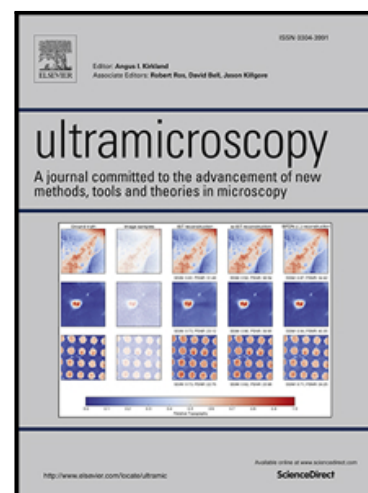


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Correlative Raman Spectroscopy and Focused Ion Beam for Targeted Phase Boundary Analysis of Titania Polymorphs

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Abstract

Site-specific preparation of specimens using focused ion beam instruments for transmission electron microscopy is at the forefront of targeting regions of interest for nanoscale characterization. Typical methods of pinpointing desired features include electron backscatter diffraction for differentiating crystal structures and energy-dispersive X-Ray spectroscopy for probing compositional variations. Yet there are situations, notably in the titanium dioxide system, where these techniques can fail. Differentiating between the brookite and anatase polymorphs of titania is either excessively laborious or impossible with the aforementioned techniques. However, due to differences in bonding structure, Raman spectroscopy serves as an ideal candidate for polymorph differentiation. In this work, a correlative approach utilizing Raman spectroscopy for targeted focused ion beam specimen preparation was employed. Dark field imaging and diffraction in the transmission electron microscope confirmed the region of interest located via Raman spectroscopy and demonstrated the validity of this new method. Correlative Raman spectroscopy, scanning electron microscopy, and focused ion beam is shown to be a promising new technique for identifying site-specific preparation of nanoscale specimens in cases where conventional approaches do not suffice.

Keywords: Raman spectroscopy; focused ion beam; transmission electron microscopy; correlative; site-specific preparation; polymorphs

Abbreviations: TEM, transmission electron microscopy; FIB, focused ion beam; EBSD, electron backscatter diffraction; EDS, energy-dispersive X-ray spectroscopy; RISE, Raman imaging-scanning electron, SEM scanning electron microscopy; SAED, selected area electron diffraction

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