



Peculiarities of Obtaining Nanostructured Materials Compacted by the Method of Hot Pressing Due to the Passage of Direct Electric Current

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The paper investigates the peculiarities of microstructure formation in tungsten monocarbide nanopowders during hot pressing with direct passage of electric current. On the basis of the conducted research, the optimal modes of hot pressing by direct current passage during sintering of tungsten monocarbide nanopowders were selected: heating at a rate of 50 °C/min to 300 °C, then 250 °C/min to 900 °C, then isothermal holding at 900 °C for 2...3 min. After that, it is necessary to raise the temperature at a rate of 500 °C/min to 1650 °C with a holding time of 1 min at this temperature. Hot pressing with direct passage of electric current (electroconsolidation) has common features with conventional and activated sintering, hot pressing, and at the elementary volume level – with microelectric welding. However, this method has some features that favorably distinguish it from other methods of powder consolidation. The passing electric current contributes to the release of heat according to the Joule-Lenz law. Turning on the current (single or multiple) often has a positive effect on the sintering results. In addition to Joule heating, heat due to dielectric losses is released in the sintering samples. Conducted studies have shown that the passage of alternating current accelerates the cauterization of particles and contributes to a higher and more uniform density in volume than in the case of any other electrical influence. Due to the formation of the so-called skin effect during alternating current, the temperature of the cross-section of the sample is equalized. As a result, the cross-section of the sample is uniform.

Keywords: hot pressing, electroconsolidation, tungsten monocarbide.

1. Introduction

Numerous studies have established that nanostructured ceramics have unique properties and operational characteristics due to the formation of a fundamentally different structure compared to large-crystalline counterparts [1, 2].

Nanometer and submicron sizes of structural elements (grains, aggregates) lead to increased crack resistance (impact toughness), strength and hardness of ceramics, composites and potentially allow to reach the parameters of "ceramic steel". Functional nanoceramics (piezo-, ferro-, dielectric, superconducting, etc.) improve electrophysical, magnetic, and other properties [3, 4]. Taking this into account, the task of developing competitive technologies for the manufacture of products for various purposes from nanostructured ceramics is very urgent.

The most important stage in the technology of manufacturing nanoceramics is the formation of high-quality pressings (compacts) of a given shape from ceramic nanopowders. Nanopowders of ceramic compositions (often very complex) are characterized by metastability of the structural-phase state, developed specific surface area and, as a result, high surface activity and tendency to agglomerate [5, 6]. Dust-like nanopowders, as a rule, are characterized by poor molding and pressing due to the specificity of their physicochemical properties – in particular, high interparticle and wall friction (caused by a high specific surface area), agglomeration and a significant amount of sorbed impurities [7, 8]. The task is to ensure a uniform distribution of the density of such nanopowders in compacts of even a complex shape, to preserve the nanostructure in pressings for the formation of nanosized grains during sintering, i.e. to create conditions for inhibiting the growth of grains (preventing recrystallization) and for sintering high-quality nanoceramic products with specified functional properties. It is also important to ensure chemical purity and the required phase composition of finished products. In this regard, the issue of creating products from dry nanopowders that ensure uniform density distribution in pressings of a complex shape without the use of any plasticizers, which are potential sources of impurities and additional porosity in sintered products, is relevant. In pressings, internal stresses and macrodefects (delamination, cracks, etc.) are minimized, thereby eliminating the germs of such macrodefects even during sintering of pressings. These factors make it possible to produce high-quality ceramic and composite products from nanopowders. One of these methods is the production of products from nanopowders by the method of direct current transmission.

Electroconsolidation has common features with conventional and activated sintering, hot pressing, and at the elementary volume level – with microelectric welding [9, 10]. However, this method has some features that favorably distinguish it from other methods of powder consolidation.

The effect of electric current on powders and other dispersed materials gives rise to a number of interesting phenomena from a scientific point of view. The study of the effect of electric current on sintered metal powders is well studied in papers [11, 12]. Some features of the processes of sintering powders of refractory compounds are considered in papers [13, 14].

The regularities of the sintering mechanism of tungsten monocarbide nanopowders have not been studied much. Earlier studies showed that it is possible to obtain a material with high

physical and mechanical properties [15, 16]. It is assumed that intense mass transfer occurs at the contact areas between neighboring particles under the influence of the current. As a result, there is a rapid sintering process in powder pressing.

The purpose of the paper is the selection of optimal modes of hot pressing by direct current passage during sintering of tungsten monocarbide nanopowders.

2. Materials and methods

Imported tungsten monocarbide powders produced by Wolfram (Austria) obtained by plasma chemical method (Catalog number 74-0601, lot number IMC6002-2, purity 99.95 %) with a grain size of 40...70 nm, aluminum oxide nanopowders with a grain size of 60...80 nm produced by Infarmat (USA).

For the formation of ceramic products, a device for hot vacuum pressing was used, designed and manufactured at the department "Materials and technologies for manufacturing products for transport purposes" of the Ukrainian State University of Railway Transport.

Structural studies were carried out by the method of raster electron microscopy (JSM-840) on fractures of hot-pressed samples, grinds, as well as initial powders. The error in density measurement is less than 1 %.

X-ray phase analysis was carried out on an XRD unit using monochromatic $\text{Cu}\alpha$ radiation.

To study the physical and mechanical properties, samples and sections were prepared from the central part of the sample. To measure HRA, indentation was performed by indenting a diamond pyramid on a TM-12 hardness tester.

The stress intensity factor was chosen as a criterion for crack resistance, the limit value of which in a flat deformed state is denoted as K_{IC} [17, 18]. Its value determines the material's resistance to destruction [19, 20]. When determining the K_{IC} stress intensity factor, the test method was used for three-point bending of beam-shaped samples with a rectangular cross-section 3.5 mm wide, 5 mm thick, and 45 mm long with a thin side cut of 0.2 mm. During the test, the sample, mounted on two supports, was loaded with a force P using a stop (at the point of application of the force). The sample was loaded up to the critical load P_c , which corresponds to the start of the crack from the top of the notch. The critical values of the K_{IC} stress intensity factor were calculated according to the equation:

$$K_{IC} = 1.5 \cdot a^{1/2} \cdot \frac{Y(a/h) \cdot P_c \cdot L}{2 \cdot b \cdot h^2}, \quad (1)$$

where a is the crack length; h is the sample height; L is the distance between supports; $Y(a/h)$ is the function determined by the geometric parameters of the sample and its loading conditions.

3. Theoretical background

In some cases, due to uneven compaction in some parts of the sample, rapid heating can lead to the formation of large isolated pores. For this purpose, it is necessary to heat during hot pressing at a controlled rate, first at a rate of 50 °C/min to a temperature of 300 °C, then 250 °C/min to 900 °C, and then hold isothermally at a temperature of 900 °C for 2...3 min and raise the temperature at a rate of 500 °C/min to 1700 °C. In this case, residual CO gas manages to leave the sample, and in the future, an almost non-porous structure is formed.

The probability of pore formation is especially high in cases where the temperature rises at a rate of 500 °C/min. The increase in fracture toughness of materials obtained from WC nanopowders is caused, first of all, by highly dispersed grains and strong boundaries between them, which is caused by a short sintering time and the formation of contact necks between neighboring grains. X-ray phase analysis showed that, in addition to the WC phase, compounds W_2C , $W_6C_{2.54}$ are present. The X-ray pattern of WC samples is shown in Fig. 1.

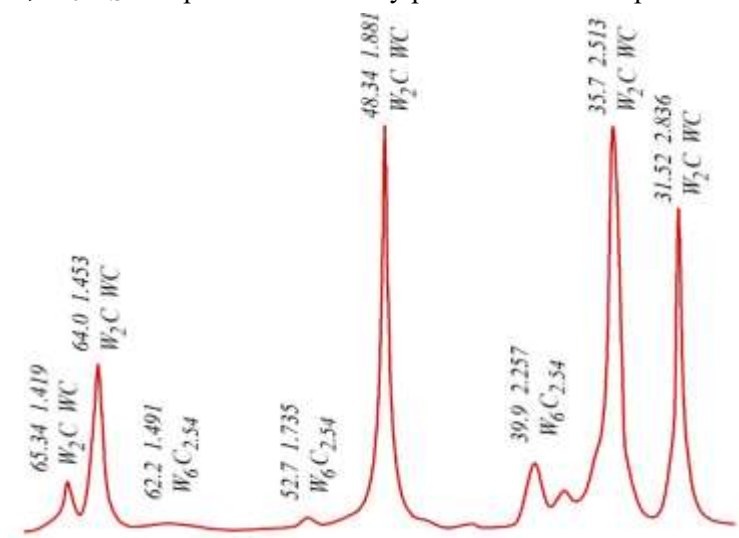


Figure 1. X-ray pattern of WC samples

Depending on the parameters of the sintering process (pressure, current, voltage, heating rate, heating time), the course of sintering of tungsten carbide nanopowder can occur in different ways. In this regard, the structure and properties of the material can vary widely. In conventional sintering, the main driving force of shrinkage is surface tension. In contrast to conventional sintering, during electroconsolidation, as a result of a non-uniform temperature field, there is a non-uniformity in the distribution of vacancies, which causes mass transfer; in addition, thermal diffusion makes a certain contribution to mass transfer [21, 22]. The contributions of surface tension and forces caused by electric heating to mass transfer and, therefore, to the change in pore size depend on the initial pore size. For vacancy diffusion and mass transfer, where the primary cause is surface tension, the picture is reversed.

From this can conclude that during electrosintering, the reduction of large pores proceeds at an accelerated rate. Obviously, it can be assumed that large pores do not grow at the expense of small ones, which usually happens with ordinary sintering. In this case, there is a reduction in size, and the number of small pores decreases. The total number of pores during electrosintering decreases to a lesser extent than during conventional sintering. The temperature of the powder is increased near the surface of the pore, so the temperature gradient during heating promotes the flow of vacancies in the pore, that is, the growth of the pore.

At the same time, the increased concentration of vacancies near the surface of the pore leads to the flow of vacancies from this surface, that is, to the reduction of the pore. It can be assumed that the pore further from the surface of the powder annihilates. This is probably

due to the fact that the equilibrium concentration of vacancies, which corresponds to high temperature, does not have time to be established, while on the surface of the pore such a concentration is established almost instantly.

4. Results and discussion

In a vacuum chamber (Fig. 2), tungsten monocarbide was sintered to a density of 99 % (Fig. 3). A nanodisperse powder obtained by a plasma chemical method was used as the starting powder.

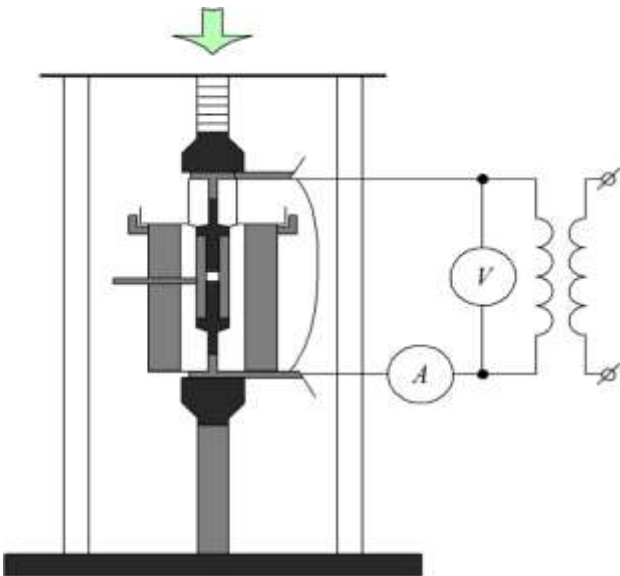


Figure 2. Vacuum chamber

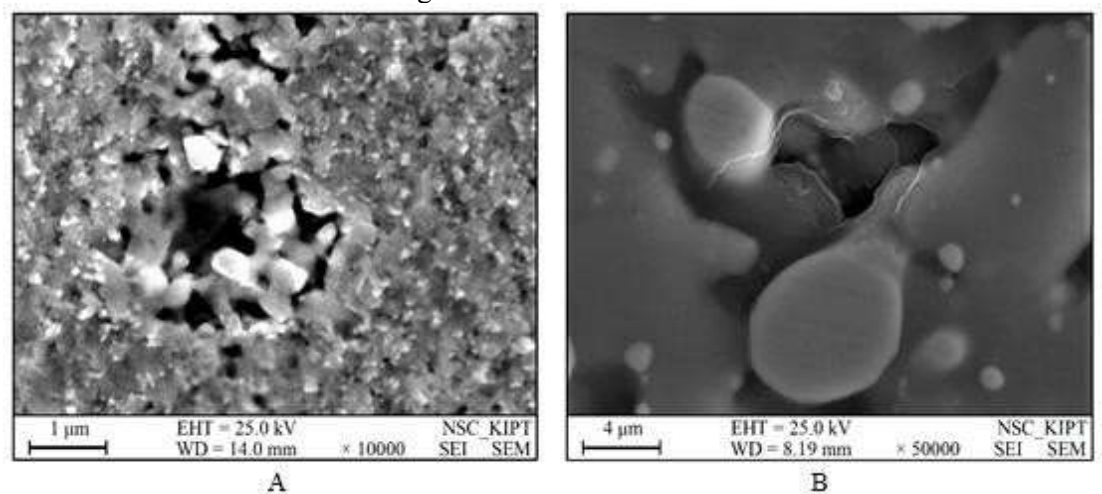


Figure 3. Fractogram of fracture (A) and microstructure (B) of sintered WC nanopowder by the electroconsolidation method at temperature $T = 1600\text{ }^{\circ}\text{C}$, pressure $P = 45\text{ MPa}$, holding time 2 min

These facts indicate that a tetragonal-monoclinic transformation takes place during loading. That is, the addition of 30 wt.% alumina contributes to the strengthening of the material based on zirconium oxide while restraining abnormal grain growth and forming a finer structure with a high content of the tetragonal phase, capable of transformation into a monoclinic phase (under the action of stresses) near the crack tip. Additives of 30 wt.% alumina contribute to the strengthening of the material based on zirconium oxide while restraining abnormal grain growth and the formation of a finer structure with a high content of the tetragonal phase, capable of transformation into a monoclinic phase (under the action of stresses) near the crack tip. The results of measuring the mechanical properties of some composites are shown in the table, which presents the results for hot pressing by electrosintering, when the maximum pressure on the graphite mold is 40 MPa, and the sintering time at the maximum temperature is 2 minutes.

It is known that the sintering process is accompanied by a number of phenomena determined with the help of various devices and tools [23, 24]. Observing these phenomena and their change when the technological parameters are changed allows to control the sintering process in the necessary direction in order to obtain materials with a given level of properties, which ensures obtaining a product of given shape and size. Refractory superhard substances, in particular, and tungsten carbide are hard and brittle substances [25, 26].

During sintering of the blanks, shrinkage can reach up to 60 %, depending on the initial density of the blanks. Shrinkage of workpieces is accompanied by a decrease in the number of defects in the crystal structure, particle surface roughness, porosity, as well as grain growth and homogenization of alloys. Only two phenomena are directly related to the sintering process: the growth of the contact area between the particles and the convergence of the particle centers – shrinkage, the other listed phenomena are concomitant. As know, sintering brings a system consisting of individual particles closer to thermodynamic equilibrium, that is, it is a process during which the excess energy of the system is reduced. In homogeneous pressing, one can only imagine the excessive energy of the free surface of the crystal lattice boundaries and defects. This energy is the main driving force of sintering. The magnitude of this driving force can be estimated based on the size of the particles from which the workpiece is formed. For example, the specific surface of carbide powders after grinding is usually 1...5 m²/g, powders obtained by plasma chemical synthesis – 10...40 m²/g.

Schematically, the phenomenon of pressure distribution can be depicted as shown in Fig. 4.

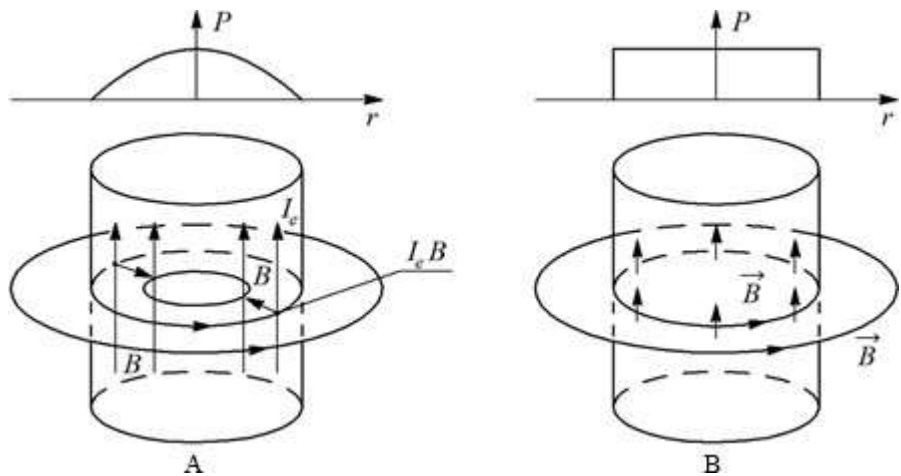


Figure 4. Pinch effect in sintering powder and pressure euphemism: (A) uniform distribution of current I in the conductive powder column $I_e = I/s$ is the constant; s is the cross-sectional area; the induction B has an azimuthal direction, the force $J_e \cdot B$ has a radial direction to the axis of the conductor; (B) conductor with surface current

With increasing power on the secondary winding of the transformer (Fig. 3), the mechanical properties of tungsten monocarbide increase, since the sintering temperature increases with increasing power.

The hardness and fracture toughness of samples obtained from monocarbide tungsten nanopowders is higher than that of samples from submicron powder (Table 1).

Table 1. Comparative properties of samples from submicron (0.5 μm) and nanopowders (40...70 nm) of WC, as well as the usual hard alloy BK8

WC samples	1	2	3	WC nano	BK8
Sintering temperature, $^{\circ}\text{C}$	1630	1750	1800	1740	1450
Holding time, min	1	20	20	1	60
Relative density, %	98.7	99.1	99.8	99.2	99
Average grain size, μm	0.5	2.1	5.5	0.1	3...4
Vickers hardness HV_{10} , GPa	24.3	20.3	18.4	26.4	16
Fracture toughness, $\text{MPa} \cdot \text{m}^{1/2}$	9.1	8.2	7.6	10.9	12

Additions of chromium and vanadium carbides lead to a slight increase in hardness and practically do not affect fracture toughness. The relative density also decreases, which, apparently, is associated with a decrease in the activity of the grain boundary sliding process during hot pressing [27, 28]. Even a small amount of carbide impurities of such refractory elements as chromium and vanadium leads to grain growth. This is obvious, and can be explained by the fact that the addition of these compounds activates diffusion processes during hot pressing, which ultimately promotes grain growth.

The high physical and mechanical properties of the obtained materials from tungsten monocarbide nanopowders compared to the known ones are due, first of all, to highly dispersed grains and strong boundaries between them, which is due to a short sintering time and the formation of contact necks between the grains. The method of hot pressing of nanopowders when heated by direct current passage accelerates the flow of vacancies on the

surface of the pores, and the rapid reduction of porosity at the grain boundaries increases the mobility of the boundaries and the compaction of the material due to stacking of the grains due to their sliding along the boundaries. The passing electric current contributes to the release of heat according to the Joule-Lenz law. Turning on the current (single or multiple) often has a positive effect on the sintering results. In addition to Joule heating, heat due to dielectric losses is released in the sintering samples. Conducted studies have shown that the passage of alternating current accelerates the cauterization of particles and contributes to a higher and more uniform density by volume than in the case of any other electrical influence. Due to the formation of the so-called skin effect during alternating current, the temperature of the cross-section of the sample is equalized. As a result, the cross-section of the sample is uniform.

It has been established that in the case of electroconsolidation, time dependences of mechanical and electrical influence and their interrelation are of great importance. The simplest example of non-constant mechanical impact is the increasing load, when the punch electrodes slowly move towards each other. At the same time, the final pressing pressure is applied at a temperature above 1400 °C. This electroconsolidation process includes two stages:

1) passing an electric current through a weakly pressed powder; at the same time, large contact resistance and relatively weak contact connections occur in the interparticle contacts at a mechanical pressure below 10 MPa, which causes a spatial discharge inside the powder filling during the passage of current. Normal current of industrial frequency is used for sintering;

2) the second stage proceeds with an increased current and under a higher pressure than the first. The temperature reaches its maximum value at the end of the high pressure period. After that, the current is turned off and the pressure is reduced during the cooling period.

In the first stage, the current density is several amperes per square centimeter; during the second, so-called consolidation stage, it reaches several hundred amperes per square centimeter. Powders are compacted according to equation (1). The main driving force of sintering is free energy. The magnitude of this driving force can be estimated based on the size of the particles from which the workpiece is formed.

In the heating process, physical contact is first formed between the particles, as in the case of traditional sintering, and then a branched system of boundaries is formed, i.e. the initially free surface energy, causing compaction of the system, is also spent on the formation of boundaries, the excess energy of which is the driving force of the sintering process. The formation of a branched system of boundaries occurs as a result of thermally activated sliding along grain boundaries in the initial period of sintering. As a result of rapid heating by the mechanism of thermally activated sliding along the boundaries of the workpiece, the surface of boundary separation is 1...2 orders of magnitude smaller than the surface of the powder, so the further compaction of pressing by 10...20 % proceeds very slowly by the mechanism of creep.

During electroconsolidation, at the moment of passing an electric current in the zones of interparticle contacts, a zone of increased temperature is formed, which causes the formation of a clean surface of the grains, which leads to the activation of the surface of the sintering powders. Convergence between the centers of particles, i.e. compaction of pressing occurs as a result of the action of external mechanical pressure, simultaneously with the action of an

electric current. In contrast to conventional sintering, during electroconsolidation due to a non-uniform temperature field, there is a non-uniformity in the distribution of vacancies, which causes mass transfer, in addition, thermal diffusion makes a certain contribution to mass transfer. The contributions of surface tension and forces caused by electric heating to mass transfer and, therefore, to the change in pore size depend on the size of the pores.

5. Conclusions

It was established that during electroconsolidation, large pores do not grow at the expense of small ones, as is usually observed during conventional sintering. In this case, there is a reduction in size, and the number of small pores slows down. The total number of pores during electrosintering decreases to a lesser extent than during conventional sintering.

On the basis of the conducted research, the optimal modes of hot pressing by direct current passage during sintering of tungsten monocarbide nanopowders were selected: heating at a rate of 50 °C/min to 300 °C, then 250 °C/min to 900 °C, then isothermal holding at 900 °C for 2...3 min. After that, it is necessary to raise the temperature at a rate of 500 °C/min to 1650 °C with a holding time of 1 min at this temperature.

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