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Research article
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Dissolution-precipitation and cobalt grain growth during liquid phase sintering of Cu–Sn–Co and Cu–Sn–Co–W powder materials

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Abstract. The study presents the results of the dissolution-precipitation process and cobalt grain growth during liquid phase sintering of Cu–Sn–Co and Cu–Sn–Co–W powder materials. The samples were obtained by static pressing of mixtures of technically pure copper, tin, cobalt, and tungsten powders. The average particle size of cobalt was 1.6 μm, and tungsten was 20 μm. Some of the samples contained mechanically activated tungsten with an average particle size of 0.14 μm. Sintering of the materials was carried out in a vacuum at temperatures of 820 and 1100 °C for durations of 5, 20, and 120 min. The structure of the sintered materials was studied using scanning electron microscopy and optical metallography. Elemental distribution maps in the materials were obtained through *X*-ray microanalysis. The grain sizes of cobalt were measured using specialized software. The largest grain size was observed in the Cu–Sn–Co material: after sintering at the specified temperatures and durations, it ranged from 8 to 46 μm. It was found that the most intensive grain growth occurred within the first 20 min of sintering. The addition of tungsten powder to the Cu–Sn–Co material contributed to the formation of finer cobalt grains. This is explained by the fact that tungsten particles, possessing high surface energy, act as nucleation centers for cobalt crystallization from the liquid phase. Mechanical activation of the tungsten powder increases its free surface area and enhances the mass transfer of Co through the liquid phase to the W particles. This helps to reduce the deposition of material on large Co particles and prevent their growth. As a result, in the Cu–Sn–Co–W material containing mechanically activated tungsten, the minimum average cobalt grain sizes were obtained, ranging from 3 to 25 μm.

Keywords: liquid phase sintering, dissolution-precipitation, mass transfer, nucleation centers, mechanical activation, grain size

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Растворение–осаждение и рост зерна кобальта при жидкофазном спекании порошковых материалов Cu–Sn–Co и Cu–Sn–Co–W

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Аннотация. Представлены результаты исследования процесса растворения–осаждения и роста зерна кобальта при жидкофазном спекании порошковых материалов Cu–Sn–Co и Cu–Sn–Co–W. Образцы получали статическим прессованием смесей порошков технически чистых меди, олова, кобальта и вольфрама. Средний размер частиц кобальта составлял 1,6 мкм, вольфрама – 20 мкм. Часть образцов содержали механоактивированный вольфрам со средним размером частиц 0,14 мкм. Спекание материалов проводили в вакууме при температурах 820 и 1100 °С с выдержками 5, 20 и 120 мин. Структуру спеченных материалов исследовали методами сканирующей электронной микроскопии и оптической металлографии. Карты распределения элементов в материалах получали путем микрорентгеноспектрального анализа. Размеры зерна кобальта измеряли с помощью специализированного программного обеспечения. Наибольший размер зерна наблюдался в материале Cu–Sn–Co: после спекания при указанных температурах и выдержках он составлял 8–46 мкм. Установлено, что наиболее интенсивный рост зерна происходит в течение первых 20 мин спекания. Добавка порошка вольфрама в материал Cu–Sn–Co способствует формированию более мелкого зерна кобальта. Это объясняется тем, что частицы вольфрама, обладающего высокой поверхностной энергией, играют роль центров кристаллизации кобальта из жидкой фазы. Механическая активация порошка вольфрама увеличивает площадь его свободной поверхности и способствует усилению массопереноса Co через жидкую фазу к частицам W. Это позволяет ослабить осаждение вещества на крупных частицах Co и предотвратить их рост. В результате в материале Cu–Sn–Co–W, содержащем механоактивированный вольфрам, получены минимальные значения среднего размера зерна кобальта, находящиеся в диапазоне 3–25 мкм.

Ключевые слова: жидкофазное спекание, растворение–осаждение, массоперенос, центры кристаллизации, механическая активация, размер зерна

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Introduction

The production of sintered materials with a fine-grained structure and enhanced mechanical properties is a current challenge in powder metallurgy.

During the liquid phase sintering of various materials, a dissolution-precipitation process occurs, which is observed when the solid phase material is soluble in the liquid phase [1–3]. The dissolution-precipitation of the solid phase material contributes to the shrinkage and densification of the material, but it can also lead to the formation of a coarse-grained structure and deteriorate its mechanical properties [4–6].

Various methods are employed to achieve a fine-grained structure in sintered materials, including those aimed at suppressing the dissolution-precipitation process. These methods include homogenizing the sintered mixture by the size and shape of the powder particles [6; 7], reducing the sintering duration [8; 9], and introducing grain growth inhibitors into the powder material [10; 11].

To reduce the duration of the material's exposure to high temperatures, spark plasma sintering is used [8; 9]. However, this method is not applicable to all materials and products. During spark plasma sintering, the molds wear out rapidly, negatively affecting the dimensional accuracy of the products.

To inhibit grain growth, small amounts of chromium, vanadium, niobium, or other refractory metal carbides are added to tungsten-cobalt hard alloys [10; 11]. There is also evidence of grain growth inhibition in hard alloys with the addition of aluminum oxide nanoparticles [12]. During liquid phase sintering, these substances precipitate on the surface of tungsten carbide particles, inhibiting their growth. However, the addition of these substances leads to the formation of brittle layers around the tungsten carbide particles, which negatively affects the mechanical properties of the sintered materials.

Some composite materials include diamond-graphite nanoparticles, which serve as nucleation centers for the liquid phase crystallization during cooling after sintering [13]. The addition of such particles allows for the formation of fine grains from the liquid phase, but it does not prevent dissolution-precipitation and grain growth of the solid phase during sintering. Thus, there is a need to develop new methods for forming a fine-grained structure in sintered materials.

The study [14] demonstrated that when two dissimilar solid metals, which are only partially soluble in the liquid phase, are placed in a melt, mass transfer occurs towards the metal with higher surface energy. This phenomenon provides opportunities to influence the dissolution-precipitation process by introducing particles with high surface energy into the powder material.

Cu–Sn–Co powder materials are used as metal binders in diamond abrasive tools [15–17], which exhibit resistance to abrasive wear and good adhesion to diamond grains [16]. During the liquid phase sintering of these materials, cobalt grain growth occurs, which negatively impacts their mechanical properties. To prevent this effect, it is advisable to introduce tungsten particles into the Cu–Sn–Co material. Compared to other components of this alloy, tungsten has a higher surface energy, ranging from 2.7 to 5.57 J/m^2 according to various sources [18; 19]. Additionally, tungsten is a carbide-forming metal, and its addition improves the adhesion of Cu–Sn–Co binders to diamond abrasive grains [17].

The objectives of this study were as follows:

– to identify the patterns of dissolution-precipitation and cobalt grain growth during the liquid phase sintering of Cu–Sn–Co and Cu–Sn–Co–W powder materials;

– to develop a new method for producing sintered materials with a fine-grained structure.

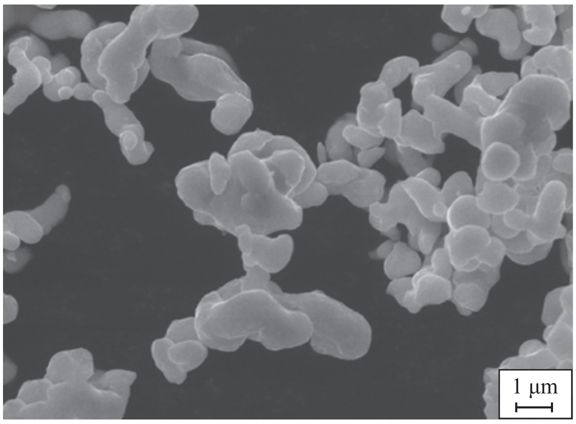

Fig. 1. Shape and size of Diacob-1600 cobalt powder particles

Рис. 1. Форма и размер частиц порошка кобальта Diacob-1600

Research methodology

For the research, the following metallic powders were used: PMS-1 copper powder (GOST 4960–75), PO1 tin powder (GOST 9723–73), and Diacob-1600 cobalt powder (Dr. Fritsch Kg., Germany). The latter was obtained by the carbonyl method, with rounded particles averaging 1.6 μm in size (Fig. 1).

W16.5 special tungsten powder from “Pobedit” JSC (Russia), consisting of equiaxed particles with an average size of about 20 μm, was mechanically activated using a AGO-2U planetary centrifugal mill (NPO “NOVITS”, Russia) for 60 min at a rotational speed of 800 rpm [20]. After treatment, the tungsten particles retained an equiaxed shape and ranged in size from 0.025 to 12.0 μm, with an average size of approximately 0.14 μm.

Mixtures were prepared from the specified powders, and their compositions are given in the Table.

Powder samples weighing 20 g were compacted by uniaxial static pressing under a load of 850 MPa. The resulting cylindrical samples, with a diameter of 21 mm, were sintered in a vacuum at temperatures of 820 and 1100 °C for 5, 20 and 120 min, and then microsections were prepared. To reveal the microstructure, an etchant containing 5 g of $FeCl_3$, 15 ml of HCl, and 100 ml of water was used.

Composition of powder materials

Состав порошковых материалов

Material composition	Content of powder components, wt. %				
	Copper PMS-1	Tin PO1	Cobalt Diacob-1600	Tungsten W16.5	Mechanically activated tungsten
Cu–Sn–Co	46	21	33	–	–
Cu–Sn–Co–W	43	20	30	7	–
Cu–Sn–Co–W(*m*)	43	20	30	–	7

The microstructure of the sintered alloys was studied using a scanning electron microscope EVO HD 15 and a metallographic microscope AxioObserver.A1m (both manufactured by Carl Zeiss AG, Germany) at magnifications of 50–1000×. Grain size measurements in the sintered materials were carried out using AxioVision Rel.4.8 software (Carl Zeiss AG).

The elemental distribution in the samples was investigated by *X*-ray microanalysis using the EVO HD 15 microscope.

Results and discussion

In Fig. 2, the microstructure of the Cu–Sn–Co–W material containing tungsten W16.5, sintered at 820 °C for 20 min, is shown. The phase composition of the Cu–Sn–Co and Cu–Sn–Co–W alloys has been investigated in studies [20; 21].

After sintering at temperatures of 820–1100 °C, the materials consist of the following phases: a copper-based solid solution (Cu), the intermetallic phase $Cu_{10}Sn_3$ (ξ-phase), and α-Co. In the Cu–Sn–Co–W samples, α-W and metastable β-W are present. Intermetallic compounds of Co–Sn and Co–W were not detected in the sintered materials. *X*-ray microanalysis showed that tungsten does not dissolve in the copper-tin phases (Cu and ξ) or in α-Co. The W particles observed in Fig. 2 retained their original shape characteristic of W16.5 powder. In the structure of the material containing mechanically activated tungsten powder, fine W particles remain after sintering, including some with transverse sizes less than 100 nm.

The structural formation of the materials during sintering consisted of several stages:

– formation of the liquid phase: melting of tin, its diffusion into copper particles, and subsequent melting of surface layers enriched with tin;

– viscous flow of the liquid and rearrangement of Co and W particles;

– dissolution-precipitation of cobalt;

– during subsequent cooling, crystallization of the liquid phase into a solid solution (Cu) and intermetallic ξ-phase.

According to *X*-ray microanalysis, the cobalt content in the (Cu) and ξ phases is 2 wt. % and 3 wt. %, respectively. This indicates its solubility in the liquid phase at sintering temperatures.

The dissolution-precipitation process leads to noticeable changes in the shape and size of Co particles. In Fig. 3–5, they appear as the brightest component. With increasing temperature and duration of sintering, Co particles become more equiaxed and more uniform in size.

Studies [22; 23] present an equation showing a nonlinear relationship between the duration of liquid phase sintering and the size of the solid phase particle surrounded by the melt:

$$D^n - D_0^n = k\tau,$$

where D^n and D_0^n are the particle sizes after and before sintering; τ is the sintering duration; k is a constant; and the exponent n depends on which stage determines the intensity of the solid phase material's dissolution-precipitation:

– if the slowest process is the dissolution of the solid phase in the liquid, then $n = 2$;

– if the determining stage is the diffusion of the dissolved substance in the liquid phase, then $n = 3$.

The kinetic curves of Co particle growth, shown in Fig. 6, align well with the given equation if the parameter $n = 3$. This indicates that the rate of Co particle growth is limited by the diffusion rate of Co atoms through the liquid phase.

In Fig. 6, it is evident that the size of Co particles grows most intensively during the first 5–20 min of sintering. During the dissolution-precipitation process, Co

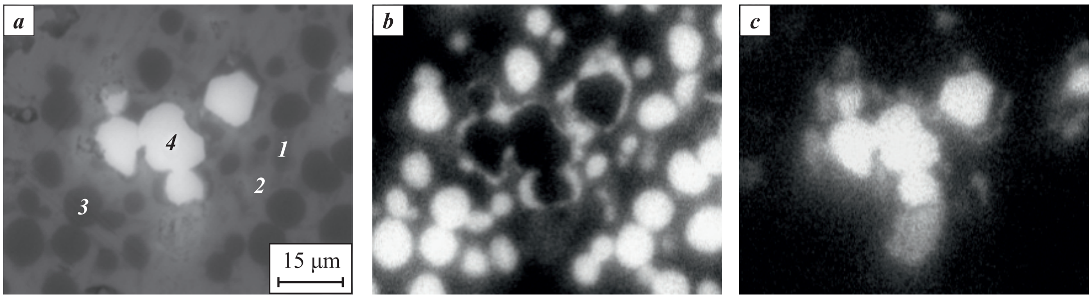

Fig. 2. Microstructure of the sintered Cu–Sn–Co–W material (***a***) and distribution maps of cobalt (***b***) and tungsten (***c***)
1 – solid solution (Cu); ***2*** – ξ-phase; ***3*** – α-Co; ***4*** – tungsten particles

Рис. 2. Микроструктура спеченного материала Cu–Sn–Co–W (***a***) и карты распределения кобальта (***b***) и вольфрама (***c***)
1 – твердый раствор (Cu); ***2*** – ξ-фаза; ***3*** – α-Co; ***4*** – частицы W

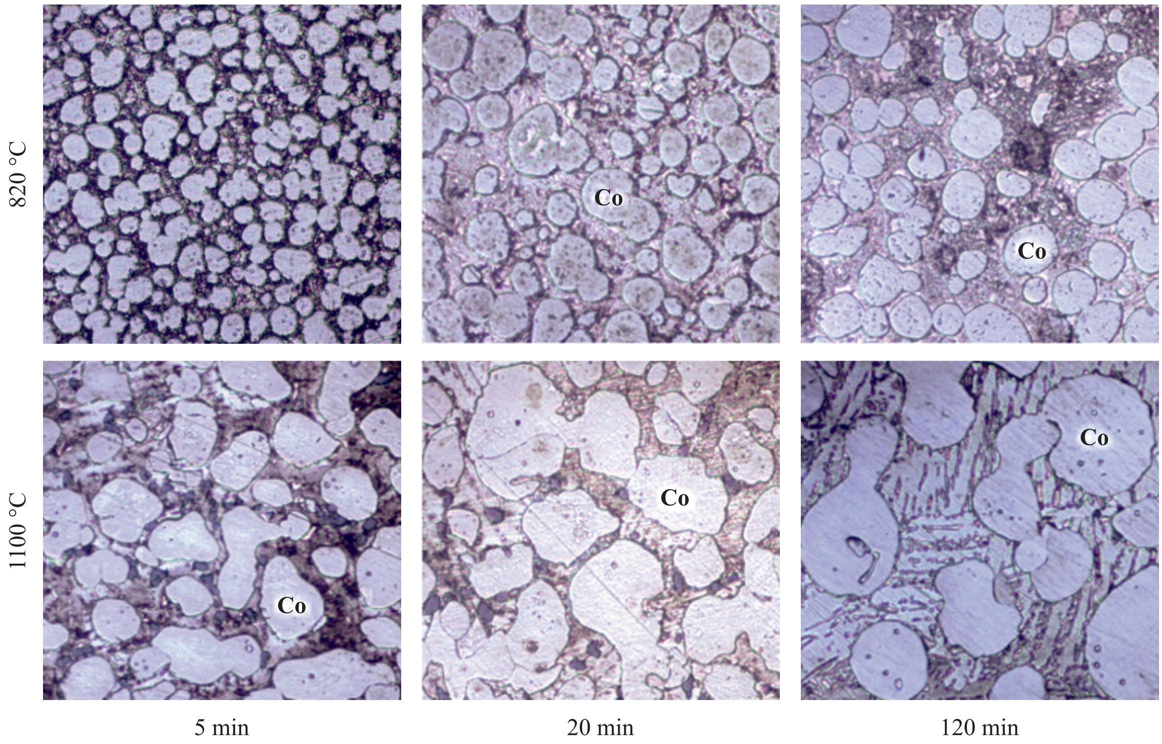

Fig. 3. Structure of Cu–Sn–Co alloys
Light image (×500)

Рис. 3. Структура сплавов Cu–Sn–Co
Световое изображение (×500)

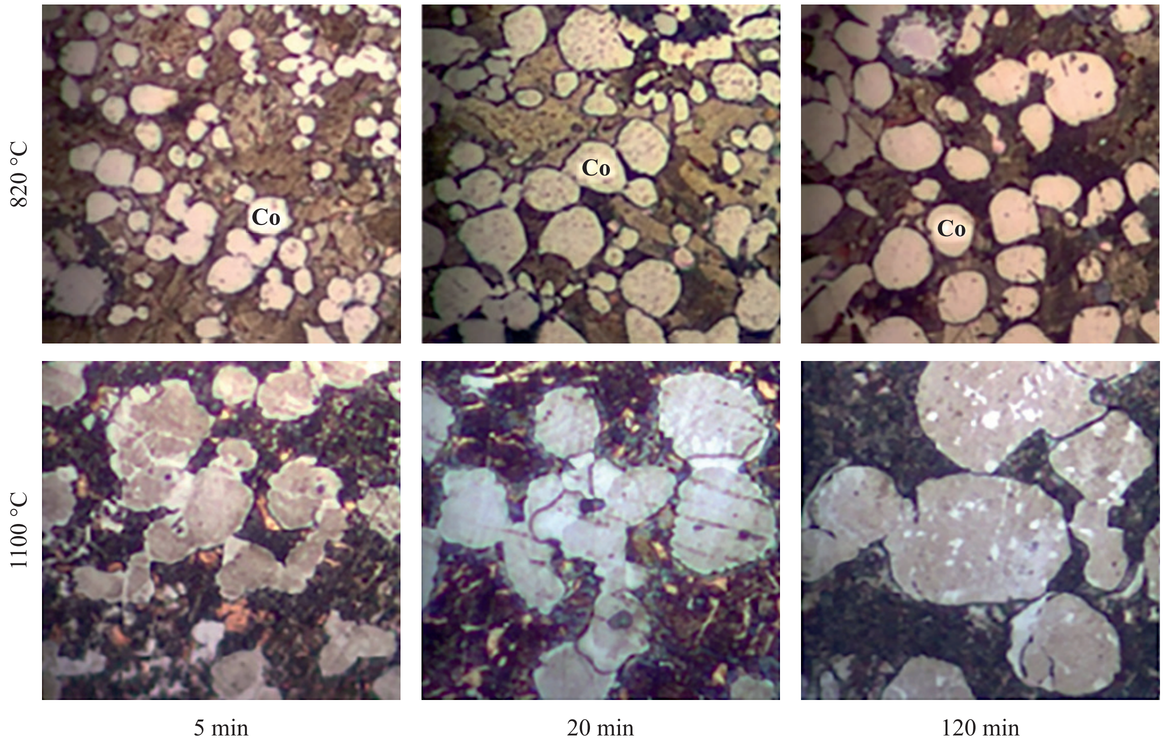

Fig. 4. Structure of Cu–Sn–Co–W alloys containing tungsten W16.5
Light image (×1000)

Рис. 4. Структура сплавов Cu–Sn–Co–W, содержащих вольфрам W16,5
Световое изображение (×1000)

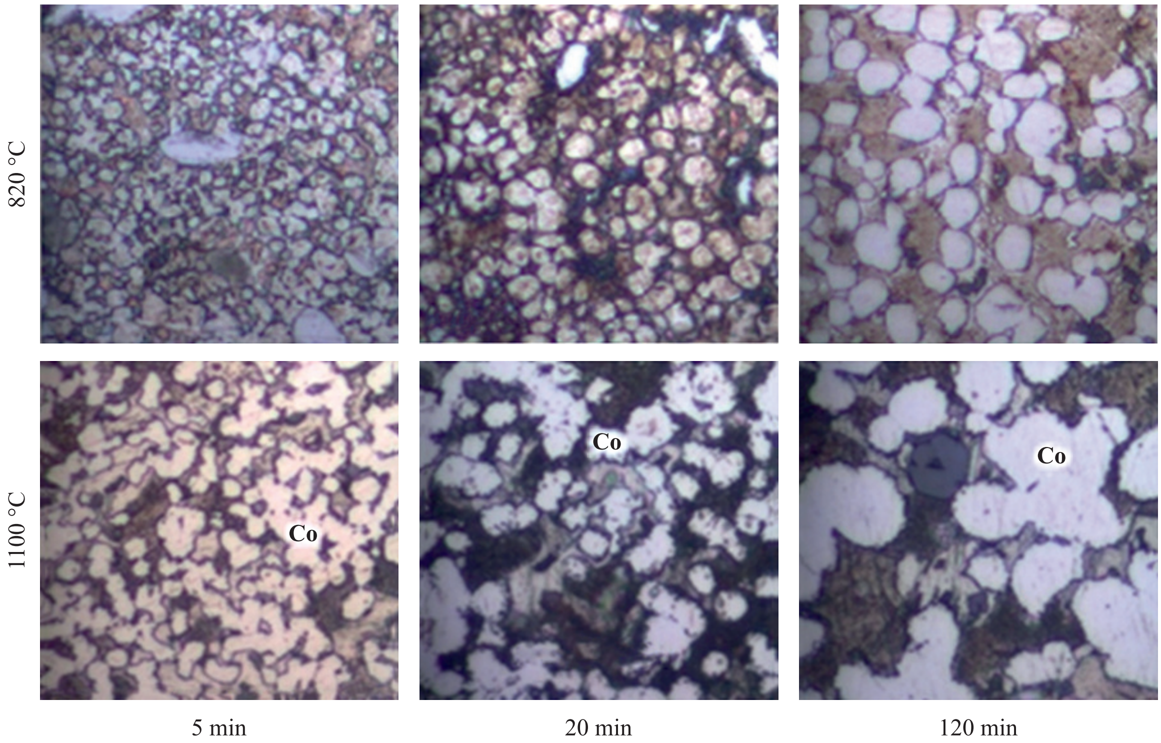

Fig. 5. Structure of Cu–Sn–Co–W(*m*) alloys containing mechanically activated tungsten
Light image (×1000)

Рис. 5. Структура сплавов Cu–Sn–Co–W(*m*), содержащих механоактивированный вольфрам
Световое изображение (×1000)

particles become more uniform in shape and size, leading to a decrease in the difference in chemical potentials of cobalt within the particles. As a result, during further isothermal holding, the driving force for mass transfer weakens, and the rate of Co grain growth slows down.

The addition of tungsten to the Cu–Sn–Co powder material significantly reduces the size of Co grains. *X*-ray microanalysis of the sintered material showed a cobalt layer on the surface of W particles (visible in the component distribution maps, Fig. 2). It is known that tungsten has a higher surface energy than cobalt [18; 19], which facilitates the mass transfer of cobalt through the liquid phase to the surface of W particles during sintering. The thickness of the Co layer is predominantly uniform (Fig. 7, *a*). Some uneven areas with greater thickness formed due to the sintering of Co particles to W particles (Fig. 7, *b*). The probable mechanism of sintering is the dissolution of Co at con-

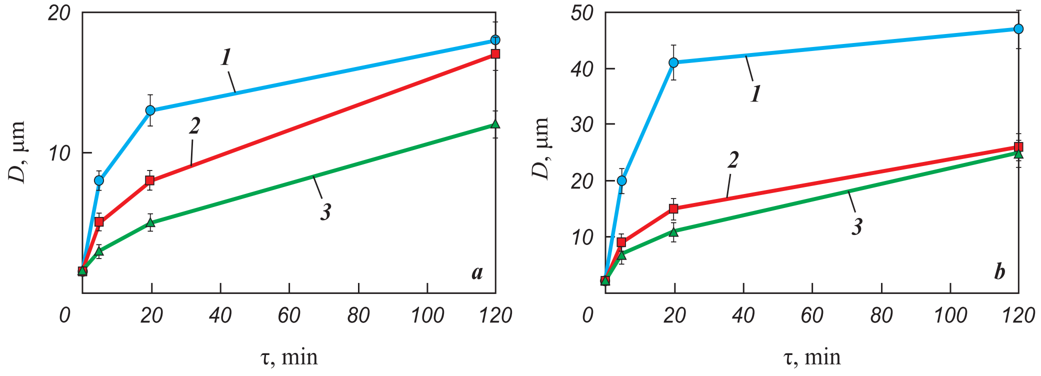

Fig. 6. Dependence of cobalt grain size on sintering duration at 820 °C (***a***) and 1100 °C (***b***)
1 – Cu–Sn–Co; ***2*** – Cu–Sn–Co–W; ***3*** – Cu–Sn–Co–W(*m*)

Рис. 6. Зависимость размера зерна кобальта от продолжительности спекания при *t* = 820 °C (***a***) и 1100 °C (***b***)
1 – Cu–Sn–Co; ***2*** – Cu–Sn–Co–W; ***3*** – Cu–Sn–Co–W(*m*)

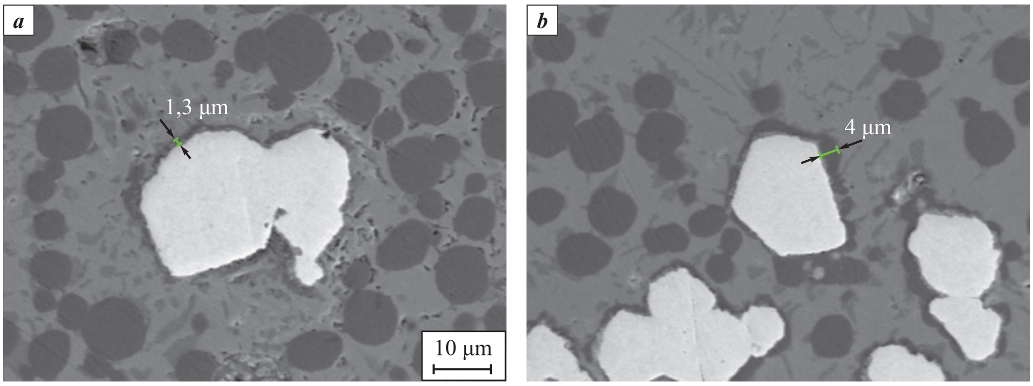

Fig. 7. Cobalt layer on tungsten particles formed by the precipitation of Co from the liquid phase (***a***) and sintering of Co particles (***b***)

Рис. 7. Кобальтовый слой на частицах вольфрама, сформированный за счет осаждения Co из жидкой фазы (***a***) и припекания частиц Co (***b***)

tact points and its precipitation on the free surface of W particles [1; 3].

It should be noted that, due to their high density, tungsten particles occupy only 2 % of the volume of the sintered material at a mass fraction of 7 %. As a result, the diffusion flux directed from Co particles to W particles is limited by the relatively small free surface area of the tungsten particles.

After mechanical activation under the above conditions, the free surface area of the tungsten powder increases significantly. In the mechanically activated powder, the average particle size of W is about 0.14 μm. Calculations using known geometric formulas show that reducing the diameter of equiaxed (spherical) particles from 20 to 0.14 μm increases the free surface area of the powder by approximately 140 times.

Increasing the free surface area of tungsten enhances the mass transfer of cobalt through the liquid phase to the W particles. As a result, deposition on large Co particles decreases, leading to the formation of a fine-grained structure in the material (see Fig. 5).

The identified patterns of cobalt mass transfer were utilized in developing a new method for producing sintered materials with a fine-grained structure [24]. This method involves introducing fine refractory particles with high surface energy into the powder material. The addition of these particles alters the direction of mass transfer during liquid phase sintering and inhibits the growth of solid phase grains.

The positive effect of introducing mechanically activated tungsten powder into the Cu–Sn–Co material is evident in the following example. At a sintering temperature of 820 °C and a holding time of 20 min, Co grains with an average size of 13 μm form in the material. The introduction of 7 wt. % mechanically activated tungsten powder under the same sintering conditions results in Co grains with an average size of 5 μm.

Conclusions

1. The study determined the patterns of the dissolution-precipitation process of cobalt during the liquid phase sintering of Cu–Sn–Co and Cu–Sn–Co–W powder materials.

2. It was found that the addition of tungsten powder to the Cu–Sn–Co alloy promotes the formation of finer cobalt grains. This is explained by tungsten particles with high surface energy acting as nucleation centers for cobalt crystallization from the liquid phase.

3. Mechanical activation of tungsten powder increases its free surface area and enhances the mass transfer of cobalt through the liquid phase to the W particles. This helps to reduce the deposition of material on large Co particles and further decreases the size of the cobalt grains.

4. Based on the identified patterns, a new method for producing sintered materials with a fine-grained structure was proposed. The essence of this method is that the addition of fine refractory particles with high surface energy to the powder material changes the direction of mass transfer and inhibits the growth of the solid phase grains during liquid phase sintering.

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D. A. Golius – planned experiments, prepared powder mixtures, pressed and sintered experimental samples, and prepared microsections.
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