In Situ Transmission Electron Microscopy

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Guest Editors

Abstract

The articles in this issue of *MRS Bulletin* provide a sample of what is novel and unique in the field of *in situ* transmission electron microscopy (TEM). The advent of improved cameras and continued developments in electron optics and stage designs have enabled scientists and engineers to enhance the capabilities of previous TEM analyses. Currently, novel *in situ* experiments observe and record the behavior of materials in various heating, cooling, straining, or growth environments. *In situ* TEM techniques are invaluable for understanding and characterizing dynamic microstructural changes. They can validate static TEM experiments and inspire new experimental approaches and new theories.

The technique of *in situ* transmission electron microscopy (TEM) refers to a broad class of experiments whereby the dynamic response of a material to an externally applied stimulus is observed as it happens inside the microscope. Whereas *in situ* TEM may be one of the most novel but best kept secrets of materials science, the key to its importance lies in something we encounter on a daily basis. The following thought experiment may help make this clear:

> Imagine looking at two different pictures of a billiard table, taken within five seconds of each other. The first picture shows a white ball and two colored balls on the billiard table. The second picture shows the white ball alone on the table, in a different location than first pictured. If asked to figure out what happened between shots, we might first ask 1) what happened to the colored balls and 2) why is the white ball in a new spot? We might assume a play was made, and the colored balls are in the pockets. However, from the snapshots taken, we see only the outcome and know nothing about the process that led up to it. To know more about the trajectories, the rebounding or angles of play, we would need to observe, *in situ* (i.e., as the process is happening in real time), the events that took place in the five-second gap between one picture and the next.

The ability to observe, film, and record events as they occur in real time is what *in situ* TEM offers to the world of materials science research. As illustrated in Figure 1, *in situ* TEM enables real-time observations of structure–property–processing relationships, at high magnifications, by employing *in situ* TEM sample holders, which are essentially no more than small laboratories placed in the column of the microscope. These holders, available commercially or in specific research groups, enable environmental changes to the sample, such as heating, cooling, gas and liquid exposure, straining and indentation, and electrical and magnetic biasing (Table 1). In each of these circumstances, the sample environment is controlled from outside the microscope while the sample is observed and its responses are recorded in real time.

From nanoscale observations to biological interactions, advancements in *in situ* TEM are enabling us to observe the known world on a tiny scale. Although *in situ* TEM microscopy has been used for quite some time, emerging aberration-corrected TEMs/ scanning TEMs (STEMs) and the fabrication of microelectromechanical systems–based and piezo-actuated *in situ* holders are profoundly impacting the way *in situ* experiments are performed and the types of observations we are able to make.

In the case of TEM/STEMs corrected for spherical aberration (C_s), additional

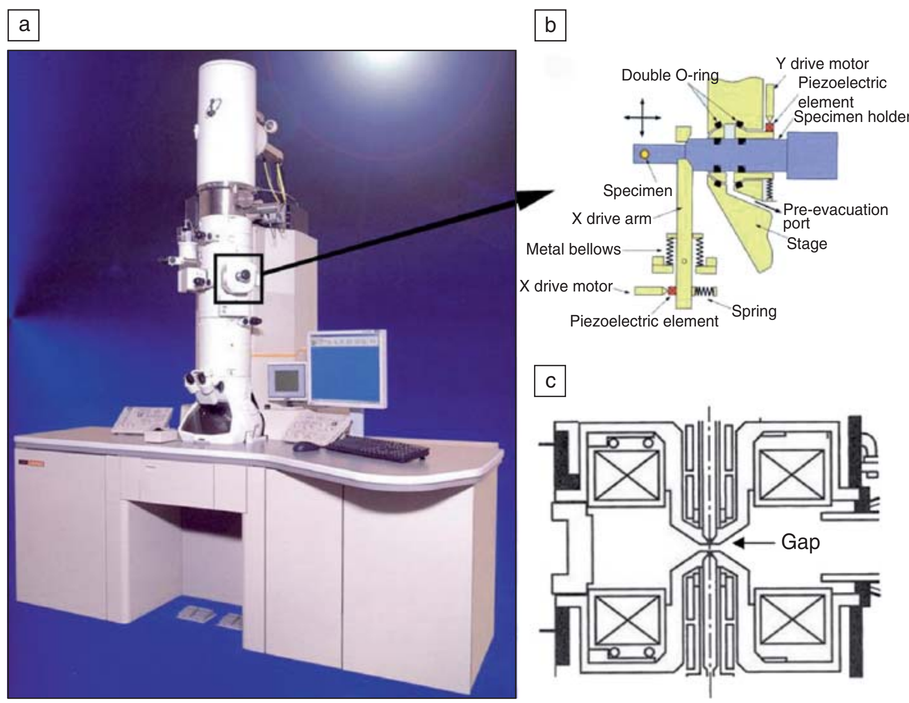

Figure 1. Conventional transmission electron microscope showing the capability for performing *in situ* experiments. (a) The area around the black square depicts the location in the microscope where the specimen holder is inserted into the column for *in situ* observation. (b) A more detailed side view of the specimen holder can be seen. (c) A front view of the pole piece and the narrow pole-piece gap within which the specimen holder shown in (b) needs to be inserted. In the case of aberration-free microscopes, the pole-piece gap can be significantly increased while keeping the resolution high.

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Table I: Variety of specimen holders for *in situ* TEM experiments and possible applications in materials science.

Types of Holders	Possible Applications
In situ heating (up to 1273 K)	Thermal stresses, coarsening of nanoparticles, sintering, grain growth, strain relaxation in heterostructures and nanostructures
In situ nanoindentation	Indentation response of thin films, nanoparticles and nanopillars
In situ straining in combination with cooling (down to 5 K) or heating (to 1300K)	Ductile-to-brittle transitions, microstructural origins of mechanical response, measurements of lattice strains by convergent beam diffraction
In situ STM	Electrical properties of nanostructures, field emission of carbon nanotubes, electromechanical properties of nanowires.
In situ tomography	3D visualization of porosity, nanoparticles, virus morphology.
In situ liquid cell holder	Cell growth and biological interactions, wet chemistry

lenses are incorporated in the microscope column to reduce and/or eliminate the spherical aberration of the objective lenses. The aberration correctors are inserted either above or below the objective lenses to correct the illumination (STEM mode) and imaging (TEM mode) systems, respectively. In the past, the spherical aberration of the TEM lens was minimized by reducing the pole-piece gap. The pole-piece gap is the distance between the magnetic pole pieces, which comprise the magnetic electron lenses and within which the *in situ* holder is located (Figure 1c). When the pole-piece gap is too narrow, as in ultrahigh-resolution pole pieces (~2 mm), only a selected group of *in situ* TEM holders can be accommodated. Pole pieces with a larger gap in uncorrected machines will decrease the resolution significantly; for example, the point resolution in Scherzer defocus for an uncorrected 300-kV TEM decreases from 0.16 nm to 0.27 nm if the gap is widened from 2.5 mm to 20 mm, which is sufficient to eliminate the possibility of high-resolution imaging of most metals, semiconductors, and ceramics. This decrease in resolution can be attributed to the higher chromatic aberration (C_c) coefficients of lenses with larger gap and therefore large focal length. C_c-correction as being developed by the U.S. Department of Energy TEAM project [http://ncem.lbl.gov/TEAM-project/] has the potential to enable sub-Angstrom resolution even for objective lenses with a gap of 20 mm.

While less obvious, C_s and C_c correction provide additional important advantages in improving the spatial coherence and the energy width of the electron beam. By improving the spatial coherence with C_s correction, it is possible to provide higher beam current densities to the sample without converging the illumination to an extent that it limits the transfer of higher order spatial frequencies in images. This enables a higher signal-to-noise ratio and improved time resolution while still maintaining a high information limit—the inherent information conveyed in the image. Additionally, the simpler contrast-transfer function of a C_s-corrected instrument improves the interpretability of the images, an important parameter when considering the large number of images that can be produced at 30 frames per second acquisition rates. The advent of C_c correction compensates for information transfer losses due to inelastic scattering of the electrons. This will improve our ability to image thicker samples, as is desirable in understanding mechanical responses, and in environmental studies where the presence of a gaseous or liquid layer can cause scattering of the electron beam.

Although the goal of the community is to enable widespread use of C_s-corrected and perhaps even C_c-corrected TEM/STEMs, this will still take some years due to the high cost associated with the purchase, installation, operation, and maintenance of such machines. In the meantime, advances in *in situ* holders are enabling advances on non-C_s-corrected machines.

Like all advanced and sophisticated technology, *in situ* TEM works with precise specifications. Whereas the C_s-corrector lenses enable wider pole-piece gaps, the use of MEMS-based and piezo-actuated *in situ* holders permits the integration of devices with a suitable size to be accommodated within narrow pole-piece gaps. In addition, these systems enable a range of experiments to be performed where simultaneous real-time TEM observations and property measurements can be made. These include, for example, measuring electrical current as a function of strain and temperature, measuring stress and strain as a function of temperature, and measuring stress and strain as a function of a magnetic field.

Because of the dynamic capabilities of *in situ* TEM observations, imaging and recording equipment are instrumental parts of the observation process. As needs vary, some experiments profit from a wide field of view, others require high magnification. To accommodate both requirements, *in situ* TEM digital media is continually improving to monitor and record dynamic experiments via video camera, yet retain the ability to record high-quality still images when needed. Wide-angle cameras in the 35-mm port along with a high-resolution charge-coupled device (CCD) camera and an intensified video system below the film camera of the TEM can meet dynamic and still requirements. For dynamic *in situ* events, wide-angle cameras are most often lens-coupled CCD cameras with a fast read out. On the other hand, high-resolution fiber-coupled CCD cameras have relatively slow read outs and narrow areas of view, making them fit for high magnification and slower dynamic experiments. New cameras are now becoming available that combine the high resolution of CCD with reasonable read-out speeds and are expected to greatly improve both the signal-to-noise ratio and the general ease of data collection and manipulation.

Ask any trained TEM student and you will hear that sample preparation is one area where more creative problem solving is always welcome. For the typical 200-keV and 300-keV TEMs, the sample thickness is normally around 50–100 nm to permit electron transparency. Conventional methods, such as jet polishing, microtoming, or ion milling, are still widely used but require highly experienced users and are not well suited for samples thicker than 3 mm. In this regard, the focused ion beam (FIB) is a major advancement for TEM sample preparation. Whereas advanced training is still essential, the FIB permits a high level of flexibility for sample preparation because of its ability to prepare samples from specific locations and with controllable and reproducible geometries, even for the fabrication of MEMS samples.

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In recent years, developments in nanoscience and nanotechnology have brought about an exciting resurgence of interest in *in situ* TEM. This technique plays a crucial role in nanomaterials research, where high resolutions are needed to observe atomic/nanostructures and their properties. Additionally, the fields of TEM and nanomaterials are particularly well suited because the crystal size of nanomaterials is typically comparable or below the thickness of electron transparent samples required by TEM.

Focusing on these and other exciting topics, we have included five articles in this issue of *MRS Bulletin* to illustrate how a variety of *in situ* TEM techniques can be used to solve materials science problems. As the range of materials and applications is extremely broad, this issue provides several different material examples using different *in situ* TEM techniques rather than attempting to cover specific areas of materials research. The articles focus on particular *in situ* TEM techniques and their application to materials. In most of the cases, each technique requires specific *in situ* TEM holders and configurations, whereas dedicated TEM/STEMs are necessary in a few situations, as, for example, for environmental cell studies.

In the first article, H. Saka et al. review the area of *in situ* TEM heating experiments to illustrate the important effect of temperature on the behavior of materials. In fact, many materials phenomena of both fundamental interest and commercial importance occur at elevated temperatures, such as solid–solid and solid–liquid transformations, nucleation and growth processes, sintering, and thermal-induced stresses. In particular, this article discusses the fabrication of specimen-heating holders for successful *in situ* heating in the TEM, the high-resolution observation of solid–state reactions at high temperatures and solid–liquid interfaces. In addition, the authors describe the size dependence of melting temperatures in one-, two-, and three-dimensionally reduced systems, as well as the size dependence of the contact angle of fine liquid metals. The article then turns to a discussion of solid–gas and liquid–gas reactions at high temperatures and the transformation of specific dislocation core reconstructions in silicon at high temperatures.

The second article, written by J. Cumings et al., describes *in situ* TEM capabilities for uncovering information about electric and magnetic properties of materials for operational devices. Magnetic materials encompass a variety of materials that are used in a diverse range of applications, such as media storage, sensors, and actuators. In parallel, many materials are currently required to transport electrical current, such as interconnect structures and various transducers, sensors, and actuators. In order for these materials to be considered for specific applications, it is crucial to understand and monitor their response to an electric and/or magnetic field. Often, these electrical and magnetic properties are determined by nanoscale features that can be most effectively understood through electron microscopy studies, particularly when devices are operated during *in situ* TEM observations, for which a wealth of information is available about dynamics, including metastable and transitional states. Additionally, because the imaging beam is electrically charged, it can directly capture information about the nanoscale electric and magnetic fields in and around devices of interest. This is perhaps most relevant to the growing areas of nanomaterials and nanodevice research. The article describes various methods of obtaining electrical and magnetic information, the dynamics of magnetic domains, and the fabrication of *in situ* devices. Several specific examples of materials systems that have been investigated with these techniques are presented.

The third article, written by P. L. Gai et al., reviews the development of time-resolved, high-resolution environmental scanning/TEM and related methods for directly probing dynamic gas–solid, liquid–solid, and gas–liquid–solid interactions at the atomic level. The unique information available from such experiments has allowed the dynamic nature of nanostructures to be visualized during reactions. This has enabled the development of advanced nanomaterials and processes, including the design of novel green routes to polymers, and the identification of the important processes during catalysis, chemical vapor deposition, and electrochemical deposition.

The fourth article, written by J. M. Howe et al., addresses the use of *in situ* TEM techniques under high-resolution imaging, which is particularly important for the study and development of nanomaterials and nanotechnology. Using various examples, these authors show how *in situ* high-resolution TEM (HRTEM) can be used to understand alloy phase formation in isolated nanometer-sized particles, to understand and measure the mechanical and transport properties of carbon nanotubes and nanowires, and to determine the dynamic behavior of interphase boundaries in nanoscale materials. The use of special holders, the role of C_s correctors, better time resolution, and image storage and processing in such studies are also discussed.

The fifth article, written by I. M. Robertson et al., discusses the capabilities of *in situ* TEM deformation experiments in providing critical and valuable real-time dynamic information for direct investigation of the link between deformation mechanisms, microstructure, and properties. Observing dislocation behavior in real time as a material is deformed provides exceptional insight into how the complex and coordinated behavior of dislocations controls the macroscopic mechanical properties of materials. The transmission electron microscope provides a unique environment in which to observe dislocation motion, which has been a common use of transmission electron microscopy since the instrument was first introduced. Since then, this technique has been used to reveal dislocation behavior and interactions in a range of materials, and it has been instrumental in providing a fundamental basis for modeling mechanical properties under different stimuli and environments. Advances in instrumentation, stage design, recording media, computational power, and image manipulation software are providing new opportunities for not only stimulating the motion of the dislocations but also measuring the macroscopic mechanical response while observing how dislocations behave. Insight gained from past studies and new capabilities are described briefly.

In summary, the articles in this issue of *MRS Bulletin* provide a sample of what is novel and unique in the field of *in situ* TEM. The advent of improved cameras and continued developments in electron optics and stage designs have enabled scientists and engineers to enhance the capabilities of previous TEM analyses. Currently, novel *in situ* experiments observe and record the behavior of materials in various heating, cooling, straining, or growth environments. *In situ* TEM techniques are invaluable for understanding and characterizing dynamic microstructural changes. They can validate static TEM experiments and inspire new experimental approaches and new theories.

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Wang's most recent research focuses on oxide nanobelts and nanowires, *in situ* techniques for nanoscale measurements, self-assembly nanostructures, fabrication of nanodevices and nanosensors for biomedical applications, and nanogenerators for self-powered nanosystem.

He was elected a fellow of American Physical Society in 2005, has received the 2001 S.T. Li prize for Outstanding Contribution in Nanoscience and Nanotechnology, the 2000 and 2005 Georgia Tech Outstanding Faculty Research Author Awards, Sigma Xi 2005 sustain research awards, Sigma Xi 1998 and 2002 best paper awards, the 1999 Burton Medal from Microscopy Society of America, and 1998 China-NSF Oversea Outstanding Young Scientists Award. Details can be found at: http://www.nanoscience.gatech.edu/zlwang. He has authored and co-authored four scientific reference and textbooks and more than 500 peer reviewed journal articles, 55 review papers and book chapters, edited and co-edited 14 volumes of books on nanotechnology, and held 20 patents and provisional patents. Wang is among the world's top 25 most cited authors in nanotechnology from 1992-2002 (ISI, Science Watch). His entire publications have been cited for more than 12,000 times. The H-factor of his publications is 54.

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