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SYNTACTIC AND COMPOSITE FOAMS

Radius ratio effect on high-strain rate properties of syntactic foam composites

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Abstract The high-strain rate compressive properties of syntactic foams are characterized in this study. This study is performed using a pulse-shaped Split-Hopkinson Pressure Bar technique. Nine different types of syntactic foams are fabricated with the same matrix resin system but three different size microballoons and three different microballoon volume fractions. The microballoons have the same outer radius of 40 μm, but different internal radii leading to a difference in their densities. The volume fractions of the microballoons in the syntactic foams are maintained at 0.1, 0.3, and 0.6. Analysis is carried out on the effect of the microballoon radius ratio at each volume fraction on the high-strain rate properties. This approach is helpful in separating and categorizing the contribution of matrix and microballoons to the dynamic compressive properties of syntactic foams. The results at high-strain rates are compared to quasi-static strain rate compressive properties of the same material. The results show that there is little or no significant change in both compressive strength and modulus of syntactic foams at all radius ratios when tested at strain rates of 400–500/s compared to quasi-static rates. However, higher dynamic strength and stiffness values are obtained consistently at all radius ratios when tested at 800–1000/s compared to quasi-static values. It is observed that the radius ratio does not affect the syntactic foam properties significantly when tested at the same high-strain rate and volume fraction. Scanning electron microscopy is carried out to understand the fracture modes of the syntactic foams.

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Introduction

Polymeric syntactic foams, composed of both open- and closed-cell porosity, are suitable for weight saving aeronautical and marine structural applications [1]. Syntactic foam, type of closed-cell foam with properties such as high damage tolerance, low density, and high specific strength, has increased its use in composite sandwich panels for civil, automobile, and aircraft structural applications [2]. Depends on loading and environmental conditions either open- or close-cell structured foams can be selected for a specific application. Both foams are widely used in sandwich composites as core materials [3]. However, moisture absorption and thermal expansion coefficients of close-cell foams are lower than open-cell foams, and therefore provide more dimensional stability during service [4, 5].

Syntactic foam is fabricated by incorporation of hollow particles (microballoons) in a matrix material. It is one of the most widely used close-cell structured foams because of properties such as high strength-to-weight ratio, excellent thermal insulation, low radar cross-section, and vibration damping. Syntactic foams can be considered as particulate reinforced composites composed of matrix and microballoons only. However, they can also be considered as to be composed of matrix, microballoons, and voids [6]. Syntactic foams give great design flexibility as microballoons and matrix can be made up of several types of materials and quantity depending on the desired composite properties. Wall thickness of microballoons, volume fraction of constituents, and interfacial properties can also be accustomed to fabricate syntactic foams exactly as required in specific applications. Large numbers of studies and research activities on the mechanical properties of syntactic foams are mainly focused on the quasi-static properties, such as tension, compression, and flexural properties, and the associated fracture-mode behavior [6–13]. Polymeric materials and composites are found to be strain rate sensitive and therefore the properties of syntactic foams can be dependent on the loading rate.

Large number of studies can be found in the published literature on the high-strain rate testing of several types of polymeric and metallic foams performed by using various techniques, but not much on syntactic foams. Dynamic tests are conducted in industry to characterize the impact energy behavior of a variety of rigid polymers using drop weight tower [14] or simulated head impact using dynamic impact sled [15]. High-strain rate mechanical properties of metallic foams have been studied by many researchers using the Split-Hopkinson Pressure Bar (SHPB) technique [16–19]. These studies suggest that compressive flow stress of the Al-foam is a function of the relative density but does not exhibit strain rate sensitivity. High-strain rate compressive behavior of a rigid polyurethane foam with various densities was determined by Chen et al. [20]. They found the peak stress to be strain rate sensitive and expressed it in terms of the square of the foam density. Tensile and compressive properties of polystyrene bead foams at various temperatures and strain rates were studied extensively by Rinde and Hoge [21]. Some of the previous studies on the dynamic properties of honeycomb structures can also be found in the published literature [22, 23]. These studies found an increase of 20–70% in the dynamic crush strength at impact the velocities of 30 m/s.

However, there is only limited dynamic or high-strain rate studies of syntactic foams found in the published literature [24, 25]. There are several applications such as in aircraft and marine structure where the components undergo multiple dynamic or impact loading during service. These applications require a fundamental understanding of dynamic mechanical properties of syntactic foams, because the impact loading conditions may cause unexpected response unlike quasi-static condition. Therefore, syntactic foams should extensively be characterized for high-strain rate or dynamic properties using experimental apparatus that would provide stress–strain to strain rate relationships. The data provided from such an experiment are critical for efficient design of structures that could go through impact loading and use in numerical modeling for more realistic simulations.

In the present work, an SHPB apparatus is used to determine the high-strain rate properties of syntactic foams of nine different types of syntactic foams. The syntactic foams have three different radius ratios (related to the wall thickness of the microballoons), and the study is carried out at 10, 30, and 60% microballoon volume fractions. The specimens are cylindrical in shape with an aspect ratio of 1. The high-strain rate test results provide the relationship of dynamic properties of syntactic foams and radius ratio. The effect of radius ratio on the high-strain rate values of compressive strength, failure strain, and compressive modulus is observed and analyzed. Extensive scanning electron microscopic observations are performed to establish the modes of failure. Additionally, these high-strain rate properties are compared to the results of the quasi-static tests to evaluate the effect of radius ratio at different strain rates.

Syntactic foam—radius ratio

The fractured surface of typical syntactic foam that clearly shows the microballoons and the epoxy matrix is shown in Fig. 1. Syntactic foams can be considered as two-phase materials having microballoons dispersed in a matrix material or three-phase material with void being considered as an additional component. The density of syntactic foams can be changed without changing the volume fractions of microballoons and matrix material in the structure. This is achieved by using microballoons of different wall thicknesses. The same volume fraction provides a constant interfacial area between matrix and microballoons, and variation of properties can directly be related only to the difference in wall thickness. Gupta and Woldesenbet [9] have introduced the concept of the radius ratio, η, parameter and the relationship of the radius ratio, η, and microballoon wall thickness is given by Eq. 1.

$$\eta = \frac{r_1}{r_0}, \tag{1}$$

where r_1 is the internal radius and r_0 is the outer radius of the microballoon. The sketch of microballoons of varying η with notations used is shown in Fig. 2. The radius ratio, η, varies between 0 and 1. The wall thickness decreases correspondingly when η increases leading to a decrease in

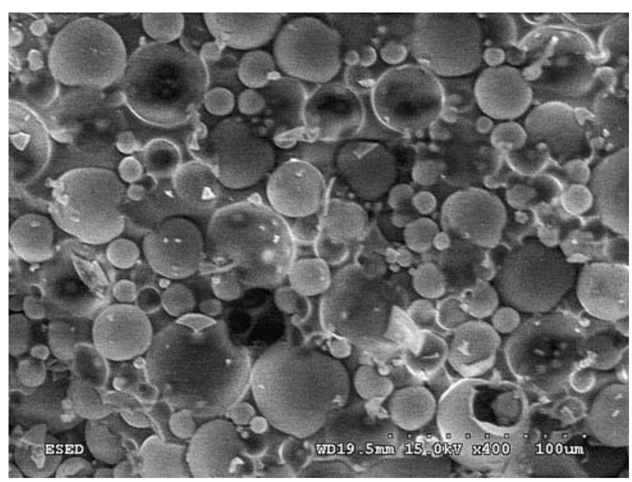

Fig. 1 Structure of syntactic foam

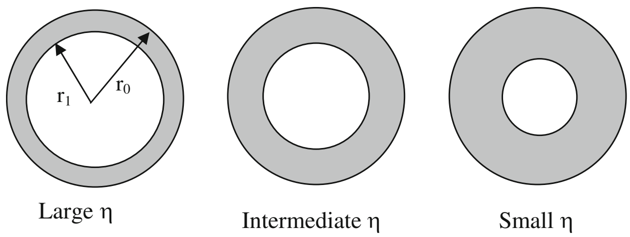

Fig. 2 Microballoons of varying η with notations

the density of the microballoon. Similarly, when η decreases, the density of the microballoon increases, and therefore the syntactic foam's density increases. All selected types of microballoons have η value more than the critical value of 0.71 to make the direct comparison of experimental results meaningful [26]. It was theoretically established that syntactic foams having η value higher than 0.71 experience similar stress states in the specimens during compression testing, where the fracture of the microballoons did not induce compression on the matrix.

Experimental procedure

Material and specimen

Three types of microballoons used for fabrication of syntactic foams are hollow spherical particles of chemically stable soda-lime borosilicate non-porous glass, manufactured and supplied by 3M Company under the trade name "Scotchlite." All three types of microballoons have similar outer radius, thus having a mean diameter of 40 μm. However, η is different for each of them. The radius ratio and particle density of the microballoons (as supplied by the manufacturer) are given in Table 1. Diglycidylether of bisphenol A-based epoxy resin D.E.R. 332 manufactured by DOW Chemicals with hardener D.E.H. 24 is used as the matrix material. The molecular weight of the hardener is 146.4 and weight per active hydrogen is 24.4. Phr (parts per hundred parts of resin) of amine for 95:5% by weight resin-diluent mix is calculated to be 13.74. For the selected combination of epoxy resin and hardener the curing schedule is to gel at room temperature and then post cure at 100 °C for 1–2 h. A diluent C_{12}–C_{14} aliphatic glycidyl ether, commercially known as ERISYS-8, is used to lower the viscosity of the resin, which is desired to properly mix and wet the microballoons. It is difficult to mix large volume of microballoons in the resin if the viscosity is very high. Adding 5% by weight diluent C_{12}–C_{14} aliphatic glycidyl ether brings down the viscosity of the resin from about 4000 cps at 20 °C to about 2000 cps at the same temperature. The diluent was supplied by CVC Specialty Chemicals. Average equivalent epoxide weight (EEW) of the diluent is 285. For a 95 wt.% resin and 5 wt.% diluent mixture, the EEW is calculated to be 17.75. The volume fraction of microballoons is maintained at 0.1, 0.3, and 0.6, creating a total of nine different types of syntactic foams.

A slurry mixture of microballoons, resin, hardener, and diluents is cast in stainless steel molds of 240 × 240 × 13 mm^3 dimensions and cured for at least 36 h at room temperature. The slabs are removed from the molds and post cured at 100 °C for approximately 3 h. It is quite possible that the fabricated syntactic foams have some entrapped air, called voids, due to mechanical mixing. The total void content varies from 1 to 6% volume fraction for the fabricated syntactic foams with the higher amount of voids appearing in the 60% microballoons volume fraction syntactic foams. The measured densities of fabricated syntactic foams are presented in Table 2.

Cylindrical specimens of 9.5 mm in diameter and 9.5 mm in length are core drilled from the syntactic foam slabs for testing. The diameter of the specimens is kept slightly less than that of the SHPB apparatus bars, 9.65 mm, to make sure that the specimens are fully impacted. There is a slight Poison's ratio effect causing lateral expansion during high-strain rate testing using the SHPB, and therefore the specimen diameter never exceeds the pressure bar diameter up to its fracture strain. The specimen ends are carefully polished with 400-grit polish paper.

Testing methods

Quasi-static compression testing of the syntactic foams is carried out using MTS 810 Material Test System. The compressive strength and modulus are calculated using the load and displacement data obtained from the machine. Constant crosshead velocity of 1.3 mm/min was maintained

Table 1 Properties of microballoons used to fabricate syntactic foams

Microballoon type	Microballoon density (kg/m^3)	Microballoon size distribution (μm)			Calculated radius ratio (η)
		10th Percentile	50th Percentile	90th Percentile	
S32	320	20	40	75	0.907
S38	380	15	40	75	0.888
K46	460	15	40	70	0.863

Table 2 Density of fabricated syntactic foams

Microballoon type	Volume fraction (%)	Corresponding foam nomenclature	Syntactic foam density (kg/m^3)
S32	10	SF 3210	1,020
S38		SF 3810	1,035
K46		SF 4610	1,043
S32	30	SF 3230	861
S38		SF 3830	877
K46		SF 4630	902
S32	60	SF 3260	545
S38		SF 3860	575
K46		SF 4660	685

following the recommendation by ASTM D 695-94 standards. This compression rate corresponds to a strain rate of about 3 × 10^{-4}/s. Test specimens have cross-sectional area of 25.4 × 12.5 mm^2 and height of 25.4 mm.

An SHPB equipment that is popularly known as Kolsky bar well described in the literature is used to carry out the high-strain rate tests [27]. The SHPB is modified with pulse shapers to minimize wave dispersions and obtain the right shape of the wave. In this technique, a cylindrical specimen is mounted between long incident and transmitter bars of very high yield strength, while a short striker bar is used to produce an impact on one end of the incident bar. The overall specimen dimensions are required to be small enough to minimize the effects of longitudinal and lateral inertia and wave dispersion within the specimen. In addition, a frictional constraint at both pressure bar–specimen interfaces due to the radial expansion of the specimen during loading is significantly reduced by applying a thin film of lubricant at the interfaces. A molybdenum disulfide is applied as a lubricant. The details about testing by using this technique for high-strain rate testing of materials can be found elsewhere in the literature [28].

Results and discussion

The radius ratio, η, effect on the high-strain rate compression properties of syntactic foams is presented. The quasi-static results of the same type of syntactic foams are also compared to the dynamic results in order to understand the effect of radius ratio at different strain rates. A total of nine types of specimens are tested both at quasi-static and three different high-strain rates. Typical stress versus strain curves for the three syntactic foams at 10, 30, and 60% volume fractions obtained at approximately 800/s strain rate are shown in Figs. 3–5. The results show that there is some effect of radius ratio on the high-strain rate properties of syntactic foams. These figures indicate that varying the

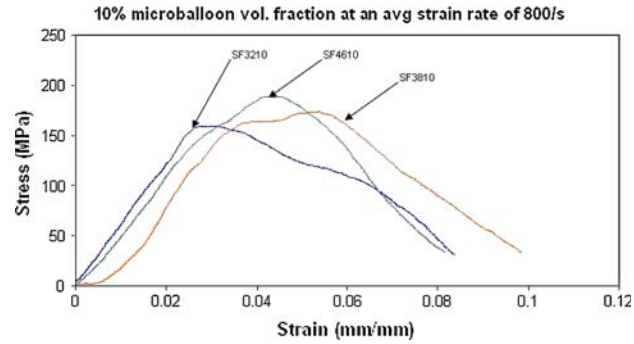

Fig. 3 Stress versus strain curves for three types of syntactic foams at 10% volume fractions obtained at approximately 800/s strain rate

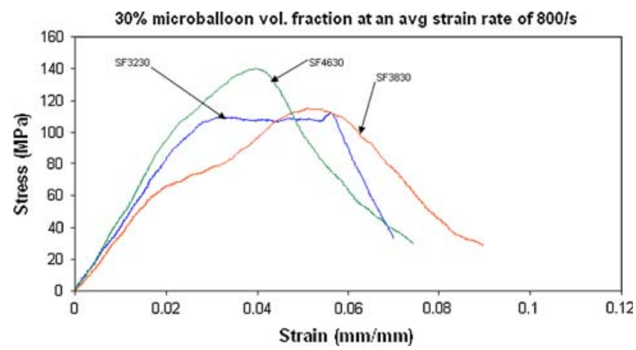

Fig. 4 Stress versus strain curves for three types of syntactic foams at 30% volume fractions obtained at approximately 800/s strain rate

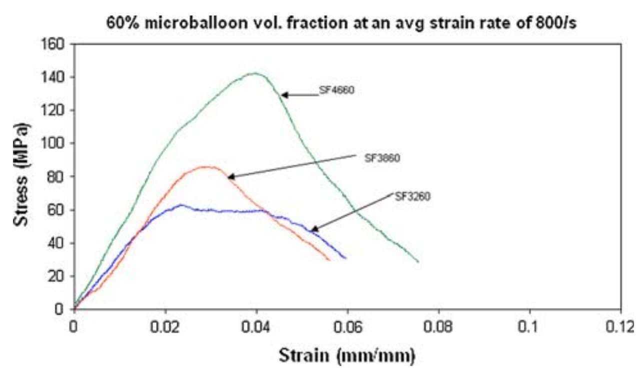

Fig. 5 Stress versus strain curves for three types of syntactic foams at 60% volume fractions obtained at approximately 800/s strain rate

radius ratio changes the behavior of the syntactic foam, even though the volume fractions of the microballoons and the matrix do not change. The peak stress and the corresponding strain at 800/s strain rate change depending on the radius ratio of the microballoons. However, there is no clear trend. For example, the strain at peak stress for SF46 is smaller than that of SF38 at 10% volume fraction but the reverse is true at 60%. The fact that there is no trend in the strain values strongly indicates that the critical strain at which peak strength is observed does not depend on the type of microballoons and can be primarily recognized as the matrix property as similarly concluded by Woldesenbet et al. [29] previously. It is shown that the SF46 type syntactic foam, with dense microballoons having $\eta = 0.863$, has the most superior peak stress and modulus at the volume fractions tested. The superiority of the SF46 seems to be more apparent as the microballoons volume fractions increase. Another distinct observation from Figs. 3–5 is the fact that at all three volume fractions, the SF32 syntactic foams, have an extended plateau region compared to the other two types of syntactic foams. The plateau region indicates that there is more extensive crushing of microballoons in SF32 than in SF38 and SF46 syntactic foams. This observation corresponds to the fact that SF32 is composed of microballoons with the largest radius ratio which is equivalent to the thinnest walls, and therefore the easiest to crush. Figure 6 shows the scanning electron microscopy (SEM) images of fractured surfaces of SF3260 and SF3860 specimens tested at strain rates in the range of 780–820/s.

The discussed stress versus strain graphs provide a general overview of the effect of microballoons radius ratio at high-strain rates. However, it is critical to understand the magnitude of this radius ratio effect at different strain rates including quasi-static strain rates. Figures 7–9 and 10–12

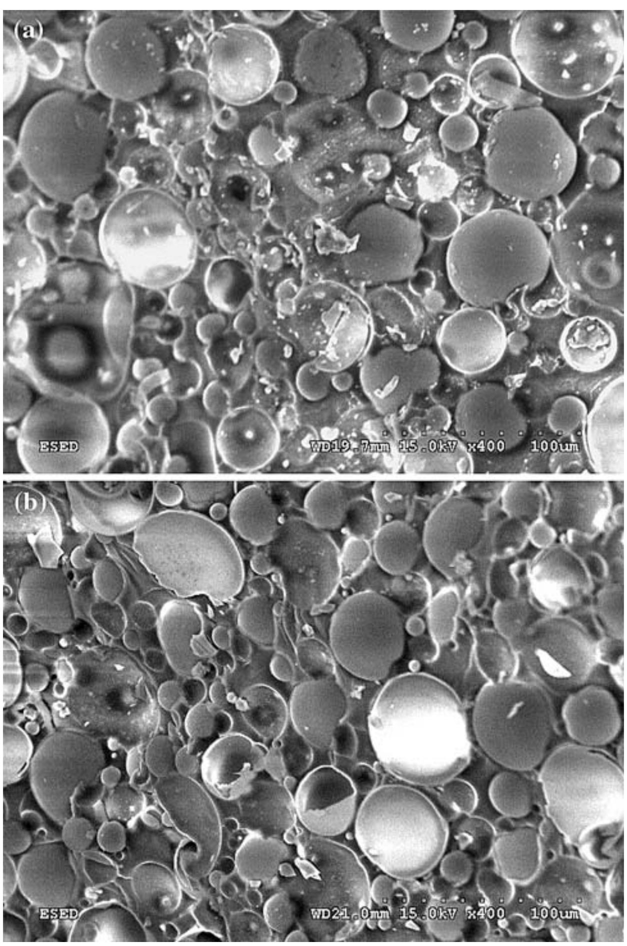

Fig. 6 SEM pictures of fractured surfaces of **a** SF3260 and **b** SF3860 specimens, respectively, at ~800/s strain rates

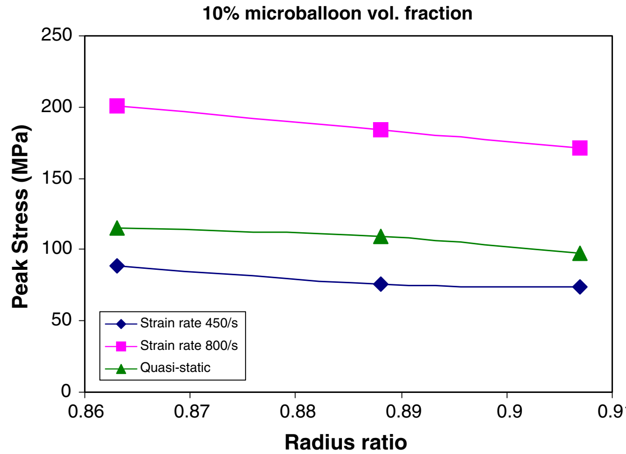

Fig. 7 Maximum stress versus radius ratio at 10% microballoon volume fractions and varying strain rates

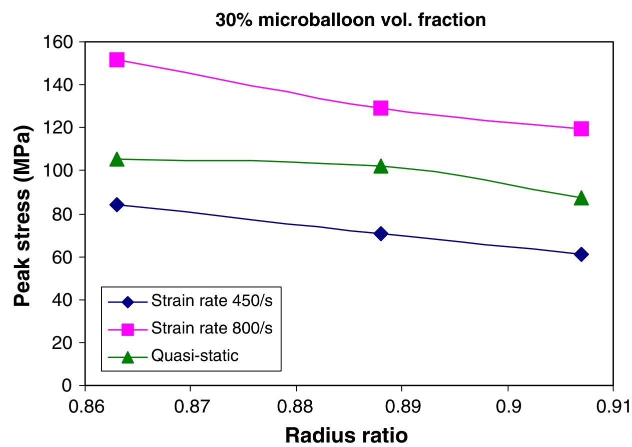

Fig. 8 Maximum stress versus radius ratio at 30% microballoon volume fractions and varying strain rates

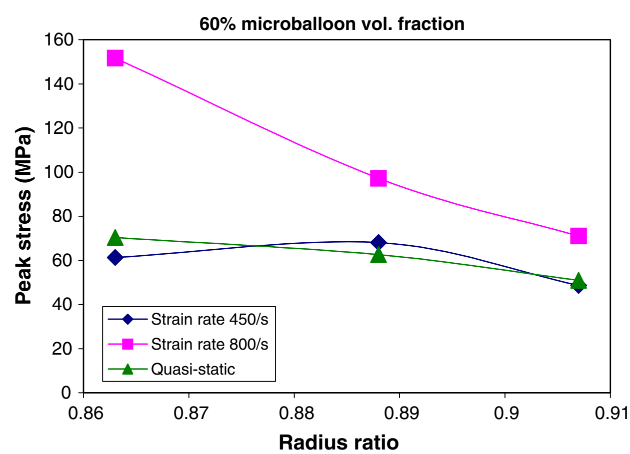

Fig. 9 Maximum stress versus radius ratio at 60% microballoon volume fractions and varying strain rates

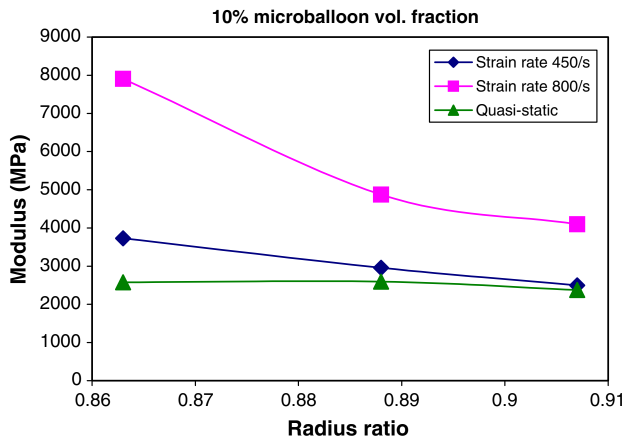

Fig. 10 Modulus versus radius ratio at 10% microballoon volume fractions and varying strain rates

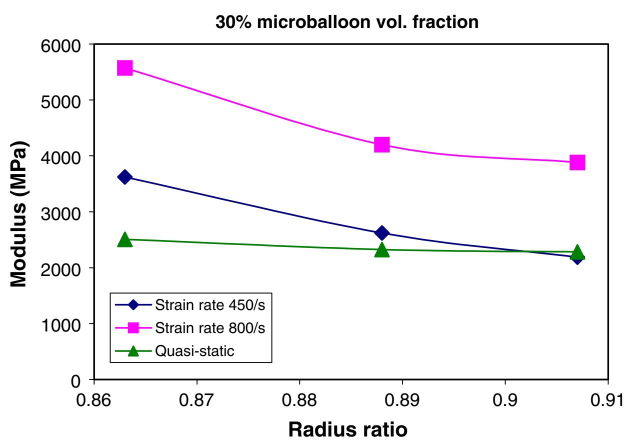

Fig. 11 Modulus versus radius ratio at 30% microballoon volume fractions and varying strain rates

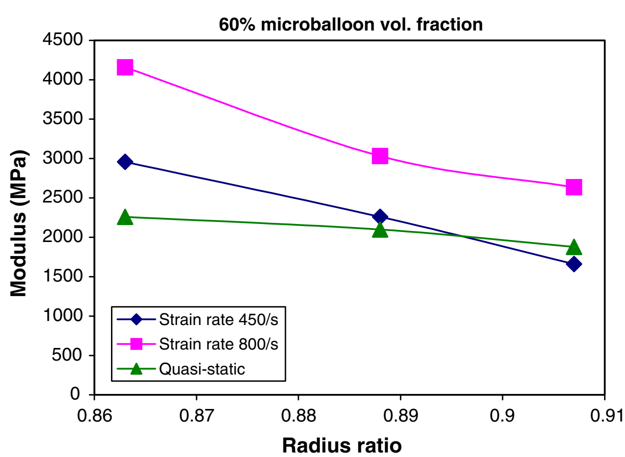

Fig. 12 Modulus versus radius ratio at 60% microballoon volume fractions and varying strain rates

show the maximum stress versus radius ratio and modulus versus radius ratio, respectively, at 10, 30, and 60% microballoon volume fractions and varying strain rates. The results including the standard deviations are also given in Tables 3–5.

The maximum stress values show no significant variation for all types of syntactic foams and strain rates, when the radius ratio changes except for SF46 samples at 800/s that has the most dense microballoons at $\eta = 0.863$. At strain rate of 800/s, the effect of radius ratio on the maximum stress increases as the microballoon volume fraction increases from 10% where there is almost no effect at all, to 30% where a slight decrease is observed, and to 60% where a significant decrease occurs as the radius ratio increases. The peak stress at 450/s and quasi-static strain rate show no significant variation at 10 and 30%

Table 3 Radius ratio effect on the modulus and peak stress values at varying strain rates and 10% volume fraction

Syntactic foam type	10% Microballoon volume fraction		
	Strain rate (s^{-1})	Modulus (MPa)	Peak stress (MPa)
SF32	578.4	2950 ± 55	74 ± 12
	617.38	3631 ± 51	133 ± 13
	775.69	4945 ± 60	168 ± 4
	Quasi-static	2430	107
SF38	495.73	3121 ± 33	76 ± 5
	591.13	4046 ± 63	145 ± 4
	742.97	5239 ± 66	184 ± 17
	Quasi-static	2551	110
SF46	454.23	3503 ± 39	78 ± 5
	519.56	4545 ± 69	148 ± 3
	601.34	6062 ± 45	190 ± 10
	Quasi-static	3719	113

Table 4 Radius ratio effect on the modulus and peak stress values at varying strain rates and 30% volume fraction

Syntactic foam type	30% Microballoon volume fraction		
	Strain rate (s^{-1})	Modulus (MPa)	Peak stress (MPa)
SF32	504.57	2311 ± 31	61 ± 4
	676.8	3344 ± 29	126 ± 10
	794.95	4024 ± 36	155 ± 6
	Quasi-static	2281	87
SF38	512.74	2619 ± 39	68 ± 7
	651.01	3883 ± 60	129 ± 10
	830.82	4454 ± 29	158 ± 9
	Quasi-static	2325	102
SF46	441.54	2856 ± 57	71 ± 2
	605.03	4552 ± 31	132 ± 9
	694.37	4859 ± 25	161 ± 5
	Quasi-static	2508	105

Table 5 Radius ratio effect on the modulus and peak stress values at varying strain rates and 60% volume fraction

Syntactic foam type	60% Microballoon volume fraction		
	Strain rate (s^{-1})	Modulus (MPa)	Peak stress (MPa)
SF32	474.03	1845 ± 45	42 ± 3
	654.12	2657 ± 40	54 ± 3
	942.89	3364 ± 77	81 ± 10
	Quasi-static	1878.11	50.96
SF38	480.57	2259 ± 60	55 ± 4
	647.34	2744 ± 24	58 ± 6
	954.55	3881 ± 47	130 ± 15
	Quasi-static	2099.33	62.62
SF46	483.52	2378 ± 21	55 ± 6
	624.67	3000 ± 33	59 ± 2
	722.56	4335 ± 54	130 ± 15
	Quasi-static	2259.66	64.158

microballoon volume fraction. At 60% microballoon volume fraction, a slight decreasing trend is observed in peak stress values at 450/s and quasi-static strain rates where air voids affect the results.

Therefore, this can suggest that the peak stress obtained at high-strain rate loading of syntactic foams does not depend on the microballoons radius ratio but rather depends on the matrix material. The exception to the above conclusion occurs for 60% microballoon volume fraction syntactic foams when tested at 800/s. These specimens show significant dependence on the radius ratio because of the reduced volume fraction of the matrix decreasing the role of the matrix and increasing the probability of crack propagation through microballoons. Therefore the peak stress will vary based on the type of microballoon, with SF4660 having the highest value and SF3260 the lowest. Based on all tests carried out at different strain rates and 60% volume fraction, it can be concluded that the higher the strain rate the more the effect of radius ratio.

An unexpected result of this experiment is the lack of change or slight decrease in the peak stress of all types of syntactic foams at 450/s compared to the peak stress obtained at quasi-static. SEM images of fractured surfaces of SF4660 tested at quasi-static and 450/s strain rates are shown in Fig. 13. These micrographs exhibit different dominant fracture modes. In Fig. 13a, corresponding to the fracture surface of SF4660 specimen tested at quasi-static loading, there is considerable crushing of the microballoons. In Fig. 13b, corresponding to the fracture surface of the specimen tested at lower strain rate of 470/s, it is observed that the crack propagates through either the matrix material or the matrix–microballoon interfaces completely bypassing the microballoons. Cracks passing

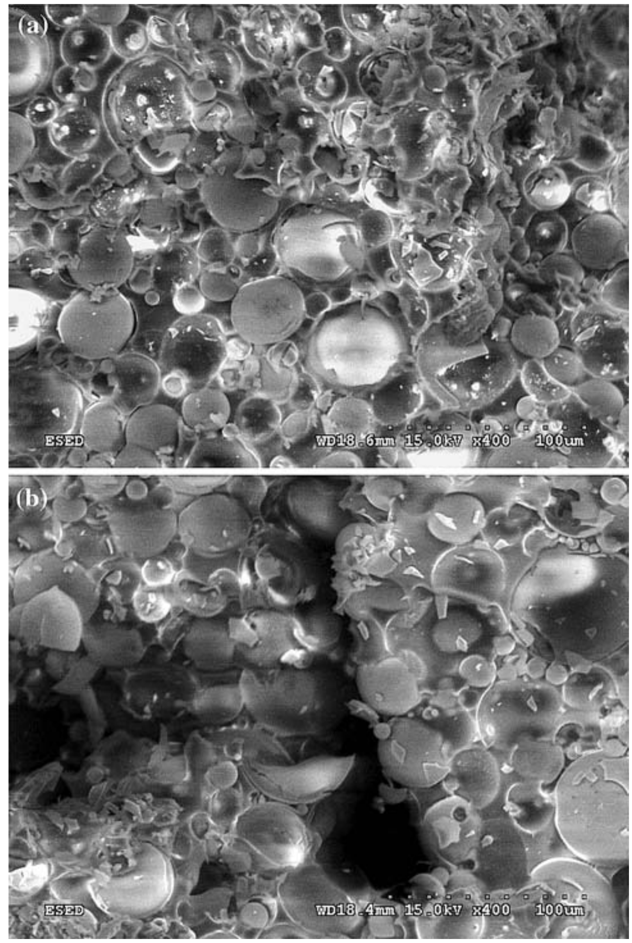

Fig. 13 SEM images of fractured surfaces of SF4660 tested at **a** quasi-static and **b** 470/s strain rates

through the matrix–microballoon interface cause limited fracture of microballoons as compared to the extensive fracture observed in quasi-static loading. Figure 9 depicts the nature of the above behavior where the peak stress of the syntactic foam at quasi-static loading is found to be equivalent to the peak stress at medium strain rate loading.

The result is as expected for strain rate loading at 800/s for all types of syntactic foams as shown in Figs. 7–9. The peak stress values are in general significantly higher for 800/s tests compared to quasi-static tests at all radius ratios and volume fractions, consistent with prior results [24, 25]. The increase in maximum strength with increase in strain rate can be attributed to the fact that at slower strain rates, the damage propagates more slowly expending most of the applied energy. However, at higher strain rates, the damage is constrained to propagate in a particular path without dispersion, and a higher amount of energy is absorbed under this situation. This is accompanied by increase in stress level as compared to quasi-static conditions for similar strain values. At the higher strain rate of 800/s, most of the fracture is a result of straight cleavage.

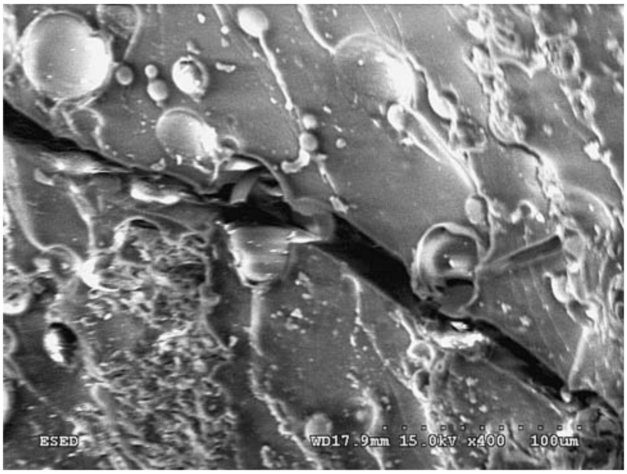

Fig. 14 SEM image of the fractured surface of SF4630 syntactic foam tested at 740/s

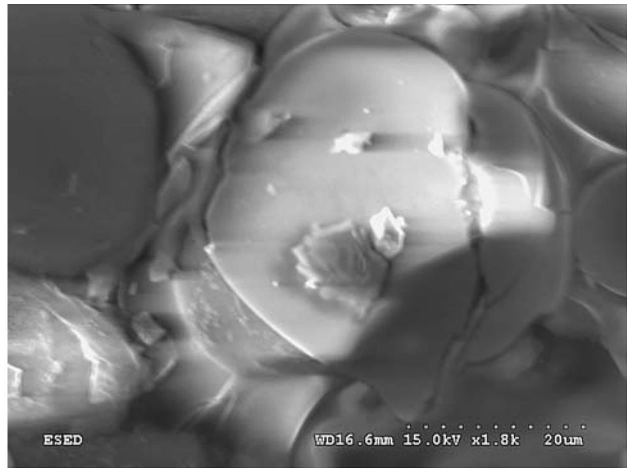

Fig. 15 SEM image of a broken microballoon in the path of the crack

Figure 14 shows the fractured surface of SF4630 syntactic foam tested at 740/s. In the higher strain rate test specimens cracks start from one end and propagate to the other end across the specimen length. Therefore, in the higher strain rate test, the cracks tend to fracture the microballoons while propagating fast.

In regards to the radius ratio effect on the dynamic modulus of the various syntactic foams, Figs. 10–12 show the variations at both 450 and 800/s. The effect of radius ratio on the dynamic modulus is more significant on tests carried out at 800/s compared to those carried out at 450/s. In all high-strain rate tests, the modulus decreases as the radius ratio increases. The higher radius ratio microballoons have thinner walls, and therefore the cracks tend to fracture these microballoons more than the lower radius ratio microballoons when tested at the same high-strain rates as indicated in Fig. 6. Also, the modulus values at 450/s and quasi-static strain rate tests are virtually the same. As in the case of the peak stress, the crushing of the microballoons creates higher modulus in quasi-static testing because of the higher stiffness of the glass microballoons than the epoxy. This increase is similar to the modulus values of specimens tested at 450/s. The increase at 450/s is attained from the matrix due to its strain rate sensitivity. It has been proven that composite materials have higher dynamic modulus because of matrix strain rate sensitivity [30, 31].

The total energy absorbed by syntactic foam samples can be considered as the area under the stress versus strain curve at 800/s strain rate as shown in Figs. 3–5. It is found that total energy absorbed is independent of radius ratio at 10 and 30% microballoon volume fraction. It only shows dependence on radius ratio for SF46 specimens with $\eta = 0.86$ and SF38 specimens with $\eta = 0.888$ at 60% microballoon volume fraction. Several factors play a role in determining the total energy absorbed based on the strain rate and radius ratio. For quasi-static tests, there is significant amount of crushing for all syntactic foams composed of microballoons of the three radius ratios, where the variation in radius ratio does not affect the energy absorbed in any significant manner. For 450/s experiments, the specimens show less microballoon crushing and not much of microballoons fracture. The cracks tend to propagate through the matrix or microballoon–matrix interface, and therefore the total energy does not depend on the radius ratio. For 800/s tests, the fracture behavior of the syntactic foam mainly involves vertical crack propagation from one end to another end of the specimen. The crack propagates fast and fractures the microballoons in the vertical path of the crack. Figures 14 and 15 show the vertical crack plane passing through microballoons and a closer view of a broken microballoon, respectively. Thus, at high-strain the amount of energy absorbed is affected by radius ratio of microballoons.

Conclusion

The effect of radius ratio on the high-strain rate compressive properties of syntactic foams is established in this study. Altering the radius ratio changes the behavior of the syntactic foams to varying degree, even though the volume fractions of the microballoons and the matrix do not change. It is found that the dynamic material properties obtained from tests carried out at quasi-static and 450/s strain rates show no major dependence on radius ratio, while those at 800/s do, especially at 60% volume fraction. The critical strain at which peak stress is observed does not depend on the type of microballoons and can be primarily recognized as the matrix property. Even though not significant, the effect of radius ratio on the maximum stress and dynamic modulus is more on tests carried out at 800/s compared to those carried out at 450/s. In all strain rate tests, the maximum stress and modulus decrease as the radius ratio increases. Syntactic foams are in general found to be strain rate sensitive. However, the sensitivity depends on the strain rate achieved. The maximum stress and modulus values at quasi-static and 450/s strain rates show no significant differences for syntactic foams made of microballoons of the same radius ratio. However, there is a large difference in the values between quasi-static and 800/s strain rates. The effect of radius ratio at varying strain rates is better explained by the modes of fracture as observed using scanning electron images.

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