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Experiment and identification of thermal conductivity and extinction coefficient of silica aerogel composite

Hua Liu [a], Xinlin Xia [a, *], Xiangqian Xie [b], Qing Ai [a, **], Donghui Li [a]

[a] School of Energy Science and Engineering, Harbin Institute of Technology, Harbin, Heilongjiang, 150001, China
[b] Science and Technology on Space Physics Laboratory, Beijing, 100076, China

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ABSTRACT

Highly porous silica aerogel composites have shown excellent potential as insulation materials for various insulation systems, the knowledge of their conductive and radiative properties is of essential importance. In this study, several experiments were performed to measure the transient heat transfer behavior of silica aerogel composite at temperature between 290 and 1090 K with various gas pressures ranging from 0.01 Pa to 100 kPa. The temperature dependent conductive part of thermal conductivity $k_c(T)$, and global extinction coefficient of the silica aerogel composite were simultaneously determined from the experimental temperature data by solving an inverse problem employing the genetic algorithm as optimization method. The retrieved conductive thermal conductivity fell within 0.013–0.032 W/(m·K), increased nonlinear with increasing gas pressure, and showed an approximate linear increasing trend as temperature increases. The global extinction coefficients were independent of gas pressure and showed relatively good agreement with those predicted from Mie theory. These properties could be used to predict the effective thermal conductivity $k_{eff}(T)$ for evaluating thermal performance of the insulation systems, and to separate contributions of conductive and radiative heat transfer in silica aerogel composite at various temperatures and gas pressures.

1. Introduction

Silica aerogels are nanoporous amorphous materials prepared by the sol-gel technique and supercritical drying [1], featuring high porosity, low density, high specific surface area, and ultralow thermal conductivity at ambient temperature [2–5]. The pure silica aerogels are brittle and easy broken, thus different types of fibers are generally doped into the aerogels to reinforce their mechanical properties [6–9]. Since the extinction coefficient of pure silica aerogel is extremely small in the near-infrared spectral range (3–8 μm) [1,10,11], the role of thermal radiation is expected to be significant for applications at elevated temperatures. In order to extend their application at elevated temperatures, various infrared radiation opacifiers such as carbon black, silicon carbide, and TiO_2 particles, usually featuring spherical shape with average diameters of about 2–5 μm, are added into the aerogel matrix to attenuate thermal radiation [11–13]. The fibers and opacifier particles are usually occupy very small volume fractions to maintain high porosity of the materials. Silica aerogels doped with fibers and opacifiers are also called silica aerogel composite (SAC), which have found excellent potential applications as high temperature insulation used in applications including aerospace [14], high temperature energy storage [15], and solar energy utilization systems [16,17].

A good evaluation of thermal performance of the SAC based high temperature thermal insulation systems is required in the process of designing the systems thus improve their efficiencies. Since thermal radiation in the SAC used in high temperature insulation systems must be taken into consideration as it becomes significant, heat in the SAC is transferred simultaneously by conduction and radiation. Convection can always be neglected due to their nanoscale pores with average diameter of about 2–50 nm [18,19].

As regards the conductive heat transfer in the SAC, numerous theoretical and numerical studies have been conducted based on the microstructure of the solid matrix as well as the fractions and properties of the constituents. Most of these studies considering simplified 3D structures such as the artificially designated [20,21] and the randomly generated [4,22] solid matrixes, the predicted

* Corresponding author.
** Corresponding author.
E-mail addresses: Xiaxl@hit.edu.cn (X. Xia), hitaiqing@hit.edu.cn (Q. Ai).

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results were usually validated by effective thermal conductivity measurements. This subject is beyond the scope of the present study, one can refer to He et al. [3], Bouquerel et al. [19], Tang et al. [21], and Ebert et al. [23] for more details, the authors conducted detailed review of the analytical models and numerical methods.

Many theoretical studies were conducted uniquely on radiative transfer of SAC, mainly focus on predicting the radiative properties of aerogel matrix, fibers, opacifiers, and the composites based on their microstructures and optical properties [5,13,21,24]. These results were mainly validated either by experimental measurements or by results predicted from different methods. Compared with theoretical studies, the experimental measurements are relatively limited. Fu et al. [25,26] obtained the absorption and scattering coefficients of silica aerogel and silica-doped alumina aerogel from normal-hemispherical transmittance and reflectance measurements by solving an inverse problem combining a two-flux approximation solving the radiative transfer equation (RTE) and an optimization method based on least-squares algorithm. Several similar experiments were performed to determine the extinction coefficient of silica aerogels [20,27–30] from transmittance measurements, but their experiments did not permit to separate absorption and scattering contributions. Determination of extinction coefficient of SAC opacified with a large volume fraction (3.9% for the present SAC specimen) of SiC opacifier from transmittance measurements might be challenging as the specimen extinction coefficient would be very large, thus the reliable transmittance data would be difficult to be measured.

Several researchers were interested in investigating the coupled conductive and radiative heat transfer in SAC by performing effective thermal conductivity measurements based on either steady-state [7,31,32] or transient methods [18,20,33–36]. Yuan et al. [7] measured the effective thermal conductivity of silica aerogel composite from the steady-state method, the temperature range investigated was 300–700 °C. Lee and Cunnington [31] investigated the combined conduction and radiation in fiber-filled silica aerogels, and performed several experiments to measure the effective thermal conductivity between 350 K and 1200 K by using a guarded heat flow meter based thermal conductivity apparatus, and various gas pressures were considered in the experiments. Smith et al. [32] measured the effective thermal conductivity of carbon opacified silica aerogel under various gas pressures between 1.0 Pa and 10^5 Pa using a guarded hot plate method. Employing transient methods, Wei et al. [20] performed the effective thermal conductivity measurements of silica aerogel and xonotlite-aerogel composite insulation at temperature between 300 and 970 K and gas pressure ranging from 0.045 Pa to atmospheric pressure using the transient hot-trip method. Kwon et al. [33] investigated the effective thermal conductivities of aerogel composite for thermal insulation between 25 and 400 °C, by employing transient hot wire technique. Besides, the transient plane source (TPS) method [37] was also widely employed in SAC effective thermal conductivity measurements [18,34–36]. The above steady-state and transient methods based experiments gave a unique measured parameter characterizing both conductive and radiative heat transfer globally, they did not permit to distinguish the conductive and radiative heat transfer contributions.

It can be seen that the literature on modeling radiative or conductive heat transfer in SAC is abundant, while the experimental investigations remain relatively limited. Moreover, the experimental studies generally focus on either the radiative alone or the effective thermal conductivity measurements. Besides, to the best of the authors' knowledge, there is no experimental work to evaluate simultaneously both the temperature dependent radiative and conductive contributions in SAC at various temperatures up to 1100 K from a unique measurement. This paper proposed an inverse method for retrieving the temperature dependent conductive thermal conductivity $k_c(T)$ and two global extinction coefficients $\beta^{tr}_{R,1}$ and $\beta^{tr}_{R,2}$ characterizing radiative transfer from experimentally measured transient temperature. The basic principle of the inverse method is to minimize the discrepancy between the experimental data and the temperature prediction obtained by solving combined heat transfer in SAC. First, we simplified the combined conductive and radiative heat transfer in SAC, and established the number of conductive and radiative properties required to simulate the transient temperature. Then, we validated numerically that the thermal behavior of the SAC can be very well reproduced by using the simplified model and the parameters chosen. Last, we retrieved the conductive thermal conductivity and two global extinction coefficients of SAC at temperature ranging from 290 K to 1090 K and gas pressure between 0.01 Pa and 100 kPa from experimental data, and separated the conductive and radiative contributions.

2. Experiments and analysis

2.1. Specimen and experimental setup

The porosity φ of the present SAC specimen is 83.3%, and the apparent density ρ_{com} is 402 kg/m^3. The compositions and their volume fractions of the specimen are as follows, silica particle 12.3%, silica fiber 0.5%, and SiC 3.9%. The mean diameters of the silica fibers, silica particles, and SiC opacifiers are 7 μm, 7 nm, and 3 μm, respectively, these data were measured by the producer by analyzing SEM photos. The specimen featuring cuboid shape with the same measured length and width of 300 mm, and the thickness of the specimen is 30 mm. The SAC specimen is somewhat hydrophilic and it can absorb water vapor in atmospheric environment, while the effect of humidity on the SAC thermal behaviors has been shown to be non-ignorable [38]. In order to reduce the impact of humidity on the thermal behavior of SAC specimen, the specimen was placed in a vacuum cavity for supercritical drying at 150 °C for 24 h, then it was installed on the experimental setup, and was heated at 800 °C in a quasi-vacuum environment (<0.01 Pa) for several hours before the experiment. During the experiment, the specimen did not contact with the atmospheric environment thus no water vapor was absorbed.

An experimental setup was built to measure the heat transfer behaviors of the SAC at various temperatures and gas pressures. Fig. 1 shows the schematic of the experimental setup, the coordinate system, and the positions of thermocouples installed on the specimen. The radiant heater was made of graphite plate which can reach a high temperature of 2300 K in vacuum or nitrogen atmosphere. The temperature of the heater, labeled as the furnace temperature T_f, was measured using a type C thermocouple and was then digitally controlled by a proportional-integral-derivation (PID) controller [39]. The SAC specimen was placed between two graphite septum plates with very large thermal conductivities, thus the plates can provide uniform thermal boundary conditions. The heater, the septum plates, and the specimen were installed on a high temperature insulation box placed in a vacuum cavity in which the gas pressure was measured and automatically controlled at arbitrary values between 0.01 Pa and 200 kPa. Six Type K thermocouples with limiting error of either 2.2 °C or 0.75% were installed at strategic locations (Fig. 1(b)) of the specimen to monitor the temperatures during the experiments. The temperatures were recorded and saved by an Omega multiple channel USB data acquisition module (OM-DAQ-USB-2401). The temperature measurement system was calibrated before performing the experiments and the thermocouples used to measure temperatures at different specimen positions were checked experimentally at two