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**MULTIFUNCTIONAL HYBRID CARBON FOAMS:  
PROCESSING, CHARACTERIZATION AND MODELING  
GRANT NUMBER FA9550-04-1-0323**

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## **INTRODUCTION**

Carbon and ceramic foams are at the center of intensive development for thermal management, thermal protection, filtration, packing, sensor and structural applications [1,2]. The objective of this research is to develop and characterize multi-functional foams with optimal thermal, electrical, magnetic and oxidation resistant properties.

Four broad categories of carbon foams are under investigation. The initial specimens investigated are pitch based carbon foams and are used as a baseline data set. The second category consists of carbon foams coated either with metals or ceramics. The third category is carbon foams infiltrated with fibers with or without surface treatment. Possible surface treatments include metallic or ceramic coatings, pitch or other chemical surface treatments. The fourth category is carbon foams with metal or ceramic particulates.

These specimens are investigated using an integrated "Processing-Characterization-Modeling" approach to enable characterization and quantification of the foam microstructure to measured properties and expand our modeling effort to capture multiple scale results.

## **PROCESSING AND CHARACTERIZING OBSERVATIONS**

The initial samples undertaken are carbon foams with varying mass fractions of carbon fibers. Samples of 1% through 10% mass fractions are processed, however a representative sample of 1%, 5% and 10% fiber content are chosen for further analysis. The samples are cut into 5mm x 5mm x 25mm blocks and then vacuum filled with epoxy to create sample plugs. These plugs can then be polished to examine the 2D microstructure. Example images obtained from the three representative fiber content samples are shown in Figure 1 at 50x magnification. The black circles represent areas where the epoxy did not fully infiltrate the foam.

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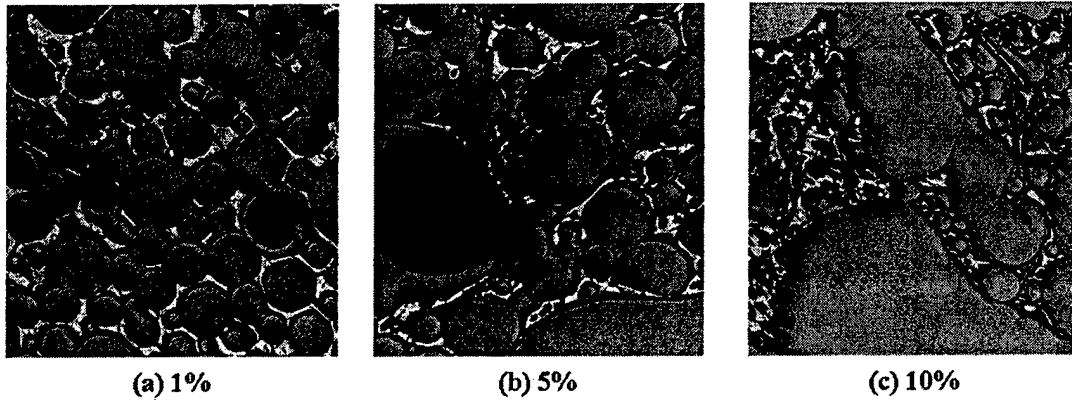


Figure 1. Carbon foam with 1% (a), 5% (b) and 10% (c) fiber mass fractions under OM

The pore distribution in the 1% fiber content carbon foam is the most uniform. As the fiber content increases, the specimen becomes more heterogeneous with a non-uniform distribution of pore sizes. In addition the pores evolve from circular shape into a more elliptical shape. This is likely due to constraints from the presence of fibers on pore growth during processing. The 10% fiber content carbon foam sample illustrates this point well. The fibers are not distributed well and in the high content areas the bubbles are very small, fitting in between the fibers as they can as shown in Figure 1. Another interesting observation is the lack of uniformity on fiber aspect ratio. In some instances, very long fibers are present and in other areas a shorter fiber is seen.

In addition to optical microscopy images, fiber carbon foams were investigated under SEM to determine the location of the fibers with respect to the pore walls. For this study, fractured samples of 1%, 4% and 10% were used. Figure 3 shows the 1% and 10% sample images taken of the overall microstructures. The same observations of limited non uniform pore growth and elliptical pores at the higher fiber mass fraction are seen in this study.

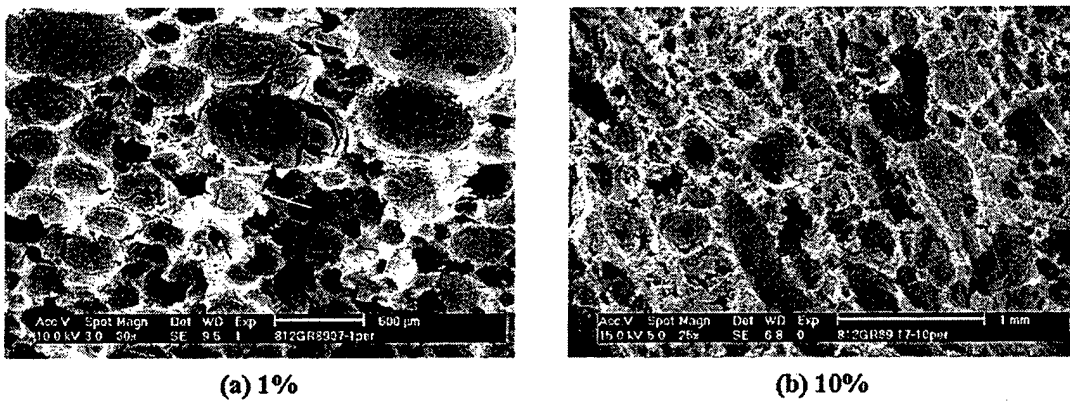


Figure 3. Carbon foam with 1% (a) and 10% (b) fiber fractions under SEM

The SEM pictures reveal that the fibers are not embedded into the pore walls. In general, the fibers are too long and tend to cut across the pores and penetrate the ligaments/nodes. Figure 4a shows a representative example of this behavior occurring in the 4% fiber mass fraction specimen. The final observation from the SEM study is that cracking is prevalent in the carbon foam pore walls. These cracks seem to occur with or without the presence of carbon fibers and in some cases appear to be shrinkage cracks resulting from thermal exposure. Example images obtained are shown in Figure 4b. In addition, Figure 4b depicts an interface problem between the fiber and carbon foam.

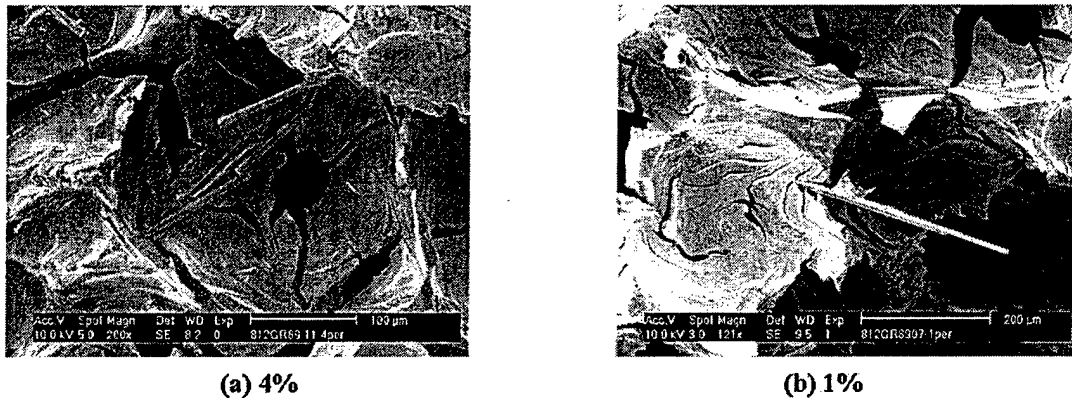


Figure 4. Carbon foam under SEM showing fiber detail

## IMAGING TECHNIQUES TO OBTAIN MICROSTRUCTURE

### *Hand Polishing*

In order to capture slices of the carbon foam microstructure to build into a 3D representation, images of the foams must be obtained at regular intervals. This process is initially completed using the carbon foams consisting of 1, 5 and 10% weight fraction of fibers. The surface is polished to flatten the epoxy plugs and prepare them for imaging. The specimens are then oriented on the microscope stage.

The carbon foam surfaces are captured through 9 individual images at 50x magnification which map the area and are then combined in Photoshop. In addition to these images, higher magnification images are obtained of interesting feature present on this layer. Finally, polarized light images are obtained to characterize the graphitic alignment/orientation of the carbon foams as seen in Figure 6.

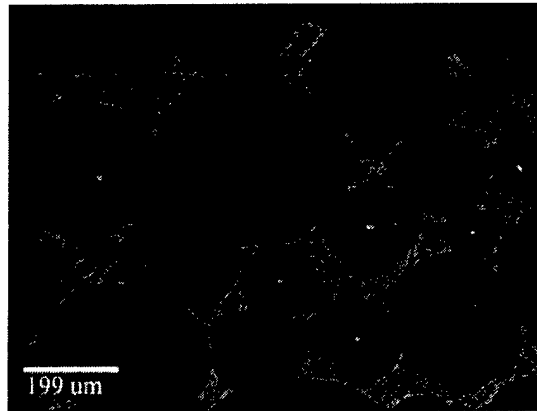


Figure 6. Polarized light image of carbon foam with 1% fiber fraction

The specimens samples are then removed from the stage and measured with dial calipers for specimen thickness/height. The samples are then polished to remove material and the procedure begins again with imaging, followed by measurement.

In general, this technique has proven difficult in that by controlling the polishing parameters, one is not guaranteed a uniform slice thickness from layer to layer. In addition, the dial calipers make the slice thickness measurements suspect and may contribute to the wide variation of slice thickness obtained.

More complication arises in using this method to obtain 2D images which stack into a 3D microstructural description when one considers positioning and alignment of the slice images. The technique described above does not guarantee that the same area can be isolated from the mapped images from layer to layer. In fact, from the slices obtained thus far a noticeable shift is present as the layers progress. This is partially due to bubble orientation; however, the technique used to obtain the stacked images is also questionable.

#### *Automated Polishing (Robo-Met.3D)*

An alternative to this hand polishing technique of obtaining 2D slices of the foam geometry is to use a system known as Robo-Met.3D [3]. This system combines a polishing wheel, clean-up station and microscope with imaging optics. The specimen is moved among the various areas via a robotic arm, thus eliminating human error associated with polishing as well as specimen orientation. Robo-Met.3D is controlled by computer software which is capable of returning to the same position with the optical microscope each time. In addition, the control system allows for multiple overlapping images to be taken in order to map the specimen surface with each layer. The user inputs the various variables and Robo-Met.3D proceeds to obtain 10 slices. The specimen is then removed and measured with micrometers to determine the amount of material removed, or the individual slice thicknesses. This process is repeated to obtain as many slices as required to capture the microstructure of interest. With the flexibility of this system, it is possible to obtain multiple scale representative volume elements from 3 pore

by 3 pore areas to ligament interface scales or at the smallest scale, a fiber interface with consistency.

### FROM 2D IMAGES TO 3D FEA STUDIES

In order to go from 2D slices of the carbon foam specimens to a 3D volume representation, it is necessary to interpolate between the layers. Visualization Tool Kit (VTK) provides a method to transform the 2D slices to a 3D volume [4]. VTK has been used in similar applications such as medical imaging, where 2D images are obtained, spaced along a third axis and then joined.

After the images are read into VTK, those images must be aligned in 3D space with the proper slice thickness spacing. Then a procedure is undertaken to render the image, or fill in the gaps between layers. This rendering can be completed as either a surface rendering as a volume rendering. With either method of rendering, VTK can output a variety of file formats, including stereo lithography files. An example of surface rendering is shown in Figure 7, along with the original 2D image that was extruded.

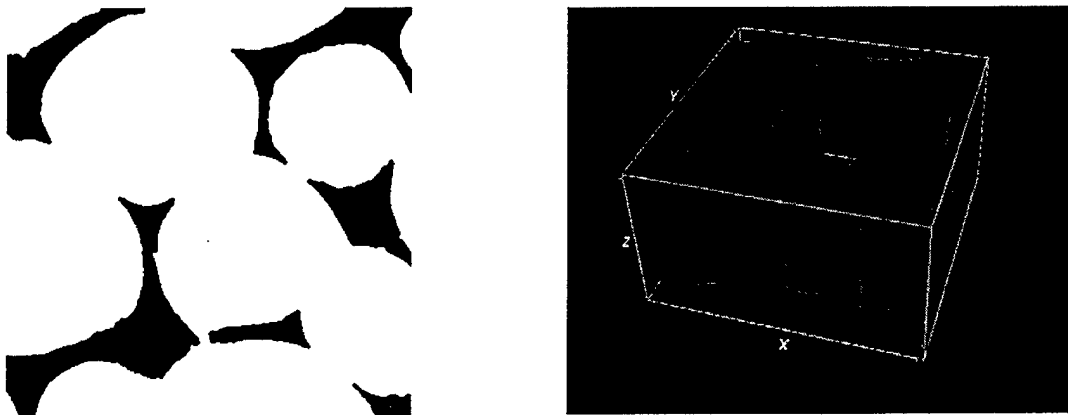


Figure 7. VTK surface rendering of extruded 2D image obtained from RoboMet

This stereo-lithography file is a useful output for importing into finite element programs. For example, the stereo lithography files of the carbon foam 3D microstructure will be imported as geometry into Patran. Following this, Patran can mesh, assign material properties, and load the geometry obtained from Robo-Met.3D.

Initially, mechanical analysis will be conducted to obtain deformation behavior of the microstructure. In addition, thermal and or electrical analysis can be conducted to obtain the respective conductivities. Eventually coupled fields will be applied to the microstructure to predict behavior of the carbon foam, especially the interfacial stresses between the various fibers or coatings and the carbon foam ligaments.

Eventually this work hopes to address materials design applications. The initial models of the 3D carbon foam microstructure can be altered to achieve a new category of material. For instance, the pores can be filled with a metallic material and the analysis above can be repeated to determine what properties would occur if this material existed. In addition, the porosity could be altered to show the effect on mechanical, thermal or electrical properties.

This integrated "Processing-Characterization-Modeling" approach will enable us to characterize and quantify the foam microstructure to measured properties and expand our modeling effort to capture multi-cell behavior to study random cell size and defects. Furthermore homogenization techniques will be enlisted to assess the multifunctional, multi-gradient design of demonstration devices such as sensors and thermal protections systems.

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