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Compressive behaviors and morphological changes of resorcinol–formaldehyde aerogel at high strain rates

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ABSTRACT

The dynamic compressive properties of resorcinol–formaldehyde (RF) aerogel were investigated using a spilt Hopkinson pressure bar. The effects of strain rate, water absorption and sample basal area on the dynamic behaviors of RF aerogel were investigated through a series of dynamic experiments. Morphological changes of RF aerogel under compression were studied by SEM, TEM, BET and BJH methods. Results show that the compressive behaviors of RF aerogel display a remarkable strain rate strengthening effect. The water-saturated RF aerogel shows stiffened behavior at high strain rates in comparison with the dry RF aerogel, but the dynamic failure strain is small. The dynamic compressive behaviors of RF aerogel display a remarkable size effect. The stress increases with the sample basal area at the same strain. At high strain rates, the pores shrink rapidly; RF particles fuse together to form larger particles and surface area reduces rapidly. It is the fusing of gel particles that allows RF aerogel to be much more ductile than silica aerogels and not to break into fragments at high strain rates.

1. Introduction

Aerogels are novel porous materials with many interesting characteristics, such as low bulk density, high surface area, continuous porosity and highly crosslinking structure [1]. Because of their unique microstructure consisting of particles and pores with nanometer size, aerogels are widely used in thermal insulation, nuclear particle detection, light-guides and electronic device [2,3], etc. Aerogels have also been proposed as a shock absorbing material. The low density of aerogels allows higher compression and thus more internal energy could be deposited in it [2]. Nevertheless, their engineering applications are limited due to their brittleness and low strength. Pure silica aerogels are generally considered to have poor mechanical properties. The compressive strength of pure silica aerogel with density of 0.112 g/cm^3 is only 1.8×10^4 Pa [4].

Recently, several methods were used to improve the mechanical properties of aerogels. For example, fibers [4,5] and organic matter (epoxy resin [6], polyvinyl butyral [7]) were added into aerogels. Fibers play a role of compartments containing aerogels, which could support the aerogel structure. The organic matter crosslinks the nanoparticles and reinforces the weak connections between the nanostructures without clogging the pores. Organic aerogels are new kinds of aerogels with better mechanical properties [8]. Resorcinol–formaldehyde aerogel is a kind of organic aerogel, which has better mechanical properties than native silica aerogel [9].

Recent studies have focused on the compressive properties and morphological changes of aerogels. The quasi-static compressive properties of fiber reinforced silica aerogel were studied by Parmenter and Milstein. They considered that for specimens in the low range of matrix density (less than approximately 180 kg/m^3), high fiber reinforcement had a beneficial effect on strain at fracture. The compressive strength did not show discernible trends with fiber percentage for a given matrix density [5]. Structural and textural evolution of the silica aerogels depends on the densification process. Densification by isostatic compression leads to a pore size decrease while the size of the particles remains unchanged. However, densification caused by sintering results in an increase in solid particle size [10,11]. Katti et al. investigated the quasi-static compressive properties of isocyanate cross-linked amine-modified silica aerogels: they found that these aerogels were suitable candidates for energy absorption in structures subjected to impact/penetration loadings [12]. Luo et al. found that the dynamic compressive behavior of isocyanate-crosslinked silica aerogel displayed a remarkable strain rate strengthening effect. This aerogel failed generally by splitting, but was much more ductile than native silica aerogel [13]. The previous studies indicated that some kinds of aerogels were suitable for force protection. Resorcinol–formaldehyde (RF) aerogel with better mechanical properties may have potential for shock absorption as lightweight structural materials. For ballistic applications, energy absorption

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of RF aerogel at high-speed impact is important. Thus dynamic compressive experiments using a split Hopkinson pressure bar would be necessary. To date, however, the compressive properties and the morphological changes of RF aerogel at high strain rates, have rarely been reported.

The present paper investigated the dynamic compressive behaviors of RF aerogel using a large-sized split Hopkinson pressure bar (SHPB). The effects of strain rate, water absorption and sample basal area on the dynamic behaviors of RF aerogel were investigated through a series of dynamic experiments. Morphological changes of RF aerogel were studied by SEM, TEM, BET and BJH methods. The research results might potentially shed some light on the ballistic applications of aerogels.

2. Experimental

2.1. Material preparation

RF hydrogel was synthesized by the polycondensation of resorcinol [$C_6H_4(OH)_2$] (R) with formaldehyde [HCHO] (F) [14]. RF solutions were prepared by mixing resorcinol, formaldehyde, sodium carbonate and distilled water under vigorous stirring for 45 min. The molar ratio of R to F was 1:2. The RF solutions were poured into sealed glass container to prevent the evaporation of water during gelation and gelled by aging for 72 h at 90 °C into an oven to obtain RF hydrogel. To remove water from their structure, the gel was solvent exchanged by immersing in acetone for 72 h prior to the vacuum drying. Finally, the hydrogel was dried in a vacuum oven at 80 °C for 2 days to obtain RF aerogel. The density of RF aerogel was 0.24 g/cm^3.

2.2. Quasi-static and dynamic compressive experiments

Quasi-static compressive experiments were conducted at a computer controlled electronic universal testing machine. Dynamic compressive experiments were conducted on a large-sized SHPB facility, which was used to measure the stress–strain relationship of RF aerogel during deformation. The schematic diagram of SHPB facility is shown in Fig. 1. The bars of SHPB facility were made of hard aluminum alloy with Young's modulus of 70.25 GPa, density of 2.81 g/cm^3 and wave speed of 5000 m/s. The diameter of all bars is 37 mm. The lengths of the incident bar, transmission bar and striker bar are 2000, 2000 and 800 mm, respectively. The duration of the pulse was 320 μs. The specimen was sandwiched between the incident bar and the transmission bar. The striker bar was projected towards the incident bar, by means of a gas gun. Upon impact, a compressive incident stress pulse was generated in the incident bar and propagated towards the interface between the incident bar and specimen. At the incident bar/specimen interface, the incident pulse was partially reflected back into the incident bar and the rest was transmitted through the specimen into the transmission bar. Resistance strain gauges were mounted on the incident and transmission bars to measure the incident, reflected and transmitted pulses. Because RF aerogel has much lower strength and impedance compared with the bars, the initial transmitted pulse signal was weak. In order to capture the initial signal on the transmission bar effectively, a semiconductor strain gauge was mounted on the transmission bar to measure the initial transmitted pulse signal. Since the sensitivity coefficient of the semiconductor strain gauge is 100, which is about 50 times greater than that of the resistance strain gauge, the signal-to-noise ratio of the signal measured by the semiconductor strain gauge is about 50 times greater than that measured by the resistance strain gauge, providing accurate pulse signal on the transmission bar. The output signals from strain gauges were recorded on a digital oscillograph. The stress, strain and strain rate histories were calculated according to the strains measured on the incident and transmission bars as follows [15].

$$\sigma(t) = E\left(\frac{A_b}{A_s}\right)\varepsilon_T(t) \tag{1}$$

$$\dot{\varepsilon}(t) = -\left(\frac{2C_0}{L}\right)\varepsilon_R(t) \tag{2}$$

$$\varepsilon(t) = -\left(\frac{2C_0}{L}\right)\int_0^t \varepsilon_R(t)dt \tag{3}$$

where $\varepsilon_T(t)$ and $\varepsilon_R(t)$ denote the amplitudes of the transmitted and reflected strain pulses. E, A_b and C_0 denote the Young's modulus, cross-sectional area and longitudinal wave speed of the bars, and A_s and L are the cross-sectional area and length of the specimen, respectively. The history of stress according to Eq. (1) and the history of strain according to Eq. (3) are combined to yield the stress–strain relation of specimen. RF aerogel samples were compressed at several strain rates to determine the effects of strain rate. RF aerogel samples saturated with water were used in SHPB experiments to determine the effect of water absorption on the dynamic compressive behavior of RF aerogel. Both the dry and water-saturated RF aerogel samples had a diameter of 37 mm and a thickness of 5 mm. RF aerogel samples of several basal areas were used in SHPB experiments to determine whether the basal area played a role in the stress–strain relationship at high strain rates. The thickness of the RF aerogel cylindrical samples was 5 mm. The diameters of the samples were 37, 28 and 18 mm, respectively.

2.3. Characterization of RF aerogels before and after compression

Microstructure investigation of RF aerogels before and after compression (under quasi-static and dynamic conditions) was carried out on a S4800 scanning electron microscope (SEM) and a Tecnai F30 transmission electron microscope (TEM). The surface morphology of RF aerogel was studied by SEM. The samples were coated with Pt and were observed on scanning electron microscope. The internal structure of RF aerogel was observed by TEM. The samples for TEM were prepared by grinding in an agate mortar and subsequent dispersing the powder in ethanol. The suspension was treated in ultrasonic bath for about 10 min. A drop of very dilute suspension was applied on carbon-coated copper grid for microscopic examination by TEM. Surface area/Pore size analysis of RF aerogel samples before and after compression (under quasi-static and dynamic conditions) was conducted on a NOVA 4200e

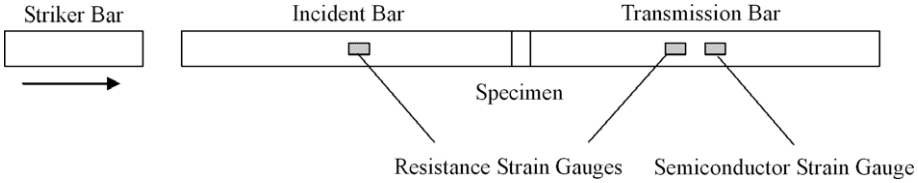

Fig. 1. Schematic diagram of SHPB facility.