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Development & Characterization of Styrene Butadiene Rubber (SBR) –Hollow Glass Microballoons (HGM) Syntactic Foams

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Abstract—*Polymer syntactic foams are a subset of polymer composites that are formed by introduction of hollow microspheres in a resin. Presence of hollow microspheres bestows light weight capabilities to these syntactic foams. The properties of these syntactic foams are therefore governed by the properties of the constituent materials and on their volume fraction. Syntactic foams have made their way into a multitude of applications ranging from underwater to aerospace. These foams are used in the construction of light weight parts of automobiles and aerospace where they serve to increase the payload capacity and reduce the fuel consumption. They are being used as core materials in the construction of sandwiched structures for shock mitigation applications. This paper is an attempt to develop elastomeric syntactic foam containing styrene butadiene rubber as the matrix material. Hollow microballoons of glass (ρ= 0.46 g cm^{-3}) are employed as light weight hollow fillers to impart porosity to the system. SBR glass microballoons (40-60 % v/v) syntactic foams are prepared by solution mixing approach wherein SBR is dissolved in toluene followed by addition of measured quantities of vulcanizing ingredients and hollow glass microballoons. The formulation is vulcanized. This method prevents the breakage of hollow microballoons.*

Characterization of syntactic foams is being done by Scanning Electron Microscope(SEM), thermo gravimetric analyser(TGA).

Keywords: *Styrene butadiene rubber(SBR), Hollow glass microballoons(HGM), Syntactic foams*

I. INTRODUCTION

Syntactic foams are a fascinating category of polymer foams in which foaming is accomplished by insertion of hollow microballoons of glass in resin matrix[1]. This category of material are depicted by presence of low weight and good specific mechanical properties by inserting hollow microballoons which are of different sizes ranging from 10 μm to 300 μm. They are used as core material in sandwich composites for automotive, aerospace, marine applications[2]. Syntactic foams are chiefly closed cell foams compared to conventional foams which are mostly open celled. The mechanical properties of syntactic foams are far superior than open cell foams, therefore the former are used in sandwiched structures[3]. Hollow microballoons of glass, polymer, flyash, etc. are inserted into various matrix system including both polymer, metal and ceramic[4-7].

A variety of polymer matrices have been exploited for preparation of syntactic foams. However, the studies on elastomeric matrix filled glass microballoons has not been investigated. We anticipate that preparation of styrene butadiene rubber (SBR) glass microballoons syntactic foams will not only improve the mechanical properties of syntactic foams but also improve the damage tolerance which will be be helpful in the engineering applications of such foams in the future. Elastomeric composites are usually prepared either by two roll mill and compression moulding or by solution mixing approach[8-10].

The use of two roll mill may lead to the excess shearing of glass microballoons which might result in their fracture. Hence, for the present study, solution mixing technique has been followed.

This study is therefore aimed at development and characterization of SBR glass microballoons syntactic foams by solution mixing process containing varied volume percentages (40-60 % v/v) of glass microballoons. The mechanical properties of the foams have been studied which may serve as the base for the development of such elastormeric based foams in the future.

II. MATERIALS USED

Material used is as follows:

- Styrene butadiene rubber(SBR)
- Hollow Glasss Microballoons(HGM)
- Stearic Acid($C_{18}H_{36}O_2$)
- Sulphur Powder
- TBBS
- Zinc Oxide(ZnO)
- Organic Solvent- Toluene

Sulphur powder acts as a vulcanising powder, zinc oxide and stearic acid act as an activators, TBBS acts as an Accelarator.

III. MATERIALS AND PROCESSING

Styrene butadiene rubber (SBR) grade-JSR 1502 was procured from Japan Synthatic Rubber Co. Stearic Acid was obtained from Burgoyne Burbidges& Co. Mumbai. Zinc Oxide has been purchased from Merck Ltd. Mumbai. Sulphur powder was obtained from Merck Specialities Pvt Ltd. Mumbai. TBBS was obtained from Merchem Ltd.Hollow Glass Microballoons K 46 type were obtained from 3M™ America. Four categories of samples were prepared. The specimen designation and its compositional details are mentioned in Table 1.

TABLE-1 Sample Designation And Compositions Of Neat Sbr And SBR K46 Syntactic Foam

COMPOSITION	SBR (% v/v)	HGM (% v/v)
SB0	100	0
SB40	60	40
SB50	50	50
SB60	60	40

Here, SB stands for SBR rubber which is followed by the volume percentage of glass microballoons employed. As an example, SB0 refers to a specimen containing only SBR rubber without glass microballoons. Similarly, SB40 refers to a composition containing 40 volume percentage of hollow glass microballoons in SBR matrix.

IV. PREPARATION OF NEAT SBR AND SBR SYNTACTIC FOAM

Neat SBR film was prepared by dissolving requisite amounts of SBR in toluene at room temperature. This was followed by incorporation of the compounding ingredients. The actual amounts of the ingredients is presented in Table 2. The solution was cast into aluminium moulds and cured in an oven at 160^{o}C for 2 hours. SBR glass microballoons syntactic foams were prepared by a similar process described above. Following the complete dissolution of SBR in toluene at room temperature, measured amounts of hollow glass microballoons (K46) (40-60 % v/v) was added. Rest procedure was similar to that used for neat SBR film. Once cured, the films were cut into dumble shaped specimens for tensile testing.

TABLE-2 Compounding Ingredients for SBR

Sample	Zinc Oxide(g)	Stearic Acid(g)	Sulphur(g)	TBBS(g)
SB0	2.5	1	1.125	0.35
SB40	2.11	0.847	0.95	0.29
SB50	1.96	0.787	0.88	0.27
SB60	1.77	0.711	0.8	0.24

The actual amounts of SBR rubber and hollow glass microballoons was calculated as per the formula:

$$\frac{Mass of SBR}{Mass of composite} = \frac{\rho_{SBR} \times \Phi_{SBR}}{\rho_{K46} \times \Phi_{K46} + \rho_{SBR} \times \Phi_{SBR}}$$

Where ρ and Φ refer to the density and volume fraction of the constituent respectively, and the subscripts 'SBR' and K46 refer to styrene butadiene rubber and microballoons respectively. For the purpose of calculation, the density of SBR and K46 microballoons 1.7 g cc^{-1} and 0.46 g cc^{-1} respectively.

V. MECHANICAL TESTING

QUASI-STATIC TENSILE TESTING

This test was done to check the ability of material to resist the fracture under stress applied at high speed.For the purpose of tensile testing, three samples of each compostion were tested to bring out elongation,specific tensile strength, specific tensile modulus toughness specific toughness. Dumbbell shapedspecimens were made as per ASTM D412C and tested at a crosshead speed of 50 mm/min.

VI. CHARACTERIZATION

The thermal behaviour was investigated using Perkin Elmer (Pyris 1 TGA) under N_2 atmosphere in the temperature range 50-600°C. A heating rate of 20 °C/min and sample mass of 5.0 ± 0.5 mg was used for each experiment. The surface morphology was studied using a scanning electron microscope (JEOL- JCM6000PLUS) under an acceleration voltage of 1 kV. Samples were mounted on aluminium stubs and sputter-coated with gold and palladium (10 nm) using a sputter coater (DII-29030SCTR Smart Coater) at 10-12 mA for 120 s.

VII. DENSITY DETERMINATION

The theoretical density was calculated using the standard rule of mixtures.

$$\rho_{th} = \rho_{K46} * \Phi_{K46} + \rho_{SBR} * \Phi_{SBR}$$

Experimental density (ρ_{ex}) was determined by calculating the mass: volume ratio of three specimens as per ASTM D1622-98[11]. The difference between the theoretical and experimental density was used to determine the air-void porosity trapped within the foam.

$$Void\ volume\ (\%) = \frac{\rho_{th} - \rho_{ex}}{\rho_{th}} \times 100$$

VIII. RESULTS AND DISCUSSION

Tensile Testing

Quasi-static tensile testing of neat SBR film and SBR syntactic foams is presented in Figure 1 (a-d).Test result is shown below of tensile strength, specific tensile strength and tensile elongation(%) of four samples.

Graph (a) represents the tensile strength of syntactic foam samples. As it is seen by graph tensile strength of sample having 40%(v/v) of hollow glass microballoons(HGM) shows relative increase (~ 4 %) in the tensile strength compared to neat SBR. This is attributed to the presence of high strength glass microballoons (41 MPa) compared to SBR. However, owing to the lack of proper interfacial adhesion between the matrix and glass microballoons, the extent of improvement is lower. In samples, containing higher volume percentage of glass microballoons (50-60 % v/v), the observed values are lower due to a reduction in resin content and its subsequent inability to hold the microspheres together, resulting in tensile failure.Similar results have been obtained with epoxy glass microballoon syntactic foams wherein an increase in the hollow glass microballoons content leads to a concominant reduction in the properties of syntactic foams[12,13].

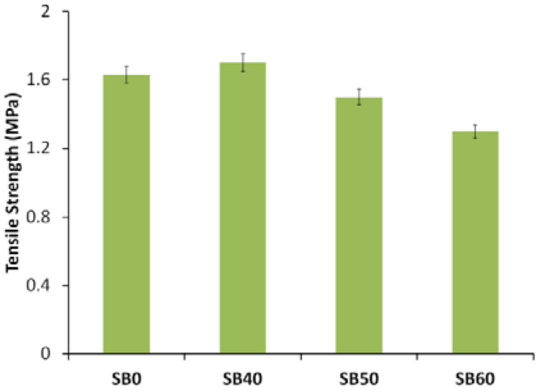

Fig. 1(a): Tensile strength of neat and syntactic foam specimens

Specific tensile strength of syntactic foams are obtained by dividing tensile strength of each sample by their density. Inview of the substantial reduction in the density of syntactic foams owing to the presence of glass microballoons, the specific tensile strength values are significantly higher than neat SBR. An improvement of ~ 47 % is recorded for SB40 compared to neat film.

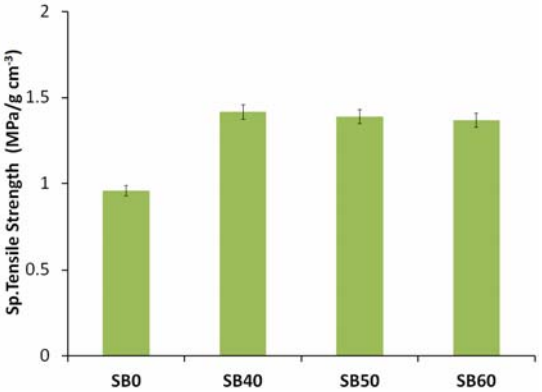

Fig.1(b): Specific tensile strength of neat and syntactic foam specimens

The tensile modulus of neat and SBR- HGM syntactic foams is presented in Figure 1(c). Tensile modulus of syntactic foams is calculated by the ratio of stress to corresponding strain in the linear elastic region of the stress-strain curve. The presence of brittle domains i.e. glass microballoons in a softer phase i.e. elastomer improves the tensile modulus of syntactic foams. At 40 percent loading of glass microballoons in SBR matrix, an improvement of 50 % is achieved compared to neat sample without glass microballoons.

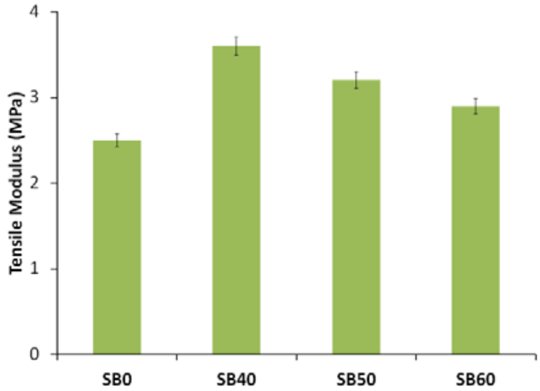

Fig. 1(c): Tensile modulus of neat and syntactic foam specimens

Figure 1(d) represents specific tensile modulus of syntactic foam samples. Specific tensile modulus of sample is determined by dividing tensile modulus of each sample by their experimental density which results in an increase of104 % in specimens containing 40 volume percent of hollow glass microballoons.

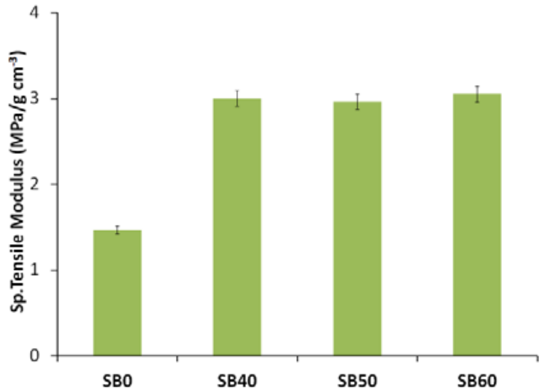

Fig.1(d): Specific tensile modulus of neat and syntactic foam specimens

Microstructural Characterization

The scanning microscopy images of SBR without hollow glass microballoons (K 46) and in the presence of K 46 at different magnifications are presented in Figure 2 (a-c). Neat SBR film has a smooth texture (Figure 2(a-b)) whereas in the SEM images of syntactic foam specimens, spherical microballoons can be clearly seen embedded in the matrix of SBR. The presence of voids can also be seen in Figures (2 (c-e)).

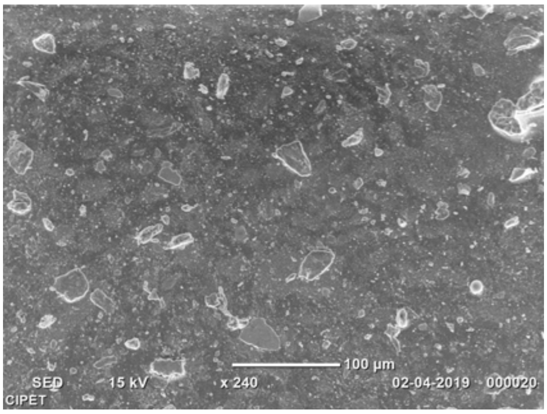

Fig.2(a) SEM image of syntactic foam without HGM (240 X)

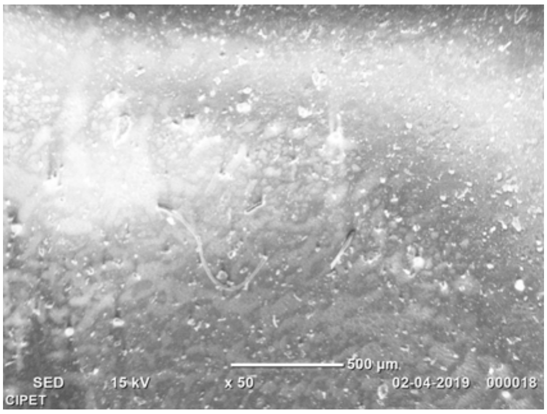

Fig.2(b) SEM image without HGM (50 X)

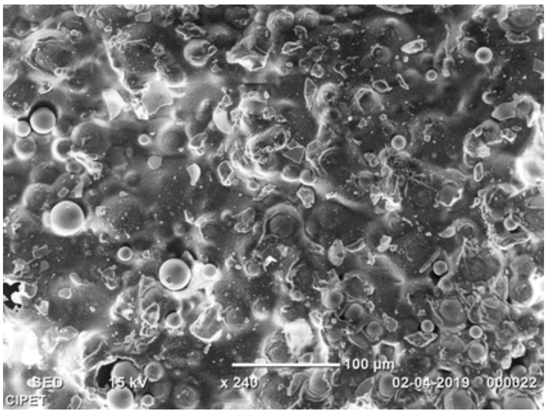

Fig 2 (c)SEM image of syntactic foam with 40%(v/v) HGM (240 X)

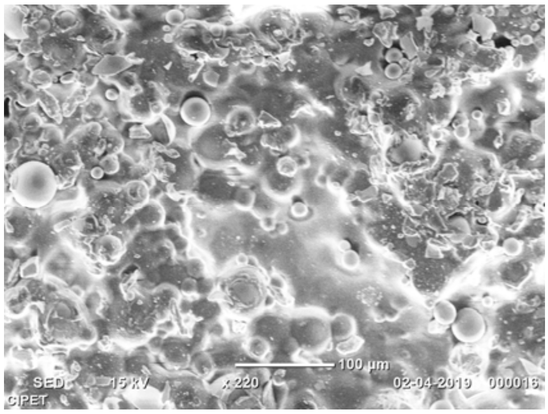

Fig 2 (d)SEM image of syntactic foam with 50%(v/v) HGM (220 X)

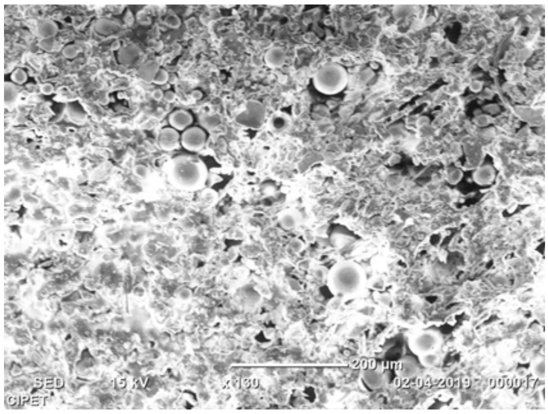

Fig 2(e)SEM image of syntactic foam with 60%(v/v) HGM (130 X)

Density Determination

The densities of syntactic foams containing varying volume percentage (40-60 % v/v) of hollow glass microballoons is presented in Figure 3. In all cases, the theoretical density is higher than the experimental density. This difference between the theoretical and experimental densities is used to estimate voidage[14,15]. During the processing of SBR by solution mixing process, the solvent evaporation leads to high voidage than would normally be caused by processing using two roll mills. It is also worth noticing that the voidage is high in samples containing higher volume percentage glass microballoons. The same can be evidenced from the SEM micrograph of syntactic foam specimens.

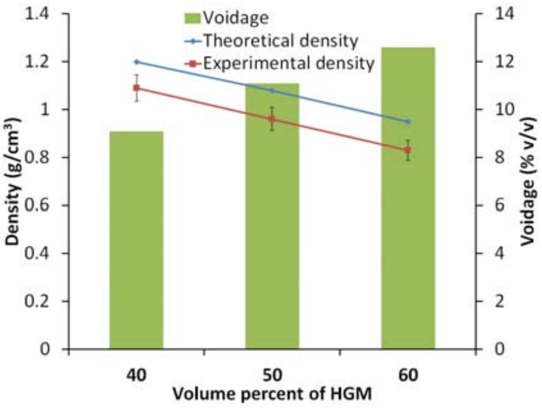

Fig.3: Theoretical and experimental densities and voidage of syntactic foams

Thermal Characterization

Thermo gravimetric traces of syntactic foams and neat SBR is presented in Figure 4. The presence of glass microballoons lead to an increase in the char content of syntactic foam specimens. All the specimens exhibit a single step degradation profile.

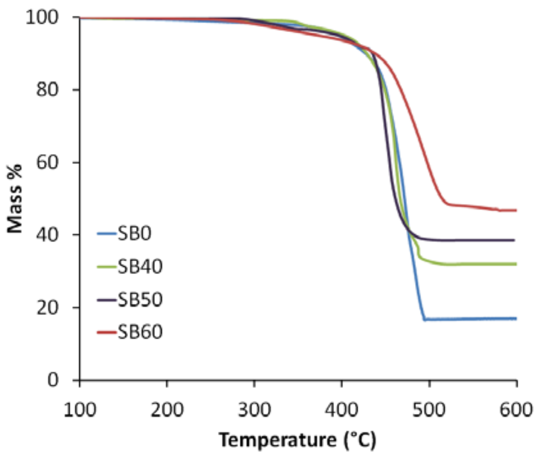

Fig.4: Thermogravimetric traces of neat and SBR syntactic foams

IX. CONCLUSION

Styrene butadiene rubber –glass microballoons syntactic foams were prepared by a solution mixing technique. The amount of hollow glass microballoons was varied from 40-60 % v/v. Incorporation of hollow glass microballoons led to appreciable increments in the quasi-static tensile properties of syntactic foams. Optimal property enhancements were obtained for 40 volume percent of glass microballoons. Enhancements of the order of 47 percent and 104 percent were obtained for specific tensile strength and specific tensile modulus respectively. These highlight the potential of development of elastomeric syntactic foams for advanced applications.

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