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TECHNOLOGY FOR MACHINING HIGH-REFRACTORY CERAMIC PARTS BASED ON SILICON NITRIDE

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The results of the experimental study of mechanical treatment of highly refractory ceramic parts made of various compositions based on silicon nitride are described. The revealed regularities of the surface layer and defect formation have been used to develop a two-stage process for working these parts, which ensures high machining efficiency and a minimum level of surface layer defects. The proposed technology has been used in making cutting plates from nitride ceramics, which has increased their average resistance by 15 – 20%.

The service reliability of parts made of highly refractory silicon nitride ceramics to a large extent depends on the type and conditions of their machining [1]. These parts are usually worked by diamond grinding, which produces an enhanced force and thermal impact on their surface layer. Large stresses are formed in the surface layer of the part under loading, which generates cracks and local fractures [2]. Such technological defects significantly modify the structure and properties of the surface layer of high-refractory ceramic parts, which is eventually reflected in their service parameters. The number of defects in the surface layer of ceramic parts could be reduced by decreasing the grinding parameters and, accordingly, lowering the grinding efficiency. However, this substantially increases the cost of the parts, which in some cases is unacceptable.

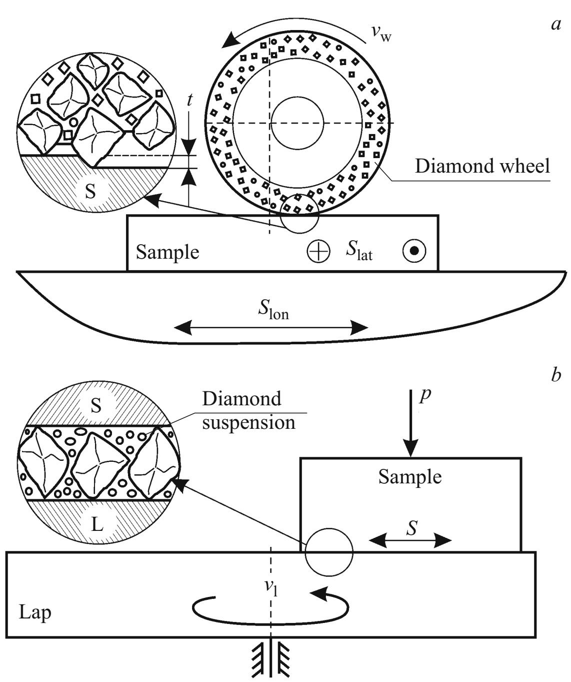

Fig. 1. Schemes of diamond grinding and free abrasive lapping of ceramic samples based on silicon nitride: v_w) rotational speed of the wheel; v_l) rotational speed of the lap; S_{lat}) lateral feed; S_{lon}) longitudinal feed; t) grinding depth, O) sample; L) lap.

A technology for treating parts made of highly refractory silicon nitride ceramics intended to minimize the number of surface defects has been developed involving a rational choice of a kinetic scheme and regimes of treatment, as well as design and parameters of diamond tools meeting the preset efficiency and service parameters of ceramic parts. The proposed two-stage process of mechanical treatment of high-refractory silicon nitride parts includes highly efficient diamond grinding (Fig. 1*a*) and subsequent lapping of the surface with a free abrasive (Fig. 1*b*). Lapping was chosen as the final stage of treatment, since it yields the best roughness parameters ($R_a < 0.05$ μm) and an optimum profile of the treated surface, which has a positive effect on the service parameters of the parts [3, 4]. Therefore, in our study we analyzed the correlation of diamond grinding and free abrasive lapping parameters with the morphology and roughness of flat surfaces, as well as the bending strength of silicon nitride ceramics of different compositions.

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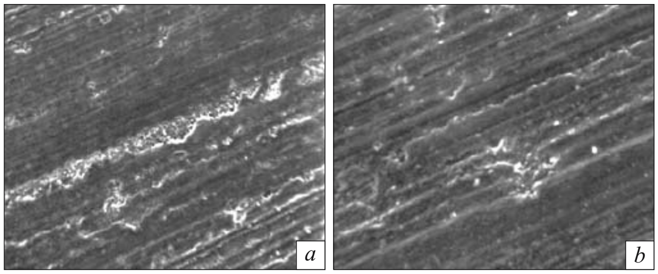

Fig. 2. Morphology of treated surface of ceramics No. 2 (*a*) and No. 3 (*b*) after grinding using wheel APP ASV125/100 K1 100% (v_w = 25 m/sec, S_{lon} = 10 m/min, S_{lat} = 1.20 mm in a double pass, and t = 0.03 mm).

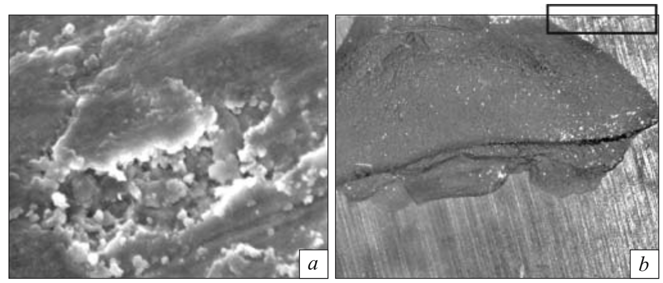

Fig. 3. Spalling on the surface and at the edge of a part made of ceramic No. 3.

Ceramic samples were ground on a 3G71 surface grinder and then lapped with a free abrasive on upgraded Neris machine. The reciprocating motion *S* and the pressure *p* (Fig. 1*b*) of ceramic samples under lapping was carried out by a specially designed mechanical device. This device made it possible to vary the pressure upon the sample within a lapping cycle. Grinding and lapping regimes varied within wide limits. In grinding we used diamond wheels APP with different parameters and a lubricant-coolant with a mean flow rate of 3 – 5 liters/min. Lapping was performed using diamond pastes ASM with different grain sizes and a working liquid (kerosene) supplied to the working zone at the rate of 15 g/min.

The allowance was uniformly removed in grinding from ceramic samples of size 6 × 6 × 50 mm fixed in a multicavity device to a size of 4.5 × 4.5 × 40 mm. Free abrasive lapping removed the allowance of 50 μm from ceramic sample surfaces. In each experimental series 15 ceramic samples were ground and lapped. The composition and main properties of the experimental ceramic materials are listed in Table 1.

The bending strength of ceramic samples was determined under three-point loading at a rate of 0.6 mm/min. The roughness of the treated surface was measured with a Kalibr 202 profilograph-profilometer. The morphology and defects of the surface layer were analyzed by optical and electron-scanning microscopy. The depth of the defective layer was measured by the consecutive polishing method, which made it possible to identify the depth of the defect propagation in the surface layer of the product.

It has been established that the topography of the polished surfaces of silicon nitride samples has numerous protrusions and grooves formed as a consequence of microcuts by diamond grains comprising the working surface of a grinding wheel. The sample surface has an oriented somewhat blurred contour, which indicates that the surface layer of nitride ceramic samples is formed not only via brittle destruction but plastic deformation as well. According to the data in [5], the thickness of the surface layer, in which residual stresses arise in grinding, is more than 5 μm; moreover, this parameter depends on the kinetic scheme of the grinding machine and the treatment regimes.

The composition and properties of nitride ceramics have a substantial effect on the morphology of ground surfaces, which is manifested in the type and intensity of their destruction (Fig. 2). This becomes especially evident comparing the morphology of the polished surfaces of samples with a different content of the intergrain phase. For instance, ceramics with 3% Y_2O_3 exhibits a highly fractured surface under grinding, whereas a ceramic sample with 15% Y_2O_3 in grinding forms a surface with a sufficiently smoothed profile.

We have identified numerous cracks on the ground surfaces of nitride ceramic samples, which propagate into samples to a depth of 40 μm (Fig. 3*a*). It is established that the cracks produce a local destruction of surface layer volumes; the depth of these sites, as a rule, is 10 – 15 μm. In certain conditions these sites merge and form local destruction grooves. The merging of the grooves and cracks at the edges of the intersecting surfaces loosens the bonds between individual surface grains. Under the effect of thermal and concentrated force loads from the diamond grains, the boundary silicon nitride grains and their conglomerates are chipped off and form a notched surface on the edges of the sample (indicated with a frame in Fig. 3*b*). In some cases nitride ceramics sample are fully or partly destroyed.

TABLE 1

Ceramic number	Composition of ceramics	Bending strength σ_b, MPa	Critical stress intensity coefficient K_{1c}, MPa · $m^{1/2}$	Heat resistance (1200 – 20°C), cycles
1	$Si_3N_4 – Y_2O_3$	658	5.4	620
2	$Si_3N_4 – Y_2O_3 – Al_2O_3$	754	6.2	890
3	$Si_3N_4 – Y_2O_3 – Al_2O_3$ – TiC	790	7.3	1060
4	$Si_3N_4 – Y_2O_3 – Al_2O_3$ – SiC	880	7.4	1120

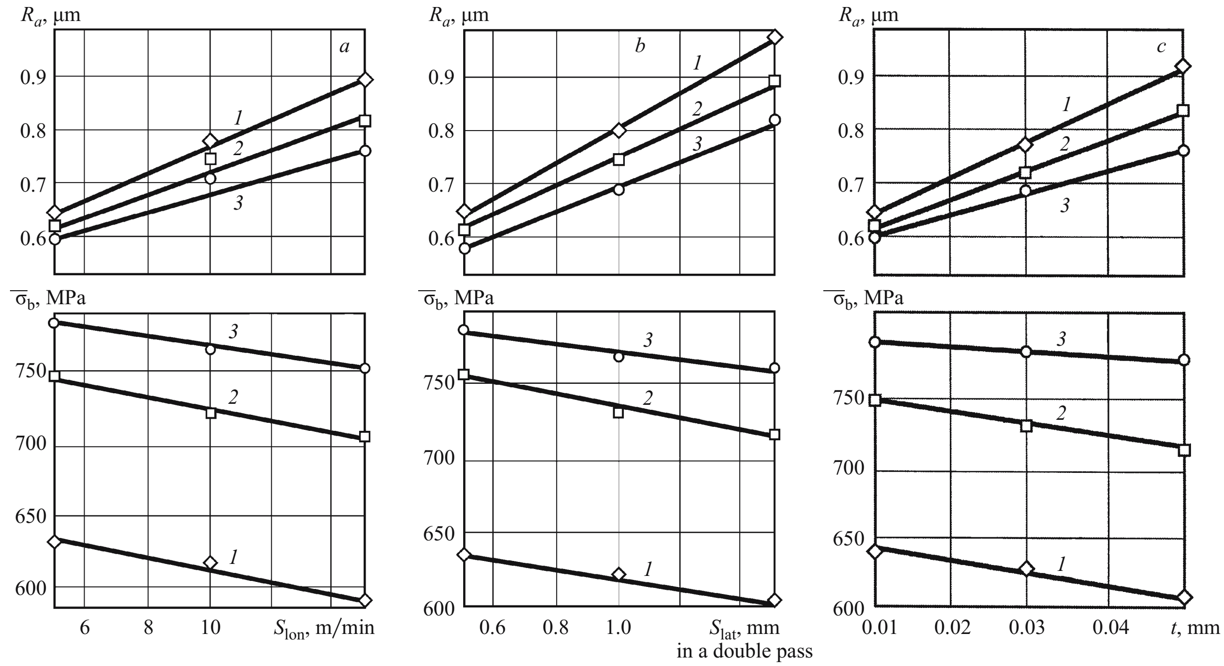

Fig. 4. The effect of longitudinal feed [*a*) S_{lon} = 1.0 mm in a double pass, t = 0.03 mm], lateral feed [*b*) S_{lat} = 10 m/min and t = 0.03 mm], and grinding depth [*c*) S_{lon} = 10 m/min, S_{lat} = 1.0 mm in a double pass] on the roughness and average bending strength $\overline{\sigma}_b$ of ceramic samples (the number of the ceramic is indicated on the curves).

The number of technological defects formed in machine treatment of ceramic parts and the depth of their propagation in the surface layer to a large extent depend on the grinding regime and the diamond wheel parameters. By way of example in Fig. 4 we show the dependences reflecting the effect of the longitudinal S_{lon} and lateral S_{lat} feed, as well as the depth t of grinding using the wheel APP ASB125/100 K1 100% (v_w = 25 m/sec) on the roughness parameter R_a and the mean bending strength $\overline{\sigma}_b$ of nitride ceramic samples of different compositions. The plots show that when the grinding regime is intensified, the surface roughness grows and the strength of the sample decreases.

The longitudinal and lateral feed, as well as the grinding depth, have virtually equal effect on the roughness of the sample surface. An increase in S_{lon}, S_{lat}, and t raises the roughness R_a from 0.64 to 1 μm. The most significant effect on the morphology of the treated surface and σ_b of nitride ceramic samples is exerted by the graininess of the wheel. An increase in the size of synthesized diamonds perceptibly increases the surface roughness R_a and decreases the σ_b of ceramic samples.

The optimum roughness of the treated surface is achieved by the wheels with diamond grains ASO, whereas the wheels with stronger grains (ASV) to some extent increase the roughness R_a of the surface. The grade of synthetic diamonds has no significant effect on the strength of nitride ceramic samples, although we observe a slight tendency to its decrease after grinding wheels with synthetic diamonds ASV compared to the wheels with grains ASO and ASR. Metallization of diamonds has virtually no effect on the roughness and strength of nitride ceramic samples.

An increase in the size of diamond grains raises the number of defects at the edges of nitride ceramic parts, increasing both the size and number of spallings. The grade of diamond grains has virtually no effect on the defects of ceramic parts at the start of the wheel operation. However, the wheels with diamond grains ASO get blunted more intensely than the grains ASV, which lowers the diamond wheel profile height. A significantly larger number of diamond grains participate in the formation of ceramic surfaces; part of these grains enter in the friction process, increasing the unit pressure and temperature. This leads to a more intense formation of defects in the surface layer of nitride ceramic parts. Therefore, the advantages of the wheels with stronger diamonds (ASV) in grinding nitride ceramics become obvious.

Experimental results were used to construct a regression model relating the initial average bending strength $\overline{\sigma}_{b\,in}$, heat resistance TC_{1200}, the stress intensity coefficient K_{1c} of sintered ceramics based on silicon nitride, the depth t of the longitudinal S_{lon} and lateral S_{lat} feeds in grinding, and the grain size G of the diamond wheel to the average bending strength $\overline{\sigma}_b$ of samples after grinding. This regression model has the following form:

$$\overline{\sigma}_b = 1.18\overline{\sigma}_{b\,in} - 31.42S_{lon} - 2.82S_{lat} - 641t - 0.07G - 68.17.$$

Analysis of the regression model shows its adequacy and high regression significance: multiple correlation coefficient 0.096, determination coefficient 0.992, corrected determina-

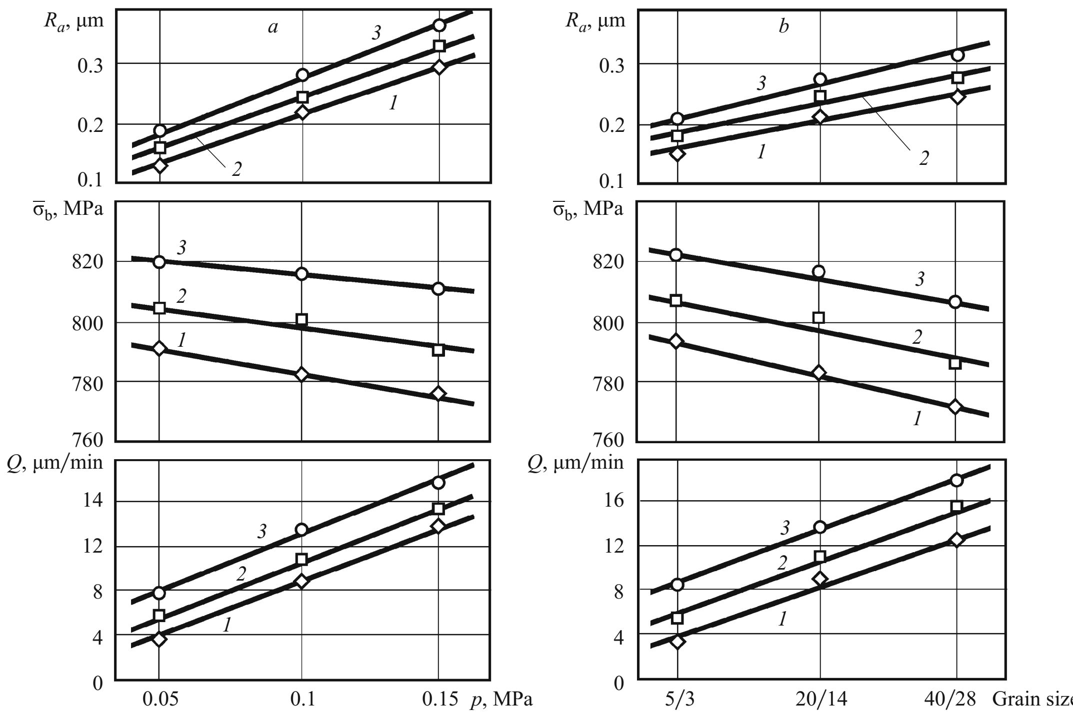

Fig. 5. The effect of pressure p (a) in lapping with free abrasive ASM 20/14 (rotational speed n = 100 rev/min) and grain size (b) of diamond paste (n = 100 rev/min, p = 0.1 MPa) on R_a, $\bar{\sigma}_b$, and Q of ceramic lapping process (the number of the ceramic is indicated on the curves).

tion coefficient 0.99, F-criterion (5.23) — significance level 615.7 — $p < 0.0000$, and standard estimate error 6.68. The pairwise correlation coefficients are given below:

	$\bar{\sigma}_{b\,in}$	TC_{1200}	K_{1c}	S_{lon}	S_{lat}	t	G
$\bar{\sigma}_b$	0.983	0.974	0.92	– 0.093	– 0.104	– 0.084	– 0.163

Analysis of the pairwise correlation coefficients revealed the following. The initial bending strength and heat resistance of silicon nitride ceramics have the maximum effect on the properties of samples after grinding, The stress intensity coefficient K_{1c} of sintered samples has a lower effect on the strength of these samples after grinding. The grain size of diamond wheels has a perceptible effect on $\bar{\sigma}_b$ of nitride ceramic samples. As for the grinding regimes, the biggest effect on $\bar{\sigma}_b$ of silicon nitride ceramics after grinding is exerted by the lateral feed and the lowest effect by the grinding depth.

Thus, the study of diamond grinding of nitride ceramic samples indicates that their mean bending strength decreases after grinding. This is related to the nature of the emerging surface layer and the propagation of technological defects to a depth of 40 μm. A decrease in the grinding parameters and the use of dwelling passes to a limited extent can influence the defective layer thickness and bending strength of nitride ceramics. For instance, the bending strength of ceramic sample No. 1 that had been ground (v_w = 25 m/sec, S_{lon} = 10 m/min, S_{lat} = 1.0 mm) in a double pass with t = 0.03 after three dwelling passes with t = 0 grew from 616 to 621 MPa. However, these measures are not effective in the technical and economical aspects, since the increased production cost does not provide a corresponding increase in their service parameters.

Free abrasive lapping offers significantly greater possibilities for controlling the state of the surface layer in nitride ceramic samples. The process involves a complex interaction between the lap, the diamond grains (unfixed and charging the lap), the working liquid, and the ceramic part, which contacts the lap through a diamond suspension layer. All elements of the system have low mutual migration velocities; therefore, the contact of the diamond grains with the ceramic part surface occurs at normal temperature. The absence of thermal stresses in the surface layer and the low contact pressure make it possible to meet the high requirements imposed on the surface layer quality and on the precision of the sizes and shape of the treated ceramic surfaces.

The results of studying the effect of the technological parameters of free abrasive lapping of ceramic sample No.3 on the treated surface roughness R_a, the mean bending strength $\bar{\sigma}_b$, and the process efficiency Q are shown in Fig. 5. Analysis of the data shows that when the pressure p exerted on the samples in lapping increases, the surface roughness R_a and the process efficiency Q grow, while the $\bar{\sigma}_b$ of ceramic samples decreases (Fig. 5a). This is related to the growing number of diamond grains positioned in the state of limited mobility between the sample and the lap. As a consequence, characteristic local destruction grooves are formed on the sample surface.

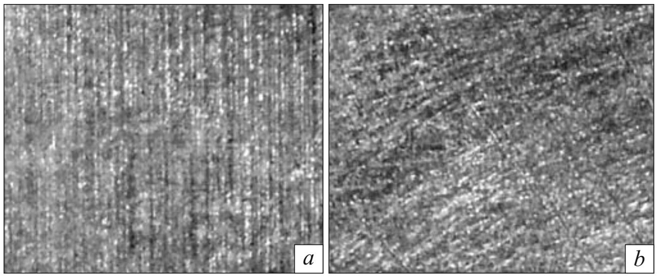

Fig. 6. The state of the surface of sample No. 3 after grinding with a diamond wheel (*a*) and free abrasive lapping (*b*).

A decreasing grain size of the diamond paste ASM decreases the roughness R_a and the process efficiency. It should be taken into account that the diamond suspension in lapping loses its cutting properties due to the wear and destruction of the diamond grains and their charging the lap. As the result, the low efficiency of this process decreases even more. However, in this case σ_b of the samples registered an increase; moreover, in some cases the strength of the samples after lapping exceeded the strength of the sintered samples.

It has been established that it is possible to raise the process efficiency while ensuring the high quality of the surface layer of ceramic samples by a periodic pressure variation according to the following scheme: a gradual pressure increase from 0 to p_{max}, a working cycle at p_{max}, a gradual decrease from p_{max} to p_{dw}, and dwelling at p_{dw}. Such procedure ensures an effective removal of the main allowance (the defective layer formed in grinding) in a short time and satisfies the prescribed lapping parameters. The uniform wear of the lap is achieved by choosing an appropriate path for the motion of ceramic preforms over its working surface.

By way of example Fig. 6 shows the surface of ceramic sample No. 3 after grinding with a diamond wheel and after the additional free abrasive lapping.

Thus, the proposed two-stage process ensures high efficiency of mechanic treatment of highly refractory parts based on silicon nitride with a minimum level of surface defects. The use of this technology for producing cutting plates of nitride ceramics in two classes of tolerance has made it possible to lower wear intensity and to improve the operating stability at the initial stage of service. This has increased the average resistance of the cutting plates by 15 – 20% compared to the resistance of cutting plates ground according to the traditional technology.

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