# EFFECT OF MULTI-WALLED CARBON NANOTUBES ON MECTANICAL PROPERTIES OF CARBON FIBER POLYURETHANE FOAM SANDWICH STRUCTURES: OPTIMIZATION OF PARAMETERS USING TAGUCHI'S DESIGN

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#### Abstract

Dispersing of the nanoparticles into the polymer can improve their mechanical and thermal properties. Due to having high surface energy and tendency to agglomeration of nanoparticles, dispersing method is the most critical issue. Sonicator is the one of the efficient method for dispersion of nanoparticles using acoustic cavitation principle. Homogenizer is the effective method and high shear forces are used in this method. In present work, multi-wall nanotubes (MWNTs) are dispersed in the rigid polyurethane (PU) foam to use as core materials. Two different L9 Taguchi design are performed to obtain best dispersion parameters and their levels for both PU foam and epoxy resin. According to mechanical test results of nanophased foam and epoxy, nanoreinforced sandwich structure are produced and tested.

#### 1. Introduction

Sandwich composites have been an increasing interest in aerospace, automotive and other high tech industries. Main advantages of sandwich composites are high rigidity, excellent thermal insulation, ease of machining, low density etc. [1]. Honeycombs and foams are the most widely used core materials, however foams provide higher surface area for bonding with composite sheet and manufacture is easier. Polymer foams has been used for many years as lightweight supporting in varied applications and polyurethane (PU) foam is widely used in thermal insulation materials for buildings as the high energy saver [2]. Compared with high performance structural foam cores, they have lower mechanical properties (such as polymethacrylimide foam) and neat PU is not suitable as core materials [3]. Integrating nanoparticles to PU can improve mechanical, thermal and electrical properties of core materials, because of extraordinary properties of carbon nanotubes (CNTs) [4, 5]. Although CNTs have high mechanical properties, they tend to agglomerate due to their high surface energy. Therefore, homogeneous dispersion and distribution of nanoparticles in the matrix are the most critical steps [6].

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Figure 1. Nanophased PU foam manufacturing process.

In this study, multi-walled carbon nanotubes (MWNTs) are dispersed in rigid polyurethane foam system. PU foams are formed from two components named as diphenylmethane diisocyanate (MDI) and polyol. In previous studies, nanocomposite PU foam showed better mechanical properties when the MWNTs were dispersed in polyol component, so nanoparticles are dispersed in polyol using homogenizer and horn sonicator, simultaneously (see Fig. 1). An experimental design is performed to obtain best dispersion parameter level doing minimum number of experiment with Taguchi method which is shown in Table 1. Neat and nanoreinforced PU foam in which showed highest mechanical properties are characterized using scanning electron microscopy (SEM) and thermogravimetric analysis for the cell size and distribution and thermal stability to investigate the CNT effect. Besides MWNTs are dispersed in epoxy resin to improve mechanical and thermal properties of sandwich composites and another Taguchi design is set for homogeneous MWNTs dispersing in epoxy as shown in Table 2. Several mechanical test are performed to obtain highest mechanical properties both foams and epoxies, and according the test results sandwich composites (MWNTs-PU/MWNTs-epoxy/carbon fiber) are manufactured using vacuum infusion process (VIP) and also compression and 3 point bending tests are performed to the sandwich composites. In the next steps shear and climbing drum peel tests will be performed to determine shear strength and adhesion properties between core materials and composite sheets.

 Table 1. Experimental design of nanocomposite foam manufacturing.

Parameters	Level-1	Level-2	Level-3
Amount of MWNTs (%)	0,01	0,05	0,1
Homogenizer time (min.)	0	30	60
Horn sonicator time (min.)	0	5	10
Extra mechanical stirrer process position	А	В	С

## 2. Experimental

## 2.1. Materials

 $80 \text{ kg/m}^3$  liquid form two component PU foam materials are supplied by Flokser Textile. Part A and part-B are named MDI and polyol respectively. MWNTs have innermost diameter of  $4.5\pm0.5$  nm, outer diameter of  $10\pm1$  nm and length of  $3-6\mu$ m and they are supplied from Sigma Aldrich (773840). As the matrix, Hexion MGS L160 epoxy resin and MGS H 160 are used.

Parameters	Level-1	Level-2	Level-3
Amount of MWNTs (%)	0,1	0,2	0,4
Homogenizer time (min.)	30	45	60
Homogenizer revolution (rpm.)	10.000	15.000	18.000
Horn sonicator time (min.)	5	10	15

**Table 2.** Experimental design of MWNTs/Epoxy.

#### 2.2. Preparation of MWNTs/PU nanocomposite foams and MWNTs/Epoxy nanocomposites

Manufacturing of nanocomposite foam is a two-step process as shown in Fig. 1. First step is dispersing of MWNTs in part-B using 7.000 rpm homogenizer and 20 Hz. 150W horn sonicator. L9 Taguchi design includes nine different experiments using three parameters with their three diverse levels. Parameters and their levels are detailed given in the Table 1 and the nine experiments are given in the Table 3. Besides, A, B and C shows extra mechanical stirrer process position in the first step. A means before using homogenizer process MWNTs and part-B will be mixed 2200 rpm 20 min. using mechanical stirrer, B means same process will be performed after homogenizer and C means after sonicator process mechanical stirrer will be performed. During the whole dispersion step, temperature rise is resisted using ices. In the second step obtained liquid and part-A are mixed ( polyol and MDI weight ratio 100:100) using mechanical stirrer at 2200 rpm for about 15 sec and the mixing are poured into 23 °C aluminum open mold at atmospheric pressure.

MWNTs/Epoxy manufacturing process is the same with nanocomposite foams except additional mechanical stirrer process. The parameters and levels are given in details in Table 2. In the first step nanoparticles are dispersed in MGS L160 according to Table 4 and then the hardener are added into the mixture (MGS L160 and H160 weight ratio 100:25) and hand mixing are applied until homogeneous distribution is achieved. At the end of the process the mixture are poured into the silicone mold (as desired shape shown in the Fig. 2a.) and post cure is applied at 50  $^{\circ}$ C for 15 hours.



Figure 2. a) Images of the baseline and nanophased epoxy test specimens, b) tensile and c) 3 point bending tests figures.

#### 2.5.1. Compression Tests

Flatwise compression tests are performed according to ASTM C365/C365M- 11a using Shimadzu AG-Xplus 100kN universal test machine. Neat PU foam, nanophased PU foams and nanoreinforced sandwich composites are tested and specimen dimensions of compression test are about 27x27x20 mm3. The deformation rate of compression test are set as 0.5 mm/min., 45N initial load are performed and force-displacement changes are recorded. At least 5 specimens are tested for each sets.

#### 2.5.2. Tensile Tests

Tensile test are performed according to ASTM D368-14 using Shimadzu AG-Xplus 100kN universal test machine. Test specimens (baseline epoxy and all MWNTs/Epoxy) are poured into the dog born shape silicone mold (D: 368-14 type 4, shown in the Fig.2b.). The deformation rate of compression test are set as 1 mm/min. and force-displacement changes are recorded. At least 5 specimens are tested for each sets.

## 2.5.3. Three Point Bending

Three point bending tests are carried out according to ASTM D790 using Shimadzu AG-Xplus 100kN universal test machine at room temperature. Test specimens (baseline epoxy and all MWNTs/Epoxy) are poured into the silicone mold (type 4 ASTM D:368-14) and preferred specimen dimensions for molding materials is 12.7 mm wide, 3.2 mm thick and 127 mm long as illustrated in Fig. 2c. The deformation rate of compression test are set as 1.5 mm/min. and force-displacement changes are recorded. At least 5 specimens are tested for each sets

#### 2.5.4. Mechanical Test Results

Mechanical test results of nanophased PU foams and MWNTs/Epoxy nanocomposites are listed in Table 3. and Table 4., respectively.

#	Amount of MWNTs (%)	Homogenizer time (min.)	Horn sonicator time (min.)	Mechanical stirrer position	Compressive strength (MPa)	Compressive modulus (MPa)
1	0,01	0	0	A		
2	0,01	30	5	В		
3	0,01	60	10	С		
4	0,05	0	5	С		
5	0,05	30	10	А	T	BD
6	0,05	60	0	В		
7	0,1	0	10	В		
8	0,1	30	0	С		
9	0.1	60	5	А		

**Table 3.** Mechanical test results of nanophased PU foams.

#	Amount of MWNTs (%)	Homogenizer time (min.)	Homogenizer rpm.	Horn sonicator time (min.)	Tensile strength (MPa)	Tensile modulus (MPa)	Flexure strength (MPa)	Flexure modulus (MPa)
1	0,1	0	10.000	5	86	4412	142	3706
2	0,1	45	15.000	10				
3	0,1	60	18.000	15		TDD		
4	0,2	0	15.000	15	IBD			
5	0,2	45	18.000	5				
6	0,2	60	10.000	10	74	4212	134	3481
7	0,4	0	18.000	10				
8	0,4	45	10.000	15	77,5	4224	135,5	3510
9	0,4	60	15.000	5				

<b>Fable 4.</b> Mechanical	test results	of MWNTs/Er	oxv.
	test results		/onj.

#### 2.6. Characterization

## 2.5.1. Morphological characterization by SEM

Morphological characterization are investigated using scanning electron microscopy. Neat PU and nanophased/PU which is having best mechanical properties TBD. Fig. 3 shows 0.1wt.% MWNTs/PU foams.



Figure 3. SEM images of 0.1wt.% MWNTs/PU foams.

## 2.5.1. Thermal properties by TGA and DSC

TGA is performed using TA instruments SDTQ 600 to measure the thermal stability of MWNTs, neat PU and nanophased PU foam which is having highest mechanical properties. Specimens are heated to 700 °C from room temperature in a platinum pan at a heating rate  $10^{\circ}$ C/min. in air. Fig. 4b shows that ten percentage of thermal degradation (Td 10%) is found ~528°C).

DSC measurement, to achieved information about effects of the MWNTs on the crystallization behavior of MWNTs/Epoxy nanocomposites. DSC analyses were conducted on TA instrument DSC Q200 and test specimens (baseline epoxy and MWNTs/Epoxy which is having highest mechanical properties) are heated from 0 to 200°C at scan rate 10°C/min.



Figure 4. TGA results of neat and MWNTs/PU and MWNTs.

## 2.7. Manufacturing and Testing of the Sandwich Composites

Sandwich composite is produced used vacuum infusion method. TBD

## 4. Conclusion

TBD

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