

Summary

In the preparation of this book and the organization of the symposium from which it grew, there has been an attempt to look in some detail at various facets of a complex field. Some papers have dealt with mechanistic studies of interest to researchers in materials science. Other papers have addressed rather pragmatic questions involved in day-to-day engineering practice. Still others have provided examples of how microindentation testing may be used in creative, less conventional ways to probe the near-surface mechanical properties of materials.

All along the path from testing concepts to implementation and data analysis, there are fertile areas for study. Advances in the utility of microindentations can result from both improved machine designs and improved understanding of how properties and microstructures of materials affect their observed behavior.

Fundamentals of Indentation Testing

Samuels begins this section of the book with a review paper which questions some of the traditional models of the indentation process in metals. He examines indentation mechanisms involving cutting, elastic behavior, and compression. In this chapter, the strengths and limitations of modeling using the three mechanisms are considered, and the compression mechanism is favored. Implications from this model are discussed regarding matters of impression size, interconversion of hardness scales, impression shape, friction, and surface topography. The need to characterize better the deformation of the material adjacent to the impressions is also emphasized.

Marshall and Lawn review the use of microindentation methods in investigating the fracture and deformation behavior of brittle materials, such as ceramics and glasses. They argue against the shortcomings of traditional models of hardness phenomena, which are largely based on plasticity considerations which presume volume conservation and homogeneity. The authors present an interpretation based on intermittent "shear faulting" and also include, where appropriate, a contribution from structural compaction or expansion. Examples relating indentation behavior to elastic modulus and fracture toughness are provided, along with an illustration of surface residual stress determination in brittle materials.

The next three papers all involve some aspects of the load versus indenter displacement behavior of materials. *Pollock, Maugis, and Barquins* use a

three-faced pyramidal indenter under loads of 0.01 to 30 mN to study a range of behavior in submicrometre surface layers. The authors also use a friction instrumented scratch hardness tester. Both instruments operate under computer control. Studies of small-scale microstructural features, such as grain boundaries and dislocation configurations, are summarized. The influences of elastic-plastic response, elastic recovery, and indentation creep are discussed.

Continuing on with the topic of indentation elastic-plastic relationships, *Loubet, Georges, and Meille* provide models for the interpretation of load versus microdisplacement curves. Young's modulus and Vickers indentation pressure are calculated for hardened 52100 steel and annealed aluminum from their analysis. The authors discuss the correlation between depth-based measurements and traditional light optical measurements for hardness determination. By examining the residual impression depth after off-loading, estimates of the ratio of work supplied to that retained in the material can be made.

Oliver, Hutchings, and Pethica complete the series of three papers dealing with load-displacement behavior by describing a set of experiments on hardness as a function of depth on gold, nickel, lithium fluoride, and silicon. Detailed examinations of impressions as shallow as 20 nm are made using both transmission and scanning electron microscopes. Modifications to Meyer's law are proposed, based on a consideration of the increasing influence of microstructural features (such as dislocation cells) when indentations become very small.

Dislocation aspects of indentations are the focus of the contribution from *Armstrong and Elban*, who model the crystallographic aspects of microindentations in two materials: magnesium oxide and cyclotrimethylenetrinitramine. These authors extend the earlier continuum mechanics approaches to account for the interactions of moving dislocations in accommodating the localized strains associated with the indentation process.

Techniques and Measurements

The paper by *Tabor*, which introduces the section on testing methodology, reviews many of the aspects of indentation processes and provides an overall perspective from which the limitations of microindentation testing can be viewed. It deals with the concepts of the expanding spherical cavity approach to elastic-plastic indentation behavior and with problems associated with load application and crack formation in brittle materials. The paper ends with some cautionary comments related to the interpretation of low-load penetration depth data.

Sargent carries forward some of the concerns of the previous authors on indentation size effects and introduces an empirical indentation size effect (ISE) index which is defined in terms of the applied load and indentation diameter (or some similar dimensional parameter). He applies the analysis of

the ISE to a series of metals, ceramics, single crystals, and polycrystals at various temperatures to generate error ellipsoid plots. These plots are suggested as a compact way to display rigorous interpretations of the ISE in a range of materials.

The effects of external stresses imposed during microindentation testing procedures are investigated by *Vitovec*. This author uses a tensile stage on a bench-top microindentation machine to vary the externally applied load on the test specimen. He found that hardness decreased linearly with increasing imposed tensile stress. Yielding, surface films, and residual stresses all seemed to affect the hardness-load relationship and the trend of the stress effect.

Standardization of microindentation hardness testing procedures is extremely important if useful engineering data are to be obtained. *Kelley, Johnson, and Lashmore* discuss the fabrication and certification of copper and nickel electroformed standard reference materials at the National Bureau of Standards. Concerns relating to the choice of testing procedures for material certification are discussed. The need to prepare standard samples within well-known ranges of microindentation hardness numbers for both Knoop and Vickers testing presents particular problems in fabrication.

The testing of thin, hard coatings presents particular challenges to those who need to determine microindentation hardness numbers. Extremely small impressions tax traditional optical microscopy. *Westrich* describes an alternative approach to measuring very small impressions using a scanning electron microscope and a diffraction grating as an internal size standard. This greatly reduces the scatter in data frequently associated with hard coating testing. A comparison between light optical and electron optical measurements is made for titanium nitride and hafnium nitride coatings, and correction factors are provided. Computerized curve fitting is used to facilitate conversion of hardness scales.

Engineering Applications

The last section of the book begins with a paper by *Blau* on the application of microindentation techniques to tribology. Three aspects of microindentation methods are discussed: first, the use of indentations and scratch methods in measuring small amounts of wear loss from the surfaces of metals; second, the characterization of surfaces of test pieces prior to wear testing, in which it is shown that, depending on the type of wear, increasing hardness does not always lead to increased wear resistance; and third, several uses of microindentation tests in the study of the subsurface deformation of metals from wear.

The contribution by *Kosel* relates to the use of scratch tests of controlled depth in the study of the fracturing process of carbides during abrasive wear and machining. Instead of using conventional diamond indenters, particles similar in kind to the actual abrasives encountered in the field are used. This

provides insight into the mechanisms of actual field abrasion conditions. A specially designed instrument for scratch testing in the scanning electron microscope permits observations of prescribed areas on carbide-laden specimens to be examined after successive scratches.

During powder metal processing, the earlier the properties of materials can be measured, the better one is able to monitor and adjust parameters for quality control of the final product. *Shives and Smith* discuss several of the practical problems of mounting and testing powder metal particles. A novel technique involving the embedment of particles into an electrodeposit prior to polishing and hardness testing is described. Commercial nickel alloy, stainless steels, and aluminum powders are used to illustrate various preparation techniques and sources of error in the data.

The final trio of papers in this book deals with various kinds of coating hardness and adhesion testing problems. *Vingsbo, Hogmark, Jönsson, and Ingemarsson* treat the problems of thin metal coatings by proposing a model in which a mixture-rule relationship is derived for coatings of various thickness. A linear relationship is seen to hold for indentations larger than a limiting value which depends on the materials being tested. Separate hardness values for the coating and substrate are estimated.

The adhesion of polymer films to glass-epoxy layered circuit-board substrates is quantified by *Engel* using conical or ball indenters. The extent of the annulus of de-adhered coating surrounding the impressions is the means by which relative film adhesion is obtained. The paper contains an analysis of the contribution of the coating to the hardness of the test piece surface. The methods described quantify debonding tendencies in laminar boards due to mechanical handling.

The final paper in this book describes an engineering approach to the quantification of what has been known as "pencil hardness." *Walker* describes a study in which Knoop microindentation hardness numbers for various grades of pencils are measured and correlated with data from painted surfaces. The linear correlation between Knoop and pencil hardness scales permits a useful method for assessing the hardness of painted surfaces.

Obviously, this book could not hope to cover the breadth and depth of a field so great as microindentation testing in all of materials science and engineering, but what it provides is a series of fundamental insights, testing guidelines, and creative applications of many techniques to engineering practice. The editors hope that similar texts will follow to stimulate greater scientific enlightenment and enable the development of effective solutions to the many challenges of surface science and engineering.

Peter J. Blau

Brian R. Lawn

National Bureau of Standards, Gaithersburg,
MD, 20899; symposium cochairmen and
editors.