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Carbon-carbon composite foams with high specific strength from sucrose and milled carbon fiber

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ABSTRACT

Carbon composite foams prepared by the thermo-foaming of milled carbon fiber dispersions in molten sucrose were evaluated for microstructure and mechanical properties as a function of average fiber length and fiber concentration. The density and specific compressive strength of the carbon composite foams increased with a decrease of average fiber length and reached a maximum at an average fiber length of 33 μm. A maximum increase in compressive strength of 125% and specific compressive strength of 92% was obtained at a carbon fiber concentration of 2 wt.%.

1. Introduction

Carbon foams are new generation materials having applications in thermal protection system, EMI shielding, catalytic support, electrode, host structure for phase change materials, core material for light-weight sandwich composite structure, etc. [1–2]. They are prepared from coal tar pitch, petroleum pitch, synthetic polymers and natural renewable molecules such as tannin and sucrose [1–5]. The coal, petroleum and **phenolic resin** based carbon foams have higher mechanical strength **(up to 64 MPa)** compared to the carbon foams prepared from the naturally renewable precursors **(up to 8.4 MPa)** [1–8]. **Moreover, the carbon foams prepared from sucrose showed compressive strength (3.6 MPa) inferior to that of tannin based carbon foams** [2,3,5,8,9]. Various reinforcements have been attempted for the improvement of mechanical strength of coal and petroleum based carbon foams [10–16]. However, studies on reinforcement in naturally renewable precursor based carbon foams are rarely reported [6]. The present work reports the preparation and characterization of carbon fiber reinforced carbon composite foams from sucrose and milled carbon fiber (CF).

2. Experimental

Sucrose and acetone were analytical reagent grade and procured from Merck (India), Mumbai. Polyacrylonitrile based milled CF was purchased from Zoltex (Germany). The average length and diameter of the CF are 300 and 7.2 μm, respectively (**Fig. S1**). The sucrose-CF mixtures were prepared by planetary ball milling of sucrose and CF in acetone medium using zirconia grinding media. The CF dispersions in molten sucrose were prepared by heating the sucrose-CF mixtures at 170 °C under stirring. The carbon composite foams were prepared by the thermo-foaming of the CF dispersions in molten sucrose at 140 °C followed by dehydration at 200 °C in an air oven and carbonization at 900 °C in an ultrapure argon atmosphere (The flow chart (**Fig. S2**) and other details of carbon foam preparation are given in supplementary information). The CF length in the planetary ball milled sucrose-CF mixture was measured from the images (**Fig. S3**) recorded using an optical microscope (Leica DM2700, Germany). The microstructure of the carbon composite foams was observed using a Scanning Electron Microscope (SEM, FEI Quanta FEG200). The cell size was measured using ImageJ software from the SEM microstructures. The compressive strength of the carbon foams was measured using a universal testing machine (Instron 5050, Instron USA) at a crosshead speed of 0.5 mm/min with 25 mm × 25 mm × 12 mm samples (ASTM standard C365/C365M-05). The maximum stress in the stress-strain graph was taken as the compressive strength. **The XRD analysis of the carbon fiber and carbon composite foams was carried out using X-ray diffractometer (XRD) (PANalytical 3 kW X'pert PRO X-ray diffractometer) with a Cu Kα of 1.5418 Å at 40 kV and 30 mA.**

3. Results and discussion

The average length of CF in the sucrose-CF mixture planetary ball milled for various times is given in Table 1. The average length of the CF decreases from 300 to 15 μm when the ball milling time increases from 0 to 3 h. The CF dispersion in molten sucrose

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obtained by heating the sucrose-CF mixture at 170 °C undergoes slow foaming and setting when heated at 140 °C in an air oven for 96 h to form a solid organic foam [2]. The density and cell size of the carbon composite foams obtained by the dehydration followed by carbonization of the solid organic foams are given in Table 1. The incorporation of 4 wt.% CF of 300 μm length decreases the density of carbon foams from 0.19 to 0.16 g.cm^{-3}. However, a decrease in the average fiber length from 300 to 15 μm by planetary ball milling increases the foam density from 0.16 to 0.23 g.cm^{-3}. On the other hand, the cell size of the carbon foams decreases from 0.67 to 0.43 mm when the CF length decreases from 300 to 15 μm. The low density observed at the CF length of 300 μm is due to the pores produced in the strut region as a result of bridging of longer fibers [13]. Fig. 1 shows the SEM photomicrographs of carbon foams prepared from sucrose-CF mixtures containing 4 wt.% CF of various lengths. The carbon foam without CF contain spherical cells with well-defined dense struts whereas the cells in carbon composite foam prepared at CF length of 300 μm show large distortion from spherical shape. Further, the struts in the carbon composite foam prepared at a CF length of 300 μm are not well defined and contain a large number of pores due to the fiber agglomeration by bridging of longer CF. In addition, a large number of the CF are protruding out from the cell walls. On the other hand, the carbon composite foams prepared from sucrose-CF mixtures containing CF of average length lower than 70 μm show spherical cells with well-defined dense struts. That is, CF with average length less than 70 μm disperses in the molten sucrose without much fiber bridging.

Fig. 2**a** shows the stress-strain graphs of carbon composite foams at various CF lengths at a CF concentration of 4 wt.%. The stress-strain curves show a typical behavior of the elastic, rigid, solid foam with an initial linear elastic region followed by a stress maximum and finally a densification region [11]. The compressive strength of the composite foams increases from 1.23 to 3.11 MPa when the average fiber length decreases from 300 to 33 μm. A further decrease in the fiber length decreases the compressive strength. This indicates that a maximum reinforcement, due to the effective matrix to fiber stress transfer, is achieved at a fiber length of 33 μm. The effect of fiber length on compressive strength of the carbon composite foams prepared at a CF concentration of 4 wt.% is shown in Fig. 2**b**. It is worthy to note that the maximum compressive strength is obtained at a CF length much lower than the strut thickness (Table 1**).** The fiber length lower than the strut thickness enables the CF to uniformly disperse and orient in all possible directions without fiber bridging and protruding out from the cell wall surface. However, at a length lower than 33 μm the CF acts more like particles because the aspect ratio is only in the range of two to three. This decreases the effective stress transfer which results in a decrease in compressive strength of the carbon composite foams. **The XRD (Fig. S4) analysis of the carbon foams with and without CF showed broad reflections at 2θ values of 25.7 and 43.8° corresponding to (0 0 2) and (1 0 1) peaks, respectively, of turbostratic graphite structure. There is no indication of crystallization of carbon matrix induced by the CF.**

The effect of CF concentration on the density and cell size of the carbon composite foams prepared at the optimum CF length of 33 μm is shown in Fig. 3**a**. The density of the carbon composite foams increases from 0.19 to 0.22 g.cm^{-3} and cell size decreases from 0.89 to 0.38 **mm** when the fiber concentration increases from 0 to 2 wt.%. A further increase in CF concentration does not make much change in the density and cell size. The effect of CF concentration on the compressive strength and specific strength of the

Table 1
Effect of planetary ball milling time on CF length and carbon foam properties (CF concentration 4 wt.% of sucrose).

Milling time (h)	0	0.5	1.5	2.5	3
Average CF length (μm)	300	70	33	20	15
Density (g.cm^{-3})	0.169	0.196	0.219	0.223	0.232
Cell size (mm)	0.67 ± 0.11	0.52 ± 0.08	0.38 ± 0.05	0.45 ± 0.04	0.43 ± 0.06
Strut thickness (μm)	60	81	82	87	95

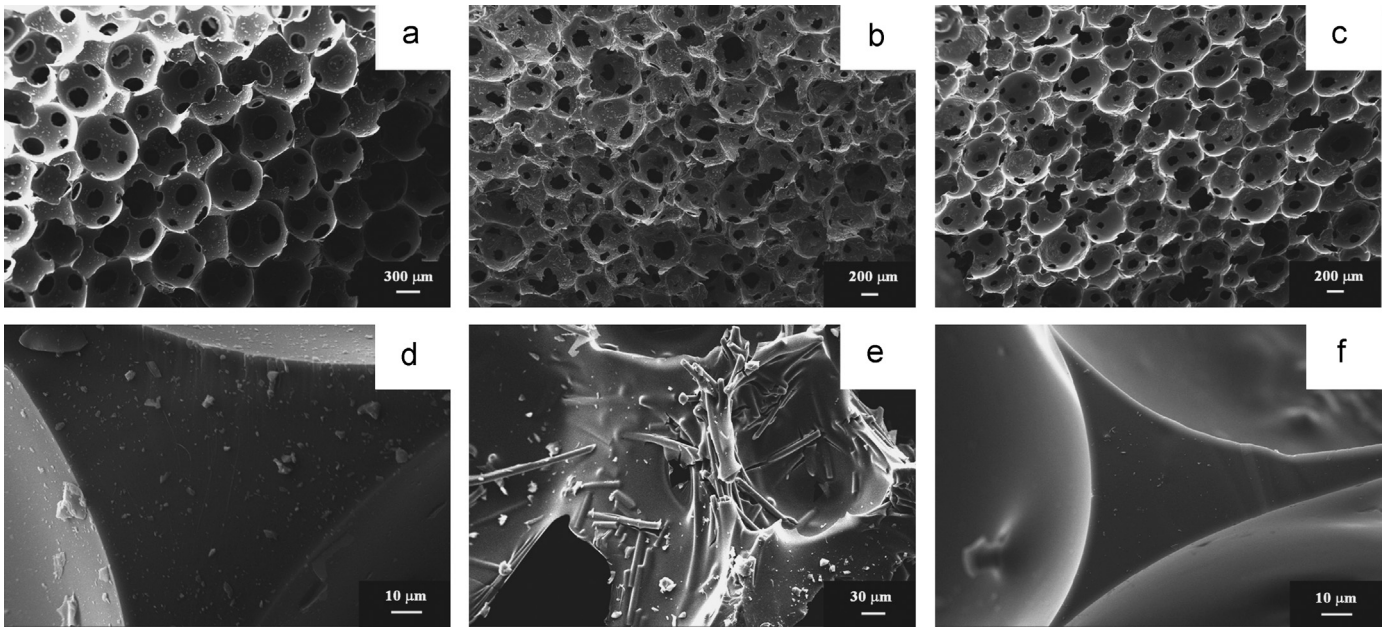

Fig. 1. SEM microstructure of the carbon foams (a and d) without CF, (b and e) 4 wt.% 300 μm CF and (c and f) 4 wt.% 33 μm CF.