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The Effect of Boron Nitride Reinforcement on Physical and Mechanical Properties of Polyethylene Tetraphalate (PET)

Mahesh A. Kori [1, 2*], Anand N. Sonnad[1], Shravankumar B. Kerur[1]
[1] *Mechanical Engineering Department, Basaveshwar Engineering College, Bagalkot, Karnataka, India*
[2] *KLS Gogte Institute of Technology, Karnataka, India*

Abstract:
Polymers are economical, easy to fabricate, low weight, decent strength, good thermal and electrical insulating properties, and many more advantages made polymers reach the highest magnitude and replace all traditional materials with various types of reinforcing elements as desired. In many capacities, they have significantly suppressed traditional materials. There are numerous advantages that make polymers very promising contenders for domestic, commercial, and engineering applications. In the present research, Polyethylene terephthalate (PET) polymer as a matrix and hexagonal boron nitride (h-BN) as reinforcement were used, and are fabricated using ball milling and injection molding process. The properties of h-BN addition to (0 – 40 wt. %) were studied, recorded, and analyzed the physical and mechanical properties. Optical microscopy images showed good distribution of BN particles in the PET matrix. The Vickers microhardness, impact strength, were found to increase by 28% and 70%. Tensile strength and flexural bending test results of composites were found to increase approximately 54% and 52%, and percent elongation has reduced by almost 46% by an increase in the BN particles compared to pure PET matrix.

***Keywords**: polymer composite, boron nitride, injection molding, Optical Microscopy, mechanical properties.*

1. Introduction

Polymer composite materials are a combination of matrix and reinforcement which have different physical and chemical properties. The need of adding materials in the pure polymer is to take advantage of the better properties of both materials without compromising on the properties of either. The reinforced particle composites absorb the major load and the matrix assists as the mediocre for transfer of the load. The addition of reinforcing materials further increases the purposeful properties of the composites. These composite materials find applications in aerospace components, industrial, automotive, naval, defense, electric insulation, transportation, etc. Particle reinforced polymers have to function efficiently and effectively in the corrosive environment as all traditional cannot function for a longer period and this leads to functional failure as a result it leads to larger economical damage. Harder reinforcement particles usually high strength metals, oxides, and ceramics particles are getting used these days to enhance the surface damage resistance, even up to a few orders of magnitude [1]. Many researchers have carried out extensive research on Polymer matrix composites and investigated and determined polymer composites play a vital role in decreasing the applications of corrosion resistance and wear-resistant properties by the addition of reinforcement materials [2-6]. Therefore polymers have become an issue of extended interest. The reinforcement materials generally added are metallic, organic, and inorganic particulate materials in numerous shapes (fibers, particles, particulates) and sizes (micro and nano). Polymer composites with thermal durability at high temperatures are most desirable for industrial applications [6]. Polymer composites are most desired due to their flexibility in manufacturing design, low cost of fabrication, corrosion resistance, and majorly its low density, [3, 4, and 5]. For almost the last decade ceramic reinforced particles in a polymer matrix have the potential substance of extensive research. The mainly intended ceramic reinforced polymers have properties of stiffness improvement, cost reduction, simplicity of fabrication, and sustainable applications [6, 7]. Polymers have been widely used owing to their advantages such as low cost, low weight, endurance, hygiene, and design adaptability. The specified properties of polymer composite are achieved by numerous reinforcing materials of chemical compounds to achieve the physical, mechanical, electrical, thermal, and tribological applications [8]. For many engineering applications of polymer composites, the tribological behavior is important for understanding the mechanical properties [9].

PET material is the most common polymer used in day-to-day life. It's one of a leading polymer having applications starting from domestic to aerospace, and extensively used for electronics as PCB's, automotive as decorative interiors, aerospace as ergonomic structures, marine as corrosion-resistant materials, food packaging, lightweight fabrics, domestic usage molded parts, and plenty of applications in numerous fields [10]. Even in domestic applications, PET polymer is prominently used as a general pure plastic bottle container. Due to the versatility of PET polymer, many researchers have studied extensively to increase the properties of PET polymer as matrix material. Various materials are reinforced in PET polymer, like commonly used reinforcement are oxides such as ZrO2, Al2O3, SiO2, TiO2, Cu, CuO, CuS, CuF2, PBS, CaS, and boron nitride [1-12] for prerequisite applications, however, very limited research is done with PET polymer reinforced with BN ceramic particles processed by an injection molding process. Hence an attempt is made to enhance the physical properties and mechanical properties of PET polymer by BN particles of average 50 microns size as reinforcement was used.

2. Materials and methods of Experimental work

2.1 Materials

Commercial purity of 99% PET powder Accucomp PET008L grade of 1.38 g/cm3 density is used as matrix material and h-BN (purity: > 99%) powder of 2.1 g/cm3 density received from Intelligent Materials Pvt. Ltd, India, and M/s Nanoshell LLC were used as reinforcement material.

2.2 Preparation of PET/BN composites

The powdered PET polymer and BN particles were mixed and ball milled at room temperature together in correct proportions as per calculated wt. % (0 – 40%) by using eqn. 1 in an lab developed ball milling setup. The zirconia balls were used for mixing in the ball mill PET to BN powder at a 10:1 ratio. The ball mill was rotated at a speed of 60 rpm for 4 hours under dry conditions, BN micro size powder was added in 0, 5, 10, 15, 20, 30, and 40 wt. % in the PET matrix. During the ball milling process, the temperature of the mixture is maintained at room temperature by turning off the machine for 15 minutes every hour. The mixed powders from the mill were taken out and dried at 180 °C in the oven (INLAB Equipment (Madras) Pvt. Ltd. Chennai, India) to ensure complete moisture is removed from the composite mixture if any. The composite powder after drying was taken for processing in the molding machine. The injection molding machine barrel has 3 heating zones of temperature 200, 250, and 290 °C, and a pressure of 200 bar pressure is applied. The samples are cooled in the die only are ejected from the machine. All the test specimens are fabricated as per ASTM standards. The schematic of the injection molding machine process is as shown in Fig. 1. Commercially injection molding machines are used widely for polymer processing for several applications by varying the die molds. The benefits of the Injection molding process are process automation, low cost, permitting better-quality contours details, and cost of manufacturing is also economical, produce larger volume components and mass production in contrast to compression or hot compacting molding processes [18-20].

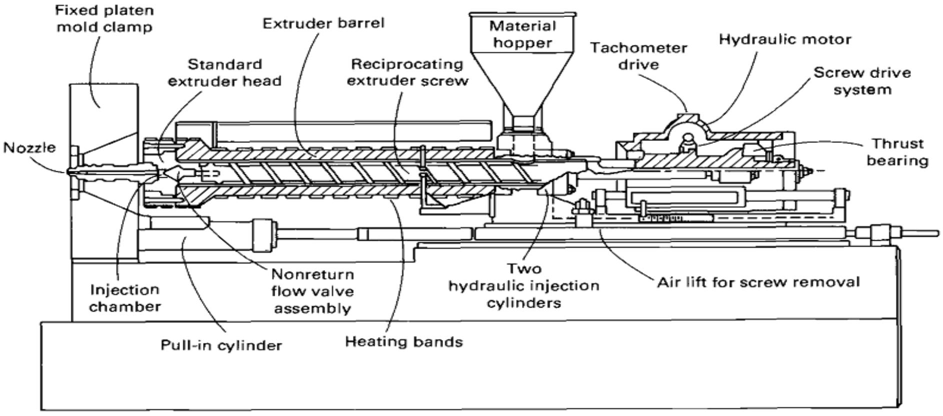

Figure 1. Schematic diagram of molding machine

The composite powder mixture was taken in the machine hopper and the barrel is preheated to 200, 250, and 290^0 C. The melting temperature of PET polymer is 280^0 C and was achieved in three phases in the barrel heaters, the barrel were at 200^0 C and 250^0 C the composite material becomes softer by the heat transfer from the barrel wall heaters, as the lead screw rotates it applies the high-shear forces on the powder and transfers the material to the die cavity. The powder turns completely into the molten state at 290^0 C and is injected through runner and gates into mold die with high pressure. Later the die mold is allowed to cool for solidification at room temperature. The specimens are prepared as showed in Table 1 for different weight percent, by using Equation 1. The specimens were fabricated asper ASTM standards, Impact test-ASTM D256, tensile test-ASTM D628, and flexural test-ASTM D790,

Table 1. Specimen codes and compositions.

Specimen code	PET- 0	PET-5	PET-10	PET-15	PET-20	PET-30	PET-40
Wt. % of BN	0	5	10	15	20	30	40
Vol. % of BN	0.0	3.34	6.8	10.4	14.1	22.0	30.5

Determination of wt.% and vol. %of PET/BN composite, The equivalence vol. fraction (V_f) of wt. fraction (W_f) of BN was calculated by using equations:

For pure PET, required weight for sample preparation is calculated by

Mass (m) = Volume (V) * Density (ρ), gms (Eqn. 1)

V = volume of sample, cm^3

ρ = density, g/cm^3

m = mass, g

$$Vf = \text{Volume fraction} = \left[\frac{W_f}{(W_f + (1-W_f))\left(\frac{\rho_f}{\rho_m}\right)}\right] \quad \text{(Eqn. 2)}$$

Where, W_f = wt. fraction of reinforcement, ρ_f = density BN (2.1 g/cm^3), ρ_m= density PET (1.38 g/cm^3).

Table 1. shows the W_f Composition and equivalent V_f in percent. Theoretical density $\boldsymbol{\rho_c}$(th), of PET/BN composite and weight of matrix and filler required for sample preparation of PET/BN composite are calculated by "Rule of Mixture (ROM)" [11] by using Archimedes principle experimental density was calculated [11,12]. The average of 3 readings were considered for calculations, specimen is weighed in air and later when immersed in medium and its weight is recorded.

2.3 Characterization

PET/BN composite theoretical densities were calculated by using rule of mixtures (ROM) [11]. Densities of PET matrix 1.38 g/cm^3 and BN reinforcement 2.1 g/cm^3 were used. It was presumed that no loss and no cavities were formed in the processing of specimen fabrication. A high precision electronic balance AFCOSET model: ER200A is used to weigh the specimen. XRD peaks of BN powder (pure PET-100), PET powder (PET-0), and its composite comprising 5, 10, 15, 20, 30, and 40 wt% BN were carried out at room temperature by Philips X'Pert X-ray diffraction system with Cu Kα radiation (λ = 1.54) for 2θ. Microhardness of PET/BN composite were calculated using (Model-MVH–S-AUTO-Z, Metatech Industries. Pune, India,) at 100 g load and 10 s dwell time. Average of five readings were used for calculations of microhardness of PET/BN composites. Tensile tests and flexural tests are carried out by using a UTM machine (Model-UNITEK-16100-STS) until specimens breaks. 5 mm/min were speed rate was used for applying a load, and Percent elongation and applied forces were recorded and results obtained from the tests generally suggest or predict the material behaviors and responses under other different loadings, this helps to select composite for a suitable applications. In tensile test, some of properties can be directly measured are tensile strength, elongation, and % reduction in area. Some properties can be determined indirectly are Young's modulus, yield strength, Poisson's ratio, and strain hardening characteristics, similarly flexural load, maximum deflection are studied from flexural testing and flexural strength, flexural modulus are determined. The impact test of PET/BN composite results was obtained using a Fine testing Machine (FTM), Model FIT-14, Miraj, India, the average of three specimens was considered for the impact test. The energy (J) absorbed by the specimen before fracture determines the impact toughness of a material to its cross sectional area. The weight and height at which the pendulum is released (potential energy) and swing of pendulum after it has struck the specimen start and end of energy pointer determines the energy absorbed (J). The PET/BN material impact strength is determined by energy absorbed before fracture to the cross-sectional area of the specimen. Optical microscopy images were observed by model SuXma-Met III, Metallurgical inverted microscope, Conation Technologies, Pune, India.

3. Results and discussions

3.1 Density

The theoretical and experimental densities of PET/BN composites are shown in Fig. 2. The increase in BN percent linearly increases the composite densities. The higher density of BN particles increases the pure PET matrix. The results shows good samples of defect free specimens during injection molding process and proper ball mill blending. Therotical and experimental densities of PET/BN are nearer to therotical densities at lower vol.% (PET-0, PET-5, PET-10 and PET-15) and at upper vol.% (PET-20, PET-30 and PET- 40) differs from linearity as some porosity was observed, and higher concentration (PET-30 and PET- 40) more porosity was observed by addition of BN percent. The porosity confirms by addition of BN wt.% the interparticle space reduces and in turn resulting in bigger agglomerations of BN elements. The controlling of uniform distribution of BN particles in molten polymer at machine barrel becomes more challenging as a result agglomeration is observed as cnfirmed in optical microscopy, which leads to porosity. The maixmum 4.21 % porosity was observed at PET- 40 composite compared with therotical density. The results obtained are inline with other researchers.

Theoretical density and weight of matrix and filler required for sample preparation of PET/BN composite are calculated by “Rule of Mixture (ROM)”

For a given weight fraction, the volume fraction of reinforcement can be calculated
Theoretical density, $\rho_{th} = (\rho_m * V_m + \rho_f * V_f)$, g/cm^3 (Eq^n. 3)
V_m = (1- V_f)
V_m = volume fraction of matrix
ρ_m = density of matrix, g/cm^3
V_f = volume fraction of filler
ρ_f = density of filler, g/cm^3

Determination for experimental density (ρ_{exp}), by Archimedes principle
The experimental density is obtained by using Archimedes principle.
Experimental density, ρ_{exp}= $\left[\left(\frac{W_{air}}{W_{air}+W_{medium}}\right) * \rho_{medium}\right]$, g/cm^3 (Eq^n. 4)
ρ_{med} = density of ethanol = 0.791 g/cm^3
W_{air} = weight of specimen in air = gms
W_{med} = weight of specimen in ethanol = gms
ρ_{exp} = experimental Density = g/cm^3

Determination of Porosity
Porosity in the specimen prepared is calculated by
Porosity = $\left[\left(\frac{\text{Theoretical density } (\rho_{th}) - \text{Experimental density}(\rho_{exp})}{\text{Theoretical density } (\rho_{th})}\right) * 100\right]$ (Eq^n. 5)

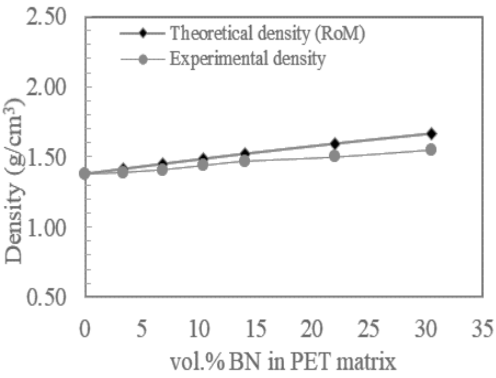

Figure 2. Density of PET-BN - vol. % BN of composites

3.2 Microhardness Hardness

Fig. 3, shows theoretical and experimental microhardness of PET/BN composites, by using rule of mixtures (ROM), theoretical values were calculated. The microhardness of pure PET is 7.9 kg/mm2 and BN microhardness is 76 kg/mm2 the microhardness of composites increases with addition of BN percent in the virgin PET matrix. The experiemntal hardness ic calculated from the machine readings as shown in Fig. 3 and average of 5 readings were considered.

Determination of experimental and theoretical micro hardness

Determination of experimental micro hardness

$$\text{Experimental micro hardness, Hv} = \left[\frac{(1.8544 * P)}{d^2}\right], \text{kg/mm}^2 \qquad (\text{Eq}^{\text{n}}.\ 6)$$

P = Applied load (in kg)
d = Average indentation diameter in mm.

Experimental Vickers micro hardness were recorded from machine directly.

Determination of theoretical micro hardness

Theoretical calcualtions of microhardness were predicted from the rule of mixtures (ROM).

$$\text{Hardness of composite}, H_c = \left[\mathrm{H_m}\left(1 - V_f\right) + \left(H_f * V_f\right)\right], \text{kg/mm}^2 \quad (\text{Eq}^{\text{n}}.\ 7)$$

H_m=microhardness of matrix, (pure PET is 7.9 kg/mm^2 [experimental hardness])
H_f = microhardness of BN particles, (pure BN particles 67 kg/mm^2 [46])
V_f = volume fraction of the BN particles.

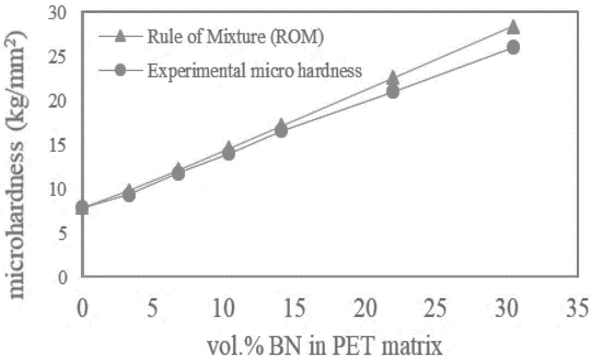

Figure 3. Microhardness - vol. % BN of composites

The vickers micro hardness tester uses micro indentation technique and extensilvely for polymer composites to get micromechanical properties of polymers and polymer composites. The microhardness of PET/BN composite progressively increases with addition of BN reinforcement. The maximum microhardness values of the PET-40 composite increased to 21 kg/mm2, approximately 35% increase compared to pure PET matrix. By addition and uniform dispersion of BN particles increase the microhardness and attributing to the higher hardness. The BN particles uniform dispersion resists the deformation to externally applied load. However, increasing BN percent in the PET matrix reduces the inter-particles spaces of BN thus, resulting in increased resistance to indentation as observed presence of agglomaration and voids in optical microscopy. At higher vol. % (PET-40) the maximum porosity was around 4.21%. The therotical microhardness were calcultaed by using rule of mixture [11] and were compared with experimental microhardness values. The presence of voids in the matrix offers least resistance to indenter and resulting lower microhardness values related to void free specimens.

3.3 Impact strength

Impact test results at room temperature of PET/BN composite are shown in Fig. 4. Fracture energy and impact strength increases and absorbs more energy for higher concentration BN powder compared to pure PET matrix before failure. As BN particles in per unit volume increase with wt. % obstructs the crack propagation, (i.e. absorbed energy increases). The fracture energy and Impact strength are increasing as the contact surface area of PET are decreasing for increased wt. % as wettability of BN particles increases resulting in increasing in transmitting energies from matrix to BN reinforcement. The grain boundaries are increasing as an agglomeration of BN reinforcement addition at higher wt. % resulting in the better impact strength of PET/BN composite.

Determination of theoretical Impact strength

$$\text{Impact strength} = \left[\frac{\text{Impact Energy (Joules)}}{\text{Area of cross section (cm}^2\text{)}}\right], (\text{J/cm}^2) \qquad (\text{Eq}^n.\ 8)$$

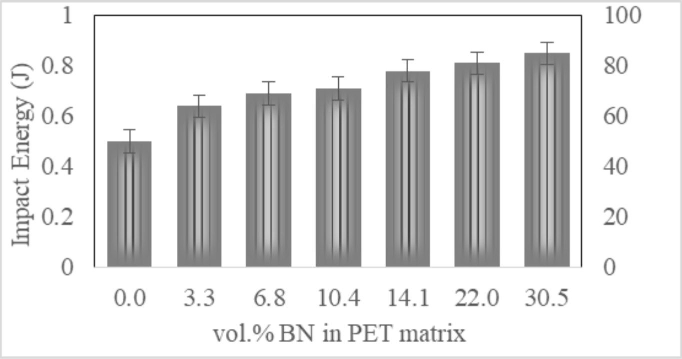

Figure 4. Impact energy and impact strength versus vol. % BN of the PET/BN composites

3.4 Tensile strength

Mechanical properties - The most basic characteristics of semi crystalline polymers are tensile behavior. Generally, the predictable that microscopic properties depend closely on the strain rate, hence the dependence of strain rate behavior is necessary to promote the wide engineering applications. The necking of the polymeric material generally signifies softening and reduces the elastic module [13], the morphology changes of a particle structure with increase in elastic modulus known as strain hardening, as the neck grows and reaches a limiting region. Similar trend was reported by other researchers [13, 14].

Determination of stress and strain

The *engineering stress*, σ, and *strain,ε*, are calculated by following equations:

$$stress, \sigma = \left(\frac{F_n}{A}\right), \text{N/mm}^2 \qquad (\text{Eq}^n.\ 9)$$

F_n is the force and A is the cross-section of the gauge section

$$strain, \varepsilon = \left(\frac{\Delta L}{L_o}\right) = \left(\frac{L-L_o}{L_o}\right) \qquad (\text{Eq}^n.10)$$

ΔL is the change in gauge length,

L_0 is the initial gauge length,

L is the final length.

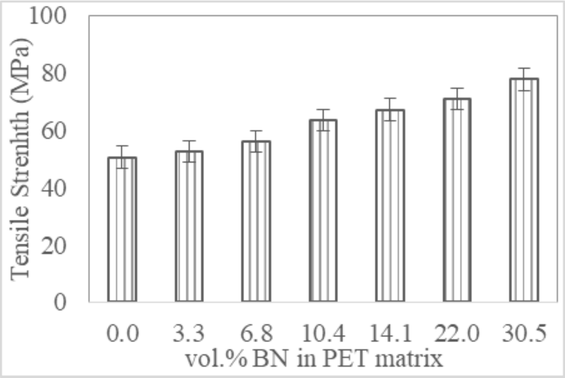

Figure 5 (a) Tensile strength and % elongation versus vol. % BN of the PET/BN composites

Fig. 5 (a, b) shows, tensile strength and % elongation of the PET/BN composites, this shows the tensile strength of composite has increased by almost 54%, and percent elongation reduced by almost 39%, this attributes the behavior is brittleness by addition of BN particles resulting into agglomeration resulting in insufficient transfer of stress to PET matrix and the lack of proper load distribution between the matrix and the BN. Moreover, BN particle reinforcement has shown to contribute to an enhancement of Young's modulus.

3.5 Flexural bending

The addition of BN reinforcement increases the flexural modulus or stiffness of pure PET polymer as shown in Fig. 6. The properties of reinforcement majorly depends on its particle size and aspect ratio.

Higher the aspect ratio, higher the stiffness for improving flexural strength and flexural modulus [14].

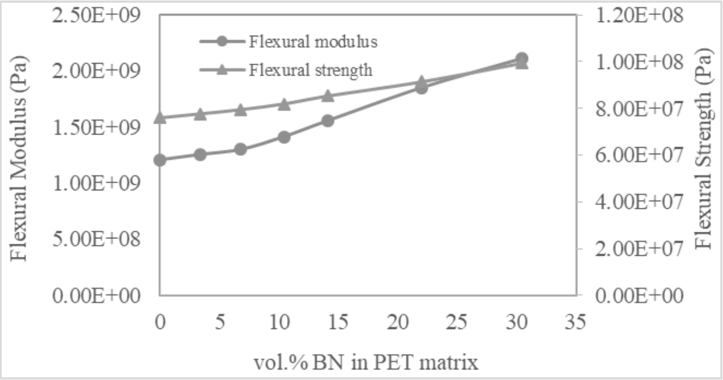

Figure 6. Flexural modulus and flexural strength - vol. % BN of the PET/BN composites

Pure PET samples are ductile and yielding higher deformation compared to BN reinforcement composites. By the addition of BN particles yielding is reduced and flexural strength and flexural modulus are increased, it’s observed from results due to BN addition tensile strength and flexural strength, microhardness, impact strength are increasing and resulting in a decrease in elongation at break and deformation this is due to ductile to the brittle transition of composite specimens.

3.6 XRD analysis

The (2θ) diffraction peaks (JCPDS: 34-0421[15]) are predominant crystallographic reflections for virgin PET polymer (PET-0), and virgin BN (PET-100), and compositions containing 10, 20, and 30 wt. % (PET-10, PET-20 and PET-30 respectively). Fig. 7 shows 2θ values of XRD patterns for pure PET and BN reinforced compositions. The patterns of XRD test shows a superimposition of crystalline peaks. This suggests that the PET is a semi-crystalline polymer [16]. This PET/BN powder mixture no longer shows any peaks of elements present in the system as impurity. All compositions sows diffraction peaks of PET and BN elements only. This confirms the BN morphology is unchanged in the fabrication process. It is likewise determined that the peak intensity of BN vol. % increase in the PET matrix.

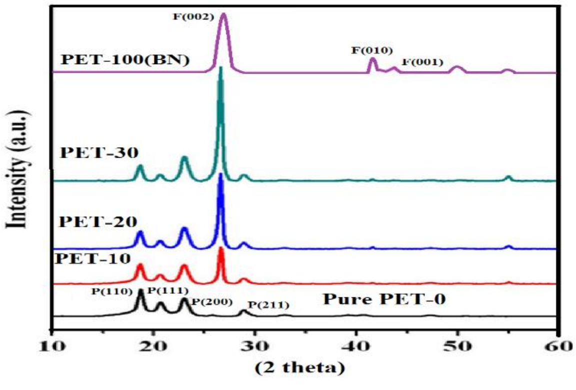

Figure 7, XRD (2θ) peaks of PET and BN powder PET/BN composites

3.7 Optical microscopy.

Fig. 8 (a-f) shows the distribution of BN in PET matrix prepared by ball milling. BN is homogeneously dispersed throughout the PET matrix to lower concentration (a-d) and at higher concentration agglomeration is observed, BN particles tend to form a fine 3D network and causing in increasing impact strength, hardness, and tensile strength of the PET/BN composite [17]. The porosity present in BN vicinity, leads to agglomeration affecting decreased density, and lower elongation of composite results in brittleness and higher vol. % decreases particle distances between the BN particles.

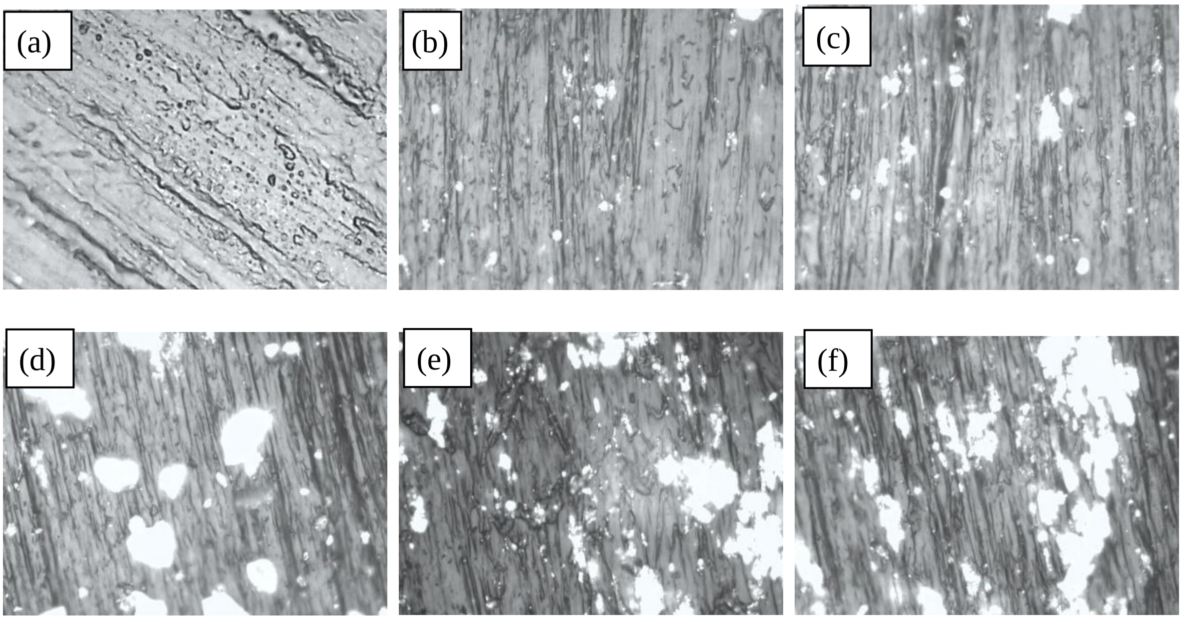

Figure 8 (a-f). Optical micrographs of (a) PET-0, (b) PET-5 (c) PET-10 (d) PET-20 (e) PET-30 (f) PET-40 vol. % BN of the PET/BN composites (Scale bar 100 µm, at 100 X magnification)

4. Conclusions

The PET/BN composites added up to 40 wt.% (30.5 vol.%) BN particles were successfully fabricated by injection molding process are almost defect-free samples, theoretical and experimental densities are identical at lower vol.% and 4% porosity was observed at higher vol. % due to agglomeration of BN particles forming a three-dimensional network structure, as observed by the optical microscopy. Optical microscopy photographs shows uniform dispersion of BN particles at lower vol. % and agglomeration in higher vol. %. XRD confirms the absence of new materials in the composites. i.e., the BN particle presence didn't affect PET morphology. Microhardness was increased by 28% and are close to theoretical hardness. Impact energy and impact strength were increased nearly 70% compared to virgin PET, tensile strength (50 to 77MPa) and flexural strength increased by 22%. Ductility/Elongation of the composite is decreased (115 to 61%) and brittle fracture nature was observed at a higher vol. % of the polymer.

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