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Effect of moisture on nanoparticle packed beds

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ABSTRACT

Nanoparticle packed beds are of interest because of their extremely low thermal conductivities. However, measurements for nanoparticle packed beds exposed to humid air have shown anomalously high thermal conductivity. This work examines the cause for that increase in conductivity. Both experimental measurements and an analytical model for the hygrothermal properties of nanoparticle packed beds are presented. Sets of silica and copper nanoparticles are first characterized using electron microscopy (SEM, TEM), X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS). Sorption isotherms for the nanoparticle beds were then found by exposing them to controlled humidity environments. The thermal conductivities of the nanoparticle beds were measured both with the beds under vacuum and then at ambient pressure over a range of controlled humidity. The thermal conductivities of the nanoparticle beds are found to be monotonic functions of humidity, increasing more than order of magnitude. An analytical model of nanoparticle packed bed heat transfer controlled by the nanoscale constriction resistances between nanoparticles is extended to account for liquid condensation between nanoparticles for packed beds exposed to humid air. Good agreement is found between the analytical model and experimental conductivity measurements for 200 nm silica nanoparticle packed beds under vacuum and over the range of humidity tested. Good agreement is also found between the analytical model and the experimental measurements for 20 nm silica nanoparticle packed beds under vacuum and for humidity up to 20%. However, the model underpredicts the experimental measurements of bed conductivity for humidity above 20%. Together the model and the measurements demonstrate the impact of moisture on the thermal properties of these materials and identify important limitations on the use of nanoparticles for very low thermal conductivity insulating materials.

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1. Introduction

One area in which the use of nanoparticles has generated great interest is in their potential for the creation of novel thermal insulation materials. Nanoparticle-based materials like nanoparticle packed beds have been identified as having extremely low thermal conductivities, among the lowest of any solid material [1]. Nanoparticle beds, like other nanostructured materials such as beds of nanowires or nanofibers, porous nanofoams, and aerogels take the form of repeated building blocks connected through nanoscale constrictions that effectively inhibit heat transfer [2–7]. Prasher first identified the mechanism of ballistic phonon transport through the nanoscale contacts between particles that lead to the extremely low thermal conductivity of nanoparticle beds [8]. Based on this analysis, Hu et al. [1] assembled packed beds of alumina nanoparticles ranging in diameter from 11 to 500 nm and experimentally measured thermal conductivities as low as 0.035 W/m °C. Voges et al., set out to create ultra-low conductivity composite insulation materials by bonding together mixtures of silica, alumina and carbon black nanoparticles ranging in size from 30 to 50 nm. Measurements of these nanoparticle composites resulted in thermal conductivities that ranged from 0.01 to 0.035 W/m °C [9].

These studies demonstrated the great potential for insulating materials based on nanoparticles to provide thermal conductivities lower than the conductivities of many gases. However, some of the nanoparticle-based materials reported in the literature have shown thermal conductivities much higher than expected. For example, Voges et al. measured an unusually high thermal conductivity of 0.07 W/m °C for their nanoparticle composite materials after being exposed to air [9]. Work in our lab has given similar results. Thermal conductivities of 0.018–0.054 W/m °C were measured for 200 and 300 nm nanoparticle packed beds under vacuum (0.1 Torr), while the conductivities of the same beds exposed to

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ambient air were found to much higher, in the range of 0.09–0.5 W/m °C [10].

Theoretical work has clearly identified the dominant mechanism for the very low thermal conductivities of nanoparticle beds as the nanoscale constriction resistance between particles [8,11]. In this model, heat transfer between adjacent nanoparticles occurs through two parallel paths, one path through the contact area between nanoparticles and a second path through the fluid filling the interstitial pores between nanoparticles. These two thermal resistances, the constriction resistance due to the nanoscale contact area between particles and the interstitial fluid resistance together determine thermal transport rates from one particle to the next. The constriction resistance can be found assuming a model for particle mechanics to determine the contact area between adjacent nanoparticles. The interstitial fluid resistance depends solely on the pore size between particles and the gas pressure. Given the particle packing geometry, the thermal resistance across the entire packed bed of nanoparticles may then be determined from these two interparticle resistances.

In previous work, our group demonstrated that an analysis based on this nanoscale constriction resistance model could be used to predict the thermal conductivity of a packed bed of nanoparticles under some but not all circumstances. For example, the model was successful in predicting the conductivity of 200 nm silica nanoparticles, for measurements made under vacuum (0.1 Torr), by calculating the contact radius between nanoparticles assuming Hertz's classical mechanics model for contacting spheres, and assuming a simple cubic packing geometry. However, the theory underpredicted experimental measurements of nanoparticle beds exposed to ambient air by more than a factor of four. In addition, the measurements for beds exposed to ambient air showed a temperature dependence not predicted by the theory. The theory also significantly underpredicted the thermal conductivity measurements reported by Hu et al. of alumina nanoparticle packed beds exposed to ambient air [10].

Based on these results, it is clear that exposure to ambient air may cause the thermal conductivity of nanoparticle-based insulation materials to rise by an order of magnitude or more. However, the reason behind this increase in conductivity has been unclear. Identifying the source of the increased conductivity is fundamental to effective use of nanoparticle-based insulation materials. Previous work has indicated that one possible explanation for the increase in thermal conductivity at ambient pressure might be the effect of moisture infiltrating the samples [9,10]. However, there has been no clear experimental evidence to support this hypothesis. Testing this conjecture, and determining the cause of the anomalously high thermal conductivity measurement for these nanoparticle-based materials is the motivation behind the present work.

In the present work, the effect of humidity on the hygrothermal properties of nanoparticle-based insulation materials is documented. First, the physical properties of two nanoparticle packed beds are characterized. The nanoparticle beds are then exposed to a range of humidity and their moisture uptake and thermal conductivity determined. The relationship between humidity, nanoparticle packed bed moisture content and bed thermal conductivity is mapped out. An improved analytical model for nanoparticle bed heat transfer is introduced that accurately predicts the thermal conductivity of a dry nanoparticle packed bed. The model for nanoparticle beds under dry conditions is then extended to include the effect of liquid water condensing between nanoparticles upon exposure to humid air. This model is used to test the hypothesis that liquid bridges formed of condensed water provide an added path for heat transfer between nanoparticles, resulting in the increased thermal conductivity measured for nanoparticle packed beds exposed to moisture.

2. Approach

Three sets of nanoparticle packed beds were assembled of 300 nm copper nanoparticles, 200 nm silica nanoparticles and 20 nm silica nanoparticles. The shape and size distribution of the nanoparticles as well as the packing geometry of the particles in the beds was characterized via electron microscopy (SEM, TEM). The elemental composition of the nanoparticles was verified using both X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS). The effect of ambient humidity on the moisture content of the nanoparticle bed was characterized by gravimetrically determining sorption isotherms for each bed. The thermal conductivity of the nanoparticle packed beds was then characterized using a guard heated calorimeter (GHC). Thermal conductivity measurements were made first under vacuum (0.1 Torr) and then at ambient pressure. To isolate the effect of humidity on nanoparticle bed conductivity, a series of conductivity measurements were then made under controlled humidity ranging from 0% to 50% relative humidity. An improved analytical model, based on the nanoscale constriction resistance developed by Prasher, the JKR model for contact between spheres with significant surface forces, and the packing geometry model for conduction through packed spheres by Tien and coworkers is shown to accurately predict the thermal conductivity of a dry nanoparticle packed bed [12–15]. The model is then used to interpret the present set of measurements of nanoparticle beds exposed to humid air as well as discuss previously published measurements on nanoparticle-based insulating materials.

2.1. Nanoparticle bed assembly

Packed beds were assembled from copper and silica nanoparticles. The copper nanoparticles, purchased from SkySpring Nanomaterials Inc., were nominally 300 nm in diameter, and had a purity of 99.5%. The silica nanoparticles (NanoXact nanoparticles) purchased from NanoComposix had diameters of 200 ± 7 nm and 22.5 ± 2.8 nm respectively. All nanoparticles were delivered with nonfunctionalized, clean surfaces and used as received from the vendors.

To avoid any contamination, the packed beds were assembled by pouring the nanoparticles directly from the sealed vendor packaging into a hardened steel mold as seen in Fig. 1. The nanoparticles were poured into the steel mold and a micrometer spindle inserted in the mold. The micrometer dial was rotated until the nanoparticles were pressed in the mold with an applied pressure of 15.4 MPa. The diameter of the mold, 6.75 mm, determined the diameter of the disk-shaped packed bed. The extension of the micrometer spindle into the mold determined the height of the packed bed disk. Once the bed was packed tightly together, the steel mold was opened and the nanoparticle bed extracted using clean tweezers.

2.2. Sorption isotherm measurements

The effect of humidity on the moisture content of the nanoparticle beds was characterized by determining sorption isotherms for each. The nanoscale pores between nanoparticles in a packed bed provide energetically favorable sites for condensation. For this reason, the moisture content of a nanoparticle bed would be expected to increase as the water vapor pressure of the surrounding air rises. Sorption isotherms were determined by measuring the equilibrium moisture content of the nanoparticle packed beds over a range of ambient humidity levels at a constant temperature of 20 °C following the procedure described by Richards et al. [16].

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