

## IMPACT MODIFICATION OF NYLON-6

### I. Object

Non-plasticized Nylon-6, poly( $\epsilon$ -caprolactam), has a glass transition temperature,  $T_g$  near 73 °C, based on dynamic mechanical property analysis at 1 Hz. Partly for this reason, Nylon-6 can fail in a brittle manner when rapidly stressed at room temperature. Nylon-6 can be "impact modified" by the addition of a rubber phase through melt compounding in an extruder. In addition to improved impact strength, the resulting three phase blend (a crystalline Nylon-6 phase, an amorphous Nylon-6 phase, and an amorphous rubber phase) has reduced modulus and heat distortion, relative to pure Nylon-6.

To illustrate the modifications to physical properties that result from addition of a rubber phase, blends containing several rubber contents will be prepared and tested. The importance of interfacial adhesion and surface energy will be investigated by comparing the properties of a blend containing chemically-grafted rubber with those of a blend where no grafting has occurred.

### II. References

1. A. J. Oshinski, Rubber Toughening of Polyamides using Functionalized Block Copolymers, Ph.D. Dissertation, The University of Texas at Austin, 1990.
2. D. R. Paul and S. Newman, Polymer Blends, Vols. I and II, Academic Press: New York, 1978.
3. D. R. Paul, H. Keskkula, and J. W. Barlow, "Polymer Blends" in Kirk-Othmer Encyclopedia of Chemical Engineering, 4th Ed., Vol. 19, J. Wiley & Sons: New York, 1996, 837-881.
4. G. P. Shaver, Compatibilization of Immiscible Blends Through Chemical Reaction, M.S. Thesis, The University of Texas at Austin, 1989.

### III. Equipment and Supplies

#### Part 1. Preparation (1 period)

1. One-inch Killion single screw extruder with intensive mixing screw, cooling bath, and strand chopper (or equivalent).

2. Half ounce, Arburg screw injection molding machine with mold to prepare ASTM D-638 tensile and impact specimens.
3. Nylon-6, Allied Capron 873WP, or equivalent.
4. Impact Modifier, Exxon Exxelar 1803, EPR-g-MAH (1.14%).

#### **Part 2. Physical Property Testing (1 period)**

Tensile Testing Machine with 1000 lb load cell, Instron 1137 (MTI- Phoenix) or equivalent.

#### **Part 3. Impact Testing (1 period)**

1. T.M.I. Computerized Pedulum Impact Tester (CPE 3.460)
2. Izod Notch Forming Machine (CPE 3.460)
3. Dynatup Computerized Falling Weight Impact Tester (CPE 3.460)

### **IV. Experimental Procedures**

#### **Part 1. Blend Preparation**

1. Follow the general operating procedures for operating the extruder. Set all extruder zones to 240 °C (464 °F). Be sure to turn on the hopper cooling water.
2. Be sure that the Nylon-6 feed stock has been dried overnight at 80 °C and full vacuum prior to use. Remove the feed from the vacuum oven and place it in a convection oven, set at 80 °C. As you process material, remove small batches from the oven. Size the batches to minimize exposure to air to 20 min or less. This should prevent any significant resorption of moisture by the Nylon-6.
3. After extrusion, return the product to the convection oven in (2) above. Repeat the procedure in (2) when injection molding the product to minimize moisture sorption.
4. Set up the Arburg Injection Molding Machine. Useful initial parameters are:
  - a. Barrel temperature, all zones =240 °C
  - b. Injection Pressure =1500 psig
  - c. Holding Pressure = 500 psig
  - d. Holding time = 2 sec
  - e. Cooling time= 35 sec
  - f. Injection time= 7 sec
  - g. Screw speed= 300 RPM

h. Mold temperature = 70 °C

i. Dosage= 15.5-17.0 units

j. HOPPER AND HYDRAULIC COOLING WATER ON

5. While the machines are heating (this takes about 1/2 hour), prepare 1 Kg dry blends with the following compositions:

Blend Designation	Impact Modifier, phr
A	5
B	10
C	15
D	20

(phr is parts per hundred parts of base resin on a weight basis)

To prepare the blends, place appropriate amount of Impact modifier (50 g for blend A) in a zip-lock polyethylene bag, add 1 Kg of hot dry Nylon-6, then seal the bag. Dry mix by shaking the ingredients in the bag.

6. Extrusion blend each mix. A typical extruder screw speed for this operation is 35-50 RPM. Do not exceed 5 amps (continuous) under any circumstances. Either raise the processing temperature or reduce the screw speed to keep the armature current below 5 amps. Extrusion blend each batch by following each batch directly with the next without intermediate purge. The best procedure is to allow the extruder to "empty" itself. Then start the next batch. Allow about 5-8 min of extrusion of the new batch before beginning to save the material. This eliminates most of the transition material between batches. Remember to return each blended batch to the oven so that it remains dry, prior to molding in the injection molding machine.

7. When finished blending, purge the extruder with polyethylene. Shut down the system and sweep up the polymer pellets that have inevitably spilled.

8. After two batches have been blended, begin to injection mold what has been made. Obtain at least 10 tensile bars and 10 impact bars for each composition. Also mold a comparable number of non-modified Nylon-6 bars for later comparison. These bars should be labeled and saved in sealed polyethylene bags for testing over the next two periods.

9. Half of the bars may be boiled in water for at least 1 hour prior to testing to simulate "wet" Nylon-6. Weigh a few samples before and after boiling to determine water pick-up. The boiled samples can be stored in a "zip-lock" polyethylene bag prior to testing at room temperature. You may wish to return the dry specimens to the vacuum oven to ensure that they remain dry prior to test. Be sure to record the actual molding conditions and try to maintain the same conditions for all batches.

10. When molding is completed, power down the molding machine, the mold temperature control unit, valve off the cooling water, and clean the area of spilled pellets.

### **Part 2. Physical Property Testing (1 period)**

Your instructor will show you how to use the MTI Instron tensile testing machine to obtain room temperature tensile properties of the blends. Obtain stress vs. strain diagrams for at least five specimens from each batch. Use the testing program to obtain modulus, yield strength, ultimate strength, strain at yield, strain at break, and energy per unit volume to break for each blend. Use a cross-head velocity of 2.0 inches/min, in accordance with the ASTM D638 test protocol for Nylon-6. If the bars have been stored in the vacuum oven, be sure to let the bars return to room temperature before testing them.

### **Part 3. Impact Testing (1 period)**

Obtain both notched and unnotched Izod Impact strengths for all blends. Your instructor will show you how to prepare the specimens. Use the computer controlled TMI Pendulum Impact Testing Machine. Test at least 5 samples in each notch mode for each blend. Report the averages of your determinations in the conventional units of the test, ft-lbf/in. notch. The bars are 1/8 in. thick. If the bars have been stored in the vacuum oven, be sure to let the bars return to room temperature before testing them.

## **V. Report**

1. Briefly describe the mechanisms for impact strength enhancement of Nylon-6 by incorporation of a rubbery second phase. How does the addition of rubber affect other mechanical properties, such as tensile modulus, yield strength and elongation at break? Try plotting the energy to break observed in the tensile test vs that observed by impact testing. Are the two energies related? Why or why not?

2. Compare the impact energies between notched and non-notched specimens of the same material. Is Nylon-6 "notch sensitive"? That is, is the energy to break, corrected for ligament length, lower for the notched material? What about the blends?

3. The standard ASTM Heat Distortion Test loads a flexural specimen to either 66 psi or 266 psi in bending, then watches for "beam sag" as the temperature is slowly raised. Essentially, this test is watching the effect of temperature on the elastic modulus of the material. Based on this insight, do you expect the HDT of these blends to increase or decrease with increasing loading of Impact Modifier?

4. Compare the impact and mechanical properties of "wet" vs. "dry" Nylon-6. Is an impact modifier still required to impact modify "wet" Nylon-6? Is water a good plasticizer for Nylon-6?