Small-Scale Mechanical Testing at Michigan Technological University
Houghton, MI

The small-scale mechanical testing systems at Michigan Tech enable state-of-the-art ambient, non-ambient, in-operando and in-situ mechanical testing of materials at nanometer and micrometer length scales. Both standard and highly customized test methods are routinely used to investigate metals, ceramics, polymers, composites, semiconductors, biomaterials and structured devices. Suitable test specimens come in a wide variety of forms and physical geometries: bulk materials, thin films, coatings, modified surfaces, micro-pillars, fibers, membranes, hydrogels, MEMS, microfluidic and micromachined devices to name just a few. Testing can be performed over the temperature range of -20 to 100 °C and wet cell capability is available to mimic physiological environments for biomaterials or to create a protective oil barrier on highly reactive surfaces such as lithium. Tech's modular actuators are capable of routinely performing but not limited to quasi-static and dynamic nanoindentation (both have high-speed mapping capability), multi-axis loading, micro-pillar compression and micro-tensile testing on small diameter fibers and wires as well as the ability to utilize feedback control for operation as a load cell. Among the properties that can be measured are the following:

- Hardness, H
- Elastic modulus, E
- Storage modulus, E'
- Loss modulus, E''
- Tan δ
- Creep compliance function, $D(t)$
- Stress relaxation modulus, $E(t)$
- Constitutive creep parameters, A and n
- Fracture toughness, K_c
- Yield strength/stress-strain curve, σ_y and n
- Residual stress, σ_r

Our primary objective is to provide the critical information that will directly enable transformative insights into the complex coupling between the microstructure, its defects and the mechanical behavior of materials. To that end, we systematically investigate how the test specimen's mechanical properties change near free surfaces and interfaces or how they change as a function of strain, strain rate, temperature, processing, microstructural length scale, molecular structure, volume constraints, aging, disease, hydration or loading conditions, e.g., shear, tension, compression or some combination of all three. Beyond advancing structure-property relationships, quantifying these effects leads to improved inputs for modeling and design work and advances our overall understanding of material and device performance. For our industrial partners, this directly translates into a competitive advantage in the marketplace by enabling more accurate simulations and minimizing the time required to develop successful products. Performing meaningful experiments at micron and sub-micron length scales is, however, fraught with numerous experimental challenges. Therefore, we invest significantly in the process of developing and vetting novel experimental methods. When experimental results cannot be verified using standard reference materials, confidence in our data is established through rigorous efforts to identify and eliminate potential sources of experimental error. In the end, we strive to implement the best possible set of experimental conditions that enable the most reliable measurements possible.

Capabilities at Michigan Tech

The flagship characterization facility at Tech is the Applied Chemical and Morphological Analysis Laboratory (ACMAL), which houses sample preparation equipment and an extensive array of optical and electron microscopes, atomic force microscopes and x-ray diffraction instruments. In addition to ACMAL, Tech operates a versatile suite of 6 small-scale mechanical testing platforms in which the user has direct control over the way the test is performed and how the raw data are recorded, reduced and analyzed. Collectively, these systems equip Tech's students and faculty with the tools needed to help solve the most relevant and challenging problems in science and industry. The small-scale mechanical testing systems at Tech are:

Nanomechanics, Inc. (Oak Ridge, TN)
1. iNano, nanoindentation system
2. InSEM III, mechanical properties microprobe
3. Non-ambient system on the InSEM III platform
4. Multi-axis system on the InSEM III platform

MTS Systems Corporation (Minneapolis, MN)
5. NanoIndenter XP
6. NanoIndenter DCM

Overview of the iNano

The iNano, shown in Fig. 1, is latest generation of the highest performance ambient nanoindentation system currently available on the market today. Its key specifications are the following:

- InForce 50 nanomechanical actuator (electromagnetic actuator and capacitive displacement sensor)
  - Travel: ~50 μm
  - Displacement noise floor: 0.25 nm at 20 μs (well controlled laboratory conditions)
  - Maximum load: 50 mN
  - Typical dynamic characteristics: mass = 350 mg, stiffness = 200 N/m, damping coefficient = 0.1 Ns/m, resonant frequency = 120 Hz
  - Load frame stiffness ≥ $1E^{+}6$ N/m
- Frequency specific measurement capability
  - Frequency range of 1 to 120 Hz
  - Time constant at 100 Hz is 50 ms (5 waves of data at the drive frequency)
- InQuest high speed controller electronics
  - Data acquisition rate: 100 kHz
  - Closed loop CPU control rate: 500 Hz (2 ms)
  - Displacement time constant: 20 μs
- InView control software and CPU
  - Contains all aspects of instrument control, test design, reporting and data analysis. InView allows the user to easily create and implement virtually any load or displacement-time history.

FIG. 1. The iNano mechanical testing system.

Among the enabling technology of the iNano is its ability to achieve less than 1 nm of noise in the displacement signal while using a measurement time constant of 20 μs, i.e., there is no averaging of fast time constant data to lower the noise floor. Coupled with a data acquisition rate of 100 kHz, the critical outcome is that the iNano offers unparalleled ability to perform high-speed testing and unmatched ability to capture sudden events such as strain bursts, fracture, film delamination, and dynamic ringing. A sampling of iNano's unique testing capabilities are:

- Basic load-unload experiments performed in less than 1 s per test
  - Mitigates thermal drift
  - Thousands of indents in a single night of testing
    - Spatially resolved hardness and modulus maps
    - Remarkably robust statistics
- Frequency specific experiments (f = 100 Hz, $\dot{P}/P$ = 1 s^{-1}) performed in less than 20 s per test
  - Minimizes thermal drift
  - Spatially resolved hardness and modulus maps as a function of indentation depth
- Frequency specific experiments performed at strain rates ranging from 0.01 to 1 s^{-1}
  - The range in $\dot{P}/P$ is currently limited to 2 orders of magnitude, but work is underway to push the upper limit by at least a factor of 10. Note that significantly higher strain rates for creep testing can be achieved by utilizing ramp or step loading rather than maintaining a constant value of $\dot{P}/P$.
- Frequency sweep experiments performed over the range of 0.01 to 120 Hz
  - Over this entire frequency range, the dynamic motion of the actuator in free space can be accurately modeled by a single degree of freedom, simple harmonic oscillator to within 3% or better
    - Measured properties over 4 decades in frequency without having to resort to time-temperature superposition

Overview of the InSEM III

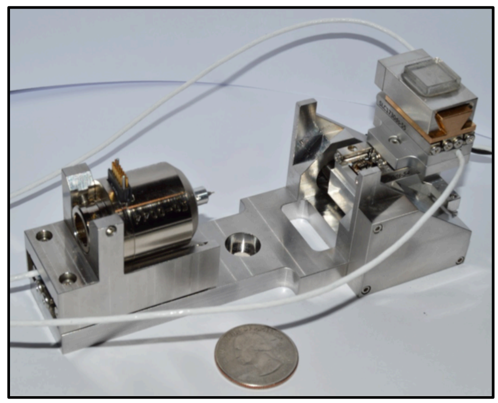

FIG. 2. The InSEM III mechanical testing system.

The InSEM III, shown in Fig. 2, is the world's most technologically advanced in-situ mechanical testing platform. In addition to running in the scanning electron microscope, InSEM III's universal microscope-to-indenter calibration procedure allows it to be operated in conjunction with any microscope capable of accommodating its 13 x 5.4 cm footprint and 5 cm height; e.g. optical, optical interference, raman and atomic force microscopes are among the possibilities. InSEM III can also be operated in a glove box, as it requires only two electrical feedthroughs (LEMO and Fischer). Like iNano, InSEM III incorporates the InForce 50 nanomechanical actuator, InQuest high-speed controller electronics and InView control software and CPU. The compact system is housed in a stiff cradle assembly that attaches to the microscope stage. The test specimen's X-Y position and the gross normal motion of the actuator are controlled by 3 piezo micropositioners, 2 mounted behind the test specimen and 1 mounted behind the actuator. Equipped with optical encoders and closed loop control, the micropositioners provide a minimum step size <10 nm, 20 mm of travel, vacuum compatibility and an axial stiffness ≥ $1E^{+}6$ N/m. By virtue of the higher resonant frequency of InSEM III's load frame, the useable frequency range for dynamic characterization extends from 120 to 500 Hz.

InSEM III's temperature capability

The InSEM III platform further extends state-of-the art nanomechanical characterization by providing temperature testing capability over the range of -20 to 100 °C, which spans the usable temperature range for all biomaterials, the vast majority of polymer and battery systems and a significant fraction of fuel cell applications. The accuracy and stability of the temperature system are ± 0.5 and 0.05 °C, respectively. The upper limit of 100 °C was chosen by design because it enables 3 important advantages over systems designed to achieve higher temperatures. These advantages are:

1. *Superior stability*. The entire measurement system is temperature controlled in a sealed copper block. This simple design significantly reduces thermal drift and completely mitigates the need to incorporate complex thermal management systems. The key outcome is a testing environment that is significantly more stable than that of most ambient systems.
2. *Lower operational costs*. At temperatures of 100 °C and below, the system is much less expensive to operate because it uses fewer consumables such as tips, heaters and custom mounting fixtures.
3. *Higher productivity*. The absence of localized, independent heating and cooling systems for the test specimen and indenter tip make the instrument easier to operate. As a result, its productivity is nearly on par with that of any ambient system.

<u>InSEM III's multi-axis capability</u>

The InSEM III platform can also be arranged with a second actuator mounted orthogonal to the normal loading axis. In this configuration, the InSEM III becomes a novel 2-dimensional system that enables the second actuator to create shear stresses precisely at the surface of an ultra-thin film (5 to 100 nm thickness) or at a prescribed depth relative to any number of microstructural features such as the average dislocation spacing. The system is specifically designed to minimize crosstalk between the axes and deliver nearly identical dynamic characteristics in both directions. These experiments will serve to further advance our basic scientific understanding of the fundamental mechanisms of plasticity and how they can be uniquely probed and investigated through small-scale experiments.

While the most challenging experiments are generally best suited for the iNano and InSEM III, the well established NanoIndenter XP and DCM provide complimentary capabilities. Among the most notable features of the XP is it's 600 mN load limit, which is over 10 times more than that of the InForce 50. In addition, the modularity of these platforms enables the assembly of a system that is adaptable to in-situ x-ray diffraction experiments at the Advanced Photon Source in Argonne National Lab. Collectively, the iNano, InSEM III, XP and DCM provide state-of-the-art small-scale mechanical characterization capabilities at Michigan Tech that meet or exceed the ability of top labs the world over.

<u>Next Generation Nanomechanical Measurement System</u>

Michigan Tech is also a partner institution in a 5-year, $2.2M instrumentation project funded by the National Science Foundation, the University of Tennessee (UT) and Nanomechanics, Inc. The 2014 award is titled, "Development of and Broad-Based Materials Research with the Next Generation Nanomechanical Testing Laboratory." When fully developed, the instrument will be operated as a national shared user facility in the Joint Institute for Advanced Materials at UT. The new instrument, schematically illustrated in Fig. 3, will be capable of performing a wide variety of mechanical tests including indentation, compression testing, tensile testing, and dynamic mechanical analysis, all with nano-scale precision and accuracy. Among the instrument's significant new capabilities are the following:

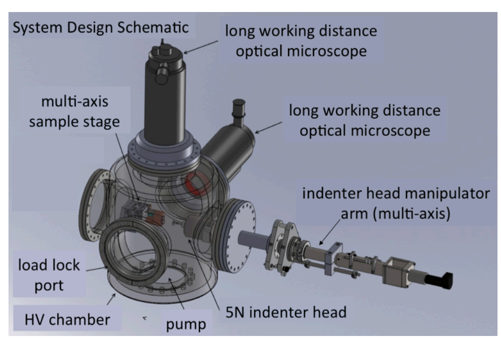

FIG. 3. Schematic illustration of the new nanomechanical testing laboratory.

- Localized, independent heating of the sample and actuator probe to at least 1,100 °C, significantly greater than any instrument on the market today.

- A revolutionary laser interferometric displacement measurement system (LIDM) with sub-nanometer resolution to eliminate longstanding problems related to thermal drift and load frame compliance.
- High vacuum (~10^{-7} Torr) to protect specimens from oxidation and contamination and to aid in thermal stability and LIDM performance.
- Provisions for testing in some gaseous environments at controlled pressures.
- Multiple tip, grip, and probe geometries made from a variety of materials that will perform well when diamond becomes unstable at elevated temperatures.
- Ultra high-speed data acquisition (~1 MHz and the requisite time constants) enabled by the LIDM to permit characterization of rapid deformation events such as pop-in, pop-out, shear banding and fracture.
- Rapid throughput hardness and modulus mapping with a spatial resolution better than 100 nm for detailed property mapping of surfaces.
- 5 N load capability with load resolution of ~10 nN to permit testing of a variety of specimen sizes
- Enhanced dynamic measurements using phase lock amplifiers and fast Fourier signal analyzers for development of transformative dynamic mechanical characterization methods.
- Two, synchronized orthogonal long working distance (LWD) optical systems with submicron resolution to provide enhanced alignment capabilities and to facilitate digital image correlation.
- Digital image correlation (DIC) for measurement of localized strain at the specimen surface.
- Five axes of piezo motion control with nanometer resolution for unprecedented alignment of indenter tips and probes relative to the specimen or specimen features.
- Tunable damping of the force actuator that will revolutionize our ability to perform dynamic mechanical tests and enable novel investigations of rate effects on deformation and fracture.
- Lateral force and displacement measurement for conducting nano-scale friction and wear studies and assessing thin film adhesion.