FATIGUE OF PURE AND NANOPHASED SANDWICH COMPOSITES UNDER SHEAR LOADING

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Abstract Sandwich structures are widely used in marine, automotive, and aerospace structures because of their high stiffness and strength to weight ratio. In all of these applications, core plays an important role in controlling the extent of damage in sandwich structures especially when subjected to repetitive dynamic loading. When a sandwich structure is subjected to transverse loads, the face sheets carry bending moments as tensile and compressive stresses and the core carries transverse forces as shear stresses. The core is typically the weakest part of the structure and is first to fail in shear. Hence strengthening of core materials will essentially enhance the overall performance of sandwich structures. In this study foam core materials have been strengthened with the infusion of acicular nanoparticles such as carbon nanotubes and carbon nanofibers in the polymer precursor. This infusion has been carried through a sonic cavitation process. Once the core was modified, sandwich composites were fabricated through a traditional resin transfer molding (RTM) process. Shear fatigue behavior of sandwich composites having both pure and nanophased polyurethane foams as core materials have been investigated. The density of the core materials was identical in both cases. Static shear tests reveal that nanophased foams are more ductile, have higher strength and stiffness, and better crack propagation resistance when compared to pure foams. Shear fatigue tests were conducted at room temperature, at a frequency of 3 Hz and at a stress ratio, $R = 0.1$. S–N curves were generated and shear fatigue characteristics were determined. The number of cycles to failure for the nanophased sandwich was substantially higher than that of the neat ones. SEM micrographs show that the cell structures of nanophased polyurethane foams are stronger and larger in size with thicker walls and edges. These stronger cell structures subsequently strengthen the sub interfaces when the sandwich composite is fabricated. The high intrinsic toughness of the sub interface delays the initiation of fatigue cracks and thereby increases the fatigue life of the nanophased sandwich composites. There was no volume change for either the neat or the nanophased foam during shear deformation, and the material failed by shearing in the vicinity of the centerline of the specimen along

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the longitudinal axis. In both cases numerous 45◦ shear cracks formed across the width and the cracks traversed through the entire thickness of the specimen signaling the final failure event during fatigue.

Keywords: nanoparticles, polyurethane foam, sandwich, fatigue, shear.

1. INTRODUCTION

In most of the applications, sandwich beams are subjected to repetitive transverse loading. Because of this, sandwich beams constituents are subjected to various kinds of loading. The face sheets exhibits membrane tension/ compression behavior and the core exhibit the most critical stress i.e. pure shear [1]. The most common failure of sandwich construction is the core shear failure that occurs when the shear stress reaches its critical value [2]. Many researchers have extensively studied sandwich structures emphasing face sheets and it is generally agreed that behavior of face sheets is well known. On the other hand, comparatively very less has been done to study the core behavior of sandwich structures. It has been demonstrated over time [3, 4] that either flexural or shear loading, of core basically controls the failure of the sandwich. Studies [5, 6] on the flexural behavior of foam core sandwiches showed that numerous cracks initiated in the core sub-interface area. These cracks grew together and propagated on the compression side of the beam, immediately below the sub-interface. Cracks then propagated in the core, parallel to the beam. Shipsha et al. [7] performed tests on H100 foams and found that crack continuously propagated along the interface in the core material below the resin rich cells with diagonal secondary fracture cracks. In all these cases, the core shear stresses produced global deformation. Therefore it is understood that if the core shear properties can be enhanced, the overall performance of the sandwich structure will be improved.

Also in recent years, infusion of nanoparticles into polymer foam cores showed considerable enhancement in mechanical properties [8–11]. It has been shown that by infusing a small percentage of nanoparticles in the foam core, materials, the static properties of sandwich structures can be improved significantly [5]. Similar trend has been shown when nanophased foam core sandwich structure tested under compression and high strain loadings.

In this study, polyurethane foam core properties have been modified by dispersing nanoparticles. Modified core materials were then used to fabricate sandwich composites using the VARTM process. In parallel, control sandwich panels were also fabricated using core materials without any particle infusion. Quasi-static and fatigue behavior of these sandwich composites have been studied under shear loading.

2. MANUFACTURING OF SANDWICH COMPOSITES

The following materials systems, as shown in Table 1, were used for making various sandwich panels:

Face sheet (skin)			Core materials	
Fiber		Resin	Foam	Nanoparticles $(1\%$ w)
No. of	Tvpe	Epoxy SC 15	Polyisocyanurate	CNF
layers 3	S-2 Glass 240 F		with density	Dia: 200 nm,
			80 kg/m^3	aspect ratio: 500

Table 1. Materials used in the sandwich composites.

The manufacturing of sandwich composites was carried out in three steps; the first was the dispersion of nanoparticles into liquid polyurethane, the second, casting of the foam (core materials) and the final, fabrication of sandwich panels. In addition, a number of sandwich panels were also made with pure polyurethane foam without having any nanoparticle infusion.

2.1 Dispersion of nanoparticles into liquid polyurethane

The liquid foam used in this investigation is Polyisocyanurate. It has two parts, part A (Diphenylmethane Diisocynate) and part B (Flurocarbon blown Polyol). Part-A was selected for infusion of nanoparticles since it is less reactive than part-B. Carbon nanofibers (CNF) nanoparticles were first carefully measured along with Part-A to have a 1% loading by weight. The mixing was carried out in a Sonic Vibra Cell ultrasonic liquid processor (Ti-horn, 20 kHz, 100W/cm2) as shown in Figure 1 for about 30 minutes at 5◦C temperature. At this time it was found that nanoparticles were uniformly dispersed in Part-A. In order to avoid temperature rise during sonication, external cooling system was used. After infusion of nanoparticles, the modified Part-A was mixed with Part-B at a ratio of 48:52 by using a high speed mechanical stirrer. The mixture was then cast in a steel rectangular mold as shown in Figure 2. The mold was heated to about 65◦C prior to pouring the mixture. After about 8–9 hours the cast foam was demolded and post cured for about 20 minutes at 80◦F.

2.2 Sandwich fabrication

The resin transfer molding (RTM) process was employed to fabricate the sandwich panels. Since both the top and bottom face sheets had to be infused simultaneously, a co-injection resin infusion process was used to process the sandwich composites. A schematic of the co-injection process is shown in Figure 3. Dry fabric preforms with required orientations were first laid out on the top of a flat aluminum tool. The fabric used in this investigation was plane

Figure 1. Ultrasound mixing. *Figure 2.* Casting of foam in a rectangular mould.

Figure 3. CIRT process for sandwich manufacturing.

weave S2-Glass fibers. Three layers of fabrics were used for each face sheet. The core was then placed on the top of the bottom face sheet fabrics, and upon which the preforms for the top face sheets were stacked. Two types of core materials were used during the fabrication; one was pure polyurethane, and the other was doped with CNF. After stacking, infusion lines were installed and the assembly was vacuum bagged. Before infusion the system was debulked for several hours. SC-15 epoxy resin (Part-A: epoxy, Part-B: Hardener, Alkyl Polyamine) manufactured by Applied Poleramic, Inc. was used in this study. After the resin infusion, the vacuum was kept on until the complete cure took place. No additional adhesives were used for the skin-core bonding, since it developed during the cure process. It is to be noted that the surfaces of the nanophased foams had to be sanded prior to setting them up in the RTM mold. This allowed better adhesion between the core and the skin. Several panels were fabricated in this manner, and were machined for shear characterization.

Figure 4. (a) Schematic of shear test fixture. (b) Photograph showing experimental setup.

3. EXPERIMENTAL

3.1 Quasi-static shear tests

Five replicate of pure and nanophased polyurethane foam sandwich specimen of dimension 40 mm \times 160 mm were cut from 12.5 mm thick panels, using a diamond coated steel blade, as per ASTM C273-61 [12] standard test method. The specimen was bonded between the two parallel loading steel plates as shown in the test set up in Figure 4a which shows a schematic of the shear fatigue test fixture. The arrows indicate the direction of loading. The steel plates were truly parallel since a small deviation in parallelism of the loading plates can cause considerable errors in the calculation of the shear strength and shear modulus. A two-part epoxy, Hysol EA 9309.3 NA was used as the adhesive to bond the foam to the steel plates. The epoxy was allowed to cure at room temperature for a minimum of 48 hours prior to testing. The fixture was fitted into a servo hydraulic testing machine (MTS) equipped with a 100kN load cell as shown in Figure 4b. The tests were conducted at room temperature in displacement control at a crosshead speed of 1.27 mm/min. A Keyence laser displacement unit coupled to a RD-50R controller was installed to measure the sliding movement of the loading plates relative to each other in the direction parallel to the loading plates.

3.2 Fatigue test

Shear fatigue tests were performed at room temperature under load control on the foam specimens at a load ratio of $R = |P_{min}|/|P_{max}| = 0.1$, using the

MTS machine at a frequency of 3 Hz. The run out cycle number was set at 106 cycles. Fatigue data for each specimen were generated at stress levels of: 90%, 80%, 70%, 60%, and 50% of the static shear strength.

4. SCANNING ELECTRON MICROSCOPY ANALYSIS

Virgin surfaces were examined in a JEOL JSM 5800 scanning electron microscope. The specimens were glued to an aluminum base and coated with gold to prevent charge build-up by the electrons absorbed by the specimen. Micro-structural analysis were performed on both pure and nanophased polyurethane foams.

5. RESULTS AND DISCUSSION

5.1 Static tests

These tests were conducted primarily to obtain strength values for the fatigue tests. The load was applied to the pure and nanophased sandwich specimen via the steel plates as shown in Figure 4a. The laser displacement unit recorded displacement of the moving steel plate relative to the fixed plate. This displacement was used to calculate the shearing strain, γ . Figure 5 shows representative stress-strain (τ - γ) curves for pure and nanophased sandwiches. The failure loads of nanophased specimens were higher then the pure foam sandwich specimens. Small cracks were noticed immediately in the core but upon reaching the critical load, the specimen elongated up to a threshold value whereupon these cracks intensified. Rapid shearing of the core occurred at this stage causing failure from the sub-interface section. Similar trend was also noticed with sandwiches made from pure foam.

The stress strain behavior of both nanophased and pure sandwich composites as seen in Figure 5 is more or less identical except that the nanophased sandwich has higher strength and stiffness. Data from static shear tests are shown in tabular form in Table 2.

The shear strength and shear modulus of the nanophased foam sandwiches were approximately 33% and 19% higher than that of the pure foam sandwiches, respectively. An approximation of the area under the stress-strain curves indicates that the energy absorption capability of the nanophased foam sandwich is almost 30% higher than the pure foam sandwich.

Figure 6 shows a schematic of a foam specimen subjected to static shear load. The foam specimen deformed as shown in Figure 6. The first crack initiated at the free edge in the uppermost section of the specimen adjacent to the epoxy interface. The crack then propagated parallel to the plate for approximately 15 mm after which, it kinked into the core moving diagonally toward the opposite end. In a few specimens free edge effects occurred, i.e. the

Figure 5. Stress-strain curves for pure and nanophased foam sandwich.

Property	Neat polyurethane foam sandwich	1% CNF polyurethane foam sandwich	% Improvement
Shear strength	0.55	0.83	$+33$
(MPa)	0.64	0.81	
	0.66	0.78	
	Ave.: 0.61	Ave.: 0.81	
	Std. Dev. -0.014	Std. Dev -0.02	
Shear modulus	11.7	13.7	$+19$
(Mpa)	11.9	14.9	
	12.4	14.4	
	Ave.: 12.0	Ave.: 14.3	
	Std. Dev. -0.35	Std. Dev -0.32	

Table 2. Static shear test data.

foam specimen tore away from the plate at either of the free ends in the upper corners.

5.2 Fatigue Tests

S–N diagram with normalized shear stress is presented in Figure 7.

Under constant amplitude loading many engineering materials exhibit a plateau in the stress life plot typically beyond about 10^6 cycles, which was also true in this case. Accordingly, the run out cycle number was taken as $10⁶$ cycles. The fatigue limit was found to be about 55% of the ultimate strength for nanophased specimen and 50% for pure specimen. It is seen in Fig 7 that at each of the stress levels, the number of cycles to failure for nano-

Figure 6. Schematic of deformed specimen showing crack propagation.

Figure 7. S–N curves for neat and nanophased specimens. Stress ratio, $R = 0.1$ and frequency $= 3$ Hz.

Figure 8. Side views of a failed nanophased foam sandwich and its schematic.

phased foam was higher than that of pure foam specimen. The damage formation process in both the nanophased foams and pure foams were similar.

Shortly prior to failure, numerous small cracks formed in the foam just below the interface area on the side of the fixed plate (side b). The cracks then coalesce into a more dominant crack, which propagate parallel to the steel plate. In the next stage, the crack kinks at an angle of approximately 45° into the core, advancing towards the moving plate (side a). The crack arrests at the interface area (side a), apparently having insufficient energy at the crack tip to penetrate the cured epoxy. Upon the onset of the first crack, similar cracks appear in the core on side b at fairly equidistant locations along the length of the specimen. Each of these cracks propagates in the same way as the first one until final tearing and separation from the face sheet. Figure 8 shows the damaged surface of nanophased foam specimen.

Figure 9. Micrographs of cell structures: (a) neat foam and (b) nanophased foam.

It is believed that as the resin gets filled into the partially opened cells, it soaks around and down the cell walls and edges. When the resin is cured, these soaked cell materials become stronger than the regular dry foam cells just underneath. A sub-interface is therefore created between these so-called soaked and dry foam cells, which are apparently weaker than the actual coreskin interface mentioned earlier. It is also known that infusion of nanoparticles acts as catalyst and increases the cell size and edge thickness which is shown in SEM micrographs of Figure 9. This might be the reason that formation of cracks in nanophased foams during fatigue is somewhat delayed as a result of more resin absorption in thicker cell walls and edges when compared with pure polyurethane foams.

5.3 SEM analysis

To investigate the microstructural effect of nanoparticles infusion in polyurethane foam, SEM analyses were carried out on both the pure and nanophased foam as shown in Figures 9a and b. The micrographs show that both foams have a fairly uniform distribution of regular cells. The cell sizes are found to be larger for the nanophased polyurethane foam by about 30%.

The cell walls and cell edges of the nanophased foams appear thicker than the pure foam. This was estimated from enlarged photographs (same scale) using a caliper. It was found that on an average of five specimens that the cell wall thickness of the pure and nanophased foams was 3μ m and 5.3μ m while the cross sectional area of the cell edges were approximately $217 \mu m^2$ and 345μ m², respectively. Stronger cell walls and edges eventually make the nanophased foam tougher and less prone to premature failure.

6. SUMMARY

In summary, this study reveals that

1. Nanoparticle infusion improves the static shear strength and stiffness of the sandwich composites by $20-30\%$ over the over their neat counterparts.

- 2. This improvement is carried also to the cyclic loading when the sandwich composites are subjected to shear fatigue.
- 3. The improvement in the performances both at static and cyclic loading comes from the fact that nanoparticle infusion causes significant changes to the cell dimension, thereby strengthening the cell walls and cell edges.
- 4. Changes in the cell geometry and dimension are due to the presence of nanoparticles which apparently work as catalysts and control the rate of formation of $CO₂$ during the blowing reaction.
- 5. There is no significant difference in the failure mechanisms of sandwich composites due to nanoparticle loading, but because of the relatively stronger core materials, the initiation of failure is significantly delayed in nanophased sandwich composites.

REFERENCES

- [1] Zenkert, D., "The Handbook of Sandwich Construction", EMAS Ltd. Solihull, UK, 1995.
- [2] Gdoutos, E.E., Daniel, I.M. and Wang, K.A., "Failure of Cellular Foams under Multiaxial Loading" J. of Comp. Part A, Vol. 33, 2002, pp. 163–176.
- [3] Kulkarni, N., Mahfuz, H., Jeelani, S. and Carlsson, L.A., "Fatigue Crack Growth and Life Prediction of Foam Core Sandwich Composites under Flexural Loading", Composite Structures, Vol. 59, No. 4, March 2003, pp. 499–505.
- [4] Krishnan Kanny, Hassan Mahfuz, Tonnia Thomas and Shaik Jeelani, "Static and Dynamic Charecterization of Polymer Foams Under Shear Loads" J. of Composite Materials, Vol. 38, No. 8, 2004.
- [5] Mahfuz, H., Islam, M.S., Rangari, V.K., Saha, M.C. and Jeelani, S., "Response of Sandwich Composites with Nanophased Cores under Flexural Loading", Composites Part B: Engineering, Vol. 35, Nos 6–8, September–December 2004, pp. 543–550.
- [6] Kanny, K., Mahfuz, H., Carlsson, L.A., Thomas T. and Jeelani, S., "Dynamic Mechanical Analyses and Flexural Fatigue of PVC Foams", Composite Structures, Vol. 58, No. 2, November 2002, pp. 175–183.
- [7] Shipsa, A., Burman, M. and Zenkert, D., "Interfacial Fatigue Crack Growth in Foam Core Sandwich Structures", International Journal of Fatigue & Fracture of Engineering Materials & Structures, Vol. 22, 1999, pp. 123–131.
- [8] Reynaud, E., Gautier, C. and Perez, J., "Nanophases in Polymers", Review Metallurgie, Vol. 96, 1999, pp. 169–176.
- [9] Chun, L.W., Ming, Q.Z., Min, Z.R. and Klaus, F., "Tensile Performance Improvement of Low Nanoparticle-Filled Polypropylene Composites", Composites Science and Technology, Vol. 62, 2002, pp. 1327–1340.
- [10] Mahfuz, H., Chisholm, N. and Jeelani, S., "Mechanical Characterization of Nanophased Carbon Composites", 2002 ASME winter Annual Meeting, New Orleans, Louisiana.
- [11] Mahfuz, H., Rangari, V., Islam, M., and Jeelani, S., "Fabrication, Synthesis and Mechanical Characterization of Nanoparticles Infused Polyurethane Foams", Composites Part A: Applied Science and Manufacturing, Vol. 35, 2004, pp. 453–460.
- [12] ASTM Standard: C 273-61 Standard Test Methods for the Shear Properties in Flatwise Plane or Flat Sandwich Constructions or Sandwich Cores.