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Effect of arctic environment on flexural behavior of fly ash cenosphere reinforced epoxy syntactic foams

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ABSTRACT

In this paper, the effect of arctic conditions on the flexural response of cenosphere/epoxy syntactic foams is investigated. Understanding the behavior of such foams under extreme conditions is critical for exploring their suitability for constructing lightweight platforms used in arctic explorations. Such platforms are exposed to subzero temperatures for extended periods of time potentially degrading their mechanical properties. In the research study presented here, samples of cenosphere/epoxy syntactic foams were conditioned under arctic environment at −60 °C temperature for a period of 57 days. Flexural tests were then conducted at room temperature as well as in-situ −60 °C on the conditioned samples and compared against unconditioned samples. Combinations of surface modification and cenosphere volume fractions were considered. Experimental findings showed that an increase in flexural modulus can be observed at room temperature with increasing cenosphere volume content for both untreated and treated cenosphere reinforced syntactic foams. In contrast, a decrease in flexural strength was observed as compared to neat resin. For the case of arctic exposed samples, an apparent increase in flexural modulus was recorded between 7-15% as compared to room temperature cenospheres/epoxy syntactic foams. In addition, an apparent increase of 3–80% in the flexural strength was observed under arctic environment. The conditioning of cenosphere/epoxy syntactic foams under low temperatures manifested lower strains to failure as compared to neat epoxy and they exhibit quasi-brittle behavior leading to sudden failure in the post peak regime.

1. Introduction

Sandwich composites with foam cores are of interest in applications like aircraft and naval applications. These foam cores are typically made from closed-cell and low-density polymers and are sandwiched between fiber-reinforced polymeric composite facesheets. Such sandwich constructions are extremely lightweight, which increase the buoyancy of the ship-structures. However, extended period of exposure to sea environment in marine applications often results in mechanical property degradation due to moisture absorption and temperature variations in these materials. Structural components in arctic marine applications encounter these major concerns and are the focus of the present work. Dispersion of hollow microballoons/microspheres in resin matrix forms a special class of composite known by name syntactic foams [1,2]. The spectrum of engineering applications of these foams is very broad as elaborately discussed by Gupta et al. [3–5]. Components like boat decks, ribs, hulls and floatation modules are some of the widely known and proven applications in naval structures. Nevertheless, syntactic foams are also utilized in remotely or humanly operated vehicles used for sea explorations. These closed cell foams are also promising material systems in pipelines laid deep in sea demanding thermal insulation [6].

Developing structure-property correlations and understanding failure mechanisms therein in tailoring syntactic foam properties for various applications has been extensively dealt with in the past decade [7–10]. Thermal and electrical behavior of syntactic foams [11–14] have also been investigated in addition to mechanical properties. Further, syntactic foams reinforced with micro and nano scale fillers (fiber and particle) have been studied extensively, which were beneficial towards tailoring the properties as compared to plain syntactic foams [15,16]. Recently thermoplastic foams have been developed using industrial scale injection molding machine [17–23], compression molding [24], 3D printing [4,5] and characterized for mechanical properties. These closed cell foams are tested under 3 point bending in flexural

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[25–28] or with short beam test condition [29–31] in recent past. The relationship of volume fraction and reinforcement surface treatment with the modulus in flexure is often complex. Relative modulus of the particle material, geometrical parameters and the matrix properties are influenced by the matrix stiffening due to lower density hollow microballoons. Effect of particle shell thickness and interfacial bonding between the constituents also have significant influence on the flexural properties. Published literature reports several experimental and analytical studies on hygrothermal effects [32–34], impact loading [35], microstructural characterization [36,37] and polymer cure cycle effect [38] of syntactic foams. Variations in cenosphere shell thickness and built-in porosities therein makes structure-property correlations of cenosphere embedded thermosetting foams quite complex and challenging to address especially with additional temperature effects like in arctic conditions. Marine vessels are often subjected to arctic conditions. Understanding mechanical behavior in arctic scenario is necessary and demanding as these naval structures are made of syntactic foams. Further, overall mechanical behavior and water uptake is significantly governed by operating temperatures [39,40] particularly in arctic condition wherein low temperature prevail.

Engineered glass microballoons and fly ash cenospheres as filler materials have been widely studied [14,41]. Degradation of glass microballoons syntactic foams has been reported recently due to dealkalization of glass [42]. Fly ash is a potential environmental pollutant and an industrial waste comprising of hollow cenosphere particles that are made of alumina and silica primarily. These hollow microballoons are by-product of coal combustion in thermal power plants. Though, they have numerous defects on and in their walls with marginal deviations from perfect sphericity, the alumino-silicate composition might compensate the limitations caused by such defects. Further, fly ash cenosphere properties have been found to be in the range of widely used glass microspheres. Developing utilitarian syntactic foams with such industrial waste can help the environment, minimize landfill burden and create foams with better properties [43–45]. The current work uses fly ash cenospheres for manufacturing syntactic foams.

Majority of studies on the mechanical property characterization of syntactic foams is conducted at room temperature (RT) [46,47]. However, for marine vessels for operation in the arctic or antarctic oceans, it is critical to investigate these materials in such harsh conditions of subzero temperatures. The effect of arctic environment on cenosphere/epoxy foams has not been studied before, and is crucial for marine structural components operating in Arctic regions. The present study explores this possibility by investigating the flexure behavior of syntactic foams owing to changes in operating temperatures. Hollow fly ash cenospheres, epoxy matrix, filler treatment and filler loading are held constant while altering the exposed temperatures. Thereby, changes in the fracture pattern are governed by temperature variations. The behavior of syntactic foams in such temperatures can be explored by such an investigation, which has not been reported in the literature yet. Present work focuses on the development of environment friendly syntactic foams subjected to arctic conditions along with the influence of filler (cenosphere) surface modification.

2. Sample preparation and test method

2.1. Sample preparation

Table 1
Chemical, physical and sieve analysis details of cenospheres[a] [48].

Physical properties		Chemical analysis		Sieve analysis	
True particle density	920 kg/m^3	SiO_2	52–62%	+30# (500 μm)	Nil
Bulk density	400–450 kg/m^3	Al_2O_3	32–36%	+60# (250 μm)	Nil
Hardness (MOH)	5–6	CaO	0.1–0.5%	+100# (150 μm)	Nil
Compressive strength	180–280 kg/m^3	Fe_2O_3	1–3%	+150# (106 μm)	0–6%
Shape	Spherical	TiO_2	0.8–1.3%	+240# (63 μm)	70–95%
Packing factor	60–65%	MgO	1–2.5%	- 240#	0–30%
Wall thickness	5–10% of shell dia.	Na_2O	0.2–0.6%		
Color	Light grey – light buff	K_2O	1.2–3.2%		
Melting point	1200–1300 °C	CO_2	70%		
pH in water	6–7	N_2	30%		
Moisture	0.5% max.				
Loss on ignition	2% max.				
Sinkers	5% max.				
Oil absorption	16–18 g/100 g				

[a] As specified by supplier.

CIL 150 grade cenospheres are procured from Cenosphere India Ltd., Kolkata and their basic properties are presented in Table 1 [48]. Lapox is used as matrix (L-12 grade, K-6 hardener) and is bought from Atul, Valsad, Gujarat. Two configurations of cenosphere/epoxy foams are prepared in the present work i.e. with cenospheres in as received condition and their surface modified counterparts. Surface treatment procedure and the confirmatory tests of silane coating are outlined in Ref. [49]. The built-in void space within cenospheres, volume fraction and size play a critical role in lowering the material density substantially. Further, interfacial adhesion owing to surface modification of cenosphere promotes effective load transfer mechanisms between the constituents [50]. It's interesting to analyses the effect of arctic temperature on the interface and if it could sustain the integrity under such lower operating temperatures. Cenospheres in desired proportion are dispersed gently in epoxy resin until homogenous slurry is formed. Hardener is added by 10 wt % to initiate polymerisation and further stirred for 2 more minutes, degassed for 5 min and finally poured in the aluminium molds. Curing time of 24 h and post curing at 90 °C for 3 h is adopted. Syntactic foams with cenosphere variation of 20, 40 and 60 vol %, both in as received and surface modified conditions are prepared having dimensions as outlined in ASTM D790. Neat epoxy samples are also casted by following similar procedure for comparative analysis. At least five specimens each are tested in flexure under room and arctic temperatures. Samples are coded as per the EXX-Y convention where epoxy, cenosphere content and filler surface treatment is represented by letters E, XX and Y (U - Untreated, T - treated) respectively. Experimental density of all the samples is estimated using ASTM D792-08. Rule of mixture is used to compute theoretical density and is given by,

$$\rho_c = \rho_f V_f + \rho_m V_m \tag{1}$$

where, ρ, V, c, f and m denote density, volume fraction, composite, filler and matrix respectively. Experimental (ρ^{exp}) and theoretical (ρ^{th}) densities are utilized further to calculate void volume % (ϕ_V) and is given by Refs. [9,26],

$$\phi_V = \frac{\rho^{th} - \rho^{exp}}{\rho^{th}} \tag{2}$$

2.2. Arctic conditioning and flexure test

All the samples prepared are conditioned in accordance to ASTM C272 and D5229 standards as there are no standards available for arctic tests. Specimens are dried at 100 °C prior to conditioning. As part of arctic conditioning, all the samples are maintained at −60 °C (Thermo Scientific TSU Series −86 °C Upright Ultra-Low Temperature Freezer) for 57 days. Post arctic conditioning, all the samples are tested at −60 °C in-situ. Flexure test is conducted as per ASTM D790 standard at both room (30 °C) and arctic condition (−60 °C) using Instron 5969 Tabletop UTM. Crosshead displacement is maintained constant at 1.4 mm/min. Flexural strength and modulus of elasticity in flexure is