

Enhancement in Mechanical Behavior of Nylon/Teflon Composites by Addition of Nano Iron Oxide ($\gamma\text{-Fe}_2\text{O}_3$)

Karan Agarwal¹, Nirmala Akhil², Regalla Srinivas³, A. Chennakesava Reddy⁴

^{1, 2, 3}Students, Department of Mechanical Engineering, JNTUH College of Engineering
Kukatpally, Hyderabad – 500 085, Telangana, India

⁴Professor, Department of Mechanical Engineering, JNTUH College of Engineering
Kukatpally, Hyderabad – 500 085, Telangana, India

Abstract: Iron oxide as a nano filler material to Nylon 6/Teflon matrix doubles the tensile strength and increases the hardness of the composites. There is the occurrence of agglomeration among iron oxide particles owing high surface energy. The addition of 3% of iron oxide yields better mechanical properties.

Keywords: Iron oxide, Nylon 6, Teflon, tensile strength, hardness, agglomeration, flow lines, injection molding

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]. 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 [2].

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. [3] prepared PTFE/ZnO nano composites. The wear resistance was doubled up with a maximum wear resistance at ZnO concentrations of 15vol%. Sawyer et al. [4] used 38 nm Al_2O_3 filler to improve the wear 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 [5]. Wang et al. [6] 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 [7] reported that the addition of 2vol% nano-CuO could enhance the wear resistance of short fiber-reinforced polyphenylene sulfide. The use of Nylon 6 and Teflon as a matrix material and graphite as a filler material to fabricate sleeve bearing and thrust washer is presented [8].

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 Teflon varying

the percentage by weight. The mechanical properties are tested.

2. Materials and Methods

The matrix material was Nylon 6 and the filler material was Teflon. The melting temperature of Nylon 6 is 220°C and crystalline density at 25°C is 1.23 g/cm^3 . The melting point of Teflon 327°C and crystalline density at 25°C is 2.20 g/cm^3 .



Figure 1: Torque rheometer with a twin-rotary mixer

2.1 Preparation of Nylon/Teflon Composites

Different matrix/filler blends were prepared in a torque rheometer with a twin-rotary mixer as shown figure 2. The Teflon content in the composite was fixed to be 25%. The nylon 6 and iron oxide were varied. Prior to the melt processing, Nylon 6, Teflon, and iron oxide were dried in the oven at 75°C for 4 hr. Iron oxide nano powder were pre-mixed 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 iron oxide 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 and hardness tests were prepared using an injection molding machine with 10 ton clamping pressure at 220-230°C and an injection pressure of 50 bars.



Figure 2: Injection molding machine.

2.2 Conduction of Tests

The following tests were conducted on the metal matrix composites:

- Tensile test for elastic modulus and ultimate tensile strength
- Vickers hardness test

The as-mold test samples (according to ASTM D 618) are shown in figure 3 along with two die halves. The computer-interfaced tensometer was used for the tensile test as shown in figure 4. 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 Vickers hardness was used to measure the hardness of test samples.



Figure 3: Two mold halves and specimens.

The optical microscope was used to study the porosity and voids in the test samples. The scanning electron microscope was employed to examine the fracture of the tested tensile specimens.



Figure 4: Tensometer.

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.

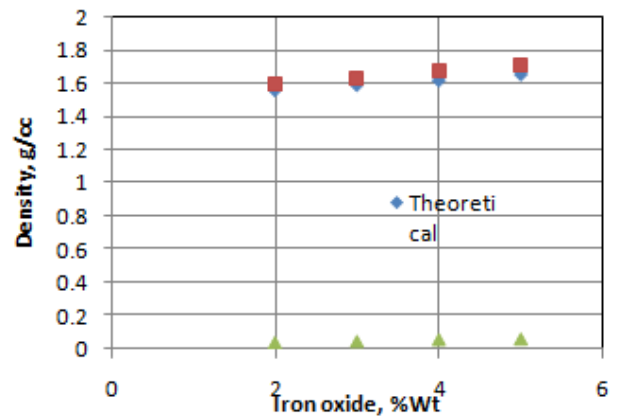


Figure 5: Density and voids of Nylon/Teflon/Iron oxide composites

3.1 Density and Voids

The density depends on the composition of the Nylon 6/Teflon/Iron oxide polymer composite. The calculated density values of the Nylon/Teflon/Iron oxide polymer composites were higher than the measured values as noticed from figure 4. There is nucleation of crystal growth in the composite as the density of the composite is less than theoretical density calculated by the rule of mixture. This might be due to the formation of voids in the composites. The density of composites increases with increases in the content of iron oxide. The void content increases with increase in the iron oxide content in the composite. The densities of Teflon, Nylon and iron oxide are 2.20 g/cc, 1.29 g/cc and 5.24 g/cc respectively. The optical microscope images shown in figure 5 reveals the presence of voids in the composite having (a) 10%, (b) 15%, (c) 20% and (d) 25% Teflon respectively.

3.2 Mechanical Behavior of Nylon/Teflon/Fe₂O₃ Composites

The load vs displacement of Nylon/Teflon/Iron oxide composites is shown in figure 6. It is observed that the load increases with an increase in the iron oxide content in the composite. The reinforcing particles in each composite seem to be isolated from each other, but their distribution is relatively non-uniform (figure 6).

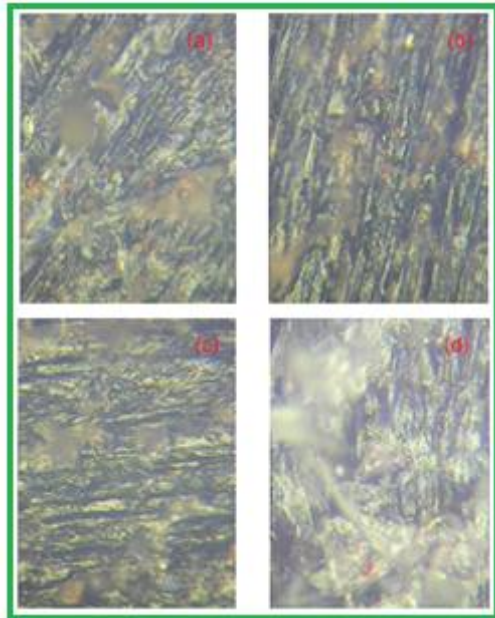


Figure 5: Optical micrographs showing voids or porosity in Nylon/Teflon/Iron oxide composites

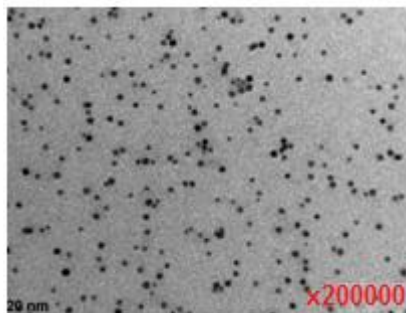


Figure 6: Iron oxide nano particle distribution in the nylon/Teflon/Iron oxide polymer composite

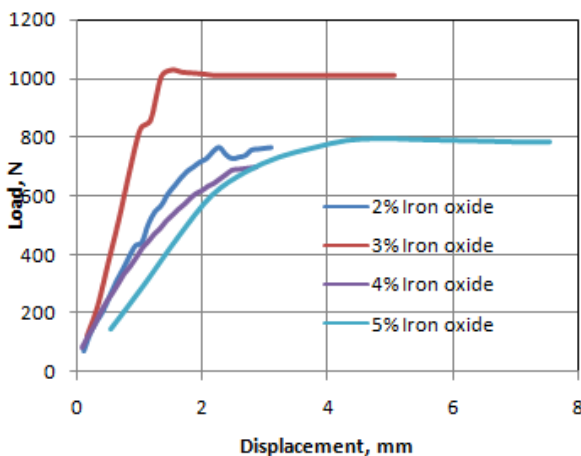


Figure 7: Displacement vs load curve of Nylon/Teflon/Iron oxide composites

The influence of iron oxide addition on the tensile strength is shown in figure 7. The engineering tensile strength, true tensile strength and breaking strength increase with increasing Teflon content in the composite. The strengthening mechanism is caused by grain refinement and dislocations interaction by the introduction of the iron oxide particles. The higher the volume fraction of the Fe₂O₃ particles, the more apparent the grain refinement and dislocations interaction.

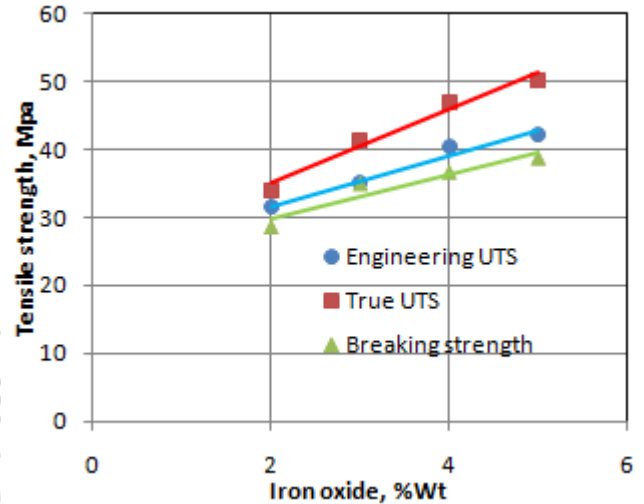


Figure 8: Influence of Teflon on tensile strength

Filler orientation strongly affects the strength of polymer composites. This is important because injection molded ASTM standard 'dog-bone' shaped test specimens give high filler orientation. The most important filler parameter affecting the tensile strength is its shape. Unfortunately, when the filler is non-spherical, the theories become much more complicated. Figure 9 shows the stresses acting on an iron oxide particulate (assumed to be spherical) reinforcement in polymer matrix composite.

$$\sigma_p = V_p(\sigma_m + 1.25\tau_m) + V_m\sigma_m \quad (1)$$

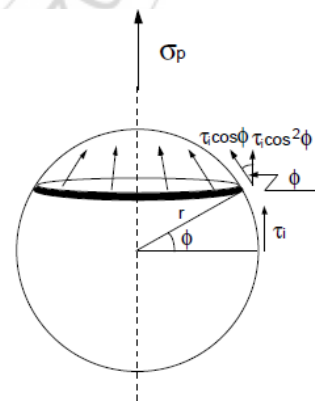


Figure 9: Schematic diagram showing stress components acting on an iron oxide particle (assumed to be spherical).

Pukansky examined the effects of 11 different fillers in polypropylene [10] and concluded that Young's modulus is affected by the amount of bonded polymer, which is in turn related to surface area, and therefore to both particle size and shape. That observation helps to explain the strong effect that nano-fillers have on the modulus of a composite. Schreiber and Germain showed that modulus depends on the strength of interaction between the polymer and the filler surface [11].

Polymers interact with the filler surface, forming an interphase of adsorbed polymer. The thickness of the interphase can vary widely from system to system. That is to be expected; for example polar polymers such as polyamides are capable of strong, specific interactions with groups on the filler surface. In contrast, non-polar polymers such as PE and PP have weaker interactions with fillers. The apparent thickness of the interphase also depends strongly upon the measurement method. Lower values of around 0.004 μm are reported from extraction experiments, whereby all non-adsorbed polymer is solvent extracted.

Several other factors determine the level of reinforcement attained by adding filler. These include the volume fraction of filler added, the surface area of the filler, particle shape, the level of adhesion between the filler and polymer [12], as well as the thickness and nature of the interphase between the two phases. It is well known that spherical fillers give least reinforcement, platy fillers are better and fibrous fillers are best of all. This improvement must be due to the high surface area of the filler as the filler is isotropic. The high surface area increases overall polymer-filler adhesion and thereby improves the tensile strength. The filler creates an additional complication especially for injection molded parts. Namely, during mould filling, the filler distribution becomes non-homogeneous due to the flow. One consequence is flow lines and weld lines. The flow lines are observed in Nylon/Teflon/ Iron oxide polymer composite as seen in figure 10.

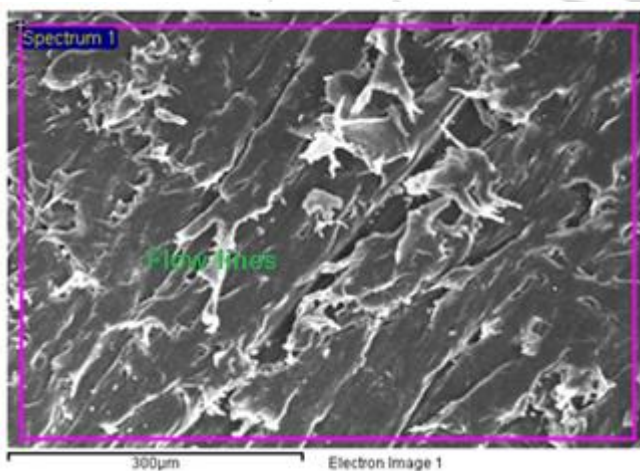


Figure 10: Flow lines in Nylon/Teflon/Iron oxide composite.

The influence of iron oxide on percentage elongation (ductility) and hardness is shown in figure 10 & 11 respectively. The peak percent elongation (ductility) decreases with an increase content of iron oxide in the composite while the break percent elongation increases. The increase in the strength of the composites by the introduction of iron oxide particles is accompanied by the decrease in the plasticity, since iron oxide particles are the micro-crack initiators during deformation. Fe_2O_3 is brittle. During deformation, two types of the micro-cracks are initiated by the Fe_2O_3 particles. First, if the interfacial cohesion between the Fe_2O_3 particles and matrix is strong, the Fe_2O_3 particles fracture to nucleate micro-cracks when the local strain and dislocation density reach the critical values by the high stress concentration. Second, if the interfacial cohesion between the Fe_2O_3 particles and matrix is weak, decohesion between the Fe_2O_3

particles and matrix happen to nucleate micro-cracks before the Fe_2O_3 particles are fractured. Thus, a strong interfacial cohesion can improve both the strength and plasticity of the composites since weak interfaces nucleate micro-cracks at a rather low external applied stress. The addition of iron oxide increases the hardness of the composite as shown in figure 12.

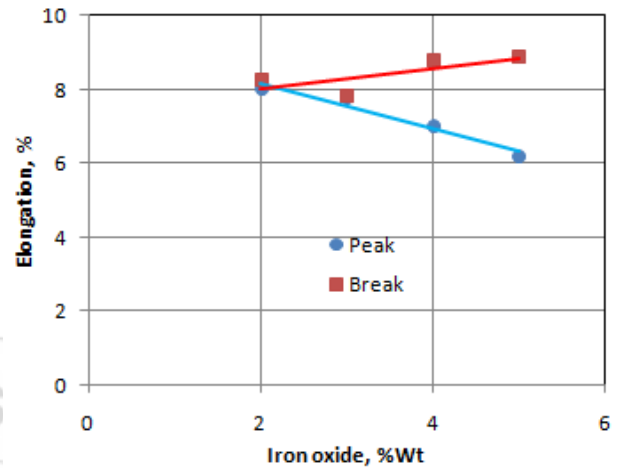


Figure 11: Influence of Teflon on ductility

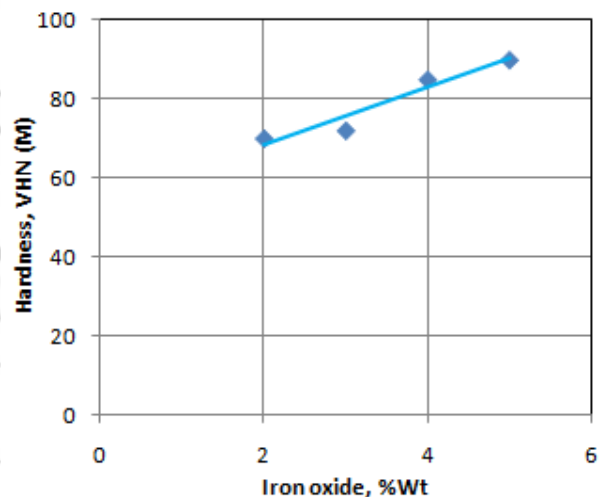


Figure 12: Influence of Teflon on hardness.

3.3 Concept of Strengthening Mechanism

The concept of strengthening mechanism is the surface energy (or surface tension) of a liquid, which is the amount of excess energy at the surface of the liquid. Surface tension exists because molecules in the bulk liquid are in a lower energy state than at the surface. When a liquid is placed on a solid surface the phenomenon therein depends on the relative surface energy of the liquid compared to the surface energy of the solid. If the liquid has a higher surface energy than the attractive forces between the liquid and the solid surface, the liquid will prefer to maintain its spherical form. When this phenomenon happens between an adhesive and a substrate the adhesive will not spread and make intimate contact with the surface to be bonded; rather, the liquid molecules will tend to remain associated with themselves rather than the surface. The result is lower bond strengths. In contrast if the surface energy of the adhesive is less than that of the sub-

strate the adhesive will spread out and wet the substrate thus making the intimate contact necessary for good bonding.

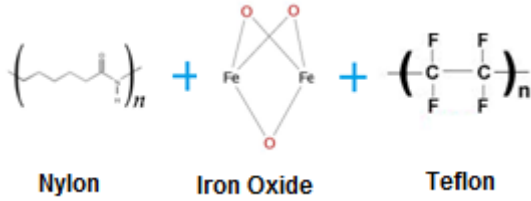


Figure 13: Structure of Nylon/Teflon/Iron oxide composite

The structure of Nylon/Teflon/Iron oxide polymer composite is shown in figure 13. The structure of iron oxide (Fe_2O_3) is shown in figure 14. In Maghemite ($\gamma\text{-Fe}_2\text{O}_3$) most of the iron is in the trivalent state (Fe^{3+}) and by the presence of cation vacancies in the octahedral sites. Maghemite has a cubic unit cell in which each cell contains 32 O ions, $21\frac{1}{3}$ Fe^{3+} ions and $2\frac{2}{3}$ vacancies. The cations are distributed randomly over the 8 tetrahedral and 16 octahedral sites. The strength of Nylon/Teflon/Iron oxide polymer composite depends upon the adhesive bonds between Nylon and iron oxide, Teflon and iron oxide and Nylon and Teflon. Being highly susceptible to oxidation, magnetite (Fe_3O_4) is transformed to maghemite ($\gamma\text{-Fe}_2\text{O}_3$) in the presence of oxygen

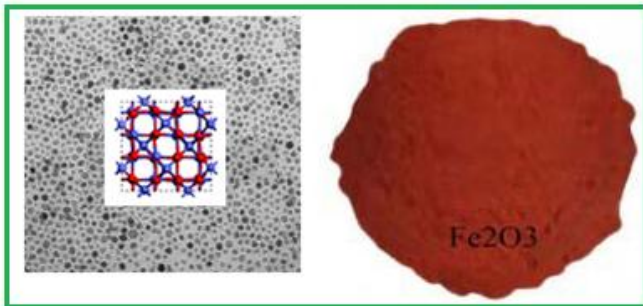
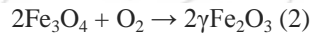


Figure 14: Structure of iron oxide nano particle.

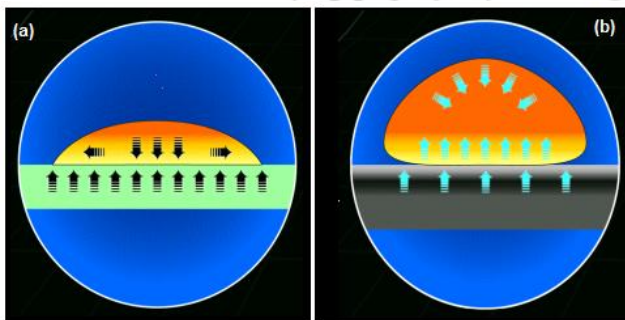


Figure 15: Adhesive bonding based on surface tension (a) solid surface has high surface energy; Liquid will spread or wet out the surface (b) solid has low surface energy; adhesive will bead up on the surface.

The high surface energy (HSE) materials such as metals and glasses can be readily bonded with a variety of adhesives. The critical surface tensions of Teflon, Nylon and iron oxide are 18.5, 43 and 1350 mN/m respectively. As the surface tension of iron oxide is greater than those of Nylon and Teflon, the adhesive bonding occurs between these ingredients in the Nylon/Teflon/Iron oxide polymer composites. The phenomenon of strengthening mechanism in the Nylon/Teflon/Iron oxide polymer composites is, therefore, of category shown in figure 15(a).

3.4 Fracture Behavior of Nylon/Teflon Composites

The fractured surfaces of nylon/teflon/iron oxide specimens revealed with SEM are shown in figure 16. The SEM images reveal that the fracture is due to interface debonding between iron oxide to Teflon, iron oxide to Nylon and Nylon to Teflon. It is also the fracture of matrix only but not iron oxide nano particles. Figure 16(a) is of 2% iron oxide; figure 16(b) is of 3% iron oxide; figure 16(c) is of 4% iron oxide; and figure 16(d) is of 5% iron oxide. The breakage of clustering is also observed among iron oxide nano particles in the composite. The agglomeration is observed in the composites due 4% to 5% iron oxide.

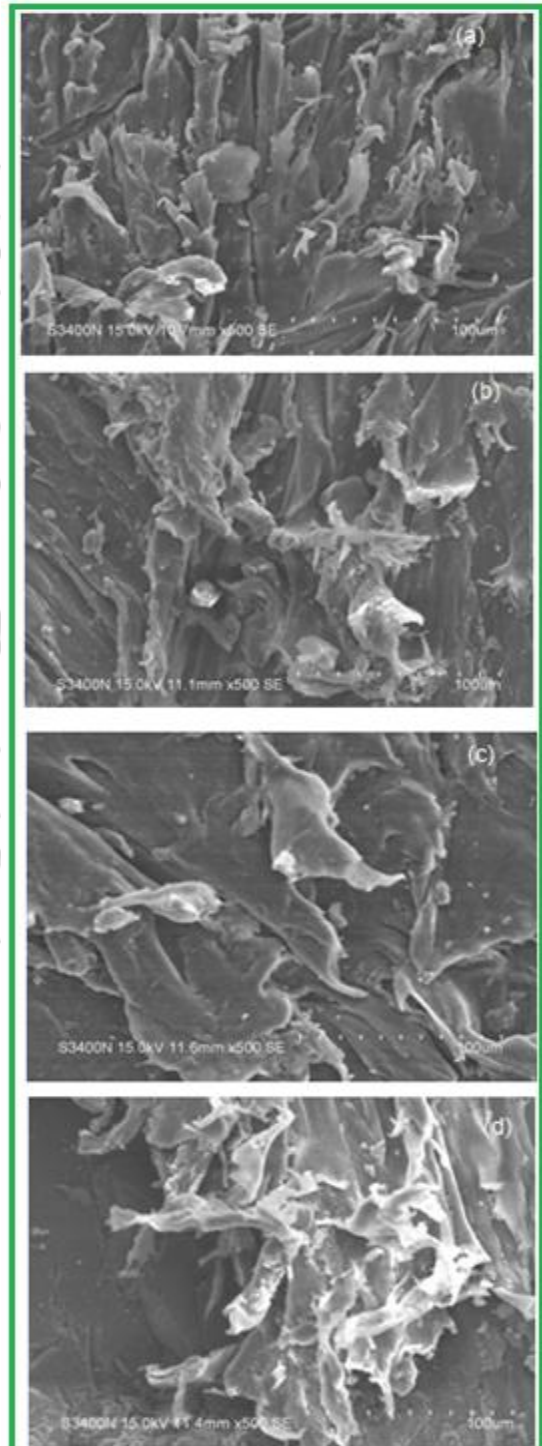


Figure 16: Fracture surfaces of Nylon/Teflon composites

4. Conclusions

The addition of iron oxide to Nylon 6/Teflon matrix doubles the tensile strength and the hardness while it reduces the ductility of the Nylon/Teflon polymer composites. There is the occurrence of agglomeration of iron oxide particles owing to high surface energy. The flow lines have been observed in the Nylon 6/Teflon/Iron oxide on account injection molding process.

5. Acknowledgements

The author acknowledges with thanks University Grants Commission (UGC) – New Delhi for sectioning R&D project.

References

- [1] Q. H. Wang, Q. J. Xue, W. M. Liu and J. M. Chen, "The Friction and Wear characteristics of Nanometer SiC and Polytetrafluoroethylene Filled Polyetheretherketone," *Wear*, vol. 243, no. 1-2, pp. 140-146, 2000.
- [2] J.K.Lancaster, Polymer-based bearing materials, the role of fillers and fiber Reinforcement, *Tribology* vol. 5, no.6, pp.249-55, 1972.
- [3] F. Li, K. Hu and J. Li, "The Friction and Wear Characteristics of Nanometer ZnO Filled Polytetrafluoroethylene," *Wear*, vol. 249, no. 10-11, pp. 877-882, 2002.
- [4] W. Sawyer, K. Freudenberg, P. Bhimaraj and L. Schadler, "A Study on the Friction and Wear Behavior of PTFE Filled with Alumina Nanoparticles," *Wear* vol. 254, no. 5- 6, pp. 573-580, 2003.
- [5] J. Bijwe, J. J. Rajesh A. Jeyakumar, A. Ghosh and U. S. Tewari, "Influence of Solid Lubricants and Fiber Reinforcement on Wear Behavior of Polyethersulphone," *Tribology International*, vol. 33, no. 10, pp. 697-706, , 2000.
- [6] Q. H. Wang, Q. J. Xue and W. C. Shen, "The Friction and Wear Properties of Nanometer SiO₂ Filled Polyetheretherketone," *Tribology International*, vol. 30, no. 3, pp.193-197, 1997.
- [7] M. H. Cho and S. Bahadur, "Study of the Tribological Synergistic Effects in CuO-Filled and Fiber-Reinforced Polyphenylenesulfide Composites," *Wear*, vol. 258, no. 5-6, pp. 835-845, 2004.
- [8] A. Chennakesava Reddy, "Characterization of Mechanical and Tribological Behavior of (Nylon 6 + Graphite + Teflon) Nano Particulate Composite: Application Perspective," *International Journal of Scientific & Engineering Research*, vol.6, no.4, pp. 378-386, 2015.
- [9] B. Kotiveerachari, A. Chennakesava Reddy, Interfacial effect on the fracture mechanism in GFRP composites, CEMILAC Conference, Ministry of Defence, India, 20-21st August, B85-87, 1999.
- [10] B. Pukansky, J. Kolarik and F. Lednicky, "Polymer Composites," *Proceedings of the 28th Microsymposium on Macromolecules*, Prague, Czechoslovakia, vol.67, pp.553, 1985.
- [11] H.P. Schreiber and F. St. Germain , "in Acid-Base Interactions: Relevance to Adhesion Science and Technology," Eds., K.L. Mittal and H.R. Anderson, Jr., VSP, Utrecht, The Netherlands, 1991, 273.
- [12] S.J. Porter, C.L. DeArmitt, R. Robinson, J.P. Kirby and D.C. Bott, "The Surface Characterization of Polyacrylonitrile-

based Carbon fibers by Electrochemical Techniques," *High Performance Polymers*, vol. 1, no.1, pp.85-93, 1989.