MICROINDENTATION TECHNIQUES IN MATERIALS SCIENCE AND ENGINEERING

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Foreword

This publication, *Microindentation Techniques in Materials Science and Engineering*, contains papers presented at the Microindentation Hardness Testing Symposium and Workshop, which was held 15–18 July 1984 in Philadelphia, PA. The event was jointly sponsored by ASTM, through its Committee E-4 on Metallography, and the International Metallographic Society. Chairing the symposium were Peter J. Blau and Brian R. Lawn, both of the National Bureau of Standards, who also served as editors of this publication.

Related ASTM Publications

Practical Applications of Quantitative Metallography, STP 839 (1984), 04-839000-28

MiCon 82: Optimization of Processing, Properties, and Service Performance Through Microstructural Control, STP 792 (1983), 04-792000-28

MiCon 78: Optimization of Processing, Properties, and Service Performance Through Microstructural Control, STP 672 (1979), 04-672000-28

Damage Tolerance of Metallic Structures: Analysis Methods and Applications, STP 842 (1984), 04-842000-30

A Note of Appreciation to Reviewers

The quality of the papers that appear in this publication reflects not only the obvious efforts of the authors but also the unheralded, though essential, work of the reviewers. On behalf of ASTM we acknowledge with appreciation their dedication to high professional standards and their sacrifice of time and effort.

ASTM Committee on Publications

ASTM Editorial Staff

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Introduction

Microindentation hardness testing and its associated methodology continue to be used widely in materials evaluation. The subject matter in this book, however, goes beyond the mere obtaining and interpreting of microindentation hardness numbers. It deals with the use of indentation methods in the study of intrinsic deformation properties, residual stress states, thin-film adhesion, and fracture properties in a variety of materials.

The last such collection of contributions to the general field of microindentation hardness testing in the United States was published more than a decade ago.[1] Since then, a considerable body of work has improved our understanding of indentation behavior as it relates to fundamental material properties and has extended the range of applications to engineering practice. The symposium from which the content of this book derives was organized as a joint venture between the International Metallographic Society, the American Society for Metals, and ASTM. It was held on 15 and 16 July 1984, in Philadelphia, PA, in conjunction with the 17th annual International Metallographic Society technical meeting. Contributors and attendees at the symposium represented eleven countries in addition to the United States, and their technical interaction provided a forum for discussion of microindentation research and technology.

This volume is organized into three sections dealing with fundamentals, testing techniques, and engineering uses of microindentation-based methods for metals, ceramics, and polymers. The reader will find that the classification of papers into the three sections is somewhat arbitrary. Nevertheless, as one proceeds through the book one will note something of a progression from scientific principles to practical applications.

The papers in the section on fundamentals question some of the traditional theories of indentation behavior and examine how these theories relate to intrinsic material properties. This section covers metals, ceramics, and polymers. There is an emphasis in many of these papers on a relatively new approach to quantifying microindentation behavior through the use of the load-displacement response of materials. The section on techniques addresses such topics as hardness scale interconversions, measurement methods, errors, standardization, and time and size effects. The third section, on applications, contains six papers which exemplify some of the many engineer-

[1] *The Science of Hardness Testing and Its Research Applications*, American Society for Metals, Metals Park, OH, 1973.

ing uses of microindentation techniques. Two of these papers deal with sliding wear and abrasion damage assessment, one with the mounting and microindentation testing of small particles, and three with various aspects of coatings testing.

The editors would like to express their sincere gratitude to the contributors and reviewers of this volume for their cooperation and efforts. The International Metallographic Society and the U.S. Office of Naval Research provided travel support for some of the symposium contributors from outside of the United States. Chris Bagnall and James McCall of the International Metallographic Society are also acknowledged for their help with many of the necessary details required to organize the symposium facilities and funding.

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Fundamentals of Indentation Testing

Leonard E. Samuels[1]

Microindentations in Metals

REFERENCE: Samuels, L. E., "**Microindentations in Metals,**" *Microindentation Techniques in Materials Science and Engineering, ASTM STP 889,* P. J. Blau and B. R. Lawn, Eds., American Society for Testing and Materials, Philadelphia, 1986, pp. 5–25.

ABSTRACT: The mechanisms involved when an indentation is made in the surface of a metal by a blunt indenter have received a good deal of attention, but little of this work has appeared in publications that might easily come to the attention of those who actually carry out hardness tests. Even less of the work has been analyzed for its implications in practical hardness testing. The main models of indentation which have been proposed to date are reviewed, with particular attention to the one that most closely relates to the realities of hardness testing. This model, developed by Mulhearn, proposes that indentation occurs by radial compression, the formation of the indentation being likened to the expansion of a hemispherical cavity. The implications of this model to indentation hardness testing, and specifically to microindentation testing, are considered. This involves consideration of the current status of views on the effects of indentation size on the apparent hardness number.

KEY WORDS: microindentation hardness testing, indentation, indentation hardness test, microhardness test, mechanical properties, compression, models

The distinguishing feature of the hardness indentations with which this publication is concerned is their size. A microindentation can be arbitrarily defined as one which has a diagonal length of less than 100 μm, noting that increasing interest is being taken in indentations with diagonal lengths less than 10 μm. The force that has to be applied to an indenter to produce indentations of this size is important to the design and operation of a hardness testing machine but not necessarily to the mechanism of the indentation process.

Little or no work has been carried out on the mechanism of indentation at this scale. It is, consequently, necessary to draw on the work carried out on larger macroindentations and extrapolate downwards, relying in the first instance on the principle of geometric similarity. This principle is fundamental

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to macroindentation testing and, although it does not follow that it will be applicable down to the smallest indentations, the principle should not be abandoned lightly. It will be accepted here as applying unless there is good evidence to the contrary.

Some of the investigations referred to in the treatment which follows were carried out on Brinell macroindentations. Only pyramidal indenters, principally of the Vickers (136° included face angle) and Knoop types, are used in microindentation tests, but the general principles that emerge from the study of Brinell indentations can still be applied because of the geometric similarity of the indentations. This is a particularly reasonable assumption if the radial compression mechanism of indentation which will be discussed is accepted. Note also that some of the investigations have been carried out using two-dimensional wedge indenters. This is done for experimental simplicity and because it is possible to treat theoretically only the two-dimensional situation; it is then assumed that a pyramidal indentation has a radial symmetry. Finally, little work has been carried out directly on Knoop indentations. It has to be assumed that a Knoop indenter behaves in essentially the same way as a Vickers indenter, noting that it is the blunter of the two.

Mechanisms of Indentation

Cutting Mechanisms

Analyses of the mechanism of indentation of metals by blunt pyramids commonly has been based on a slip-line field solution developed originally by Hill, Lee, and Tupper [*1*]. This is a two-dimensional treatment of an ideal rigid-plastic material; that is, a material that is perfectly rigid up to a yield stress but then deforms plastically without work hardening. The application of this treatment, and several of its subsequent developments, have been reviewed by Shaw [*2*].

The slip-line field solutions are all based on the supposition that the indenter cuts the specimens, for example, along Plane ab in Fig. 1. It follows that this creates two new surfaces, which rotate about Point b as the indentation forms. The material originally located at Point a is thus relocated to Point c, and the material in Volume bdefc is plastically deformed with a sideways and upward motion. The rigid-plastic boundary is bdef. Relative movement is required between the surfaces of the indenter and the specimen, and consequently friction should have a significant effect, which can be taken into account by the solution (compare the left and right sides of Fig. 1). Work hardening of the specimen material is predicted to have a similar effect to an increase in the coefficient of friction.

Mulhearn [*3*] carried out a detailed quantitative analysis of indentations made by wedges, cones, and pyramids with a range of included angles, investigating a material whose mechanical characteristics closely approached

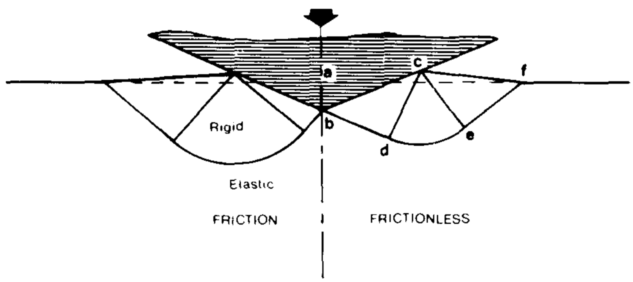

FIG. 1—*Slip-line field solution for the indentation of a rigid-plastic material by a blunt wedge, assuming a cutting mechanism of indentation. The angles of the slip-lines have been modified slightly in the lefthand half of the sketch to account for the effects of friction.*

those of an ideal elastic-plastic solid—that is, a solid which is elastic up to a yield stress and then deforms plastically with no work hardening; slip-line field theory can reasonably be assumed to apply to such a material. Mulhearn [*3*] established that the characteristics of the indentations conformed to the cutting model when the wedge angle was less than about 60°. Indentations made with wedges that had larger angles, however, did not conform. It is worth recounting the evidence that established this:

1. A cutting mechanism requires that the elastic-plastic boundary should not extend much below the tip of the indentation. In fact, as described later in more detail, it extends for a considerable depth below the tip of the indentation (Fig. 2).
2. A cutting mechanism proposes that the displacement of points on the specimen surface would have a large component parallel to the surface. In fact, the displacement is relatively small (Fig. 3 [*3,4*]).
3. If cutting occurred, the height of the lip raised adjacent to the indentation would be approximately one third the depth of the indentation. In fact, it is only a fraction of this, never more than half the required amount.

The discrepancy between predictions and observations increases as the wedge angle increases above 120° and becomes marked by a wedge angle of 140°. Mulhearn concludes that the cutting model is not valid at this point.

Mulhearn [*3*] has also established that the same discrepancies arose with a material (annealed 30% zinc) whose mechanical characteristics are considerably different from those of an ideal elastic-plastic solid. Brass has a low yield stress and work hardens to a considerable extent. Woodward [*5*] subsequently investigated in more detail the locations of various isostrain boundaries beneath indentations made in a similar brass and compared his observations with the boundaries predicted by several slip-line field solutions based on a cutting mechanism. He concluded that the discrepancies became so large when the included angle of the indenter exceeded 90° that the slip-line field solutions could no longer reasonably be applied. Hirst and Howse [*6*]

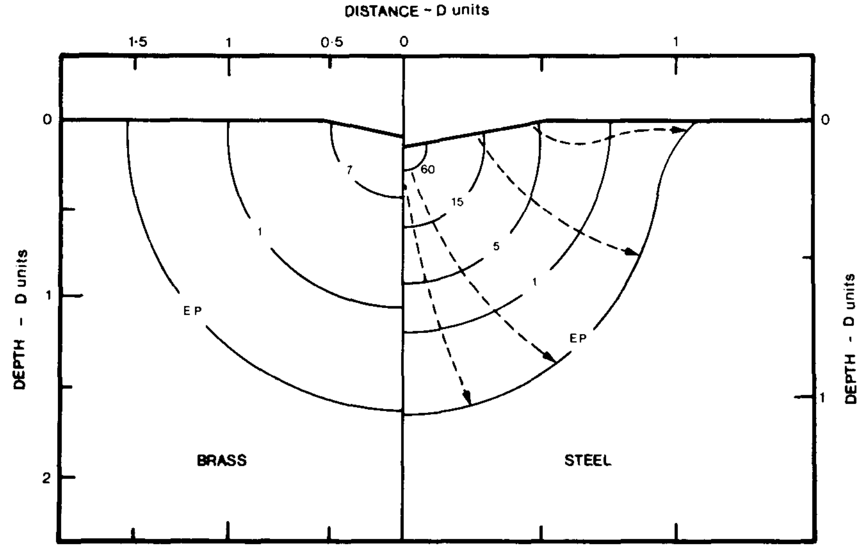

FIG. 2—(Left) *Isostrain boundaries in the deformed zones beneath a Vickers indentation in annealed 70-30 brass. The strain boundaries were determined by the metallographic investigation of a section cut through the indentation* [4]. (Right) *Isostrain boundaries in the deformed zone beneath a Vickers indentation in a cold-worked low-carbon steel. The isostrain boundaries were determined on a split specimen on the parting surface of which a grid had been ruled. The broken lines are displacement trajectories* [3]. *The figure associated with each boundary is the engineering strain as a percentage of compression.*

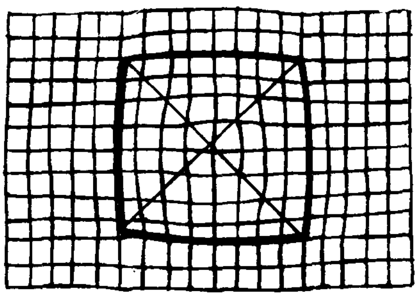

FIG. 3—*A tracing of a square grid which had been ruled on the surface of a cold-worked low-carbon steel and then distorted when a Vickers indentation was made on the surface. The outline of the indentation is indicated by the heavier lines.*

have also confirmed this point for a range of materials and have shown that the transition wedge angle is smaller as the ratio of Young's modulus to the elastic limit of the material becomes smaller.

The evidence that slip-line field analyses, based on the hypothesis that indentation occurs by a cutting mechanism, cannot usefully be applied to indentation hardness testing appears to be conclusive. It is time this type of analysis be dropped from the hardness-testing literature.

Elastic Mechanisms

Shaw and De Salvo [7] have also noted some of these disqualifying deficiencies of the slip-line field solutions and have sought an alternative mechanism of indentation. The model that they advanced was based on two presumed features of unloaded indentations: (1) that the form of the plastic zone beneath the indentation is very similar to a line of constant shear stress derived by a Hertzian analysis of elastic contact of a blunt indenter on a flat surface and (2) that the specimen surface adjacent to the indentation remains flat. Their proposal was as follows:

1. Plastic deformation beneath the indentation occurs during loading in a zone of the form sketched in Fig. 4, the whole plastic zone sinking into the specimen. There is no upward flow adjacent to the indentation.
2. The elastically strained zone surrounding the plastic zone decreases in volume (increases in density) to account for the volume of the indentation. A proviso is that the specimen should extend for at least ten indentation diagonals in all directions from the indentation.
3. A second phase of plastic deformation during unloading occurs in a volume smaller than the loading plastic zone and in the opposite direction. Biaxial residual stresses are thereby induced in planes parallel to the free surface, and these internal stresses maintain the indentation.

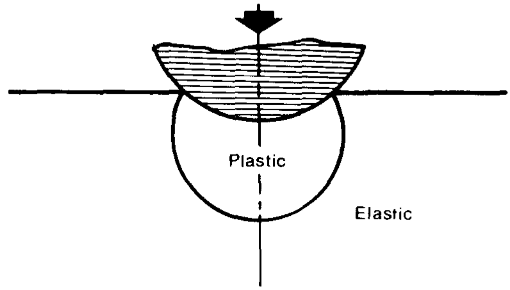

FIG. 4—*Illustration of the elastic mechanism of indentation proposed by Shaw and De Salvo* [7].