Preface

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Nanoindentation provides an effective and convenient quantitative technique for probing the mechanical behavior at the nano, micro, and macroscales. Nanoindentation instrumentation has become increasingly accessible in the past three decades, and numerous techniques have been developed. This special issue includes a collection of papers on new experimental methods, contact mechanics theories, numerical simulations, and applications on nanoindentation for a range of materials, including brittle and ductile alloys, high wear resistance coatings, geomaterials, biomaterials, nuclear fuels, ceramics, semiconductors, and electronic packaging materials.

The twelve papers are divided into three categories: (1) nanoindentation techniques for probing the mechanical behavior at room and elevated temperatures, including high temperatures; (2) Numerical methods for contact mechanics analysis to determine the constitutive and failure behaviors; and (3) Nanoindenter as a loading device to enable materials testing experiments and machining processes. We provide a brief summary of the included papers following this sequence.

In the paper by Jeon, et al., the authors proposed a new approach to analyze contact mechanics in indentation on metals in order to determine the onset of both brittle and ductile fracture and the fracture toughness. They established a criterion by mean pressure in brittle materials, and also developed a failure criterion based on the fracture strain and critical plastic zone size for ductile fracture. The authors validated their methods using experiments on a number of alloys. Their technique is suitable for measurement of fracture toughness on small

samples and for nondestructive in-situ measurements on structures without recourse to notched specimens that are typically used for fracture toughness measurements.

Cole, et al. investigated the local mechanical behavior of steel experiencing large amplitude harmonic loading in a clamped steel beam, and evaluated the modulus and plastic work evolution by indentation. They correlated the grain microstructural evolution with the modulus reduction and other property changes as a function of loading cycles and attributed the reduction to grain fragmentation and reorientation during fatigue.

Silicon is used in electronic and photovoltaic devices typically in diamond cubic crystal and amorphous phases. In the paper by Wong, et al., the authors investigated the phase transformation of silicon under the high pressure induced by a spherical tip. They used cross-sectional transmission electron microscopy and micro-Raman measurements to characterize the indenter tip and phases, and identified two different pathways that induce the phase transformation of diamond cubic crystals into a mixture of body-centered cubic and rhombohedral phases. One pathway nucleates a phase transformation while the other induces crystalline defects followed by a phase transformation at higher applied stress. They further showed that one pathway leads to a larger phase-transformed volume than the other, thereby providing such transformed materials for a range of applications.

DelRio and Cook used nanoindentation in forensic case studies on hair, documents, fingerprints, and explosives via atomic force microscopy (AFM). They measured the indentation modulus and pull-off force, and determined that a unique time-evolving quantitative nanoindentation load-displacement relationship exists. That relationship is the “fingerprint” of the material. Their study established the use of AFM as a quantitative nanoindentation tool for obtaining evidentiary information, which is anticipated to provide a new methodology in forensic science.

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In the paper by Csanádi, et al., the mechanical properties of a tungsten carbide coating on steel substrate were measured by an inverse method. A Berkovich tip was used to press into the coating to induce yielding in both the coating and the substrate. The extended finite element method was used to simulate the contact mechanics problem. An excellent agreement was reached for both the nanoindentation load-displacement curves and the indent profiles, both in the coating and the steel. This inverse approach provided measurements of hardness, modulus, and yield strength for the coating and the steel substrate.

Tertuliano, et al. measured the mechanical properties of niobium carbides with high hardness and high modulus. Two configurations were used, carbide particles dispersed in steel, and sintered carbide particles. Nanoindentation was made to reach a depth high enough to introduce a residual indent for hardness measurements. Consistent data were obtained for the mechanical properties for sintered carbide, while variations were observed in property data for dispersed carbide particles.

In a paper by Frazer, et al., the authors used two different nanoindentation techniques to measure the mechanical properties of a nuclear fuel particle with a diameter less than 1 mm. They used a micro-machining technique to prepare a micro-ring specimen, and a nanoindenter to apply compression to crush the ring while recording the resulting load – displacement response. In this technique, they were able to determine the stiffness and convert it into the modulus. In another technique, they used a nanoindenter to press directly into a polished fuel particle surface. The two sets of experiments provided consistent results. Their study has established nanoindentation as a rapid tool for assessment of the mechanical properties of nuclear fuel particles.

Hydraulic fracturing, or fracking has revolutionized the petroleum industry in the recent years. In fracking, the procedures used depend on the mechanical properties of the geomaterials involved. Indentation at different length scales is especially suitable for probing the heterogeneous properties of geomaterials such as shales. Han, et al. conducted an investigation to evaluate the role of cohesion, friction angle, and tensile strength on the contact response. They compared their simulation data with micro-indentation results on a shale. Their work indicated that the Mohr-Coulomb constitutive law, with the use of appropriate cohesion and frictional parameters is appropriate for the analysis of the spherical contact behavior of shale.

In data analysis for nanoindentation, a perfect tip geometry is usually considered to determine the mechanical properties. In reality, however, a nanoindenter tip has defects, a Berkovich tip does not necessarily have an atomically sharp apex, and a spherical indenter tip does not necessarily have a perfectly spherical shape at its front. In addition, an indenter

tip undergoes deformation that contributes to errors. Keryvin, et al. developed a method to take into account both tip imperfections and deformations by modifying the experimental nanoindentation response curve to generate artificial results for a workpiece material under perfect tip geometry, and from there to extract accurate property data. Implementation of their method is expected to increase the accuracy of indentation-determined properties.

Nanoindentation under environmental conditions provides mechanical properties necessary in material design optimization and reliability analysis. Harris, et al. described a nanoindentation system that is capable of reaching temperatures up to 950 °C in vacuum, and conducted nanoindentation tests on polycrystalline tungsten. Their results showed that at lower temperatures, the elastic-plastic analysis of contact mechanics provided modulus values that were consistent with literature results, while at higher temperatures the viscoelastic compliance of the material has to be taken into account in the calculation of elastic modulus.

Jeon, et al. used dynamic indentation as a tool for micromachining. They used dual nanoindenter heads in a 250 mm × 250 mm area, and demonstrated this capability by machining a mold for an array of lenses with depths between 1 μm and 6 mm. To reduce the pile-up associated with ductile metals, they introduced thermal annealing, resulting in a high precision geometry for the lens array in the metal mold. This development opens a new path for using dynamic nanoindentation as a tool for nanoscale and microscale machining.

Xu, et al. investigated the mechanical behavior of a heterogeneous molding compound in packaged integrated circuits, consisting of assorted glass beads in an epoxy. A large spherical tip was used to press into the molding compound. Nanoindentation was made on a grid of sites. The grid size was increased until the average mechanical properties did not change further with the increase in grid size in order to allow for identification of the representative volume size. A linear viscoelastic contact mechanics analysis was implemented to extract the mechanical properties. Their approach allows for direct measurement of process-dependent mechanical properties of a molding compound actually used in an integrated chip for reliability analysis.

The papers in this Special Issue show a trend moving from obtaining basic mechanical properties, such as hardness and modulus, in nanoindentation to obtaining the comprehensive constitutive and failure behaviors of materials. This trend is expected to continue in numerous current and emerging applications, such as nano/microscale machining, and forensic science and engineering.