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In-situ nanomechanical testing using X-ray microscopy

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Micron-scale X-ray tomography, classically referred to as MicroCT, is a well-established 3D imaging technique and has seen various applications of *in situ* testing due to flexible sample types, sizes, nondestructive imaging, and lack of need for a vacuum enclosure. Recent advances in lab-based nanoscale X-ray microscopy (XRM) have moved beyond some of the physical constraints of traditional MicroCT by incorporating synchrotron-style optics and detection systems, extending spatial resolution down to 50 nm for samples tens to hundreds of microns in size. These nanoscale X-ray microscopes provide high resolution, nondestructive 3D imaging of interior structures on samples of tens to hundreds of microns in cross-sectional dimension. This new length scale of nondestructive investigation complements existing *in situ* mechanical testing capabilities by filling an additional portion of the span between the sub-nanometer and bulk experimental regimes, and also provides new opportunities for understanding material deformation behavior at this scale.

In a complementary fashion to the variety of imaging tools, *in situ* mechanical testing also covers a broad range of scales. At the high resolution end of the spectrum, testing is often coupled with techniques such as SEM or TEM for direct observation of the deformation processes. While offering excellent spatial resolution, *in situ* SEM is limited to observations of the sample surface, and TEM is restricted by very thin samples, which also generates unique material properties due to the small size effects. In an effort to provide new, unique information by merging the worlds of high resolution 3D imaging and nanomechanical testing, a new *in situ* load stage has been designed and tested in a ZEISS nanoscale X-ray microscope.

This work will cover early material investigations performed to uniquely connect bulk material properties with detailed, direct observation of discrete, internal deformation events occurring at the nano- and micron-scale. A case study of the nanoindentation of a sample of elephant dentin will be presented. Dentin is a naturally-occurring nano-composite material (found in teeth) consisting of a collagen matrix, mineralized hydroxyapatite, and anisotropic tubule structures. This natural structure is of interest for biomimetic applications due it's remarkable mechanical properties. In this study, 3D tomography was performed multiple times at increasing levels of applied load to monitor crack initiation and growth processes, and gain insight into the connections between the novel microstructure, crack shielding mechanisms, and the material's fracture toughness. As an example of the cracking process, figure 1 provides two orthogonal virtual cross sections of the dentin sample during indentation, revealing the tubule structure as well as the initiation of cracks in the vicinity of the indenter tip.

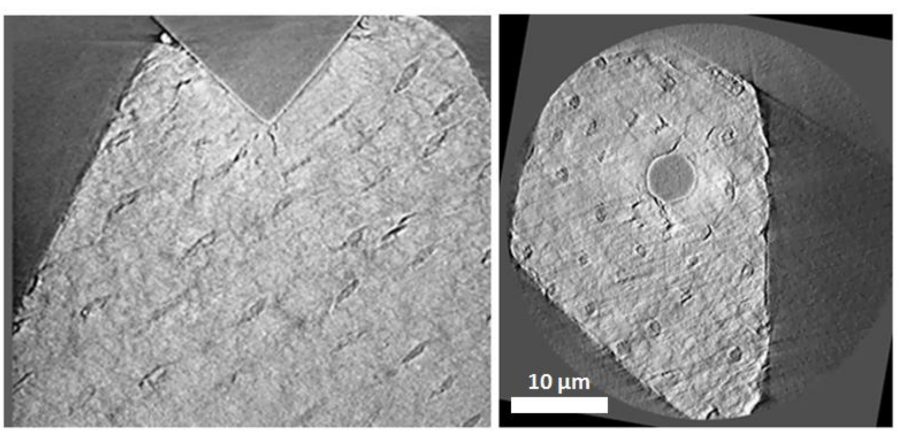

Figure 1 – Virtual cross sections of the nanoindentation of elephant dentin, acquired using a nanoscale X-ray microscope. Crack development around the indenter tip, as well as the neighboring tubule structures, can be observed and contributes to the material's fracture toughness.