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Wear-Resistant TiN – Si_3N_4 Nanocomposites Consolidated By Electric Discharge Sintering

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Dense nanocomposites on the basis of silicon nitride with the size of structural elements less than 150 nm have been consolidated by Electric Discharge Sintering. The improvement of materials properties is expected: wearproofness, fracture toughness (~ 5 – 6 MPa·м$^{1/2}$), hardness (~ 22 GPa), bend stress (800-1100 MPa), stability to the aggressive environments (acids at a room and high temperatures) as compared to traditional materials which now use as frictional unit.

Keywords: Electric discharge sintering, Nanocomposite, Nanorod, Hardness, Linear wear.

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1. INTRODUCTION

Electric Discharge Sintering (EDS) is a very fast method to consolidate different materials via direct heating of press instrument together with green pellet by pulse electric current[1, 2]. Presence of pressure makes this method similar to the hot pressing one. The typical scheme of EDS consists of two stages: preliminary and the main. At the preliminary stage relatively low pressure is used to treat the powder. On this stage interparticle contacts play the main role as local overheating takes place here. On the second stage the sample is subjected to a higher pressure; on this stage the sample is heated by Joule heat. The EDS features help to decrease the process time and temperature as well as to consolidate nanomaterials.

2. EXPERIMENTAL

In our investigation we use silicon nitride and titanium nitride nanopowders (PCT Ltd., Latvia), nanorods of silicon nitride (Nanoamor inc., USA) and their mixtures. Initial powder properties of the nanopowder compositions used in this research are summarized in Table 1.

Table 1 – Properties of the initial nanopowders

Nanopowder	Specific surface, m²/g	Average, nm	Chemical analysis, wt. %	
			[O]	[N]
TiN	25,7	~ 70	1,81	19,2 ± 0,3
Si_3N_4 nanopowder	70	30 – 50	2,1	38,4 ± 0,3
Si_3N_4 nanorod	103	d = 30-70 l =300 – 800	< 1	-

Mixtures of TiN (nanoparticle) - Si_3N_4 (nanoparticles) - Si_3N_4 (nanorods) were prepared by mixing in the planetary mill (Pulverizette-6, Fritch, Germany), 600 rpm, 4 h. EDS experiments have been carried out using SPS apparatus FCT-HP D 25 manufactured by FCT Systems GmbH (T_{max} = 2400 °C, P_{max} = 250 kN, I_{max} = 8000 A, U_{max} = 10 V, mediums: vacuum – 5·10^{-2} mbar, nitrogen).

Table 2 – Nanocomposites consolidated by EDS

№	Nanocomposite	P, MPa	Heating rate, °C/min.	T, °C	Soak time, min.	ρ, g/cm³	Open porosity, %
1	38,1 wt. % Si_3N_4 – 57,1 wt.% TiN - 4,8 wt. % Si_3N_4 rods	50	200	1700	1	3,400	0,0
2	16,6 wt. % Si_3N_4 – 66,6 wt. % TiN – 16,8 wt. % Si_3N_4 rods	50	200	1600	1	3,140	0,0
3	87,5 в wt. % Si_3N_4 – 12,5 wt. % TiN	50	200	1700	1	2,462	21,7

Regimes of EDS consolidation and characteristics of the sintered nanocomposites are presented in table 2.

The X-ray diffraction method (XRD) was used for the qualitative phase analysis (XRD – 7 with Cu Kα radiation, Seifert-FPM, Freiberg, Germany). The particle size distribution of investigated nanopowders was estimated by a light scattering particle size analyzer (Zetasizer 1000 HS, Malvern Instruments, United

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Kingdom) and by a transmission electron microscope (JEOL JEM-2100F, Japan). Vickers hardness and fracture toughness of sintered composite ceramics were measured at 50 g and 2 kg load conditions by MMT-3 (Buehler, USA) hardness testers. Microstructure of the polished surface of sintered composites was examined by a Field Emission Scanning Electron Microscopy NVision 40 (Carl Zeiss SMT AG, Germany) and HD 2700 (Hitachi, Japan). Density of the SPS consolidated nanocomposites has been measured by Archimedes method in deionised water at room temperature.

3. RESULTS

The typical microstructure of EDS consolidated TiN - Si_3N_4 nanocomposites is presented on fig. 1. As one can observe, grains of TiN (average size 100 – 150 nm) and Si_3N_4 (average size ~ 70 nm) are distributed homogeneously in the nanocomposite structure.

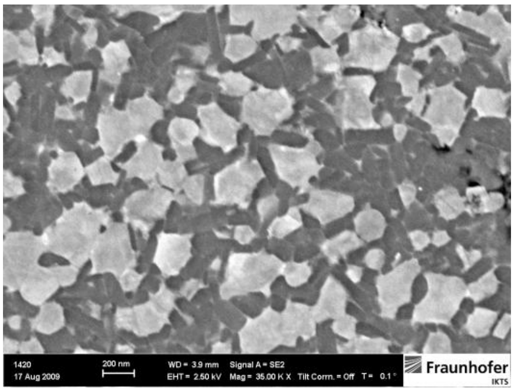

Fig. 1 – Microstructure of EDS consolidated TiN – Si_3N_4 nanocomposite

Consolidated nanocomposites were tested on wear resistance at unlubricated friction in the pair with steel 15. All composites with Si_3N_4 nanorod additives showed good wear resistance and relatively low friction coefficient (Fig. 2-3).

It should be mentioned that the wear of friction pair is mainly determined by the wear of counterbody: the gravimetric wear of samples makes ~ 0,3-0,5 mg/km and depends on loading, and a gravimetric wear of counterbody is in a range of 1-5 mg/km and increases with loading increase. The most preferable composite to use in the condition of high loads and wear in pair with steel 15 is composition 16,6 wt. % Si_3N_4 – 66,6 wt. % TiN – 16,8 wt. % Si_3N_4 rods. For the composite the coefficient of friction and linear wear are the lowest.

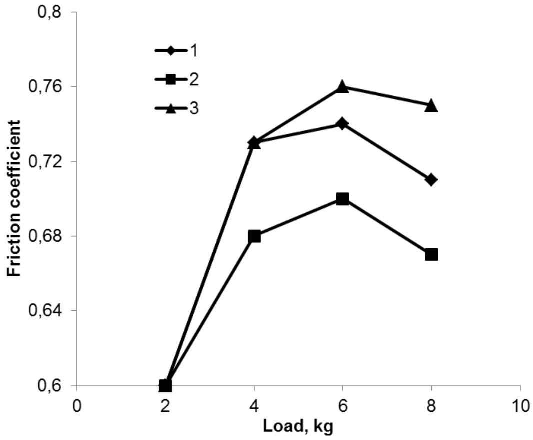

Fig. 2 – Friction coefficient as a function of load at V = 0,5 m/s

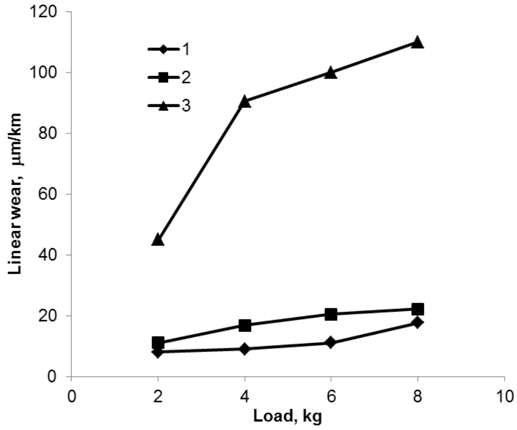

Fig. 3 – Linear wear as a function of load at V = 0,5 m/s

4. CONCLUSIONS

Dense nanoceramics on the basis of silicon nitride with the size of structural elements less than 150 nm are consolidated by EDS. The improvement of materials properties is expected: wearproof in the pair with steel 15, hardness, bend stress as compared to traditional materials which are now used as frictional units.

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