

Processing and Compressive Properties of Aerogel/Epoxy Composites

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ABSTRACT

Aerogels are ultra-lightweight materials composed of almost 99.98 vol% of porosity and present excellent opportunities for developing lightweight composite materials. Special properties such as electrical insulation and controlled dielectric constant can be generated in such composites. Ultra-low density and hydrophobic nature of aerogels make it difficult to mix them in epoxy resins. The present research is aimed at developing fabrication methods for aerogel/epoxy composites. The development of such methods requires encapsulating the aerogel particles in epoxy resin without infiltrating them. Compression tests are conducted on these composites and their deformation and fracture behavior is studied. The composites are found to have compressive failure strain of over 25%. It is observed that the higher density composites showed higher compressive modulus but lower yield strength. The reduction in the yield strength is attributed to the early crack initiation in these specimens.

Key words: Aerogel, polymer matrix composite, compression.

1. INTRODUCTION

Aerogels were first developed in 1930s but the interest in these materials was renewed in 1970s when the possibility of storing oxygen and rocket fuel in its porous structure was explored. Currently, these materials are being studied for possibility of using them as thermal and electrical insulators, ballistic armors, and structural materials. The Stardust mission of NASA used aerogel for the purpose of arresting and collecting interstellar and comet dust particles from a comet "Wild 2" [1]. These high speed and high energy particles, traveling at a speed of about six times the speed of a bullet could be trapped in an aerogel. It is also known that silica aerogels are about 40 times more insulator than fiberglass. NASA has used it on the Mars

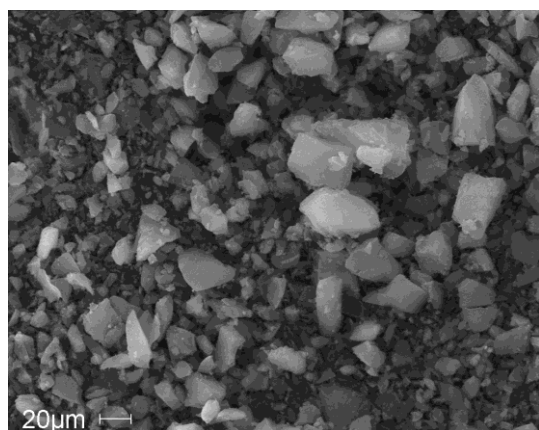
Pathfinder Sojourner rover and Mars Exploration rovers for insulation purposes. Additional applications of aerogels are observed in battery electrodes [2], catalysts [3] and electronic devices [4].

Aerogels are ultra-light weight open-celled mesoporous materials. Their structure is composed of an interconnected network of channels made of thin ligaments. The thickness of ligaments determines the final density and porosity of the aerogel. Currently, silica, alumina and carbon aerogels are available. Possibility of fabricating aerogel/epoxy composites for insulation purposes [5], development of active biosensors using aerogels [6, 7], and development of low dielectric lightweight materials [8] are being studied by various researchers. Aerogel/epoxy composites can provide controlled thermal, electrical and dielectric properties in addition to lightweight and tailored mechanical properties. Clay aerogel/polymer composites can also be found studied by several researchers [9]. However, such efforts are limited in scope and understanding of processing parameters for such composites is still lacking. The present study is focused on developing synthesis methods for aerogel/epoxy resin composites. The fabricated composites are characterized for compressive properties.

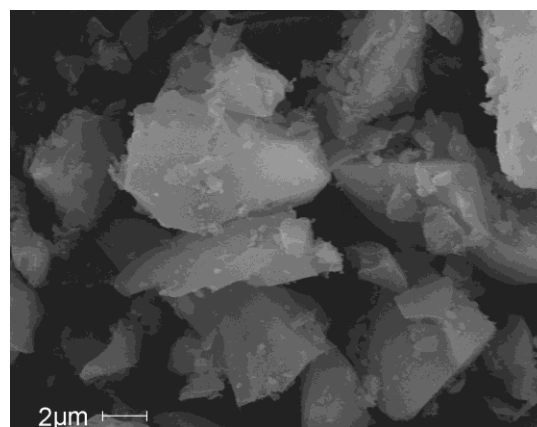
2. MATERIALS AND METHODS

2.1 Constituent Materials

DER 332 epoxy resin and DEH 24 hardener, manufactured by Dow Chemical Company, are used as the matrix material and curing agent, respectively. The resin to hardener volume ratio is maintained at 14:1 as recommended by the manufacturer. Silica aerogel particles supplied by Cabot Corporation are used. These particles have an average pore diameter of about 20 nm and bulk density of about 40 kg/m³ as reported by the manufacturer. Some of the particles are shown in Figure 1.



(a)



(b)

Figure 1. Scanning electron micrographs of aerogel particles.

2.2 Compression Tests

The fabricated specimens of aerogel/epoxy composites are tested for compressive properties using Instron 4467 test system. The test specimens are in the shape of cubes with each dimension of 10 mm. Specimens with similar dimensions have been used in some previous studies on porous materials [10]. Load-displacement data was obtained during the test and used for developing stress-strain relations, and calculating compressive modulus and strength.

3. RESULTS AND DISCUSSION

3.1 Specimen Fabrication

The first step of this study was to develop a fabrication method for aerogel/epoxy composites. The aerogel particles are composed of an interconnected network of channels. Hence, the method of incorporating them in the polymeric resin requires that these particles are encapsulated by the resin without infiltrating them. Stir-mixing is the most widely used method for fabricating particulate composites. This method was considered suitable for synthesizing aerogel composites also. Other techniques such as pressure or vacuum assisted infiltration methods have a disadvantage that the resin may infiltrate in the nanosized pores of aerogel particles. Infiltration of pores is undesirable because it will lead to increase in the density of the composite.

In the initial mixing studies it was observed that the wetting of the surface of aerogel particles by the resin system was poor. The particles were observed to immerse into the vortex generated by the high-shear impeller used in the mechanical mixing process, but emerged again as dry powder. Even prolonged mixing and variation in mixing speed did not provide any favorable results. These mixing experiments were conducted at the room temperature. Only a small volume fraction of aerogel particles (<5 vol%) could be incorporated in the specimens processed by this method. The scanning electron micrograph presented in Figure 2 shows that particle-epoxy bonding is not good and particles are easily separated from the matrix. The surface modification is not an option for making them compatible with epoxy resin because of the presence of open-cell porosity. Modification of aerogel surface to make it compatible with epoxy may lead to enhanced infiltration of pores with the resin.

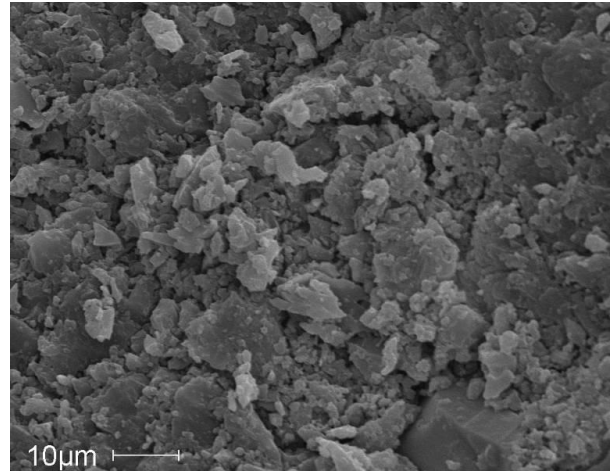
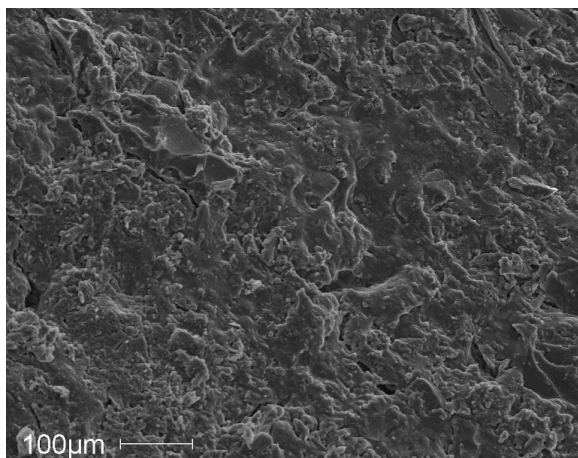
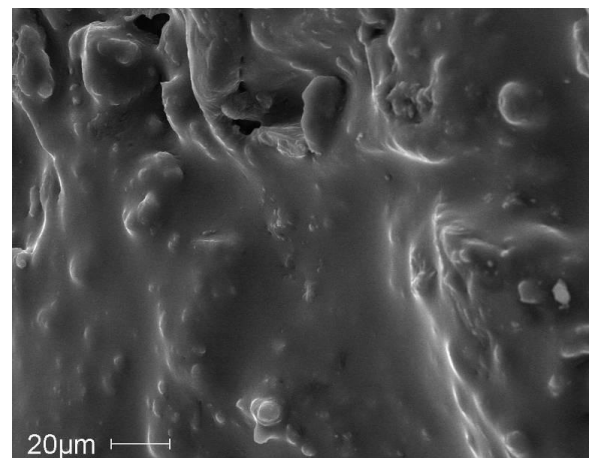


Figure 2. Scanning electron micrograph showing surface of an aerogel-epoxy composite. The bonding between particles and matrix appear to be inadequate.

After mixing aerogel in the resin-solvent mix, the slurry was kept at 50°C overnight for the removal of acetone. It is recommended by the manufacturer that the epoxy resin used as the matrix material should be maintained below this temperature to avoid self polymerization. After removal of acetone the hardener was mixed to initiate curing. The final composite showed considerable amount of porosity and the composite was of very poor quality. The porosity appeared due to the removal of residual amount of acetone during curing. One of the possibilities for this kind of behavior may be that acetone penetrates in the nanoporous structure of aerogel and does not get completely removed in the heating step. Heating time was varied to improve the quality of the final composite, and curing schedule was also altered but results were not satisfactory even after considerable number of experiments. The solution processing method demonstrated that the mixing can be improved by reducing the viscosity of the matrix resin. Hence, in the third set of experiments increase in temperature was adopted as a means of decreasing the viscosity of the resin. In these experiments the resin was heated to 50°C to reduce its viscosity from about 8,000 cps to about 360 cps and then the aerogel particles were mixed in it. Again, mixing was carried out using a high shear impeller in a variable speed mixer.



(a)



(b)

Figure 3. Scanning electron micrographs of an aerogel-epoxy composite fabricated by processing at higher temperature (a) appearance of the surface at lower magnification (b) higher magnification figure showing good wetting of aerogel particles with resin.

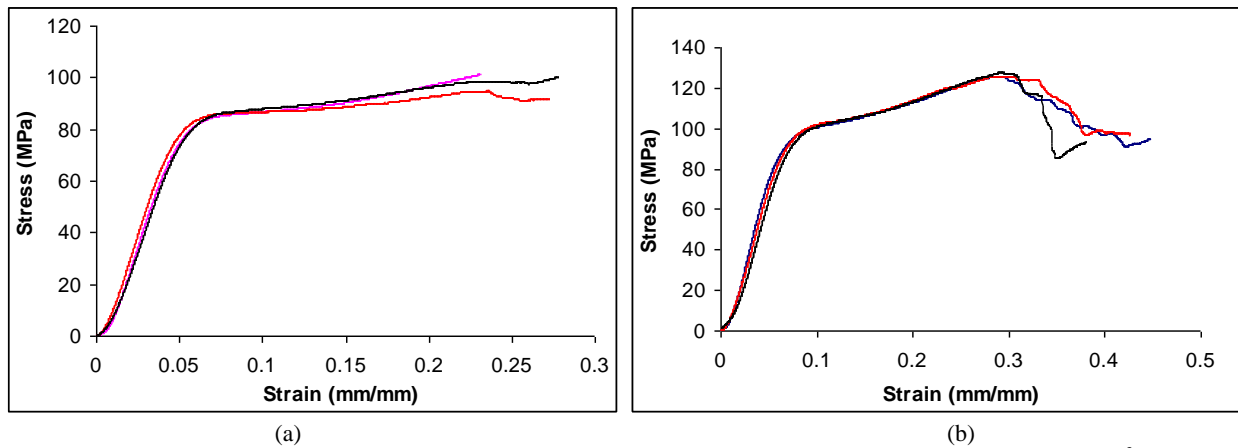


Figure 4. Stress strain curves for aerogel-epoxy composites having densities of (a) 980 and (b) 1070 kg/m³.

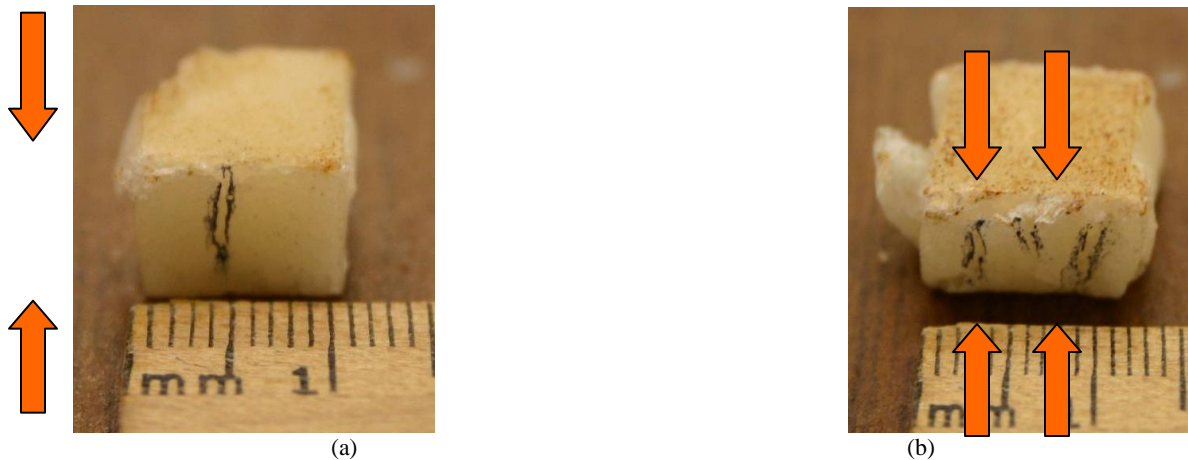


Figure 5. Fractured specimens of (a) lower density and (b) higher density aerogel-epoxy composites.

This set of experiments resulted in engulfment of aerogel particles in the epoxy resin and relatively better quality composites compared to the previous two methods. Microscopic observations taken on the surface of one of these composites are shown in Figure 3. The micrographs presented in Figure 3 show that the aerogel particles were engulfed in the matrix resin and were encapsulated.

Composites containing 20-30% aerogel by volume were fabricated by this method. Compared to the density of resin, which is 1160 kg/m³, the density of composite was found to be lower because of the presence of lighter weight particles. However, some penetration of resin inside the porous structure of aerogel seems to have taken place because the reduction in density is small. The values of densities for three compositions of composites prepared in this study are measured to be 980, 1070 and 1130 kg/m³.

3.2 Compression Testing

Two compositions of aerogel/epoxy composites fabricated in this study were tested for compressive properties. The specimens having the density values of 1130 kg/m³ were not tested because their density is very close to that of the neat resin system. The stress-strain curves for the two types of composites tested in this study are presented in Figure 4. These figures show curves for at least three specimens of each type of composite. A comparison shows that variation in data for various specimens of one type of composites is very small and results are consistent.

An important feature of these curves is that these composites can be compressed to about 25% strain without any loss in strength. The stress-strain curves shown in Figure 4 contain a stress plateau region that indicates that these composites have an ability to absorb compressive damage. These curves are similar to those observed for other porous composites such as syntactic foams, which also show a stress-plateau [11, 12].

Table I. Compressive properties of aerogel-epoxy composites.

Specimen Type	Density (kg/m ³)	Compressive Modulus (MPa)	Compressive yield Strength (MPa)
A	980	1806 ± 62	101 ± 6.2
B	1070	1875 ± 94	83 ± 3.7
Neat Polymer	1160	1813	109.2

Compressive yield strength and modulus presented in Table 1 are calculated using the stress-strain data. The data for the neat polymer is obtained from the material selection sheets provided by the manufacturer [13]. The compressive yield strength is calculated as 0.2% Yield Strength. It is observed that higher density composites showed higher compressive modulus. Similar trend is observed for most cellular materials and composites [14]. However, the strength is found to be lower for the higher density composites. Incorporation of ceramic particles increases the modulus but decreased the strength of the

composite. The load partitioning effects between mesoporous materials such as aerogel and matrix polymer are not well understood at this time. At macroscopic levels, it is observed that higher density specimens show extensive crack initiation in the direction of compression as shown in Figure 5. The direction of compression is marked by arrows in this figure. Cracks are marked with black ink in these figures. Such behavior is consistent with polymeric syntactic foams containing the same matrix resin system [15, 16]. Similar to these observations syntactic foams also show that that cracking becomes more prominent as the specimen modulus increases, especially when the increase in stiffness is due to higher volume fraction on matrix material in such composites.

4. CONCLUSIONS

The outcomes of various synthesis methods adopted for fabricating aerogel/epoxy composites are presented. Success is achieved in fabricating such composites. However, infiltration of nanosized pores present in aerogel particles limits weight reduction achieved by incorporation on ultra-light aerogel particles. Behavior of these composites under compressive loading conditions is similar to other porous composites such as syntactic foams. The compressive stress-strain curves indicate that there is a potential for developing highly damage tolerant ultralight composites using aerogel particles.

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