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Numerical–experimental method to study the viscous behaviour of ceramic materials

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Abstract

The aim of the work deals with the development of a numerical–experimental method to study the viscous behaviour of ceramic materials and to estimate the viscosity evolution as a function of temperature.

The current state of the art regarding the study of viscous behaviour of ceramic materials is carried out by using optical methods able to perform measurements without contact with the sample to be analyzed. The two point bending configuration is the conventional test currently used. Despite the use of the optical technology, the test specimen, because of its viscous behaviour, interferes with the measuring device. The experimental test results are inevitably compromised.

The developed numerical–experimental method aims at overcoming the drawbacks of the conventional experimental test configuration and at determining the material viscosity. This method consists of an *ad hoc* developed experimental test configuration and of a numeric simulation of the experimental test.

Keywords: Ceramic material; Sintering; Material viscosity; Pyroplastic deformation; FE numerical analysis

1. Introduction

The viscosity of ceramic materials undergoing densification is typically measured by applying compressive or tensile stress to the specimen, which causes creep deformation. In uniaxial compression tests, it is difficult to maintain a uniaxial stress state because the friction at the loading platens causes "barrelling" whereas tensile creep tests are complicated by problems associated with gripping of brittle samples. To avoid gripping or alignment problems, bending creep test has been proposed.[1,2] The difficulty with bending tests to characterize creep is that the constitutive relationship for uniaxial compression differs from that in uniaxial tension. It has been shown that a porous material undergoing densification can be treated as a linear viscous material at low stresses (about 1 MPa).[3] For such materials, it is expected that the creep rates in uniaxial tension and compression will be the same. Thus, the bending test is an attractive method to measure the uniaxial viscosity of porous materials.

The viscosity of materials during sintering has been demonstrated, both theoretically[4–6] and experimentally,[7–9] to depend strongly on the relative density and temperature. Generally, the viscosity of a material decreases as the temperature increases. However, densification of porous materials results in a viscosity increase.

The aim of the work deals with the development of a numerical–experimental method able to solve the problems concerning the bending test and to accurately determine the viscosity evolution as a function of temperature and material density.

The numerical–experimental method has been developed to study the viscous behaviour of traditional ceramic materials (*i.e.* Vitreous China) during firing; however, this method is applicable as well to study all the materials, that during a process, exhibit shrinkage and viscous deformation at the same time, as for example polymeric materials, composite materials and others.

2. Theoretical background

Ceramic materials, undergoing densification, exhibit a linear viscous behaviour. For a two point bending creep test, by

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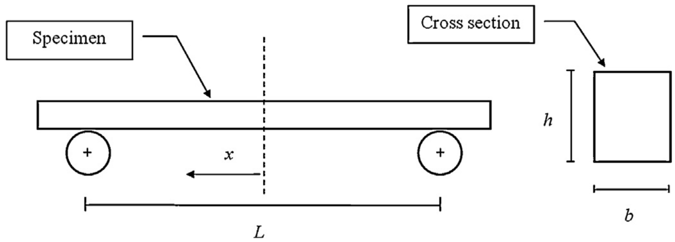

Fig. 1. Two point bending creep test.

using the beam deflection theory and the linear elastic to viscous analogy, it is possible to derive the following relationship[10]:

$$\eta = \frac{5\rho g L^4}{32\dot{\delta}_{x=0} h^2} \tag{1}$$

where η is the material viscosity, ρ the density, h the specimen thickness, g the gravity acceleration, L the span length and $\dot{\delta}_{x=0}$ is the deflection rate at beam centre (see Fig. 1).

Dilatometric tests are used to determine material shrinkage and to derive density evolution during the sintering cycle. Starting from dilatometric test results and the previous relation, by measuring the deflection of a specimen during a two point bending creep test (see Fig. 1) the viscosity of a ceramic material during the sintering cycle can be assessed.

3. Experimental characterization

Experimental tests have been carried out by means of a combined optical instrument able to perform bending and dilatometric tests (Fig. 2).

The traditional ceramic materials during sintering are highly deformable. The choice of a non-contact measurement method allows to investigate the thermo-mechanical behaviour of the material without compromising the measure, so that the material is completely free to expand or contract or to deform under its own weight.

In this work the instrument has been used to perform both thermal expansion (Fig. 2a) and pyroplastic deformation measurements (Fig. 2b).

3.1. Experimental tests results

Dilatometric tests are performed to measure sintering shrinkage and to derive density of ceramic material during sintering. Fig. 3 shows the density evolution of the traditional ceramic material during sintering cycle. From room temperature up to 800 °C the material behaviour is characterized by a density decrease due to the weight loss caused by the residual water evaporation and by the decomposition of organic components. At higher temperature the density increase as consequence of porosity closure (sintering).

Bending creep tests are performed to investigate the pyroplastic behaviour of traditional ceramic material during firing and to derive the bending rate curve $\dot{\delta}_{x=0}$ (see Eq. (1)). At temperatures of about 900–1000 °C the feldspar content of raw materials melts; this turns into a viscosity decrease that causes the

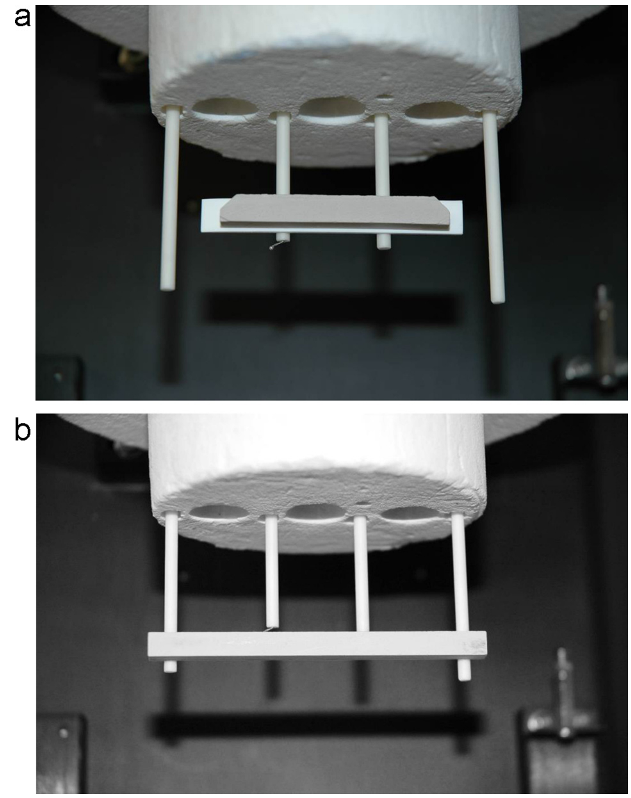

Fig. 2. Combined optical instrument: (a) dilatometric test configuration; (b) two point bending creep test configuration.

pyroplastic deformation (see Fig. 4). On the other end, during the thermal cycle, the sintering process involves an increase of the density and as a consequence an increase of the viscosity. Finally, the deflection stops during the cooling phase.

Despite the use of the optical technology, during the conventional bending test the specimen interferes with the measuring device because of its viscous behaviour. The most recurring problem is that at high temperatures the ceramic specimens adhere to the supporting rods. Because such phenomenon does not occur equally at both ends, the specimen midpoint, due to the material shrinkage, translates horizontally invalidating the experimental test results. The characteristic effect of the above

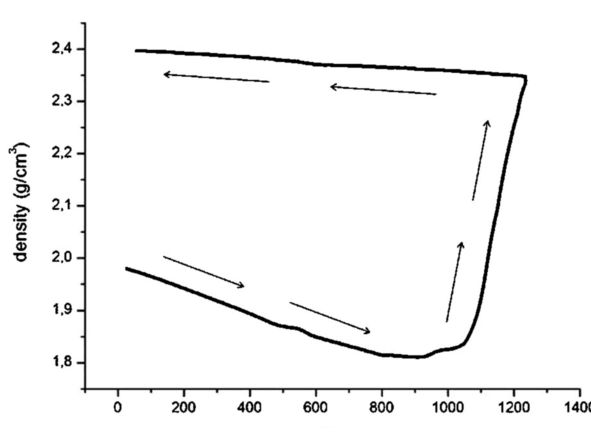

Fig. 3. Density evolution during sintering cycle.