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Compressive behavior of fly ash based 3D printed syntactic foam composite

Balu Patil [a], B.R. Bharath Kumar [b], Mrityunjay Doddamani [a,*]

[a] Advanced Manufacturing Laboratory, Department of Mechanical Engineering, National Institute of Technology Karnataka, Surathkal, India
[b] Department of Mechanical Engineering, Jain College of Engineering and Technology, Hubballi, India

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ABSTRACT

Syntactic foams are widely used in damage tolerance and low-density applications. In present work compressive behavior of 3D printed three-phase syntactic foams under quasi-static strain rates (0.001, 0.01 and 0.1 s^{-1}) are investigated. Extruded filaments of High density polyethylene (HDPE) with environmentally pollutant fly ash cenospheres (0, 20, 40 and 60 vol%) are used for 3D printing. Micrography reveal that syntactic foam filament and 3D printed samples are three phase systems comprising matrix, cenosphere and porosity. Matrix porosity of about 7% makes these foams lightweight and suitable for buoyant applications. The compressive properties are extracted from the stress-strain plots. It is observed that modulus and specific modulus increases with strain rate and cenosphere content. Specific compressive strength increases with strain rate and decrease with cenosphere content.

1. Introduction

Syntactic foams are lightweight composites synthesized by dispersing hollow microspheres in neat resin exhibiting higher damage tolerance and energy absorbing capabilities [1]. Their properties depend on constituent materials, particle volume fraction and wall thickness. Existing applications of syntactic foams include ballistic and blast resistant armors, marine structures, buoyancy modules in deep-sea vehicles, undersea pipe insulation, automobile and spacecraft components. Thermoplastic syntactic foams are processed using extrusion [2], injection molding [3] and compression molding techniques [4]. Additive manufacturing (AM) may provide substantial leverage especially when complex shaped lightweight parts are required in short time intervals [5]. Over the past decade AM has spread across aerospace, medical, automotive, architecture, education, and fashion industries [6,7]. It provides no restriction on part complexity, zero tool cost and reduced development cycle time [8]. Fused Filament Fabrication (FFF) method in AM allows quick development of functional parts using solid thermoplastic filaments [9]. The range of commercially available filaments for 3D printers are limited, although wide range of composite filaments have been successfully developed [10]. In the authors recent work, filament with 40 vol% of cenospheres embedded in HDPE is developed successfully for printing [2,11]. Infusion of hollow particles into resin lowers overall composite density and quantity of expensive neat resin in addition to the improved specific properties [4]. Achieving better compressive properties with lower density is possible with porosity into the foam sample. Syntactic foam density reduction through infusion of microballoon is limited by volume fraction and number of survived particles [12]. In addition, voids formed during processing will further reduce density. Three-phase foams are very useful in submarine and underwater applications where buoyancy can be achieved using low density foams [13]. Present study investigates quasi-static compressive behavior of 3D printed three-phase syntactic foams that find application in sub 4000 m range buoyancy modules. Emphasis of this investigation is to provide wide material choices for commercial 3D printers without any hardware modifications in printing lightweight foams.

2. Experimental

HDPE (HD50MA180) having 20 g/10 min MFI and 950 kg/m^3 density is procured from Reliance Polymers, Mumbai. Cenosphere of CIL-150 grade (920 kg/m^3) obtained from Cenosphere India Pvt. Ltd., Kolkata is used as hollow fillers (as received) and sieved to 75 µm [14]. Homogenous blend of HDPE/cenosphere having different cenosphere volume fraction (20, 40 and 60%) is prepared using optimized blending parameters (165 °C, 10 min) in brabender [3] to be filament extruded using single screw extruder. Average die and barrel temperatures are maintained at 150 and 160 °C

* Corresponding author.
E-mail address: mrdoddamani@nitk.edu.in (M. Doddamani).

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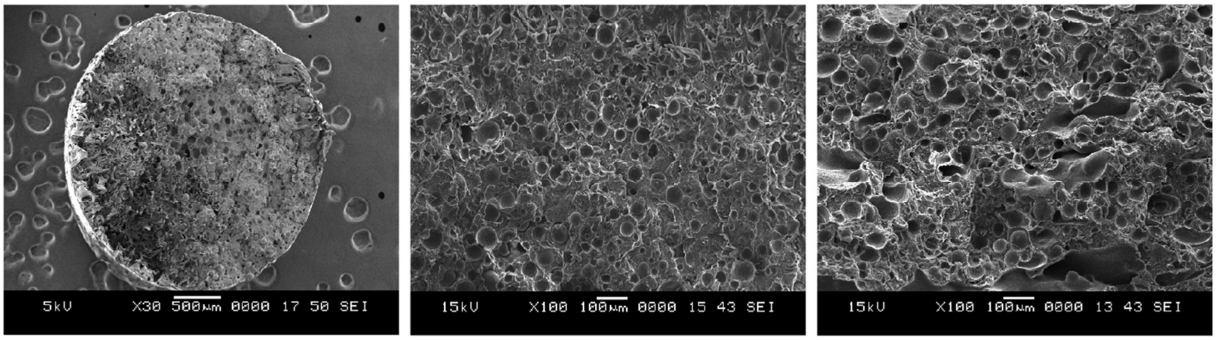


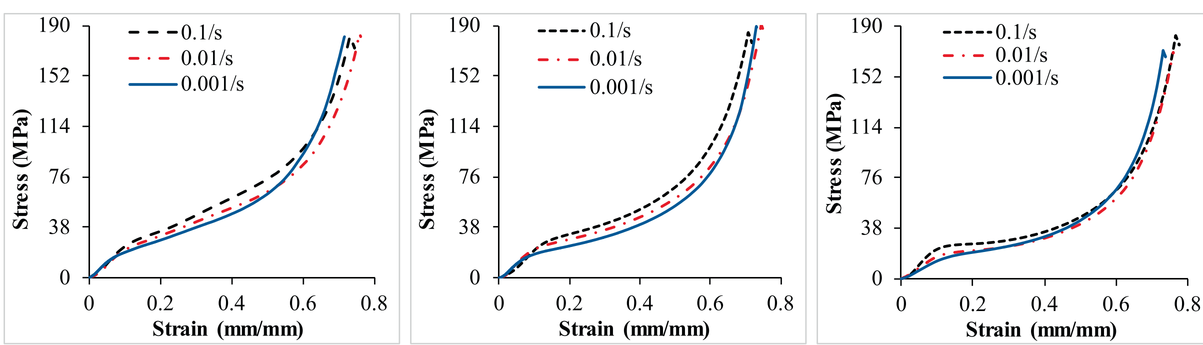
(a) (b) (c)

Fig. 1. Micrograph of freeze fractured (a) extruded syntactic foam filament showing circular cross-section of representative H20 filament (b) 3D printed H20 and (c) H60 sample.

with screw and take-up unit speed of 25 and 12.5 rpm respectively to extrude filament of 2.85 ± 0.05 mm diameter. Samples are codes as HXX (H – HDPE, XX – cenosphere content). Neat HDPE and syntactic foam specimens are printed to 10 × 10 × 3 mm [12] dimension with 100% infill (ensures translation of filament porosity to the printed part without forming additional pores between the deposition layers) and 0.35 mm layer thickness using STAR 3D printer. Nozzle temperature, printing speed and bed temperature are maintained constant at 250 °C, 27 mm/s and 70 °C respectively. Quasi-static compression test is conducted with load cell capacity of 20 kN at 0.001, 0.01 and 0.1 s^{-1} strain rates using Z020 Zwick Roell UTM. Average values of minimum five samples are reported.

3. Results and discussion

Measured densities (ASTM D792-13) of HDPE, H20, H40 and H60 filament are 949.32 ± 0.049, 940.73 ± 0.041, 897.41 ± 0.049 and 886.28 ± 0.051 kg/m^3 and are respectively lower by 0.071, 0.34, 4.32 and 4.90% compared to the theoretical densities (rule of mixture) of 950, 944, 938 and 932 kg/m^3 signifying entrapped porosity during extrusion. Fig. 1 presents circular cross section of H20 filament post freeze fracture. Matrix porosity clearly indicates three-phase structure in the extruded filament. Such a structure might help in additional cushioning effect during compression and might enhance damping. Cenospheres are seen to be intact post extrusion



(a) (b) (c)

Fig. 2. Representative stress-strain plots for (a) HDPE (b) H20 and (c) H60.

Table 1
Quasi-static compression data of 3D printed HDPE and their foams.

Material	Strain rate (s^{-1})	Modulus (MPa)	Yield strength (MPa)	Yield strain (%)	Energy absorbed at 40% strain (MJ/m^3)	Densification stress (MPa)	Densification strain (%)	Specific modulus (MPa/kg/m^3)	Specific yield strength (MPa/kg/m^3) × 10^{-3}
H	0.001	239.84 ± 10.32	16.79 ± 0.87	8.53 ± 0.11	13.09 ± 0.76	–	–	0.253	17.69
	0.01	239.36 ± 10.28	23.31 ± 1.03	12.01 ± 0.18	13.09 ± 0.87	–	–	0.252	24.84
	0.1	271.32 ± 11.47	27.45 ± 1.14	12.16 ± 0.25	13.28 ± 0.94	–	–	0.286	28.93
H20	0.001	151.86 ± 6.13	15.34 ± 0.79	7.95 ± 0.12	11.96 ± 0.65	68.45 ± 2.14	54.45 ± 1.44	0.161	16.34
	0.01	187.56 ± 8.61	21.86 ± 1.14	10.13 ± 0.11	12.13 ± 0.64	70.42 ± 2.41	56.43 ± 1.07	0.200	23.29
	0.1	208.81 ± 10.02	24.54 ± 1.65	11.69 ± 0.14	12.21 ± 0.45	76.87 ± 2.73	58.27 ± 1.95	0.222	26.15
H40	0.001	214.15 ± 10.41	11.09 ± 0.88	5.76 ± 0.07	12.22 ± 0.45	58.42 ± 2.08	55.43 ± 1.57	0.240	13.34
	0.01	219.03 ± 11.68	19.85 ± 1.02	10.01 ± 0.08	12.23 ± 0.59	72.31 ± 3.07	58.41 ± 1.74	0.245	22.25
	0.1	241.08 ± 11.17	22.98 ± 1.44	10.47 ± 0.16	12.28 ± 0.56	72.39 ± 3.18	58.42 ± 1.45	0.270	25.76
H60	0.001	222.94 ± 11.26	14.58 ± 0.98	10.12 ± 0.06	13.05 ± 0.38	52.58 ± 1.09	52.04 ± 1.87	0.255	16.71
	0.01	239.34 ± 21.15	19.35 ± 1.05	9.57 ± 0.11	13.23 ± 0.55	48.42 ± 2.45	48.12 ± 1.44	0.274	22.18
	0.1	242.54 ± 25.56	22.6 ± 1.12	10.12 ± 0.12	13.35 ± 0.22	52.58 ± 2.11	52.40 ± 2.25	0.278	25.91

and are uniformly distributed in HDPE as observed from Fig. 1a and is also confirmed earlier through micro CT scans [2]. Subsequently these filaments are used for 3D printing. 3D printed HDPE, H20, H40 and H60 have densities of 948.93 ± 0.012, 938.32 ± 0.008, 892.14 ± 0.017 and 872.11 ± 0.016 kg/m^3 respectively. Measured values of printed specimens are 0.04, 0.26, 0.59 and 1.6% lower compared to filament due raster gaps. Closer density values of 3D printed samples as compared to their respective filaments indicate the porosity survival post 3D printing (Fig. 1b-c). Rheology of material flow gets affected by higher filler content resulting in elongated pores (Fig. 1c) as compared to more circular pores in H20 (Fig. 1b). Elongated pores are formed for H60 resulting in lower densification strain as these pores under test conditions collapse much earlier as compared to more uniform circular pores. 3D printed foams follow similar trend in quasi-static compressive mode (Fig. 2) as reported in fully dense two-phase foams [12,15]. Modulus of neat HDPE is higher for all strain rates as compared to foams (Table 1) which is due to HDPE's viscoelastic behavior [15]. H60 registered highest modulus at all strain rates among foams. With increasing filler content, stress plateau region becomes distinguishable signifying lower strain hardening resulting in higher energy absorbing capabilities. Yield strength of neat HDPE is comparable with all foam composition at all strain rates indicating potential of complex 3D printed parts to be replaced with compression and injection molded components in marine systems. Specific yield strength and yield strain values confirms that 3D printed foams may lower the overall structural weight by further increasing porosity at lower filler loadings. During compression, initial densification is initiated by matrix porosities collapse (Fig. 3a, c and e). As stress level rises, cenospheres start to break resulting into further densification. At higher magnification (Fig. 3b, d and f) deformed resin, intact cenospheres and debris are visible at 0.1 s^{-1} strain rate. Since all the tests are

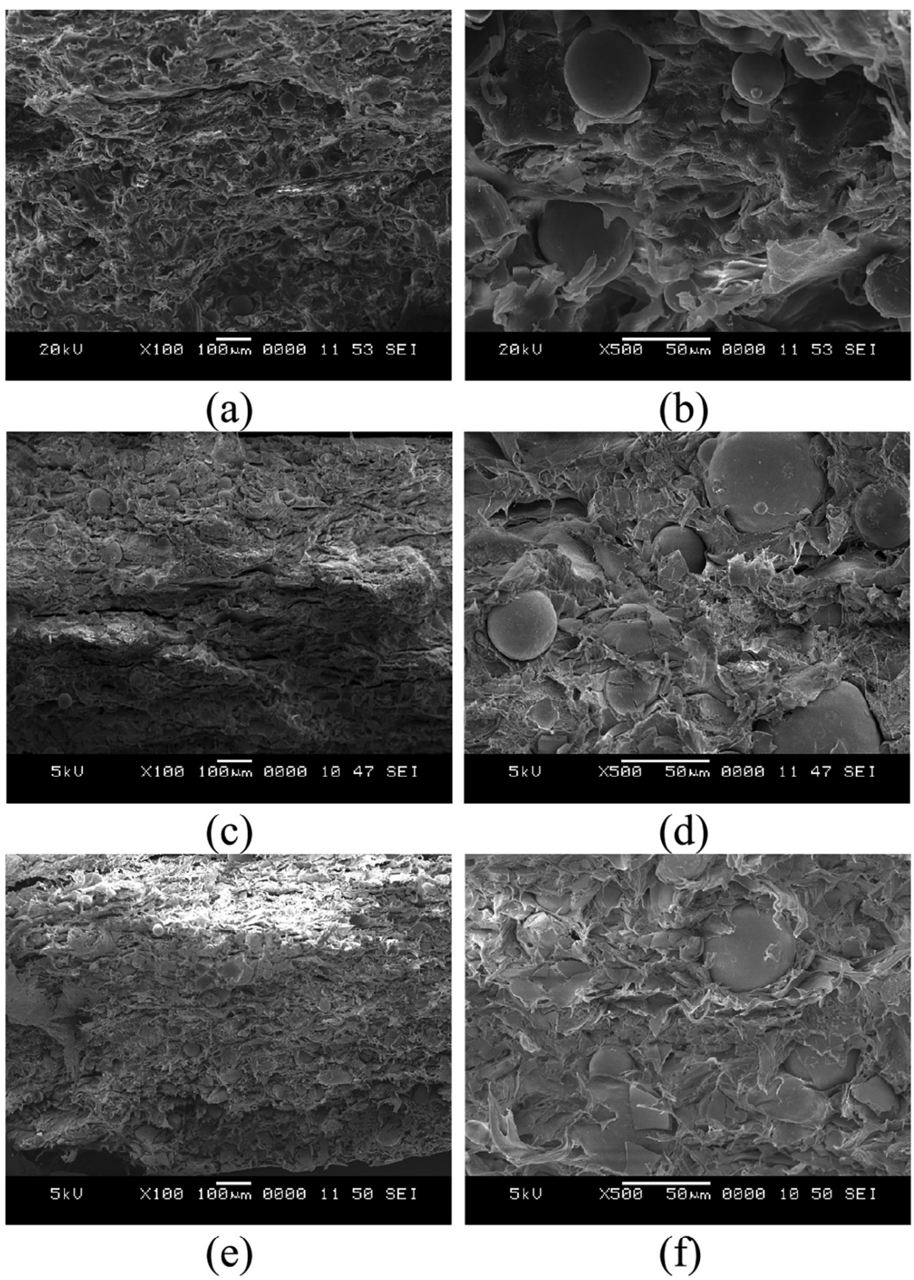


Fig. 3. SEM of compression tested samples at 0.1 s^{-1} for H20 (a-b), H40 (c-d) and H60 (e-f) at lower and higher magnifications.

conducted within quasi-static strain range, micrographs have no much difference in appearance of fractured surfaces with respect to strain rate. Stress-strain curve shows similar level of strains in all the specimens at all strain rates when test is stopped making deformation and densification features appear similar. 3D printed foams exhibit good strain rate sensitivity and is of great interest in developing complex lightweight materials based on the applications.

4. Conclusions

Environmental pollutant fly ash cenosphere based three-phase filaments are developed to be used in commercial 3D printers for potential lightweight applications. These foams are printed and studied for quasi-static compressive behavior. Entrapped porosity in the filaments is retained in 3D printed samples lowering the density. At 100% infill and H60 3D printed samples are 6.43% lighter than the theoretical density. Yield strength of neat HDPE is comparable to foams indicating 3D printing potential over expensive injection and compression molding for complex geometries. Highest specific compressive modulus and yield strength is observed for H60 and H20 respectively at 0.1 s^{-1} among foams. 3D printed syntactic foams show good strain rate sensitivity. Developing complex geometries with 3D printed three-phase syntactic foams make them potential candidate materials for buoyant weight sensitive structures.

Declaration of Competing Interest

None.

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