

# Advances in multi-scale mechanical characterization

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**Note:** This paper is part of the Special Topic on Advances in Multi-Scale Mechanical Characterization.

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The multi-scale mechanical characterization of nanostructured, innovative structural and functional materials, biomaterials, self-reporting and adaptive structures, hierarchically architected systems, and miniaturized devices are intriguing due to their unusual and scale-dependent behavior. As evident from the significantly increasing publication output over the last two decades, the research community showed tremendous interest in advancing small-scale mechanical testing instrumentation and procedures to employ them for probing the local mechanical properties of advanced materials and devices. This is imperative because the reliability and performance of such intricate devices in various fields require in-depth knowledge regarding their properties and respective sensitivity to different service conditions, which are commonly only addressable by applying advanced *in situ* or *in operando* experimental techniques and/or simulation methods at small length scales. The insights obtained via such experiments, computations, theory, and simulations using empirical-based atomistic methods have led to the conceptualization and realization of new structures with tunable properties, thereby enabling the desired applications. Since this field is rapidly developing, the guest editors felt an update was required on new innovative advanced instrumentation, new theoretical models, and methods for improving the current understanding and applications thereof, which would be attractive for researchers working in these areas. This Special Topic covers high-quality contributions from renowned researchers across the globe on topics that provide fundamental insights into the mechanisms responsible for the scale-dependent mechanical behavior of advanced materials and their applications in deciphering complex challenges.

One of the important topics covered in this special collection relates to multi-scale mechanical testing and characterization of materials (experiment, instrumentation, theory, modeling and simulation, and applications). Several articles related to this are published in this collection. For example, the advanced structural performance of spider webs due to the unique combination of strength and toughness fascinated researchers, aiming to understand their mechanics. Earlier, several articles discussed mechanical properties modeling and structural design of spider webs for improving structural performance. However, 3D spider web structures are poorly understood compared to simple 2D structures. Buehler's group<sup>1</sup> has contributed an excellent article on developing computational methods to create synthetic 3D spider web structures. Their work reports a computational approach combining advanced modeling techniques to relate spider web graph microstructures to effective mechanical properties, focusing on strength and toughness. The web property prediction was accomplished using deep neural networks, which are trained on graph-structured web data and simulated mechanical properties. This study provides a basis for rapid digital analysis of spider web structures, aiding synthetic web design and optimization tools for structural design exploration and inspiration.

A further article by Polycarpou's group<sup>2</sup> reports the characterization of multi-scale physical properties of multilayer coatings of Ni and Cr onto Inconel 617 using high temperature tribological experiments, nanoindentation, and micro scratch testing. It was observed that aging (950 °C in a He environment) in both Ni/Cr (50 nm) and Ni/Cr (200 nm) films resulted in improved

nanomechanical and tribological properties. The abundant formation of a chromium oxide layer of higher thickness was found to enhance the hardness and wear resistance significantly.

Investigation of the indentation-induced deformation behavior of oxide single crystals is an interesting topic, as they are inherently brittle materials. However, several studies have shown that oxides undergo plastic deformation before fracture. Yet, Velazquez's group<sup>5</sup> observed a lower Young's modulus for the brittle materials' category. Interestingly, no clear evidence of plastic deformation was seen before cracking, even when approaching the melting point.

Pop-ins or sudden displacement bursts at constant loads during nanoindentation gained much attention, as the occurrence of pop-ins during loading is typically attributed to processes such as nucleating dislocations in the indented zone, occurrence of phase transformations in semiconductors such as Si, and fracture (usually at higher penetration depths). Though many models and experiments were carried out on various materials to understand these special events during loading, Mukhopadhyay's group<sup>4</sup> reported the physics behind the loading-rate dependency on the nano-scale plastic events in titania-densified alumina ceramics. They realized pop-in events at lower loading rates occur due to shear-induced homogeneous dislocation nucleation. In contrast, shear band formation, the interconnection between nucleated dislocations and microcracking in titania-densified ceramics, are the primarily responsible mechanisms at higher loading rates.

Silicon alloyed steel, known as electrical steel, is typically used in electrical power transformers, generators, and motors. However, the production of thin sheets is highly challenging due to crack formation during the rolling process. Jain *et al.*<sup>5</sup> investigated the microstructure, crystallographic texture, and stress state plays on the formation of alligator and edge cracks during cold rolling of high Si electrical steel. It was found that alligator cracks and edge cracks could be controlled by intermediate and hot band annealing, respectively. Alligator cracks start from the shear bands during cold rolling due to increased strain incompatibility. In addition, the Taylor factors of different slip systems assist the crack following the hot band interface.

Knowledge of the indentation size-dependent dislocation-mediated plasticity is essential in many materials and technologies. Ishikawa *et al.*<sup>6</sup> performed an interesting study to understand the dislocation structure-imprint size relation by Vickers-indentation induced 3D dislocation structures (flower pattern, rosette pattern, and a triangular area), investigated by luminescence and electron microscopy studies. Intriguingly, a geometric similarity between imprint size and the dimension of the dislocation structure was observed. This study can help determine the maximum thickness of the indentation-affected layer over the entire wafer area.

Low Poisson's ratio (0.1–0.25) materials fracture easily, and high Poisson's ratio (0.35–0.45) materials offer higher fracture resistance. Oxide glasses typically have a Poisson's ratio  $<0.3$ ; however, the brittle-to-ductile transition that can also be seen in oxide glass is entirely unknown. To unravel this, Smedskjaer's group<sup>7</sup> measured the fracture energy of six high Poisson's ratio oxide glasses, but no such relationship was observed between Poisson's ratio and fracture energy. Instead, they found a relation between Poisson's ratio and Young's modulus of oxide and metallic glasses.

This special collection also contains a tutorial article that discusses the new instrumentation development for improved *in situ* mechanical characterization of materials. Useinov's group<sup>8</sup> combined optical microscopy with instrumented nanoindentation to visualize the real-time indenter penetration through the material. In this advanced instrumentation, a laser can focus through the transparent indenter shaped to special double-edge geometry and provide real-time video imaging of the indenter penetration to visualize the cause of pop-in occurrence, fracture initiation, pile-up or sink-in formation, shear band formation in bulk metallic glasses, phase transformations, or slip lines in the vicinity of the indenter. This tutorial article discusses the preparation and characterization of such a transparent diamond indenter, the importance of such combined experimentation, and a few results and promising application areas.

Furthermore, this Special Topic includes studies on materials testing in harsh environments, high-pressure research, and emerging materials for mechanical applications. An article published in this collection is related to the theme of x-ray wavelength calibration in synchrotron and x-ray-free lasers by using cerium oxide as standard. Therefore, it demands a better understanding of the high-pressure deformation mechanism. Sturtevant's group<sup>9</sup> measured the yield strength of Ceria using static compression in radial diamond anvil cells. They observed that the [001] direction had a slightly preferred orientation along the compression axis as pressure increased.

The collection also features several papers on the fabrication and mechanical manipulation of micro- or nano-scale objects and devices, *in situ* observation of the mechanical deformation behavior, and structure-property correlation studies. For example, environment-friendly lead-free ceramics with attractive properties and high thermal and mechanical stabilities are of great interest to the industry. Barium titanate ( $\text{BaTiO}_3$ ) is a material of this type with the piezoelectric property used in patterned and thin film forms in micro- and nano-scale devices. However,  $\text{BaTiO}_3$  fails due to cracking and/or interface delamination by the development of stresses during deposition. To better understand the cracking behavior of  $\text{BaTiO}_3$  at the nanoscale, Jaya's group<sup>10</sup> conducted *in situ* microcantilever bending-based fracture and tensile tests based on shear lag tests in combination with digital image correlation and quantified the fracture toughness, fracture strength, and interface shear stresses. Interestingly, the damage tolerance depends on film thickness. Further, fracture resistance was found to decrease for nanocrystalline  $\text{BaTiO}_3$  compared to its bulk counterpart.

The high-volume fraction of grain boundaries in nanocrystalline materials significantly changes materials' physical, mechanical, and chemical properties compared to bulk or coarse-grained counterparts. Consequently, nanostructuring of metals/alloys substantially enhances the strength due to the suppression of dislocation activity. Nevertheless, twin formation is complex in metals such as Al due to high stacking fault energies. Zhang's group<sup>11</sup> fabricated Al–Zr alloys with a high density of growth twins. The *in situ* micropillar compression tests demonstrate that nanotwinned Al–Zr alloys reach a flow stress of  $\sim 1$  GPa. The Zr solute was identified as key to the formation and deformation of nanoscale twins.

This collection also covers articles on mechanical investigations of various materials: from ceramics and alloys to cells, tissues,

and hydrogels. For instance, multiple principal element alloys, known as high-entropy alloys (HEAs), are currently of outstanding research importance in materials science and engineering. There are several practical challenges regarding their usage in energy, aerospace, and advanced manufacturing sectors. One of the fundamental challenges is compositional inhomogeneity, which impacts their defect movements. The groups of Ding and Salje<sup>12</sup> have utilized acoustic emission spectroscopy and demonstrated the differences between the  $\text{Fe}_{40}\text{Mn}_{40}\text{Co}_{10}\text{Cr}_{10}$  high-entropy alloy and conventional 316L stainless steel. Under tensile loading, they observed the coexistence of dislocation movement and detwinning/twinning processes in the HEA. The study evidenced that dislocation movements in the HEA last longer than in 316L steel. However, the aftershock rates for dislocation–movement signals are the same for both materials.

Piezoelectric materials find vast applications in advanced solid-state devices. The microstructural length scales can tailor the properties (dielectric, piezoelectric, and electromechanical); therefore, they are likely candidates for transducers, sensors and actuators, and many advanced miniaturized electronic and energy harvesting devices. It is imperative to have in-depth knowledge of mechanical, electrical, and electromechanical behavior under various complex conditions. In a perspective article category, Kathavate *et al.*<sup>13</sup> provided a systematic outlook on piezoelectric materials' *ex situ* and *in situ* deformation behavior and ferroelectric/elastic distortion during deformation. The multi-functionality of the piezoelectric materials, microstructural length scales contribution to the properties, and mechanical characterization techniques from mesoscale to nanoscale and the wealth of information obtained through experiments are summarized in conjunction with the current challenges and future perspectives.

Another central platform focused on in this Special Topic includes tuning functional properties by manipulating material defect structures. Mechanical strain-induced tuning of electronic and photonic performance of 2D layered materials is an exciting topic for achieving the ultimate performance of 2D materials-based devices. Strain engineering in 2D materials is well-studied, and researchers are developing new strategies to address the challenges. Layered  $\text{MoS}_2$  can withstand large strains up to 11%; therefore, physical properties can be well-tuned. However, slippage of these atomically layered materials on the substrate restricts further strain tuning. Xie's group<sup>14</sup> developed a facile three points approach combined with a dry transfer method that can apply uniaxial strain to 2D materials. They studied the slippage behavior of  $\text{WSe}_2$  on a polycarbonate substrate and observed that a larger lateral friction force suppresses slippage following thermal annealing.

In summary, this Special Topic offers readers a glimpse of several notable recent advancements in small-scale mechanical testing instrumentation for probing the local mechanical properties of advanced materials and devices, new methods, analyses,

interpretations, as well as the diverse perspectives of the challenges and opportunities offered at the frontiers of multi-scale mechanical testing.

In conclusion, the guest editors hope the readers will benefit from the wealth of information published in this focused special collection and serve to improve this multidisciplinary field to a more advanced level.

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## AUTHOR DECLARATIONS

### Conflict of Interests

The authors have no conflicts to disclose.

## DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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