

# Cracking behavior of ZrB<sub>2</sub>-SiC-Graphite sharp leading edges during thermal shock



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## ABSTRACT

Crack initiation and propagation of ZrB<sub>2</sub>-SiC-Graphite (ZSG) sharp leading edges (SLEs) subjected to thermal shock were systematically evaluated by the water spraying method followed by a crack dyeing treatment. Distinct differences in the crack patterns among different test conditions were observed, and the cracking behavior of ZSG SLEs (including crack initiation time, crack number and critical failure temperature) was revealed to be strongly dependent on both the cooling rate and the microstructure. The crack propagation during thermal shock could be considered as a quasistatic process (crack speed was lower than 1 cm/s) that needed to be driven by continuous cooling.

## 1. Introduction

Due to the extremely high melting point coupled with excellent thermal (high thermal conductivity and low thermal expansion coefficient) [1], physical (high strength, hardness, Young's modulus and melting point) [2], chemical (good chemical stability) [3] and oxidation resistance [4] properties, ZrB<sub>2</sub>-SiC-Graphite (ZSG) ceramics, known as ultra-high temperature ceramics, have received considerable attention for structural applications in many extreme environments, such as leading edges and nose tips of missiles, reusable launch vehicles or hypersonic vehicles [5–7]. Although the ZSG ceramics have many advantages, their intrinsic characteristics such as low fracture toughness (premature failure due to brittle fracture), low toughness induced poor thermal shock resistance are still obstacles for them to be widely used, especially for applications in extreme environment [8]. Thus, it is essential, in order to ensure the safety of the aircraft, to thoroughly understand the thermal shock behavior of ZSG ceramics before actual use.

To date, significant progress has been made in this field and the available methods, which are used for the measurement of thermal shock resistance, can be broadly classified into two groups: thermal shock during heating [9–11] and cooling [12–16]. For thermal shock during heating, rapid resistance heating via a high current electrical setup is the most commonly used method, and the specific approach is measuring the strength retention of test specimens after rapid heating. By contrast, the cooling tests are more widely used for the evaluation of thermal shock behavior of ceramics because the heating thermal shock

process always involves the oxidation and ablation behaviors [17,18], which make the problem more complex. In cooling test, quenching method is the most frequently-used approach and the effect of graphite content [19], graphite flake diameter [20], quenching medium [21] and atmosphere before quenching [22] on the thermal shock resistance of ZSG ceramics with simple shapes (i.e., bar, plate and thin column) have been thoroughly investigated by this method.

However, thermal shock behavior of many components in actual engineering applications (such as leading edges and nose tips) differs significantly from that of specimens with simple shapes, because the geometry effects on the thermal shock behavior are great [23,24]. Recently, the thermal shock resistance and the failure mode of ZSG ceramics in shapes of sharp leading edges (SLEs) were successfully investigated by water quenching [8] and a novel water spraying method [25], respectively, wherein the later has been verified that is more reliable for evaluating the thermal shock behavior of SLEs because the specimens in water spraying test have the similar boundary condition and failure mode to that of the samples under simulated environments [26,27]. However, to the authors' knowledge, there is little, if any, literature on the study of the cracking behavior (such as crack initiation and propagation) of ZSG SLEs during water spraying or similar approaches, while this has been recognized as a basic issue to give full play to the potential of SLEs in actual engineering applications.

In this paper, the cracking behavior of ZSG SLEs was systematically investigated by the water spraying method as proposed in our previous work [25]. The entire crack patterns under different thermal shock

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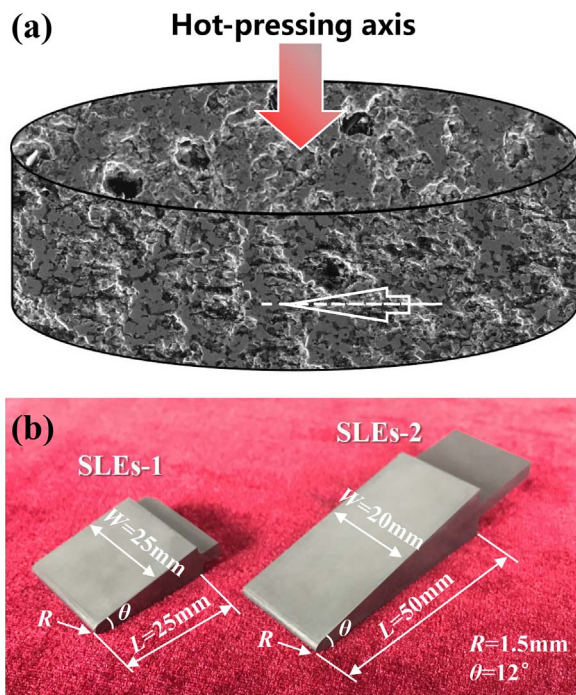


Fig. 1. (a) Schematic diagram of the hot-pressed ZSG ceramic and (b) the macrographs of ZSG SLEs-1 and SLEs-2 with the same tip radius of curvature ( $R$ ) and wedge angle ( $\theta$ ), while different front-edge widths ( $W$ ) and side-edge lengths ( $L$ ).

times were observed successfully. The effects of test condition and flaw on crack initiation were analyzed and discussed in combination with the temperature measurement. The crack propagation (including crack hierarchy and crack speed) was intuitively understood at the same time.

## 2. Experimental procedures

### 2.1. Materials and test samples

The following commercially available powders were used to prepare the  $ZrB_2$ -20 vol% SiC-15 vol% Graphite (ZSG) ceramic:  $ZrB_2$  powder (purity > 99.5%, Northwest Institute for Non-ferrous Metal Research, China) with an average particle size of  $2\ \mu\text{m}$ , SiC powder (purity > 99.0%, Aladdin Industrial Corporation, China) with an average particle size of  $0.5\ \mu\text{m}$  and graphite flake (purity > 99.0%, Kaier Nanometer Energy & Technology Co. Ltd., China) with an average diameter and thickness of  $15\ \mu\text{m}$  and  $2\ \mu\text{m}$ , respectively. The preparation procedures were described elsewhere [28]. The ZSG SLEs were cut from the billet ( $\Phi 150\ \text{mm} \times 40\ \text{mm}$ , as shown in Fig. 1a) using electric discharge machine, then polished to  $0.5\ \mu\text{m}$  with diamond slurries. Because the thermal shock resistance of ZSG SLEs was extremely sensitive to the sampling orientation [25], all of the test samples were machined with the same sampling orientation, that was, the main direction (highlighted with white dotted line in Fig. 1a) perpendicular to the hot-pressing direction. Here, two types of SLEs (as shown in Fig. 1b) were fabricated in this study, wherein the SLEs-2 was mainly used for the investigation of crack propagation behavior because it could provide enough room for crack extension. Furthermore, in order to explore the relationship between the crack initiation and the microstructure, the thermal shock resistances of SLEs-1 with no obvious flaws and SLEs-1 with a notch at the center of the front-edge were compared by the water spraying method, where the notch was fabricated via the laser notching method (see details in Ref. [28]) and could be regarded as an obvious flaw. All edges of the specimens were chamfered to eliminate stress concentration due to machining flaws.

### 2.2. Thermal shock procedures and temperature measurement

Thermal shock tests were conducted on the self-built water spraying platform (see Fig. 2), and the test procedures were described elsewhere

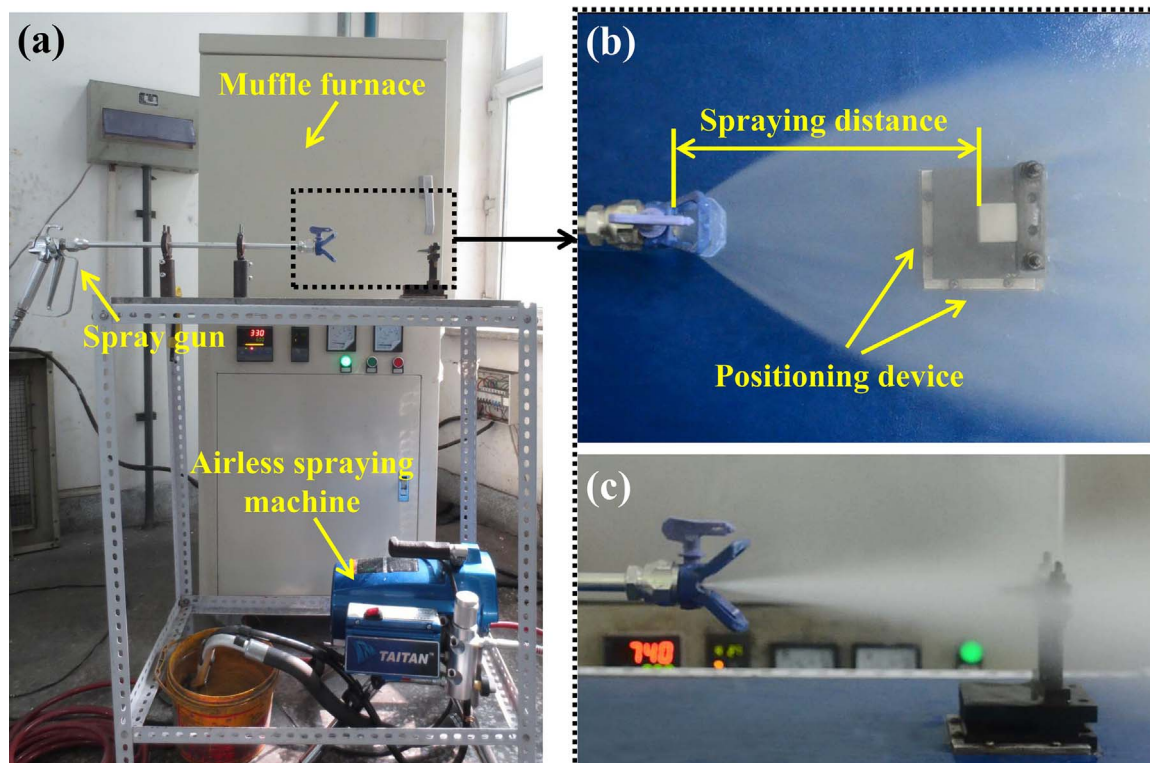


Fig. 2. (a) The self-built water spraying platform and (b) the top view as well as (c) the side view of typical screenshot for the video recording of SLE-1 during thermal shock.

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