Mechanical properties of fiber-reinforced silica aerogels and its phase change nano-composite

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ABSTRACT

Both new nano-porous silica with high performance insulation and advanced nanotechnology-based phase change materials (PCM) will strikingly contribute to the utilization of thermal energy efficiency. The present study is devoted to the investigation of mechanical properties of fiber-reinforced silica aerogels and its phase change composite materials due to their fewer reports in the literatures. The preparation process of fiber-reinforced silica aerogels and PCM/silica composite was firstly reported, and then the bending and compression strength properties of the two types of materials were performed via two techniques: three-point flexural bending and uni-axial compression tests. Meanwhile, the effects of fiber content on mechanical properties of silica aerogels were investigated. The result indicates the bending strength and compression strength of silica aerogels increased firstly when adding fiber volume fraction from 5.8% to 9.4%， and then decreased when adding fiber volume fraction from 9.4% to 11.5%. Furthermore, the result shows the bending strength and the compression stress of fiber-reinforced aerogles were improved by impregnating PCM in its porous structure.

Keywords: Fiber; Silia aerogels; Nano-composite; Scanning electron microscopy (SEM); Mechanical properties

1. Introduction

Silica aerogels have broad applications or applications prospect in the fields where have strict limit of space, weight or thermal insulation, for example in aeronautics and aerospace [1], transportation of off-shore oil and gas [2], and buildings energy saving [3]. They are sol-gel derived porous inorganic materials recognized for their low density (as low as 0.01-0.02 g/cm^3)[4], high porosity (>90%)[5], and high specific surface area (600-1000m^2/g)[6]. These structural properties result in a few thermal conductivity and high optical transparency [7]. The properties of silica aerogels that make them such good insulators also make them inherently fragile and brittle. Thus, their use in load-bearing applications presents a challenge. Currently, attention is being placed on improving the mechanical properties of silica aerogels without sacrificing other unique properties.

Many works focused on the methodology that improves the strength of silica gels through extended aging and ultimately particle growth [8]. While percolation of the particle has a profound effect on the physical characteristics of the aerogels, often times resulting in a decrease in surface area, increase in pore size and increase in density at extended aging times [9, 10]. Secondary reinforcement materials such as fibers, particles etc. may be introduced into the alcogel before the gel is supercritically dried [11-14]. Among of them, silica fiber is inorganic fiber that contents more than 96% of SiO_2, it's resistant to high temperature, soft point 1700 ^{0}C, long term service temperature 900 ^{0}C, and it can work 10 minutes at 1450 ^{0}C and keeps good state at 1600 ^{0}C for 15 seconds. For these properties of chemical stability, high temperature resistance and obtain resistance, it is prime candidates used as reinforcement. The reinforcement materials behave as a reinforcing scaffold for the aerogel structure creating a robust physical framework that helps support the fragile aerogel network.

Furthermore, PCM/silica composite though impregnating PCMs into silica nano-porous network structure, which store excess heat generated from the work components and release it reversibly when needed, provide a smart approach for more efficient temperature management and energy utilization [15,16]. However, relatively few experiments to determine the mechanical properties of PCM/silica composite have been carried out to date. No results for the PCM/silica composite have been found in the literature.

To investigate the mechanical properties of PCM/silica composite as well as fiber-reinforced silica aerogels, experiments most applicable to the present work include work on the properties of fiber-reinforced silica aerogels. Two processing parameters, the volume percentage of fiber reinforcements and the matrix density, were altered to obtain silica aerogels with differing physical characteristics. The target density is a readily controllable processing variable, based on the mass of the aerogel phase and the volume of the original mold. The mechanical behavior of the fiber-reinforced silica aerogels and its phase change composite was studied by uni-axial compression and three-point flexural bending. Particular attention was paid to the effects of volume fraction of fiber reinforcements on the mechanical behavior of silica aerogels.

2. Experimental

2.1 Preparation of fiber-reinforced silica aerogels and PCM/silica composite

The preparation process of fiber-reinforced silica aerogels is shown in Fig.1. The source of silica for the aerogels that were prepared in this study was tetraethylorthosilicate (TEOS). The aerogels were synthesized by first dissolving TEOS in ethanol. The mixture was next hydrolyzed with water at room temperature, and a cross-linked silica network was produced. The fiber reinforcements were then mixed into the solution, while it was being stirred at a constant rate. The volume percentage of reinforcements was varied from 0 to 12 % and the target density was varied from 0.1 to 0.2 g/cm^3. The silica fibers used in this study were supplied by Universal Star Group Ltd Ningbo in China. The reinforced aerogels is inorganic fiber that contents more than 96% of SiO2, it's resistant to high temperature, soft point 1700 ^{0}C, long term service temperature 900 ^{0}C, and its density is 1.28 g/cm^3.

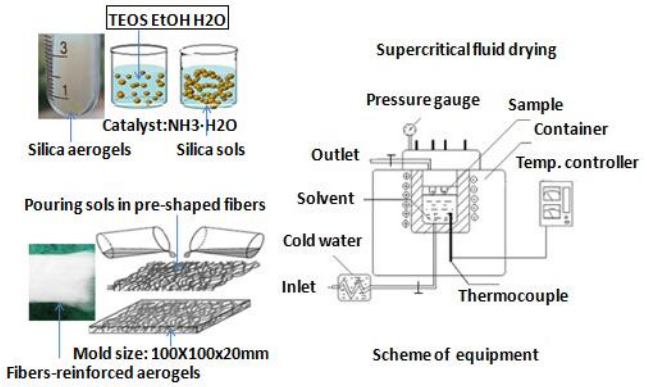

Fig.1. Experimental preparation process of fiber-reinforced silica aerogels

The solution was base-catalyzed with ammonia and ammonium fluoride until it reached a pH of 6.5 and then it was poured into molds. The molds were rectangular, with dimensions of 100x100x20 mm. The target density is the mass of the aerogel phase divided by the mold volume (target density does not include the mass of the fibers). Gelation occurred within 30 min, after which the gels further solidified overnight in the molds. The samples were aged for three days in ethanol before they were supercritically dried (The illustration of supercritical fluid drying equipment is shown in Fig.1.). Gels were supercritically dried by placing them in an autoclave in an ethanol bath, and liquid carbon dioxide was introduced to displace the ethanol. The autoclave was then heated to above critical temperature to vaporize the solvent while eliminating its saturated vapor phase. The vapor was then evacuated and the aerogels were cooled. In the preparation of these materials, the autoclave was pressurized to 3.8 MPa and then the temperature was increased to 278 ^{0}C causing the pressure to rise to 7.6 MPa.

Finally, the PCM (A white waxy solid, paraffin) was heated to over its melting temperature, and then the fibers-reinforced silica aerogels was put into the melted PCM. As a result, the PCM/silica composite was obtained through liquid PCM impregnation due to capillary force of silica pore.

2.2. Structural characterization

The morphologies of fiber, fiber-reinforced silica aerogels and PCM/silica composite were observed with a field emission scanning electron microscope (FE-SEM: JSM-6700F, JEOL, and Japan, with a thin Pt–Pd coating).

2.3. Mechanical properties

2.3.1. three-point bending test

For advanced ceramic matrix composites, a more suitable transverse flexural bending test is most frequently employed, in which a rod specimen having a rectangular cross section is bent until fracture using a three-point loading technique. Three-point bending test were performed using Instron 3366 according to the standard method used for flexural properties [17]. The three-point loading scheme is illustrated Fig.3. The specimen size was fixed at 50×5×4mm. The distance between support points is 40mm. The cross-head speed for Instron machine set at 500um/min.

2.3.2. uni-axial compression

Uni-axial compression tests have been done using a RHEOTXT2/25/SMS machine. The accuracy of the uni-axial compression test [18] depends on the planarity and the parallelism of the contact area. The samples have been checked by a micrometric sensor (±0.1um), which measures the different points on the flats show that this distance is constant. The test specimen's size was fixed at 10×10×15 mm.Load-displacement data was obtained during the test and used for developing stress-strain relations, and calculated compressive strength.

3. Results and discussion

3.1. Volume fraction of fiber and the matrix density of aerogels

The flexural bending and compression strength with varied fiber volume fraction, as shown in Table1, illustrate some experimental fact. The mechanical strength of silica

aerogels will be improved by introducing some range content of fibers. Table1 shows an increase in mechanical strength quantities with addition quantities of 5.8 vol%, 7.6 vol% and 9.4 vol% fibers. However, the mechanical strength quantities are no more increase with addition quantities of more than 9.4 vol%. It means that the flexural bending and compression strength has a turning point with varied content of fibers. We found there is a decrease in mechanical strength quantities with addition quantities of 11.5 vol% fibers compared with that of 9.4 vol% fibers. These findings keep similar when showing the matrix density of silica aerogels. Table1 also shows an increase in matrix density quantities with addition quantities of 5.8 vol%, 7.6 vol% and 9.4 vol% fibers. However, the matrix density quantities are no more increase with addition quantities of more than 9.4vol%. Thus, relations may exist between the content of fibers and matrix density of silica aerogels. Correlations between mechanical properties and the content of fibers/matrix density will be explained as below from the microstructure and preparation process.

Table 1 Mechanical properties of silica aerogels with varied volume percent fiber
(omitted)

Fig.2 shows the microstructure of silica fibers. These fibers disperse randomly each other and many open spaces among them can be found. The spaces among the fibers were filled with silica sols and fibers were sufficiently covered with the sols after sol-gel precursor was immersed into the fibers. Fig2 (a), (b) show that silica aerogels integrated closely with the fibers after supercritical fluid drying process. We found the discrepancy in terms of integration when compared Fig.2(a) with Fig.2(b). The integration of aerogels using 9.4 vol% fibers (Fig.2(a)) combined more densely than that of the 11.5 vol% fibers(Fig.2(b)).

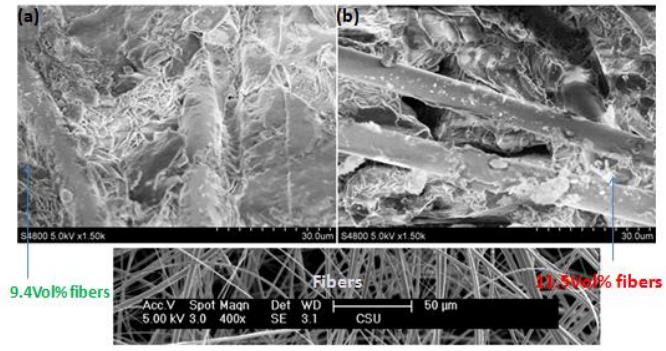

Fig.2 Micrographs of silica fibers and fibers-reinforced aerogels

Fig.3 shows the effect of fibers on the mechanical properties of silica aerogels. Fibers are the main mechanical support of the silica aerogels. The fracture surfaces of the silica aerogels correspond well according to the load-displacement experiments. As shown Fig.3, cracks firstly appeared in aerogels after fiber-reinforced silica aerogels were under flexural bending load. And when the cracks reached the fibers, then they propagated across the interface of fibers-aerogels along the significant pull-out of fibers. Thus, fibers improve the mechanical properties of silica aerogel due to their pull-out energy. The mechanical strength was enhanced with increase of the fiber volume fraction. However, Table1 shows the matrix density was decreased when fiber volume percentage reaches to 11.5% and Fig.2 shows that fibers were not uniformly dispersed into the aerogels. As a result, the mechanical strength of fiber-reinforced silica aerogels was no longer increased until fiber volume percentage reached to 11.5%.

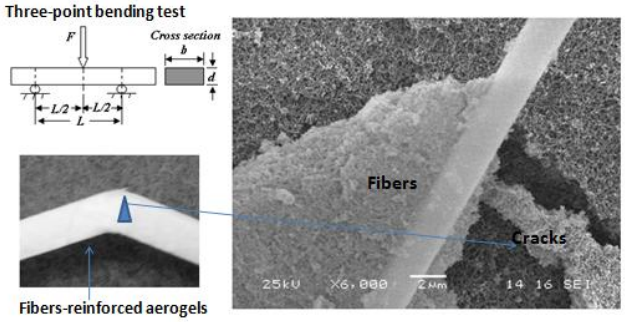

Fig.3 The fracture surface of fiber-reinforced aerogels under bending load

During the preparation of silica aerogels, the porosity of the aerogels was modified by the molar ratio of EtOH/TEOS. In our previous study [19], we found the matrix density of the aerogels decreases with increasing the molar ratio of EtOH/TEOS while the porosity and the average pore size correspondingly increase. The relatively larger pore or cracks consequently resulted in defects of the mechanical strength of aerogels. Because the molar ratio of EtOH/TEOS with five shows good structure of aerogels [19], it was adopted during this present experiment. Meanwhile, there is volume shrinkage during the preparations of silica aerogels. For example, the matrix density was 0.132 g/cm^3 while the target density was 0.082 g/cm^3 when adding 7.6 vol% fibers. Final fiber-reinforced silica aerogels composite densities are significantly larger than the target densities owing to shrinkage during processing and to the additional mass of fiber reinforcements. The final density is taken to be the final mass (including the fibers) divided by the final volume. Additionally, the final matrix density (which is simply called the matrix density) was calculated by subtracting the mass of the fibers and neglecting their volume (i.e. by assuming that the final volume of the matrix is approximately equal to the final volume of the composite). The matrix density and final density was 0.119 g/cm^3, 0.193g/cm^3, respectively when adding 5.8 vol% fibers. Volume shrinkage of silica aerogels happened when compared with the target density. However, the variation of volume shrinkage is less than that of silica aerogels without

any content of fibers. The results also show the fibers effectively retarded the volume shrinkage of aerogels, thus increase the matrix density and enhance the mechanical properties of the silica aerogels.

According to above analysis and results of our experiments, we performed the mechanical properties of fiber-reinforced silica aerogels and the PCM/silica composite where the fibers volume fraction is 9.4 % and the matrix density of aerogels is 0.141 g/cm^3.

3.2. Mechanical properties of PCM/silica composite

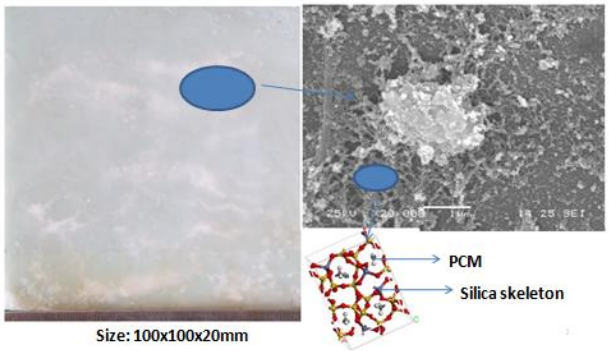

Fig.4. Real photo and SEM of the PCM/silica composite

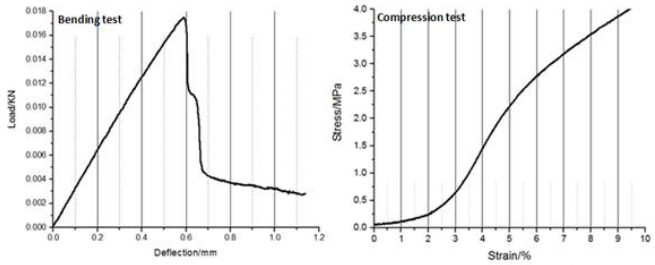

Fig.5. Three-point flexural bending and compression tests of PCM/silica composite
(omitted)

4. Conclusion

A sol-gel synthesized method to fabricate fiber-reinforced silica aerogels was introduced to prepare phase change composite through impregnating PCM into silica nano-porous network structure. SEM results showed the PCM as well as the fibers improved the structural integrity of silica aerogels. The flexural bending and compression strength tests indicated both the fibers and PCM contributed to enhance the mechanical strength of the silica aerogels. The results of the effects of fiber content on mechanical properties of silica aerogels showed the bending strength and compression strength of fiber-reinforced aerogels firstly increased when adding fiber volume fraction from 5.8% to 9.4%, and then the increased quantities was intercepted when adding fiber volume fraction with 11.5%.

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