Foam Sandwich Composites with Cyanate Ester Based Syntactic Foam as Core and Carbon-Cyanate Ester as Skin: Processing and Properties

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ABSTRACT: Foam sandwich composites were processed using cyanate ester-based syntactic foam as core and carbon fabric-cyanate ester composite as skin. They were processed by a one-step compression-molding technique. The mechanical performance of the sandwich composites was evaluated in terms of flatwise tensile strength (FTS), flatwise compressive strength, and edgewise compressive strength. The dependency of these properties on the core composition was investigated. FTS initially increased with the increase in resin content of the syntactic foam core. However, higher resin content in the core led to a diminution in FTS due to high void content. The flatwise compressive strength and edgewise compressive strength and the corresponding moduli values showed an increasing trend with increase in resin content of the core despite the presence of voids at high resin content. The failure modes of the composites under different loading conditions have been examined. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 1366–1374, 2008

Key words: cyanate ester; syntactic foams; foam sandwich composites; lightweight composites

INTRODUCTION

Sandwich composites are widely used in marine, aerospace, and other structural applications because of their high stiffness and strength and low-specific gravity. Their areas of application include various types of transportation vehicles and packaging. They consist of two thin, but stiff skins attached to a lightweight core.[1,2] The skins are usually made of glass- or carbon fabric-reinforced composites of a suitable resin. The core is made of cellular or syntactic foam of various resin system. Recently, syntactic foams have gained considerable importance as core materials in sandwich composites. Syntactic foams are composite materials consisting of hollow microspheres that are dispersed in a resinous matrix. These microspheres are commonly made from inorganic materials such as glass and silica and polymeric materials such as epoxy resin, unsaturated polyester resin, silicone resin, phenolics, polyvinylalcohol, polyvinyl chloride, polypropylene, and polystyrene. The important polymer systems used in syntactic foams are epoxy resin, phenolic resin, polystyrene, etc.[3–14] The choice of matrix depends on the desired properties and applications. A suitable combination of matrix and microballoon materials at specified volume fractions can lead to syntactic foam of desired properties.[2]

Among various foam materials, syntactic foams have an excellent combination of compressive strength, low density, low radar detectability, and low-moisture absorption coefficient.[10] In sandwich composites, the mechanical properties of syntactic foam-based cores are several orders of magnitude higher than those of the traditional foams.[11] The closed cell structure has the advantage of continuous contact between the skin and the core, providing better interfacial strength compared to cellular foam cores used in sandwich constructions. Sandwich composites with different densities can be fabricated either by simply varying the volume percentage of resin and microballoon or by using microballoons of different shell thickness. These features make the syntactic foams better candidate as core material for sandwich composites in aerospace and other structural applications.[12]

Highly damage tolerant sandwich constructions can be obtained by using carbon fiber-reinforced plastic (CFRP) as skins and a syntactic foam as core.[2] CFRP skins, which are the structural backbones, provide high-specific strength and stiffness to the sandwich. The syntactic foam core provides excellent shear tie between the skins. It supports the skin against buckling, localizes the impact damage, and absorbs energy through a microballoon-crushing mechanism.[11] Although several resin systems have been attempted for forming the foam core, there are only few reports on the use of cyanate ester for this purpose. In this work,

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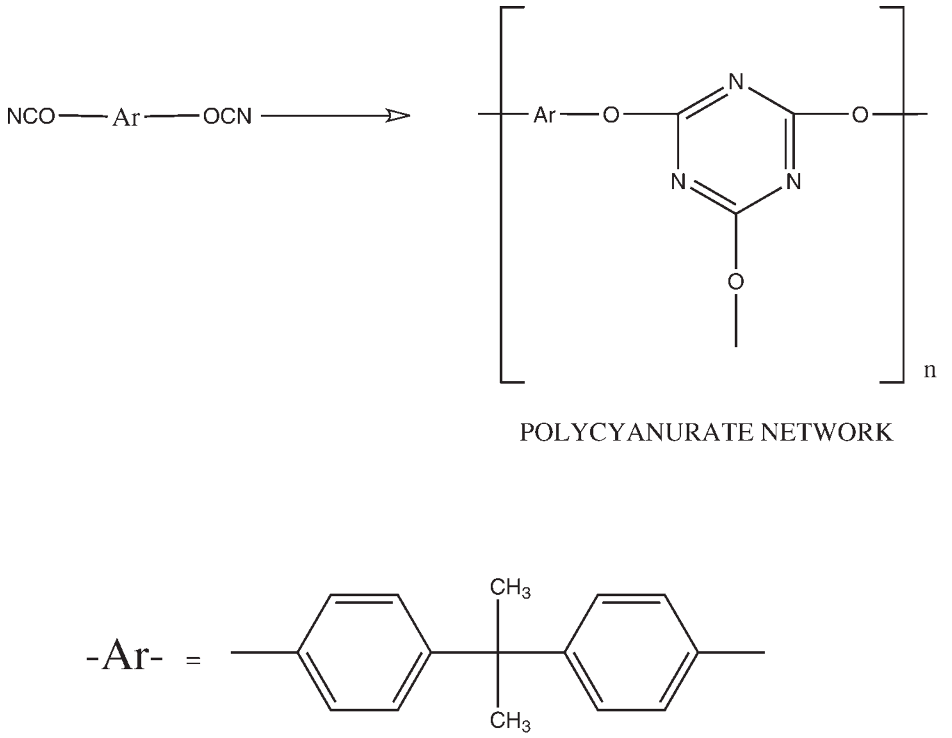

Scheme 1 Polymerization reaction of cyanate ester.

we have explored cyanate ester to constitute both the core and skin composites of the sandwich composites. Cyanate ester is known for its built-in toughness, microcrack resistance, and ease of processing. Cyanate esters cure via addition polymerization to produce a heterocyclic ring referred to as a polycyanurate network as shown in Scheme 1. This polycyanurate network is responsible for the excellent properties of cyanate ester.[15] A large variety of commercial dicyanates are available, and the most versatile among them being bisphenol A dicyanate (BACY), which was used as matrix in the syntactic foam core in this study. A major advantage of using cyanate ester as matrix is that there is no adhesive required for bonding the skin to the core due to the inherent adhesive property of the resin. They have higher T_g in comparison with epoxy matrices. All these justify the use of cyanate ester-based syntactic foam as core in sandwich composites envisaged as lightweight structures for possible aerospace applications.

The mechanical properties of syntactic foams have been extensively studied by various researchers. In a previous study, we have reported the mechanical properties of syntactic foams of cyanate ester.[16] Sandwich structures with syntactic foam as core has also been reported. Thus, Sankaran et al.[17] have developed cyanate ester-based syntactic foam as core for E-glass sandwich composites for radome applications. Gupta et al.[2,10,12,18,19] have studied the effect of microballoon radius ratio and composition on the properties of sandwich composites. They also reported the enhancement of energy absorption by way of nanoclay incorporation in syntactic foams for use as core of sandwich composites.[20] Post impact damage and residual strength of syntactic foam-based sandwich composites have been studied by Hiel et al.[11] Elastic design of syntactic foam sandwiches has been done by Bardella and Genna.[21] Corigliano et al.[1] have reported the experimental characterization and numerical simulation of sandwich composites with syntactic foam as core and glass fabric as skin.

The objective of this work is to develop sandwich composites with cyanate ester-based syntactic foam as core and to investigate the effect of composition of syntactic foam core on the mechanical properties of the sandwich composites. The external facings of the sandwich (skins) are made of carbon fabric/cyanate ester composites. Sandwich composites with different core compositions were processed. The mechanical performance of the sandwich composites has been evaluated in terms of flatwise tensile strength (FTS), flatwise compressive strength, and edgewise compressive strength. The fracture features of the composites under different loading conditions are also examined.

EXPERIMENTAL

Materials

BACY [2,2-bis(4-cyanatophenyl) propane] with a melting point of 79°C supplied by Lonza

TABLE I
The Density and Properties of K-37 Glass Microballoons

Microballoon size distribution (μm, by volume)					
10th percentile	50th percentile	90th percentile	Effective top size (μm)	Target fractional survival (%)	Average true particle density (kg/m^3)
20	45	80	85	90	370

(Switzerland) was used as received. The density of the cured resin system is 1200 kg/m^3. Zinc octate (Amruth Industries, Mumbai, India) and nonyl phenol (Fluka, Switzerland) were used as catalysts for the oligomerization of BACY. Acetone (AR, Nice Chemicals, India) was used as received. Glass microballoon K-37 supplied by 3M Company, USA, were used as closed pore material. The density and particle size distribution of glass microballoon supplied by the manufacturer are given in Table I. Plain weave carbon fabric (T-300) supplied by Toray, Japan, with a density of 1780 kg/m^3 was used as received. A proprietary adhesive developed by adhesive section of VSSC, based on a modified urethane-epoxy adduct that cures at room temperature with an amine hardener, was used for bonding the specimens to aluminum blocks for flatwise tensile testing.

Processing

BACY was oligomerized in the presence of 4-nonyl phenol and zinc octate catalyst (40 : 3, by weight) under thermal conditions. Oligomerization was done by heating BACY in the presence of catalyst and cocatalyst at 90°C. The extent of oligomerization was 45% as estimated by FTIR. Oligomerized cyanate ester was very viscous. Therefore, the required amount of resin was dissolved in acetone for proper dispersion of microballoon. Then, weighed quantity of microballoon was added to it and thoroughly mixed to get a uniform dispersion. Mixing was done gently to avoid breaking of microballoons. Acetone was then removed in a vacuum oven at 60°C. Carbon fabric was impregnated with a solution of cyanate ester in acetone. It was then dried at room temperature for 18 h. The prepreg composition was maintained at carbon fabric: cyanate ester = 60 : 40 (by weight). The prepregs were then cut into a square of 100 mm side. The average thickness of each skin was 0.5 mm. The skin and cyanate ester microballoon mixture were arranged in such a manner that the cyanate ester-microballoon mixture was placed in between two prepreg plies. It was then compression molded to the required thickness. The curing was done according to the following cure schedule: 100°C (1/2 h), 125°C (1/2 h), 150°C (1/2 h), 200°C (1 h), and 250°C (2 h). The physical properties such as density and volume fraction of the constituents were determined. The average thickness of the sandwich composites is 14 mm.

Determination of void content

The void content in the sandwich composites was calculated using eq. (1).

$$\text{Void percentage} = \frac{\left[(V_{SA} - V_{SK}) - \left[(W_{SA} - W_{SK}) \times W_R/\rho_R + (W_{SA} - W_{SK}) \times W_M/\rho_M\right]\right] \times 100}{(V_{SA} - V_{SK})} \quad (1)$$

where V_{SA} and W_{SA} are the volume and weight of sandwich block, V_{SK} and W_{SK} are the volume and weight of sandwich skin, W_R and W_M are the weight fractions of resin and microballoon, and ρ_R and ρ_M are the densities of resin and microballoon, respectively.

Specimen preparation and mechanical testing of the sandwich composites

The mechanical properties of the sandwich composites were determined in Universal Testing Machine, Instron Model 4202.

Flatwise tensile strength

ASTM standard C 297-94 was used for the determination of flatwise tensile strength (FTS). Samples of dimension 25 × 25 × 14 (mm) were cut from the processed sandwich composites. The loading blocks of aluminum of dimension 25 × 25 × 25 mm were bonded to the facings of the test specimen using a proprietary epoxy adhesive. The crosshead speed was 0.50 mm/min.

TABLE II
The Density and Composition of the Sandwich Composites

Sample code	Core composition (in volume percentage) Cyanate ester	Microballoon	Void	Overall density (kg/m^3)
SA1	10.9	82.1	7.0	560
SA2	16.3	79.4	4.3	610
SA3	21.1	68.3	10.6	630
SA4	26.5	57.4	16.1	640
SA5	33.0	46.0	21.0	680

Edgewise compressive strength

ASTM C 364-99 was followed for determining edgewise compressive strength. The dimension of the specimen was 30 × 27 × 14 mm. The rate of cross head movement was 0.50 mm/min.

Flatwise compressive strength

The standard used was ASTM C 365-00. The testing was done at a crosshead speed of 0.50 mm/min. Samples of dimension 25 × 25 × 14 mm were used for testing.

Three specimens were tested in all the above three cases for each sandwich composites, and the average value is reported.

RESULTS AND DISCUSSION

Table II gives the composition and physical characteristics of the various foam sandwich composites. As the resin concentration in the core increases, the density of the sandwich composite increases. The void content also increases with resin enrichment in the core. The void content in composites is an important factor affecting the mechanical properties. This is true with syntactic foams too. There are different possibilities for void formation in syntactic foams. In some cases, where the microballoon concentration is very high, insufficient resin in between the microballoons leads to voids. Also, during the mechanical mixing of microballoon and resin, there is a possibility for entrapment of air. This entrapped air could act as voids in the foam structure. The void content in the syntactic foam core is inevitable even after the application of vacuum to the resin-microballoon mixture as it is not possible to completely remove the entrapped air. In some other cases, a thin film of resin may surround a cluster of microballoons preventing penetration of resin into this cluster. The void content has been calculated using eq. (1). At high-resin volume percentage, the void content is very high. This has been attributed to the partial oozing of resin and microballoon from the core during compression molding. This points out the practical difficulty in the processing of syntactic foam sandwich composites with high resin content using the one-step compression-molding method. The void content makes the syntactic foam core a three-phase system. The observed density of the sandwich composites was lower than the theoretical values calculated from the rule of mixtures due to the presence of voids. Sandwich composites based on syntactic foam as core with a void content up to 31% have been reported in literature.[22] Earlier studies on syntactic foam showed that the presence of voids in the core adversely affects the mechanical properties.[3,16] The presence of voids decreased the flexural and compressive strength of cyanate ester syntactic foams.[16]

Mechanical properties of the sandwich composites

The mechanical properties of sandwich composites are greatly influenced by the properties of skin and core. The skin composition was fixed throughout the study (i.e., carbon fabric : cyanate ester = 60 : 40, by weight). The only change was in the composition of the core by varying the volume percentages of resin and microballoon. Because the properties of syntactic foams depend on the density, which, in turn, depends on volume percentages of microballoon and resin, these factors are also likely to determine the properties of the resulting sandwich composites. Because these sandwich composites could find use as structural materials in aerospace applications, it is required that the mechanical properties of the sandwich composites be optimized by way of composition of the core. Hence, this aspect was examined in this study. A detailed investigation of the mechanical properties of the sandwich composites and their dependencies on the core composition form the focus of this study.

Flatwise tensile strength

Flatwise tensile strength (FTS) provides information on how well the facings are bonded to the core. It also reflects the FTS of the core, which is a critical factor in the design of sandwich structures. The test

TABLE III
Flatwise Tensile Strength and Failure Mode of the Sandwich Composites

Sample code	Flatwise tensile strength (MPa)	Specific flatwise–tensile strength (MPa cm^3 g^{-1})	Failure mode
SA1	1.4 ± 0.3	2.5 ± 0.5	Core failure
SA2	3.0 ± 0.3	4.9 ± 0.5	Skin-core debonding
SA3	3.8 ± 0.2	6.0 ± 0.3	Skin-core debonding
SA4	4.2 ± 0.2	6.6 ± 0.3	Skin-core debonding
SA5	2.2 ± 0.2	3.2 ± 0.3	Skin-core debonding

consists of subjecting the sandwich composite to a tensile load normal to the plane of the sandwich, such load being transmitted to the sandwich through thick loading blocks bonded to the sandwich facings. In the case of syntactic foam-based sandwich composites, the strength of the core as well as the interfacial strength between skin and core are of great importance in determining the FTS.

The FTS, specific FTS (ratio of FTS to density), and failure modes of the sandwich composites are given in Table III. The FTS of the sandwich composites increased with increase in concentration of resin in the core. However, the FTS showed a diminishing trend on enhancing the resin content beyond 26.5%. Two types of failure modes were observed when the sandwich composites were subjected to flatwise tensile loading. The failure mode was found to be core failure in the case of SA1 having the lowest resin content. The low-resin concentration in SA1 caused the resin-to-microballoon bonding weaker than skin-to-core bonding. Therefore, the failure of core took place in preference to skin-to-core debonding when a tensile load is applied normal to the plane of the sandwich. Skin-to-core debonding has been the failure mode in the rest of the cases. Here, the high-resin content makes the resin-to-microballoon bonding stronger than skin-to-core bonding. Figure 1(a) shows the core failure in the case of SA1, and Figure 1(b) shows the skin-core debonding in the case of SA-3. The low value of FTS for SA5 is attributed to the high void content. The high void content has resulted in a reduction in area of contact between the skin and the core at the skin-core interface. As a result, the skin is peeled off easier from the core. The specific FTS values also manifested a similar trend.

Typical stress–strain curves for SA-1 and SA-5 tested under flatwise tensile loading are shown in Figure 2(a,b). Although the failure modes in both cases were different, the nature of stress–strain curve is similar. In both cases, the point up to A corresponds to the region of elastic deformation. The point A refers to the point of core failure and skin-to-core debonding in SA-1 and SA-5, respectively. The systematic increase in the FTS of the sandwiches up to SA-4 implies a stronger interfacial bond with increase in resin content of the core.

Flatwise compressive strength

This test method essentially covers the estimation of compressive strength and modulus of sandwich cores, which are the fundamental mechanical properties of sandwich composites. It was observed that large amounts of debris are generated in compressive fracture, whereas tensile fracture shows only a

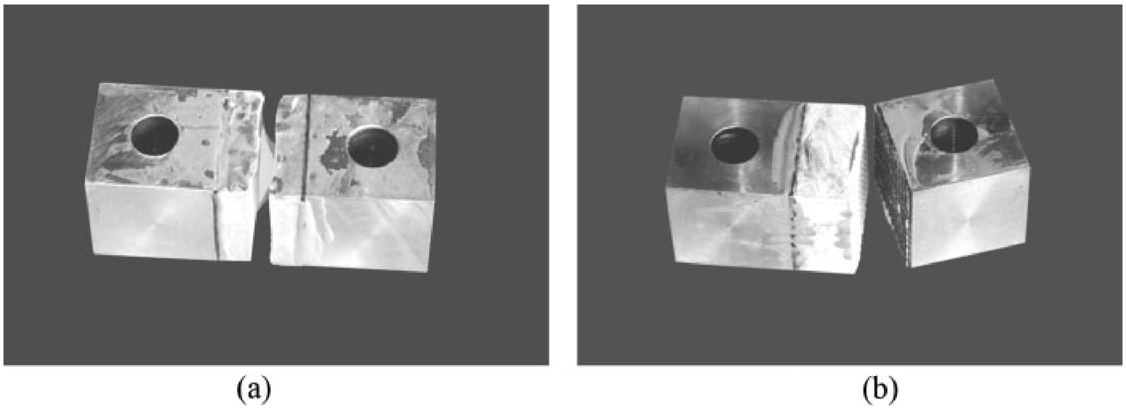
(a) (b)

Figure 1 Sandwich composites failed under flatwise tensile load. (a) Failure of core in the case of SA1. (b) Skin-to-core debonding in the sample SA3.

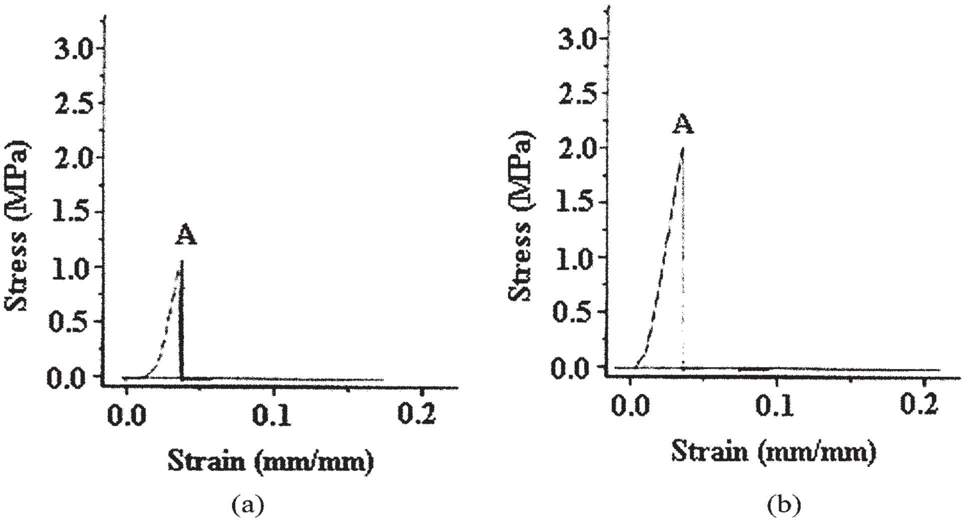

Figure 2 Typical stress–strain curve of SA-1 (a) and SA-5 (b) on flatwise tensile loading.

small amount of broken pieces on the fracture surface. When a syntactic foam core-based sandwich is subjected to compressive loading, the microballoon breakage is the prominent failure phenomenon. The contribution of skin in load bearing is negligible in this case. The flatwise compressive strength values are found to increase with increase in resin concentration in the core. This is due to the better load bearing property of the resin compared to that of the low strength microballoons. The flatwise compressive strength was not affected by the high void content as in the case of SA5. This shows that the load-withstanding ability of resin overcomes the likely adverse effect of high void content in this case. The corresponding specific strength values also followed the same trend. The flatwise compressive modulus and the corresponding specific modulus values increased with increase in resin concentration. However, the high void content in the case of sample SA5 caused a slight diminution in the modulus.

To explain the various failure features, the photographs of the sandwich samples subjected to flatwise compressive testing have been presented in Figure 3. During flatwise compression, it is the material at the central part of the sandwich that is likely to carry more load, due to stress concentration from both sides. In SA1 (where the microballoon volume percentage is high) when a compressive load is applied, the microballoons absorb the energy through uniform breaking. Therefore, no visible cracks were seen in the tested sample [Fig. 3(a)]. In the case of SA3, which is having higher resin content than SA1, the stresses from both sides concentrate on the central part generating a horizontal crack on the sandwich core [Fig. 3(b)]. In the case of SA5, (with the highest void content) when a compressive load is applied, the voids tend to close and, as a result, no outburst of the core is observed [Fig. 3(c)].

The nature of the stress–strain curves for the sandwich composites under flatwise compressive loading was found to be the same in all the cases. The typical stress–strain curves for SA-2 are depicted in Figure 4. The stress values initially increase linearly with strain, attain a maximum, and then become almost constant. The maximum stress value in the curve is taken as the ultimate flatwise compressive strength. The region up to the peak stress

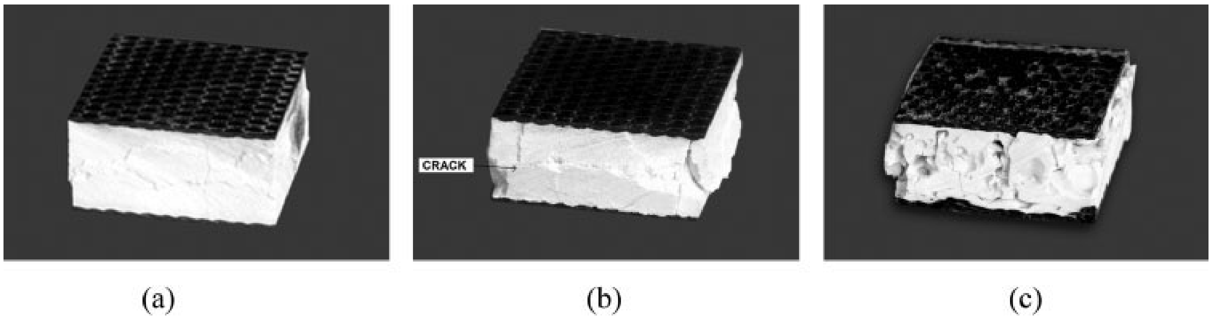

Figure 3 Different responses of the sandwich composites to flatwise compressive load.

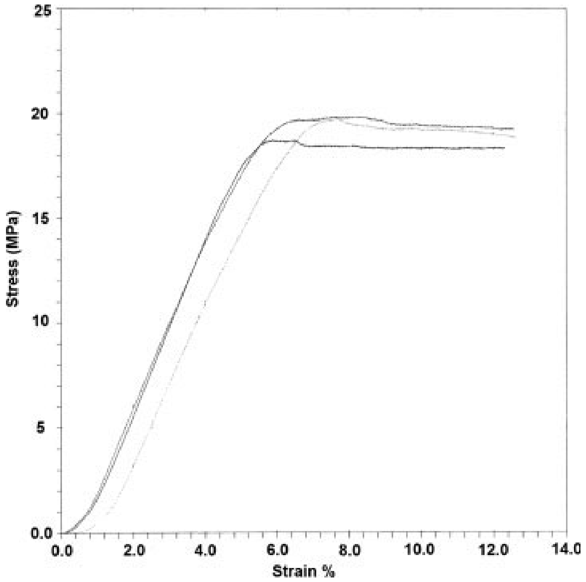

Figure 4 Stress–strain curves for SA2 under flatwise compressive loading.

corresponds to the elastic deformation regime of the syntactic foams. The slope of initial linear portion of the stress–strain curve is taken as the modulus of the composites. Although the maximum stress value for the sandwich composites is shown by SA-5 under flatwise compression, it is attained at a high percentage of strain, due to the uniform closing of voids in addition to the crushing of microballoons. This leads to a reduction in slope of the stress–strain curve for SA-5, thereby causing a slight decrease in modulus value.

The flatwise compressive strength values reported in this study (Table IV) are higher than those reported for sandwich composites composed of epoxy resin-based syntactic foam core (average value = 20 MPa) with a core density of 550 kg/m^3.[1,22] However, the modulus values are lower in the present case.[22]

Edgewise compressive strength

This test involves the determination of compressive strength of sandwich constructions in a direction parallel to the sandwich-facing plane. The edgewise compressive strength and modulus values of the sandwich composites are given in Table V. The edgewise compressive strength increased with the increase in concentration of cyanate ester in the core. There is not much increase in the strength value for SA5 due to high void content. The edgewise compressive strength values are lower than flatwise compressive strength, but the modulus values for edgewise compression are comparatively higher. This is due to the fact that unlike in the case of flatwise compressive testing, during edgewise compressive testing, a good proportion of the load is carried by the edges of the skin. When the stress increases, the microballoons in these two edge faces undergo crushing, and the load is transferred to the skin. The tensile component of the compressive forces in the lateral direction causes the skin to bulge outward. Therefore, further application of load results in the peeling of skin from the core. After the peeling of skin, the failure of core is initiated by a crack parallel to the plane of the skin as shown in Figure 5. Thus, the failure mode in the case of edgewise compressive strength is through peeling of skin from the core followed by failure of the core. In the case of compressive loading on bare syntactic foams, the compression takes place in a slow manner with the breaking of microballoons. Even at the high microballoon volume fraction, the syntactic foams are not completely broken. But in the case of sandwich structures with high microballoon content, the syntactic foam-based core is completely destroyed. This is due to the sudden transfer of load to the syntactic foam core as the skin gives way.

Previous reports show that the nature of the reinforcement that forms the skin plays an important role in edgewise compressive properties of the sandwich composites.[19] Woldesenbet et al.[12] reported that the failure during edgewise compression occurs by the breaking of skin, followed by failure of core for sandwich composites with E-glass skin. In the present case, the skin-to-core binding strength is inferior to the longitudinal compressive strength of the skin composite material.

TABLE IV
Flatwise Compressive Strength and Modulus of the Sandwich Composites

Sample code	Ultimate flatwise compressive strength (MPa)	Flatwise compressive modulus (MPa)	Specific flatwise compressive strength (MPa cm^3 g^{-1})	Specific flatwise compressive modulus (MPa cm^3 g^{-1})
SA1	16 ± 2	155 ± 3	28 ± 4	276 ± 5
SA2	19 ± 1	181 ± 2	33 ± 2	297 ± 4
SA3	21 ± 1	182 ± 1	33 ± 2	289 ± 2
SA4	27 ± 1	381 ± 1	42 ± 2	595 ± 2
SA5	42 ± 5	374 ± 2	62 ± 7	550 ± 4

TABLE V
Edgewise Compressive Strength and Modulus of the Sandwich Composites

Sample code	Ultimate edgewise compressive strength (MPa)	Edgewise compressive modulus (MPa)	Specific edgewise compressive strength (MPa cm^3 g^{-1})	Specific edgewise compressive modulus (MPa cm^3 g^{-1})
SA1	12.5 ± 0.5	620 ± 15	22.3 ± 0.9	1100 ± 25
SA2	14.8 ± 0.5	640 ± 20	24.2 ± 0.8	1050 ± 30
SA3	17.8 ± 0.5	720 ± 20	28.3 ± 0.8	1140 ± 30
SA4	19.0 ± 0.4	750 ± 40	29.7 ± 0.6	1170 ± 60
SA5	19.4 ± 0.4	540 ± 10	28.5 ± 0.6	790 ± 15

Figure 5 Photograph of sandwich composite failed under edgewise compressive loading.

The stress–strain curve for the sandwich composites under edgewise compression can be related to visual observation for drawing conclusions regarding the failure modes. Figure 6(a,b) shows the stress–strain curve for SA-1 and SA-3, respectively, which exhibited different failure features. In the case of both SA-1 and SA-3, the region from A to B refers to the elastic deformation of the sample. From B, the slow delamination of the skin from the core takes place for SA-1. This continues up to C. At C, the skin is fully delaminated, and the load is suddenly transferred to the core. In the case of SA-1, because the resin content is very low, the sudden transfer of load results in catastrofic failure of the core. Whereas in SA-3, after the delamination of the skin, the stress–strain profile is akin to that of a high resin content syntactic foam under compression.[16] In other words, the stress–strain curve after the delamination of skin can be compared to that of syntactic foams under compression. After the delamination of the skin, the stress starts increasing, becomes maximum, and then decreases before becoming almost constant due to the densification of the microballoons. This is the stage where microballoons are crushed exposing their internal hollow volume. The maximum stress value in the curve is taken as the ultimate edgewise compressive strength of the sandwich composites. The edgewise compressive modulus and the corresponding specific modulus increase with resin content, but exhibit lower value for the highest resin-containing system (SA-5), due to the presence of high void content.

CONCLUSIONS

Sandwich composites with cyanate ester syntactic foam as core can be processed using a one-step compression-molding method. Sandwich composites with different core compositions and property profiles are realizable by varying the volume percentage of microballoon in the core. A good skin-to-core adhesion could be achieved due to the inherent

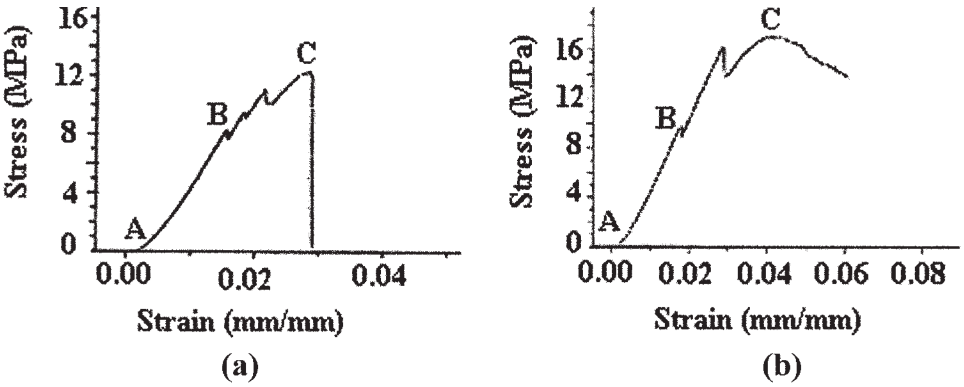

Figure 6 Typical stress–strain curve for SA-1 (a) and SA-3 (b) under edgewise compressive loading

adhesive property of cyanate ester. Moderate resin content is better in yielding low void content core.

The composition of the foam core influences the FTS, flatwise compressive strength, and edgewise compressive strength of the sandwich composites. FTS increases with the increase in resin content of the core. However, the presence of voids at high resin concentration reverses the trend. The failure mode in the case of flatwise tensile loading changes from core failure in the case of low resin content system (SA-1) to skin-to-core debonding in the case of high resin content systems (SA-2 to SA-5). The flatwise compressive strength and edgewise compressive strength and the corresponding modulus values increase with resin content of the core and are not much affected by the presence of voids at high resin loading. The failure mode under edgewise compression occurs by skin delamination followed by core crushing. Although, the flatwise compressive modulus and edgewise compressive modulus of the sandwich composites increase with resin content, higher resin content is detrimental for these properties. Depending on the application and strength requirement, the suitable composition of core can be selected. A theoretical modeling of the properties of these composites is under investigation and will be the subject of an ensuing publication.

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