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Conference Proceedings: International Workshop on Instrumented Indentation San Diego, CA April 22-23, 1995

Douglas T. Smith, Editor

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[2] Some elements at Boulder, CO 80303.

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Douglas T. Smith, Editor and Workshop Chairman

Ceramics Division
National Institute of Standards and Technology
Gaithersburg, MD 20899-0001

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[a] Extended abstracts for the presentations by John Pethica and Hans-Hermann Behncke were not available at the time of publication.

[b] This abstract covers material presented in both of Dr. Oliver's presentations.

Acknowledgments

The workshop would not have been possible without the financial support of both the Institute for Mechanics and Materials (IMM) at the University of California, San Diego, and the Standard Reference Materials Program (SRMP) at NIST. Thanks go to Dr. Richard Skalak, Julie Bennett and Amy Graves at IMM for funding and administrative support, and to Nancy Trahey at the NIST Standard Reference Materials Program for financial support. Additional funds from Warren Oliver at Nano Instruments, Inc., and Steven Webb at Instron Corporation for student travel were greatly appreciated. Thanks go also to Bob Tucker, Mary Gray and Phyllis Greene of ICMCTF 95, for letting the workshop be part of their program on such short notice. Vanessa L. Williams, of the NIST Ceramics Division, was responsible for the recording and transcribing of the discussions.

Finally, the workshop would not have succeeded without the efforts of an informal planning committee, which met at NIST in August 1994, largely at the members' individual expense, to begin the organizational process. Those committee members included: Richard Colton (NRL), William Gerberich (U. Minnesota), Chris Johnson (NIST), Jean-Luc Loubet (CNRS), Larry Mosiman (MTS Systems), Warren Oliver (Nano Instruments, Inc.), William Ruff (NIST), Michael Swain (CSIRO) and Steve Webb (Instron Corp.).

Executive Summary

1. Introduction

An international workshop was held on April 22-23, 1995, at the Town and Country Hotel in San Diego, to discuss the scientific and standardization issues associated with instrumented indentation, also known as dynamic hardness testing or depth-sensing, ultra-low-load, or nano indentation. The workshop was sponsored jointly by the NIST Standard Reference Materials Program and the Institute for Mechanics and Materials in San Diego, with additional support for student travel from Nano Instruments, Inc., Oak Ridge, TN, and Instron Corporation, Canton, MA, and was part of the program of the 1995 International Conference on Metallurgical Coatings and Thin Films (ICMCTF95). The 96 attendees represented 14 U.S. and 6 foreign companies, 18 U.S. and 7 foreign universities, and 15 national laboratories, including NIST, the National Physical Laboratory (NPL) in England and the Commonwealth Scientific and Industrial Research Organization (CSIRO) in Australia. The two-day program consisted of 22 oral presentations, 22 poster presentations, and several open discussion sessions.

2. Background

Indentation has been used for many years to measure the hardness of materials. The technique involves pushing an indenter tip, typically a sphere, cone or diamond pyramid, into a material under controlled load, then measuring the size of the residual impression. This testing is economical, both in terms of equipment and time, and produces a reliable hardness measurement in macroscopic specimens.

Recently, however, a more sophisticated form of indentation testing, known as "instrumented" or "depth sensing" indentation, has been developed that offers significant advantages over traditional indentation. In an instrumented indentation system, an indenter tip is loaded onto a specimen under computer control of either the load or the displacement or both, and load, displacement and time are recorded continuously throughout the loading–unloading cycle. These data form what is known as a "load-displacement curve," and they contain a wealth of information about the elastic, plastic and time-dependent deformation behavior of the material being tested. Imaging of the residual impression is not necessary, although scanning and transmission electron microscopy and atomic force microscopy can yield useful information about deformation mechanisms. The technique is routinely used to determine mechanical properties from indentations that are sub-micrometer in size, and is considered to be of particular value in

evaluating mechanical properties at high spacial resolutions (*e.g.*, in functionally graded or structurally modulated materials) or for assessing the mechanical properties of materials of limited dimensions (thin films on substrates, nanostructures). When used to make sub-micrometer-scale indentations, the technique is often referred to as ultra-low-load indentation or nanoindentation. Several national standards laboratories, including NIST, NPL and CSIRO, have built their own instrumented indenters, and at least four companies now market commercial machines with varying levels of performance and sophistication. Instrumented indenters are currently used in a number of industrial research laboratories, including 3M, Intel, Kodak, Rockwell, United Technologies and several IBM sites.

Despite the ability of instrumented indentation to make micrometer- and nanometer-scale mechanical properties measurements, use and acceptance of the technique is hampered by a lack of standardization in the field. Not only are there no standard reference materials for use with instrumented indenters, but there also are differences of opinion about exactly how best to analyze load-displacement curves to yield quantities such as hardness and elastic moduli. The purpose of the workshop was to bring together researchers from universities and from standards and other government laboratories with industrial users of the technique to outline areas of agreement and disagreement, and to begin the process of establishing standard, or at least recommended test methods and data analysis techniques, and, ultimately, standard reference materials. The specific goals of the workshop were:

- to assess the current industry use of instrumented indentation, and to hear from industry users about their needs for standardization in the field;
- to provide attendees, particularly those from industry, with detailed, up-to-date information on what material properties are currently being measured with the technique, and how those properties can be extracted from load-displacement curves;
- to discuss the "state of the art" in analytical and finite-element modeling of the indentation process; and
- to discuss standardization issues in instrumented indentation, including a) current standardization efforts within international standards committees, b) machine and tip shape calibration techniques, c) the effects of test parameters (loading rates, dwell times, etc.) on results, and d) possible candidates for standard reference materials.

3. Workshop Program

The workshop ran for two full days and included four sessions of oral presentations and discussion plus an evening poster session. Abstracts from the oral and poster presentations begin on pages 10 and 48, respectively. A brief summary of each session follows.

3.1 Industrial Applications. Discussion Leader: Trevor Page, University of Newcastle.

The workshop began Saturday morning with a set of four talks from industrial users of instrumented indentation. These presentations described how instrumented indentation techniques are currently being used to characterize materials in industrial research and development, quality assurance and process environments, together with problems encountered (both experimentally and in terms of interpretive methods) in producing useful data. Richard White, from IBM's Storage Systems Division, San Jose, began by describing how he is using instrumented indentation to characterize the mechanical properties of the various hard materials used in layered magnetic hard disk drive systems, and how those properties correlate with wear performance. Kevin O'Connor, from Eastman Kodak, Rochester, then described the problems of studying much softer coatings, such as the polymers and gelatins used in photographic materials. These materials often exhibit strong creep and stress relaxation effects. Dr. O'Connor suggested that additional work in the analysis of highly dissipative, viscoelastic systems, particularly layered systems, would be a great help to him in interpreting his indentation data. Harry Fujimoto presented work being done at Intel Corporation on the use of indentation to induce delamination in layered systems as a means of measuring interfacial adhesion. Results on several systems, including a tungsten coating on a softer metal substrate, were discussed. Finally, Clark Cooper, from United Technologies Research Center (UTRC), summarized his experience with instrumented indentation. His comments were of particular interest because UTRC purchased one of the first commercial indenters from Nano Instruments, Inc., and has logged 10 years of use with it.

3.2 Determining Material Properties. Discussion Leader: William Nix, Stanford University.

The purpose of the second session, "Determining Material Properties," was to present a series of talks from an experimental perspective that described which material properties can, in principle, be determined using instrumented indentation, and how they should be determined. The session actually began before lunch, with two overview talks to set the tone for the afternoon. First Warren Oliver, from Nano Instruments, Inc., gave a brief overview of the use

of sharp diamond indenters. He described in general terms the elastic and plastic response of materials to sharp indenters, methods of calculating hardness and Young's modulus, and problems inherent in determining the tip shape and contact area between tip and sample. His talk was followed by one from Michael Swain, CSIRO. Dr. Swain focused primarily on the use of spherical indenters and their advantages over sharp indenters for determining the full stress-strain response of materials. He also described errors that can result if the indenter tip shape is not perfectly spherical and suggested a procedure to correct for this in the analysis.

Six talks were presented in the afternoon, each presenting a detailed discussion of an experimental method for determining a specific material property or set of properties. David Rowcliffe, from the Royal Institute of Technology (KTH) in Stockholm, outlined a method of analyzing Vickers load-displacement curves, supported by three-dimensional finite element analysis, to determine hardness, yield stress, strain hardening and plastic zone size.

Two talks then followed that dealt specifically with coated systems. First, Trevor Page, from the University of Newcastle, gave a detailed account of the problems associated with the use of instrumented indentation to characterize coating/substrate systems and stressed the great care that must be taken to interpret results sensibly. Various strategies for both performing experiments and interpreting data were put forward. Neville Moody, of Sandia National Laboratories, described how his group had combined nanoindentation, continuous scratch testing, and high resolution transmission electron microscopy to study tantalum nitride films and their interface to sapphire.

After a short break, William Gerberich presented work by his group at the University of Minnesota on the yield of materials at very low loads. He showed evidence for initial yield in Fe with mass fraction of Si, $w(Si)$, of 0.03 at a load of 150 μN under a Berkovich diamond indenter.

The final two talks in the session dealt with the determination of time-dependent properties. Jean-Luc Loubet, of the Centre National de la Recherche Scientifique (CNRS) in France, described his current work using a load oscillation technique to study viscoelastic behavior. Howard Poisl, from the University of Arizona, showed, using experiments on amorphous selenium, how indentation creep and indentation strain rate measurements could be related to more conventional creep measurements.

3.3 Poster Session.

A poster session, containing 22 entries, was held Saturday evening. The posters covered a broad range of experimental and theoretical topics, from standardization, calibration and instrumentation issues to analytical analyses of contact stresses in layered systems. The session was well-attended and generated a great deal of animated discussion.

3.4 Modeling of the Indentation Process. Discussion Leader: John Pethica, Univ. of Oxford

The Sunday morning session consisted of four talks on theoretical and modeling aspects of instrumented indentation. It began with a talk by George Pharr, of Rice University, comparing experimental and finite element simulation results on the effect of residual stress in a material on its measured hardness and modulus. Although the experimental results indicated a dependence of both properties on residual stress, the finite element simulation indicated that the effect was not real, but was instead a result of the residual stress changing the amount of pile-up around the contact site, thus causing the actual contact area to differ significantly from the calculated area.

Joost Vlassak then described his work at Stanford University on the effects of elastic anisotropy on the measured "indentation modulus," which is not, in general, the Young's modulus in the direction of indentation for anisotropic materials. In the next talk, Antonios Giannakopoulos presented results of three-dimensional finite element modeling of the sharp indentation process, with and without friction at the contact, and with different degrees of strain hardening.

Finally, Subbiah Ramalingam, from the University of Minnesota, presented an analytical approach to the calculation of elastic stresses that develop in film/substrate systems. The approach permitted an analysis of the stress fields within each material and at the interface between film and substrate.

3.5 Methodology and Standardization. Discussion Leader: Douglas Smith, NIST

The final session combined several aspects of instrumentation and standardization. John Pethica, from the University of Oxford, opened the session with some cautionary remarks about problems that are encountered when performing indentation experiments on length scales approaching atomic dimensions. He cited meniscus and surface adhesion forces as having significant effects on measurements in that regime.

The next two talks dealt with the use of atomic force microscopy (AFM) technology for mechanical property measurement. Steve Hues presented work from NRL on the use of AFM's to make quantitative modulus measurements, by replacing piezoelectric actuators with electrostrictive materials, in an effort to eliminate hysteresis and creep, and by using small glass spheres as indenters, for their more easily characterized tip geometry. Jack Houston, from Sandia, then described a novel force-balanced AFM tip support, developed at Sandia, that is capable of accurately recording attractive tip-substrate interactions through a feedback system that gives the tip support a near-infinite effective stiffness. Warren Oliver returned to finish the instrumentation part of the session with a discussion of techniques for calibrating the shape of sharp indenter tips.

The session then concluded with two talks directly addressing standardization efforts in the instrumented indentation community. First, Stuart Saunders, from NPL, presented the results of research, partly funded by the European Commission, on the "Measurement of Hardness (Mechanical Properties) of Surfaces." The work involved developing machine and tip calibration procedures, as well as conducting round-robin testing with three commercial machines at 14 sites. Hans-Hermann Behncke, from Helmut Fischer Company, described work being done in Germany towards a draft ISO standard (TC 164, WG 3) for a quantity referred to as "Universal Hardness," based on Vickers indentation at depths greater than 3 micrometers. Dr. Behncke also presented results from round robin testing of samples consisting of titanium nitride coatings on steel.

4. Summary

The workshop concluded with an open discussion period at the end of the Sunday afternoon session. Although many topics were addressed, the theme throughout the discussion was to determine to what extent "standardization" of instrumented indentation, in any sense of the word, was desirable, or even possible in the near future. Just the array of names for the technique itself highlights the lack of consensus in the field; it is referred to as *instrumented indentation*, *dynamic hardness testing*, *depth-sensing indentation*, *continuously recording indentation technique*, and more. At very low loads, the terms *nanoindentation*, *ultra-micro-indentation* and *ultra-low-load indentation* are all used. The views of the participants on issues like standard test procedures and standard reference materials ranged from considering them to be essential and long overdue to considering such efforts premature and essentially impossible to implement until the indentation process is better understood. One of the more memorable comments in the discussion came from Bill Nix when he stated that the many different approaches to the problems presented in the

workshop represented a healthy level of scientific activity, and that people should not be overly concerned about the lack of consensus. He felt that we should be pleased not only with the progress that has been made so quickly in this new field, but also with the wide range of applications involved and the amount of sensible scientific criticism that underpins the discussions of how data should be interpreted. His remarks were greeted with applause.

There was however one area where many participants agreed. It was noted that very often when values for hardness, modulus or other quantities obtained by instrumented indentation are reported in the literature, little information is given on the test parameters that were used in the measurements. It would be easier for people to compare their results to those by others in the literature if authors gave, and editors required, a minimum amount of information on technique from those publishing indentation results. That information should include details of specimen preparation (sample mounting, surface roughness), experimental parameters like the loads used, the depths of the indents at those loads, loading rates, and calibration corrections applied (*e.g.*, tip shape, machine compliance and drift), as well as the precise definition of the reported result (*e.g.*, hardness, modulus) and how it was obtained from the load-displacement curves. Trevor Page felt strongly that a representative load-displacement curve should always be published, to allow the reader to assess the overall quality of the data and to help identify any obvious problems arising from conditions such as soft or rough surfaces, thermal drift or crack "pop-in" behavior. If this information is available, people will at least be able to determine whether they can directly compare their results to those from another group.

There was also discussion at several points during the workshop about what materials might make good standard reference materials. An ideal material should be easy to polish reproducibly, as surface finish becomes critical at low loads, and should be as elastically isotropic as possible, so that the precise orientation between sample and indenter geometry is not critical. Single crystal tungsten and several glasses, including pure fused silica and BK-7 glass, have been used in round-robin testing and are possible candidates.

By all comments received by the Chairman during and following the workshop, the participants felt that the workshop had been a very valuable meeting, despite the failure of the group to adopt any firm recommendations for test or analysis procedures. It brought together many of the most experienced people in the field to exchange ideas, and many participants expressed a desire to meet on a more regular basis. Several sessions at 1996 ICMCTF meeting will include talks on instrumented indentation.

Final Agenda

SATURDAY, APRIL 22, 1995

8:00-8:30	Registration, outside California Room.	
Session 1:	**Industrial Applications: California Room** Session Chairman/Discussion Leader: **Trevor Page**, U. Newcastle	
8:30-8:45	Douglas Smith, NIST	Introduction
8:45-9:15	**Richard White**, IBM	"Nanoindentation of Magnetic Storage Materials"
9:15-9:45	**Kevin O'Connor**, Kodak	"The Use of Instrumented Indentation in the Design of Multilayer Coating Structures for Imaging Applications"
9:45-10:00	Break	
10:00-10:30	**Harry Fujimoto**, Intel	"Microindentation for Electronic Thin Films"
10:30-11:00	**Clark Cooper**, UTRC	"The Use of a Nanoindenter I: Case Histories and Lessons Learned"
11:00-11:30	**Warren Oliver**, Nano Instruments	"Measuring Hardness, Modulus, Creep and Fracture Toughness Using Instrumented Indentation Tests"
11:30-12:00	**Mike Swain**, CSIRO	"Simple Procedures for Obtaining and Analysing Force Displacement Data With Small, Spherical-Tipped Indenters"
12:00-1:00	Lunch Break	
Session 2:	**Determining Material Properties: California Room** Session Chairman/Discussion Leader: **William Nix**, Stanford	
1:00-1:30	**David Rowcliffe**, KTH	"Controlled Indentation: A General Approach to Determine Mechanical Properties of Materials"
1:30-2:15	**Trevor Page**, U. Newcastle	"Procedures for the Nanoindentation Testing of Coated Systems"
2:15-2:45	**Neville Moody**, Sandia	"Thin Film Property Measurements Using Nanoindentation and Microscratch Techniques"
2:45-3:00	Break	
3:00-3:30	**Bill Gerberich**, U. Minn	"Multiple Yield Phenomena Under Light Contacts"
3:30-4:00	**Jean-Luc Loubet**, CNRS	"Some Measurements of Creep and Viscoelastic Behavior With the Help of Indentation Tests"
4:00-4:30	**Howard Poisl**, U. Arizona	"Determination of the Relationship Between Indentation and Uniaxial Creep Strain Rates in Amorphous Selenium"
4:30-5:00	Open Discussion	
Session 3:	**Poster Session, 5:00-7:00: Council/Chamber/Cabinet Rooms.** Refreshments.	

SUNDAY, APRIL 23, 1995

Session 4: **Modeling of the Indentation Process: California Room**
Session Chairman/Discussion Leader: **John Pethica**, Oxford

8:30-9:15	**George Pharr**, Rice	"Effects of Residual Stress on the Measurement of Mechanical Properties Using Instrumented Indentation"
9:15-10:00	**Joost Vlassak**, Stanford	"Measuring the Indentation Modulus of Elastically Anisotropic Solids"
10:00-10:15	Break	
10:15-11:00	**A.E. Giannakopoulos**, KTH	"3-D Finite Element Modeling of Sharp Indentations"
11:00-11:45	**S. Ramalingam**, U. Minn	"Analytical Modeling of Stresses in the Film/Substrate System, and Their Significance for Indentation Testing"
11:45-12:00	Open Discussion	
12:00-1:00	Lunch Break	

Session 5: **Methodology and Standardization: California Room**
Session Chair/Discussion Leader: **Douglas Smith**, NIST

1:00-1:15	**John Pethica**, U. Oxford	"Detecting the First Contact - The Role of Adsorbed Molecules" (abstract not available)
1:15-1:45	**Steve Hues**, NRL	"Nanoindentation Using the AFM: Considerations for the Quantitative Measurement of Mechanical Properties"
1:45-2:15	**Jack Houston**, Sandia	"Interfacial Force Microscopy Measurements of the Nanomechanical Properties of Materials"
2:15-2:45	**Warren Oliver**, Nano Instruments	"Tip Geometry Calibration: Is Fused Silica an Effective Standard for Ultra-Low Load Indentation?"
2:45-3:00	Break	
3:00-3:30	**Stuart Saunders**, NPL	"Calibration and Validation of Depth-Sensing Indentation: The European Dimension"
3:30-4:00	**Hans-Hermann Behncke**, Fischer Tech.	"European Efforts Under ISO TC164 for a Dynamic Loading Hardness Test Specification" (abstract not available)
4:00-5:00	Open Discussion and Wrap Up	

Extended Abstracts for Oral Presentations

NANOINDENTATION OF MAGNETIC STORAGE MATERIALS

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Nanoindentation data are presented for three different but interrelated components of the thin film disk magnetic recording system -- the air bearing slider/recording head, the disk substrate, and the sputter deposited thin film disk recording media. Hardness traces across the slider trailing edge (figure 1) demonstrate the hardness of the NiFe, sputtered Al2O3, and the ceramic Al2O3/TiC are 8, 10 and 24-40 GPa, respectively. Lapping under non-optimum, aggressive conditions can lead to significant recession in these components which is directly related to their hardness.

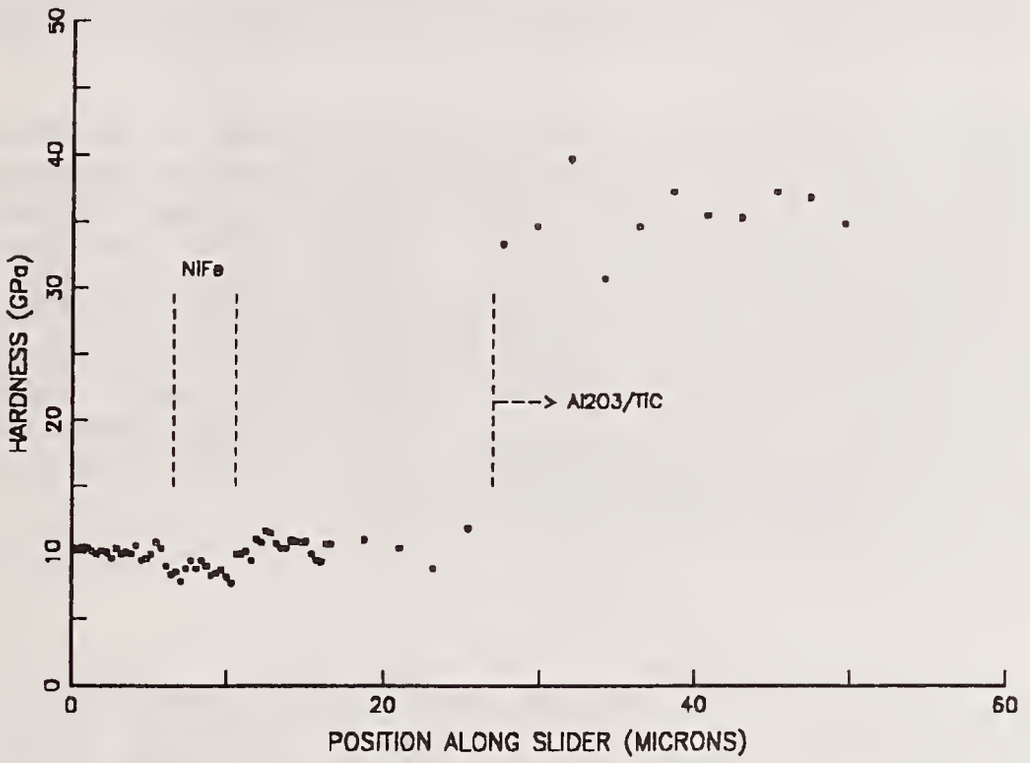

Figure 1. Hardness trace along slider air bearing surface and head.

The hardness and modulus have been measured for a number of alternate substrate materials ranging from AlMg/NiP to glass and glass ceramic. The ability of these substrates to resist damage from slider shock forces generally increases with substrate hardness, although other criteria (fracture toughness and plasticity initiation) can be important. Finally, hardness and modulus data for carbon overcoat films are presented which have been sputtered under various conditions. The process variations lead to variations in hardness, the hardness/modulus (H/E) parameter, and tribological performance in slider/disk testing. The applicability of these mechanical property parameters to the wear degradation is discussed.

THE USE OF INSTRUMENTED INDENTATION IN THE DESIGN OF MULTILAYER COATING STRUCTURES FOR IMAGING APPLICATIONS

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Media used for the capture, storage and display of images are generally multiple-layer structures coated on flexible substrates. Examples include conventional photographic film and paper, photoconductor belts for electrophotography, optical storage disks, and thermal dye-transfer media. The layers are often polymer-based and highly filled with funtional components. In both manufacturing and use, surface contact stresses and resulting physical damage can result in image defects and degraded information content.

Instrumented indentation is a useful technique for determining fundamental material properties such as hardness, modulus, yield, creep and stress relaxation. The sensitivity and depth resolution of modern indentation instruments allows the characterization of thin coatings, and advances in the field hold the promise that meaningful properties may be obtained from multiple-layer coatings as well.

In our work, we take advantage of additional capabilities including a variety of indenter tip geometries and sizes, use of the AC method for continuous measurement of contact stiffness, and tangential force measurement during micro-scratching. Our objective in using indentation techniques is to gain mechanistic understanding of practical multilayer performance features such as adhesion and resistance to scratching and abrasion. In addition to functional layers related to imaging, special-purpose layers such as protective overcoats and adhesion-promoting layers are commonly used. Their effectiveness can be limited by the properties of underlying or adjacent layers. It is critical to treat the design of a multilayer coating as a layered composite structure rather than a collection of individual layers. Strategies such as matching adjacent layer modulus and Poisson's ratio, or creating intentional gradients in properties, are part of the overall design of new imaging media with excellent mechanical performance.

MICROMECHANICS OF THIN FILMS IN ELECTRONIC DEVICES

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Silicon process technology's rapid advance has enabled the design and manufacture of ever increasing more powerful microprocessors and computers. This trend is driven by geometries of features getting smaller. Current manufacturing uses 0.35um technology, but major advances in speed must take place and interconnects are not scaling with feature size. As linewidths decrease aspect ratios of lines increase, surface to volume increases, new methods to pattern, clean and fill are developed, and more interconnect levels are needed. All these affect the stresses in these semiconductor structures and lead to potential cracking and voiding problems.

Comprehensive modelling is necessary. The model should include intrinsic material stresses, interfacial adhesion, time dependent plasticity, crack propagation and fatigue. Integration of the model into process and electrical models will be required to allow interactive modeling capabilities. In order for this model to be useful, it is critical to measure the effects to verify the model and materials parameters for input into the model. Stresses, interface adhesion energy, fracture toughness, and elastic moduli are a few of the parameters required.

Stresses in the materials can be measured by conventional techniques, but there is an appalling lack of tools and techniques to measure the adhesion of thin films. Several techniques are being explored. This paper focuses on microindentation as a technique for differentiating adhesion in certain thin film stacks.

Microindentation was developed to measure the materials properties of thin films. Using the force displacement curve we show results determining adhesion between films in a thin film stack.

Our results show that the force displacement curve can be used not only to determine materials properties but to determine adhesion of the W to metal interface and the oxide to salicide interface. Using a 20 μm spherical indenter tip, the samples were indented to a maximum load of 500mN. When the interface is strong, the force displacement curve is smooth without any discontinuities. In samples with weak W to metal interfaces, a jump in the displacement is observed in the force displacement curve indicating debonding has occurred at the interface.

Optical microscopy shows that the good interface shows an indentation in the W the size of the indenter tip. For the poor interface, a large area around the indentation appears to be debonded. SEM cross-sections confirm that W has debonded from the metal. The Al has been pushed away from the indentation location under the W. This pileup then levers the tungsten until debonding occurs. SEM cross-sections of the good interface samples do not show any debonding and also show the Al pileup under the tungsten.

The force at which there is a jump in the displacement curve, can be correlated to the relative adhesion strength of different interfaces and can be used to develop processes which result in good interfacial strength between the thin film stacks.

Two limitations for microindentation are described. One of the critical issues in indentation is tip wear and tip calibration. Data indicate that the diamond tip in our experiments shows a decrease in the standard deviation and the magnitude of the force required for decohesion as a function of the number of indents. Results also show that different tips with nominally the same radius give different forces for decohesion of the same samples. This is explained by the different shapes of the nominally 20um spherical tips. SEM observations show that one tip was more faceted due to tip wear from many indents that the second tip. This explains the increase in scatter in the data using the same tip and decreasing load for debonding.

Tip wear is also an issue for scratch testing. Results show wear for sequential scratches of the same material, making data analysis very difficult. As we scratched the thin film being tested the force at which the film debonds under the diamond tip increases as a function of the number of scratches. Five scratches were done sequentially and the last two scratches were done the next day showing the same trend of increasing load to debond. Different tips also show very different scratch characteristics as well.

We have shown that indentation can be used to determine the difference in adhesion between thin films. Tip calibration and tip wear have been shown to be issues. This problem of tip calibration needs to be addressed if indentation is to be used as a repeatable technique for both materials properties and adhesion testing. Other limitations are that no quantitative interface energies are determined, the geometry of the test is not submicron, system to system calibration is needed, and the technique doesn't work for all materials systems. We are investigating chip structures and potential micromachining techniques to determine the interface adhesion of semiconductor thin films. This values will be critical for comprehensive finite element method modeling of thermal mechanical stresses and issues in thin film structures.

THE USE OF A NANOINDENTER I: CASE HISTORIES AND LESSONS LEARNED

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This presentation will focus on several salient applications of a Nanoindenter I, the original instrument as made commercially available through Nano Instruments, Inc., at United Technologies Research Center. This instrument was among the first of its kind delivered to a commercial customer almost a decade ago, and it has been a workhorse since its delivery. The representative application examples, described below, have their own requirements and difficulties for the instrumented indentation system, and a discussion of these and the maintenance requirements which accompany a highly utilized instrument is the focus of the presentation. As motivation for the application examples presented, an overview of United Technologies Corporation is given, in which the various operating divisions and the major products manufactured by each are identified. A clear transition over the past ten years from a corporation highly dependent on and supported by defense initiatives to one which realizes far more sales and profit from its commercial/industrial sector is noted.

An overview of the use of a Nanoindenter I for the determination of indentation hardness and modulus is presented, including the experimental procedure required for a determination of the indenter area function, the geometric similitude between Berkovich and Vickers indenters is noted, and the positioning, force, and depth sensing transducers are presented and discussed. Equations are provided to account for machine compliance, allowing the user to determine the reduced elastic modulus, E_r, from the force-displacement data [1]. Briefly presented and discussed are hard, inorganic films, such as TiN deposited by various PVD processes, which appear to exhibit an indentation size effect, and explanations and theories devised to account for this phenomenon are offered [2, 3].

Other application examples are given in which the Nanoindenter is used to characterize the properties of a wide variety of materials. For example, nanoindentation has been applied to thermal-barrier coatings composed of stabilized ZrO_2 to detect the presence of undesirable phases or porosity. The complex, multiaxial stress state caused by the indentation process is shown to lead to the accommodation of significant plastic deformation in quite brittle amorphous and crystalline oxide and fluoride ceramic materials [4]. The use of nanoindentation to determine the properties of a SiC_f-reinforced, lithium aluminosilicate glass composite material, including the interfacial sliding resistance of the SiC fibers and the influence of processing on this measurement. A final application example is provided, in which the Nanoindenter I is used to determine differences in hardness and elastic modulus of diamond films synthesized by hot-filament CVD as a function of methane concentration [5, 6]. A clear, inverse relationship between indentation hardness and methane concentration emerges, as does an imperative for the presence of smooth surfaces for the acquisition of repeatable and low-scatter indentation data.

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INDENTATION TECHNIQUES FOR THE MEASUREMENT OF SEVERAL MECHANICAL PROPERTIES

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Great strides have been made over the past few years in the development of techniques for probing the mechanical properties of materials on the sub-micron scale [1-3]. The advances have been made possible by the development of instruments which continuously measure force and displacement as an indentation is made [4-7]. The indentation load-displacement data thus derived can be used to determine mechanical properties even when the indentations are too small to be imaged conveniently. Since the indentation positioning capability of some of the instruments is also in the sub-micron regime, a means is available by which the mechanical properties of a surface can be mapped with sub-micron resolution. An instrument with such capabilities can be described as a mechanical properties microprobe [8-10].

In this document we will describe some analysis techniques for obtaining the hardness, modulus, indentation strain rate sensitivity and the fracture toughness of brittle materials from the experimental data obtained from mechanical properties microprobes. The first step towards calculating all of these material properties from force-displacement information is some way of determining the area of the contact at any point during the indentation process. One approach, using a Berchovich indenter, that has been documented to be reasonably accurate for a fairly wide variety of materials is the solution of the following set of equations [11].

$$H = \frac{P_{max}}{A}$$

$$S = \frac{2}{\sqrt{p}} E_r \sqrt{A}$$

$$E_r = \left[\frac{\left(1 - \upsilon_s^2\right)}{E_s} + \frac{\left(1 - \upsilon_i^2\right)}{E_i} \right]^{-1}$$

$$h_c = h_t - \varepsilon \frac{P_{max}}{S}$$

$$A = 24.5 h_c^2 + \sum_{i=0}^{7} C_i h_c^{\frac{1}{2^i}}$$

Where:
- H = hardness
- P_{max} = the maximum load that has been applied to the contact
- A = the area of contact under load
- E_r = the reduced modulus
- S = the elastic stiffness of the contact
- E_s = Young's modulus of the sample
- ν_s= Poison's ratio of the sample
- E_i = Young's modulus of the indenter
- ν_i = Poison's ratio of the indenter
- h_t = The displacement of the indenter beyond the point of contact

h_c = the vertical distance between the indenter tip and the perimeter of the indentation under load

ε = a constant equal to approximately 0.72

C_1-C_8 = experimentally determined constants used to describe the geometry of the indenter

A typical experiment involves slowly loading the indenter to some specified load or depth and then unloading it. The experimentally determined parameters are P_{max} , S, and h. The value of S is available only at the point from which the loading is reversed. The stiffness of the contact at P_{max} is obtained from the slope of the unloading curve at peak load. The values of the constants in the area function (C_1-C_8) must have already been determined in separate experiments. This will be discussed bellow. The indenter properties must also be known quantities. Finally, Poison's ratio for the sample and ε must be known or assumed. With these conditions H, E, A, and h_c can be calculated. If the area function is accurately determined, and the effects of the testing system are properly removed from the data, reasonably accurate results can be obtained [11]. There are several limitations of this technique. At high loads, the contact stiffness can approach that of the load frame. When this occurs it becomes difficult to determine the modulus of the sample with accuracy. This problem has only a small effect on the hardness measured as long as the stiffness can be determined within an order of magnitude. The second limitation occurs at very low loads where surface effects (roughness and physical effects of the surface) can make it very difficult to determine the geometry of the tip of the indenter with accuracy. This problem degrades the accuracy both the hardness and the modulus result. This technique should work well for other indenter geometries for which pile up of material around the indenter is not a large effect; however, its accuracy for other indenters has not been documented. Finally, this analysis is only valid for samples that are effectively semi-infinite.

The constants in the area function can be determined using the same set of equations and making the additional assumption that the modulus measured from a semi-infinite homogeneous sample should be constant. The most up to date, detailed procedures for this technique are available elsewhere [12].

Clearly, the stiffness of the contact must be known at the point of loading at which the model described above is to be applied. As suggested above this can be accomplished with an unloading procedure; however, this means that the contact must be unloaded from each point at which the hardness and the modulus are to be calculated. An alternative technique using very small oscillatory excitation of the contact during the loading process together with alternating signal handling techniques can yield the stiffness of the contact continuously as the mean load on the indenter is continuously increased. This leads to the data required to calculate stiffness, hardness and modulus continuously during loading[13].

The indentation strain rate sensitivity can be obtained by performing a different experiment and analysis. One experiment that can be used is to load the indenter at a constant loading rate to a specified load or depth and then reverse the loading. The indentation strain rate is obtained by using the displacement data to determine the rate of change of the displacement divided by the displacement itself at each point in time. The hardness, at the same point in time, can be considered to be proportional to the stress. The slope of a plot that is logarithmic in both the X and Y axis of the hardness verses the indentation strain rate is the indentation strain rate sensitivity. Under certain conditions this quantity has been shown to equal the strain rate sensitivity determined by uniaxial tests[14-15]; however, this is certainly not always the case and the conditions under which the equality exists are not at present understood.

Finally, indentation tests can be used to make a measure of the fracture toughness of brittle materials. The indenter geometry that yields the most accurate results for which the best documentation of those results exists is the corner of a cube [16]. To make these measurements an indentation is made and fracture must occur around the indentation. These fractures should be radial in nature and extend from the corners of the indentation to at least a distance equal to the distance from the center of the indentation to its corner into the surrounding material. If these

conditions are met, microscopy must be used to measure the length of the cracks and the following equation can be applied.

$$K_c = 0.04\left(\frac{E}{H}\right)^{1/2}\left(\frac{P_{max}}{C^{3/2}}\right)$$

Where: K_c = the fracture toughness
C= the crack length

This technique will be effected by crystalline anisotropy of the sample, residual stresses, defects in the material and possibly the environment.

The techniques discussed above are useful; however, they all suffer loss of accuracy under certain conditions. Although some of these conditions are mentioned above this document is not intended to be a critique of the techniques. The techniques should all be considered "under development".

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SIMPLE PROCEDURES FOR OBTAINING AND ANALYSING FORCE DISPLACEMENT DATA WITH SMALL SPHERICALLY TIPPED INDENTERS

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Force-displacement data of high precision are now readily obtainable with a number of micro-mechanical probe systems. The emphasis to date has been on nominally pointed indenters, although spherically tipped indenters have distinct advantages. These advantages include a more well defined and analytical response during the initial elastic loading of the material. The approach also enables the entire unloading response for an elastic-plastic contact to be predicted, rather than the usual asymptotic response as favored for pointed indenters. A material's response to spherical indentation has many similarities with traditional mechanical testing of materials in that the initial elastic response precedes the material's non-linear behaviour. Often, however, the transition from one regime to the other is asymptotic and difficult to distinguish. Another inherent problem with the interpretation of all monotonic loading and unloading force-displacement data is that of partitioning the elastic and inelastic contributions to the displacement at a specific force. Such partitioning usually relies on the assumption of constant elastic modulus throughout the test, with the modulus determined during the final unloading. A simple procedure to overcome this limitation with spherical tipped indenters is via partial unload steps throughout the penetration to maximum load. This procedure enables the contact pressure and modulus to be determined independently throughout the excursion to maximum load. Using the simple relationships proposed by Tabor (1953), and more recently rigorously confirmed by Hill et al (1989), the materials stress-strain response may be determined. It is also possible to further extend the analysis to determine work hardening response and also to incorporate "pile-up" and "sinking-in" about the impression. As with all analysis of indentation force-displacement data, interpretation of spherical contact requires a detailed knowledge of the indenter tip shape. Examples of procedures used for such characterization and even their extension to nominally sharp Berkovich indenters are discussed. Application of spherically tipped indenters to the investigation of the stress-strain response of polymers, metals and ceramics will also be shown and, in the case of polymers and metals, compared with conventional stress-strain curves. Issues involved with the extension of the spherical indentation approach to materials that fracture and to very thin films will also be discussed.

CONTROLLED INDENTATION: A GENERAL APPROACH TO DETERMINE MECHANICAL PROPERTIES OF MATERIALS

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In recent years, depth sensing indentation techniques have been developed and widely used, with both Vickers and Berkovich geometries (nano-indenter). Regardless of the shape of the indenter the most important result from such a test is the indentation load-depth (P-h) curve. Various mechanical properties, not only hardness, but also modulus, yielding stress, and strain hardening must affect the form of the penetration curve. Thus it should be possible to derive these properties for any material provided an appropriate analysis of the penetration curve is available. This paper presents a general methodology to analyse the loading cycle of experimentally-obtained indentation P-h curves for Vickers indentation. The approach uses results from a recent 3-D FEM analysis to derive various mechanical properties, including hardness, yielding stress, strain hardening and plastic zone size. The validity of the approach can be tested by reconstructing the P-h curves from these data.

The method is based on two assumptions which were confirmed by the 3D-FEM analysis: (1) there is no elastic recovery from the diagonal of the plastic imprint after unloading, therefore the generally-defined hardness value is equal to the average pressure during loading; (2) the average pressure during loading, in this case, the hardness, is a constant during indentation loading, hence the true projected contact area can be found from the load and hardness values. An important aspect is that surface displacement occurs during indentation and must be accounted for in the analysis. This is done with a surface displacement factor which relates the geometrical contact area to the true contact area under load. So far, various ceramics have been investigated and it is found that, during loading, indentation is accompanied by different amounts of sinking-in just outside the contact boundary. The study of the surface displacement suggests, as a first approximation, that the Mises elastoplastic solution needs to be re-scaled by the surface displacement factor in order to explain the results of indentation behaviour on ceramic materials.

Values for compressive plastic properties derived in this way agree well with the limited data available in the literature. Recent work suggests that this approach can be extended to include other effects such as cracking or material densification during indentation. Ultimately it should be possible to obtain a complete characterisation of all of the important compressive plastic and elastic properties of materials from an analysis of a single indentation cycle.

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PROCEDURES FOR THE NANOINDENTATION TESTING OF COATED SYSTEMS

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Indentation testing methods where load, depth and time are continuously recorded (eg. nanoindentation) have demonstrated considerable ability for studying the near-surface mechanical properties of materials[eg1-2]. Such continuously recording indentation techniques (CRIT) have a unique ability to probe mechanical properties at shallow penetration depths and, consequently, appear particularly well-suited to the study of surface-engineered and coated systems [eg3-8]. Indeed, for thin film coated systems, they offer one of the few possible means of making mechanical property measurements at depths sufficiently shallow to allow the mechanical response of the films alone to be studied with minimal contribution from the substrate. In addition, at increasing displacements, the effects of film and substrate can be measured together, and thus the behaviour of the coated system investigated over ranges of contact-affected volumes and stresses. Further, the technique offers the possibility of experimentally determining properties which may be very difficult to ascertain in other ways - eg. the elastic moduli of coatings in the microstructural and residual stress states in which they are used in various coated systems[eg3,9,10]. Furthermore, since software control allows many indentations to readily be made on quite extensive sample areas, CRIT are also ideally suited to assessing the point-to-point reproducibility of coated systems though, as yet, this seems an under-exploited application.

At the outset, we have to face a central underlying problem which is that there are currently few soundly-based mechanistic models for predicting 'how coated systems work'. Indeed, the pace of technological demand for surface engineering has led to the development of many coated systems where expectations of properties and behaviour are based upon experience and empiricism. Thus, in the absence of well-founded models, it can be difficult both to plan how best to utilise CRIT to probe the properties of coated systems - and to interpret the resultant data - especially since many intuitive models simply fail to work. For example, the enhanced hardness of coated systems can be strongly influenced by elastic bending and flexure of the coating rather than its own hardness or indentation plasticity; thus typical 'law of mixtures' hardness models are inappropriate. Similar comments can also apply to understanding the effective elastic modulus of coated systems[eg9] in various loading modes and even the effect of substrate properties on system behaviour[eg3,10]. Thus, before we can properly either predict or interpret the nanoindentation response of coated materials, we need to develop good models of systems behaviour based on understanding not only the differing deformation mechanisms controlling behaviour in various systems, but also the changing balance between dominant deformation mechanisms over a range of contact scales and stresses. Arguably, this is the single

largest factor limiting current progress with applying CRIT techniques to the study of coated systems.

On the positive side, there is certainly a wealth of information which, *currently, we can access* using nanoindentation. This presentation will highlight these areas which prescribe our present approaches to, and procedures for, the CRIT testing of coated systems as follows.

(a) The general use of the 'fingerprinting' approach[3] whereby the responses of the coated system can be compared to those of the substrate alone (as a function of indentation scale etc). This underlines the importance of 'knowing your substrate' and recognising its profound influence on coating structure and properties[10-13].

(b) The examination of load-displacement curves for signs of behaviour peculiar to the coated system (eg. the 'pop-in' (displacement discontinuity) behaviour often associated with fracture and delamination of the coating (or even nucleation of plasticity in the underlying substrate))[3,5].

(c) By making quantitative comparisons between the load displacement data for both the coated system and the substrate alone, quantifying the enhancement provided by the coating to the system (eg. comparing δ_{max} δ_{res}, stiffnesses, plastic work expended etc)[3].

(d) Performing (a)-(c) over a range of indenter displacements (δ). Since experience suggests that many coatings flex elastically up to displacements approximately equal to the coating thickness (t), it is suggested that at least three series of tests are performed (namely with $\delta << t$ (the regime where the coating is mainly flexing to generate membrane stress[13]), with $\delta \simeq t$ (where the onset of coating fracture may occur and the substrate is playing a significant role[12]) and with $\delta >> t$ (where the sections of broken coating may simply act as blunt extensions to the indenter-profile)).

(e) The essential use of microscopy techniques to study both the initial states of test surfaces and post facto evidence for deformation mechanisms. The preliminary assessment is necessary to ensure that the surfaces are both flat enough for examination (eg. only exhibit topography small in scale compared to the indentation sizes) and to identify features (eg. metal globules in some coated systems) which could lead to artifacts in the data. The post facto observations are critical in determining the occurrence and extent of, for example, any coating fracture or delamination, evidence of substrate pile-up beneath the coating etc[3].

(f) Considering the use of both cross section and taper section techniques as well as testing coatings as stand-alone membranes with the substrate removed[7,8,14].

(g) Assessing both the possible role of the substrate in supporting the contact load[15] and using quantitative modelling techniques - especially those based on a proper understanding of deformation and mechanisms - to predict and interpret the load-displacement data[12,13,16].

(h) Using indenters of different geometry to investigate differing aspects of contact response - eg. using hemispherical indenters to quantitatively explore elastic properties[eg17].

There are also areas for future development and assessment and these include

- The development of better models for predicting load-displacement response[eg13,16]. In particular the shapes of unloading curves for many coated systems are not always amenable to the analytical approaches often used for calculating the stiffnesses and elastic moduli of simpler systems[18,19]. In these cases, the generally more curved appearance of the unloading curves is presumed due to the inhomogeneous nature of elastic recovery in these systems and, in these cases, conventional analysis must either be approached with caution or alternatives sought[eg16].

- Addressing the problem of deconvoluting properties as a 'function of depth', especially in those cases where the coating is simply bent and flexed into the substrate under the indenter contact. Even for isotropic specimens simply penetrated by the indenter, different portions of the indenter surface are sampling properties of the specimen at various depths. Thus, at best the load-displacement data can only supply some complex summation of all properties experienced at all the depths sampled by the indenter profile. This problem is even more complex with either inhomogenous samples or with coatings which flex and bend to follow the contact profile[3].

- Furthering an understanding of the work of indentation approach[eg20] (conveniently provided by the area under the load-displacement curve) to quantitatively describe the enhancements provided by the coating to the system[eg21]. Much of the problem here is concerned not with measuring the reversible and irreversible components of the work done, but on establishing the sample volume in which the work has been dissipated by deformation. It should be noted that, since some of the earliest attempts to analyse indentation behaviour[eg22], the equivalence of indentation hardness as both a contact pressure and work done per unit volume has been appreciated but under-used.

- Assessing how best to characterise multi-layer systems where the full system performance may only be experienced when deformation spans scales larger compared to the microstructural scale or characteristic length.

- Undertaking contact fatigue and creep experiments, especially in controlled environments[eg4].

In order to obtain useful property data, many of these concepts need approaching with caution but, given the right experimental approaches, continuously recording load-displacement instruments can provide a wealth of unique information regarding coated systems. However, more informed and imaginative use of nanoindentation techniques is required if we are to learn more about coated systems and escape from the self-imposed shackles of simply trying to assess their hardnesses.

Finally, it must be emphasised that CRIT do not exist in isolation but form part of the spectrum of techniques (eg. bend tests, adhesion tests etc) available for studying different aspects of coating performance[eg23,24].

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DETERMINATION OF THIN FILM PROPERTIES USING NANOINDENTATION AND MICROSCRATCH TECHNIQUES

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The reliability of many microelectronic thin film components is governed by the composition and structure of the film and the film-substrate interface. Thin tantalum nitride films for resistor applications are of particular interest, for miniaturization and high heat generation increase the probability that changes in composition and structure over long service lives will degrade properties. However, the lack of small volume test techniques have precluded quantifying the effects of these changes on properties. We have therefore combined nanoindentation, continuous microscratch testing, and high resolution transmission electron microscopy to determine how film and film-substrate interface structure affects properties in thin tantalum nitride films.

The thin tantalum nitride films employed in this study were sputter deposited on $(11\bar{2}0)$ single crystal sapphire substrates to nominal thicknesses of 100 and 600 nm. The elastic modulus of each film was determined as a function of depth with a Nano Indenter™ II using a Berkovich indenter. Each test included a 60 s hold at maximum load to minimize time-dependent effects on property measurements, and a 60 s hold during the unloading segment to determine thermal drift. The plastic contact area for each indentation in the 600-nm-thick as-sputtered film was measured directly in a JEOL 840 SEM. The plastic contact areas in the 100-nm-thick films were calculated using an indenter tip shape function determined from fused silica and verified with values measured in the thick tantalum nitride film. Stiffness and elastic modulus were determined from the slopes of each unloading curve as described by Oliver and Pharr.[1] Fracture toughness was then determined on a Nano Indenter™ II and a microindentation test system by IBM, configured to control normal and lateral displacements. These tests employed a conical diamond indenter with a 1 μm tip radius and a 90° included angle that was simultaneously driven into the films at a rate of 15 nm/s and across the films at a rate of 0.5 μm/s until a portion of the film spalled from the substrate. The work of adhesion and interfacial fracture toughness values of the thin Ta_2N films were determined from the data using the elasticity based approach of Venkataraman et al.[2].

The elastic modulus of the as-sputtered thin tantalum nitride films varied from area to area on each wafer and between wafers from different deposition runs. For the 600-nm-thick films, the elastic modulus ranged from 350 to 450 GPa. The elastic modulus of the 100-nm-thick film was slightly higher at almost 500 GPa, with a smaller variation in values. With Poisson's ratio set equal to the value for tantalum at 0.35, the work of adhesion and fracture toughness of both the 100 and 600-nm-thick films were near 0.6 J/m^2 and 0.5 $MPa \cdot m^{1/2}$ respectively, with one sigma approaching 50 percent.[3] The variation in elastic modulus of the 600-nm-thick films is consistent with a highly variable columnar structure that forms near the film surface, while the less variable values observed in the 100-nm-thick films are consistent with the somewhat uniform

amorphous structure observed. The variability in adhesion values can be directly attributed to the variability in the several-monolayer-thick zone of damage that characterizes the film-substrate interface.

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MULTIPLE YIELD PHENOMENA UNDER LIGHT CONTACTS

W.W. Gerberich, J. Nelson, E. Lilleodden and S. Venkataraman

Yielding in small volumes under sharp contacts has been observed for about 25 years with the first direct observations in gold by Gane[1] using direct transmission electron microscopy of tip deformation. Later Pethica and Tabor[2] used resistivity techniques. More recently, such small volume yielding has been observed by others using depth sensing instruments. Notably, Pharr and Oliver[3] showed indirect evidence in silver, Page, *et al.*[4] directly imaged dislocation loops in Al_2O_3 after nanometer level indentations and Tangyunyong, *et al.*[5] used an interfacial force microscope to monitor plastic deformation in gold as produced by μN level forces.

Probably the most convincing evidence of a yield point due to dislocation nucleation is Page, *et al.'s* since they show a 10 nm "pop-in" displacement onto $\{10\bar{1}2\}$ sapphire and the approximate 10 dislocation loops associated with it. While we had previously shown dislocation loops in Fe-3wt%Si[6] and yield "pop-ins" at similar loads to the Al_2O_3 in both Fe-3wt%Si[7] and GaAs,[8] in all of these experiments there was additional evidence that deformation had occurred prior to the yield point. The present result sought to find if there was even a lower load level yield point, particularly in Fe-3wt%Si, that might be associated with the initial yield. In fact very clear evidence of two yield points was found, these being shown in Figures 1 and 2. The first yield point occurred near 150 μN in over 10 contacts and the second yield point occurred near 1500 μN for many more than 10 repeat experiments.[9]

Using sharp diamond tips with radii in the range of 60 to 200 nm, we saw no evidence of either phase transformation (densification) or microcracking which could be associated with these yield phenomena. Clearly, the cavities produced cannot be associated with surface diffusion considering the rapidity with which the displacement excursions occurred at room temperature. Furthermore, as explained in more detail elsewhere[8] time dependent indentation and relaxation in GaAs can best be explained by a dislocation nucleation mechanism.

Based upon previous information[6] we propose that oxide breakdown is the most likely process for the second yield point. We do recognize that with sharp indenters and the

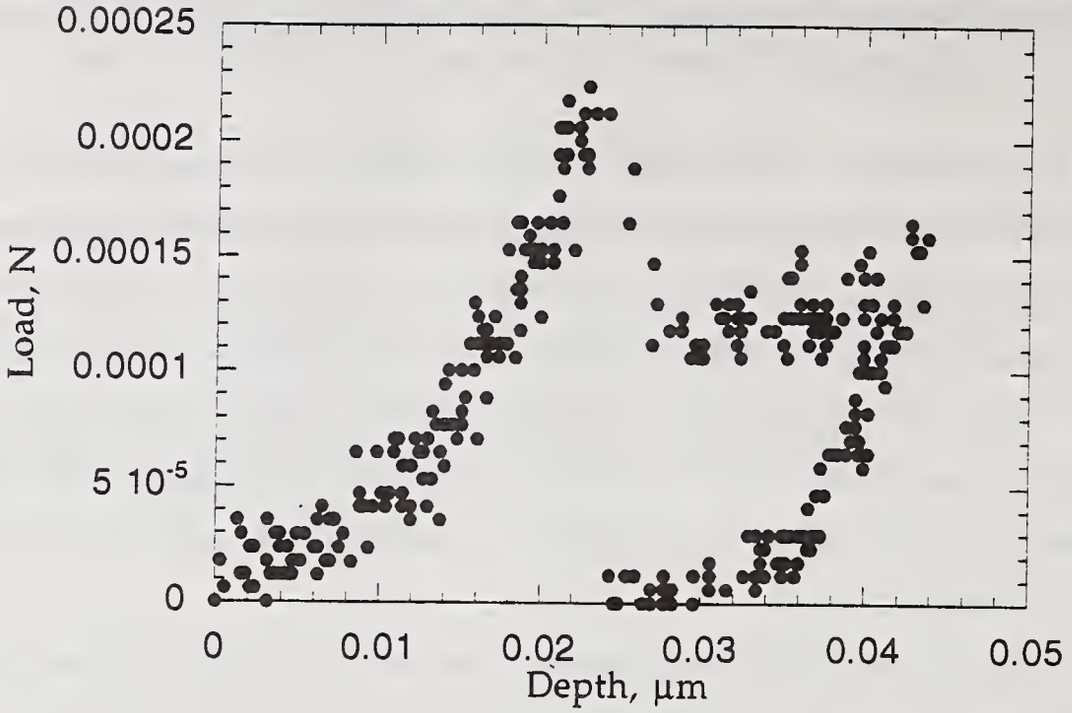

Figure 1: Initial yield point near 150 μN in Fe-3wt%Si ⟨100⟩ loaded at 1.5 nm/s. Hertzian behavior is followed for the initial loading portion to 0.023 μm depth.

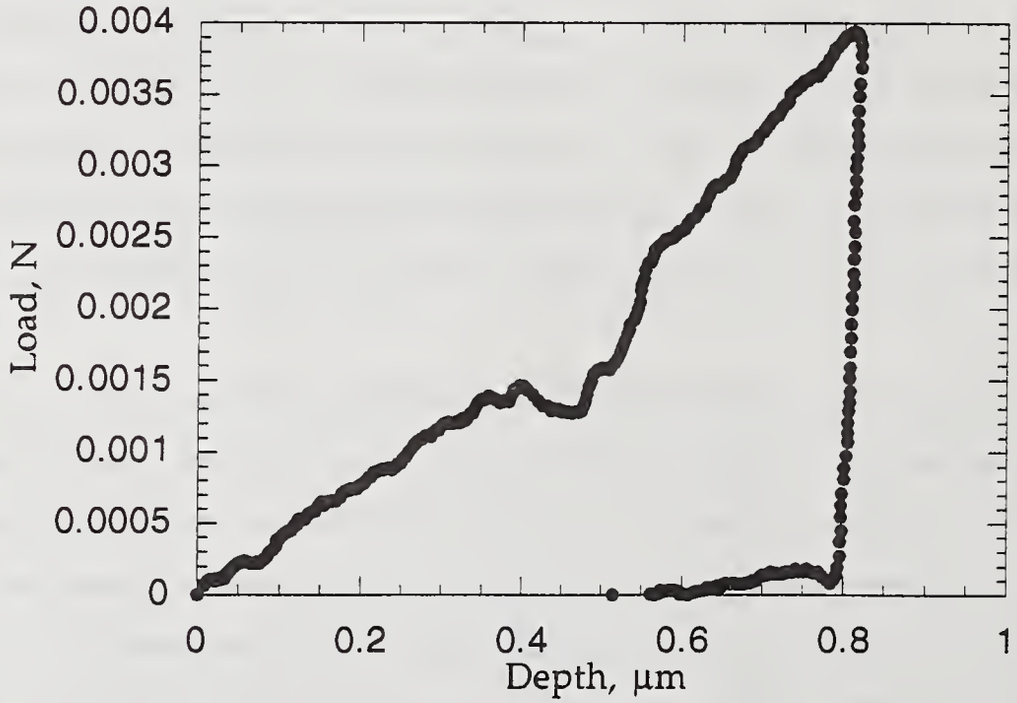

Figure 2: Second yield point near 1500 μN in Fe-3wt%Si ⟨100⟩ loaded at 1.5 nm/s. Curve irregularities are partially due to the slow rate of loading.

changes in the stress distribution associated with the elastic bending of an elastic film on a ductile substrate, that the second yield may be associated with a shear stress shift nucleating yield at either different locations or on different slip systems under the contact. Irrespective of the reason for the second yield, we propose the oxide film to have major effects on the initiation of yield in metallic systems. That is, the first dislocations are proposed to nucleate at a load under a cylindrical plane strain contact, as given to first order by

$$P_{UYP} = \frac{E^*}{R}\left\{\left(\frac{h_{fo}^2}{h_{fo} - \frac{\mu_s R}{4E^*}}\right)^2 - h_{fo}^2\right\}^{3/2}$$

where E^* is the reduced modulus, R is the tip radius, h_{fo} is the oxide layer thickness and μ_s is the shear modulus of the plane that is involved in tip nucleation of the first dislocation. While there are a number of simplifying assumptions here that relegate image force contributions in Fe-3wt%Si to secondary effects, the major variables of moduli film thickness and tip radius all should have major effects on the nucleation condition. Data demonstrate yield points in the range of 70 to 300 μN, the scatter being attributed to small variations in the oxide film.

Upon nucleating the first dislocation, others follow until an arrest occurs associated with an equilibrium of forces between the indenter tip and the dislocation back force. To first order, this gives a good agreement with observed forces at arrest when utilizing the number of dislocations estimated from residual displacement excursions at yield. This is given by

$$P_{LYP} \simeq \frac{3\mu_s bNa}{4f(\alpha)}$$

where μ_s is the shear modulus on the slip plane, N s the number of dislocations, b is the burgers vector, a is the contact radius at the lower yield point and $f(\alpha)$ is obtained form the shear stress distribution. While this fit the Fe-3wt%Si data, one can also show that it comes close to reproducing Page, *et al.*'s[4] average force of 1500 μN for nucleating dislocations in $\langle 10\bar{1}2\rangle$ Al_2O_3. Note here in this load controlled instrument that the force for nucleating dislocations is also the force at arrest. There is a caveat here in that $f(\alpha)$ is smaller in the Al_2O_3 case compared to Fe-3wt%Si, consistent with the fact that dislocation loops less than 200 nm in diameter were observed for Al_2O_3.

FUTURE DIRECTIONS

We believe that a number of complementary instruments currently exist. These provide microindentations which scale from the mN to the kN regime, nanoindentation types which scale from μN to N levels and interfacial force or atomic force types which can scale from the nN to mN regime. Note that each of these may have a 10^6 range of force capability with approximately a three order of magnitude overlap. While not all instruments have the ranges given above, there is sufficient overlap in each case with other instruments that scale effects can be appropriately examined. It is clear to us that there are scale effects with near surface contacts at the atomic scale, as measured by STM, IFM or AFM, being best modeled by molecular dynamics; small contacts at the mezoscopic scale as measured by modified AFM, IFM or nanoindenters being best modeled by discretized dislocations (in crystals); and large contacts at the continuum scale as measured by depth-sensing microindenters being modeled by FEM. We do not mean the above pairings to be exclusive as much can be learned by using microindenters at the Newton level using large spherical tips. For example, dislocation modeling may be best at some mezoscopic scales by using a combination of many spherical tip indenters. Only by appropriate pairing of experimental instruments, theoretical modeling and understanding of scale will the power of instrumented indentation bring broad understanding and therefore usage by both the academic and industrial communities.

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SOME MEASUREMENTS OF VISCOELASTIC PROPERTIES WITH THE HELP OF NANOINDENTATION

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The viscoelastic behavior of surfaces is fundamental in fields dealing with interfaces, such as adhesion, polymer blending, composite materials and tribology. As a result, there has been considerable recent effort devoted to investigating the viscoelastic properties of thin films using surface force apparati (SFA) [1,2] and atomic force microscopy (AFM) [3,4]. Using a nanoindentation instrument, the Nano Indenter® II, we present results of the first measurements of the linear viscoelastic properties of a polymer obtained with of a trigonal diamond tip (Berkovich indenter). Small, oscillatory motions of the indenter yield a shear field in the material from which the complex elastic modulus (G*) of the polymer can be deduced. The main objective of this presentation is to report how this was accomplished. The polymer tested was a unvulcanized natural rubber, an amorphous polyisoprene of molecular weight 800,000. It has the chemical structure poly-*cis* 1,4-isoprene.

If a small, constant harmonic load oscillation is imposed on the specimen during the indentation process and displacement response is measured at the same frequency (f), it is possible to determine the experimental dynamic response of the system. The amplitude and phase relationship between the excitation and the response are a composite of the response of the apparatus and the material being indented. To separate the two, an accurate dynamic model for the apparatus must be determined. Such a model has been established for the Nano Indenter® II [5] but some improvements in the method and the mechanical modeling are still needed. Having removed the response of the apparatus, the remaining dynamic response can be divided into two parts [6]. The portion of the displacement signal which is "in phase" with the excitation is the elastic response of the contact. It is related to the stiffness (S) of the contact and to the elastic modulus, or storage modulus (G'), of the material. The "out of phase" portion characterizes the energy being absorbed by the material, i.e. the damping ($C\omega$) of the contact, and thus the loss modulus (G") of the material where ω is the angular frequency or pulsation of the harmonic oscillation of frequency

f ($\omega=2\pi f$). We show that both the "in phase" and the "out of phase" portions of the response scale according to the square root of the contact area. Using the *Principe de correspondance* of linear viscoelastic theory we propose that the storage modulus G' and the loss modulus G" can be written as:

$$G' = \frac{\sqrt{\pi}}{6}\frac{S}{\sqrt{A}}$$

$$G'' = \frac{\sqrt{\pi}}{6}\frac{C\omega}{\sqrt{A}}$$

where A is the contact area.

Two different experiments were conducted at room temperature (T=23.9°C) to test the proposed method. The first one was performed at constant area of contact and different frequencies. Small harmonic oscillations of the load were imposed while the area of contact was held constant. The ratio of the amplitude of each force excitation and the amplitude of the resulting harmonic displacement were measured. The phase angle between the force and the displacement were measured simultanously. The range of the frequencies tested was 50 mHz to 50 Hz. This gives the viscoelastic properties as a function of frequency, as they are determined from the complex mechanical modulus G*=G'+iG" (see figure 1).

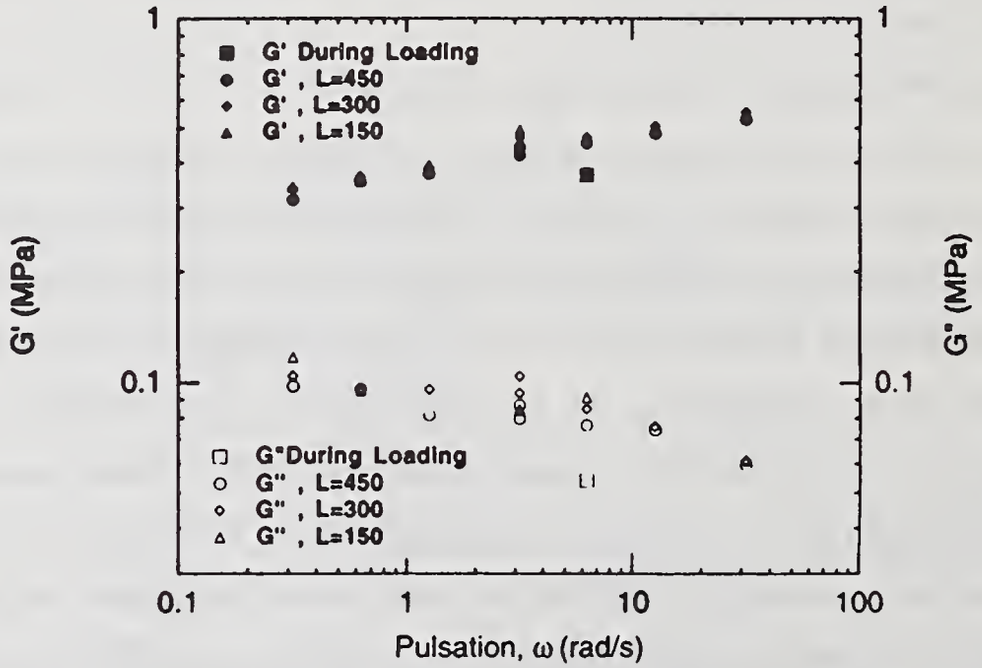

Figure 1: Changes of the storage modulus (G') and the loss modulus (G") as a function of frequency at room temperature (T=23.9°C) for unvulcanized natural rubber, poly-*cis* 1,4-isoprene (Mw=800,000) as measured in a microscopic contact between the material and a diamond tip, Berkovich indenter.

The second set of experiments was performed at a constant frequency of about 1 Hz and different indentation depths. A fixed frequency for the harmonic oscillations of the load was imposed and the ratio and the phase angle between the harmonic force and the resulting harmonic displacement were measured as a function of increasing indentation depth (with a corresponding increase of the contact area). The indentation velocity was 10 nm/s. This gives the stiffness (S) and the damping coefficient (C) of the contact as a function of the indentation depth (see figure 2). It was found that both (S) and (C) vary linearly with the indentation depth. The slopes of these straight lines are respectively proportional to the storage and loss modulus of the indented material, as can be easily deduced from equations (1) and (2).

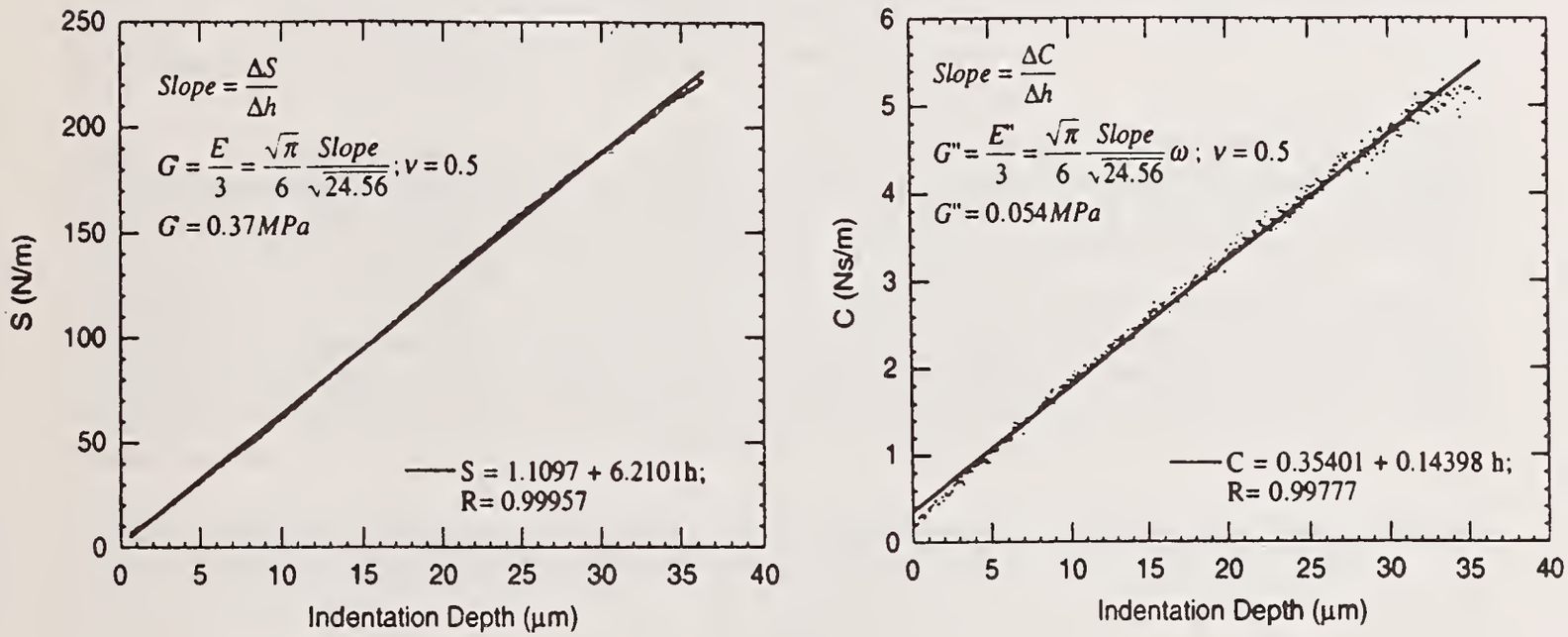

Figure 2: Changes of the stiffness (S) and the damping coefficient (C) of the contact between a Berkovich diamond and an amorphous polyisoprene with indentation depth. The slopes of the curves are proportional to the storage (G') and loss modulus (G") of the material as shown in the insert formulas. The measured values of the storage and loss modulus (G'=0.37 MPa and G"=0.054 MPa) at 1Hz are in good agreement with data in the literature [7].

In conclusion, it is shown for the first time that it is possible to measure the viscoelastic behavior of materials such as solid polymers with the help of nanoindentation techniques. Accurate measurements of the storage modulus and the loss modulus can be obtained using this method.

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INDENTATION CREEP IN AMORPHOUS SELENIUM

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Indentation techniques can be used to obtain time-dependent, or indentation creep, properties of materials. Since the development of instrumented indentation techniques, it is now easier to obtain indentation creep data as the indentation size is directly obtainable from the displacement of the indenter, obviating the need to stop the experiment in order to measure the indentation size. Using instrumented indentation it has been found that many materials, even ceramics, creep at temperatures well below half of their melting temperature.

While previous studies[1-6] have indicated that the activation energy and stress exponents for indentation creep are similar to conventional creep, there is no accepted, validated method for analyzing indentation creep data. The power-law creep equation

$$\dot{\varepsilon} = k\sigma^n \tag{1}$$

where $\dot{\varepsilon}$ is strain rate, σ is stress, n is the stress exponent and k is a constant for a given material/temperature, has been used by many researchers to describe the time-dependent properties of materials during indentation creep. Values for the stress on the material under the indenter during indentation creep can be obtained from the well known relationship between stress and hardness, $H = c_1\sigma$, where c_1 is a constant of about 3 for most material/indenter systems. However, the strain rate of the material under the indenter during the indentation creep test has not been accurately determined. It would be useful to extend the concept of representative strain under an indenter to define a representative strain rate for the material undergoing indentation creep. The representative strain rate could then be related to the indentation strain rate as $\dot{\varepsilon}_{rep} = c_2\dot{\varepsilon}_I$.

Newtonian viscous materials have a constant stress exponent of 1.0, irrespective of the stress and strain rate applied. The relationship between stress and strain rate for a viscous material is

$$\dot{\varepsilon} = \frac{\sigma}{3\eta} \tag{2}$$

which is similar to the power law creep equation with a stress exponent of 1.0. Using the relationships shown above, the coefficient relating indentation strain rate to the representative strain rate in a viscous material can be found from the following equation

$$c_2 = \frac{H}{3c_1\eta\dot{\varepsilon}_I} \tag{3}$$

Indentation creep data on amorphous selenium at temperatures of 32.1 and 34.3 °C with a Berkovich indenter have been obtained.[7] Creep data were obtained from a step load procedure, in which a load of 10 mN was applied to the indenter in less than a second and held constant for 1 hour. Load and displacement as a function of time were monitored, from which hardness and indentation strain rate were calculated. Hardness was calculated as the load divided by the projected area of the indentation, while indentation strain rate was calculated as

$$\dot{\varepsilon}_I = \frac{1}{h}\frac{dh}{dt} \tag{4}$$

where h is the current displacement. Indentation strain rate versus hardness curves for these procedures are shown in Figure 1. It can be seen that there are two regions to these curves - a linear region at low hardness levels, and a non-linear region at higher hardness values. The non-

linear region is due to two effects. When the load is suddenly applied to the indenter, the deformation of the material is initially dominated by the elastic and delayed elastic response of the material. In addition, amorphous selenium exhibits non-Newtonian viscous flow for high stress, or hardness, levels.[8] Once deformation is occurring only due to Newtonian viscous flow, the log $\dot{\varepsilon}_I$ - log H curves become linear with a slope of 1.0. Using experimental values of hardness and indentation strain rate, c_1 equal to 3, and published viscosity values,[8,9] Equation 3 results in a coefficient c_2 of 0.09, or

$$\dot{\varepsilon}_{rep} = 0.09\dot{\varepsilon}_I \tag{5}$$

The coefficient relating indentation strain rate to strain rate should vary with the power-law creep exponent as $x^{1/n}$.[10] Therefore, as n increases the representative strain rate will approach the indentation strain rate.

Equation 3 can now be rewritten to obtain the viscosity as a function of hardness and indentation strain rate during a constant load indentation creep test, or

$$\eta = \frac{H}{9(0.09)\dot{\varepsilon}_I} \tag{6}$$

The constant load indentation creep test has been shown to be characterized with a constant strain of approximately 10%.[4] Therefore, the constant load indentation creep procedure is equivalent to a stress relaxation test. The relaxation function for a stress relaxation test can be defined as the instantaneous stress divided by the initial stress applied. For an instrumented indentation test, this can be expressed as

$$\Psi = \frac{H(t)}{H(0)} \tag{7}$$

where $H(t)$ is the hardness at a given time and $H(0)$ is the initial hardness. The relaxation function of linear viscous materials can be well described by

$$\Psi = \exp\left(-\left[\frac{t}{\tau}\right]^b\right) \tag{8}$$

where τ is the relaxation time for the material/temperature and b is a constant usually between 1/3 and 1/2.[11] Equation 8 has been fit to the experimental relaxation function, as defined in Equation 7, for the 34.3 °C data with τ and b as adjustable parameters. The resulting curve fit is shown as the dashed line in Figure 2, with curve fit parameters τ = 7 seconds and b = 1/3. The calculated relaxation time is of the right order of magnitude for an amorphous material above its glass transition temperature, and b is in the range found previously for oxide Newtonian materials. It can be seen that the fit is quite good, especially considering that some of the non-Newtonian data is included in the curve for the experimental relaxation function.

Indentation creep experiments on amorphous selenium has led to a better understanding of the relationship between indentation and compression creep. Indentation creep procedures can also be used to obtain viscoelastic parameters of materials, especially viscosity and relaxation functions.

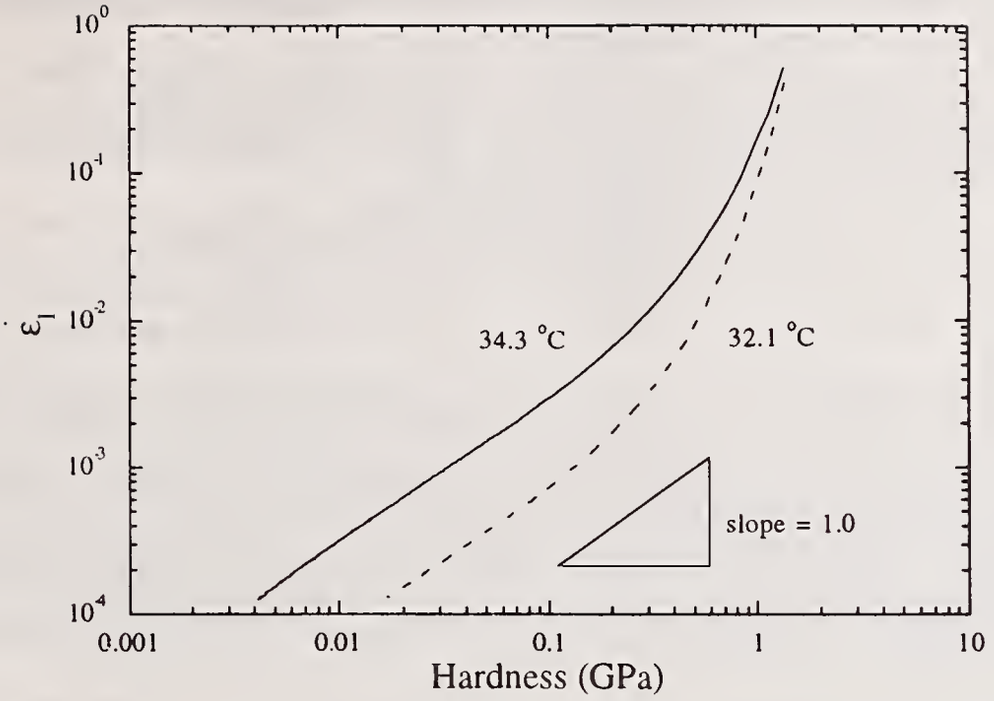

Figure 1. Indentation strain rate versus hardness for amorphous selenium at the temperatures indicated. The slope of the curves are equal to 1.0 at low hardness values, indicating that deformation is occurring due to viscous flow.

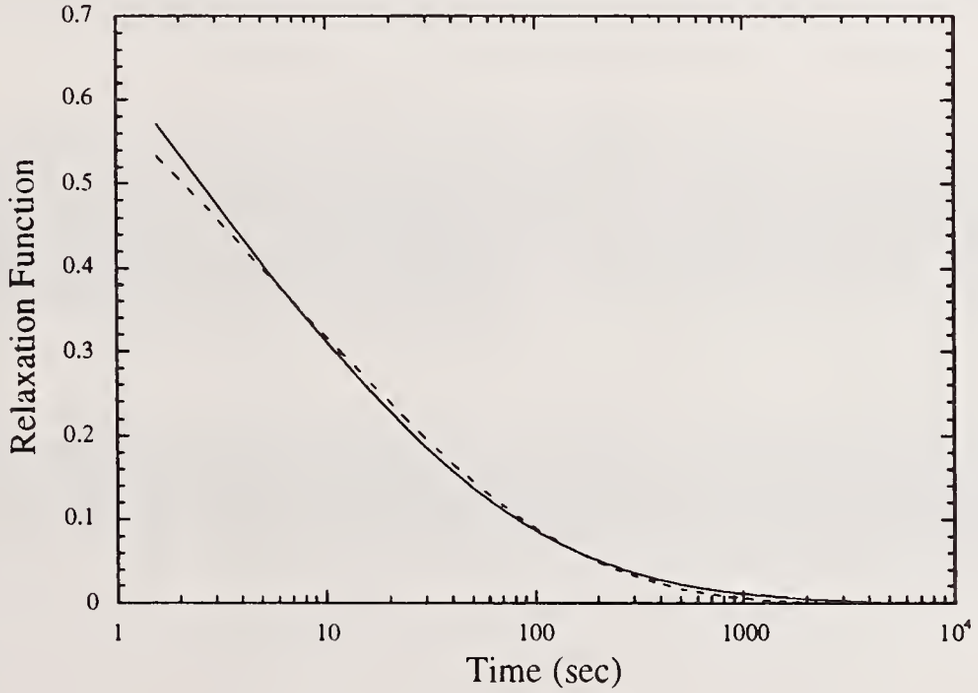

Figure 2. Experimentally determined relaxation function for indentation at 34.3 °C (solid line) and a fit to the experimental data using Equation 8 (dashed line). Curve fit parameters are τ = 7 seconds and b = 0.33.

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EFFECTS OF RESIDUAL STRESS ON THE MEASUREMENT OF MECHANICAL PROPERTIES USING INSTRUMENTED INDENTATION*

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The effects of stress on the measurement of hardness and elastic modulus using instrumented indentation have been studied experimentally and by finite element simulation for a rapidly solidified aluminum alloy (alloy 8009). The experiments were performed by making a linear array of nanoindentations on the side of a stressed bend bar, sampling regions of high uniaxial tension, high uniaxial compression, and a variety of stresses in between. When analyzed according to standard methods, the nanoindentation data reveal a decrease in both hardness and modulus with increasing stress from compression to tension. While the decrease in hardness is consistent with previous observations made in conventional hardness testing, the modulus decrease was unexpected.

Finite element simulation revealed that the decreases in hardness and modulus are not real, but occur because the procedure for determining contact area from the nanoindentation load-displacement data does not account for pileup around the indentation. The simulation shows that large compressive stresses enhance pileup while tensile stresses reduce it, and this must be properly accounted for if accurate hardnesses and moduli are to be obtained. Re-evaluation of the experimental results based on the finite element simulation further supports this point of view.

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MEASURING THE INDENTATION MODULUS OF ELASTICALLY ANISOTROPIC SOLIDS

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Nanoindentation testing is one of the most popular methods for measuring the mechanical properties of materials that are difficult to test with more conventional techniques. It has been used successfully to study hard coatings, ceramics and thin films used in microelectronic devices and for magnetic data storage. The indentation process is well understood for elastically isotropic materials and simple formulae can be derived to determine the elastic modulus of a material. We have studied the indentation of elastically anisotropic materials. The modulus measured in an indentation experiment is then a weighted average of the elastic properties of the material and is very different from Young's modulus in the direction of the indentation. We define the indentation modulus for anisotropic materials and show how it can be calculated for arbitrary anisotropic solids. We present the results from calculations for materials with cubic and hexagonal crystal symmetry and for textured films. We have calculated the contact stiffness for a flat triangular indenter on a half space for various anisotropic materials. The indentation modulus for a triangular indenter is typically 5-6% higher than that for an axisymmetric indenter and varies only slightly with the orientation of the indenter in the plane of the indentation. We have also conducted nanoindentation experiments in various single crystals. Results are in good agreement with theoretical calculations.

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THREE-DIMENSIONAL FINITE ELEMENT MODELING OF SHARP INDENTATIONS

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In recent years, pyramid micro and nano-indentation tests have been used on materials such as metals, polymers, ceramics and glasses and were found to give valuable mechanical and other physical information which may be otherwise difficult to obtain. The present talk concerns analysis of the commonly used Vickers and Berkovich tests [1,2], as well as the cone indentation test, of homogeneous, isotropic materials that are initially stress free. The mechanical information included in the experimental observations was explored with the finite element method in combination with theoretical results. A large strain elasto-plastic formulation was used, assuming small elastic strains. Contact with a rigid indenter with and without friction was accounted for. Different degrees of material strain hardening were examined. The material pressure sensitivity (e.g., compaction, transformation) was modeled according to the classic Drucker-Prager plastic potential [3]. The results concern the indentation force-depth curve for a complete load-unload cycle, the imprint morphology (i.e., sinking-in, elastic rebound, surface lift and contact area), stress and strain 3-D distributions and macro-cracking commencement. The results are presented in compact universal forms and 3-D maps which can be used for the inverse mechanical problems of finding the mechanical properties of materials (e.g. elastic modulus, yield and ultimate stress, toughness) given the output of their indentation test.

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MODELING STRESSES AT THE FILM-SUBSTRATE INTERFACE – SIGNIFICANCE TO INDENTATION TESTING

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Differences in displacements within the substrate and the coating are common when coated solids are loaded, due to differences in elastic constants. This is usually a serious problem when compliant solids are covered with hard coats, since hard compounds used for protection are invariably much stiffer than the common substrate materials. The problem is especially serious in concentrated contacts where Hertz-like contact conditions prevail and hard coats are employed to secure wear protection. Integrity of the coated composite then depends on flexure stress in the film, shear stress at the film-substrate interface or the film lift-off stress at or close to the contact. This presentation is concerned with the modeling of film-matrix interface stresses and the computation of stresses at and close to a two-dimensional or axisymmetric, high stress contact in a thin-film coated solid.

Earlier work is first reviewed. Approaches based on Fourier Transform of the stress function have been used widely. Notable works include those of Gupta and Walovit, Kennel and Dow, Ling, and Wu and Ling. Focus in these and other studies has principally been on contact stress calculations. Wu and Ling have determined the von Mises stresses at and below an axisymmetric contact. Keer and co-workers have recently made an important contribution using the transform (stress function) method.

Since the stress function transform method originally introduced by Sneddon leads to severe convergence problems when very thin coatings (sub-micron coating thickness) are modeled, (noted by Gupta and Walovit), a displacement formulation is developed and applied systematically to a number of contact problems in the present work. Navier equations of elasticity are solved for single and multi-layer composites with very thin coatings. The displacement field is first determined and the stresses are then calculated from the displacement derivatives. It is shown that the flexure stress in the film and the lift-off stress at the film-matrix interface close to the contact can lead to coating failure in many combinations of contact geometry, loading and contact friction conditions. Modeling results also indicate that the likely problems can be avoided with a judicious combination of film and substrate materials, choice of coating thickness and film deposition conditions (control of residual stresses during film deposition). Calculated results support the views advanced and discussed. It is suggested that an analytical basis is now available to design coatings for a range of loaded contact applications.

Approaches to model and analyze instrumented indentation are noted. It is noted that geometric similarity, which holds in frictionless contact, no longer holds in indentation testing. Significance of stick and slip zones at the contact is recognized. Attention is also called to the importance of Dunder's constant.

The presentation is concluded by postulating possible film failure modes during indentation testing based on the von Mises stresses calculated at and close to an indent. Results presented were obtained by modeling a two-dimensional contact – a cylindrical indentor acting on a semi-infinite coated composite. It is suggested that the plastic collapse is initiated on the upper surface of the film and grows beneath the contact and into the substrate. Shear mode failure appears to prevail during growth of plastic volume. Initial results of axisymmetric modeling (sphere on a semi-infinite coated composite) tend to support this view as well. Since elastic-plastic transition is initiated at small strains, it is noted that there is a need for relatively high precision calculations to model the indentation process.

NANOINDENTATION USING THE AFM: CONSIDERATIONS FOR THE QUANTITATIVE MEASUREMENT OF MECHANICAL PROPERTIES

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The useful application of a material depends, in many cases, on the mechanical properties of those few nanometers comprising the surface or surrounding an internal interface. Examples of this would include material failure due to grain boundary embrittlement and polymer surface coating delamination. Nanoindentation provides us for the first time with a window through which we may view the mechanical properties of these nanometer-scale volumes. Nanoindentation using an atomic force microscope (AFM) allows us the highest degree of both force and penetration depth resolution.[1]

Initially, the nanoindentation measurements which were obtained via the AFM were qualitative only. That is, the mechanical properties derived from the force versus penetration depth curves were higher for stiffer materials, but the actual measurements for a given material varied over a fairly large range. Upon further study, it was determined that this lack of precision was due to large degrees of hysteresis and creep in the lead zirconia titanate piezoceramic actuators used to translate the sample during the measurement. Upon replacing the piezoelectric actuators with electrostrictive lead magnesium niobate actuators, the hysteresis and creep effects were eliminated.[2] This resulted in highly reproducible measurements which allowed us to quantitatively compare the relative magnitudes of surface mechanical properties, i.e., one surface was 2.3 time stiffer than another.

The next evolution of this technique involved the determination of absolute mechanical properties without regard to a reference surface, i.e., surface A has an elastic modulus of 4.3 GPa. Determinations of this type require knowledge of the magnitude of the contact area between the indentor tip and the surface. Spherical indentors have several advantages for nanoindentation.[3] Among these is an easily characterized contact area. Through the use of spherical glass indentors we have been able to quantify the absolute elastic modulus of highly-orientated pyrolytic graphite (HOPG) and commercial polycarbonate as a function of strain. Our measurements have excellent agreement with those taken on the same samples using the commercial UMIS-2000 instrument.[4]

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INTERFACIAL FORCE MICROSCOPY MEASUREMENTS OF THE NANOMECHANICAL PROPERTIES OF MATERIALS

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Scanning probe techniques have shown dramatic growth over the last several years and are presently making a significant impact on surface and interfacial problems in material science. The most widely used of these techniques is Atomic Force Microscopy (AFM), which can measure interfacial forces down to the molecular level and which is applicable to virtually all materials. In its present embodiment, however, the AFM is limited in applicability by its use of a deflection force sensor which is mechanically unstable under rapidly varying interfacial forces and which has a very large compliance when measuring repulsive contact forces. In contrast, Interfacial Force Microscopy (IFM) utilizes a self-balancing force sensor which eliminates the instability and permits operation in both the attractive and repulsive modes with effectively a zero compliance. In recent studies, we have taken advantage of these unique sensor properties and applied the IFM to the study of the mechanical properties of materials at the nano-meter scale. In this presentation, we show results demonstrating the enhanced capabilities of the IFM in application to studies of the elastic and plastic deformation of single nano-scale grains in polycrystalline films of Au deposited on various substrates. The probes consisted of characterized W tips and the Au surfaces were all passivated by a single monolayer of self-assembling alkanethiol molecules. These molecules eliminate the adhesive interaction of the tip and substrate and permit a classical Hertzian analysis of the loading data. Interestingly, we are able to create significant indentations in the Au surfaces without detecting any tip/substrate bonding interaction. The molecular monolayers have a truly impressive breakdown resistance.

Loading data for single grains, along with repulsive-force images of the surface before and after loading, permit a quantitative evaluation of the effective local elastic modulus, the shear-stress threshold for plastic deformation and various modes of grain-boundary slipping. The results show a considerable variation in values obtained for the various substrates. However, the modulus and threshold shear-stress values vary in a consistent way such that the ratio of the latter divided the former is a constant 4%. The implication of this constant ratio is that the deformation to plastic threshold should be a constant 12 nm (for the 200 nm tip radius used), which is confirmed experimentally. We discuss these curious results in terms of the structural properties of the films – average grain size, substrate adhesion, etc.

In addition, we present preliminary studies of the affect of probe radius on the threshold shear stress for plastic deformation for W probes on thiol-passivated single-crystal Au(111) surfaces. We find that for tip radii less than about 400 nm the shear stress at threshold is not constant but rises sharply with decreasing radius. These findings will be discussed in terms of defect nucleation. Finally, we include a brief discussion of projected future work using the IFM in application to studies of the mechanical properties of materials.

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DEVELOPMENT OF TRACEABLE CALIBRATION GUIDELINES FOR LOAD DISPLACEMENT INSTRUMENTS, AND THEIR VALIDATION BY AN INTERCOMPARISON TEST

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The results of a research project partly funded by the European Commission on the "Measurement of Hardness (Mechanical Properties) of Surfaces" are described. The objective of the study was to develop calibration procedures for measurement of the mechanical properties of surfaces using load displacement indentation instruments; both nano- and micro-range instruments were included in the project.

The work was carried out in three phases:

i) development of calibration procedures;

ii) production and homogeneity testing of reference samples for an interlaboratory test, and;

iii) conducting an intercomparison using the agreed procedures and samples.

A primary objective in the calibration phase of the work was only to use methods that were traceable to primary standards. While for the intercomparison, the following reference materials were produced: single crystal tungsten and fused silica (isotropic materials) and single crystal aluminium (anisotropic) and 2 μm thick TiN coated tool steel was also used to examine the behaviour of a bilayer system.

Load calibration procedures for all types of instrument used in this study were relatively straightforward, with the use of calibrated weights. In some cases the weights were suspended on the indenter holder, while in others it was possible to introduce a calibrated top loading balance into the system and thus measure the load directly. Displacement calibration was carried out using a laser interferometer or a linear variable displacement transducer (LVDT) which had been previously calibrated using traceably calibrated slip gauges.

Calibration of the frame stiffness was more complex and consisted of measuring the composite stiffness as a function of load and determining the intercept at zero load in the manner described by Oliver and Pharr, for example. For this work the single crystal tungsten samples were used. There was some evidence that the expected linear behaviour was not found, and in that case the intercept was determined from the extrapolation of the best fit to the data. The departure from linearity may be related to instrument derived thermal drift during loading or use of the flat punch model (at low displacements)

Calibration of the indenter tip shape was carried out using a calibrated AFM and by indentation into fused silica. Both methods agreed at contact depths greater than about 200 nm, but the divergence increased up to 10 % at about 20 nm. This result was considered very satisfactory. Possible reasons for this discrepancy were related to uncertainty about the AFM/indenter tip interactions, and the assumption of lack of pile-up or dishing during the indentation method, and, of course, the assumption of depth independent property behaviour of the silica.

A round-robin was carried out on three types of instrument, two nano-scale and one micro-scale. When all participants have completed the tests, it is hoped that up to 14 data sets will be available. The results of three, are, however, presented. These data showed that for the isotropic materials reproducibility was such that the one standard deviation of the mean of five measurements fell within 10%. A complete error analysis considering both calibration (systematic) and measurement (random) errors showed that in the best case hardness and Young's modulus values could be obtained to 15% uncertainty for a confidence of probability of not less than 95% (2 sigma).

Abstracts for Poster Presentations

DETERMINATION OF THERMODYNAMIC RESPONSE OF POLYMERS BY MICRO-INDENTATION RATE CHANGES

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The characterization of polymers by thermodynamic response via direct activation volume measurements can be performed by means of strain rate change tests. The difficulty in performing these tests has been to make the measurement without inelastic strain transients. This has been accomplished by the use of a step-ramp procedure and validated by measuring the unrelaxed modulus of polymers during tensile testing. This step-ramp technique has been adapted for micro-indentation testing of polymers such that thermodynamic response from small volumes can be measured. The use of this technique to examine Polyethylene resins and polyester paints will be demonstrated.

ARTIFACTS IN NANOINDENTATION PROCEDURES

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Mechanical Properties Microprobes (MPM) have become a useful means of measuring hardness and elastic moduli in materials. Precision positioning tables on these devices allow the user to assess small changes in mechanical properties over predetermined distances and locations in a sample. Some MPMs have sophisticated options and allow the user to vary parameters such as load/unload rate, depth of penetration and length of hold segments which opens new possibilities for investigators. The versatility of controls on these MPMs may, on the other hand, lead inexperienced users to obtain erroneous microhardness measurements. Not only may their results differ from standard Vickers or Knoop hardness values, but variations in hardness measurements which are solely dependent on the indentation procedures programmed by the user of the MPM may be observed. This study addresses the effects of the manipulation of the operating parameters of an MPM on the microhardness and elastic modulus of a number of material systems.

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DEVELOPMENT OF STANDARDS FOR THE VALIDATION OF HARD THIN COATINGS

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In Round-Robin-Experiments the qualification of thin hard coatings for the standardisation of chemical analysis and hardness tests was investigated. PVD-TiNx-coatings of different compositions were proofed. The coating thickness amount to 10 µm. Substrate types were M2 and 1015. After finishing these investigations samples will certificate as standards. These standards then are provided for the calibration of instruments by end users.

The integral coating composition was obtained by wet chemical analysis. Element-depth profiles have been tested by several spectroscopic techniques (GDOS, AES, XPS, etc.).

The quantification of the results of the spectroscopic techniques shows systematic deviations in the concentrations of nitrogen and titanium. These deviations were determined by the used technique and increases with increasing distance to the stoichiometric composition of TiN. The results of the investigations were located by the mean value of the quantificated results of the spectroscopic techniques.

The hardness of the coated samples was examined by optical methods inspecting the indentation profile. Furthermore the load-indentation depth-technique was tested by round robin trials.

The intercomparison of hardness measurements of thicker coatings (10 µm), deposited on polished M2-substrates, showed good reproducibility. Nevertheless, by lower loads the roughness of the coating leads to increasing error paths of the results. Coated 1015-substrates are not suitable for standard samples, because of the soft substrate.

GROWTH AND MECHANICAL ANISOTROPY OF TiN THIN FILMS

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We have grown TiN thin films on Si(100) and Si(111) substrates by ultra high vacuum dc reactive sputtering. TiN grows on Si(111) epitaxially while almost completely textured growth of TiN on Si(100) is achieved. Elastic constants of TiN are measured by measuring the longitudinal sound velocity in these TiN films using a time resolved transient piezoreflectance technique. The mechanical response of these TiN films was further measured with a sub-micron depth-sensing indentation technique. Measured hardness and indentation modulus as a function of indentation depth are compared with existing theories.

ADHESION OF METAL INTERCONNECTS BY MICROWEDGE INDENTATION

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Indentation in thin film fine lines such as metal interconnects is most conveniently analyzed by using a sharp wedge-shaped probe as opposed to a tip which comes to a point as in the case of planar thin films. Two new techniques for obtaining the interfacial work of adhesion, G_c, using microwedge probes are compared in this work. In the first, microwedge indentation, we present the mechanics for adhesion, and compare the results to experiment. The system of choice is a tungsten thin film on SiO_2, commonly used in the microelectronics industry. The mechanics show that compared to axisymmetric analysis of a planar film, the microwedge technique has much higher driving force. This allows for good accuracy in measuring crack lengths using Nomarski microscopy, because crack lengths on the order of 10 to 40 microns are predicted and measured. A crack stability analysis is performed, and the effect of buckling during and after indentation is analyzed. The experiment shows an unexpected dependence of crack length versus metal line linewidth at a given indentation depth, which is resolved by accounting for microwedge tilt. Spallation of the fine lines at larger indentation depths can be detected in the indentation curves. This result is used to calculate critical bending strains in the thin films. While reasonable values of G_c are found by this technique, accuracy depends on measuring the indentation volume V_o precisely, which is best achieved by AFM. In order to improve on the accuracy of G_c, a procedure which directly measures the driving lateral force has been developed. In this technique, precracked fine lines are probed by microwedge scratching at the end of a fine line. By taking lateral load cell measurement data, G_c is well known, and compared to the result from the indentation technique.

ASSORTMENT OF TECHNIQUES IN INSTRUMENTED INDENTATION

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In this poster we review experimental techniques we have developed for: (1) calibrating both machine compliance and tip shape without having to directly image indents in the microscope; (2) performing experiments and analyzing data characterizing the creep properties of the material; and (3) performing experiments and analyzing data characterizing the hardnesses and moduli of thin coatings.

IN SITU IMAGING OF ULTRA-LIGHT LOAD INDENTS INTO GaAs AND Fe-3wt% Si

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Nanomechanical devices constitute an important and growing field, as they allow for new understanding of the mechanical properties at interfaces and surfaces. As an example, a newly developed nanoindentation device has been used to accomplish ultra-light load indents into GaAs and iron single crystals. This unique force/displacement transducer designed by Hysitron Incorporated, Minneapolis, MN, allows STM or AFM imaging directly before and after indentation, and force/displacement measurements during indentation. It is shown here that a plastic zone can be measured and is comparable to theory. Also, it is shown that the rate of indentation affects both the depth and upset zone of low load indents, implying a strain-rate sensitivity effect at room temperature. The unique problems this presents with regard to assessment of tip shape and penetration on hardness measurements of metallic surfaces is discussed. This is reinforced by observation of what appears to be a glide-based relaxation process.

MODIFICATION OF THE NIKON QM HIGH TEMPERATURE MICROHARDNESS TESTER TO OBTAIN LOAD-DEFLECTION CURVES

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Micro indentations will be made on a grown single crystal of zirconia at temperatures up to 300°C. The crystal grown by the flux method at Case, presumably monoclinic, will be cut in to bend bars. Indentations will be made on certain crystallographic planes, primarily the {100} planes, based on recent studies done at Case. The bend bars will then be bent and released prior to fracture. T.E.M. analysis of the indent area should reveal patterns of dislocation motion and generation. A secondary goal based on modifications done to a hot furnace Nikon micro indenter will enable the generation of load verses displacement curves done during the indentation. Such curves won't be affected by elastic recovery and thereby provide true hardness measurements. A load cell and displacement gages connected to an I.B.M. acquisition system will provide and record the data.

NEW ANALYTICAL PROCEDURE TO DETERMINE STRESS-STRAIN CURVE AND ELASTIC MODULUS FROM INSTRUMENTED BALL INDENTATION

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A multiple ball indentation process performed at a single penetration location was analyzed by finite element method. The response surface methodology was implemented to analyze the influence of various material properties on load-displacement (F-h) curve. A new analytical approach for relating the ball indentation data to the true-stress/true-plastic-strain (σ_t-ε_p) curve, where ε_p could be also higher than 0.2, is presented. Also, a new correlation between F-h curve, after indenter compliance subtraction, and material elastic modulus was obtained. The correlation was verified by experimental data measured by the automated ball indentation technique, for several metallic materials (Al, Cu, Steel) and the results were within ±5%.

DYNAMIC HARDNESS TESTING OF INFRARED MATERIALS

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Dynamic hardness represents a novel form of material property evaluation where the hardness of the sample is calculated based on the depth of indenter penetration while the test piece is still under load. Since this form of testing yields information about the resistance of a material to both elastic and plastic deformation and the property measurement is carried out at the time of point load application, the data obtained is highly relevant to the grinding mechanisms of these materials. Dynamic hardness measurements have been carried out for a host of infrared materials like ZnS, ZnSe, Si, Ge, etc. The hardness variation at low loads which are pertinent in the grinding process, has been examined and various possible causes for this behavior have been investigated. The influence of the surface preparation on the dynamic hardness due to various modes of grinding operations has also been investigated.

This work was carried out with the support of the Center of Optics Manufacturing (COM), American Precision Optics Manufacturers Association (APOMA) and the University of Central Florida

EFFECT OF INDENTER SHAPE, YIELD STRESS AND MODULUS ON THE INDENTATION PROCESS

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The indentation process is simulated by finite element analysis using the elastic/perfect plastic constitutive relation and the ABAQUS software. Conical indenters of different included angles are used. To correlate with the yield stress, the hardness should be calculated by dividing the applied force to the projected area (rather than the contact area used in some conventional hardness tests) measured by including the pileup of the indentation (similar to the area observed in the optical microscope). The area determined by using the plastic depth, namely, the extrapolated depth from the initial part of the unloading curve is about 72% of the projected area just mentioned. The estimation of Young's modulus from the slope of the initial part of the unloading curve is better if the projected area is used instead of the area determined by the plastic depth. The ratio of hardness to yield stress decreases with the ratio of yield stress to Young's modulus if the area determined by the plastic depth is used.

This work was supported by NSF through DMR 9221326 monitored by Dr. Bruce MacDonald and by the Manufacturing Sciences Group in the Center for Optics Manufacturing at the University of Rochester.

VISCOSITY MEASUREMENT BY IMPRESSION TEST

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A new method of measuring Newtonian viscosity is proposed. In this method, a flat cylindrical punch is pressed onto the surface of a semi-infinite medium by a small load. The relationship between the steady penetration velocity and the applied load is independent of the slip or stick boundary condition at the punch-material interface. In this relation, the steady state punch velocity is proportional to the punching stress and, for the same stress, it is proportional to the punch diameter and inversely proportional to the viscosity. By using this method, the viscosity of amorphous Se was measured in the temperature range of 29.2 to 50.7 °C and under the punching stress of 0.25 to 15 MPa. The viscosity of amorphous Se obtained this way is comparable to the literature data and its flow behavior under these conditions is Newtonian.

This work was supported by NSF through DMR 9221326 monitored by Dr. Bruce MacDonald.

CONTACT STRESS ANALYSIS OF A LAYERED SOLID UNDER A RIGID INDENTER

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The problem of a layered elastic solid indented by a rigid body involves mixed boundary conditions. The distributed tractions between two bodies and contact length are determined by the geometries and material properties of the punch, films coated and substrate. An approach based on the displacement formulation is used. The contact pressure and contact length are calculated from the governing integral equations. The problem then becomes that of a layered solid under boundary tractions. Calculated results of stresses within film and substrate are to be presented. The effects of the elastic modulus and Poisson's ratios of film and substrate are investigated and discussed.

MEASUREMENT OF STRAIN-RATE SENSITIVITY BY NANOINDENTATION TECHNIQUES

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For this work, the nanoindentation device uses a piezoelectric transducer to drive the indenter and a double spring system to measure mechanical load. The instrument is characterized by a displacement resolution of 0.4 nm and a load resolution of 5 μN. The indenter system was modified to perform strain-rate sensitivity measurements by a software-driven step ramp technique. The procedure involves the application of an indenter-displacement step in the ramp while changing the indentation rate. The magnitude of the displacement step is selected to compensate the elastic response of the indenter system and to eliminate any transient in indentation rate. The change in load response is then truly material dependent and provides information on microstructural properties of the specimen.

ANALYSIS OF NANOINDENTATION LOAD-DISPLACEMENT LOADING CURVES

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Nanoindentation load-displacement curves provide a 'mechanical fingerprint' [1] of a materials response to deformation. Over the last few years, much attention has been focused on understanding the shape of the unloading curve to obtain values for true contact area, Young's modulus and hardness. When the unloading curve is well behaved, i.e. approximates to linear behaviour [2] or alternatively fits a power-law relationship [3] then this approach can be very succesful. However, when the material is very stiff and hard (such as for many thin hard coatings), the unloading curve fits neither of these two models particularly well. Therefore, there can be considerable difficulty in obtaining an accurate final plastic depth from which the area of contact between the coated system and the indenter can be calculated. Thus it can be extremely complex to calculate meaningful mechanical property data (i.e. E and H numbers) for these types of material.

Recently, an alternative approach has been to try and understand the shape of the nanoindentation loading curve and the relationship between modulus, hardness, indenter geometry and the resultant maximum displacement for a given load. This used an original model by Loubet [4] who proposed the relationship $P = K_{ep}h^n$ for describing the shape of loading curves, where K_{ep} is a constant which describes the elasto-plastic nature of the loading curve. This model was used to predict the rank position of loading curves for a range of materials including a TiN coating on Si and a CN_x coating on Si [5].

However, when compared to real nanoindentation loading curves it was found that there was some discrepancy between the rank order of the actual loading curves and those predicted by the Loubet model. The Loubet model used a power law fit to the loading curves, which gave indexes of usually < 2. For indentation by a cone however, the relationship between the load and displacement is predicted to follow the relationship $P \propto \delta^2$ and therefore we chose to plot P versus δ^2 to see if this relationship holds. The plots of load versus displacement squared showed excellent correlation over the whole loading range. This correlation then formed the basis for our analysis. We followed a similar approach to Loubet *et al* where we decomposed

the total deformation into elastic and plastic parts. The plastic and elastic contributions can be represented by

$$\delta_p = \phi\sqrt{\frac{P}{H}} \qquad \text{and} \qquad \delta_e = \psi\frac{P}{E}\sqrt{\frac{H}{P}}$$

For these two equations δ is the displacement, ϕ and ψ are constants, P is the load, H the hardness and E the Young's modulus. Summing these two parts and a little manipulation of the equation gives the relationship

$$\delta^2 = \frac{P}{E}\left(\phi\sqrt{\frac{H}{E}} + \psi\sqrt{\frac{E}{H}}\right)^2$$

This equation is of the form

$$P = K_I\delta^2$$

We found the gradients, m, of the load versus displacement squared plots from nanoindentation data for a number of materials and compared this to the value of K_I predicted from their modulus and bulk hardness values. This allowed us to determine the constants given below

$$\frac{1}{K_I} = \frac{2}{E}\left(\sqrt{\frac{H}{E}} + 0.1\sqrt{\frac{E}{H}}\right)^2$$

This equation was found to reliably predict the loading curves of a wide range of materials with varying combinations of modulus and hardness.

This model therefore, helps to analyse and understand the fundamental factors which control the shape of the loading curve during nanoindentation experiments with a pointed (Berkovich) indenter. This is particularly valuable for assessing the mechanical properties of systems with thin hard coatings, where there are difficulties in estimating the real plastic depth.

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MEASUREMENT AND THEORY OF THE ORIENTATION DEPENDENCE OF KNOOP MICROHARDNESS IN A LAYERED SINGLE CRYSTAL

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The room-temperature red phase of mercuric iodide crystallizes in a layered structure, with monoatomic planes of iodine (I) and mercury (Hg) atoms stacked perpendicular to the [001] (or c) axis in the sequence ...IHgIIHgIIHgI.... Each mercury atom is covalently bonded to two iodine atoms in the layer below and two iodine atoms in the layer above; bonding between adjacent iodine layers is van der Waals in nature. Because of its unusual bonding characteristic and resulting high anisotropy, single crystal mercuric iodide provides an interesting system for studying fundamental deformation processes. The scientific interest in mercuric iodide's behavior is enhanced by its technological importance as a wide band-gap, high atomic mass semiconductor which finds application as a room-temperature detector material for gamma-ray, X-ray and visible radiation. In the present work, measurements were made of the orientation dependence of the Knoop microhardness on faces of single crystals of red (tetragonal) mercuric iodide that were vapor-grown for use in radiation detectors. Theoretically, the size of a microhardness indention is presumed to depend on the volume of material in which appropriate slip systems are stressed sufficiently to cause appreciable slip. To test this concept and determine which particular slip systems dominate the indention process, the "infinite flat punch" model was used to calculate the orientation and volumetric variations of shear stress on various potential slip systems in mercuric iodide. Excellent agreement was obtained between theory and experiment.

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INDENTATION AND STRENGTH OF FIBER-REINFORCED AEROGELS

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Aerogels are extremely low density solids that are characterized by a high porosity and by pore sizes on the order of nanometers. Their low thermal conductivity and sometimes transparent appearance make them desirable for applications such as insulation in cryogenic vessels and between double paned glass in solar architecture. An understanding of the mechanical properties of aerogels is necessary before aerogels can be used in load bearing applications. Experiments to test the resistance to indentation (hardness) and the compressive strength of various types of fiber-reinforced silica aerogels have been undertaken. The indentation pressures of traditional techniques such as Vickers and Knoop were too large for the fragile aerogels. An alternative approach using an Instron 1123 testing machine equipped with a 19.05 mm steel ball as the indenter proved successful. Initial results of measurements of hardness, compressive strength, and stiffness, and their dependence on processing variables and fiber percentage, are presented. For specimens tested to date, the addition of fibers to the aerogel matrix resulted in weaker materials.

STUDY OF ROCK INDENTATION FRACTURE IN MECHANICAL EXCAVATION

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This paper represents part of research effort to establish a method for prediction of penetration rate of tunnel boring machines (TBMs). Focus is on single disc cutters working in medium to very high strength rock (uniaxial compressive strength 20 to more than 200 MPa) . The objective is to relate achieved penetration rate to geometry and material properties of the disc cutter, and mechanical properties of the intact rock; effects of discontinuities on penetration rate will be investigated once the model is working for intact rock. Our proposed work will focus on basics of indentation fracture and rock crushing.

Indentation of brittle materials using sharp indentors leads to inelastic deformation beneath the indentor, and radial-median cracks during loading. Lateral cracks grow from the area of inelastic deformations and propagate toward free surface upon unloading (Lawn, 1993). Similar observations have been made during linear cutter tests (Snowdon *et al*, 1982, Nelson, 1986).

Proposed methodology contains three phases: identification of the potentially most important factors in disc-rock interaction, developing analytical solution(s) to determine state of stress under cutter, and a finite-element model to model rock crushing and crack propagation from contact zone. A simple two-dimensional indentation problem will be modeled, in conjunction with laboratory experiments. In this problem, four distinct substructures can be identified: cutter and machine, crushed rock, moderately-cracked rock, and intact rock. Interface elements will be used between elements of different substructure types. Data from similar loading conditions are sought even if tests done on other brittle materials.

The model needs then to be extended to three-dimensional, dynamic problems in order to be able to predict penetration of cutters. This is left as a future project once the two-dimensional, static model gives insight into the mechanics of problem and provides good answers.

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INDENTATION TESTING OF THE MAGNETIC THIN-FILM HEAD ALUMINA SUBSTRATE

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The complex layered structure of the thin-film head contains many materials and interfaces. Interaction of the head with the rotating disk surface causes the crack initiation which leads to the head failure. We report on the application of the microindentation technique for the characterization of the crack resistance of the head alumina substrate. Effects of the head current, laser processing on the fatigue crack location and geometry discussed.

BULK AND INTERFACIAL CRACK-PROPAGATION BEHAVIOR IN CVD DIAMOND

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The fracture toughness, stress corrosion and cyclic fatigue properties of CVD polycrystalline diamond have been investigated on thick (100 to 300 μm) free-standing films. Since all reported toughness and crack growth measurements on CVD diamond to date have been derived using approximate indentation techniques, in the present study ASTM-standard compact-tension (fracture-mechanics) specimens were tested in addition to indentation methods. Resulting values of the fracture toughness, K_{IC}, for the free-standing films were found to be of the order of 5 MPa$\sqrt{m}$. Preliminary studies on subcritical crack growth under sustained load indicate that CVD diamond is essentially immune to stress-corrosion cracking, although more sophisticated experiments need to be tried out in order to prove this definitively. Fatigue tests are currently being conducted by loading indentation-precracked free standing CVD diamond cantilever beams in three point bend. Indentation techniques have also been applied to measure and compare the interfacial adhesion and toughness of thin (~2 μm) CVD diamond films deposited on various metallic substrates such as molybdenum and titanium.

RESIDUAL STRESS FIELDS AT INDENTATIONS

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This presentation outlines an indentation method to measure residual stress in ceramics. The analysis is based on the premise that the observed lengths of radial cracks at Vickers indentations depend on the residual stress due to the indentation and a component due to any imposed residual stress field. The expressions to evaluate the imposed residual stress are:

$$\sigma_r = K_c \frac{1-(c_0/c_1)^{3/2}}{\sqrt{\pi c_1}} \qquad \text{(tensile stress)}$$

$$\sigma_c = -K_c \frac{1-(c_0/c_2)^{3/2}}{\sqrt{\pi c_2}} \qquad \text{(compressive stress)}$$

where K_c is the fracture toughness, c_0 is the unstressed crack size and c_1 and c_2 are crack sizes in the stress field.

The technique is used to explore residual stress fields around Vickers, Berkovich and Knoop indentations made in glass and several ceramics. The data are presented as isostress contour maps for both the tensile and compressive components of the biaxial stress fields. In general the fields are not axisymmetric but reflect the shape of the indenter and the influence of cracking. The toughness of the material appears to be an important factor influencing the residual stress field produced by sharp indentation.

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INTERFACIAL FORCE MICROSCOPY STUDY OF THE NANOMECHANICAL PROPERTIES OF THIN FILMS FORMED ON CARBON STEEL FROM ZINC DIALKYL DITHIOPHOSPHATE ANTIWEAR/EXTREME PRESSURE OIL ADDITIVES

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Zinc dialkyl dithiophosphates (ZDDPs) are the most commonly used antiwear/extreme pressure additives in commercial motor oils. These compounds break down under conditions of sliding contact, and form thin antiwear films on carbon steel surfaces from the decomposition products. The chemical makeup of the antiwear films has not been elucidated to any great extent, nor has the mechanism by which ZDDPs dramatically decrease the wear rate.

In this study, we have used the interfacial force microscope (IFM) to investigate the nanomechanical properties of thin films formed on carbon steel from decomposition of ZDDPs in base oil. Films formed by thermal means only and films formed in the presence of sliding contact have been examined. Preliminary results indicate that these two methods of preparation result in vastly different mechanical properties on the nanometer scale. In the case of the thermal samples, the elastic portion of the loading curve can be interpreted in terms of Hertzian contact mechanics for a parabolic probe. Both the indentation modulus of the film and the maximum shear stress at the onset of plastic deformation have been determined. The sliding contact samples, on the other hand, exhibit a range of indentation behavior that suggests the presence of three distinct materials. Two of these are likely to be exposed steel and material quite similar to the thermal films, but the third is unique to the sliding contact samples. The latter material is particularly exciting as its force-displacement curve is elastic to high peak loads, although not in a Hertzian manner. These observations allow us to speculate that the material unique to the sliding contact samples may come about by transformation of thermal film material under the influence of high local stress and temperature at contact asperities. This is an important statement in that it contradicts the belief held by some researchers that the two methods of preparation result in identical films.

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