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High temperature internal friction measurements of 3YTZP zirconia polycrystals. High temperature background and creep

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Abstract

This work focuses on the high-temperature mechanic properties of a 3 mol% yttria zirconia polycrystals (3YTZP), fabricated by hot-pressureless sintering. Systematic measurements of mechanical loss as a function of temperature and frequency were performed. An analytical method, based on the generalized Maxwell rheological model, has been used to analyze the high temperature internal friction background (HTB). This method has been previously applied to intermetallic compounds but never to ceramics, except in a preliminary study performed on fine grain and nano-crystalline zirconia. The HTB increases exponentially and its analysis provides an apparent activation enthalpy which correlates well with that obtained from creep experiments. This fact shows on the one hand the plausibility of applying the generalized Maxwell model to ceramics, and on the other hand indicates the possibility of using mechanical spectroscopy as a complementary helpful technique to investigate the high temperature deformation mechanism of materials.

Keywords: Mechanical spectroscopy; ZrO_2; Ceramics; Internal friction; Creep

1. Introduction

Ceramics materials possess a unique blend of physical, chemical and mechanical properties (high wear-resistance, hardness, strength, thermal-chemical stability, etc.) which makes them suitable in a wide variety of technological applications, especially under extreme conditions. Moreover, they can exhibit a superplastic behavior by decreasing the grain size. This fact was first reported for yttria tetragonal zirconia polycrystal,[1] and then this material has been the subject of numerous studies.[2–5] As a result of these investigations, grain boundary sliding has been recognized as the main deformation mechanism for yttria tetragonal zirconia polycrystal, with an apparent activation enthalpy between 5 and 7 eV, depending on the experimental conditions.[6] From a physical and technological point of view it becomes essential identifying the deformation mechanisms that operates in a material in order to predict its mechanical response under certain conditions. Creep provides information to understand how a material deforms at high temperatures because some parameters intrinsically related to the operating deformation mechanisms, like the activation energy and the stress exponent, can be obtained from these experiments.

Mechanical spectroscopy is a technique which operates in the anelastic range, applying an oscillating stress to a specimen and then inducing the structural defects motion (point defects, dislocations, etc.). The mobility of these defects, which is intrinsically related to the mechanical properties of material, can be investigated by this technique. Furthermore, mechanical spectroscopy has also the advantage of being a non-destructive technique, and therefore very useful when not much material is available and many experiments have to be performed under different conditions. At high temperatures, the HTB analysis provides

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an apparent activation enthalpy similar to the one measured by creep tests. Thus, Schaller et al.[7] studied 3 mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP), with an average grain size of 0.29 μm, obtaining an apparent enthalpy energy of about 5.6 eV. Donzel et al.[8] studied the same material but with different grain size and they managed to observe a relaxation peak, called Peak II, related to the well-known threshold stress exhibited by this material. A more extended study was performed by Donzel et al.[9] on YTZP with different grain sizes and sintered with different amounts of intergranular phase. They observed another relaxation peak (Peak I) in samples with low purity which was attributed to the glass transition of the amorphous pockets. These two peaks were already observed in silicon nitride,[10,11] where a relationship between standard equation for high temperature strain rate and mechanical loss was established. Recently, a preliminary study performed on micro- and nano-crystalline zirconia was reported, where nano-3YTZP specimens exhibited a higher increase of the internal friction HTB compared to micro-3YTZP, due to a higher effective grain boundary area associated to the former.[12]

In this work we show the experimental results obtained by mechanical spectroscopy performed on 3YTZP and the analysis using the generalized Maxwell model. We compare them to the ones previously obtained by means of others analytical methods, and also to the activation parameters obtained by creep in 3YTZP. So, the purpose of this paper is twofold. Firstly, we want to show that mechanical spectroscopy is a complementary technique together with creep experiments which seems to be very valuable to investigate the high temperature deformation mechanisms, so important for technological applications. And secondly, on the light of the coherent results obtained in this work, the generalized Maxwell model can be applied for the experimental data analysis in ceramics.

2. Experimental details

2.1. Material

High purity commercial 3 mol% yttria-zirconia powder, with 30 nm particle size, was supplied by Tosoh, Japan. Powders were prepared by cold isostatic pressing at 300 MPa. The compact pieces were sintered in a conventional super-kanthal furnace at 1450 °C for 4 h, with a heating and cooling rate of 600 °C/h. Dense specimens were fabricated with a density, measured using Archimedes' method, close to 100%.

2.2. Microstructural characterization

The microstructure of the sintered samples was observed by high resolution scanning electron microscopy HRSEM (Model HITACHI S5200, Electron microscopy service, University of Seville, Spain) operating at 5 kV using backscattered and secondary electrons. Prior to observation, the samples were polished with diamond paste of grain sizes down to 1 μm. Then, they were annealed for 30 min at 1400 °C to reveal grain boundaries. To study the 3YTZP grain morphology, the grain diameter (d; defined as $d = (4 \times \text{area}/\pi)^{1/2}$), and the form factor ($F$; defined as $F = 4\pi \times \text{area}/(\text{perimeter})^2$) were measured from HRSEM micrographs.

2.3. Mechanical spectroscopy

Sintered samples were cut for mechanical spectroscopy with dimensions of 30 mm × 5 mm × 1.3 mm. The equipment used in this work has been developed few years ago[13] and is based on an inverted torsion pendulum. It can measure the internal friction and the dynamic shear elastic modulus in two different working modes: (a) as a function of temperature (300–1800 K) at imposed frequency, and (b) as a function of frequency (10^{-3}–10 Hz) in isothermal conditions. Here we describe its main components:

- An anti-vibratory system to provide stability to the pendulum, composed by three Newport I-2000 laminar flow pneumatic dampers and two synthetic marble flagstones.
- A high vacuum system (10^{-4} Pa) is essential for many reasons: (i) to reduce the noise coming from the transmission of external acoustic vibrations (ii) to avoid overheating of the mechanical system and (iii) to prevent sample oxidation or contamination at high temperatures. This vacuum system consists of a Boc Edwards RV8 rotary vane pump and a Leybold Turbovac 340 M Turbomolecular pump, provided by two pirani and penning vacuum sensors.
- A graphite resistance tubular furnace, which by radiation can heat up to 1800 K. The temperature control system is carried out with a stability of ±0.1 K by a Eurotherm 3504 PID controller, which regulates the power sent to the furnace by a SM 300-10D Delta Electronika supply.
- An excitation–detection system. A permanent magnet is excited by two Helmholtz coils driven by a KEPCO power supply, which amplify the sinusoidal signal from a Solartron frequency analyzer. The response signal due to the torsion angle is detected by the displacement of the laser beam spot on a photocell. This response is filtered, magnified and sent to the frequency analyzer which measures the phase lag between the excitation and the response signal. The oscillating strain is measured through an Agilent 34970A voltmeter.

In this work the internal friction of 3YTZP has been measured at temperatures between 800 and 1650 K and oscillating stress frequencies between 0.01 and 3 Hz.

3. Experimental results and analysis

The microstructure of the sintered-3YTZP shown in Fig. 1 predominantly consists of equiaxed grains in the submicrometric size range. The analysis of the grain size and form factor distributions showed an average grain size of 0.6 μm and a shape factor around 0.7. Neither pores nor cavities are observed between grains, which is coherent with the full densification measured by Archimedes' method.

Fig. 2 shows the mechanical loss measurements performed on 3YTZP as a function of the temperature for an oscillating stress frequency of 0.3 Hz. The dynamic shear elastic modulus