HARDNESS AND INDENTATION FRACTURE TOUGHNESS OF ALUMINA-SILICON CARBIDE NANOCOMPOSITES

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Abstract
The present work deals with the study of mechanical properties of a range of alumina-silicon carbide composites. The materials were prepared using two different types of alumina powders: CR1 with grain size 500 nm and BMA with grain size 150 nm, into which various amounts of silicon carbide nanoparticles were admixed.

The Vickers indentation method was used to determine hardness and indentation fracture toughness for different Al2O3-SiC nanocomposites.

The results showed a negligible influence of the SiC volume contents on the hardness and indentation toughness. Only in the case of material with BMA matrix an important increase between 2.5 and 5 vol.% SiC was found.

Keywords: hardness, indentation fracture toughness, alumina-silicon nanocomposites

1 Introduction
Alumina (Al2O3) is widely used material in the engineering ceramics. An excellent combination of properties as hard, wear-resistance, high strength and stiffness, good thermal conductivity lead to a very wide range of applications for alumina. Typical uses are gas laser tubes, wear pads, high temperature electrical insulators, electronic substrates, instrumentation parts for thermal property test machines, etc. The inherent brittleness of aluminium oxide crystal structure is still one of its main drawbacks. There have been explored many ways how to improve the fracture toughness and lower the brittleness of these materials. Among them the production of various types of composites proved to be very fruitful. One of the widely used methods is introduction of silicon carbide nanoparticles into the alumina matrix and preparing Al2O3-SiC composites.

Silicon carbide (SiC) has been developed into a high quality technical grade ceramic with very good mechanical properties. It is used in abrasives, refractories, ceramics, and numerous high-performance applications. The silicon carbide has low density, high strength, low thermal expansion, high hardness, thermal shock resistance. SiC is also a very hard material, extremely stable, particularly when protected by the matrix from oxidation environment. The residual stresses created as a consequence of difference in thermal expansion factors of both components of the composite tend to deflect, slow down and possibly to stop the cracks propagation, thus leading to toughening of the whole system.
Nano-size particle reinforced ceramics represent a new class of materials with improved mechanical properties, even at high temperatures, compared to monolithic ceramics. A system of particular interest, and the focus of this paper, is the Al$_2$O$_3$-SiC system because it has been reported with the most improved properties: Al$_2$O$_3$ ceramics containing several vol.% SiC particles of size 30 and 60 nm [1].

A related factor is that Al$_2$O$_3$-SiC nanocomposites are harder to sinter to full density than Al$_2$O$_3$ as the SiC particles retard densification and grain growth. Therefore, the nanocomposites tend to have significantly finer grain sizes when fully dense than Al$_2$O$_3$ [2-9].

The indentation fracture (IF) method has been widely used for determining fracture resistance of ceramic materials. This method is particularly useful when the sizes of available specimens are limited. The IF method is considered to be an alternative technique to single edge-precracked beam (SEPB) methods [10]. Compared with other conventional fracture mechanics methods, the advantages of the indentation methods include the small size of the test specimen, the ease of the specimen preparation, and the simplicity of the test. Consequently, numerous semi-empirical equations relating material fracture toughness to the measured indentation parameters, such as the applied indentation load, final radial crack length, or indentation print size, have been derived based on experimental observation and/or theoretical considerations [11,12].

The aim of the present work is to study and compare the hardness, fracture toughness and crack propagation behaviour of different Al$_2$O$_3$-SiC composites.

## 2 Experimental materials and methods

Materials were prepared in the Universite de Lyon, INSA de Lyon. As starting materials two alumina powders were used: CR1 (grain size 500 nm) and BMA-15 (150 nm). Two SiC powders with 30 nm (SiC 30) and 60 nm (SiC 60) grain sizes were used as toughening additives. The mixing ratios are given in the Table 1. The powders were mixed in ball mill for > 20 h, then slip casted, dried for 50 h, ground and sieved. Then they were pressed into a die and spark plasma sintered at various temperatures (see Table 1) for 30-40 min. The resulting samples were of disc shape with 20 mm diameter and 3 mm height.

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Additive</th>
<th>Additive contents (vol.%)</th>
<th>Sintering temperatures (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CR1 Al$_2$O$_3$ (grain size 500 nm)</td>
<td>SiC 30</td>
<td>2.5, 5, 7.5</td>
<td>1550, 1650</td>
</tr>
<tr>
<td>BMA-15 Al$_2$O$_3$ (grain size 150 nm)</td>
<td>SiC 60</td>
<td>2.5, 5, 7.5</td>
<td>1550, 1650</td>
</tr>
<tr>
<td>BMA-15 Al$_2$O$_3$ (grain size 150 nm)</td>
<td>SiC 30</td>
<td>2.5, 5, 7.5</td>
<td>1550, 1650</td>
</tr>
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<td>2.5, 5, 7.5</td>
<td>1550, 1650</td>
</tr>
</tbody>
</table>

Hardness was measured by means of Vickers method using 5 kg loading and holding time was 10 s. The hardness was calculated using Eq. 1.

$$HV = 1,8544 \cdot \frac{P}{a^2}$$

where: $HV$ [GPa] – hardness  
$P$ [N] – indentation load  
$a$ [mm] – diagonal length.
Fracture toughness was measured using indentation method from crack lengths. The 10 indents for each material were performed. The length of cracks was measured by 3D Optical Profiler SENSOFAR PLu neox. Young’s modulus 360 GPa was obtained independently by instrumental nanoindentation [13]. The indentation fracture mechanics had been studied previously in this material. By gradual grinding of indented samples it was found that it is type of half-penny cracks [13]. From the length of the indentation cracks fracture toughness was calculated by applying the Anstis’ equation [14]:

\[
K_{IC} = \eta \left( \frac{E}{H_V} \right)^{1/2} \frac{F}{c^{3/2}}
\]

where: 
- \(K_{IC}\) [MPa.m\(^{1/2}\)] – Fracture toughness 
- \(\eta\) [-] – shape factor (0.016) 
- \(E\) [GPa] – Young’s modulus 
- \(H_V\) [GPa] – hardness 
- \(F\) [N] – indentation load 
- \(c\) [\(\mu\)m] – crack length

### 3 Results and discussion

The typical Vickers indent and typical cracks on the surface of an experimental material with high amount of SiC are illustrated in Fig. 1. The cracks are straight without clear deviation of the crack path. The microstructure of the material is shown in Fig. 2, where arrows show SiC nanoparticles.

![Fig. 1 CR1 1550°C-7.5% SiC-30nm](image)

**Fig.1 CR1 1550°C-7.5% SiC-30nm**

**Figures 3 and 4** shows dependence of hardness on volume content of SiC. For both CR1 materials near linear dependencies for both temperatures were determined. Only the sample BMA with grain size 30 nm SiC showed significant increase for values of hardness. The increase of hardness from 14.7 GPa for 2.5 vol. % of SiC to 19.2 GPa for 7.5 vol. % of SiC at
sintering temperature 1550°C (Fig. 3) was found. For the samples sintered at 1650°C values from 14.9 GPa for 2.5 vol. % of SiC to 20.8 GPa for 7.5 vol. % of SiC were found (Fig. 4).

The indentation toughness was determined and only small differences for the materials shown in Fig. 5 and Fig. 6 were found. In the case of material CR1 with 30 nm SiC prepared at 1550°C indentation toughness had slight maximum for 5% SiC. However, the opposite tendency for both CR1 with 60 nm SiC and BMA with 30 nm SiC was found (Fig. 5). It seems that these values are basically the same within the errors of measurement. The scatter of the measured values is not illustrated in the plots, because it would make the figures confusing but typically the standard deviations for indentation toughness were about 8.11% for the samples sintered at 1550°C and 6.42% for those sintered at 1650°C.

The indentation toughness for material at sintering temperature 1650°C (Fig. 6) decreased with increasing SiC content. The most significant loss of indentation toughness with increasing SiC content had BMA with 30 nm SiC.

Fig. 2 BMA 1650°C-5%SiC-30nm

Fig. 3 Hardness and content SiC, sintered at 1550°C

Fig. 4 Hardness and content SiC, sintered at 1650°C
Figure 7 shows chipping of effect on test material. Typically, this is a sign of low fracture toughness and possibly of presence of residual stresses. In our case, this effect increases with increasing sintering temperature and decreasing SiC content. For this reason, 1 indent out of 10 indents was unusable (due to extensive chipping) for sample sintered at 1550°C with 7.5% of SiC, but 3 out of 10 indents chipped out in the sample sintered at 1650°C with 2.5% of SiC. Figure 8 and Fig. 9 show some details from Fig. 7.

4 Conclusions
Generally, the results showed in most cases only a negligible influence of the SiC contents on the hardness and fracture toughness. Only in the case of material with BMA matrix an important increase between 2.5 and 5 vol. % SiC was found. This matrix tended to have higher hardness for higher SiC volume contents, what is shown in Fig. 3 and Fig. 4. Fracture toughness did not show any noticeable increase because of SiC additive. Higher sintering temperature did not result in better properties, thus suggesting that lower sintering temperature (1550 °C) should be sufficient for the material preparation.

References

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