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Fabrication technique and thermal insulation properties of micro- and nano-channeled polymer composites ☆

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ABSTRACT

Sustaining the extra-terrestrial presence of humans in the solar system will require the ability to provide structural, thermal insulation composites (STICs) that have thermal insulation properties in the range of 10–50 mW/m K and mechanical properties at least capable of supporting an anticipated lunar habitat internal pressure of 100 kPa. A technique has been developed which permits the introduction of micro- and nano-scale channels in polymers, in controlled configurations that will allow for optimization of both thermal insulation and mechanical properties. In this approach, conductive heat transfer mechanisms are being limited by the introduction of crisscrossed channel obstacles into an epoxy matrix, which creates a tortuous pathway and inhibits thermal conduction. By varying the volume fraction of micro-channels, control over final density and effective thermal conductivity can be achieved. The micro-channeled materials were created via sacrificial removal of poly(lactic acid) (PLA) fiber networks embedded in a high-temperature epoxy matrix, using either selective thermal degradation or solvation of the thermoplastic fiber. These processes allowed for acceptable rigidity and strength to be retained by the epoxy matrix. Furthermore, it is expected that as channel diameter decreases from micro-scale to nano-scale, gas diffusion will be constrained by the Knudsen effect, since the gas molecules will increasingly collide with the channel wall, and less frequently with each other, as channel diameter becomes comparable to, or smaller than, the molecules' mean free path. This effect, which may limit convective heat transfer through the channels, may be augmented by an even higher degree of tortuosity (or pathlength) imposed by the tightly packed nano-channels. In this report, the thermal conductivities of micro-channeled specimens were compared over a range of channel fractions to provide insight into the mechanisms controlling and limiting heat transfer in these systems.

1. Introduction

Although NASA has a goal to establish a permanent human presence in space beyond the International Space Station, limitations in current materials make extended human survival in deep space difficult. New materials are needed which must demonstrate excellent thermal insulation properties in the range of 10–50 mW/m K, have suitable mechanical properties for large pressure-vessel construction, and are lightweight so that long-range space transport is economically feasible. The high tailorability of polymer materials is being utilized to design new multi-functional polymer composites which meet these property requirements. Additionally, novel micro- and nano-scale morphologies are being developed which are expected to

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further improve these desired properties, with the present study specifically targeting high thermal insulation capability through a strategy utilizing micro-dimensional channeled network structures.

1.1. Approach

The channeled materials were created via removal of a sacrificial template of nonwoven thermoplastic fiber from a cured thermoset composite. The fiber template can be removed by either thermal degradation or solvation, with both methods leaving behind the intact, thermoset matrix. The resulting hollow channels increase the insulation ability of the bulk matrix material by forcing conduction to occur around the channels, thus increasing the pathlength and reducing the conductive energy transfer through the matrix. The hollow channels can also lower convective heat transfer by restricting the molecular mobility of the gas molecules contained within the channels. These effects can be enhanced, respectively, by increasing the number of channels per unit volume and by decreasing channel diameter into the sub-micrometer and nanometer range. The current study is focused on the formation of micro-channel structures and characterization of their thermal insulation properties, as well as on the creation of hybrid micro-channel/nano-channel materials. The mechanical properties of these materials will be the subject of a future report.

1.2. Theory

Typical insulation materials contain high fractions of trapped air, which create limitations in achievable mechanical properties, such as the tensile strength observed in brittle aerogels [1]. The concept of using cylindrical channels instead of pockets may allow for robust mechanical properties to be retained. In the present study, having reduced to practice the creation of micro-channels, the primary interest revolves around the effect of bulk micro-channel density on the effective thermal conductivity of the matrix. By increasing the number of micro-channels per unit volume, the size of the matrix regions located between channels is reduced, thereby decreasing the available pathways for conductive energy transfer to occur. Since the thermoplastic fibers are nonwoven and thus display random orientation, conduction is already difficult due to the curvature and crisscrossed nature of the resulting channels. Packing these randomly curving fibers closer together further decreases the size of the matrix regions, restricting thermal energy transfer and decreasing thermal conductivity. This effect is additionally enhanced when the matrix regions become sub-micrometer, since according to molecular heat transfer theory, non-constant thermal conductivity will result from the closeness of the interface boundaries [2]. Together with enhanced insulation properties, a substantial reduction in density can be achieved, while still retaining a mechanically intact matrix network.

Another area of interest arises from the notion that insulation ability may be augmented by decreasing the diameter of the random channels. When the diameter of the hollow channels becomes smaller, the convective heat transfer becomes more difficult as molecular gas motion becomes more restricted. As the channel diameter approaches nanoscale, it becomes of the same order of magnitude as the mean free path, λ, of the gas molecules. When this occurs, gas flow becomes rarefied and molecular transport becomes difficult, limiting both mass and energy transfer [3–5]. This is referred to as the Knudsen effect and is expected to become evident at channel diameters of approximately $10\lambda_{air}$, or 670 nm, for molecules of air at standard temperature and pressure [3–6]. At these small scales, continuum theory and traditional boundary conditions of the macro-scale begin to break down [7]. This is evidenced by the equation presented in work by Qiao and colleagues, in which the effective thermal conductivity of air, k', is reduced by decreasing the characteristic length, d (channel diameter), of the system, while keeping temperature, T, and pressure, P, constant [6]

$$k' = k'_0\left(\frac{1}{1+C\left(\frac{T}{Pd}\right)}\right) \tag{1}$$

In this equation, k_0' is the effective thermal conductivity of air at standard conditions and C is a coefficient for air, with $C = 2.5\times10^{-5}$ Pa/m K [6]. This equation leads to Fig. 1, which can be used to visualize when the transitional regime between continuum and molecular transfer modes will occur at varying temperatures and pressures. Although a pronounced Knudsen effect is not expected until characteristic lengths decrease to less than about 100 nm, the transitional region is predicted to begin appearing at dimensions just less than one micrometer in standard temperature and pressure scenarios. At low temperatures and low pressures, such as those encountered in space applications, the thermal conductivity of air decreases due to less molecular kinetic energy and less molecules present, respectively, and the Knudsen effect may be used to further augment insulation capabilities at a given channel diameter. In the present study, a preliminary specimen was created for visualization of a hybrid micro- and nano-channel structure, in which the nano-channels were smaller than 670 nm. Future work will examine the ability of similar nano-channel networks to limit thermal conductivity in the manner described above, but the current work is focused on establishing a thermal insulation baseline with nonwoven micro-channel material, for which 18.2 ± 0.8 μm diameter poly(lactic acid) micro-fibers were used as the template.

2. Experimentation

2.1. Materials

Poly(lactic acid) (PLA) nonwoven microfiber mats with fiber diameters of 18.2 ± 0.8 μm were obtained from Unitika, Ltd. Using a Vacuum Assisted Resin Transfer Molding (VARTM) technique, fiber mats were infused with an epoxy resin, consisting of diglycidyl ether of bisphenol F and 4,4′-diaminodiphenylsulfone curing agent.

2.2. Procedure

Nonwoven mats of PLA microfiber were cut and laid up for a VARTM infusion. To lower the viscosity of the resin