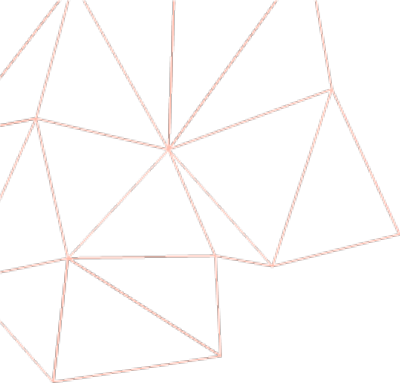




Whitepaper

Nanomechanical Testing: Techniques, Applications, Advantages and Limitations

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“Nanomechanical testing” is an umbrella for a family of cutting-edge techniques used in materials science for resolving the mechanical properties of materials at the nanoscale. This whitepaper provides an overview of the main techniques, their working principles, typical applications, as well as their advantages and limitations. It aims to guide researchers in selecting the most appropriate technique for a given material and research question.

This whitepaper is a token to the activities of COST CA21121 MecaNano (“European network for the mechanics of matter at the nanoscale”) and is freely available at www.mecanano.com.

1. Introduction

As modern materials are increasingly engineered bottom-up from the nanoscale, understanding their mechanical properties is paramount. Nanomechanical testing provides the means to probe these properties with high spatial resolution, enabling insights into nanoscale phenomena such as plasticity, fracture, and deformation mechanisms that ultimately control the failure and lifetime of bulk materials. This review describes various nanomechanical testing methods, their typical applications, advantages and limitations. The methods discussed include nanoindentation, micropillar compression, micro-beam bending, in situ testing techniques and methods specific to thin films. Focused ion beam (FIB) milling is discussed as a specimen-preparation method supporting several of these techniques. Nanomechanical measurements must be interpreted in the context of specimen dimensions, microstructure and loading conditions [1].

2. Nanoindentation testing

Nanoindentation is the most widely used technique for measuring the hardness and Young's modulus of materials at the nanoscale. Its robustness has been established by applications to a wide range of materials, which has been documented by >20 000 publications over the past 30 years [2,3]. It is extensively used for characterizing thin films, coatings and small volumes of materials. Nanoindentation is also extremely valuable in research fields like biomaterials and polymers science.

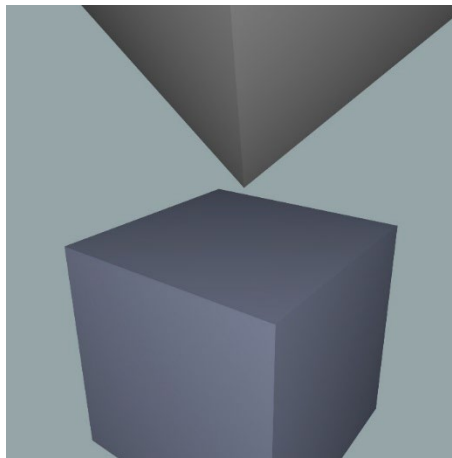


Figure 1: Nanoindentation testing with a Berkovich indenter tip

The nanoindentation technique involves pressing a hard tip into the surface of a material while continuously recording the load and displacement. Berkovich diamond tips are most commonly used. The resulting data allow for the calculation of mechanical properties such as hardness and Young's modulus [4]. The method has also been extended to investigate the fracture toughness, the strain rate sensitivity and creep properties, while spherical nanoindentation is increasingly used to access the stress-strain yield behavior of materials at the nanoscale [5–8]. In recent years, nanoindenters have been designed to operate under non-ambient conditions, allowing investigations at high, low temperatures, under vacuum or at high strain rates [9–12]. Under such extreme conditions, measurements require careful correction for instrumental and environmental effects.

Nanoindentation usually provides very high lateral ($\sim 1\ \mu\text{m}$) and depth ($\sim 1\ \text{nm}$) resolutions, allowing the investigation of very small volumes of material, on the order of its plastic zone. Sample preparation is comparatively straightforward for many bulk materials, and mostly

requires a flat, smooth surface. Measurements and data evaluation with the Oliver-Pharr method are usually automated, which allows for high-throughput testing and mapping of mechanical properties [13]. Calibration of the tip area function and instrument compliance is essential, with the ISO 14577-1:2026 norm providing a standardized framework for these.

On the downside, the accuracy of nanoindentation can be affected by poor sample preparation, e.g. excessive surface roughness, preparation-induced damage, surface tilt and compliant fastening of the sample. In addition, in soft and strain hardened materials, pileups can form around the indents, leading to an overestimation of the measured hardness and modulus evaluated by the Oliver-Pharr method [14].

3. Focused Ion Beam (FIB) sample structuring

Building upon the original nanoindentation hardware, several advanced nanomechanical testing techniques have been developed that rely on pre-structuring a sample by focused ion beam (FIB) milling prior to testing. SEM-FIB dual-beam systems are advanced scanning electron microscopes (SEM) that combine the usual electron column with a focused ion gun. These microscopes allow for the targeted removal of material from the sample surface, thereby allowing for the fabrication of microscopic structures, such as micropillars. Mechanically loading precisely shaped nano-samples, instead of indenting a plain surface, allows to substitute the complex stress field found below a nanoindenter tip with a simpler and well-defined deformation field in these objects. Typical structures are micropillars and micro-beams, but a variety of other geometries have also been proposed in the literature, which are specifically tailored to access further mechanical properties [15].

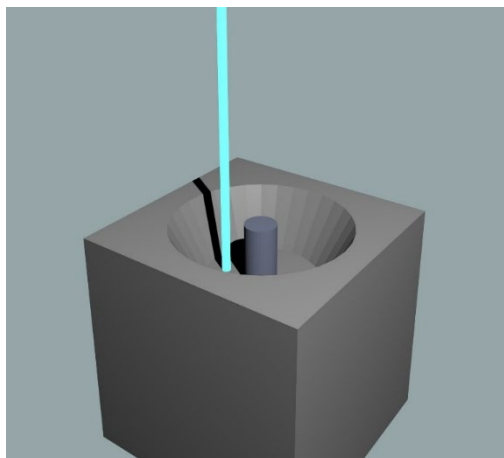


Figure 2: Focused Ion Beam (FIB) milling of a micropillar from a bulk sample

A downside of FIB-assisted techniques is the damage inflicted to the specimen by the ion beam. Its affected range and severity strongly depend on the material, ion species, beam energy and incidence angle. The damaged zone typically extends over a few tens to hundreds of nanometers, which is small compared to the micrometer size of the fabricated micro-specimen [16]. Under specific circumstances, e.g. when using a gallium-based FIB on aluminum samples, new phases might be formed which lead to an embrittlement of the specimen. The latter can be mitigated by using a plasma FIB, e.g. based on xenon or argon [17]. Generally, the mechanical implications of the FIB preparation method should be assessed for each material and testing technique.

4. Micropillar compression

Micropillar compression testing involves fabricating small, often cylindrically shaped specimens using focused ion beam (FIB) milling or photolithographic methods. These pillars are then subjected to uniaxial compression in the nanoindenter, using a flat punch, to measure their stress-strain behavior. This method is particularly useful for studying incipient plasticity, as well as work hardening, which are not captured by pyramidal nanoindentation. Micropillar compression testing has given rise to an abundant literature on mechanical size effects in all classes of materials [18,19]. It is also routinely used to single out microstructural features and measure their contribution to strength and plasticity. This makes it possible to study the effects of grain boundaries, phase interfaces, and other crystallographic defects [20,21]. By applying a small deformation and imaging the resulting slip traces by SEM, it is possible to identify the activated glide system and measure parameters as fundamental as their criticality resolved shear stress [20].

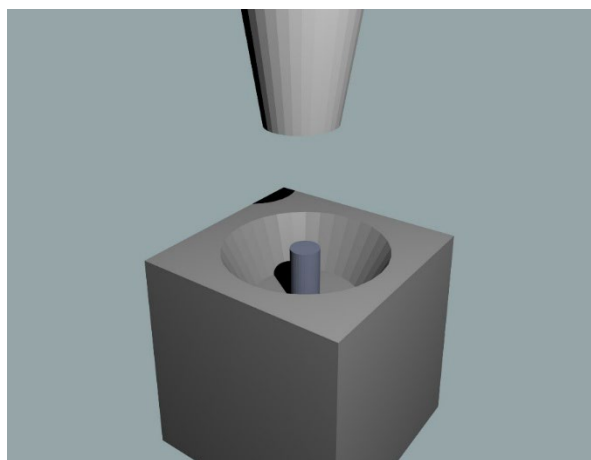


Figure 3: Micropillar compression with a flat punch

FIB milling of micropillar is straightforward for conductive materials, such as metals. It can also be performed with insulating material, provided an electron flood gun is available, or the sample is coated with a thin conductive layer prior to processing. The most common milling pattern is annular milling, where the ion beam is moved along increasingly small concentric circles. It is more time efficient than the lathe milling alternative [22]. However, it results in a slightly conical shape of the pillars, which can potentially distort the uniform straining and lead to strain localization in the top section of the specimen. Similar strain localization effects can result from poor alignment between the flat punch and the top surface of the pillar. Excessively long micropillars may experience buckling, while excessively short ones are prone to barreling. An aspect ratio of 3:2 (length-diameter) is usually considered a good trade-off [1,22].

5. Microbeam bending

Microbeam bending is a technique widely used to measure the fracture toughness of materials at the micrometer scale. It is particularly useful for characterizing individual layers or interfaces within a complex stack of coatings. A common finding is that their microscopic behavior does not necessarily mirror the bulk fracture toughness. Fracture testing requires the fabrication of notched microcantilevers by FIB milling. The beams are subsequently loaded with a pointed or wedge indenter, with both load and displacement being continuously recorded. The technique has most often been applied to brittle materials, such as ceramics and hard metals. Evaluation of the data using linear elastic fracture mechanics (LEFM) requires a small yielding condition, i.e. the crack must be sharp and the fracture process zone small compared to the

beam dimensions [23]. An extension to the elastic plastic fracture mechanical (EPFM) framework has recently been enabled by the continuous measurement of the cantilever stiffness (CSM). This allows monitoring the crack extension and measuring crack-resistance curves with moderately ductile materials [24,25]. Highly ductile materials remain a challenge because their plastic zone typically extends throughout the whole beam width.

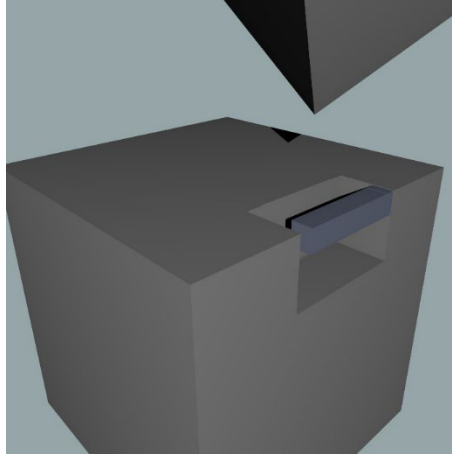


Figure 4: Microcantilever bending with a cube-corner indenter

There are two main limitations of the notched microcantilever technique, whose severity depends on the level of ductility of the samples. First, the finite radius of FIB-milled notches can lead to an overestimation of the fracture toughness of brittle samples [26]. Second, the proximity to the free surface might limit the extension of the process zone in ductile samples, thereby reducing the energy dissipation and the measured fracture toughness [27]. In addition, conventional FIB milling of rectangular cantilevers is restricted to the vicinity of a sample edge. Advanced geometries, such as triangular and chevron cantilevers, broaden the scope of accessible locations, but require a more careful evaluation [28].

Besides fracture, microbeam bending is also used for fatigue investigations. In this case, notched or unnotched micro cantilevers are cyclically loaded in the nanoindenter, allowing the investigation of both fatigue crack initiation and cyclic lifetime [29].

6. In situ testing in electron microscopes

Performing nanomechanical testing in situ in a scanning electron microscope (SEM) or a transmission electron microscope (TEM) yields crucial information about the evolution of the microstructure during testing. High-resolution imaging makes it possible to correlate features in the mechanical data with specific deformation events and to identify the fundamental mechanisms of deformation and failure at the nanoscale. In situ SEM testing is widely used to observe the deformation of micro and nano-scale structures under compression, tension, and bending. In situ TEM testing allows for direct observation of dislocations interactions, phase transformations, and crack propagation [30]. These techniques are particularly valuable for investigating advanced materials such as nanowires, thin films, and composites.

The main limitations of in situ testing are the need for specialized equipment, and the complexity and time duration of its operation. The mechanical setup must be compact enough to fit into the microscope chamber or specimen gap. The vacuum environment of the microscope and the exposure of the sample to high energy electron irradiation can introduce artefacts, including charging, beam-induced damage or heating [31]. Faulty interpretations can be avoided by confirming the measurements with beam-off repetitions.

7. Thin films testing

While nanoindentation and micropillar compression are well suited for coatings thicker than a few micrometers, submicron films require dedicated testing techniques. Some of them are based on Micro-Electromechanical Systems (MEMS), which are compact platforms combining actuators, sensors and thin film specimen within a few square millimeters. Depending on their design, they can either support parallel high-throughput testing or be integrated into scanning electron microscopes (SEM) or transmission electron microscopes (TEM) for in situ investigations [32,33]. MEMS platforms have been mostly used to investigate the plasticity behavior and fatigue behavior of thin films. More recently, they have been applied to the fracture and creep characterization [34]. On the downside, the fabrication of MEMS devices is complex, and requires special facilities and expertise. There is limited flexibility for optimizing the geometry of the samples during a fabrication cycle.

As an alternative to MEMS tensile testing, bulge testing is widely used for measuring the mechanical properties of thin films. In this method, a freestanding film is clamped over an aperture, and a gas pressure is applied to deform the membrane. Using an appropriate model, the pressure and deflection data subsequently yield the stress-strain diagram of the films and properties such as elastic modulus, residual stress, and fracture strength [35]. Bulge testing is particularly useful for studying the mechanical behavior of ultra-thin films (ca. 100 nm thickness), which are challenging for any other method. In recent years, the bulge method was expanded to the characterization of the creep, fracture and fatigue behavior of such films [27,36]. The accuracy of bulge testing depends largely on pressure and deflection calibration, membrane dimensions, film thickness and uniformity, initial curvature and alignment of the laser sensor.

Both MEMS and bulge testing techniques are well suited for the characterization of thin films that are essentially stress-free or exhibit a tensile residual stress. Highly compressive residual stresses can cause out-of-plane buckling, which complicates the evaluation of the experimental data [37].

8. Conclusions

The nanomechanical testing methods discussed in this whitepaper have distinct strengths and limitations. The choice of method further depends on the material being studied, the sample dimensions and the targeted property. Nanoindentation is best suited for hardness and modulus measurements, e.g. on thick coatings. Micropillar compression is best suited for studying small-scale plasticity, e.g. within a specific grain in polycrystalline material. Microbeam bending provides access to the fracture behavior. In situ SEM and TEM testing provide high-resolution insight into the mechanisms governing material deformation. Compact mechanical testing systems such as MEMS are of interest when such in situ observations are required. Bulge testing provides complementary capabilities for ultrathin films.

Nanomechanical testing is a dynamic field, and future developments and refinements of the techniques are expected to continue supporting innovation in materials science.

Researchers interested in further details are encouraged to consult the MecaNano (“European network for the mechanics of matter at the nanoscale”) website www.mecanano.com for training resources and events announcements.

Acknowledgement

This publication is based upon work from COST Action CA21121 MecaNano, supported by COST (European Cooperation in Science and Technology) and funded by the Horizon Europe Framework Programme.

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