POLYAMIDE 11-CARBON NANOTUBES NANOCOMPOSITES: PROCESSING, MORPHOLOGICAL, AND PROPERTY CHARACTERIZATION

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Abstract

The objective of this research is to develop a multifunctional polyamide 11 (PA11) polymer with enhanced electrical, thermal, mechanical, and flammability properties for selective laser sintering (SLS) rapid manufacturing through the use of nanotechnology. In the present study, a nanophase was introduced into PA11 powder via rotation & revolution and twin screw extrusion techniques to disperse the MWNTs in the PA11 pellet. Arkema Rilsan® polyamide 11 molding polymer pellets were used with 1, 3, 5, and 7 wt% loadings of Arkema’s Graphistrength™ multi-wall carbon nanotubes (MWNTs) to create a family of PA11-MWNT nanocomposites using twin screw extrusion technique. Arkema Rilsan® polyamide 11 powders were blended with 5 wt% loading of Graphistrength™ MWNTs using the rotation & revolution technique to create another set of PA11-MWNT nanocomposites.

Transmission electron microscopy and scanning electron microscopy were used to determine the degree and uniformity of dispersion of MWNTs in the PA11 polymer. Injection and compression molded test specimens were fabricated for physical, electrical, thermal, mechanical, and flammability properties characterization. Thermal stability of these polyamide 11-MWNT nanocomposites was examined by TGA. Kinetic parameters were calculated using isoconversion technique. Electrical properties were measured using ASTM D257. Mechanical properties such as ultimate tensile strength, rupture tensile strength, and elongation at rupture were measured. Flammability properties were obtained using the UL 94 test method. All these different test methods and subsequent polymer characteristics are discussed in this paper.

1. Introduction

Rapid Prototyping (RP) technology has allowed the product designer access to a vast arsenal of tools and technologies unprecedented in the history of manufacturing. The phenomenal pace of desktop computing with the market availability of 3D software and
the development of RP technology allows product design to engage more freely with new ideas, to produce prototypes, and to carry out testing in a shorter time. RP has received much attention in recent years and has been embraced as a preferred tool for not only product development but, in some cases, "just-in-time manufacturing."

There are several different RP platforms, of which selective laser sintering (SLS) is the one that provides the widest commercial material capabilities [1-5]. The development of RP, defined as the creation of a 3D part directly from computer aided design (CAD) data without special tooling, has generated interest in moving to the next step, where fully functional parts could be created in the same way. The concept is widely known as rapid manufacturing (RM). A key objective in transitioning RM is the creation of an SLS RM capability for high-performance materials by leveraging existing commercial RP machines.

In recent years an emerging vision of RM has occurred within the SLS industry. The SLS method involves the fabrication of a part by building up layers of powder, whereby each thin layer (0.003” to 0.006”) is added across a part build chamber as a computer controlled laser fuses particles in a layer region together with the previously fused regions. A three-dimensional (3D) computer aided design (CAD) file is sliced into layers with each of those layers becoming one of the fused regions. In this way, a 3D part is built from successive layers of small, uniform particles. The SLS process has been refined to support greater accuracy and duplication, which sustains a modest array of manufacturing applications, such as ventilation ducts for aircraft crew. Yet these advances have been confined to special nylon (polyamide) materials as the precursor material used in SLS. In other words, the current embodiment of RM represents a small segment of utility, since RM has been optimized for a narrow group of materials [nylon (polyamide) 11 and nylon 12]. RM has the capability for greater penetration in many market areas, especially the high performance advanced materials areas encompassing military and aerospace structures.

Direct manufacturing successes include Boeing’s use of PA11 SLS powder to manufacture air ducting systems for F18 fighter jets [5] and Siemen’s use of PA12 SLS powder to manufacture hearing aid shells to fit individual user’s ears. PA11 parts produced by Boeing’s SLS technology are directly used in aerospace structures that meet stringent aerospace performance requirements, in contrast with prototypes or other designs typically associated with RM processes. These parts are primarily environmental control system ducts (ECS).

Commercially available polymers for SLS include PA11, PA12, and glass-filled PA12 from 3D Systems. The PA12 materials are typically used for design verification models and for limited functional prototypes. The Boeing Company has developed laser sintering techniques using PA11 powders and launched a manufacturing business [On Demand Manufacturing, Inc. (ODM), Camarillo, CA. ODM is now owned by RBM Products, Inc., Fountain, CO] to support large-scale production of functional parts for military aircraft. PA11 or PA12 materials are limited to a use temperature of about 71ºC with brief excursions to 121ºC, are flammable, and possess mediocre modulus and strength compared to high-performance thermoplastics such as PEEK.
The introduction of selected nanoparticles, such as carbon nanofibers (CNFs) and MMT nanoclay into PA11 or PA12 by compounding, has resulted in a family of intumescent polyamide nanocomposites. Small amounts of nanoparticles (<7%) are required to make polymer nanocomposites (PNCs) exhibit enhanced flame retardant (FR) properties when compared with the modified thermoplastic processed by conventional methods [6-17]. However, unlike the conventional FR thermoplastics, the resulting PNCs exhibit enhanced mechanical properties, such as high strength/modulus, moisture resistance, higher heat deflection temperature, etc. Therefore, nanotechnology can be considered as a unique technique to develop novel FR thermoplastic structural components with high performance characteristics.

Our previous studies showed that PA11-clay nanocomposites and PA11-CNF nanocomposites exhibited better mechanical, flammability, and thermal properties than neat PA11 polymer [18,19]. SLS parts of some of these PA11 nanocomposites have been fabricated successfully; however some fabricated formulations did not exhibit optimal properties that are necessary for the intended application [19]. Our present study is expanded to include the use of MWNT [20] in PA11 polymer and expected to result in superior property characteristics due to the higher aspect ratio of MWNT compared to CNF. Pending the outcome of examining different wt% of MWNT in PA11 by twin screw extrusion and injection molding, a few, selected formulations will be chosen for SLS fabrication and complete characterization.

The PA11-MWNT masterbatch (containing 20wt% MWNT) provided by Arkema was diluted with Rilsan® BMNO PA11 to 1, 3, 5, and 7% using twin-screw extrusion. The resulting pellets were injection molded into different test specimens. The extent and uniformity of dispersion of MWNT was examined by transmission electron microscopy (TEM). Flammability properties were studied by conducting UL 94 tests. Thermal properties were analyzed by thermogravimetric analysis (TGA). Tensile properties were obtained from the stress-strain behavior of the specimens and measured by automated tensile testing system. Another set of PA11-MWNT nanocomposites was prepared by mixing the neat PA11 powder with 5wt% MWNT using rotation & revolution technique with grinding media of different sizes. Thin films were fabricated by compression molding the resulting powder mixtures for electrical conductivity measurement.

2. Experimental

2.1 Materials

**Polymer Resin** Arkema’s Rilsan® BMNO PA11 was used. Rilsan® PA11 is one of the few polymers that are produced from ‘green’ raw materials – castor beans. It has earned a preferred material status in some demanding applications, due largely to its unique combination of thermal, physical, chemical, mechanical properties, and ease of processing. This BMNO grade of PA11 possesses very similar properties to the grade of PCG LV that was used in previous studies [18,19], but it has higher a viscosity than PCG LV and duplicates the one used in the PA11-MWNT masterbatch.

**Nanoparticle** Arkema’s Graphistrength™ C100 MWNT has outstanding mechanical properties combined with electrical and thermal conductivities. It can be used in a variety of applications, such as high-strength thermosetting composites [20]. No surface
treatment/functionalization was performed on the C100 MWNT. It is available in its natural black powder form with an apparent density of 50 to 150 kg/m$^3$ and a mean agglomerate size of 200 to 500 µm. The C100 MWNT has a mean number of walls of 5 to 15, an outer mean diameter of 10 to 15 nm, and mean lengths of 0.1 to 10 µm. This C100 MWNT is used in the preparation of the PA11-MWNT masterbatch.

**Polymer-MWNT Masterbatch** Arkema’s PA11-20wt% MWNT masterbatch was supplied by Arkema. Arkema’s Graphistrength C100 MWNT [20] was melt-blended into Rilsan® BMNO PA11 via twin-screw extrusion to obtain the masterbatch.

2.2 Measurements

**Morphological Microstructures Analysis** The cross-sections of the PA11 nanocomposites were investigated by TEM to examine the dispersion of MWNTs in the PA11 polymer matrix. Uniform distribution of the nanoparticles within the polymer matrix is essential to yield the best enhancement of material properties of the polymer blend. SEM was used to examine the morphology of PA11-MWNT powder mixtures.

**Mechanical Testing** In order to compare the mechanical properties of PA11 nanocomposites with the baseline material, stress-strain behavior was examined using an automated tensile testing system (Instron model 3345) in accordance with ASTM D638. Ultimate tensile strength, rupture tensile strength, tensile modulus, and tensile elongation at rupture were obtained from the stress-strain data.

**Thermal Stability Testing** Thermal stability of the PA11 baseline and PA11-MWNT nanocomposites were examined by thermogravimetric analysis (TGA) using the Perkin Elmer TGA 7. Weight changes in sample materials are measured as function of temperature or time in TGA. The sample is heated by a furnace with a nitrogen atmosphere, while the loss or gain of sample weight is monitored by a sensitive balance. Weight, temperature, and furnace calibrations were carried out within the range of the TGA (100-900°C) at scan rates of 10°C/min and 20°C/min.

**Flammability Testing** UL 94 is a standard test for flammability of plastic materials in industry that serves as a preliminary indication of plastics acceptability for use as a component of a device or appliance with respect to its flammability behavior [21]. UL 94 is not intended to reflect the hazards of a material under actual fire conditions but is considered as a preliminary step toward obtaining plastic recognition and subsequent listing in the “Plastics Recognized Component Directory” (formerly known as “Yellow Cards”). The materials are tested in a vertical setting to determine the UL 94 V-0 rating. Five specimens are tested for each formulation.

**Electrical Conductivity** Electrical sheet resistivity of the polymer nanocomposites was measured using a four-point probe setup. The results were normalized by the thickness of the thin film specimens to obtain the volume resistivity. Electrical conductivity is the inverse of the volume resistivity.
3. Results and Discussion

3.1 Processing and Characterization of Polymer Nanocomposites

PA11-MWNT masterbatch was diluted to 1, 3, 5, and 7% using twin screw extrusion. A 30mm Werner Pfleider corotating twin screw extruder that is configured for a wide variety of materials was used. The extruder L/D can be varied from 21 to 48, with options of multiple feeds and vents. The energy profile of the screw is adjusted to optimally meet the needs of the target product. Approximately 4.6 kg (10 lbs) of each formulation were produced. Separate volumetric feeders were used for the masterbatch and the base resin. The PA11 was dried in a desiccant drier before compounding. Injection molded specimens of each blend were prepared and examined by TEM. Figures 1 and 2 show the TEM micrographs of PA11 with 20% MWNT and PA11 with 7% MWNT, respectively. MWNTs seem well dispersed in the polymer matrix, although some aggregates of MWNTs of about 200nm in size were still observed. It is believed that better dispersion can be achieved by using optimized processing procedures or screw profiles. Since individual MWNTs are observed throughout the TEM scanned specimens, these MWNTs appear to be well compatible with the PA11, even though there no MWNT surface treatment/functionalization was applied.

![Figure 1. TEM micrographs of polyamide 11 with 20% MWNT (PA11-MWNT masterbatch).](image1)

![Figure 2. TEM micrographs of polyamide 11 with 7% MWNT.](image2)

3.2 Mechanical Properties of the Polymer Nanocomposites
Ultimate tensile strength, rupture tensile strength, tensile modulus, and tensile elongation at rupture of the PA11-MWNT nanocomposites are shown in Figures 3 through 6, respectively. Samples with 5 wt% MWNT were delayed in the extrusion sequence and not included in the present study. Table 1 shows a summary of the mechanical properties of the PA11 and PA11-MWNT nanocomposites. A general trend is observed and indicates that the ultimate tensile strength, rupture tensile strength, and tensile modulus properties increase as the loading of MWNT increases. The ultimate tensile strength of PA11 neat and PA11-7%MWNT are 46±2 and 53±5 MPa, respectively. The rupture tensile strength of PA11 neat and PA11-7%MWNT are 34±4 and 53±5 MPa, respectively. The largest enhancement occurred for the tensile modulus of PA11 neat compared to PA11-7%MWNT: 1,284±28 and 1,669±45 MPa, respectively. There is a substantial decrease in tensile elongation at rupture of PA11 neat versus PA11-MWNT, from 177±74 down to 6±2%. This observation is similar to our previous studies of PA11 with other nanoparticles (e.g. clay, CNF, etc.) [22].

Figure 3. Ultimate tensile strength of PA11 MWNT nanocomposites.
Figure 4. Rupture tensile strength of PA11 MWNT nanocomposites.

Figure 5. Tensile modulus of PA11 MWNT nanocomposites.

Figure 6. Tensile elongation at rupture of PA11 MWNT nanocomposites.

Table 1 Mechanical Properties of PA11-MWNT Nanocomposites

<table>
<thead>
<tr>
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<th>Ultimate Tensile Strength (MPa)</th>
<th>Rupture Tensile Strength (MPa)</th>
<th>Tensile Modulus (MPa)</th>
<th>Tensile Elong. at Rupture (strain%)</th>
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<tbody>
<tr>
<td>Ave</td>
<td>Std Dev</td>
<td>Ave</td>
<td>Std Dev</td>
<td>Ave</td>
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<tr>
<td>PA11 neat</td>
<td>46</td>
<td>2</td>
<td>34</td>
<td>4</td>
</tr>
<tr>
<td>PA11+1%MWNT</td>
<td>47</td>
<td>5</td>
<td>35</td>
<td>5</td>
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<td>52</td>
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<td>39</td>
<td>2</td>
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<tr>
<td>PA11+7%MWNT</td>
<td>53</td>
<td>5</td>
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</table>
3.3 Thermal Stability of the Polymer Nanocomposites

TGA was performed on all six formulations under nitrogen using a scan rate of 10°C/min (Figure 7). A general trend of increased thermal stability is observed (higher decomposition temperatures) as the amount of MWNT is increased. Table 2 summarizes the 10% mass loss, onset, and 50% mass loss decomposition temperatures of the PA11-MWNT nanocomposites. The 10% mass loss, onset, and 50% mass loss decomposition temperatures of PA11-7%MWNT nanocomposite increase significantly versus the neat PA11, from 418° to 440°C, 425° to 457°C, and 439° to 475°C, respectively.

![Figure 7. Thermogravimetric analysis of PA11 MWNT nanocomposites at 10°C/min in nitrogen.](image)

<table>
<thead>
<tr>
<th>Polymer Blend</th>
<th>Decomposition Temperatures (°C)</th>
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<tr>
<td></td>
<td>at 10% mass loss</td>
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<tr>
<td>PA11 neat</td>
<td>418</td>
</tr>
<tr>
<td>PA11+1% MWNT</td>
<td>431</td>
</tr>
<tr>
<td>PA11+3% MWNT</td>
<td>437</td>
</tr>
<tr>
<td>PA11+5% MWNT</td>
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<tr>
<td>PA11+7% MWNT</td>
<td>440</td>
</tr>
<tr>
<td>PA11+20% MWNT</td>
<td>456</td>
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</table>
3.4 Flammability Properties of the Polymer Nanocomposites

The materials were tested “as received with no additional conditioning/drying” prior to UL 94 testing. The test was performed in our lab with the UL 94 testing requirements and procedures followed as stringently as possible. Our lab is not certified for UL 94; the results serve as a screening tool. Five specimens were tested for each formulation (the one with 5% MWNT was lacking). The testing was performed in a fume hood with a preset airflow of 90-105 ft/min. The fume hood sash was pulled down as much as possible to prevent airflow from the outside environment. The erratic exhaust airflow from the fume hood and the surrounding area made the reproducibility of the testing challenging. The burner is lit during the time of this experiment to keep the applied flame constant between each specimen.

Figure 8 shows photos of specimens before and after the test while Figure 9 shows a burning specimen during the test. All formulation specimens with MWNTs burned longer than 100 seconds after the first flame was applied and continued to burn. Therefore, none of the formulations passed any rating of the UL 94 test. However, it should be noted that the PA11-MWNT nanocomposites did not exhibit any dripping when they were burned, while the neat PA11 dripped copiously during the test. This indicates the enhanced char forming characteristics of PA11-MWNT compared to neat PA11.
Figure 9. Flame burned all the way to the clamp in the UL 94 test.

3.5 Electrical Resistivity of the Polymer Nanocomposites

Specimens were pressed into thin films using a heated hydraulic press. Figure 10 shows the volume resistivity of PA11-MWNTs nanocomposites prepared via twin screw extrusion and planetary mixing.

![Volume Resistivity of PA11-MWNT Nanocomposites](image)

**Figure 10.** Volume resistivity of PA11-MWNT nanocomposites compounded by twin-screw extrusion and planetary mixer (Thinky mixer).

The resistivity of the neat PA11 was $10^{13}$ Ohms-cm. By incorporating 1wt% of MWNT, the resistivity decreased by three orders of magnitude, which was close to the requirement of ESD. In general, the volume resistivity decreased with increasing amount of MWNTs; thus formulations with higher weight loadings of MWNTs achieved the ESD requirement. The volume resistivity of PA11-20%MWNT masterbatch even achieved the requirement for EMI. However, it might not be practical because the amount of MWNT used is too high, thus the viscosity of the nanocomposites might be too high for processing. Furthermore, the planetary mixer prepared samples do not seem as effective as their twin-screw extruded counterparts. Selected samples from both methods will be further evaluated by SLS fabrication.

3.6 Morphology of PA11-MWNT Powder Mixtures

As-received MWNT were de-bulked by the Thinky NP-100 Pulverizer using grinding media of different sizes, (a) 5mm, (b) 10mm, and (c) 20mm, respectively, in order to investigate the effect of different media sizes on the pulverizing process. The de-bulked MWNT was then mixed with PA11 powder using the Thinky ARE-310 mixer. The PA11
was mixed in gradually in three steps of 30g, 30g, and 35g, to 5g of MWNT, each time spinning at 2,000rpm for 10 seconds, resulting in 5wt% PA11-MWNT mixtures. SEM was used to examine the distribution of the nanotubes on the PA11 particles of the resulting mixtures (A, B, and C). The microanalysis along with thermal study would serve as a screening test to select the best pulverizing procedure among the three used. PA11-MWNT mixtures of different weight loadings of MWNT could then be made using MWNTs prepared by this procedure for further characterization. Figures 11 to 12 show the SEM images of neat PA11 and the PA11-MWNT mixtures at 250X, 10kX and 50kX magnification.

From Figure 11b-d, MWNT clusters were observed attaching to the PA11 particles. There seemed to be a higher concentration of MWNT clusters in mixtures B and C. In Figure 12, the MWNTs can be observed on the surface of an individual PA11 particle. From simple observation, it appeared that mixture B had a slightly thicker layer of MWNTs on the surface of PA11 particles. In Figure 13, contrary to the other micrographs, it seems that at this magnification it was mixture A that had a little higher concentration of MWNTs on the surface.

In conclusion, it was difficult to determine any one mixture as the “best” based on the SEM micrographs. In all the blends, the MWNTs were dispersed on the surface of the PA11 particles quite evenly, as seen in Figure 13. The only difference was in the concentration of pure MWNT aggregates seen at 250x magnification. There was a higher amount of clusters observed in mixtures B and C. Based on this observation, mixture A would probably be the best choice, since it showed a better distribution of MWNT particles among the PA11 powder, with less MWNT clusters.
Figure 1. SEM micrographs taken at 250X magnification of (a) neat PA11, (b) PA11+MWNT-a, (c) PA11+MWNT-b, (d) PA11+MWNT-c.

Figure 12. SEM micrographs taken at 10kX magnification of (a) PA11+MWNT-a, (b) PA11+MWNT-b, (c) PA11+MWNT-c. Note that there are no higher magnification micrographs of the neat PA11 powder, since increasing the magnification made the beam focus more strongly on the sample, causing it to melt and making clear images impossible.
Figure 13. SEM micrographs taken at 50kX magnification of (a) PA11+MWNT-a, (b) PA11+MWNT-b, and (c) PA11+MWNT-c.

3.7 Thermal Analysis of PA11-MWNT Films from Powder Mixtures

The thermal stability of the PA11-MWNT mixtures was studied using TGA. The mixtures, as well as the neat PA11 powder, were first pressed into thin films using a heated press. A small amount of mixture was placed between aluminum foil sheets coated with 3% silicone spray mold release, and then compression molded between two 200°C plates at 5,000psi for 10 minutes. The films were then analyzed using TGA with heating rates of 10°C/min and 20°C/min in nitrogen. Figures 14 and 15 show the TGA results while Table 3 shows the decomposition temperatures of the PA11-MWNT films. In both cases, Film B had the best thermal stability and decomposition temperatures compared to the neat PA11. Film A showed the next best improved thermal stability while Film C showed very little improvement, remaining approximately the same decomposition temperatures as the neat material. Therefore, mixture B was recommended for the next round of study, since the MWNT distributions in all three mixtures were about the same according to the SEM micrographs.
Table 3. Decomposition Temperatures of MWNT Reinforced PA11 Films (TGA).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Decomp. Temp. at 10% Mass Loss</th>
<th>Decomp. Temp. at 50% Mass Loss</th>
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<tr>
<td></td>
<td>10°C/min</td>
<td>20°C/min</td>
</tr>
<tr>
<td>Neat PA11</td>
<td>404</td>
<td>418</td>
</tr>
<tr>
<td>A film</td>
<td>409</td>
<td>427</td>
</tr>
<tr>
<td>B film</td>
<td>415</td>
<td>430</td>
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<tr>
<td>C film</td>
<td>403</td>
<td>419</td>
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</table>

Figure 14. Thermogravimetric analysis of PA11-MWNT films at 10°C/min in nitrogen.
3.8 Electrical Resistivity of PA11-MWNT Films from Powder Mixtures

The powder mixtures of PA11-MWNT processed by Thinky mixer were also pressed into thin films using a heated hydraulic press. The volume resistivity was measured by the four-point probe setup. Figure 10 shows that, in general, the conductivity of these formulations were enhanced as the amount of MWNT increased. An exception was the PA11-1wt%MWNT. This could be due to poor sample preparation since air bubbles were observed on the film specimens. More investigation is needed to verify this. It was found that PA11-7wt%MWNT passed the ESD requirement. This formulation of the powder mixture method will be chosen for further SLS fabrication.

4. Summary and Conclusions

A total of five formulations of PA11-MWNT nanocomposites were compounded via twin screw extrusion. Injection molded specimen of PA11 baseline and PA11-MWNT nanocomposites were fabricated for physical, mechanical, thermal, flammability, electrical properties measurements. TEM showed that MWNTs were well dispersed into the PA11 matrix, although it is possible that better dispersion can be achievable by using a more aggressive screw design. MWNTs enhanced the mechanical (except tensile elongation at rupture), thermal stability, and electrical conductivity properties of the PA11. However, all PA11-MWNT nanocomposites failed the UL 94 test. To make them truly flame retardant, a second additive (such as conventional intumescent flame retardant additives) to act as a synergist is necessary. It has been demonstrated that thermal and flammability can be enhanced substantially as long as synergism is established between the nanoparticles and conventional flame retardant additive in the polyamide matrix [22]. Furthermore, to improve the tensile elongation at rupture, an elastomeric component may be required. If, indeed, an elastomeric component is introduced, then its effect on the corresponding mechanical, thermal, and flammability properties must be determined.

The other set of PA11-MWNT nanocomposites were fabricated by mixing PA11 powder with pulverized MWNT using a Thinky mixer. A network of MWNT, which could serve as a conductive path, should be formed after MWNT-coated PA11 powder was compression molded into specimens. Comparing the two sets of volume resistivity data, however, the set processed by twin-screw extrusion had much better electrical conductivity values than the set processed by the planetary mixer. Select samples of the twin screw extruded and the planetary mixed PA11-MWNT will be further studied in the SLS process.

5. References


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masterbatch, neat PA11, and MWNT for this study; and Thinky Corp. for mixing PA11-MWNT powders using their rotation & revolution mixer.