Advances in *in situ* nanomechanical testing

Andrew M. Minor and Gerhard Dehm, Guest Editors

In situ nanomechanical testing provides critical insight into the fundamental processes that lead to deformation phenomena in materials. Often, *in situ* tests are performed in relevant conditions such as high or low temperatures, tribological contact, gas environments, or under radiation exposure. Modern diffraction and imaging methods of materials under load provide high spatial resolution and enable extraction of quantitative mechanical data from local microstructure components or nano-sized objects. The articles in this issue cover recent advances in different types of *in situ* nanomechanical testing methods, spanning from dedicated nanomechanical testing platforms and microelectromechanical systems devices to deformation analyses via *in situ* diffraction and imaging methods. This includes scanning electron microscopy, advanced scanning transmission electron microscopy, electron diffraction, x-ray diffraction, and synchrotron techniques. Emerging areas such as *in situ* tribology enable novel insights into the origin of deformation mechanisms, while the evolution of microelectromechanical systems for controlled *in situ* testing provide opportunities for advanced control and loading strategies. Discussion on the current state of the art for *in situ* nanomechanical testing and future opportunities in imaging, strain sensing, and testing environments are also addressed.

Introduction

Methods for nanomechanical testing have become a critical part of materials research, with the ever-increasing importance of nanomaterials and nanoscale phenomena in modern technological applications.[1–3] Small-scale mechanical testing can also enable a "minimally invasive" approach to collecting mechanical data from bulk materials, providing information from individual microstructural components to areas of damage accumulation in materials under harsh environments.[4] By simultaneously observing deformation phenomena and measuring mechanical response *in situ*, it is possible to connect nanomechanical testing information to models that describe both the subtlety and complexity of how materials respond to stress and strain. Furthermore, the *in situ* approach is crucial to obtain insight into the quality of a mechanical test, as small inaccuracies can lead to completely false conclusions. Therefore, quantitative mechanical testing conducted while observing deformation *in situ* is uniquely suited for studying mechanical properties since the fundamental deformation mechanisms are readily observed.

Starting in the late 1950s, *in situ* straining stages for electron microscopes were developed that provided dynamic observations of dislocation motion in metals.[5–7] Electron microscopy is well suited for studying the mechanical properties of materials, since high spatial resolution is required for imaging individual defects in heterogeneous materials. However, complex defect structures lead to complications in connecting high-resolution observations to deformation phenomena that are inherently multiscale in nature. Therefore, multiscale analysis of materials is required to fully understand how a material responds to stress. Nonetheless, *in situ* nanomechanical testing provides the opportunity to apply controlled loading geometries, environments, and sensitive measurements that provide clarity in dynamic microstructural changes.

The study of deformation phenomena has a history going back to the start of high-voltage electron microscopy.[8–10] More recently, the introduction of quantitative measurement devices has enabled precise knowledge of the stress inside a sample.[11,12] Recent reviews have described the impact of combined quantitative measurement and imaging at the individual defect level.[13,14] *In situ* transmission electron microscopy (TEM) nanomechanical testing makes use of the sensitivity of TEM to lattice distortion that allows for visualization of both elastic and plastic deformation via strain fields from a length scale of

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several hundred nanometers down to the atomic-length scale. Novel correlated imaging and diffraction scanning transmission electron microscopy (STEM) techniques have emerged due to the development of fast detection systems of several hundred frames per second. *In situ* scanning electron microscopy (SEM) testing allows for observation of the overall morphology of the deformation. With the recent progress in electron channeling contrast imaging (ECCI), transmission SEM, and electron backscatter diffraction (EBSD), SEM techniques have advanced into length scales that were historically covered only by conventional TEM. *In situ* x-ray diffraction (XRD) testing can identify defect structures and phases at a larger scale than that of TEM and provide access to subtle changes in misorientation. In synchrotrons, focused x-ray beams can probe strains and orientation changes with beam diameters down to ~10 nm.

Direct observations of defect structures

Physical metallurgy has certainly benefited from direct observations of defect structures, as Kacher et al.[15] show in their article in this issue. They discuss mostly TEM-based observations that led to insights into defect–defect interactions and their impact on mechanical properties. Grain boundaries are one of the most common defects in structural materials, but many details of the complex strain accommodation mechanisms at grain boundaries are still unsolved. *In situ* experiments have been reported that deal with inherent mechanisms in metals that relax stress at grain boundaries, such as grain-boundary migration and grain rotation. In this respect, automated crystal orientation mapping (ACOM) performed by electron diffraction inside the TEM gives information on the evolution of grain size and grain orientation under load and guides the analysis of different grain-boundary mechanisms. While this is especially important for fine-grained and nanocrystalline materials, dislocation–grain-boundary interactions are another topic highlighted in their overview. In service, engineering materials are exposed to complex stress states and high strain rates. The recent advances of *in situ* nanomechanical testing covering both of these topics is another central point of their overview.

In situ imaging is most useful in the context of connecting defects and structural features with deformation studies. Due to the multiscale and complex nature of defect structures in deformed materials, correlative microscopy techniques in scanning electron microscopy can provide unique insights. In their article in this issue, Gianola et al.[16] discuss some of the emerging SEM-based methods for identifying defects, including EBSD, ECCI, and diffraction-contrast STEM. EBSD continues to evolve with faster detectors that enable *in situ* mechanical testing combined with ECCI and diffraction-contrast STEM, and higher resolution imaging that allow for defect characterization.
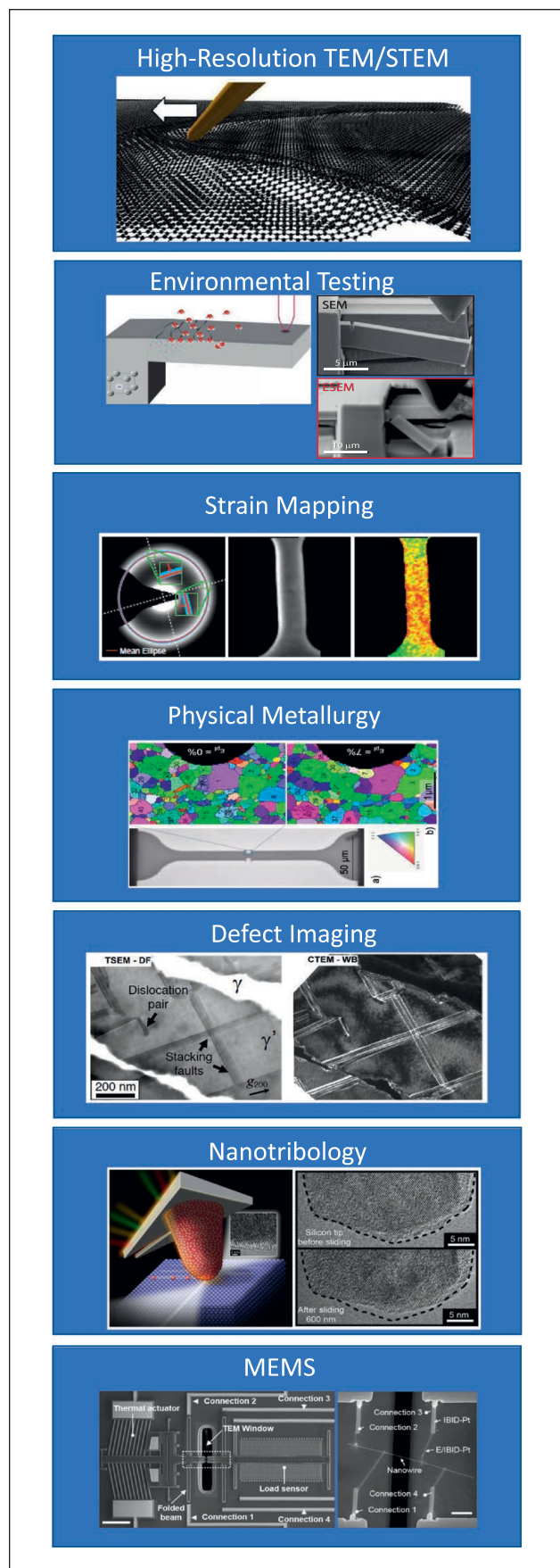
Atomic-scale resolved imaging

In their article, Spiecker et al.[17] carry the discussion to the atomic scale by employing atomic resolved *in situ* TEM/STEM

experiments to study dislocation generation, multiplication, and interaction with hydrogen as well as dislocation–grain-boundary interactions. The strength of these methods is the direct visualization of defects during deformation. An example provided in their article is screw dislocations intersecting a low-angle tilt grain-boundary-inducing kink formation in the transmitted dislocations and jogs in the grain-boundary dislocation array. Such studies are crucial to understand dislocation–grain-boundary transmission stresses and possible resulting strain-rate dependence. Interestingly, functional two-dimensional (2D) materials such as bilayer graphene can also form dislocations as inherent growth defects, which may alter charge transport in devices. Spiecker et al.[17] show that *in situ* nanomechanics allows for manipulation of individual dislocations, which in turn leads to mechanical switching of stacking sequences of the 2D material,[18] a previously unobserved effect.

Local strain measurements

Emerging techniques of measuring local strain around individual defects or groups of defects are discussed by Gammer et al. in their article in this issue.[19] Digital image correlation is one of the most readily available methods for strain measurements using light optical or electron microscopy imaging techniques. At the (sub-)nanometer length scale where high-resolution TEM is employed, geometric phase analysis (GPA),[20] dark-field electron holography (DFEH),[21,22] and four-dimensional (4D) STEM using nanobeam electron diffraction[23–25] have been developed to measure strains. These techniques can also correlate defect structures and mechanical properties. GPA requires an atomically resolved image to map the strains in reciprocal space. DFEH and in-line DFEH take advantage of the phase change of the electron wave traveling through a strained lattice of the sample and need dedicated hardware or computational techniques to acquire the projected strain tensor, and are not yet easily implemented as routine *in situ* techniques.

In contrast, 4D STEM, where an annular detector is used for dark-field imaging and an ultrafast camera is used to simultaneously record the nanobeam electron diffraction pattern of the rastering beam at every position, is a robust correlative technique. Video camera speeds of several hundred frames per second are required for *in situ* experiments and are nowadays available by fast complementary metal oxide semiconductor or direct-detection camera systems. In return, the user must be prepared for large data sets to be analyzed. Big data is routine now for synchrotron experiments, where strain mapping uses either monochromated or polychromated x-rays as probes. Laue diffraction with its polychromatic x-rays avoids tedious sample tilting compared to monochromated x-ray techniques such as nano-XRD, as the polychromatic spectrum always contains wavelengths that fulfill the Bragg diffraction condition. Submicron beam sizes and a high sensitivity to probe local orientation gradients make μLaue diffraction a versatile tool.

High-Resolution TEM/STEM

Environmental Testing

Strain Mapping

Physical Metallurgy

Defect Imaging

Nanotribology

MEMS

While deviatoric strains are straightforward to extract, hydrostatic strain remains undetected as the positions of the diffraction spots remain unchanged.[26] The full strain tensor requires knowledge of the corresponding diffracting wavelength by an energy scan or an energy-sensitive detector. µLaue has been successfully applied for *in situ* compression, tension, and low-cycle fatigue tests.[27–29] New developments using monochromated x-rays encompass fast scanning x-ray microscopy and full-field diffraction x-ray microscopy combining imaging and diffraction as well a coherent Bragg diffraction.[29] While strains are measured with unprecedented accuracy, experimental challenges and tedious data analysis currently prevent routine application of these methods.

Environmental *in situ* nanomechanical testing

The impact of environment is a critical area of *in situ* nanomechanical testing that has expanded in recent years. In their article, Barnoush et al.[30] report on recent progress in extending *in situ* nanomechanics to low (–140°C) and elevated temperatures (1000°C) and the challenges that need to be considered. Similarly, recent advances in strain-rate-dependent *in situ* nano- and micromechanical experiments ranging from creep at 10^{-6} s^{-1} to impact testing at 10^6 s^{-1} are summarized. Barnoush et al.[30] report longstanding challenges in understanding the complex mechanisms of hydrogen embrittlement. This gained new momentum by *in situ* nanomechanical testing. *In situ* nanomechanical testing allows individual microstructure components (grain boundaries, phases, and grain interior) to be separately addressed.

Another topic reported in their article revolves around *in situ* nanomechanical testing of materials exposed to radiation. For harsh environments, *in situ* nanomechanics are key in discriminating individual deformation mechanisms for complex engineering materials and may in future present a tool to monitor materials by extracting only small volumes. Advances in mechanical testing instrumentation and testing methods under such special environments are discussed in the Barnoush et al. article.[30]

Nanotribology

Insights into nanotribology from *in situ* experiments have enabled identification of physical processes occurring at and below a wear surface. In their article in this issue, Jacobs et al.[31] describe key advances in *in situ* nanotribology that have enabled direct observations of processes in the sliding contact, including tribochemistry and subsurface deformation. For example, *in situ* TEM nanotribology has revealed a number of material transformations in layered materials typically used as lubricants such as MoS$_2$[32] and graphite.[33] Studies of

Figure 1. Topical areas related to advanced *in situ* nanomechanical testing techniques covered in this issue. Note: TEM, transmission electron microsopy; STEM, scanning transmission electron microscopy; MEMS, microelectromechanical systems; ESEM, environmental scanning electron microscopy; CTEM-WB, conventional TEM-weak beam; TSEM-DF, transmission scanning electron microscopy-dark field.

corrosive environments have benefitted from *quasi-in situ* AFM-based probing investigations coupled with *ex situ* analysis. Subsurface dislocation processes in metal contacts during *in situ* nanotribology demonstrate the impact of *in situ* testing, and surface adhesion phenomena are readily observed with *in situ* TEM. As Jacobs et al.[31] discuss, breakthroughs in *in situ* nanotribological characterization have the potential to impact a wide array of applications and technologies.

Integrated micro-/nanofabrication

Bhowmick et al.[34] describe how integrated micro-/nanofabrication can lead to incredible control and opportunities for complex testing schemes. Lab-on-chip testing provides material testing schemes with accurate mechanical testing and precise measurements of load and displacement. For example, it is possible to investigate rate effects[35] or combine multimodal testing capabilities such as simultaneous electrical and mechanical measurements.[36] The flexibility of microelectromechanical systems devices leads to adaptation for high cycle fatigue, *in situ* wear and environmental testing. Future designs integrating control of temperature, liquids, and electrochemistry will provide insight into *operando* conditions of materials deformation.

Conclusion

This issue of *MRS Bulletin* describes the state of the art with regard to experimental techniques that provide direct observation and measurement of materials deformation phenomena (**Figure 1**). New methods of imaging defects, measuring local strain and loading samples *in situ* can directly couple to computational modeling and theoretical studies of mechanical behavior. The field of *in situ* nanomechanical testing has proven to be critical for our understanding of structure–property relationships at the most critical length scales for mechanical properties, and future progress in this area will increase the fidelity, resolution, and impact of these techniques.

Acknowledgments

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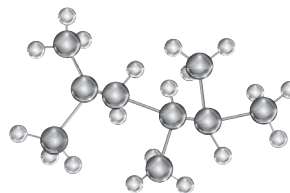
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