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An estimate of quartz content and particle size in porcelain tiles from young's modulus measurements

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ABSTRACT

The influence of quartz particle size, weight content and firing temperature on the Young's modulus of porcelain tiles was studied. To simulate a porcelain tile microstructure, an albite glass matrix with added crystalline quartz particles was developed. Average particle size of quartz (3.4 and 31 μm) and volume content (18.5 and 37.6 vol %) were varied. An acoustic impulse excitation technique was used to measure the elastic modulus from room temperature up to 700 °C. Results showed that quartz has a major influence on the elastic modulus of porcelain tiles. At temperatures below 573 °C, a hysteresis area between the Young's modulus curves during heating and cooling was closely related to quartz particle size. Between 573 and 700 °C, the variation of the Young's modulus was related to the quartz volume fraction. By using those correlations, a prediction of quartz content and quartz particle size in commercial porcelain materials can be carried out from Young´s modulus data.

1. Introduction

Ceramic materials undergo brittle fracture, with little or no plastic deformation. Non-crystalline materials, such as the vitreous phase component of most traditional ceramics are brittle below the softening temperature [1,2].

The linear elastic behaviour found in ceramic materials is described by Hooke's law, as expressed by Eq. (1) for uniaxial loads

$$\sigma = E\varepsilon \quad (1)$$

where σ is the stress (Pa) and ε is the strain or elastic deformation.

Hooke's law simply shows that within the elastic region, stress and strain are related by a constant, the Young's modulus, E. [3].

Knowing the Young's modulus is a key factor during the design, development and quality control of ceramic materials. Its measurement might be carried out by either destructive or non-destructive testing. Three-point bending is a common example of a destructive method [4,5], and acoustic impulse excitation technique (IET) [6,7] is a non-destructive test for the measurement of elastic moduli.

Young's modulus in heterogeneous materials is affected by composition, processing [8] and microstructure [9–11]. Therefore, the presence of porosity, cracks and crystalline particles of different nature may change the elastic modulus of those materials.

Budiansky & Connel described the relationship between the density of cracks and the modulus of elasticity, analysing cracks in dry, saturated, and partially saturated solids [10]. Štubňa et al. analysed the mechanical behaviour of porcelain materials during the cooling step and found out that there is a variation of the elastic modulus around 573 °C associated to the allotropic transformation of quartz [12]. Moreover, Boccaccini et al. [13] established a correlation between Young's modulus and porosity of materials such as glass and ceramic oxides. The influence of temperature on the elastic modulus of silica polymorphs has been investigated by Ohno et al. and Lakshtanov et al. [14,15]. They confirmed that the allotropic transformation of α/β quartz caused a wide variation on the elastic modulus around this temperature. Štubňa et al. [16] analysed the elastic behaviour of ceramic materials subjected to different heating cycles, evidencing that during heating and cooling the elastic modulus presented a hysteresis. This behaviour was related to the cracks caused by the difference in thermal expansion coefficient between the glass phase (matrix) and quartz (disperse phase). Oliveira et al. [17] assessed the variation of the elastic module in porcelain materials, and confirmed the hysteresis

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caused by quartz during heating and cooling, and proposed a mechanism to explain this behaviour.

In spite of the extensive research on mechanical properties of porcelain materials containing quartz particles, the effect of the addition of quartz particles on the elastic modulus in heterogeneous materials has been hardly addressed. This is probably due to the fact that elastic modulus is not commonly used as a quality control tool. However, the importance of this mechanical property in ceramic products has been extensively reported [18–21].

Quartz is a very common raw material for the ceramic industry [22]. Very frequently quartz occurs as secondary mineral in clays, feldspars or other raw materials [23]. Regardless of the way in which the quartz is incorporated, this crystalline phase is part of most traditional ceramic compositions.

The interest on investigating the effect of quartz (content and/or particle size) on mechanical properties of porcelain materials has been intensified in the last 10 years. Bragança et al. [24] found that the particle size of quartz influenced the fracture toughness in triaxial ceramics composed of quartz, feldspar and kaolin. There was an increase in fracture toughness by reducing the particle size of quartz. The effect of the crystalline quartz particles on mechanical strength was also investigated by De Noni et al. [25], who found a relationship between the compression caused by the quartz crystal in a glassy phase and microstructural defects caused by cracks around (or inside) quartz particles. Moreover, Gilabert et al. [26] modelled the fracture patterns of quartz particles added to a glassy (albite) matrix.

Thus, the quantification of the effect of quartz particle size and content on the elastic modulus of an albite glass matrix is the main scope of this work. The measurement of the elastic modulus was carried out by a non-destructive, in situ thermomechanical method (IET) which has been previously described [17]. Conversely, as an innovative approach, the methodology proposed in this work may enable the estimation of the mean particle size of the quartz particles present in a porcelain tile matrix from elastic modulus data.

2. Experimental procedure

2.1. Materials

Sodium feldspar (Mario Pilato, Spain) was used to sinter a porcelain matrix comprised of albite glass with low porosity and a little residual crystalline phase. Crystalline quartz particles (Sibelco, Spain) were added to study the influence of particle size and volume content of quartz on the elastic modulus of the porcelain matrix. The respective chemical analyses are shown in Table 1, according to the supplier. Four quartz particle size ranges were used, SE-8, SE-12, SE-100 and SE-500 (Table 2).

Moreover, the volume fraction of quartz in the matrix was varied in three levels: 6.3 wt%, 24.8 wt% and 43.9 wt%.

Table 1
Chemical analyses of the raw materials used.

Oxides	Feldspar (wt%)	Quartz (wt%)
SiO_2	69.90	98.9
Al_2O_3	18.70	0.51
Fe_2O_3	0.06	0.05
TiO_2	0.12	–
CaO	0.50	0.03
MgO	0.10	–
Na_2O	10.00	0.01
K_2O	0.30	0.06
Other	0.02	0.17
Loss of ignition	0.30	0.27

Table 2
Mean particle size of quartz particles used.

Quartz	D_{50} (μm)
SE-500	3.4 ± 0.5
SE-100	13.4 ± 0.6
SE-12	20 ± 1
SE-8	31 ± 4

2.2. Processing

The particle size of sodium feldspar was reduced using a planetary mill with water and alumina balls. The milling time was 30 min at 260 rpm, resulting in a mean particle size of about 6 μm. Then, the feldspar was dried and granulated with 8 wt% aqueous solution containing 5 wt% polyvinyl alcohol (PVA, Mowiol 4-88, 31000g/mol, Sigma-Aldrich). Each sample comprised of ground feldspar and quartz at the given volume fraction was homogenized in water in a ball mill for 10 min at a low speed. The feldspar and quartz mixture was then granulated and pressed at 35 MPa using a uniaxial press, resulting in specimens with dimensions of 67×16.5×5.5 mm.

The samples were then sintered in an electric furnace (Pirometrol R-series, Spain), with an initial heating rate of 210 °C/min between room temperature and 500 °C, followed by a second heating rate of 25 °C/min up to 1200 °C. The maximum sintering temperature was adjusted for each quartz sample to obtain the maximum densification of any of the ceramic material. The optimal sintering schedule was found by analysing at which temperature a highest linear shrinkage and lowest water absorption was reached. This sintering temperature was maintained for 6 min, followed by a natural (convective) cooling.

2.3. Characterization

The technique of impulse excitation of vibration [6,7] was used to measure the elastic modulus as a function of temperature. This technique relies on the natural vibration frequency as response to a mechanical impulse, in which the relationship between the frequency of the first vibrational mode and the Young's modulus can be established [6,27]. The sample experiences a small deformation, and the resulting vibration is then analysed as a mass-spring system. The natural vibration frequency with which the sample vibrates is a function of Young´s modulus, material density and sample geometry.

In this study, the maximum temperature was 700 °C, and the heating rate was 400 °C/h, while cooling occurred by natural convection. The measurement of the natural vibration frequency of the sample was carried out at an interval of 2 °C during heating and cooling using a Grindosonic apparatus fitted within an oven. According to ASTM E1876-09 [7], the Young's modulus (E, in GPa) of a dry sample can be calculated as

$$E = \frac{0.9465\Phi\rho L^4 f^2}{w^2} \quad (2)$$

where Φ is a correction factor for fundamental flexural mode to account for finite thickness; ρ is the apparent density (g/mm³); L is the length (mm); f stands for the natural vibration frequency (kHz); and w is the thickness (mm) of the sample, respectively [7].

X-ray diffraction analyses were performed using powdered material to identify potential crystalline phases. The X-ray diffractometer (D8 Advance, Bruker, Germany) used CuK$\alpha_{1,2}$ (1.5406 Å) radiation, with a goniometer radius of 250 mm. The data were collected using the Bragg–Brentano geometry (θ/θ), between 5 and 90° (2θ), with a step size of 0.015° and 1 s per step. A rapid solid-state detector (Vantec, Bruker, Germany) fitted with a system of Soller slits and nickel filter was used. The X-ray tube operated at 40 kV and 45 mA. Fluorite was used as internal standard for quantification of crystalline phases by the