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CHARACTERIZATION OF LOW DENSITY CARBON FOAMS BY X-RAY
COMPUTED TOMOGRAPHY (CT) AND ION MICROTOMOGRAPHY (IMT)

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ABSTRACT

Two NDT techniques were used to characterize low-density, microcellular, carbon foams fabricated from a salt replica process. The two techniques are x-ray computed tomography (CT) and ion microtomography (IMT); data are presented on carbon foams that contain high-density regions. The data show that densities which differ by <10% are easily observable for these low density (<100 mg/cm³) materials. The data reveal that the carbon foams produced by this replica process have small density variations; the density being ~30% greater at the outer edges than when compared to the interior of the foam. In addition, the density gradient is found to be rather sharp, that is the density drops-off rapidly from the outer edges to a uniform one in the interior of the foam. This edge build-up in carbon density was explained in terms of polymer concentrating on the foam exterior during drying which immediately followed a polymer infusion processing step. Supporting analytical data from other techniques show the foam material to be >99.9 % carbon.

INTRODUCTION

EG&G Mound Applied Technologies has produced carbon foams from a salt replica process that has been developed at Lawrence Livermore National Laboratory.^[1] The uses of such carbon foams are described elsewhere by Williams et al^[2] and will not be addressed in this paper. In the replica process, salt particles are pressed into a bar and then infused with a polymer. The polymer is then cured and the salt removed. This creates small voids, microcells, within the cured polymer. Finally the cured polymer is carbonized primarily into an amorphous carbon. The carbon foams are light in weight and can be produced to a designed density between 15 and 100 mg/cm³.

In this paper, we are interested in determining the density distribution throughout the light-weight carbon foams. Two NDT techniques are used to measure the density variation; they are x-ray CT (computed tomography) and IMT (ion microtomography). The first measures the distribution of carbon atoms in the foam from x-ray absorption^[3] and the latter determines the electron distribution from ion energy loss due to small angle scattering.^[4]

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EXPERIMENTAL

All of the carbon foams examined in this study are made by the salt replica process.^[1] In order to identify the contaminants and also to determine the purity of the carbon produced by this replica process, the foams are characterized by several analytical techniques. The residual organic materials that might be present after pyrolysis are found by exchanging the foams with several polar and non-polar organic solvents; the extracted solvent is concentrated and then analyzed by gas chromatography/mass spectroscopy (GC/MS). No extractable contaminants, <10 ppm, are detectable. In addition, foams are weighed and burned in O₂, and the weight of the residual ash is determined. The measured weight loss is >99.9%. Most ash, but not all, dissolved in aqua regia. The composition of the acid-soluble ash is determined by inductively coupled plasma (ICP) spectroscopy. The results are given in Table I. They show the major constituents to be Ca and P, and

TABLE I

Inductively Coupled Plasma (ICP) Data (in ppm) on Ashed Samples of Carbon Foams Made From Replica Process

Elemental Concentration	Element										
	<u>Al</u>	<u>Ca</u>	<u>Cr</u>	<u>Cu</u>	<u>Fe</u>	<u>Mg</u>	<u>Mn</u>	<u>Na</u>	<u>Si</u>	<u>Ti</u>	<u>P</u>
	23	385	13	10	95	21	105	10	140	3	168

the total concentration of the impurities to be <1000 ppm. That part of the ash that is not soluble in the strong acid is measured by energy dispersive spectroscopy (EDS) and found to be primarily alumina, which is only very slightly soluble in aqua regia. A JEOL 840 spectrometer is used to record the electron images and the EDS measurements.

The CT studies were performed at the Wright Research Development Center, Materials Laboratory x-ray Computed Tomography facility located at Wright-Patterson AFB, OH. The LAM/DE operates with a 420-kV bremsstrahlung x-ray source and has a spatial resolution of ~0.25 mm. Because of the low-density of these carbon foams, the x-ray source was not filtered and detectors only used a 1.5 mm thick aluminum filter. The LAM/DE machine has two sets of detectors for each line of sight, a thin front detector to monitor low-energy x-ray attenuation and a thick back detector to monitor the higher energy x-ray attenuation. This study used the front detector and utilized a detector preamp setting which assumed low x-ray attenuation through the material. This allowed for accumulation of CT data with high signal to noise ratios while still operating at the 420-kV level with the x-ray source.

The IMT data were collected using a 200 micron square, 8 MeV proton beam at the Sandia Microbeam Analysis Laboratory at Livermore.^[4] The specimens were translated and rotated through the stationary beam, with the median of 100 proton energy loss determinations used to determine each line density (ray) through each specimen. Energy losses, measured with a solid state detector, were converted to density values using tabulated stopping powers for protons in carbon. Approximately 10,000 line density determinations were used for each tomographically reconstructed slice.

RESULTS AND DISCUSSION

Figure 1 illustrates a typical, high purity (>99.9%) carbon foam produced at Mound. A high-magnification secondary electron photomicrograph (SEM), Figure 2, shows the typical microcellular structure within a foam. The carbon manifests itself as the cell walls; the wall thickness is typically 0.1 micron or less. The diameter of the microcells is on the average a few microns and corresponds to the diameter of the salt particles. Some contaminants are dispersed throughout the foam as very small particulates. These can be seen as tiny bright spots in the backscatter electron (BSE) image shown in Figure 3. These spots are examined with EDS and are found to consist of Ca, P, Si, Mn, Fe and S. These impurities are likely present in the original salt and are left behind in the salt extraction process.

Figure 1. Optical Photograph of a Typical Carbon Foam Produced at EG&G Mound.

Figure 2. High-Magnification SEM Photomicrograph Showing Typical Cell Structure in Carbon Foam.

Figure 3. BSE Image Showing Bright Spots That Are Higher Atomic Number Elements Than Carbon Matrix

As stated previously, we are interested in determining the density distribution in these carbon foams. In the past, density distributions have been routinely obtained by radiography. Two typical digitized radiographs are shown in Figures 4a and 4b; the first is of a low-density foam with a bulk density of 38 mg/cm^3 and the second is of a foam with an even lower bulk density of 16 mg/cm^3 . The first radiograph shows a variation in carbon density in the foam, that is Figure 4a shows the density to be higher at the outer edges when compared to the interior. The second radiograph, Figure 4b, shows no obvious high and low density regions. The foam represented in Figure 4a was prepared with the polymer infusion step and the one in Figure 4b was not. Radiography, or digitized radiography, has very good spatial resolution (x and y) but very poor depth of field (z), since the density is summed over a large volume element that extends the length (or width) of a foam. Therefore, it is impossible to quantify where on the edge the density gradient is the highest. Two techniques, CT and IMT, are able to give density information with a volume resolution of ≤ 0.125 and $\leq 0.008 \text{ mm}^3$, respectively. In addition, these two techniques are quantitative. The next paragraphs discuss the CT and IMT results on the same two foams.

Several CT slices are recorded on each specimen; all give similar results, thus, the data given in Figures 5a and 5b can be considered as representative of what would be expected for the 38 mg/cm^3 foam and 16 mg/cm^3 foams. For the former material (Figure 5a), the CT slice shows the "outside" edge to have a higher carbon density than the interior of the foam. This was not the case for the low-density one (Figure 5b) where "no" carbon build-up on the "outside" surfaces could be detected. In fact, all foams that have been examined with CT that have bulk densities $\geq 30 \text{ mg/cm}^3$ exhibit similar high-density "outside" edges. However, no high density edges are detected in foams with bulk densities of $< 20 \text{ mg/cm}^3$.

Relative Density
200
50

255
205
50

255
184
71
163

255
163
92

Figure 4. Digitized Radiographs

Figure 5. Typical CT Slices Illustrating Carbon Foams With:
 (a) Bulk Density of 38 mg/cm³ and (b) Very Low
 Density Foam of 16mg/cm³

From the line scans of the CT slice for the 38 mg/cm³ material (Figure 6a), densities on the periphery are found to be ~30% higher than the densities on the inside of the foam. In fact, the corners show the greatest density of carbon. These results can be explained by examining more closely the polymer infusion step of the replica process. Polymer is dissolved in a solvent. The salt bars are placed in the solution and the polymer diffuses into the center of the bars. After equilibrium, the wet bars are allowed to dry. The solvent evaporation begins on the outside surface of the bar. As the solvent evaporates, more solvent migrates from the interior of the bar to the surface. In doing so, some of the polymer is dragged towards the edges of the foam causing an enrichment of the polymer on the outside surfaces. After curing, salt removal and pyrolysis, the CT images show these high density regions on the edges and the corners. Figure 6b depicts a CT line scan from the very low-density foam. Since this foam does not include a polymer infusion step in its production, a high density edge region would not be expected. The CT data support this finding. From a practical point-of-view, these CT results suggest that machining ~1.5 mm from each side of the 38 mg/cm³ would produce a foam with a reasonably uniform density.

The IMT examinations of the same two specimens discussed above are given in Figures 7a and 7b. Since IMT measures the energy loss of monoenergetic protons that have scattered from the electrons of a material, the line scans are a measure of variations in electron density in the foam. For the energy range used with the CT scans, the amount of x-ray attenuation is primarily due to Compton scattering which in turn is also proportional to electron density. The former is a very highly probable process whereas the latter is not. Although the probabilities of the scattering events are much different between the two techniques, similar conclusions can be drawn from the IMT data, that is a high-density edge is observable for the 38 mg/cm³ foam but is not present in the very low density one. Also the IMT data show the increase in electron density at the edge to be ~30% over that of the interior, the same value extracted from the CT line scans.

SUMMARY

Analytical data from various techniques that were taken on carbon foams produced by the salt replica process are shown to be very pure, >99.9% carbon. The density distributions are determined with two NDT techniques, x-ray CT and IMT; data from both techniques showed carbon enrichment at the edges and corners of a foam with a bulk density of 38 mg/cm³. The density increase at the edges are determined to be ~30% when compared to the bulk density. This density variation is explained in terms of a polymer infusion processing step. A foam that was prepared without the polymer infusion step, which subsequently results in a very low density of only 16 mg/cm³, showed no build-up of carbon on the periphery of the material.

(b)

(a)

Figure 6. CT Line Scans Showing Density Variations in: (a) 38 mg/cm³ and (b) 16 mg/cm³ Carbon Foams.

Figure 7. IMT Line Scans for (a) 38 mg/cm³ and (b) 16 mg/cm³ Carbon Foams.

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