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Hybrid Effect of Micro Fillers on the Mechanical Behavior of Polyamide66/Polytetrafluoroethylene Blend

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ABSTRACT

The present investigation thermoplastic blends such as polyamide66 (PA66) and polytetrafluoroethylene (PTFE) in 80/20 weight percentage proportion was selected for the study. Three micro composites were prepared by reinforcing fine particles (micro fillers) such as molybdenum disulfide (MoS_2) PA66/PTFE/MoS_2, PA66/PTFE/MoS_2/silicon carbide (SiC), and alumina (Al_2O_3) PA66/PTFE/MoS_2/SiC/Al_2O_3 of different geometric shapes to form the mixture. These hybrid micro composites were prepared by melt mixing method using twin-screw extruder followed by injection moulding. The studied mechanical properties as per ASTM are a tensile strength, flexural strength, and impact strength including the hardness of the blend micro composites. Results revealed that these hybrid micro fillers decreased the tensile strength and tensile modulus of PA66/PTFE blend composites. However, the incorporation of MoS_2 into the blend increased the flexural strength and the flexural modulus of PA66/PTFE blend appreciably, but the hybrid effect of three micro fillers decreased the flexural strength and the modulus slightly below the value of PA66/PTFE/MoS_2 composites but above the values of PA66/PTFE blends. But, the tensile strain at break gradually decreased after the hybrid fillers addition into the blend. The effect of SiC addition to PA66/PTFE/MoS_2 composites increased the impact strength appreciably, but decreasing trend was observed due to the hybrid effect of three fillers. However, the different shape micro fillers exhibited a synergic effect on the tensile and flexure properties of PA66/PTFE-based composites, respectively. The density of the studied blend increased due to denser nature of micro fillers. The hardness of the blend increased by 3% by the addition of three micro fillers. However, the hybrid effect of micro fillers on PA66/PTFE blend influenced the flexural properties and the hardness than tensile properties. Among these micro composites, PA66/PTFE/MoS_2 showed better mechanical properties. The fractured surfaces are studied by using scanned electron microscopy photographs.

Key words: *Polyamide66/Polytetrafluoroethylene, Hybrid effect, Fillers, Mechanical properties, Blends.*

1. INTRODUCTION

The mechanical industries are always under constant pressure to develop a creative material which is good at both mechanical strength and tribo-performance. Polymer and their composites are finding ever increasing usage for numerous industrial applications such as bearing material, rollers, seals, gears, cams, wheels, and clutches [1]. The use of polymers and polymer-based composites which are having a combination of good mechanical and tribological properties can only prove themselves as worthy. It is often found that such properties are not attainable with a homopolymer. This has led to the development of polymer blends. Polymer blends are mixtures of at least two macromolecular species, polymers, and/or copolymers. Polyamide66 (PA66) is a semi crystalline, thermoplastic commodity polymer that finds widespread use in applications that require considerable strength but low toughness. Polytetrafluoroethylene (PTFE) is a linear polymer with high crystallinity, strong, stiff, and tough engineering material with a lower coefficient of friction. Polymeric composites filled with inorganic fillers are the most important engineering materials today. Incorporating filler and/or fibers to a base polymer material provides substantial improvements in terms of the mechanical properties. Attempts to understand the modifications in the mechanical behavior of the polymers with the addition of fillers

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have been made by many researchers. It was found that incorporation of fillers as reinforcements effectively changes the various properties of thermoplastics. The role of filler deformability, filler – polymer bonding on the flexural strength of polyphenylene sulfide (PPS) was reported by Schwartz and Bahadur [2]. They used Ag_2S, CuS, ZnF_2, and SnS as micro fillers with PPS. They found that the flexural strength of PPS decreased by the addition of these fillers. But, Ag_2S and CuS composites had flexure strengths much higher than those of the ZnF_2 and SnS composites. Ravikumar *et al*. [3] reported the effect of particulate fillers on the mechanical behavior of PA/polypropylene (PP) nanocomposites. They reported that PA66/PP blend showed lower tensile strength and higher strain. But, the addition of particulate clay into the blend decreased the tensile strength and the strain of the composites. The mechanical behavior of PP/wood flour (PP/WF) composites were studied by Zhang *et al*. [4]. They showed that the tensile strength of unmodified PP/WF composites lowered slightly with the addition of WF and elongation at break dropped significantly. However, the flexural strength and the flexural modulus of unmodified PP/WF composites increased greatly than that of PP, respectively. The mechanical properties of PP are modified by adding various mineral fillers. The most studied fillers types being talc and calcium carbonate [5,6]. PP hybrid effects reinforced particulate filler was studied by Hartikainen *et al*. [7]. They studied the mechanical behavior of PP filled with $CaCo_3$. Decrease in the tensile strength and fracture toughness was observed by filling $CaCo_3$ into PP composites. The effect of PTFE filler on the mechanical properties of 80/20 blend of PA6/HDPE blends was studied by Palabiyik and Bahadur [8]. They showed that the addition of 5-10 wt.% of PTFE practically had no effect on the tensile strength of the blends. The effect of PTFE on tensile strength, hardness, and elongation to break is fairly small. Chen *et al*. [9-11] systematically studied the mechanical and tribological properties of PA66/PPS blend filled with PTFE particles. Addition of PTFE particles is beneficial from friction and wear behavior point of view and deteriorated the mechanical properties. The effect of glass powder on some mechanical properties of engineering plastics was studied by Karunanayake [12]. They studied the compatibility of glass powder on four thermoplastics PA6, PA66, poly(butylene terephthalate, and PAA. They found that the polymer PA's has good compatibility. But, the effect of glass filler impaired the improvement of mechanical properties such as tensile strength, impact strength, and thermal expansion. Sun *et al*. [13] studied the mechanical properties of polyoxymethylene modified with nano particles and solid lubricants. They used PTFE, molybdenum disulfide (MoS_2), and nano alumina as fillers for the study. The hybrid effect of these fillers decreased the tensile strength and fracture strain but increased the hardness and the flexural strength. Stuart [14] has recently published the review article on various particulate filled polyblends. Bijwe *et al*. [15] investigated the role of PTFE on mechanical properties of poly(ether-ether-ketone) (PEEK)/PTFE blends. They showed that addition of 30 wt. % of PTFE in the blend has the maximum impact strength, but other properties were impaired. Rong-Guo *et al*. [16] reported the role of PTFE in PA66/PTFE and PA6/PTFE on the mechanical properties. They showed that incorporation of PTFE content in the blend reduced the tensile strength, flexural strength, and the impact strength of the blends. Sreekanth *et al*. [17] explored the role of inorganic fillers such as Mica and Fly ash added to the polyester thermoplastic elastomer. They concluded that there was a significant improvement in flexural strength and the modulus with increase in filler concentration. Alhareb and Ahmad [18] studied the effect of incorporation of alumina (Al_2O_3) and zirconium oxide (ZrO_2) in poly (methyl methacrylate) (PMMA). The addition of these fillers improved the fracture toughness, tensile modulus, and flexural properties of PMMA-based composite materials. Hemanth *et al*. [19] studied the effect of fibers and fillers on thermoplastic composites. They showed that POM-based composites exhibited better tensile strength and flexural strength than thermoplastic copolymers. PTFE is one of the most important and promising material to improve the fracture toughness of polymer-based composites. In spite of the fact that polymer composites are used in such structural applications, no data are reported on the influence of Teflon in PA66 other inorganic particulate fillers *viz*. MoS_2, silicon carbide (SiC), and alumina (Al_2O_3). Keeping this in view, PA66/Teflon blends with ceramic fillers were investigated for tensile, flexure, and impact properties.

PA66 is a semi crystalline, thermoplastic commodity polymer that finds widespread use in applications that require considerable strength but low toughness. It is a widely used engineering thermoplastic. It possesses an outstanding combination of properties such as low density, easy processing, good strength, and solvent resistance.

2. EXPERIMENTAL

2.1. Materials

The materials used in the present investigation are PA66, PTFE, MoS_2, Al_2O_3, and SiC are listed in Table 1. The details of materials and their source are also tabulated in the given table. The material formulations based on the weight percentage are reported with the designation of the materials used in the present study are also reported in Table 2.

2.2. Fabrication of Blends and their Micro Composites

The polymers and fillers were dried at 85°C for 48 h to avoid plasticization, hydrolyzing effects from

Table 1: Data and the source of the materials used in this study.

Material	Designation	Form	Size (µm)	Trade name	Manufacturer	Density (g/cm^3)
Polyamide 66	PA66	Granules	-	Zytel 101L NC010	DuPont Co. Ltd	1.14
Polytetrafluoroethylene	PTFE	Particles	12	Grade MP1000	DuPont Co. Ltd	2.16
Silicon carbide	SiC	Irregular	5-10	-	Carborundum India Ltd.	3.21
Aluminum oxide	Al_2O_3	Particles	5-10	-	Aldrich, Bangalore	3.95
Molybdenum disulfide	MoS_2	Particles	5-10	-	Advanced Engineers, Bangalore	-

Table 2: Formulations of composite blend PA66/PTFE and microcomposites in weight percentage.

Composition	Material ID	Weight percentage				
		PA66	PTFE	MoS_2	SiC	Al_2O_3
PA66/PTFE	1F	80	20	-	-	-
Blend (PA66/PTFE)/MoS_2	2F	80	20	2.5	-	-
Blend (PA66/PTFE)/MoS_2/SiC	3F	80	20	2.5	2.5	-
Blend (PA66/PTFE)/MoS_2/SiC/Al_2O_3	4F	80	20	2.5	2.5	2.5

PTFE=Polytetrafluoroethylene, PA66=Polyamide 66, MoS_2=Molybdenum disulfide, SiC=Silicon carbide, Al_2O_3=Aluminum oxide

humidity and to obtain the sufficient homogeneity. The materials were mixed, and the mixture was extruded using Barbender co-rotating twin-screw extruder (Make: CMEI, Model: 16CME, SPL, chamber size 70 cm^3) (Figure 1). The temperature maintained in five zones of the extruder barrel were Zone 1 (220°C), Zone 2 (235°C), Zone 3 (240°C), Zone 4 (265°C), and Zone 5 (270°C), respectively, and the temperature at the die was set at 75°C. The extruder screw speed was set at 100 rpm which yielded a feed rate of 5 kg h^{-1}. The extrudates obtained was quenched in cold water and then palletized (Figure 2). Before injection moulding, all the pallets were dried at 100°C in a vacuum oven for 24 h. All test specimens were injection molded from the pelletized polyblend material obtained from the co-rotating extruder. The temperature maintained in the two zones of the barrel was Zone 1 (265°C) and Zone 2 (290°C), and mold temperature was maintained at 65°C. The screw speed was set at 10-15 rpm followed by 700-800 bar injection pressure. The injection time, cooling time, and ejection time maintained during injection molding were 10, 35, and 2 s, respectively. All the molded specimens as per ASTM were inspected and tested visually, and those found defects were discarded for the testing.

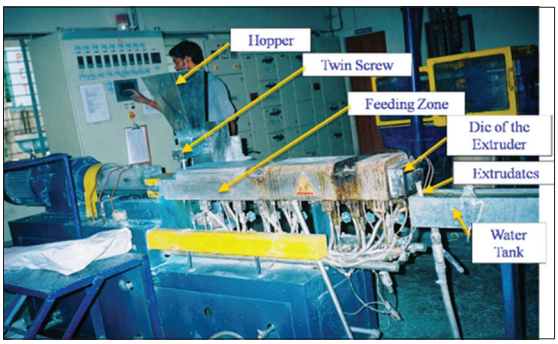

Figure 1: Barbender co- rotating twin -screw extruder.

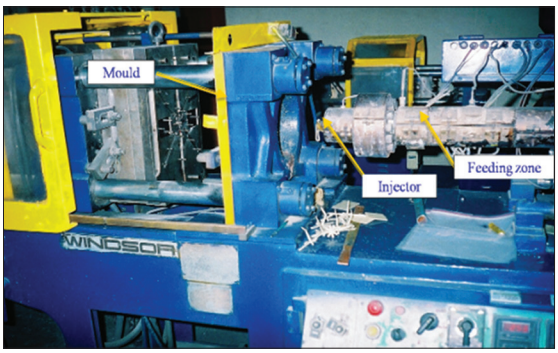

Figure 2: Injection moulding machine.

2.3. Measurement of Mechanical Properties

The mechanical properties such as tensile strength, flexural strength, impact strength along with density and hardness of the blends were measured as per ASTM. The tensile strength and the tensile elongation at break were measured using the Universal testing machine (JJ Lloyd, London, United Kingdom, capacity 1-20KN) in accordance with ASTM D638. Tests were performed at a constant strain rate of 5 mm/min. ASTM D638 Type 1 standard dimensions are used. Flexural strength or three point bending were carried out on the same machine by changing the jaws of the setup, and the specimen acts as simply supported beam subjected to point load at the middle. The flexural strength and flexural modulus were determined at the rate of 2 mm/min as per ASTM D790. The standard specimen dimensions for the flexural strength is 125 mm × 12.7 mm × 3.2 mm. The

Izod impact strength was determined using ASTM D256 by using Izod impact testing machine at the striking rate of 3.2 mm/s. The densities of the blend composites were determined as per ASTM D756. The ASTM standards for these mechanical testing is shown in Figure 3. All these tests were conducted at the room temperature. Minimum of three samples was tested for the data representation. On the other hand, the density and the hardness (Shore D) of the blended composites were determined as per ASTM D792 and ASTM D224, respectively.

3. RESULTS AND DISCUSSIONS

3.1. Hybrid Effect of Micro-Fillers on the Density and Hardness of the Blend PA66/PTFE

The addition of MoS_2 into 80/20 wt. % PA66/PTFE blend increased the density of the blend (Figure 4a). Further inclusion of fine particulates of SiC, Al_2O_3 into the blend, increased the density of the blend linearly. Addition of fine particles of SiC, Al_2O_3, and MoS_2 into the blend, improved the density of PA66/PTFE blends. This can be attributed to dense nature of MoS_2 particulates and hard fine particles. Therefore, the hybrid effect of the micro fillers on the density PA66/PTFE blend has increased the density of the microcomposites. The influence of PTFE in PEEK-PTFE blend increased the density of the blend [15]. The effect of micro fillers on the hardness of the blend is reported in Figure 4b. The hardness of PA66/PTFE blend increased after the fillers addition.

By loading 2.5 wt. % of MoS_2 into the blend, the hardness was improved by 3%. Further addition of the fillers SiC and Al_2O_3 into MoS_2 filled blend did not show much improvement in the hardness of the blend. The increase in hardness is due to the hard nature of hybrid fillers. MoS_2 and SiC are the two major constituents among the fillers responsible to improve the hardness of the neat blend. However, an appreciable change in the hardness value was not observed in the entire blend based composites by the influence of fillers [13]. Furthermore, the degree of transformation of the material phase from ductile to brittle was not appreciable by the hybrid effect of micro fillers.

3.2. Combination Effect of Micro Fillers on Tensile Behavior of PA66/PTFE

The tensile behavior of micro filled PA66/PTFE blend composites are reported in Figure 5a and b. The tensile strength of pure PA66/PTFE blend was 66.5 N/mm^2. After the addition of 2.5 wt. % of MoS_2 into the blend, it was 62.5 which is 6% decrease. Further inclusion of 2.5 wt. % SiC into PA66/PTFE/MoS_2 composites decreased the tensile strength of the composites by 1.6% and 8% against the blend. This shows that the bi filler addition into the blend decreased the tensile strength of the material by very little extent (2%). Furthermore, loading of associate filler into the filled composite did not respond to the brittle nature of the material. This modified effect of the properties may be due to the influence of synergic effect between the fillers and the plastics. The effect of adding fine particles of Al_2O_3 into the composite PA66/PTFE/MoS_2/SiC further decreased tensile strength. It was 49.5 N/mm^2.

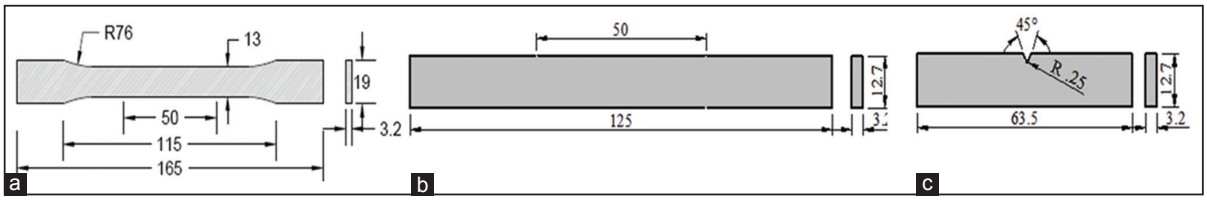

Figure 3: Specimen standards: (a) ASTM D638 (tensile test) (b) ASTM D790 (flexure test), and (c) ASTM D256 (impact test).

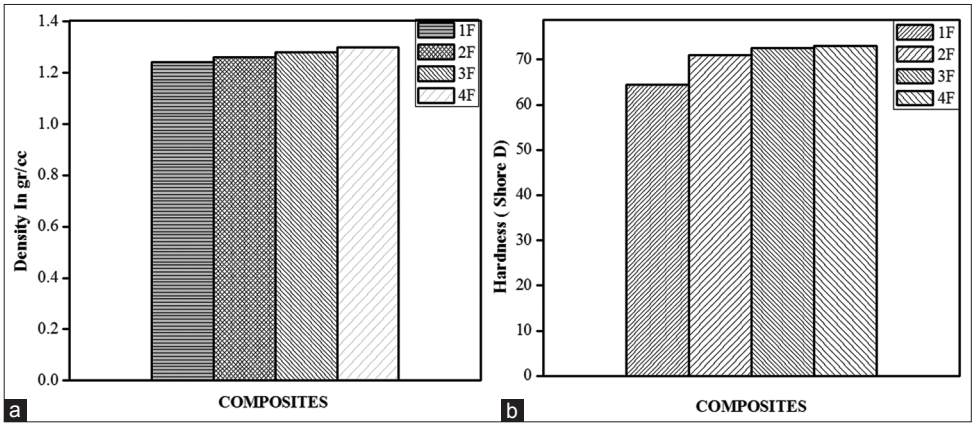

Figure 4: Variation of properties of polyamide 66/polytetrafluoroethylene and their micro composites: (a) Density and (b) hardness.

This is 25% decrease in strength against pure blend PA66/PTFE. The hybrid hard fillers did not have the much compatibility with the thermoplastics to obtain the good results in terms of strength. This is due to the introduction of voids and discontinuities in the blend due to the filler addition. The effect was to create stress risers to concentrate the stress at the point which could lead the material to influence the crack growth. In addition, less amount of strength is decreased after the addition of MoS_2. This can be attributed purely to the strength properties of MoS_2. It can act as strength and tribo carrier during the performance of the material. The same observations were made after SiC inclusion into the composites. But, slightly greater amount of deterioration in strength was noticed after alumina as filler into PA66/PTFE/SiC/MoS_2 composites. This is due to the refractory nature of the alumina.

3.3. Influence of Rigid Fillers on the Flexural Strength of PA66/PTFE Blend

The flexural behavior of PA66/PTFE blend against the effect of hard fillers is shown in Figure 6. The flexural strength of pure PA66/PTFE blend was 93 N/mm^2. After the addition of MoS_2 into the blend, it was 103 which is 11% increase. Further inclusion of SiC into PA66/PTFE/MoS_2 decreased the tensile strength of the composites by 2% against the filled blend, 7% increase against neat blend. The sole modification of the flexural behavior of the blend is mainly attributed to the synergistic effect of the hard fillers and ductile nature of the blend based composites. But, after adding fine particles of Al_2O_3 into the composite PA66/PTFE/MoS_2/SiC, further decrease in flexural strength was observed. It was 93 N/mm^2. This is almost equal to the strength of neat blend. This shows the interaction of fillers and the compatibility with the associates of the blends was uniform and equally contributable. Increase in flexural strength after adding MoS_2 into the neat blend can be attributed to the strength and malleable nature of MoS_2.

Decrease in flexural strength of composites was observed after adding SiC into the MoS_2 filled PA66/PTFE micro composites. This is due to the brittle and the hard nature of SiC. But still, the composites maintained the strength above the value of neat blend. The effect of hybrid fillers after incorporating, alumina into the filled

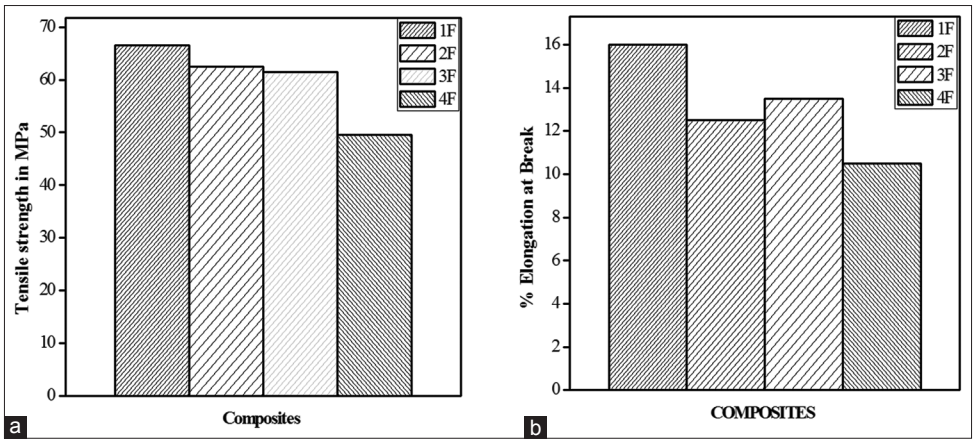

Figure 5: Variation of properties of polyamide 66/polytetrafluoroethylene (PTFE) and poly (methyl methacrylate)/PTFE micro composites: (a) Tensile strength and (b) strain at break.

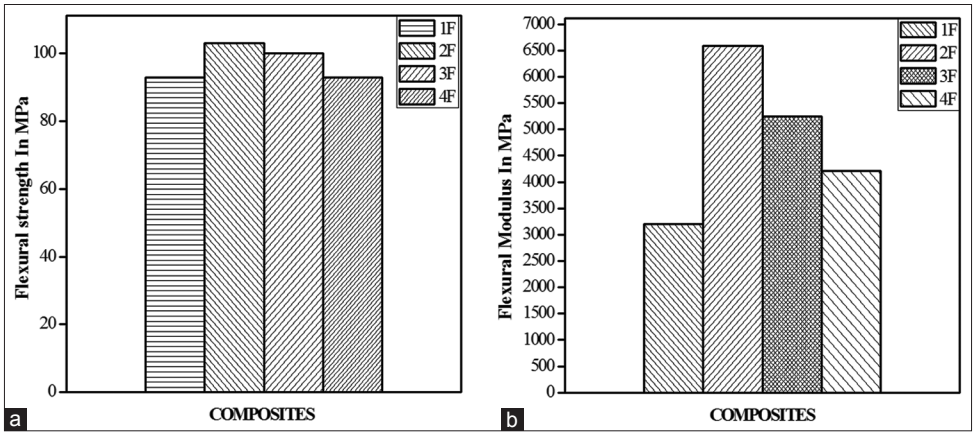

Figure 6: Variation of properties of polyamide 66/polytetrafluoroethylene and their micro composites: (a) Flexural strength (b) flexural modulus.

composite appreciably impaired the flexural strength (Figure 6b). The hybrid effect on the flexural modulus of PA66/PTFE tends to have its value to 4210 N/mm^2; it was around 36% decrease against PA66/PTFE/MoS_2, 19% against PA66/PTFE/MoS_2/SiC composites but increase of 31% against the pure blend PA66/PTFE. However, the individual and the hybrid effect of the filler improved the flexural strength and the modulus of the neat blend PA66/PTFE. The flexural strength of the composites were in the order of PA66/PTFE/MoS_2 > PA66/PTFE/MoS_2/SiC > PA66/PTFE/MoS_2/SiC/Al_2O_3 < PA66/PTFE. This is in good agreement with the studies done by Hui *et al*. [13].

3.4. Impact Strength of Micro Filled PA66/PTFE Blend Composites

The impact strength of neat blend PA66/PTFE was 54 J/m. But, after the inclusion of MoS_2 as filler, the impact strength of the composite was reduced by 2%. After adding SiC into the MoS_2 filled composites, slight increase in impact strength was observed, this may be attributed to the ductile nature of PA66/PTFE/MoS_2, which absorbs more impact energy to break and had more flexural modulus. However, the hybrid effect of fine particles on the blend impaired the impact strength of PA66/PTFE blend (Figure 7). It was around 7% decrease. This may due to the synergic effect of micro fillers and also the brittle nature of the composites by adding fine particulates into the blend. The hybrid effect of fillers on the hardness of the composites is one of the major effects to decrease the impact strength of the composite. However, PA66/PTFE/MoS_2/SiC composites had better impact strength among studied composites.

3.5. Study on Fractured Surfaces of the Filled Micro Composites Using Scanned Electron Microscopy (SEM)

The fractured surfaces during the test as per ASTM are studied and reported in the following section by using SEM micrographs. Figure 8a shows the SEM of the surfaces of the pure blend subjected to tension load. From the SEM, it was clear that phase morphology of PA66 and PTFE was heterogeneous. The interfacial tension due to plastic deformation was severe which had led to the impairment of the mechanical properties. PTFE is filled with good bonding with PA66. The compatibility of the blend was well witnessed by the continuous phase deformation which was shown in Figure 8a. Addition of MoS_2 into the blend influenced the brittle nature of the material. The introduction of this filler into the blend introduced the concept of stress raisers by creating voids in the blend. During tension, the interfacial force of attraction between the fillers and the plastics would be very weak and supported the mechanism of formation of voids, which was very visual in the SEM graphs of Figure 8b. The synergistic force between the fillers and the plastics found to be very less due to the introduction of the hard fillers such as SiC. The filler interfacial reaction found to be heterogeneous because of more fillers lead the material to become brittle rather than the ductile which was well witnessed by the SEM graph (Figure 8c). The fillers seem to be embedded in the matrix of the blend. Figure 8d showed the compatibility of the fillers with that of the blend. On the other hand, the severe deformation due to brittle nature of the composites supported the cracks growth which tends the material to become irregular.

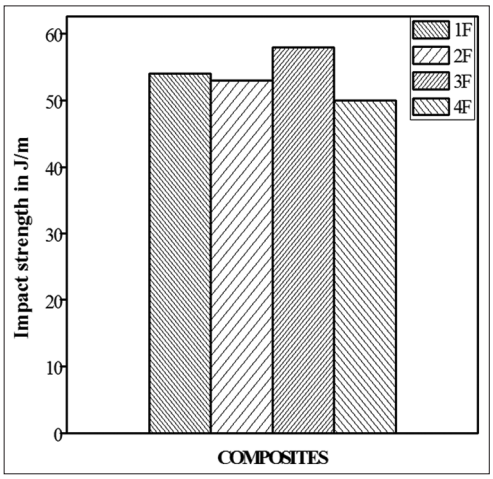

Figure 7: Impact strength behavior of polyamide 66/polytetrafluoroethylene and their micro composites.

4. CONCLUSIONS

- The hybrid effect of micro fillers MoS_2, SiC, and Al_2O_3 on the mechanical properties of PA66/PTFE blend are appreciable
- The tensile strength and strain of PA66/PTFE blend were impaired due to the hybrid effect of micro fillers
- The flexural strength and the modulus of PA66/PTFE blend were effectively improved due to the hybrid effect of micro fillers
- Increase in hardness and density of PA66/PTFE blend noticed due to the hybrid effect of fillers
- There was a moderate effect on the impact strength of PA66/PTFE due to hybrid fillers. PA66/PTFE/MoS_2/SiC composites had better impact strength among studied composites
- Composite PA66/PTFE/MoS_2 showed better mechanical properties among the studied one
- The SEM pictures revealed the synergistic effect of the hybrid fillers and their compatibility with the thermoplastic matrix.

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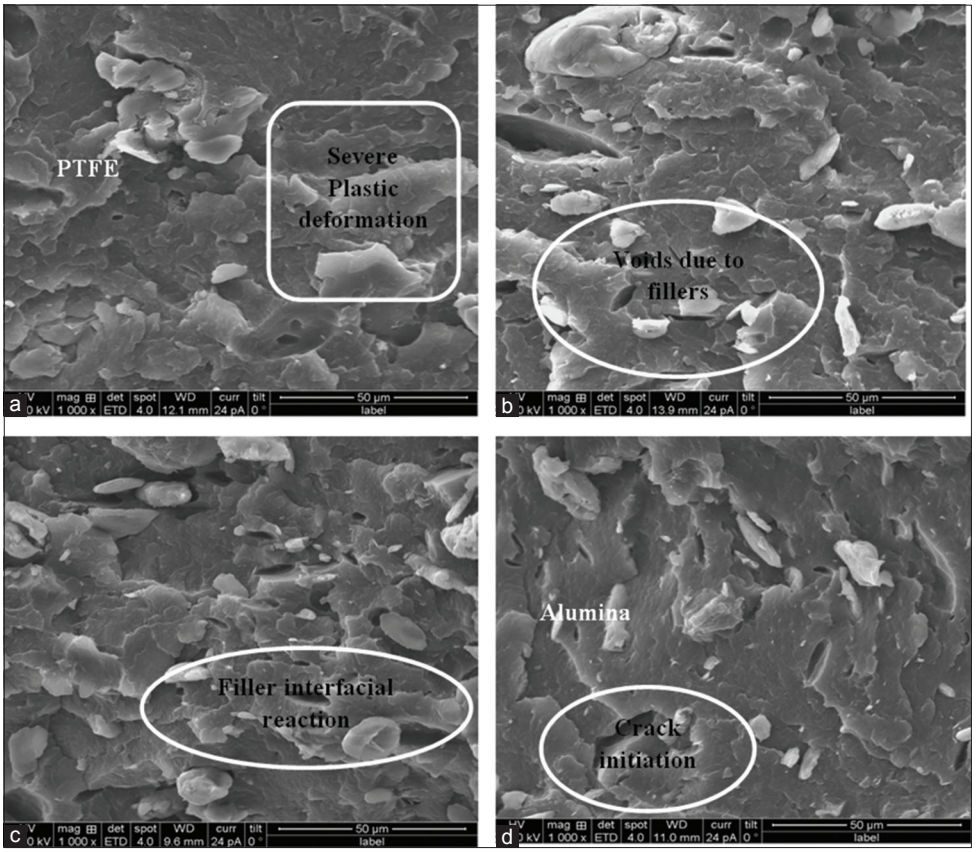

Figure 8: Scanning electron microscopy photographs of different composites (a) 80/20 wt.% polyamide 66 (PA66)/polytetrafluoroethylene (PTFE) blend, (b) blend (PA66/PTFE)/2.5% molybdenum disulfi de (MoS2), (b) blend (PA66/PTFE)/2.5% MoS2/silicon carbide (SiC) and (c) blend(PA66/ PTFE)/2.5%MoS2/2.5% SiC and (d) PA66/PTFE/2.5% MoS2/2.5% SiC/2.5% aluminum oxide.

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