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Epoxy interface method enables enhanced compressive testing of highly porous and brittle materials

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Abstract

The compressive mechanical characterization of highly porous ceramics is problematic due to sensitivity to stress concentrations and localized fractures. In this paper a review of the methods used by the scientific community is done and their typical results, advantages, and shortcomings are discussed. Here, a new methodology that can address some of the problems associated with the current measurement procedures is presented. The proposed method is applied to highly porous barium titanate foams. This procedure produces more consistent data that can be analyzed and interpreted objectively. We show that properties such as the crushing or collapse stress as well as the compressive modulus cannot be extracted correctly by the conventional methods. By contrast, the proposed method collects data that is more accurate, consistent with what is expected of porous ceramics and that yields smaller measurement to measurement variation.

Keywords: C. Mechanical properties; Ceramic foams; Compliant layer

1. Introduction

Highly porous ceramics (> 50% porosity) are used for many applications including catalyst structures, bio-applications, and refractories [1–4]. Although porous ceramics have been investigated for decades, characterization of their mechanical properties remains challenging. This limitation is particularly relevant since the elastic and compressive behavior is critical for load bearing applications. For example, accurate effective modulus and compressive collapse stress are necessary for determining if certain porous ceramics are useful as bone regeneration matrices, catalytic structures, or filters. Accurate compressive testing of highly porous ceramics is difficult due to surface stress concentrations at the sample and test platen interface [2,5–8]. These surface stress concentrations during compressive testing are less of a problem in ductile materials such as metallic foams. This is partially due to easier polishing and sample preparation but more importantly due to the fact that the ability of metallic foams to deform plastically prevents a cascade of localized fractures at the surface. These stress concentrations arise from the uneven load distribution due to small variations in the height of the sample at the platen interface, as seen in Fig. 1. Ideally, these small variations could be eliminated with proper sample preparation. However, the brittle nature of ceramics makes polishing of highly porous samples challenging, and does not allow for deformations to ensure a uniform contact and load distribution at the surface [2]. Therefore, as later described in detail, typical test data will be rather noisy and primarily consisting of cascaded micro-fractures and in most cases a distinct collapse or crushing stress cannot be discerned.

It is important to note that currently there are testing standards for ordered and highly porous ceramics, e.g. honeycomb structures (ASTM C1674), but none for highly porous ceramics with randomly oriented porosity such as ceramic foams. Further, according to a news release in 2008 by ASTM International, subcommittee C28.04 was going to develop standards for porous ceramics with three-dimensional cellular ceramic foam structures [9]. However, to date no new

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standards have been published. It is evident that a better methodology to measure compressive response of highly porous brittle materials is necessary, such that more consistent and quantifiable data can be collected and analyzed. In this paper, two of the most widely used methods to measure compressive behavior of cellular ceramics will be reviewed; case I in which the samples are simply compressed in a universal testing machine (UTM), and case II in which compliant testing surfaces are utilized. Furthermore, a new methodology that can address the issues with both cases and collect more consistent data will be proposed, tested, and analyzed.

2. Case I: direct UTM compression

The most common method for characterizing the compressive behavior of porous ceramics utilizes cylindrical samples with diameters on the order of a centimeter with heights ranging from millimeters to centimeters [10–16]. In some cases rectangular samples in the same size range are used [17–25] and in some cases sample size is not mentioned [26]. Samples are usually mentioned to have been polished flat and parallel but the quality, extent, or the importance of this step is rarely emphasized. It is noteworthy that polishing can be very challenging for highly porous or weak samples due to the fact that struts tend to break instead of being polished in the grinding process [2]. These samples are then placed between two platens of a mechanical testing load frame as shown in Fig. 1.

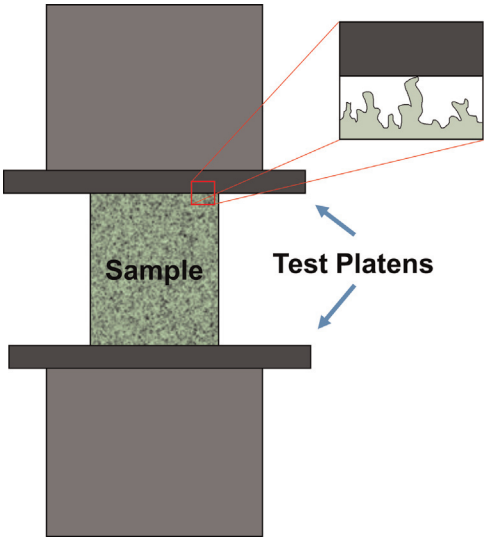

Fig. 1. Schematic of case I type of compressive testing where stress concentrations may arise when samples are placed directly between hardened steel test platens.

The load and displacement data from the instrument are analyzed and an effective modulus (Young's modulus of the structure as a whole, not Young's modus of the struts, since stresses are defined as force divided by the area of the foam surface not accounting for porosity) and fracture/collapse stress, mostly referred to as compressive strength, is extracted from the data. It is notable that the actual stress–strain curves are often not published and only 7 of the 17 publications cited in this section present the raw data [11,12,14,15,17,25,26]. Moreover, the methodology by which the effective modulus and the collapse stress are extracted is rarely discussed. It can be inferred in most cases that the effective modulus is defined by the ratio of stress to strain before a large drop in stress. This is commonly referred to as the collapse stress, crushing stress, fracture stress, or compressive strength. Two examples of published raw data by Zarkoob et al. [15] and Gómez de Salazar et al. [25] from the type of measurement described here are reproduced after digitization in Fig. 2. Zarkoob et al. investigated the effect of hydroxyapatite coatings on 78% porous disk shaped barium titanate samples. The raw mechanical data is presented by force (exerted by the samples) versus crush (displacement of testing platens) graphs. Gómez de Salazar et al. show the stress (σ) versus displacements (Δx) of rectangular $12.7 \times 12.7 \times 25.4$ mm^3 silicon carbide foams with 3 different ppi (pores per inch) porosity levels in which they used ASTM standard D695-96 (Standard Test Method for Compressive Properties of Rigid Plastics).

As seen in Fig. 2, interpretation of stress–strain data from highly porous ceramics is challenging. The data tends to be noisy and consists of many small drops in stress which are

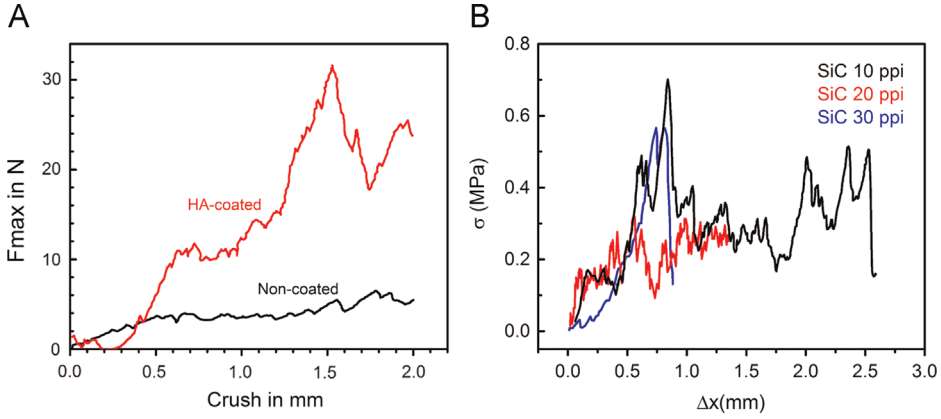

Fig. 2. (A) Force vs. displacement data of two different porous barium titanate samples under compression by Zarkoob et al. [15] (B) SiC ceramic foam compression data by Gómez de Salazar et al. [25].

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