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Original Article

Mechanical Behaviour and Morphological Studies of Infused Carbon Fillers with Different Weight Ratios in Polyurethane Foam

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Abstract *- Polyurethane foams with short carbon fiber materials are being used in shape memory, aerospace, sports goods, and biomedical applications. This study emphasises the tensile behaviour of Polyurethane (PU) foam added with Chopped Carbon Fibers (CCF) mixture of 250 microns with different weight ratios 0.25%, 0.5% and 1%. Four configurations are cast in a mould, tensile samples are prepared and experimentally validated in a universal testing machine by capturing cross-head displacements and gauge strains. A load displacement drop is noticed to 1.61 times for a maximum weight percentage of 1% CCF mixture. Experimental results showed that the addition of chopped carbon fibers to PU foam has improved the tensile strength. CCF content of 0.5 wt% % showed a tensile strength increase to 73% and a tensile modulus increase to 56% compared to specimens with 0 wt% of CCF content. A morphological examination was carried out, and the developments in TS25C, TS50C, and TS100C specimens showed improvement in cell density from 1.95 to 6 times and a drop in cell size from 13.68 to 75.89%. Maximum density increased in TS100C specimens is upto 19.6% as compared to TS0C. Agglomeration is noticed with a percentage increase of CCF in PUF with more than 0.5 wt%.*

Keywords *- Chopped carbon fibers, Polyurethane foam cast, Tensile behavior study, Displacement and strain characteristics, SEM morphology.*

1. Introduction

A mixture of chopped carbon fiber and PU foam finds a place in lightweight structural applications in aerospace, automotive, and sports goods. The addition of CCF enhances the strength characteristics and conductivity, making the product more suitable for the intended use. Fundamentals of the tensile test method and the experimentation standard procedures are adopted through ASTM standards [1]. The fundamental aspects of lightweight structures using foam and the new challenges with in-process methodologies are investigated [2]. Carbon nanofibers in composite applications add up advantages and are reviewed [3]. The advantages of using nano-composite foams in both industrial and biomedical applications, as well as meeting current and future trends, were discussed [4]. Synthesising the polymers is a special technique, and such processing techniques of nanocomposite-based based polymers are elaborated [5]. Dispersing these nano compounds is very intensive. A new method of dispersion of carbon nanotubes in polyurethane matrix and mixing them by sonification is studied [6]. During the process of mixing or reactive foaming, certain defects may be introduced in the structure. The reactive foaming of nano dispersions resulted in structural defects in the foam [7]. Fundamental structure/property relationships and the overall manufacturing techniques to meet the end applications are of the utmost importance, and such studies are made [8] in polymer nano-composite materials.

After producing these polymeric nano-composite foams, their detailed characterization needs to be studied and reviewed [9]. Carbon nanofibers can act as filling agents between the foam bubbles during the foaming process. A method of preventing the collapse of synthesised PU foam is important by adding a small amount of carbon nanofibers [10]. The dispersion of carbon nanotubes in polyol by the mechanochemical approach, with the assistance of a specific dispersing agent, and the tensile strength is investigated [11]. A combination of polyurethane foam with nano-composite fillers has led to an increase in electrical conductivity, thermal conductivity, and mechanical strength. These nano fillers are more prone to the biomedical field; studies have been done to expand into automotive and aerospace [12]. To meet the structural requirements, the orientation of these fillers plays a vital role, wherein the enhancement of mechanical properties by low and medium-density nanophased polyurethane foams with randomly oriented nanofibers as compared to the neat

ones is studied [13]. As a part of extension with multi-addition of particles, the effect of different types of nanoparticles on the mechanical performance of rigid polyurethane foam is also investigated [14].

Foam is soft in nature compared to carbon nanoparticles, so the improvement in mechanical performance with low deformations in polymer/carbon nanotube composites [15] is also important. Different types of fibers, such as magnetised fibres, showed an increase in interfacial strength, and with the introduction of aligned CF, Microcrystalline Cellulose (MCC) fibers as fillers, the tensile strength and stiffness of the composites [16, 23]. Tensile strength and Young's modulus have also increased with increasing Al2O3 nano-composites and are investigated [17]. The water-activated polyurethane–glass fiber composite, when reinforced with amino-functionalized carbon nanofibers, has improved the tensile strength. This study also found a reduction in Young's modulus at higher applied load levels [18]. Multiple choices of filler addition were attempted, and it was found that PU foam with carbon fillers has better tensile strength compared to the PU foam with glass fibers [19]. Finally, the addition of fillers in polyurethane foam has increased the tensile strength [20 - 22].

A literature survey reveals several advantages of using carbon fibers and their performance when mixed with foam. The current study is aimed at investigating the addition of CCF in different weight ratios of PU foam solution. Three different percentages were studied, tensile specimens were manufactured, and experimentally tested. Morphological studies were examined, and dispersion and failure behaviour were also studied. Post tests were examined, and tensile strength characteristics were plotted.

2. Materials Constituents

2.1. PU Foam

PU foam is blended with liquid precursors of Polyol (JD 98025) and Isocyanate (MDI 5005) by preparation of 1:1.2 by volume. The constituents are manufactured by m/s Hunstman (India) Private Limited. The parameters of material constituents are shown in Table 1.

Table 1. Parameters of material constituents

JD	MDI
Hydroxyl, 410 mg KOH/gm	Viscosity, 350 cPs at 25˚C
Viscosity, 3500 cPs at 25˚C	NCO, 35% content
PH, 8	
Specific Gravity, 1.2	

2.2. Chopped Carbon Fiber (CCF)

CCFs are considered for this study, and the basic material is prepared from intermittent modulus carbon fiber, and the specifications are tabulated in Table 2.

Table 2. Specifications of CCF

Parameter	Value
Length of the fiber (microns)	250
Diameter of the filament (µm)	8
Density (g/cc)	1.78
Modulus (GPa)	230
Carbon content	90%

3. Preparatory Process

3.1. PUF-CCF Mixture

The addition of CCF in 0.25%, 0.5% and 1% weight ratios with respect to the PU foam mixture is studied. Mechanical stirring is done at 1400 rpm for a duration of 5 minutes to properly distribute CCF in JD. The compound is added to MDI and stirred for about 10 seconds. The whole mixture is poured into an open free-size mould, and the lid is closed to maintain block dimensions of 170X100X10 mm. After leaving for 24 hours at room temperature, the foam blocks are extracted and trimmed at the edges. The process is repeated for every weight ratio of 0.25%, 0.5% and 1% of CCF Mixture, and the typical manufacturing process is represented in Figure 1.

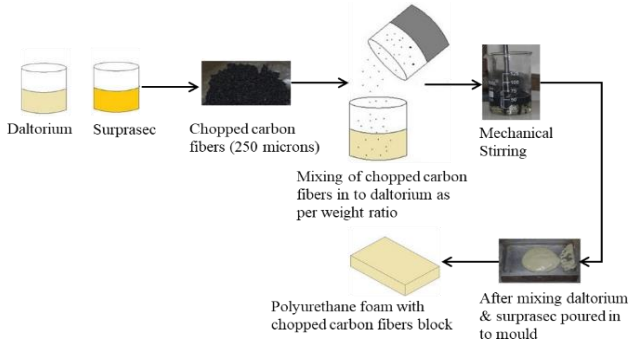
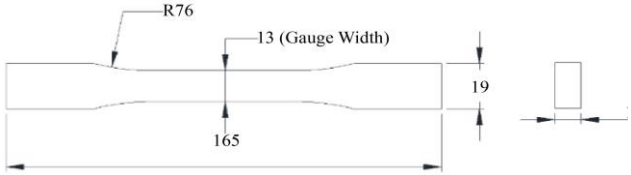


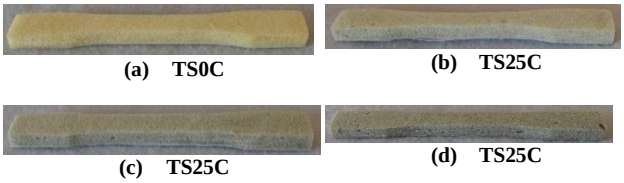
Fig. 1 Schematic image of polyurethane with carbon nanoparticles preparation

3.2. Specimen Sizes

The specimen sizes are cut as per the ASTM standard [1] dimensions. The length of specimens is maintained at 165 mm, and the thickness is 10 mm with a gauge width of 13 mm.



(a) Specimen dimensions



(a) TS0C **(b) TS25C**

(c) TS25C **(d) TS25C**

Fig. 2 Specimen configuration

Four different configurations are manufactured and named TS0C, with no addition of CCF. CCF was added in percentages of 0.25, 0.5, and 1.0, which were named TS25C, TS50C, and TS100C, respectively. The specimen dimensions are represented in Figure 2 (a), and the types of specimens are shown in Figure 2(b) to 2(e). The colour variation in specimen types is noticed from a pale yellow colour in TS0C and gradually to dark blackish in TS100C, indicating the addition of CCF percentages.

4. Mechanical Testing

Mechanical tests, including density examination and tensile tests, are conducted on all specimen configurations.

4.1. Density Measurement

Density of specimens ρ is measured taking into consideration physical mass, volume according to ASTM standard D792.

$$\rho = \frac{m}{v} \tag{1}$$

Where m represents the measured mass (kg) of each specimen and v represents the volume (m^3) obtained by multiplying the length, width, and thickness of the specimen. It is observed that the plain foam TS0C has a density of 106 kg/m^3, whereas mixing of CCF resulted in an increase in density up to 128 kg/m^3. Linear increment is noticed as per the percentage weight addition of CCF in PU foam. 5.6, 15, and 19.6% increase in density is noticed in TS25C, TS50C, and TS100C as compared to TS0C, and the variation is plotted in Figure 3.

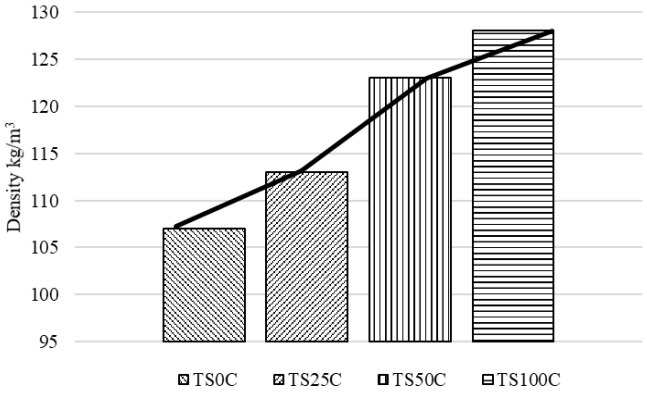


Fig. 3 Measured density of specimens

4.2. Tensile Test

A tensile test is calculated using ASTM D638 and a universal machine. Being the foam soft in nature compared to pure composites or metals, rubberised grips are preferred for tests.

The specimens are loaded quasi-statically till failure, maintaining a cross-head speed of 0.5mm/min. 2.5 kN load cell in an ADMET model 2656 machine with a data acquisition system 5000 model, and M-test Quarto software is used for the purpose. The alignment of the specimen between the upper and lower grips in both planes is ensured during specimen gripping. Biaxial strain gauges are bonded on the specimen at the centre to record strains along the longitudinal and lateral directions. The test setup is represented in Figure 4 with the specimen position.

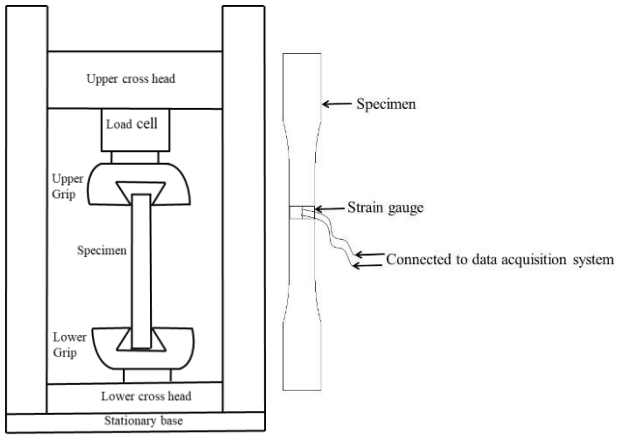


Fig. 4 Experimental test setup

5. Results and Discussions

5.1. Displacement and Strains

The specimens are loaded into the testing machine and tested up to failure. Cross-head displacements, loads, and strain are recorded during the experiment. The specimens are tested to failure, and continuous recording of values is done. The maximum load of 100N is noticed for the TS0C specimen. The load capability of the CCF added specimens gradually increased to 125 N for TS25C and 166 N for TS50C. However, the highest addition of CCF in TS100C specimens has failed at 161 N load. On the other hand, the maximum displacements are recorded as 3 mm for plain TS0C, 1.79 mm for TS25C, 2.6 mm for TS50C, and 2.3 mm for TS100C, respectively. Load displacement curves are plotted in Figure 5 (a).

The tensile strength σ_{ij} of the specimens is computed by taking into account the initial cross-sectional area (A) and the maximum load P_{max}.

$$\sigma_{ij} = \frac{P_{\max}}{A_{ij}} \tag{2}$$

The tensile strength has increased for the specimens from 0.74 MPa to 1.28 MPa for TS50C, and a drop in strength is noticed in the TS100C specimen to 1.24 MPa. The tensile strength variations are plotted in Figure 5(b).

The load capability is directly proportional to the maximum tensile strength of the material and is noticed with

the addition of CCF. However, with the addition of more than 0.5 wt% of CCF, agglomeration with non-uniformity spread resulted in a decrease of tensile strength as well as failure load. The longitudinal and lateral strains are also recorded against the load and are plotted in Figures 5(c) and 5(d), respectively. Positive trend of lateral strain recorded from 4000 με, 3650 με, and 2809 με, with a sudden increase to 4400 με, is observed in TS0C, TS25C, TS50C, and TS100C specimens.

All the curves in the graph followed a similar trend, and a drop in trend for TS100C is noticed following the curve of TS25C. CCF has provided more resistance, and in turn, lower strains are noticed than in pristine foam specimens, except in highly CCF-stuffed specimens of TS100C. In similar lines, the lateral strain showing negative values (compressive in nature) was also recorded.

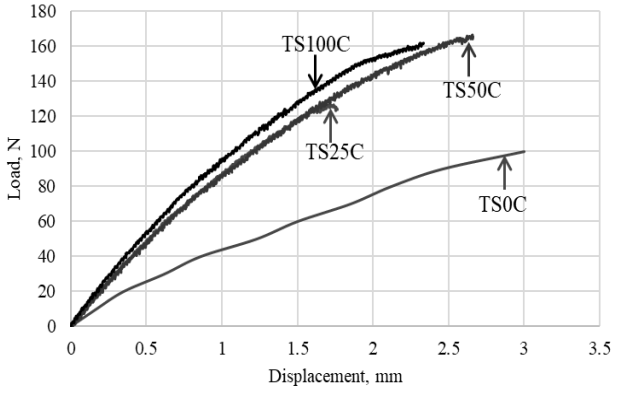
Fluctuating values in incremental and decremental of 1289 με, 1878 με, 1344 με, and 1570 με are noticed in TS0C, TS25C, TS50C, and TS100C, respectively. The ratio of lateral $-\Delta\varepsilon_T$ to longitudinal $-\Delta\varepsilon_L$ strain showed a decremental trend from 0.32 to 0.47 and then back to almost 0.35 in specimens as plotted in Figure 5(e), and Poisson's ratio, ν is computed.

$$\nu = \frac{-\Delta\varepsilon_T}{\Delta\varepsilon_L} \tag{3}$$

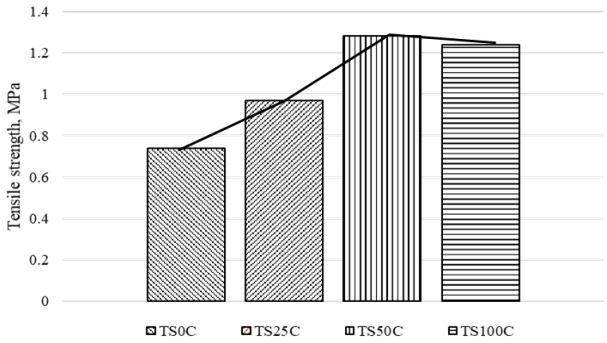
The tensile modulus of these samples is also measured from the slopes obtained in the linear portion and is plotted in Figure 5(f). The values have increased to TS50C and then dropped for TS100C, resulting in a limit to 0.5% addition of CCF in PU foam.

Tensile Modulus, E of the specimens is computed taking into account the change in strain ($\Delta\varepsilon_{ij}$) and the change in stress ($\Delta\sigma_{ij}$) within the elastic limit.

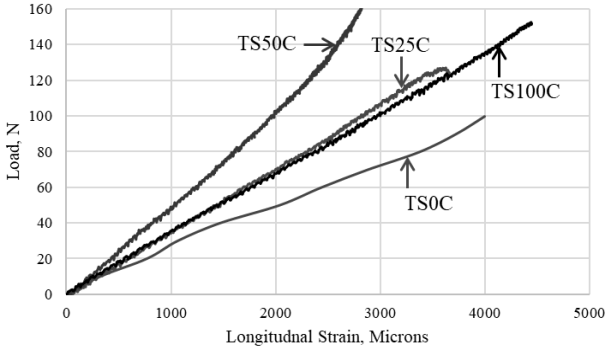
$$E = \frac{\Delta\sigma_{ij}}{\Delta\varepsilon_{ij}} \tag{4}$$



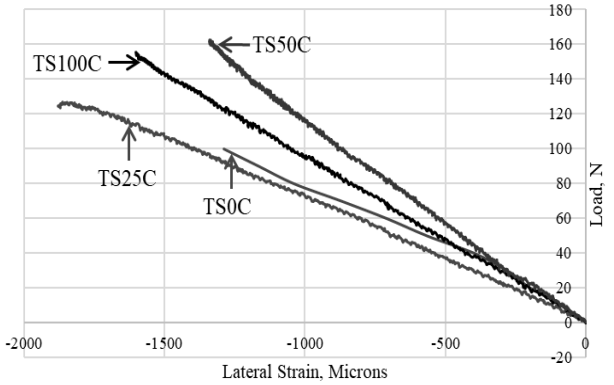
(a) Load vs Displacement



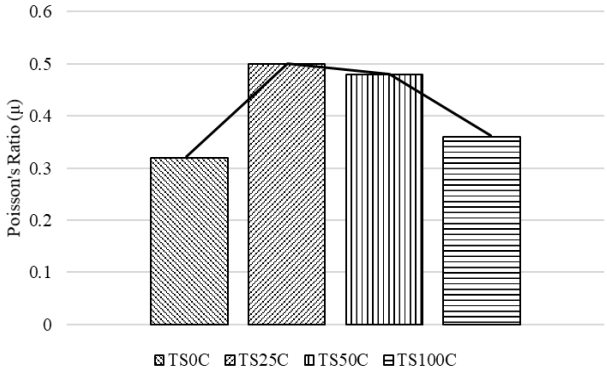
(b) Tensile Strength



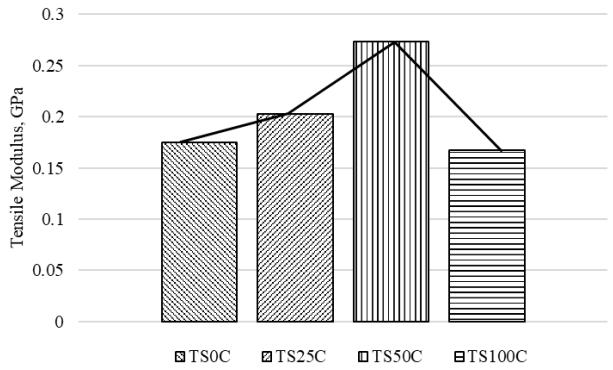
(c) Longitudinal Strain



(d) Lateral Strain



(e) Poisson's Ratio



(f) Tensile Modulus

Fig. 5 Experimental results graphical representation (a) Load vs Displacement, (b) Tensile strength, (c) Longitudinal strain, (d) Lateral strain, (e) Poisson's ratio, and (f) Tensile modulus.

5.2. Failure Behaviour of Specimens

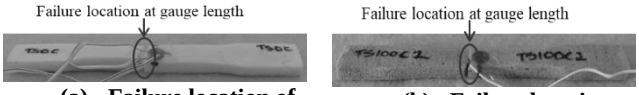
After the test, the specimens are physically examined, and a lateral crack is found at the center along the full width of the specimen. TS0C, TS25C, TS50C, and TS100C noticed a similar type of failure behaviour within the gauge lengths, and the specimens are separated into two pieces at the mid region of the overall specimen length.

The comparison before and after the test is shown in Figure 6. The loaded specimen is represented in Figure 6(a), and the tested specimens of TS0C and TS100C are represented in Figures 6(b) and 6(c), respectively.

(a) Loaded Sample **(b) Post Test of TS0C** **(c) Post Test of TS100C**

Fig. 6 Failure pattern

All the specimen types failed in gauge length and were normal in the loading direction. Visual examination of post-test specimens representing failure is shown in Figures 7(a) and 7(b).

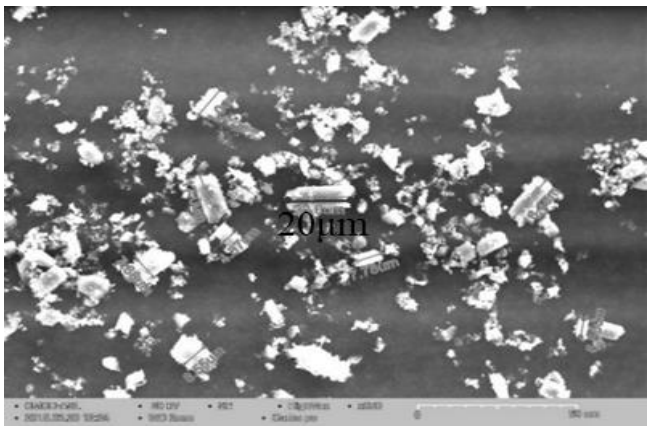


(a) Failure location of TS0C **(b) Failure location TS100C**

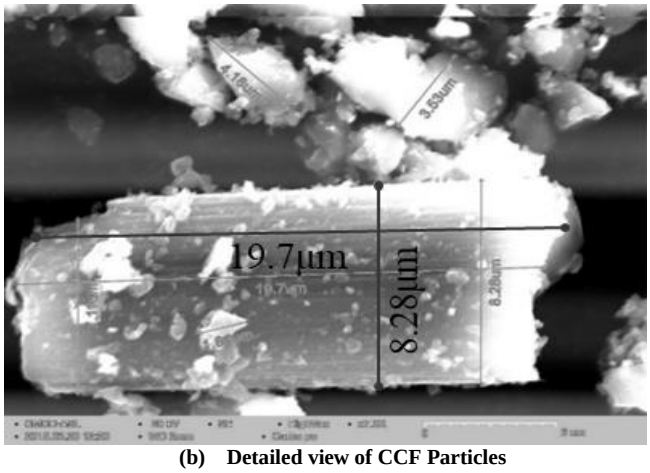
Fig. 7 Specimens after the test

5.3. Morphological Examination

The Scanning Electron Microscope (SEM) image of the samples was obtained from SEM (SNE-4500M Plus, Acceleration voltage 1-30 KV, Magnification range 30X – 1,50,000X, Made in Korea).



(a) CCF Particles

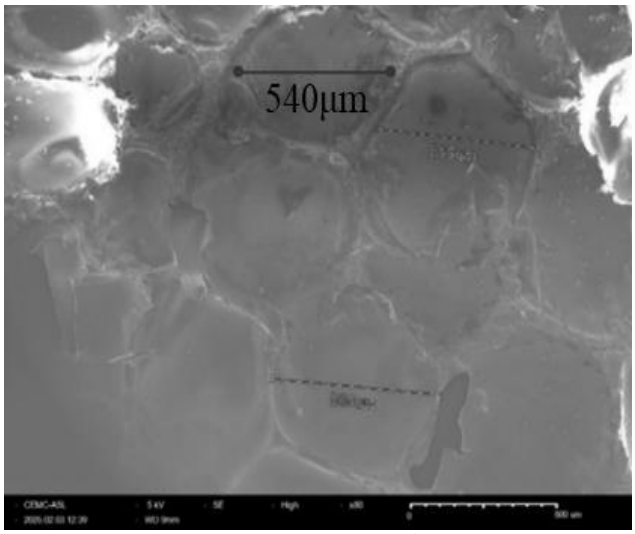


(b) Detailed view of CCF Particles

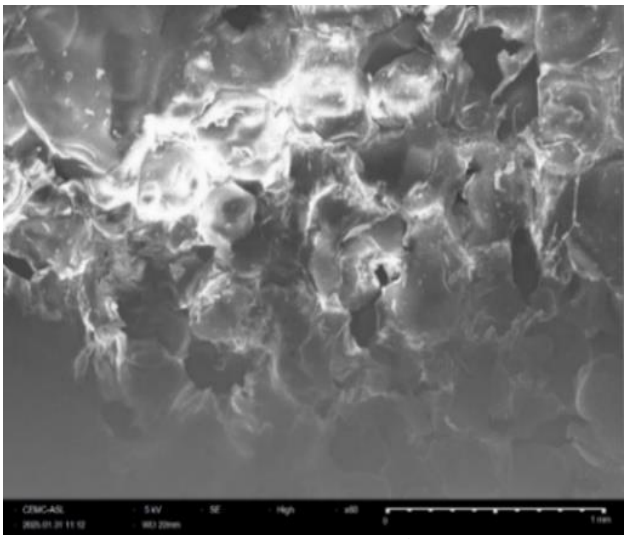
Fig. 8 SEM micrograph of chopped carbon fibers

The SEM images of CCF particles, along with a detailed view, are shown in Figure 8(a)-(b). The diameter of the fiber is 8.28 microns, and the length is about 19.7 microns, which is in correlation with Table 2.

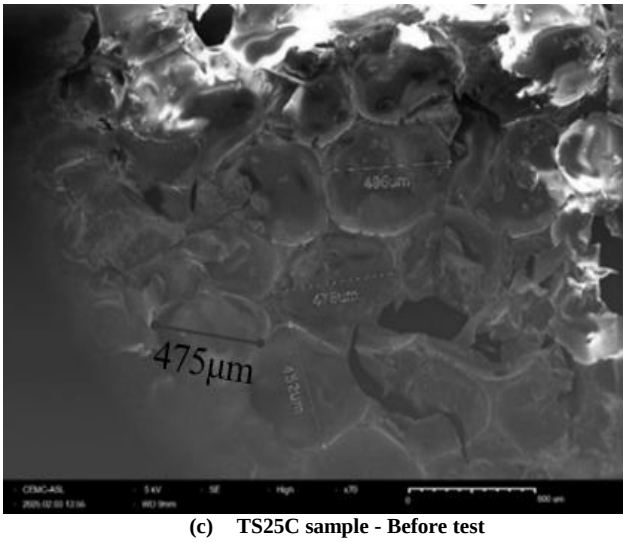
The images in Figure 9(a)-(h) show variation in the morphology of the foam cells with the increased loading of the carbonaceous fillers. When there are no fillers, the shape of the cell was observed to be a defined, uniform shape with an approximate size of around 500 to 560 microns. One image showing about 540 microns is depicted in Figure 9(a). These foams without any fillers were also observed to have defined cell boundary layers between the cells. As the carbonaceous fillers were added to the foam, the cell size gradually decreased. This can be attributed to the fact that the fillers act as nucleating sites for the reacting species, thereby providing increased avenues for reactions and thus cell growth [10].



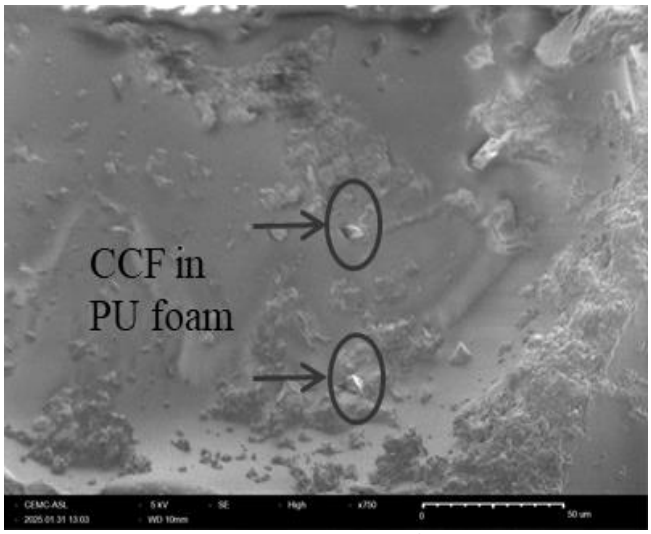
(a) TS0C sample - Before test



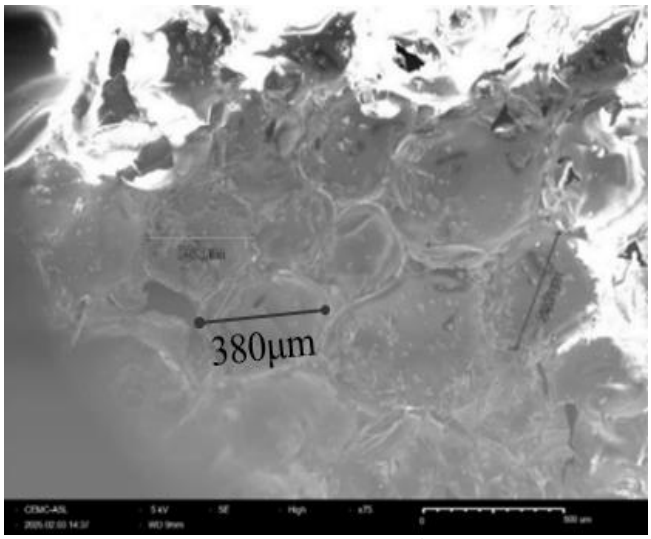
(b) TS0C sample - After test



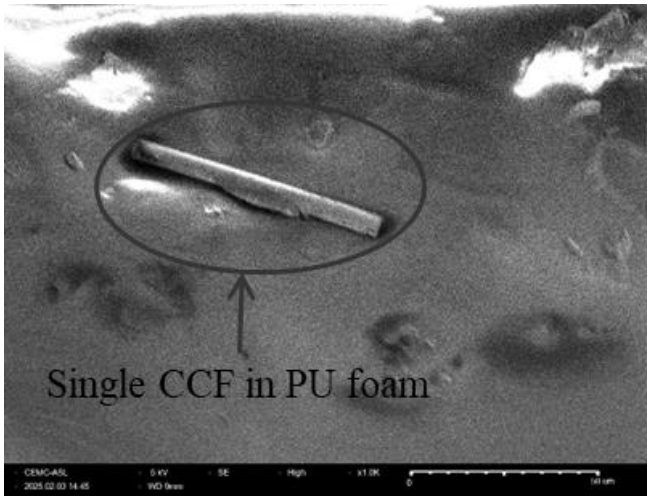
(c) TS25C sample - Before test



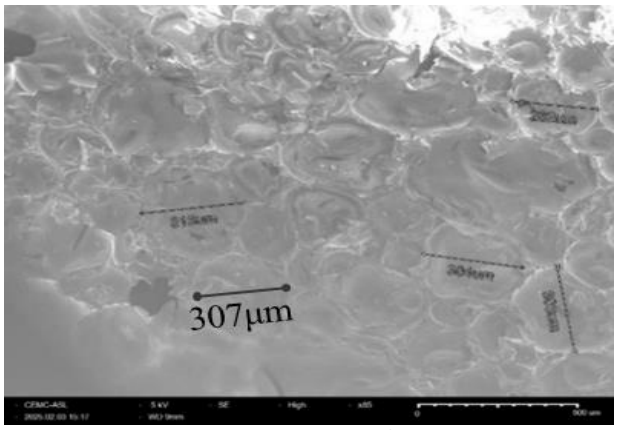
(d) TS25C sample - After test



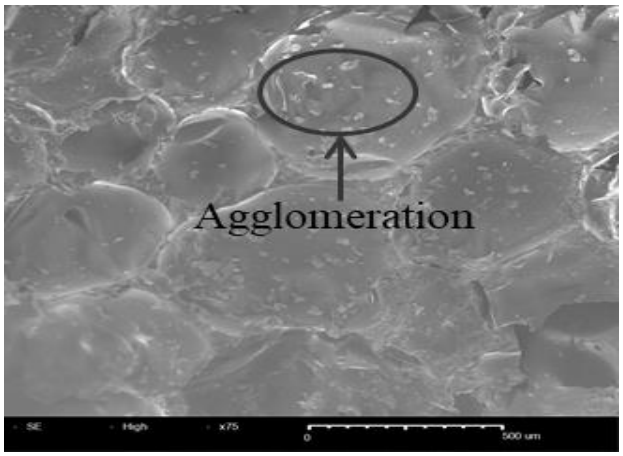
(e) TS50C sample - Before test



(f) TS50C sample - After test



(g) TS100C sample - Before test



(h) TS100C sample - After test

Fig. 9 (a)-(h) SEM micrograph of pristine and tested samples of PU foam with carbon nanofibers

At lower concentrations of carbonaceous fillers Figure 9(c), the reduction in cell size is marginal. The majority of the cells were found to have around 475 microns in size, resulting in an about 88% drop compared to the original cell size. This indicates that the fillers have not obstructed basic diffusion mechanisms of the reacting species (foaming chemicals).

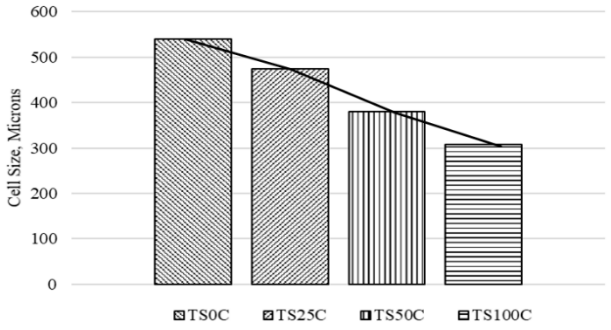
However, as the concentration of the fillers has increased, the sizes have come down drastically, and have also been found to have non-uniform cell sizes spread across the space. Many number of small-sized cells were found in Figure 9(e), which are randomly positioned along with the larger-sized cells.

As the concentration of the filler was increased further, Figure 9(g), structural reversal of the foam took place; the foam cells were predominantly small in size, with large-size cells randomly dispersed in them. The overall reduction of about 56% is noticed in TS100C samples.

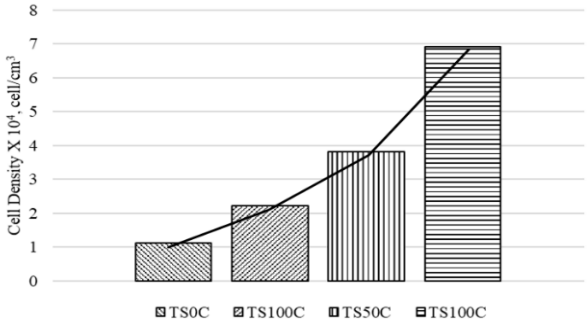
For instance, at 0% concentration of the fillers, the cell structures were found to be a larger number of cells with around 50 microns in size, with a few cells having 300 microns. This indicates that, at higher concentrations of the fillers, due to more availability of the nucleating sites, the available crosslinking groups get diffused randomly towards the nearest nucleating site, resulting in a smaller size. Cell collapse, though observed at higher concentrations, is very low compared to the reference sample. Cell boundaries are also not prominent after the structural reversal of the foam. The significance of this structure reversal is that the collapsed cells and their sizes become insignificant at this concentration. After the test, in the TS0C specimen, Figure 9(b), the collapse of foam cells all along the loading direction due to elongation is noticed in the SEM micrograph. A good dispersion of chopped carbon fibers in PU foam is seen in TS25C and TS50C, as shown in Figures 9(d), (f). Some spotting indications representing the probable start of agglomeration are noticed with the detailed view of CCF in foam structure in Figure 9(f). More enhancement of agglomeration is noticed in the TS100C specimen, though the SEM micrograph in Figure 9(h). Due to a smaller number of collapsed cells and more cell boundary areas, which can act as crack deflectors, such transformed cell-structured foams can display higher mechanical properties. It can be seen that, at a concentration of 0.5 wt% of CCF, where structural reversal has happened, tensile strength and modulus were found to be highest.

5.4. Cell Size and Density

With an increase in the chopped carbon fiber content in PU foam, cell size started decreasing, as shown in Figure 10(a). The cell density of the TS50C specimen has increased up to 3.2 times compared with the TS0C.



(a) Cell Size



(b) Cell Density

Fig. 10 Graphical representation of cell size and cell density

The cell density has a reverse effect compared to cell size. The overall increment of 6 times of cell density is noticed in TS100C as compared to TS0C, and the variation is represented in Figure 10(b). The cell density (N_f) is calculated [10] using the following equation:

$$N_f = \left(\frac{nM^2}{A} \right)^{\frac{3}{2}} \quad (5)$$

Where n – the number of cells, A – the area of the micrograph (cm^2), M — the magnification factor.

6. Conclusion

This study focused on evaluating the tensile strength of 0%, 0.25, 0.5, and 1 wt% CCF-infused polyurethane foam. The inclusion of CCF increases the mechanical characteristics of the foam. The results of the tensile test showed that the addition of CCF improved the tensile strength to a specific CCF content of 0.25, 0.5 %. However, the further addition of 1 % caused an inverse effect. The inclusion of CCF up to 0.5 % has a maximum tensile strength increase of 73 % and a tensile modulus of 56 % compared to plain PU foam. A good dispersion of carbon nano fibers throughout the PU matrix and the strong interfacial adhesion between fibers and the matrix are proposed to be responsible for the significant mechanical enhancement. The scanning electron microscopic test shows the decrease in cell size, which means an increase in cell density, compared to 0% CCF in PU foam. There is no evidence of agglomeration in 0.25% and 0.5 %, but 1 % of the sample shows the agglomeration of CCF in the foam. SEM micrograph of TS25C, TS50C, and TS100C specimen results showed a decrease in cell size to 13.68, 42.1, and 75.89% and an increase in cell density to 1.95, 3.2, and 6 times compared with TS0C. Temperature-dependent studies and interfacial behavior can be explored in detail for futuristic studies.

Declaration of Conflicting Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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