HIGH STRAIN RATE BEHAVIOR OF HYGROTHERMALLY CONDITIONED SYNTACTIC FOAMS FOR MARINE APPLICATIONS

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1 General Introduction

Syntactic foams are widely used in aerospace, defence and marine structures due to their superior mechanical properties, light weight and enhanced thermally insulating behaviour. They are widely employed in low density applications, such as buoyancy aid materials for deep sea exploration and naval vehicles [1–4]. Vinyl ester based syntactic foams have gained considerable momentum in this decade primarily due to their employment in marine/submarine and structural applications, and low cost when relative to their epoxy counterparts. Understanding the dynamic compressive behavior of vinyl ester based syntactic foams when subjected to hygrothermal conditioning is of critical importance as these systems will eventually find applications in deep seawater devices. Thus, this study aims at processing vinyl ester based syntactic foams and characterizing their dynamic behavior by conducting high strain rate tests employing a Split Hopkinson Pressure Bar (SHPB) setup. Additionally, uniaxial compression tests, Dynamic Mechanical Analysis (DMA) and microstructural analysis (SEM) will be performed to gain a better understanding of the underlying damage mechanisms involved during crack propagation.

2 Raw Materials and Processing Method

2.1 Raw Materials

The matrix resin used in this study was HYDREX 100® 33350 vinyl ester (density = 1100 kg/m^3), manufactured by REICHHOLD Company and supplied by US Composite Company. Nanomer I.30E (nanoclay platelets with diameter of 1µm and thickness of 1nm, density=1710kg/m^3) supplied by Nanocor Inc. was used as the nanoparticle reinforcement. The glass microballoon used in this study is termed as "S22" and supplied by 3M Company under the trade name 'Scotchlite'. S22 microballoon has a mean outer diameter of 40 µm and a density of 220 kg/m^3.

2.2 Processing Method

The processing method begins by mixing required amount of nanoclay in vinyl ester using an ultrasonic probe (VIBRACELL) resonating at a frequency of 20kHz±5Hz and amplitude of 40% with a pulse "ON" for 30 seconds and, "OFF" for 5 seconds. Due to improper dispersion issues that may arise as a result of localized ultrasonic mixing, the nanoclay-vinyl ester mixture was passed through a three roll mill. This mixing is envisaged to facilitate better dispersion and intercalation of nanoclay platelets by introducing shear forces in the system and eventually resulting in enhanced dispersion. Further, a measured quantity of glass microballoons were added and mixed with a spatula until a uniform mixture was obtained. Then, MEKP (hardener) was added and mixed. This mixture was poured into a mold and cured at room temperature for 36 hours. For comparison purposes, syntactic foams without nanoclay were also fabricated. The weight fraction of nanoclay used in this study was 1%, 2% and 5%. The volume fraction of glass microballoons used was 60%. One set of specimen were subjected to hygrothermal conditioning by immersing them for a prolonged period in salt water and de-ionized water. The specimen was immersed in these harsh sea water environments until saturation was reached. Specimen with different nanoclay weight fractions were named as Nanoclay Syntactic Foams (NCSF) in this study.

3 Experimental Procedures

3.1 Hygrothermal Moisture Conditioning

Moisture conditioning was performed by immersing one set of specimen in salt water and another set of specimen in de-ionized water. The water absorbed was measured periodically. When variation in

weight of the specimen due to moisture absorption reached less that 0.1% in 7 days (ASTM D 5229-92), it was assumed that saturation was attained. At this juncture, conditioning was stopped.

3.2 High Strain Rate (HSR)

High strain rate testing of both dry and moisture conditioned specimen were performed on a Split Hopkinson Pressure Bar (SHPB) apparatus at strain rates of $875s^{-1}$ and $1200s^{-1}$. The SHPB system basically consists of three bars, (a) a striker bar, (b) an incident bar and (c) a transmitter bar aligned in line and made of maraging steel. The specimen was sandwiched between the incident and transmitter bars, and the striker bar was released from a pressure barrel, which was allowed to hit the incident bar. This results in a stress wave been transmitted through the incident bar, to the incident bar-specimen interface, and this wave further passes through the specimen to the specimen-transmitter bar interface. The strain in the system was measured by strain gauges attached at equal distance from the interface. Details of the test and analysis procedures can be found elsewhere [5]. From these tests the dynamic compressive strength of the vinyl ester syntactic foams was determined. A typical SHPB test data is depicted in Fig. 1.

3.3 Uniaxial Compression

Strain controlled uniaxial compression tests of dry and moisture conditioned specimen were conducted on an MTS Q TEST 150 universal testing machine. The test was done at a cross-head speed of 1.3mm/min according to ASTM C365-94. Five effective specimens (25.4mm x 25.4mm x 12.7mm) were tested for consistency in results. The load-deflection data obtained from these tests were converted into equivalent stress-strain plots and analyzed for their compressive strength (strength at yield point).

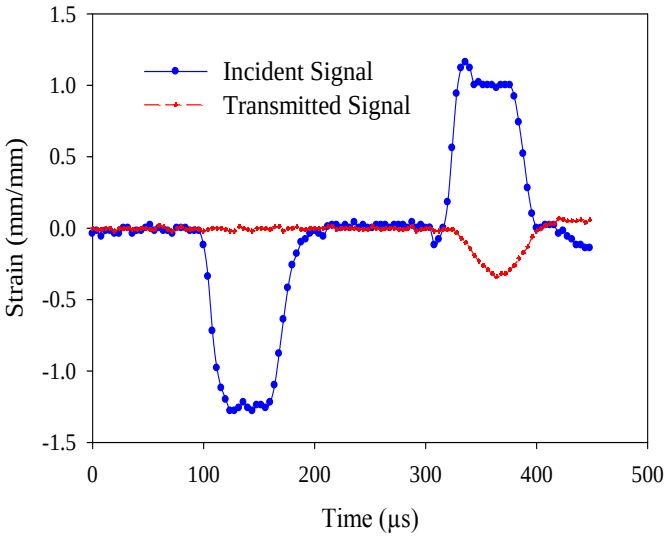

Fig.1. Strain signals from HSR testing on 0% dry specimen at a strain rate of 875 s^{-1}

3.4 Dynamic Mechanical Analysis (DMA)

Viscoelastic properties like storage modulus, E' (measure of energy stored elastically, elastic modulus), loss modulus, E'' (measure of energy dissipated, viscous modulus) and tanδ (damping coefficient) of vinyl ester syntactic foams were determined by conducting DMA tests. Glass transition temperature of the specimen was evaluated from these tests by finding the temperature corresponding to the peak of the tanδ curve. Dynamic Mechanical Analysis of dry and wet specimen was carried out on a Rheometric Scientific Solids Analyzer, RSA III, operating under tension mode at a frequency of 1Hz and a heating rate of 1^{o}C/min, as per ASTM E 1640-04. The temperature was ramped from 30^{o}C to 180^{o}C, while tensile testing was carried out at a constant frequency. The specimen were machined to dimensions of 1.5 x 12.7 x 35 mm^3, using a diamond cutter.

3.5 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) images of fractured high strain rate tested specimen were captured using a JEOL SEM machine. This was done to understand the failure modes occurring in the specimen when subjected to high strain rate dynamic testing conditions.

4 Results and Discussions

4.1 Hygrothermal Conditioning

From the moisture absorption studies conducted on salt water (SW) and de-ionized (DI) water specimen, it was concluded that the DI water conditioned specimen had enhanced moisture intake (50% increment) when compared with their SW conditioned counterparts. The ionic species of salt water (Na^{+}) is large enough to reduce diffusion rate as compared to the ionic species of de-ionized water.

It is envisaged that the presence of large amounts of Na^+ ions in SW conditioned specimen interfered with the diffusion process and hence resulted in lower moisture absorption. Also, in the HSR, compression and DMA test results, it was visualized that the DI water specimen had a larger reduction in properties when compared with their SW counterparts.

4.2 HSR and Uniaxial Compression

HSR and uniaxial compression results are summarized in table 1 and 2, respectively. The compressive strength values obtained from these tests are very critical in determining the safe workable limit of a material. It can be seen that the dynamic strength is about 40-135% higher than their corresponding static compressive strength. With an increase in strain rate, the compressive strength increases irrespective of the type of conditioning (dry, SW & DI (table 1)). The reduction in dynamic compressive properties of the DI conditioned specimen (40-46%) was high when compared with SW specimen (27-42%).
Typical uniaxial compression stress-strain plots for dry syntactic foams with and without nanoclay reinforcement are depicted in Fig. 2.

Table 1. Variation of Peak Compressive Strength with nanoclay weight fraction and different moisture conditionings for HSR tests

Dynamic Compressive Strength (MPa)				
NCSF,%	**Strain rate, s^{-1}**	**Dry**	**SW**	**DI**
0	875	65.93	42.54	38.28
	1200	72.31	51.04	42.54
1	875	65.22	44.24	40.41
	1200	70.72	47.64	38.28
2	875	65.66	41.51	37.00
	1200	73.91	45.09	44.66
5	875	63.80	43.39	34.74
	1200	70.18	51.04	40.41

The addition of nanoclay did not have any major effect on the uniaxial compressive properties, even though there were some improvements for the 2% reinforced specimen. The reduction in compressive strength of salt water conditioned specimen was 27%, 24%, 25% and 24%, respectively for 0%, 1%, 2% and 5% nanoclay reinforced syntactic foams. The corresponding reduction for the DI water conditioned specimen was 26%, 24%, 28% and 24%, respectively.

Table 2. Variation of Yield Strength with nanoclay weight fraction and different moisture conditionings for Quasi-static Compression tests

Compressive Yield Strength (MPa)			
NCSF, %	**Dry**	**SW**	**DI**
0	31.80	23.24	23.54
1	30.04	22.96	22.83
2	32.53	24.51	23.42
5	31.88	24.10	24.12

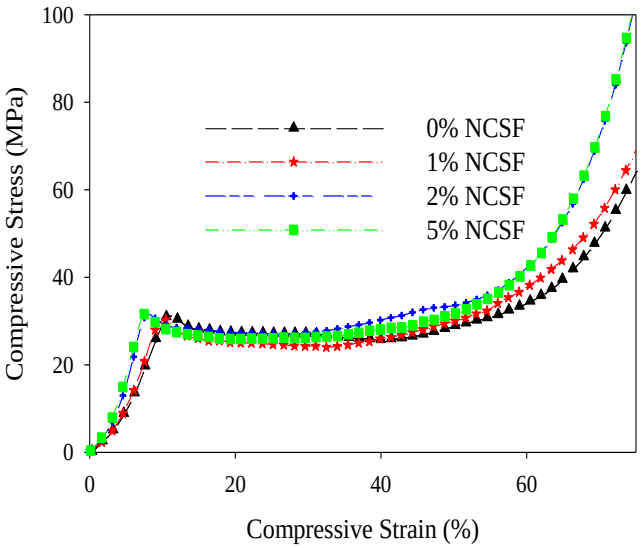

Fig.2. Compressive stress-strain behavior of nanoclay reinforced syntactic foams

4.3 DMA

The variation of storage modulus with nanoclay weight fraction for SW and DI water conditioned specimen is shown in Fig.3 and Fig.4, respectively. From these figures it can be visualized that storage modulus was a constant initially, until it gradually start sloping down. Once the material passes through the T_g region, an abrupt fall in the storage modulus curve can be noticed. This corresponds to the softening/relaxation of polymeric chains. Thus, in a more relaxed state, the polymeric chains can be easily deformed due to the tensile loading condition employed in DMA testing. The T_g values (temperature corresponding to the peak of tanδ curve) for both SW and DI specimen in comparison with their dry counterparts is summarized in table 3.

From Figures 3, 4 and table 3, it can be seen that, the storage modulus and T_g for nanoclay reinforced syntactic foams up to 2% by weight increased when compared with their pure syntactic foam counterparts, irrespective of whether they were dry or moisture conditioned. The nano-scale distribution of clay platelets in the polymeric matrix with glass microballoons resulted in better reinforcement, and hence higher stiffness and T_g values.

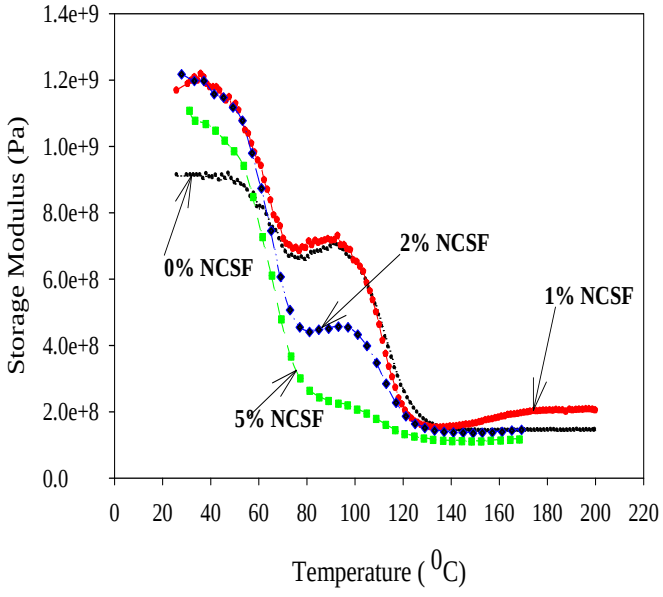

Fig.3. Variation of storage modulus for SW specimen with nanoclay weight fractions

The reduction in storage modulus and T_g was higher for the DI water conditioned specimen compared to SW conditioned specimen. Moisture absorption resulted in deteriorating or weakening chemical bonds between polymeric chains, resulting in lower viscoelastic properties, compared to dry syntactic foams. Due to moisture absorption, the free volume of the conditioned specimen will be higher than that of the dry specimen. This swelling of the syntactic foam resulted in a reduction of T_g of the material [6].

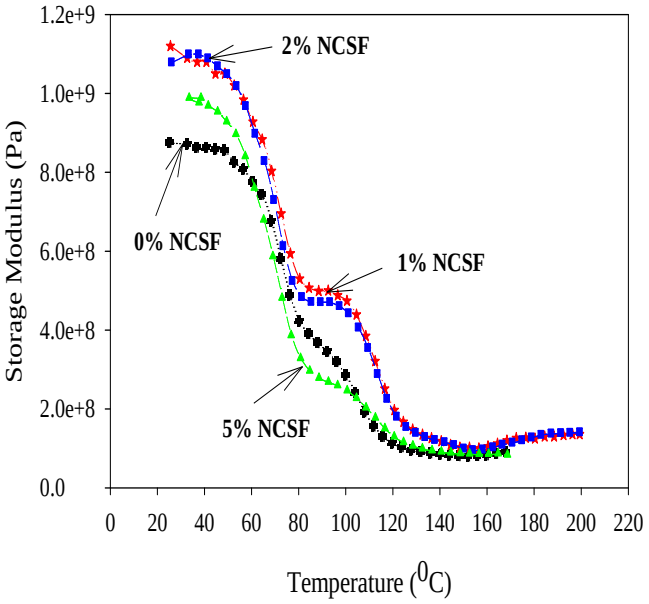

Fig.4. Variation of storage modulus of DI specimen with nanoclay weight fractions

Table 3. Variation of T_g due to different moisture conditionings and nanoclay weight fractions

NCSF	Dry T_g (^{0}C)	SW		DI	
		T_g (^{0}C)	% Redn	T_g (^{0}C)	% Redn
0	119	109	8.6	106	11.3
1	125	111	9.6	110	11.5
2	126	112	10.7	112	11.1
5	125	107	14.6	106	15.2

4.4 Microstructural Failure Analysis

Fracture surfaces of plain syntactic foam subjected to HSR testing at strain rates of 857 s^{-1} and 1200 s^{-1} is depicted in Fig.5 and Fig.6, respectively. From these figures it can be visualized that the extent of damage due to crack propagation for 1200 s^{-1} tested specimen is higher than that of their 875 s^{-1} tested specimen. This can be visualized by the presence of more debris from broken glass microballoons in Fig.6.

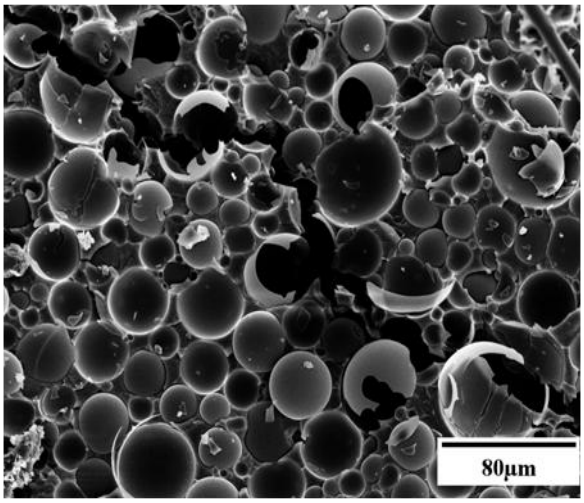

Fig.5. SEM image of plain syntactic foam tested at 875 s^{-1}

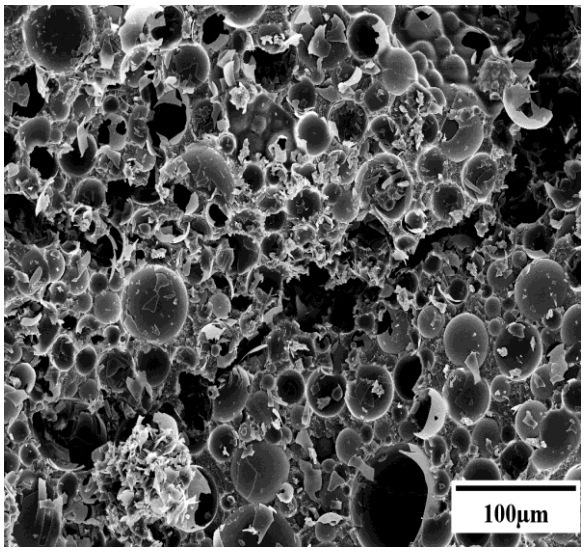

Fig.6. SEM image of plain syntactic foam tested at 1200 s^{-1}

5 Conclusions

(a) Syntactic foams subjected to salt water conditioning had less moisture absorption when compared with their de-ionized water counterparts.
(b) The compressive strength (static and dynamic) reduced when the specimen is subjected to hygrothermal conditioning and the reduction is the highest for the de-ionized water specimen when compared with the sea water specimen.
(c) The dynamic compressive strength of syntactic foams increased with increase in strain rate, irrespective of the nanoclay weight fraction or hygrothermal conditioning.
(d) Viscoelastic properties of nanoclay reinforced syntactic foams up to 2% weight fraction were enhanced when compared with their plain syntactic foam counterparts.

References

[1] J.R.M. d'Almeida. "An analysis of the effect of the diameters of glass microspheres on the mechanical behavior of glass-microsphere/epoxy-matrix composites", *Composites Sci. Technol.*, Vol. 59, pp 2087–2091, 1999.
[2] J.R. Vinson. Lancaster: *Technomic*, 1999.
[3] L. Bardella, and F. Genna. "On the elastic behavior of syntactic foams", *Int. J. Solid Struct.*, Vol. 38, pp 7235–7260, 2001.
[4] N. Gupta, and W. Ricci. "Comparison of compressive properties of layered syntactic foams having gradient in microballoon volume fraction and wall thickness", *Mater. Sci. Eng. A.*, Vol. 427, pp 331–342, 2006.
[5] S. Peter, and E. Woldesenbet. 2008. "Nanoclay syntactic foam composites—high strain rate properties", *Mater. Sci. Eng. A.*, 494:179–187.
[6] A. Haque and M.K. Hossain. 2003. "Effects of moisture and temperature on high strain rate behavior of S2-glass-vinyl ester woven composites", *J. Compos. Mater.*, 37: 627-647.