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Porous Silica Nanotube Thin Films as Thermally Insulating Barrier Coatings

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ABSTRACT: The fabrication and examination of a porous silica thin film, potentially for use as an insulating thin film, were investigated. A vertically aligned carbon nanotube (CNT) forest, created by chemical vapor deposition (CVD), was used as scaffolding to construct the porous film. Silicon was deposited on the CNT forest using low-pressure CVD (LPCVD) and then oxidized to remove the CNTs and convert the silicon to silica for electrical or thermal passivation (e.g., thermal barrier). Thermal conductivity was determined using a 1D heat-transfer analysis that equated radiative heat loss in a vacuum with conduction through the substrate and thin film stack. A comparison of the surface temperature differences between a sample film and a reference of comparable thermal resistance enabled determination of the increase in the thermal resistance and of the thermal conductivity of the films. For film thicknesses of approximately 55 μm , the cross-plane thermal conductivity was found to be 0.054–0.071 $\text{W m}^{-1} \text{K}^{-1}$ over 378–422 K. This thermal conductivity value is in the range of other silica aerogels and consistent with the low gravimetric density of 0.15 g cm^{-3} for the samples. The film is also relatively smooth and flat, with an average arithmetic mean roughness of 1.04 μm .

KEYWORDS: silica, carbon nanotube, thin films, insulation, MEMS, thermal barrier, aerogel

Carbon nanotubes (CNTs) can achieve incredible length-to-diameter ratios. With a diameter of just a few nanometers wide, practical length-to-diameter ratios can be greater than 10000:1, providing relatively large surface area with little volume.^{1,2} The thermophysical and geometric properties for CNTs make them attractive for constructing thin-film materials for use in thermal applications. Robust, high-aspect-ratio microstructures, including microscale films, have been fabricated in a process called CNT-templated microfabrication (CNT-M).^{3–6} In CNT-M, a patterned CNT forest acts as a scaffold, which is infiltrated or coated with additional materials. The conditions and duration of an infiltration procedure can be controlled to result in highly dense or highly porous forest regions. Control of the porosity of the structure and the ability to use insulating materials as scaffolding in a porous thin film makes CNT-M intriguing for use in the creation of a thermally or electrically insulating layer for a microelectromechanical system (MEMS) and other microscale sensors.

Traditional insulating materials in MEMS, such as silicon oxide or silicon nitride, while having a much lower thermal conductivity than silicon, have values larger than that required for many MEMS applications. Further, thermal or electrical cross-talk can affect the sensor sensitivity and accuracy. Attempts to incorporate other insulating materials, such as silica aerogels, into microdevices have failed to preserve the

material's low thermal conductivity.⁷ Aerogels are lightweight, synthetic materials that can be fabricated from a variety of chemical compounds [including silica (SiO_2)⁸], but they generally lack structural integrity and do not have a clear path for MEMS integration. Recent explorations into using nanostructures to create spectrally selective surfaces for radiative cooling indicate promise in tailoring thermal and structural properties.^{9,10} Silica-based, porous materials constructed from nanoparticles have also appeared in recent literature, demonstrating low thermal conductivities of 0.10–0.16 $\text{W m}^{-1} \text{K}^{-1}$ using sol–gel methods to obtain free-standing silica monoliths.¹¹

This work explores the capability of using insulating thin films, constructed using CNT-M, as a thermal barrier in MEMS sensors and similar applications by determining the thermal conductivity of porous silica nanotube thin films. CNT-M affords the method to create insulating thin films that can be integrated into MEMS/nanoelectromechanical system (NEMS) sensor architectures by infiltrating CNT forests with

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Figure 1. (a) Vertically aligned CNTs grown using a lithographically patterned catalyst. (b) Uniform silicon infiltration of the CNT forest using LPCVD. (c) Oxidation in an O_2 -rich furnace, burning away the CNTs and leaving a (d) porous silica structure.

low-pressure chemical vapor deposition (LPCVD) of silicon and then oxidizing at high temperatures to simultaneously burn out the carbon and oxidize the silicon. The result is a porous silica structure similar to an aerogel. However, a structure of this kind offers several specific benefits relative to aerogels. First, porous silica films in this work offer the ability to easily and preferentially locate insulating regions on a substrate through lithographic patterning or liftoff of the iron catalyst used for CNT growth. Second, control of the CNT growth height readily offers control of the film thickness (and associated thermal and electrical resistance); the terminal film thickness or CNT growth height is achieved in less than a 1 or 2 min (typically on the order of tens of seconds). Third, our approach offers a rigid, surface-mounted insulating layer. It is this flexibility in patterning and control of the dimensions that make this material particularly attractive, while also providing a low thermal conductivity. It is envisioned that these porous silica thin films may be used to isolate measurements that would otherwise require a suspended MEMS structure, as in the case of a thermal conductivity detector^{12–14} or a microbolometer.¹⁵ Patterned silica structures of this kind have been demonstrated to be effective for use in chromatography.⁴ The current work demonstrates that structurally stable, insulating thin films can be created using CNT scaffolds to achieve a porous insulating material and seeks to quantify the cross-plane thermal conductivity.

Porous silica films were produced following the CNT-M procedure shown in Figure 1a–c and procedures similar to those in previous CNT-templated works.⁴ Aluminum oxide (50 nm, e-beam evaporation) and iron (4 nm, thermal evaporation) were deposited onto a silicon wafer. CNTs were grown using a chemical vapor deposition (CVD) process of flowing ethylene (C_2H_4) over an iron catalyst at elevated temperature. In a 2.54-cm-o.d. tube furnace, samples were brought to 750 °C in the presence of flowing hydrogen (H_2) at 310 sccm. At temperature and while still flowing H_2 , 340 sccm of ethylene was introduced for 50 s, growing CNTs of 40–60 μm height (55 μm average). After growth, H_2 and ethylene were turned off, and argon was flowed over the samples for 1 min. The furnace heating was then turned off, while argon continued to flow in order to cool the sample. Once grown,

CNTs were treated with ozone to improve silicon deposition.¹⁶ Gas-phase ozonation oxidizes the CNTs, which primes and increases the number of nucleation sites on the tubes and facilitates conformal growth of silica along the CNT. To ozonate the CNTs, the O_3 gaseous reagent is passed over the forest with an Atlas 30 ozone generator (Absolute Ozone, Edmonton, Alberta, Canada) at an O_2 flow rate of $6.7 \times 10^{-6} m^3 s^{-1}$ with the current set at 45%, producing approximately $4.4 g h^{-1}$ of ozone. CNT samples were placed in a 2.54-cm-o.d. fused-silica tube, purged for 1 min with oxygen (O_2), then exposed to the ozone flow for 45 min, before finally being purged again for 1 min under an O_2 flow and then removed. Sample scanning electron microscopy (SEM) images of the CNT template are shown in the Supporting Information.

After CNT growth to the desired height and ozone treatment, CNTs were coated in silicon using LPCVD (Figure 1b). Samples were placed in a sealed furnace tube and evacuated to 2×10^{-4} Torr. The remaining air in the furnace was purged from the tube by flowing nitrogen (N_2) for 30 s to prevent the combustion of CNTs that would occur in air at high temperatures. The N_2 flow rate was slowly reduced and then held constant as the pressure returned to 2×10^{-4} Torr. The furnace was then heated to 535 °C, and the N_2 flow was turned off and replaced with the same flow rate of silane (H_4Si) for 15 min, keeping the pressure at 200 mTorr. The silane flow was then turned off, and the temperature was cooled to ambient. When the temperature dropped below 150 °C, the system was flushed with N_2 for 1 min. The vacuum pump was then turned off, and N_2 was introduced to the system until the pressure exceeded 500 mTorr, at which point the N_2 flow was turned off and the furnace tube seal was opened to reintroduce atmospheric conditions. The resulting CNT structures were conformally coated with silicon; SEM images of conformally coated CNT structures at this intermediate step were shown in previous work and appear to be similar to that in Figure 1d shown after oxidation.⁴

To burn out the carbon and convert the silicon to an insulating silicon oxide, the films were placed in a furnace and heated to 850 °C in ambient air for 2 min, allowing the silicon to oxidize (Figure 1c). After cooling to room temperature, the porous silica thin films were complete for testing. Samples of

approximately 1–4 cm² area were used for thermal conductivity testing.

The density of the porous silica films was determined by separating the thin film from the substrate with a razor blade and weighing it. The volume of the film was determined from the base area of the growth substrate and measured height of the film. When the mass of the film was divided by its volume, the density of the porous silica film was obtained. Density measurements indicated that the porous silica film is similar to that of an insulating silica aerogel. The volume for a 2 × 2 cm film grown to 15 μm height was 0.006 cm³. A typical sample of this kind weighed 0.9 mg, resulting in a density of 0.15 g cm⁻³. For comparison, the density of silica aerogels can range from 0.001 to 0.7 g cm⁻³.¹⁷ Silicon deposition successfully infiltrated the entire depth of the CNT forest, but deposition was observed to be somewhat thicker at the top of the CNTs than at the base (Figure 1d).

The surface roughness and film height were measured with an optical profilometer (Zeta Instruments 20). Samples were scraped along a segment of the thin film to expose the substrate and measure the height disparity between the substrate and thin film. Sample thicknesses were approximately 55 μm and approximately the same height as the original CNT forest before infiltration and burnout. The surface roughness was determined by interrogating an area of 71 × 95 μm (the largest available aperture area). The average arithmetic mean roughness and root-mean-square roughness of four tested films were 1.04 [standard deviation (SD) = 0.19] and 1.33 μm (SD = 0.28), respectively. The maximum peak height and valley height were 4.93 μm (SD = 0.72) and 5.62 μm (SD = 2.28), respectively. Additional specifics regarding the surface morphology, roughness characteristics, and surface imaging is provided in the Supporting Information.

As a governing parameter in the conduction of thermal energy and to quantify the porous silica's thermal insulating properties, the thermal conductivity was measured. 1D heat transfer was established, and the thermal resistance across the thin film was determined with the aid of a reference measurement. The layers of material present in the test condition and the associated thermal resistance for each layer are shown in Figure 2 (with the thermal resistance associated with the porous silica film highlighted in blue; Figure 2d).

To heat the silicon substrate to steady state and establish 1D heat transfer, samples were placed on a 5.1 × 2.7 × 1.3 cm copper block to act as a large, isothermal substrate that was heated by an attached thin-film heater. Four T-type thermocouples were placed in the copper block 1.3 cm from each side edge and 0.3 and 0.8 cm from the top surface, embedded half-way into the block to ensure isothermal conditions in the copper. Thermal insulating foam encased the sides and bottom of the copper block and heater to reduce heat loss and further ensure isothermal conditions. Test samples were placed on top of the isothermal copper block and heated via conduction. Accompanying temperature measurements of the copper block at steady state for each test confirmed temperature uniformity, as noted in the Supporting Information.

During testing, a reference sample and one or more test samples were placed on the heated copper substrate. The copper substrate was painted black in regions where the test samples were not located (Figure 2). The black paint (emissivity $\epsilon = 0.97$; Krylon Ultra Flat Black) enabled use of an IR camera (FLIR SC6103) to measure surface temper-

Figure 2. (a) Schematic of the reference conditions used to determine the thermal resistance of all layers except the porous silica film. (b) Schematic of the test conditions used to determine the thermal resistance of the porous silica film. (c) Schematic of the benchmark conditions using a material of known thermal conductivity (G10) to confirm the measurement method. (d) Resistance network for the material stack for the test conditions, including the porous silica film.

atures. The top of each test sample (or reference/benchmark sample) was also painted black in the same manner in order to determine the temperature difference across test samples (ΔT_{test}) using the IR camera during heating. Each film was sprayed twice by hand with a smooth, even pass. Each pass lasted approximately 0.5 s over the 1 cm length of the film. Control and benchmark samples were prepared in the same way. The surface temperature for each test was determined using an average over the top surface of each sample (see an explanation in the Supporting Information).

The isothermal copper substrate with reference and test samples were placed in a vacuum chamber (measuring 70 cm in diameter and 76.2 cm in length) and evacuated to a pressure of less than 4×10^{-5} Torr in order to neglect convective heat transfer from the surface.¹⁸ The IR camera was located exterior to the vacuum chamber with a direct line of sight to the sample surfaces through a 7.5-cm-diameter sapphire window. The window had a band transmissivity of 0.81–0.85 for source temperatures between 323 and 423 K in the spectral band of 3–5 μm, corresponding to the wavelength range of measurement by the IR camera.¹⁹ A transmissivity of 0.83 was used for IR measurements through the sapphire window, and the impact of the transmissivity variation on the IR measurement of the sample was accounted for by incorporating possible transmissivity variation with temperature into the IR measurement uncertainty (see the Supporting Information).

Because the test and reference samples were thin (<600 μm), heat loss from the sides of the samples was neglected, resulting in 1D conduction heat transfer through the sample thickness and heat loss from the samples radiated to the vacuum chamber walls at ambient temperature. The thermal resistances in the stack are identified by each layer comprising the sample (Figure 2), namely, silicon substrate (R_{Si}), alumina diffusion barrier (R_{Al}), iron catalyst (R_{Fe}), porous silica (R_{ps}), and black paint (R_{p}). The conduction resistance for these

layers can each be calculated using $R = L/kA$, assuming 1D, steady-state conduction and constant properties (where L is the thickness, k is the thermal conductivity, and A is the area of each layer). An additional contact resistance (R_c) is present and includes a thin layer of vacuum grease used for good contact between the substrate and sample.

The reference test, which excludes the resistance associated with the porous silica film, can be used to determine a composite resistance associated with all other layers and the contact resistance combined, creating R_{comp} in eq 1.

$$R_{\text{comp}} = R_c + R_{\text{Si}} + R_{\text{al}} + R_{\text{Fe}} + R_p \quad (1)$$

This composite resistance can be determined by measuring the temperature difference across the reference sample (ΔT_{ref}) and calculating the radiative heat rate, leaving the surface of the reference sample, as given in eq 2.

$$q_{\text{comp}} = \frac{\Delta T_{\text{ref}}}{R_{\text{comp}}} \quad (2)$$

This heat rate (q_{comp}) is equivalent to the net radiative heat-transfer rate (q_{rad}) shown in eq 3 because the heated surfaces are small relative to the isothermal vacuum chamber²⁰

$$q_{\text{rad}} = \varepsilon \sigma A (T_t^4 - T_{\text{surr}}^4) \quad (3)$$

where $T_{\text{surr}} = 296.15$ K (as measured by a thermocouple placed on the aluminum chamber wall), ε is the emissivity, A is the surface area, and σ is the Stefan–Boltzmann constant ($5.67 \times 10^{-8} \text{ W m}^{-2} \text{ K}^{-4}$). With q_{comp} and ΔT_{ref} obtained, the composite resistance is readily calculated and used to determine the resistance to heat transfer across the layers of material in the test conditions excluding the resistance associated with the porous silica film.

A measurement similar to that outlined above for the reference conditions is performed simultaneously for test surfaces that include a porous silica film, introducing an additional layer of resistance. With R_{comp} known from measurements associated with the reference conditions, the rate of radiative heat loss (calculated using a new surface temperature T_t for the test conditions) can be equated to the measured ΔT_{test} divided by the sum of the composite resistance and the resistance associated with the porous silica film, yielding eq 4.

$$\varepsilon \sigma A (T_t^4 - T_{\text{surr}}^4) = \frac{\Delta T_{\text{test}}}{R_{\text{comp}} + R_{\text{ps}}} = \frac{\Delta T_{\text{test}}}{\frac{L_{\text{comp}}}{k_{\text{comp}}A} + \frac{L_{\text{ps}}}{k_{\text{ps}}A}} \quad (4)$$

The only unknown in eq 4 is the cross-plane thermal conductivity of the porous silica film (k_{ps}), which can now be obtained by inserting the known expression for the thermal conductivity of the composite, yielding eq 5.

T_t and T_{comp} are the top surface temperatures, read from the thermal camera, of the porous silica and reference composite sample, respectively.

$$k_{\text{ps}} = \frac{L_{\text{ps}}}{\frac{\Delta T_{\text{test}}}{\varepsilon \sigma (T_t^4 - T_{\text{surr}}^4)} - \frac{\Delta T_{\text{ref}}}{\varepsilon \sigma (T_{\text{comp}}^4 - T_{\text{surr}}^4)}} \quad (5)$$

The copper substrate temperature was increased using a thin-film heater with wire leads passing through a vacuum feedthrough until a desired temperature was reached. Each test was allowed to reach steady-state behavior, defined as less than a 0.5 K temperature change in the copper block over 10

min. Repeat testing at several copper block temperatures was performed, with steady-state behavior reached for each condition. Higher copper block temperatures resulted in larger ΔT_{test} values and greater confidence in the temperature differences measured.

In order to verify the measurement approach for obtaining the thermal conductivity, benchmark samples of Garolite fiberglass laminate (G10, McMaster-Carr) were cut from sheets of thickness of 0.79, 1.59, and 3.18 mm. The 0.79-cm-thick samples were used for reference condition measurements with the 1.59 and 3.18 mm samples used as test condition samples following the approach outlined above for determining thermal conductivity for the porous silica film. This approach of using both a thinner reference condition and a thicker test condition with G10 eliminated the requirement of obtaining the contact resistance. In this manner, the thermal conductivity for G10 could be determined and compared with a published thermal conductivity value, thereby validating the measurement approach. Further, the thermal resistance of G10 in the thicknesses selected above ($R_t'' = 2.6 \times 10^{-3} \text{ m}^2 \text{ K W}^{-1}$) was on the same order as that anticipated from the porous silica layer based on calculations using the thermal conductivity of silica aerogel and a film thickness of 55 μm .

The measured thermal conductivity for G10 averaged 0.31 and 0.25 $\text{W m}^{-1} \text{ K}^{-1}$ for the 1.59- and 3.18-mm-thick samples, respectively, over the temperature range of 391–434 K. Published values of the thermal conductivity for G10 are approximately 0.29 $\text{W m}^{-1} \text{ K}^{-1}$.²¹ Using this value as the correct thermal conductivity, the current measurement approach for the two thicknesses provided results with errors of 7% and 14%. While additional refinements could be made to the measurement method, the good agreement between the measured and published thermal conductivity for G10 yields confidence in using the measurement method for obtaining reasonable thermal conductivity values for the porous silica films.

Thermal characterization of the porous silica films was performed in a manner similar to the benchmark tests using G10. On the basis of the surface temperature measurements of the reference condition and a test sample with a porous silica film, the thermal conductivity was determined using eq 5. Thermal conductivities for two porous silica films are shown in Figure 3 (PS1/PS2), obtained using two reference samples (REF1/REF2). The resulting four measurements obtained from a combination of different samples and references vary by $\lesssim 15\%$ relative to the mean at a given substrate temperature with a mean of 0.062 $\text{W m}^{-1} \text{ K}^{-1}$ over the range of 378–422 K. The thermal conductivity of the porous silica films seems to increase only slightly with increasing copper substrate temperature over the range of temperatures tested, with a range of thermal conductivity values of 0.054–0.071 $\text{W m}^{-1} \text{ K}^{-1}$ over all measurements. Table 1 shows a comparison of the density and thermal conductivities for several related materials, including the silica aerogel and porous silica film. The results indicate that the thermal conductivity obtained for the porous silica films created and characterized using the methods proposed here are consistent with silica aerogels, while also offering a method to integration on a silicon platform.

The uncertainty in the measured thermal conductivity was calculated by using the partial derivative of each variable in eq 5 and the associated uncertainty of that variable. Uncertainty values and calculation methodology are provided in the

Figure 3. Thermal conductivity results of two porous silica films (PS1/PS2) when compared to two separate control samples (REF1/REF2). A condition of “PS1 (REF1)” indicates a result for porous silica film 1 (PS1) relative to reference sample 1 (REF1).

Table 1. Density and Thermal Conductivity of Several Comparable or Constituent Insulating Materials,^{17,22} Including the Porous Silica Film of the Current Work

material	density (g cm ⁻³)	thermal conductivity (W m ⁻¹ K ⁻¹)
air	0.001	0.024
G10	1.800	0.290
silicon	2.329	150.0
silicon dioxide	2.196	1.400
silica aerogel	0.001–0.700	0.004–0.030
porous silica (current work)	0.150	0.054–0.071

Supporting Information. For these conditions, the total uncertainty was determined to be 0.0075 W m⁻¹ K⁻¹.

Experiments for the porous silica film were performed in a vacuum, resulting in a mean thermal conductivity of 0.062 W m⁻¹ K⁻¹. The thermal conductivity of air is 0.024 W m⁻¹ K⁻¹.²³ As an example, Maxwell’s formula for the effective thermal conductivity of a porous medium indicates the potential for a ~25% increase in the thermal conductivity when including the influence of the interstitial fluid (air), assuming a ~93% porosity material.²⁴ However, the Knudsen effect, which occurs when a material’s pore diameter is less than the mean-free path of the gas molecules, may reduce the overall thermal conductivity.²⁵ In air, this occurs in pores of 40 nm diameter and smaller and can reduce the thermal conductivity to less than 0.004 W m⁻¹ K⁻¹.²⁵ Additionally, it should be noted that this thermal conductivity measurement method yields an average value over the film thickness at the micron scale for an inhomogeneous material at the nanoscale. It is possible that changes in the thermal conductivity may also be observed for different porous silica thicknesses if the plasma-enhanced CVD coating thickness at the top and bottom of the film is appreciably different or if burnout of the carbon varies for different thicknesses.

This work reports on a fabrication method for porous silica films and determines the thermal conductivity of these films, created using CNTs as a scaffold. The porous silica film may be used in MEMS/NEMS structures because it has excellent thermally insulating properties, with a thermal conductivity of approximately 0.062 W m⁻¹ K⁻¹ in the range of 378–422 K.

This thermal conductivity value is in the range of other silica aerogels and has a very low density of 0.15 g cm⁻³. The film is also relatively smooth and flat, with an average arithmetic mean roughness of 1.04 μm. The porous silica films demonstrate the ability for CNTs to be used as a structural template for the fabrication of micro/nanostructures. Future work includes the use of alternative coating materials besides silicon/silica, opening interesting possibilities in material creation.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsnm.0c00596>.

Images of the CNT template, details on the surface roughness/morphology, results from transmission electron microscopy studies, details of the temperature measurements, and uncertainty analysis (PDF)

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Notes

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