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Creep-related micromechanical behavior of zirconia-based ceramics investigated by nanoindentation

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Abstract

The creep-related micromechanical behavior of zirconia ceramics (ZrO_2) was investigated by nanoindentation based on continuous stiffness measurements at different strain rates at room temperature. The elastic modulus was found to decrease continually and the hardness reached a constant value with increasing depth. Moreover, the elastic modulus and hardness both increased at a given indentation depth with increasing loading rate and exhibited similar trends. The creep strain rate sensitivity was independent of the loading rate. The primary deformation mechanism is grain boundaries sliding accompanied by intergranular dislocation due to the existence of amorphous phases in the grain boundaries. Meanwhile, the mechanism of stress-directed diffusion acts an important role during the creep process.

Keywords: ZrO_2; Nanoindentation; Creep; Grain boundaries

1. Introduction

Zirconia ceramics are increasingly being considered for structural and engineering applications owing to their excellent properties, such as high hardness, low wear resistance, low coefficient of friction, high elastic modulus, chemical inertness, low thermal conductivity, and high melting point [1–3]. The time-dependent plastic deformation of materials, termed creep, is an important design criterion for high-temperature, high-stress, or long-term service conditions [3]. A great deal of research on the plastic behavior of zirconia ceramics at high temperature, especially for yttria-stabilized tetragonal zirconia, has been explored [3–8]. Grain boundary sliding has been consistently recognized as the primary deformation mechanism. Extensive grain boundary sliding needs 'accommodation processes' such as dislocation or diffusion to prevent cavitation [7]. Plastic deformation in zirconia ceramics is also considered to relate to tetragonal-to-monoclinic (T–M) phase transformation under critical stress [8].

Despite its importance, the creep related to micromechanical behavior in zirconia ceramics at room temperature has received little attention. Nanoindentation has been widely used to analyze the mechanical properties for various materials, such as metals, ceramics, polymers, and composites [9,10]. The nanoindentation testing apparatus is also an effective tool for investigating room-temperature creep behavior of materials with very high force and displacement resolutions. Based on the application of nanoindentation on studying elasticoplastic deformation, some researchers attempted to utilize such technique to investigate time-dependent deformation (i.e., creep) behavior [11,12]. Additionally, the stress exponents obtained by the nanoindentation technique have been found in general agree with those from conventional creep tests because the underpinning deformation mechanism was the same in a wide range of materials [13–16]. These consistencies were useful to measure micromechanical creep deformation and also gave an attraction of understanding the deformation mechanisms of the materials by nanoindentation [17]. Meanwhile, nanoindentation based on continuous stiffness measurements (CSM), as proposed by Oliver and Pharr [18], can be used to continuously measure the hardness and elastic modulus with

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increasing the indention depth. Hence, nanoindentation is a convenient method to investigate the creep-related micromechanical characteristics of materials.

Considering the descriptions above, the creep-related micromechanical behavior of zirconia-based ceramics at room temperature was investigated using nanoindentation based on CSM in the present study. On the basis of the results obtained, the strain rate sensitivity was demonstrated and a possible mechanism for indentation creep was discussed as well.

2. Experimental procedures

Commercial zirconia-based ceramics stabilized with 2.5 mol % CeO_2 were used and the sample surface was mechanically polished to a mirror finish. Nanoindentation tests were carried out using a Nano Indenter G200 test system with triangular pyramid Berkovich diamond indenter (Agilent Technologies). There are three common ways to carry out indentation tests; namely, with constant load (P), constant loading rate ($\dot{P}$), and with constant strain rate ($\dot{\varepsilon}$; i.e., CSM). It has been proposed that the constant indentation strain rate condition should be applied by performing load-controlled indentations with a constant loading rate ($\dot{P}/P$) [19,20].

To understand the effect of strain rate on the micromechanical behavior of the ceramics, indentation tests were performed based on CSM under five loading rates ($\dot{P}/P$) of 0.01, 0.02, 0.05, 0.1, and 0.2 s^{-1}. The maximum indentation depth and Poisson's ratio of the material were 2750 nm and 0.20, respectively. As shown in Fig. 1, for each strain rate, the indenter remained at the respective peak load, P_{max}, for 200 s before it was withdrawn from the specimen. To eliminate the influence of thermal drift, the peak load was first reduced to 0.1 P_{max} at which it was held for 60 s before being reduced to zero during the unloading stage.

In Oliver and Pharr's nanoindentation model [21], the contact area, A_c, is defined by a polynomial function $\left(A_c = \sum_{n=0}^{8} C_n h_c^{\frac{1}{2^n-1}}\right)$ of the contact depth, h_c, which is given by:

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S} \tag{1}$$

where P_{max} is the peak load, h_{max} is the displacement at the maximum load, ε is a constant equal to 0.75 and S is the contact stiffness. Based on the CSM technique, S can be obtained as:

$$S = \left[\frac{1}{(P_0/Z_0)\ \cos\ \phi - (K_s - m\omega^2)} - \frac{1}{K_f}\right]^{-1} \tag{2}$$

where P_0 is the amplitude of the harmonic excitation force, Z_0 is the response to the displacement amplitude (≈ 2 nm), Φ is the phase shift between the harmonic displacement and the harmonic excitation force, $\omega = 2\pi f$ is the angular frequency (45 Hz), and K_s, K_f, and m are the spring constant in the vertical direction, frame stiffness, and mass of the indenter, respectively.

For a Berkovich indenter, A_c is given by:

$$A_c = 24.56 {h_c}^2 \tag{3}$$

The hardness is defined as:

$$H = P/A_c \tag{4}$$

The reduced modulus, E_r, is calculated from the unloading data as:

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A_c}} \tag{5}$$

where β is a constant equal to 1.034 for a Berkovich indenter. The modulus, E, of the sample can easily be determined from the following relationship:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \tag{6}$$

where E_i and ν_i are Young's modulus and Poisson's ratio of the material and the indenter, respectively ($E_i = 1141$ GPa and $\nu_i = 0.07$).

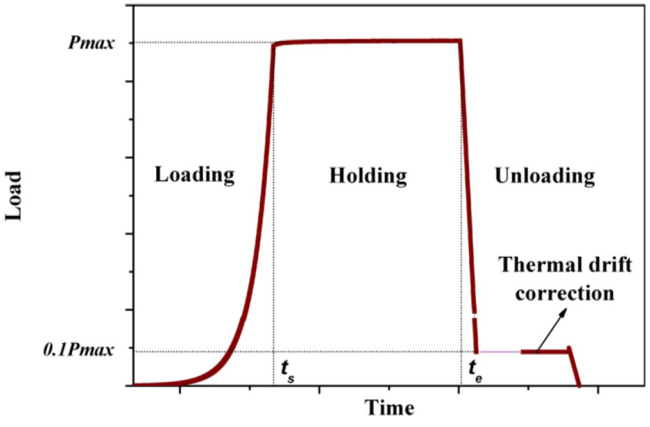

Fig. 1. Schematic load–time curve for the indentation tests.

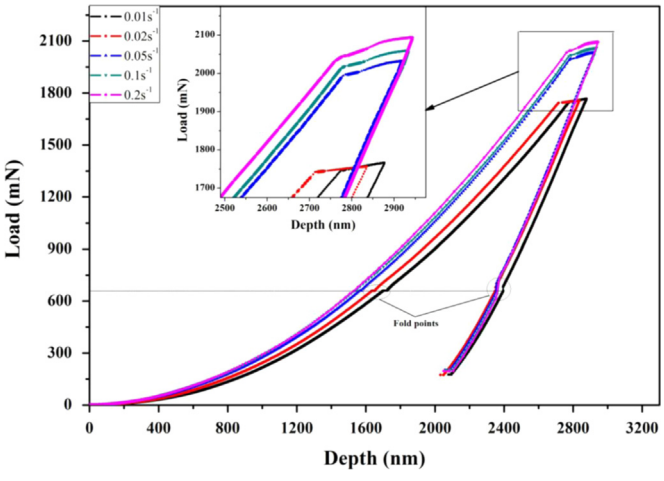

Fig. 2. Nanoindentation load–depth curves at different $\dot{P}/P$.