FRACTURE TOUGHNESS BEHAVIOR OF ALUMINA MATRIX COMPOSITES AT ELEVATED TEMPERATURE

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1. Introduction

Advanced ceramics products usually offer superior performance that cannot be replicated by other materials. Despite the relatively small sales advanced ceramics products form an integral part of modern technology. Advanced ceramics are materials tailored to obtain exceptional properties (superior mechanical properties, corrosion/oxidation resistance, thermal, electrical, optical or magnetic properties) by controlling their compositions and internal microstructure. So, they are subdivided into structural ceramics, electrical ceramics, ceramic coatings, chemical processing and environmental ceramics. Structural ceramics include wear parts, cutting tools, engine components and bioceramics [1]. According to analysis of marked report research the total technical and advanced structural ceramics marked in USA was worth $3.2 billion in 2010 and $3.4 billion in 2011. This figure is projected to reach $4.4 billion in 2016 yielding a compound annual growth rate (CAGR) of 5.1 between 2011 and 2016 (Fig.1) [2].

In 2011, the global market for ceramic cutting tools was over $1 billion and equaled 8.5% of the total market for metal-cutting tools (Fig.2).

Relative growth rates will be stronger than that of hardmetals, high-speed steel (HSS) and cermet tools, but lower than the growth rates for super-hard tools such as polycrystalline cubic boron nitride (PcBN) and diamond (PCD and single crystal). By 2016, the global market for ceramic cutting tools is expected to reach $1.5 billion [3]. Ceramic cutting tools are constructed mainly from alumina (Al2O3) and silicon nitride (SiN). Recent advances have also introduced the use of silicon carbide (SiC) and ceramic matrix composites (CMCs) in order to enhance the performance of the cutting tool. Each of these ceramic materials has its own particular characteristics, but, in general, they all exhibit excellent hardness, toughness and thermal conductivity. In fact, the advantages of using ceramic materials in manufacturing revolve around ceramic’s greater ability to withstand much higher
temperatures than tools made from hardmetals or high-speed steel. Generally speaking, ceramic tools’ heat resistance exceeds 2200°C vs. about 870°C for tools made from carbide powder. In addition, ceramic’s ability to operate at higher temperatures can result in a softening of the workpiece material, which allows for deeper and cleaner cuts. Thus, ideal machining temperatures can be accommodated by ceramic. However, these high temperatures are unattainable for hardmetals tools because they exceed the melting point of the cobalt binder.

The manufacturing of ceramic tools also offers advantages over typical manufacturing of hardmetals tools in that they are typically prepared using powder metallurgy techniques and can be manufactured closer to near-net finish, thus saving the tool manufacturer hard machining costs. The main drawback of ceramic tools is that they are brittle and do not withstand thermal shock very well, which means that they are better applied to continuous, long-run applications. Cutting tool ceramics are used in thermal and structural applications requiring high temperature resistance, high hardness and chemical inertness. Ceramic composite design can also be used to tailor other important properties such as high-temperature strength and thermal shock resistance, wear resistance, friction, thermal and electrical conductivity. By an appropriate adjustment of the concentration of its components, alumina matrix composites (AMCs) can offer a good strength properties, high hardness, wear resistance and chemical inertness but their brittleness (low value of fracture toughness) usually leads to a short tool life [4]. Initially, only alumina ceramics (oxide ceramics) were used but to improve their toughness level alumina matrix composites are reinforced with a wide range of ceramic phases including TiC, TiN, ZrO₂, WC, Ti(C,N), SiC, TiB₂ [5,6,7]. In the early 1970’s aluminum oxide/titanium carbide composites (carboxide ceramics) were introduced. They provided improved results in finish turning of ferrous metals and turning of hard ferrous metal. Optimization of the composition including the introduction of new sintering technologies resulted in further improvements in this group of cutting tool materials. Titanium nitride (TiN) and carbide (TiC), classified as refractory metal compounds, possess an unusual combination of physical properties. Both TiN and TiC are extremely hard, have very high melting points and exhibit metallic-like electronic conductivity [8]. These properties make them attractive both fundamentally and technologically, and are closely connected with their electronic structure. Titanium carbide (TiC), is ceramics with NaCl-type face centered cubic crystal structure-cF8 with octahedral coordination geometry (Fig.3) [9].

Other properties, such as high hardness (34 GPa), low density (4.93 g/cm³), relatively high thermal conductivity (30 W/mK), Young’s modulus 439 GPa cause these materials attractive for cutting tools [10]. Titanium nitride (TiN) with high thermal and mechanical stability exhibits the Vickers hardness values of about 18-19 GPa, modulus of elasticity of 251 GPa and thermal conductivity about of 19 Wm⁻¹K⁻¹ and is used together with TiC to improve mechanical properties of pure alumina. The crystal structure of TiN is cubic (cF8), while the coordination geometry is octahedral (Fig.4) [11].

![Fig.3. Crystal structure of TiC (C atoms are smaller) (a); projection of Ti-C along [111] direction (b).](image)

![Fig.4. Crystal structure of TiN.](image)
Each nonmetal atom is situated in regular octahedron which consists itself with six titanium atoms. A critical feature of cutting tool materials with content of titanium nitride or carbonitride is decreased thermal conductivity which in turn holds down tool inserts temperature and allows higher surface machining speeds without serious edge deformation [12]. These ceramic composites, to their great thermodynamic stability, high hardness, and compression strength of the TiC, Ti(C,N) additives, have a better cutting properties in comparison to oxide ceramics, and can be used for precision machining of hard work-pieces (Fig.5).

A commercial Alcoa alumina powder (containing 85.0 % α-phase, of 99.8% purity) type A16SG with a submicron particle size of below 0.5 μm and a nano particle size of 40 nm, with average size of agglomerates of 150 nm produced by Inframat Advanced Materials, USA were used to prepare the tested composites. Experiments were carried out on alumina-based composites (type B) with the addition of: 10 mass% ZrO₂ with modification of the zirconia phase (partially stabilized with 5% mass Y₂O₃ – PYT05.0 (ZY5) produced by Unitec Ceramics, England, with an average particle size of 0.9-1.1 μm, a monoclinic phase of zirconia in submicron (m-ZrO₂) and nano scale produced by Fluka, Germany. A commercial TiC powder with different particle sizes as a standard TiC (grade ST120 produced by H.C. Starck, Germany) with an average particle size of 1.0-4.0 μm and TiC_{nano} with agglomerates of ≤130 nm produced by Aldrich Sigma, Germany were added to compounds. Second kind of composites (type C) based on alumina contain 30 wt% carbonitride titanium Ti(C,N) 30/70 produced by H.C. Starck in micro scale size, Ti(C,N) 50/50 produced by Neomat with an average particle size of 40 nm and 2wt% of m-ZrO₂ in micro and nano scale sizes as well. Sintering additives, such as MgO_{nano} (0.3 mass%), were introduced to inhibit grain growth. The initial composition of compounds selected for testing are presented in Table 1.

![Image](image.png)

**Fig.5.** Comparison of cutting speed versus tool life for ceramic tool materials.

The advantages of such tools are the possibility of high duty working, obtaining of surfaces with very low roughness (in many cases grinding is eliminated) and the possibility of ecological “dry cutting” without the use of cutting fluids. Thus the main aim of this study is to obtain new titanium carbide and titanium carbonitride-reinforced alumina-based composites that combine excellent toughness with increased hardness. Today, different carbide tool manufacturers have tailored TiC and Ti(C,N) properties to satisfy the industrial requirements for specific applications.

### 2. Experimental procedure

Alumina, zirconia partially stabilized with yttria, non-stabilized zirconia, titanium carbide and titanium carbonitride were used as raw materials to manufacture the tested alumina matrix composites.

<table>
<thead>
<tr>
<th>Compound composition, wt%</th>
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<tbody>
<tr>
<td><strong>Compounds</strong></td>
</tr>
<tr>
<td><strong>Micron Size</strong></td>
</tr>
<tr>
<td>B</td>
</tr>
<tr>
<td>B1</td>
</tr>
<tr>
<td>C</td>
</tr>
<tr>
<td>C1</td>
</tr>
</tbody>
</table>

Components were mixed for approximately thirty hours in alumina mills with zirconia balls, with the addition of a plasticizer. Materials, uniformly set, after plasticizing and drying, were granulated. Green compacts with dimensions of 5.5x3.0x35 mm and of 16.5x16.5x10 mm were uniaxially pressed at 100 MPa and then cold isostatically pressed at 200 MPa. Ceramic composites were sintered in vacuum at a maximum temperature of 1973 K at constant heating and cooling rates of the furnace. Following
measurements were performed on the tested specimens: Vickers hardness \(HV_1\), Young’s modulus (by measuring the transmission velocity of longitudinal and transversal ultrasonic waves through the sample), fracture toughness at room temperature \(K_{IC}\) (based on 3PB) and at an elevated temperature of 873K, \(K_{IC(ET)}\), wear resistance \(V_n\) (determined by speed of mass loss), friction coefficient \(\mu\) and apparent density \(\rho_p\). Single Edge Notched Beam (SENB) specimens (mechanically notched) with dimensions of 1.5x4.0x35.0±0.1mm were used to determine fracture toughness by means of a conventional method based on three-point bending of specimens (3PB). An initial 0.9 mm deep notch was produced with a diamond saw (thickness 0.20 mm) and then the notch tip was pre-cracked with a thin diamond saw (thickness 0.025 mm). The total initial notch length was approximately 1.1 mm (Fig.6).

![Fig.6. Single edge notch bending specimen (SENB) for fracture toughness testing](image)

The relationship \(K_{IC} = f(c)\) is given by the equations (1,2) [13,14]:

\[
K_{IC} = 1.5 \frac{P_c}{W^2 B} Y c^{1/2}
\]  

\[
Y = \sqrt{\frac{\pi}{\beta}} \left[ 0.3738 \beta + (1 - \beta) \sum_{i,j=0}^4 A_{ij} \beta \left( \frac{W}{S} \right)^i \right]
\]

where: \(P_c\)=critical load, \(S\)=support span, \(W\)=width, \(B\)=specimen thickness, \(Y\)=geometric function, \(c\)=crack length, \(\beta= c/W\) and \(A_{ij}\) are the coefficients given by Fett [14].

Measurements of the fracture toughness at an elevated temperature of 873K were carried out on a ZWICK 1446 instrument with mounted electrical furnace (Fig.7).

![Fig.7. Measuring position for determination of fracture toughness at elevated temperatures.](image)

Modulus of elasticity (Young’s modulus) of the tested composites was determined by measuring the transmission velocity of longitudinal and transversal ultrasonic waves through the sample. Probe sets work together with a Panametrics Epoch III ultrasonic flaw detector connected to a controlling computer. Calculations were made using the following formula:

\[
E = \rho C_T^2 \frac{3C_L^2 - 4C_T^2}{C_L^2 - C_T^2}
\]

where: \(E\)-Young’s modulus, \(C_L\)-velocity of the longitudinal wave, \(C_T\)-velocity of the transversal wave, \(\rho\)-density of the material.

The velocities of transversal and longitudinal waves were determined as a ratio of sample thickness and relevant transition time. The accuracy of calculated Young’s modulus from equation (3) was estimated to be below 2%. Wear resistance was determined by a method based on the measurement of mass decrement rate during wear of the specimen against an SiC80 abrasive cloth. The following formula (4) was used to calculate mass decrement rate (\(V_n\)):
\[ V_{n} = \frac{1000 \cdot \Delta m}{\rho_{p} \cdot F \cdot T} \]  \[ \text{(4)} \]

where: \( \Delta m \)-absolute mass wear, \( \rho_{p} \)-apparent density, \( F \)-specimen contact surface, \( T \)-time.

In ball-on-disc tests, the coefficient of friction for contact with an \( \text{Si}_{3}\text{N}_{4} \) ball were determined using a CETR UMT-2MT universal mechanical tester. In the ball-on-disc method, sliding contact is produced by publishing a ball specimen onto a rotating disc specimen under a constant load. Tests were carried out without lubricant. The loading mechanism applied a controlled load \( F_{n} \) to the ball holder (Fig.8) and the friction force was measured continuously during the test using an extensometer.

![Fig.8. Material pair for the ball-on-disc method: 1- \( \text{Si}_{3}\text{N}_{4} \) ball; 2-sample (disc)](image)

For each test, a new ball was used or the ball was rotated such that a new surface was in contact with the disc. The surface of the discs flat and parallel to within 0.02 mm and the roughness of the test surface not more than 0.1 µm \( R_{a} \). Friction coefficient was calculated from the following equation (5):

\[ \mu = \frac{F_{t}}{F_{n}} \]  \[ \text{(5)} \]

Where: \( F_{t} \) is the measured friction force, and \( F_{n} \) is the applied normal force.

Microstructure observations of the specimen were carried out using a JEOL JSM-6460LV scanning electron microscope. X-ray diffraction analysis for the characterization of the ceramic composites were made as a completion study. The Rietveld method with X’Pert Plus programme made possible of quantitative phase analysis of ceramics. The Philips program APD-3.5B-Fit profile was applied for observation wt% content of revealed phases. X-ray diffraction was used both to identify phases and to assess the ratio of tetragonal zirconia phase (t) to monoclinic zirconia phase (m) in selected tested composites. Preliminary cutting tests of \( \text{Al}_{2}\text{O}_{3}-\text{Ti}(\text{C},\text{N}) \) (C) in the turning of tool steel steel NC6 grade with hardness 50 HRC were carried out for CSRNL2525-12 type inserts. \( \text{Al}_{2}\text{O}_{3}-\text{ZrO}_{2}-\text{TiC} \) (type B, B1) composites were carried out for SNGN 12 04 08 type inserts in the turning of the constructional alloy steel 40H with 252 HB hardness. Tool life (\( T_{\text{mean}} \)) of the insert cutting edge was measured for tested composite inserts at following cutting parameters: speed (\( v_{c} \)) = 150 m/min, feed (\( f \)) = 0.13, depth (\( a_{p} \)) = 0.3, wear criterion -wear on flank face from measured \( V_{B} \) values up to \( V_{B_{\text{max}}} = 0.30 \) mm. were accepted. Cutting tests were performed without cooling lubricant.

### 3. Results and Discussion

The results obtained from the tests concerning apparent density (\( \rho_{p} \)), Young’s modulus (\( E \)), Vickers hardness (\( HV_{1} \)), fracture toughness at room temperature (\( K_{\text{IC}} \)) and at elevated temperature 873K - \( K_{\text{IC}}^{\text{CUT}} \) of the tested alumina matrix composites with various composition are presented in Table 2.

<table>
<thead>
<tr>
<th>Material</th>
<th>Apparent density ( \rho_{p} ) [g/cm(^3)]</th>
<th>( HV_{1} ) [GPa]</th>
<th>( E ) [GPa]</th>
<th>( K_{\text{IC}} ) [MPa-m(^{1/2})]</th>
<th>( K_{\text{IC}}^{\text{CUT}} ) at 873K [MPa-m(^{1/2})]</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>4.13</td>
<td>18.8</td>
<td>378</td>
<td>4.6</td>
<td>4.3</td>
</tr>
<tr>
<td>B1</td>
<td>4.09</td>
<td>18.3</td>
<td>358</td>
<td>4.2</td>
<td>4.0</td>
</tr>
<tr>
<td>C</td>
<td>4.18</td>
<td>19.3</td>
<td>385</td>
<td>4.2</td>
<td>4.0</td>
</tr>
<tr>
<td>C1</td>
<td>4.23</td>
<td>17.0</td>
<td>394</td>
<td>4.6</td>
<td>4.2</td>
</tr>
</tbody>
</table>

Tested titanium carbide-zirconia and carbonitride reinforced alumina matrix composites exhibit: high Vickers hardness (in the range 17.0-19.3 GPa), similar values of critical stress intensity factor \( K_{\text{IC}} \) at room temperature (in the range 4.2-4.6 MPa m\(^{1/2}\)), \( K_{\text{IC}}^{\text{CUT}} \) at elevated temperatures of 873K (in the range
4.0–4.3 MPa m$^{1/2}$), similar values of elastic modulus (358–394 GPa). Further, the lower mass decrement rate $V_n$ is observed for $\text{Al}_2\text{O}_3$-$\text{Ti}(\text{C}, \text{N})$ where $V_n$=4.7–4.9 $\mu$m/h for C and C1 respectively in comparison to $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$-$\text{TiC}$ composites $V_n$=5.7–7.8 $\mu$m/h for B and B1 respectively. Analysis of results indicates higher fracture toughness (by approx. 10%) for ceramic composites based on submicro powders (B) than for those containing mixture nano and submicro powders (B1). One of the reasons of differences in fracture toughness is various ratio of tetragonal zirconia phase (t) to monoclinic zirconia phase-(m) observed in tested composites. The $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$-$\text{TiC}$ (B1) composite reveals higher amount about 40% of monoclinic phase in comparison to $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$-$\text{TiC}$ (B) (Fig.9).

Fig.9. X-ray diffraction analysis of $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$-$\text{TiC}$ (B) composite.

The ratio of tetragonal to monoclinic phase is 6.6 for $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$-$\text{TiC}$ (B) and 3.4 for $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$-$\text{TiC}$ (B1). Increasing of the zirconia tetragonal phase-(t) fraction in comparison to the zirconia monoclinic phase-(m) fraction in the $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$-$\text{TiC}$ (B) composite effects improvement of the fracture toughness. The values of fracture toughness is higher (about 10%) for $\text{Al}_2\text{O}_3$-$\text{Ti}(\text{C}, \text{N})$ (C1) composites with addition of 2wt% ZrO$_{2\text{nano}}$. This increase can result from lower Vickers hardness of tested composite. The fracture toughness at elevated temperature (873 K) exhibits lower values (by approx. 5-10%) in comparison to composites tested at room temperature. The similar effect is observed for both tested composites: the alumina composites reinforced by means of titanium carbonitride and composites reinforced by means of titanium carbides. The fracture toughness remains on the same level with increasing of the temperature up to 1073 K. The friction coefficient values for the $\text{Al}_2\text{O}_3$-$\text{Ti}(\text{C}, \text{N})$ (C,C1) composites at the contact with the Si$_3$N$_4$ ball were changed in the range from 0.38 to 0.48 respectively (Fig.10).

Fig.10. Friction coefficient of the $\text{Al}_2\text{O}_3$-$\text{Ti}(\text{C}, \text{N})$ (C) and $\text{Al}_2\text{O}_3$-$\text{Ti}(\text{C}, \text{N})$ (C1) composites.

The $\text{Al}_2\text{O}_3$-$\text{Ti}(\text{C}, \text{N})$ (C) composite reveals higher (about 20%) value of the friction coefficient ($\mu$=0.48) than the $\text{Al}_2\text{O}_3$-$\text{Ti}(\text{C}, \text{N})$ (C1) composite ($\mu$=0.38). The microstructure of the $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$-$\text{TiC}$ and the $\text{Al}_2\text{O}_3$-$\text{Ti}(\text{C}, \text{N})$ is presented in Fig. 11-13.

Fig.11. SEM surface micrographs of $\text{Al}_2\text{O}_3$-$\text{ZrO}_2$-$\text{TiC}$ composites: a) (B); b) (B1).
Fig.12. SEM micrographs of the fracture surface of the tested composites: a) Al₂O₃-ZrO₂-TiC (B), b) Al₂O₃-ZrO₂-TiC (B1).

Fig.13. SEM micrographs of the fracture surface of the tested composites: a) Al₂O₃-Ti(C,N) (C); b) Al₂O₃-Ti(C,N) (C1).

Fine particles of the zirconia and of the titanium carbides, homogeneously distributed in the alumina matrix, are visible in the micrograph of the Al₂O₃-ZrO₂-TiC (B1) composites (Fig.11a,b). The uniformly distributed of the ZrO₂ particles with agglomerates are observed as well (Fig. 11a). An intergranular and transgranular fracture is observed for alumina matrix composites reinforced TiC and Ti(C,N) with dominate of transgranular fracture for composites based on mixture nano and micro powder (Fig. 12,13). Cutting tests carried out on the titanium carbide-reinforced ceramic composites with alumina matrix reveal more than twice increase in tool life ($T_{\text{mean}}=24$ min) for the Al₂O₃-ZrO₂-TiC (B) composite in comparison to the Al₂O₃-ZrO₂-TiC (B1) (for which tool life achieves values 11 min). The high tool life is observed for the alumina matrix composite with Ti(C,N) as well. Preliminary industrial cutting tests in the turning of NC6 steel exhibit tool life ($T=253$ min) of the composite with the Ti(C,N) and 2 wt% ZrO₂ nano (C1) at cutting speed $v_c = 150$ m/min, (Fig.14). The cutting tests were carried out without cooling lubricant.

Fig.14. Relationship of the wear on flank face ($V_B$) against cutting time ($t$) for the C1 composite.

4. Conclusions

Results, obtained from analysis of the mechanical properties of tested alumina matrix composites reinforced by means of the titanium carbide zirconia and titanium carbonitride permit to evaluate these materials usefulness for cutting tools edges. Alumina-zirconia-titanium carbide composite with mixture powders in micro- and nano-scale of size not improve the fracture toughness. Alumina-titanium carbonitride with 2wt% zirconia composites reveal the fracture toughness on same level in comparison to alumina–zirconia-titanium carbides.
composites. The elevated temperature of 873K decreases the fracture toughness of tested composites (by approx. 5-10%). Preliminary cutting tests carried out on the alumina matrix composites prove their good cutting properties. The results of the presented investigations allow rational use of existing ceramic tools.

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