Microscopy Techniques (cont.)

Polymer Blend Morphology (LOM / TEM)
Rubber and plastics can be cryogenically microtomed to obtain sections thin enough to be observed in the TEM. Larger features can be analyzed with the LOM. By staining with osmium or ruthenium, polymer morphology of blends can be seen. Subsequent preparation of the microtomed bulk specimen by etching with the appropriate organic solvent, acid or base will make certain morphologies more apparent using the SEM.

Room Temperature & Cryogenic Microtoming (LOM / TEM)
Thin sections of most rubber and plastic materials can be obtained using our new RMC Powertome XL with RXL cryo attachment. Sections under 100 nm can be obtained and viewed with the TEM or LOM and micromachined bulk sections can be viewed with the SEM.

Surface Analysis (LOM / SEM)
Surface features of samples can be obtained using the LOM or the SEM, allowing features such as surface roughness and crack propagation to be seen. Discoloration problems can be detected in the LOM and sometimes identified elementally by using atomic number contrast in the SEM. Atomic number contrast uses the backscattered electron detector, which can determine differences in relative atomic number with high atomic number elements (for example, iron or lead) being lighter in color and lower atomic number elements (carbon, silicon, etc.) depending on the matrix of the sample. Specific elements can then be identified using the IXRF Systems - EDS2004 energy dispersive X-ray system (EDX). High-resolution secondary electron SEM images can also be obtained to further characterize the sample.

Where Do I Go From Here?
Whether you need failure analysis or just want to obtain compound properties, ARDL is ready to help you with all of your microscopy needs. Call, email or go online now to find more information or to request a quotation.
Dispersion Analysis (LOM)

Dispersion of carbon black or inorganic fillers can be determined by cutting or microtoming the rubber and analyzing with reflected light, transmitted light or electron microscopy. In the case of LOM, carbon black dispersion can be determined using the reflected light method commonly known as the Phillips dispersion rating. More precise carbon black dispersion can be determined by microtoming thin sections and analyzing with transmitted light for the analysis. SEM analysis uses atomic number contrast from backscattered electron images to determine the dispersion of inorganic fillers with the fillers appearing lighter (high atomic number) than the surrounding rubber (low atomic number).

Elemental Analysis & Multi-Element Dot Mapping (EDX)

Determining elemental composition (anything over sodium in the periodic table) of contaminants in samples is important when other means of analysis such as FTIR and GC/MS did not work. FTIR and GC/MS are useful to determine the organic structure of contaminants present, but do not do as well as SEM/EDX for identification of inorganic contaminants. Positions and concentrations of different elements in a composite can be located with either multi-elemental X-ray dot mapping or line scan analysis.

Coating & Film Thickness (LOM / OC / SEM / EDX / TEM)

Many modern rubber and plastic products are manufactured with different layers (coatings, laminating, and surface coatings or treatments) that perform specific functions. By embedding and microtoming cross-sections through these different layers and analyzing them microscopically using the optical comparator or light optical microscope, one can determine the number of layers present and determine the thickness of each. Subsequent analysis of the microtomed sections by microscopy FTIR and SEM/EDX can also determine organic and inorganic compositions of the different layers. Analyzing sections in the TEM can resolve very thin sections.

Metal-to-Rubber Bonding (SEM / EDX)

The extent of tire cord adhesion and other rubber-to-metal bonds can be looked at in the SEM using image and elemental analysis. Techniques such as freeze fracturing and polishing of sample cross-sections can be employed to get a close look at the interfaces in question. Multi-elemental X-ray dot mapping and line scan techniques can then be used to measure layer thickness and identify elements present that are specific to primers and adhesives.

Foam Cell Size (LOM)

Open and closed foam cell size can easily be determined using the light microscope by taking reflected light images of the foam cross-section, measuring a fixed distance and counting the number of cells in that distance. Using a specific mathematical formula, an accurate determination of the three-dimensional volume of the cells can be made and the number of cells per unit measure can be obtained.

Particle Size & Particle Size Distribution (LOM / SEM / TEM)

In addition to carbon black typing, the light microscope and TEM can also determine particle size distribution and morphology of inorganic fillers and other compound additives as long as they don’t interact too much with the electron beam. Examples of other types of particles analyzed include silica, recycled rubber, PVC and latex.