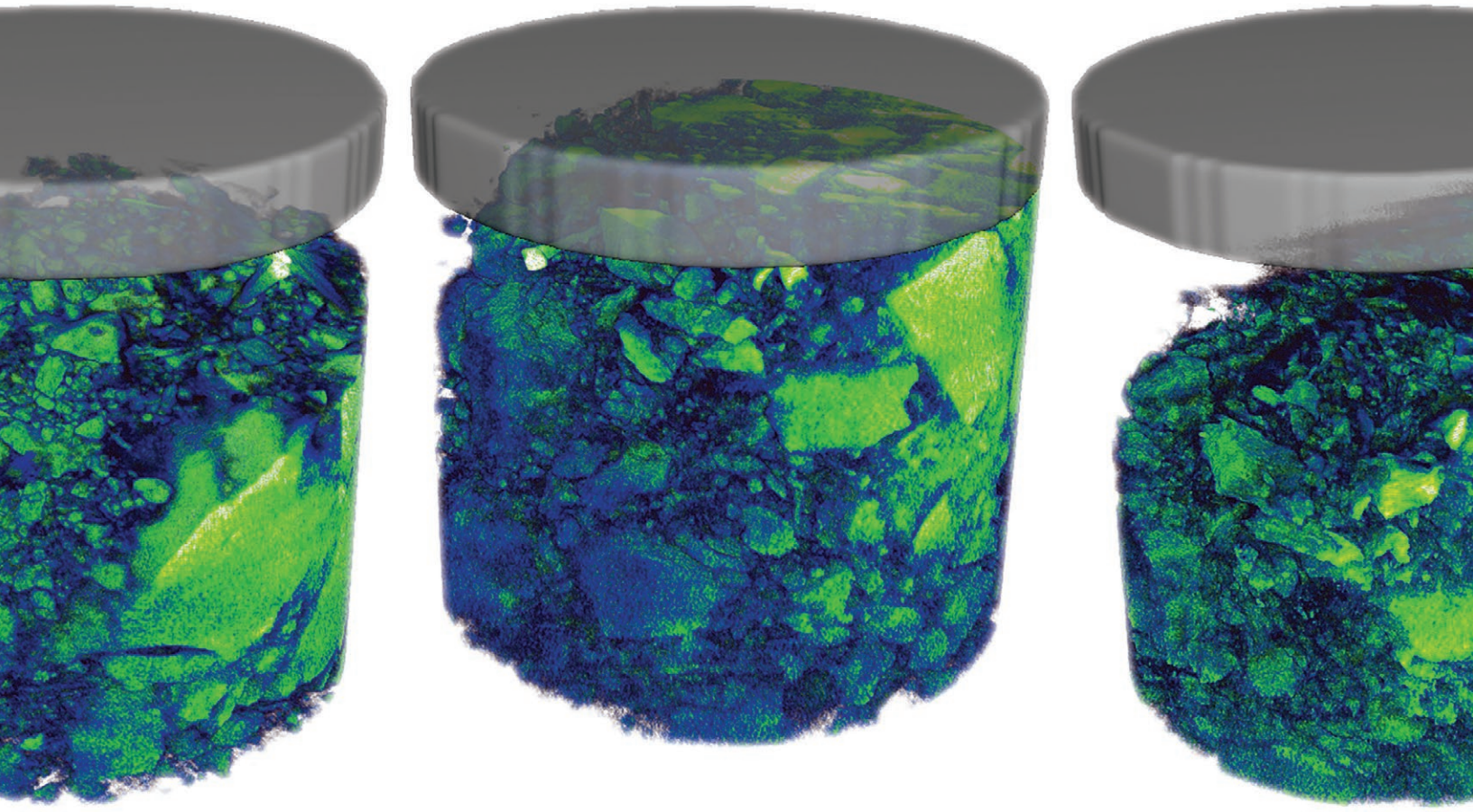
Technical Note



In Situ Observation of Mechanical Testing

at the Nanoscale

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Date: March 2015

X-ray microscopy (XRM) enables nondestructive 3D investigation of a variety of samples across multiple length scales. By adapting advanced X-ray optics from synchrotron developments, 3D X-ray tomography with resolution down to 50 nm is now available in the laboratory. As a nondestructive technique, X-ray tomography uniquely enables 4D studies by observing 3D structure at multiple points in time and under varying realistic conditions. These conditions can include a variety of external stimuli, including mechanical loads. In this Technical Note we introduce the concept of *in situ* mechanical testing integrated in nanoscale XRM. This approach is complementary to established *ex situ* or *in situ* testing methods in SEM or TEM, but covers a unique 3D length scale, offering new opportunities to connect small scale evolution processes with those observed in micron scale XRM and bulk material testing. This connection of micro and macro deformation behavior, with direct 3D visualization, has promising applications covering a variety of materials from metals to biomaterials to thin coatings. Xradia Ultra Load Stage, a new nanomechanical test stage, is now available for integration into ZEISS Xradia Ultra, the only nanoscale XRM available for the laboratory.

Nanomechanical testing seeks to answer the question: How does a material yield, deform, and fail on small scales?

By combining with *in situ* 3D observation, we now seek to answer the next question: how do these mechanisms vary locally within the sample, and what role does the microstructure play in determining the bulk properties and behavior?

This broad concept can be further refined with several more targeted questions:

- How is the behavior of individual micro- and nano-features such as struts, films, "walls", particles, etc. different from the collective bulk? How does this limit the interpretation of material properties from a purely geometric perspective based on simple morphological properties like porosity, surface area, etc.?
- What effects do these features have on the local deformation of a material? For example, do some locations display brittle behavior and others ductile? Do these events occur with sufficient frequency or prevalence that the effects cascade up to larger length scales?
- In terms of engineering a material with desirable properties (modulus, hardness, toughness, etc.) are some defects and features acceptable and others not?

Investigating Under Load

To investigate the deformation and possible structural failure under various loading conditions, a nanomechanical testing rig has been designed that can be easily configured by the user to enable three different operating modes—compression, tension,and nanoindentation. Xradia Ultra Load Stage operates on samples of sizes typical of the nanoscale X-ray microscope, on the order of a 10-100 micron diameter cross section.

This length scale simultaneously satisfies two experimental constraints by:

1) providing optimal X-ray attenuation characteristics for a wide variety of sample types, and

2) effectively connecting the length scales of material deformation spanning resolutions from tens of nanometers up to sample sizes approaching bulk material response.

The Nanomechanical Testing Gap

The Ultra *in situ* load stage explores a new critical length scale of materials characterization, observing the types of internal features, such as nanoscale cracks and voids, that initiate as well as accentuate material deformation and failure, and subsequently connecting these features with properties that are observed on the macroscale.

This new capability satisfies a void in the current availability of mechanical testing and imaging approaches. On the spectrum of force and length scales, Xradia Ultra Load Stage is positioned between, on the one hand, that of macro scale to microtomography testing[1], and on the other, established nanomechanical testing such as *in situ* SEM, TEM[2,3] (Figure 1), or standalone nanoindenters. Of the currently existing *in situ* techniques, micron scale X-ray tomography provides spatial resolution down to the single-micron or submicron scale and can accommodate samples on the millimeter or larger scale subjected to kilonewton loads. On the small end of thespectrum, electron microscopy enables *in situ* mechanical testing on the nanometer scale. Despite providing excellent spatial resolution, the electron techniques suffer from some inherent limitations. SEM is capable of nondestructive surface imaging only, and TEM can provide 3D data but only on very thin samples, typically less than one micron, where surface effects dominate the deformation behavior and strongly influence the results. For both EM methods, there is a lack of understanding of internal micro and nanoscale deformation within a sample which is sufficiently large to minimize surface effects. Conversely, the larger sample sizes typical of the Xradia Ultra microscope (10-100 microns) approach, in many instances, bulk material behavior, providing a new length scale of investigation as well as complementary information to connect length scales.

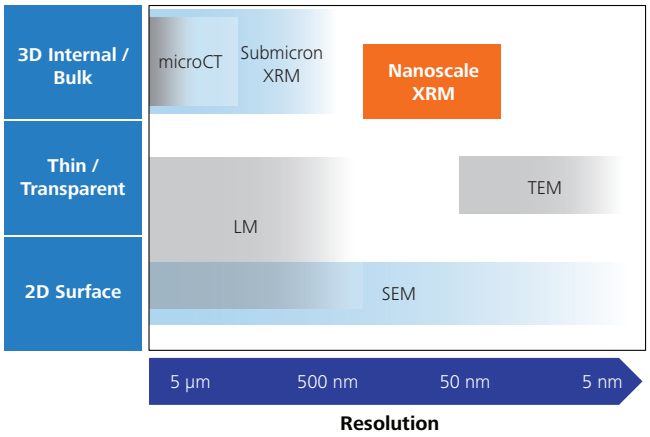


Figure 1. Approximate imaging resolution for common in situ *mechanical testing methods, categorized by sample thickness and transparency. The* in situ *Xradia Ultra Load Stage uniquely fills a gap between single nanometer resolution SEM/TEM methods that are restricted to surface imaging or extremely thin samples, and micron/submicron-scale tomography.*

Modes of Operation

Next, we focus on the following three modes: compression, tension and indentation. In the implementation of these three modes in nanoscale XRM (ZEISS Xradia Ultra), the sample is mounted between two anvils, one of which is stationary and one of which can be moved in a controlled manner. A sensor measures the force on the sample as a function of anvil displacement.

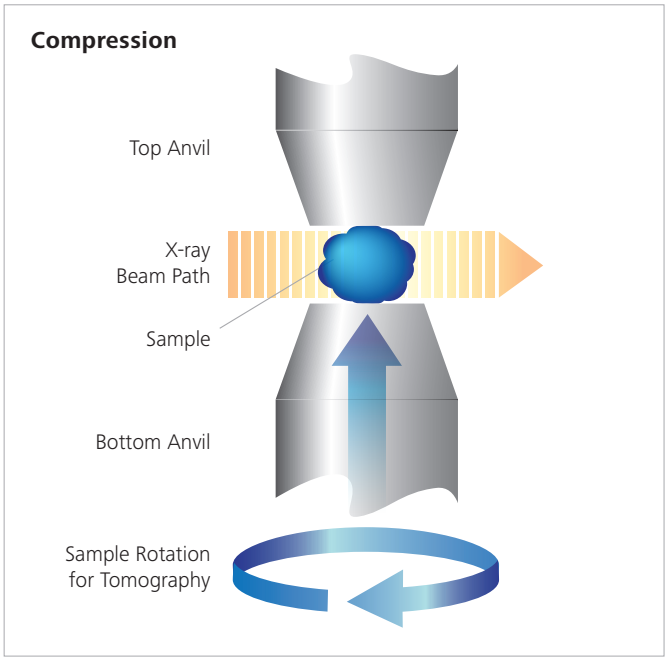
- Compression: the sample is "pinched" between the two anvils that move towards each other. In this mode, structural deformation under uniaxial compressive load is studied.

- Tension: the two anvils move away from each other, pulling on the sample. In this mode, structural deformation under uniaxial tensile load is studied.

- Indentation: a sharp nanoindenter mounted to one of the anvils is pushed into the sample to initiate cracks, delaminate coatings etc. Common types of indenters include cone, cube corner, or wedge.

Compression

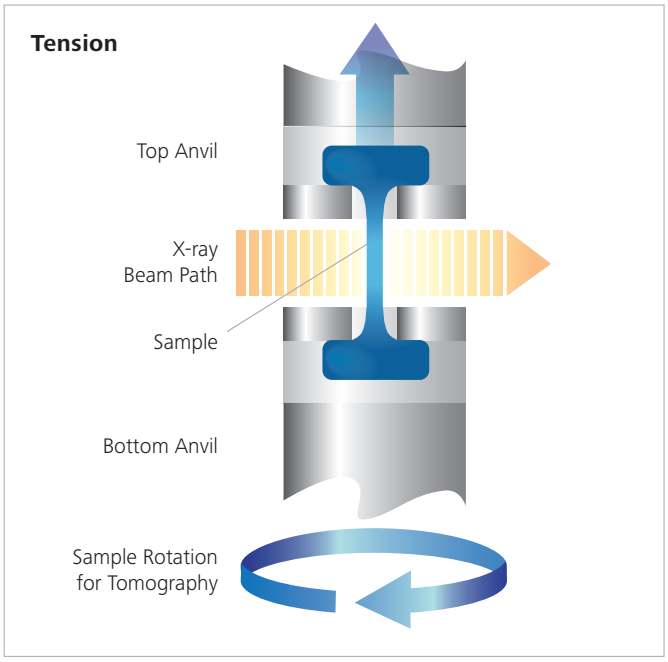
In situ nanomechanical compression combined with X-ray tomography can be used to observe the deformation of materials under compressive load. This type of mechanical test has been commonly used in *ex situ* applications to study the behavior of samples such as metal pillars and porous foams, but a direct 3D image of the deformation process on this length scale has not been previously available. This has left a number of unanswered questions in terms of the relevant local mechanisms. Do micro and nanoscale features undergo uniaxial compression in a similar manner to the bulk sample, or are there local buckling/yielding events? Likely there is varying behavior especially for small, high aspect ratio features such as struts or pore walls. Pores of different sizes may also yield at different rates. Furthermore, if there are anisotropic structures what is the relationship between loading direction, deformation direction, and feature orientation?



These types of structures could have implications for the lateral expansion of the sample orthogonally to the loading direction. And finally, how do these microstructures affect relaxation after load? It is now possible to observe if certain features relax elastically, while others remain permanently deformed.

Tension

Tensile testing can reveal an additional wealth of information about a sample's deformation and failure mechanisms, including critical properties like elastic modulus and tensile yield strength. While these properties can be traced back to atomistic level deformation events in the form of dislocations, at what level do these processes combine in a fashion that dictates bulk behavior? Xradia Ultra Load Stage can uniquely help address this question by operating at an intermediate length between atomistic information and bulk properties; at this scale nano/microstructural features can constitute critical locations where isolated deformation events likely begin to occur on a length scale capable of propagating up to macro-level response.
In a tensile scenario, these types of critical locations include micro-voids, where increased loading can lead to growth and coalescence of neighboring voids, as well as possible crack initiation sites in high stress regions of surrounding material.
It can also include thin and anisotropic features such as struts and fibers which can undergo local rupture and failure at different rates and under different loading than larger domains. It is also possible, particularly in the case of composite materials, to consider regions of varying chemistry or grain structure, resulting



possibly in effects such as locally ductile expansion in one region, but locally brittle failure in a neighboring region. These detailed microstructural effects could indeed have implications for a wide variety of sample types including the life sciences (bone, natural fibers, animal setae, hair), geosciences (mechanics of rock and ore), and materials science (metals, polymers, ceramics, MEMS).

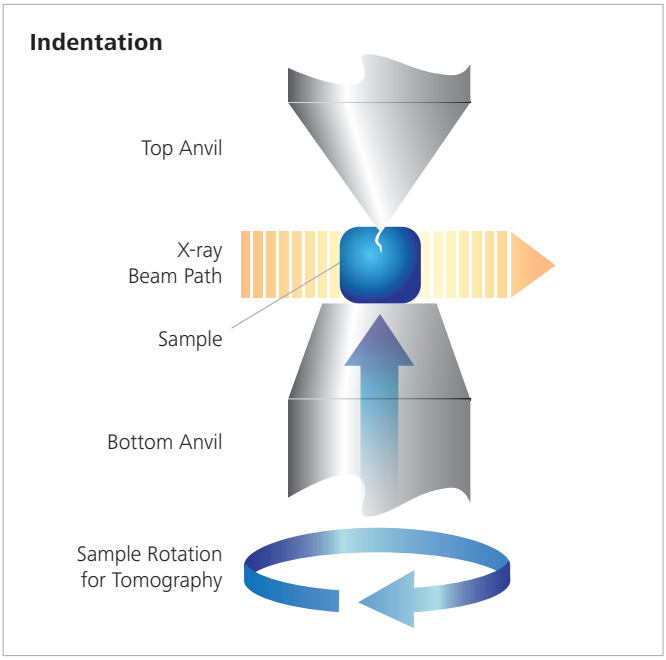
Indentation

Nanoindentation offers the opportunity to study very isolated deformation and failure events surrounding the indentation site. This type of test has a number of distinct advantages, including the study of samples with small linear dimensions, such as coatings and layered structures. In addition, the capability to apply the load in a carefully isolated manner enables the user to locally select the position where failure should initiate. For example, in a composite material consisting of a matrix and distributed inclusion particles, indentation provides the researcher with the ability to initiate deformation of the matrix either in close proximity or extended distance from an inclusion. In a general sense, the indentation can be targeted near particular features of interest, like voids, micro-channels, or interfaces. The exact location in which force is applied often has great impact on such phenomena as crack initiation, growth, and coalescence.

By combining the indentation test with the 3D imaging capability of nanoscale X-ray microscopy, it is possible to observe these processes as they occur not only on the surface but also within the sample interior. For thin films and coatings, this can provide insight into localized delamination processes that occur at sub-surface interfaces. For a variety of additional sample types such as metals, concrete building materials, bone, and teeth, it reveals the impacts that internal heterogeneities and defects near the indentation site have on the deformation and failure of the overall sample.

Practical Considerations

Sample sizes for the described methodology are on the order of tens to hundreds of microns, and based on the Young's modulus for elastic deformation over a range of sample types, such sizes typically require forces in the millinewton to single Newton range to produce deformation on the micron scale. To track this behavior quantitatively during loading, load-displacement curves are acquired to relate the applied force to the deformation or damage events directly observed from the imaging.



As another consideration, data acquisition times in lab based nanoscale XRM are on the order of hours for 3D tomographic datasets. Therefore, real time observation of dynamic processes is not practical. Instead, nanoscale XRM lends itself to so-called "interrupted 4D" experiments, where 3D datasets are acquired at multiple, discrete steps when the loading condition is held static. The *in situ* configuration is still critical for this type of measurement as the load must be maintained during imaging to properly represent deformation processes such as the opening of crack tips. To advance scientific understanding during dynamic loading between tomography acquisitions, the sample can be monitored in 2D radiography mode on the time scale of seconds to minutes.

Lastly, while X-ray tomography ideally is performed over a rotation range of at least +/- 90 degrees, nanomechanical testing requires a structurally rigid connection of the two anvils between which the sample is mounted. The lack of mechanically strong but X-ray transparent materials in the X-ray energy range of interest (5.4 or 8 keV in currently available instruments) requires a narrow but strong support post. This limits the angular range for tomographic data acquisition to about +/- 70 degrees.

Demonstration

The concept of *in situ* nanomechanical testing in XRM is demonstrated here on two types of materials intended to indicate the possible range of applications. A compression experiment was performed on a porous elastomer as shown in Figure 2. The sample is mounted on the bottom anvil (not visible) and occupies most of the field of view, while the stationary top anvil is also visible at the top of the field of view. The three panels display the results of three separate tomography scans conducted before, during, and after a compressive loading process. Panel (a) depicts the original, uncompressed sample. In panel (b), the sample has been moved vertically upward, putting the sample in contact with the top anvil causing compression. The loading was maintained in this state during the collection of the tomography. After tomography, the sample was moved back down to the original position to remove the compressive force. A third tomography, depicted in panel (c), reveals the final deformation of the sample after removal of the applied load, suggesting permanent changes of the microstructure.

In a second experiment, nanoindentation was performed on a sample of dentin. Dentin is a natural microstructured composite material found in teeth, serving functions of transport, cell communication, and structural reinforcement. The material displays excellent mechanical and fracture toughness, making it a prime candidate for biomimetic engineering. In this work, *in situ* X-ray microscopy was performed with Zernike phase contrast to observe both the sample and the diamond indenter tip. Evidence of the internal tubular dentin structure, which is hypothesized to be responsible for many of the material's impressive properties, is also visible from the surface renderings. Two data sets were collected, first at light load and second after partial fracture initiated by cracking around the indenter tip, as shown in Figure 3 below. Investigation of the internal structure can reveal where these cracks initiate and propagate relative to the tubules, helping to explain why bulk dentin displays such exceptional mechanical toughness.

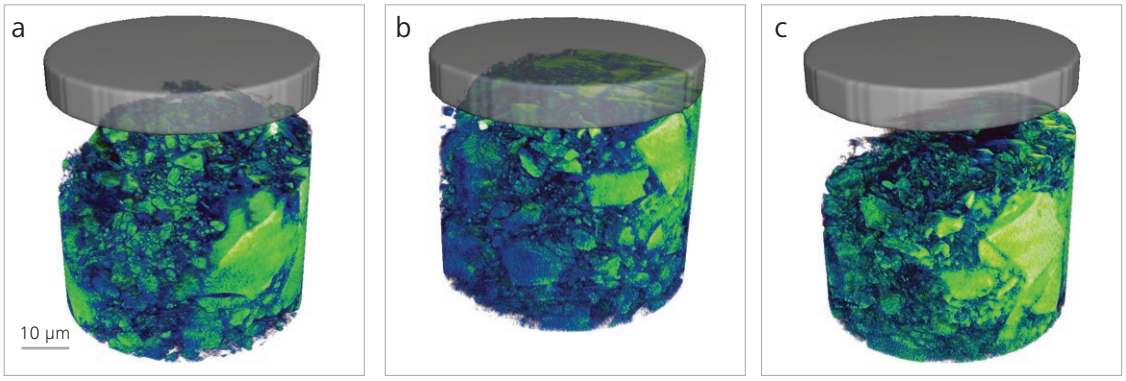


Figure 2. Compressive loading of a porous elastomer. (a) Uncompressed, (b) Compressed, (c) Decompressed

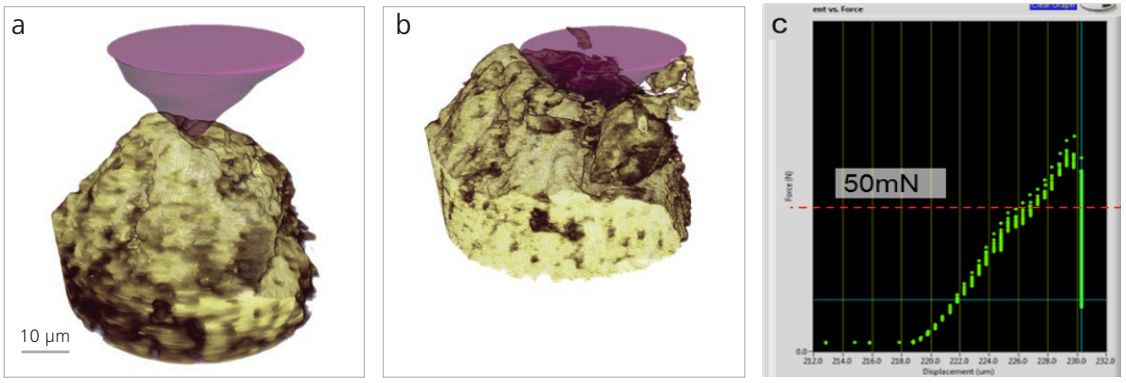


Figure 3. Nanoindentation of dentin. (a) 3D rendering of dentin sample before cracking. The indenter tip is visible above the sample. (b) 3D rendering of the same sample after the indenter tip has been driven into the sample and a piece of dentin has fractured off. (c) Force-displacement curve. The fracture event is clearly visible. Courtesy The University of Manchester.

Outlook

In situ mechanical testing in a nanoscale X-ray microscope applies to a wide range of interests, covering both engineered and natural materials. Many such materials display hierarchical structures, with the behavior at the bulk scale intimately linked to the structure and properties of multiple smaller scales. Nanoscale XRM, sitting in the unique nanometer to micron nondestructive 3D imaging regime, has the potential to help link observations across this vast range of scales, and complement existing methods including *in situ* SEM, TEM, and micron scale XRM. Specifically, Xradia Ultra Load Stage, capable of compression, tension, and indentation loading, offers new capabilities to observe internal processes such as elastic and plastic deformation, crack initiation and propagation, and surface delamination down to the 50 nm scale. While not an inclusive list, this is envisioned to have strong applications in such fields as high strength alloys, coatings, fiber composites, biomaterials, building materials, and engineered foams.

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