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Spherical indentation of porous ceramics: Cracking and toughness

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ABSTRACT

A combined experimental-numerical approach is used to characterise the fracture of a porous bulk ceramic material ($La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$) with porosities of 5–45%, undergoing spherical indentation. The Gurson model was used in FEM to describe the porosity densification. Indentation-induced radial cracks were observed, when the applied contact pressure exceeded threshold values, with no Hertzian ring-cone cracks found. FEM analysis indicated that the cracks propagated mainly during unloading, driven by the tensile hoop stress generated near the contact circle. The stress intensity at the crack tip was estimated using an approximate analysis of the FEM stress field to derive toughness values that were consistent with values determined by conventional methods, provided that the crack length is sufficiently large compared with the contact radius and can be measured accurately. The absence of ring-cone cracks during loading is due to the material's high modulus-to-hardness ratio and the small indenter radius as predicted by established theory.

1. Introduction

The development of porous ceramics is aimed at demanding engineering applications, such as insulation, filtering, catalysis, microelectronics, bioengineering and membranes [1–6]. Porous bulk ceramics are generally prepared by the partial sintering of powders at elevated temperatures. The influence of the resultant microstructure on typical elastic properties of such porous ceramics has been widely studied [7–12]. Although the property-microstructure correlation is complex in detail [13], the elastic and fracture properties depend mainly on the volume fraction of porosity [14].

Nanoindentation measurements, typically with the use of sharp indenters, have been extensively employed to characterise the mechanical properties (e.g. elastic modulus and hardness) of nominally dense materials [15,16]. However, the application of indentation to the mechanical characterisation of porous materials is not so common. This is because the presence of the porous microstructure brings added complexity to obtaining and interpreting reliable data. The complication arises from collapse and densification in the compressive field of the indenter, as observed in the indentation of highly porous silica foams [17] and ceramics [18]. Nevertheless, in previous studies we have used sharp indenters and conventional methods (single edge-notched beam) to measure the fracture toughness of a typical porous ceramic used in solid oxide fuel cell (SOFC) technology and found that the two approaches gave results in acceptable agreement [19]. However, in many contact situations the contacting component does not have a sharp profile (like a sharp indenter), but is rather blunt and more like a spherical indenter. For example, in SOFC technology this might be the contact between a rib on a bipolar plate and the current collector layer of the cell. Therefore it is important to understand contact damage when a blunt (spherical) indenter is loaded into a porous ceramic.

In an earlier paper from this study, the influence of porosity on the elastic and plastic properties deduced from spherical indentation of a typical porous ceramic was investigated [20]. The interpretation was based on finite element modelling of the indentation employing the Gurson model [21] to describe the collapse and densification of the porous structure in the compressive plastic zone under the indenter. In the present paper we extend this approach to investigate indentation-induced cracking of the same porous ceramics when higher contact pressures are applied.

Very few studies are reported in the literature regarding spherical indentation-induced damage in porous ceramics. For example, Latella et al. [22] studied contact damage in porous and dense liquid-phase-sintered alumina using large spherical indenters. Their results showed that in the low porosity specimens (2.5% porosity) the spherical contact induced classical Hertzian ring and cone cracks penetrating below the surface with no detectable residual surface depression caused by the indenter, indicating negligible

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Table 1
Properties of LSCF specimens and thresholds for radial cracking.

Sintering Temperature (°C)	Porosity (%)	Threshold Load (mN)	Threshold Depth (μm)	Threshold Pressure (GPa)	*E* below Threshold (GPa)	*E* above Threshold (GPa)
900	44.9 ± 0.3	600	5.33	0.88 ± 0.03	33.4 ± 1.2	28.9 ± 1.2
1000	36.3 ± 1.1	800	5.31	1.16 ± 0.06	52.7 ± 3.7	47.4 ± 2.3
1100	28.7 ± 0.9	2000	5.01	3.28 ± 0.59	86.8 ± 3.1	84.1 ± 2.0
1200	5.2 ± 0.1	800	1.03	7.01 ± 0.21	178.2 ± 2.3	162.9 ± 2.2

plastic deformation. Increasing porosity (up to 17.8%) induced a transition to a quasi-plastic response in which there was collapse of the porous material and formation of surface ring cracks in the indented region that did not penetrate appreciably below the surface. No radial cracks were seen in any specimen.

In the current study, spherical indentation at a wide range of loads was carried out to generate cracks in the porous ceramic material $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF), which was sintered from powder at different temperatures between 900 and 1200 °C, resulting in pore fractions between 45 and 5%. Contrary to the behaviour of alumina observed by Latella et al. [22] indentation-induced radial cracks were observed above a threshold contact pressure and their geometry was characterised using FIB-SEM. Numerical analysis (FEM) was performed to determine the stress field in the absence of cracks during both loading and unloading; using the Gurson model to describe the collapse and densification of the porous material. This stress field, together with the dimensions of the cracks observed experimentally, was then used to estimate the fracture toughness of the porous material. Finally, the values of fracture toughness obtained in this way were compared with values obtained using sharp indenter (Berkovich) and notched beam methods.

2. Experiments and simulations

2.1. Indentation tests

The perovskite material $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) is used widely as a porous cathode in solid oxide fuel cells (SOFCs). Bulk LSCF specimens with different porosities were prepared by sintering LSCF powders at different temperatures. The sintering temperatures and the corresponding percentage porosities are shown in Table 1. Indentation experiments over the load range 50–10000 mN were carried out using a spherical diamond indenter (radius 25 μm). Table 1 also shows the values of critical load, indentation pressure and depth of indentation when cracking was first observed (by microscopic examination after unloading). The apparent elastic modulus (derived in the standard way from the unloading stiffness) was calculated for maximum indentation loads below and above the critical load and the results are also displayed in Table 1. They show a small, but significant, reduction at loads above the critical value indicating that cracking has reduced the local stiffness in the neighbourhood of the indentation. Focused ion beam-scanning electron microscopy (FIB-SEM) was used to study the surface and cross-sectional microstructures of the specimens before and after indentation. Details of the specimen preparation, indentation and FIB-SEM procedure can be found in [18]. Technical aspects of the indentation process are fully described in [18] and [20].

2.2. Finite element modelling

Axisymmetric finite element simulation of the indentation process, taking into account the collapse and densification of the porous material under the indenter, was developed in the earlier part of the current study [20]. That work showed that the Gurson model could provide a suitable description of densification in the "plastic zone" of the indentation, despite the fact that the Gurson model is normally applied to plastic deformation of porous metals and is only strictly valid when the porosity is lower than approximately 10%. As a result, although application of the Gurson model in the present study describes the densification behaviour acceptably, the critical materials parameter used in the Gurson model (the yield stress of the fully dense material) has no fundamental significance and is regarded as an adjustable parameter whose value is determined by fitting to the experimental indentation curve. In the present study, FE simulations were carried out using Abaqus CAE 6.12 (Dassault Systemes, USA) for indentation loads (or the corresponding indentation depths) equal to, or greater than, the threshold values that were observed to trigger the radial cracks found in the indentation experiments. The full description and implementation of the models can be found in our earlier paper [20].

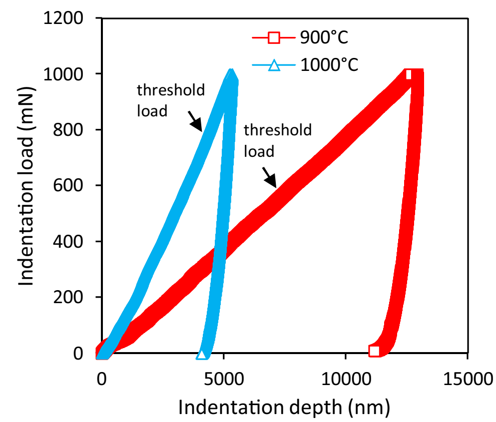

Fig. 1. Experimental indentation load-depth curves generated at 1000 mN load (beyond each corresponding threshold load) for LSCF bulk specimens sintered at 900 °C and 1000 °C.

3. Results and discussion

3.1. Crack characterisation

The LSCF specimens were subjected to spherical indentation at a series of loads ranging from 50 to 10000 mN. In addition to the densification deformation (regarded as the permanent plastic deformation) resulting from crushing of the particle-pore networks under indentation, as described in [20], indentation-induced radial cracks were also found in the specimens when the applied load surpassed a threshold value, determined by microscopic characterisation after indentation. Below the threshold loads, permanent "pseudo plastic" deformation without any radial cracks was observed in all samples.

Some typical experimental indentation curves are shown in Fig. 1. In general, the loading-unloading curves did not show any obvious evidence of long-range fracture events (such as clear pop-in responses), and therefore microscopic inspection of the specimens was required to detect whether any fracture had