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The influence of thermal treatment on the microstructure and thermal insulation performance of silica aerogels

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ABSTRACT

In this work, thermal treatment to the as-prepared silica aerogels was explored as a strategy to reduce the macropores in aerogels, thus to improve the thermal insulation performance. The experimental results show that the pore size exhibits a slight shift to the value lower than the pore size of no-heat-treated silica aerogels and the macropore volume fraction experiences sharp decline from 63.05 to 24.82% after thermal treatment at 800 °C. The thermal conductivity doesn't increase with the enhancement of density but decreases from 0.0090 W/(mK) for the no-heat-treated sample to 0.0080 W/(mK) for the 400 °C- treated aerogels. The reason is that the reduction of macropores results in the restriction degree of gaseous thermal transfer being enhanced in aerogels, which is proved through theoretical calculation by two models. However, the thermal conductivity dramatically climbs to 0.030 W/(mK) after 800 °C owing to the pore structures being damaged and solid heat transfer gets significantly enhanced, which is confirmed by the theoretical calculation. This study suggests that appropriate high-temperature treatment to the as-prepared aerogels could improve the thermal insulation performance and the usability of silica aerogels at room temperature.

1. Introduction

Since the first preparation by Kistler in 1931 [1], silica aerogels, as a kind of sol-gel derived nanostructured materials with low density, high specific surface area, low thermal conductivity, and interconnected nanopore network filled with air, have been regarded as a most promising candidate for super thermal insulating materials [2–5]. Thermal insulating materials are widely needed in the high-tech field demanding stable working environment for spacecraft and in building area requiring low energy consumption [6–9]. Generally, thermal treatment is used to investigate the thermal stability and thermal conductivity of materials in the high-temperature environment to avoid unexpected and uncontrollable damage in the thermal insulating field. However, the investigations have ignored that appropriate pore contraction of as-prepared aerogels during high-temperature treatment may be favorable for improving the thermal insulation performance at room temperature. Macropores in aerogels have an important effect on the thermal insulation performance of aerogels. The existence of substantial macropores within the network will weaken the suppression of gaseous heat transfer of silica aerogels because the size of macropores generally exceeds the gas mean free path (ca. 70 nm). Thus the

reduction of macropore contents possesses a large potential in improving the thermal insulation performance of aerogel.

It is generally believed that different degrees of structural variation occur as a function of temperature due to the system being prone to energy minimization when aerogels are exposed to varying temperature environment. With regard to silica aerogels, the porous structures will be severely damaged due to the sintering behavior when it is subjected to the environment over 650 °C, which is referred to as sintering temperature [10]. Investigations suggest that smaller pores are expected to shrink and finally disappear faster than larger pores when materials experience high-temperature environment [11]. Other investigations support an opposite viewpoint that the larger pores may preferentially shrink and disappear in aerogels, especially at the early stage of thermal treatment process, when the fractal build-up network of aerogels is taken into account [12,13]. This is attributed to the complex and unique hierarchical nanostructure of aerogels. In general, the nanostructure of silica aerogel is modeled as multilevel fractal structure, which is constructed with aggregates. The aggregates are composed of interconnected porous secondary particles, and the secondary porous particles are assembled by dense primary particles and micropores inside it [14–16].

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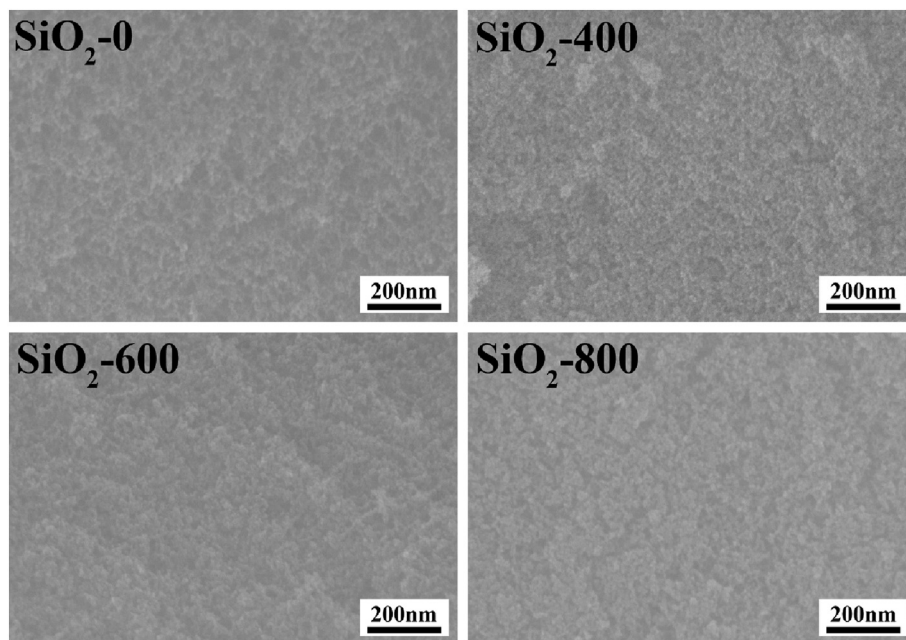


Fig. 1. SEM images of SiO₂ aerogels by varying temperature.

In this work, we suppose and explore that appropriate high-temperature treatment to as-prepared aerogels as a processing technique to decrease the room-temperature thermal conductivity of silica aerogels by reducing the macropores, on the basis of close relationship between the pore structure and thermal conductivity. To the best of our knowledge in this category, no investigation focusing on thermal treatment as a method to improve the room-temperature thermal insulation performance of silica aerogel has been reported so far.

2. Experimental

2.1. Thermal treatment to the as-prepared aerogels

The starting material of silica aerogel includes tetraethoxysilane (TEOS), ethanol, and oxalic acid solution (0.01 mol/L). In a typical synthesis, the precursor and water, as the mole ratio was 1:6, were mixed with ethanol and diluted oxalic acid solution to hydrolyze for 24 h under stirring. The sol after the ammonia was added under stirring will be transferred into an ampoule bottle (inner diameter is 20.5 mm) at the close height of liquid level (15–20 mm), and the gelation happens and then the gel is dried. Thus the aerogels in cylindrical shape were prepared. Subsequently, the as-prepared aerogel monolith was calcined in a muffle furnace in air atmosphere at 400 °C, 600 °C and 800 °C for 2 h, respectively. The resulting samples are denoted as SiO₂-x in this work, where the x refers to the thermal treatment temperature. Specifically, the SiO₂-0 represents the sample without thermal treatment.

2.2. Characterization

The bulk density of the SiO₂-x is determined by the ratio of mass to volume, where the mass is weighed by electronic balance and the corresponding volume is obtained by measuring the dimension of samples using vernier caliper. In order to reduce the errors in the calculation, we use the average value of three parallel samples for each relevant parameter in this work. The porosity is calculated using the following formula:

$$\Phi(\%) = (1 - \rho_b/\rho_s) \cdot 100\% \quad (1)$$

where the ρ_b and ρ_s represent the bulk density and skeletal density of

SiO₂-x aerogels, respectively. The ρ_s is 2190 kg/m³ [17]. We assumed that skeleton compactness of silica experienced a small fluctuation in the moderate treatment conditions, thus the skeletal density taken as constant value in this work is an approximate treatment. The morphology of sample is observed by a scanning electron microscope (SEM, ZEISS SUPRA 55) and a high-resolution transmission electron microscope (HRTEM, JEM-2100). The aerogels are ground into powders and then dispersed in ethanol in a centrifuge tube. The centrifuge tube is placed in the ultrasonic dispersing instrument (KQ-700D) for 5 min and the solution is dropped onto lacey support films. After the ethanol volatilizes, the HRTEM samples were obtained. The organic groups composition is investigated by Fourier transform infrared spectroscopy (FTIR, NICOLET-IS50) covering the range from 4000 to 400 cm⁻¹. The nature of phase with different thermal treatment temperature is monitored by X-ray diffraction (XRD, 2500VB2 + PC). The pore structure is characterized using specific surface and pore analyzer (Micromeritics ASAP 2020). The specific surface areas and pore size distribution are analyzed by the BET (Brunauer–Emmett–Teller) and the BJH (Barrett–Joyner–Halenda), respectively. The total pore volume per unit of mass is calculated by the formula:

$$V_T^* = 1/\rho_b - 1/\rho_s \quad (2)$$

The thermal conductivity is evaluated by Hotdisk (TPS 2500S, Sweden), and the instrument in this work is based on the principle of transient heat flow. The lower limit of the instrument is 0.005 W/(mK).

3. Results and discussion

3.1. The physical and structural properties of aerogels

The morphology of silica aerogel with different thermal treatment temperatures presents apparent variation, as shown in Fig. 1. The microstructure of pristine silica aerogels (SiO₂-0) exhibits well-developed interpenetrating network composed of interconnecting necklace-like skeleton. The local inhomogeneity of pores can be observed in SiO₂-0. The high-branched hierarchical structure can be illustrated in Scheme 1. The secondary particles, covalently bonded by primary particles, join together to form chain aggregation structure, and the diameter of secondary particles is smaller than 10 nm. The secondary particles contributing to the aggregates are derived from condensation

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