



# Current trends in nanomechanical testing research

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## Introduction

Small-scale mechanical characterization is essential for ensuring the service, performance, and lifetime of microscopic components, such as thin films and coatings, electronic sensors, and MEMS (Micro-Electro-Mechanical Systems). The first mechanical measurements on the submicrometer scale were enabled by the development of nanoindentation in the 1980s and early 1990s. From the beginning [1], Journal of Materials Research (JMR) has been the flagship journal for this field. In addition to countless contributed articles, the four previous focus issues published between 1999 and 2012 [2–5] have strongly contributed to disseminating the latest in method developments and trends in the field. This new focus issue on Advanced Nanomechanical Testing is a unique opportunity to identify and reflect upon the current research trends.

## Nanoindentation

Nanoindentation has been considered the reference small-scale mechanical characterization method for almost 30 years. Its robustness has been repeatedly proven on a wide range of materials [6]. In 2002, it became the first and, to date, the only nanomechanical testing method sanctioned with an ISO norm (ISO:14,577 [7]). Still, a non-negligible number of the contributions to this focus issue are devoted to further improving its reliability [8–13], based on emerging technologies. These include more accurate imaging [8, 10, 11] and faster data processing [9], which, for example, allow for improved characterization of the geometry of the indenter tip and the resulting contact area. In addition, this focus issue documents new method developments, which aim at further expanding the capabilities of nanoindentation beyond the measurement of hardness and Young's modulus, e.g., accessing the local creep properties [14], ductility [15], and surface free energy [16] of materials.

## Focused Ion Beam (FIB)-based nanomechanical testing

Complex mechanical behaviors and associated materials parameters have become accessible through advanced nanomechanical testing techniques relying on a preliminary structuring of the samples via focused ion beam (FIB) milling. The most popular application in the focus issue is measuring the local fracture behavior of materials [17–20]. In addition to providing a desired complex microsample geometry, the FIB micromachining technique allows one to visualize the deformation field that would otherwise be concealed under the surface [21].

As evidenced by the high number of related contributions, the use of the FIB has become essential in the field of nanomechanical testing [17, 18, 20–22]. This has inevitably raised concerns about possible measurement artifacts due to ion damage incurred during the specimen preparation [20, 23–25]. While detrimental effects can be ruled out for some applications [20], in other cases, workarounds are actively being developed to minimize [24] or to completely avoid [23] exposure of the samples to highly energetic ion beams.

## Big data

A significant trend pervading this focus issue is the strongly increasing use of nanoindentation as a high-throughput method for producing large amounts of data [26–31], which are then analyzed with emerging data science algorithms. The former is made possible through recent technical developments by hardware manufacturers which aim at performing a single indentation within no more than 1 s (KLA: Express Test and NanoBlitz, Bruker: XPM, Alemnis: UHSR, and others). This allows for the measurement of large mappings of up to 100,000 indents within 1–2 days. Such large datasets are then statistically analyzed, e.g., by machine learning algorithms [26]. The aim of such studies

is generally to access the intrinsic mechanical properties of the different components of modern alloys and composite materials.

### Correlative investigations

Because a statistical analysis does not always yield an unequivocal identification of the material phases, there is a growing trend of performing correlative analyses, which combine nanoindentation mapping with imaging of the sample surface using one or more analytical techniques [27–29, 32, 33]. Alternatively, numerical simulations can be used to inform the experiments and data analysis [32, 34]. The most widely used imaging techniques are backscattered electron diffraction (EBSD) for mapping the crystallographic orientation and energy dispersive X-ray spectroscopy (EDX) for local chemical characterization [27–29, 32, 33]. This focus issue also witnesses the use of other emerging techniques, such as atom probe tomography (APT), which provides chemical composition mapping with strongly enhanced spatial resolution [29, 33]. Regardless of the nanomechanical testing method, post-mortem analyses appear to greatly benefit from the increasing availability of image processing algorithms, such as digital image correlation [11, 25, 35, 36].

### In-situ investigations

In-situ experiments in a microscope offer an attractive alternative to combinatorial approaches, since they allow one to unequivocally correlate a given mechanical behavior to a given deformation mechanism. Their development has been a dominant trend in nanomechanical research throughout the past decade, with their application seemingly only being limited by the high costs associated with the techniques. This trend is continued in the focus issue, which features applications both inside scanning electron microscopes (SEM) [17, 18, 21, 22] and transmission electron microscopes (TEM) [23, 24].

### Extreme testing environments

Combining high spatial resolution with extreme environments is arguably the greatest experimental challenge currently faced by the nanomechanical testing community. Owing to intense research and development activities during the past decade, nanoindentation has been successfully expanded to applications at high temperatures. This has proven highly beneficial for investigating not only the deformation behavior of high-temperature materials [37, 38], but also the fundamental effects of heat treatments [31]. In addition, the focus issue features a study conducted at cryogenic temperatures [39], which underscores that progress is also being made in this direction. Environmentally controlled experimental setups also allow investigating the influence of humidity [19] on mechanical properties. The frontier of

extreme testing currently appears to lie with very high strain rates [40] and very high cycle fatigue [41] applications, with both requiring significant improvements to hardware in order to deal with the acquisition of mass data within a short time, as well as with their subsequent evaluation.

### Conclusions

This focus issue on Advanced Nanomechanical Testing documents how actively developments are taking place in the field of nanomechanical testing research. Most of the current developments aim at deriving benefits from the latest advancements in data acquisition, data processing, as well as the new environmental capabilities of nanoindentation hardware. The overarching goals are to improve the statistical significance of the results and to reproduce the extreme environments that are responsible for the failure of many systems.

In summary, this focus issue presents a diverse perspective of the challenges and opportunities offered at the frontiers of nanomechanical testing, which we hope demonstrates to the reader the exciting future of this topic.

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