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Learning Isolating Techniques for Microscopical Analyses

Kristie Diebold* and Kristen Partin*

*McCrone Associates, Inc., 850 Pasquinelli Drive, Westmont, IL 60559

Industrial contract laboratories receive samples from a wide variety of industries, including polymer, pharmaceutical, forensic, electronic, and art, that may require sensitive manipulation and isolation.

The sample preparation requirements for many microscopies are demanding, and critical to the success of the analysis. Polarized Light Microscopy (PLM) requires the microscopist to study particulate with transmitted light on a glass slide in a useful mounting medium. Electron Microprobe Analysis (EMA) and Scanning Electron Microscopy (SEM) require particles to be mounted on conductive material such as carbon planchets, carbon tabs, or beryllium. Infrared microspectroscopy (IR) analysis requires an infrared transparent substrate such as NaCl or KBr for analysis in transmission mode, while an e-glass slide may be used for reflectance measurements. Transmission Electron Microscopy (TEM) requires a minute amount of sample dispersed on a carbon coated copper grid in order to resolve any nano-scale structures and determine chemical and structural features of isolated regions. Secondary Ion Mass Spectrometry (SIMS), as used for particle analysis, often requires a dispersion of particles to facilitate particle counts and size distribution measurements as well as trace chemical and isotopic analysis. Therefore, the light microscopist using a variety of microscopical techniques is essential for all types of microscopical analysis.

The defect and contaminant problems presented to the laboratory may consist of: rubber, glass delamination, fibers, hair, residues, and pigments. Isolation of these substances requires skill and patience in order to remove and transfer them onto the desired substrate for microscopical analysis. Substrates include carbon, beryllium (Be), KBr crystals, carbon coated copper grids, silicon wafers and glass microscope slides.

Using precise tools such as micro-blades, scribes, fine tungsten (W) needles, adhesives, and solvents, the light microscopist must isolate the defect of interest using a variety of techniques, depending on the sample in question, including:

- Rubber stopper defects can be removed with a W needle and micro-blades
- Glass delamination may be removed with a W needle and soluble gum, or replicated onto Be or Silicon wafer with water
- Hairs and fibers can be moved to a slide and mounted in various refractive index liquids
- Residues from microelectronic devices are isolated using a W needle and a solvent, such as amyl acetate, to create a dispersion for SIMS analysis
- Extractions of different paint pigments can be done using a variety of solvents and a W needle on KBr to authenticate a painting

While experience is mandatory, the skills helpful for isolating contaminants or defects can be learned relatively easily. There are just a few basic principles for handling small particles that can be applied to common contaminants and defects, techniques originally developed by Dr. Walter C. McCrone and Anna Teetsov. A skilled microscopist, with a steady hand, and a very sharp pointed needle, can pick up and deliver particles as small as 1 µm in diameter. Techniques for a particular particle depend on where it is; that is, it may be embedded, lying loose on a surface, or in a mounting medium. There are a few helpful hints that may be applied to each unique situation, including:

- Never take your eyes from the particle
- Adhesives may be used to isolate particles
- Particles are removed from the needle by releasing it into an appropriate solvent
- Ingenuity, patience, and self-confidence are essential for success

McCrone Associates' Cleanroom Microscopists teach these techniques at the College of Microscopy in Westmont, Illinois. A number of laboratory exercises are used in each class to build the handling and manipulation skills required. For example:

- Two handed technique (Figure 1)
- Manipulating solvent drops with a tungsten needle
- Isolating fine particles from filters

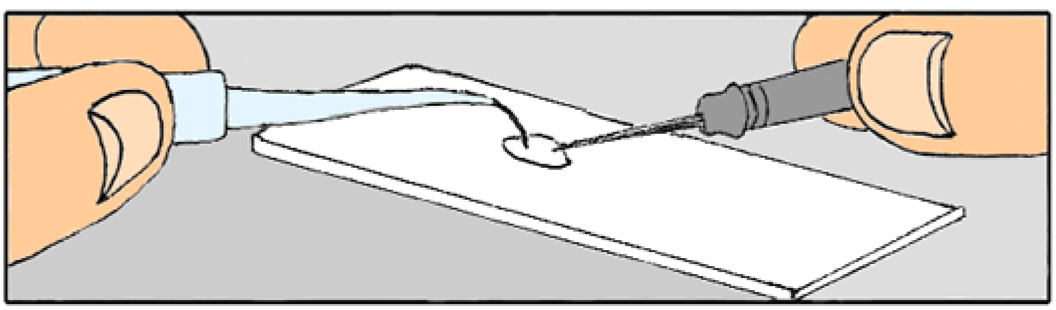

Figure 1: Two Handed Technique

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