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Mechanical, thermal and flammability properties of glass fiber film/silica aerogel composites

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ABSTRACT

Aerogels are the best thermal insulating materials ever known because of their extremely low thermal conductivity. However, their applications are limited because of their brittleness and low strength. For maintaining the integrity, we prepared glass fiber film reinforced silica aerogel composites have been prepared by the sol–gel method via ambient pressure drying. The composites were characterized by scanning electron microscopy, bulk density analysis, Brunauer–Emmett–Teller method, thermal constant analysis, and compression and cone calorimeter tests. The H_2O: TEOS molar ratio of the silica aerogels was found to affect the properties of the composites significantly. As the H_2O: TEOS molar ratio increased from 2 to 6, the density of the composites first decreased dramatically and then increased slightly. The thermal conductivity also showed the same trend. The mechanical properties of the composites were improved greatly compared to the pure aerogels without compromising their thermal insulation properties. Moreover, the composites prepared in this work exhibited more elasticity and flexibility than the conventional thermal insulating materials. The data obtained from the cone calorimeter test showed that with an increase in the H_2O: TEOS molar ratio, the fire hazard of the composites decreased.

1. Introduction

Recently, energy-saving thermal insulation materials have attracted immense attention. Particularly, fireproof thermal insulation materials with high efficient thermal insulation are gaining attention and have promising prospects in the field of heat insulation [1–3]. Silica aerogels are ultralight materials with a three-dimensional network of interconnected nanometer-sized primary particles. Owing to their remarkable characteristics, such as low density (0.003–0.5 g cm^{-3}), high porosity (80–99.8%), high specific surface area (500–1000 m^2 g^{-1}), and extremely low thermal conductivity, typically of the order of 0.005–0.015 W m^{-1} k^{-1} [4–6], Silica aerogels are regarded as the best thermal insulating materials ever known. However, because of high porosity, they exhibit poor mechanical properties, such as low strength, poor toughness and brittleness. This seriously limits their practical applications

Several methods have been used to improve the mechanical properties of silica aerogels [7–9]. One of the most convenient and effective methods is the addition of fibers to the silica sols as supporting skeletons, resulting in the formation of fiber reinforced aerogel composites [10]. Various fibers such as ceramic [11,12], mullite [13], glass [14,15] and aramid fibers [16] can be combined with aerogel for reinforcement. Although aramid fibers exhibit good mechanical properties, the organic components in these fibers may contribute to combustion. Inorganic glass fibers, which exhibit high mechanical strength and are non-combustible, heat insulating, and corrosion resistant, have been widely used. It has been reported that the introduction of fibers in aerogel increases their thermal conductivity [13,17]. The increase of thermal conductivity is a disadvantage to the thermal insulation performance of the aerogel composites. A cheaper, simpler, and safer method to introduce glass fibers into aerogel composites is to handle the glass fibers with a carding machine. The resulting films are lighter than blankets and can reduce the contact between the fibers and the thermal conductivity of the composites. Moreover, reducing the effective pore size is an efficient method to suppress the heat transfer in silica aerogels. The molar ratio of water to silica precursors plays an important role in controlling the nanostructure of silica aerogels. Therefore, in this study, we investigated the effect of H_2O: TEOS molar ratio (S) on the thermal conductivity and mechanical properties of GF/aerogel composites.

Currently, the introduced hydrophobic organic groups during the preparation of silica aerogels are inevitable for acquiring the hydrophobicity. These organic components just contribute a potential fire risk to silica aerogels exposed to a heat flux or other flame propagating materials in an existing fire. However, the flammability of hydrophobic silica aerogels has been neglected till date and a very limited literature is available to discuss the fire reaction properties and burning behavior of SA composites under radiation. It is generally believed that the heat

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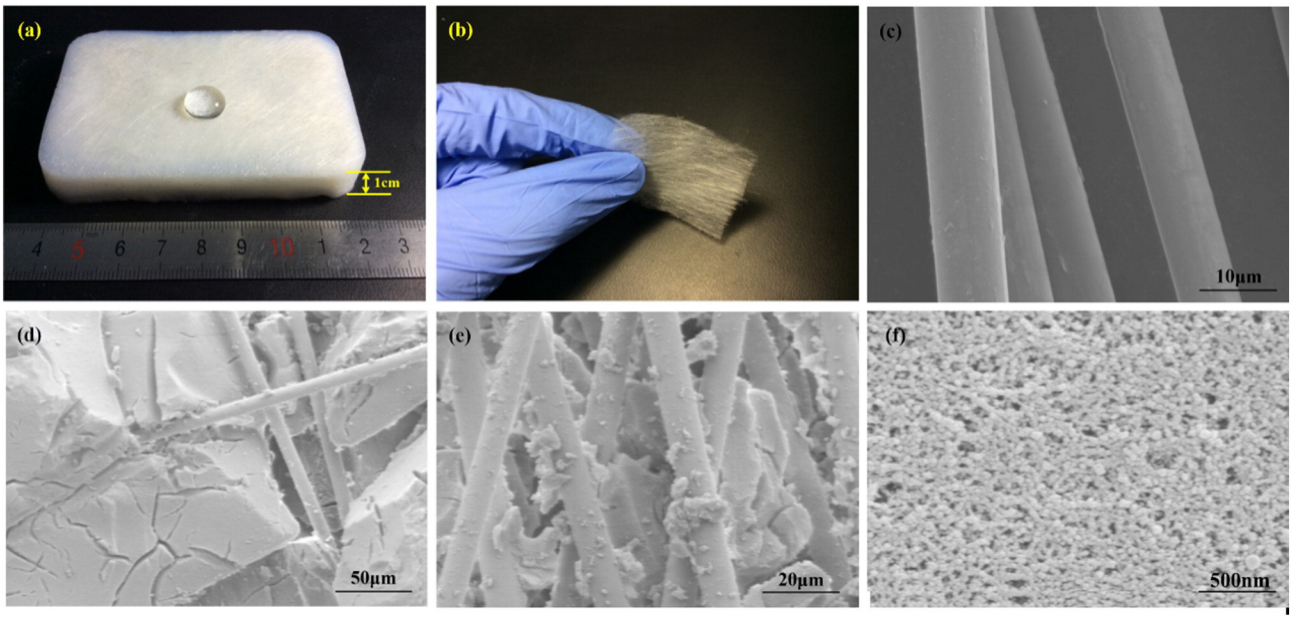

Fig. 1. GF/aerogel (a), glass fiber film (b), glass fibers (c), overall view of the composite (d), fiber surface covered with aerogels (e), and magnified view of the silica aerogel (f).

release rate (HRR), which corresponds directly to the intensity of fire, is the most important factor in controlling fire hazards. Thus, we propose that by estimating the HRR of the composites with different S values using a cone calorimeter, their burning behaviors can be investigated.

In this paper, glass fiber film reinforced aerogel composites were fabricated by the sol-gel method via ambient pressure drying. The effects of water content on the thermal conductivity, elastic modulus and fire resistance of the GF/aerogel composites are analyzed, aiming at obtaining flexible fiber-reinforced aerogel composites with low thermal conductivity and good fire resistance.

2. Experimental procedures

2.1. Materials

Glass fibers (8–10 μm diameter) in excess of 5 cm were obtained from Sinopharm Chemical Reagent Co., Ltd (China), and were used as reinforcement. TEOS (AR, ≥98%), EtOH (AR, ≥99.7%), n-hexane (AR, ≥97%), trimethylchlorosilane (CP, ≥98%, TMCS), hydrochloric acid (36–38%, HCl) and ammonia (25–28%, $NH_3 \cdot H_2O$) were purchased from China National Medicines Co., Ltd.

2.2. Preparation of aerogel composites

In order to obtain glass fiber films, the thick entangled glass fibers were dealt with fiber carding machine in advance. TEOS, EtOH, water and 0.1 mol/L HCl (aq) was mixed for under vigorous stirring 40 min. The molar ratio of TEOS: H_2O: EtOH: $NH_3 \cdot H_2O$: HCl was fixed at 1: (2–6): 10.55: 9.7×10^{-3}: 1.6×10^{-3}. After sufficient hydrolysis of TEOS, 0.5 mol/L NH_4OH (aq) was added to the obtained sol under stirring for 10 min for the condensation reaction. The as-prepared sols were poured into rectangular molds with inner dimensions of 83 mm × 54 mm × 15 mm and the resulting glass fiber films were immersed into the resulting alcosol slice by slice. The molds were set for 30 min and were aged with EtOH for 8 h. Solvent exchange was done twice with n-hexane for 6 h. Surface modification was carried out with a hexane/TMCS solution for 24 h. Finally, the aged alcogel was dried at 60, 80 and 110 °C for 8 h in an oven to finally obtain the GF/aerogel composites.

Table 1
Physicochemical parameters of the aerogel with the various S values.[a]

Mole ratio of H_2O: TEOS (S)	BET surface area ($m^2\ g^{-1}$)	Average pore size/nm	Pore volume ($cm^3\ g^{-1}$)	Water contact angle/°
2	867.6 ± 13.6	8.6 ± 0.2	2.5 ± 0.1	168.3 ± 1.1
3	963.8 ± 21.4	9.8 ± 0.3	2.7 ± 0.3	166.3 ± 0.1
4	917.2 ± 10.1	10.3 ± 0.2	2.8 ± 0.1	159.1 ± 0.2
5	904.6 ± 14.9	13.7 ± 0.4	3.7 ± 0.2	155.5 ± 0.6
6	887.5 ± 11.2	13.9 ± 0.3	3.8 ± 0.1	148.5 ± 0.6

[a] All the values are given in a form of average value plus standard error (with three repeated tests) for each specimen.

2.3. Methods of characterization

The bulk densities of the composites were measured from their weights and dimensions. The surface morphology of the composites was studied by using a field emission scanning electron microscope (SEM, SIRION200, FEI). The porosity, P%, was calculated according to Eq. (1) [18]:

$$P\% = \frac{1/\rho_b - 1/\rho_s - 1/\rho_f}{(1/\rho_b)} \quad (1)$$

where ρ_b, ρ_s and ρ_f are the density of the composites, silica aerogel and glass fibers, respectively. The thermal conductivity was calculated by using the transient hot wire method at 25 °C in a vacuum container (TC3000E, XIATECH, China). The contact angle measurements were performed using a contact angle meter (SL200KS contact angle meter and interfacial tensiometer, KINO, USA) to quantify the degree of hydrophobicity. The specific surface area and pore size distributions were calculated by the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) nitrogen gas absorption and desorption (NOVA 2200e, Quantachrome, USA) methods. The elastic modulus and compressive strength were calculated using a dynamic and static fatigue testing machine (E3000K8953, Instron). For uniaxial compression test, the samples with the dimensions of Φ30 mm × 30 mm × 10 mm and loading rate of 4 mm/min were used.

For the investigation of the flammability of the GF/aerogel composites, the corresponding tests were carried out according to the ISO 5660 standard with a cone calorimeter (Fire Testing Technology, UK). The flammability parameters investigated were the time to ignition