

Nano - Engineered Fire Resistant Composite Fibers

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ABSTRACT:

We undertook the investigation of fire resistant nanocomposite fibers by: (1) Melt compounding of Nylon 6 and organically modified montmorillonite (OMMT) nanoparticles and polypropylene-grafted polypropylene - fumed silica nanoparticles in Brabender twin blade roller mixer and extruding fibers through Sterling single screw extruder. (2) Solution mixing of polypropylene-grafted polypropylene fumed silica nanoparticles via ultrasonication and fiber formation. The morphology and thermal stability of the nanocomposites and nanocomposite fibers were studied. Transmission Electron Microscope (TEM) images revealed the intercalation of OMMT by nylon 6 polymer chains. Scanning Electron microscope (SEM) images of the nanocomposite fibers confirmed the presence of nanoparticles on the fiber surface and cross section. Differential Scanning Calorimetry (DSC) studies on the nanocomposites and nanocomposite fibers indicated no significant difference in percentage crystallinity or melting temperature compared to their neat nylon. However the peak crystallization temperature of the nanocomposites was 10-15°C higher for nylon 6 nanocomposites and the crystallization peak is narrow compared to nylon6 revealing a higher crystallization rate due to the heterogeneous nucleation effect of clay. In case of both polypropylene nanocomposites and fibers decreased percentage crystallinity was observed compared to neat counterparts. Thermo gravimetric analysis of the nanocomposite fibers also confirmed the formation of a considerable residual char, which was significantly higher, compared to neat fibers. In case of nylon 6, no significant improvement in thermal stability was observed for the nanocomposite fibers. However TGA analysis of polypropylene composite fibers showed improvement in thermal stability as compared to neat polypropylene fibers. The temperature for 5% weight loss is increased by around 20°C for composite fibers to that of neat polypropylene fibers. To get further insight on morphology of composite fibers, X-ray diffraction studies are in progress. Further investigation on the nanocomposite fibers is focused on thermo-mechanical properties and flammability characteristics.

GOAL STATEMENT

The objective of the research is to obtain a molecular understanding of thermal stability and degradation of heat and fire resistant polymeric composite fibers embedded with nano-scale hard particles. Heat transmittance through polymer (e.g. nylon6, polypropylene, polyester etc) rich composites under exposed flame or high temperature is complex due to the various modes of heat transfer. These research addresses issues related to the mechanisms of heat/flame propagation through polymer-filler assemblies via overall structural investigation and interfacial interaction between nanoparticle and polymer phases. Hence, we are in the process of establishing the link between microstructural parameters and fire resistance behaviors by experimental flammability studies of nanocomposites and developing heat penetration model that takes into account various modes of heat supply and their influence on final fiber geometry.

INTRODUCTION

Nanocomposites have been in the helm of research since 1961 owing to its enhanced mechanical, thermal and barrier properties over the conventional micro composite counterparts. They are showing great promise as effective fire retardants for some types of materials. Because of the upcoming restrictions on some of current popular flame retardants, the flame retardancy of nanoparticle filled systems or any non-halogen alternatives need to be improved to meet the new standards. As a result interest in this area of research has recently increased. Though the significant reduction in heat release rate of nanocomposites was reported quite long time before, the flame retardancy of polymer filled with nanoparticles such as clay, carbon nanotubes or silica

has not further improved to achieve industrial acceptance. Also the effect of nano fillers on thermal stability and flame retardancy of nanocomposite fibers is not clear.

Introduction of fillers into polymers may either accelerate or inhibit different stages of the decomposition process, and also may change the mechanisms of these reactions. Polymer decomposition is a highly complex process. To achieve reasonably good fire resistant property of nanocomposite materials it is essential to have clear enough understanding of their decomposition behavior. Thermal stability of the polymers can be improved by the introduction of chemically active or inert fillers. In the later case only physical factors, such as mobility of the macromolecules on the interface or orientating influence and aspect ratio of the filler, contributes to overall thermal stability, whereas, in the former case, interplay of both physical and chemical factors determines the filler activity on polymer thermal stability. Introduction of chemically active fillers improves thermal and thermal-oxidative stability of polymers by the formation of thermally stable chemical bonds between polymer and the filler. It is believed that formation of cross-linking bonds and of a three-dimensional polymer-filler network at elevated temperatures increase the thermal stability of the polymer. Besides, chemically active filler may promote the decomposition of thermally unstable groups present in the polymer to form more thermally stable bonds or these filler may play an important role in retarding the kinetics of radical decomposition. Also, relatively higher thermal conductivity and heat capacity of filler is supposed to dissipate the heat energy, and improve the thermal stability of the polymer-filler composite [1].

The properties of the nanocomposites depend on the chemistry of the nano fillers, polymer - filler compatibility and nanocomposite preparation method and process conditions. The morphology and dispersion of the fillers are critical factors in determining the properties of the nanocomposite and hence the behavior of the nanocomposite fibers will be different depending on the extrusion conditions. In order to enhance our understanding on the influence of extrusion and drawing on morphology and dispersion of fillers in the fibers and the effect of these conditions on the thermal stability and flame retardant behavior we undertook to synthesize Nylon 6/ montmorillonite fiber and polypropylene /grafted polypropylene/ fumed silica fiber and make a comparative investigation of nanocomposites and nanocomposite fibers.

EXPERIMENTAL MATERIALS AND METHODS

Materials and Sources

1. Montmorillonite (1.34TCN) were supplied by Nanocor Inc
2. Nylon 6 and nylon 6/clay composite- supplied by Honey well company.
3. Fumed silica supplied by Sigma-Aldrich
Avg. individual particle size 7 nm.
4. Isotactic Polypropylene beads, supplied by Philip Sumika.
Fiber grade polymer Mn ~ 150,000
5. Maleic anhydride grafted polypropylene supplied by Sigma-Aldrich
6. Xylene

Equipment and Services Used

1. Brabender-plasticorder twin blade roller mixer (intellitorque) , Brabender, Hacknshack, NJ (ATMC, UMD).
2. Sterling WB2122 single screw extruder L/D 24:1
3. Thermo-gravimetric analyzer (TGA) Q-500, TA Instruments, (UMD)
4. Differential scanning calorimeter (DSC) Q-1000, TA Instruments (UMD)
5. Scanning electron microscope (SEM) JEOL, JSM 5610 (ATMC, UMD)
6. Ultrasonic processor, , Cole Palmer Inc. (UMD)
7. Cone Calorimeter (WPI)
8. X-ray diffractometer (Rigaku, 300) (MIT)
9. Transmission electron microscope and electron diffraction (JEOL, TEN, 2010F field emission, 200kV) (Boston College)

RESULTS AND DISCUSSION

Nylon6/OMMT nanocomposite fibers

We prepared nylon 6/ Montmorillonite nanocomposites by melt compounding in a twin roller blade mixer. Montmorillonite (1.34 TCN), modified with quarternary ammonium salt (methyl dihydroxyethyl hydrogenated tallow ammonium) was used. Nylon6 pellets were dried in vacuum chamber at 80°C for 16 hours and melt compounded with the organic montmorillonite at 240°C for 10 minutes at 30, 50, 90 rpm. The diffusion of polymer chains into the galleries of layered silicate and eventual delamination and dispersion of the layered silicates in the polymer matrix is influenced by the chemistry of the clay, rheology of polymer melt and shear forces. In case of melt compounding selection of a compatible polymer layered silicate system and optimization of the process conditions could lead to nanocomposites with properties comparable to in-situ polymerized systems. In our studies and observation nanocomposite melt intercalated at 90 rpm had better properties as evidenced by TGA and DSC results. Transmission electron microscopy (TEM) studies of the nanocomposites in fine powder form revealed the polymer intercalation into the silicate layers. (Fig 1)

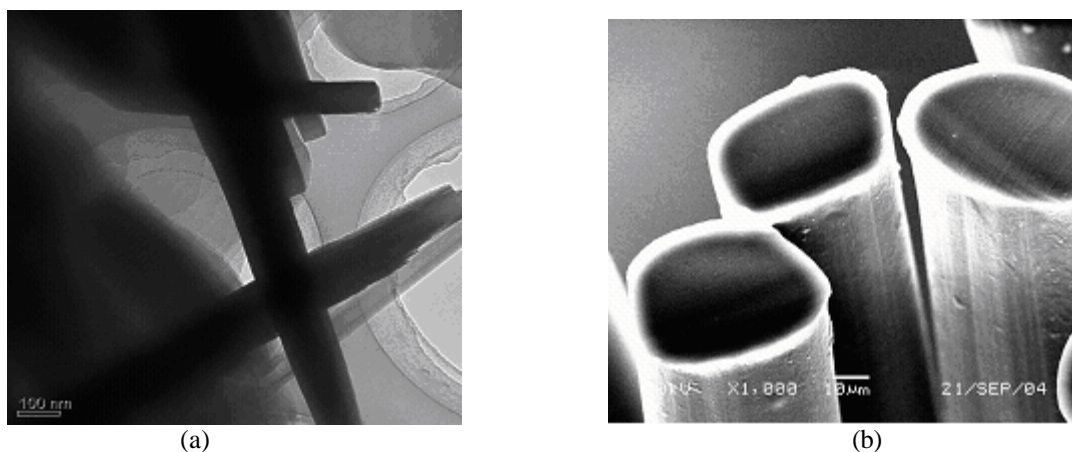


Figure 1: (a) TEM images of nanocomposite showing polymer intercalation into clay platelets (b) SEM image of nanocomposite fiber cross section.

The nanocomposites synthesized at 90 rpm were extruded into fibers using a single screw extruder. The screw rpm was 10 and pump speed was 30rpm. The temperatures of the 6 zones from feeder to spinneret were 230°C, 240°C, 240°C, 240°C, 240°C, 240°C respectively. The fibers were subjected to inline drawing. Draw ratio was 3 and drawing temperature was 120°C. Monofilament denier was around 12. Scanning electron microscopy images of the longitudinal surface and cross section of nanocomposite fiber shows the presence of particles on the cross section and longitudinal surface (fig2).

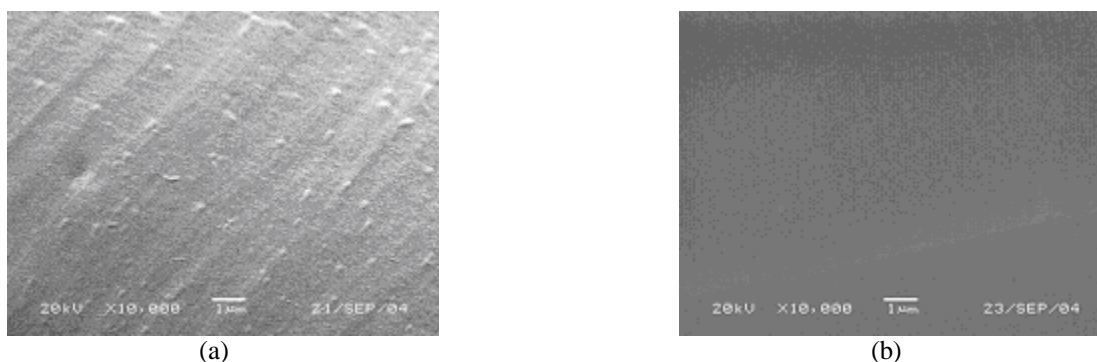


Figure 2: (a) SEM images of nanocomposite fiber cross section and (b) SEM image of neat fiber cross section.

Thermogravimetric analysis (TGA) was conducted on the nanocomposites and neat samples. The decomposition temperature (5% and 10% wt loss temperature) of nanocomposites is slightly less than neat nylon 6(fig). The difference is 15-20°C for 30 and 50rpm but for 90rpm it is 5-7°C. The amount of additional carbonaceous residue was also negligible. TGA analysis of nanocomposite fibers conducted in inert

atmosphere showed that montmorillonite neither increased the decomposition temperature nor decreased the decomposition rate of nylon 6 (fig 3). However, samples of nanocomposite fibers constantly yielded a residue of 4.5 -5% which was not observed in neat fibers which could be taken as an indirect indication of uniform distribution of nanoclay platelets in the fiber. Further experiments to confirm the observations and morphological studies of nanocomposites and nanocomposite fibers using X ray diffraction is in progress.

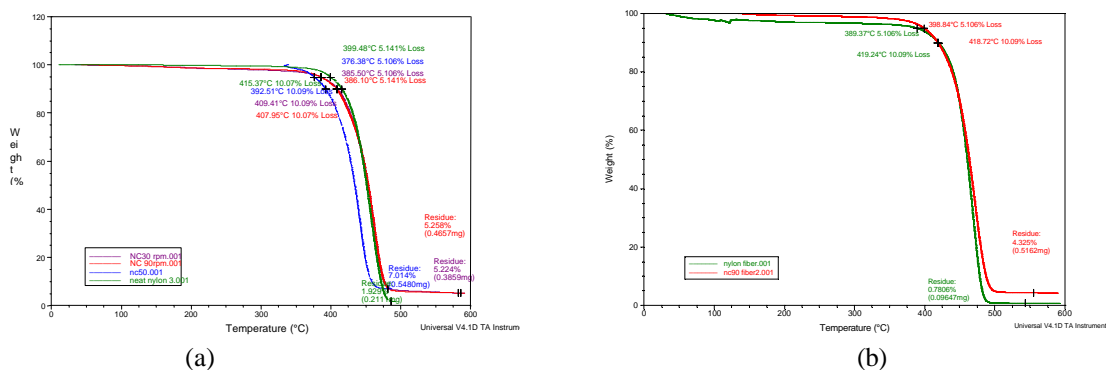


Figure 3: (a) TGA graph of nanocomposite and neat polymer and (b) TGA graph of nanocomposite and neat fiber.

Differential Scanning calorimetry (DSC) studies were conducted on the nanocomposite and neat samples. We used a heat/cool/heat method for nanocomposites and only heating cycle for fibers. The heating rate and cooling rate were 10°C / min respectively. The peak crystallization temperature of all the nanocomposites was 14-18°C higher than neat nylon. Narrow crystallization peak is observed in nanocomposites, indicating heterogeneous nucleation effect of layered silicates. The melting temperature was around 220°C which is almost same as that of neat nylon used in study. DSC studies conducted on the fibers extruded from the neat nylon and synthesized nanocomposites is given in fig 4. The curve profile of both nylon and nanocomposite fiber is similar with no significant difference in melting temperature or percentage crystallinity.

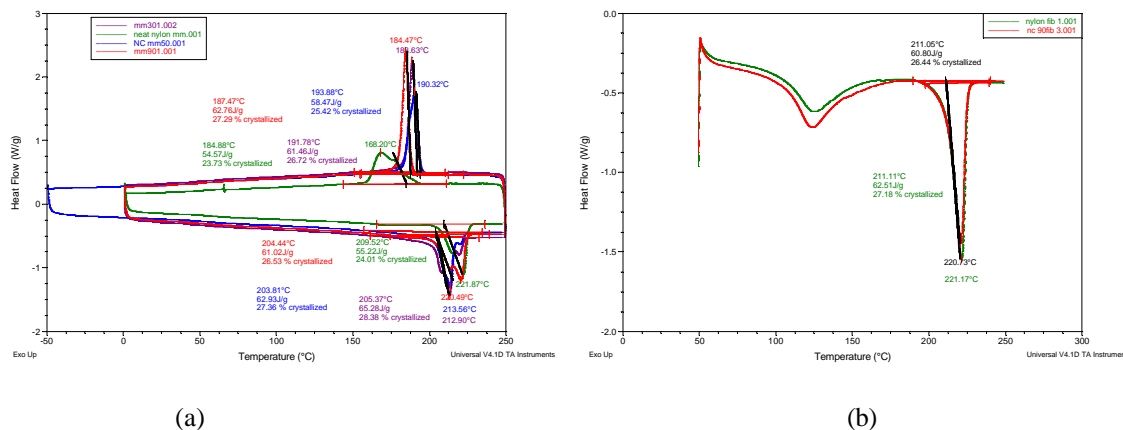


Figure 4: (a) DSC graph of nanocomposite and neat polymer and (b) DSC graph of nanocomposite and neat fiber.

Polypropylene/ fumed silica nanocomposite fibers

It is shown that silica in various forms, like fumed silica and silica gel is effective flame retardant inorganic additive for polypropylene. Fumed silica is almost spherical in size with avg. individual particle diameter around 7 nm. It tends to agglomerate and form chain like structures of around 100-200 nm. The surface of it

is reactive and consists of silanol groups projecting outwards. The maleic anhydride grafted polypropylene is added up to 10% by weight of nanocomposites to enhance chemical interaction between polymer and silica. It is proposed that the silanol group of silica and anhydride groups of polypropylene forms hydrogen bonding, which gives better particle dispersion, enhanced chemical interaction between 2 phases and lead to char formation. Our research work focuses on extruding the polypropylene-silica nanocomposite fibers and characterizing those for thermal stability, morphology and flame retardant characteristics.

5% silica-Polypropylene nanocomposites are prepared by 2 methods, namely melt mixing and solution mixing. The maleic anhydride grafted polypropylene is added up to 10% by weight of nanocomposites to enhance chemical interaction between polymer and silica. It is proposed that the silanol group of silica and anhydride groups of polypropylene forms hydrogen bonding, which gives better particle dispersion, enhanced chemical interaction between 2 phases and might lead to char formation. Also nanocomposites of ungrafted isotactic polypropylene were prepared. In solution mixing method, silica particles are dispersed in xylene by application of ultrasonic waves for 15 min. with 85% amplitude. Then polypropylene and grafted polypropylene were added in the bath and mixture is heated with simultaneous application of ultra sonic waves. At 140°C solvent is evaporated and nanocomposite is formed. In melt mixing method, nanocomposites were prepared from isotactic polypropylene and 5% fumed silica, at 180°C, screw rpm 50, for 20 min. under inert nitrogen atmosphere.

The nanocomposites synthesized by both methods were extruded into fibers using a single screw extruder. The screw rpm was 10 and pump feed rate was 4.8cc/min. The temperatures of the 6 zone from feeder to spinneret were 160°C, 160°C, 170°C, 170°C, 170°C and 170°C respectively. The fibers were drawn with draw ratio 4 at 120°C. monofilament denier was around 10. The composite fibers are characterized with Scanning Electron Microscopy (SEM), Differential Scanning Calorimetry (DSC), Thermogravimetric Analyzer (TGA).

SEM analysis

SEM images of composite fibers and neat polypropylene fibers are shown below.(fig. 5,6)

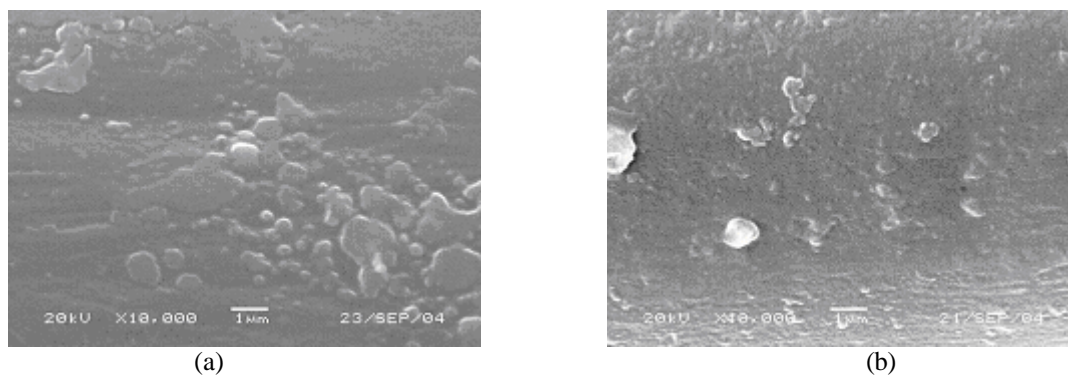


Fig. 5 (a) Neat i-PP fiber surface image and (b) Nanocomposite fiber surface image

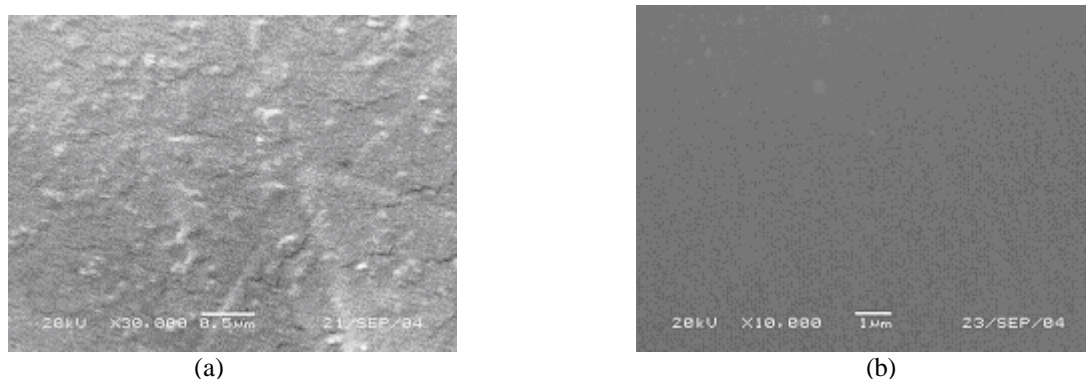


Fig.6 (a) nanocomposite fiber cross section and (b) neat i-PP fiber cross section

The SEM images are quiet revealing. The cross section of composite fiber showed the presence of filler which are not visible in neat fiber image.

TGA analysis

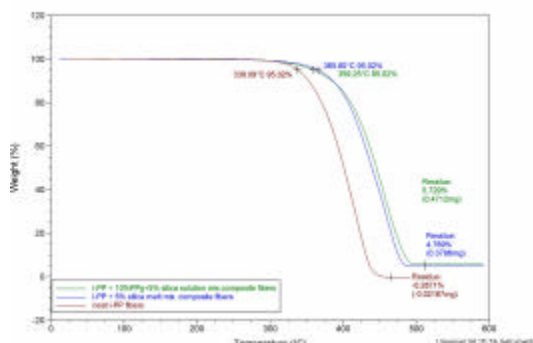


Fig. 7 TGA analysis of fibers

TGA analysis is carried out on both nanocomposite fibers as well as neat polypropylene fibers. It is observed that the decomposition temperature (temperature at 5% weight loss) is around 20°C higher for the nanocomposite fibers than that of neat polypropylene fibers. This indicates that fillers are playing role to give polymer, thermal stability. Also by the addition of grafted polypropylene the amount of residue is increased. The possible reason behind it is chemical interaction of silica with polymer, which is helping in retaining part of the polymer in residue. The decomposition temperatures are almost same for both melt mixing and solution mixing nanocomposite fibers.

DSC analysis

DSC analysis is carried out on both nanocomposites as well as nanocomposite fibers.

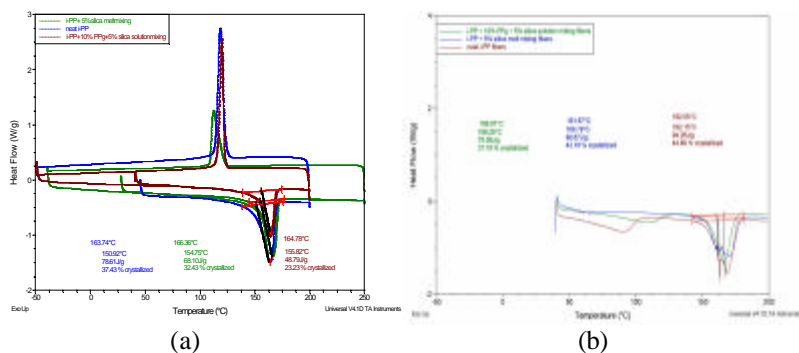


Fig. 8 (a) DSC analysis of fibers and (b) DSC analysis of nanocomposites

From DSC curves it is observed that the fillers have reduced the percentage crystallinity in nanocomposites as well as composite fibers, as compared to that of neat polypropylene samples. It can be inferred that fillers are hindering the crystal growth and hence reducing the crystallinity. In fiber DSC curves, small valley is observed at 100-120°C. This transition can be attributed to melting of beta crystals in the fiber, which are not seen in nanocomposite slabs. There is small reduction in onset melting temperature of nanocomposite as compared to neat polypropylene.

Heat Transfer modeling of nanocomposite fibers

Thermal conductivity is important to define flammability properties. A recent study done by Kashiwagi et al., shows that, by measuring thermal properties, we can understand the flammability properties [2]. Heat transfer takes place when there is a temperature difference in the material, and heat is transferred by conduction, convection or radiation. When the size of the material decreases, the importance of the molecular behavior

becomes much higher, the free path plays an important role, and the conventional belief of heat transfer disappears.

In nanoscale, the thermal conductivity of homogeneous material can be calculated by three different techniques of molecular dynamics. (1) Equilibrium molecular dynamics with the Green-Kubo's formula, (2) Non-equilibrium molecular dynamics (NEMD) developed by Evens and (3) Non-equilibrium molecular dynamics with direct temperature differences [3]. Cahill et al. critically reviewed the studies about the thermal transport in nanostructures. Although this work focused on electronic devices, it gives a brief understanding about thermal transport [4]. Mirmira et al. summarized the studies related to thermal conductivity of thin films in nano scale [5]. There are many thermal conductivity models for thin films [6]. Berber et al combined the equilibrium and non equilibrium molecular dynamic simulations and found a thermal conductivity of a 6600 W/mK, which is unusual [7]. In macro modeling, many researchers have discussed heat behavior either by using analytical [8] or numerical modeling [9]. Springer and Tsai used a theoretical approach to find out the transverse thermal conductivity by using shear loading analogy and square packing array [10]. In another analytical study, Rayleigh and Maxwell's theories were modified by Hasselman and Johnson [11]. In their approach, they assumed a continuous behavior and derived relations for a single coated fiber, which is surrounded by matrix. There is no experimental comparison of these approaches. One of the important factors to understand the difference between nano and macro structures is interface effect. To support this, it is necessary to obtain either experimental or simulation results. It seems from the studies that much of the information could be gathered from the simulations because of the lack of analytical calculations. There is still no clear explanation to make the interface clear in minds. What do we see at the interface? What kind of bonding could appear? What kind of topology, transport across it, deformation, chemical activities, and forces also could appear?

Van Assche and Van Mele discussed the effect of the interphase. They emphasized that macroscopic properties of composite materials, including fiber-reinforced polymers, blends, and multilayer systems, were often strongly affected by the development of interphase regions with properties differing from the properties of the constituent materials. Interphases could arise due to preferential adsorption, catalytic influences of a surface, inter-diffusion, phase separation, etc. The resulting gradients in composition (polymer blends) or crosslink density (thermosets) lead to gradients in the microscopic properties[12]

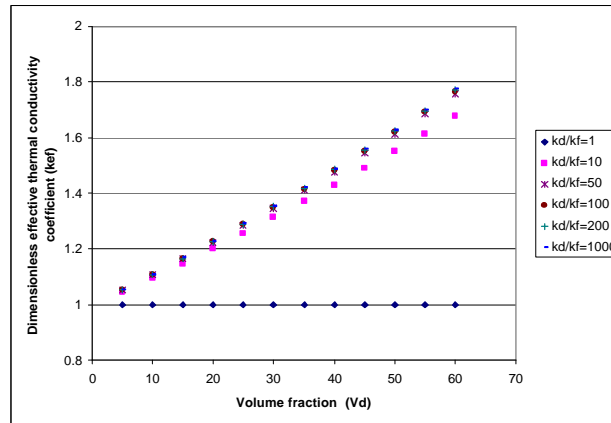


Figure 9. Effective thermal conductivity by using thermal resistance model

When the effect of volume fraction on thermal conductivity is compared in Maxwell and Lewis-Nielsen models, it is seen that Lewis- Nielsen method gives more accurate results when the filler ratio increases in the composite. Our model gave almost a linear relationship compared to the other models.

Computer Modeling

The problem consists of two substances: the fiber and the particle. Both of them have their thermal conductivity coefficients and so the aim is to get an efficient thermal conductivity coefficient. In order to avoid nano size effect and to provide a continuous modeling, we choose the size that finite element analysis can be applied.

In modeling part, we compared analytical and finite element models. Finite element modeling was done by using ANSYS7.0. As the element type, we used SOLID90, which allows us to model cylindrical shape. The constant heat flux was applied as 50 kW/m². Meshing was done automatically and the symmetry was used to avoid memory effect (Figure 10).

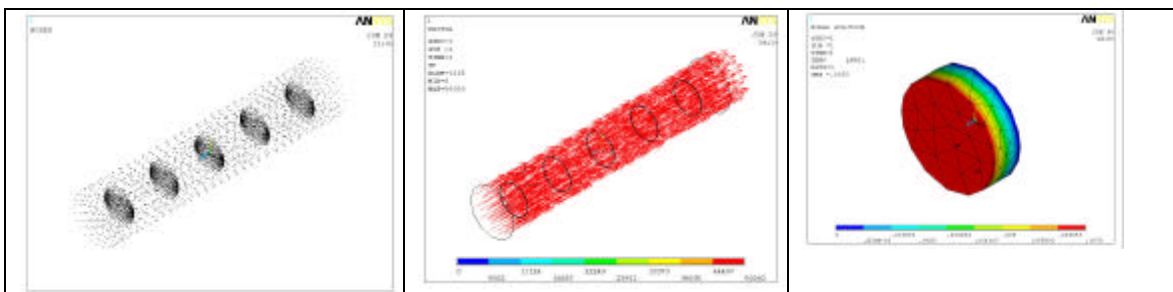


Figure 10. Heat conduction model for nano-filled composite fiber

Conclusions and future considerations

Conclusions

Nylon 6/OMMT nanocomposite fibers constantly yielded a residue of 4.5 -5% unlike neat fiber, which may be because of distribution of nanoclay platelets in the fiber. SEM images of nanocomposite fiber cross section showed the presence of nanoclay and its statistical distribution in the fiber. PP/silica nanocomposite fibers showed thermal decomposition temperature (temperature at 5% weight loss) about 20°C higher than that of neat polypropylene fibers. Also by the addition of grafted polypropylene the amount of residue increased indicating a possible chemical interaction between polymer and silica.

Future Considerations

We are investigating the orientation of nanoparticles in the polymer matrix and exploring the morphological influence of composite fiber microstructure on their fire retardancy. Experiments are underway to determine the filler migration and thermal conductivity effect of composite fibers on its over all flammability. We hired one more graduate student (Yue Huo) to work along these directions.

Acknowledgement

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