

# IN-SITU SEM/FIB

## NANOINDENTER

FT-NMT04 NANOMECHANICAL TESTING SYSTEM



Product Brochure

## **FT-NMT04 NANOMECHANICAL**

### **TESTING SYSTEM**

The FT-NMT04 Nanomechanical Testing System is a versatile in-situ SEM/FIB nanoindenter capable of accurately quantifying the mechanical behavior of materials at the micro- and nanoscale.

As the world's first MEMS-based in-situ SEM nanoindenter, the FT-NMTO4 is based on the patented FemtoTools Micro-Electro-Mechanical Systems (MEMS) technology. Leveraging over two decades of technological innovations, this in-situ nanoindenter features unmatched resolution, repeatability and dynamic stability.

The FT-NMT04 in-situ SEM nanoindenter is optimized for the mechanical testing of metals, ceramics, and thin films, as well as micro-scale structures such as metamaterials and MEMS. Furthermore, the FT-NMT04 is modular with extendable capabilities to accommodate therequirements of various research fields.

Typical applications include the quantification of plastic deformation mechanisms by compression testing of micro-pillars or tension testing of dog-bone shaped specimens, thin films, or nanowires. Furthermore, continuous stiffness measurement (CSM) during bending enables the J-integral quantification of the fracture toughness and crack growth events during fracture testing of microcantilevers.

With an unmatched noise floor of only 500 pN in force (guaranteed real world values) and 50 pm in displacement (guaranteed real world values) and comparatively large ranges of 200 mN and 25  $\mu$ m, the FT-NMT04 enables the comprehensive study of mechanical behavior of materials with an unprecedented accuracy and repeatability.

CITATION CONTRACTOR

Nanoindentation, compression testing, tension testing, fracture testing, fatigue testing

Quantitative mechanical testing with simultaneous imaging via SEM, EBSD or STEM

EATURES

Patented MEMS-based sensing technology enables the highest resolution and repeatability in both force from 0.5 nN to 200 mN and displacement from 0.05 nm to 21 mm

Continuous stiffness measurement (CSM) or fatigue measurements up to 500 Hz without complex, dynamic calibrations

True displacement-controlled testing, enabling the quantification of fast stress drops (optional force-controlled measurements are possible as well)

3-Axis closed-loop sensor for sample alignment using positioning encoders on all axes

High temperature isothermal testing up to 800°C

Simple determination of the indenter area function and frame compliance

Powerful data analysis tools for evaluating measurement results and applying fits and functions to determine material properties

SEM synchronization enabling simultaneous acquisition of SEM images and video with nanomechanical data

Compact, modular design enables integration into SEMs even with small chambers

Customizable measurement procedures and principles

## APPLICATION OVERVIEW



#### MICRO-PILLAR COMPRESSION

- Determination of critical-resolved shear stresses (CRSS) of slip systems
- Characterization of deformation mechanisms under uniaxial stress
- Quantification of plastic damage and strain localization



### MICRO-CANTILEVER FRACTURE TESTING

- Determination of sub-micron fracture toughness with continuous J-integral method
- Characterization of monotonic and cyclic fracture behavior
- Quantification of individual crack initiation and propagation events



#### NANOINDENTATION

- Determination of hardness and Young's modulus in small volumes
- Quantification of contact mechanics and dynamic response
- Characterization of deformation mechanisms under multiaxial stresses



#### MICRO-TENSILE TESTING

- Determination of yield stress, ultimate tensile stress and elongation to failure of specific phases and structures
- Characterization of fracture modes under monotonic and cyclic loading
- Quantification of strain localization effects and crack initiation and propagation events



#### **CORRELATE MECHANICAL TESTING WITH STEM/EBSD**

- Quantitative study of strain localization
- Quantitative study of phase transformations
- Quantitative study of texture evolution
- Quantitative study of dislocation dynamics
- Quantitative study of grain boundary migration

## FT-NMT04

## SYSTEM BUILDUP



#### SYSTEM COMPONENTS

- 1 One-axis nanopositioning stage with high-resolution positioning encoders, enabling movements over a range of 21 mm with 1 nm noise floor
- 2 Two-axis nanopositioning platform with high-resolution positioning encoders, enabling movements over a range of 12 x 12 mm with 1 nm noise floor
- 3 Flexure-based, linear piezo scanner with capacitive positioning encoders for continuous and fast movement over a range of 25 μm with 0.05 nm noise floor
- 4 FT-S Microforce Sensing Probe with a force sensing range from 0.5 nN to 200 mN

## **MEMS-BASED NANOINDENTATION**

1-Axis Microforce Sensing Probe



2-Axis Microforce Sensing Probe



1-Axis Microforce Sensing Probe with integrated tip heater



The T-NMT04 Nanomechanical Testing System is a high-resolution nanoindenter based on Micro-Electro- Mechanical Systems (MEMS) technology.

While typical nanoindentation systems feature force-sensing technologies based on precisionmachined and assembled components, Femto-Tools is using semiconductor fabrication technology to machine the entire force sensor out of single crystal silicon wafers. This approach enables the fabrication of much smaller structures, making load cells with high sensitivity, resolution, and repeatability therefore overcome the limitations oftraditional technologies. Furthermore, the small size of the MEMS sensing element results in a mass orders of magnitude lower than conventional load cells. In combination with the high stiffness of silicon flexures, the FemtoTools FT-S Microforce Sensing Probes provide a high natural frequency (up to 50 kHz) and the related ability to measure fast events or to conduct fatigue or cyclic tests at high frequencies.

One additional benefit of the compact MEMSbased force sensing technology is the small form factor of the FT-NMT04 (120 mm x 72 mm x 44 mm). This enables the integration of the FT-NMT04 in most SEM / FIB systems.

#### MEMS-BASED MICROFORCE-SENSING PROBE



### FT-NMT04

## ACCESSORIES

#### **FT-S MICROFORCE SENSING PROBES**

The FemtoTools FT-S Microforce Sensing Probes are sensors capable of measuring forces from sub-nanoNewtons to 200 milliNewtons along the sensor's probe axis. Both compression and tension forces can be measured. SI-traceable pre-calibrations for each probe in combination with outstanding long-term stability guarantees significantly higher measurement accuracy than other force-sensing systems in this force range. Specialized versions are also available, including 2-Axis Microforce Sensing Probes or High Temperature Probes (HT) up to 800°C.

The FT-S Microforce Sensing Probes are available with a wide variety of tip materials and geometries including diamond Berkovich, cube corner, flat punch, wedge, conical and more.



#### HIGH TEMPERATURE TESTING MODULE

The FT-NMT04 can be upgraded with the FT-SEM-HT04 In-situ SEM High Temperature Module. This module enables heating specimens up to 800°C. It is used in combination with the Microforce Sensing Probe with an integrated tip heater, which provides localized heating of the tip to match the temperature of specimens. This module allows for all of the FT-NMTO4 capabilities at temperatures up to 800°C, including: high-resolution and high-repeatability nanoindentation, micro-pillar compression, micro-tensile testing and micro-cantilever fracture testing. The In-situ SEM High Temperature Mod-

ule enables the quantitative study not only of changes in local materials properties, but also of plastic deformation, softening and fracture mechanisms at high temperature. Typical studies include the evolution of hardness, elastic modulus or fracture toughness with temperature.





#### SCRATCH TESTING MODULE

The FT-NMT04 can be upgraded with the FT-SEM-ST04 In-situ SEM Scratch Testing Module to allow for nanoscratch, nano-wear and nano-friction testing, as well as scanning probe microscopy (SPM). The diamond tip of a 2-Axis Microforce Sensing Probe is moved across the sample surface while applying a ramped or constant normal load at a given speed.

Scratch testing yields quantitative insights into various properties such as failure mechanisms at the nanoscale, thin film adhesion, friction coefficients, and abrasive or wear resistance of materials. Furthermore, highresolution SPM imaging can be used for topographic imaging before and after scratch, wear, or nanoindentation testing. It provides direct visualization of pre-test surface roughness and post-test surface deformation or damage.

Combining a 20 x 20 x 25 µm imaging range with a 50 pm scanning noise floor, this module is coupled to the 2-Axis Microforce Sensing Probes featuring a 100 nN noise floor for a 20 mN force range. In addition, the module benefits from a long-range probe positioning noise floor of 1 nm to target specific testing or scanning locations. Adding to the technical superiority of the FT-NMT04 for high-resolution, high-repeatability nanoindentation, the Scratch Testing Module enables the comprehensive study of materials properties at surfaces and interfaces, as well as the quantitative characterization of elasto-plastic deformation and fracture mechanisms at the nanoscale. Typical examples include the characterization of critical loads for cohesive and adhesive failures, the effect of surface roughness on shallow indents or the impact of near-surface plasticity on friction.

### **MICRO-PILLAR COMPRESSION**

In-situ SEM micro-pillar compression tests provide a way to measure the uniaxial mechanical response of low volumes of materials and to directly correlate the stress-strain data to individual deformation events. It enables to quantify specific phases and particles or to study size effects, in terms of deformation behavior and strengthening mechanisms. In order to resolve individual deformation events, key requirements for the measurement system are high load and displacement resolution, as well as fast data acquisition rate.

After identification of a suitable location in terms of microstructure or crystal orientation using SEM and EBSD, micro-pillars are prepared by top-down milling with focused-ion beam (FIB). Decreasing ion currents are used from the machining of the structure to the final surface polishing in order to reduce FIB damage.

During compression, linear elasticity is observed in the initial loading stage, before yielding and plasticity. In the plastic regime, a serrated plastic flow behavior with sudden stress drops followed by reloading periods is often characteristic of dislocation slip events. For example, a correlation is shown between stress drops in the stress-strain data and shear-band formation seen in SEM with a) elastic loading, b) nucleation of the first slip event, c) intersection with top surface and d) multiplication of slip events.

It is worth noting that load-controlled testing systems show strain bursts (not stress drops) in the stress-strain curve, preventing the quantitative study of these mechanisms. A key requirement of the system is therefore true displacement control. In combination with an ultra-low load noise floor, the statistical analysis of even smaller magnitude stress drops is possible. This enables to gain new insight into the nature of the interactions between dislocations and various lattice defects. Work conducted by: D, Gianola et al., Gianola Lab, Materials Department, UC Santa Barbara, USA.



X. Zhao, D.J. Strickland, P.M. Derlet, M.R. He, Y.J. Cheng, J. Pu, K. Hattar, and D.S. Gianola. "In situ measurements of a homogeneous to heterogeneous transition in the plastic response of ion-irradiated <111> Ni microspecimens." Acta Materialia, 2015

### HIGH TEMPERATURE TESTING



High temperature microcompression testing is a great tool to study plastic deformation in materials, even semiconductors like Silicon that are normally brittle. The combination of small length scales and high temperatures suppresses crack formation in many materials, especially in single-slip orientations, and allows plastic deformation mechanisms to be investigated in great detail

In the still images on the left, taken from in-situ videos, the brittle to ductile transition from fracture at room temperature to plasticity at high temperatures can be seen. The room temperature image shows the elastically deformed pillar just prior to fracture, while the higher temperatures display clear evidence of plastic slip and deformation. This is clearly visualized insitu in a Zeiss EVO scanning electron microscope (SEM).

After contact detection in the rapid approach, all pillars were deformed at a constant strain rate of 0.004/s. Above the transition temperature of 200 °C, dislocation motion is initially localized on a few slip planes as seen at 400 °C, but at high temperatures the dislocation motion becomes delocalized and deformation becomes more homogeneous. This is also observed in the stress-strain behavior, where considerable plasticity is observed at temperatures above 200 °C.

A strong indicator of quality in this data is the initial elastic loading region of the stress-strain curves. This closely matches the expected elastic modulus for [100]-oriented Silicon, as shown. This is due to the lithographic fabrication of these pillars, which allows high geometric accuracy with minimal taper or rounding of the pillar tops. The modulus appears to be approximately constant with temperature, showing that the measurements are independent of thermal drift. In reality, the elastic modulus decreases slightly with temperature to 125 GPa by 500 °C, but this requires higher precision measurements, such as continuous stiffness measurement (CSM), to be resolved.

### **FRACTURE TESTING**

Fracture toughness is a key property in most engineering applications. Fracture experiments at small scale using micro-cantilever bending tests are crucial to determine fracture toughness in low volumes of materials. Furthermore, these tests provide crucial information to quantify the contribution of specific microstructural features to the overall crack resistance of a material. Moreover, in-situ SEM microcantilever bending tests provide new insights into the micromechanisms of fracture by combining the force-displacement data with direct crack path observations.

The typical micro-cantilever bending test uses compression loading of a free-standing notched cantilever beams, prepared with lithography or Focused Ion Beam (FIB). For brittle fracture, the fracture toughness  $K_{\mbox{\tiny III}}$  is determined from the stress intensity factor K, at the maximal load. A key reguirement of the measurement system is true displacement control to avoid catastrophic failure of specimens once the crack becomes unstable, which is typical with force-controlled systems. For elastic-plastic fracture, the J-Integral method is typically used to analyze the crack growth resistance curve (J-R curve) and elastic-plastic fracture toughness  $(J_{\mu})$ . Generally, higher  $K_{\mu}$ ,  $J_{\mu}$  or steeper J-R curve indicate that a material is more resistant to fracture. Micro-cantilever bending testing using continuous stiffness measurement (CSM) enables to monitor the evolution of crack length and compute continuous J-integral from periodic unloading segments, and therefore to build continuous J-R curves.

By combining true displacement control, high load and displacement resolutions, a wide harmonic frequency range and fast data acquisition rate, FT-NMT04 enables the precise control of the fracture process and the quantification of the effect of individual microstructural features on the fracture toughness of materials. Work conducted by: S. Gabel, B. Merle, M. Göken, Department of Materials Science and Engineering, Institute I, University of Erlangen-Nuremberg, Germany



Technique based on: Ast J., Merle B., Durst K., Göken M. "Fracture toughness evaluation of NiAl single crystals by microcantilevers - A new continuous J-integral method" (2016) Journal of Materials Research, 31 (23), pp. 3786-3794.

## MICRO-TENSILE TESTING

Work conducted by: J. L. Wardini, T. Rupert, Rupert Lab, University of California Irvine, USA



Z. Fu, L. Jiang, J. L. Wardini, B. E. MacDonald, H. Wen, W. Xiong, D. Zhang, Y. Zhou, T. J. Rupert, W. Chen, E. J. Lavernia, "A high-entropy alloy with hierarchical nanoprecipitates and ultrahigh strength." Science Advances, 2018

Large scale tensile testing is a commonly used test to quantify the elastic modulus, yield-, ultimate-, and fracture-strength of materials. However, while these tests provide valuable insights into the overall material properties, they average out the effects of constituents such as individual phases and interfaces.

To quantify the properties of a single phase or interface, micro-tensile testing is required, as shown in the image sequence on the left. Furthermore, by scaling down these experiments even more, single plastic deformation mechanisms can be detected and investigated.

For sample preparation, focused-ion beam (FIB) can be used to create dogbone shaped samples with a uniform cross-section that remain attached to the original substrate. FIB can also be used to machine a gripper shape into the tip of the force-sensing probe. This gripper shape enables the interlocking with the dog bone sample in order to conduct micro-tensile tests.

During tensile loading, linear elasticity is observed in the initial stage, before yielding, plasticity and, eventually, fracture.

A critical testing requirement to measure the full stress-strain curve is true displacement-controlled testing, which prevents catastrophic failure of the specimen during unstable crack propagation. As a result, the material's behavior after the ultimate strength (with decreasing slope of the stressstrain curve) can be characterized.

By combining this micro-tensile testing method with the deposition of speckles or line markers (shown on the left) along the sample, digital image correlation (DIC) can also be used to obtain the local strain along the loading axis.

### NANOINDENTATION

Nanoindentation is a standard and efficient method to study the mechanical properties of materials at small scale with minimal sample preparation. The technique is ideal for the study of thin films or low material volume. In addition to hardness and elastic modulus, nanoindentation gives useful insight into the creep, fracture and fatigue properties of materials. The multiaxial stress-field underneath the indenter enables the activation of slip systems in different planes and therefore comprehensive investigation of complex plasticity mechanisms. Making use of size effect in miniaturized samples also enables to study the plastic behavior of quasi- brittle materials.

By combining ultra-high load and displacement resolutions with in-situ SEM observation, FT-NMT04 enables to measure the mechanical properties of specific sub-micron-scale microstructural features and to directly visualize the formation of pile-up, slip bands and cracks.

While standard nanoindentation provides measurement data at the onset of unloading, using Continous Stiffness Measurement (CSM) enables to record both hardness and elastic modulus as a function of the indenter penetration depth. With high load and displacement resolutions, CSM nanoindentation with FT-NMT04 enables to quantify the evolution of the mechanical response from shallow penetration depths and the onset of plasticity, to the bulk material. Furthermore, the extended harmonic frequency range of the FT-NMTO4 system (up to 500 Hz with very little contribution from the measurement system), combined with a fast data acquisition rate, enables the unprecedented quantitative dynamic mechanical analysis of the viscoelastic and viscoplastic behavior of materials.



### MECHANICAL MICROSCOPY



Hardness map (CSM nanoindentation, 1 indent/s, 60000 indents).



EBSD mapping of crystal orientation (same specimen, different location).



1200 µm

To demonstrate the capability of the FT-NMT04, results from CSM nanoindentation measurements on a Ni-Ti alloy are presented here. Ni-Ti alloys are designed with near-equal atomic percentages of nickel and titanium that display unique temperature-dependent properties: the shape memory effect and super-elasticity. Shape memory relates to their capacity to undergo plastic deformation and retain the deformation upon unloading until heating up to a critical temperature. Upon reaching this critical temperature, the alloy undergoes phase transformation and recovers from plastic deformation back to its original state. At temperatures higher than this critical temperature, Ni-Ti alloys exhibit superelasticity where they undergo large deformations via reversible phase transformation and immediately recover on unloading. Since they combine high biocompatibility, corrosion resistance, and wear resistance, Ni-Ti alloys are amongst the most well-known shape memory alloys. They are used in a wide range of commercial applications from medical implants to intelligent reinforced concrete and damping components. Many research efforts are devoted to developing a full mechanistic understanding of the shape memory effect and its limitations, particularly, the relationship between crystal orientation and elastic modulus appears to be critical in explaining local strain accommodation and localization which leads to irreversible deformation and the progressive loss of the shape memory effect. Here, the FT-NMT04 enables the quantification of hardness and modulus variations with crystal orientation. Remarkably, it also enables quantification of the narrow and soft gradients of properties (<3%) near grain boundaries. Featuring a high spatial resolution over large areas and maintaining high precision even at high mapping speeds, FT-NMT04 results can be directly compared to EBSD and EDX maps for a full understanding of the link between microstructures and materials properties.

### **FATIGUE TESTING**

Work conducted by: S. Singh, A.S. Singaravelu, and N. Chawla, Center for 4D Materials Science, Arizona State University, USA

In-situ micro-fatigue testing enables the quantitative study of fatigue crack initiation and propagation events, with direct crack path observations and continuous monitoring of the dynamic mechanical response. It provides unprecedented insights into fatigue micro-mechanisms and their influence on the fatigue life of materials and complex structures, such as nano-composites and laminates.

With its wide dynamic range (without system resonance), fast data acquisition rate and true displacement control, the FT-NMT04 enables low- and high-cycle in-situ fatigue testing up to 500 Hz and more than 5 million load cycles in less than 3 hours.

A typical experimental set-up is the bending of a microcantilever. Other possibilities include cyclic compression of micropillars or cyclic tensile and compression testing of tensile sample such as e.g. thin lamellae.

Cyclic loading is then applied on the specimen. The test is interrupted at regular intervals for high-resolution SEM imaging, in order to observe indications of crack initiation, propagation, deflection at interfaces or surface damage.

The dynamic stiffness response of the specimen is monitored to provide crucial insights, into e.g. short or long crack initiation and growth, or cyclic hardening and softening mechanisms. In addition, correlative EBSD mapping enables to follow strain accumulation ahead of the crack tip and its link with subsequent crack propagation along critical planes.

The fatigue life can be evaluated with changes in the specimen's stiffness. The data can then be compared to ob-

servations of fracture surfaces after final failure.



S. Singh, A.S. Singaravelu, and N. Chawla, "Deformation Behavior of Co-Sputtered and Nanolaminated Metal/Ceramic Composites" Dissertation for the Doctor of Philosophy Degree, Center for 4D Materials Science, Arizona State University, 2018

## **CORRELATIVE STEM / EBSD IN SEM**

Work conducted by: D, Gianola et al., Gianola Lab, Materials Department, UC Santa Barbara, USA.





The FT-NMT04 is specially-designed to combine the study of the stress-strain response of materials not only with the observation of surface events, but also with EBSD, TKD and STEM characterization to gain unprecedented quantitative insight into phase transformation and dislocation dynamics.

Micro-tensile testing, pillar compression and cantilever bending in correlation with EBSD enable to monitor and quantify dynamic phase transformations and strain localization. To further explore plasticity at the dislocation scale, micro-tensile testing of electron-transparent specimens and thin films can be performed in correlation with TKD and STEM detectors.

After selecting a proper location in terms of microstructure and crystal orientation using SEM and EBSD, a specimen is extracted by focused-ion beam (FIB) machining. It is then placed on a testing sample support, such as the FemtoTools Nano-Tensile Testing Chip, and secured using ion (or electron) beam induced deposition. FIB carving and thinning of the micro-tensile specimen down to electron transparency is then performed with low voltage to minimize FIB damage and enable for STEM imaging.

During loading, linear elasticity is observed first, before a plastic regime characterized by multiple load drop events and the final fracture of the specimen.

With true displacement control, fast data acquisition rate and ultra-high load and displacement resolution, the analysis of the amplitude and time of individual load drops is possible. In correlation with STEM images, it provides the characteristic load (down to less than 0.5 µN) attributed to specific plastic events. It gives the unique opportunity to study distinct dislocation interactions with lattice defects, such as Orowan bowing or shearing of precipitates by partial or full dislocation pairs. It enables to build a statistical understanding of plastic localization mechanisms down to the dislocation scale.

## **MICROSTRUCTURE 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 their 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 (applying 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.



#### STABLE COMPRESSION TESTING OF MICROSCAFFOLDS

Microscaffolds are used in various areas such as in materials 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



#### 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.





# TECHNICAL

# SPECIFICATIONS

### FT-NMT04 NANOMECHANICAL TESTING SYSTEM

Number of axes (coarse)	3
Actuation principle (coarse)	Piezoelectric stick slip
XYZ actuation range (coarse)	21 mm x 12 mm x 12 mm
Min. motion increm. (coarse)	l nm
Encoder noise floor (10 Hz)	l nm
Actuation principle (fine)	Piezoelectric scanning
Actuation range (fine)	25 µm
Min. motion increm. (fine)	0.05 nm
Encoder noise floor (10 Hz)	0.05 nm
Digital resolution	0.05 pm
Position measurement range	0.05 nm - 21 mm
Maximum force range*1)	± 200 mN
Force noise floor (10 Hz)*2)	0.5 nN
Digital resolution*2)	0.5 pN



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







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