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HOT PRESSING OF TUNGSTEN MONOCARBIDE NANOPOWDER MIXTURES BY ELECTROCONSOLIDATION METHOD

Key words: nanopowders, electroconsolidation, compaction, hot pressing, nanostructure.

Abstract: The paper presents theoretical and experimental results in the field of the manufacturing of cemented tungsten carbide materials. The important issue of avoiding any additional substances like plasticizers was challenged in order to reach the maximal possible density of sintered material while keeping its purity. To solve the problem, the electroconsolidation method of hot pressing supported by direct current was applied. The respective apparatus was constructed that enabled WC nanopowders to be sintered under pressure and high temperature during a very short time of ca. 3 minutes. In the experiments, because of the short heating time, grain size of the sintered bulk WC increased insignificantly, in general, remaining smaller than 1 μm. Similarly, sintering under hot pressing with direct current, a mixture of 3% by weight Y2O3 stabilized ZrO2 and 50% by weight WC, produced a fine structure with a uniformly distributed WC grains. The applied electric field led to the formation of a temperature gradient around the pores, with a favourable impact on the compaction of large pores and an increase in the final density of the bulk material. The experimental research confirmed that the main mechanism of the densification of nanodispersed powders of tungsten monocarbide was a locally inhomogeneous diffusion-viscous flow with intergranular slipping.

Spiekanie mieszanek nanoproszków węglików wolframu metodą elektrokonsolidacji

Słowa kluczowe: nanoproszki, elektrokonsolidacja, kompakcja, spiekanie, nanostruktura.

Streszczenie: W artykule przedstawiono rozważania teoretyczne i wyniki badań eksperymentalnych dotyczących wyrobów z węglików wolframu uzyskiwanych metodą spiekania. Podjęto próbę rozwiązania jednego z problemów, jakim jest obecność substancji uplastyczniających, która wspomagając proces spiekania jednocześnie utrudnia uzyskanie maksymalnej gęstości gotowego materiału. W celu rozwiązania tego zagadnienia zastosowano metodę eloktrokonsolidacji, polegającą na spiekaniu wspomaganym przepływem prądu elektrycznego. Skonstruowana aparatura umożliwia spiekanie proszków węglika wolframu w bardzo krótkim czasie rzędu 3 minut. W badaniach eksperymentalnych wykazano, że krótki czas oddziaływania wysokiej temperatury na wzrost ziaren w strukturze spieku jest nieznaczny i rozmiary ziaren pozostają na poziomie 1 μm. Podobnie mieszanka 3% masy Y2O3 stabilizowanego ZrO2 z 50% masy WC umożliwiła uzyskanie spieku o strukturze zawierającej równomiernie rozłożone ziarna węglika wolframu. Zastosowanie prądu elektrycznego powoduje wytworzenie gradientu temperatury wokół porów, korzystnie wpływając na proces kompakcji i zwiększając wynikową gęstość spieku. Wyniki eksperymentów potwierdziły główne założenia stosowane w opisie teoretycznym kompakcji nanodyspersyjnych proszków węglika wolframu.

Introduction

Nanostructure science and technology is a multidisciplinary field of research that includes bulk nanostructured materials [1]. Many materials, like polymers, by virtue of their structural features are always nanostructural systems [2]. The nanostructured ceramic materials are not a new development, because the very nature of the minerals taking part in the porcelain formation has resulted in features of the dimensions below 100 nm, which is considered “nanoscale” [3]. Recently, nanostructured materials found their

applications in many advanced technologies, such as thermomechanics, aerospace, nuclear power plants, and biomedicine [4]. In traditional engineering and manufacturing processes, nanostructure applications are especially found in the form of nanoceramic and nanocomposite coatings [5] or thin films [6].

Numerous researches have shown that nanostructured ceramics have unique properties and performance characteristics in comparison with coarse-grained analogues [7–10]. It has an increased crack resistance (toughness), strength, and toughness, and potentially allows the parameters of the "ceramic steel" [11] to be reached. Functional nanoceramics (piezoelectric, ferroelectric, dielectric, superconducting, etc.) significantly improve the electro-physical and magnetic properties.

In the cemented tungsten carbide materials, to obtain a unique combination of exceptional properties, a cobalt matrix is traditionally used as a bond, and a non-hazardous iron-based alloy binder phase was proposed [12]. Among manufacturing methods of nanostructured ceramics, spark plasma sintering (SPS) technology is reported to be very successful and promising [13–14]. As a modification, high pressure pulsed electric current activated equipment with a large volume chamber was proposed, reportedly capable of reaching high pressures up to 6 GPa and temperatures up to 1800°C [15]. Chuvildeev et al. published a paper stating that, when using the SPS method with direct current, they were able to obtain samples of high-density nanostructured tungsten carbide with high hardness and improved fracture toughness [16]. It was demonstrated that the increase in the sintering time up to 2 hours ensured the increased density of the nanostructured WC pieces, and a temperature increase from 1300° up to 1400°C increased their microhardness [17].

The challenging task of developing competitive manufacturing technologies for products for various purposes made out of nanostructured ceramics is very relevant. One of the propositions supported by the experimental researches is presented below.

1. Theoretical background

Technologically, the most important operation in the process of manufacturing products out of nanopowders is the formation of compacts of the required form and quality. Both single-phase nanopowders and multiphase compositions (often highly complex) have metastability of the structural-phase state, a developed unit surface, and, as a result, a high surface activity, and agglomeration ability. As a rule, they are characterized by poor mouldability and compressibility due to the specificity of their physical and chemical properties. In particular, high interparticle and near-wall friction caused by a high unit surface area, agglomeration, and a significant amount of absorbed impurities should be mentioned. Therefore, it is technologically difficult to ensure uniform density distribution in compacted nanopowders even in a simple form, and thus to preserve the nanostructure in the compacts in proper form during the sintering process. In other words, it is difficult to create conditions for inhibiting the growth of grains (preventing recrystallization) and for sintering high-quality nanoceramic products with specified functional parameters. It is also important to keep the chemical purity and the required phase composition of finished products.

In this respect, the issue of obtaining compacts with a uniform distribution of density in products of relatively complex shapes without the use of any plasticizers is relevant. The plasticizers should be avoided, because they are potential sources of impurities and additional porosity in the sintered samples. In this way, the internal stresses, delaminations, and cracks are minimized and, thus, the embryos of such macro-defects are eliminated during the sintering of compacts. One of these methods ensuring the production of products from nanopowders with a minimal and uniform distribution of defects is the method of hot pressing supported by direct current, which is called *electroconsolidation* [18]. In the present work, the process of hot pressing under direct current through a billet of nanopowders of tungsten monocarbide WC underwent investigation.

Materials based on tungsten carbide are most often used as wear-resistance for dragging and cutting tools. The composition of these materials includes a cobalt binder that binds the WC grains and allows the material to be compacted at relatively low temperatures. However, the cobalt bond reduces the hardness of the material, which is a parameter that largely determines the future wear resistance. It was demonstrated that the cutting inserts made out of pure WC have ca. 8–10 times higher wear resistance compared to WC-Co plates when cutting the steel X12M [9, 19]. Products made out of pure WC powders are often obtained by hot pressing and hot isostatic pressing [20]. The pressing temperature is usually 200–500°C above the sintering temperature of the WC-Co powder mixture, and as a result, materials with increased brittleness are obtained. To increase the toughness of the WC powder, some tungsten, carbon, carbides, and nitrides are added. The most effective methods for compacting hard-to-weld materials are methods of compacting with heating by electric current, such as SPS (Spark Plasma Sintering) and FAST (Field Activated Sintering Technique), which have been developed recently [21]. In these methods, a pulsating electric current is applied together with an external pressure (up to 100 MPa), which allows sintering at different heating rates. The sintering cycle is very short, usually 1–5 min, which ensures only a slight increase in grain growth. The explanation was proposed that,

during sintering, electric discharges could occur under electric current in the zones of interparticle contacts, which caused the formation of plasma, and lead to the cleaning and activation of the surface of the sintered powders [22]. Previous studies indicated that purification of the surface of the powder particles led to the formation of pure grain boundaries [23]. For example, when sintering Al powders, which have an inhomogeneous layer of Al_2O_3 on the surface about 5 nm thick, this layer was removed by an electric field, and the powder was compacted to full density [24].

The applied electric field leads to the formation of a temperature gradient around the pores with a favourable impact on the compaction of large pores. In the intermediate stages of sintering, a charge gradient is formed in the pore region of different sizes. The electrical resistance increases as the concentration of equipolar lines increases, as was illustrated by Raychenko [25] in Figure 1 (left). The corresponding increased heating in contact points between grains, caused by the electrical current, is illustrated in the Fig. 1 (right).

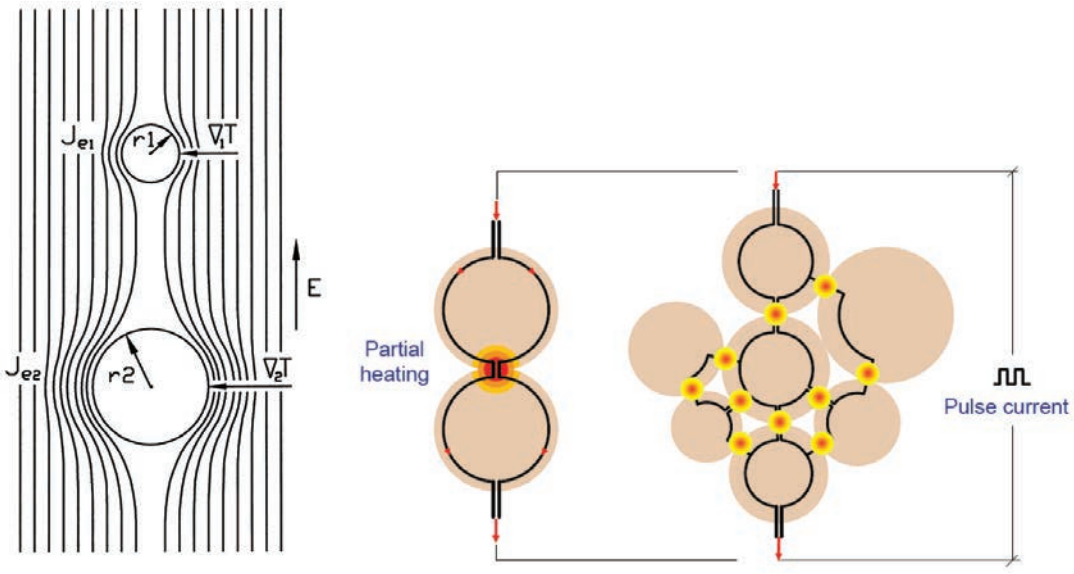

Fig. 1. Schemes of the electric field density in the temperature gradient in the work of large and small pores (left) [25] and partial heating in the contact zones between grains (right)

Source: left [25], right Authors.

The electric current density is higher in the region of large pores. This creates a temperature gradient, so the temperature is higher in the region of large pores. Raychenko obtained the following equation for calculating the temperature gradient ΔT near the pore [25]:

$$\Delta T \approx \frac{1}{R}\sqrt{\frac{\rho_0}{2C_M}\cdot\frac{T_0 E_0 \Delta\tau}{n}} \tag{1}$$

where R is the radius of the pore, ρ_0 is the electrical resistivity, C_M is the heat capacity, T_0 is the initial temperature, E_0 is the electric field strength, $\Delta\tau$ is the time of action of the electric field, and n is the number of electrical pulses.

The temperature gradient around the pores leads to the formation of the vacancy gradient ΔC_v. More vacancies are formed in the region of large pores [25]. The flow of vacancies J, can be represented in the following form:

$$J = D_v\left(\frac{k_T}{T}\Delta T - \Delta C_v\right) \tag{2}$$

where D_v is the diffusion coefficient of vacancies, and k_T is the coefficient of thermal diffusion.

According to Equation (2), vacancies diffuse from large pores to smaller ones, which leads to a reduction in the size of large pores. This process is the opposite of the process of vacancies diffusion under conditions of conventional furnace sintering, when the large pores grow due to small pores. In this case, the coalescence of the pores decelerates.

When hot pressing is performed under electric heating conditions (electroconsolidation), the compaction takes place during a short holding time, which prevents grain growth [26–27]. For example, SnO_2 powder was sintered to a density of 93% at 890°C for 10 minutes. At the same time, this powder

was compacted in ordinary furnace sintering at a temperature of 1000°C for 3 hours only to a density of 61% [27]. When electrosintering the TiN nanopowder, the total density was reached at a temperature of 1200°C and the obtained grain size was an order of magnitude lower than that obtained at traditional sintering at 1400°C [28]. The absence of a bundle during electrosintering made it possible to eliminate the process of cold pressing and stripping of the bundle. This stage took up to 30% of the cost of production by the powder metallurgy method [27]. Sintering of ceramics without additives by electric current made it possible to fabricate high-density ceramic, to increase significantly the wear resistance of the material, and to obtain high mechanical properties because of the direct binding of grains. High-density materials with a fine microstructure were obtained at relatively low temperatures and a short sintering time.

2. Experimental apparatus

To fabricate high-density products, a device for hot vacuum pressing with direct electric current was designed and manufactured. Figure 2 presents its overview (left) and scheme (right) with a description of the main units.

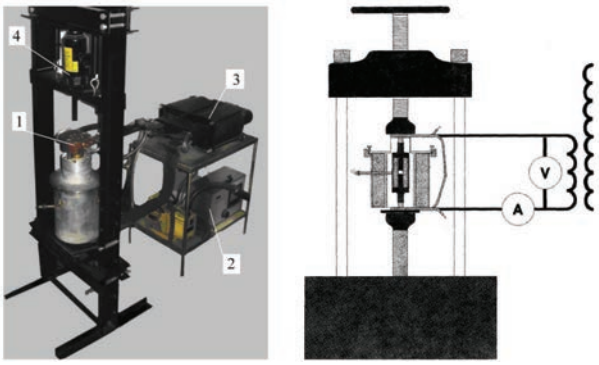

Fig. 2. Hot pressing machine: overall view (left) and scheme (right): 1 – hot vacuum pressing chamber, 2 – power supply unit, 3 – transformer TBK-75 with water-cooled current leads, 4 – hydraulic press HLR-12

Source: Authors.

With the developed apparatus, WC powder was sintered to a density of 99%. The obtained samples were of 20 mm diameter and 5 mm thickness. During the process, pressure was regulated manually and controlled with a special manometer. The initial powder was tungsten monocarbide obtained by the plasma-chemical method with the trade mark Tizit produced by the Austrian company Bergbau-und Hutten-GmbH Nfg as shown in Figure 3.

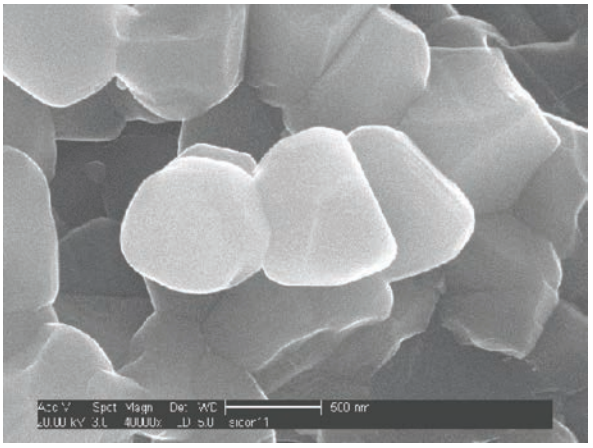

Fig. 3. Photomicrograph of pure WC initial powder

Source: Authors.

The specific surface area of the pure WC powder was 7 m^2/g, and the apparent density was 1.22 g/cm^3, with a particle size distribution of 60% – 40 nm, 30%–70.1% – 100 nm. According to the manufacturer of the powder, the abovementioned measurements were made with a CILAS HR850 granulometer. The content of tungsten monocarbide was 99.95%, and specific gravity was γ = 15.63 g/cm^3. Pre-tableted tungsten carbide powder was sintered to the limit for the graphite MPG-7 at a pressure of about 50 MPa and a temperature above 1200°C. Therefore, the maximum pressure in the mould reached ca. 45 MPa. The shrinkage cessation temperature was 1700°C. Figure 4 presents the thermogram of the sintering procedure.

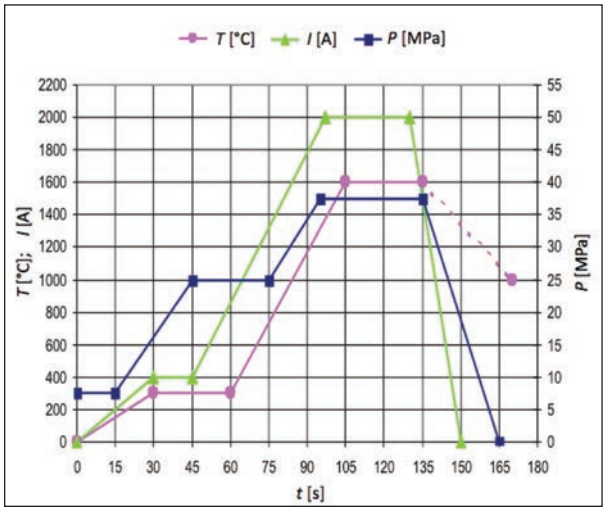

Fig. 4. Thermogram of sintering process under pressure and direct current

Source: Authors.

The density of the samples after grinding the surfaces were determined by hydrostatic weighing. The chips on their surface, like the powder form, were examined using a scanning electron microscope

JSM-840. The obtained material showed a high hardness HV = 24.3 GPa and K_{1c} = 6.1 MPa·m$^{1/2}$. The grain size in the obtained samples was between 250 and 420 nm.

3. Results and discussion

Measurements of ρ density, hardness of HRA, tensile strength at bending (σ_{bd}), and grain size $d_{av.}$ of the samples obtained for different *P* and *T* were reported previously [24]. Table 1 presents the results for three samples sintered at *T* = 1700°C and *P* = 40 MPa during different time spans *t*. The dependence of grain size on the processing time is clear and unequivocal: When *t* was increased from 5 up to 10 min, grain sizes grew three times larger. Simultaneously, hardness HV and fracture toughness K_{1c} decreased significantly, even below the values obtained after 2 minutes of a sintering process.

Table 1. Some physical and mechanical characteristics of the samples produced at T = 1700°C and P = 40 MPa

t [min]	ρ [g/cm^3]	*HV* [GPa]	K_{1c} [MPa·m$^{1/2}$]	d_{av} [nm]
2	96.2	23.0	5.5	350
5	99.0	24.3	6.1	400
10	99.0	21.0	4.5	1,200

The data presented in [24] and in the Table 1, as well as the structure observed with the electron microscope led to the conclusion that the process of high-temperature sintering under pressure should last 3–5 minutes. Under these conditions, the sintered WC grain size increased insignificantly, in general, remaining smaller than 1 μm, and the obtained material performed favourable characteristics. There is practically no porosity in the sintered material, as can be seen in the Figure 5.

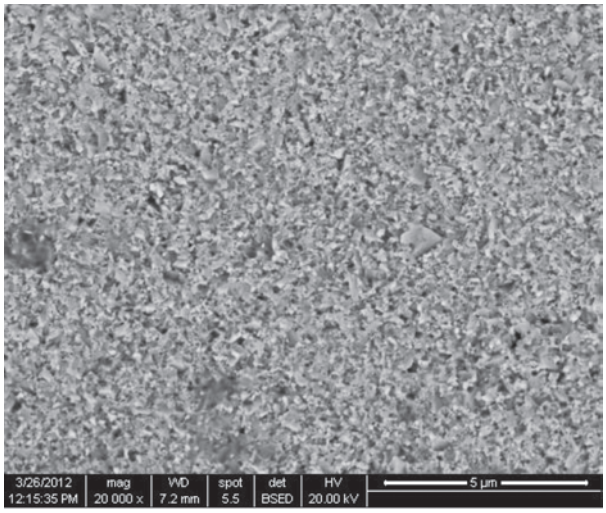

Fig. 5. Photomicrograph of pure WC structure sintered at *T* = 1700°C under pressure *P* = 45 MPa

Source: Authors.

That is why high values of σ_{bd} = 720 MPa were obtained. When WC the initial powders had a grain size between 2 μm and 5 μm, hot pressing at *T* = 2500°C, during *t* = 10 min, under pressure *P* = 12 MPa, it produced a much smaller σ_{bd} value between 350 and 520 MPa. The examined nanostructured samples also exhibited a very high value of fracture toughness K_{1c} = 6.1 MPa·m$^{1/2}$ and a high HRA value, which is important for the ceramics applied in cutting tools.

The increase in the fracture toughness of the obtained materials made out of WC nanopowders in comparison with other reported values can be ascribed, first of all, to highly disperse grains and strong boundaries between them. The latter is predetermined by the short heat exposure time and not by the high sintering temperature. The method of hot pressing of nanopowders heated by direct current transmission accelerates the flow of vacancies on the pore surface [27]. However, a rapid decrease in porosity at the grain boundaries increases the mobility of the boundaries; therefore, the compaction due to grain deposition is a result of their sliding on the boundaries.

Interesting results are obtained by sintering nanopowder mixtures of tungsten monocarbide and partially stabilized yttria with zirconia. Some investigations were reported on the influence of WC particles on the microstructural and mechanical properties of 3mol% Y_2O_3 stabilized ZrO_2 matrix composites produced by hot pressing, where hard WC particles were added with various proportions up to 40 vol% [29]. In our research, during sintering under hot pressing with directly applied current, ZrO_2 stabilized with the addition of 3 wt% Y_2O_3, which was mixed with WC powder in proportion of 50% by weight, forming a fine structure with uniformly distributed WC grains. Figure 6 presents the initial mixture of nanopowders, and Figure 7 shows the final nanostructured material obtained by hot pressing electroconsolidation at *T* = 1450°C and a pressure of 30 MPa, during the sintering time *t* = 3 min.

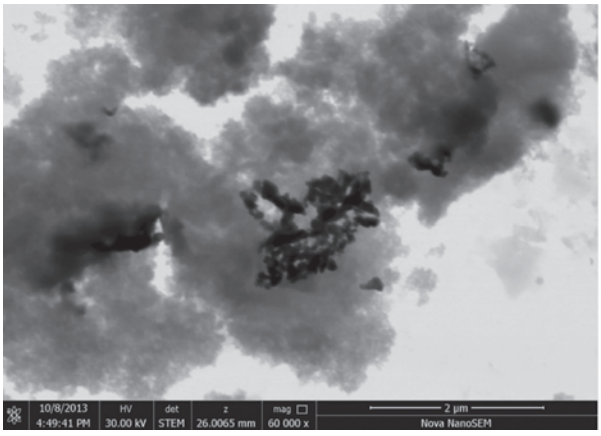

Fig. 6. The initial nanopowder mixture of 50 wt% WC and ZrO_2 stabilized with 3 wt% Y_2O_3

Source: Authors.

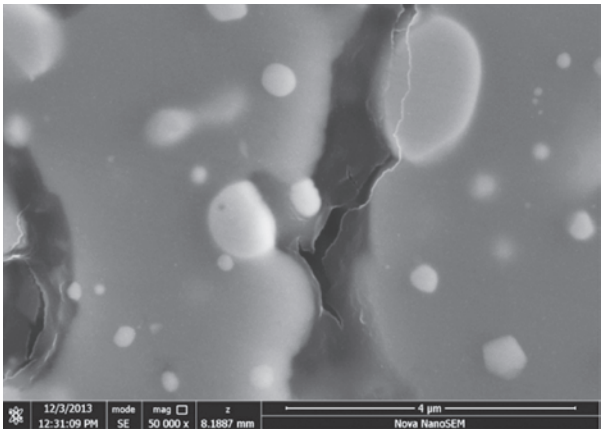

Fig. 7. The sintered nanostructure, obtained by hot pressing electroconsolidation at T = 1450˚C and P = 30 MPa, sintering time 3 min

Source: Authors.

From the practical perspective, it is highly desirable to obtain better strength and toughness of the sintered material with less energy consumption. In the case of cutting tools inserts, their material and structure have an impact both on chip formation mechanisms [30] and on surface hardness [31]. The presented results demonstrated that the electroconsolidation method provided a nanostructural tungsten monocarbide compound of high properties with minimized energy losses. It can be assumed that the rapid decrease in porosity at the grain boundaries increases the mobility of the boundaries and improves the compaction of grains as a result of their sliding along the boundaries. In the initial stage of heating process, physical contact is formed between the particles and a branched boundary system, i.e. the free surface energy. That causes the system to become denser and to expand with the formation of boundaries, which subsequently releases excessive energy that plays the role of the driving force of the sintering process.

Conclusions

The presented researches on hot pressing electroconsolidation revealed improved physical and mechanical properties of the materials obtained from nanopowders of tungsten monocarbide WC in comparison with other reported ones. The following can be pointed out:

– The obtained properties are conditioned, first of all, by highly dispersed grains and strong boundaries between them, which is predetermined by short time and low sintering temperature.
– The method of hot pressing of nanopowders supported by direct current heating accelerates the flow of vacancies on the pore surface that resulted with higher density.
– As a consequence, better mechanical properties were obtained at lower energy consumption.

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