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Acta Materialia 60 (2012) 5867–5876

Acta MATERIALIA

www.elsevier.com/locate/actamat

Elastic properties of β-eucryptite in the glassy and microcracked crystalline states

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Received 30 December 2011; received in revised form 21 May 2012; accepted 10 July 2012
Available online 23 August 2012

Abstract

Amorphous and crystalline β-eucryptite ($LiAlSiO_4$) specimens were prepared with controlled grain sizes and varying levels of microcracking, and their elastic moduli were determined using resonant ultrasound spectroscopy. It was found that the relationship between Young's modulus, Poisson's ratio and degree of microcracking in these materials can be described well with fracture-mechanics-based models. It was also found that if glassy β-eucryptite is considered to be a microcracked medium in which broken Si–O bonds, with respect to the crystalline material, constitute microcracks, then its elastic properties can be described equally well by these models. Such considerations are explained by noting the differences in atomic bond density among the different states of the material and by accounting for differences in strain energy release rate measurements on glass and ceramic specimens.
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Keywords: Ceramics; Silicates; Elastic behavior; Fracture; Modeling

1. Introduction

The relationship between the elastic properties of a precursor glass and the corresponding crystalline ceramic is not known, even though it is recognized that the nature of the atomic bonding forces between the two types of materials are similar [1]. Glasses form extended three-dimensional (3-D) networks like crystals, but the network does not possess translational periodicity [2]. The elastic properties of glassy oxides have been widely studied and the effects of the network structure and short-to-medium-range order on their elastic properties were recently reviewed by Rouxel [3]. Charles [4] demonstrated that the Young's modulus of various silica glasses was directly proportional to the density of Si–O–Si bridges. Makishima and Mackenzie [5,6] proposed a model to calculate the Young's modulus of oxide glasses based on the dissociation energy of the oxide constituents per unit volume and the atomic packing density. The Makishima and MacKenzie model was developed by analogy with ionic crystals, where the elastic modulus is twice the binding energy per unit cubic volume of r_o^3, where r_o represents the atomic spacing in the crystal [7]. Bridge et al. [8] developed a bond compression model based on the argument that several oxide glasses are three-dimensionally connected ring structures with the average ring size of the glassy solid being similar to the corresponding crystalline form of the oxides. The above models have been successfully applied to describe the elastic properties of numerous glassy systems as a function of their composition [9–11].

In general, the Young's moduli of glasses are lower than those of the corresponding crystalline ceramic. Microcracking is another phenomenon that lowers the Young's modulus and Poisson's ratio value of solids [12,13]. Microcracking is often displayed by ceramics that possess anisotropic coefficients of lattice thermal expansion, leading to grain-level internal stresses that cause bond rupture [14]. β-Eucryptite ($LiAlSiO_4$) is a well-characterized ceramic [15,16] where tailored levels of microcracking can be induced in the crystalline state by thermal treatments [17].

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1359-6454/$36.00 © 2012 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.actamat.2012.07.028

In the present investigation, microcracked structures of β-eucryptite were produced via devitrification. This processing route was chosen to prevent confusing effects like porosity, which might result from other processing methods, from influencing the elastic properties. In addition to glass with composition of β-eucryptite, crystalline material with three different grain sizes (and microcracking levels) were produced. The Young's modulus and Poisson's ratio of all four materials follow model predictions of effective elastic properties of microcracked materials [18]. In this paper, we discuss the reasons that make glassy $LiAlSiO_4$ follow the effective elastic property trends of microcracked solids. In particular, it is argued that the glassy state of this material can be considered a microcracked condition of the crystalline state in which microcracks are of the size range of broken bonds/rings in silica. This argument is supported by additional physical considerations, including model calculations and fracture surface energy measurements on glass and ceramic specimens.

2. Experimental procedure

Glass specimens in plate form were provided by Corning Glass Company (Corning, NY) for crystallization to β-eucryptite. The approximate composition of the glass specimen was 48 wt.% SiO_2, 38 wt.% Al_2O_3, 10 wt.% Li_2O, and 4 wt.% TiO_2 [17]. The glass was sectioned into rectangular parallelepiped specimens with approximate dimensions of 15 mm × 25 mm × 4 mm. A series of specimens were prepared with varying grain sizes and levels of microcracking by following prescribed thermal treatments [17]. The characteristics of the specimens, their heat treatments and resultant grain sizes are summarized in Table 1. The ceramic specimens were prepared ceramographically and examined by optical and scanning electron microscopy (SEM). Grain-size measurements were performed by the intercept method on optical images of as-processed and thermally etched ceramic specimens. Microcrack populations on specimens with different thermal treatments were characterized by SEM imaging on ceramographically prepared specimens. Linear crack densities were determined by introducing ten randomly oriented line scans in two SEM images and then counting the number of cracks intersecting the lines. The chemical composition of crystalline phases was determined by energy dispersive X-ray spectroscopy (EDS).

The elastic properties of the prepared specimens were determined as a function of temperature by the resonant ultrasound spectroscopy (RUS) technique [19]. A Quasar RI system was employed for gathering the RUS spectra [20]. The specimens were assumed to be elastically isotropic and the elastic constants were calculated with a weighted non-linear error minimization routine (Magnaflux, Albuquerque, NM). The root mean square (RMS) error of all the RUS spectra fits was less than 1%. Values of the Young's modulus, Poisson's ratio and shear modulus were determined every 100 °C up to 1000 °C for the ceramic specimens and up to 600 °C for the glass specimens because above 600 °C, the peaks from the RUS spectra of the glass specimens became too broad for RUS analysis. For the ceramic specimens, additional RUS measurements were performed every 200 °C during the cooling portion of the test. RUS analysis for specimens at high temperature required accounting for temperature-induced volumetric changes of the specimen dimensions. Thermal expansion tests were performed with a thermomechanical analyzer (TMA, QA Systems) for the glass and ceramic specimens from room temperature to 1000 °C. The thermal expansion of the materials was non-linear with temperature, and values of the average linear coefficients of thermal expansion values are listed in Table 1.

Plate-shaped double-torsion specimens with approximate dimensions of 20 mm × 40 mm × 0.70 mm with a notch on one side of length 12 mm were machined for fracture toughness determination. The Young's modulus and Poisson's ratio of two glass and ceramic plate specimens were determined by RUS analysis prior to notching the specimens. Double-torsion tests were performed on the glass and small grain-sized (SGS) ceramic specimens. The specimens were initially precracked in an MTS electromechanical test machine at a cross-head displacement rate of 1 μm s^{-1}. Deviation from non-linearity was indicative of precrack formation. The precracked specimens were then loaded at a cross-head displacement rate of 0.01 mm s^{-1} to failure. The maximum load (P_{IC}) was used to calculate the plane stress fracture toughness according to:

$$K_{IC} = P_{IC} S_m \sqrt{\frac{3(1+\nu)}{St^4\psi}} \quad (1)$$

Table 1
Characteristics and heat treatments of the investigated specimens. SGS – small grain size. MGS – medium grain size. LGS – large grain size. Note that even though the linear coefficients of thermal expansion are quoted below, the curves are highly non-linear.

Specimen type	Heat treatment	Mean grain size (μm)	Microcracking	Room temperature density (g cm^{-3})	Room temperature Young's modulus (GPa)	Room Temperature Poisson's ratio	Linear coefficient of thermal expansion (per °C) (temperature range in °C)
Glass	None	–	None	2.46	89.65	0.235	7.4×10^{-6} (25–600 °C)
β-Eucryptite – SGS	1100 °C for 4 h	<4.0	None	2.43	115.51	0.296	0.7×10^{-6} (25–1000 °C)
β-Eucryptite – MGS	1300 °C for 1 h	8.1	Moderate	2.38	23.73	0.064	-4.9×10^{-6} (25–1000 °C)
β-Eucryptite – LGS	1300 °C for 16 h	23.3	Severe	2.37	13.57	0.044	-6.5×10^{-6} (25–1000 °C)

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