

Raman Spectra and Mechanical Properties of Graphene/Polypropylene Nanocomposites

Siti R. Ahmad, Robert J. Young, and Ian A. Kinloch

Abstract—Graphene/Polypropylene nanocomposites were prepared at different filler loading and different average surface diameter 5, 15 and 25 μm of graphene nanoplatelets by using Haake Minilab mixer at 180°C and rotor speed 50rpm. Besides, Haake MiniJet is used to obtain dumbbell shape specimen. The effect of filler loading and average surface area of filler in PP/GnP composites on Raman spectrum and tensile properties were studied. Raman spectrum of graphene particles indicate three major spectrums such as D, G and 2D band. In addition, PP/GnP composites shows the Raman band shift quite strong by increasing GnP loading. In general, increased of graphene nanoplatelets loading have increased the value of modulus of elasticity, whereas tensile strength, elongation at break of composites reduced.

Index Terms—Graphene, polypropylene, Raman spectroscopy.

I. INTRODUCTION

Nanocomposites in polymeric materials offer superior mechanical properties at a lower amount of loading rather than microsize filler [1], [2]. However, the properties is depends on proper selection of nanofiller/matrix weight percentage and the homogenous distribution of filler in polymer matrix due to van der Waals's bonding alignment of nanosizes filler in the matrix [3], [4]. Besides, the fabrication cost of nanofiller exhibit higher compared to microfiller.

The incorporation of graphene in polymer composites such as polypropylene receives interest from industries and research laboratories due to improve of mechanical properties and relatively low cost which is widely used in automobile, household appliance and construction industry [4]. In future application of graphene composites material based on graphene filled polymer composites expected to be an ideal material for several applications such as lightweight gasoline tanks, plastic containers, aircraft component (more fuel-efficient), car parts, medical implants, stronger wind turbines and sport equipments [5], [6].

Graphene is a multifunctional material which is considered

as better nanofiller compared to nanotubes and other conventional fillers. It improves the mechanical and thermal properties of nanocomposites to a great extent with a very small loading. The mechanism of polymer-graphene interaction is mainly governed by polarity, molecular weight, hydrophobicity, polymer functionalities, graphene functionalities and graphene-solvent interaction [7], [8].

Graphene is a basic structure of all graphitic form of carbon in single layer atom (SAL) of sp^2 hybridized carbon atom which is tightly packed into a two-dimensional (2D) in honeycomb structure [9]. It has low density that related to lightweight material with 2.3% light absorbed [10]. Some of the amazing properties of graphene are it is the purest form of carbon, large theoretical specific area (2360 m^2/g), high intrinsic mobility (200,000 $\text{cm}^2\text{V}^{-1}\text{s}^{-1}$), extremely high Young's modulus (~ 1.0 TPa), thermal conductivity ($\sim 5000\text{Wm}^{-1}\text{K}^{-1}$) and optical transmittance ($\sim 97.7\%$) [11].

Polypropylene (PP) is a commodity polymer which offers a combination of outstanding physical, chemical, mechanical, thermal and electrical properties not found in any other thermoplastic [2], [12]. The use of fillers in the preparation of polymeric compositions increases every year as its contribute to the reduction in the final price of the product, improvement in process ability and capability to use for specific applications [12]-[14].

In this paper, the effect of filler loading and surface diameter GnP particles on Raman spectra and tensile properties of Graphene Nanoplatelets (GnP)/Polypropylene (PP) composites were investigated.

II. SAMPLES PREPARATION

A. Material

A commercially polypropylene used was grade 100-CA50 Polypropylene Homopolymer from Ineos Polyolefins Europe (Ineos Olefin and Polymers Europe). The density of this thermoplastic was specified as 0.9 g/cm^3 [15].

Graphene Nanoplatelets (GnP): Grade M with average particle diameters of 5, 15 or 25 μm were supplied by XG Sciences, Michigan, United States of America. The average thickness of Grade M GnP particles is approximately 6 nm, a typical surface area about 120 - 150 m^2/g and 2.2 g/cm^3 of density [16].

B. Mixing and Injection Moulding

Compounding of the composite was carried out by using Haake Minilab Rheomax CTW5 Mixing machine at temperature of 180°C and rotor speed of 50 rpm for 5 minutes per sample.

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Pure PP was prepared using Haake minilab mixing machine. PP was charged into the mixing chamber immediately after the motor started and was allowed to melt for 5 minutes.

For PP/GnP composites, firstly, polypropylene was charged into the mixing chamber immediately after the motor started and was allowed to melt for 3 minutes. GnP was then charged into the mixing chamber and continue the melt mixing for another 2 minutes. The total mixing time was 5 minutes. The total number of the compositions prepared was 10.

After that, the composite samples were injection moulded in a Haake Minijet Injection Moulding machine to perform Dumbbell Shape (Standard: ISO 527-2-1BA), thickness 1.5mm (as opposed ≥ 2 mm) of composites. Injection moulding procedures involve cylinder temperature at 200°C, mould temperature at 40°C under 400 bar of pressure for 10s. All raw materials were prepared and weighed accordingly as indicated in the formulations in Table I.

TABLE I: FORMULATION OF PP/GnP COMPOSITES WITH DIFFERENT FILLER LOADING FOR AVERAGE GnP DIAMETER 5 μ m IN POLYPROPYLENE

Materials	Composites (PP/GnP-5)			
	100/0 (wt %)	100/1 (wt %)	100/3 (wt %)	100/5 (wt %)
Polypropylene, PP (g)	100	99.01	97.08	95.24
Graphene Nanoplatelets (GnP-5) (g)*	0.00	0.99	2.91	4.76

*Note that: The same amount of GnP is used for PP/GnP-15 and PP/GnP-25 composites at three different compositions.

III. CHARACTERIZATION AND MECHANICAL PROPERTIES

A. Raman Spectroscopy

Raman Spectroscopy is used to study the spectrum of pure PP, particles of GnP and PP/GnP composites. Renishaw 1000 Raman Spectrometers with Olympus BH2-UMA microscopes system were used with Renishaw 1000 Raman Spectrometers with Olympus BH2-UMA microscopes system were used with 632 nm HeNe. A spot size between 1 to 2 μ m was obtained by the objective lens with x50 magnification of long working distance (LWD).

The Raman system was calibrated using a silicon sample which shows a sharp peak at 520 cm^{-1} . After the focus point on a silicon sample was obtained, the Renishaw software was setup to perform a scan for a 10% laser power, 3 accumulation and 15 sec exposure time. Then, the samples were prepared.

Firstly, the small amount of GnP particle was compressed to obtain a thin flat layer of GnP using two glass slides. Next, one of the slides which have slightly uniform flat layer was located on the test section. After that, the same step as calibration of silicon samples was repeated to obtain the spectrum of each sample (GnP-5, 15 and 25).

For PP pure and composites, the flat surface of dumbbell shape was placed on the test section. The data was recorded and analyzed using WIRE 3.3 software.

B. Tensile Test

Tensile test of composites was carried out according to ASTM D638 using a Universal Testing Machine (UTM), Instron Model 1121 with Series IX software. The test was performed at $25 \pm 3^\circ\text{C}$, room temperature. A cross head speed of 5 mm/min was used and the gauge length was set at 5 mm.

In addition, the *extensometer* or *extension-meter* was used to measure changes in the length of specimens in stress strain measurements. The tensile properties were measured on 5 identical samples (dumbbell shapes) for each composition and the average value was reported. Tensile strength, elongation at break and modulus of elasticity were recorded and calculated.

IV. RESULT AND DISCUSSION

A. Raman Spectroscopy

Fig. 1 shows the Raman spectrum for pure polypropylene. A Raman spectrum for polypropylene material indicates several peaks. First positions of several peaks were located between 800 and 1500 cm^{-1} . The others of peaks were observed between 2700 and 3000 cm^{-1} . The similar trend of PP spectrum was observed by Bhattacharyya *et al.* [17]

A Raman spectrum is used to allow the ratio of ordered crystalline and disordered sp^2 carbon in the material. It can be seen clearly 3 typical peaks for GnP materials called D, G and 2D (G') bands in Fig. 2.

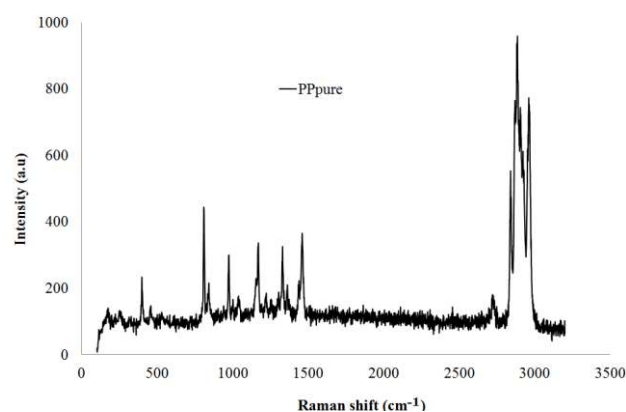


Fig. 1. Raman spectra for Pure polypropylene (PP).

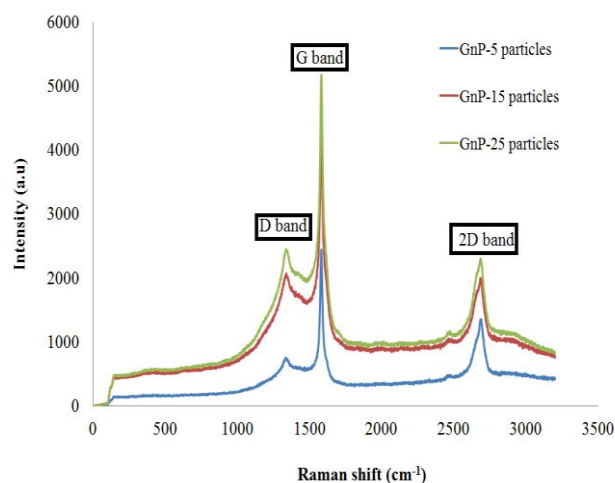


Fig. 2. Raman spectra for Graphene nanoplatelets (GnP).

The value of D band of GnP located at $\sim 1338 \text{ cm}^{-1}$. D band

also known as defect band which is typically found in different forms of graphitic carbon. D band is resulted from one phonon lattice vibrational process. D band is resonant. Normally this band is very weak in graphene and level of defect in sample is about 1300-1400 cm^{-1} [18]-[20].

The primary peak in graphene and graphite is G band. This G-band related to the planar configuration sp^2 bonded carbon that forms graphene. The layer thickness of graphene can be determined using G-band [21].

The 2D band is used to identify the stacking layers of graphene. 2D band is resonant like D band. In this case, the small peak occurred before 2D band position remains that many layers of graphene are likely graphite which is consisting of an AB-type stacking order [21].

The GnP particles with 25 μm of surface diameter exhibit higher Raman spectrum compared to GnP-15 and GnP-5. It might be due to high surface diameter of GnP particles strongly interact with the monochromatic light (laser). Some of researcher state that the Raman shift of a mode of dispersion effect of the D band and the 2D band are depends on the resonant Raman process [18], [22].

Fig. 3 to Fig. 5 shows the Raman spectrum for PP/GnP-5, 15 and 25 composites at different filler loading. All composites shows D, G and 2D bands of the spectrum located at ~ 1335 , ~ 1585 and ~ 2750 cm^{-1} , respectively. It can be seen that the combination of PP_{pure} spectrum and GnP particles spectrum at each composites. The location of D band is increased by increasing filler loading.

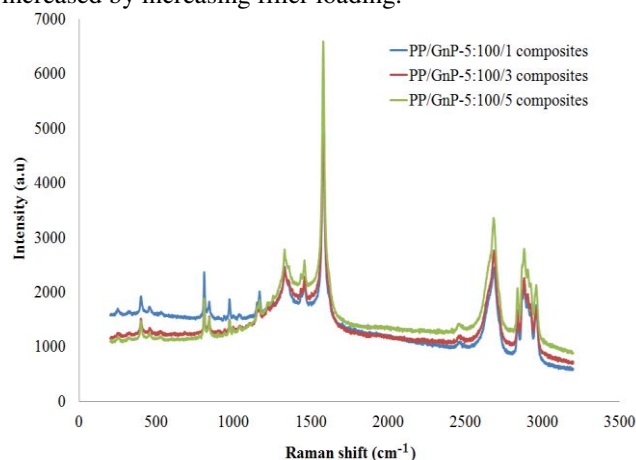


Fig. 3. Raman spectra for PP/GnP-5 composites at different filler loading.

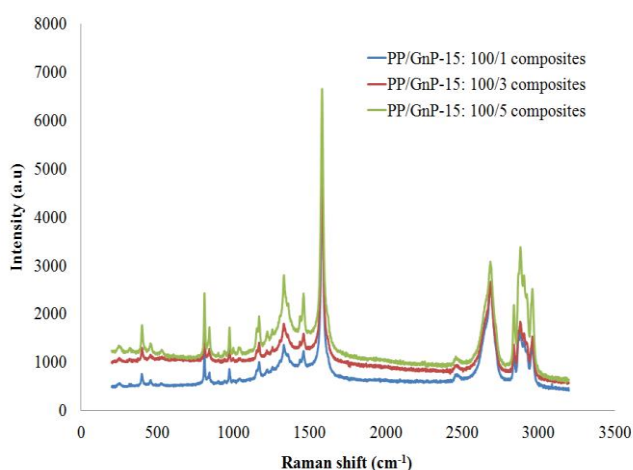


Fig. 4. Raman spectra for PP/GnP-15 composites at different filler loading.

At 2D band, the location of Raman shift is decreased. At higher filler loading, the effect of PP as a matrix is exhibit lower due to the high modulus of GnP. However, there is slightly small effect at G band that can be observed using Lorentzian curve fitting.

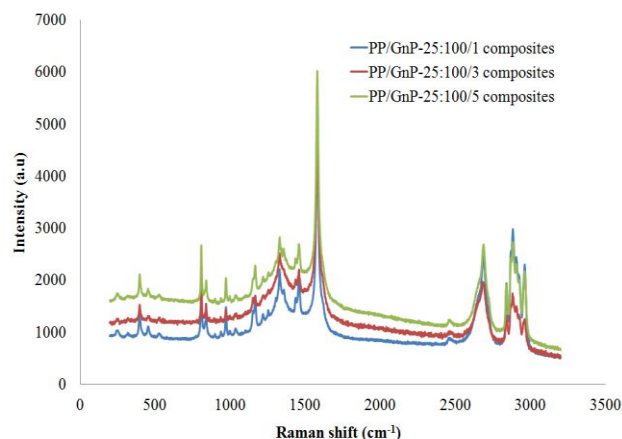


Fig. 5. Raman spectra for PP/GnP-25 composites at different filler loading.

B. Tensile Testing

Fig. 6 shows the effect of filler loading on modulus of elasticity of PP/GnP composites. It can be seen that all the composites indicate increasing Young's modulus with increasing filler loading.

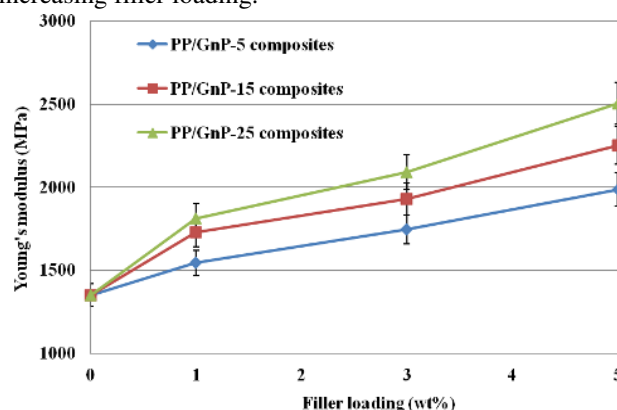


Fig. 6. Young's modulus of PP/GnP composites at different surface area and filler loading.

The addition of 5% of Graphene nanoplatelets results in an increase of the Young's modulus about 57% to 67%, as compared with the PP composites without graphene. The increasing of Graphene nanoplatelets (GnP) loading increases the stiffness of the composites. In addition, the small addition of graphene increased the modulus of composites.

The Young's modulus of the composites increases drastically at this low filler contents with strong polymer-filler interfacial action. In general, the modulus increased by inorganic fillers is achieved at the expense of tensile strength, due to the better dispersion of GnP filler in PP matrix, thus improved filler-polymer interfacial stress transfer [2], [23]. At the similar filler loading, PP/GnP-25 composites indicate higher Young's modulus compared to PP/GnP-15 and PP/GnP-5 composites due to high aspect ratio and surface area.

The effect of filler loading on tensile strength of PP/GnP composites is shown in Fig. 7. The tensile strength of composites with graphene is lower than the composites

without graphene nanoplatelets. This might be due to the weak interfacial interaction between the filler and matrix of the composites. Therefore, the presence of filler that acts as foreign materials in the composites that caused the initial crack propagation in the composites itself.

The huge surface area of graphene nanofillers suggested significant improvement in the mechanical properties of the nanocomposites [18]-[25]. Grade M graphene nanoplatelets particles have an average thickness of approximately 6 nanometers and a typical surface area of 120-150 m²/g. The average particle diameter is varies about 5, 15 and 25 μ m. Therefore, the PP/GnP-25 composites shows lower tensile properties compared to the PP/GnP-15 and PP/GnP-5 due to the larger surface area.

Fig. 8 and Fig. 9 shows the effect of filler loading on elongation at break of PP/GnP composites. The composite without graphene indicates the value of elongation at break is 253.38%. However, the composites with graphene the value of elongation at break are range between 5.5 to 8 %. Consequently, the composites with graphene nanoplatelets exhibited brittle fracture, whereas the PP composites without graphene revealed plastic behaviour.

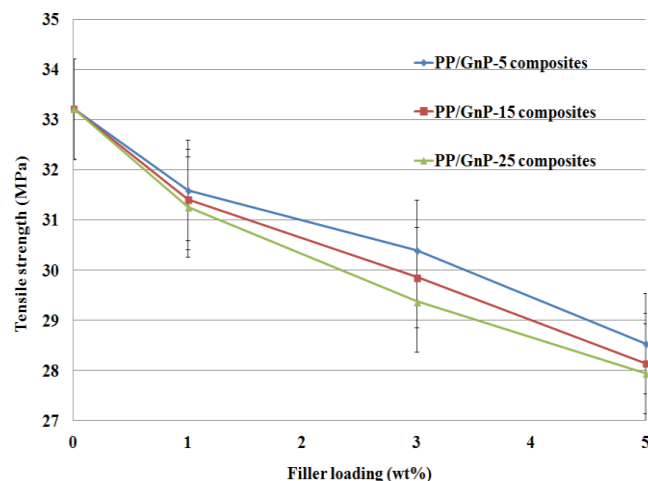


Fig. 7. Tensile strength of PP/GnP composites at different average filler diameters and filler loading.

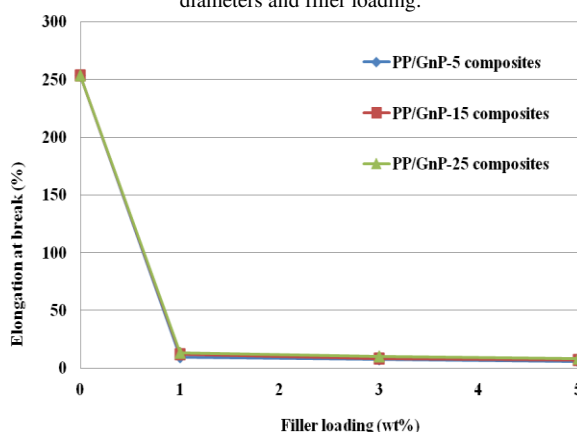


Fig. 8. Elongation at break of PP/GnP composites at different surface area and filler loading.

The incorporation of graphene nanoplatelets in the polypropylene matrix resulted in a drastic decrease in the elongation at break of the composites. The reduction of elongation at break with increasing filler loading might be due to the decreased deformability of a rigid interface between the filler and PP matrix [26]. When filler loading is increased,

more weak interfacial regions between the filler surface and the PP matrix are formed. These occur because cracks travel more easily through the weaker interfacial regions, the composite fracture at a lower elongation with increasing filler loading.

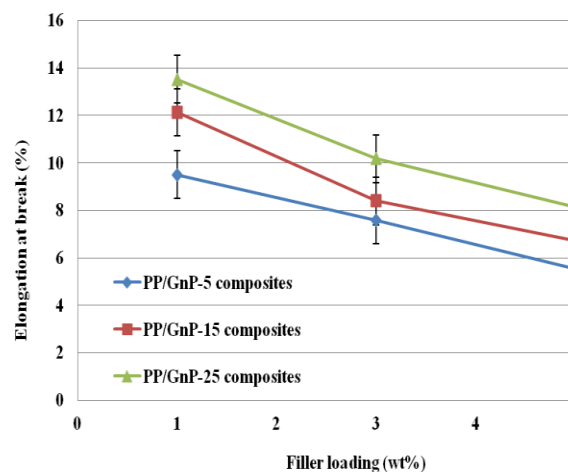


Fig. 9. Elongation at break of PP/GnP composites at different surface diameter and 1, 3 & 5wt% filler loading.

At high filler loading it may become difficult to avoid aggregates. Aggregates lead to less ductile behaviour. The dispersion could be a dominating factor in this threshold; when large aggregates are present the voids that are created by debonding are not stable and grow to a size where initial crack occurs [25]. Composites with 25 μ m of Graphene nanoplatelets diameter seem to be more elastic than composites with 15 and 5 μ m diameter composites. This related to the lowest value of tensile strength of composites with 25 μ m GnP than others.

V. CONCLUSION

In conclusion, the Raman spectrum of pure polypropylene (PP), graphene nanoplatelets (GnP) particles and PP/GNP composites at different filler loading were obtained. Raman spectroscopy analysis indicates three main peaks of GnP such as the D, G and 2D bands. The strongest Raman spectrum of GnP were occurred at the highest graphene loading (5wt%). The presence of GnP into PP matrix has resulted in the improvement in the Young's modulus but the tensile strength and elongation at break decreased with increasing filler loading. The excellent performance of Young's modulus was attributed to the better filler-matrix adhesion and the effect of improved in crystallinity.

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