

Heat-resistant, strong alumina-modified silica aerogel fabricated by impregnating silicon oxycarbide aerogel with boehmite sol

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ABSTRACT

In order to overcome the fragility and sintering behavior of silica aerogel at high temperature, heat-resistant and strong alumina-modified silica aerogel was fabricated by impregnating silicon oxycarbide (SiOC) aerogel with industrial boehmite sol. The partial sintering of SiOC aerogel during its pyrolysis endowed alumina-silica aerogel its high strength. Meanwhile, the carbon in SiOC can also act as a pore former due to oxidation with O₂ during the subsequent calcination in air. This approach is straightforward and economical. The as-prepared composite aerogel exhibited high surface area (278 m²/g) and compressive strength of up to 2.17 MPa. Compared to SiO₂ aerogel prepared using the same method, the composite aerogel was found to be more heat resistant. After being heated to 1000 °C and 1200 °C for 2 h, the surface area of composite aerogel reached up to 183 and 22 m²/g, respectively. On the other hand, the corresponding values for linear shrinkage were as low as 6.93% (for 1000 °C) and 18.90% (for 1200 °C). The XRD patterns indicated that amorphous SiO₂ and γ-Al₂O₃ remained in the composite aerogel after heat-treatment at 1200 °C.

1. Introduction

Among various aerogels that have been explored to date, silica based aerogels are the most widely developed and used material system. Due to the high porosity and nanoporous nature of aerogel, which evidently reduces the solid and gaseous thermal conductivities, silica aerogel exhibits extremely low thermal conductivity (0.01–0.03 W/m/K at room temperature) [1–3]. This key property leads to its many applications in various fields, including insulation for advanced industrial devices, space vehicles, hypersonic vehicles, solar energy systems, and architectural purposes [3–6]. SiO₂ aerogels are usually synthesized using sol-gel technology, which is also widely employed to prepare other functional materials [7–10]. In the sol-gel process, the interparticle connecting zones in the framework of aerogel, which are referred to as the neck zones, are formed by coagulation of particles upon gelation and grow to a certain point by dissolution and reprecipitation of silica [11]. Due to this reason, the particles in silica aerogel are held together only by a few Si–O–Si bonds between these neck zones, thus making the aerogel fragile. Beside the inherent fragility of silica aerogel, the sintering at temperature above 600 °C also limits the practical applications of silica aerogel at elevated temperatures [12–14].

In recent decades, many efforts have been made to improve the mechanical properties of aerogel by fiber reinforcement (such as

mullite fiber, carbon fiber, glass fiber), particle reinforcement (such as Al₂O₃, ZrO₂, Si₃N₄), polymer cross-linking and other chemical or physical means [15–24]. Table 1 summarizes some selected properties of mechanical enhanced silica aerogel reinforced by different strategies. It can be observed that the mechanical properties of silica aerogel can be improved to a certain extent by adding fiber or particle in the initial sol [12,15,21,24]. However, this method has some shortcomings. The short fiber or powders, which are used as reinforcement, are normally incorporated by mechanical stirring, hence these powders inevitably exhibit non-uniform dispersion in the aerogels, which leads to the deterioration of performance of aerogels. As for the fiber felt reinforcement, different shrinkage rates between the fibers and aerogel matrices would lead to large residual stresses, which would lead to large cracks. These cracks may be detrimental for aerogels using as thermal insulators, as they may increase the gaseous thermal conductivity. In addition, overlapped cracks may decrease the mechanical strength of composites [25]. Compared to the method of secondary reinforcement, the enhanced effect of polymer cross-linking on the mechanical properties of aerogel is very impressive [16–18,23]. In reference [17], by cross-linking with a polyuria, the elastic modulus of amine-modified aerogel could reach a value of 129 MPa. Such a high strength is attribute to a conformal polymer coating on the silica framework, which widens the neck regions between the adjacent secondary particles. For pure SiO₂ aerogel, the neck zones, formed between the secondary spherical silica

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Table 1
Comparison of the select properties of as-prepared aerogels with other silica aerogels reinforced by various methods in references.

	Bulk density (g/cm ³)	Surface area (m ² /g)	Compressive strength (MPa)	Elastic modulus (MPa)	Reference
SiO ₂ aerogel	0.292	287	2.39 ± 0.13	110 ± 3.7	As-prepared
Al ₂ O ₃ -SiO ₂ aerogel	0.343	278	2.17 ± 0.16	78 ± 4.2	As-prepared
Fiber-reinforced SiO ₂ aerogel	0.320	–	1.45 ± 0.18	36 ± 3.4	[12]
ASPEN Pyrogel® XT-E	0.20	–	0.26 ^a	–	[24]
Glass fiber/SiO ₂ aerogel	0.35	–	0.3 ± 0.04	–	[21]
Si ₃ N ₄ /SiO ₂ aerogel	0.320	170	3.20	–	[15]
Poly(hexamethylene diisocyanate)/SiO ₂ aerogel	0.447	165	–	47.5	[18]
Polyurethane/SiO ₂ aerogel	0.155	227	0.55 ^a	2.50	[23]
Isocyanate/SiO ₂ aerogel	0.478	261	4.26	129 ± 8	[17]
CVD treated SiO ₂ aerogel	0.288	784	–	8.77 ± 1.1	[22]
Pure SiO ₂ aerogel	0.240	–	1.01 ± 0.03	16.4 ± 1.8	[12]

^a Stress at 25% strain.

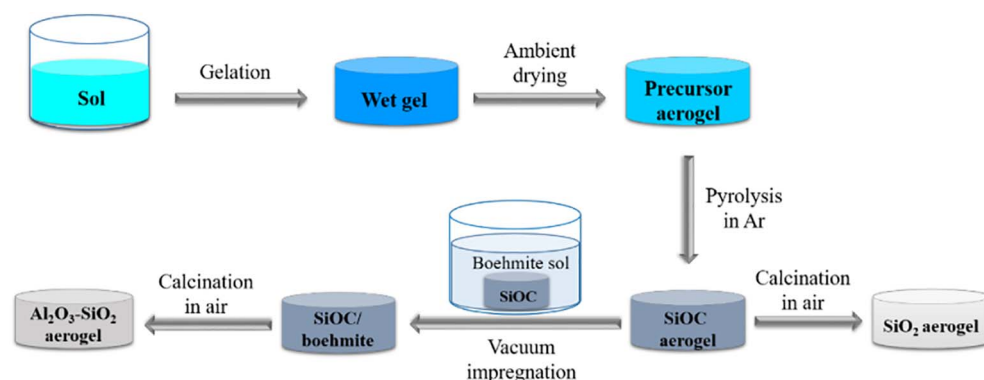


Fig. 1. Schematic of the fabrication process for SiO₂ and Al₂O₃-SiO₂ aerogel.

particles, are held together by only a few Si–O–Si bonds which are responsible for the fragility of aerogel. When the neck zones are widened by some means (such as, compounding with organic polymer), the specific stiffness and strength of monolithic silica aerogels could be improved. Although polymer cross-linking is an effective way to strengthen the aerogels without significantly increasing the density, it does not apply to those aerogels, which are used for high temperature applications. This is due to the thermal pyrolysis of the polymer, which occurs above a temperature of 500 °C.

With regards to the improvement of heat resistance at high temperature, the addition of some metal oxides (such as Al₂O₃, Y₂O₃) to silica is an effective way to enhance high temperature resistance [26–30]. Among most of the recent composite aerogels, Al₂O₃-SiO₂ aerogel exhibits excellent heat resistance, and retains a high specific surface area of 125–136 m²/g after being heated to 1300 °C [31]. However, the preparations of composite aerogels are often sophisticated and high-cost due to different hydrolysis rates of the precursors and expensive supercritical drying processes.

SiOC aerogel is a kind of aerogel with nanoporous structure and high heat resistance, which can be prepared by pyrolysis of cross-linked polysiloxane networks containing Si–R bonds under inert atmosphere [32]. Owing to some degree of sintering (or partial sintering) between the adjacent particles during the pyrolysis at high temperature, the neck zones between the particles in the skeleton of aerogel were rendered wider, which resulted in better mechanical strength of SiOC aerogel than silica aerogel [33]. Recently, inspired by the nanoporous structure and good mechanical properties of SiOC aerogels, a novel and convenient method to fabricate alumina-modified silica (Al₂O₃-SiO₂) composite aerogel by impregnating boehmite sol into SiOC aerogel is developed. The proposed method can simultaneously improve the strength and thermal stability of silica aerogel. In the proposed method, SiOC aerogel was prepared from triethoxyvinylsilane/tetraethoxysilane (VTES/TEOS) using sol-gel technology and ambient drying, and industrial boehmite sol was used as the alumina source. It is noteworthy

that the carbon in SiOC, which prevented the excessive sintering of aerogel during pyrolysis process, will act as a pore-forming agent through oxidation with O₂ during the subsequent calcination in air. This ingenious strategy can make for high porosity and low density of the composite aerogel. The resultant Al₂O₃-SiO₂ exhibits excellent mechanical strength and high heat-resistance, which can retain a high specific surface area of 91 m²/g after being heated to 1100 °C for 2 h. The present work provides a low-cost and simple strategy to fabricate high-strength and high heat-resistance Al₂O₃-SiO₂ composite aerogel.

2. Experimental

2.1. Sample preparation

Al₂O₃-SiO₂ aerogel was prepared by calcining the SiOC aerogel impregnated with boehmite sol. The synthesis steps are shown in Fig. 1. In addition, the SiO₂ aerogel without alumina was also prepared by the same method.

The SiOC aerogel monolith, used in this paper, was fabricated through the pyrolysis of precursor aerogel, which was prepared from triethoxyvinylsilane/tetraethoxysilane (VTES/TEOS) using sol-gel process and ambient drying. Based upon the process description given in a previous study [33], VTES (Huaian Heyuan Chemical Co.), TEOS (Sinopharm Chemical Reagent Co.), deionized water, ethanol and *N,N*-dimethylformamide were mixed together with a molar ratio of 1:1.8:3:1.2 respectively. The pH value of the solution was adjusted to 2 by adding 2M HCl. After stirring for 2 h, 2M NH₄OH was added dropwise into the solution to adjust the pH value to 7. Then the sol was immediately poured into a polystyrene mold for gelation at room temperature. After aging and solvent exchange, the gels were dried slowly at 60 °C for 48 h, and then pyrolyzed at 1000 °C under Ar atmosphere. Then, the as-prepared SiOC monolith was used as a preform for subsequent impregnation process. The boehmite sol (Zibo Jinqi Chemical Co.) with a concentration of 25 wt% was diluted with distilled

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