MICROCELLULAR GRAPHITIC FOAM

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Introduction

Structural composites consist of individual fibers bound in a matrix. The fibers are melt spun from a pitch precursor, which give the composite its strength and stiffness along the fiber directions. A microcellular foam containing a network of interconnected struts, that simulate graphite fibers, would create a unique material. Hall and Hager [1] predicted graphitic foams with a density of approximately 0.1 gm/cm^3 to have a modulus of 2000 MPa.

Microcellular, open cell foams are produced from an anisotropic pitch with graphitic planes aligned along the struts. The blown foams undergo the same processing steps as do pitch fibers, which include stabilization, carbonization and graphitization.

Experimental

Materials

The foam precursor is an anisotropic pitch, produced by Mitsubishi Gas Chemical Co., is denoted by the trade name AR Resin. The pitch is created by the catalytic polymerization of naphthalene and is supplied in pellet form. The data supplied by the manufacturer states the softening point of the material is 239°C and it is 100% anisotropic. The glass transition temperature of AR Resin resides in the range of 230°-260°C.

Processing

The pitch pellets were jet milled into 1-3 μm particles and then pressed to create a disk. The pitch disk was then placed into a Parr® pressure vessel, and purged with carbon dioxide gas (CO_2). The reactor was then pressurized with CO_2 and heated to the desired final temperature. Once the disk reached the final temperature, it was held for a determined length of time. The gas was then rapidly vented to the atmosphere. The foamed disk was removed and placed in a forced air oven. Further processing treatments were conducted to transform the pitch foam to a stable carbonaceous foam. Then they were carbonized in a nitrogen environment to 900° C at 1°C/min.

Characterization

Weights and dimensions of the foam samples were taken between each processing step for physical evaluation to give a non-destructive indication of the effects of each processing step. The carbonized foam samples were viewed under an optical microscope and a Scanning Electron Microscope (SEM) to evaluate the pore size and shape as well as porosity. Compression tests were also performed on carbonized foam samples, from which both strength and modulus were calculated.

Design of Experiment

A 2^3 factorial design of experiment matrix was employed to determine how the morphology is effected by each process variable. Conditions for the test matrix were based upon earlier work completed using nitrogen gas to blow foams [2]. However, the test matrix was modified for the CO_2 blown foams, so that they achieved the same final pressures as the nitrogen foams. The initial pressure was varied only to arrive at the final pressure and was not considered in the test matrix.

Results and Discussion

Altering the process conditions produced foams with varying morphologies. The foams contained both small and large open cells. Small celled foams had uniform porosity while large celled foams did not.

Figure 1 is a SEM of a foam that was processed at 250°C, 10.3 MPa and had a hold time of 30 min. The size of the cells were approximately 40-50 μm and were uniformly distributed. The foam in Figure 2 was blown at 270°C, 6.9 MPa and had a hold time of 30 min. The cells were not uniform throughout the foam and were much larger.

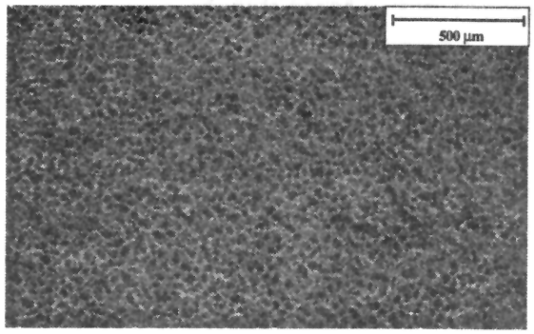

Figure 1. SEM of uniform microcellular foam.

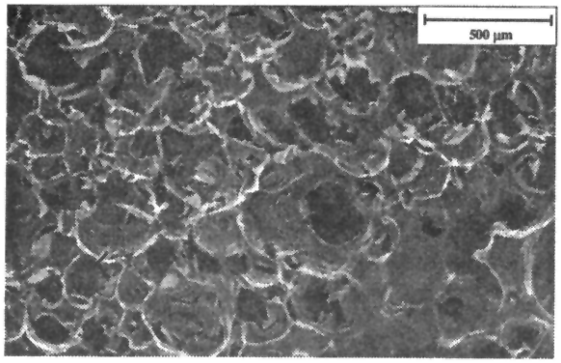

Figure 2. SEM of non uniform large celled foam.

The foams processed at the higher temperature increased in volume by approximately 680% and those processed at the lower temperature increased only about 55% by volume as seen in Figure 3.

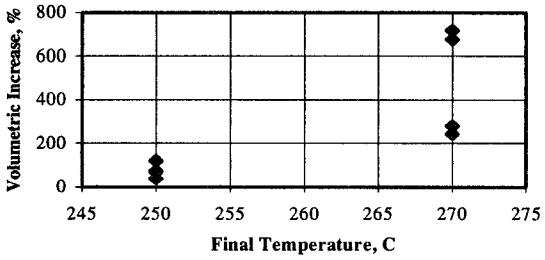

Figure 3. Volumetric change as a function of final temperature.

The mechanical testing showed similar extremes between the two processing temperatures, Figure 4. Foams blown at 270°C had moduli of 4-8 MPa and densities of approximately 0.1 gm/cm^3. At the other extreme, foams blown at 250°C had a moduli of 4000-4900 MPa, with densities ranging from 0.7-1.0 gm/cm^3.

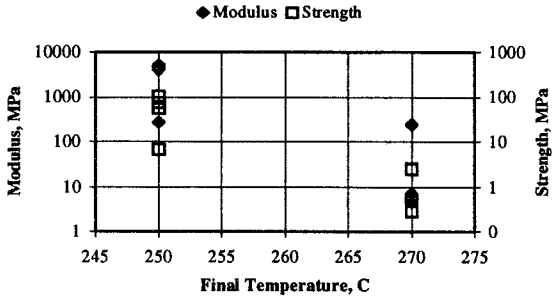

Figure 4. Modulus and strength as a function of final temperature.

Conclusions

The sudden release of pressure causes the gas to expand the pitch and form bubbles or pores. The growth of the bubbles depends on the amount of gas, nucleation density and pitch viscosity. The latter depends on the temperature of the material.

The viscosity is higher at the lower temperature, limiting the expansion of the gas, which suppresses bubble growth. Consequently, the cells are small and the volume expansion is limited. At 270°C, the converse occurs. The gas is able to diffuse out of the material at a much faster rate which enables the pores to grow more freely, producing large cells and giving the foam a high volume expansion.

Foams blown at 270°C had significantly lower mechanical properties as compared to the foams blown at 250°C. This is attributed to the bulk density. The foams processed at the higher temperature also had lower densities. Conversely, those foams processed at the lower temperature approached their solid properties achieving higher bulk densities and mechanical properties.

The results reported here are preliminary and need to be evaluated further. Additional experiments are required before any conclusions can be drawn between the processing conditions and the foam morphology.

References

1. Hall, R.B. and Hager, J.W., *21st Biennial Conference on Carbon Extended Abstracts*, 1993, pp. 100-101.
2. Kearns, K.M., and Anderson, D.P., Microcellular Graphitic Foam Processing, *ICCM*, 1997 (in press).