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TITLE THE PHYSICAL PROPERTIES OF MICROCELLULAR COMPOSITE FOAMS

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MASTER

DESCRIPTION OF THE PHYSICAL PROPERTIES OF MICROCELLULAR COMPOSITE FOAMS

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THE PHYSICAL PROPERTIES OF MICROCELLULAR COMPOSITE FOAMS

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ABSTRACT

Recently we reported on a method of preparing microcellular composite foams. In this procedure an open-celled polystyrene foam is prepared by the polymerization of a high-internal-phase water-in-oil emulsion containing styrene, divinylbenzene, surfactant, free-radical initiator and water. After drying, the cells of the polystyrene foam are then filled with other materials such as aerogel or resoles. The physical properties of these materials e.g., surface area, density, thermal conductivity, and compressive strength will be presented.

INTRODUCTION

Physics experiments require very special materials. For inertial confinement fusion experiments, foams have been suggested as ideal material to hold liquid DT fuel uniformly in a fusion target.¹ To do this, the foam must be a low Z (carbon, hydrogen), material with very small cells (< 0.1 micron), low density (< 40 mg/cm³) but handleable. Other experiments require open-celled foams with specific densities that can range from 7 mg/cm³ (five times the density of air) to nearly full density, and range in length from a millimeter to meters. One type of foam that we use extensively is a polystyrene emulsion foam² prepared by a technique introduced by Unilever.³ The foam is prepared from monomers of styrene, divinylbenzene, sorbitan monooleate and free radical initiator which form a water-in-oil emulsion upon mixing that can be polymerized by heating at 60 C. After drying to remove the water, the resulting foam has an open-celled structure with a density that can range from $0.012 - 0.20$ g/cm³ and have cell diameters of $1 - 100$ microns depending on the concentration of monomers, surfactant, initiator and salt (Fig. 1)

Composite foams were prepared to reduce the cell size of the foam matrix by backfilling the cells of the polystyrene foam with materials such as aerogel, or resole.⁴ The preparation of the composite foams have been reported on in detail elsewhere.⁵ The polystyrene foam can be filled completely (Fig. 2,3), or the cell walls can be coated depending upon the concentration of filler. This backfilling method has been extended to other filler materials including PS/DVB⁶ which is used to prepare foams of higher density. In the remainder of this paper we will examine some of the physical properties of the composite materials.

Fig.2

Fig. 3

Figure 2. Composite foams prepared by backfilling polystyrene emulsion foam with (10%) Si aerogel.

Figure 3. Composite foams prepared by backfilling polystyrene emulsion foam with (6%) Resorcinol-Formaldehyde (RF).

RESULTS AND DISCUSSION

Specific surface area measurements were made on a Monosorb (Quantachrome Corporation) single point surface analyzer using 30 mole percent nitrogen in helium. The single point method was used to obtain specific surface areas; therefore, the data (Table I) should be considered as relative values only.

Compression analyses were determined on 0.75 cm right cylinders using a Materials Testing System (MTS) with a 500 lb. load and a platen speed of 0.005 inch/minute. Results are reported in Table II. The composite foams were found to handle much as the polystyrene foam. Compression testing data show the filler increased the density, but in the case of the Si aerogel filler, added nothing to the modulus. Conversely, the resole matrix increased the modulus. These effects are consistent with the poor interaction of the Si aerogel with the polystyrene and the much better interaction of the resole with the polystyrene. Although intrinsically weak, the resole appears to stiffen the walls of the polystyrene foam. This effect is consistent with the results of the compression testing.

Table 1. Specific surface area measurements on foam composites

Sample I.D.	Foam Type	Density (g/cm ³)	Specific Surface Area (m ² /g)	SEM Observations
C-1	PS	0.105	3	empty cells
141-3	PS+(2%)RF	0.116	35	coating on cell walls
141-2	PS+(4%)RF	0.118	27	coating on cell walls
141-1	PS+(6%)RF	0.116	28	filled cells
146-3	PS+(2%)PF	0.121	30	coating on cell walls
146-2	PS+(3%)PF	0.126	14	filled cells
146-1	PS+(7%)PF	0.158	20	filled cells
14-5	PS+(5%)Si aerogel	0.119	127	partially filled cells
14-3	PS+(7.5%) Si aerogel	0.139	138	coating on cell walls
Control	PS	0.100	5	empty cells
118G	PS+(4%)RF	0.149	105	filled cells
92-1	PS+(10%) Si aerogel	0.111	391	filled cells
92-2	PS+(7.5%) Si aerogel	0.090	538	filled cells
92-3	PS+(5%)Si aerogel	0.089	309	partially filled cells
C-2	PS	0.021	9	empty cells
28A	PS+(2%)RF	0.048	252	filled cells
67-1B	PS+(1%)RF (carbonized)	0.032	800	filled cells

Table II. Compression strength on foam composite

Foam Type	Density (g/cm ³)	Yield Strength (psi)	Compression at Yield (psi)	E-mod. (psi)
PS	0.105	174	4.7	5800
PS+(10%)Si aerogel	0.139	183	6.4	5800
PS+(7.5%)Si aerogel	0.126	181	6.4	5900
PS+(5%)Si aerogel	0.106	181	6.4	5900
PS+(2%)RF	0.111	248	6.2	7600
PS+(4%)RF	0.122	294	6.8	9500
PS+(6%)RF	0.131	367	6.4	10600

Since aerogels are known to be good insulating materials,⁹ we have attempted to make thermal conductivity measurements on the composite foams. Thermal diffusivity (α) of the composite samples was measured using a laser flash thermal diffusivity system at the Applied Thermal Physics Laboratory (ATPL), University of Delaware. This system and its associated analytical software is described elsewhere.¹⁰ The samples in the form of discs 1 cm in diameter by 1.5 mm or 2.5 mm thick were used in the ATPL

sides with copper, followed by a thin graphite surface to ensure uniform lateral heat dispersion on both surfaces.

Following energy deposition by the infra-red laser pulse on the front face of the sample, the temperature rise of the second face of the sample was monitored and showed a time dependence distinctly different from that expected of a homogeneous isotropic material. Analysis of the time dependent temperature rise was effected by calculating the theoretical thermal diffusivity obtained from the fractional temperature rise at each 0.1 of the time lapse (t_v ; $V=0.1$ to 0.9) between the pulse and the peak temperature rise of the second face. We note that in many measurements of α only $t_{1/2}$ is reported and these are indeed the values reported in Table III for these composites since we are concerned there only with relative value of α .

We note, however, that for values of V between 0.1 and 0.5 the calculated values of α decreased steadily from approximately three times the values in Table III for $V=0.1$. This initial high measured diffusivity may be due to residual effects of rapid radiant heat transfer through the composite materials since there is no component in the composite which would give such a high initial diffusivity. For values of V from 0.5 to 0.9 the average value of α calculated was typically within 10% of the value at $V=0.5$. This suggests that thermal transport in this time range is more typical of a homogeneous, isotropic material and is presumably due to a combination of skeletal conductivity and thermal transport through the entrapped air. Further analysis and modeling of the transient thermal response of the composite foams is in progress.

Pending further transient analysis we can gain a qualitative understanding of the changes in heat transport mechanism in these composite foams by comparing the $t_{1/2}$ values of α listed in Table III. We assume that radiation contribution to thermal transport are small while the skeletal diffusivity and entrapped air diffusivity contributions are comparable to each other. Using the blank foam value of 0.0018 as a reference we note that only for sample 146-1 is there a reduction of α below the blank foam value. We assume that in this material the phloroglucinol-formaldehyde (PF) foam completely filled the foam thus reducing the cell-size for the entrapped air. The resulting reduction in gas molecule mean-free-path reduced the entrapped air diffusivity while the skeletal diffusivity is not enhanced by the fine-textured added organic aerogel skeleton.

At the other extreme we note that sample 14-5 has the highest α value despite the low density. This suggest that the silica filler (which as a thermal conductivity about ten times that of the polystyrene) has coated the walls densely thus enhancing the skeletal conductivity without inhibiting the air diffusivity since the cell-size remains about the same.

The other two PF aerogels and the three RF aerogels give values equal to, or slightly above, those of the blank foam. This relatively small enhancement could be due to partial filling of the foam cells possibly providing thermally conductive bridges across the cells. This effect may be partially compensated by a reduced gas mfp due to some reduction in cell size. Similar comments apply to the other Si aerogel sample 14-3 though in this case, wall coating give enhanced skeletal diffusivity over the organic fillers.

Table III. Measurement and Analysis of Thermal Diffusivity

Sample I.D.	Foam Type	Density (g/cm ³)	Thermal Diffusivity (cm ² /sec)	Comments
C-1	PS	0.1044	0.0018	Blank foam: provides reference level for interpretations below.
14-3	PS+(7.5%)Si aerogel	0.1386	0.0022	Partial filling, partial coating. @ enhanced by wall-coating but not as much as 14-5.
14-5	PS+(5%)Si aerogel	0.1190	0.0025	Well-coated walls enhance skeletal diffusivity; air diffusivity unaffected.
146-1	PS+(7%)PF	0.1554	0.0014	Best and most complete filling. The reduced cell-size reduces mfp and thus reduces @ below the blank foam value.
146-2	PS+(3%)PF	0.1262	0.0019	Values very close to blank foam suggest that filling not effective in reducing mfp but little enhancement due to wall coating since filler has lower diffusivity than 14-3,5.
141-1	PS+(6%)RF	0.116	0.0022	Enhanced value above blank foam suggests that some enhancement of skeletal diffusivity has taken place despite the low bulk diffusivity of the filler.
141-2	PS+(4%)RF	0.1175	0.0020	Similar to 146 series.
141-3	PS+(2%)RF	0.1159	0.0019	Similar to 141-2.
23A	PS+(2%)RF	0.048	0.0063	

CONCLUSIONS

Although the materials needed for laser fusion targets or other physics experiments are highly specialized, we envision that composite foam technology can be extended to other foam combinations and that novel filters, insulation, catalytic devices and chromatographic materials could be made from these materials. At this time we are continuing to study a way to optimize these materials.

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