

Alignment of Si₃N₄ nanorods in Polypropylene single fibers

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ABSTRACT

In the present investigation, we have aligned the Si₃N₄ nanorods in the polypropylene (PP) fibers through extrusion process to fabricate PNCs. The alignment of Si₃N₄ in polypropylene/Si₃N₄ nanocomposite filaments has been carried out in a systematic manner to produce uniform monofilaments. The nanocomposite filaments were first uniformly stretched and stabilized using a two-set godet machine. The as extruded fibers were tested for their thermal and mechanical properties. The results of nanocomposite polymer filaments were compared with neat polymer filaments. These results clearly show that the nanocomposite polymer filaments are much (307 %) higher in tensile strength and modulus (>1000%) as compared to the neat polymer filaments

Keywords: polypropylene, silicon nitride, nanocomposite fibers, mechanical properties

1 INTRODUCTION

Polymer nanocomposites (PNCs) have garnered great academic and industrial interest since their inception [1-6]. PNCs have been shown to enhance physical, thermomechanical, and processing characteristics compared to their neat polymer counterparts. These nanocomposites can be tailored according to the applications and requirements by selection of nanoparticles morphology and compatible polymer matrix. Nanostructured silicon nitride particles exhibit a high-potential for the reinforcement of polymers [4,7-10]. These covalently bonded material in its larger form exhibits high strength and high toughness. It has been used in many industrial applications, such as engine components, bearings and cutting tools [11,12]. Polypropylene fibers have been widely used in apparel, upholstery, floor coverings, hygiene medical, geotextiles, car industry, automotive textiles, various home textiles, wall-coverings [13].

A significant progress has been made in dispersion of acicular or spherical nanoparticles in polymer matrix by surface modification and other techniques. While alignment of acicular or rod shape particles in polymeric fiber and manufacturing advanced macroscopic structures remain a challenge. Most commonly use techniques for alignment are wet spinning [14], magnetic alignment [15] electrospinning [16] and melt processing [17,18]. To align

the nanoparticles while extruding, many aspects should be considered. Such as the starting materials, chemical compositions, mixing techniques, type of extruder, material loading process, extrusion temperature, material residence time within the extruder, the die and its orifice shape and size, extrusion rate, extrusion direction, surrounding air temperature and its speed, fibers cooling type and process, filament draw ratio and winding speed. Proper combination of such factors leads to the production of fine fibers.

In the recent years, much attention has been paid to the synthesis of different morphologies of compounds of silicon nitride nanoparticles. The spherical and rod shaped morphologies are studied extensively among them. Our present interest deals with the study of rod-silicon nitride nanoparticles along with PP. Both neat and nanoparticles infused PP single fibers were produced using a single screw extrusion technique. Morphological and mechanical properties are presented in this paper.

2 EXPERIMENTAL

Materials

The Si₃N₄ nanorods (light gray color, Si₃N₄ alpha, 99%, Size 100X800 nm) and spherical shaped (white color, Si₃N₄ amorphous, 98.5+%, Size 15-30 nm) particles were obtained from *Nanostructured & amorphous Materials, Inc.* Polypropylene (Microthene FP-800-00 microfine powder ~20 µm, melt flow rate 35g/10min and density ~ 0.909 g/cc) powder was purchased from Equisart Chemicals, LP, USA.

Rod shaped Si₃N₄ particles and PP powder were carefully measured in the ratio of 1:99 by weight (1wt%) and dry mixed using a mechanical blender at a speed of 2300 rpm with a circulating cold base container (5°C). The mixing process was paused for 6 minutes after 3 minutes of continuous mixing and then resumed for further mixing. This was done to avoid temperature rise, which may result in softening the PP. This technique is repeated 20 to 30 times until uniform mixing was observed. The effective time for which the mixture was blended was between 75 to 90 minutes. At this time, the mixture took a light gray appearance and it seemed that the rod Si₃N₄ particles were thoroughly mixed with the PP.

The mixture of PP and rod-Si₃N₄ nanoparticles was then further air dried in a dryer for 12 h and extruded using a *Wayne Yellow Label Table Top single screw extruder*.

3 RESULTS AND DISCUSSION

Tensile tests were carried out to study the effect of alignment of rod-Si₃N₄ particles in PP polymer on tensile strength and modulus. The single fiber tests were carried out according to the ASTM standard D 3379-75 [31] using Zwick/Roell tensile testing equipment with a 20 N load cell. Test specimens were cut each of fibers were aligned to the two ceramic grips, with a distance of separation of 102mm (gauge length). Tensile test results are shown in Figure 1. The ultimate tensile strength of 1wt% rod-Si₃N₄/ and 2wt% rod-Si₃N₄ in PP is 249.48 MPa, and 307.21 MPa respectively. Where as the neat PP is 63.58MPa. These results show that the ultimate tensile strength is increased by 383% as compared to that of neat PP.

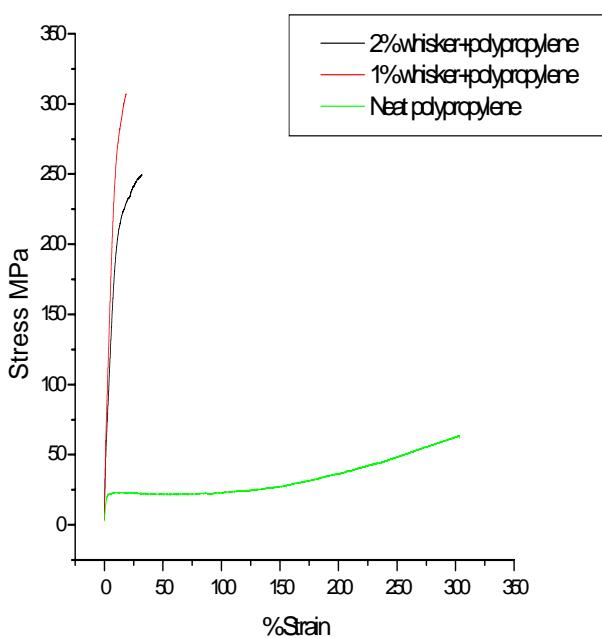


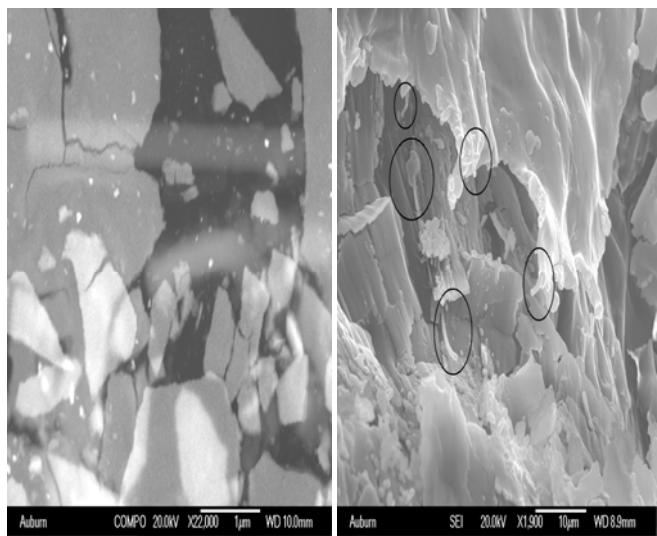
Figure 1. Tensile response of 1wt% and 2wt% of rod-Si₃N₄ Polypropylene and neat polypropylene filaments

These significant increments in tensile properties are assigned to the alignment and exfoliation of rod-Si₃N₄ particles in PP polymer. The tensile modulus was also calculated from the stress vs. strain curve of neat PP, 1wt%, and 2wt% of rod-Si₃N₄ are 0.49GPa, 4.03GPa and 5.03GPa respectively. These significant improvements of tensile

modulus were attributed to the infusion of high strength Si₃N₄ nanoparticles and their alignment in extrusion direction of PP polymer fibers. This strength/modulus of nanocomposite fiber (307.21MPa/ 5.03.4GPa) is more than that of the neat PP (63.58MPa/0.49GPa) which indicates the exceptional load bearing capability of Si₃N₄ and their potential applications in structural composite materials. When rod-Si₃N₄ nanoparticles are increase in strength and modulus of the PP at the cost of a loss in ultimate PP polymer strain.

Morphological Studies

FESEM (*JEOL FE-JSM7000F*) studies have been carried out to study the alignment of rod-Si₃N₄ in PP fibers. To corroborate the alignment of the Si₃N₄ rods in PP matrix the polymer was etched and coated with gold palladium before the FE-SEM analysis. Etching of the specimens was carried out in a HUMMER 6.2 plasma etching system with aluminum target. The etching process was done in the argon atmosphere for a span of 1 hour. The vacuum was maintained between 60to 80 millitorr and the current flow was maintained between 10 to 15 Amps. The samples for FE-SEM experiments were prepared by etching the as extruded rod-Si₃N₄/PP fibers in argon gas for 30 minutes. Due to the etching most of the polymer that covered the matrix layer facing the target was etched out and the Si₃N₄ rods were clearly seen in the FE-SEM. The Figure 2 (a)



(a) (b)

Figure 2. SEM micrographs of (a) aligned silicon nitride nanoparticles in the polypropylene matrix and b) particles pullout from the polymer matrix

very clearly shows that the rod shaped Si₃N₄ particles are well aligned in the fiber extrusion direction. These Si₃N₄ particles are surfaced only after deep etching of the surface polymer. Such alignment of the rod shaped particles proves the ability of the extrusion method to align the particles in the direction of extrusion. To find out the alignment of rod-Si₃N₄ particles deep in the fiber a higher magnification FE-SEM was carried out. Figures 2(b) micrograph shows the rod-Si₃N₄ particles are pulled out after tensile testing. These rod-Si₃N₄ particles are aligned in the fiber in different layers one below the other layers. The micrograph clearly shows that the rod shaped particles are aligned in the various levels of the fiber thickness. This phenomenon plays the leading role in enhancing the mechanical properties of composite fibers.

4 CONCLUSIONS

We have successfully aligned the silicon nitride nanorods in polypropylene single filaments through melt extrusion process. Significant enhancement in tensile properties has been observed for 2wt% rod shaped Si₃N₄ particles. The tensile results clearly show that the nanocomposite polymer filaments are much (307 %) higher in tensile strength and modulus (>1000%) as compared to the neat polymer filaments. The FE-SEM study proves that the rod shaped particles can be successfully aligned in the extrusion direction. These results are consistence with our previous studies on rod- Si₃N₄ nanoparticles infused in Nylon-6 polymers and CNTs infused Nylon6-polymer filaments.

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