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Grain bridging locations of monolithic silicon carbide by means of focused ion beam milling technique

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ABSTRACT

A slice and view approach using a focused ion beam (FIB) milling technique was employed to investigate grain bridging near the tip of cracks in four silicon carbide (SiC) based materials with different grain boundary chemistries and grain morphologies. Using traditional observations intergranular fracture behaviour and hence clear evidence of grain bridging was found for SiC based materials sintered with oxide additives. More surprisingly, in large grain materials, the FIB technique reveals evidence of grain bridging irrespective of the grain boundary chemistry, i.e. also in materials which macroscopically fail by transgranular failure. This helps to explain why the toughness of large grained materials is higher even if failure is transgranular.

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1. Introduction

The strong covalent bonding between silicon and carbon atoms gives SiC materials an excellent oxidation resistance, strength retention to high temperatures, high wear resistance, high thermal conductivity and good thermal shock resistance. Nowadays, SiC is recognized as one of the leading structural ceramics for gas turbines [1], for mechanical seals and brakes [2] and for bioengineering application (i.e. implants) [3,4]. On the other hand, there is a limitation to the usefulness of silicon carbide materials due to their low fracture toughness and high strength dispersion, i.e. an inhomogeneous and large distribution of flaws and defects.

During the last 30 years a lot of attention has been devoted to improving the toughness of ceramic materials to ensure that the damage during processing and post-processing can be acceptable without compromising the structure's reliability. Ceramic materials have been toughened using different approaches. A very successful approach consists of crack tip shielding through grain bridging in the wake of the crack tip [5]. The toughness increase arises because crack deflection leaves grains connected to both crack faces and these grains transmit a closing force across crack walls trying to prevent the crack from further extension by reducing the applied stress intensity at the crack tip [6]. The resistance to crack propagation due to grain bridging in general increases with increasing crack length, which leads to the so called Resistance-curve (R-curve) [7–9]. During the last 30 years, the R-curve behaviour of ceramic materials has been extensively studied for long cracks. What may happen for short cracks ($<100\ \mu m$) and its effect on toughness has until recently largely been overlooked. A material with flat R-curve may still have a steep rise at the beginning of the test as suggested by Fett et al. [10]. In materials with a steep rising R-curve, one may expect that toughness will behave differently for short cracks than for long cracks. The understanding of this concept is highly relevant as ceramic materials are generally produced with small natural defects in the order of few microns [11]. While there is general agreement that the action of grains a long distance behind the crack tip is a consequence of friction between the grains and the matrix from which they are being pulled, the steep rise in R-curve at the onset of failure is increasingly attributed to elastic bridging, i.e. where the grain bridging the crack faces are still to some extent fully bonded to both fracture surfaces. Different approaches were considered to study the effect of elastic bridging i.e. intact grains still bonded to both crack faces bridging the crack near its tip. For example Foulk et al. [12] proposed a 2D model of bridging by a single elongated grain based on finite element analysis during the early stage of the rising R-curve. This increase in crack resistance in very short cracks before forming grain bridges is in line with Fett et al. [10]. Fünfschilling and co-workers [13] observed that silicon nitride materials sintered with different additives show different R-curve behaviour ranging from flat, moderate and rising R-curve. However, the main common factor between the studied microstructures is that they all have a steep rising R-curve in the first $10\ \mu m$ of crack extension. Still, direct evidence from observation of such elastic grain bridging sites close to the tip of the crack has so far not been reported.

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2. Experimental methods

In this work, four silicon carbide based materials were produced: two using solid state sintering with boron and carbon (SS) and two using liquid phase sintering using alumina and yttria as sintering additives (LP), see Fig. 1. The processing details are described in [14]. The LP-Fine consists of homogenous fine equiaxed grains with average grain size of 1.0 ± 0.1 μm while LP-Coarse has elongated grains with a length of 16 ± 1 μm and a width of 5 ± 0.3 μm. For the SS-Fine, the grains have an average size of 4.0 ± 0.4 μm and the grains in the SS-Coarse have a length of 18 ± 1 μm and a width of 5 ± 0.3 μm. The samples were ground and polished up to 1 μm using diamond suspension. A crack is introduced by placing an indent of 30 kg on the polished surface. Once the crack tip was found an area with dimensions of 4 μm by 5 μm by 5 μm, Fig. 2, was selected for investigation with a slice and view approach, i.e. progressive milling in small steps (20 nm) along the X-axis and taking micrographs in between each milling stage.

The R-curve measurements were carried out in situ in the SEM (vacuum level of 1×10^{-3} Pa) using a constant moment double cantilevered test rig, built in line with the set-up proposed by Sorensen et al. [15]. The silicon carbide specimens were 65 mm by 10 mm by 5 mm. One side of the samples was polished to facilitate the observation of the crack tip. The speed of the stage motor was 0.1 mm min^{-1}. Every time crack propagation was observed, the applied load was recorded and a SEM micrograph was taken.

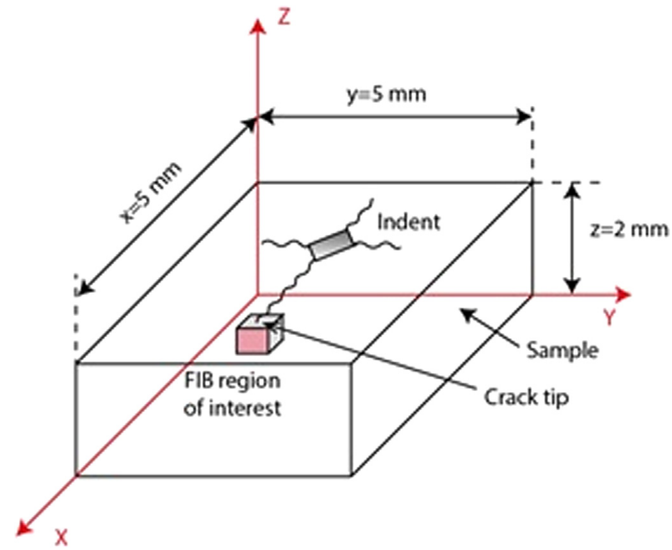

Fig. 2. Sketch showing the selected coordinates to identify planes and region of interest for the FIB slice and view process.

3. Results and discussion

As expected in the LP-sintered materials, cracks are deflected at grain boundaries, whereas in the SS-sintered materials, the cracks grow much straighter and do not appear to be influenced strongly by grain boundaries, see Fig. 3. However, a closer look at the coarse grained materials revealed that close to the tip of the crack, fracture is slightly complicated. While fracture continues to be along the grain boundaries in LP-Coarse and more cleavage-like in SS-Coarse, ligaments of material which remain solidly bonded to both sides of the fracture surface can be found in both materials.

In LP-Coarse sequence it is clear that the main crack is found to

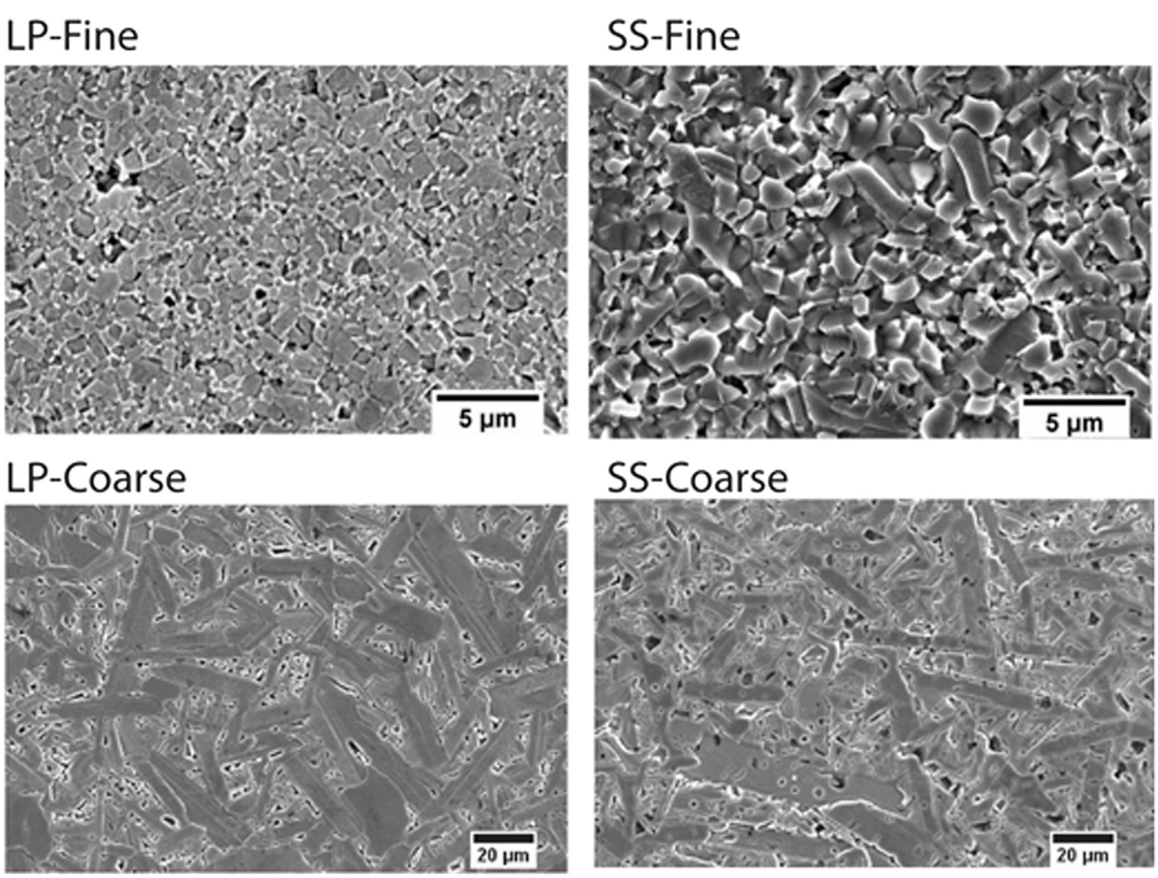

Fig. 1. Scanning electron micrographs of chemically etched silicon carbide materials of this work.

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