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Characterization of Young's modulus and fracture toughness of albite glass by different techniques

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Abstract

This work compares values of Young's modulus and fracture toughness of amorphous albite measured from different experimental techniques. Sodium feldspar powder was used to synthesize the albite glass. After quenching, the microstructure of the amorphous solid was analyzed via scanning electron microscopy and X-ray diffraction. Several nanoindentations on albite samples were carried out with two purposes: determination of Young's modulus by continuous stiffness measurement, and critical stress intensity factor by crack size evaluation. Concurrently, impulse excitation technique and three point bending single-edge notched test were adopted for providing the comparison of elasticity and toughness, respectively. Young's modulus measured by continuous stiffness yielded a value of 71.8 ± 0.9 GPa, which was in agreement with the value obtained by impulse excitation (72.5 ± 1.3 GPa). The fracture toughness determined by nanoindentation was slightly higher (0.86 ± 0.06 MPa m$^{1/2}$) than that (0.78 ± 0.06 MPa m$^{1/2}$) obtained by single-edge notched beam.

Keywords: B. Non-destructive evaluation; C. Mechanical properties; D. Glass; Nanoindentation

1. Introduction

The mineral albite belongs to the family of plagioclase feldspars and it is widely used for producing ceramic coatings [1,2], ceramic glaze [3], dental porcelain [4], abrasives [5] or in compounds with recycled materials [6,7]. Sintering temperatures higher than 1100 °C [1] cause the albite crystal to melt, after which albite glass can be generated using fast cooling processes.

The thermal and rheological behavior of albite glass, such as thermal diffusivity [8,9], heat capacity [10] and viscosity [8] have been extensively investigated. In contrast, important mechanical properties of this material, such as Young's modulus and fracture toughness, are still scarcely reported in the literature [11]. The former property quantifies how easily the material experiences elastic deformations, whereas the latter property quantifies the susceptibility for crack propagation, being often used to evaluate the material brittleness.

According to Navarro [12], typical values of toughness for most glasses lie within a range from 0.6 to 0.9 MPa m$^{1/2}$. However, it is well established that both chemical composition and previous processing steps have a great influence on the mechanical properties of ceramics, and this influence plays a key-role in designing new materials or components.

In recent years several studies have been made for understanding and optimizing the mechanical strength [13–15], Young's modulus [16,17] and fracture toughness [18,19] of ceramic materials with heterogeneous microstructure. Such materials are generally composed of crystalline particles dispersed in a glassy phase. Knowing the individual responses

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of each existing phase is an essential factor for controlling or minimizing the damaging process throughout the material, both in micro- and macroscales. Furthermore, such knowledge is also required for the modeling and evaluation of mechanical systems of heterogeneous materials using numerical methods [20].

Currently two experimental methods are widely used for the experimental determination of fracture toughness (K_{IC}) and Young's modulus (E) of brittle materials. These methods are the Single Edge Notched Beam (SENB) and the Impulse Excitation Technique (IET), respectively.

However, improvements in instrumented indentation technique through electromagnetic and electrostatic systems have brought substantial control over the indentation process [21]. At the same time, further developments of fundamental theories on the material response under indentation have revealed the potential of the nanoindentation technique as a practical alternative for the characterization of mechanical properties.

The main advantage of the nanoindentation technique is the possibility of measuring both E and K_{IC} relatively fast and with low costs. In addition, this technique requires very small samples, offers punctual characterization ($< 40\ \mu m^2$) in heterogeneous materials, and also enables an "in situ" characterization of the microstructural constituents.

However, previous works showed that these results may differ for both Young's modulus [22,23] and toughness [24,25] when different theoretical models or measuring techniques are used. An investigation specifically devised to perform such experimental comparison still lacks in bibliography.

In this context, the present work intends to compare values of the mechanical properties E and K_{IC} of albite glass measured by different experimental techniques. Nanoindentation technique was therefore adopted for determining Young's modulus and also fracture toughness. Results based on nanoindentations were then compared against those from the classical experimental methods: IET for Young's modulus and SENB for fracture toughness, so that the viability of using nanoindentation could be properly evaluated for both properties.

2. Theoretical description

The experimental techniques based on nanoindentation considered in this work are the Continuous Stiffness Measurement (CSM) [26] and the Crack Length Evaluation (CLE) procedure, for measuring Young's modulus and fracture toughness, respectively. Short overviews of those techniques are given in this section.

2.1. Young's modulus determination

2.1.1. Continuous stiffness measurement (CSM)

In the nanoindentation CSM technique (also called dynamic stiffness measurement), stiffness is measured continuously during loading of the indenter on the material, by superimposing a small dynamic oscillation on the load signal and measuring the amplitude and phase of the corresponding displacement signal by means of a frequency specific amplifier [26,27]. In this technique, the indentation system is modeled as a harmonic oscillator.

Provided the dynamic response of the testing instrument is well-known, the contact stiffness (S) can be isolated from the equations governing the dynamic behavior of the system [21,28]. The contact stiffness can be calculated using the following equation:

$$S = \left| \frac{1}{\frac{L_o}{h_o}\cos\delta - (K_s - m\omega^2)} - \frac{1}{K_f} \right|^{-1} \quad (1)$$

where L_o and h_o are the force and displacement amplitudes, respectively δ is the phase angle between load and displacement, ω is the frequency, K_s is the stiffness of the supporting springs, K_f is the load frame stiffness of the instrument and m is the system mass [28,29].

The contact stiffness S is then used to calculate the elastic modulus of the sample by using the equations derived by Oliver and Pharr for Young's modulus from nanoindentation [26]. First, the so-called reduced elastic modulus E_r is calculated, which comprises the combined response of the indenter tip and the sample as follows:

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \quad (2)$$

where A is the projected contact area measured constantly as a function of the indentation depth, and β is a constant depending on the geometry of the indenter used [26]. Subsequently, Young's modulus E of the sample can be calculated, provided that the properties of the indenter tip are well-known.

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \quad (3)$$

where ν is Poisson's ratio of the test material, and E_i and ν_i are the elastic modulus and Poisson's ratio of the indenter tip, respectively [26].

A great advantage of the CSM technique is the possibility of measuring Young's modulus and hardness of the material as a function of indentation depth from single indentation experiments [26,27].

2.1.2. Impulse excitation technique

This technique is based on the natural vibration frequency as response to a mechanical impulse in macroscopic scale. The relationship between the frequency of the first vibrational mode and Young's modulus was established by Lemmens [30]. The sample experiences a small deformation, and the resulting vibration is then analyzed as a mass–spring system. The natural frequency with which the sample vibrates is a function of its Young's modulus E, the material density, as well as the sample geometry. Admitting the international unit system, E can be expressed according to the following equation [30,31]:

$$E = \rho\left(f\frac{U^2}{e}\right)^2 C_f \quad (4)$$

where f is the natural frequency of the object, ρ is the material density, U and e are length and thickness of the specimen,