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ПОВЕРХНЕВІ ВЛАСТИВОСТІ АЛМАЗНИХ ШЛІФПОРОШКІВ МАРКИ АС250 РІЗНИХ ЗЕРНИСТОСТЕЙ

У статті представлені результати дослідження поверхневих властивостей алмазних шліфувальних порошків марки АС250 з розміром зерна 630/500 та 500/400 (згідно з нормативною документацією України). Дослідження структурно- сорбційних властивостей поверхні шліфувальних порошків проводилося об'ємним методом адсорбції-десорбції азоту за стандартною процедурою. Показано, що, незважаючи на більший розмір кристалів алмазу, зразок з розміром зерна 630/500 має більшу питому площу поверхні (0,21 та 0,18 м^2/г відповідно) та об'єм пор 1,99 проти 1,75×10-4 мл/г, порівняно зі зразком з розміром зерна 500/400. Цей результат зумовлений меншим середнім радіусом пор для зразка з розміром зерна 630/500, порівняно зі зразком з розміром зерна 500/400. Водночас, досліджувані зразки мають питому поверхню приблизно в 50–70 разів більшу, ніж площа поверхні ідеальних кристалів алмазу відповідного розміру зерна, та підвищену гідрофільність, що є результатом наявності точкових дефектів у поверхневому шарі зразків.

Ключові слова: *алмазні шліфувальні порошки, питома поверхня, об'єм та розмір пор, гідрофільність, концентрація азотовмісних дефектів.*

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SURFACE PROPERTIES OF DIAMOND GRINDING POWDERS OF THE AC250 BRAND OF DIFFERENT GRAIN SIZES

The paper presents the results of the study of the surface properties of diamond grinding powders of the AC250 brand with grain sizes of 630/500 and 500/400 (according to the normative documentation of Ukraine). The study of the structural and sorption properties of the surface of the grinding powders was carried out by the volumetric method of nitrogen adsorption-desorption according to the standard procedure. It is shown that despite the larger size of the diamond crystals, the sample with grain size of 630/500 has a larger specific surface area (0.21 and 0.18 m^2/g, respectively) and a pore volume of 1.99 versus 1.75×10^{-4} ml/g, compared to the sample with grain size of 500/400. This result is due to the smaller average pore radius for the sample with grain size of 630/500, compared to the sample with grain size of 500/400. At the same time, the studied samples have a specific surface area approximately 50–70 times larger than the surface area of ideal diamond crystals of the corresponding grain size, and increased hydrophilicity, which is the result of the presence of point defects in the surface layer of the samples.

Keywords: *diamond grinding powders, specific surface area, pore volume and size, hydrophilicity, concentration of nitrogen-containing defects*

Introduction and problem statement. In industry, during mass production of machine parts, precision plunge grinding of difficult-to-machine materials is becoming increasingly widespread, which ensures high-performance and high-quality processing of parts of complex shape, high accuracy and stability of the quality of the processed products. To carry out such processing, straightening rollers are used. At present, considerable experience has been accumulated in the design and manufacture of straightening rollers, which are equipped with high-strength diamond grinding powders of grades AC200 - AC400.

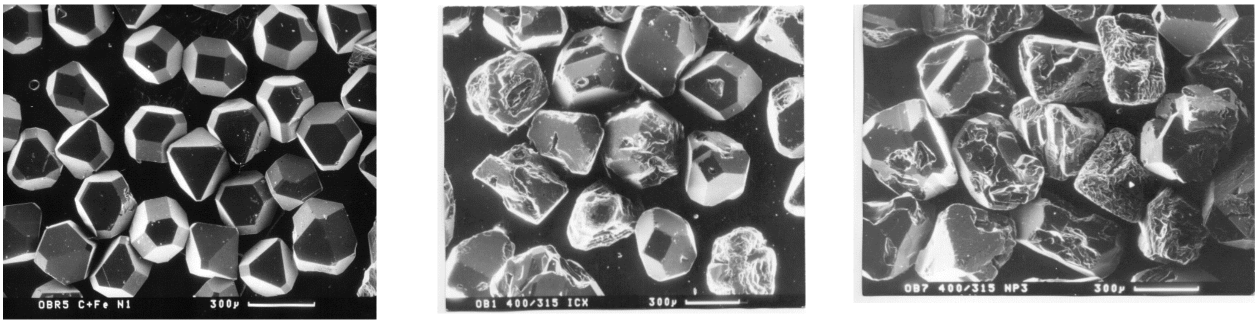
When growing diamond crystals in various growth media, depending on the growth conditions and after their extraction, sorting and processing, grinding powders with varying degrees of surface defects are formed - from atomically smooth to rough. Surface defects significantly affect the strength characteristics of crystals and the performance of any diamond tool.

The purpose of this work is to study the physico-mechanical and physico-chemical properties of high-strength diamond grinding powders of the AC250 brand with grain sizes of 630/500 and 500/400, which are used for the manufacture of straightening rollers.

Analysis of recent research and publications. High-performance and high-quality processing of parts of complex shape, including those made of difficult-to-machine materials, is provided by precision deep plunge grinding with straightening rollers. When performing such processing, the use of diamond tools is of great importance. In the works of V.I. Lavrinenko, O.O. Pasichny, V.V. Smokvina, it is shown that the performance of such a tool largely depends on the quality of the diamonds used, which is due to the need to provide the straightening tool with the maximum possible stability [1-3]. For the manufacture of straightening tools, natural diamond powders of increased strength and grain size are usually used.

At the Institute of Superhard Materials of the National Academy of Sciences (INM of the National Academy of Sciences) for the manufacture of straightening rollers, work was carried out to study the possibilities of replacing natural diamonds with high-strength diamond grinding powders of the AC250 brand, which are obtained in growth environments at high pressures and temperatures (HPHT method). Real diamond crystals obtained from various growth media have surface defects that significantly affect the strength characteristics of the crystals and the performance of any diamond tool [4-6].

Thus, the research of scientists of the Institute of Metallurgical Materials of the National Academy of Sciences of Ukraine A.A. Shulzhenko, A.L. Maistrenko, G.P. Bogatyreva, G.F. Nevstruev and others convincingly showed a significant influence on the strength characteristics of diamond grinding powders of grain shape, the presence of metal-solvent inclusions and other volumetric and surface defects. The general appearance of diamond grinding powders of grain size 250/200 with a smooth (a), with a partially defective (b) and defective surface is shown in Fig. 1 [6].



a) **b)** **c)**

Fig. 1. **General appearance of diamond grinding powders with a smooth (a), partially defective (b) and defective surface (c)** [6].

In Fig. 1, one can visually note their noticeable differences, which should be considered as extreme manifestations of the defectivity of the surface of diamond crystals. In fact, there is a continuous series of the state of the surface of diamond crystals from smooth to defective. Moreover, there is no sharp boundary between crystals with a smooth surface and crystals with only a defective surface. It can be observed that on the surface of one crystal there are both areas of a smooth surface and areas with defects. Therefore, when characterizing the defectivity of the surface, it is necessary to take into account the state of the entire surface area of diamond grains.

Materials and methods of research. The research was conducted on diamond grinding powders of the AC250 brand with grain sizes of 630/500 and 500/400 , determined according to the regulatory documentation of Ukraine. For diamond samples, the physical and mechanical characteristics were determined using the methods developed at the Institute of Mechanical Engineering of the National Academy of Sciences: strength, as an indicator of strength under static compression (P, H), uniformity in strength (Codn.mits., %); the specific magnetic susceptibility χ (m3/kg) was estimated using the Faraday method; the free energy of water saturation of 1 g of powder was used to estimate hydrophilicity (ΔCS, J/mol-g).

The appearance of diamond grinding powders of different grain sizes was studied using an Axioscope 5 optical microscope (Zeiss, Germany).

The structural and sorption properties of the surface of diamond grinding powders were studied by the volumetric nitrogen adsorption-desorption method at –196 °C using a Quantachrome NovaWin gas adsorption automatic analyzer (Quantachrome, USA) according to a standard procedure. All samples were degassed at 150 °C for 20 hours before measurement. The surface area by the BET method (SBET), the total pore volume (VS) at p/p0 = 0.995…0.999 and the average pore radius (width) (R) were determined from the adsorption isotherms using the NovaWin software. The pore size distribution curves were calculated from the isotherms by the QSDFT method within the standard equilibrium model for a carbon surface with slit-like pores.

Spectrometric studies of synthetic diamond powder samples were carried out in the mid-IR range (4000–700 cm–1) with a resolution of 2 cm–1. A Nicolet 6700 Fourier spectrometer (Thermo Fisher, USA) was used to record the diffuse reflectance infrared (DRIFT) spectra of the samples.

Presentation of the main material of the study. Microphotographs of individual crystals of the initial diamond grinding powders of the AC250 brand with grain sizes of 630/500 and 500/400 are shown in Fig. 2 (a, b). Analysis of the surface morphology of individual crystals and their macrostructure did not reveal significant differences between materials of different grain sizes. Most diamond crystals, regardless of size, have macrodefects - chips, inclusions, violation of face symmetry, etc.

a) **b)**

Fig. 2. **Micrographs of diamond crystals of the AC250 brand with grain sizes: 630/500 (a) and 500/400 (b).**

The results of measuring the physical and mechanical characteristics of diamond grinding powders of the AC250 brand with grain sizes of 630/500 and 500/400 are presented in Table 1.

Table 1.

Physical and mechanical characteristics of diamond grinding powders of the AC250 brand of various grain sizes

Grain size, μm	Name of indicators			
	Average particle diameter, *d*average, μm	Strength, *P*, H	Specific magnetic susceptibility, $\chi \cdot 10^{-8}$, m³/kg	Hydrophilicity, ΔC_S, J/mole-g
630/500	565	520	3,8	0,48
500/400	450	395	2,5	0,38

As follows from Table 1, diamond abrasive powders of grits 630/500 and 500/400 differ in strength by 1.3 times, which corresponds to the range of the AC250 grade for these grits. The results of the studies showed that the specific magnetic susceptibility of abrasive powders of grits 630/500 is 1.5 times greater than the specific magnetic susceptibility of diamonds of grits 500/400. The hydrophilicity of the surface of diamonds of grits 630/500 is approximately 1.3 times greater than the hydrophilicity of diamonds of grits 500/400. Since the specific magnetic susceptibility and hydrophilicity of diamond are associated with bulk and surface inorganic impurities, the presence of a correlation between strength, specific magnetic susceptibility, and hydrophilicity indicates that these impurities do not have a negative effect on the strength of AC250 diamond grinding powders.

Nitrogen adsorption-desorption isotherms for the studied samples are similar (Fig. 3). The isotherms have a close to linear form with maximum adsorption of 0.11–0.13 cm^3/g at p/p0=0.99. From the nitrogen adsorption-desorption isotherms, pore size distribution curves were calculated (Fig. 4) and structural and sorption characteristics were determined (Table 2).

The results of measuring the surface characteristics of AC250 diamond samples of different grit sizes are given in Table 2. The results obtained show that AC250 diamond grinding powders of grit sizes 630/500 and 500/400 are characterized by a small specific surface area within 0.18–0.21 m^2/g and a small pore volume of $1.75–1.99 \times 10^{-4}$ ml/g. Despite the larger size of the diamond crystals, the AC250 sample of grit size 630/500 has a larger specific surface area and pore volume compared to the sample of grit size 500/400. This result is due to the smaller average pore radius for the sample with grit size 630/500 compared to the sample with grit size 500/400.

For diamonds of the AC250 brand, regardless of grain size, a broadened maximum is observed on the pore size distribution curves at 1.57–1.64 nm (half-width of the pore) (Fig. 4). The position of this maximum shifts to the region of larger values with a decrease in the grain size of the sample. The studied samples contain a small number of mesopores of various sizes - from 2 to 32 nm.

Table 2.

Average particle diameter and structural and sorption characteristics of diamond grinding powders of the AC250 brand of various grain sizes

Grain size, μm	Name of indicators			
	Average particle diameter, daverage, m	Specific surface area, S_{BET}, m^2/g	Pore volume 10^4, V, cm^3/g	Average pore radius, R, nm
630/500	565	0,21	1,99	18,7
500/400	450	0,18	1,75	19,3

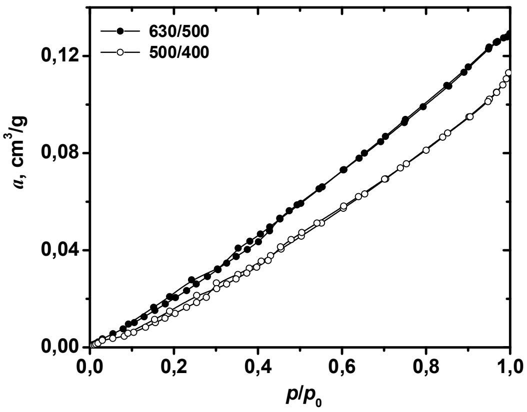


Fig. 3. **Nitrogen adsorption-desorption isotherms for AC250 diamond grinding powders of various grain sizes.**

The absence of micropores is fully consistent with the small values of the specific surface area and the ordered crystal structure of the samples. At the same time, the studied materials have a specific surface area approximately 50–70 times larger than the surface of ideal diamond crystals of the corresponding grain size. Thus, the results of adsorption studies indicate the presence of point defects in the surface layer of the samples.

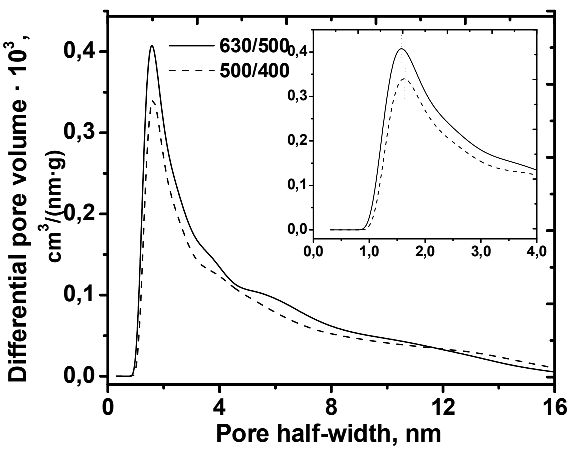


Fig. 4. **Pore size distribution curves for AC250 diamond grinding powders of various grit sizes.**

The diffuse reflectance IR spectra of the studied diamond grinding powders are shown in Fig. 5. The intense bands at 2160, 2030 and 1970 cm–1 and in the range 2540–2430 cm–1 are due to vibrations of the diamond crystal lattice [7, 8]. The spectral region 4000–3000 cm^{-1} is uninformative and the type of spectra in this region coincides with the zero line. In the studied spectra, no significant absorption was detected in the range 1570–1480, which indicates the absence of a graphitic or amorphous carbon phase. The low-intensity bands at 2925 and 2848 cm^{-1} correspond to symmetric and asymmetric stretching vibrations of the C–H bond. These bands for the 500/400 grit sample are larger than those for the 630/500 grit sample, which is fully consistent with the hydrophilicity of these samples. The intensity of these bands can be used as a measure of the hydrogenation of the diamond surface and the presence of surface C–H defects.

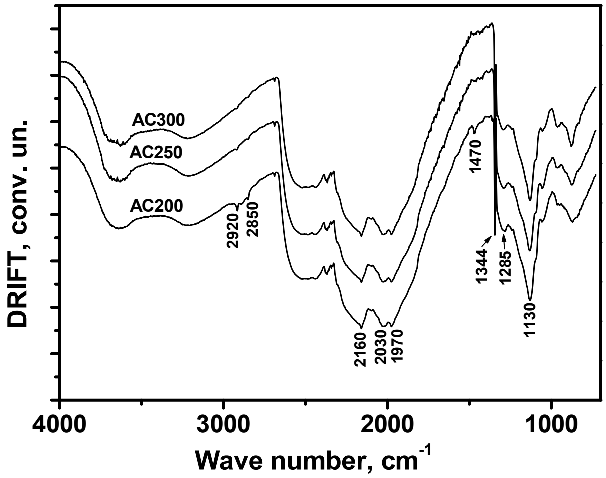


Fig. 5. **IR diffuse reflectance spectra of synthetic diamond grinding powders of AC250 grade of various grain sizes.**

A series of intense bands in the range of 1360–700 cm^{-1} corresponds to multiphonon vibrational lattices of diamond and/or nitrogen-containing clusters [9, 10]. Absorption bands at 1289, 1344 and 1130 cm^{-1} are due to nitrogen impurities in the form of the so-called A-, B- and C-centers, respectively. For yellow diamonds (type Ib) obtained by the HPHT method, the band at 1130 cm^{-1} has the highest intensity, which indicates the presence of single nitrogen atoms in the diamond crystal lattice. The relative content of nitrogen substitution defects was calculated as the ratio of the band intensity for the nitrogen-containing defect (1130, 1289 and 1344 cm^{-1}) to the intensity of the diamond band (2160 cm^{-1}) (Table 3).

Table 3.

Relative content of defects in diamond grinding powders of AC250 grade of different grain sizes

Grain size, μm	Relative content of nitrogen substitution defects		
	A-centers (1285 cm^{-1})	**A-centers (1285 cm^{-1})**	**A-centers (1285 cm^{-1})**
630/500	0,560	0,558	0,850
500/400	0,585	0,587	0,916

As can be seen from the data in Table 3, there is a clear relationship between the relative content of nitrogen-based defects and the strength of the samples (Table 1). The 620/500 grit diamond grinding powder sample has a lower content of A-, B- and C-centers and a higher strength compared to the 500/400 grit sample. Therefore, the use of diffuse reflectance IR spectroscopy to determine the relative concentration of nitrogen-based defects is the best method for predicting the mechanical properties of crystalline diamond powders of this grade.

Thus, the physicochemical and physicomechanical characteristics of diamond grinding powders of the AC250 grade with grain sizes of 630/500 and 500/400 were investigated. Based on the nitrogen adsorption-desorption isotherms, it can be stated that these diamond grinding powders have a surface area close to geometric (less than 1 m2/g), which indicates an ordered crystal structure with a small number of pores. However, despite the larger specific surface area and a larger number of point defects, the strength of the sample with grain size of 630/500 is 1.3 times higher than that of the sample with grain size of

500/400. The established correlation between strength, specific magnetic susceptibility and hydrophilicity requires further research, including for diamond grinding powders of other grades. One possible explanation for such an unusual combination of a larger number of impurities and higher strength for crystals of grain size 630/500, compared to 500/400, may be the different, depending on the grain size, distribution of impurities in diamond crystals. The presence of metallic impurities, which are located directly in the reaction zone, has a positive effect on the size of diamond crystals due to catalytic acceleration of the speed of transport processes and growth of diamond nuclei. It is likely that the high speed of carbon transfer processes during the growth of the diamond phase will contribute to the formation of more ordered crystals, in which impurities do not form intracrystalline inclusions and do not have a negative effect on the structure and strength.

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