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OPTIMIZATION OF HOT-DEFORMATION MODES FOR SILICON NITRIDE CERAMICS

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The aim of this article is to develop silicon nitride ceramics with improved cutting properties by optimizing their composition and microstructure.

Experimental Procedure

The preparation and investigation of Si_3N_4-based ceramics included the next stages. The first step included the choice of composition of Si_3N_4-based ceramics. The composition of silicon nitride-based ceramics with additives is given in Table 1.

Table 1

The composition of silicon nitride-based ceramics

Si_3N_4, wt.%	Y_2O_3, wt.%	Al_2O_3, wt.%	Additives, wt.%
92	5	3	–
92	5	3	–
87	5	3	5% B_7O
82	5	3	10% B_7O
72	5	3	20% TiN
72	5	3	20% TiB_2
52	5	3	40% ZrB_2
82	5	3	10% ZrN
82	5	3	10% TiCN

Grinding of mixture of certain composition were done in a ball mill for 5 h. Grain size of powders was measured by a laser micron sizer. Grain size distribution and shape of powders were observed by scanning electron microscopy.

Samples for investigations and tool materials from Si_3N_4 (HCStarck LC10) and additive powders (Table 1) were prepared by uniaxial hot pressing: T = 1750–1870°C; t = 15–30 min; P = 30 MPa. The graphite dies were used for the hot pressing without protective atmosphere.

The hardness of the Si_3N_4-based ceramics was measured using a Vickers indenter with a load of 196 N. The hardness is the average of 6 indentations for each sample. The indentation fracture toughness K_{1c} was determined according to [1]. The four-point bending strength σ_f was determined with chamfered bars (3.5 × 5 × 40 mm) at a load speed of 0.2 $mm{\cdot}s^{-1}$. The present values of σ_f are the average of three results for each material. The Young's modulus E was determined at room temperature by measuring the deflection of samples during bending tests according to the ASTM 855-90 standard. Optical and scanning electron microscopy were used for a microstructure investigation. X-ray analysis and micro-Raman spectroscopy were used for the phase characterization.

The mechanisms of wear in extreme cases for the performance of Si_3N_4-based cutting tools were investigated. The most favorable case is grey cast iron machining and the trial tests were tool steel machining. Grey cast iron and high-carbon tool steel were selected for the wear and cutting tests.

A technique of investigating the service properties of cutting tools during the turning process is developed. An experimental technological system which consists of an universal turning thread-cutting lathe (model 16K20) with height of centres i = = 200 mm and the maximum length of a bar under machining l = 710 mm has been created. The methods which allow a user to estimate cutting-force components affecting the tool under half-time processing conditions and the stresses arising in the cutting wedge were developed.

The technique based on parameter determination of tool relative durability has been developed. This parameter is determined by construction subjection of cutting path from time for specific criterion bluntness. To construct the adequate mathematical models which show the influence of cutting modes on tool durability, mathematical methods of planning of multifactor investigations were used. In this case, the experimental determination of the period of tool durability for specific cutting model unaffected in the process of manufacturing the given tool has to be done.

Cutting test with grey cast iron and annealed tool steel were carried out without coolant. Average cutting speeds were calculated by account for the workpiece radius reduction at constant angular speed. The removal rate Q is the product of the cutting speed, the feed (0.1 mm) and the depth of cut (DOC = 0.35 mm). The volume of removed material M was calculated as Qt, were t is the time when flank wear reaches 0.35 mm or 0.45 mm.

Scientific Results

Phase analysis is made by X-ray diffraction technique using diffractometer DRON-2. Silicon nitride in all samples is shown to be in stable β-state. Addition of strengthening phases does not influence the β-state stability. Lattice parameters of Si_3N_4 are about theoretical values.

Relative densities and microstructural parameters are given in Table 2.

Strength, hardness and fracture toughness of Si_3N_4–B_7O ceramics are lower than those of silicon nitride-based ceramics without B_7O (Table I). Presence of Si and B phases as well as a high porosity of materials can explain such a decrease. Mechanical properties of Si_3N_4-based ceramics with additives are given in Table3.

Table 2

Relative densities and microstructural parameters of ceramics

Composition	Relative density	Grain size, μm
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)	0.98	1.6
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)	0.99	1.4
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–5% B_7O	0.94	2.1
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–10% B_7O	0.96	2.0
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–20% TiN	0.99	2.4
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–20% TiB_2	0.96	2.6
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–40% ZrB_2	0.99	1.5
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–10% ZrN	0.99	1.4
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–10% TiCN	0.98	2.7

Taking into account the results of structural analysis and mechanical tests the main attention was paid to investigation of systems β-Si_3N_4 + TiN and β-Si_3N_4 + + $TiC_{0.65}N_{0.25}B_{0.1}$. This compound has a high resistivity to oxidation and forms favourable phase structure in the silicon nitride composition.

Composites of β-Si_3N_4 + TiN and β-Si_3N_4 + TiC are most frequently used for particulate reinforcement of silicon nitride matrix [2,3]. It was shown by O.N. Grigoriev [4], that the thermal expansion mismatch between added particles and Si_3N_4 matrix will produce compression stresses in the matrix during cooling from high temperature. The introduction of TiN particles into silicon nitride matrix can increase its fracture toughness, hardness, and Young's modulus.

Table3

Mechanical properties of Si_3N_4-based ceramics

Composition	σ_f, MPa	E, GPa	H_v, GPa	K_{1c}, MPa·$m^{0.5}$
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)	723	315	13.45	5.95
Si_3N_4 (5%Y_2O_3–3% Al_2O_3) - 5% B_7O	520	221	10.3	4.7
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–10% B_7O	358	182	7.95	3.2
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–20% TiN	600	323	12.68	7.7
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–20% TiB_2	450	260	–	–
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–40% ZrB_2	–	–	16.4	4.9
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–10% ZrN	–	–	15.43	6.8
Si_3N_4 (5%Y_2O_3–3% Al_2O_3)–10% TiCN	533	333	13.52	7.0

It was found by X-ray analysis of Si_3N_4–TiN specimens, that TiN is characterized by an increased lattice parameter compared to tabular data. The lattice parameter increasing is more pronounced in titanium carbonitride with boron additions. The discovered effect seems to be associated with tensile stresses in analysed phases. One should note that matrix compression is accompanied by residual stresses in TiN and

TiCN particles. This can result in particle microcracking and bending strength decreasing owing to microdefects formation.

The alteration of the Si_3N_4 microstructure by the dispersed additions may then offer a potential for improvement of the composite's mechanical properties at room temperature. High temperature effect may be adverse [5]. In the earlier work on silicon nitride it has been demonstrated that oxidation may have a profound influence on the microstructure of these materials by modification of the intergranular phase. This phase plays a crucial role in the creep behaviour of these materials. But Yu. Gogotsi et al. [8] have shown that the presence of fine isolated TiN particles in the silicon nitride matrix (< 30 wt.% TiN) only slightly increases the creep rate.

Capability of such additives to form a protective oxide layer under conditions of high-temperature treatment is the important advantage of Si_3N_4-ceramics [6]. It is well known that turning of Fe-based alloys is accompanied by the considerable temperature rise of processed pieces and cutting tool up to ~ 1200°C. This effect is especially characteristic of steel treatment. In this case the cutting process is accompanied by intensive dislocation shears; the chip looks as continuous tape, that makes heat abstraction from cutting edge essentially difficult.

The cutting parameters for «ceramics–cast iron» and «ceramics–steel» cutting tests are presented in Figs. 1,*a* and 1,*b*. Cutting tools made of nitride ceramics with TiN and TiCNB additions compare favourably with our base composite and industrial cutting instrument («Elektrobosna», Yug.). In «ceramics–cast iron» cutting tests (Fig. 1,*a*) the cutting time increases by a factor of 2 and 3.5 for TiN and TiCNB, respectively. «Ceramics–steel» cutting tests show pronounced improvement of wear behaviour of the Si_3N_4-based tools with TiN and TiCNB additives (Fig. 1,*b*). Particularly, weight of removed material is 2.4 and 3.1 kg at flank wear of 0.3 mm for the developed cutting tool, whereas this value is 0.16 kg for base material.

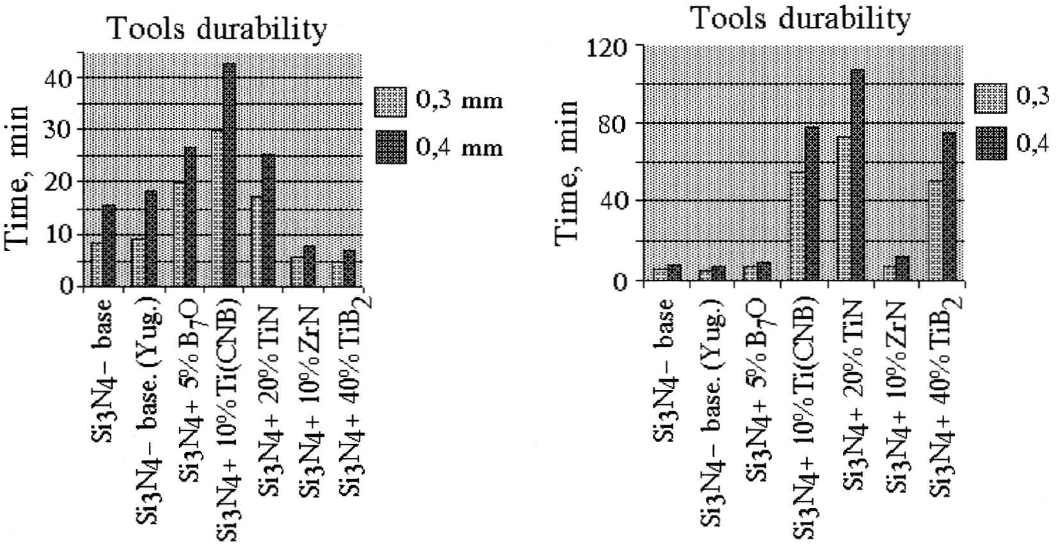

Fig. 1. Silicon nitride-based ceramic tools durability: ceramics–cast iron cutting tests (*a*); ceramics–steel cutting tests (*b*)

The results obtained corroborate the proposed hypothesis about the cutability increase of instruments containing TiN and TiCN, due to enhancement of their low-temperature properties as well as resistance to high-temperature corrosion and chemical wear.

Conclusion

Results of this article corroborate the proposed hypothesis about the cutability increase of instruments containing TiN and TiCN due to enhancement of their low-temperature properties, as well as resistance to high-temperature corrosion and chemical wear.

1. *A.G. Evans*, Mater. Sci. Eng. **71**, 3 (1985).
2. *G.G. Gnesin, I.I. Osipova, G.D. Rontal, V.P. Yaroshenko*, Ceramic Tool Materials, Tehnika, Kiev (1991) (in Russia).
3. *V. Yaroshenko, Yu. Gogotsi, I Osihova*, in: Ceramics Today – Tomorrow's Ceramics, P. Vincenzini (ed.), Elsevier, Amsterdam (1991), pp. 2821–2830.
4. *O. Grigorjev, Yu. Gogotsi, V. Yaroshenko*, Silikattechnik **41**, 156 (1990).
5. *P. Peni, J. Crampon, R. Duclos, B. Cales*, J. Eur. Ceram. Soc. **8**, 311 (1991).
6. *Yu. Gogotsi, G. Grathwohl*, J. Mater. Sci. **28**, 4279 (1993).