

Beyond Indentation Hardness and Modulus: Recent Advances in Nanoindentation Techniques: Part I

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Instrumented indentation testing has long been used for determining hardness and modulus at submicrometer-length scales. Recently, advances in indentation-based mechanical testing have enabled quantitative characterization of more complex mechanical behaviors at the nanoscale, such as stress–strain relation, visco-elastic/plastic property, creep and stress relaxation behavior, strain-rate sensitivity, fracture toughness, interfacial adhesion, and in situ nanoindentation in electron microscopes. The ability to characterize quantitatively and tailor the mechanical properties of individual microstructures/phases/constituents/interfaces in bulk materials at the nanoscale, as well as thin films and low-dimensional materials, has been critical for making revolutionary advances in materials development. The scope of Part I of this *JOM* special topic is to review some of these recent advances in nanoindentation techniques on measuring various nanoscale mechanical properties. The topic will continue with Part II scheduled to publish in early 2018.

Recently the measurement of the creep response of materials at small scales has received renewed interest. Despite an increasing capability to perform high-temperature nanomechanical testing on advanced instrument, several significant experimental and modeling challenges remain in small-scale mechanical testing at elevated temperatures. In this regard, relating the creep response probed by high-temperature instrumented indentation experiments to macroscopic uniaxial creep response is of great practical value. The article titled “On the Measurement of Power Law Creep Parameters from Instrumented Indentation” by Sudharshan Phani et al. reviews various methods currently being used

to measure creep with instrumented indentation, with a focus on geometrically self-similar indenters, and their relative merits and demerits from an experimental perspective. Additionally, a comparison of various methods that use instrumented indentation results to predict the uniaxial power law creep response of a wide range of materials is presented to assess their validity.

Instrumented indentation or nanoindentation can also be employed to measure thin film interfacial adhesion energies by producing well-defined areas of delamination. When combined with the proper mechanics-based model and characterization of the failing interfaces, nanoindentation induced delamination becomes a powerful tool to quantify interfacial fracture properties. The review article on “New Insights into Nanoindentation-Based Adhesion Testing” by Kleinbichler et al. presents new development of the technique, which was first introduced by Marshall and Evans in the 1980s. The improvements are demonstrated using a WTi/Si₃N₄ film system on a rigid silicon wafer where the WTi acts as a stressed overlayer. Furthermore, focused ion beam cross-sectioning and confocal laser scanning microscopy were used to characterize failing interfaces and additional fracture events, as well as to determine the adhesion energy.

By adapting standard nanoindentation test methods, simple protocols capable of probing thermally activated deformation processes can be accomplished. Abrupt strain-rate changes within one indentation allow the determination of the strain-rate dependent variation of hardness at various indentation depths. To exclude thermal drift at low strain rates, long-term creep experiments can be performed by using the dynamic contact stiffness for determining the true contact area. From both procedures, hardness and strain-rate, and consequently strain-rate sensitivity and activation volume, can be reliably deduced within one

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indentation, permitting information on the locally acting thermally activated deformation mechanism. The article titled “Advanced Nanoindentation Testing for Studying Strain-Rate Sensitivity and Activation Volume” by Maier-Kiener and Durst reviews various testing protocols, including possible challenges and improvements. Moreover, different examples showing the direct influence of the crystal structure and/or microstructure on the underlying deformation behavior in pure and highly alloyed material systems are presented.

To understand the phenomena observed during nanoindentation tests, modeling and simulation methods have been developed to predict the mechanical response of materials during nanoindentation. Nevertheless, challenges remain in these computational approaches because of their length scales, predictive capabilities, and accuracy. The article titled “Modeling and Simulation of Nanoindentation” by Huang and Zhou reviews recent progress and challenges for modeling and simulation of nanoindentation, which includes an overview of molecular dynamics (MD), quasicontinuum method (QCM), discrete dislocation dynamics (DDD), and crystal plasticity finite element method (CPFEM). It discusses the way to integrate the multiscale-modeling approaches seamlessly with experiment studies to understand the length-scale

effects and microstructure evolution during nanoindentation tests and create a unique opportunity for establishing new calibration procedures to nanoindentation techniques.

The following articles are published under the topic “Beyond Indentation Hardness and Modulus: Recent Advances in Nanoindentation Techniques: Part I” in the November 2017 issue (vol. 69, no. 11) of *JOM* and can be accessed via the *JOM* page at <http://link.springer.com/journal/11837/69/11/page/1>.

- “On the Measurement of Power Law Creep Parameters from Instrumented Indentation” by P. Sudharshan Phani, W.C. Oliver, and G.M. Pharr.
- “New Insights into Nanoindentation-Based Adhesion Testing” by A. Kleinbichler, M.J. Pfeifenberger, J. Zechner, N.R. Moody, D.F. Bahr, and M.J. Cordill.
- “Advanced Nanoindentation Testing for Studying Strain-Rate Sensitivity and Activation Volume” by Verena Maier-Kiener and Karsten Durst.
- “Modeling and Simulation of Nanoindentation” by Sixie Huang and Caizhi Zhou.