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SYNTACTIC AND COMPOSITE FOAMS

Compressive and ultrasonic properties of polyester/fly ash composites

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Abstract The addition of hollow fillers having appropriate mechanical properties can decrease the density of the resulting composite, called syntactic foams, while concurrently improving its mechanical properties. In this study, hollow fly ash particles, called cenospheres, are used as fillers in polyester matrix material. Cenospheres are a waste by-product of coal combustion and, as such, are available at very low cost. In this study, the composites were synthesized by settling cenospheres in a glass tube filled with liquid polyester resin and subsequently curing the resin. This process resulted in a functionally graded structure containing a gradient in the cenosphere volume fraction along the sample height. Uniform radial sections were cut from each composite and were characterized to observe the relationship between cenosphere volume fraction and compressive properties of the composite. The composite was also tested using ultrasonic non-destructive evaluation method. Results show that the modulus of the composites increases with increasing cenosphere volume fraction. The modulus of composites containing more than 4.9 vol% cenosphere was found to be higher than the matrix resin. In general, the modulus of composites increased from 1.33 to 2.1 GPa for composites containing from 4.9–29.5 vol% cenospheres. The specific strength of the composite was found to be as high as 2.03 MPa/(kg/m^3) compared to 0.96 MPa/(kg/m^3) for the neat resin. Numerous defects present in fly ash particles caused a reduction in the strength of the composite. However, the reduction in the strength was found to be only up to 22%. Increase of over 110% in the specific modulus and only a slight decrease in the strength indicates the possibility of significant saving of weight in the structures using polyester/fly ash syntactic foams.

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Introduction

Incorporation of rigid solid fillers is known to increase Young's modulus and other mechanical properties of polymers [1, 2]. Most of the rigid fillers are made of ceramic materials, which have higher density than the polymer matrix materials. Inclusion of high volume fractions of solid fillers results in an undesirable increase in the density of the composite material. Because of this there is a tradeoff between any improvements in the modulus and other mechanical properties of composites relative to the increase in density when compared with the properties obtained in the neat polymer system. This tradeoff can be minimized by using stiffer particles, such as those made of glass. In general, the ratio of Young's modulus of the particle and the matrix material, termed as relative Young's modulus, determines if the presence of particles would enhance or degrade the mechanical properties of the composite.

The hollow particles used in making syntactic foams can be made of polymers, metals, or ceramics and are available in a wide range of sizes and densities. Glass microballoons and fly ash cenospheres are the most commonly used hollow particles used in such composites [3–7]. Both types of particles are made of ceramic materials and, in spite of their hollow nature, have a high Young's modulus relative to the encapsulating resin and thus provide an improvement in the mechanical properties of the composite when compared to those exhibited by the neat resin.

Fly ash cenospheres are obtained as a waste by-product of burning coal in thermal power plants [7] and their cost is primarily associated with cleaning and transportation. Fly ash particles are crystalline compounds of quartz, mullite, hematite, and glassy compounds such as silica and other metal oxides. The results of energy-dispersive X-ray spectroscopy (EDX) and X-ray diffraction spectroscopy (XDS) analyses have shown that fly ash is a mixture of SiO_2, Al_2O_3, Fe_2O_3, and several metallic oxides, and its microstructure is very complex [8–12]. Over 70 million tons of fly ash is produced in the United States each year, about half of which is dumped in landfills. The other half is primarily used as filler in cement and concrete. If other constructive uses of this waste fly ash could be identified then more of this waste material could be diverted from landfills.

In their primary application, cenospheres are mixed with cement to decrease the mixture density. Moreover these cement–fly ash mixtures have improved thermal insulation and better vibration damping properties [13, 14]. Use of fly ash particles as a filler and reinforcement in metal-matrix composites is also expanding [5, 15–17]. Incorporation of fly ash in an aluminum alloy matrix is observed to increase hardness and density, and to improve wear resistance [15]. The same improvements can also be obtained by incorporating fly ash in polymer matrices. The yield strength and Young's modulus of hollow particle-filled polypropylene matrix composites increase and elongation decreases as the filler volume fraction increases [18–20]. Epoxy/fly ash composites have also been characterized and similar results are obtained [21–23]. These studies provide an important insight into the parameters that can lead to enhanced properties in the composite.

In recent studies, mathematical models have been used to find relationships between mechanical properties of composites and wall thickness and volume fraction of hollow particles [24, 25]. These relations help in optimizing the weight saving and mechanical property enhancement in composites. The models show that enhancement in modulus of composites is obtained when hollow particles of a minimum wall thickness are used in more than a minimum volume fraction [24]. The critical volume fraction depends on the particle wall thickness and also on the relative modulus of the particle and the matrix material.

Polyester resins are used in a wide variety of applications, including construction, marine, transportation, electrical, and sanitary ware because of their ease of fabrication, high strength, corrosion resistance, and low cost. In spite of their widespread use, there is no systematic information on how the properties of cenosphere-filled polyester matrix composites are improved by the cenosphere additions. In this study, polyester resin is used as the matrix material, and the synthesized composites are characterized using compression testing and ultrasonic non-destructive testing to determine their mechanical properties and elastic constants. Specific modulus and specific strength of polyester/cenosphere composites are analyzed as a function of cenosphere volume fraction.

Experimental procedure

Material preparation

Fly ash cenospheres, obtained from Dayton Power & Light Company, OH, were sieved to separate different particle size ranges. The particle size range of 45–75 μm was selected to fabricate composites. These cenospheres had an average true particle density of 600 kg/m^3. Cenospheres were demagnetized using a handheld magnet, and then they were mixed with unsaturated polyester resin Polylite® 32462-00, procured from Reichold Chemicals, NC. This resin has specific gravity of about 1210 kg/m^3. The composites were cast into glass tubes (14-mm diameter) and cured for 24 h using 0.5 wt% of a 6% cobalt naphthenate solution in styrene (Aldrich Chemicals) at room temperature. The processing was carried out at 70 °C to decrease the viscosity of the resin and facilitate settling of particles so that a gradient in the particle volume fraction can be obtained.

During the curing process, cenospheres start rising in the tube because of their lower density compared to the matrix resin, leading to a gradient in the volume friction of particles along the height of the composite. The graded composite bars were removed from the glass tubes and then cut into radial pieces (14-mm diameter and 8-mm height) using an Enco water-cooled cutting machine. Each of these pieces contained a different volume fraction of cenospheres, as schematically shown in Fig. 1. The cut specimens were used to study the effect of cenosphere volume fraction on the mechanical properties of composites.

The density of specimens was measured using ASTM C693-93(2003) standard [26] and was used to calculate the cenosphere volume fraction in specimens. While each specimen has a gradient of fly ash volume fraction along its height, the average fly ash volume in each section was used to characterize the section. The calculated volume fraction of cenospheres is, therefore, an average value over the entire thickness of the specimen. Due to the small specimen thickness, the volume fraction is assumed to be

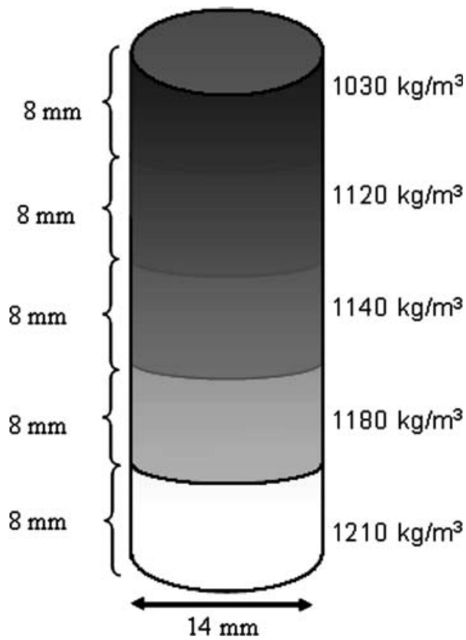

Fig. 1 Schematic representation of gradient structure of synthesized polyester/cenosphere composite. The composite was sectioned into 8-mm thick specimens for compression testing

constant within each specimen. It has been shown that some cenospheres break during composite synthesis; such breakage was not accounted for in this study. Consequently, the volume fraction is only an approximate value calculated from the measured density and is used to distinguish the density differences between various compositions, and its values are not used in any theoretical calculations. A typical structure of the composite is shown in Fig. 2. In this optical micrograph, some fly ash cenospheres are seen embedded in the polyester matrix. This micrograph will serve as a reference while interpreting the microscopic fracture features of various composite compositions.

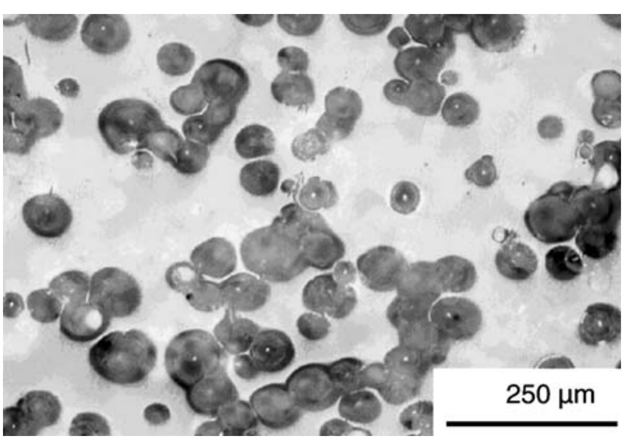

Fig. 2 An optical micrograph of a typical polyester/fly ash composite containing 29.5 vol% fly ash

Compression test

The compressive properties of polyester/cenosphere composites are measured in accordance with ASTM, D695-02 standard [27]. The thickness and diameter of each compression test specimen cut from the fabricated graded composites were 8 and 14 mm, respectively. The compression tests were conducted using an Instron test system at a constant crosshead speed of 1.3 mm/min. Five specimens of each type of composite were tested. Load and displacement data were obtained from the machine, which were converted to stress and strain information. These data were used to calculate modulus and other properties of composites.

Ultrasonic velocity measurements

Velocities of longitudinal and transverse ultrasonic waves in polyester/cenosphere composite specimens were measured using pulse-echo technique. The longitudinal and shear wave velocities were measured in separate experiments. A Physical Acoustic Ultrapac immersion type system was used for the longitudinal wave velocity measurement. The same system with contact type shear wave transducers, supplied by Technisonic Inc., was used for shear wave velocity measurements. The transducer frequency and sampling rate used in the study were 5 MHz and 500 ms/s, respectively. A typical ultrasound wave signal for a polyester/cenosphere composite is shown in Fig. 3. This waveform contains peaks corresponding to the front wall and the back wall of the specimen. The location of these peaks on the time axis and their amplitude were measured. The longitudinal (V_L) and shear (V_S) velocities (in m/s units) can be calculated by Eq. 1 [28].

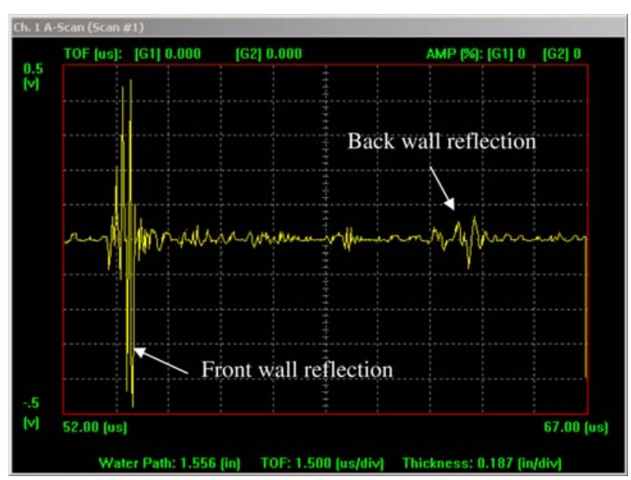

Fig. 3 A typical ultrasonic pulse–echo response waveform

$$V_L = \left(\frac{2t}{T_L}\right) \times 10^{-6} \quad (1a)$$

$$V_S = \left(\frac{2t}{T_S}\right) \times 10^{-6} \quad (1b)$$

where t, T_L, and T_S represent the specimen thickness, the time of flight for longitudinal wave, and the time of flight for the shear wave, respectively. The attenuation coefficient (α) is calculated using Eq. 2 [28].

$$\alpha = \frac{10 \times \log\left(\frac{I_0}{I}\right)}{2t} \quad (2)$$

where I_0 and I are the amplitudes of the front wall and the back wall reflection peaks in the A-scan waveform. The raw data obtained from the equipment was processed to calculate the wave velocities and attenuation coefficient.

Results and discussion

Microstructural observations

Cenospheres have lower density than the polyester resin (1210 kg/m^3) leading to their flotation during synthesis of composite specimens. This density difference caused a variation in their volume fraction along the sample height leading to a functionally graded microstructure. While the bottom part of the composite contains no cenospheres, the upper parts contained approximately 29.5 vol% of cenospheres. The average volume fractions of cenospheres in various sectioned specimens, calculated from the measured density using rule of mixtures, are presented in Table 1. It is shown that the specimen taken from the top part of the cast composite has a lower density because of higher cenosphere concentration.

The flotation velocity of cenospheres in the polymer melt determines their distribution in the composite. The flotation velocity is a function of the resin viscosity, which is determined by temperature and curing reaction rate, and cenosphere size, density, and sphericity. The terminal velocity of a spherical particle in a fluid is given by

$$V = \frac{g}{18\mu}(\rho_w - \rho_0)D^2 \quad (3)$$

where V, g, and μ are the terminal rise velocity of a sphere, the acceleration of gravity, and the dynamic viscosity of the fluid, respectively. Symbols ρ_w, ρ_0, and D denote the density of the fluid, the sphere density, and the sphere diameter, respectively. The extent of flotation of particles in the polymer can be effectively reduced by using smaller particle size, because the terminal velocity is a function of second power of the particle diameter. In hollow particles such as cenospheres, the parameter, sphere density, is dependent on the wall thickness. For a narrow particle size distribution, a gradient created by settling methods will be significantly dependent on the wall thickness of cenospheres.

A uniform distribution of particles is desired in the composite without formation of aggregates to obtain uniform properties. Particle agglomeration can be caused by three adhesion forces, which are mechanical, electrostatic, and molecular [29]. As particle size is decreased, the gravitational force decreases proportional to the third power of the diameter but the natural adhesion force decreases only between the first or second power of the diameter. Therefore, as the particle size decreases, the adhesion forces begin to dominate and the particles agglomerate naturally [30]. This effect can be evidenced by the fact that the microstructures containing the larger diameter cenospheres have a lesser occurrence of agglomeration. The microstructure presented in Fig. 2 did not show extensive agglomeration of cenospheres and the dispersion seems to be uniform.

Compressive properties

Compressive modulus

Typical compressive stress–strain curves for a neat polyester and a polyester/cenosphere composite are shown in Fig. 4. These curves are randomly selected from the five specimens of the same type of material. The stress–strain curves for all compositions of composites show similar trends. The curves for composites show a trend that is typical of syntactic foams [31, 32]. The compressive

Table 1 Measured compressive modulus and strength of polyester/fly ash composites

No.	Composite density (kg/m^3)	Cenospheres (vol%)	Modulus (GPa)	Yield strength (MPa)
1	1210	0	1.41	87.8
2	1180	4.9	1.33	98.0
3	1140	11.5	1.46	80.0
4	1120	14.8	1.76	79.1
5	1030	29.5	2.10	71.7

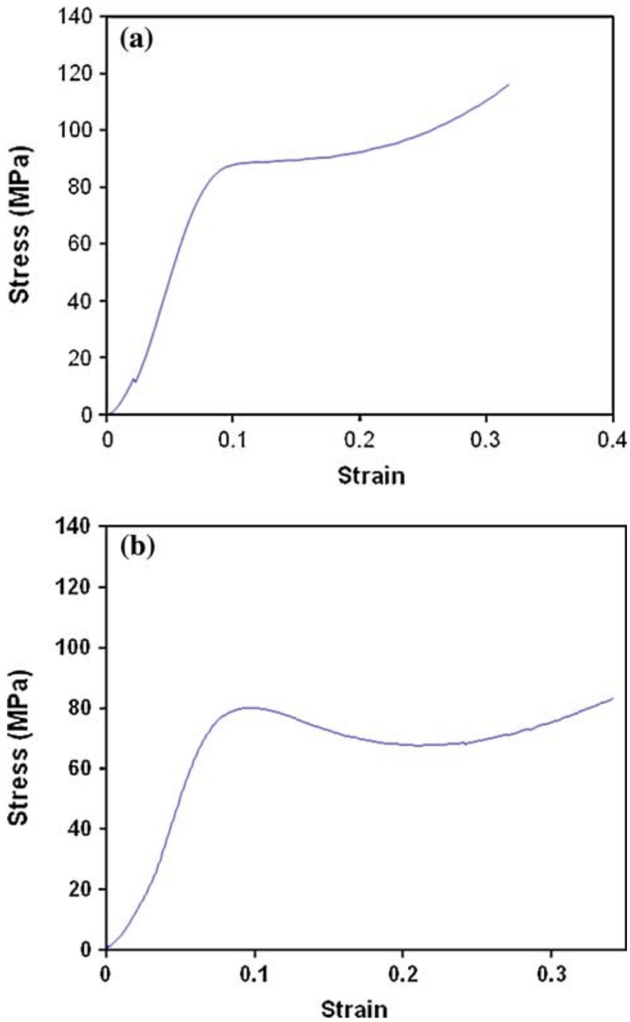

Fig. 4 Representative compressive stress–strain curves for **a** unsaturated polyester and **b** polyester/containing 4.9 vol% cenosphere fly ash particles. These graphs are randomly selected from the repeat tests on the same type of material

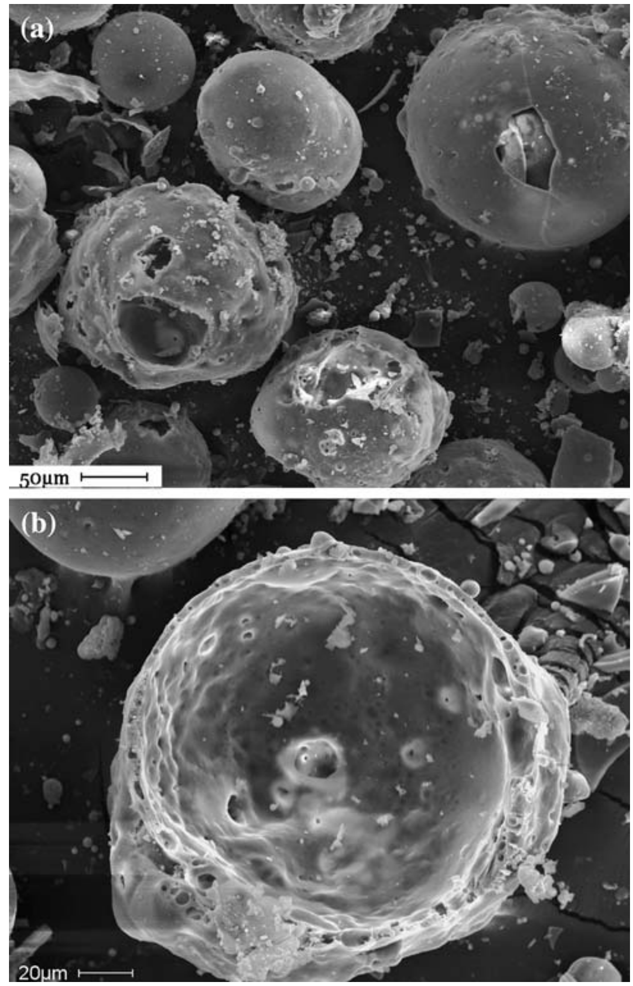

Fig. 5 SEM micrograph showing **a** some fly ash particles of irregular shapes and **b** numerous pores in the wall of a broken particle

modulus and yield strength obtained from the experimental data for various compositions of the composites are presented in Table 1. The modulus is found to be lower than that of the matrix material only for the composite containing 4.9 vol% cenospheres. For higher volume fractions of cenospheres, the modulus of composites is found to be higher than the matrix. In general, the modulus of composites increases with increasing particle volume fraction when the particles are stiffer than the matrix [33]. It has also been observed previously that the use of hollow particles in less than a critical minimum volume fraction results in reduction in the modulus of the composite [24].

The modulus of a hollow particle is lower than that of a solid particle of the same material because of the presence of a cavity. The value of effective modulus depends on the particle wall thickness [24, 25, 34, 35]. The classical approaches, such as those proposed by Christensen [36], can be extended to calculate the modulus of hollow particle-filled composites. However, in the case of fly ash-filled composites, the real challenge is that the presence of defects in the wall of cenospheres reduces their mechanical properties [37]. Several cenospheres can be observed in Fig. 5a. Most of these particles show irregular shapes and rough surface. A broken micrograph present in Fig. 5b contains significant porosity in its walls. The defects present in the walls play an important role in determining the strength of these particles. The estimation of these defects is a significant challenge, and experimental data can be the best source for such information because of unique characteristics of each particle [35].

Compressive yield strength

The compressive yield strength of the polyester/cenosphere composites decreases with increasing volume fraction of particles, as given in Table 1. This trend is similar to that reported by previous studies on thin-walled hollow glass particle-filled syntactic foams [38]. The yield strength of polymers containing particles can be expressed by the following equation [39]

$$\sigma_c = \sigma_m(1 - b\phi) \quad (4)$$

where σ_c is the yield strength of the composite, σ_m is the yield strength of the matrix, b is a constant, and ϕ is the volume fraction of particles. According to Eq. 4, for positive values of b the yield strength decreases with increasing particle volume fraction. In polyester/cenosphere composites, the yield strength of all compositions, except one, has decreased with the increasing particle volume fraction. Thus, the magnitude of b is positive, and its value, determined from the data shown in Table 1, is around 0.5.

A decrease in yield strength of polyester/fly ash composites with increasing volume fraction of cenospheres is expected to be related to low fracture strength of cenospheres due to the small wall thickness and weakening of particles by the presence of defects in their walls. In addition, the yield strength of particulate-reinforced polymer matrix composites depends on the particle–matrix interfacial bonding characteristics. An improvement in the interfacial bond strength leads to enhanced strength of the composite [40].

Fly ash-filled low density composites can be used as core materials in sandwich structures. Bending stiffness and compressive strength are important parameters in such applications. The mass of a sandwich beam of a given bending or axial stiffness can be reduced by selecting the material with higher values of E/ρ and E/ρ^2. The variations of E/ρ and E/ρ^2 for polyester/cenosphere composites are plotted against volume fraction of cenospheres and shown in Fig. 6. The maximum values of E/ρ^2 and E/ρ in the current date set are observed to be 2.0×10^{-3} MPa/(kg/m^3)^2 and 2.03 MPa/(kg/m^3), respectively, for composite containing 29.5 vol% cenospheres. The E/ρ^2 and E/ρ values for the neat resin are 1.16×10^{-3} MPa/(kg/m^3)^2 and 0.96 MPa/(kg/m^3), respectively. The values of E/ρ^2 and E/ρ steadily increase with increasing volume fraction of cenospheres. In view of this, it can be concluded that hollow particle-filled composites allow the design of sandwich structures with higher bending and axial stiffness at reduced weight.

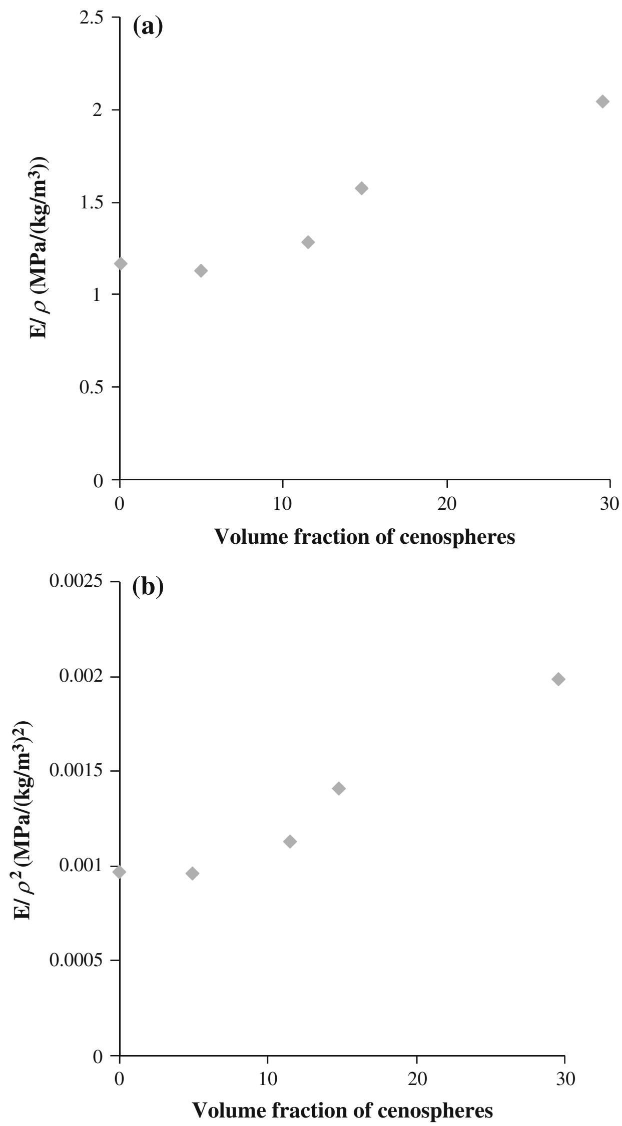

Fig. 6 **a** E/ρ and **b** E/ρ^2 for polyester/fly ash composites

Fracture features

Figure 7a, b show the microstructure of a composite containing 29.5 vol% cenospheres before and after the compression test, respectively. The micrographs are taken on polished sections. The compression test was stopped for the specimen shown in Fig. 7b at 0.1 mm/mm strain to observe the fracture features. It is observed in this micrograph that several cenospheres are broken due to compression. Several cracks in the matrix can also be observed in Fig. 7b as indicated by arrows. These cracks seem to have initiated near cenospheres and propagated into the matrix. The formation of cracks around particles is a general trend which can be observed in several polymer systems containing rigid particles [41]. Increase in cenosphere volume fraction increases the possible fracture locations in composites leading to prominent cracking under compression.

Ultrasonic measurements

The longitudinal and shear velocities of the composites, measured using ultrasonic A-scan technique, are listed in Table 2. For comparison, the longitudinal and shear wave

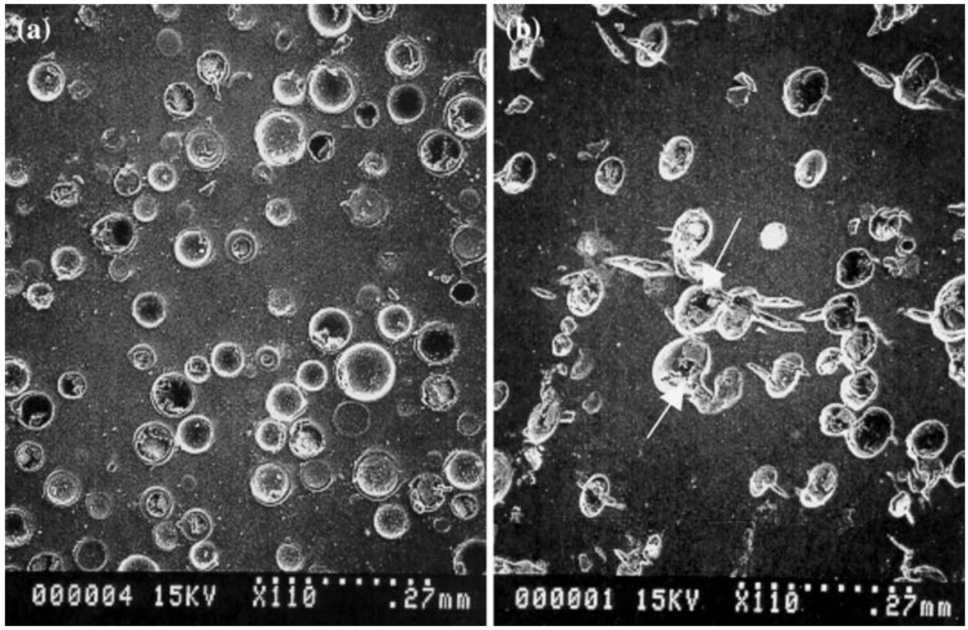

Fig. 7 SEM microphotograph of polymer/cenosphere composites containing 29.5 vol% cenospheres **a** before and **b** after compressive testing. Arrows show broken cenospheres and cracks in the vicinity

Table 2 Values of Young's modulus, shear modulus, and bulk modulus calculated from the measured ultrasonic properties

Cenospheres (vol%)	Longitudinal velocity (m/s)	Shear velocity (m/s)	Ultrasonic modulus (GPa)	Shear modulus (GPa)	Bulk modulus (GPa)
0	2321	1128	4.14	1.46	4.46
4.9	2463	1166	4.36	1.70	5.12
11.5	2578	1253	4.82	1.93	5.14
14.8	2573	1233	4.61	1.61	5.43
29.5	2753	1356	5.06	2.02	5.39

velocities in epoxy resin are about 2.5×10^3 and 1.1×10^3 m/s, respectively [42]. These values are close to those measured for the neat polyester resin, also shown in Table 2. It is observed that the ultrasonic wave velocities in polyester/cenosphere composites increase with increasing volume fraction of cenospheres. The longitudinal and shear wave velocities can be used to calculate various elastic constants of materials using Eq. 5 [42]:

$$v = \left[\left(1 - 2\left(\frac{V_S}{V_L}\right)^2\right) \Big/ \left(2 - 2\left(\frac{V_S}{V_L}\right)^2\right)\right] \quad (5a)$$

$$G = \rho V_S^2 \times 10^{-6} \quad (5b)$$

$$K = \left(\rho V_L^2 - \frac{4}{3} G\right) \quad (5c)$$

$$E = \frac{(\rho V_L^2)(1 - 2v)(1 + v)}{(1 - v)} \times 10^{-6} \quad (5d)$$

where v, G, K, and E represent Poisson's ratio, the shear modulus, the bulk modulus, and Young's modulus, respectively. In addition, ρ represents the density of the composite specimen. However, these equations are mainly used for monolithic metallic materials and their applicability for composite materials is limited.

A comparison of Young's moduli of various composites, calculated using the ultrasonic velocities, shows that the moduli increases with an increase in the volume fraction of cenospheres, as given in Table 2. The moduli calculated from ultrasonic measurements are much higher compared to the experimentally measured values for the corresponding composite compositions. Some of the recent studies have suggested that in particulate composites the ultrasonic modulus is close to the dynamic modulus that is obtained at high strain rates measured by techniques such as Split-Hopkinson pressure bar [42]. The dynamic modulus is higher than quasi-static modulus and depends upon the strain rate until a certain maximum strain rate is reached [43]. In these studies, the modulus value that becomes insensitive to further increase in the strain rate is reported to correspond to the values measured from the ultrasonic data.

Figure 8 compares the compressive and ultrasonic modulus values for composites, normalized with the modulus of the neat resin measured by the respective

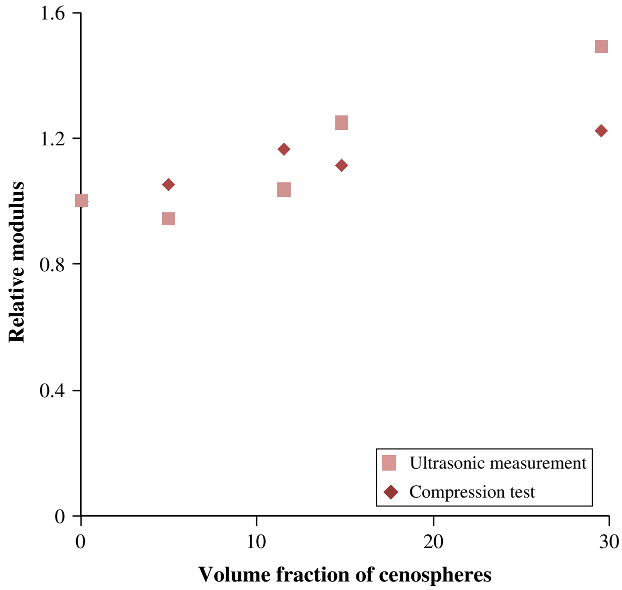

Fig. 8 Modulus of composite materials normalized with the modulus of the matrix resin

methods. It is observed that the relative modulus values measured using both methods are close to each other with less than 10% difference. This provides additional information that the main source of difference is the ultrasonic response of the polymeric resin. Presence of viscoelasticity in polymers may be a reason contributing to such a difference and needs further study. It is also noticed in Fig. 8 that the trendlines for the relative ultrasonic and compressive modulus values cross over. However, because of a small difference (less than 10%) in the values this observation is not considered conclusive.

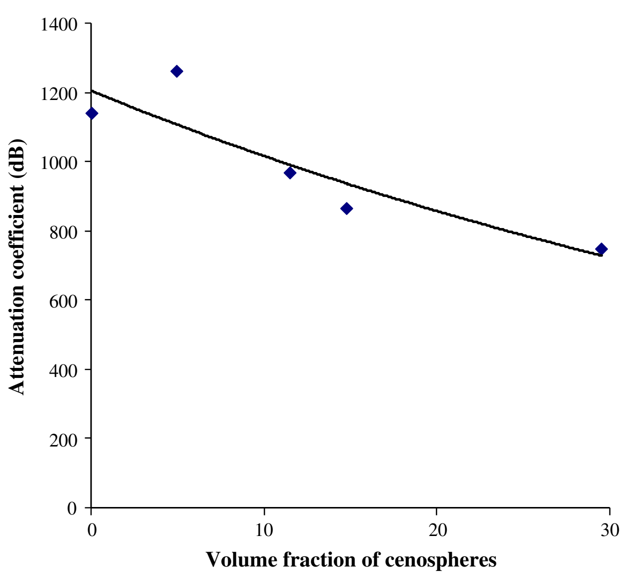

Fig. 9 Ultrasonic attenuation coefficient of composites

Figure 9 shows that the attenuation coefficient decreases with increasing cenosphere volume fraction in composites. This indicates that the attenuation coefficient of fly ash is lower than that of the polyester, implying that the velocity of ultrasound is higher in cenospheres than in the polyester matrix. These results are specific to the parameters of this study and depend on the particle size, test frequency, volume fraction range, and particle/matrix interfacial strength. In the same material system, a change in any of these parameters can affect the results.

Conclusions

Polyester/fly ash composites are fabricated and characterized in this study. The composite material was fabricated by releasing cenospheres in a tube filled with polyester resin. Such fabrication method resulted in functionally graded structure where cenosphere volume fraction varied from 0 to 29.5 vol% in the composite. Test specimens were obtained from various sections of the graded composite to have different cenosphere volume fractions. Compression and ultrasonic tests were conducted on these specimens.

Results show that the compressive modulus increases and compressive strength decreases as the volume fraction of fly ash increases in the composite. However, numerous defects present in fly ash particles contribute to their early fracture and reduce the strength of the composite materials. The increase in the specific modulus is found to be up to 110%, whereas the decrease in the strength is found to be up to 22% compared to the neat resin. This result shows that considerable weight saving in structures can be obtained by using polyester/fly ash syntactic foams. From the trends of the modulus change, it also seems possible that higher loading of cenospheres can make composites lighter and can further increase the modulus. Functionally graded microstructure containing a volume fraction gradient would contain a gradient in the modulus along the specimen height as indicated by the results on various sectioned specimens.

The modulus values computed by the ultrasonic measurements and measured by compression tests showed significant difference. However, the difference in the normalized modulus (modulus of composite/modulus of resin) obtained from both these methods was less than 10% for each composite. This result shows that the measurement technique is not optimized for polymers, where viscoelasticity may be playing an important role in determining material properties.

The study shows that fly ash can be used as a filler to enhance the mechanical properties and to reduce the density of polyester matrix composite.

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