









Micromechanical testing

Electrical nanoprobing

Correlated AFM-SEM imaging

SEM signal acquisition and processing

Workshop on In-Situ Microscopy Solutions

This series of one-day workshops will take place in two locations in the USA, providing a unique opportunity to discover the in-situ solutions developed by the four partners of the alliance. If mechanical, electrical and optical characterizations methods are of interest then please join us! A wide variety of applications will be presented to cover different aspects of your research projects.

Locations

09:00 - 09:45

23 April 2024 | Sunnyvale, CA | 9am – 3pm | Covalent Metrology – <u>Sign up here</u>
25 April 2024 | Cambridge, MA | 9am – 3pm | Massachusetts Institute of Tech.
(MIT.Nano) – <u>Sign up here</u>

Cambridge Program (25 April 2024)

Registration and Coffee

09:45 - 10:00	Introduction of the In-situ Microscopy Alliance (IMA)
10:00 - 10:30	Recent innovation in small-scale in-situ mechanical properties testing, Dr.
	Nicholas Randall, Alemnis
10:30 - 11:00	Mechanics of architected materials through the lens of in situ
	characterization, Prof. Carlos Portela, MIT
11:00 - 11:15	Break and networking
11:15 - 11:45	Latest updates in electro-optical characterizations and failure analysis, Mr.
	Karl Boche, Imina Technologies
11:45 - 12:15	Metal layers short localization with EBAC and FIB circuit modifications, Mr.
	Karl Boche
12:15 - 13:30	Lunch
13:30 - 14:00	AFM-in-SEM - step forward for in-situ correlative microscopy, technology
	and applications, Mr. Jan Neuman NenoVision
14:00 - 14:30	Benefits of AFM-in-SEM for applications in material science and battery
	research, Mr. Jan Neuman, NenoVision
14:30	End of the seminar, open discussion

Abstracts

Recent Innovation in Small-Scale In-Situ Mechanical Properties Testing

Nicholas Randall

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In situ SEM micro- and nanomechanical testing is an indispensable technique for materials design as well as for fundamental mechanics research. Many new protocols and testing geometries beyond traditional nanoindentation now enable the study of microstructure– property relationships, material intrinsic behaviour including orientation-dependence and plasticity, fracture dynamics, or the performance of novel micro-3D-printed metamaterials, to name but a few.

Thanks to its versatility, in situ SEM-based micromechanics is contributing to numerous scientific domains, including thin films and coatings, metallurgy, glasses and ceramics, semiconductors, biomechanics, or architectured materials. Performing micromechanical tests in situ in a SEM offers two important advantages: (1) unmatched control, stability, and positioning accuracy, and (2) the possibility to perform unique correlative experiments based on, for example, the combination of mechanical data with direct imaging or EBSD measurements.

An increasingly important branch of micromechanical testing can be found in the simulation of real-world, extreme operation conditions, such as high temperatures in engines, cryogenic temperatures in hydrogen storage, dynamic loading under shock or impact, high frequency cyclic fatigue, or a combination thereof. Progress in the understanding of material behaviour at such conditions is clearly linked to the availability of laboratory equipment that can perform reliable tests under such conditions.

New correlative methods of in-situ micromechanical testing will be presented, including the combination of the Alemnis ASA with the Nenovision AFM and Imina electrical nanoprobing systems.

Mechanics of architected materials through the lens of in situ characterization

Somayajulu Dhulipala¹, Rachel Sun¹, Thomas Butruille¹, Yun Kai², Jet Lem¹ and Carlos M. Portela^{1,2}

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Architected materials across length scales—from nanometers to centimeters—have demonstrated unique mechanical properties enabled by a variety of 3D material morphologies. Significant advances in our understanding of these materials have pointed to structure-property relations that lead to unique macroscopic mechanical properties. Characterization of these novel materials has relied on stress-strain curves which have proven useful to determine effective mechanical properties. However, only in situ observation of these materials has uncovered the complex mechanisms that lead to said responses. As interest in their extreme-condition responses grows, the requirement for nanosecond temporal resolution in imaging techniques adds another challenge.

We present our efforts leveraging in situ characterization with high spatial and temporal resolution to characterize architected materials across various regimes. Quasi-statically, we present in situ nanomechanical characterization on two types of architected materials, where visualization evidences surface-stiffening and nano-shell-buckling responses. Dynamically, we present microparticle impact experiments on architected materials, where ultra-high-speed imaging sheds light on energy dissipation mechanisms. Finally, we present efforts employing a custom laser-ultrasonics characterization technique to obtain nanometer and nanosecond vibrational signatures of 3D micro-architected materials, providing a unique non-destructive characterization route for dynamic mechanical properties and structural health monitoring.

Latest updates in electro-optical characterizations and failure analysis

Karl Boche

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Increasingly complex semiconductor device developments, such as three-dimensional device architecture, pose serious challenges in failure analysis. To ensure efficient and safe device operation, engineers need to localize and understand electrical failure in elements with complex shapes and overlapping structures and fields. It becomes increasingly hard to interpret images and to correctly distinguish between Electron Beam Induced Current (EBIC) and Electron Beam Absorbed Current (EBAC), or between Resistive Contrast Imaging (RCI) and Electron Beam Induced Resistance Change (EBIRCH). This trend poses two somewhat opposite requirements on the failure analysis workflow: on one hand, more complex data has to be collected, but at the same time, there is a need for more intuitive data visualization and interpretation.

In this talk, we will show several examples that illustrate how to combine multi-channel imaging and color coding to bring this much-needed improvement.

AFM-in-SEM - step forward for in-situ correlative microscopy, technology and applications

Jan Neuman

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Correlative in-situ microscopy, which combines the benefits of different imaging systems, has become an essential tool helping us to understand the complexity of the sample properties. For these reasons, correlative microscopy is one of the hot topics of nowadays research. When we imagine a combination of two complementary techniques, atomic force microscopy (AFM) and scanning electron microscopy (SEM), this setup has several advantages, such as the complexity of the measurement, in in-situ conditions, and with precise localization to the area of interest.

To be able to combine these techniques, NenoVision company has developed a unique Atomic Force Microscope (AFM), LiteScope[™], for easy "plug & play" integration into the SEMs. The connection of AFM and SEM enables the merging of the strengths of both techniques, resulting in effective workflow and possibilities of complex sample analysis that was difficult or readily impossible by conventional, separate AFM and SEM instrumentation.

During the presentation, we will select and demonstrate the performance and capabilities of the AFM-in-SEM technique on several examples chosen from a broad range of applications such as batteries, semiconductors, and material science. We will show in-situ correlative characterization of different material properties analyzed by the AFM, such as high-resolution topography, mechanical, electrical, and magnetic properties, and correlated with SEM images.

Benefits of AFM-in-SEM for applications in material science and battery research

Jan Neuman

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During the talk, we will demonstrate the benefits of AFM-in-SEM characterization capabilities on various examples from the material science area with a focus on battery materials and components.

In-situ correlative microscopy using AFM-in-SEM enables the analysis of various properties such as mechanical, electrical, or magnetic. In combination with SEM, we will present how such characteristics benefit the analysis of 2D materials, metals, nanostructures, and other field analyses.

Highly air-sensitive samples from the battery industry are difficult to work with, especially when the analysis requires multiple instruments. Such is the case with the cathode active material (NCM) dispersed within the solid electrolyte SE. While Atomic Force Microscopy (AFM) can map the conductivity of the grains in electrolytes, it is not feasible to perform such measurement on a sample degraded by exposure to air and humidity. We will demonstrate how in-situ analysis can reveal crucial information about cathode materials, their degradation, and characteristics of failures significantly influencing the overall battery performance. Additionally, we will present the overall analytics workflow, enabling the analysis of battery materials and components at the nano/microscale level.