DOI: 10.1017/S143192760551064X
Microsc Microanal 11(Suppl 2), 2005

Polymer Sample Preparation for Electron Microscopy

Amanda M. Schrand*

*Graduate Department of Materials Engineering, The University of Dayton, 300 College Park, Dayton, OH, 45469

Microscopy generally involves either the examination of a surface in reflection or the transmission of radiation through a thin sample [1]. The quality of the information obtained with microscopy is dependent on resolution and contrast [2]. Therefore, complementary techniques such as light and electron microscopy can impart valuable scientific insights. Light microscopy can initially save time and effort by providing a lower magnification overview to locate specific areas, check orientation, or discard uninteresting or poorly processed material [3]. However, features smaller than the wavelength of light are not resolvable with light microscopy. In order to visualize details that are orders of magnitude smaller, the scanning electron microscope (SEM) and transmission electron microscope (TEM) are utilized (Table 1).

Unlike light microscopes, electron microscopes require high vacuum levels in order for electrons to reach the sample without being scattered by atmospheric particles. Once the electrons reach the sample, they must interact in such a way to produce useful information. This information is generally in the form of a grayscale image. Image contrast in electron microscopes is produced through electron scattering by the atomic nuclei of the sample. In organic polymers, which consist exclusively of light atoms such as carbon, hydrogen, oxygen, and nitrogen, the scattering is weak and therefore produces poor contrast [3]. To meet these conditions needed for successful electron microscopy, polymers require special sample preparation (Table 2).

Ideally, samples for electron microscopy should be non-volatile, free of contaminants, able to withstand electron beam irradiation, produce atomic-based contrast, and act as conductors or be thin enough for electron transmission. Most polymers do not naturally possess these characteristics, which creates obstacles to having the best imaging conditions. These obstacles can be combated and overcome by proper cleaning and drying, etching and staining of low contrast samples, lightly coating to prevent charging and protect from beam damage, making a pathway to ground with conductive paint or tape, and sectioning with an ultramicrotome for ultrathin sections [1-7].

A successful SEM sample has a clean, supported, dry, conductive surface with a path to ground that can withstand electron beam irradiation and produce atomic contrast. A successful TEM sample is very thin (<100 nanometers) in addition to all of the above characteristics. Although the sample is typically altered in the sample preparation process, the end goal is to retain its structural integrity and to not compromise the electron microscope [4].

References
[1] A. Vaughan, Polymers and Microscopy, RMC/Boeckeler 7th Annual Materials Microtomy Short Course, Tucson, 2002.
[2] D. T. Grubb and L. C. Sawyer, Polymer Microscopy, 2nd ed., Chapman and Hall, London, 1996.
[3] N. Reid, Ultramicrotomy, In: A. Glauert (editor), Practical Methods in Electron Microscopy, Vol 3, North Holland Publishing Col, Amsterdam, 1974.
[4] J. Goldstein, D. Newbury, D. Joy, C. Lyman, P. Echlin, E. Lifshin, L. Sawyer, and J. Michael, Scanning Electron Microscopy and X-ray Microanalysis, 3rd ed., Kluwer Academic/Plenum Publishers, New York, 2003.
[5] T. Malis, TEM Specimen Preparation "At the Cutting Edge", Leica 2002 Ultramicrotomy of Industrial Materials, 1996.
[6] A. W. Robards and A. J. Wilson, Procedures in Electron Microscopy, John Wiley & Sons, Chichester, 1993.
[7] C. Schooley, Materials Microtomy, RMC/Boeckeler 7th Annual Materials Microtomy Short Course, Tucson, 2002.

TABLE 1. Comparison of Optical and Electron Microscopes [2]

Ranges	Stereo binocular	Compound light	SEM	TEM
Useful magnifications	5-100x	30-1500x	20-60kx	3-250kx
Typical resolutions	10 microns	1 micron	10 nanometers	1 nanometer
Best resolution	2 microns	0.2 microns	4 nanometers	0.2 nanometers
Smallest observable polymer structures	Macroscopic	Spherulitic	Lamellar	Crystal lattice
Field of view	Very large 5mm, 50x	Large 2mm, 50x	Large 20 microns, 5kx	Small 2 microns, 50kx
Imaging system	Light optical Glass lenses	Light optical Glass lenses	Non-optical Raster	Electron optical Magnetic lenses
Sample environment	Ambient	Ambient	High vacuum	High vacuum
Radiation damage	None	None	Little	Severe
Chemical analysis	Not usually	Not usually	Yes, x-ray	Yes, x-ray

TABLE 2. Summary of Sample Preparation Techniques for Electron Microscopy [1-7]

Preparation	Specific Methodology
Cleaning	Alconox detergent, plasma (etch), CO_2 snow, acetone and alcohols, air duster
Fracturing	Controlled with machine or cool sample to temperature < Tg, then break
Polishing	Silicon carbide, aluminum or chromium oxide, diamond paste, colloidal silica
Etching	Benzene, toluene, xylene, acetone, acids (nitric, chromic, sulfuric, orthophosphoric)
Staining	Osmium or ruthenium tetroxide, phosphotungstic acid, hydrazine, silver sulfide
Embedding	Epon 812 (epoxy), Spurr (epoxy), Araldite 6005 (epoxy), LR White (acrylic)
Sectioning	Ultrathin samples to 10's of nanometers, cut with diamond knife, can be cryo
Supporting	Aluminum or carbon stubs, carbon paint, copper tape, silver epoxy, copper grids
Coating	Carbon, gold, gold-palladium, tungsten, chromium, platinum, titanium
Storing	Vacuum dessicator or dry, dust-free, airtight container, constant temperature, label

https://doi.org/10.1017/S143192760551064X Published online by Cambridge University Press