Mechanical properties of hot-pressed Si_3N_4-TiN grain composites

Właściwości mechaniczne kompozytów ziarnistych Si_3N_4-TiN otrzymywanych metodą HP

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Dense Si_3N_4-TiN composites were prepared from commercial powders using hot pressing method. Titanium nitride grains were homogenously distributed in material's microstructure. Flexural strength of materials increased with TiN amount and reached 880 MPa. Fracture toughness of materials varied between 7 and 8 MPa m^{0,5}.
KEYWORDS: composites, titanium nitride, silicon nitride, mechanical properties

W pracy zbadano właściwości mechaniczne kompozytów ziarnistych Si_3N_4-TiN otrzymywanych metodą prasowania na gorąco z komercyjnych proszków. Drobnoziarnisty azotek tytanu był homogenicznie rozmieszczony w mikrostrukturze spieków. Zaobserwowano poprawę wytrzymałości na zginanie materiałów. Odporność na kruche pękanie kompozytów nie uległa poprawie.
SŁOWA KLUCZOWE: kompozyty, azotek krzemu, azotek tytanu, właściwości mechaniczne

Silicon nitride is a common structural ceramic material for wear and high temperature application. Its good mechanical properties, wear resistance and reliability at room and high temperature enable several applications for example cutting tools, engineering components, crucibles for molten metal, ball bearings [1]. To improve mechanical properties of Si_3N_4 based materials some other ceramic phase can be added to silicon nitride matrix. Titanium nitride and silicon carbide are most often used for this application [2, 3]. Moreover addition of a conductive phase may enable EDM machining, which is great alternative in case of hard, difficult to machine materials [4, 5]. Hot pressing seems to be very effective way to achieve dense silicon nitride based composites with good mechanical properties [6]. Addition of rather coarse TiN (3÷6 µm) is reported to increase fracture toughness of Si_3N_4-TiN composites, but to decrease flexural strength [7]. Addition of fine titanium nitride to silicon nitride lowers friction coefficient and wear rate [8]. In this research mechanical properties of hot pressed Si_3N_4-TiN grain composites consisting of fine TiN grains were measured.

Materials and methods

■ **Samples preparation.** Composites with varying volume friction of TiN (Tab. 1) and constant amount of sintering additives (6 wt.% of Al_2O_3 and 4 wt.% of Y_2O_3 count to the amount of Si_3N_4) were hot pressed from the following commercially available powders: silicon nitride (SILICUM NITRIDE Si_3N_4 GRADE M 11 HP, H.C. Starck GmbH), titanium nitride (TITANIUM NITRIDE TiN GRADE A H.C. Starck GmbH), aluminium oxide (Taimicron TM-DAR, TAIMEI CHEMICALS CO. LTD), yttrium oxide (Y_2O_3 GRADE C, H.C. Starck GmbH). Titanium nitride powder was milled in high-energy rotary-vibratory mill using Si_3N_4 milling media in the isopropanol alcohol environment for 15 h to obtain average grain size around 1 µm. Proper amounts of powders were then homogenised in high-energy rotary-vibratory mill in conditions mentioned above, for 1.5 h. Compositions of prepared materials are listed in the table. Homogenized and dried mixtures were hot-pressed (Thermal Technology LLC) at 1750 ºC for 1 h under 25 MPa in nitrogen flow. The heating rate was 10 º/min and the cooling rate was resulting from thermal inertia of furnace. Sintered bodies with a diameter of 75 mm were obtained.

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TABLE. Composition and density of samples

Sample	TiN, vol.%	Si_3N_4 (6 wt.%Al_2O_3 4 wt.%Y_2O_3), vol.%	Density, g/cm^3
Si_3N_4	0	100	3,20±0,01
5TiN	5	95	3,30±0,01
10TiN	10	90	3,39±0,01
15TiN	15	85	3,52±0,01
20TiN	20	80	3,58±0,03
30TiN	30	70	3,81±0,01
40TiN	40	60	4,03±0,01

■ **Samples examination.** Densities of samples were determined using Archimedes method. Phase composition of sinters was analysed using X-ray diffraction (Philips equipment with X-Pert HighScore software). Microstructure of materials was observed on polished samples using SEM technique (FEI Nova Nano SEM). The EDS method was used for point element distribution to identify phases in the material microstructure. Banding strength was measured on polished beams using three point bending method. Fracture toughness was measured using double-torsion method.

Results and discussion

■ **Microstructure and phase composition.** The densities of materials are listed in the table. The XRD analysis revealed presence of two crystalline phases in all composite samples after sintering: β-Si_3N_4 and TiN. In reference sample only β-Si_3N_4 phase was detected (Fig. 1). Sintering additives probably formed glassy phase during cooling.
Results of EDS analysis and XRD phase analysis enabled identification of phases visible in microstructure (Fig. 2). White grains are titanium nitride and grey grains are silicon nitride.

Exemplary microstructures are shown in Fig. 3. Materials were fully dense. Titanium nitride grains were well dispersed in sinters. Elongated silicon nitride grains were surrounded by amorphous oxynitride phase.

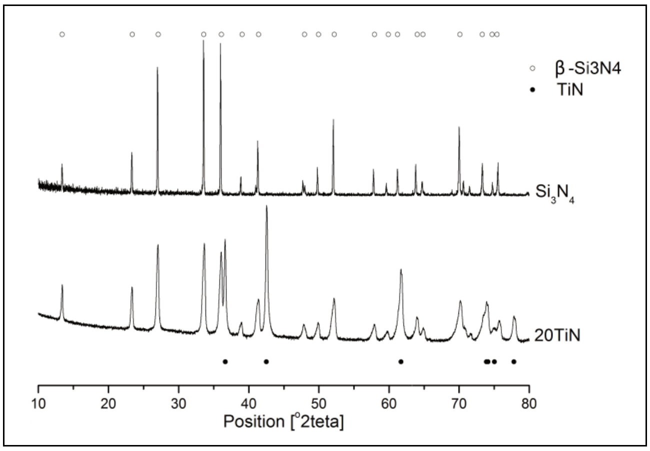

Fig. 1. XRD analysis of 20TiN sample

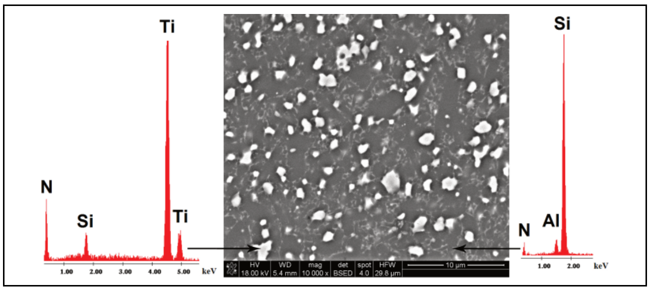

Fig. 2. SEM microstructural observation and EDS analysis of 15TiN sample

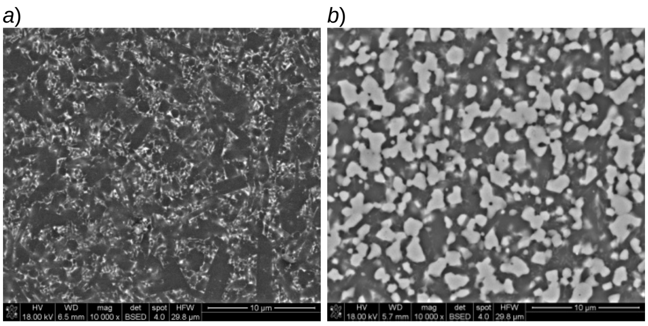

Fig. 3. SEM observations of *a*) Si_3N_4, *b*) 40TiN samples

Mechanical properties. Fracture toughness and flexural strength of materials are presented in Fig. 4. Fracture toughness of composites was lower than fracture toughness of reference sample. Presence of fine TiN grains did not caused the elongation of crack propagation Fig. 5. Flexural strength was higher for composites and increased with TiN addition.

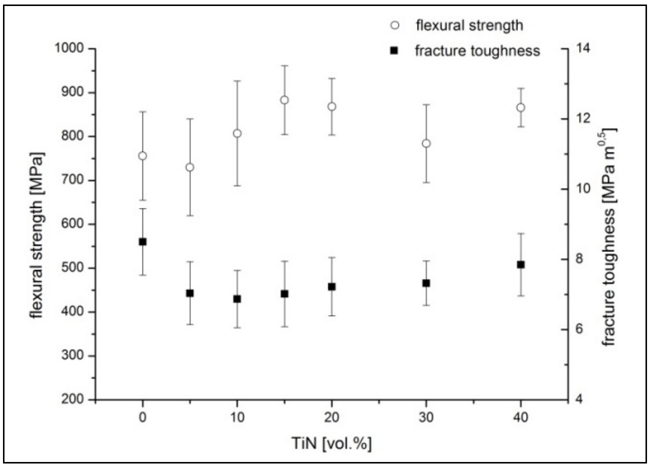

Fig. 4. Mechanical properties of materials

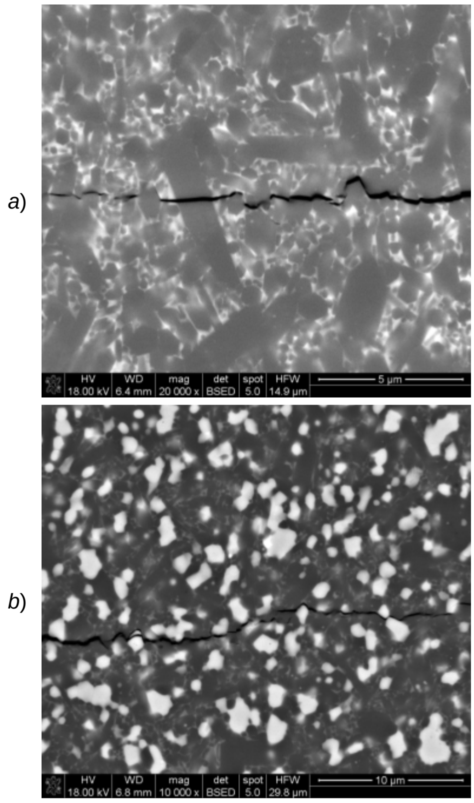

Fig. 5. Crack propagation *a*) Si_3N_4, *b*) 20TiN

Conclusions

Dense Si_3N_4-TiN grain composites were sintered from commercial powders. Usage of fine TiN powder resulted in increase in flexural strength of composites. Fracture toughness of composites was lower than fracture toughness of reference sample.

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