IN-SITU SEM/FIB
NANOMECHANICAL TESTING

FT-NMT03
NANOMECHANICAL TESTING SYSTEM
The FT-NMT03 Nanomechanical Testing System is a nanorobotic system for the direct and accurate in-situ SEM/FIB measurement of the mechanical properties of nanostructures. Testing principles such as compression, tensile, cyclic or fracture tests are enabled by applying a load with a microforce sensor onto the nanostructures while using position encoders to measure their deformation. From the resulting force-versus-deformation (stress-versus-strain) curves, the material properties of these nanostructures are quantitatively determined. Furthermore, through the combination with sample holders that feature electrical connections, the combined electrical and mechanical properties of nanostructures can be quantified.

Most nanomechanical metrology applications require complex sample preparation steps prior to the measurement. For this reason, the FT-NMT03 also features micro- and nanohandling capabilities using either force-sensing microgrippers or sharp, force-sensing tungsten tips, which enable the pickup, placing and attaching of nanostructures to a testing substrate. This combination of electro-mechanical metrology and nanohandling capabilities provides a complete solution for most nanomechanical testing applications.

**Features**

- Quantitative nanomechanical testing and simultaneous imaging with the SEM
- Force sensing range from 0.5 nN to 200 mN (9 orders of magnitude)
- Displacement sensing range from 0.05 nm to 21 mm (9 orders of magnitude)
- 5-Axis (x, y, z, rotation, tilt), closed-loop sensor-sample alignment
- Measurements at the analytical working distance of the SEM/FIB (down to 4 mm) for optimal imaging resolution and simultaneous ion beam milling and deposition (FIB)
- Ability to perform a large range of mechanical tests, such as compression/tensile, bending, shear, cyclic, fracture tests in both position and force controlled mode
- The position encoders on all axes (x,y,z and rotation/tilt) enable measurement automation for the testing of a large sample number and property maps on samples
- Simultaneous mechanical and electrical analysis of nanostructures enabled by the included electrical testing module (sample holder with 6 electrical connections)
- Electrically conductive force sensing probe tips to reduce charging effects
- Correlation between mechanical measurements and other in-situ SEM/FIB analysis such as EDX or EBSD and the simultaneous ion beam deposition or milling (FIB)
- SEM sample stage mount enables fast installation and removal
- Modular design enables the adaptation of the instrument to the testing requirements of various sample types and SEM/FIB configurations
- Compact instrument design enables the integration into most SEM/FIB chambers
- Customizable measurement procedures and principles
The FT-NTP Nanomechanical Testing Platform is a 5-axis nanorobotics system that can be integrated into most full-sized SEMs and enables the accurate nanomechanical testing of micro- and nanostructures.

The FT-nMSC Modular System Controller interfaces the Nanomechanical Testing Platform and acquires the synchronized force and position data. A key feature of the Modular System Controller is the hardware-level sensor protection mode to prevent damaging the Microforce Sensing Probes and Microgrippers.

The FT-nHCM Hand Control Module with two joysticks enables the user-friendly control of the Nanomechanical Testing Platform.

The SEM-specific flange with electrical feedthrough connectors.

The FT-SH Sensor Head Set, consists of 4 different sensor heads for the user-friendly mounting of the FT-S Microforce Sensing Probes for either in-plane or out-of-plane nanomechanical measurements at different working distances.

The FT-SB Sample Base Set, consists of 4 different sample bases for the user-friendly mounting of the samples at different orientations and heights using either a 1” or ½” SEM stub.

The FT-ETB Electrical Testing Base Set, consists of 2 different electrical testing bases that allow the electrical connection to the sample chip (by wire bonding) and their user-friendly mounting into the Nanomechanical Testing Platform.

The FT-S Microforce Sensing Probes and FT-G Microgrippers (sold separately) are available for mechanical testing and microassembly at different force and size scales.

Due to the compact size of the MEMS-based FT-S Microforce Sensing Probes, the FT-NMT03 Nanomechanical Testing System can operate at the analytical working distance of the SEM. Furthermore, the compact design also enables the simultaneous ion beam milling or deposition while mechanical testing in a FIB. This is crucial for the attachment and removal of samples to the force sensing probe (tensile testing application).
The FT-NMT03 Nanomechanical Testing System is a 5-axis instrument for in-situ SEM measurement of the mechanical properties of micro- to nanometer sized structures. The small instrument form factor of 100 mm x 35 mm x 71 mm enables the integration into most SEM/FIB chambers. A MEMS-based microforce sensor is mounted on a 3-axis piezoelectric actuator with a range of 21 mm x 12 mm x 12 mm. The microforce sensor enables the measurement of forces ranging from 0.5 nN up to 200 mN. The angular alignment of the sensing probe to the sample is enabled by two rotation/tilt stages on which the sample is mounted. All five actuators are equipped with optical-grating-based position encoders for high resolution closed-loop control and measurement automation. A linear, flexure-based piezo-scanner with capacitive position feedback enables measurements with a resolution down to 0.05 nm. The combination of these six actuators with the MEMS-based force sensor enable force-displacement measurements ranging from 0.05 nm to 21 mm (9 orders of magnitude). Due to the small size of the microforce sensor and the compact configuration of the actuators, the measurements can be visualized at the optimal (native) working distance of the SEM, which is usually 5 mm to 10 mm.

### Mechanical Testing Specifications

- **Force sensing**
  - Maximum force: 200 mN
  - Force resolution: 0.5 nN (at 10 Hz)
  - Measurement frequency up to 96 kHz

- **Displacement sensing**
  - Coarse
    - Displacement range: 21 mm
    - Displacement resolution: 1 nm (at 10 Hz)
    - Measurement frequency up to 50 Hz
  - Fine
    - Displacement range: 25 μm
    - Displacement resolution: 0.05 nm (at 10 Hz)
    - Measurement frequency up to 96 kHz

- **5-axis force sensor to sample alignment**
  - X, Y, Z closed-loop positioning with a range of 21 x 12 x 12 mm and a resolution of 1 nm
  - Tilt and rotation by 90° and 360° respectively with 35 micro-degrees resolution

- **Force sensing tip options**
  - Flat punch with a size of 50 x 50 μm
  - Tungsten tip with tip radius of <0.1 μm or <2 μm
  - Spherical tips with radius of 125 μm or 25 μm
  - Do-it-yourself customization option

- **Overall system dimensions**
  - Length ~ 100 mm
  - Width ~ 71 mm
  - Height ~ 35 mm
Three-axis nanopositioning platform with high-resolution position encoders, enabling movements over a range of $12 \times 12 \times 21$ mm with 1 nm resolution

2 FT-S Microforce Sensing Probe with a force sensing range from 0.5 nN to 200 mN (sold separately, see page 9)

3 Sample tilt stage with rotational encoder (optional) for the closed-loop angular sample alignment with 35 micro-degrees resolution

4 Flexure-based, linear piezo scanner with capacitive position encoders for continuous and fast movement over a range of 25 $\mu$m with 0.05 nm resolution

5 Sample holder base compatible with 1” and ½” SEM stubs

6 Sample rotation stage with rotational encoder (optional) for the closed-loop angular sample alignment with 35 micro-degrees resolution

**SELECTED SYSTEM CONFIGURATIONS**
The FT-W1003 Mechanical Testing and Handling Software Suite is provided together with the FT-NMT03. The software suite consists of three parts:
- FT-WFS Micromechanical Testing Software
- FT-WGS Microhandling Software
- FT-WMS Modular Mechanical Testing Software
The FT-WFS and the FT-WGS are graphical user interfaces (GUIs) for MS Windows, which enable user-friendly plug-and-play type micromechanical testing and microassembly. The FT-WMS is a library based on National Instruments’ LabVIEW for the creation of customized micromechanical testing programs.

**FT-WFS Micromechanical Testing Software**

The FT-WFS Micromechanical Testing Software has been developed to perform automated force-position-time measurements for the testing of mechanical properties such as:
- Stiffness
- Elastic and plastic deformation
- Linearity
- Hysteresis
- Breaking strength
- Dimension/topography
- Deflection range
- Adhesion force
- Actuation force

The user-friendly graphical user interface (GUI) features an automated contact detection mode that enables a fast and safe approach of the sensor to the sample. The software enables the measurement visualization (e.g. force vs. displacement or force vs. time data), recording and data exporting (.txt or .xls).

The FT-WFS Micromechanical Testing Software Suite enables automatic, repeated measurements at a single position or at multiple positions on the sample. This function is used for cyclic testing or for array measurements in the area of interest. Typical applications for array measurements are the creation of topography maps or stiffness maps.

**FT-WGS Microhandling Software**

The FT-WGS Microhandling Software enables the user to perform sophisticated pick-and-place operations and microassembly tasks in an intuitive way, using a graphical user interface (GUI). The most important software features are:
- High-precision positioning of the microgripper
- Closed-loop gripping force control
- Microgripper force feedback, measurement data visualization and exporting (.txt or .xls)
- Software limit switches to define a safe working area and prevent crashing the gripper
- Position save and return function for repetitive handling and assembly tasks
The FT-WMS Modular Mechanical Testing Software enables the user-friendly development of customized micromechanical testing programs. This National Instruments’ LabVIEW based software library consists of predefined building blocks that can simply be assembled and reconfigured to develop custom micromechanical testing sequences. The simple drag-and-drop process of building blocks requires only a minimum of programming knowledge.

While creating a new and customized testing sequence, the FT-WMS Modular Mechanical testing software automatically generates the elements for the graphical user interface such as controls and graphs. Furthermore, the mechanical testing software library can be combined with any other LabVIEW compatible software and hardware. An extensive documentation together with sample programs are provided to further simplify the development of your own testing sequence.

1. Graphical user interface of a custom micromechanical testing program
2. Selection of the graphical user interface controls of the FT-WMS Modular Mechanical Testing Software
3. Block diagram of a custom micromechanical testing program
4. Selection of the function library in the FT-WMS Modular Mechanical Testing Software

FEATURES

- User-friendly graphical user interfaces (GUIs) for MS Windows
- Visualization, recording and exporting of measurement data
- Contact detection allows a safe sensor to sample approach
- Compression / tensile testing, adhesion testing, deflection testing
- Cyclic, fatigue and creep testing
- Automated line, surface and volume measurements
- Software library for customized testing principles and sequences
- Documentation and tutorials for the creation of customized testing principles and sequences
APPLICATION EXAMPLES

NANOMECHANICAL TESTING

CHARACTERIZATION OF LIGHTWEIGHT NANOCOMPOSITE MATERIALS

The systematic development of new, lightweight, high-strength materials requires the accurate investigation of their mechanical properties. Complex composites based on nanoscale structures such as fibers, particles and bubbles are receiving growing attention due to their ability to increase the specific strength and stiffness of materials. The depicted work by Prof. Daniel S. Gianola from UC Santa Barbara demonstrates the incorporation of stiff, hollow microparticles (also known as bubbles) into a polymeric matrix. For the development of such lightweight materials, tailoring the mechanical properties of the hollow microparticles is crucial. Through the use of in-situ SEM nanomechanical testing, Prof Gianola could demonstrate that thermal treatment of these nanoparticle-shelled bubbles will result in an enhancement of the stiffness and strength by a factor up to 14.

IN-PLANE NANOMECHANICAL TESTING OF 3D METALLIC NANOPILLARS

Because of the high surface-to-volume ratio, nanostructures are ideal for the creation of high sensitivity sensors, such as gas detectors. Using a combination of nanolithography and electroplating, 3D metallic nanostructures, can be fabricated. Both, the resulting material properties and geometry have high variations. Quantitative insights into the behavior of these nanopillars is obtained by nanomechanical testing. Image courtesy: Prof. Bradley Nelson, ETH Zurich, Switzerland

STABLE COMPRESSION TESTING OF MICROSCAFFOLDS

Microscaffolds are used in various areas such as in material science for the creation of ultra-light materials or in biology as a cellular environment with a predefined mechanical rigidity, used for the growth of artificial tissues. Compression testing of scaffolds enables the determination of their elastic and plastic behavior even beyond the collapsing point. Image courtesy: ETH Zurich, Switzerland
The standardized mechanical investigation of materials in the micro- and nano-scale such as the depicted chevron notch fracture test (Prof. Mortensen, Laboratory of Mechanical Metallurgy, EPFL, Switzerland) is important for the determination of their mechanical behavior. Especially for crystalline materials, the nanoscale mechanical investigation offers new opportunities to understand the effect of stress onto the material’s structure-property relationship.

Nanomechanical testing can be combined with specialized SEM detectors such as Electron Backscatter Diffraction (EBSD) detectors. This combination enables investigators to correlate the applied mechanical stress with the material’s crystallographic properties e.g. grain structure and orientation using Transmission Kikuchi Diffraction.

For applications in environments with high radiation fluxes, such as in nuclear reactors and space, a quantitative understanding of the impact of irradiation onto the material of the load bearing structures is required. In-situ SEM nanomechanical testing enables the compression testing of micropillars and the high-resolution recording of stress versus strain curves. The depicted image sequence by Prof. Daniel S. Gianola from UC Santa Barbara shows the stress-strain curve of the micropillar and its plastic morphology with high irradiation dose.

After the linear elastic regime (a), macroscopic yielding can be observed as well as the nucleation of shear localization (b). With a further increase of the strain, the shear band propagates until it reaches the pillar surface (c), indicated by the stress drop. Subsequently, smaller stress drops correlate to intermediate slip events on parallel planes as shown in figure d.
**UNIAXIAL COMPRESSION TESTING OF CNT MICROPILLARS**

Vertically aligned carbon nanotube (CNT) pillars are a promising material for applications such as flexible batteries, compliant thermal interface materials and 3D super capacitors. The mechanical properties of CNT arrays do not only depend on the individual nanotubes, but also on their packing density and the interaction forces between the individual tubes. To experimentally study the mechanical properties of a CNT pillar, a compression test is performed. First, the CNT pillar is aligned to the microforce sensing probe of the testing system (a). A load is applied to the CNT pillar and the deformation is measured and simultaneously viewed by the scanning electron microscope (b). The plastic deformation of the pillar after the load application is shown in (c). Additionally, stress relaxation effects are shown in (d). (Image courtesy: Prof. Daniel S. Gianola from UC Santa Barbara)

**TENSILE TESTING OF NANOSTRUCTURES**

For the quantification of the mechanical properties of nanowires (left) or nanohelical spirals (right), tensile testing along the long axis is an important testing principle. The FT-NMT03 offers the capabilities to pick and place the structures and attach them between the microforce sensing tip and the sample holder. For fixation of the samples either ion beam induced deposition (FIB) or different types of SEM adhesives can be used. Image courtesy: ETH Zurich, Switzerland

**MANIPULATION AND ASSEMBLY OF NANOSTRUCTURES**

The preparation of a nanostructure for its mechanical test may require to mount it in a certain location and orientation. Due to the 5-axis microforce sensor tip to sample alignment capabilities, the FT-NMT03 offers the capability to handle and assemble nanostructures. For small structures, the natural adhesion to the microforce sensor tip and sample holder can be used for the pick-and-place. Larger structures can be manipulated with the FemtoTools FT-G Microgrippers.
ELECTRO-MECHANICAL TESTING

THERMOMECHANICAL CREEP TESTING OF INDIVIDUAL METALLIC GLASS NANOWIRES

Metallic glasses are receiving growing attention due to their unique mechanical properties such as a large elastic limit and high fracture toughness. Furthermore, the large supercooled liquid region enables superplastic forming, opening up new material processing strategies. Therefore, a quantitative understanding of its thermomechanical behavior is crucial. The depicted work from Prof. Daniel S. Gianola at UC Santa Barbara investigates the superplastic-like flow of metallic glass. For this purpose a metallic glass nanowire is attached between the FT-S Microforce Sensing Probe and a second substrate by Pt-EBID. While performing a creep test (apply a constant tensile load while measuring the deformation), the temperature is increased stepwise by passing an electric current through the nanowire. With this method, the creep behavior is analyzed at different nanowire temperatures.

PIEZORESISTIVE MICROCANTILEVER ARRAY TESTING

Piezoresistive microcantilever arrays have been developed for various applications such as gas sensors, memory storage devices and nanolithography. In-situ nanomechanical testing is used to automatically quantify the mechanical properties (stiffness, linearity, breaking force) as well as the electro-mechanical properties (resistance change vs. load) of each cantilever in the array. Image courtesy: NTB, Switzerland

IN- AND OUT-OF-PLANE MECHANICAL TESTING OF MEMS / NEMS

The continuous miniaturization trend has been driving the typical feature size of MEMS towards the nanoscale. As a result, conventional mechanical testing principles based on optical microscopy have reached their limit. Due to the higher resolution imaging capabilities of SEMs, in-situ SEM mechanical testing enables direct quantification of mechanical and electro-mechanical properties of MEMS / NEMS.
FT-S MICROFORCE SENSING PROBES

The FemtoTools FT-S Microforce Sensing Probes are microforce sensors capable of measuring forces from 200 millinewtons \((10^{-3} \text{ N})\) down to sub-nanoneutons \((10^{-10} \text{ N})\) along the sensor’s probe axis. Both compression and tension forces can be measured. The FT-S Microforce Sensing Probes are available with five different tip options:

- Flat silicon tip with a tip size of 50 µm by 50 µm
- Sharp tungsten probe tip with a tip radius smaller than 2 µm or 100 nm
- Spherical tips with either 125 µm or 25 µm radius

The individual calibration in combination with an outstanding long-term stability guarantees significantly higher measurement accuracy than any other force sensing system in this force range. A calibration data sheet is delivered for each individual sensor.

<table>
<thead>
<tr>
<th>Model</th>
<th>Range</th>
<th>Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>FT-S200</td>
<td>+/- 200 µN</td>
<td>0.5 nN</td>
</tr>
<tr>
<td>FT-S2'000</td>
<td>+/- 2'000 µN</td>
<td>5 nN</td>
</tr>
<tr>
<td>FT-S20'000</td>
<td>+/- 20'000 µN</td>
<td>50 nN</td>
</tr>
<tr>
<td>FT-S200'000</td>
<td>+/- 200'000 µN</td>
<td>500 nN</td>
</tr>
</tbody>
</table>

FT-G FORCE SENSING MICROGRIPPER

The FT-G Microgripper series is designed to handle micro- to nanometer-sized objects. The initial openings of the gripper arms are 30 µm for the FT-G33 and 100 µm for the FT-G 103. The opening can be controlled with nanometer precision, such that the gripper arms are fully closed upon applying the maximum actuation voltage.

Both the FT-G33 and the FT-G103 Force Sensing Microgripper feature an integrated force sensor to measure the gripping force. The force feedback greatly enhances the efficiency and reliability of automated microhandling systems and assembly processes.

<table>
<thead>
<tr>
<th>Model</th>
<th>Opening</th>
<th>Force Sensor</th>
</tr>
</thead>
<tbody>
<tr>
<td>FT-G33</td>
<td>0 - 30 µm</td>
<td>yes</td>
</tr>
<tr>
<td>FT-G103</td>
<td>0 - 100 µm</td>
<td>yes</td>
</tr>
</tbody>
</table>
# Technical Specifications

## FT-NMT03 Nanomechanical Testing System

### FT-NTP Nanomechanical Testing Platform

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of axes (coarse)</td>
<td>5</td>
</tr>
<tr>
<td>Actuation principle (coarse)</td>
<td>Piezoelectric stick slip</td>
</tr>
<tr>
<td>XYZ actuation range (coarse)</td>
<td>12 mm x 21 mm x 12 mm</td>
</tr>
<tr>
<td>Min. motion increment (coarse)</td>
<td>1 nm</td>
</tr>
<tr>
<td>Actuation principle (fine)</td>
<td>Piezoelectric scanning</td>
</tr>
<tr>
<td>Actuation range (fine)</td>
<td>25 µm</td>
</tr>
<tr>
<td>Min. motion increment (fine)</td>
<td>0.05 nm</td>
</tr>
<tr>
<td>Encoder resolution</td>
<td>0.05 nm</td>
</tr>
<tr>
<td>Position measurement range</td>
<td>0.05 nm - 21 mm</td>
</tr>
<tr>
<td>Sample tilt range</td>
<td>90°</td>
</tr>
<tr>
<td>Sample tilt resolution</td>
<td>35 µ°</td>
</tr>
<tr>
<td>Sample rotation range</td>
<td>360°</td>
</tr>
<tr>
<td>Sample rotation resolution</td>
<td>35 µ°</td>
</tr>
<tr>
<td>Maximum force range*1)</td>
<td>± 200 mN</td>
</tr>
<tr>
<td>Smallest force resolution*2)</td>
<td>0.5 nN</td>
</tr>
</tbody>
</table>

*1) Using a FT-S200’000 Microforce Sensing Probe  
*2) Using a FT-S200 Microforce Sensing Probe

### FT-nMSC Modular System Controller

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
</table>
| Key functionality | Force acquisition  
Control of the manipulation unit  
Position readout  
Control of the microgripper  
Data synchronization |
| Sensor protection mode | Axial sensor protection  
Automated sample approach |
| Electrical sample testing | Sample holder with 6 electrical contacts |
| Joystick control | Hardware joysticks  
Software joysticks  
Computer keyboard |
| Interface | USB |
| Power supply | 12 V power supply with Europlug (110 V/230 V) |