Characterization of Mechanical and Tribological Behavior of (Nylon 6 + Graphite + Teflon) Nano Particulate Composite: Application Perspective

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Abstract— At higher content of graphite the strength of composite has been found reduced because of its highly porous structure and high surface area. The flexural strength has been observed to be sensitive to flaws or defects. An increase in flexural strength on account of increasing teflon has been attributed to its dimensional stability. The introduction of graphite has increased the storage modulus of the composites. The anisotropic nature of graphite was the result of the two types of bonding actions in different crystallographic directions. The ability of graphite to form a solid film lubricant has been attributed to these two contrasting chemical bonds. Two products namely sleeve bearing and thrust washer were fabricated from the optimum values of control parameters estimated by the design of experiments using Taguchi's technique. The properties of the nylon 6/teflon/graphite composites have been optimized by the confirmations tests.

Index Terms— Nylon 6, graphite, teflon, Wear, mechanical, tribological, DMA, sleeve bearing, thrust washer.

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1 INTRODUCTION

NYLONS are favored materials for gears, cams, bearing applications. The wear behavior of nylons is adequate for dry sliding conditions and using lubrication at higher speeds. When nano-solid lubricants are added to a polymer matrix to form a composite, they play an important role in enhancing tribological characteristics of nylons. The inclusion of the ceramic nano-solid lubricants into the more flexible and lower thermal resistance polymer can significantly increase its stiffness and thermal stability [1, 2]. The solid lubricants reduce friction coefficient and wear rate through the decrease of adhesion with a counterface or by creating a transfer film with low shear strength at the interface [3].

performance of PTFE, and the wear resistance increased 600 times than that of unfilled PTFE at a loading of 20wt%. Graphite and molybdenum disulfide (MoS_2) have been successfully used with PTFE to reduce the friction coefficient and the wear rate [6-8]. Wang *et al.* [9] have studied the composites made of poly-etheretherketone (PEEK) with various weight fractions of SiC, Si_3N_4, SiO_2, and ZrO_2. They found an improvement in the wear resistance and reduction in the friction coefficient with the addition of the filler in fractions less than 10% by weight. Cho and Bahadur [10] reported that the addition of 2vol% nano-CuO could enhance the wear resistance of short fiber-reinforced polyphenylene sulfide.
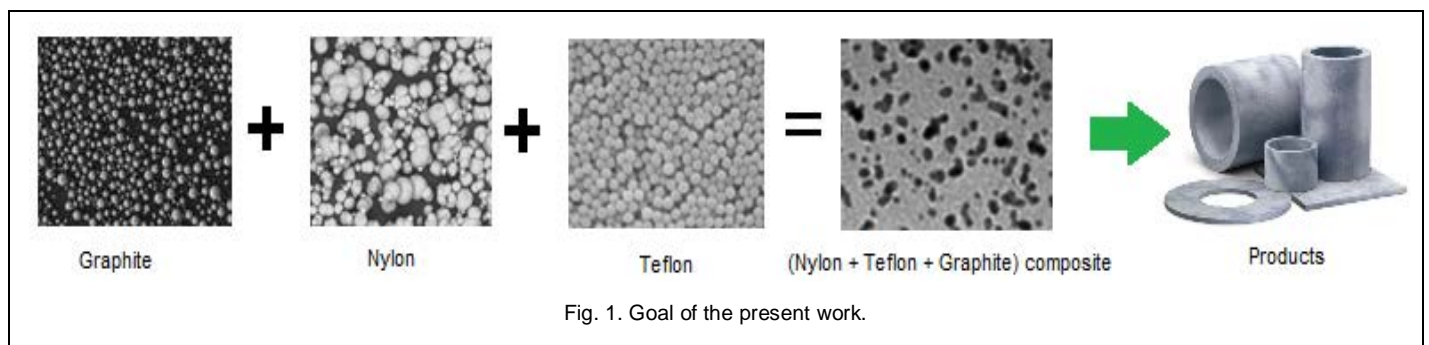

Fig. 1. Goal of the present work.

Polytetrafluoroethylene (PTFE) is an excellent solid lubricant and used commonly in bearing and seals applications. PTFE exhibits high wear rate under normal friction conditions limiting its application fields. Li *et al.* [4] prepared PTFE/ZnO nanocomposites. The wear resistance was doubled up with a maximum wear resistance at ZnO concentrations of 15vol%. Sawyer *et al.* [5] used 38 nm Al_2O_3 filler to improve the wear

Zhang et al. [11] studied the effect of nano-TiO_2 on short fiber-reinforced epoxy under different loading conditions. They found that the addition of 5vol% nano-TiO_2 could significantly reduce the friction coefficient and the wear rate of epoxy composites than filling only with traditional fillers. Avella et al. [12] filled polymethylmethacrylate (PMMA) with nano-scale $CaCO_3$. The abrasion resistance increased as the filler content was increased, improving by a factor of 2% with 3% $CaCO_3$ by weight. Nucleation and growth of ZnS nano-particles were achieved in a poly (diallyl dimethylammonium chloride) (PDDA)-poly (acrylic acid) (PAA) film prepared by the layer-by-layer deposition technique [13]. It was found that

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ZnS nano-particles within PEMs possess load-carrying capacity and enhance antiwear life.

Nylon 6 finds application in a broad range of products requiring materials of high strength. It is widely used for gears, fittings, and bearings. The present experimental study involves the testing of Nylon 6 blended with graphite and teflon varying the percentage by weight. The wear, friction, impact strength, flexural and tensile strengths properties and dynamic mechanical analysis are tested. The nylon- graphite-teflon composites are used to fabricate sleeve bearing and thrust washer form optimum values of control parameters evaluated by the Taguchi techniques (figure 1).

2 MATERIALS AND METHODS

Nylon 6 is used in applications where toughness, lubricity, and wear are important, such as gear wheels. The Melting temperature of nylon 6 is 220°C and crystalline density at 25°C is 1.23 g/cm^3. The filler materials were graphite and teflon. Control parameters are those parameters that a manufacturer can control the design of the product, and the design of process. The levels chosen for the control parameters were in the operational range of the polymer matrix composites process. Each of the three control parameters was studied at three levels. The chosen control parameters are summarized in table 1.

TABLE 1
PARAMETERS AND LEVELS

Factor	Symbol	Level–1	Level–2	Level–3
Graphite, %Wt	G	5	10	15
Teflon, %Wt	T	5	10	15
Particle size of graphite, nm	P	55	255	475

The orthogonal array (OA), L$_9$ was selected for the present work. The parameters were assigned to the various columns of O.A. The assignment of parameters along with the OA matrix is given in Table 2. One interaction among graphite and teflon of filler (GxT) was also considered.

TABLE 2
Orthogonal Array (L9) and control parameters

Treat No.	A	B	P	AxB
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

2.1 Preparation of nylon-graphite-teflon composites

Different matrix/filler blends were prepared in a torque rheometer with a twin-rotary mixer as shown figure 2. Prior to the melt processing, nylon 6, teflon, and graphite were dried in the oven at 75^{0}C for 4 hr. Graphite powder were premixed with teflon powder in tumbling mixer for 15 min at 50 rpm. After nylon 6 was melted at 220°C for 5 min at 75 rpm, the mixture of graphite and teflon was added in 3 min at the same temperature and at 50 rpm. The blend was kept at 75 rpm for another 5 min. The extruded sample was palletized and stored in sealed packs containing desiccant. The test specimens for tensile tests, impact test, and water test were prepared using an injection molding machine with 50 ton clamping pressure at 220-230^{0}C and an injection pressure of 100 bars. After molding, the test specimens were conditioned at $25 \pm 2^{\circ}$C and 50 ± 5% RH for 40 h according to ASTM D 618 prior to testing.

Fig. 2. Torque rheometer with a twin-rotary mixer

2.2 Conduction of Tests

The following tests were conducted on the metal matrix composites:

- Tensile test for elastic modulus and ultimate tensile strength
- Three point flexural test
- Impact test
- Wear test
- Estimation of friction coefficient
- Dynamic Mechanical Analysis (DMA) test for storage and loss moduli.

The samples were machined to get dog-bone specimen for tensile test. The computer-interfaced UTM (Universal Testing Machine) was used for the tensile test. The loads at which the specimen has reached the yield point and broken were noted down. The extensometer was used to measure the elongation.

The load v/s deflection graph was also obtained for each specimen from the computer attached to the machine. The three-point flexural test was conducted on UTM. Two specimens were used for each trial. Impact (Izod) test was carried out to find impact strength in Joules on a standard pendulum-type impact machine.

The wear test was carried out on a pin-on-disc machine with the pin held stationary against a rotating steel disc [14]. The frictional force due to load was measured with a load cell. The cylindrical specimens for the wear test of different compositions were injection molded from the prepared blends. The specimens are 10mm diameter and 50 mm long held in the chuck perpendicular to the disc surface. The experiments were carried out at a nominal speed of 0.5 m/s under a constant load of 1000 N for a period of 30 minutes. The changes in the lengths of the specimens were recorded using LVDT to calculate the volumetric wear rate. Also, the loss of weight due to wear for each composition was recorded.

The viscoelastic property of a polymer was studied by dynamic mechanical analysis where a sinusoidal force (stress σ) was applied to a material and the resulting displacement (strain) is measured. Viscoelastic polymers have the characteristics in between where some phase lag will occur during DMA tests. The storage modulus measures the stored energy, representing the elastic portion, and the loss modulus measures the energy dissipated as heat, representing the viscous portion. The tensile storage and loss moduli are defined as follows:

- Storage Modulus: $E' = (\sigma_0/\varepsilon_0)\cos\delta$ (1)

- Loss Modulus: $E'' = (\sigma_0/\varepsilon_0)\sin\delta$ (2)

- Phase Angle: $\delta = \arctan(E''/E')$ (3)

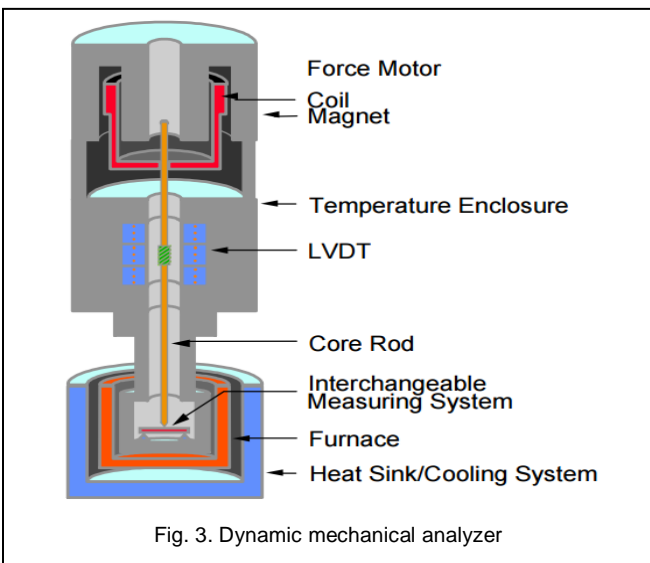where δ is phase lag between stress (σ_0) and strain (ε_0).

Fig. 3. Dynamic mechanical analyzer

the sample. This was applied using an electric motor moving linearly (up and down), and contained a frequency (speed of oscillation), and contained a force (energy input into sample). Resulting strain (displacement) was measured using LVDT. The size of the test specimens were of 56 x 13 x 3 mm.

3 RESULTS AND DISCUSSION

The experiments were scheduled on random basis to accommodate the manufacturing impacts (like mixing of ingredients and variation of temperature, density and particle size). Two trials were carried out for each experiment.
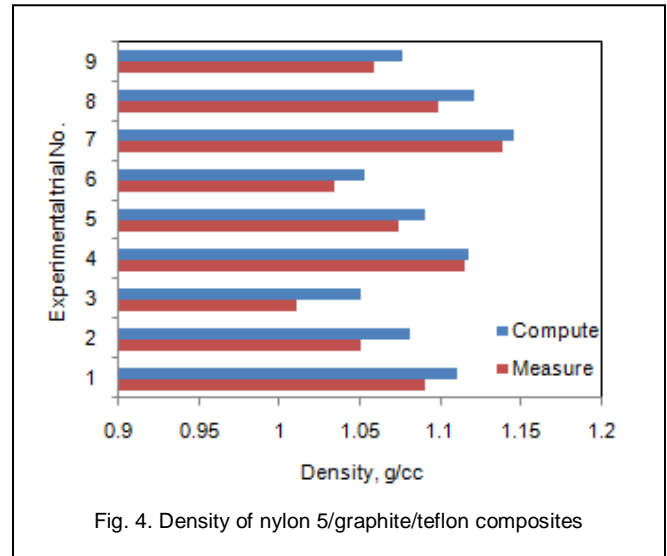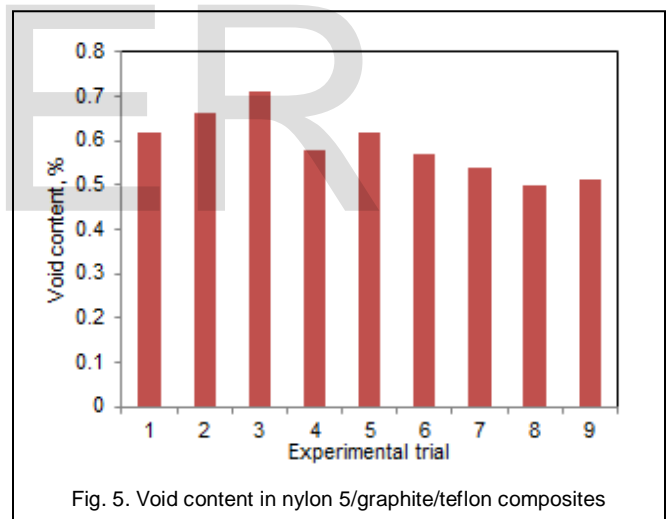

Fig. 4. Density of nylon 5/graphite/teflon composites

Fig. 5. Void content in nylon 5/graphite/teflon composites

3.1 Density and voids

The density depends on the composition of the nylon 6/graphite/teflon polymer composite. The calculated density values of the nylon 6/graphite/teflon polymer composites were higher than the measured values as noticed from figure 4. This might be due to the formation of voids in the composites. The density of composites increases with increases in the content of graphite. The void content increases with increase in the particle size and the teflon content in the composite as shown in figure 5.

3.2 Mechanical Behavior

Table – 3 gives the ANOVA (analysis of variation) summary of raw data. The Fisher's test column establishes all the parameters (A, B, and P accepted at 90% confidence level. The percent

contribution indicates that the composite parameter, G (%wt graphite) contributes 35.2% of variation, T (%wt teflon) aids 30.47% of variation, P (particle size) influences 24.21% of variation. The error is owing to formation of voids in the composite. The ANOVA summary of the elastic modulus is given in Table 4. The Fisher's test column ascertains all the parameters (G, T, P, GxT) accepted at 90% confidence level influencing the variation in the elastic modulus. The parameters, G and P contribute 17.24% of variation each, the parameter, T aids 39.36% of variation, and the interaction between parameters GxT is significant over the variation in the elastic modulus of composites.

TABLE 3
ANOVA summary of the tensile strength

| Source	Sum 1	Sum 2	Sum 3	SS	v	V	F	P
G	395	445	425	211.12	2	105.56	42.22	35.2
T	448	414	403	183.45	2	91.72	36.69	30.47
P	438	429	398	146.78	2	73.39	29.36	24.21
GxT	416	417	432	26.78	4	6.69	2.68	2.87
Error				17.48	7	2.5	1.00	7.25
T				585.61	17			100

Note: SS is the sum of square, v is the degrees of freedom, V is the variance, F is the Fisher's ratio, P is the percentage of contribution and T is the sum squares due to total variation.

TABLE 4
ANOVA summary of the elastic modulus

Source	Sum 1	Sum 2	Sum 3	SS	v	V	F	P
G	17.12	17.7	17.03	0.05	2	0.02	12.42	17.24
T	17.9	17.2	16.75	0.11	2	0.05	31.06	39.36
P	17.6	17.4	16.85	0.05	2	0.02	12.42	17.24
GxT	16.96	17.14	17.75	0.05	4	0.01	6.21	16.06
Error				0.0113	7	0.0016	1	10.1
T				0.2713	17			100

The effect of teflon and graphite addition on the tensile strength and elastic modulus is shown figure 6. The tensile strength of composites increases from 65.83 to 74.17 MPa with increasing graphite content from 5 to 10%. The elastic modulus of composites increases from 2.85 to 2.95 GPa with increasing graphite content from 5 to 10%. Still, increasing of filler content from10 to 15%, the tensile strength and elastic modulus decrease to 70.83 MPa and 2.83 GPa respectively. The elastic moduli of nylon 6, graphite and teflon are respectively 2.76, 17.5 and 0.50 GPa. At higher content of graphite, the reduction of strength is owing to its highly porous structure and high surface area, low content of polymeric matrix is insufficient to infiltrate in graphite. There is an interaction between teflon and graphite as shown in figure 7. The interaction effect leads infiltration of teflon into graphite. The tensile strength and

elastic modulus decrease with increase in the content of teflon as shown in figure 6. It may follow due to the poor interface bonding between the nylon 6 and teflon resulting in poor adhesion of particles and the softness of teflon filler. The detrimental mechanical properties of HDFE-graphite composites are described on the basis of aggregation theory [15].

Fig. 6. Effect of graphite/teflon on (a) tensile strength and (b) elastic modulus
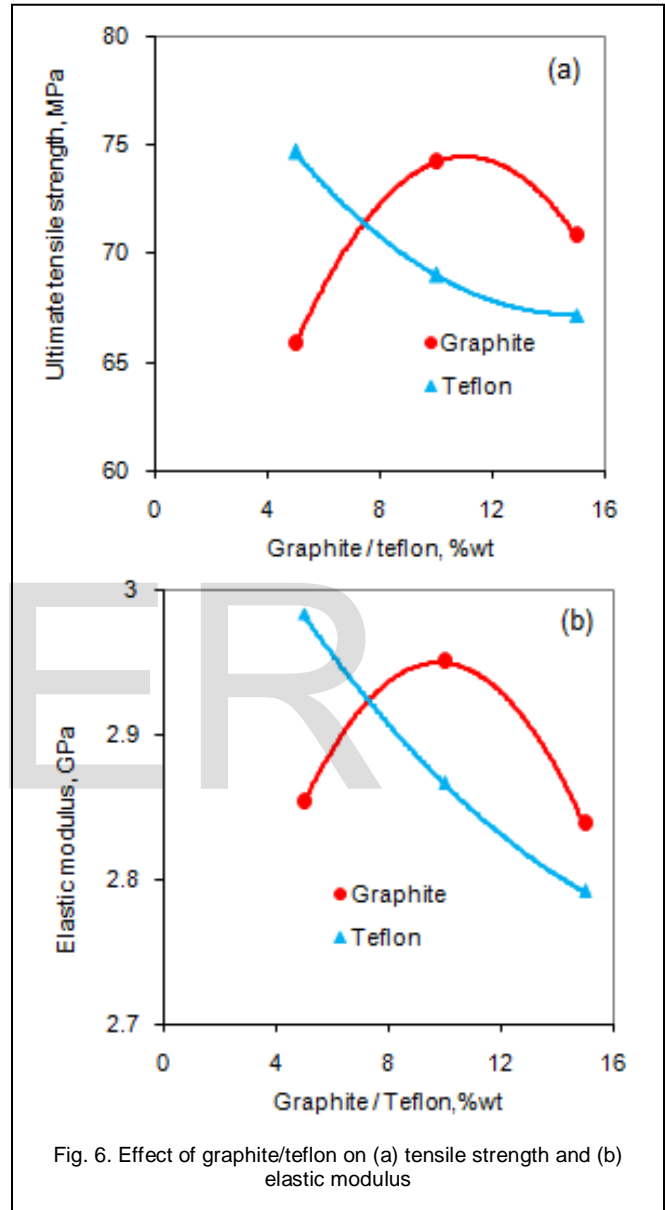
The tensile strength and elastic modulus decrease with increase in the particle size of filler as shown in figure 8. Hoshino *et al.* [16] discussed the effects of size and shape of silica particle on the strength and fracture toughness based on particle-matrix adhesion and they found an increase of the flexural and tensile strength as specific surface area of particles increased. The specific surface area is higher for smaller particles [17].
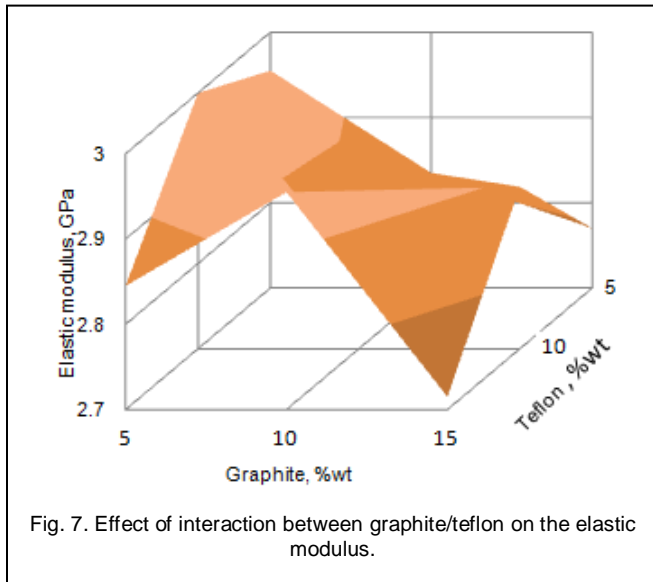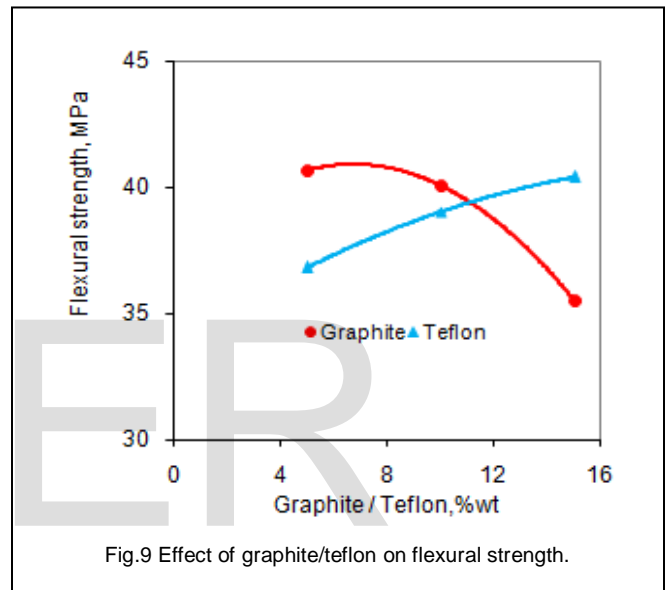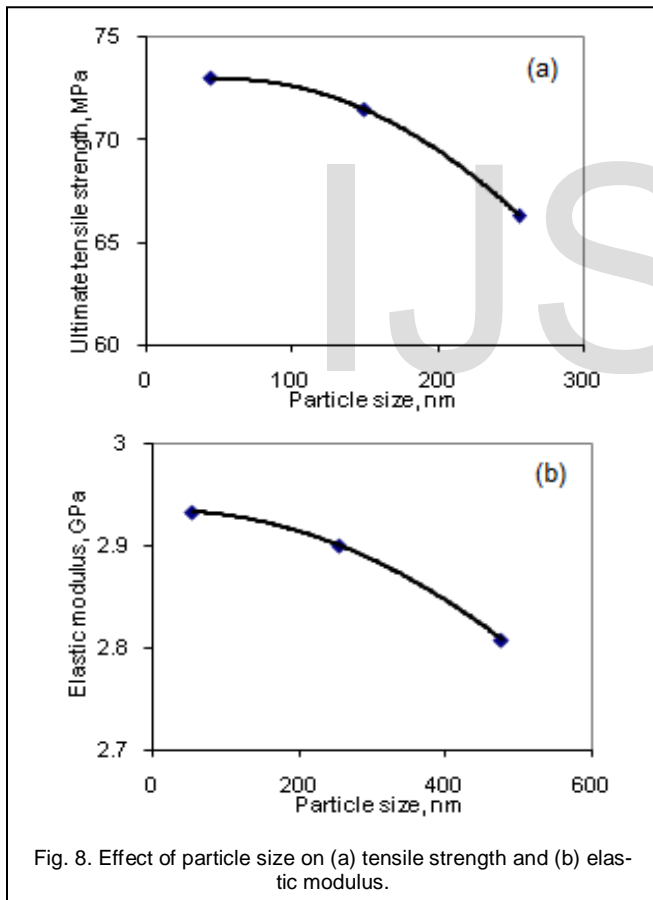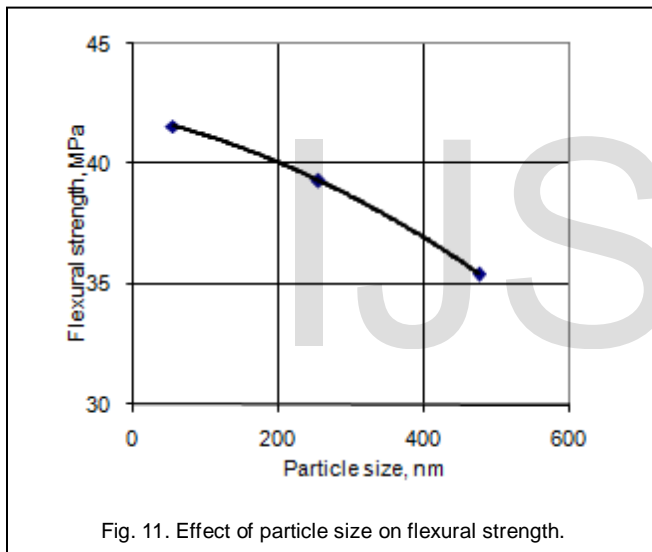
Fig. 7. Effect of interaction between graphite/teflon on the elastic modulus.

TABLE 5
ANOVA summary of the flexural strength

Source	Sum 1	Sum 2	Sum 3	SS	v	V	F	P
G	244.08	240.51	213.23	94.92	2	47.46	42.378	29.89
T	220.99	234.32	242.51	39.33	2	19.67	17.56	11.96
P	249.48	235.77	212.57	116.03	2	58.01	51.80	36.7
GxT	225.46	247.02	225.34	51.94	4	12.98	11.59	15.31
Error				7.814	7	1.12	1	6.14
T				310.034	17			100

Fig. 8. Effect of particle size on (a) tensile strength and (b) elastic modulus.

Fig.9 Effect of graphite/teflon on flexural strength.

The ANOVA summary of the flexural strength is given in table 5. The Fisher's test column ascertains all the parameters (G, T, P, and GXT) accepted at 90% confidence level influencing the variation in the flexural strength. The percent contribution indicates that G (%wt graphite) gives 29.89% of variation, T (%wt teflon) supports 11.96% of variation, P (particle size) controls 36.70% of variation, and the interaction between parameters G and T contributes 15.31% of variation.

The effect of teflon and graphite addition on the flexural strength is shown figure 9. The flexural strength of composites decreases from 40.68 to 35.54 MPa with increasing graphite content from 5 to 15%. The flexural strength of composites increases from 36.83 to 40.42 MPa with increasing teflon content from 5 to 15%. At higher content of graphite, the reduction of strength is owing to its highly porous structure. As the graphite is a brittle material, the flexural strength is sensitive to flaws or defects. If a flaw is present within the span of the flexural test specimen, then the load required to break the specimen is reduced. When failure occurs, it is catastrophic. An increase in flexural strength on account of increasing teflon is due to its relative stiffness supporting dimensional stability. There is an interaction between teflon and graphite as shown in figure 10. The interaction of 5% graphite and 10% teflon results in the flexural strength of 43.895 MPa. The flexural strength decreases with increasing particle size (figure 11). The attribution is same as the case of tensile strength.

Fig. 10. Effect of interaction between graphite/teflon on flexural strength.

Fig. 11. Effect of particle size on flexural strength.

TABLE 6

ANOVA summary of the impact strength

Source	Sum 1	Sum 2	Sum 3	SS	v	V	F	P
G	2.32	1.70	1.30	0.06	2	0.03	41.18	68.79
T	2.13	1.68	1.51	0.02	2	0.01	13.73	21.79
P	1.58	1.72	2.02	0.00	2	0	0.00	-1.71
GxT	1.75	1.82	1.75	0.00	4	0	0.00	-3.42
Error				0.01	7	0.00073	1.00	14.55
T				0.09	17			100.00

The ANOVA summary of impact strength is given in table 6. The Fisher's test column ascertains the parameters (G, T, and P) accepted at 90% confidence level influencing the variation in the impact strength. The percent contribution indicates that the composite parameter, G (graphite) contributes 68.79% of variation, parameter and T (teflon) aids 21.79% of variation. The effect of particle size and the interaction between parameters G and T contribute negligible variation.

Fig. 12. Effect of interaction between graphite/teflon on impact strength.
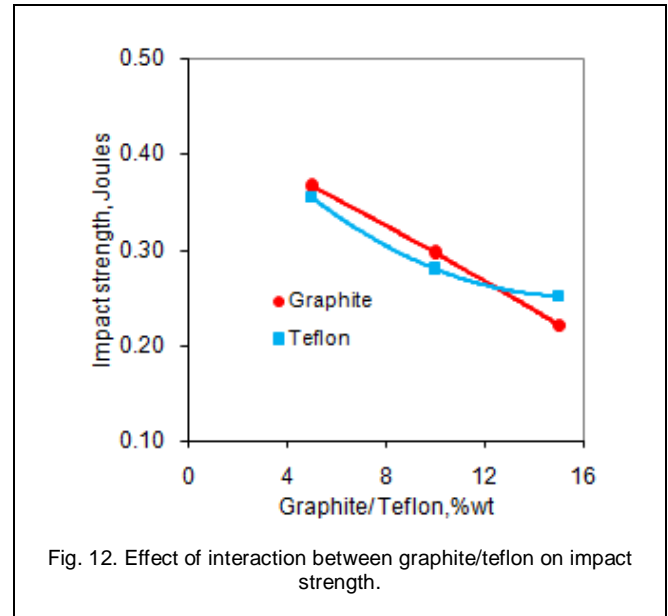
The effect of teflon and graphite addition on the impact strength is shown figure 12. The impact strength decreases with an increase in the content of both the Teflon and graphite. It is the ability of the material to resist the fracture under stress applied at high speed. The specimens are deformed within a short time and therefore exposed to high strain rates. The reduction of impact of strength is owing to the increase of hardness with the addition of graphite content in the composite.

3.3 Dynamic Mechanical Analysis (DMA)

The dynamic modulus indicates the inherent stiffness of material under dynamic loading conditions. The storage modulus E' represents the stiffness of a viscoelastic material and is proportional to the energy stored during a loading cycle. The loss modulus E'' is defined as being proportional to the energy dissipated during one loading cycle. The loss factor $\tan \delta$ is the ratio of loss modulus to storage modulus [1]. It is a measure of the energy lost, expressed in terms of the recoverable energy, and represents mechanical damping or internal friction in a viscoelastic system. The loss factor $\tan \delta$ is expressed as a dimensionless number. A high $\tan \delta$ value is indicative of a material that has a high, nonelastic strain component, while a low value indicates one that is more elastic. The dynamic mechanical properties of polymers are usually studied over a wide temperature range. In the region where the dynamic modulus-temperature curve has an inflection point, $\tan \delta$ curve goes through a maximum. This dissipation is called Tg region. The effect of temperature on the storage modulus is shown in figure 14 for various trials of the design of experiments. The dynamic mechanical analysis has been studied to probe the temperature dependence of storage modulus upon blending with nylon/teflon/graphite.

For all the trails of experimentation, the storage modulus can be seen increasing with temperature. The storage modulus is high for the trial no.7 (15% graphite + 5% teflon) and low for the trial no. 6 (10% graphite + 15% teflon). This indicates that the introduction of graphite increases the storage modulus of the composites. The effect of temperature on the loss modulus is shown in figure 15. The storage modulus is low for the trial no.7 (15% graphite + 5% teflon) and high for the trial no. 6 (10% graphite + 15% teflon). A noticeable reduction in Tg with an increase in temperature is observed from figure 16. This trend is same as that of loss modulus.

Fig. 13. Effect of temperature on (a) storage modulus, (b) Loss modulus and (c) Tan δ.

3.4 Tribological Behavior

The ANOVA summary of specific wear rate is given in table 7. The Fisher's test column ascertains the parameters (G, T, P and GxT) accepted at 90% confidence level influencing the variation in the impact strength. The percent contribution indicates that the composite parameter, G (graphite) only contributes 88.30% of variation, parameter, T (teflon) aids 9.55% of variation, and the rest of parameters contribute negligible variation. The ANOVA summary of coefficient of friction is given in table 8. The percent contribution indicates that the composite parameter, G (graphite) only contributes 98.71% of variation. The effect of other parameters is negligible on the coefficient of friction.

TABLE 7

ANOVA summary of the specific wear rate, x 10^{-8} g.N-m

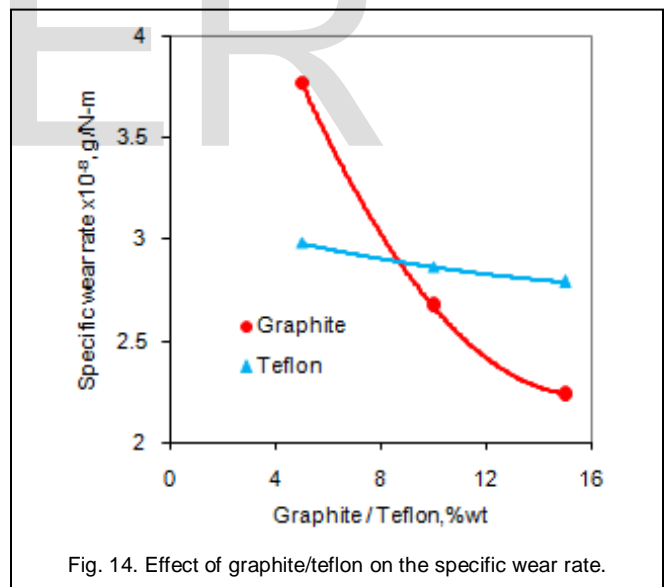| Source	Sum 1	Sum 2	Sum 3	SS	v	V	F	P
G	23.49	19.98	14.53	6.79	2	3.4	1072.6	88.3
T	20.91	19.15	17.94	0.74	2	0.37	116.7	9.55
P	18.89	19.3	19.81	0.07	2	0.03	9.5	0.83
AxB	18.83	19.58	19.59	0.06	4	0.02	6.3	0.62
Error				0.0222	7	0.0032	1	0.7
T				7.6822	17			100

Fig. 14. Effect of graphite/teflon on the specific wear rate.

The wear experiments were conducted at constant load of 100N for sliding distance of 1000m. The specific wear rate as a function of graphite / teflon content is plotted as shown in figure 14. The specific wear rate decreases with increase in graphite/teflon content from 5 to 15%. The coefficient of friction increases with increasing content of graphite in the composite (figure 15). In graphite, the carbon atoms are arranged hexagonally in a planar condensed ring as shown in figure 16. Also, the layers are stacked parallel to each other, with the atoms within the rings bonded covalently, whereas the layers are loosely bonded together by Van der Waal's forces. The

anisotropic nature of graphite is the result of the two types of bonding actions in different crystallographic directions. The ability of graphite to form a solid film lubricant may be attributed to these two contrasting chemical bonds. Also, the weak Van der Waal's forces govern the bonding between the individual layers, permitting the layers to slide over one another, making it an ideal lubricant, and resulting in a reduced coefficient of friction and, hence, wear.

TABLE 8

ANOVA summary of coefficient of friction

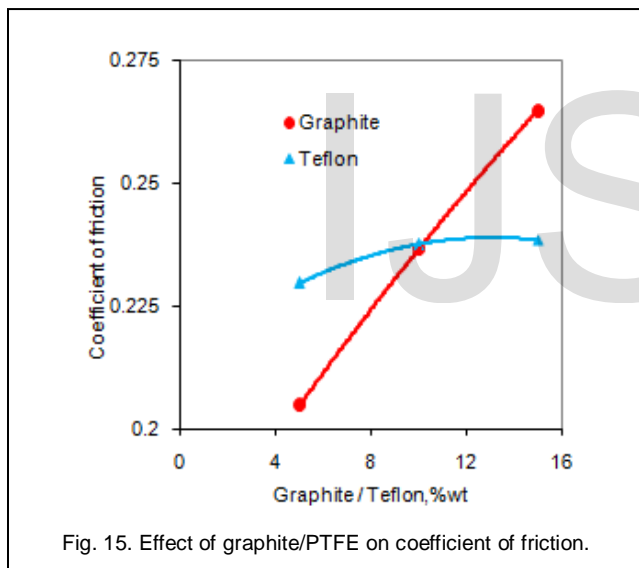| Source	Sum 1	Sum 2	Sum 3	SS	v	V	F	P
G	1.266	1.429	1.566	0.01	2	0.005	346.54	98.71
T	1.389	1.441	1.431	0	2	0	0	-0.29
P	1.528	1.408	1.325	0	2	0	0	-0.29
GxT	1.392	1.427	1.442	0	4	0	0	-0.57
Error				0.0001	7	0.00002	1	2.44
T				0.0101	17			100

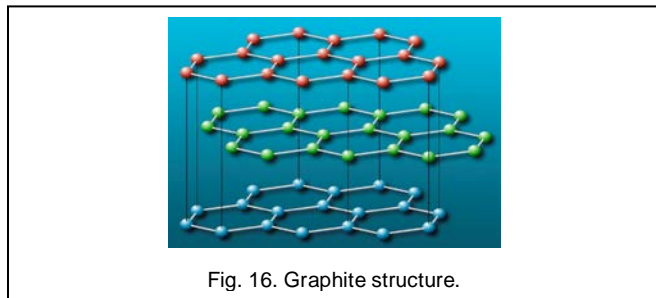Fig. 15. Effect of graphite/PTFE on coefficient of friction.

Fig. 16. Graphite structure.

According to SEM images the addition of teflon and graphite particles, the matrix damage is lessened even at high load and velocity conditions and led to an enhanced load-carrying capacity of the composite. Further, composites with higher wt % graphite shows more stable wear performance under all test conditions. It seems that although additional lubricants con-

tribute to a stable development of the transfer film even at extreme sliding conditions, meaning that uniform transfer of teflon and graphite from the sample to the counterface is observed. Occurrence and extent of agglomeration is the competition of two opposing forces acting on the particles during mixing process. These two types of forces include adhesive force between the particles, which reinforces particle agglomeration, and shear force exerted on the particles during the mixing, which leads to breakdown of aggregates [39].
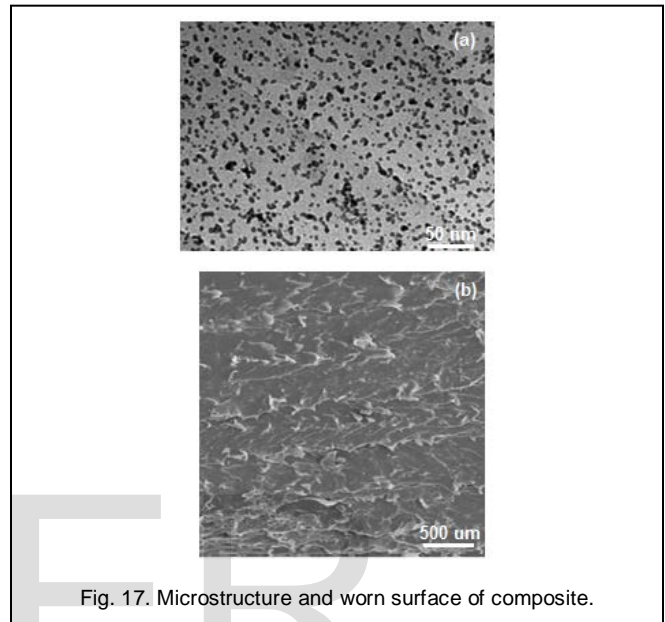

Fig. 17. Microstructure and worn surface of composite.

3.5 Confirmation of Tests

The key task was the determination of the preferred combination of the levels of the factors indicated to be significant by the analytical methods. The insignificant factors may be set at any desirable levels. The purpose of the confirmation experiment was to validate the conclusions drawn during the analysis phase.

$$\text{Confidence interval, C.I} = \sqrt{\frac{F_{\alpha;2;v_e}V_{ep}}{n_{eff}}} \tag{4}$$

Where $F_{\alpha};2;v_e$ is the Fisher's ratio, α is the risk, v_e = degrees of freedom for pooled error and V_{ep} is the pooled error variance.

Effective sample size, neff =

$$\frac{N}{1 + \text{Total degrees of freedom associated with items used in mean estimate}} \tag{5}$$

If the average of the results of the confirmation experiments was within the limits of the confidence interval; the significant factors as well as the appropriate levels for obtaining the desired result were properly chosen. If the average of the results of the confirmation experiment was outside the limits of the C.I, the chosen factors were wrong or the measurements might be with excessive error, necessitating further experimentation.

The significant factors and their levels were found to be graphite (10%), teflon (10%) and particle size of graphite

(55nm). The confirmation levels of all the characteristics are given in table 9.

TABLE 9

Typical properties of the sleeve bearing and thrust washer

S.No.	Property	Confidence levels		Experi-mental av-erage value
		Lower limit	Upper limit	
1	Young's mod-ulus, GPa	3.11	3.25	3.16
2	Tensile strength, MPA	82.15	87.85	84.36
3	Flexural strength, MPa	37.71	41.53	39.43
4	Impact strength,	0.26	0.36	0.29
5	Specific wear rate, g/N-m	1.52	1.81	1.74
6	Coefficient of friction	0.29	0.31	0.30

Typical products such as sleeve bearing and thrust washer (figure 18) were fabricated with a composite mixture of 10% graphite, 5% teflon, 80% nylon 6. The particle size of graphite was 55 nm. The properties of nylon 6/teflon/graphite composite are within the limits of confirmation levels (table 9).
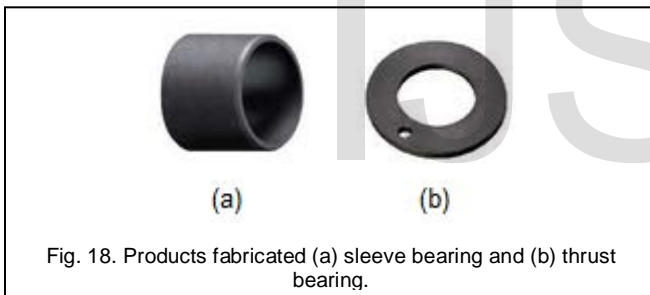

Fig. 18. Products fabricated (a) sleeve bearing and (b) thrust bearing.

4 CONCLUSION

At higher content of graphite, the reduction of strength is owing to its highly porous structure and high surface area, low content of polymeric matrix is insufficient to infiltrate in graphite. As the graphite is a brittle material, the flexural strength is sensitive to flaws or defects. If a flaw is present within the span of the flexural test specimen, then the load required to break the specimen is reduced. When failure occurs, it is catastrophic. An increase in flexural strength on account of increasing PTFE is due to its relative stiffness supporting dimensional stability. The reduction of impact of strength is owing to the increase of hardness with the addition of graphite content in the composite. This indicates that the introduction of graphite increases the storage modulus of the composites. Also, the layers are stacked parallel to each other, with the atoms within the rings bonded covalently, whereas the layers are loosely bonded together by Van der Waal's forces. The anisotropic nature of graphite is the result of the two types of bonding actions in different crystallographic directions. The ability of graphite to form a solid film lubricant may be attributed to these two contrasting chemical bonds.

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