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Mechanical characterization of aerogel materials with digital image correlation

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ABSTRACT

Silica aerogels are ultralow density materials with nano-sized skeleton network of pores. Their high brittle nature presents a major challenge for mechanical testing and a need exists for novel testing methods. Two new mechanical setups and testing techniques are proposed for measuring the aerogel elastic mechanical properties. Both techniques employ full-field Digital Image Correlation (DIC) for surface deformation measurements. The first setup uses disk compression experiment, known as diametral compression test (Brazilian disk). However, the elastic properties of the material cannot be obtained directly. Instead, an inverse mechanics computational scheme, using both a finite element (FE) model and analytical solution, is proposed. The second direct testing setup is uniaxial compression of rectangular-shaped blocks. The Young's modulus and Poisson's ratio are extracted directly from the experimental stress–strain curves. Our results of tested samples show the relation between the density and the Young's modulus to coincide with previously published trends. The direct and iterative inverse-mechanics solution methods agree well with each other. The Poisson's ratio is found to be independent of the material apparent density. Comparisons between the two methods and recommendations for expanding the disk testing approach to fracture toughness are discussed.

1. Introduction

Aerogels comprise a special class of low-density open-cell solid foams (typically, with porosity over 90%) which exhibit many unique properties such as exceptionally lightweight, high surface area, low thermal conductivity, extremely low dielectric constant, low sound wave transmission, high optical transparency in a wide range of wavelengths close to that of glass, and a very low refractive index [1–4]. These properties result from the microstructure of aerogels, which consists of a three-dimensional amorphous solid skeleton network with interconnected nanometer-sized pores in between. Silica aerogels are used for thermal and electrical insulation, especially in space applications, oxygen and humidity sensors, aerosol particle collectors, space mirror protectors, catalyst supports, battery electrodes, *etc.* [5].

The structure of the aerogel is sponge-like with nano-structured airy material whose three-dimensional network of interconnected silica particles forms cylindrical and branched open 2–50 nm pores. The elastic modulus of highly porous aerogel materials is proportional to the relative density raised by the m^{th} power ($3 \le m \le 4$). Ma et al. [6] proposed a computational model using diffusion-limited cluster–cluster aggregation (DLCA) algorithms along with FE models to estimate the elastic properties. Their computational modeling resulted in $m \approx 3.6$ for perfectly connected micro-structures.

In general, aerogels are highly brittle materials, although they can have a high stiffness-to-density ratio. Due to their low toughness, there is a major challenge to perform mechanical tests with these materials. Characterization of the mechanical properties of aerogels is important in order to design engineering applications and utilize their unique physical properties. Woignier et al. [7–9] performed both three-point bending and acoustical experiments in order to determine the static and the dynamic Young's moduli,

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respectively, of monolithic parallelepipedic non-densified silica aerogel samples. It was found that the density dependence of the Young's modulus, E, can be scaled by a power-law function $\phi^{3.8}$, where ϕ is the volume fraction, so a calibration law was proposed accordingly. Dynamic experiments have been carried out by means of the Brillouin scattering acoustic technique [7,10]. The longitudinal and transverse wave-length velocities were measured, allowing the determination of the elastic constants. Woignier and Phalippou [8] further extended the three-point bending method for densified aerogels. A general trend in which E increases with the density was observed, with E values varying between 10^{-3} and 10^{-2} GPa. Two different slopes with two different scaling exponents were observed. Thus, it was deduced that non-densified aerogels and densified aerogels have different elastic behaviors. The mechanical strength of silica aerogels has also been determined by bending tests [8].

Gross et al. [11] employed ultrasonic and static compression experiments at high frequencies in which longitudinal and transverse velocities were measured for different densities of aerogels. The Poisson's ratios thus determined were not affected by the density variations. Tillotson and Hrubesh [12] investigated the mechanical properties of ultralow-density aerogels (3–80 mg/cm^3). Scattered results for densities lower than 80 mg/cm^3 were shown since measurement in this range of low densities was more difficult to implement. The results coincided with previously published results reflecting a lower power-law slope for the ultralow-density aerogels compared with the higher-density silica aerogels. Scherer et al. [13,14] measured the shear modulus and bulk modulus of alkoxide-derived silica gels during aging and drying stages.

Parmenter and Milstein [15] investigated the mechanical behavior of fiber-reinforced and unreinforced silica aerogels using indentation and block compression tests. The direct (block) compressive strength showed correlation with the hardness results. Moner-Girona et al. [16] investigated the mechanical properties of silica aerogels by means of the micro-indentation technique. The Young's modulus for the lightest aerogel (80 mg/cm^3) was 7.0 MPa, whereas the highest density (260 mg/cm^3) yielded a modulus of 346 MPa. The four experimental points were fitted according to power-law function for the Young's modulus, with a power $\alpha = 3.0 \pm 0.2$. Wong et al. [17] investigated the elastic modulus of a wide range of hydrophobic silica aerogels in order to study the effect of density on modulus, which ranged from 40 kPa to 70 MPa. Tension and compression tests along with thermal conductivity properties were also reported. The Brazilian disk test was used for the limited purpose of measuring the tensile strength only.

To-date, very few studies have used Digital Image Correlation (DIC) for aerogels. Sun et al. [18] established guidelines for preparing shear tests of ceramic-fiber-reinforced aerogel. Iosipescu aerogel specimens with two different notches were investigated using the FE method. It was proposed (without actually applying it) that the needed mid-section strain gages can be replaced by the DIC method. Dynamic compressive tests of cross-linked silica aerogel using a split Hopkinson pressure bar (SHPB) were conducted by Luo et al. [19]. The DIC method was employed using high-speed image acquisition. The Poisson's ratio was determined to be 0.162. Ductile splitting was observed at high strain rates, but the samples were much more ductile than native silica aerogels. Luo et al. [20] studied the mechanical properties of isocyanate-crosslinked vanadia aerogels under both quasi-static loading conditions and high strain rate loading conditions using a SHPB. DIC was used to measure the surface strains, indicating nearly uniform compression at all stages of deformation during compression. In our study we apply the DIC method in a unique fashion and demonstrate the use of virtual extensometer (extension or shortening distance between two points). There are strong reasons for using the proposed DIC for aerogels. The latter are brittle, and contact gauges for deformation measurements cannot be mounted without local damage. In addition, the full-field nature of the DIC allows both overall and local strain measurements, including of the Poisson's ratio. The DIC can also be used for inhomogeneity, damage and flow detections.

Fracture toughness tests using the Single-Edge-Notched Beam (SENB) technique have been conducted [9], showing that for aerogel samples having densities between 0.1 and 0.35 g/cm^3, the fracture toughness K_{IC} ranges between 0.8 and 4.8 KPa-m$^{1/2}$ and can be scaled by a power law with an exponent equals to 1.6 ± 0.2. Alaoui et al. [21] measured the toughness of alcogels and aerogels with different porosities by SENB. The mechanical strength was also measured using the three-point bending on un-notched specimens. Both aerogel and alcogel samples were monolithic with densities ranging from 90 to 250 mg/cm^3. It was observed that, for a given density, the toughness of aerogels was higher (by a factor close to 2) than alcogels. This toughness enhancement is expected taking into account the textural and structural transformation of the gel, and was attributed to the growth of the necks between the particles.

The brittle nature of the light-weight aerogel presents several challenges for mechanical testing, such as end fixtures for mounting the specimen, contact method for applying the load, and displacement measurements during the test up to ultimate failure. Standard deformation measurement techniques (*e.g.* strain gauges, extensometers) are found unsuitable since they cause damage to the specimen. Therefore, non-contact optical measurement techniques offer a better alternative for monitoring the deformation during loading. In this study, we examine the use of the optical two-dimensional (2D) DIC technique. The 2D-DIC technique is a full-field non-contact optical technique for spatial deformation measurement on the surface of the tested sample. It directly provides the displacement components on the surface. The 2D-DIC computational algorithm divides the measured area into cells and tracks and compares features to locate their displacement vectors with time from a successive series of digital images. The DIC method has been extensively developed and applied since the early 1980's, and its principles and theoretical background have been extensively reported [22–26].

The overall objective of this work is to examine two new experimental tests and data characterization of light-weight silica aerogels in order to determine reliably their elastic properties, such as the Young's modulus. Direct block compression and indirect diametral (Brazilian disk) tests are employed. To the best of our knowledge, this is the first ever use of the Brazilian disk test for measuring the Young's modulus of aerogels. The non-contact full-field 2D-DIC strain measurement is used, and a technique is developed for applying an external coat with speckle field. A back-calculation (inverse mechanics) technique is used in the indirect tests to determine the mechanical properties of aerogels from the test results and parametric finite-element solutions.

2. Experimental

2.1. Materials and samples characterization

The tested silica aerogel samples were in the form of disks and blocks with dimensions of about 2.6 cm × 0.7 cm and 2.5 cm × 2.5 cm × 1.0 cm, respectively. These samples were obtained commercially from Aerogel Technologies™ [27]. They are hydrophilic and were supercritically dried with CO_2. Each sample was labeled 'compression aerogel disk (CAD)' followed by its number. Therefore, the first task was to perform geometrical