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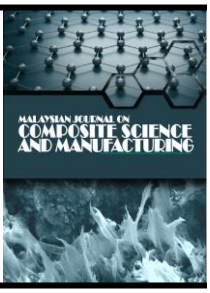

Enhancement of Mechanical and Electrical Properties in Graphene Nanoplatelet Modified Nylon 66

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ABSTRACT

Conventional thermoplastics used in electronic housings and structural members are vulnerable to electromagnetic interference (EMI). To improve the EMI shielding property in thermoplastics, conductive fillers must be incorporated. By using nano-sized conductive fillers such as graphene nanoplatelets (GNP), conductivity can be achieved without the setbacks in terms of cost, processability and mechanical properties. In this study, mechanical and electrical properties of nylon 66 added with a minimal amount of GNP are investigated. Nylon 66/GNP nanocomposites were prepared by dry mixing followed with melt compounding. Sonication was performed prior to the mixing process to reduce agglomeration of GNP. Addition of 0.3 wt% GNP significantly improved (+15.2%) the tensile strength of nylon 66. The increase at 0.3 wt% GNP is attributed to high aspect ratio and good interfacial stress transfer associated with well dispersed GNP. However, tensile strength showed a decrease when GNP amount was further increased to 0.5 and 1.0 wt%. The result is in agreement to the XRD result, in which well dispersion of GNP with no reassembly or re-aggregation of graphene layers in nylon 66 matrix is indicated at 0.3 wt%. The good dispersion level of GNP at 0.3 wt% enables the formation of an effective network for electron path transmittance, as demonstrated by the increase in electrical conductivity.

Keywords:
Nylon 66; Graphene Nanoplatelets; EMI Shielding; Polymer Composite

1. Introduction

Electromagnetic interference (EMI) is defined as a phenomenon by which disruptive electromagnetic energy is transmitted from one electronic device to another via radiated or conducted paths or both, leading to operation failures [1]. This undesired EMI effects take place when susceptible electronic devices receive electromagnetic waves discharged by other electric or electronic devices and may result in malfunction in electric or electronic system, as well as causing

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harm on human body [2]. Therefore, to prevent EMI induced functional disruption in electric and electronic devices, EMI shielding materials are required.

Previously, metal and alloy shrouds were conveniently used for this purpose. However, as the demand for low-cost and lighter electronic devices increases, attention on plastics as EMI shielding materials has been increasing. Electrical conductivity is very essential for an EMI shielding material due to physical phenomenon that electric and varying magnetic fields generate currents in the electrically conducted shielding material. However, as conventional polymeric materials are non-conductive, some modifications are necessary to enable polymers to be utilized as EMI shielding material. There are two general approaches to achieve this, i.e., one is via coating with conductive metal, and the other is blending with conductive fibers or particles [3,4]. The former has disadvantages such as delamination as well as requires additional surface preparation and special equipment. It also increases the final product cost due to the secondary expense of the coating process [5].

Generally, conducting polymer composites can be realized by incorporating high aspect ratio conductive nanofillers. Various types of conductive polymer composites (CPC) utilizing conductive fillers such carbon black, stainless steel fiber, aluminum fiber, brass fiber, nano-copper etc. have been investigated [6-9]. The electrical conductivity of a CPC determines the applications it can be used for. Highly conductive metal particles such as gold, silver and copper have been incorporated into an insulating polymer to increase the electrical conductivity. Some conductive filler such as metal powders and carbon black are not competitive as EMI shielding material because of the high filler loading required for such application, about 40 to 60 wt% [5]. In contrast, the low price and availability of pristine graphite in large quantities, coupled with relatively simple solution process makes graphene a potential choice as conductive filler in the preparation of conductive polymer composites.

Graphene nanoplatelet (GNP), as a result of its high electrical conductivity and aspect ratio, has been found to be effective for the enhancement of electrical conductivity of polymers at low filler content [10]. It also shows a much better dispersability and lower cost compared to carbon nanotube (CNT). Moreover, its two-dimensional (2-D) characteristic makes GNP to be a more attractive candidate for EMI shielding filler compared to one-dimensional CNT [11,12].

Nylon 66 is a type of engineering polymers from polyamide family and has been commonly used as housing materials for portable electric and electronic devices. It has excellent thermo-mechanical properties and has great resistance from severe atmospheric instability [13]. However, being polymer, it is an intrinsically insulating material with poor electrical conductivity. Nylon 66 with enhanced electrical conductivity is a perfect material to be used as housing material with EMI shielding capability.

Various researches have been carried out on enhancing the electrical conductivity in engineering polymers. Incorporation of 0.75 wt% of CNT into acrylonitrile butadiene styrene (ABS) polymer was reported to increase its conductivity up to 10^{-5} (S/m) [5]. Meanwhile, addition of 5 wt% nickel powder into carbon reinforced polyamide-6 managed to increase the conductivity to 10^{-9} (S/m) [14]. A larger enhancement was reported by Vankayala et al. [15], in which nylon-6 added with 1 wt% multi-walled carbon nanotubes (MWCNT) coated with polyaniline (PANI), showed an increase in conductivity up to 7.3×10^{-5} (S/m).

In this study, GNP is applied as conductive filler in nylon 66 matrix in order to enhance its electrical conductivity. The exfoliation of GNP is carried out using sonication technique. The effects of GNP loading on the conductivity as well as on the physical-mechanical properties are studied

2. Methodology

2.1 Materials

Nylon 66 (Dupont, Zytel® 101F NC010) was used as the polymer matrix, without any purification. Graphene nanoplatelets (XG Sciences US) were purchased from Terra Techno Engineering. The graphene nanoplatelets (GNP) loading was varied at 0, 0.3, 0.5 and 1.0 wt%.

Sonication was performed to exfoliate the GNP prior to the mixing with nylon 66. The GNP was dispersed in ethanol at frequency of 40 Hz for 60 minutes using an ultrasonic instrument (Fisher Scientific Sonic Dismembrator). Nylon 66/GNP nanocomposites were prepared by mixing the GNP with nylon 66 by means of dry mixing method using a table top high speed mixture at room temperature for 5 minutes. All mixtures of nylon 66/GNP were extruded by co-rotating twin screw extruder (Sino PSM 30). The materials passed through heated barrels zones and exited at the nozzle holes. The barrel temperature setting was kept constant (255-280 °C) for all sample formulations. Extruded samples were then cooled using water. Subsequently, the samples were cut into pelletized form by pelletizer machine.

Finally, the pellets were injection molded into a standard mold cavity (ASTMD 638 type 4) using Ray-Ran sample injection molding press. The samples were preheated in the barrel for 10-15 minutes prior to injection molding process. The temperatures of barrel and mold were set up to 295 and 95 °C, respectively.

2.2 Measurements

Tests on physical-mechanical and electrical properties as well as morphological analysis were performed to investigate the properties of the nylon 66 filled with various amounts of GNP.

Melt Flow Index was performed with the purpose of measuring the ease of flow or melt flow rate (MFR) of nylon 66 and nylon 66/GNP nanocomposites in molten state using melt flow apparatus (Thermo Haake, Melt Flixer). For the testing, Procedure A of ASTM D1238 was applied.

Tensile testing was carried out according to ASTM D638-Type 4 at room temperature using universal testing machine (Instron). The specimen was pulled at crosshead speed of 50 mm/min. A total of 5 tensile tests were performed for each composition and the average values were recorded.

Fourier-transform infrared (FTIR) spectra were obtained at a resolution of 4.0 cm^{-1} in the wavenumber range of 4000 – 400 cm^{-1} using an FTIR spectrophotometer (Bruker) in order to investigate the variation in the surface structure of the platelets in nylon 66 polymer. FTIR analysis was performed using the conventional KBr pellet technique. XRD analysis was carried out with an X-ray diffractometer (PANalytical, X'Pert Pro MRD) to understand the interlayer spacing and crystalline structure of GNF, graphene nanoplatelets and nylon-6/GO nanocomposites using nickel filtered copper Kα radiation with $\lambda = 0.154$ nm. The samples were scanned at a rate of 1°/min between 0-80 °C.

The resistivity of GNP/nylon 66 films was determined from the resistance values obtained using a multi-meter, known as LCR meter (Agilent, E4980A). The resistivity, ρ can be determined using the equation $\rho = RA/L$ where R is the resistance, A is the cross sectional area of the sample and L its thickness [16]. Electrical conductivity, σ is then taken as the reciprocal of resistivity. The LCR meter used was operated under 1 kHz-frequency alternating current.

Scanning electron microscopy (SEM) analysis was conducted on tensile-fractured surface of samples using a SEM (Leica Cambridge S-360) up to 1000x magnification to observe the morphology of the nanocomposite.

3. Results and Discussion

3.1 Melt Flow Index

The viscosities of nylon 66 and nylon 66/NGP nanocomposites were assessed by their MFI values. From Table 1, all nylon 66 nanocomposites show lower MFI values than the neat nylon 66, indicating an increase in viscosity. The MFI value decreases significantly from 37.8 to 25.4 (g/10 min) with the addition of 0.3 wt% GNP. However, the decrease in MFI (increse in viscosity) becomes more moderate when GNP is further increased to 0.5 and 1.0 wt%. The overall increase in viscosity with the addition of GNP is probably contributed by homogenous presence of GNP particles that effectively hindrance the movement of nylon 66 polymer chains. The presence of the interaction makes the segmental motion of the polymer chains more difficult, causing an increase in flow activation energy. As the GNP amount is increased further to 0.5 and 1.0 wt%, the platetets start to consolidate to form larger GNP agglomerates, which results in reduced number of dispersed GNP. Hence, the slight decrease in viscosity value.

Table 1
Melt flow index (MFI) values for GNP filled nylon 66 nanocomposites

Nanographene Amount (wt%)	MFI (g/10 min)
0	37.8
0.3	25.4
0.5	26.8
1.0	30.6

3.2 Tensile Properties

Figure 1(a) shows the average values of ultimate tensile strength, while Figure 1(b) shows both Young's elastic modulus and elongation at break of nylon 66 as a function of GNP amount, respectively. Neat nylon 66 shows a high ultimate tensile strength of 55.17 MPa. The value increases up to 63.56 MPa (15.2% increase) when a minimal amount (0.3 wt%) of GNP is added. However, when the amount of GNP is further increased to 0.5 and 1.0 wt%, ultimate tensile strength decreases to 44.19 and 41.34 MPa, respectivley. The decrease in tensile strength for composites with loading percentage above 0.3 wt% is probably due to agglomeration of GNP. The results of tensile properties are in agreement with the results obtained for MFI. The increased in tensile strength of 0.3% GNP is attributed to higher aspect ratio, better dispersion and interfacial stress transfer as compared to 0.5 and 1.0 wt% GNP. Good dispersion and interfacial stress transferring lead to a more uniform stress distribution and minimize the presence of the stress concentration center [17].

The effects of GNP addition on the elongation at break and Young's elastic modulus of nylon 66 are shown in Figures 1(b). Neat nylon 66 shows a very high tensile modulus (0.92 GPa) and relatively low elongation at break (39.68 %) indicating its brittle nature. The results show that elongation at break increases with the increase of GNP amount. Expectedly, Young's modulus decreases with the increase of GNP since it is always inversely proportional to the elongation at break. However, the decrease in the modulus value is relatively small with 9% of decrease at 1.0 wt% GNP. In comparison, elongation at break shows a significant 94% of increase at 1.0 wt% GNP. The results indicate improvement in ductility with an addition of minimal amount of GNP, suggesting enhanced toughness of the composite. The improvement in elongation at break is always a trade-off with

Young’s modulus [17]. However, in this study, the increase in elongation at break does not cause substantial decrease in the elastic modulus

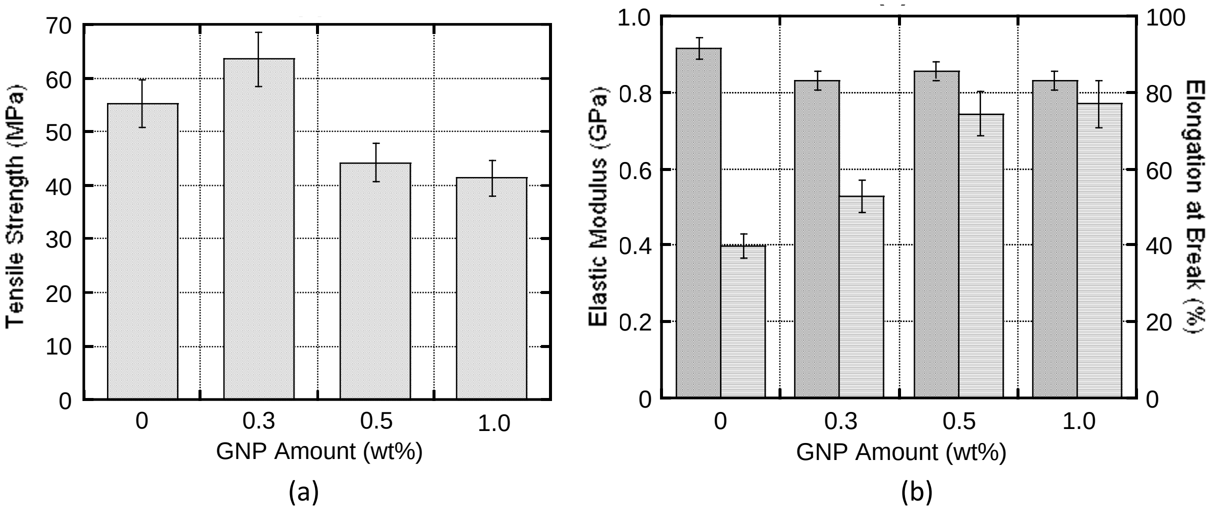


Fig. 1. The values of (a) ultimate tensile strength and (b) Young’s elastic modulus (left bar) and elongation at break (right bar) of nylon 66/GNP nanocomposites at various GNP amount

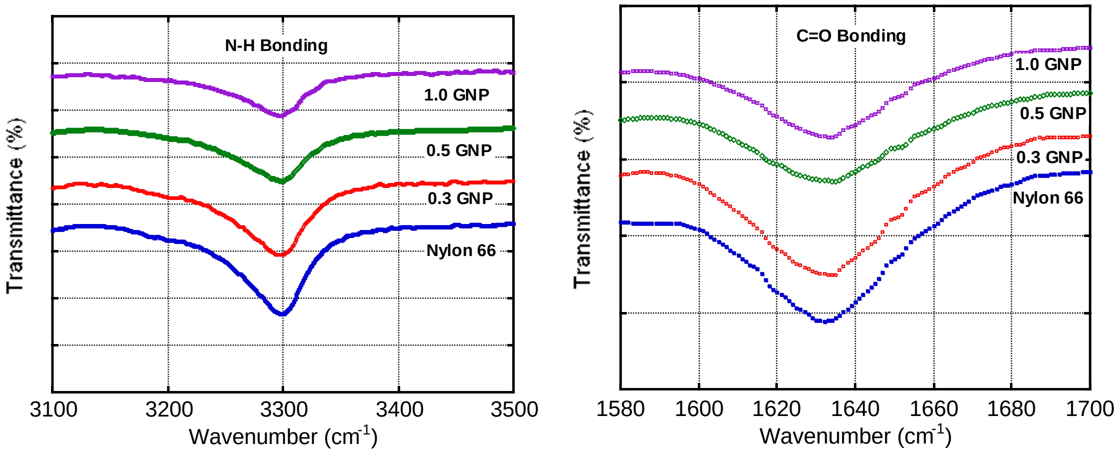
3.3 Fourier-Transform Infrared Spectra

The characteristic bands found in the FTIR spectra of nylon 66/GNP are tabulated in Table 2. Characteristics peaks are observed at 3299.45 and 1533.43 cm^{-1}, which attributed to stretching and bending vibration of the –NH group in nylon 66, respectively. The peaks at 2930 and 2860 cm^{-1} correspond to the asymmetric C–H stretching of methyl and methylene groups in nylon 66, respectively, while 1633 is attributed to –C=O group. The nylon-66 also has characteristic peaks for both crystalline (935 cm^{-1}) and amorphous (1138 cm^{-1}) regions, indicating its semi-crytallinity [18,19].

Table 2
The general characteristic bands found in FTIR spectra of nylon 66

Band Position (cm^{-1})	Functional Group
~3299	-NH (stretching vibration)
~2930	Asymmetric C–H stretching of methyl groups in nylon
~2860	Asymmetric C–H stretching of methylene groups in nylon
~1633	Amide I peak (C=O stretching and -NH stretching vibration)
~1532	Amide II peak (NH deformation, CN stretching)
~1138	Amorphous Peak
~935	Crystalline Peak

Upon the introduction of 0.3 wt% GNP, the two characteristic peaks associated with the –NH group, shift to 3299.43 cm^{-1} and 1532.07 cm^{-1}, respectively. The former is shown in Figure 2(a). Meanwhile, the peak of –C=O group shows a decrease in intensity for the GNP added nylon 66 as in Figure 2(b). Lower peak intensity indicates less hydrogen bonding. Hydrogen bonding also decreases the band intensity of amide I and amide II at 1633.88 (Figure 2(b)) and 1532 cm^{-1}, respectively, since H-bonding occurs with this functional group.

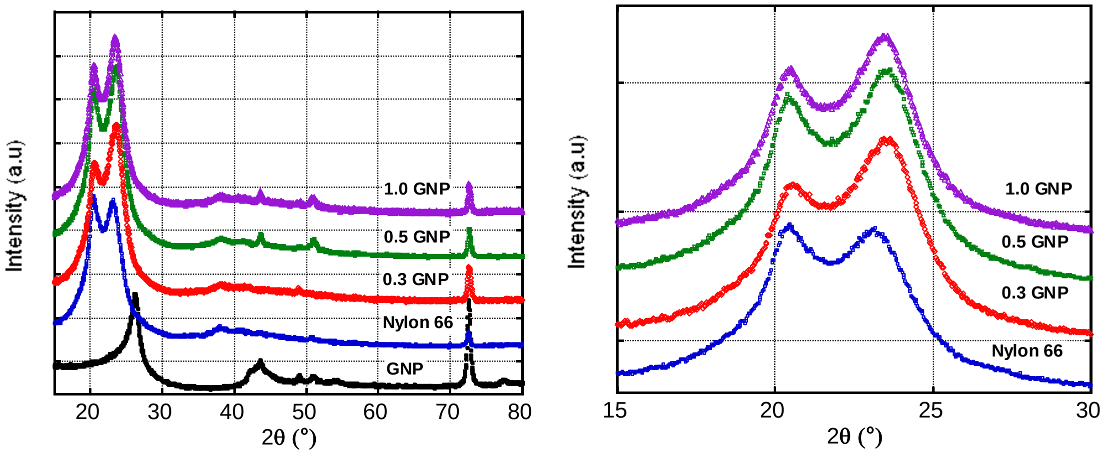


(a) (b)

Fig. 2. Enlarged FTIR spectral zones of (a) N-H bonding and (b) C=O bonding in nylon 66/GNP composites

3.4 X-Ray Diffraction

Figure 3(a) shows the XRD patterns of neat nylon 66 (control), pristine GNP and nylon 66/GNP nanocomposites. The representative diffraction peak of GNP can be observed between 26.3° to 26.4°. Meanwhile, the XRD pattern for nylon 66/GNP nanocomposite shows exceptionally wide diffraction (Peak A and Peak B) from 21° to 23° which corresponds to reflection in α-form polyamide 6 crystal [20].



(a) (b)

Fig. 3. XRD spectra for (a) (15° ≤ 2θ ≤ 80°) range, and (b) (19° ≤ 2θ ≤ 25°) range of GNP, neat nylon 66 and nylon 66/GNP nanocomposites

However, as GNP was added into the nylon 66 matrix, the diffraction peak of GNP observed around 26.4° disappeared. The XRD results suggest that GNP was fully exfoliated into individual graphene sheets in the polymer matrix and that the regular and periodic structure of graphene had disappeared, revealing the disordered and loose stacking of GNPs in nylon 66/GNP composites [18]. Morever, sharp diffraction can be observed at 45° and 51° in the XRD patterns of the nylon 66/GNP containing 0.5 and 1.0 wt% GNP, which are associated to the presence of GNP in the composites.

However, this diffraction peak does not appear in 0.3 wt% GNP filled nylon 66. This indicates that at low volume addition (0.3 wt%), GNP is well dispersed and the graphene layers are not reassembled or reagregated in the nylon 66 matrix [18].

Figure 3(b) demonstrates that upon the addition of 0.3 wt% of GNP, the diffraction peaks of α-form crystals are weaker as compared to 0.5 and 1.0 wt%, indicating a significant influence of 0.3 wt% GNP on the crystallization, aggregation or assembly behaviors of nylon 66 chains [18]. It also affirms the enhanced depressed-effect of nylon 66 crystallization in 0.3 wt% GNP nanocomposites [21].

3.5 *Electrical Conductivity*

Electrical conductivity values of the nylon 66/GNP nanocomposites are presented in Figure 4. Addition of a very small amount of GNP is found to significantly increase electrical conductivity of nylon 66. The electrical conductivity shows a significant increase by seven orders of magnitude (from 10^{-14} to 10^{-7}) at 0.3 wt% of GNP. This value is comparable to the results reported for engineering polymer based conductive polymer compsites, with reported electrical conductivity values in the range of 10^{-9} to 10^{-5} (S/m) [5,14,15]. Moreover, the yielded value is obtained with less amount of GNP as compared to other conductive fillers.

However, with increasing filler concentration (0.5 wt% and 1.0 wt%), the conductivity value decreases slightly by one order of magnitude (10^{-7} to 10^{-8}). The decrease can be associated with the increased agglomeration of GNP in nylon 66, that hinders the percolation threshold, thus slightly reduces the electrical properties of the nanocomposite.

The dispersion level of GNP is crucial in determining the final electrical properties of nylon 66 composites. The good dispersion level of GNP at 0.3 wt% in the composites produces the formation of an effective network for electron path transmittance, which is responsible for the higher electrical conductivity obtained in a highly dispersed graphene nanocomposites system.

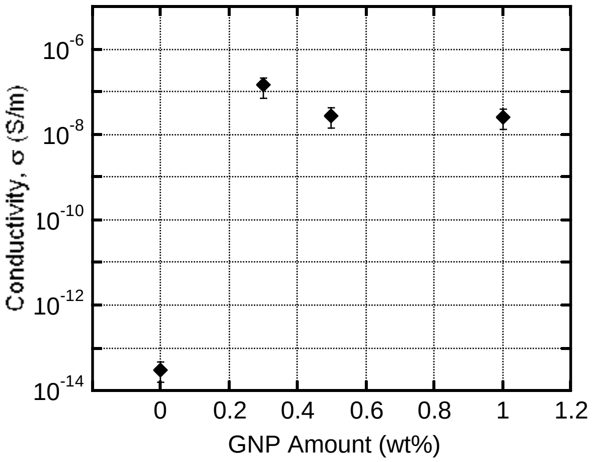


Fig. 4. Electrical conductivity of nylon 66/GNP nanocomposites at various GNP amounts

3.6 Morphological Analysis

Figure 5(a) to (d) show the SEM images of the fractured surfaces for nylon 66 and its GNP nanocomposites. The fracture surfaces of nylon 66/GNP nanocomposites display apparent shear yielding lines suggesting significant strengthening mechanism from well dispersed graphene nanoplatelets in nylon 66 matrix. While nylon 66 exhibits a smooth and brittle surface (Figure 5(a)), nylon 66/GNP nancomposites show a layered structure and rougher surface, indicating more ductility (Figure 5(b)-(d)). The fibrillar structure is an indication of ductile fracture that absorbs higher energy during deformation, explaining the improved tensile strength in nylon 66/GNP nanocompoistes [22,23]. A rougher fractured surface is always referred to the ability of nanographene to deflect crack in polymer matrix.

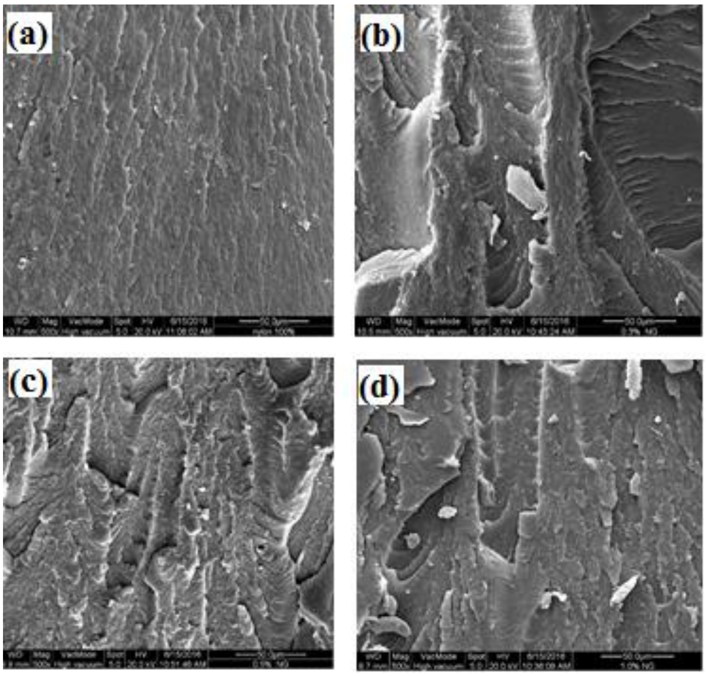


Fig. 5. SEM images of nylon 66 filled with (a) 0 wt%; (b) 0.3 wt%; (c) 0.5 wt% and (d) 1.0 wt% of GNP at 500× magnification

The GNP filled nylon 66 nanocomposites show fibrillar structure due to the shear yielding mechanism with minimal voids or cavities. In this study, an improvement in ductility is observed with the presence of GNP in the nylon 66 matrix (Refer to Figure 1(b)). The presence of as small as 0.3 wt% of GNP as reinforcement material enables the externally applied stress to be transferred and distributed to the reinforcement phase; and only small proportion of the load is sustained by the matrix phase. Therefore, incorporation of GNP into the nylon 66 has transformed the brittle fracture behaviour of neat nylon 66 into ductile fracture behaviour.

4. Conclusions

The potential of using graphene nanoplatelets (GNP) as reinforcement in nylon 66 for producing a nanocomposite with improved mecahnical and electrical performance has been explored. The incorporation of 0.3 wt% GNP is found to significantly improve the tensile strength and electrical conductivity of nylon 66. The increase in tensile strength in the composite with 0.3 wt% GNP is attributed to good filler dispersion as shown in the XRD results, thus contributes to improved interfacial stress transfer. For the same reason, at 0.3 wt% GNP, the electrical conductivity shows a significant increase by seven orders of magnitude (10^{-14} to 10^{-7}) as good GNP dispersion enables it to reach the percolation threshold, thus enhances the electrical properties. However, both tensile strength and electrical conductivity show a decrease as the amount of GNP is further increased to 0.5 and 1.0 wt%. This is due to increased agglomeration of GNP at higher amount of GNP addition.

To summarize, this study shows that a minimal amount of GNP is effective in enhancing both mechanical and electrical properties of nylon 66, and that good dispersion level of GNP is crucial in order for the addition to take effect.

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