CHARACTERISTICS OF FRACTURE ORIGINS IN SiC BASED CERAMICS

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Abstract
The paper deals with the flexural strength measurement and characterization of fracture origins of the silicon carbide materials prepared by liquid-phase-sintering. The characteristic flexural strength and Weibull’s modulus were computed using two-parameter Weibull’s distribution. Fractographic analysis of broken specimens was used to characterize the processing flaws. The strength of the investigated materials was degraded by the presence of processing flaws mainly in the form of pores, cluster of pores, silicon carbide agglomerates and sharp cracks. The fracture initiation sites are located predominantly in the volume of the specimens, sometimes close to the tensile surface. The size of fracture origins was lying in the interval from 15 µm to 400 µm.

Keywords: ceramics, silicon carbide, fracture origins, fractography

1 Introduction
Silicon carbide exhibits high strength, thermal shock resistance, chemical and thermal stability and high hardness. Its use is hence expected in numerous applications, such as aeronautic, nuclear, or wear resistant components [1]. Mechanical properties of brittle materials such as silicon nitride and silicon carbide, especially their toughness and strength but also other properties as the elastic constants or the thermal shock resistance, depend to a large extent on their microstructure [2-5]. Elongated grains of silicon carbide have been shown to increase fracture toughness by crack bridging or crack deflection due to weak interface boundaries, but coarsening leads to an increase of the size of the critical flaws which degrade strength values [6-8].

For the development and improvement of ceramic materials fractography is used as a powerful tool for optimization of materials. The identification of the fracture origins can give a clear evidence for weak points in the processing. Fracture origin is the source from which brittle fracture begins [9]. If the type of flaw is recognized, this knowledge can be used to eliminate them, i.e. to improve the processing and to get components with a higher strength. This easy finding of the mechanically weakest elements in the microstructure is probably the most important application of the fractography in the ceramic technology.

The fracture origin in each specimen is characterized by the following three attributes: identity, location, and size [10]. First attribute of a fracture origin is its identity. Fracture origins can be completely different features of the microstructure. Very often, the origins may be volume flaws like pores or porous regions. Defects can also be large grains, badly sintered grain boundaries, hard agglomerates, inorganic inclusions, local inhomogenities, cracks and more. Surface defects
can be additionally created by machining damage, corrosion pits, and corroded grain boundaries [9]. Of course, a volume-type defect can be located at the surface, and a material can contain both surface and volume flaws [11].

The second attribute of a fracture origin is its location. A volume-distributed origin type may be in the volume (bulk), at the surface, near the surface, or at an edge. Surface-distributed origins (e.g. machining damage or pits), on the other hand, can only be found at the surface or at an edge of a specimen or component.

The third attribute of a fracture origin is its size. If the origin is in the volume, for an equiaxed origin, measure the approximate diameter, for an elongated origin, measure the approximate minor and major axis lengths. If the origin is at the surface, measure the origin depth and the width [10].

![Fracture surface schematic. An initial flaw of size $a_1$ may grow to size $a_{cr}$ [11]](image)

On a fracture surface, there are five primary regions of crack growth. These are the flaw itself – fracture origin, then “mirror” region then “mist” region, the “hackle” region, and the region of macroscopic crack branching. Schematic sketch of a typical fracture surfaces is presented in Fig. 1. As the crack grows from the fracture origin with increasing velocity, the crack first propagates in a relatively smooth plane (mirror) to the boundary, and progressively gets rougher by deviating slightly out of plane in region that resembles mist (mist). Finally, the crack deviates locally from the main plane of fracture (hackle), getting very rough, and branching into two or more cracks [11].

The aim of this work is to investigate the flexural strength and to characterize the fracture origins of prepared SiC materials.

2 Experimental material and procedure

Powder batches were produced using $\beta$-SiC powder (HSC-059, Superior Graphite). The sintering additives were $\text{Al}_2\text{O}_3$ (A 16 SG, Alcoa), Y$_2$O$_3$ or AlN (grade C, H.C. Starck). The powder mixtures were ball milled in isopropanol with SiC balls for 24 hours. The suspension was dried, subsequently sieved through 25 $\mu$m sieve screen in order to avoid hard agglomerates. The samples were hot pressed at 1850°C/1h or at 1870°C/1h under mechanical pressure of 30 MPa in $\text{N}_2$ atmosphere. After sintering the specimens were cut, polished to a 1 $\mu$m finish. The chemical composition is given in Table 1.

The strength was measured using specimens with dimensions of 3x4x45 mm, tested in the four point bending mode. The specimens were ground by 15 $\mu$m diamond wheel before testing. The
two edges on the tensile surface were rounded with a radius about 0.15 mm in order to eliminate a failure initiated from an edge of the specimen. The specimens were tested in a four point bending fixture (inner span of 20 mm and an outer span of 40 mm) with the crosshead speed of 0.5 mm/min at ambient temperature and atmosphere. The flexural strength is then calculated by Eq. 1:

\[
\sigma_b = \frac{3F(S_1 - S_2)}{2b.h^2}
\]

where: 
- b - sample width
- h - sample height
- \(S_1\) - outer span
- \(S_2\) - inner span
- F - fracture load

The characteristic flexural strength and Weibull’s modulus were computed using two-parameter Weibull’s distribution. SEM and EDX analysis of broken bend specimens were used to characterize the fracture origins, their location, size and shape.

Table 1 Chemical composition of prepared materials

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sintering</th>
<th>Chemical composition (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SiC</td>
<td>(\text{Y}_2\text{O}_3)</td>
</tr>
<tr>
<td>SCYAI</td>
<td>1870°C/1h</td>
<td>91</td>
</tr>
<tr>
<td>SCYAN</td>
<td>1850°C/1h</td>
<td>85.2</td>
</tr>
</tbody>
</table>

3 Results and discussion

Representative SEM micrographs of a fracture surface of the SCYAI and SCYAN are shown in Fig. 2. The microstructures of both sintered materials consist of fine submicron-sized equiaxed SiC grains with a low aspect ratio. In the case of SCYAI the addition of \(\text{Al}_2\text{O}_3\) and lower amount of \(\text{Y}_2\text{O}_3\) resulted in a finer microstructure of the material.

![Fig.2 Fracture surface of SCYAI (A) and SCYAN (B) material](image)

The results of the statistical analysis of the flexural strength using the Weibull’s statistics are shown in Fig. 3 (SCYAI) and Fig. 4 (SCYAN). The characteristic strength and Weibull’s modulus were 424 MPa and 9.51 for SCYAI, 352 MPa and 3.43 for SCYAN. The flexural
strength ($\sigma_{\text{BT}}$) decreases with increasing of $Y_2O_3$ amount. The low values of Weibull’s modulus suggest high scatter of flexural strength values. The presence of the initial flaws caused a lower value of characteristic flexural strength. For better Weibull’s analysis at least 30 samples are necessary for four-point bend test.

Strecker [12] investigated sintered SiC ceramics with AlN-$Y_2O_3$ additives. He reported characteristic strength and Weibull’s modulus 367 MPa and 7.7, respectively. Kim [13] estimated flexural strength of 650 MPa in SiC with AlN and $Y_2O_3$. Sciiti [7] showed higher values of flexural strength in interval 594 MPa – 746 MPa in SiC materials sintered with $Al_2O_3$ and $Y_2O_3$. Suzuki [14] described that flexural strength increased with increased AlN addition up to 5 wt%.

Microfractographic observations of the fracture surface and fracture profiles of broken bars have shown predominantly mixed inter- and transgranular fracture in both materials with slightly higher transgranular portion in the system SCYAN. Macrofractographic observations have
shown that the fracture origins are often processing defects being mainly in the form of silicon carbide agglomerates (52%), pores (16%), and sharp cracks (10%). The fracture initiation sites are located predominantly in the volume of the specimens (50%), sometimes near to fracture surface (26%) and surface (21%).\textbf{Fig. 5} and \textbf{Fig. 6}. The size of fracture origins is lying in the wide interval from 15 $\mu$m to 400 $\mu$m. In most cases the shape of fracture origins was elliptical. Sciti [7] observed that critical defects in SiC ceramics are mainly clusters of larger grains. Kašiarová [15] identified two types of technological initiating flaws (agglomerates of large SiC grains and agglomerates of free carbon) in $\text{Si}_3\text{N}_4$-SiC nanocomposites. We observed in our previous work [16] that the fracture origins of SiC+$\text{Si}_3\text{N}_4$ composites are defects mainly in the form clusters of pores and SiC and $\text{Si}_3\text{N}_4$ agglomerates.

\textbf{Fig.5} SiC agglomerate found in SCYAN ceramic, located near to surface

\textbf{Fig.6} SiC agglomerate found in SCYAN ceramic, volume located

4 Conclusion

Two types of silicon carbide ceramics were prepared by liquid-phase-sintering. The strength of the both investigated materials was degraded by the presence of processing flaws mainly in the form of silicon carbide agglomerates, pores, and sharp cracks. Higher value of characteristic strength exhibits SiC material with lower amount of $\text{Y}_2\text{O}_3$ and with $\text{Al}_2\text{O}_3$ addition. After eliminating of these defects by proper technological route significant strength improvement can be expected.
References


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