Contents lists available at ScienceDirect

### Composites Part B

journal homepage: www.elsevier.com/locate/compositesb

## Temperature dependent fracture toughness of the particulate-reinforced ultra-high-temperature-ceramics considering effects of change in critical flaw size and plastic power



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#### ARTICLE INFO

Keywords: Ceramic–matrix composites (CMCs) Fracture toughness High–temperature properties Analytical modeling

#### ABSTRACT

The fracture toughness of particulate-reinforced ultra-high-temperature-ceramics changes but does not decrease by a constant gradient with the increase of temperature. The change in microstructure and the occurrence of brittle-ductile transition affect the fracture toughness of materials at high temperature. In this paper, a novel temperature dependent fracture toughness model for the particulate-reinforced ultra-high-temperature-ceramics was developed based on the Griffith energy theories and the concept of the maximum storage of energy associated with fracture. The effect of change in microstructure was considered by introducing a non-dimensional value, the ratio of critical flaw sizes of materials at room temperature and high temperature. The effect of plastic power was included in the model. It should be noted that the model has no any fitting parameter and which only needs some basic material parameters such as Young's modulus and specific heat capacity. The predictions of the fracture toughness of the composites in argon or air agreed well with the experimental measurements.

#### 1. Introduction

The particulate-reinforced ultra-high-temperature-ceramics (pUHTCs) have been developed for the leading edge and nose cap materials in hypersonic vehicles owing to their unique properties of low density, high melting temperature and good chemical and physical stability at high temperatures [1–6]. Owing to the inherent brittleness of ceramic, low toughness of pUHTCs is still an obstacle for them to be used widely, especially for the applications in severe environments.

As is well known, the fracture toughness of ceramic composite with a certain microstructure is an intrinsic property, which is supposed to be a material constant. Wang et al. developed a temperature dependent fracture toughness model for the ceramics based on a temperature dependent fracture strength model for the ceramics and Griffith energy criterion [7,8]. This model does not include the parameters of microstructures. The model predictions showed that the fracture toughness of ceramic composites is highly temperature dependent and just decreases as temperature increases. However, the experimental measurements showed that the fracture toughness of pUHTCs changes but does not decrease by a constant gradient with the increase of temperature (as shown in Fig. 1) [9-11]. For example, Neuman et al. reported the fracture toughness of ZrB2-30 vol%SiC composites from room temperature to 1600 °C in air and indicated that the oxidation damage caused by high temperature oxidation such as surface flaws affects the fracture toughness of the composites [9]. In the range of 1400 °C-1600 °C, the degree of change in fracture toughness of the composites decreases owing to the change in oxidation damage. Neuman et al. reported the fracture toughness of hot pressed ZrB<sub>2</sub>-30 vol%SiC-2vol%B<sub>4</sub>C composites from room temperature up to 2200 °C in argon [10]. They showed that the fracture toughness of the ceramic decreased from  $4.9 \text{ MPa m}^{1/2}$  at room temperature to 3.5 MPa m<sup>1/2</sup> at 2200 °C, while without the trend of monotone decreasing in the large temperature range. The occurrence of brittleductile transition above 1600 °C affected the fracture toughness of materials [10]. Neuman et al. also reported the fracture toughness of a ZrB<sub>2</sub>-10 vol%ZrC composite having a room temperature toughness of  $4.8\,\text{MPa}\,\text{m}^{1/2}$  which decreased to  $3.5\,\text{MPa}\,\text{m}^{1/2}\,\text{at}\,\,2200\,^\circ\text{C},$  further increasing to  $3.6\,\text{MPa}\,\text{m}^{1/2}\,\text{at}$   $2300\,^\circ\text{C}$  [11]. And from  $1800\,^\circ\text{C}$  the

https://doi.org/10.1016/j.compositesb.2018.09.049

Received 5 April 2018; Received in revised form 25 August 2018; Accepted 21 September 2018 Available online 22 September 2018

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**Fig. 1.** The existing experimental measurements of fracture toughness of pUHTCs at elevated temperatures [9–11].

plasticity began affect the fracture toughness of materials [11].

The above mentioned experimental measurements indicated that as the temperature increases the microstructures of materials change, such as changes in grain size and cluster size, impurities formation and liquid phase formation at the particle-matrix interface, etc., and the brittleductile transition occurs, leading to the complicated variety of fracture toughness combined with downtrend and uptrend in the measured temperature range [9–11]. It means that when predicting temperature dependent fracture toughness of ceramic composites based on that at room temperature the differences between the microstructures at a certain temperature and room temperature and the plasticity should be considered. The fracture of brittle materials is a quite complicated process, especially at elevated temperatures. It involves the growth of micro and macro voids or cracks, the interface between two phases, the temperature and the high-temperature oxidation [9-14], etc. Currently, the experimental data from different studies on fracture toughness of pUHTCs are fairly dispersed which cannot give us a systematic knowledge of effects of microstructures and their changes. The measurements of fracture toughness of materials at high temperatures are also extremely difficult and costly. On the theoretical side, generally the characterization of effects of microstructures and their changes with temperature on the fracture toughness of materials is very difficult. There has been no one set theory "set in stone" to handle all of these factors in fracture. At present, the challenge and urgent need is to build a rational model for determining fracture toughness of pUHTCs at different temperatures that can reflect the effects of change in microstructure and plasticity.

In this paper, a new model for predicting the temperature dependent fracture toughness of pUHTCs was developed. The effect of change in microstructure was considered by introducing a non-dimensional value, the ratio of critical flaw sizes of materials at room temperature and high temperature. The effect of plastic power was included in the model. The model predictions of temperature dependent fracture toughness of pUHTCs in argon or air agreed very well with the experimental measurements.

#### 2. Theoretical model

In this work, a uniaxial stress state of an external stress applied at infinity on a continuous body containing flaws is studied, and the condition of the tensile-stress distribution at the place of flaw is considered. According to the Griffith energy criterion, the change of elastic energy due to the presence of a crack can be calculated from the expression

$$W_{\rm c} = \frac{-\pi s^2 \sigma^2}{E} \tag{1}$$

where *s* is the crack size of materials; *E* is the Young's modulus of materials;  $\sigma$  is the applied stress.

The surface energy of the crack can be expressed as

$$W_{\rm s} = 4s\gamma \tag{2}$$

where  $\gamma$  is the surface energy per unit area.

τ

Now, the total energy associated with the crack can be expressed as the sum of the elastic energy and the surface energy

$$V = W_{\rm c} + W_{\rm s} = \frac{-\pi s^2 \sigma^2}{E} + 4s\gamma \tag{3}$$

According to the Griffith fracture theories, the fracture strength of brittle materials is generally thought to be controlled by the critical flaw size in the materials. The critical flaw size is the value of the strength limiting flaw i.e. critical flaw in the materials, which is determined by the microstructures such as grain, cluster, pores and the interface conditions. As temperature increases, the change in microstructure occurs, such as changes in grain size and cluster size, impurities formation and liquid phase formation at the particle-matrix interface, etc., leading to the differences in crack propagation path and finally in critical flaw sizes at different temperatures. For the critical flaw, the *s* in Eq. (3) is the critical flaw size, and the  $\gamma$  is the effective fracture surface energy. Then the total energy *W* associated with the critical flaw size can be expressed as

$$W = W_{\rm c} + W_{\rm s} = \frac{-\pi s_{\rm c}^2 \sigma^2}{E} + 2s_{\rm c} G_{\rm IC}$$
(4)

where  $G_{IC}$  is the critical energy release rate or fracture toughness of materials;  $s_c$  is the critical flaw size of materials. The fracture of brittle materials is a complicated process of energy changes. The fracture of brittle materials occurs when the elastic energy  $W_c$  is equal to or larger than the surface energy  $W_s$  as described by the Griffith energy theory. The study showed that there exists a maximum value of elastic energy associated with material failure [15]. Then for typical of brittle fracture it is assumed that there is a maximum storage of energy of materials during fracture, which should include  $W_s$ . For simplicity, here  $W_s$  is used to describe the maximum storage of energy of materials. Thus the maximum storage of energy of materials associated with fracture can be expressed by the critical flaw size. In this work, the changing of critical flaw size with temperature is introduced to characterize the effect of change in microstructure on temperature dependent fracture toughness of pUHTCs.

Bearing in mind that the fracture of brittle materials is considered as bond breaking between the fractured parts, the high temperature causative environment can make failure of materials. As the temperature increases, the microstructures of materials will evolve which thus are different from that at room temperature. This makes it more challenging for a quantitative understanding of fracture properties of materials at high temperatures. The studies have showed that the maximum storage of energy can be measured by both elastic energy and heat energy, and there exists a quantity equivalent relation between them [7,8,16–18]. Based on the above discussion about the maximum storage of energy associated with fracture, a relationship can be obtained as

$$W_{\rm max} = 2s_{\rm c}(T)G_{\rm IC}(T) + kW_T(T)$$
<sup>(5)</sup>

where  $W_{\text{max}}$  is the maximum storage of energy, which should be a constant for a specific materials;  $s_c(T)$  is the critical flaw size of materials at temperature *T*;  $G_{\text{IC}}(T)$  is the critical energy release rate of materials at temperature *T*;  $W_T(T)$  is the heat energy of materials at temperature *T* when the fracture of materials occurs; *k* is the ratio coefficient between the surface energy and heat energy.

The heat energy of materials at temperature T is expressed as

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