

Mechanical Characterisation of Polyamide 66/Graphite Nanocomposites

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Abstract

The objective of the research work was to investigate on effect of nano graphite particles on mechanical properties of polyamide 66 (PA66)/graphite nanocomposites prepared by twin screw extruder and injection molding machine. Mechanical properties such as tensile, impact and hardness tests were carried out on PA66/graphite specimens using universal testing machine, impact tester and microhardness tester respectively. The result showed that increase in the addition of graphite into PA66 increases the tensile strength from 27.33 N/mm² to 58 N/mm² and microhardness of the composite from 19.13 to 25.10 VHN; however, impact strength decreased from 406.25 J/m to 131.25 J/m with the addition of graphite. The fracture surfaces of pure PA66 and PA66/graphite composites showed rough and cavitations surfaces respectively.

Keywords: Mechanical Characterization, Nanocomposites, Twin-screw extruder, Microhardness, Impact

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1. INTRODUCTION

Graphite Nanoparticles are used as reinforcement in fabrication of polymer composites to enhance mechanical, electrical and thermal properties[1]. Some researchers tested the mechanical properties of high density polyethylene nano composites reinforced with expanded graphite [2-5]. They found that the tensile strength increased to 18.7% when 40% expanded graphite was added and the young's modulus increased with increase in graphite content [6]. Some researchers worked on blending of PA6/HDPE (polyamide 6/high density polyethylene) with varying compositions. They reported that the composition PA6/HDPE (2%) had increased modulus, hardness and strength. [7] Since PA's have great affinity towards water, researchers blended polypropylene(pp) with varying composition(0%, 10%, 20%, 30%) with carbon fiber as reinforcement. They concluded that water absorption and thickness swelling decreased as PP content increased. The ultimate tensile strength and elastic modulus (after saturation) of 10%, 20%, 30% PP were higher than neat PA.

Zhou et.al [8] studied the PA6 / PPS-CF (carbon fiber) composites and reported that increase in addition of carbon fiber (CF) content in PA6 / PPS composite decreases the impact strength. Li et.al [9] investigated the mechanical properties of PA6-polyurethane (PU) block copolymer reinforced short glass fibers (SGF) and concluded that impact strength decreased with addition of SGF. It has been found that while a lot of work has been done on development of polymer based composites but little work has been done on the development of graphite reinforced polymer composite for structural and bearing applications. The objective of the research work was to develop graphite /PP66 composites and characterised for tensile, impact and ductility behaviour.

2. EXPERIMENTAL STUDY

A commercially available PA66 in granular form and having density 1.14 g/cm^3 supplied by GLS Polymers Pvt. Ltd. Bangalore India was used as matrix material. Graphite powder with an average particle size of $45\mu\text{m}$ and having density of 2.2 g/cm^3 supplied by GLS Polymers Pvt. Ltd. Bangalore India was used as reinforcement material.

Initially, PA66 available in granules form was first preheated for 2 to 4 h at 100°C in hot furnace to remove the moistures completely. Varying amounts of graphite powder viz 0, 10, 20 and 30% by weight were mixed manually with preheated PA66 and also added 0.5% of wax for softening purpose. The mixture is then feed to intake barrel of co-rotating twin screw extruder from hopper. It consists of eight temperature barrels and the intake barrel is provided with water jacket and heating element. The material is transported through the conveying elements to melting zone where the polymer

melting takes place. The melted polymer then enters the venting zone where moisture is removed. Then it enters mixing zone where the polymer is mixed and fed to metering zone, where pressure is built at the die for extrusion. The extrudate is then quenched in cold water and cuts to form granules of PA66 and its composites. During operation the screw speed was set at 220 rpm.

The specimens of PA66 and its composites were molded by using an injection molding machine at temperatures ranges from 240 to 260 °C. The screw speed (volume of materials) was set at 50 cm³/s and the mold temperature was 70 °C. The resulting samples obtained are of size 25 mm diameter and 200 mm long. The samples are then cut and polished to the standard size. The ultimate tensile test was carried out as per ASTM D638-10, using universal testing machine. The specimens of the size overall length 165 mm, gauge length 50 mm, width 12.7 mm and thickness 3mm were used for the study. The impact test was carried out to calculate the impact strength by measuring the energy absorbed by the composite during test. Izod impact strength of PA/G composite samples was evaluated as per ASTM D256, using instrument, Impact Testing Machine, (Release angle of pendulum: 150°). The specimens of size 65.5 mm long × 12.7 mm wide × 3 mm thick were used for the study. The R1 scale was used as the impact load which is having a range of 0-2.71J. Microhardness test was carried out as per ASTM G-99 to study the hardness of polymer and their composite.

3. RESULTS AND DISCUSSION

3.1 Micro structural study (X-ray diffraction-XRD)

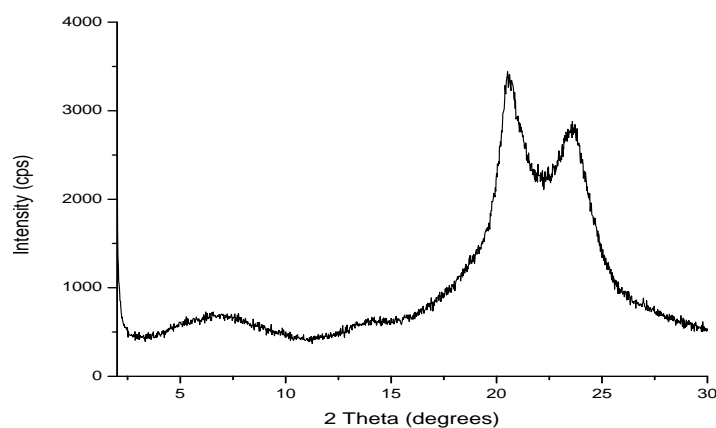


Fig. 1 XRD pattern of the pure PA-66

XRD was used to find the crystalline phase and dispersion of graphite particles in PA66 composites. The peak positions and their relative intensities indicate the crystalline phase of the PA66 and graphite particles and thus, it can be easily identified.

The intensities of the peak positions allow enumerating the phases, to detect the orientations of the crystals and to determine the atomic arrangement of the crystals. The Fig 1 shows the XRD pattern of pure PA66 with two broad peaks at 20.58° and 23.52° . The broad peaks indicate the presence of amorphous materials and also the crystals present in the polymers are very small in size.

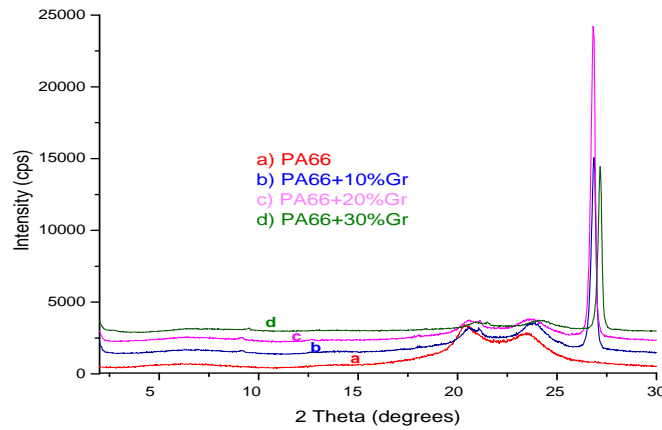


Fig. 2 XRD patterns of the PA66/Gr composites

The Fig. 2 shows the XRD patterns of PA66/Gr composites in comparison with pure PA66. While pure PA66 shows a broad peak, the PA66/Gr composites show sharp peaks at 2θ of near 26.82° which corresponds to a d-spacing of 3.32\AA . This is also the characteristic peak of pure graphite with the standard JCPDS card no 41-1487 [10-11]. Therefore, the occurrence of peaks confirms the presence of graphite. However, less intense peaks indicate uniform dispersion of graphite particles. We know that graphite is a layered material which is characterized by strong covalent bond within the carbon layers and weak van der waals interaction between successive carbon layers. Thus variety of atoms and molecules can be intercalated between carbon sheets, resulting in the formation of intercalated graphite and yielding an increased d-spacing [12].

3.2 Tensile strength

The tensile test was conducted for pure PA66 and their composites using universal testing machine. The ultimate tensile strength (UTS) for each specimen was found out by dividing the maximum load from cross sectional area of the specimen. The variation in the ultimate tensile strength (σ), tensile modulus (E) and breaking elongation (ϵ) with respect to weight % of the graphite reinforcement in PA66 is shown in the Fig. 3. From the Figure, it can be seen that the tensile strength increases with the addition of graphite in the PA66. When the content of graphite increased

from 10 wt% to 30%, the tensile strength of the PA66 changed from 42 to 58 N/mm², showing a significant increase compared with the pure PA66. Tensile modulus also improved with the addition of graphite in the PA66. Therefore it can be seen that graphite is effective to improve the tensile strength and modulus of PA66. However, the tensile strength improvement reduces the ductility of the polymer [8]. So the breaking elongation rate of PA66 / Gr composites shows a decreasing nature as the content of graphite increases.

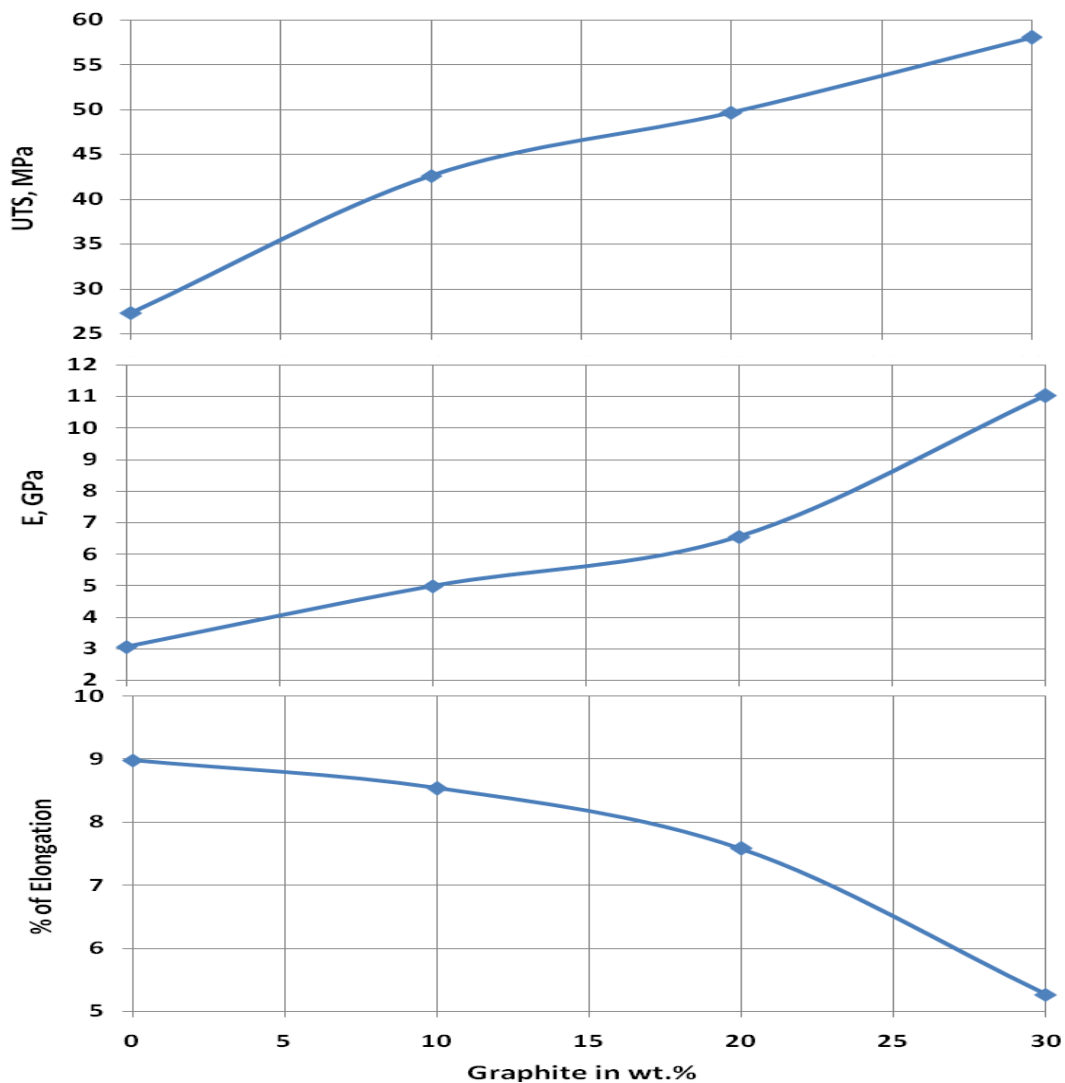


Fig. 3 Effect of Graphite on a) tensile, b) Young's modulus and c) % of elongation of PA66/Gr composites

3.3 Impact Strength

The variations in the impact strength with respect to graphite content wt% of PA66 and their composites are shown in the Fig. 4

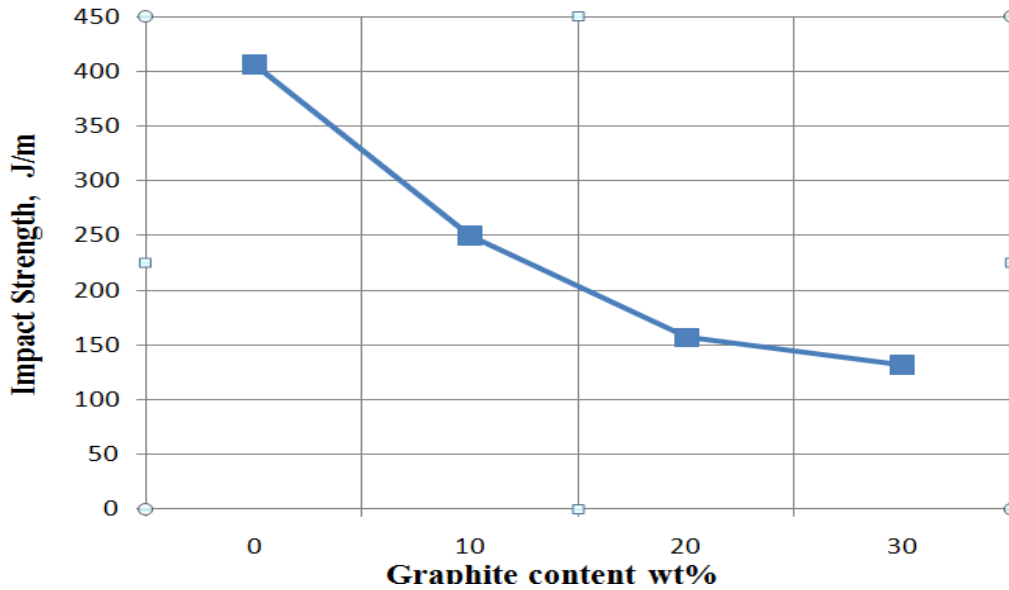


Fig. 4 Effect of graphite content on impact strength of of PA66/Gr composites

It can be found that the impact strength of PA66 / Gr composites decreases as the content of graphite increases in the PA66. When the content of graphite increased from 10 wt% to 30%, the impact strength of PA66 changed from 250 J/m to 131.25 J/m showing significant reduction in impact strength compared to pure PA66. It is due to poor compatibility between graphite and polymer matrix and also addition of graphite fillers result in the formation of agglomerates of filler in the polymer matrix, which act as internal notches [8].

3.4 Micro hardness

The microhardness for PA66 / Gr composites is shown in the Fig. 5. It is clear from the Figure that hardness of the PA66 / Gr composites improves with the increase of graphite content. The improvement of hardness of composite can be explained as follows: under the action of compressive force, the thermoplastic matrix phase and the filler will be pressed together, touching each other and offering resistance. So, even though the interfacial bond may be poor, the interface can transfer load more effectively, resulting in the enhancement of their hardness [8].

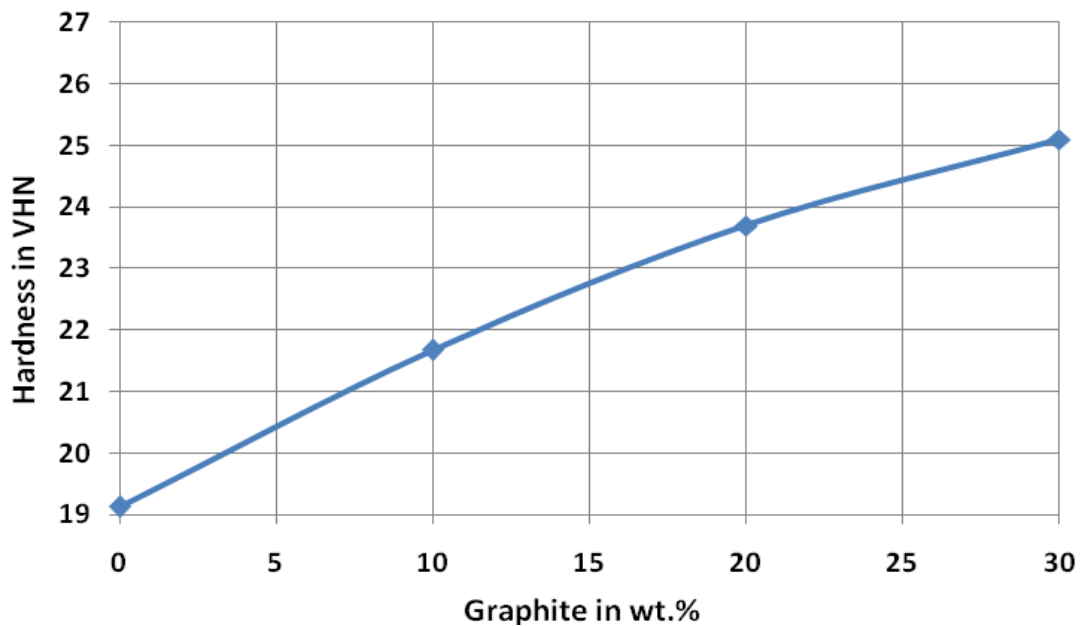


Fig. 5 Effect of graphite content on hardness of PA66/Gr composites

3.5 Fracture surface studies

Fig.6 a) , b) c) and d) show the tensile fractured surfaces of PA66/Gr composite filled with 0, 10, 20 and 30 wt.% of graphite powder respectively. The fracture surface of pure PA66 blend is not smooth and some cavitations can be seen from Fig. 6 (A). From the figure it can be seen that only few cracks (indicated by white arrows) were formed for matrix material but in composites more crack are seen at the interface which causes fracture in the specimen. The SEM morphologies of 10 wt%, 20 wt% and 30 wt% graphite powder filled PA66 composites are shown in Fig.6.B-D, respectively. Graphite with high strength and high modulus functions as the major stress concentration point. Thus, some graphite particles were detached from the polymer matrix as shown by the white circles in Fig.6. B-D.

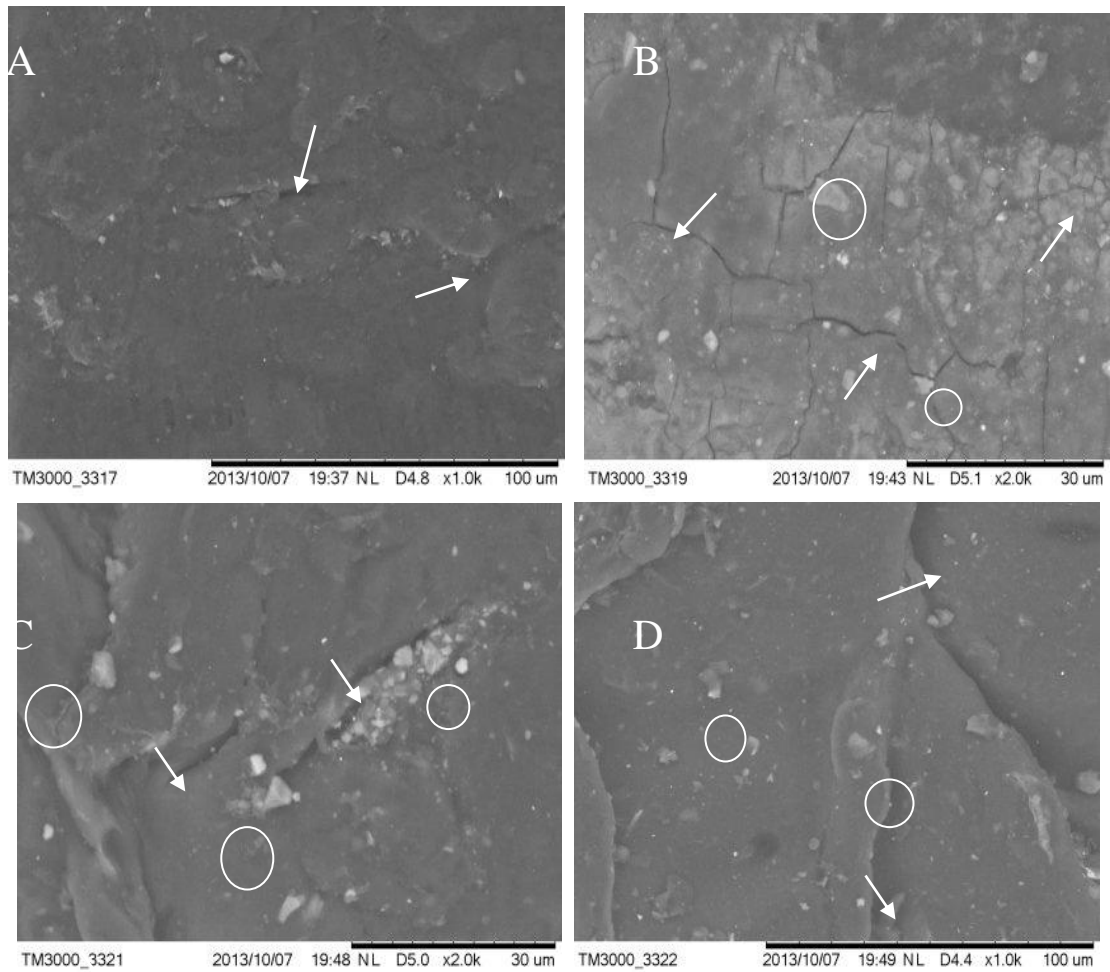


Fig.6. Tensile fractured surface of PA66/Gr composites: A) PA66, B) PA66+10%Gr, C) PA66+20%Gr, D) PA66+30%Gr. Arrow indicating cracks.

CONCLUSIONS

The effect of amount of graphite particle on mechanical properties of PA66 / Gr composites was studied. The following conclusion are drawn from the work.

- The addition of graphite (0 to 30% wt) into the PA66 matrix increases the tensile strength from 27.33 N/mm² to 58 N/mm² and also increases the tensile modulus from 304 N/mm² to 1102.71 N/mm². However it decreases the breaking elongation from 8.98% to 5.26% which leads to reduction in ductility of the composite material.
- The increase in addition of graphite also increases the microhardness of the composite from 19.13 to 25.10 HV but reduces the impact strength of the

composite from 406.25 J/m to 131.25 J/m.

- The fracture surface study shows that formation of cavities and cracks which lead to fracture of the specimens.

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