Optimization of VK10-HOM cemented carbide mixture pressing modes

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The article presents the results of a study on pressing and sintering of cemented carbide plates in order to increase the durability of metal-cutting tools. The substantiation and optimization of the cemented carbide mixture pressing conditions is shown. Various plasticizers were used in the preparation of the mixture. Pressing modes and results on pressing force and elastic aftereffect coefficient are given. After sintering the samples, the microstructure, porosity, density, and hardness are determined. Microstructures are shown with the use of plasticizers SKD II (BR-1203) and PEG-1500.
According to the granulometric composition, the initial mixture had an average particle size of 0.7601 microns. The pressing was carried out with preliminary vibration-mechanical treatment of a mold filled with a cemented carbide mixture with a plasticizer. The dependences of the density and the coefficient of elastic aftereffect on the pressing pressure are obtained. The variation ranges of pressing speed and pressure were as follows: pressing speed — from 3 to 15 kN/s, pressing pressure — from 100 to 900 MPa. Providing pressing speeds in the required pressure range was carried out on presses with loads up to 100 kN and up to 1250 kN. It has been established that polyethylene glycol-based plasticizers are preferable in terms of elastic aftereffect, density and porosity. When optimizing the parameters of pressing the VK10-HOM mixture in the selected intervals of varying factors, a plan for a complete two-factor three-level experiment for each type of plasticizer was drawn up. The density of compressed samples and the coefficient of elastic aftereffect were chosen as the optimization criteria. In the joint analysis of experimental response surfaces, a compromise task to determine the optimal parameters of the pressing process was solved for optimization criteria. The microstructure of plates obtained under the optimal pressing mode, using PEG-1500 plasticizer at sintering temperature of 1390 °C, shows low porosity, absence of undesirable η-phase at higher values of density and hardness.
Key words: cemented carbide, cutting tool, pressing, density, elastic aftereffect, plasticizer, technology, optimization, sintering, microstructure, pores.

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Introduction

Manufacturing of high-performance carbide cutting tools involves a technological sequence of about 30 different operations that affect the quality of the resulting tool, especially its durability [1–6].

The works of many authors are devoted to the problems of powder materials pressing, including metal powders [7–9]. Justification of optimal parameters and modes of carbide powder pressing process is relevant [10–12], as it affects the homogeneity of compaction on the body of cutting tools taking into account its heterogeneous cross-sections, which leads to various physical and mechanical phenomena during further sintering, for example, insufficient pressing forces lead to the appearance of pores and destruction of the carbide "skeleton".

When solving the problem of pressing, it is important to emphasize the technological capabilities of the press equipment, taking into account the tasks of controlling the speed and movement of the puncheon, control of pressing forces, depending on the composition of the powder, type of plasticizer, shape and design of the carbide cutting plate.

The density of the moulded piece depends on the pressure applied to deform the mixture [7, 13–19]. When determining the maximum possible pressing forces, it is necessary to take into account the possibility of layer cracks due to the creation of such a volume-stress state in the moulded piece, when the elastic aftereffect increases in proportion to the applied pressure, and the strength of the workpiece increases more slowly. In order to weaken the delamination effect, pressure holding is used, which leads to relaxation of internal stresses. As the cobalt content of the carbide mixture increases, the elastic expansion increases [13].

Insufficient pressing force leads to pores and uneven density in the moulded piece volume. An increase in porosity by 0.1 % reduces the bending strength by 15–20% and the cutting strength coefficient by 20–25%. In some sources, however, it is noted that porosity up to 7% does not affect the physical and mechanical properties of sintered carbide (perhaps this refers to hardness).

Investigations of the influence pressing speed of mixtures with different chemical and granulometric composition with different plasticizers are carried out [20]. The results show that the friction characteristics of carbide powders decrease at higher pressing speeds. It is also found that as the normal pressure increases, the coefficient of friction between the carbide mixture and the mold matrix decreases. The change in the friction coefficient leads to a change in the density gradient, which in turn affects the geometry of the sample after sintering [20, 21]. The dwell time after compaction before unloading is not more than 1–3 s. Stress relaxation occurs during the first 0.5 s.

Nowadays, various mathematical modeling tools are often used to predict the quality of moulded pieces [22, 23]. Physical experimentation is still the most reliable way to solve research and production problems.

The aim of the research is to study the regularities of the pressing process of carbide-alloy mixtures taking into account the peculiarities of shape, chemical and granulometric composition of the moulded piece, type of plasticizer.

Research methodology

The quality of moulded pieces and, ultimately, of sintered carbide plates is influenced both directly by pressing modes and by powder properties and conditions of its preparation at the stages preceding the pressing process [12, 16, 17, 24–28].

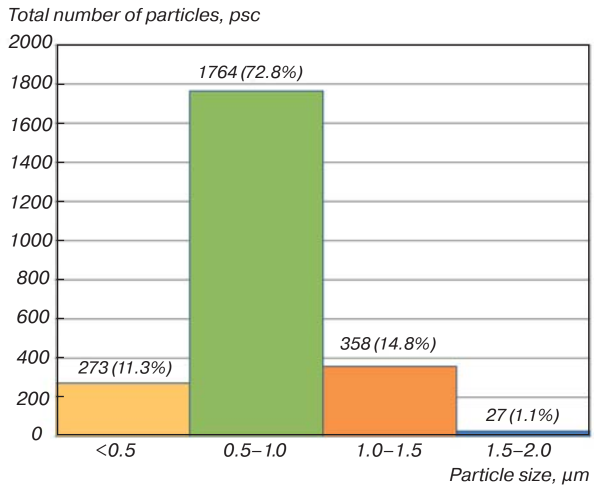


Fig. 1. Histogram of particle size distribution of initial powder VK10-HOM (based on microscopic analysis)

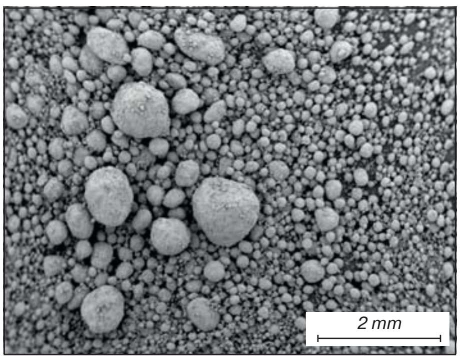


Fig. 2. Natural granulation of initial powder VK10-HOM

VK10-HOM carbide mixture was used for the research tasks.

The geometry and morphology of the powder determine such technological parameters as powder flow rate, bulk density, compressibility (compactibility).

Powder flow rate was determined according to GOST 20899–98 (ISO 4490–78) [29], bulk density was determined according to GOST 19440–94 [30].

Microscopic analysis on optical (Olympus DSX1000) and electron (JEOL JCM-6000) microscopes was used to study the shape, parameters and surface condition of powder particles. The particle size distribution of the powder was determined by microscopic methods [31], as well as using a particle size and shape analyzer Bettersizer S3 Plus, the principle of operation of which is based on laser diffraction combined with image analysis. The particle size distribution of the initial mixture is shown in **Fig. 1** (average particle size 0.7601 µm).

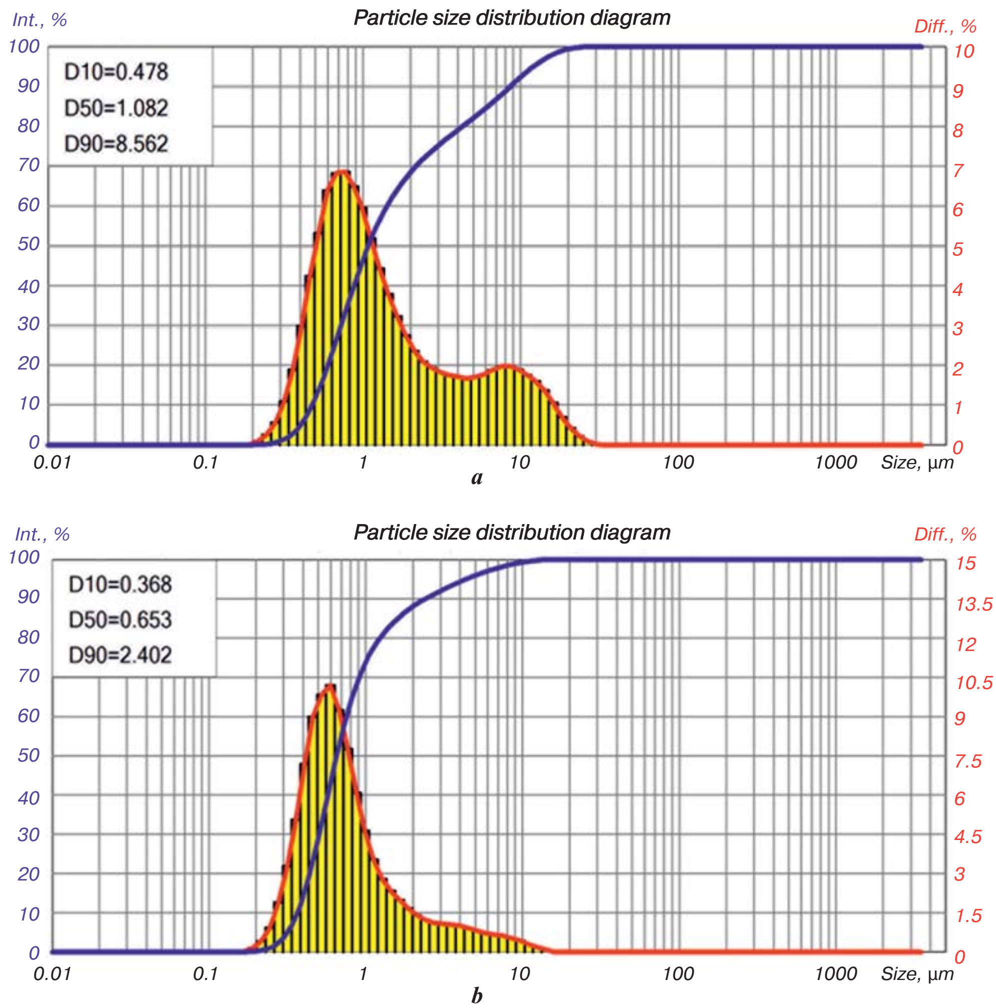
To reduce the influence of the VK10-HOM powder property to agglomerate in air (**Fig. 2**), a technology of disintegration of powder agglomerates was developed using ultrasonic and vibration-mechanical impact. As a result, the bulk density of the dried powder was 4.05 g/cm^3 and that of the undried powder was 3.62 g/cm^3.

The criterion for selecting a plasticizer for use in a carbide mixture is a number of requirements: adhesive activity to components (WC, Co, C, Cr_3C_2); high tribotechnical properties that reduce friction of WC particles; passivation of excess surface energy of finely dispersed tungsten carbides; inertness to mixture components; absence of cracking and pore formation during sintering; reduction of cohesion and agglomeration processes that deteriorate the homogeneity of moulded pieces; absence of components in the composition that change the carbon balance; complete dissociation and degassing; ability to restore surface oxides; reduction of oxygen access to the surface of carbide and cobalt; environmental friendliness; technical availability; and cost. [5, 12, 32].

The study of the influence of plasticizers on the properties of carbide press powders and sintered products is considered in the works of [33–36]. The rubber used is mainly synthetic rubber, which is introduced into the mixture as part of a solution in gasoline (or petroleum solvent (nefras)) [13]. The most cited plasticizing additive in scientific publications is ethylene glycol.

For the purposes of optimizing the pressing conditions of the VK10-HOM carbide mixture, the following types of plasticizer were used: synthetic cis-butadiene rubber (SCD II) [37] in nefras, ethylene glycol (EG-120) and polyethylene glycol (PEG-1500 and PEG-8000) in ethyl alcohol, as well as liquid paraffin.

Significant granulometric heterogeneity of the supplied carbide mixture dictates the inclusion in the technological process of grinding operation, homogenizing the composition of the mixture. Preparation of granulated mixture for subsequent pressing was carried out according to the following scheme: drying of the mixture at a temperature



Diam., µm	%
0.000 - 0.050	0.00
0.050 - 0.100	0.00
0.100 - 0.200	0.01
0.200 - 0.500	11.69
0.500 - 1.000	34.74
1.000 - 2.000	22.04
2.000 - 5.000	13.67
5.000 - 10.00	10.23
10.00 - 20.00	6.98
20.00 - 45.00	0.64

Diam., µm	%
0.000 - 0.020	0.00
0.020 - 0.050	0.00
0.050 - 0.100	0.00
0.100 - 0.200	0.13
0.200 - 0.500	28.84
0.500 - 1.000	44.74
1.000 - 2.000	14.35
2.000 - 5.000	7.89
5.000 - 10.00	3.41
10.00 - 20.00	0.64

Fig. 3. Particle size distribution of VK10-HOM carbide mixture with 3% cis-butadiene rubber SCD II (*a*) and 3% polyethylene glycol PEG-1500 (*b*)

of 120 °C in a drying cabinet in vacuum or inert gas atmosphere (argon) for 1 hour; grinding for 6 hours and mixing the powder with plasticizer for 2 hours in a laboratory ball mill; drying of the granulated mixture at a temperature of 120 °C in a drying cabinet in vacuum or argon atmosphere for 2 hours; grinding of the dried mixture; separation on a vibrating screen with a mesh of 100 µm.

Fig. 3 shows the particle size distribution of the obtained carbide mixture VK10-HOM in 3% solution of butadiene rubber (SCD II) and PEG-1500 (according to the results of analysis on the Bettersizer S3 Plus).

Compressibility of powder prepared for pressing was evaluated by determining the density of moulded pieces made at given pressures of double-action pressing in cylindrical or rectangular molds [38, 39]. To reduce the influence of edge effects during pressing, the working surfaces of the mold were lubricated with a 10% solution of lead (zinc) stearate, as proposed in the [38].

The elastic aftereffect coefficient was determined as the difference between the diameters of the press in the free state d_1 the mold hole d_0, related to the value of d_1.

Providing pressing speeds in the required pressure range was carried out on presses with loads up to 100 kN and up to 1250 kN. Pressing was carried out with preliminary vibration-mechanical treatment of the powder at a frequency of 100 Hz for 3 seconds.

To optimize the parameters of pressing of carbide mixture with different plasticizers the optimal planning of extreme multifactor experiment was carried out in accordance with the recommendations of [40].

Research results and discussion

As a result of the experiments, the compaction curves and the dependences of the elastic aftereffect coefficient on the pressing pressure process were obtained.

The use of 3% solution of SCD II cis-butadiene rubber as plasticizer allows increasing the maximum pressing pressure up to 380 MPa, which can be seen from **Fig. 4, *a***. At all pressure values, no delamination of the molds was observed, but the extrusion force and elastic aftereffect coefficient increased (**Fig. 4, *b***).

Analysis of the dependences presented in **Fig. 4**, and similar results obtained when using paraffin, demonstrates a greater influence of plasticizer SCD II on the increase of the elastic aftereffect coefficient. Consequently, paraffin has better rheological characteristics than SCD II rubber.

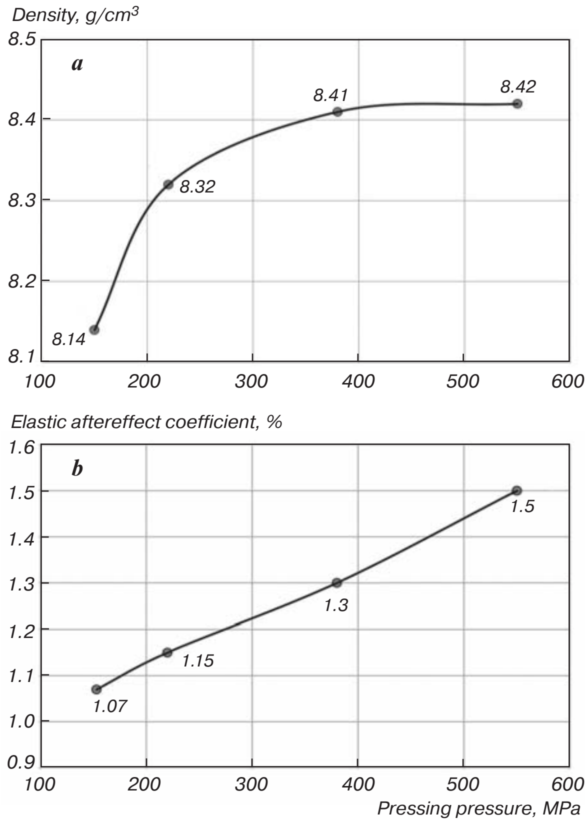


Fig. 4. Determination of pressability (*a*) and evaluation of elastic components (*b*) of carbide mixture VK10-HOM with plasticizer SKD II (sample – ∅ 9 mm moulded piece)

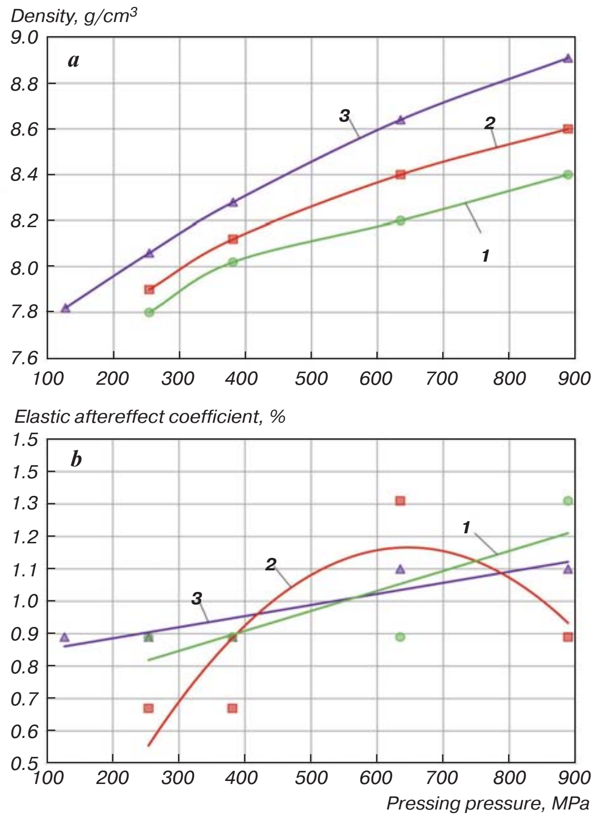


Fig. 5. Determination of pressability (*a*) and evaluation of elastic components (*b*) of carbide mixture VK10-HOM with plasticizer: *1* – 2% EG-120; *2* – 2% PEG-8000; *3* – 3% PEG-1500 (sample – ∅ 9 mm moulded piece)

When powder is pressed with plasticizers EG-120, PEG-8000 and PEG-1500 at pressures indicated in **Fig. 5, *a***, a monotonic growth of moulded piece density is observed. Plasticizer PEG-1500, previously not used in practice as a plasticizer, showed the best compressibility in the whole range of pressures (**Fig. 5, *a***, curve 3).

Polyethylene glycol-based plasticizers are preferred because of the lowest elastic aftereffect coefficient (**Fig. 5, *b***) over the entire range of pressing pressures compared to paraffin and rubber.

Analysis of the ranges of pressing speeds and pressures according to the literature data allowed us to limit the range of research: pressing speed — from 3 to 15 kN/s, pressing pressure — from 100 to 300 MPa [4, 6–8, 13–15, 17, 34, 35]. However, when pressing samples of 6 × × 6 × 40 mm in size with paraffin, their delamination was observed, which allowed us to conclude that the study of liquid paraffin as a plasticizer is futile. At 250–300 MPa, delamination was observed on samples with SCD II and PEG-1500.

Based on the data obtained, the range of studies on pressing pressure was limited to values from 100 to 200 MPa (**Fig. 6**).

To optimize the parameters of pressing of VK10-HOM mixture in the selected intervals of variation of factors the plan of the full two-factor three-level experiment is developed.

Factors, levels and intervals of their variation are given in **Table 1**. The density of compressed samples (g/cm^3), and the elastic aftereffect coefficient (%), for each plasticizer were chosen as optimization criteria. The experimental results are presented in **Table 2** (experiments were performed in triplicate).

As a result of statistical processing of the obtained experimental data in the "Statistica" statistical analysis package the regression models of the second order were obtained and presented in the coded form.

To determine the density of compressed samples using plasticizer SCD II, the regression equation, taking into

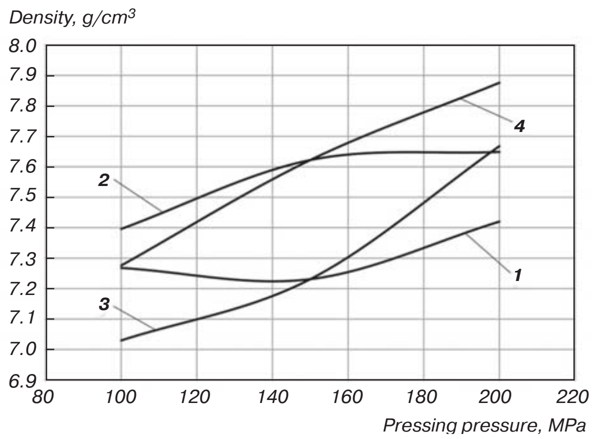


Fig. 6. Determination of pressability of VK10-HOM carbide mixture (sample – 6 × 6 × 40 mm): *1* – 3 kN/s, SCD II; *2* – 3 kN/s, PEG-1500; *3* – 9 kN/s, SCD II; *4* – 9 kN/s, PEG-1500

Table 1
Factors, levels and intervals of their variati

Factor's coded designation	Factor	Factor level			Variation interval
		lower ($x_i = -1$)	base ($x_i = 0$)	upper ($x_i = +1$)	
x_1	Specific pressing force (pressing pressure) P_{sp}, MPa	100	150	200	50
x_2	Pressing speed V_{pr}, kN/s	3	9	15	6

Table 2
Planning matrix of the full two-factor three-level experiment and experimental values of optimization criteria at the plan points

No. of experiment	x_1	x_2	Plasticizer type			
			SCD II		PEG-1500	
			ρ, g/cm^3	K, $\times 10^{-1}$%	ρ, g/cm^3	K, $\times 10^{-1}$%
1	–1	–1	7.268	6.0	7.396	6.7
2	–1	0	7.030	7.3	7.276	7.3
3	–1	1	6.743	9.0	8.200	8.3
4	0	–1	8.320	11.0	7.820	8.9
5	0	0	7.231	6.5	7.622	7.5
6	0	1	7.250	10.5	7.508	9.0
7	1	–1	8.410	13.0	7.649	7.5
8	1	0	7.668	8.7	7.876	9.5
9	1	1	8.200	8.7	8.200	8.3

account the effect of interfactor linear-linear interaction, is as follows:

$$\rho = 7.3411 + 0.5395x_1 - 0.0472x_1^2 - 0.3008x_2 + 0.3888x_2^2 + 0.0788x_1x_2 \tag{1}$$

(determination coefficient $R^2 = 0.886$),

where $x_1 = \dfrac{P_{sp}}{50} - 3$; $x_2 = \dfrac{V_{pr}}{6} - 1.5$.

The deviation of experimental values from the values calculated by equation (1) of the response function in the corresponding points of the plan is on average 2.25%.

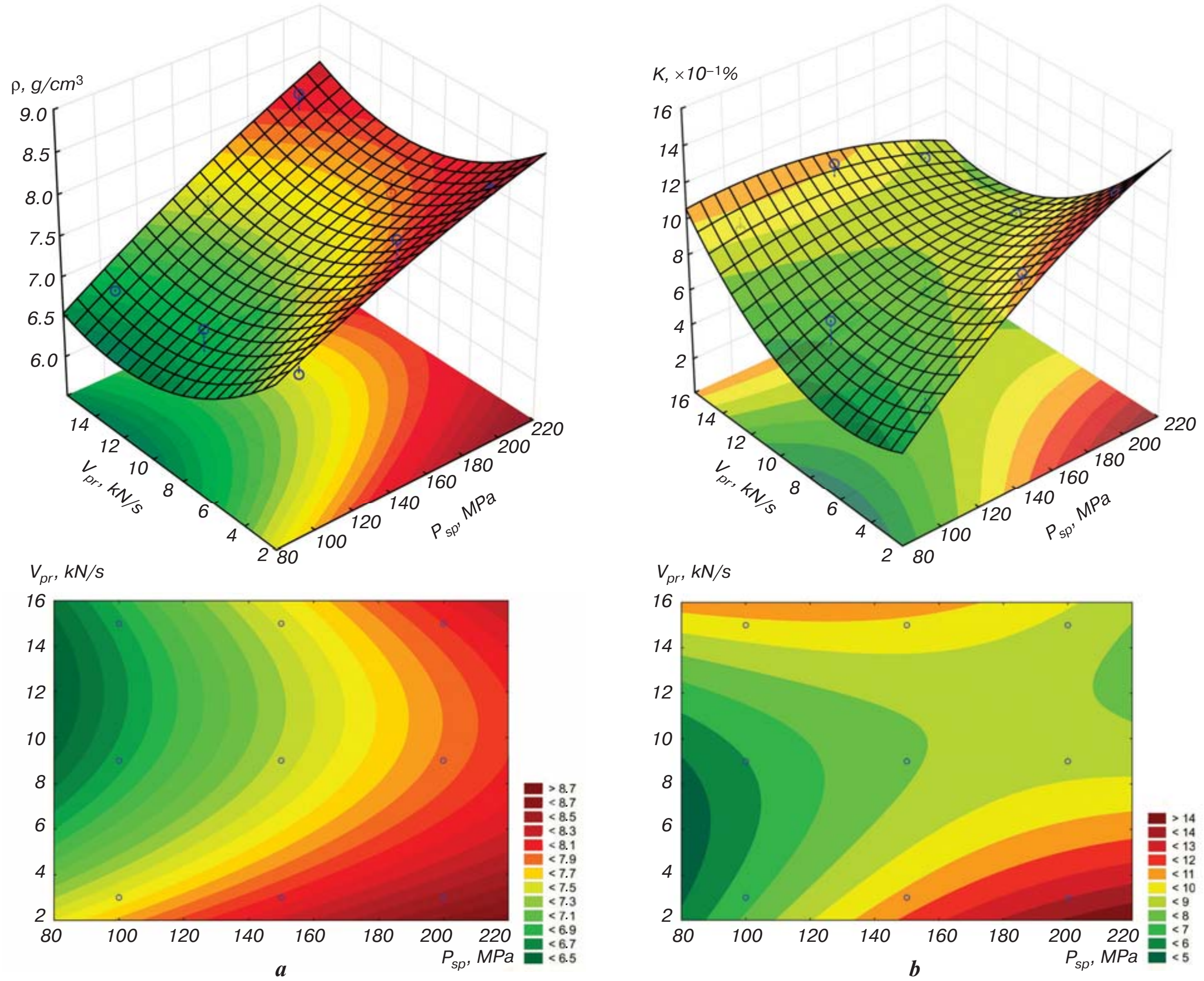


Fig. 7. Effect of pressure and pressing speed on density (*a*) and elastic aftereffect coefficient (*b*) of compressed samples (plasticizer SCD II)

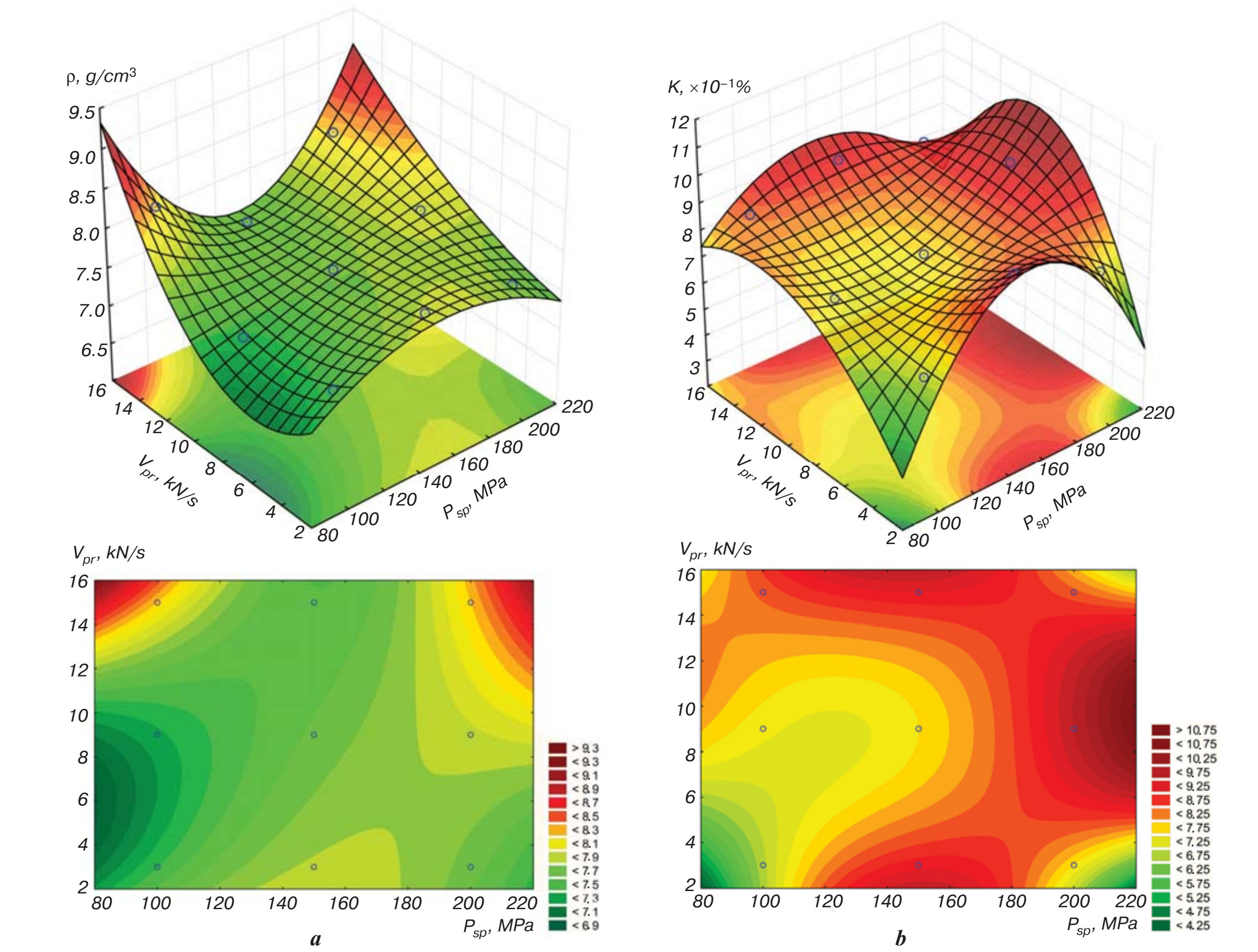


Fig. 8. Effect of pressure and pressing speed on density (*a*) and elastic aftereffect coefficient (*b*) of compressed samples (plasticizer PEG-1500)

The response surface and contour plot corresponding to model (1) are shown in **Fig. 7, *a***.

A similar model for determining the elastic aftereffect coefficient has the following form:

$$K = (7.8704 + 1.3333x_1 - 0.5556x_1^2 - 0.3056x_2 + 2.1944x_2^2 - 1.8333x_1x_2) \cdot 10^{-1} \text{ (determination coefficient } R^2 = 0.862). \quad (2)$$

The deviation of the experimental values from the values of the response function calculated by equation (2) at the corresponding points of the plan averages 7.97 %.

The response surface and contour plot corresponding to model (2) are shown in **Fig. 7, *b***.

To determine the density of compressed samples using PEG-1500 plasticizer, the regression equation, taking into account the effect of interfactor linear-linear interaction, does not approximate the experimental data accurately enough. Therefore, in order to find the values of density of compressed samples in the investigated range of variation of factors, we obtained the regression equation taking into account the effects of inter-factor quadratic-linear and quadratic-quadratic interactions (the response surface passes strictly through all experimental data points):

$$\rho = 7.6220 + 0.3000x_1 - 0.0460x_1^2 - 0.1560x_2 + 0.0420x_2^2 - 0.0633x_1x_2 - 0.2368x_1x_2^2 + 0.4948x_1^2x_2 + 0.2433x_1^2x_2^2. \quad (3)$$

The response surface and contour plot corresponding to model (3) are shown in **Fig. 8, *a***.

Similarly, for the determination of the elastic aftereffect coefficient, the regression equation taking into account the effect of interfactor linear-linear interaction has a low value of the coefficient of determination. The regression equation corresponding to the adequacy criterion is obtained taking into account the effects of interfactor quadratic-linear and quadratic-quadratic interactions:

$$K = (7.5000 + 1.0833x_1 + 0.9167x_1^2 + 0.0500x_2 + 1.4500x_2^2 - 0.2083x_1x_2 - 0.8750x_1x_2^2 + 0.5750x_1^2x_2 - 2.1583x_1^2x_2^2) \cdot 10^{-1}. \quad (4)$$

The response surface and contour plot corresponding to model (4) are shown in **Fig. 8, *b***.

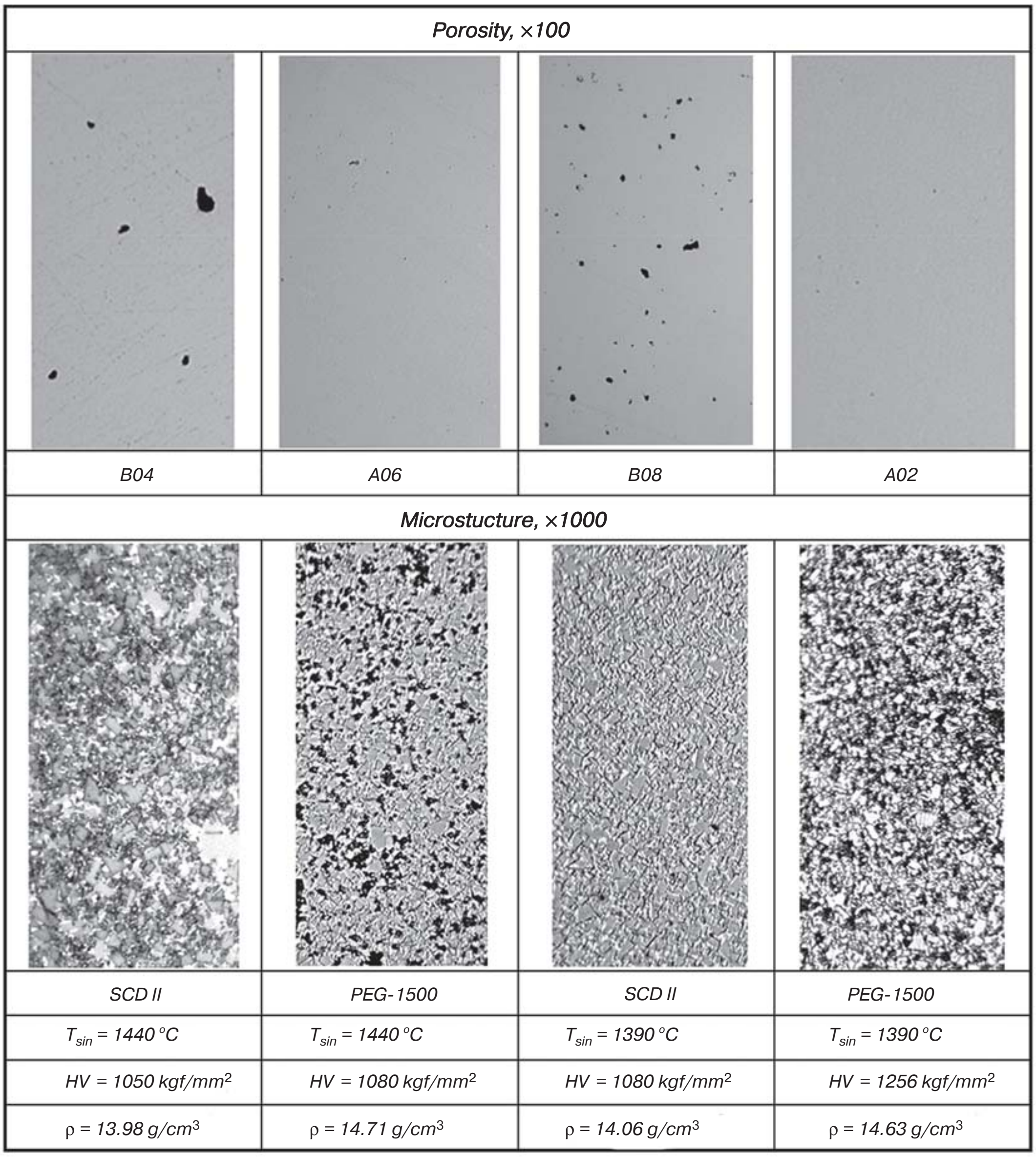


Fig. 9. Comparative analysis of porosity, microstructure, hardness and density of sintered carbide VK10-HOM

The circles in the diagrams (**Figs. 7, 8**) indicate the experimental values of response parameters at the corresponding points of the plan.

The values of density and elastic aftereffect coefficient calculated by the obtained regression models are in almost good agreement with the experimental values in the whole range of pressure and pressing speed variation.

By joint analysis of the diagrams in **Figs. 7, *a*; 7, *b*** and **Figs. 8, *a*; 8, *b***, the optimal parameters of the pressing process are determined as a result of solving the compromise problem. Experiments at a pressing speed of 15 kN/s resulted in delamination of samples on SCD II and PEG-1500 plasticizers. For the samples with PEG-1500 plasticizer, the best results were obtained at a pressing speed of 9 kN/s. Based on the results, the following modes of pressing of carbide mixture VK10-HOM with plasticizer SKD II were determined: pressing pressure — 190—200 MPa, pressing speed 9—11 kN/s; with plasticizer PEG-1500: pressing pressure – 180—200 MPa, pressing speed 8—10 kN/s.

Sintered carbide plates were fabricated with the specified parameters and porosity, microstructure, hardness and density were controlled. For convenience of analysis and perception, the results are summarized in **Fig. 9**.

The sintered plates using SCD II plasticizer show high porosity in the microstructure, and consequently, the density and hardness are lower than the optimum values.

Plates using PEG-1500 plasticizer at sintering temperature of 1390 °C show low porosity, absence of undesirable h-phase at higher values of density and hardness.

Conclusion

The quality of sintered carbide plates is influenced both directly by pressing modes and powder properties and conditions of its preparation at the stages preceding the pressing process.

For each chemical composition of carbide powder, plasticizer and carbide plate configuration, optimization studies of the pressing process are required.

When using VK10-HOM carbide powder, the main advantage of which is fine dispersity, which provides highly effective cutting properties of the tool, there are technological features that cause the complexity of mixing to a homogeneous state and shaping in a mold.

Density of moulded pieces and coefficients of elastic aftereffects as a function of pressing force are determined, which is necessary for calculation of geometrical parameters of molds.

According to the results of the experiments, the choice of plasticizer PEG-1500 (as a substitute for synthetic rubber) was justified, because it provided the best compressibility and the lowest elastic aftereffect coefficient in the whole range of pressing pressures.

As a result of pressing modes optimization by the method of optimal planning of multifactor experiment the following modes of pressing of hard-alloy mixture VK10-HOM were determined: with plasticizer SKD II - pressing pressure 190–200 MPa, pressing speed 9–11 kN/s; with plasticizer PEG-1500 — pressing pressure 180–200 MPa, pressing speed 8–10 kN/s.

When studying the microstructure, conglomerates of cobalt particles and large crystals of tungsten carbide (up to 20 microns) were noted, forming inhomogeneity. The best microstructure results were obtained with PEG-1500 plasticizer at low porosity, absence of undesirable h-phase, higher values of density and hardness on plates pressed under optimal conditions and sintered (sintering temperature 1390 °C).

Thus, the carried out researches have allowed to produce at optimum modes of pressing a carbide plate from mix VK10-HOM for cutting tools. Ongoing production tests show positive results. Implementation of the obtained results in the current tool production will allow to increase the durability of plates, reduce the costs of mechanical finishing of their size and shape, which will provide a reduction in the technological cost and generally improve the quality of finished tools.

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