Microscopy

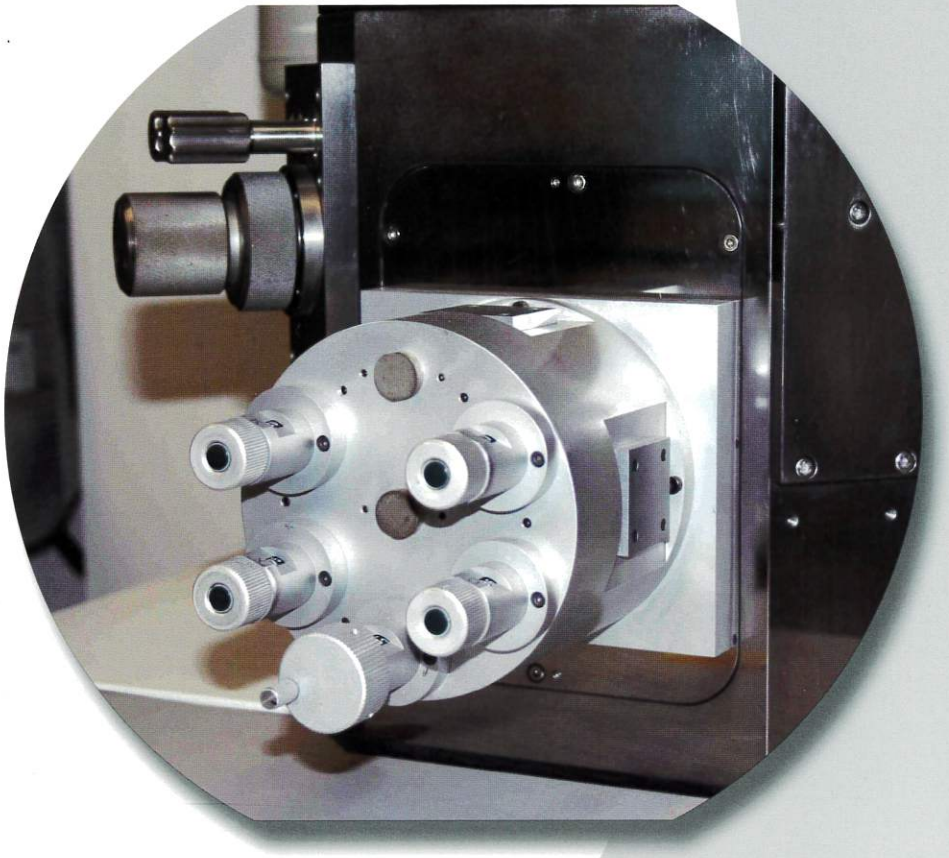
Background

Microscopy techniques are used to produce visible images of polymer micro-structures or details that are too small to be seen by the human eye.

There are three main types of microscopy: Optical, Electron and Scanning Probe. ARDL utilizes Optical and Electron microscopy including Light Optical Microscopy (LOM), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM).

Optical and Electron microscopy both work based on the fact that diffraction, reflection or refraction of radiation instigates the development of an image.

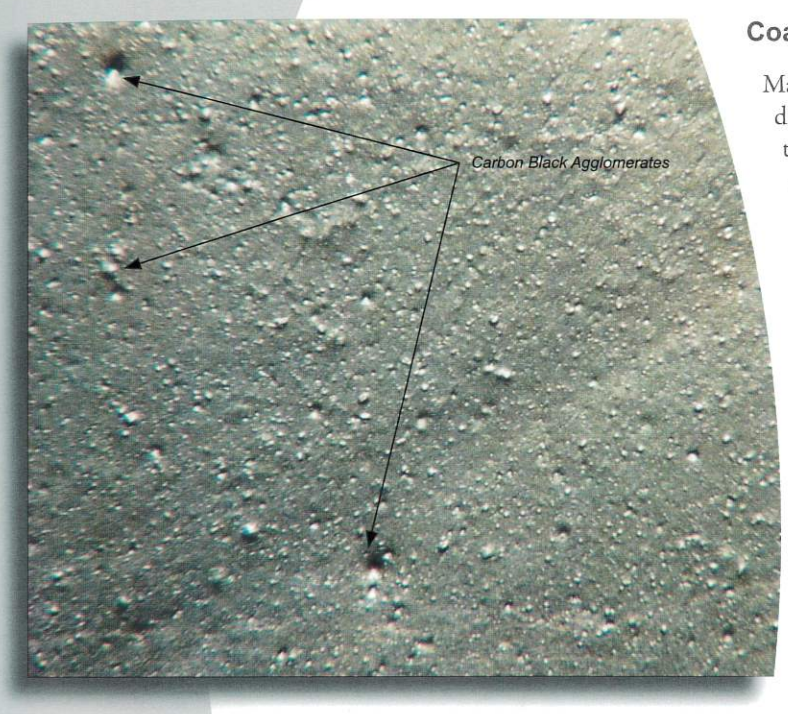
Energy Dispersive X-ray (EDX) detects elements within a sample using X-rays generated by the SEM. Optical Comparator (OC) can be used for micromeasurements on parts to make sure they are in specification.



Microscopy Techniques

Carbon Black Testing / Carbon Black Typing (TEM)

Carbon black is a major component of many rubber products and is used for reinforcement and modification of physical properties. Identifying the specific carbon black in a compound by TEM is important so that the proper carbon black can be used in formula reconstruction for optimization of the physical properties of the rubber. Determinations of type of carbon black in a compound are found by particle size distribution. Organic filler particle size can also be determined. It is important in failure analysis to determine that the appropriate carbon black/other filler has been added to the rubber compound.



Phillips Dispersion by LOM ——— *665 Micron*

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

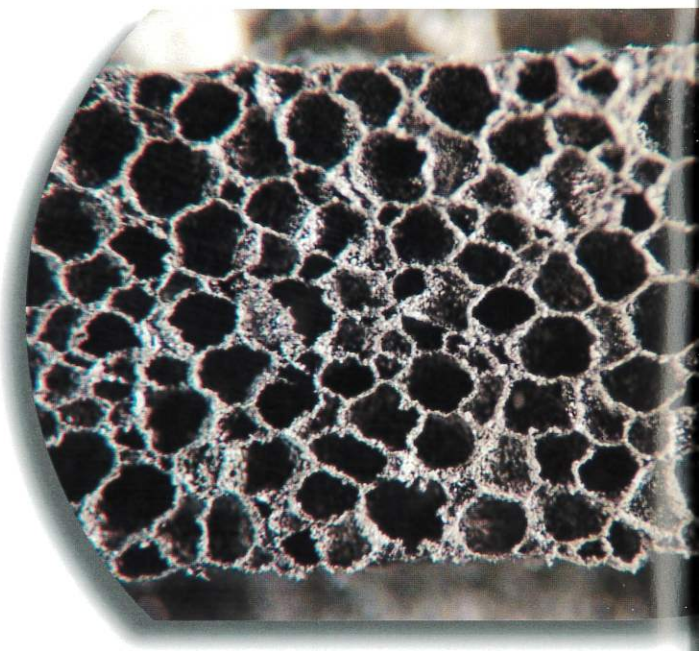
Many modern rubber and plastic products are manufactured with different layers (coextrusions, laminating, and surface coatings or treatments) that perform specific functions. By embedding and microtoming cross-sections through these different layers and analyzing them microscopically using the optical 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 microscope 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.

Dimensional Analysis (LOM / OC)

Part dimensions can be accomplished by using an optical comparator or simply by scanning an image using a flat bed scanner. Analysis of this type is usually performed to determine if a part is within the specifications determined by the manufacturer. This is important in failure analysis because if a part is out of specification, that may be the sole reason for failure.

Dispersion Analysis (LOM)

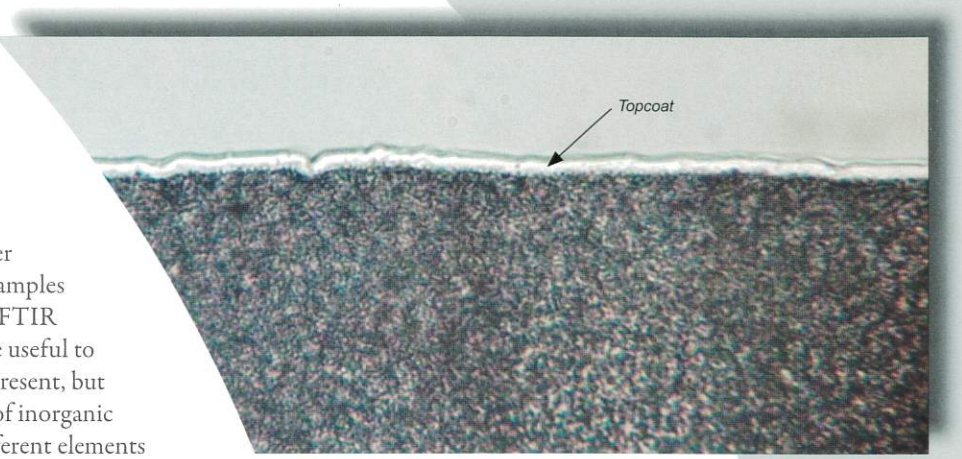
Dispersion of carbon black or inorganic fillers can be determined by cutting or microtoming the rubber and analyzing with reflected light, transmitted light or electron microscopy. In the case of LOM, carbon black dispersion can be determined using the reflected light method commonly known as the Phillips dispersion rating. More precise carbon black dispersion can be determined by microtoming thin sections and using 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).



Cell Size Determination of Closed Cell Foam Rubber by LOM
——— *665 Micron*

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.



Topcoat Thickness Measurement by LOM
15 Micron

Failure Analysis (LOM / SEM / EDX / TEM)

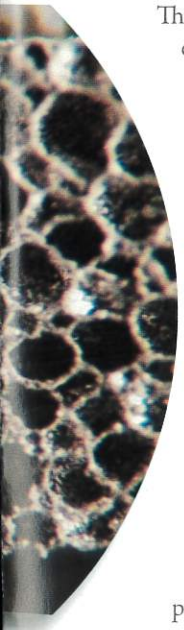
The preferred starting point of failure analysis is microscopic analysis, because it can save time and money. The microscopist first attempts to discover the root cause of failure by utilizing low magnification analysis to document macro features like crack propagation. If higher levels of magnification are needed, SEM and/or TEM analysis can be performed to allow micro features, like inorganic filler dispersion, to be seen.

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.

Internal Structure Features (LOM / SEM / TEM)

Internal structures such as crystallinity (lamellar and spherulitic structures) can be determined by using polarized light microscopy, TEM, and, in some cases, SEM depending on how the sample is fractured and the type of sample being analyzed.



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.

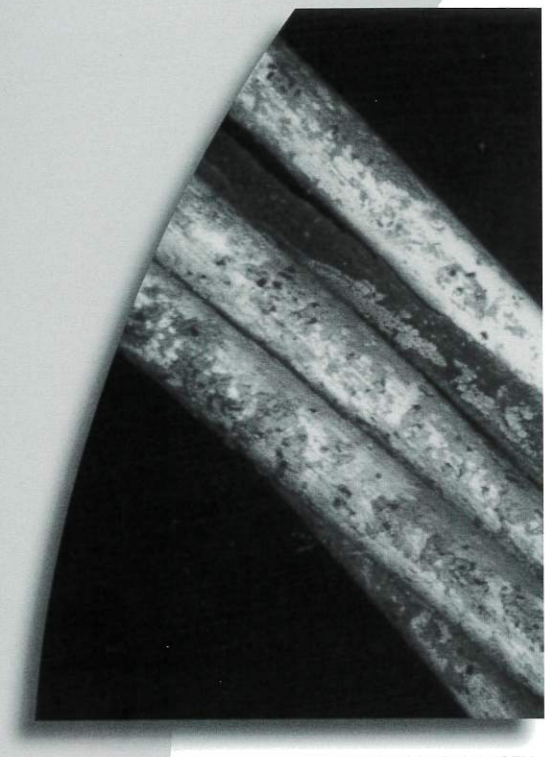
Micro-Dispersion Analysis (SEM / EDX / TEM)

Fine dispersion of inorganic fillers can be evaluated by image analysis in the SEM using backscattered electron imaging, which uses atomic number contrast in order to differentiate between low and high atomic number elements. Inorganic fillers with higher atomic number elements such as calcium and zinc would be visually lighter in color in the image and lower atomic number elements such as carbon and magnesium would be darker.

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 that could be analyzed include recycled rubber, PVC particles and latex.

Microscopy Techniques (cont.)



Tire Cord Analysis by SEM

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 microtomed 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 being darker (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.

AKRON RUBBER DEVELOPMENT LABORATORY, INC.

www.ardl.com

2887 Gilchrist Rd. | Akron, Ohio 44305 | answers@ardl.com
Toll Free (866) 778-ARDL | Worldwide (330) 794-6600 | Fax (330) 794-6610