Studying Titanium Dioxide with Correlative Raman-FIB-FESEM Microscopy

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Raman spectroscopy is a vibrational spectroscopic technique that is frequently used to characterize molecules. By combining a Raman spectrometer with a confocal light microscope, a two-dimensional array of Raman spectra can be correlated with an optical image to reveal the spatial distribution of chemical components in a material. High magnification observations of surface morphology can be directly correlated to molecular information using a fully integrated scanning electron microscope (SEM) and confocal Raman imaging system. On the Raman Integrated Scanning Electron (RISE) microscope, an area of interest is first identified in the SEM where high magnification images can be acquired. The sample is then transferred under the optical microscope of the confocal Raman imaging system for Raman imaging of the same area of interest. Lamella from this same area of interest can be created with the addition of a focused ion beam (FIB) to a SEM, for further analysis inside a transmission electron microscope (TEM).

The three most commonly found polymorphs of titanium dioxide are rutile, anatase, and brookite. While the chemical composition of the three polymorphs are exactly the same, they produce very distinct Raman signatures. The goal of this study is to use the FIB-SEM workstation to create a TEM lamella at an interface between anatase and brookite that was identified with the confocal Raman imaging system.