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Flexural Properties of Halloysite Nanotubes (HNTS) and Carbon Nanotubes (CNTS) Toughened Epoxy Composites

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Abstract*: In this research, four different concentrations of halloysite nanotubes and carbon nanotubes (0wt%, 0.2 wt%, 0.5 wt% and 1 wt%) were produced using solution casting method. Both fillers were dispersed using bath sonicator for 10 minutes. Flexural properties, surface roughness and microhardness were studied. The highest flexural modulus and flexural strength were observed in the 0.2 wt% HNTs-epoxy composites, where the maximum values were 36.6% and 82% respectively. The maximum surface roughness was recorded in the case of 0.2 wt% HNTs-epoxy composites. The highest microhardness value was found in the 0.5 wt% HNTs-epoxy composites where the microhardness improved by 80%. The results suggest, HNTs were easily dispersed in epoxy matrix than CNTs, at shorter processing time. From the SEM images, it can be observed that, HNTs significantly changed the microstructure of the nanocomposites, as there were many straight and elevated crack lines, this can be associated with the toughening mechanism offered by the filler. CNTs on the other hand, had influenced on the micro cracks and showed semi-parabolic pattern. However, the flexural properties of CNTs are slightly lower than HNTs because CNTs have strong van der Waals force and as a result very difficult to disperse by simple sonication. HNTs can be utilised as an alternative to CNTs, since the dispersion state is better even though at minimum sonication time.

Keywords*: Halloysite nanotubes (HNTs), Carbon nanotubes (CNTs), epoxy, flexural properties.

I. INTRODUCTION

Epoxy has some unique properties which include high modulus and strength, low shrinkage, as well as excellent chemical and heat resistance [1]. Epoxy has been widely used in manufacturing applications, for instance, it has been widely used as adhesives, coatings, electronic and aerospace structures [2]. However, there are some drawbacks that have been highlighted by some researchers.

Cured epoxy systems have poor impact strength, low resistance to crack initiation and propagation. Besides that, unreinforced epoxy systems also have low fracture toughness [3]. To overcome this, a new method is introduced to improve polymer properties via reinforcement of fillers in the nanometre scale into the epoxy systems [4]–[6].

Because of their outstanding mechanical, optical, electrical and magnetic properties nanoparticles reinforced in polymers have attracted great attention among the researchers [7], [8]. Among these, inorganic particles of nanometre scale in the range between 1 and 100 nm are dispersed in a matrix of polymeric material, to create new polymer nanocomposites materials [1], [9]. Nanoparticles show significant properties owing to their large surface area per unit volume, as a results of good phase interactions between the polymer matrix and the nanoparticles. Many essential chemical and physical interactions are influenced by both surfaces.

Halloysite is a clay mineral with the empirical formula $Al_2Si_2O_5(OH)_4$. This material is an unusual and rare natural nanotubular material [10]. Since the discovery, interest in the toughening mechanisms of HNTs in polymer materials has significantly increase [11]. Our previous work has suggested that this low-cost nanotubes, can significantly improve the mechanical performances of epoxy nanocomposites [12], [13]. According to Ye et al. an increase of 400% in impact strength was observed due to the large amount of micro cracking that was responsible as the toughening mechanism [14]. Deng et al. reported an improvement of fracture toughness (K_{1C}) and G_{1C} up to 50% and 127%. The improvements were due to crack bridging effect and plastic deformation of the epoxy around the HNTs clusters [15]. According to Liu et al. in future, HNTs may replace the more expensive CNTs in high performance nanocomposites [16].

CNTs were first discovered by Ijima in 1991 and were formed at the cathode during sputtering of graphite by an electronic arc [17]. Known as one of the stiffest engineering fibres, these materials have great potential as nano-fillers for aerospace application, owing to their good combination of physical and chemical properties, particularly at very small volume or percentage [18], [19].

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Many publications suggested that CNTs have tensile strengths considerably higher than steel and carbon fibre, electrical conductivity similar to silver and platinum and capable of carrying higher current densities than copper [20].

CNTs also have better thermal conductivity than diamond as well as lower density than aluminium [21]. Montazeri in his publication reported, elastic modulus and tensile strength of 1wt% CNTs-epoxy were improved up to 15% and 11% respectively [22]. Saharudin et al. reported an increase of 4% for 1wt% CNTs-epoxy composite [23]. Inam et al. reported an increase of 35% in flexural modulus in the case of 0.025 wt% CNTs-epoxy nanocomposites [20].

Mirjalili et al. in their publication reported, dispersion quality of CNTs in epoxy resin was significantly associated to fracture toughness of the modified resin and a maximum of 20% improvement was recorded [24]. An improvement of 25% in micro hardness was recorded for monolithic alumina produced after applying heat treatment process to alumina+1vol.% CNTs composite, as compared to monolithic alumina sintered without CNTs [25]. CNTs pull out and peel-off, as well as good adhesion of CNTs and polymer matrix were identified by prominent researchers from their SEM analysis as the main toughening mechanisms in their nanocomposite systems [26], [27].

The improvement of polymeric systems by nano fillers like HNTs and CNTs has created the opportunity of increasing modulus and strength of composites using a considerably lower filler laoding. Good dispersion of the nanofiller in the epoxy matrix and strong interaction between the filler and the epoxy is undeniably essential for good toughening effect. However, at higher nanofiller content, composite properties tend to decrease due to difficulties in dispersing the fillers homogenously.

To date, very limited literature currently available on a comparative study between HNTs and CNTs. Therefore, this research provides new insights on the flexural properties, surface roughness and microhardness of HNTs and CNTs reinforced epoxy. The main aim of this research is to compare the properties of epoxy composites reinforced with both fillers at low loading content (below 1 wt%). We purposely used low loading content because dispersion of nano filler at this percentage is slightly easy and also recommended by several researchers [28]–[30].

II. MATERIALS AND TESTINGS

Fig. 1. shows an image of monolithic epoxy taken by FESEM (Zeiss, Germany). It can be observed that the surface is smooth surface thus exhibits brittle failure. Miracast 1517A and Miracast 1517B are the epoxy and hardener used in this research, both materials were acquired from Miracon Sdn. Bhd., Seri Kembangan, Malaysia. The densities of the epoxy and hardener were 1.13 and 1.1 g/cm^3, respectively. Due to its low viscosity, this type of epoxy system is commonly used in the composites industry. The low viscosity of the hardener helps to improve the dispersion and the fast curing of the nanocomposites to prevent agglomeration. The gelation time of the resin was 40 min at room temperature (RT). Fig. 2 shows HNTs and CNTs images acquired using FESEM.

The HNTs used in this research were supplied by Sigma Aldrich, UK. The diameter and length of HNTs were 30–70 nm and 1–4 µm, respectively. The HNTs consist of two layers of aluminosilicate at 1:1 Al to Si ratio. The CNTs that comprised of approximately 5–20 graphitic layers used in this research were purchased from Sigma Aldrich, UK. The CNTs were 7–15 nm in diameter and 0.5–10 µm in length as declared by the supplier. The flexural tests were conducted using Universal Testing Machine, Victor Equipment Resources (Selangor Darul Ehsan, Malaysia). The crosshead speed used was 1mm/min. The flexural samples were produced according to D790 standard.

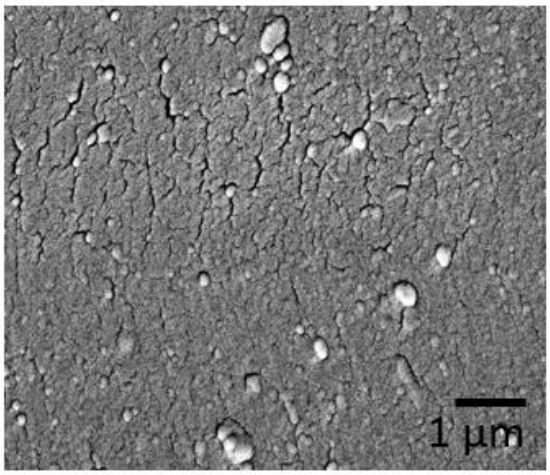

Fig. 1. SEM image of monolithic epoxy (ME).

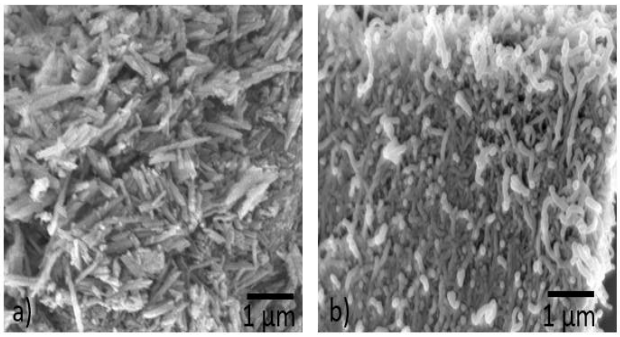

Fig. 2. Halloysite nanotubes (HNTs) (a) and Carbon nanotubes (CNTs)

III. RESULTS

Fig. 3. shows flexural modulus of monolithic epoxy, epoxy reinforced HNTs and epoxy reinforced CNTs. Monolithic epoxy recorded the lowest flexural modulus of 0.9 GPa. In the case of 0.2 wt% HNTs-epoxy nanocomposites, the flexural modulus increased 36.6% compared with monolithic epoxy. However, in the case of 0.2 wt% CNTs-epoxy nanocomposites, the flexural modulus improved 17%. At 0.5 wt% HNTs concentration, the flexural modulus increased 23% whilst at 0.5 wt% CNTs, the flexural modulus increased 7%. In the case of 1wt% HNTs and CNTs reinforcement, the flexural modulus increased 26.4% and 4% respectively.

The dispersion quality of CNT improves as the filler concentration decreases [31]. In this research, composite properties decrease as filler concentration increase showing difficulties in dispersing nano fillers through simple sonication method at shorter time.

The variation of flexural strength is presented in Fig. 4. Monolithic epoxy recorded the lowest value with 38 MPa. An improvement of 82% in flexural strength was observed in the case of 0.2 wt% HNTs-epoxy composite. In the case of 0.2 wt% CNTs-epoxy composite, the flexural strength increased 21%. At higher filler content, the flexural strength for 0.5 wt% HNTs-epoxy and 0.5 wt% CNTs-epoxy composites increased 76% and 8% respectively. In the case of 1 wt% HNTs-epoxy the flexural strength increased 60%. The lowest improvement in flexural strength was recorded in the case of 1 wt% CNTs-epoxy composite with only 3% increase compared with monolithic epoxy. The variation of flexural strain is presented in Fig. 5. Samples reinforced with CNTs tend to have low flexural strain compared with monolithic epoxy. As for the samples reinforced with HNTs, the flexural strain slightly higher than monolithic epoxy.

Fig. 6. shows the average surface roughness, R_a of the fractured samples. As expected, monolithic epoxy recorded the lowest surface roughness of 0.6 µm. In the case of 0.2 wt% HNTs-epoxy composite sample, the surface roughness increased 100% compared to monolithic epoxy. In the case of 0.2 wt% CNTs-epoxy, the surface roughness increased 85%. In the case of 0.5 wt% HNTs-epoxy, 125% increase in surface roughness was observed. In the case of 0.5 wt% CNTs-epoxy, the surface roughness increased 108%. Even though this value is slightly lower than HNTs reinforced epoxy, the difference is rather small. Similar trend was observed with epoxy reinforced 1wt% HNTs and CNTs. Samples reinforced with 1 wt% HNTs recorded 1.6 µm of surface roughness. In the case of 1 wt% CNTs, the surface roughness observed was 1.36 µm.

The variation of microhardness of epoxy and its composites is shown in Fig. 7, and Fig. 8 shows the images of monolithic epoxy and composites samples subjected to indentation (200g). It can be observed that monolithic epoxy recorded the lowest value of microhardness with 20.5 HV. The highest microhardness value was obtained in the case of 0.5 wt% HNTs-epoxy composites where the microhardness improved by 80%. For CNTs reinforced epoxy, the values were between 34 HV to 37 HV. The reinforcing effect of HNTs and CNTs was responsible for the improvement of microhardness values. Strong resistance was observed because of reinforcement effect by the HNTs and CNTs.

For a coarse surface, the indentation may possibly occur at the edge. If that is the case, less resistance shall be perceived by the material and thus hardness recorded would be low [32]. Variously, if the surface is glossy or smooth, strong resistance will be offered by the material against the penetration of indenter therefore high hardness will be recorded. These are some of the possible observations for the variation of microhardness. However, in conclusion, the microhardness was significantly increased with the incorporation of HNTs and CNTs.

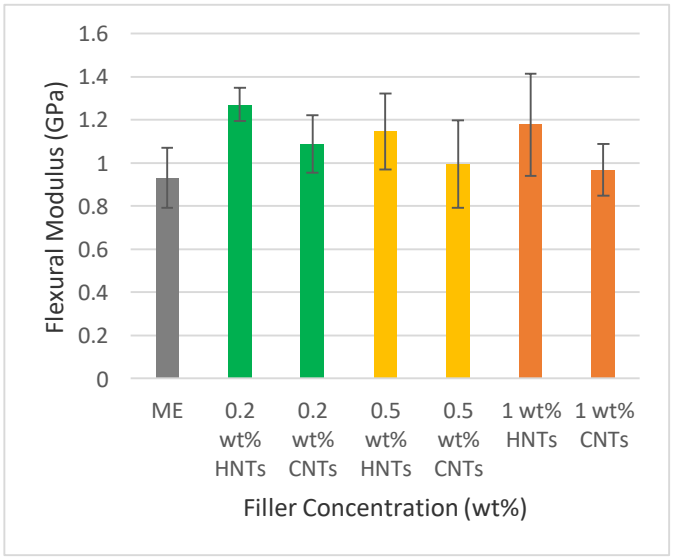

Fig.3. Flexural modulus of nanocomposites

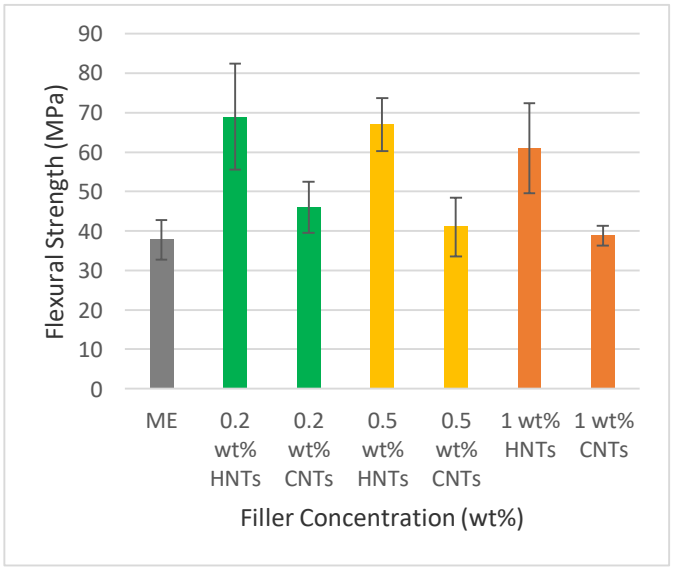

Fig.4. Flexural strength of nanocomposites

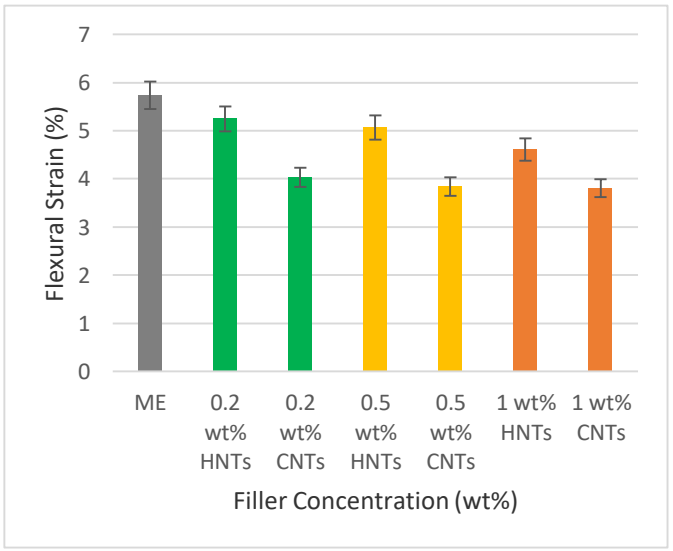

Fig.5. Flexural strain of nanocomposites

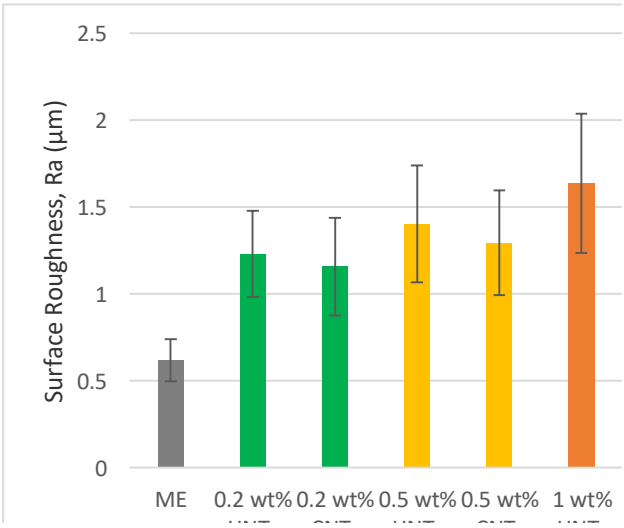

Fig. 6. Surface roughness of nanocomposites

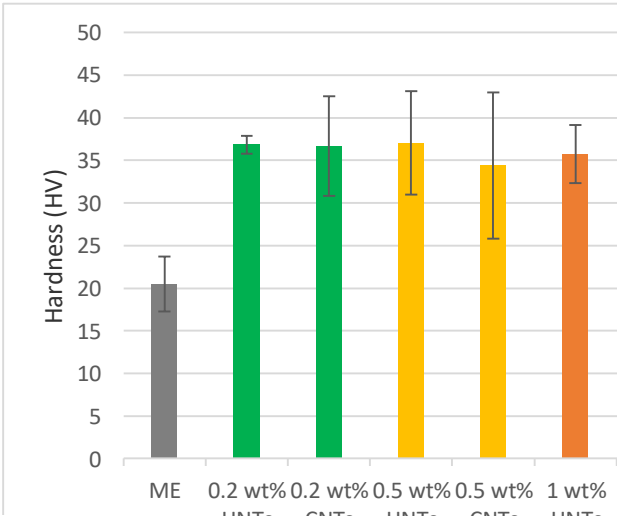

Fig. 7. Microhardness of nanocomposites

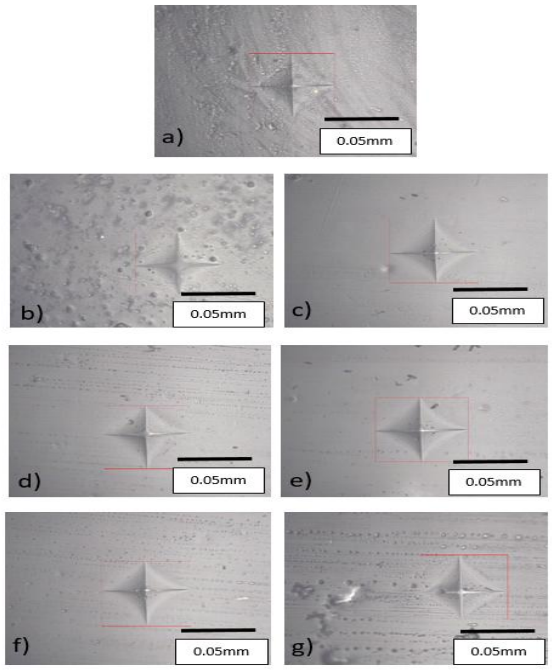

Fig. 8. Images of indentation on samples; (a) monolithic epoxy, (b) 0.2 wt% HNTs-epoxy, (c) 0.2 wt% CNTs-epoxy, (d) 0.5 wt% HNTs, (e) 0.5 wt% CNTs, (f) 1 wt% HNTs and (d) 1 wt% CNTs.

SEM Images

Images of broken samples from tensile testing specimens were analysed using Desktop Scanning Electron Microscope. Fig. 9 shows SEM images of 0.2 wt% HNT, 0.2 wt% CNTs, 1 wt% HNT and 1wt% CNTs reinforced composites. Fig. 9 (a) shows 0.2 wt% HNT-epoxy composite. It can be observed that HNT significantly changed the microstructure, as there were many straight and elevated crack lines, this can be associated with the toughening mechanism.

The formation of micro lines absorbs a large amount of energy [23] as also shown in Fig. 9 (c). In addition, the micro cracks also were bridged by rigid, strong halloysite nanotubes consequently slowing down and stopping the crack propagation [14]. As for the CNTs, the micro cracks were semi-parabolic pattern.

In the case of 1 wt% CNTs, the fracture surface slightly coarser than the sample reinforced with 0.2 wt% CNTs. Fig. 9 (b) and (c) show the fracture surface of 0.2 wt% CNTs and 1 wt% CNTs. From these two figures, it can be seen that the patterns were quite similar. The surface roughness increased in for both cases, however the number of layers for 1 wt% CNTs were coarser than the sample of 0.2 wt% CNTs. This phenomenon can be associated with crack deflection mechanism form from CNTs reinforcement. The reason why sample 1 wt% CNTs slightly lower in flexural modulus and strength, simply because there were agglomerates due to shorter sonication time.

Agglomerates create stress concentration and reduced flexural properties as also mentioned by few researchers [33], [34]. The concentration of nano filler above 0.5 wt% would also increase agglomeration [2], [3], [35]. Besides agglomerates, re-aggregation of CNTs in epoxy matrix due to insufficient time of curing could be one of possible reasons for lower flexural properties, as stated by Inam et al. [36].

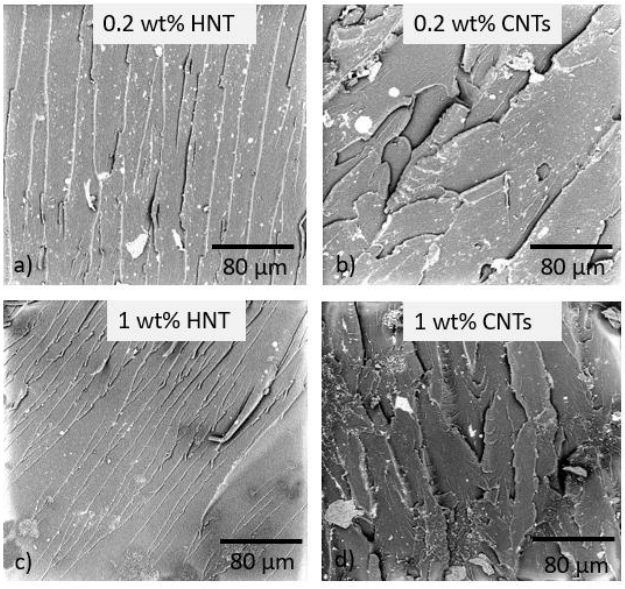

Fig. 9. SEM images of composites

IV. CONCLUSION

In this research, four different concentrations of HNTs and CNTs (0wt%, 0.2 wt%, 0.5 wt% and 1 wt%) were successfully produced using solution casting method. Both nano fillers were dispersed using bath sonicator for 10 minutes. Flexural properties, surface roughness and microhardness were compared and studied. The highest flexural modulus and flexural strength were observed in the 0.2 wt% HNTs-epoxy composites, where the maximum values were 36.6% and 82% respectively. The maximum surface roughness was recorded in the case of 0.2 wt% HNTs-epoxy composites. The highest microhardness value was found in the 0.5 wt% HNTs-epoxy composite where the microhardness improved by 80%. At higher nanofiller content, composites properties decreased, indicating difficulties in dispersing the fillers. HNTs are relatively cheaper material which can be used in automotive industry. Besides that, HNTs also can be utilised as an alternative to CNTs knowing that their flexural properties, hardness and surface roughness are comparable. CNTs might be useful in advanced composites, replacing carbon fibre when the cost is not a big concern. It can be concluded that HNTs-epoxy composites recorded the highest flexural modulus compared to CNTs-epoxy composites at low concentration, however more studies need to be carried out in future, to study the tensile properties, impact strength, and dynamic mechanical properties of both fillers in epoxy matrix system.

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