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ANALYSIS OF POSSIBLE ENHANCEMENT OF PROPERTIES OF VK15 MATERIAL USED FOR DRILLING TOOLS

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Traditionally, when drilling hard and abrasive rocks, it is recommended to use a tungsten-cobalt hard alloy VK15. The analysis of information on the possibility of improving the potential of the material has demonstrated the existence of mechanisms that provide structural transformations that enhance its strength, hardness and toughness. The use of such technology instead of traditional methods will lead to an increase in the operating efficiency and durability of the tool. During the work, experimental samples of alloy VK15 were obtained by sintering in four different modes. Then their properties were analyzed. The results of the metallographic study carried out on the «Carl Zeiss» microscope made it possible to estimate the distribution of tungsten carbide grains in cobalt bon and show the grinding of the carbide phase. Thus, with traditional sintering, the amount of tungsten carbide grains with an average size of less than 1 µm in diameter from the entire size range reaches 19.5 %, while after additional heat treatment with a holding time of 1280 °C, the value was 41.5 %; 900 °C – 59.1 %; 600 °C – 54.5 %. The maximum improvement results were the following: hardness by 18 %, a coercive force by 49 %, and crack resistance by 11 % of the traditional alloy, there were achieved at 900-1280 °C. A hypothesis has been put forward on the formation of additional structural elements not detected by the methods of optical metallography. Studies of the topology and structure of the samples on an atomic force microscope confirmed the presence of nanoscale inclusions from 20 to 40 nm (presumably tungsten carbide) in a cobalt bond.

For VK15, comparative studies of properties and analysis of the microstructure of experimental samples obtained by the traditional sintering and modified technology have shown that the sintering mode at 900 °C is a priority. Thus, the developed technology, including the sintering of powders in a hydrogen stream up to 750 °C, from 750 to 1450 °C in vacuum, feeding at a maximum temperature of 1450 °C for 15 minutes of argon at a pressure of 60 bar, subsequent cooling to 900 °C and 1 hour exposure, can be recommended as the most rational conditions for revealing the potential of the material and providing an enhanced level of properties of the drilling tool.

Key words: tungsten-cobalt hard alloy; drilling tools; temperature interval; heat treatment

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Introduction. Currently, the Russian Federation is the world leader in mining. About 90 % of drilling operations is performed by roller drill bits. Hard alloy tools are used in cutting, drilling of exploration and production wells for oil, gas, water, and drilling for blasting operations. The performance characteristics of the drilling tool are determined by the physical-mechanical properties of the material selected for its working parts. When drilling hard and abrasive rocks, it is recommended to use hard alloy VK15 [2]. It belongs to tungsten-cobalt hard alloys, the structure of which consists of grains of tungsten carbide and cobalt. This group of alloys has high characteristics of hardness, wear and heat resistance [5].

Articulation of the problem. The needs of modern production and current information on this issue have made it possible to identify the possibilities of achieving increased hardness and toughness due to the strengthened cobalt bond. For example, the use of techniques such as modification of cobalt bonds with nano-additives leads to increased durability of tools [1, 3, 4, 7, 10-13, 15-17]. These methods help to improve mechanical properties but are costly. In addition to the changes in the granulometric and chemical composition of the initial components, the structure and properties of the alloys are affected by thermal treatment [8, 14].

In this paper, we propose the justification and results of testing the modernized technological process for obtaining a nib by sintering, reheating and modified cooling operation [6, 9].

a

b

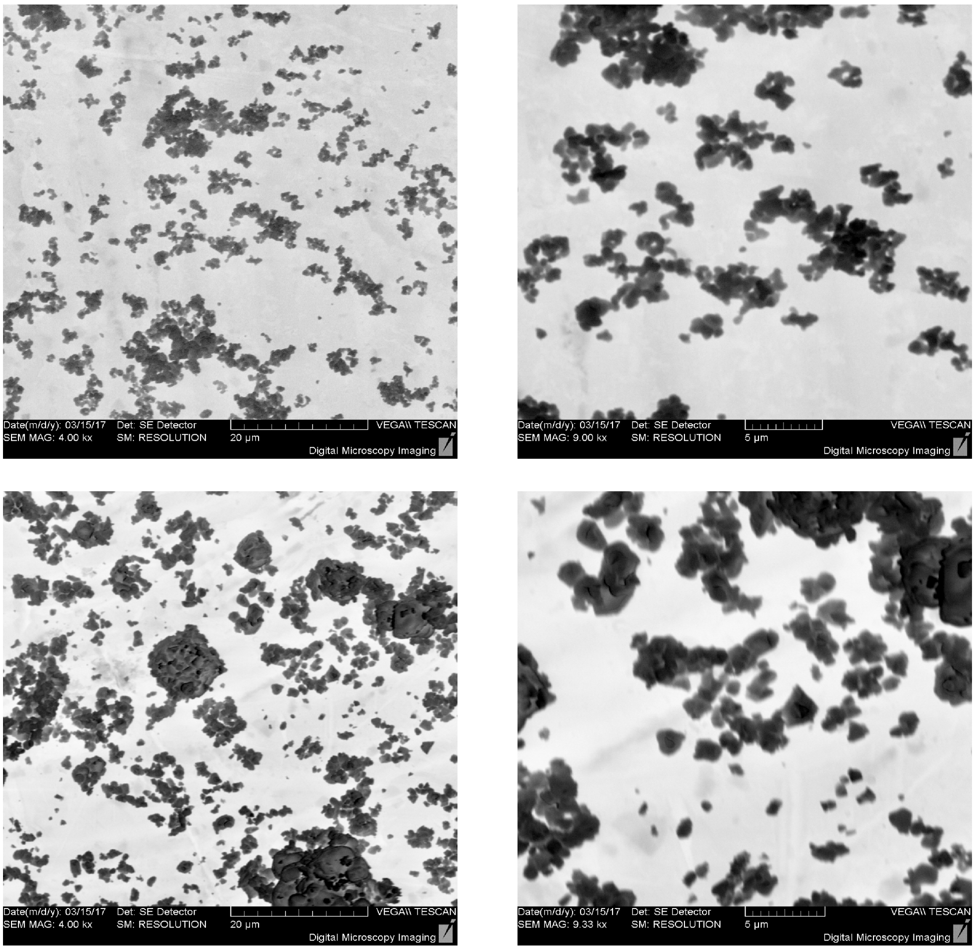

Fig.1. Powder Co (*a*) and WC (*b*), zoom in 4000^x and 9000^x

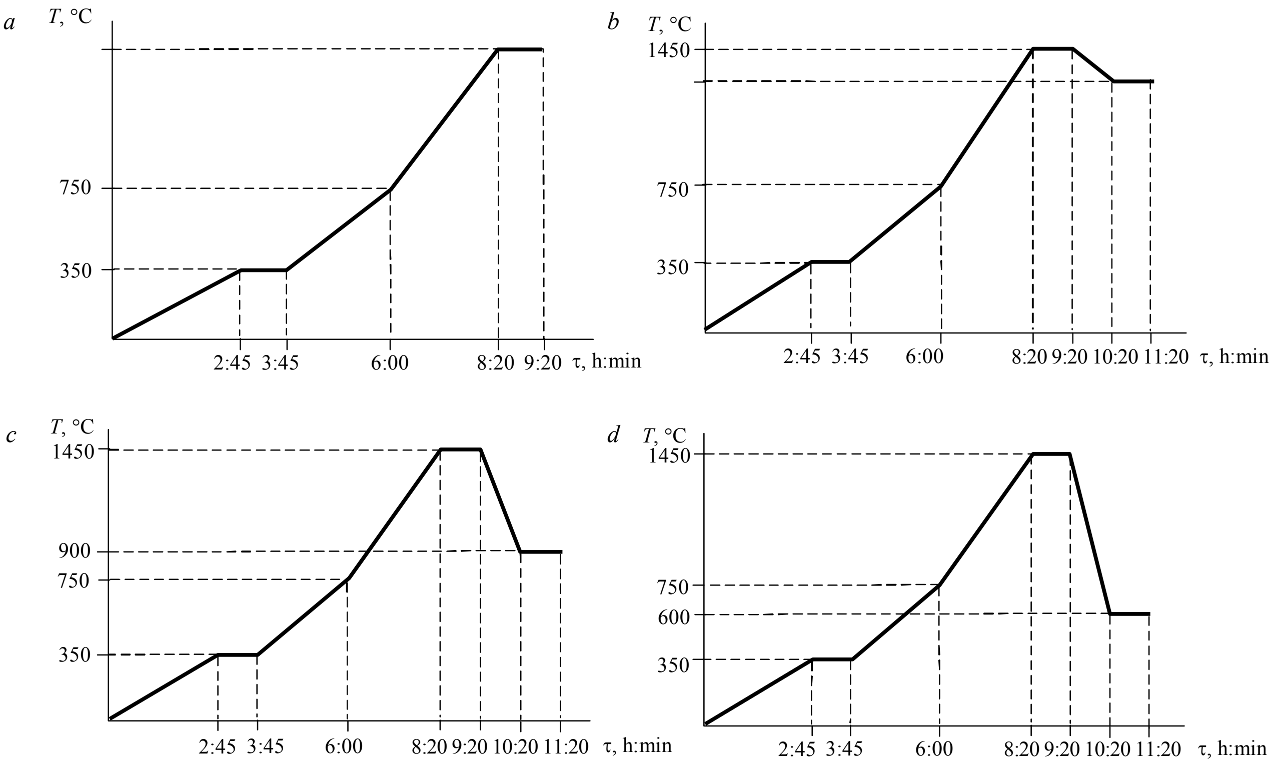

Fig.2. Treatment schedule charts: traditional (*a*) and sintering with additional exposure at 1280 °C (*b*); 900 °C (*c*); 600 °C (*d*)

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Methodology. The initial target materials are tungsten carbide powders of WC3 grade (TU 1742-002-46878311-2013) with an average powder particle size D50 = 3±1 μm and cobalt grade PK-1U (GOST 9721-79) with an average size of 1.2±0.2 μm. The control of the granulometric composition was carried out on the «Malvern Mastersizer 3000E» unit.

The chemical composition of the initial powders is as follows:

Compound	Mo	Si	Fe	Na+K	Pb	Ni	Mn	Ca	Al	S	C_{tot}	C_{free}
WC	0.18	0.003	0.100	0.010	–	–	–	0.005	0.003	0.023	6.12	0.050
Co	–	0.001	0.002	0.002	0.001	0.03	0.001	0.001	–	0.001	0.015	–

The research of the initial powders was carried out on a scanning electron microscope VEGA II LMH in the laboratory of sophisticated physical methods for studying the structure of materials of the Bauman MSTU (Fig.1).

The mixture of powders was milled in isopropyl alcohol medium in a wet grinding roller mill. Then it was dried, mixed with a solution of synthetic rubber in gasoline and pressed in steel molds at a pressure of 100 MPa. Sintering of compacts in the vacuum-compression furnace PVK-1 was carried out in various modes to determine the most effective one. All technological operations were performed on the basis of OJSC «Zavod Tekhnicheskoy keramiki (Plant of Technical Ceramics)» (Table 1).

Table 1

Sintering modes

Mode №	Temperature mode			Purpose of operation
	Temperature, °C	Heating rate, °C/min	Exposure, min	
1	≤ 350	2.0	–	Removal of plasticizer
	350	–	60	
	350-750	3.0	–	
	750-1450	5.0	–	Preliminary sintering
	1450	–	60	Final sintering
	1450-20	–5.0	–	Cooling
2	≤ 350	2.0	–	Removal of plasticizer
	350	–	60	
	350-750	3.0	–	
	750-1450	5.0	–	Preliminary sintering
	1450	–	60	Final sintering
	1450-1280	–3.2	–	Cooling
	1280	–	60	Exposure
	1280-20	–5.0	–	Cooling
3	≤ 350	2.0	–	Removal of plasticizer
	350	–	60	
	350-750	3.0	–	
	750-1450	5.0	–	Preliminary sintering
	1450	–	60	Final sintering
	1450-900	–3.2	–	Cooling
	900	–	60	Exposure
	900-20	–5.0	–	Cooling
4	≤ 350	2.0	–	Removal of plasticizer
	350	–	60	
	350-750	3	–	
	750-1450	5	–	Preliminary sintering
	1450	–	60	Final sintering
	1450-600	–3.2	–	Cooling
	600	–	60	Exposure
	600-20	–5	–	Cooling

The charts of the described modes are shown in Fig. 2.

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Up to 750 °C sintering is carried out in the hydrogen flow, from 750 to 1450 °C – in a vacuum to facilitate compaction, remove gases from porous compacts and ensure better wettability of cobalt carbides. At a maximum temperature of 1450 °C argon is supplied at a pressure of 60 bar for 15 minutes, then for the modified technology it was cooled to a temperature of 600, 900, 1280 °C, respectively, for different modes. At the end of each mode, the cage was cooled with the furnace to ambient temperature.

Discussion. Several characteristic properties of the obtained experimental samples were measured (Table 2).

Table 2

Results of measurements of physical and mechanical properties

Heating treatment mode	Density, g/cm^3	Hardness, HV 10	Coercive force, oersted	Crack resistance, MPa·m$^{1/2}$
1	13.97	1033	78	10.59
2	14.0	1088	98	10.79
3	14.1	1222	116	11.70
4	14.07	1153	107	11.05

The density was determined according to GOST 200818-74 on the VIBRA HT224RCE density meter. The Vickers hardness test was carried out on a PMT-3 micro hardness measuring unit with a maximum load of 200 g, a coercive force measurement was done on a «Cobalt-1» instrument in accordance with GOST 24916-81.

The analysis of the obtained data confirms the efficiency of the modified technology. A slight increase in density nevertheless indicates that heat treatment leads to a reduction in defects of the residual pore type. The increase in the coercive force and hardness makes it possible to judge the increase in the dispersion of grains, since it is known that these are indirect indicators of the grinding of the structure in hard alloys.

The graphic comparative analysis of these properties obtained for the three experimental regimes is shown in Fig. 3.

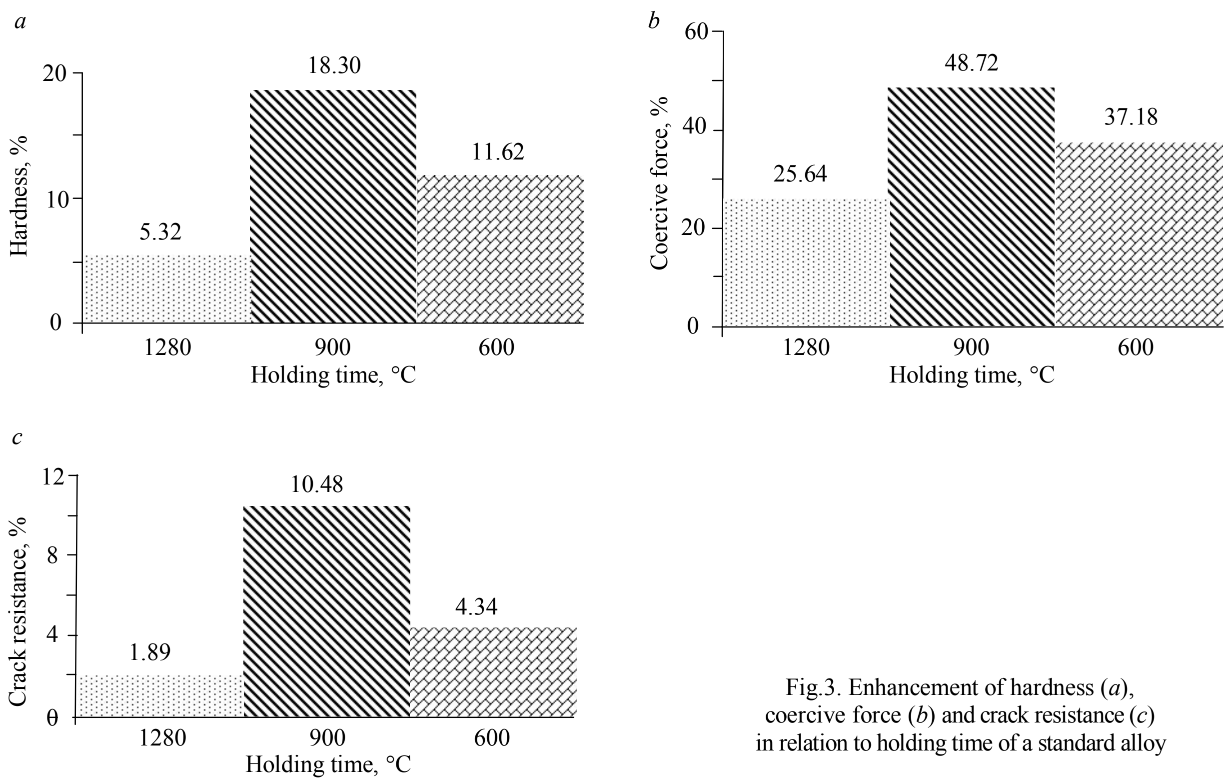

Fig.3. Enhancement of hardness (*a*), coercive force (*b*) and crack resistance (*c*) in relation to holding time of a standard alloy

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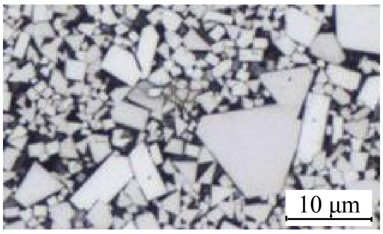
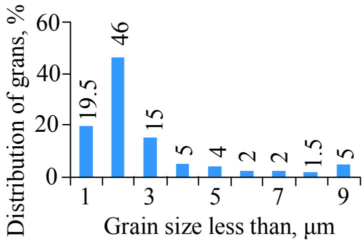
Table 3

Results of metallographic analysis and analysis of grain distribution

<table>
<tr><th>Heat treatment</th><th>Microstructure</th><th>Distribution of grains, %</th></tr>
<tr><td>1</td><td>
</td><td>
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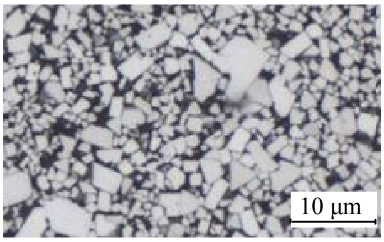
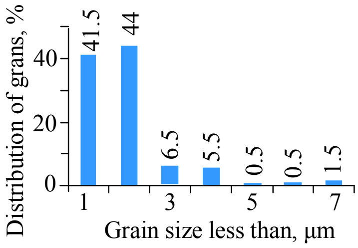
<tr><td>2</td><td>
</td><td>
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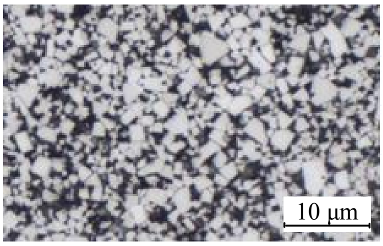
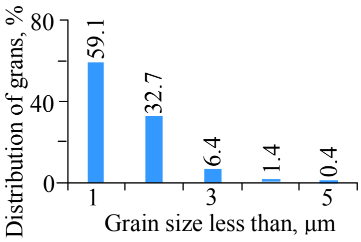
<tr><td>3</td><td>
</td><td>
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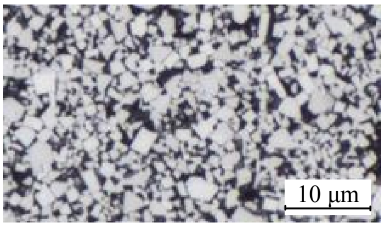
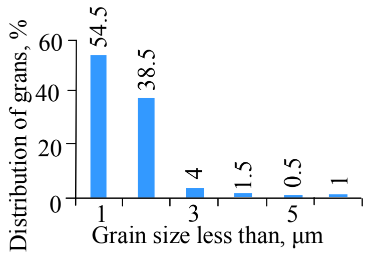
<tr><td>4</td><td>
</td><td>
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Since the grain boundaries and other structural imperfections impede the movement of the crack, the observed increase in the fracture toughness can be explained by the need for additional energy to develop a crack. The results of the metallographic study carried out on the «Carl Zeiss» microscope and the analysis of the distribution of grains of tungsten carbide in alloys are presented in Table 3.

The structure analysis demonstrates the apparent grinding of the carbide phase after the heat treatment. So, with traditional sintering, the amount of grains of tungsten carbide with an average size of less than 1 μm in diameter from the entire size range reaches 19.5 %, while after additional processing with a holding time of 1280; 900; 600 °C – 41.5; 59.1; 54.5 % respectively.

The effect of grinding, equal to the relative increase in properties, is demonstrated by the alloy after additional exposure at 900 ° C (see Table 2, mode 3).

When comparing the total data of the research, it was found that for this mode the maximum of the experimental samples studied was observed to increase the hardness by 18 %, the coercive force by 49 %, and the crack resistance by 11 % relative to the characteristics of the compact material obtained by traditional technology. A hypothesis has been put forward on the formation of additional structural elements that are not visible in optical metallography. The increase in hardness and viscosity leads to an increase in the operational stability of the drilling tool and a reduction in the costs of drilling industrial wells.

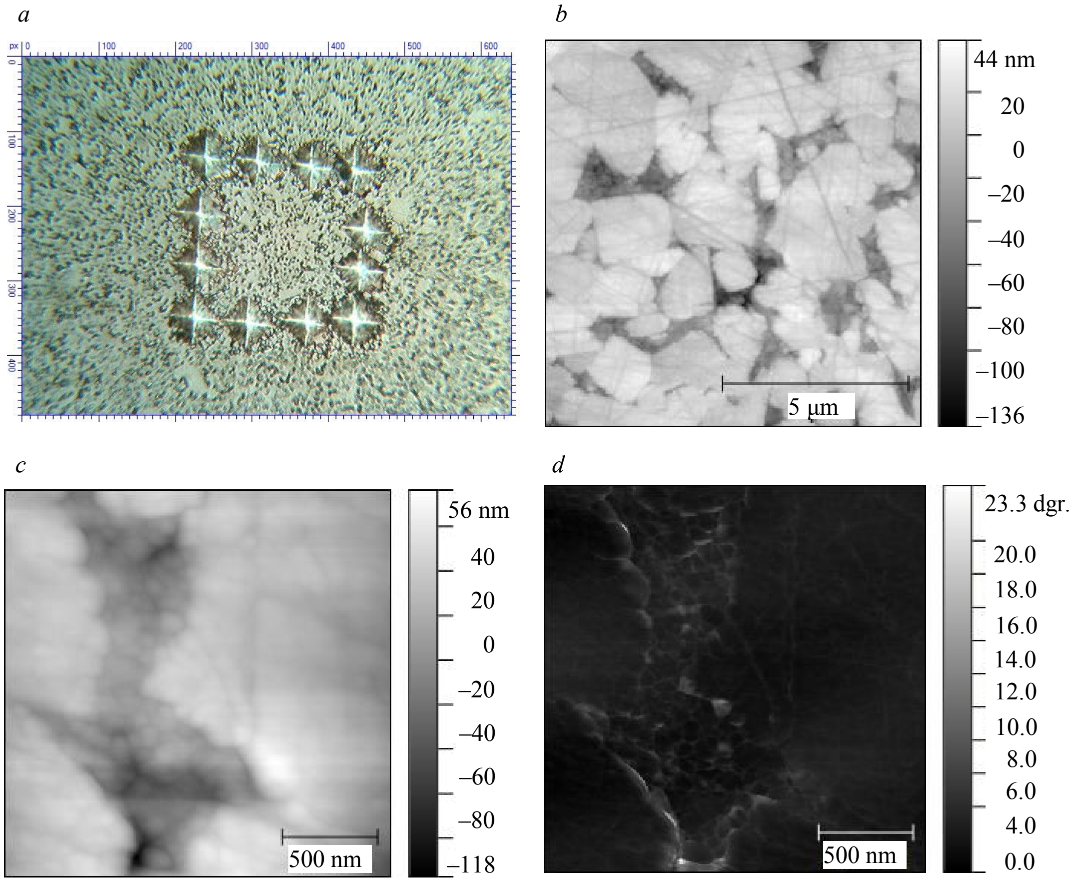

Fig.4. Scan results on an atomic force microscope: *a* and *b* are a 40 by 40 µm region on the sample; *c* and *d* – the AFM scan of the sample in the region 3 by 3 µm and with the phase difference, respectively

To confirm the assumption, a study of the heterogeneity of the structure of the cobalt bond on a sample obtained by mode 3 on an atomic force microscope using unique scientific installations was carried out at the center for collective use of the Scientific and Educational Center «Functional Micronanosystems». The results of the studies are presented in Fig. 4.

Studies of the topology and structure of the samples confirmed the presence of nanoscale inclusions, presumably tungsten carbide, in a cobalt bond. The characteristic size of inclusions is from 20 to 40 nm.

Conclusion. During the research it was found that the sintering conditions, namely the combination of temperature, heating and cooling rate, temperature and holding time, and temperature range of the additional heat treatment, significantly affect the structural transformations, and therefore can be a mechanism for controlling the properties of the hard alloy. Thus, for VK15, comparative studies of properties and analysis of the microstructure of experimental samples obtained by the traditional sintering mode and the developed sintering technology showed that the sintering mode, modified by exposure at 900 °C, is a priority.

Thus, the developed technology, including the sintering of powders in a hydrogen stream up to 750 °C, from 750 to 1450 °C in vacuum, with feeding at a maximum temperature of 1450 °C for 15 minutes of argon at a pressure of 60 bar, cooling with a furnace to 900 °C and 1 hour exposure, can be recommended as the most rational for revealing the potential of the material and providing an enhanced level of properties of the drilling tool.

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