MORPHOLOGICAL CHARACTERIZATION OF MICROCELLULAR CARBON FOAMS

SAND--89-1061C

DE89 014941

J.H. Aubert and A.P. Sylwester
Sandia National Laboratories
Albuquerque, New Mexico 87185

INTRODUCTION Low-density, microcellular carbon foams have been prepared by the high temperature degradation of polyacrylonitrile (PAN) foams in an inert atmosphere [1]. The PAN foams are first prepared by controlled phase separation of PAN solutions followed by solvent removal. Some possible applications for low-density, microcellular carbon foams are: catalyst supports, adsorbents, porous electrodes, high temperature structural insulation, and in the fabrication of inertial confinement fusion targets. To effectively design and use these carbon foams, it is necessary to characterize their morphology. In this paper, we describe two techniques which are well suited to characterize microcellular carbon foam morphologies.

THEORY The phase separation process results in carbon foam morphologies with no real "cellular" character, i.e., no spherical voids. Commonly, an open and strut-like morphology is observed. We have found that one useful measure of the "cell size" is the average spacing between surfaces in the foam, d. In theory, this could be calculated by passing a line through the foam and measuring the distances between intersections of the line and foam. If one did this for all solid angles and determined the average, then the result would be d. A well known steroelogical relationship relates d to the interfacial area per unit volume of the material [2]:

$$d = 4 / S_V \qquad (1)$$

In a typical application of Eq (1), one would measure d from a photograph and calculate S_V. Our approach has been to physically measure S_V, with BET nitrogen adsorption or mercury porosimetry, and then to calculate d.

EXPERIMENTAL BET surface areas were measured with a Quantachrome Monosorb model surface area analyzer, (Quantachrome, Syosset, NY). This technique results in a single value for the surface area, which from Eq (1) results in a single average cell size. We define this as the surface area average cell size, $\langle d \rangle_S$. For foams with a single cell size, we have shown previously that this technique is accurate [2]. For foams with a distribution of cell sizes, such as many of the carbon foams examined, we used mercury porosimetry to obtain information on the cell size distribution.

Mercury porosimetry was performed with a Quantachrome Autoscan-500 Porosimeter. The technique involves forcing mercury into the

---

foam with pressure. The fundamental measurements are the volume of mercury intruded as a function of applied pressure. An energy balance on the process equates the pressure-volume work to the surface work [3]:

$$P\ dV = -\gamma_{lv} \cos\vartheta\ dS, \qquad (2)$$

where γ_{lv} is the liquid-vapor interfacial tension of mercury and ϑ is the wetting angle. A typical intrusion curve is shown in Fig. 1. Rearranging Eq (2) shows that the surface area per unit volume as a function of pressure is directly obtained in the experiment:

$$S_V = dS / dV = -P / \gamma_{lv} \cos\vartheta \qquad (3)$$

Combining Eq (1) and Eq (3) gives the average distance between surfaces, d, as a function of mercury intrusion pressure:

$$d = 4 / S_V = -4 \gamma_{lv} \cos\vartheta / P \qquad (4)$$

Hence, mercury porosimetry is capable of determining the cell size distribution (volume as a function of cell size,d). Amazingly, no morphological assumptions are required to derive Eq (4). A useful average cell size is the volume average, $\langle d \rangle_V$, which weights the large cells very heavily. In Fig. 1, we show the calculated surface area average and volume average cell sizes for one carbon foam along with the experimental intrusion curve. As the scanning electron photomicrographs and the intrusion curve both show, the cell size distribution is bimodal. As expected $\langle d \rangle_V$ is larger than $\langle d \rangle_S$. Their ratio is an alternate to the standard deviation as a measure of the width of the distribution. Although, mercury porosimetry gives more information than just a surface area measurement, artifacts must be removed from the intrusion curve prior to analysis. The artifacts are due to surface defects and bulk compression. While the artifacts are present in the intrusion curve shown in Fig. 1, they were removed from the calculated averages.

CONCLUSIONS BET surface area measurements and mercury porosimetry are both useful techniques to quantitatively characterize open-celled carbon foam morphologies. Neither technique requires any prior morphological assumptions. The ratio of a volume average cell size (obtainable from mercury porosimetry) to a surface area average cell size (obtainable from both BET nitrogen adsorption and mercury porosimetry) is a good measure of the width of the cell size distribution.

[1] A.P. Sylwester, J.H. Aubert, P.B. Rand, C. Arnold, Jr., and R.L. Clough, ACS Polym. Mat. Sci. and Eng., 57, 113 (1987).

[2] J.H. Aubert, J. Cellular Plastics, 24, 132 (1988).

[3] S. Lowell and J.E. Shields, "Powder Surface Area and Porosity," 2nd Ed., Chapman and Hall, NY (1984).

Fig. 1. An example of a mercury intrusion curve for one microcellular carbon foam of density 0.045 g/cm^3. Both the scanning electron photomicrographs and the intrusion curve show the bimodal morphology.