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Physical, Mechanical and Thermal Properties of Hybrid Epoxy/ Multi-Walled Carbon Nanotubes/ Silicon Carbide Conductive Nanocomposites

Nurul Hani Noor Asmadi^a, Siti Salmi Samsudin^{a,*}, Mohd Shukry Abdul Majid^b, Mohd Ridzuan Mohd Jamir^b and Azlin Fazlina Osman^a

^aMaterials Department, Faculty of Chemical Engineering & Technology, Universiti Malaysia Perlis (UniMAP), Kompleks Pusat Pengajian Jejawi 2, 02600 Arau, Perlis, Malaysia

^bMechanical Department, Faculty of Mechanical Engineering & Technology, Universiti Malaysia Perlis (UniMAP), Kampus Tetap Pauh Putra, Pauh Putra, 02600 Arau, Perlis, Malaysia

*Corresponding author. E-mail: sitisalmi@unimap.edu.my

ABSTRACT

This study investigates the efficacy of an epoxy composite system that incorporates hybrid nanofillers consisting of multi-walled carbon nanotubes (MWCNTs), and silicon carbide nanoparticles (SiCs), as a means of reinforcing epoxy matrices with enhanced thermal properties. The fabrication of epoxy hybrid nanocomposites was carried out through a solution mixing process involving ultrasonication and planetary centrifugal mixing. Before proceed with the analysing synergistic effect of hybrid filler ratios, the samples were first being investigated on the effect of filler loadings to determine the optimal fillers loading, and it was discussed in other study. It was found that there was a correlation between the thermal properties of the specimens and their respective filler loadings, which an increase in filler loadings led to an increase in thermal properties. The incorporation of 4 vol.% of MWCNTs resulted in a significant enhancement of the thermal conductivity of the composites, reaching a value of 0.46 W/mK. This represents a doubling of the thermal conductivity compared to that of pure epoxy (~0.2 W/mK). Moreover, the hybrid fillers loadings of 3vol.% MWCNT+1vol.% SiC shows a higher thermal conductivity value of 0.48 W/mK which indicates the synergistic effects of hybrid fillers. The epoxy matrix exhibited uniform dispersion of MWCNTs and SiCs, resulting in the establishment of thermally conductive pathways.

Keywords: Nanocomposites, Thermal properties, Multi-Walled Carbon Nanotubes (MWCNT), Silicon Carbide (SiC), Synergistic effects

1. INTRODUCTION

Polymer nanocomposites is an interesting topic in material science due to their potential to outperform pure materials. Electrics and electronics are one industry that keeps focused on this study to solve issues like high cost, longer processing time, and design limits. Due to their versatility, lightweight, and low cost, polymers are used extensively. Thus, it has been widely used in numerous fields, including electronics. The main drawback of this material is its low heat conductivity, which is crucial for power and electrical packing. Epoxy is a thermoset polymer with strong mechanical properties and environmental resilience but limited heat conductivity. Multi-walled carbon nanotubes (MWCNTs) and silicon carbide (SiCs) thermal conductive fillers have improved nanocomposites' thermal and epoxy mechanical properties, increasing reliability. Hybrid fillers create more efficient heat conduction channels because they mix shapes and types (1).

1.1. High Thermal Conductivity Fillers

Multi-walled carbon nanotubes (MWCNT) have excellent thermal conductivity compared to silicon carbide (SiC). The experimental thermal conductivity value for MWCNTs is already high, ranging from 200-3000 W/m K at ambient temperature, whereas the theoretical value is 3000 W/m K

(2). However, multi-walled carbon nanotubes (MWCNT) also possess such a metal behaviour that it is also excellent in electrical conductivity (3). The silicon carbide (SiC) also promoted good thermal conductivity but not as high as the multi-walled carbon nanotubes (MWCNT). However, silicon carbide (SiC) is an excellent material for electrical insulation. Thus, combining these two nanofillers is expected to gain superior thermal conductivity with excellent electrical insulation polymer nanocomposites. The use of nanoscale fillers is to increase the polymer's mechanical and physical properties. The obtained results have improved the composite properties when compared to the traditional polymer composites. Nanoscience and nanotechnology provide a unique opportunity to construct novel combinations of nanoscale fillers and polymer materials to produce polymer nanocomposites with intriguing features (4). In addition, preparing these materials is quite challenging to ensure the interfacial bonding between those materials at the optimum level to create remarkable properties of the composites. The dispersion of the fillers in the matrix is also a difficulty that needs to be overcome. Hence, the sonication process was used to ensure a good dispersion of the nanofillers where the matrix's mechanical properties may also be influenced by it (5).

Nurul Hani Noor Asmadi, et al. / Physical, Mechanical and Thermal Properties of Hybrid Epoxy/ Multi-Walled Carbon ...

2. MATERIAL AND METHOD

2.1. Material

Epoxy resin referred to as (Part A: EpoxAmite 100) and hardener (Part B: EpoxAmite 103) were used in this study. Both chemical products were manufactured by by Smooth On Corporation (Pennsylvania, United State of America) and purchased from Castmech Technologies Sdn. Bhd. (Perak, Malaysia). The Multi-walled Carbon Nanotubes (MWCNT) UCNT-COOH was provided by United Nanotech Innovations Pvt. (Bangalore, India). The second filler that was used in this study is Silicon Carbide nanoparticles (SiCs) that was provided by SkySpring Nanomaterials Inc. (Houston, TX, USA). The properties of fillers are shown in Table 1 below.

2.2. Sample Preparation

Firstly, the nanofillers (MWCNT/SiC) were added to epoxy resin and stirred using a stick until the nanofillers dispersed into the resin. Then, the mixture was mixed using a planetary centrifugal mixer at a rotation speed of 2000 rpm for 5 minutes and a revolution speed of 1000 rpm for 1 minute. Next, the solution of nanofillers and epoxy resin underwent the process of ultrasonication at 100% amplitude for 30 minutes to ensure good dispersion of the nanofillers in the epoxy resin. After that, curing agents were added to the obtained dispersion mixture and remixed using a planetary centrifugal mixture at the rotation of 2000 rpm for 1 minute and a revolution at 1000 rpm for 30 seconds. Subsequently, the uncured dispersion mixture was cast into a mould and cured at room temperature for 24 hours. Then, the epoxy nanocomposites sample were post-cured by heating to 93^o C in an oven for 1 hour. Lastly, the sample was allowed to cool at room temperature before being removed from the mould. The samples then underwent physical, mechanical and thermal properties testing. All the sample formulations, pure and with fillers, were fabricated with the same steps and techniques.

2.3. Evaluation Method

2.3.1. Density

Archimedes' principle and an electronic scale were used to determine the density of the sample. The sample was first weighed to get the dry weigh (*Wdry*) and then weighed again after submerging in water to get the wet weigh (*Wwet*). From the data, Equation (1) was used to calculate the density (ρ) of the sample. The theoretical density (ρ Theo) for each composite formulation is calculate by using Equation (2). This test is following the ASTM B962.

$$\rho = \frac{Wdry}{Wdry - Wwet} \times \rho water(T) \tag{1}$$

$$\rho Theo = \frac{1}{\sum_{i} \frac{\phi i}{\rho i}}$$
(2)

2.3.2. Flexural Properties

Flexural properties of the sample were determined using the Instron 5569 Universal Testing Machine (UTM) following the ASTM D790. Three-point loading system test method was applied with the sample rests on two supports and with loading nose midway between the supports. The samples dimension was measured at 60 mm (Length) x 12.7 mm (Width) x 3 mm (Thickness). The three-point bending was carried out with loading speed of 1.3 mm. min⁻¹, at room temperature. The flexural strength and flexural modulus were calculated using the equation (3) and (4) respectively.

$$Flexural stress, \sigma_f = \frac{3PL}{2bd^2}$$
(3)

Flexural modulus,
$$\epsilon_f = \frac{L^3 m}{4bd^3}$$
 (4)

2.3.3. Thermal Properties

For each formulation, three cylindrical samples with diameters of 20 mm and thicknesses of 5 mm were prepared for thermal conductivity and diffusivity measurements. To provide the appropriate thermal contact with the sensor, these samples were hand polished using emery paper. The thermal conductivity and diffusivity of pure epoxy and epoxy nanocomposites samples were measured using the transient plane source method Hot Disk thermal analyser (Hot Disk TPS 2500s). This testing is following the ASTM D5470.

3. RESULTS AND DISCUSSION

3.1. Density

Figure 1 shows the density results as the effects of synergistic effects of hybrid fillers. The density testing is a significant physical property that offers valuable insights into the composition, structure, and overall quality of nanocomposite samples. The results depicted that the sample of 4vol.% SiC has the highest density, which is 1203.78 kg/m³. The significant high density compared to the pure epoxy shows that the SiC nanoparticles were difficult to be process, tends to form sedimentation and have high tendency to agglomerates and form voids (6). The density of composites plays a crucial role in determining their dimensional stability. This is because composites with lower density tend to have a higher number of voids and vacant spaces within their components (7). The voids content in SiC single filler epoxy nanocomposites is low due to the low SiC filler loadings in the epoxy matrix which shows by the high-density results. However, these results are not necessarily indicated that

International Journal of Nanoelectronics and Materials (IJNeaM) Volume 17, (Special Issue) December 2024 [307-313]

the dispersion of the SiC nanofillers is good as the mechanical and thermal properties of the SiC single filler epoxy nanocomposites is poor. Thus, this indicate that there are agglomerations and sendimentation of SiC nanofillers in the epoxy matrix. Agglomeration takes place when discrete nanoparticles come into close proximity and adhere to one another, resulting in the formation of larger aggregates. SiC nanoparticles tend to form agglomerations due to its high surface energy which promotes the particleparticle interactions (8). Hence, this causes the SiC nanofillers to poorly disperse which results in poor and mechanical thermal properties. Moreover, sedimentation is also likely to occur due to the significant difference of density between epoxy and SiC which contribute to the poor mechanical properties although it shows highest density. The results are best explained by the Scanning Electron Microscopy (SEM) characterization and can be seen in Figure 2. The agglomeration of SiC in the epoxy composite can be clearly seen in Figure 2. The

finding was similar to what Samsudin et al. (5) has reported in her previous study. Before this, she has found out that the fracture surface morphology of epoxy/SiC nanocomposites at 1vol.% and 5vol.% filler loadings, which demonstrates that agglomerates form in epoxy nanocomposites samples, specifically SiC, and especially at high filler loadings. The existence of nanofiller agglomeration and sedimentation could have a significant impact on the thermal and mechanical properties of the composites. (9). For the samples with hybrid fillers, the results show in corresponding that the higher the SiC filler loadings, the higher the density of the samples. Moreover, MWCNT fillers show lower density which are not far different from the pure epoxy. This is because the MWCNT fillers have low density which facilitate the fillers dispersion in the epoxy matrix, and thus provide better mechanical and thermal properties.



Figure 1. Density as the effects of synergistic effects of hybrid fillers.



Figure 2. The agglomeration of SiC in the epoxy composite.

3.2. Flexural

3.2.1. Flexural Strength

The flexural strength of the epoxy nanocomposites was examined to study the mechanical properties as the effect of synergistic effects of hybrid fillers. Flexural strength indicates the maximum stress or load that a material can withstand before failing under bending. Figure 3 shows the results of the flexural strength of the epoxy nanocomposites, which reveals that 4vol.% MWCNT has the highest flexural strength (80.41 MPa). This is due to the high aspect ratio of MWCNT given by its elongated structure, enabling it to distribute and transfer stress effectively (10). Instead, 4vol.% SiC has the lowest flexural strength (32.95 MPa) but remain higher than pure epoxy (28.37 MPa). The low flexural strength of the 4vol.% SiC is due to the low aspect ratio as it is in particle structure

compared to MWCNT with long nanotube structure. The low aspect ratio of SiC particles can result in poor stress transmission and weak interfacial interaction between the particles and the matrix, resulting in poor mechanical properties of composites (11). The aspect ratio of the reinforcement material can have a significant influence on composite mechanical properties such as stiffness, strength, and toughness (12). Moreover, SiC is ceramicbased fillers that exhibit brittle nature and inherent flaws such as pores that become the point of stress concentration prone to failure (4). Thus, the higher SiC filler loading will result in lower flexural strength. However, at the equal ratio of hybrid fillers, which is 2vol.% MWCNT+2vol.% SiC (33.08 MPa) show significantly low flexural strength. This is due to the ineffective composition of hybrid fillers at the equal ratio that causes poor dispersion and weak interfacial bonding between the hybrid fillers and matrix at this composition (13).



Figure 3. Flexural strength as the effects of synergistic effects of hybrid fillers.

3.2.2 Flexural Modulus

The epoxy nanocomposites' flexural modulus was examined which indicates the ratio of stress to strain in the elastic part of the material's stress-strain curve. It measures a material's rigidity, representing how much it will bend under a load (14). Figure 4 shows the results of the epoxy nanocomposites' flexural modulus, which reveals that 3vol.% MWCNT+1vol.% SiC (2949.01 MPa) have a higher flexural modulus which is slightly higher than the 4vol.% MWCNT (2706.55 MPa). This shows that the 3vol.% MWCNT+1vol.% SiC has a higher stiffness. This could beis due to the best composition of this hybrid fillers

ratio exhibiting synergistic effects. SiC is a ceramic-based filler with a higher stiffness than MWCNT, meaning it has higher bending resistance (15). Thus, SiC has a higher modulus of elasticity than MWCNT. Thus, incorporating the SiC with MWCNT will give a synergistic effect in flexural modulus. However, the synergistic effects only happen for MWCNT/SiC at the ratio of 3:1. Increasing the SiC fillers will lead to lower flexural modulus, which is caused by poor dispersion and agglomeration, leading to the formation of voids. This also occurs in the SiC single filler epoxy nanocomposites, which have the lowest flexural modulus and are lower than pure epoxy.



Figure 4. Flexural modulus as the effects of synergistic effects of hybrid fillers.

3.3. Thermal Properties

3.3.1. Thermal Conductivity

The results of thermal conductivity as the effects of synergistic effects are depicted in Figure 5. Thermal conductivity measures the material's capacity to transfer heat via conduction (16). Pure epoxy has relatively low thermal conductivity (~ 0.2 W/mK) due to its amorphous structures that leads to phonons scattering (17). The

3vol.% MWCNT+1vol.% SiC (0.48 W/mK) shows the highest thermal conductivity follow by the 4vol.% MWCNT (0.46 W/mK).

International Journal of Nanoelectronics and Materials (IJNeaM) Volume 17, (Special Issue) December 2024 [307-313]



Figure 5. Thermal conductivity as the effects of synergistic effects of hybrid fillers.

The higher thermal conductivity results of 3vol.% MWCNT+1vol.% SiC compared to the 4vol.% MWCNT indicate the synergistic effects of hybrid fillers at this ratio. Although the 4vol.% MWCNT contain more MWCNT fillers compared to 3vol.% MWCNT+1vol.% SiC, the thermal conductivity of the 4vol.% MWCNT are slightly lower than the 3vol.% MWCNT+1vol.% SiC. The main reason is that the mix of different types and shapes of fillers can make the hybrid thermally conductive fillers disperse better in the polymer matrix, making it easier to connect thermally conductive fillers next to each other to form thermal conduction paths (1). However, in this case, it also depends on the amount of the MWCNT filler loadings as it provides much higher thermal conductivity than the SiC nanoparticles due to its long structure. Thus, the results of 2vol.% MWCNT+2vol.% SiC (0.42 W/mK) and 1vol.% MWCNT+3vol.% SiC (0.34 W/mK) show significantly lower thermal conductivity than those with higher MWCNT fillers. MWCNT has a high aspect ratio, meaning its length is much higher than its diameter. The long structure enables it to form good alignment, which is one of the effective approaches to improve the thermal properties of the composites. The good alignment of MWCNT in the composites will enable the thermal to travel through the pathways effectively (18). Moreover, MWCNT also consists

of a carbon-based structure which also consists in the epoxy, and this similarity cause low interfacial thermal resistance between them (19). On the other hand, SiC has a low aspect ratio as it is in particles structure and has less carbon-based structure than MWCNT.

3.3.2. Thermal Diffusivity

The thermal diffusivity of the epoxy nanocomposites as the effects of synergistic effects of hybrid fillers has been examined, and the results are depicted in Figure 6. Thermal diffusivity is a measurement of heat rate through a material which relates to the time taken for heat to spread through the material (12). The thermal diffusivity is directly proportional to the thermal conductivity, as it shows that the thermal diffusivity is increased when MWCNTs' filler loadings increase. The thermal diffusivity of 3vol.% MWCNT+1vol.% SiC (0.44 mm²/s) is lower than the 4vol.% MWCNT (0.54 mm²/s), which is opposite to the thermal conductivity results that shows that 3vol.% MWCNT+1vol.% SiC higher than 4vol.% MWCNT. This is because 4vol.% MWCNT has a lower density than the hybrid fillers.



Figure 6. Thermal diffusivity as the effects of synergistic effects of hybrid fillers.

This result indicates that the 4vol.% MWCNT can distribute heat throughout its volume faster than the 3vol.% MWCNT+1vol.% SiC. The thermal diffusivity of 4vol.% MWCNT ($0.54 \text{ mm}^2/\text{s}$) is the highest compared to the 4vol.% SiC ($0.25 \text{ mm}^2/\text{s}$) and 2vol.% MWCNT+2vol.% SiC ($0.32 \text{ mm}^2/\text{s}$). The results are similar to thermal

conductivity as thermal conductivity is one of the factors affecting a material's thermal diffusivity according to the formula (α =k/ ρ c), which thermal diffusivity is the thermal conductivity divided by the density and specific heat of material (20). Material with high thermal conductivity tends to have high thermal diffusivity too. Thermal

diffusivity is also influenced by the density of the material which can be related to the density results in Figure 1. 4vol.% MWCNT has the highest thermal diffusivity because it has the lowest density. The material's volumetric heat capacity is reduced due to its lower density, resulting in less heat energy required to increase its temperature (21). As a result of possessing a lower heat capacity, the material can conduct heat at a faster rate, leading to an increase in its thermal diffusivity. Instead, the 4vol.% SiC shows a slow and small increase in thermal diffusivity due to the low thermal conductivity and high density.

4. CONCLUSION

The study successfully prepared epoxy nanocomposites at 4 vol.% of nanofillers, incorporated with single MWCNTs and SiCs as well as the hybridization of MWCNTs/SiCs fillers with three different ratios of both fillers. The testing and analysis of the epoxy nanocomposites yielded positive results. The incorporation of multi-walled carbon nanotubes (MWCNTs) and silicon carbide (SiCs) resulted in increased thermal properties as the fillers has form conductive pathways for the heat to travel more effectively in the nanocomposites. Hence, the research revealed that the thermal properties and flexural modulus of the epoxy nanocomposites were highest with hybrid fillers of 3vol.% MWCNT+ 1vol.% SiC due to the synergistic effects.

Properties	MWCNT	SiC	
Appearance	Black powder	Greyish powder	
Morphology	Nanotubes	Cubic	
Content of Carbon (%)	>97	-	
Average diameter/particle size (nm)	20.0-40.0 nm	40.0 nm	
Length (µm)	<10.0	2.0-60.0	
Thickness average (nm)	0.8-1.6	45.0-65.0	
Density given by supplier (g/cm3)	2.1-3.0	3.217	
Surface area given by supplier (m²/g)	210.0-300.0	30.0-60.0	
Thermal conductivity (W/mK)	3000	360	
Electrical conductivity (S/m)	107	-	

Table 1 Properties of nanofillers

Table 2 Formulation for single and hybrid fillers of epoxy nanocomposites

No.	Type of sample	Sample Abbreviation	Epoxy (Vol.%)	MWCNT (Vol.%)	SiC (Vol.%)
1.	Pure Epoxy	EP-MWCNT (0) SiC (0)	100	0	0
2.	Epoxy nanocomposite with fillers	EP-MWCNT (4)	96	4	0
3.		EP-SiC (4)	96	0	4
4.		EP-MWCNT/SiC (2:2)	96	2	2
5.		EP-MWCNT/SiC (1:3)	96	1	3
6.		EP-MWCNT/SiC (3:1)	96	3	1

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