FRACTURE PROPERTIES OF A FIRE RESISTANCE AND ENERGY ABSORBING CORE MATERIAL

L. C. Russell[1] and K. N. Shivakumar[2]

Summary

A process for syntactic foam made from fly ash, a waste product of coal combustion from thermal power plants, has been developed using phenolic resin binders at low levels. The fly ash consists of hollow ceramic microspheres that are to be treated to remove contaminants. The spheres are bonded together by high char yield binder by a syntactic process. The production process is easily scalable and can be tailored to produce foams of desired properties for specific applications. Complex shaped parts also are possible with appropriate compression mold tooling. Mechanical properties, compression, tension, shear and fracture toughness, have been determined in this preliminary investigation on this syntactic material and are found to be comparable or better than commercially available core materials.

Introduction

Core materials are used extensively throughout the composites industry to fabricate sandwich structures. Sandwich structures offer an order of magnitude increased flexural stiffness compared to the solid laminates. A number of multifunctional core materials are being developed for multifunctional applications [1-10]. Two main functionalities sought are fire resistance for marine structures [8, 11] and energy absorbing materials for armor applications [10]. Major drawback with most of the currently commercially available core materials is that they are not fire resistant or would emit noxious gases when exposed to a fire. Syntactic foams are made by embedding preformed hollow microspheres in a resin matrix. The lightweight hollow microspheres reduce the density of the resin and create a thick mixture that can be applied by hand, sprayed or compression molded in a suitable mold. Syntactic foams are used in many applications, such as, underwater buoyancy aids, aerospace plug manufacturing, structural components for ship hulls and bulkheads, and armor applications [5-10].

North Carolina A&T State University's Composite Materials Research Center has developed [8] a production method for syntactic foam made from a waste product generated by the utility industry. The fly ash, a byproduct of coal combustion already exposed to high temperatures of >1000^{o}C, is in the form of

[1]U.S. Army Research Office, Durham, NC, USA
[2]CCMR, North Carolina A&T State University, Greensboro, NC, USA

hollow glass or ceramic bubbles and is normally collected in the filter bag houses to reduce the particulate emissions from the thermal power plants. Details are provided in references 8 and 11. The objectives of this paper are to evaluate mechanical and fracture toughness of NC A&T SU's core material and compare them with other materials in the literature.

Experimental

The fly ash was obtained from Sphere Services Inc., Cenosphere or Recyclosphere grades CG 100 and SG 300. The binder resin in this study is a phenol-formaldehyde resole resin, Durite SC 1008 from Borden Chemical Co. The fly ash was treated with a silane coupling agent, aminoalkyl triethoxysilane, obtained from either Gelest Company or Aldrich Chemicals. An epoxy resin additive D.E.N. 431 from Dow Chemical Co. and a silicone additive, polydimethyl siloxane diglycidyl ether, obtained from Aldrich were used in a few syntactic foam panels.

The fly ash materials were treated to remove contaminants by a dilute acid (pH ~ 4) wash and the heavier than water fraction of the as-received fly ash was separated and removed by settling. The lighter floating fraction material was further washed with water 3-4 times and was separated by filtration from the water. It was thoroughly dried at 110^{o}C in a convection oven. Subsequently, the treated fly ash was treated with a silane coupling agent, as per instructions from the silane manufacturer. The fly ash after silane treatment was dried in an oven to attain a free-flowing material.

The treated and dried fly ash was admixed with typically the resole resin diluted with suitable solvents in a low-shear planetary motion mixer to uniformly coat the fly ash particles. The volatile solvents from the fly ash mixture were removed while mixing in a stream of warm air. The coated fly ash mix was subsequently placed in a compression mold of 152x152x25 mm or 330x330x25 mm dimensions and was pressed in a laboratory hot press to be finally cured at 162^{o}C. It was found that to achieve reproducibility from sample to sample the void fraction in the foam panels had to be controlled at an as low a value as possible. The foam samples were post cured at a temperature of 177^{o}C. The overall scheme is presented in Figure 1.

Mechanical testing was carried using compression, tension, shear and fracture according to ASTM standards C-365, D-3039/D-3039M, D-5379 and E-399, respectively. The density measurements were carried out on the cored specimens used in compression tests. Tensile, shear and fracture specimens were prepared by machining syntactic foam panels as per test requirements using templates. Figure 2 shows the specimen layout on a 152x152x25 mm panel. The tension test specimens were extracted from 330x330x12.7 mm panel.

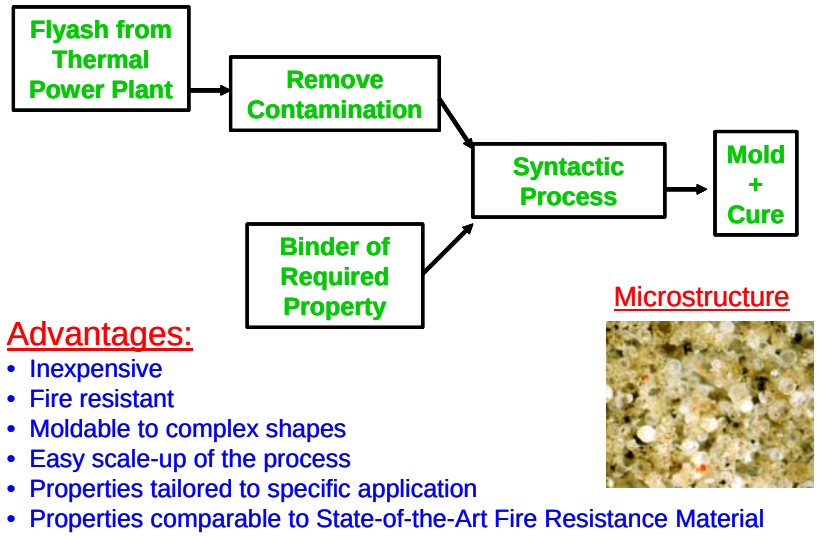


Figure 1 Process flow diagram for producing syntactic foam from fly ash

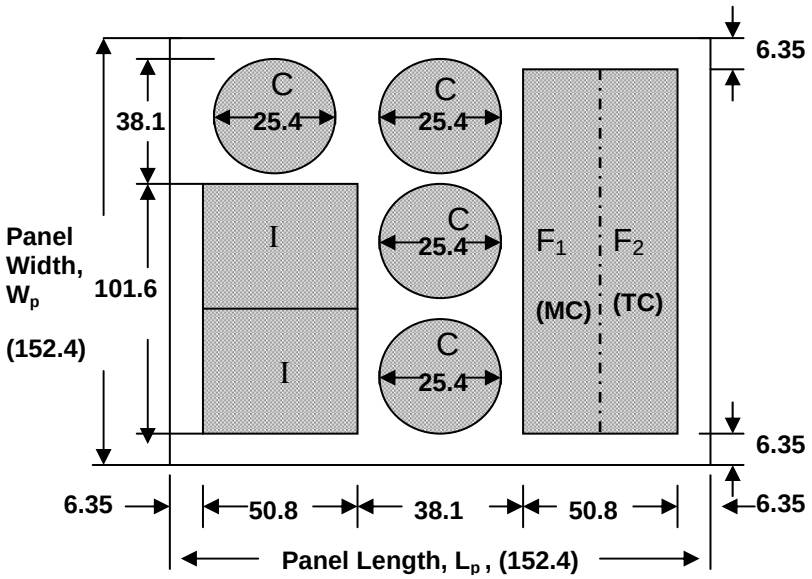


Figure 2 Specimen geometry and layout, millimeters

Results and Discussion

Fly-ash syntactic foam panels one inch (25 mm) in thickness were cored using a diamond coated hole-saw. The cylindrical core specimens were lightly polished to remove any surface irregularities and cleaned to remove the surface debris prior to making dimension and density measurements. Four core samples were taken from each panel (figure 2) and the averages of density and compression strength are determined.

The compression tests were performed according to ASTM C365 on an Instron 4204 electromechanical testing machine. Each cylindrical sample was compressed between two flat platens at a constant displacement rate of 0.51 mm/min while load and displacement were recorded every second. Compressive stress and strain were calculated as load/area and displacement/initial height, respectively.

The variation of compression strength as a function of density is shown in Figure 3. The compression strength appears to be a linear function of density. The density also is a nearly linear function of binder weight percentage [11] and hence the compression strength increases nearly linearly with density. Figure 4 exhibits compression stress-strain behavior for four typical fly ash foam core samples from the same panel. As the peak stress crushes the top layers of the samples the stress appears to decrease somewhat compressing the cenospheres and the resin. The stress becomes constant for further compression and the next layer is crushed. This constant stress-strain response shows the high ductility and/or energy absorbing capability of the material. The core's fracture strain is in excess of 25%. Similar behavior is observed by N. Gupta, et al. with syntactic foams made with epoxy resin and glass hollow microspheres [9]. Compression behavior of the fly ash foam samples changes with changing the binder material.

Tension tests were performed according to ASTM D3039 on samples designed with a dog-bone shape to ensure failure away from the grips. Tests were run on an MTS hydraulic system using mechanical (non-hydraulic) wedge grips and an extensometer for axial strain measurement. A constant displacement rate of 0.51 mm/min was used while recording load and axial strain every half second. Tensile properties for different panels are summarized in Table 1.

The shear tests were performed according to the Iosipescu, ASTM D5379 on an MTS hydraulic system using the standard fixture. Samples were machined to the size and shape required by the standard. A constant displacement rate of 0.25 mm/min was used while recording load and displacement every half second. In some cases shear strain was also measured. Properties are listed in Table 1.

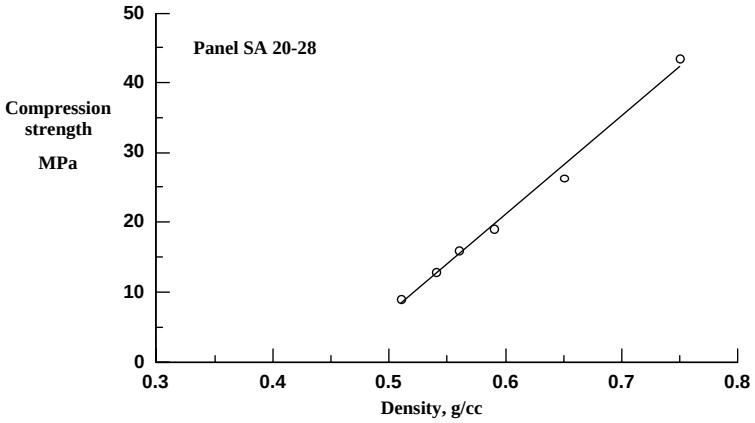


Figure 3 Average compression strength versus density of panels SA20– 28

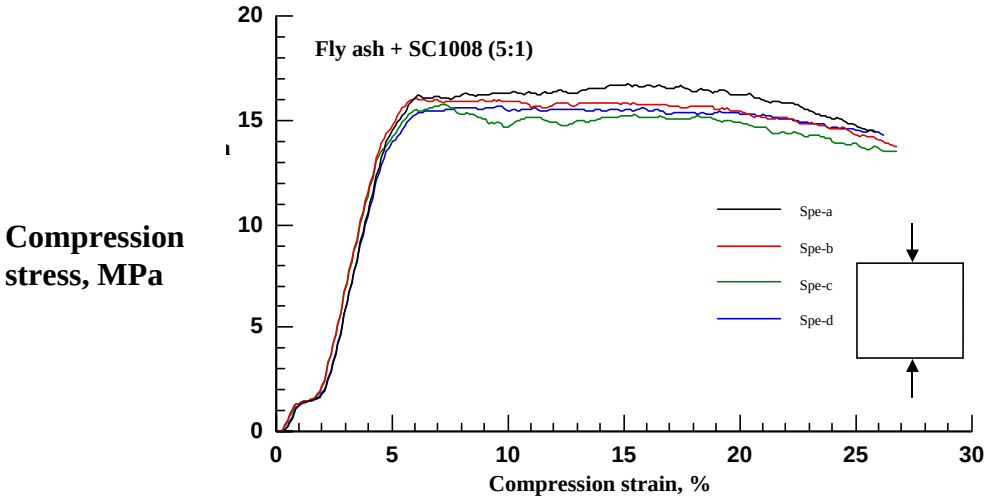


Figure 4 Compression stress versus compression strain.

Table 1. Average Tension and Shear Pro. of Foam (Fly Ash : SC-1008; 5:1)

Batch	Denstiy g/cc	Specimen #	Tension			Iosipescu Shear	
			Strength MPa	Modulus GPa	Poisson's Ratio	Strength MPa	Modulus GPa
1	0.471		**5.82**	**2.30**	0.17	**4.36**	
3	0.472		**6.07**	**2.79**		**4.56**	0.90
4	0.461		**6.81**	**2.52**	0.16	**4.71**	1.10
5	0.479		**6.95**	**2.50**		**5.00**	

* panel size: 330x330x12.7 mm

Fracture toughness tests were performed using single edge notched bend specimen according to ASTM E399. The specimen configurations and loading are shown in Fig. 5 and the specimen dimensions are listed in Table 2. Two types of cracks were considered, namely, through the thickness crack (TC) to measure the average toughness through the thickness and near mid-plane crack (MC) to measure the toughness at the mid-thickness. The crack configurations are shown in Fig. 6. These two specimens will identify any nonumiformity of the material properties. The identical toughness confirms the good quality of the manufacturing process. The crack starter notch of each sample was machined out to a width of 2.3 mm and a depth of about 10.2 mm. There the fatigue crack was made using a sharp razor blade fixture mounted in a vise. This setup ensured that a sawing motion against the end of the starter notch resulted in a fine crack extending from the center of the starter notch. These cracks were cut to about 1.3 mm beyond the machined notch, to a total length to width ratio about 0.45. Crack lengths are listed in Table 2. The tests were done using a 3-point bend fixture as shown in fig. 5 on an MTS hydraulic load frame. Peak load was used in the following equation to calculate the fracture toughness K_{IC}.

$$K_{IC} = (PqS/(BW^{3/2})) * f(a/W) \quad (1)$$

$$f(a/W)=\frac{3(a/W)^{0.5}(1.99-(a/W)(1-a/W)(2.15-3.93(a/W)+2.7(a/W)^2))}{(2(1+2(a/W))(1-a/W)^{1.5})} \quad (2)$$

Table 2. Fracture Toughness of Fly Ash Foam (Fly ash : SC1008; 5:1)

Panel #	Cut	Specimen #	w, mm	B, mm	a, mm	Span, S	Pmax, N	KIC, kPam1/2
LD_B	MC	3	25.6	25.2	11.5	102.5	154.7	354
		4	25.7	25.3	11.5	103.0	164.5	373
LD_P		1	25.7	25.5	11.5	103.0	165.1	370
		4	25.7	25.6	11.8	102.7	167.0	387
		5	25.9	25.6	11.7	103.6	184.1	416
MAT_1		F1	25.0	25.1	12.1	102.2	152.7	410
LD_B	TC	2	25.8	25.3	11.9	103.3	151.7	358
		3	25.6	25.3	12.1	102.6	161.4	391
		4	25.8	25.4	12.1	103.4	176.2	420
LD_P		4	25.7	25.6	11.9	103.0	176.3	408
		5	26.1	25.5	11.8	104.2	179.3	405
MAT_1		F1	25.4	25.1	12.2	102.2	167.9	430

Discussion of Results

Tension and shear data taken on fly ash foam samples are given in Table 1. The fracture toughness data are shown in Table 2. It appears that in batches 1 and 3 there is a greater variation in tensile strength from sample to sample most

probably due to variation in packing density. The batches 4 and 5, the samples appear to be more uniform. Nevertheless overall standard deviations for tensile strength and modulus for all samples are within 15%, which is satisfactory. The shear strength variations from sample to sample appear to be larger and overall behavior is that of a brittle material. The fracture toughness measured using through crack (TC) and middle crack (MC) specimen differed marginally, with MC value always lower. The test foam samples appear to be uniform in both the directions with similar values for fracture toughness. All three panels were made using the binder but by three individuals. Panels LD-B and DL-P were made by students and MAT by a semi- professional. The data scatter was small and confirms the repeatability of the process. Table 3 compares the mechanical and fracture properties of the present foam material with other core materials of comparable properties. Preliminary data generated compares favorably with the other materials.

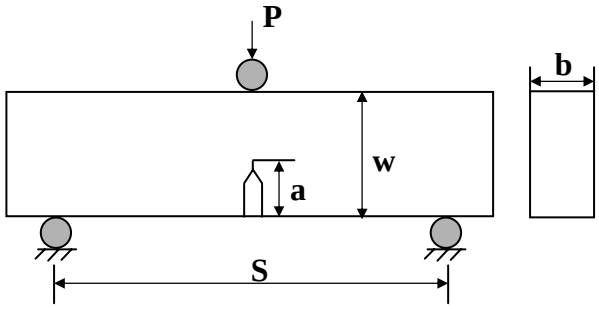
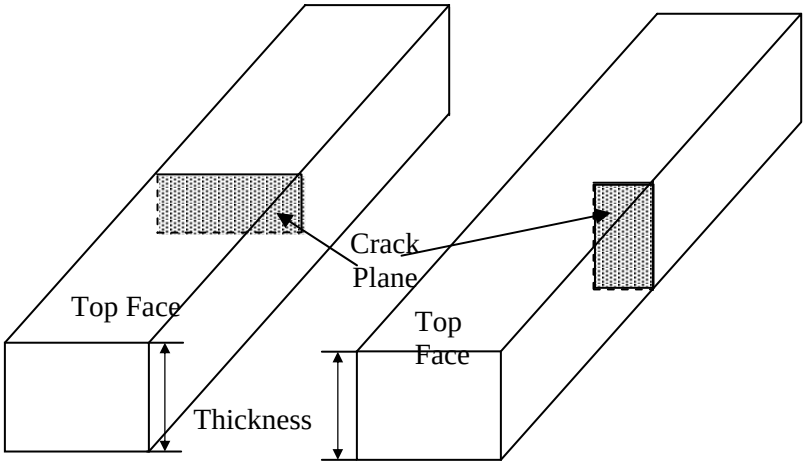


Figure 5 ASTM E399 fracture test setup and configuration

Table 3 Properties of core materials

Property	Balsa	PVC-HD	Albacore	C-Foam	Present
Density, kg/m ^{3}	220	250	200	400	470
Compression					
Strength, MPa	21.9	5.8	3.7	15.0	10.0
Modulus, GPa	6.84	0.36	0.33	0.55	1.00
Tension					
Strength, MPa	20.6	8.7	2.1	3.5	6.5
Modulus, MPa	-	280.0	230.0	550.0	2540.0
Shear					
Strength, MPa	4.5	3.3	2.8	2.1	4.6
Modulus, MPa	237	110	-	-	970
Fracture (G_{IC})					
Toughness, kJ/m ^{2}		0.8			0.05



Mid plane crack (MC) **Through the thickness crack (TC)**

Figure 6 MC and TC crack configurations

Conclusions

1. A process for low-cost syntactic foam made from fly ash, a waste product of coal combustion from thermal power plants, is developed using a resole phenolic resin binder at a low volume percentage of about 6%.
2. The compression, tension, shear and fracture toughness data on the foam samples in this preliminary evaluation indicate that the materials developed are reasonably uniform and compare favorably with other core materials.
3. Fracture toughness of the present core material is small compare to the other core materials like PVC. Further development is needed to improve the toughness.

Acknowledgements

The authors wish to thank S. D. Argade, R. L. Sadler, M. M. Sharpe, L. Dunn, G. Swaminathan of the CCMR at North Carolina A&T State University for their support. The second author acknowledges the support of the Office of Naval Research and Dr. Yapa Rajapakse, for the financial support.

References

1. R. Malloy and J. Hudson, "Foamed Composites", in S. M. Lee, ed, *International Encyclopedia of Composites*, VCH Publishers, New York, **2,** 257 (1990)
2. C. Hiel, D. Dietman, and O. Ishai, Composites, **24**, 447 (1993)
3. F. Shutov, Adv. Polym. Sci. **73**, 63 (1985)
4. R. Deanin, J. Salamone, ed., Polymeric Materials Encyclopedia, vol. 4, p. 2554, CRC Press, New York, NY (1996)
5. N. Gupta, C. Karthikeyan, S. Sankaran and Kishore, Materials Characterization, **43**, 271 (1999)
6. R. Ruhno and B. Sands, in H. Katz and J. Mileski, eds., *Handbook for Fillers in Plastics*, Kluwer Publishers, Chapter 22 , 437 (1987)
7. L. Bardella and F. Genna, *International Journal of Solids and Structures*, **38**, 7235 (2001)
8. K. Shivakumar, R. Sadler, M. Sharpe, and S. Argade, "Fire Resistant Structural Member", US Patent Filed, Serial #: 10/702 063, November, 2003.
9. N. Gupta, E. Woldesenbet and P Mensah, "Composites, Part A: applied science and manufacturing", Elsevier Publishers, in press
10. B. Gama, T. Bogetti, B. Fink, C. Yu, T. Claar, H. Eifert, and J. Gillespie, Composite Structures, **52** (2001) 381- 395.
11. S. Argade, K. Shivakumar, R. Sadler, M. Sharpe, L. Dunn, and G. Swaninathan, SAMPE 2004, May 16- 19, 2004, Long Beach, CA.