 15, 60, and 120 minutes at the carrier rotation frequencies of 400, 800, and 1,000 RPM.

Using the above powders, mixtures were prepared containing two kinds of tungsten powders – mechanically activated and non-activated; the proportion of the components was as follows (% wt.): 20 *Sn*; 43 *Cu*; 30 *Co*; 7 *W*.

The 20 g weighted samples were compacted by single-action static pressing in an all-steel mould at the 12 t/cm^2 press power. The resulting cylindrical samples of 21 mm diameter were sintered in vacuum at the temperature of 820 °C for 20 minutes.

After that, the sintered samples were weighed with the *Adventurer AR2140* assay balance (by OHAUS) to find out its density as the ratio of weight to volume.

Next, the structure of sintered materials was examined by scanning electron microscopy and optical metallography. For this, the authors used the *JSM-7500F* (by JEOL) ultrahigh resolution

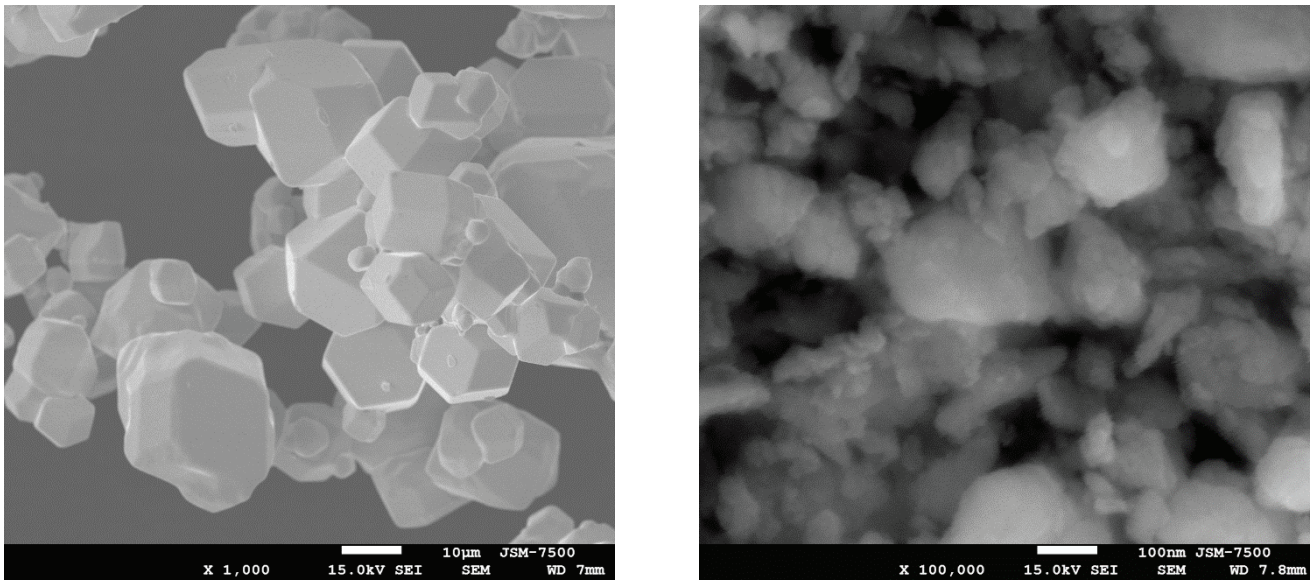
scanning electron microscope, the *EVO HD 15* electron microscope (by ZEISS), and the *AxioObserver.A1m* metallographic microscope (by ZEISS).

As for distribution of elements within the samples, it was studied by X-ray microanalysis using the *EVO HD 15* electron microscope. Microhardness of the structural constituents was measured by indentation of a tetrahedral diamond pyramid under the load of 10 g ($HV_{0,01}$) with the *DuraScan80* hardness meter (by EmcoTest). Hardness of the materials was measured according to the Rockwell method (scale B) using the *TK-2M* hardness meter.

Results and discussion

Effect of mechanical activation on the shape and size of tungsten particles

In Figure 1, one can see the changed shape of tungsten powder particles after mechanical activation. Before the activation, the particles of tungsten were equiaxial polyhedra. After being mechanically activated for 60 min at the 800 RPM, most particles have the equiaxial shape and rough surface. A small quantity of the particles has the splintery shape. As the duration of mechanical activation is increased, the quantity of splintery particles goes down.



a *b*

Fig. 1. Shape of tungsten particles:
a – before mechanical activation; *b* – after mechanical activation

Sizes of the particles were measured using the images obtained with the electron microscope. After the described mechanical activation mode, it ranges within 0.025–12 μm. The particles are distributed according to sizes as follows: d10 = 67 nm; d50 = 220 nm; d90 = 750 nm. Meanwhile, in the mechanically activated powder, the share of nanoparticles sized up to 100 nm exceeds 20% (Figure 2).

The minimum size of the particles, which equals to 25 nm, was obtained at the 800 RPM and the mechanical activation duration of 60–120 min (Table 1).

After mechanical activation, the significant proportion of the powder sticks together forming loose aggregates of up to 80 μm size. The aggregation of nanoparticles is explained by the presence of numerous uncompensated interatomic bonds on its surface. Combining such particles into aggregates contributes to a decrease in its free energy [13].

The shape and size of the resulting powders indicate the following processes occurring during mechanical activation: large particles are split up; the fragments are rolled and gain the rounded shape; small particles

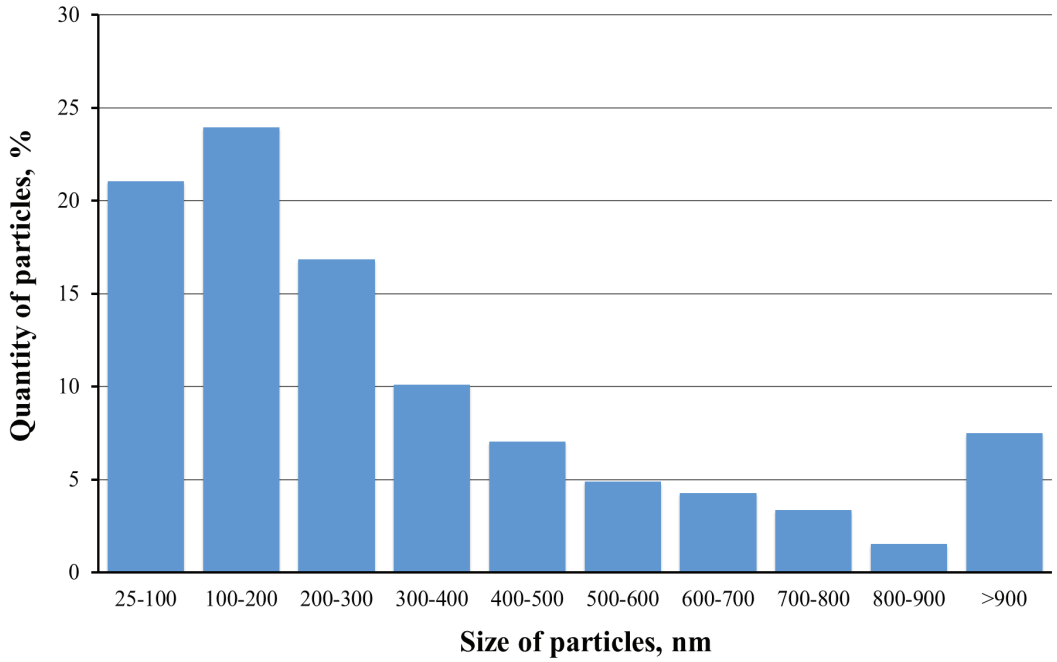


Fig. 2. Size distribution of tungsten particles after mechanical activation

Table 1

Minimum size (nm) of tungsten W16,5 particles depending on mechanical activation mode

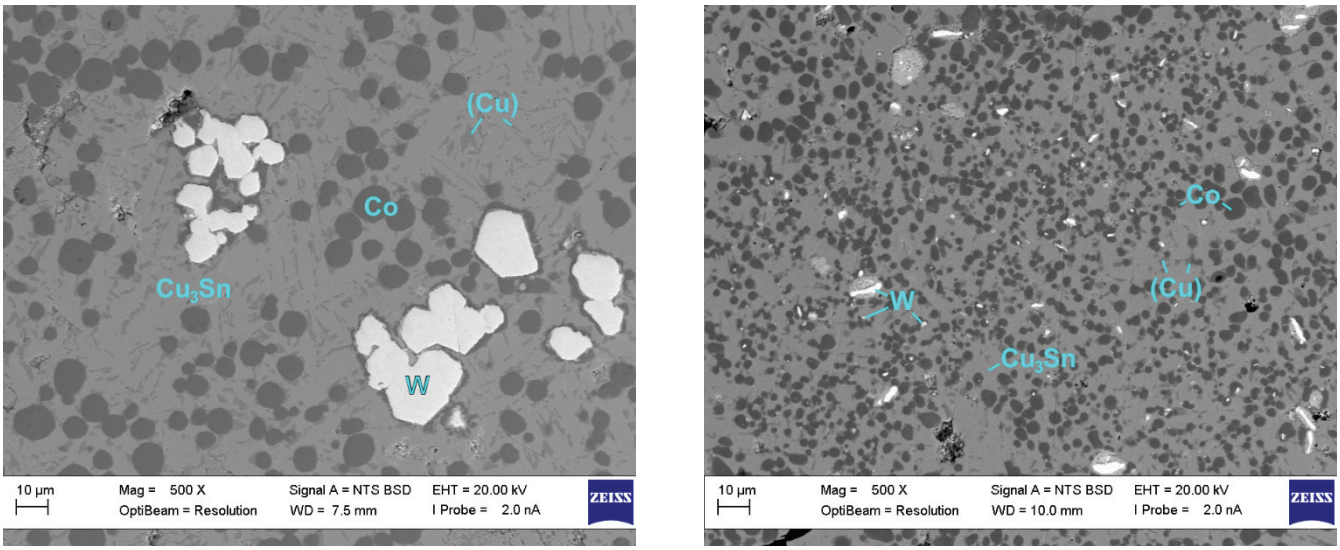
Rotation speed, rpm	Duration of mechanical activation, min			
	5	15	60	120
400	160	132	90	83
800	137	116	25	25
1,000	128	85	90	102

are aggregated. Alongside this, the material is exposed to work hardening, which hinders further milling. It can be seen from Table 1 that increasing the duration of milling from 30 to 60 min practically does not lead to a decrease in particle size. Apparently, this is explained by work hardening of the particles. Increasing the rotation frequency leads to centrifugal forces and the kinetic energy of grinding media building up that results in the above milling processes getting intensified. In particular, aggregation leading to large particle sizes mounts. Due to this, an increase in the carrier rotation frequency (from 800 to 1,000 RPM) does not give any positive effect.

Effect of mechanical activation of tungsten on the structure of sintered Sn-Cu-Co-W materials

Figure 3 shows the microstructure of two kinds of sintered materials: ones with tungsten not exposed to mechanical activation and ones with mechanically activated tungsten. Phase composition of the materials containing non-activated tungsten and its crystallization mechanism are described in works [3, 21]. After sintering, the materials contain the following phases: solid solution of tin and cobalt in copper (*Cu*), the Cu_3Sn intermetallic compound, cobalt particles, and tungsten particles.

Sintering of the materials at 820 °C proceeded with a large quantity of the liquid phase forming. Upon cooling after sintering, a Cu_3Sn compound with a melting point of 755–798 °C was formed from the liquid phase [22]. X-ray microanalysis has shown that in the examined materials, the Cu_3Sn intermetallic phase has almost the same composition, % wt.: 63.2 *Cu*; 33.5 *Sn*; 3.3 *Co*.



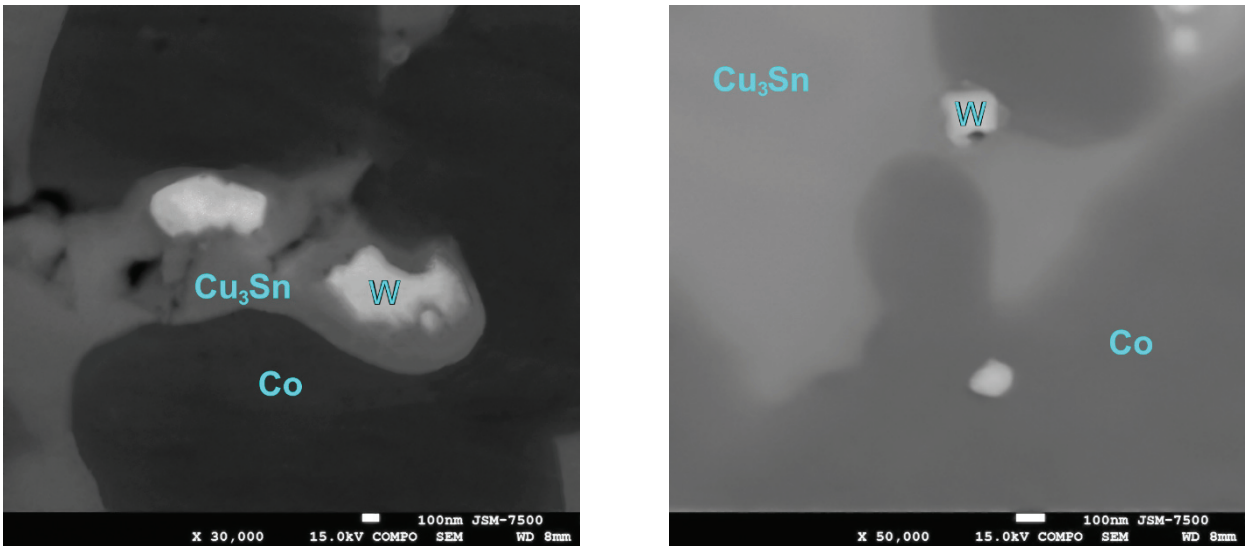
a *b*

Fig. 3. Structure of the sintered *Sn-Cu-Co-W* material:
a – without mechanical activation of tungsten; *b* – with mechanically activated tungsten

Mechanically activated tungsten occurs in the material in the form of separate particles sized 25 nm and more and sintered agglomerates having the cross dimension of up to 0.4 mm. In Figure 4 *b*, one can see nanoparticles of tungsten located within the sintered agglomerate of cobalt and at the interface of cobalt and the Cu_3Sn intermetallic phase; its cross dimension is around 100 nm. Thus, in spite of its higher reactivity and lower melting temperature, tungsten nanoparticles did not get dissolved either in cobalt or in the liquid phase during sintering.

Figure 5 demonstrates that mechanical activation of tungsten contributes to its more uniform distribution in the sintered material. Apparently, the uniform distribution of finely dispersed particles of the carbide-forming tungsten must have a positive effect on adhesion of the binder to the surface of diamond and contribute to stronger retention of diamond grains in the binder [23].

In Figure 3, the effect of mechanical activation of tungsten on the size of cobalt particles can be seen. As a rule, during liquid phase sintering of systems with limited solubility of components, what occurs in it



a *b*

Fig. 4. Particles of mechanically activated tungsten in the sintered material structure:
a – submicron, *b* – nanosized

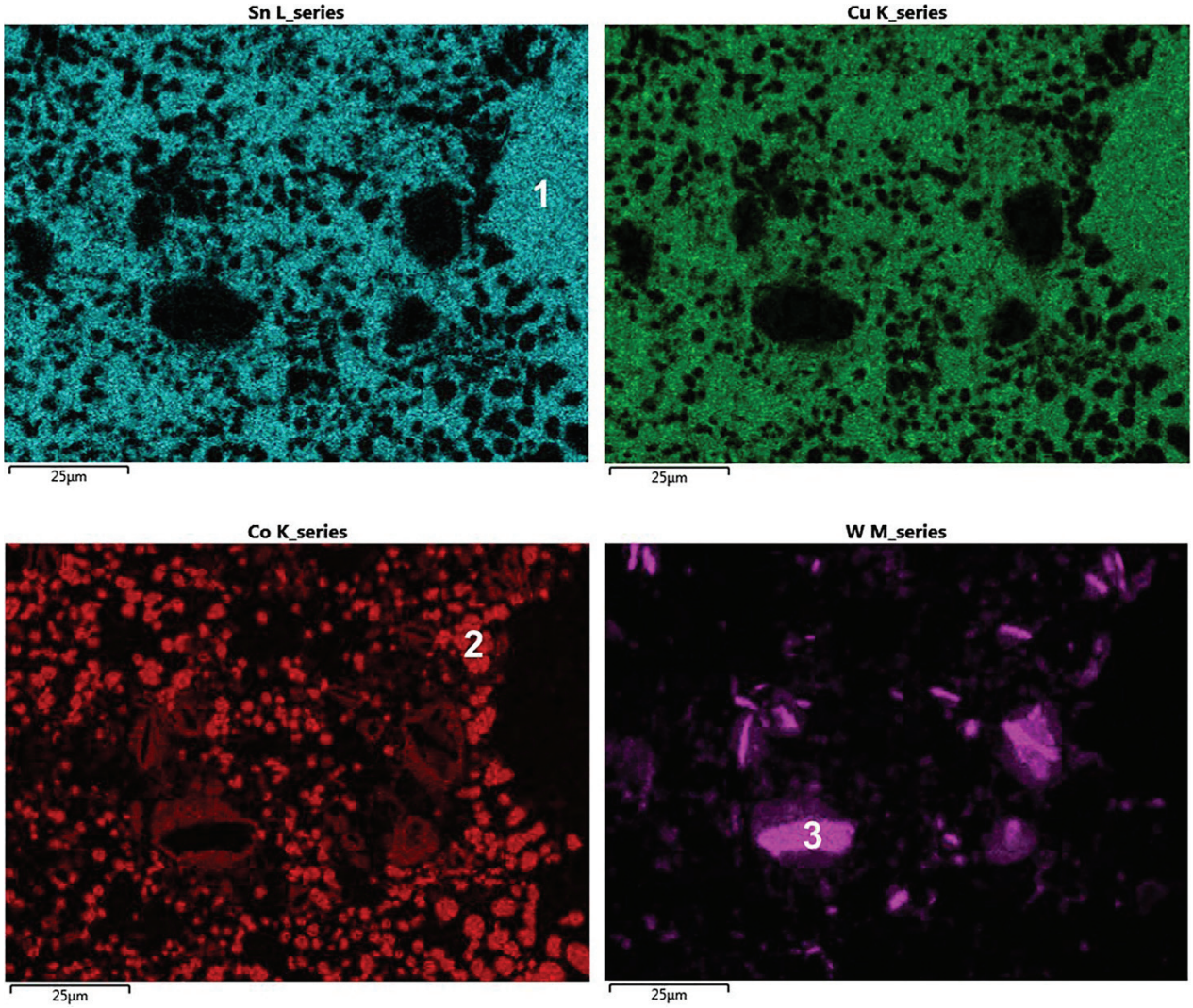


Fig. 5. Element distribution maps for the material with mechanically activated tungsten:
1 – intermetallic compound Cu_3Sn; *2* – cobalt particles; *3* – tungsten particles

is the dissolution-reprecipitation process. This process consists in small particles of the solid phase getting dissolved in the liquid phase and its substance reprecipitating against the surface of larger particles [24, 25]. In the material with tungsten not exposed to mechanical activation, the size of cobalt particles has increased owing to dissolution-reprecipitation from 1.6 μm up to 9–15 μm (see Figure 3 a). In the sintered material containing mechanically activated tungsten, cobalt particles are smaller, about 3–10 μm (Figure 3 *b*).

The effect of mechanical activation of tungsten on the dissolution-reprecipitation of cobalt is explained by the following. In systems containing two solid metals and a liquid phase, the mass transfer is directed toward the metal having the largest free energy [26]. In the ascending order of the surface energy, the components of the system in question are located as follows: *Sn*, *Cu*, *Co*, *W* [27]. Under such conditions, it is transfer of cobalt via the liquid phase to tungsten particles that is the most favorable in terms of energy.

The mass transfer of cobalt to tungsten through the liquid phase is confirmed by component distribution maps given in Figure 5. In the maps, one can see sintered agglomerates of tungsten particles; notably, spaces between the particles are mainly filled up with cobalt. Obviously, cobalt penetrated deep into the tungsten agglomerates together with the liquid phase. Precipitation of cobalt led to blocking of pores of the agglomerates. After that, cobalt could find its way deep into the agglomerates owing to its diffusion over the surface of tungsten particles. The mass transfer of cobalt to tungsten through the liquid phase occurs without mechanical activation of tungsten, too. In Figure 4 a, it can be seen that particles of tungsten not exposed to mechanical activation are surrounded by "enclosures" formed as a result of precipitation of cobalt from the liquid phase.

As it has been demonstrated above, mechanical activation has led to higher fineness of tungsten powder, with the area of its free surface increasing, respectively. As a result, precipitation of cobalt from the liquid phase against the particles of tungsten has intensified. Thus, mechanical activation of tungsten has reduced the mass transfer from small cobalt particles to larger ones and contributed to the formation of the more dispersed, fine-grained structure in the sintered material.

The effect of high-melting nanoparticles on dissolution-reprecipitation of another solid phase during liquid phase sintering needs further investigation. This phenomenon opens up new opportunities for acting on structure formation in sintering to obtain materials with the required structure and properties.

Effect of mechanical activation of tungsten on the porosity of sintered Sn-Cu-Co-W materials

In the sintered *Sn-Cu-Co-W* materials, there is a minor quantity of isolated closed pores. The material with non-activated tungsten has the density of 8.16 g/cm^3 (porosity of 8%). Meanwhile, the material with milled tungsten has the density of 7.72 g/cm^3 (porosity of 13 %). With its high chemical activity, finely dispersed tungsten tends to adsorb atmospheric gases and get oxidized. The WO_2 tungsten oxide is decomposed when heated in vacuum up to the temperature of 800 °C [28]. Apparently, at the temperature of sintering, oxides are decomposed, and gases are extracted in the closed pores subsequently. The pressure of gases in the closed pores prevents it from healing, which results in higher porosity of the sintered material.

Effect of mechanical activation of tungsten on the hardness of sintered Sn-Cu-Co-W materials

It is clear from Table 2 that the hardest structural constituent of *Sn-Cu-Co-W* materials is particles of tungsten. Mechanically activated tungsten has a 1.8–2.2 times higher hardness. For technical reasons, hardness of nanoparticles cannot be measured with the 10 g indenter load. As for larger tungsten particles, having the cross dimension of 10–12 μm, its microhardness is 823–1,162 $HV_{0,01}$. Higher hardness of mechanically activated tungsten is associated with work hardening of its particles. Recrystallization temperature of tungsten is known to be much higher than 820°C, so during sintering of the material, work hardening of tungsten particles was retained.

Table 2

Microhardness $HV_{0,01}$ of the structural constituents of the sintered Sn-Cu-Co-W material

Sintered material	Microhardness $HV_{0,01}$ of the structural constituents			
	(Cu)	Cu_3Sn	Co	W
without mechanical activation of tungsten	245±12	367±7	137±16	496±29
with mechanical activation of tungsten	259±22	384±14	140±16	992±169

A part of mechanically activated tungsten occurs within the material in the form of sintered agglomerates; its structure is given in Figure 6 (the light image; the sample was treated with the solution containing 5 g of ferrichloride, $FeCl_3$, 15 ml of hydrochloric acid, HCl, and 100 ml of water). It can be seen that necking was formed between contact tungsten particles during sintering. Microhardness of the agglomerates was measured at the 100–500 g indenter load; meanwhile, hardness impresses have been obtained with the diagonal length exceeding the size of individual tungsten particles (see Figure 6). When the indenter was pressed in, the particles of tungsten did not get disconnected or crumbled away. In spite of its porous structure, the agglomerates feature high microhardness of 582–1,223 HV.

In the structure of the materials under study, particles of tungsten occupy a small volume (less than 5 %), so its hardness has little effect on the general hardness of the material. In Figure 5, it can be seen that the largest volume in the structure of the materials belongs to the Cu_3Sn intermetallic phase. In the material with mechanically activated tungsten, hardness of the Cu_3Sn intermetallic compound is much higher

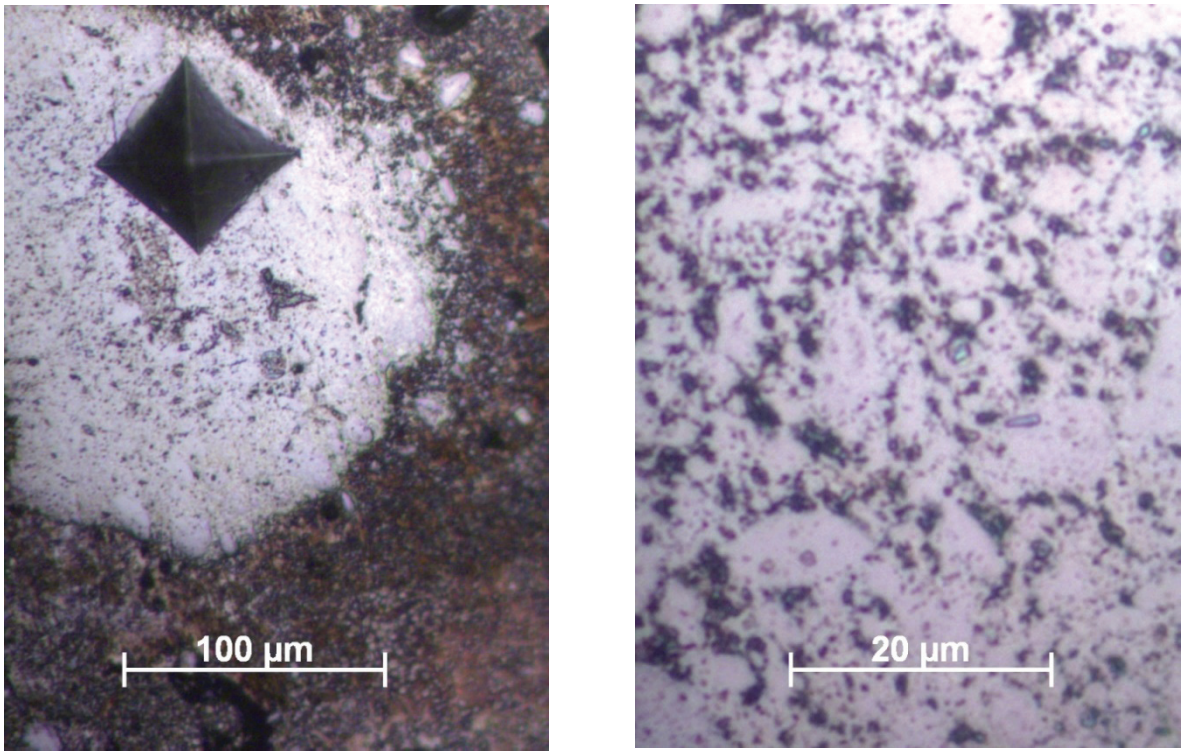


Fig. 6. Microstructure of the sintered tungsten agglomerates

(see Table 2). Obviously, this is associated with hardening of the intermetallic compound by finely dispersed tungsten particles.

The sintered material containing tungsten not exposed to mechanical activation has the macrohardness of 101–102 HRB. Meanwhile, the material with mechanically activated tungsten features the higher hardness of 105–107 HRB, which is associated with work hardening of tungsten particles and dispersion hardening of other structural constituents.

Conclusions

1. The effect of mechanical activation on the morphology of particles and fineness of the W16,5 grade tungsten powder has been studied. In the examined modes, mechanical activation is accompanied by the formation of tungsten nanoparticles with the minimum size of 25 nm. Alongside this, the powder is exposed to work hardening, which hinders further milling.

2. With its high surface energy, tungsten nanoparticles produce a noticeable effect on cobalt dissolving and depositing in liquid phase sintering of the *Sn-Cu-Co-W* powder material. Introducing nanodispersed tungsten into the material slows down the growth of cobalt particles and helps to obtain a fine-grained structure.

3. The sintered *Sn-Cu-Co-W* material containing mechanically activated tungsten features higher hardness of 105–107 HRB, which is explained by work hardening of tungsten particles and dispersion hardening of other structural constituents.

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Conflicts of Interest

The authors declare no conflict of interest.