Particle Size Analysis using Raman and AFM

The presence of unwanted particles is almost unavoidable in many situations, and unfortunately these particles can negatively affect the performance of products. Often associated with failures of medical devices, poor sterilization procedures, and contamination in general, particles have a way of working their way into even the highest class of cleanrooms. As a result, particle analysis has become widely applicable (especially in failure analysis projects), and many instruments have been developed to help analysts characterize these often submicron-sized issues.

Complete particle characterization is paramount to any particle analysis project. This typically includes chemical, size, and morphological characterization. While particle size (or hydrodynamic radius) is traditionally measured using dynamic light scattering (DLS) or particle counting instruments, these techniques give no additional information regarding the chemistry and structure of the particles. Confocal Raman Microscopy (CRM) and Atomic Force Microscopy (AFM), however, are particularly well-suited to provide the chemical and structural information, in addition to provide information on the particle size and shape. In this application note, a mixture of particles was analyzed via Raman microscopy to investigate their chemical structure. Additionally, diamond crystals were analyzed by AFM to investigate particle morphology and shape.

Figure 1. Raman image (left) of a mixture of garnet (blue), talc powder (red), and TiO$_2$ (green) and the associated Raman spectra (right).
For the Raman investigation, a mixture of particles was created using talc powder, TiO$_2$, and garnet crystals. The particles were shaken together, deposited on a standard glass coverslip, and analyzed using the Raman microscope. The results are shown in Figure 1. As can be seen from Figure 1, the garnet particles are somewhat larger (115 µm) than the smaller TiO$_2$ (10 – 50 µm) and talc (10 – 20 µm) particles. The corresponding spectra to each particle are shown to the right of the Raman image. Raman microscopy is easily able to distinguish the differing chemistry of each particle, and this comprehensive particle characterization can be obtained in a manner of minutes.

Separately, some diamond polishing crystals were deposited on a glass microscope slide for AFM characterization. As the particles could be mobilized by contact mode AFM, non-contact mode was chosen for analysis, and the resulting topographical images are shown in Figure 2. Large diamond particles approximately 2 µm in size, medium particles 400 nm in size, and small particles 170 nm in size were all observed. All particles exhibited a lobed-type fine structure; for the 170 nm sized particles, the lobes were 50 – 80 nm in size and appeared evenly distributed around the center of the particles.

Raman characterization reveals powerful insights regarding the chemical constituency of particles and size of particles. When combined with the morphological information obtained from AFM analysis, the two techniques are well-suited to provide comprehensive data in particle analysis. Particles of almost any size, shape, and constituency can be analyzed, significantly widening the breadth of applications of these techniques to almost any industry.

Figure 2. AFM images of diamond crystals on a glass slide. Small-lobed fine structures (50-80 nm in size) were observed on the surface of the crystals.