

# Crack healing in liquid-phase-pressureless-sintered silicon carbide–aluminum nitride composites

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## Abstract

Crack healing in liquid-phase-pressureless-sintered SiC–AlN composites was investigated by introducing cracks into specimens and subsequently heat-treating the specimens. It was observed that cracks were healed and the strength was recovered. Cracks were filled with silica or mullite produced by the oxidation of the composites. It was shown that the healing temperature could be fixed in the range 1100–1300 °C and that large cracks up to about 300 μm could be healed completely. Our results imply that a simple oxidation heat-treatment can improve the reliability of silicon carbide–aluminum nitride components.

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**Keywords:** SiC; AlN; Crack healing; Mechanical properties

## 1. Introduction

Several studies reported that cracks can be healed by means of a heat-treatment with a beneficial effect on the flexural strength of ceramics.<sup>1–6</sup> Gupta<sup>1</sup> reported that cracks in alumina were healed by a mechanism similar to that of densification during sintering. Choi and Tikare<sup>2</sup> reported that cracks in silicon nitride were healed by being filled with an oxidation product. In silicon carbide cracks were filled by amorphous silica<sup>3</sup> and Koros et al.<sup>4</sup> reported that the cracks in silicon carbide, once healed, surprisingly became even stronger than the original silicon carbide. Furthermore, the crack-healing ability of monolithic silicon carbide makes it possible to reduce machining costs because most of the surface flaws could be healed using a pre-oxidation technique.<sup>5</sup> Reaction-bonded silicon carbide showed the same mechanism of monolithic SiC with the possibility to recover as much as 60% of the strength of as-machined specimens without cracks.<sup>6</sup>

Crack-healing behaviour of silicon carbide–aluminum nitride (SiC–AlN) composites, one of the best candidate for high tem-

perature applications among SiC-based materials, has not been characterized up to now. These materials present high flexural strength and oxidation resistance up to 1500 °C<sup>7–8</sup> and the study of the crack-healing ability consequently represents an important issue to be investigated in order to assess the reliability of SiC–AlN components. Therefore, in this paper the crack-healing ability of pressureless-sintered SiC–AlN ceramics is studied and compared with crack-healing behaviour of sintered silicon carbide reported by other authors.

## 2. Experimental procedure

SiC–AlN composites has been prepared by means of pressureless sintering using yttria (Y<sub>2</sub>O<sub>3</sub>) as sintering aid. A batch of powder composed by 48 wt% SiC–48 wt% AlN–4 wt% Y<sub>2</sub>O<sub>3</sub> was wet-mixed in ethanol for 12 h using SiC grinding balls. After drying and sieving, the powder was compacted by die pressing at 67 MPa and subsequently pressed at 150 MPa by CIP.

Sintering was performed in a graphite element furnace in flowing nitrogen at 1 atm with green bodies put inside a graphite crucible without protective powder bed. Sintering was performed at 1950 °C, while an annealing step was conducted at 2050 °C. Thermal cycle was characterised by heating and

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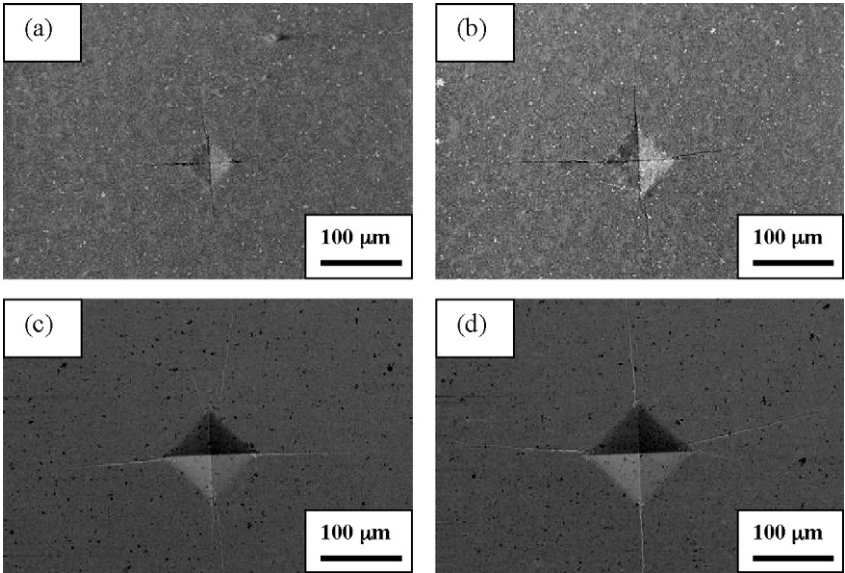


Fig. 1. SEM images of the Vickers indentations obtained with a load of (a) 49 N, (b) 98 N, (c) 147 N and (d) 196 N.

cooling rate of 20–30 °C/min and by dwell time of 0.5 h at the sintering temperature.

The sintered material was cut into test specimens measuring 3 mm × 4 mm × 20 mm. These specimens were subjected to three-points bending with a span of 16 mm. Semi-elliptical surface cracks of 60–560 μm in surface length were introduced at the center of the tensile surface of the specimens by means of

Vickers indentations. The ratio of depth (*a*) to half surface length (*c*) of the crack (aspect ratio) was *a/c* = 0.9.

The pre-cracked specimens were heat-treated for 1 h at temperature between 900 and 1300 °C in air. The specimens were furnace-cooled to room temperature. After crack-healing process, monotonic bending tests were carried out at room temperature. For a comparison, bending tests for smooth specimens

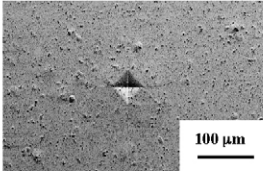
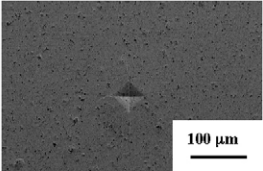
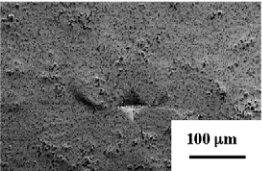
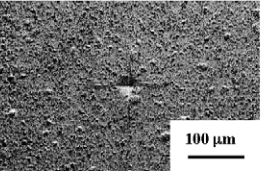
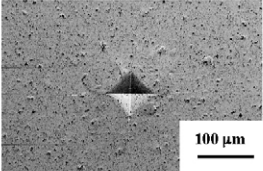
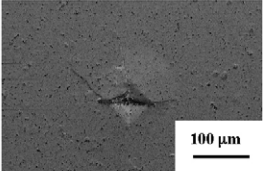
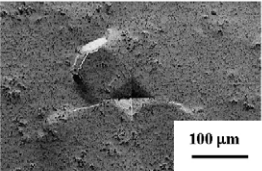
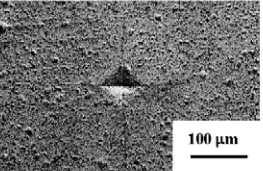
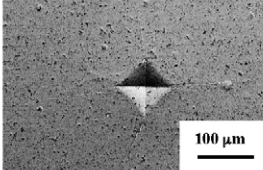
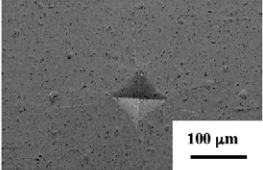
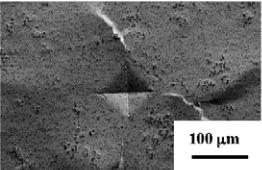
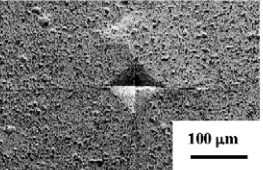
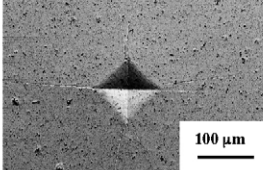
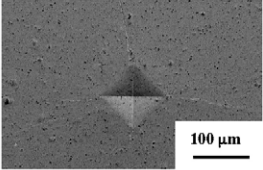
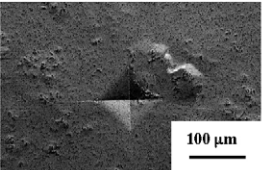
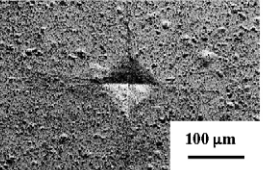
| T<br>CRACK<br>LENGTH |                                                                                     |                                                                                     |                                                                                      |                                                                                       |
|----------------------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|
|                      | 900°C                                                                               | 1000°C                                                                              | 1100°C                                                                               | 1300°C                                                                                |
| 170 μm               |  |  |  |  |
| 260 μm               |  |  |  |  |
| 370 μm               |  |  |  |  |
| 475 μm               |  |  |  |  |

Fig. 2. SEM images of the Vickers indentations after heat-treatments at 900, 1000, 1100 and 1300 °C.

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