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Microscopy of Polymers

Objective: The objective of this lab is to become familiar with microscopic analytic techniques used in polymer analysis. The simplest technique, often under utilized, is optical microscopy which observes optical scale structure, birefringence and can be used to identify the chemical composition of phases in an IR microscope or using phase contrast microscopy (**either Doug Bowling or Sathish Sukumaran will do this**). It is common to observe colloidal scale structure using SEM after gold coating polymer samples, especially for semi-crystalline or filled polymers. TEM for polymers is a fairly difficult experiment and is usually only performed by experts. Recently, with the advent of atomic force microscopy, AFM, a much less expensive, simpler and potentially more powerful microscopic technique has replaced many of these now conventional direct imaging approaches. AFM can be used across the size ranges covered by OM, SEM, and TEM under proper conditions. The department does not own an AFM and had TEM's specialized for metals which are not useful for polymer samples. Researchers in the department own several instruments which are of interest and special arrangements for the class to observe the use of these as a group will be made by Sathish and Doug.

Instruments to be used:
Optical microscope (Departmental)
SEM (Departmental)
AFM (van Ooij by special arrangement)
IR microscope (Boerio by special arrangement)

Materials:
Polyhydroxybutyrate, isotactic (Biosource) and atactic (Synthetic) (PHB). Polyethylene: HDPE, LDPE, LLDPE, Exxon Exceed Resin (Metallocene), teflon filled PE resin, toluene.

Procedure:
1) Prepare 3, 20% solutions of PHB in toluene, pure isotactic, pure atactic, a 50% blend. Solution cast these samples on microscope cover slips. Prepare similar samples for SEM and AFM.
2) Melt the other polymers on cover slips and sandwich them between 2 cover slips to make thin samples for OM. Prepare similar samples for SEM and AFM.
3) Take digital images on the OM using several magnifications and with and without crossed polars. Also, look at the samples and describe them as clear, turbid, colored etc.
4) Gold coat samples for SEM. Observe all samples in the SEM under several magnifications. Make SEM images to include in your report.
5) Determine the best IR bands to observe the teflon filled PE sample and the tactic blend PHB samples. As a group we will observe the operation of the IR microscope (Boerio's group by arrangement of Sathish Sukumaran).
6) Choose, as a group, two samples to observe using AFM at low magnifications (van Ooij's group by arrangement of Sathish Sukumaran)

Analysis:
1) Identify all morphological features observed in the images and give a brief description. These should include, spherulites, immiscible phases, spherulitic bands.
2) Identify phases which are observed, such as in the IR microscope, based on the contrast parameter for each of the microscopic techniques.

Questions:
1) Compare the size observed, contrasting mechanism and general advantages for each of the microscopes and for each of the samples.
2) What is the advantage of using crossed polars, IR wavelengths in OM?
3) What are the difficulties and disadvantages of each of the microscopies used?
4) How does a SEM work? Describe the optics.
5) How does an AFM work? Describe the basis of contrast.
6) What advantage does an IR microscope have in the Teflon filled PE sample? What advantage does it

have for the PHB blend?
7) What causes a spherulitic band? Why do polymers form spherulites rather than grains such as in a sample of aluminum