Materials Science & Engineering A 627 (2015) 290–295

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Effect of microstructure and slow crack growth on lifetime prediction of monolithic silicon carbide

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ARTICLE INFO

Article history:
Received 3 October 2014
Received in revised form
7 January 2015
Accepted 7 January 2015
Available online 14 January 2015

Keywords:
SiC
Lifetime
Strength
SCG and toughness

ABSTRACT

The lifetime of silicon carbide (SiC) based materials is strongly dependent on the presence of pre-existing flaws or cracks and their extension under an applied load during their service life. The purpose of this work is to determine the long term strength of SiC based materials with different grain morphologies and grain boundary chemistry. Solid state (SS) sintering of SiC with carbon and boron and liquid phase (LP) sintering of SiC using alumina and yttria as additives were used to produce fine and coarse grained materials to clarify the role of chemistry and grain morphology respectively. Fracture toughness, strength and slow crack growth (SCG) data were used to determine lifetime diagrams to accurately evaluate the long term strength behaviour for natural and artificial defects. The LP-SiC materials have more susceptibility to SCG compared to SS-SiC. However, the LP-SiC with coarse grains has a higher toughness and can be used at higher stresses after a large defect has been accidentally introduced. This indicates that the effect of the slow crack growth as a result of introducing oxides on the grain boundaries is not sufficient to alter the ranking between materials in terms of their deterministic allowable stress after such a damage event. On the other hand, the allowable stress in terms of the natural defect population revealed different results for using a low probability of failure (5%) and a much higher probability of failure (63.2%): the ranking of the materials alters when the stress level at which it is to be used changes.

1. Introduction

Silicon carbide's properties such as a high specific stiffness, low thermal expansion and high thermal conductivity, make it an interesting structural material for space applications. Recent examples are the 3.5 m diameter silicon carbide primary mirror on ESA's Herschel telescope [1] and the large tubular ring on the ESA's GAIA mission [2]. Reducing the mass of space components ideally requires that the materials should carry as much load as possible and therefore using a high toughness silicon carbide appears promising. Moreover, due to the wear resistance and chemical inertness of ceramic materials, silicon carbide is becoming a potential candidate for biomedical applications such as orthopaedic implants [3,4], where the components will be stressed for prolonged time in wet environments and here slow crack growth plays an important role in reducing strength and hence shortening the service lifetime of such components.

In addition to high toughness and sensitivity to slow crack growth, one should also consider strength reliability. It is known that the use of ceramic materials is limited in many applications due to the low strength reliability or the large variability in distribution of crack size and shape [5]. For identical specimens under identical loading conditions, the strength can vary unpredictably from sample to sample making it less reliable for engineering design [6]. Therefore, in order to understand the performance of silicon carbide materials and be able to design with them, one may consider the relationship between the variation in mechanical properties in terms of toughness and strength and the slow crack growth behaviour.

The aim of this paper is to evaluate the service life of the different types of silicon carbide as a function of the applied stress so that the relevance of all property measurements carried out can be highlighted. Since flaws can be introduced during production of the materials or by accidental damage after production, the material selection problem will be considered both for artificial flaws introduced by an incident as well as for the natural defects. In both cases the slow crack growth parameters in wet conditions will be used as a worst case scenario in terms of a reduction in the allowable stress for a desired service life.

2. Experimental methods

2.1. Material processing

For solid state sintering 3 wt% carbon and different contents of boron (Grade II, H.C. Starck, Germany) were used [7]. Fine grains

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http://dx.doi.org/10.1016/j.msea.2015.01.013
0921-5093/

were achieved by hot pressing α-SiC (UF-25, H.C. Starck, Germany) at 2050 °C for 30 min with 0.2 wt% boron. A bimodal microstructure with elongated grains was obtained by increasing the boron content to 0.5 wt% and hot pressing β-SiC (BF-17, H.C. Starck, Germany) at 2150 °C for 3 h. The carbon was added in the form of a phenolic resin (CR-96, Novolak, Crios Resinas, Brazil) with 50% carbon yield after pyrolysis at 400 °C for 1 h in argon atmosphere.

For the liquid phase sintered materials, a mixture of 6 wt% of aluminium oxide (AKP-30, Sumitomo, Japan) and 4 wt% yttrium oxide (Grade C, H.C. Starck, Germany) was used as sintering additives. The fine equi-axed material was produced by hot pressing α-SiC at 1950 °C for 30 min, whereas the larger grain material was obtained by hot pressing β-SiC at 2050 °C for 3 h [8–11].

The SiC powder and additives were mixed by ball milling for 24 h using silicon nitride media (Union Process, Akron, USA) for the non-oxide mixture and alumina media for the oxide mixture (Union Process, Akron, USA) both in methyl ethyl ketone (VWR, London, UK). The slurries were dried by means of rotary evaporator R-20 (BUCHI Rotavapor, Switzerland). After drying, the powders were crushed and sieved through a 100 μm sieve. Hot pressing was conducted in an 80 mm graphite dies at heating rate of 10 °C min^{-1} under flowing argon gas in a graphite hot press (FCT, Rauenstein, Germany) under 25 MPa pressure.

2.2. Characterisation

The microstructure was examined using a scanning electron microscope (SEM, S-3400N, Hitachi, Japan). The specimens were polished to 1 μm using diamond suspension and chemically etched using boiling Murakami's solution [12]. The average grain size of 300 grains was calculated using the linear intercept method according to the approach of Mendelson [13].

The density was determined by Archimedes method with distilled water as the immersion medium according to ASTM standard C8300-00 [14]. The densities were compared to a theoretical density value of 3.28 g/cm^3 and 3.21 g/cm^3 for the SiC with oxide mixture and non-oxide mixture respectively.

Fracture toughness measurements in 3 point bending were performed according to ASTM 1421 using the single notched edge beam (SENB) method [15]. Specimens were 4 mm by 3 mm by 40 mm with root notch radius of 15 μm achieved by sharpening the notch using razor blade machine. A total number of five specimens for each material were tested in air at cross head speed of 0.05 mm min^{-1}. The average notch length was measured from both fracture surfaces.

The flexural strength was measured using 4 point bending according to ASTM C1161-02C. Specimens measuring 4 mm by 3 mm by 40 mm and their edges were bevelled to eliminate any stress concentration from machining. The tensile surfaces were polished to 1 μm using diamond suspension. A total number of 17 specimens were tested in air for each material at cross head speed of 1 mm min^{-1} and the average strength was calculated. The Weibull distribution [16]

$$P_f = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{1}$$

was used to obtain the characteristic strength (σ_0) and Weibull modulus. In this expression, P_f is the probability of failure, which was estimated for each sample using [17]

$$P_j = \frac{j-0.3}{N+0.4} \tag{2}$$

where, j represents the rank number of a sample in terms of strength (j=1 for the lowest strength sample) and N is the total number of tested samples.

Slow crack growth (SCG) testing was carried out using the constant stress rate test [18]. Specimens were 4 mm by 3 mm by 40 mm and edges were bevelled and polished to 1 μm using diamond suspension. A 2 kg Vickers indent was placed in the centre of the tensile face with the orientation of the indent such that 2 of the cracks emanating from the corners created a crack perpendicular to the applied tensile stress. The indentations were made in air with 10 s holding time immediately prior to starting the bend test immersed in distilled water. The fracture stress was measured with an inner span of 10 mm and outer span of 20 mm at cross head speeds ranging from 0.001 mm min^{-1} to 1 mm min^{-1}. Additionally, inert strength values in oil were measured at cross head speed of 1 mm min^{-1}. For every test, the fracture force and time were recorded in order to calculate the fracture stress and stress rate. A total of 5 specimens were tested at each stress rate for each material. To determine the slow crack growth parameter, the following was used [19]:

$$v = AK_I^n \tag{3}$$

where, v is the crack velocity, K_I is the applied stress intensity factor, A and n are material and environment dependent subcritical crack growth parameters. The slope of log (failure stress) versus the log (stress rate), n', was converted using [20]

$$n = \frac{4n'-2}{3} \tag{4}$$

The SCG parameter A was calculated using the following approach [21,22]:

$$\sigma_f^{(n+1)} = \left[(n+1)\dot{\sigma}\sigma_{inert}^{(n-2)}\frac{2}{(n-2)AY^2K_{IC}^{(n-2)}}\right] \tag{5}$$

where, σ_f is the fracture stress, $\dot{\sigma}$ is the applied stress rate, K_{IC} is the fracture toughness, σ_{inert} is the inert strength measured in oil and Y is a geometric constant depending on the type and shape of flaw. In this analysis, Y is taken as 1.12, which corresponds to small crack length [23].

The size of the pre-existing defect, a_i can be calculated from the inert strength, σ_{inert}, and toughness, K_{IC}, of the material after introduction of the damage:

$$a_i = \frac{1}{\pi}\left(\frac{K_{IC}}{Y\sigma_{inert}}\right)^2 \tag{6}$$

During slow crack growth in wet environment, such pre-existing cracks will grow at stress level much lower than the critical value and fracture will occur once the crack length reaches a critical value at a given stress. Hence, the defect size where failure will occur can be calculated from:

$$a_f = \frac{1}{\pi}\left(\frac{K_{IC}}{Y\sigma_{service}}\right)^2 \tag{7}$$

From fracture mechanics the following applies for a specimen under tensile loading (σ) with a certain crack length (a) [19]

$$K_I = \sigma Y\sqrt{a\pi} \tag{8}$$

Substituting Eq. (8) into Eq. (3) and integrating with respect to crack length and time, the predicted lifetime, t_{life} can be obtained using the following [19]:

$$\frac{a_f^{-n/2+1} - a_i^{-n/2+1}}{1-n/2} = A\sigma^n Y^n \pi^{n/2} t_{life} \tag{9}$$

where, a_f and a_i are the final and initial crack lengths and can be calculated using Eqs. (6) and (7). The measured inert strength in oil from the constant stress rate test and the measured K_{IC} values from SENB test are used to obtain initial crack length, a_i and the variable service stress values are used to determine a_f. For each calculated final crack length and using the slow crack growth