Materials Science & Engineering A 636 (2015) 203–206

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Slow crack growth behavior of 3Y-TZP in cryogenic environment using dynamic fatigue and indentation technique

Jingya Gui [a], Sai Wei [a], Zhipeng Xie [a,*], Weijiang Xue [a], Guanwei Liu [b]

[a] State Key Laboratory of New Ceramics and Fine Processing, School of Materials Science and Engineering, Tsinghua University, Beijing 100084, China
[b] Energy Storage Tech Center, Hanergy Global R&D Center, Beijing 102209, China

ARTICLE INFO

Article history:
Received 14 March 2015
Received in revised form
25 March 2015
Accepted 25 March 2015
Available online 2 April 2015

Keywords:
SCG
Dynamic fatigue
Indentation
Cryogenic temperature
3Y-TZP

ABSTRACT

The dynamic fatigue of 3 mol% Y_2O_3 stabilized tetragonal zirconia ceramic (3Y-TZP) was assessed at 293 K and 77 K by testing the same batch of specimens in four-point flexure at various stress rates. The crack propagation velocities were also measured using Vickers indentation specimens under dynamic-loading conditions in ambient and cryogenic environment. The slow crack growth (SCG) parameter n at 77 K was larger than that at 293 K. Also, the crack propagation velocity under cryogenic circumstance is much slower than that under ambient circumstance. The experimental results suggest that the 3Y-TZP exhibits a lesser degree of SCG behavior in cryogenic environment. It should be ascribed to two reasons. The negligible effect of stress corrosion and crack tip shielding mechanism induced by temperature. The high SCG resistance in cryogenic environment makes the 3Y-TZP a promising candidate material for long-term service in cryogenic structural applications.

1. Introduction

For many structural ceramic components in service, their use is often limited by lifetimes that are controlled by a process of slow crack growth (SCG) [1]. SCG stem from pre-existing flaws, and can deteriorate the strength of the structural ceramics. The SCG behavior of zirconia-based ceramics has been extensively studied because of its widespread availability in the structural forms [2–4]. Series of studies have indicated that environmental effects, such as the influence of water in air can modify the energy required to break bonds between atoms in zirconia, leading to slow crack growth and delaying failure [5–7]. Such an effect is referred to as stress corrosion, a process involving the stable growth of pre-existing flaws. However, to date, almost all available previous investigations were carried out at ambient temperature or elevated temperatures. It is well known that in recent years, cryogenic engineering is attracting increasing attention in nuclear fusion, superconducting fields as well as in aerospace fields, etc. Our previous research revealed that 3Y-TZP is characterized not only by its excellent high strength and fracture toughness, but also by a pronounced *R*-curve behavior at cryogenic temperatures, suggesting it could be one of the most promising candidate materials in cryogenic structural applications [8,9]. Unfortunately, despite the promising properties of 3Y-TZP ceramics for cryogenic structural applications, the absent investigations of SCG behavior under cryogenic circumstance severely limit the performance and hinder the use of such materials, particularly in applications demanding long-term service. The present study was motivated by such desire to better understand the SCG behavior of the 3Y-TZP in cryogenic environment.

2. Experimental procedure

2.1. Specimens

A batch of commercial 3Y-TZP ceramic materials was procured from Sicer Advanced Materials Co. Ltd., Shandong. China. The powders were dry pressed at 50 MPa and cold isostatic pressed at 200 MPa, then were sintered at 1560 °C for 2 h. The bulk density was ~98% of the theoretical full density as determined by the Archimedes method. Subsequently, the bulk was cut into rectangular shaped specimens with dimension of 3 mm × 4 mm × 45 mm, and the prospective tensile surfaces were mirror-polished with 1μm diamond paste to optimize the surface reflectivity for crack observation.

2.2. Dynamic fatigue testing

The SCG behavior can be approximated by the empirical power-law relation [10]

$$v = \frac{da}{dt} = A\left[\frac{K_I}{K_{IC}}\right]^n \quad (1)$$

where v, a, and t are crack velocity, crack length and time, respectively. A and n are the material/environment dependent slow crack

* Corresponding author. Tel.: +86 10 62799031; fax: +86 10 62771160.
E-mail address: xzp@mail.tsinghua.edu.cn (Z. Xie).

http://dx.doi.org/10.1016/j.msea.2015.03.099
0921-5093/

growth parameters, K_I and K_{IC} are stress intensity factor and the fracture toughness of the material, respectively.

The stress intensity factor can be expressed as

$$K_I = Y\sigma\sqrt{a} \tag{2}$$

where σ is a uniform remote applied stress, Y is a geometry factor related to flaw shape and its orientation with respect to the direction of applied stress.

In this study, the SCG parameters of the 3Y-TZP under different environments were indirectly obtained through dynamic fatigue approach in which strength is measured as a function of stress rate. For dynamic fatigue testing, $\sigma(t) = \dot{\sigma}t$, using Eqs. (1) and (2) with some manipulations, a relationship under slow crack growth can be determined as follows:

$$\sigma_f^{n+1} = B(n+1)\sigma_i^{n-2}\dot{\sigma} \tag{3}$$

where σ_f is a fracture strength related to the corresponding stress rate, σ_i is an inert strength, and B is a material/environment parameter.

Making a transformation for Eq. (3), we can get an equation as follows:

$$\log\ \sigma_f = \frac{1}{n+1}\log\ \dot{\sigma} + \log\ D \tag{4}$$

where $\log\ D = (1/(n+1))\log\ [B(n+1)\sigma_i^{n-2}]$. Therefore, the SCG parameters n and D can be determined by a linear regression analysis based on Eq. (4) when the strength is plotted as a function of stress rate on a logarithmic scale.

According to ASTM C1368 [1], as displayed in Fig. 1(a), specimens were fractured in four-point rectangular beam flexure with an inner span of 20 mm and outer span of 40 mm using a universal testing machine (SUNS UTM4000, China) at room temperature (293 K) and cryogenic temperature (77 K). The low temperature was obtained by a test chamber filled with liquid nitrogen. The relative humidity was ~30%. In order to measure the environment dependent slow crack growth parameter, the specimens were tested at a displacement rate of 0.01, 0.06, 0.6 and 6 mm/min at 293 K and 0.01, 0.06, 0.1, 0.6, 2 and 6 mm/min at 77 K. There were 7 bars tested at each displacement rate. The fracture strength σ_f of each specimen was calculated from the fracture load. For each specified applied test rate σ_i, the actual loading rate was determined via a stress–time curve for each test and then calculated into the corresponding stress rate. The micro-topographies of the fracture surface after the dynamic fatigue measurement for 293 K and 77 K were obtained via Scanning

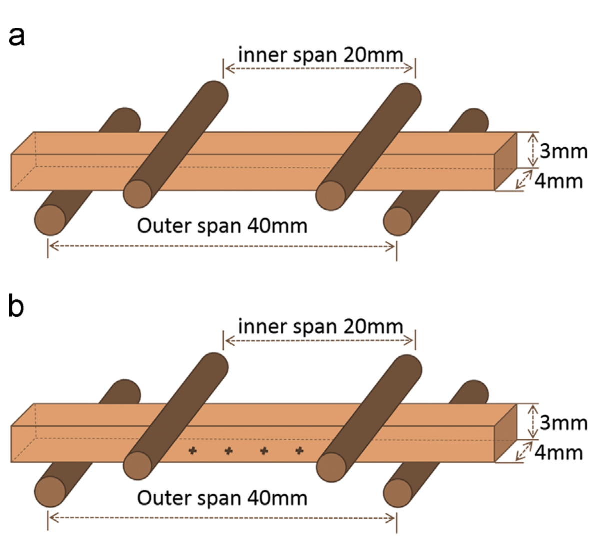

Fig. 1. Schematic illustration for (a) dynamic fatigue testing and (b) measurements of crack velocities.

Electron Microscope (SEM, Leo 1530, Zeiss, Germany), to figure out the fracture mechanism of the 3Y-TZP at cryogenic temperatures.

2.3. Measurements of crack velocities under dynamic fatigue

The crack velocities have been measured under dynamic-loading conditions in ambient and cryogenic environments. The test was aimed to identify environmental effects on crack propagation. The only difference from dynamic fatigue test is that the specimens used here have four Vickers indentations that were made on the inner span of the tensile surface uniformly using a Vickers hardness tester (Tukon2500B, Wilson). Each indentation was made in air at a load of 30 kg for 15 s to get a semi-elliptical surface crack. The indentations were carefully oriented to make sure that the cracks induced by the indentations were perpendicular to the edges of the specimen. As displayed in Fig. 1(b), the distance between neighboring indentations was 4 mm to minimize interactions among the adjacent cracks. 2 specimens were tested at each displacement rate at 293 K and 77 K, respectively. After fractured from one of the indentations, three indentations were left on each remnant bar and the cracks propagated to the instability crack length [11]. The pre-crack length c_0 and the instability crack length c_m were precisely measured by an optical microscope (OM, BX50, Olympus, Japan). The crack velocity v was calculated from the crack increment and the fracture time.

2.4. Crack growth resistance at instability crack length

The specific crack growth resistance at the instability point when the applied stress reaches the fracture stress can be calculated from a quasi-static equilibrium equation [11]

$$K_R(c) = \frac{\chi P}{c^{3/2}} + 2\Omega\sigma\left(\frac{c}{\pi}\right)^{1/2} \tag{5}$$

where P is the indentation load, the fracture strength σ_f' of each specimen is calculated from the fracture load. For each fracture strength σ_f', there is a corresponding instability crack length c_m, χ is a dimensionless indenter-material constant defined as [12]

$$\chi = \delta\left(\frac{E}{H}\right)^{1/2} \tag{6}$$

where E and H are Young's modulus and hardness, δ is a non-dimensional constant that depends on the indenter geometry and Poisson's ratio of the material, respectively. Ω is a stress-intensity coefficient that can be calculated using a method proposed by Newman and Raju [13]. Poisson's ratio of the material is 0.3 [14]. As discussed further, the mentioned above of getting a semi-elliptical surface crack would be reasonably approximated by the following equation [15,16]:

$$\frac{a}{c} = 1 - \frac{a}{w} \tag{7}$$

where a is the depth of the semielliptical crack, c is the surface crack length, and w is the bar width.

3. Results and discussion

According to Lawn et al., the n value characterizes how extensively the lifespan of the material is affected by the environment [17]. A larger n means a lesser degree of SCG on the assumption of the same K/K_{IC} level based on Eq. (1). Fig. 2 shows dynamic fatigue curves for 3Y-TZP at 293 K and 77 K. The data obtained from 293 K can be recorded by a linear regression analysis on a logarithmic plot of fracture stress as a function of the stressing rate. According to Eq. (4), the n at 293 K was determined from the slopes of the fitted curve and its value was about 28. It should be noted that the data raised at first and then declined at 77 K. To figure out the real tendency, two