



High temperature annealing for structural optimization of silica aerogels in solar thermal applications



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ABSTRACT

Optically transparent, thermally insulating monolithic silica aerogel, with its high solar transmittance and low thermal conductivity, is well-suited for solar thermal applications, particularly concentrated solar power systems. The properties of silica aerogel are directly determined by the structure of the highly porous, interconnected silica network. By using high temperature annealing to control this structure post-synthesis, we were able to optimize the material to increase solar transmittance with minimal effect on the effective thermal conductivity using an easy and scalable method. Samples made from two silica aerogel chemistries and two annealing temperatures (400 °C and 600 °C) were investigated as a function of annealing time. The annealed samples showed a maximum increase in solar spectral transmittance of over 3% while the effective thermal conductivity (including radiative and conductive contributions) was shown to increase by as much as 40%, indicating a need to optimize the annealing time for maximum performance. The properties of the characterized aerogels were used to demonstrate aerogel annealing optimization using a concentrated solar power receiver model operating at 400 °C. The model predicted a maximum receiver efficiency gain of 1% by annealing for 24 h at 400 °C, representing a significant gain in overall system efficiency.

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1. Introduction

In a world that is constantly increasing its energy demands, solar thermal power has the potential to fulfill a substantial portion of power requirements. Concentrated solar power (CSP) systems use large concentrating optics to focus sunlight onto an absorbing receiver that transfers heat at high temperatures to a working fluid for direct use or electricity generation via a heat engine. Unlike more popular wind and photovoltaic systems, CSP systems can take advantage of cheap and efficient thermal storage to provide energy throughout a 24-hour output cycle [1]. Even assuming a modest growth rate of 20–30% per year, CSP is expected to grow > 10 times by 2020, but if system efficiency is increased, CSP growth could be even more aggressive [2]. One approach to increase system efficiency is by improving the performance of the receiver which depends on absorbed solar energy and heat loss suppression. Optically transparent, thermally insulating aerogels can use these intrinsic properties to increase receiver performance with minimal cost impact or design change [3,4].

Silica aerogel can be a high-performance, optically transparent thermal insulator that consists of a cross-linked silica particle backbone supporting a mesoporous (2–50 nm) network [5]. The solid network is comprised of primary silica particles that aggregate to form secondary

particles with a diameter of 1–10 nm [6]. These secondary particles bond together to form an interconnected network that creates a structure that is 80–99% porous. With such a low solid volume fraction and pore sizes smaller than the mean free path of air, the solid and gaseous thermal conductivity of aerogels is very low (less than the thermal conductivity of air at room temperature) [7]. The radiative thermal conductivity is also very low due to the high absorptivity of the silica particles in infrared (IR) wavelengths; the particles absorb incident radiation and re-radiate in all directions, greatly reducing transmitted IR radiation [8]. However, the silica particles and the pores allow the transmission of solar wavelengths (300–2000 nm), allowing it to perform like a selective surface. The only barrier to solar transmission through the structure is the refractive index difference between the aerogel backbone (silica particles) and air, but because the sizes of the pores and particles are small relative to solar wavelengths, most of the solar light is directly transmitted and only a small fraction undergoes scattering [9,10]. These properties make silica aerogels very attractive for CSP applications [4,11].

Silica aerogel has been investigated extensively in the past, particularly for its transparency and thermal properties. Several studies have examined the effect of the sol-gel synthesis process on the final structure of aerogel [5], including investigation of different chemical precursors [12,13], different aging conditions [14], and varied drying conditions [15,16] to control the pore and particle networks. Others have investigated how the variations in the structure (pore size, particle

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size, density) relate to variations in the properties of interest, such as optical and thermal transmission [9,10,17,18]. However, for optimization in an application such as CSP, we need to consider not only how to control the structure, but how this controlled structure in turn affects all properties relevant to performance. For example, solar transmission of the aerogel can be increased by decreasing density, while radiative heat loss is reduced by increasing density, indicating a need for optimization [19]. One easy and scalable method of controlling the aerogel structure post-fabrication is through high temperature annealing [20]. Annealing at temperatures of 400 °C and above results in significant change in the structure [21], which can decrease the size of features within the aerogel structure to increase total solar spectrum transmission by up to 6% (98% transmission through a 4 mm thick sample); however, the effective thermal conductivity can also increase by up to 50%. Therefore, high temperature annealing can be used to investigate the aerogel performance change, via both structure and properties, to optimize the material in varied conditions.

In this study, we considered both the structural change and the related optical and thermal property changes caused by post-fabrication high temperature annealing time. We have developed optically transparent, thermally insulating monolithic aerogel with high solar transparency designed to operate in a concentrated solar thermal receiver. We studied the effect of annealing samples synthesized using two recipes at two different annealing temperatures (400 °C and 600 °C) over a range of annealing times as well as post-annealing resting times. Comparing sample properties and structure allows us to identify reversible and irreversible changes as a function of annealing time to optimize for a range of operating conditions. We then used this data to demonstrate how via annealing the structure and properties of the aerogel can be controlled to reach maximum efficiency for CSP applications.

2. Fabrication

The following describes our procedure for synthesizing high and low density silica aerogels by sol-gel polymerization of tetramethyl orthosilicate (TMOS, 131903, Sigma Aldrich) and Methyl Silicate 51 (MS-51, CAS#12002-26-5, Austin Chemical Company), respectively. High density aerogel (160–180 kg/m³) was synthesized using an ammonia solution (NH₃, 2.0 M in Methanol, 341428, Sigma Aldrich) as a catalyst to promote both hydrolysis and condensation reactions. TMOS was diluted by methanol (MeOH, 322415, Sigma Aldrich) followed by addition of NH₃ and water. The mixing molar ratio of chemicals was NH₃:TMOS:water:methanol = 0.004:1:4:6. Then, the sol was gelled in a disposable vial. After 2 weeks, the lid of the sol-gel mold was removed, followed by addition of ethanol. The mother solvent was replaced with ethanol (EtOH, 89234-848, VWR) in preparation for critical point drying (CPD, model 931, Tousimis) as EtOH is miscible with liquid CO₂.

To synthesize low density aerogel (65–75 kg/m³), MS-51 was used as the silica precursor with ammonium hydroxide solution (28% NH₃ basis, 320145, Sigma Aldrich) as the catalyst and H₂O source. MS-51 was diluted by a mixture (20:80 weight ratio) of dimethylformamide (DMF, 227056, Sigma Aldrich) and methanol followed by ammonium hydroxide solution (NH₄OH, 28% in water, 320145, Sigma Aldrich) at room temperature. The molar ratios of H₂O/MS-51, (DMF + MeOH)/MS-51 and DMF/MeOH were 28, 70 and 2.5, respectively. The sol was then gelled in a disposable vial for 10 min. The wet gel was kept in the solution at room temperature. After 2 weeks, the solvent was replaced with EtOH in preparation for CPD.

To dry both wet gels precursors in EtOH without cracks, it is important to dry them slowly to minimize capillary pressure during the CPD process. A bleed rate of 100 psi/h was used to decrease the CPD chamber pressure from ≈ 1300 psi to ambient pressure. After drying, the monolithic aerogels were approximately 10 × 10 × 0.4 cm in size which were then cut into several samples 2 × 2 × 0.4 cm in size for the annealing study (see Fig. 1(a) inset for image of an unannealed transparent aerogel).

3. Experimental procedure

After fabrication, we characterized samples in their initial state using small angle x-ray scattering (SAXS), and transmittance and reflectance measurements in ultraviolet (UV), visible (Vis) and IR wavelengths. The samples were then placed into an oven and ramped up to the specified annealing temperature at a rate of 10 °C/min. An unannealed reference sample was stored separately for comparison. After the predetermined annealing time for each sample was reached, we carefully removed the sample from the oven and placed it in a ceramic dish to cool to room temperature. We then measured the size, mass, and transmittance and reflectance of the sample. After characterization, the sample was stored in a plastic container alongside the reference sample. The samples were allowed to rest for a predetermined amount of time before we again measured size, mass, and transmittance and reflectance (see Supplemental Table T1 for sample annealing and resting times). After all samples were annealed and rested, we used SAXS to determine the final scattering size.

The density of the aerogels was calculated from the volume and mass measurements. The mass of the aerogels was measured on an analytical balance (Discovery DV215, OHAUS) in ambient conditions. This mass includes the adsorbed water at ambient conditions, which was measured on similar samples to be 5% by weight. The mass of the aerogel is therefore over-estimated, but this error was considered when calculating the uncertainty in the density. The cross-sectional surface area and thickness were measured by imaging the samples from both the top and the side with a 2 cm optical reference scale. The dimensions of the images were measured using ImageJ software [22] with the error estimated by the repeatability of independent measurements on a single sample.

Optical properties in the solar spectrum were determined by measuring total hemispherical reflection and transmission data in the center of the samples. This measurement was performed on an Agilent Cary 5000 UV-Vis-NIR spectrophotometer on wavelengths from 250 to 2500 nm using a polytetrafluoroethylene coated integrating sphere (Internal DRA-2500, Agilent). The IR transmittance and reflectance of the aerogels was measured using a Thermo Fisher FTIR6700 Fourier transform infrared spectrometer. Direct transmittance and reflectance was obtained through the center of the sample in the wavelength range of 1.5–18 μm.

SAXS was used to determine the scattering length distribution and estimate the average scattering radius size. Characterization was done on a SAXSLAB custom-built instrument using a Cu K-alpha x-ray source. Samples were probed in the center to best match transmission and reflection measurements, reduce edge defect effects, and maintain consistency between measurements. The scattering pattern was reduced and corrected using SAXSGUI software, and the mean scattering radius was estimated using MCSAS software [23] and verified in SasView [24].

4. Results and discussion

One of the most beneficial effects of annealing at temperatures below the glass transition temperature of the silica aerogel is the increase in visible transmittance. Fig. 1(a) shows the transmission spectra of a TMOS sample before and after annealing at 400 °C across relevant wavelengths of interest for solar thermal applications. The data shows that while the maximum total transmission was reached immediately after annealing, when the aerogel was allowed to rest at room temperature, the optical properties rebounded towards their initial state. By allowing the samples to rest for long periods of time, we were able to reach a final rested state of material properties to compare to the initial and annealed states. Examining aerogel samples at these three stages allowed us to identify properties that changed irreversibly and reversibly with annealing, and the corresponding time scales.

The spectral transmission and reflection data can be related to the intrinsic optical properties, particularly the scattering and absorption

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