# ACTIVATED HOT PRESSING BEHAVIOR OF WC NANOPOWDERS

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***Abstract***: *The questions of consolidation of nanopowders concerning hot compaction by pressing activated by electric current action are considered. Mechanisms of grain boundary creep-sliding which are sequentially prevalent in a forming of compacted structures under influence of temperature factor and in the presence of a direct electric heating are discussed. Structural-transformational sources and conditions of forming of high physical-mechanical properties of nanopowder refractory solid-state products are described.*

**Keywords:** growth, graphite, powder.

## 1. Introduction

The urgency of a consolidation problem of nanopowders refractory materials of a tool assignment is connected to formation of the structure determining physicomechanical properties of solid-state result of sintering.

In research [1] we considered questions of sintering of submicronic powders $Al_2O_3$ and WC activated by an electric field (FAST technology developed at the Californian university USA by J. Groza, et al. [2-4]). At the same time, in our opinion, from all methods of compacting nanopowder materials for refractory powders the method of hot pressing with current straight transmission is the most effective. This point of view is explained with a number of factors: an opportunity of fast heating of the graphite form up to high temperature; relative simplicity of installation and an opportunity of use of a usual alternating current of industrial frequency that excludes use of expensive pulse generators; an opportunity of reception of preparations concerning sizable; fast heating allows to limit intensive growth nanograins an initial powder, leaving their sizes in nanolimits.

If process of an electric current influence on sintering of metal powders is investigated enough [5], process of hot pressing with application of an electric current at sintering nanopowders, despite of numerous foreign publications in the field of sintering these powders, is investigated a little, is especial regarding mechanisms of refractory powders sintering connections without which it is practically impossible to present the modern tool industry. Processes of sintering of usual powders of refractory connections are in detail considered in work [6]. Authors [4, 7] investigate also the certain regularities of sintering various nonmetallic nanopowders, including refractory connections.

In given article some results of research of sintering mechanisms regularities mainly monocarbide nanopowders tungsten are submitted during hot pressing in graphite forms at transmission through a powder of an electric current (direct electroheating) with use in researches approved [1] foreign WC powders (Aldrich Co, Austria).

Carried out researches have shown, that from monocarbide nanopowders tungsten (40-70 nanometers) it is possible to receive practically a pore-free material with rather high physicomechanical properties [8]. It is obvious, that on contact sites between the next particles under influence of the big electric current should occur intensive mass transfer. In result in powder pressings there is a process of fast sintering. Depending on parameters of process (pressure, force of a current, a pressure, speed of heating, time of heating) the course of sintering can occur differently. In this connection over a wide range can change structure and properties of a material.

## 2. Experimental conditions

Preliminary preforming carbide powder tungsten was exposed to sintering in installation with the special vacuum chamber. The maximal pressure for used graphite МПГ-7 about 20 кN at temperature is higher 1200ºC, therefore for a compression mould d=20 mm created the maximal effort no more than 15 KN. Density of samples after grinding a surface determined by hydrostatic weighing, and chips on their surfaces, as well as a kind of a powder, by raster electronic microscope JSM-840.

## 3. Investigation

The temperature of the termination shrinkage WC product has made 1650ºC. The received material has shown high hardness and density. Some properties of the received samples at various effective modes of hot pressing are resulted in tab. 1. As have shown experiments, optimum modes are in rather narrow limits, in particular the temperature factor most determining from them 1700-1800 ºC. Pressure of hot pressing is limited by durability of a graphite compression mould at the given temperatures, and optimum speed of rise temperature is within the limits of 400-500 ºC/min though, with the purpose of the warning of breakage of the form, up to 900ºC the temperature rose more smoothly, with a speed of 50-250ºC/min.

Table 1 - Some physicomechanical properties received WC samples

| P, MPa | T, °C | $\frac{r_{fact}}{r_{theory}}$, % | HRA | $\sigma_{flex}$, MPa | $d_{mean}$, μm |
|---|---|---|---|---|---|
| 40,0 | 1700 | 96,2 | 93 | 530 | 0,350 |
| 50,0 | 1730 | 99,0 | 95 | 720 | 0,420 |
| 50,0 | 1800 | 99,2 | 95 | 670 | 0,750 |

Process of sintering is accompanied by a number phenomena which were fixed by us with the help of various devices and tools. Supervision over these phenomena and over their change under change of technological parameters allows to operate process of sintering in a required direction with the purpose of reception of materials with the set level of the properties providing reception of a product set form and the sizes.

The refractory body of tungsten monocarbide combines high hardness and the raised fragility. Shrinkage of preparations, as a rule, it is accompanied with reduction of quantity of defects of crystal structure, a roughness of a particles surface, porosity, and also growth of grains and homogenization. Directly it is possible to relate to the process of sintering only two phenomena: growth of the area of contact between particles and rapproachement of the centers of particles, i.e. shrinkage actually, other listed phenomena are accompanying.

## 4. Analysis and generalization of results

As is known, sintering approaches the system consisting of separate particles, to thermodynamic balance, i.e. it is process during which superfluous energy of system decreases. It is possible to assume, what exactly this energy of a free surface, surfaces of borders and defects of a crystal lattice is the main driving force of sintering.

The size of this driving force can be estimated, taking into account the sizes of particles from which preparation is formed. For example, the specific surface of carbide powders after milling usually makes 1-5 $m^2/g$, the powders received by plasmo-chemical synthesis, - 10-40 $m^2/g$ [6]. If to accept, that as a result of any formation method preparations by porosity of 40 % it is obvious turn out, that superfluous energy of such system is great enough. It also performs the basic work on condensation, causing current of substance in emptiness (pores). With some approximation, following authors [6], we can admit, that current of substance to a surface with negative curvature is carried out under some effective pressure P caused superficial tension:

$$P=2\gamma /r, \qquad (1)$$

where $\gamma$ - free superficial energy, kJ/$\mu m^2$; $r$ - the size of a grain, $\mu$m.

This pressure is great enough for pressing from nanodispersed particles where the size of pores is comparable with the size of particles. The equivalent pressure working at least in an initial stage of sintering, for nanodispersed powders is very great and, naturally, causes fast condensation $\acute{\varepsilon}$.

In general temperature $\acute{\varepsilon}$-$T$ dependences

$$\acute{\varepsilon}=A\,P^{V} exp(-E_{an}/RT), \qquad (2)$$

where A, $R$, v - constants ($R$ – a gas constant); $E_{an}$-energy of activation, kJ, in an initial stage of sintering on the mechanism of creep $P=P_{creep}$.

The following driving force of sintering is energy of a crystal lattice imperfections. It serves as the reason of the accelerated exchange of places:

$$n_a = n_o exp(-E_{аан}/RT), \qquad (3)$$

where $n_a$ - number of atoms which have left the place in a lattice and have occupied vacancy unit; $n_0$ - the structural factor, not dependent on temperature; $E_{аан}$ - energy of activation annihilator defects, kJ. This size in the broken lattice strongly goes down. Therefore for active compaction of a material it is necessary to break structure as it is possible strongly, crushing substance, as that occurs in nanopowders received in the plasmo-chemical way. During sintering the quantity of imperfections of a crystal lattice considerably decreases. So, for example, in monocarbide tungsten with a specific surface up to 1$m^2/g$ the size of coherent areas makes 20000-50000 nanometers, and after sintering at temperature 0,8 *[T]* (*[T]* – melting temperature) makes already 200000-500000 nanometers.

Besides it has been noticed, that at sintering at the moment of an electric current passage of enough the big size (up to 5000-8000 A) in zones of interpartial contacts arise electric discharges which according to physics of this process should cause formation of plasma, that naturally results in clearing and activization of a surface of sintered powders. Clearing of a surface of particles of a powder in turn results in formation of pure borders of grains and activation of the sintering process.

## 5. Conclusion

Thus, the lead researches allow to assume, that rather high physicomechanical properties of the received samples from monocarbide nanopowders tungsten are caused, first of all, by superfine grains and strong borders between them, that is provided with high speed of rise of temperature up to 1700ºC during hot pressing in graphite forms.

The given researches allow to assume, that the method of hot pressing nanopowders at heating to straight transmission an alternating current accelerates a stream of vacancies on a interstice surface, and fast reduction of porosity on borders of grains conducts to thermally activated sliding nanograins from each other, that finally, together with imposing of a variable electric field, provides the best stacking grains, and, hence, provides their faster compaction. At the same time, supervision for shrinkage of nanopowder samples at hot pressing with direct transmission a current at temperatures up to 900-1000ºC allow to assume, that the initial mechanism of consolidation is the mechanism of creep.

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