УДК 539.893.621.317.451 DOI 10.36910/775.24153966.2025.83.17

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ЕКСПЕРИМЕНТАЛЬНІ ДОСЛІДЖЕННЯ ОДЕРЖАННЯ ФУНКЦІОНАЛЬНО ОРІЄНТОВАНИХ ПОРОШКІВ СИНТЕТИЧНОГО АЛМАЗУ

Експериментальними дослідженнями порошку синтетичного алмазу марки АС20 зернистості 100/80 показано, що вплив способів модифікування вихідного порошку (модифікування високовольтними електричними розрядами або рідинофазним окисленням) відображається на зміні адсорбційно-структурних та морфометричних характеристик порошків: зокрема питомої площі поверхні; питомого об'єму пор; середнього радіусу пор; сумарного об'єму пор, розподілу об'єму пор за їх розмірам.. Показник питомої площі поверхні вихідних зразків має тенденцію до зниження після модифікування порошків. Досліджені зразки мають розвинену мезопористу структуру поверхні. Досліджені способи модифікування не виявляють суттєвого впливу на морфометричні характеристики порошків. Порівняльний аналіз результатів за коефіцієнтами лінійної апроксимації кривих розподілу пор за розміром дає змогу отримати на кількісному рівні показник (а2/а1) зміни стану поверхні порошків, що може слугувати характеристикою якості поверхні порошку при вивченні дефектності поверхні зерен синтетичного алмазу та її впливу на експлуатаційні характеристики алмазного інструменту. Отримані результати дають підґрунтя для подальшого розроблення бази даних формування оптимальних показників адсорбційно-структурних характеристик порошків синтетичного алмазу широкого функціонального призначення.

Ключові слова: *порошок синтетичного алмазу, адсорбційно-структурні характеристики, морфометричні характеристики*

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EXPERIMENTAL STUDIES ON THE PREPARATION OF FUNCTIONALLY ORIENTED SYNTHETIC DIAMOND POWDER

Experimental studies of synthetic diamond powder of the AC20 grade with a grain size of 100/80 have shown that the influence of the methods of modifying the original powder (modification by high-voltage electric discharges or liquid-phase oxidation) is reflected in the change in the adsorption-structural and morphometric characteristics of the powders: in particular, the specific surface area; specific pore volume; average pore radius; total pore volume, and the distribution of pore volume by their sizes. The specific surface area of the original samples tends to decrease after the modification of the powders. The studied samples have a developed mesoporous surface structure. The studied modification methods do not show a significant effect on the morphometric characteristics of the powders. Comparative analysis of the results by the coefficients of linear approximation of the pore size distribution curves allows us to obtain at the quantitative level the indicator (a_2/a_1) of the change in the state of the powder surface, which can serve as a characteristic of the quality of the powder surface when studying the defectivity of the surface of synthetic diamond grains and its influence on the operational characteristics of the diamond tool. The results obtained provide a basis for further development of a database for the formation of optimal indicators of the adsorption-structural characteristics of synthetic diamond powders of wide functional purpose.

Keywords*: synthetic diamond powder, adsorption-structural characteristics, morphometric characteristics*

Functional synthetic diamond powders are used in various industries. The properties of the powder are formed in the process of synthesis, extraction, sorting and modification, which determines the formation of powder properties, including the characteristics of the surface of its grains. The effectiveness of the use of powders largely depends on the adsorption-structural characteristics of the surface [1]. In well-known publications, the influence of methods for modifying synthetic diamond powders on changing their physico-mechanical and physico-chemical properties of diamond powder, morphology and chemical composition of the surface, adsorption-structural characteristics of the powder [1-4] has been studied.

The creation of new methods for modifying diamond powders is relevant and has important applied value. For the effective manufacture and operation of abrasive tools, it is necessary to study the state of the surface of diamond powder according to adsorption-structural characteristics: specific surface area, pore distribution (micro- and mesopores) by size and pore volume [1].

Another important aspect for the effective manufacture and operation of abrasive tools is also the study of the morphometric characteristics of modified powders.

The purpose of this study. The purpose of this study is to study at a quantitative level the influence of methods of modifying the original powder on the change in adsorption-structural and morphometric characteristics.

Methodology. Samples of diamond powder of the AC20 grade with a grain size of 100/80, synthesized in the Ni-Mn-C system, were studied. Two methods were used to modify the powder: a method of chemical modification using liquid-phase oxidation and a method of pulsed processing by high-voltage electric discharges in an aqueous medium (HVED). HVED modification was carried out on an experimental stand in distilled water with a pressure of 800 MPa and a total processing energy of 1000 kJ in the discharge channel.

Adsorption-structural characteristics were determined using a NOVA 2200 gas adsorption analyzer (Quantachrome, USA). The isotherm was determined by the method of low-temperature nitrogen adsorption-desorption (77 K) at $p/p_S = 0.99$, which was used to determine the specific area (S_{BET}. (m^2/g) in the region of the monolayer of nitrogen surface coverage; The specific pore volume Vp (cm^3/g), average pore radius Rp (nm), adsorption energy (J/m^2), volume and radius of mesopores; total pore and mesopore volumes, and size distribution of pores and mesopores were determined by the DFT method.

The free energy of saturation of the powder surface with water vapor, which characterizes the degree of hydrophilicity (mJ/g•mol, ΔCs) was determined by the method developed at the V.M. Bakul Institute of Materials Science and Engineering of the National Academy of Sciences of Ukraine.

The surface topography was determined using scanning microscopy methods; morphometric characteristics (compactness (form factor, Cr) and roughness (Rg) of grain projection; the number of cutting edges (n, pcs.) and the average value of their sharpening angles (φ, degrees) were determined by indirect analytical method - using the DiaInspect.OSM device from Vollstaedt Diamant GmbH to obtain the necessary initial data.

A comparative analysis was carried out based on the results of linear approximation of the pore size distribution curves. The results of the study are presented in Tables 1-4

Table 1

Adsorption and structural characteristics of the initial AC20 diamond powder with a grain size of 100/80 and powder samples after modification

Characteristic name	Sanding powder brand AC20, grain size 100/80		
	Methods of powder modification		
	Diamond powder (DSTU 3292-95)	Chemical modification	Application of HVED modification
Specific surface area, S_{BET}., m^2/g	0,5422	0,2542	0,4151
Adsorption energy, kJ/mol	7,544	8,446	0,6499
Specific pore volume, $V \cdot 10^{-3}$, cm^3/g	1,234·	0,8682	3,131
Average pore radius, nm	4,551	6,831	8,288
energy of saturation of the powder surface with water vapor (hydrophilicity) mJ/g•mol, ΔCs	264,9	187,1	119,3

According to the results of the experimental study using the HVED method, it was found that, compared with the indicators of the original powder, chemical modification using liquid-phase oxidation leads to a decrease in the specific surface area by 53%; the free energy of saturation of the powder surface with water vapor by 29.2%; the specific pore volume by 23.9%; to an increase in the adsorption energy by 11.9% and the average pore radius by 50.1%.

The use of the HVED modification method leads to a decrease in the specific surface area by 23.4%; the specific pore volume by 47.3%; the average pore radius by 31.1%; to an increase in the adsorption energy by 9.9%; to a decrease in the free energy of saturation of the powder surface with water vapor by 55.0%.

According to the results of the experimental study using the BET method, it was found that, compared with the indicators of the original powder, chemical modification using liquid-phase oxidation leads to a decrease in the specific surface area by 53%; the free energy of saturation of the powder surface with water vapor by 29.2%; The morphometric characteristics of the original AC20 diamond powder with a grain size of 100/80 and the powder samples after modification do not differ significantly. HVED modification leads to an increase in the number of cutting edges and the average value of the sharpening angles of the cutting edges.

Table 2

Morphometric characteristics of the initial AC20 diamond powder with a grain size of 100/80 and powder samples after modification

Characteristic name		Sanding powder brand AC20, grain size 100/80		
		Methods of powder modification		
		Diamond powder (DSTU 3292-95)	Chemical modification	Application of HVED modification
Compactness (form factor), Cr	Average value	1,304	1,3042	1,305
	Uniformity	0,7107	0,6979	0,6985
Grain projection roughness, Rg	Average value	1,0602	1,06001	1,06503
	Uniformity	0,7402	0,7429	0,7435
Number of cutting edge sharpenings, n		10	10	11
Average value of cutting edge sharpening angles, φ		106,37	106,32	107,32

According to the classification officially adopted by the International Union of Pure and Applied Chemistry (IUPAC), pores with sizes (diameters) less than 2 nm are micropores, 2-50 nm are mesopores, and more than 50 nm are macropores.

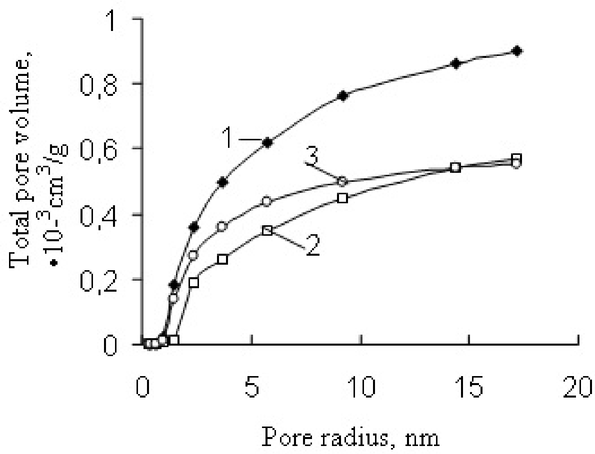

Fig. 1 **Pore size distribution, determined by pore volume using the DFT method, of synthetic diamond grinding powder of the AC20 brand with a grain size of 100/80: initial powder (1), powder after chemical modification (2) and modification by HVED treatment (3)**

According to the results of the experimental study using the BET method, it was found that, compared with the indicators of the original powder, chemical modification using liquid-phase oxidation leads to a decrease in the specific surface area by 53%; the free energy of saturation of the powder surface with water vapor by 29.2%; Fig. 1 shows the distribution of the pore radius (the calculated values of the pore radius, nm) by the value of the total pore volume. As can be seen from Fig. 1, all the studied samples have a developed mesoporous surface structure. The indicators of the pore distribution by size of the original powder Fig. 1 (curve 1), after chemical modification (curve 2) and after VER modification differ.

Table 3 presents the adsorption-structural characteristics (average pore radius, total pore volume and mesopore volume, total pore volume radius interval and mesopore volume radius interval) of synthetic diamond grinding powder samples, initial and after modification.

The range of pore size distribution in all three samples of grinding powders, the original and those obtained after modification, is almost the same and is (0.3-17.2) nm.

The average pore radius of the grinding powder of the AC20 100/80 brand after chemical modification increases to 1.55 nm from 1.42 nm (original sample). At the same time, the total pore volume decreases to 0.57•10^{-3} cm^3/g relative to the value of the pore volume of the original sample of 0.90•10^{-3} cm^3/g.

Table 3

Adsorption and structural characteristics of samples of ground and micropowders of synthetic diamond, initial and after modification

Diamond powder (DSTU 3292-95)	Modification method	Sample №	Average pore radius (DFT method), nm	Pore volume, pore size range (DFT method)			
				Total pore volume, V•10^{-3}, cm^3/g	Total pore volume radius interval, nm	Mesopore volume V 10^{-3}, cm^3/g	Mesopore radius range, nm
AC20 gritAC20 100/80	Diamond powder (DSTU 3292-95)	1	1,42	0,90	0,3–17,2	0,88	1,0–17,2
	Chemical modification	2	1,55	0,57	0,3–17,2	0,54	1,0–17,2
	HVED modification	3	1,42	0,55	0,3-17,2	0,54	1,0–17,2

The average pore radius of the grinding powder of the AC20 100/80 brand after VER modification is 1.42 nm and is identical to the average pore radius of the original sample. At the same time, the total pore volume decreases to 0.55•10^{-3} cm^3/g relative to the value of the pore volume in the original sample of 0.90•10^{-3} cm^3/g.

For comparative analysis at the quantitative level of indicators of changes in the state of the surface of the powders after modification, a linear approximation (y=ax+b) of the pore and mesopore size distribution curves was performed, and the coefficients (a, b) were established with the reliability of the approximation R^2 (Table 4). The ratio of the coefficients a_2/a_1 (where a_1 is the coefficient of the equation of the pore distribution curves by the total pore volume, a_2 is the coefficient of the equation of the pore distribution curves by the total mesopore volume) allows us to establish the proportion of the mesopore volume in the total pore volume.

Table 4

Results of linear approximation of pore size distribution curves

Sample №	Linear approximation of pore size distribution curves (y=ax+b)						The proportion of mesopore volume in the total pore volume, (a_2/a_1), %
	Coefficients of the equation of pore distribution curves by total pore volume		Accuracy of approximation, R^2	Coefficients of the equation of pore distribution curves by total vesopore pore volume		Accuracy of approximation, R^2	
	$a_1 \cdot 10^{-3}$	$b_1 \cdot 10^{-3}$		$a_2 \cdot 10^{-3}$	$b_2 \cdot 10^{-3}$		
1	0,0561	0,0932	0,84	0,0406	0,2829	0,87	72,37
2	0,0367	0,0270	0,89	0,0306	0,1019	0,86	83,38
3	0,0363	0,027	0,89	0,0223	0,2261	0,78	61,43

As follows from the results, the studied samples have a developed mesoporous surface structure, as evidenced by the proportion of the mesopore volume in the total pore volume of the original powders equal to 72–83%. After chemical modification, the proportion of the mesopore volume increases by 11%, after HER modification, the proportion of the mesopore volume decreases by 11%.

Conclusions

The results of the study established:

– the study of the influence of the methods of modifying the initial powder, in particular by chemical and VER methods, on the change in the adsorption-structural and morphometric characteristics showed that the value of the specific surface area of the initial samples tends to decrease after the modification of the powders;

– the studied samples have a developed mesoporous surface structure. The use of modification by the methods of liquid-phase oxidation and pulsed treatment with high-voltage electric discharges in an aqueous environment of the initial synthetic diamond grinding powder allows changing the values of the adsorption-structural characteristics of the powders: in particular, the specific surface area; specific pore volume; average pore radius; total pore volume; distribution of the pore volume by their sizes;

– the studied modification methods do not show a significant effect on the morphometric characteristics of the powders;

– comparative analysis of the results by the coefficients of linear approximation of the pore size distribution curves allows us to obtain at the quantitative level the indicator (a2/a1) of the change in the state of the powder surface, which can serve as a characteristic of the quality of the powder surface when studying the surface defects of synthetic diamond grains and its influence on the operational characteristics of a diamond tool;

– the obtained results provide a basis for further development of a database for the formation of optimal indicators of adsorption-structural characteristics of synthetic diamond powders of wide functional purpose.

The study was carried out on the topic III-4-25 (0789) “Study of the surface defects of synthetic diamond grains of a wide range of strength (AC6-AC160) and its influence on the operational characteristics of a diamond tool”. State registration number 0125U000040.Registration of the Bureau of the VFTPM of the NASU dated 10.12.2024, protocol No. 23; under the Memorandum of Cooperation between Lutsk National Technical University and the V.M. Bakul Institute of Superhard Materials (No. 17 dated 01/30/2024) and under the Memorandum No. 18 dated 06/19/2025 on cooperation between the IIPT of the NAS of Ukraine and the V.M. Bakul Institute of Superhard Materials of the NAS of Ukraine.

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