

# Preparation And Characterization Of Silicon Carbide Foam By Using In-Situ Generated Polyurethane Foam

Shalini Saxena, Arvind Kumar Saxena

**Abstract:** The open cell silicon carbide (SiC) foam was prepared using highly crosslinked hybrid organic- inorganic polymer resin matrix. As inorganic polymer polycarbosilane was taken and organic resin was taken as a mixture of epoxy resin and diisocyanates. The resultant highly crosslinked hybrid resin matrix on heating and subsequently on pyrolysis yielded open cell silicon carbide foam. The hybrid resin matrix was characterized by Fourier transform Infrared Spectroscopy (FT-IR) and thermal properties i.e., Thermogravimetric analysis (TGA) & Differential Scanning Calorimetry (DSC) were also studied. The morphological studies of silicon carbide ceramic foam were carried out using X-ray Spectroscopy (XRD) & Scanning Electron Microscopy (SEM).

**Keywords:** Diisocyanates, Polycarbosilane, Polyol, Polyurethane foam, Pre ceramic foam, Pyrolysis, Silicon Carbide foam.

## 1. Introduction

Silicon carbide (SiC) foam has found a variety of technological applications in the form of thermal insulating material, space mirrors, corrosive substance filters, heat shields, gas burner media, membrane supports, fusion reactors, molten melt filtration, hot gas filtration, catalyst support and light weight structural parts for high temperature applications etc [1], [2], [3], [4], [5], [6], [7], [8], [9], [10], [11], [12], [13], [14], [15], [16], [17], [18], [19], [20], [21], [22], [23], [24], [25], [26]. The general technique used for making SiC foam is by the fixing of coarse and fine slurry of SiC powder onto the Polyurethane foam which on pyrolysis yielded open cell SiC foam [21],[27], [28], [29]. In such case, the desired shape and size SiC foam could not be made to fit in the complicated engineering systems because ceramics are brittle in nature[30], [32]. Recently, it has also been found that polycarbosilane (PCS) bearing reactive Si-H functionalities on heating yielded silicon carbide ceramic foam [32], [33], [35]. Such systems, have an advantage that the resin matrix being meltable[26], can be shaped to any structure as per the mould design and can be fitted without machining in any engineering systems. But, these foams yielded closed cell structure, so cannot be used for the storage of gases and filters etc. To overcome such limitations, it has been considered worthwhile to prepare open cell SiC using meltable resin matrix. So, in the present investigation we wish to report a very convenient and potential method for the preparation of desired shape and size open cell SiC foam using hybrid resin matrix containing PCS, epoxy resin and organic diisocyanates.

## 2 EXPERIMENTAL

### 2.1 Chemicals Used

Commercially available LY-556 Epoxy resin was obtained

- *Shalini Saxena is research scholar at Central Institute of Plastics Engineering and Technology, Lucknow, Uttar Pradesh, India - 226008. shalini.du81@gmail.com*
- *Arvind Kumar Saxena is Director, DMSRDE, Kanpur, Uttar Pradesh-India 208013.*

from Huntsman, Butanediol and 2 ethyl-2(hydroxyl methyl)-1,3-propanediol (TMP) were obtained from Merck India Ltd. and Lancaster respectively. Hexamethyldiisocyanate (HMDI) 98% Methylenedianiline (MDA) 97% was procured from Acros chemicals and were used as such. Polycarbosilane (PCS) was prepared by the thermal backbone rearrangement of polydimethylsilane in strict inert atmosphere for 125 hours in the temperature range 250-450 °C as reported earlier [32], [33], [34], [35], [36], [37], [38], [39], [40], [41].

### 2.2 Sample preparation

2.5 g of 2 ethyl-2(hydroxyl methyl)-1,3-propanediol(TMP) and 22.5 g of butanediol were mixed mechanically, which was further added to 2.5 g of epoxy resin LY556. This mixture was added to 50 gm of finely powdered polycarbosilane (PCS). The composition was heated upto ~140 °C under strict argon atmosphere. Further, the above composition was cooled to room temperature. Again, it was heated to ~ 80 °C and 0.675 gm MDA was mixed to it. Immediately, 25 gm of HMDI was also added with continuous stirring thus resulting in foaming of the material because of generation of in-situ polyurethane foam. The pre ceramic foam thus obtained was pyrolysed in the furnace upto ~1100 °C under argon atmosphere at the heating rate of 2 °C/min and then soaking at 1100 °C for 2 hrs. Afterwards, the furnace was allowed to cool until it attains room temperature under argon atmosphere. On cooling, the sample thus obtained, was characterized using TGA, DSC, FT-IR, SEM and XRD.

### 2.3 Sample characterization

FTIR spectrophotometer of Perkin Elmer spectrum RX-1 model USA make was used for characterization of the material using KBr pellets in the range 4,000 – 400 cm<sup>-1</sup> and wave numbers reported in cm<sup>-1</sup>. The thermogravimetric analysis (TGA) was performed under nitrogen atmosphere (heating rate 20 °C/min, nitrogen flow 60 mL/min) using SETSYS EVO2400, SETARAM. DSC studies were carried out under high purity nitrogen atmosphere (heating rate 10 °C/min, nitrogen flow 60 mL/min) using DSC 2910 V4.4E TA instruments. The X-ray diffraction experiments were performed on thin film samples by means of Rigaku X-ray diffractometer, operating at 40kV/30 mA and having Cu-K<sub>α</sub> radiation selected by a graphite monochromator. The surface morphology,

microstructure and scanning electron microscope (SEM) studies were carried out using Carl Zeiss Evo 50 SEM.

### 3 Results and discussion

The polycarbosilane synthesized showed characteristic IR absorption peaks at  $\sim 1404$ ,  $\sim 2462$ ,  $\sim 2893$ ,  $\sim 2951$   $\text{cm}^{-1}$  ( $\nu$  C-H),  $\sim 650$ ,  $\sim 800$  and  $\sim 1246$   $\text{cm}^{-1}$  ( $\nu$  Si-CH<sub>3</sub>),  $\sim 1023$  and  $\sim 1352$   $\text{cm}^{-1}$  ( $\nu$  Si-CH<sub>2</sub>-Si-) and  $\sim 2100$   $\text{cm}^{-1}$  ( $\nu$  Si-H) [22,24]. With the progress of the reaction between polycarbosilane with epoxy, Si-H absorption peak at  $\sim 2100$   $\text{cm}^{-1}$  starts diminishing, showing other peaks at  $\sim 1024$   $\text{cm}^{-1}$  ( $\nu$  Si-CH<sub>2</sub>),  $\sim 1255$   $\text{cm}^{-1}$  ( $\nu$  SiCH<sub>3</sub>) and  $\sim 3488$   $\text{cm}^{-1}$  ( $\nu$  OH). This on further reaction with polyols, di-isocyanates and diamine shows IR peaks at  $\sim 3324$   $\text{cm}^{-1}$  ( $\nu$  NH str),  $\sim 2938$   $\text{cm}^{-1}$  ( $\nu$  CH str),  $\sim 2107$   $\text{cm}^{-1}$  ( $\nu$  SiH),  $\sim 1687$   $\text{cm}^{-1}$  ( $\nu$  Si-CH),  $\sim 1541$   $\text{cm}^{-1}$  ( $\nu$  NH def),  $\sim 1261$   $\text{cm}^{-1}$  ( $\nu$  SiCH<sub>3</sub>),  $\sim 1049$   $\text{cm}^{-1}$  ( $\nu$  Si-CH<sub>2</sub>-Si) (fig. 1).

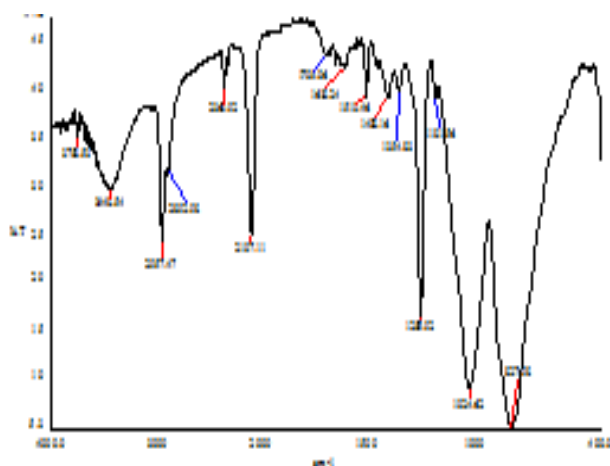


Fig. 1a. FTIR spectra of epoxy polycarbosilane

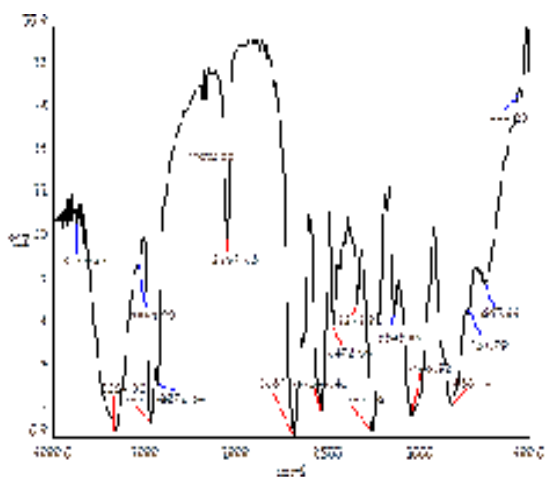


Fig. 1b. FTIR spectra of preceramic foam

Thermogravimetric analysis of preceramic foam shows a three step decomposition as shown in fig. 2. First degradation starts at  $\sim 150$  °C with a  $\sim 7\%$  weight loss which may be attributed to the trapped impurities and decomposition of low molecular weight polymers. Weight loss of  $\sim 45\%$  at  $310$  °C in the second stage, may be attributed to the evolution of methane and other

hydrocarbon gases and may be due to the decomposition of polyurethane foam formed during the reaction. Moreover, a weight loss of  $\sim 12\%$  at  $440$  °C in the third step is also seen, which may be due to the decomposition of higher molecular weight polymers. Further, there was no further weight loss upto  $1000$  °C. Thus around  $40\%$  residue was left up to  $1000$  °C.

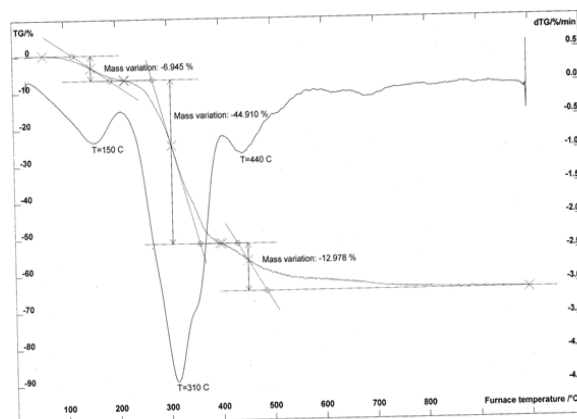


Fig. 2 TGA graph of preceramic foam

The DSC studies of preceramic foam from ambient to  $500$  °C showed (fig. 3) a very small endotherm at  $\sim 113$  °C ( $11.72$  J/g) and an exotherm transition at  $\sim 366$  °C ( $236.7$  J/g). Endotherm at  $\sim 113$  °C may be attributed to the melting of the polymer. Exotherm appearing at  $\sim 366$  °C may be attributed due to the cleavage of Si-Si bonds which is well in accordance with the significant weight loss of  $45\%$  in the range of  $310$ - $360$  °C.

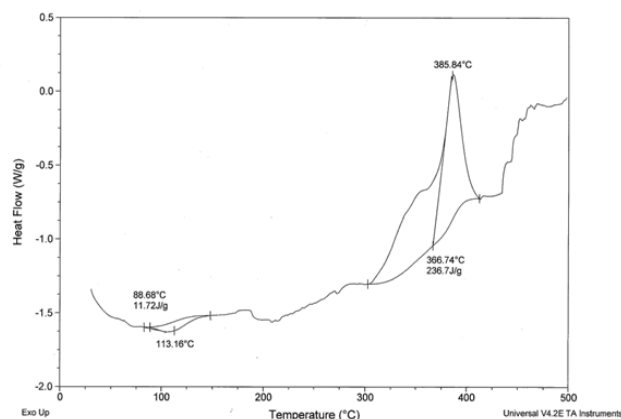
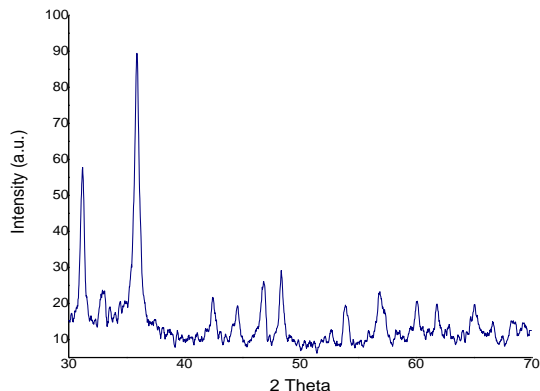


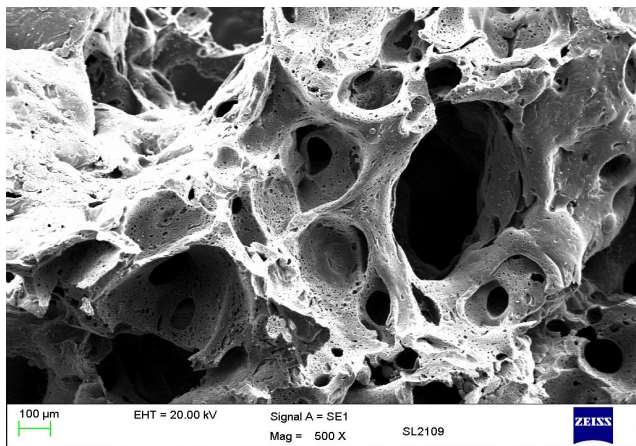
Fig. 3 DSC plot of preceramic foam

XRD spectrum of the pyrolysed product is shown in fig. 4. It showed the presence of 2 theta peaks at  $36^\circ$  (110),  $47^\circ$  (200) and  $60^\circ$  (220) which indicates the formation of  $\beta$ -SiC and the peaks are similar to the reference JCPDS.

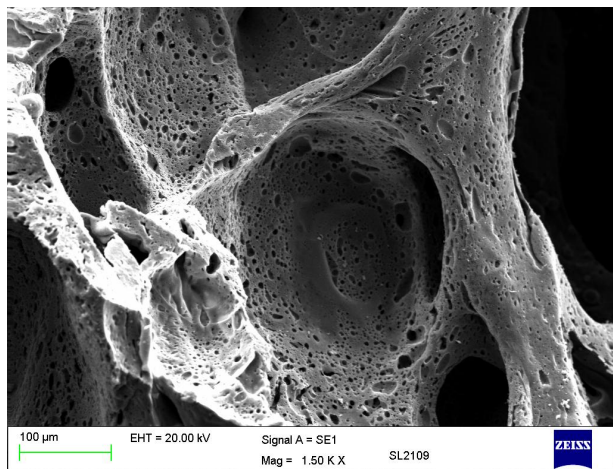


**Fig.4** XRD pattern of silicon carbide foam

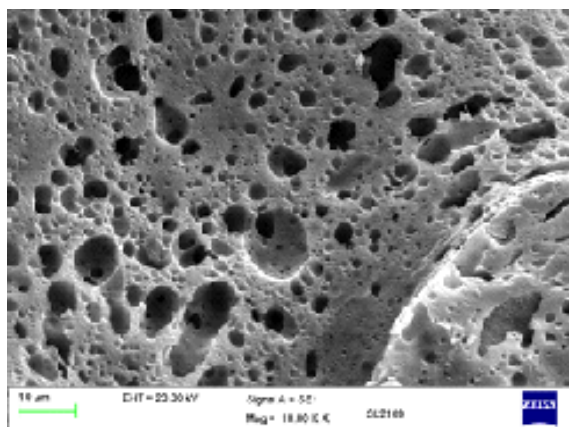
Fig. 5(a,b,c) shows a series of the SEM micrographs of preceramic foam produced after the pyrolysis at different magnifications 500X, 1.50KX, 10.0 KX. All of the figures show the open cell porous structure with interconnected pores with a few of closed cells.



**Fig. 5a.** SEM image of SiC Foam at 500X



**Fig. 5b.** SEM image of SiC at 1.50K X



**Fig. 5c.** SEM image of SiC foam at 10.00KX

## 4 Conclusion

It may be concluded out that using functional polycarbosilane bearing reactive secondary alcoholic group reacted with diisocyanate & polyols which yielded in preceramic foams & thereby on its pyrolysis under strict inert atmosphere, we have been able to synthesize open cell silicon carbide foam.

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