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Recent Advancements in Laboratory X-ray Microscopes for 3D to 4D Imaging and Analysis

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Three-dimensional X-ray microscopy (XRM) has emerged as a powerful imaging technique that reveals three-dimensional microstructure from a range of materials. The non-destructive nature of X-rays has made the technique widely appealing, with the potential for characterizing sample changes in “4D,” delivering 3D microstructural information on physically the same sample over time, as a function of sequential processing conditions or experimental treatments. This has led to a new generation of functional studies (Figure 1), with applications spanning the life sciences, physical sciences, geosciences, and electronics industry, and is in a state of rapid expansion [1]. Recently, laboratory-based X-ray sources have been coupled with high resolution X-ray focusing and detection optics from synchrotron-based systems to acquire tomographic datasets with resolution down to 50 nm [2]. This signifies an improvement of at least one order of magnitude in spatial resolution relative to the limits of ‘optic-free’ laboratory computed tomography (CT) techniques. Observing the evolution of microstructure on the same region of a single sample can rapidly benefit computational models of materials, by avoiding the requirement to extrapolate based on statistical samplings from a large number of like specimens. This talk will explore both the implementation of laboratory XRM characterization and analysis in several leading applications examples in materials imaging. Several examples of *in situ* and ‘4D’ experiments will be presented, including crack propagation in ceramics, porosity and permeability characterization, polymer electrolyte fuel cell (PEFC) electrode characterization, deformation of polymer foams under load, and the evolution of defects in electrode materials in Lithium ion batteries. By incorporating X-ray focusing lenses, laboratory nanoscale XRMs are now enabling unprecedented high-resolution studies of soft and hard materials alike without the need for synchrotron radiation [2]. This is made possible largely through the use of Fresnel zone plates, which are diffractive imaging objective lenses capable of focusing X-ray radiation. Analogous to typical transmission light or electron microscopes, condenser and objective lenses in the Xradia nanoscale UltraXRM focus and magnify the object onto a detector in the image plane (Figure 2). As a result, the UltraXRM system provides laboratory access to 3D and 4D X-ray microscopy down to the 50 nm spatial resolution scale, with ultimate voxel sizes down to 16 nm, and fields of view up to 65 µm. Soft materials, ranging from polymers to biological tissue, consistently pose challenges in generating contrast by several techniques, X-ray absorption included. Employing the Zernike method, however, enhances the visibility of grain boundaries and material interfaces when absorption contrast is low, enabling visibility of microstructures without staining. We demonstrate the application of both absorption and phase contrast techniques on such materials, including polymer electrolyte fuel cells [1, 3-4] and superconducting materials. Using the Xradia microscale VersaXRM, battery electrodes in commercially-packaged cells may be characterized nondestructively with sub-micron resolution, enabling characterization of the electrode microstructures. The tunable contrast enhancement mechanism within the VersaXRM combines optimized absorption contrast, for a uniquely flexible laboratory X-ray microscope imaging and analysis platform for modern *in situ* and 4D studies. [5]. Using this approach, researchers may characterize the changes in this microstructure as a function of operational parameters, such as charge state, thermal environment, pressure/fracture, etc., either *ex situ*, *in situ* or *in operando* [6-9]. From their lightweight properties to unique storage capabilities, polymer foams have emerged as a unique material for many industrial application. While these materials are widely utilized for their functional parameters, much about the long-term microstructural behavior remains a mystery, and degradation mechanisms are not very well understood. Using laboratory XRM, the deformations within the foam microstructure may be directly observed in 3D. This enables the 4D mapping of parameters such as elastic moduli and hysteresis curves, along with the direct observation of strut/pore deflections and responses to mechanical loading [10]. Using the XRM technique with an *in situ* compression cell (Deben, UK), the deflections of pores within a polymer foam may be directly observed in 3D [10]. Measurements of these deflections may be subsequently correlated to the loading pressures, for direct measurements of material’s mechanical properties in conjunction with finite element analysis or fluid dynamics models.

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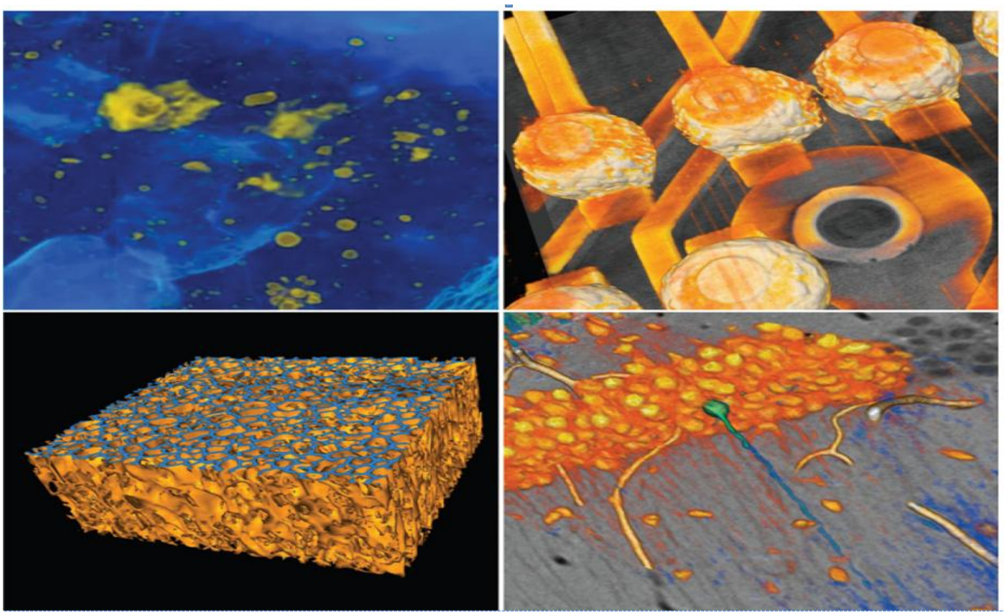

Figure 8. Xradia XRM 3D Datasets: Geology (upper left): carbonaceous chondrite meteorite that landed on April 22, 2012, at Sutter’s Mill, CA. Image width = 9 mm. Courtesy of Prof. Qing-Zhu Yin, UC Davis. Electronics (upper right): microelectronic device with short circuit between two bumps. Image width = 600 µm. Courtesy of S.T. Crolles. Materials Science (lower left): soft porous polymer with urethane backbone (blue) imaged in situ under varying temperature and compression. Image width = 200 µm. Courtesy of Natl. Chemical Laboratories, India. Life Science (lower right): mammalian brain tissue section showing individual neuron cells. Image width = 260 µm. Courtesy of the Natl. Center for Microscopy & Imaging Research at UC San Diego. Images from Microscopy Today [1].

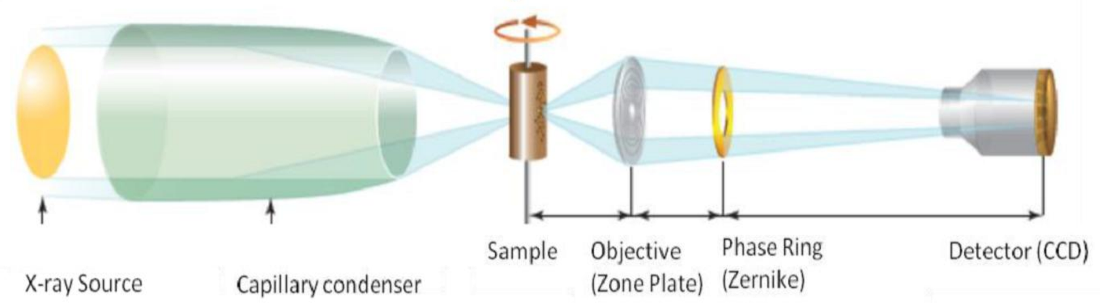

Figure 9.X-ray optical schematic of the Xradia UltraXRM-L200, achieving 50 nm spatial resolution with an 8 keV laboratory source