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Nanoindentation Round Robin on Thin Film Copper on Silicon

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Nanoindentation is used in a variety of fields to measure material hardness and elastic modulus. This test technique is especially attractive for thin films because of the difficulty of conducting tensile or other conventional mechanical characterization tests on thin film specimens, and because it requires only a small surface area for testing. However, the standardization process for this new measurement method is still in progress. To test the ability of current measurement procedures to provide comparable results, a round robin was conducted. Invitations to participate were sent to over 100 laboratories. Two specimens, a copper film on a silicon substrate and an uncoated substrate, were distributed to each of 33 laboratories. The choice of measurement procedure was left to the performing organizations. By the end of the reporting period, 27 sets of results were received. While the average reported uncertainty (1 standard deviation) among the individual participants was 4 pct of the average hardness, the interlaboratory standard deviation of the hardness values was 15 pct. Similarly, the average reported uncertainty of the modulus was 5 pct of the average value, but the interlaboratory standard deviation of the modulus was 19 pct. None of the measurement variables examined in this round robin, including instrument type, analysis procedure, time since instrument calibration, chip mounting procedure, and tip condition, and neither of the potential covariant effects, chip location in the wafer and test date, were found to have a statistically significant effect on the reported hardness or modulus.

I. INTRODUCTION

NANOINDENTATION was recently proposed as a test technique for measurement of hardness and indentation modulus of all types of solid materials.[1,2] Nanoindentation is attractive because it is a convenient, understandable test that provides a quantitative result. The over 2600 citations of Reference 1 attest to the attractiveness of this test method. Nanoindentation has been extended to studies of additional properties, in particular, to film-substrate adhesion[3] and to residual stress.[4] The advantages of the method for thin films were recognized immediately.[5] Foremost among these is the ability of this technique to characterize a thin film mechanically without creating a particular specimen geometry and freeing the specimen from the substrate, as needed for microtensile testing.

The small surface area, only a fraction of a square millimeter, damaged by the test is also a significant advantage. Standardization of this technique is still in progress.[6–13]

Tsui and Pharr[14] recognized the problem of measuring the property of the film, rather than some average property of the film and substrate, and addressed it by limiting the penetration of the indenter tip to a fraction of the thickness of the film.

Thin film materials are technologically important materials today. Metal and dielectric films on silicon substrates are basic components of ULSI devices and other semiconductors. The annual world market for semiconductor products is in the hundreds of billions of dollars; many companies around the world manufacture these products, and so are concerned with characterizing films on substrates. Metal films on silicon substrates are described as soft films on hard substrates,[15] meaning that the substrate is significantly harder than the film. A different category of films is formed by hard anticorrosion and antiwear coatings and some reflective coatings, which are harder than their substrates. Possibly because hardness characterization is traditional for hard coatings but is not traditional for electrical conductors and dielectrics, the standardization of nanoindentation for hard films has progressed further than that for soft films. This study is intended to help in moving the standardization process forward.

Hardness is a measure of the resistance of a material to permanent deformation. Various tests for hardness have been used for centuries,[16] and will not be reviewed here. The advance of nanoindentation, or instrumented indentation, is that the force on the indenter tip and the displacement of the tip into the specimen surface are measured and recorded as the indentation event progresses. Such measurements have only become possible recently, with advances in electronics and computers. The traditional analysis by Tabor[17] concludes that the hardness should be equal to 3 times the stress that is reached at a plastic strain of about 8 pct in the tensile test. This result is as valid for nanoindentation as for more traditional hardness tests.

A very large and useful group of standard test methods has been produced under the auspices of the American Society for Testing and Materials (ASTM). Efforts are in progress to standardize nanoindentation through the normal ASTM procedures. Part of the standard test method is a statement of repeatability and reproducibility. Both of these are related to the precision of the test method. Assessment of the accuracy of a test method requires knowledge of the ''true value'' of the property being measured, which is often unavailable. ASTM has published a ''Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method.''[18] Repeatability refers to the

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expected distribution of results obtained for a restricted set of test conditions, such as one instrument in one laboratory. Reproducibility refers to the expected distribution of test results that would be obtained for a more general set of test conditions, such as similar instruments in different laboratories.

A. *Specimen Material*

Because copper is now the most popular interconnect material in electronic interconnects, a copper thin film was chosen as the specimen material. The specimen material used here was a sputtered copper film, nominally 1.5-μm thick, on a silicon substrate. An uncoated substrate specimen was also distributed. Its use as a reference specimen is limited to statistical considerations. The specimen film was etched by ion milling and examined by scanning electron microscopy (SEM). Electron backscatter diffraction (EBSD) was also used to measure the grain size and the preferred orientation of the specimen film. No characterization was carried out on the {100}-oriented single-crystal silicon substrate material.

Two metallized specimen wafers were fabricated; these were silicon wafers 75 mm in diameter with a specimen film consisting of an adhesion layer 10-nm thick, a Cu layer nominally 1.5-μm thick, and a Pt passivation layer 10-nm thick. Because the Pt layer thickness is only 1/30 of the penetration depth used for the hardness measurement, and only 1/150 of the depth of the entire film, it is assumed that this layer does not influence the measured hardness values. The Pt is a face-centered-cubic metal close to Cu, Ag, and Au in the periodic table. Bahr *et al.*[19] studied the effects of 60-nm-thick hard oxide films over Al films on Si substrates; these caused discontinuous loading curves, not seen in this study. The only effect of the Pt here is assumed to be suppression of oxidation of the Cu. One of the metallized wafers, and also the uncoated wafer, was sectioned, by manual scribing and cleaving, into small chips approximately 1-cm square. An approximate record of where the film-on-substrate chip for each laboratory came from on the specimen wafer was kept. These chips were wrapped in laboratory tissue paper, placed in small plastic boxes, and mailed to the participants.

The tensile tests were conducted using procedures previously described.[20] The specimen geometry was patterned using a two-stage subtractive photolithography process. The Pt layer was removed by ion milling and the Cu layer by wet etching. For each test, the gage section was approximately 190-μm long by 10-μm wide. An offset of 0.2 pct was used to determine the yield strength.

B. *Procedure for Interlaboratory Comparison*

The present interlaboratory comparison was carried out in a manner consistent with ASTM E 691, except that each participant was requested to use the test method that they would use for a ''normal customer.'' ASTM E 691 suggests rigid control of the test method. It was part of the objective of this study to see if such rigid control was necessary. All of the instruments are supplied with computer programs that control the test operations and the acquisition of data. These programs also include routines for analyzing the data and reporting hardness and indentation modulus. The analysis routines are all based on the same original technical studies by Oliver and Pharr.[2] So it was considered possible that the test methods used by different laboratories are essentially identical. The present results place this assumption under question.

This study was designed to attract as many participants as possible; this led to certain choices in the conduct of the study. Besides not dictating a particular test method or analysis routine, as mentioned previously, we also distributed only two specimen materials to each participant, in the form of film-on-substrate and uncoated-substrate chips. Some participants did not report results for the uncoated substrate.

Each participant conducted a test and reported the results. It was requested that the hardness at a penetration depth of 300 nm be reported; this was 0.2 $\times$ film thickness. A set of questions on variables associated with the test setup, the instrumentation, and the test procedure were asked. Most participants answered all of these, although some did not.

ASTM E691 recommends that multiple materials be tested in an interlaboratory comparison, to cover a range of the property measured. The published method suggests that three materials should be used; as mentioned previously, only two were used here, the film-on-substrate and the uncoated substrate. All 27 participants reported data on the film-on-substrate; 18 sets of data for the substrate were contributed.

To explore possible effects of experimental variables, participants were requested to supply additional information, some in multiple-choice style, as follows: (1) apparatus type: make and model; (2) indenter tip geometry: spherical, Berkovich, *etc.*; (3) tip condition: new or near new, used, or heavily used; (4) experimental method: as supplied by instrument manufacturer, or literature reference; (5) analysis method: as supplied by instrument manufacturer, or literature reference; (6) time since instrument calibration for displacement: not since instrument delivery, less than 6 months, greater than 6 months; and (7) time since tip area calibration: not since delivery, less than 6 months, greater than 6 months.

II. RESULTS AND DISCUSSION

A. *Material*

The specimen films were characterized by microstructural analysis and tensile testing. An SEM view of the Cu film material is shown in Figure 1. The surface shown was prepared by ion milling, using Ar ions directed at an angle of 70 deg from the surface normal. This treatment removed the Pt surface layer and part of the Cu layer, leaving the Cu surface sufficiently flat for EBSD. The EBSD measurements showed that the visible topography in this image corresponds to crystallographic grains. Table I shows the film thickness, obtained by profilometry on patterned regions, and the grain size (average grain diameter). The grain size was obtained by averaging values from the intercept technique applied to the micrograph shown in Figure 1, the intercept technique applied to a diagram of grain boundaries obtained by EBSD, and the average diameter of the grains as obtained by EBSD. The EBSD procedure

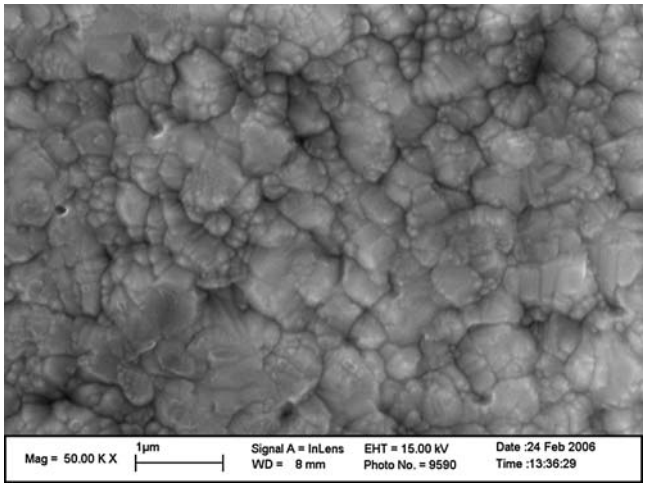

Fig. 1—SEM image of the surface of the Cu specimen film after preparation by ion milling. The imaging conditions are noted. The EBSD showed that the features correspond to crystallographic grains.

Table I. Properties of the Specimen Film

Thin film material	Sputtered Cu with 10-nm Pt passivation layer
Film thickness, μm	1.45
Average grain diameter, μm	0.48
Microtensile results	
Number of tests	7
Yield strength, MPa	421
Ultimate tensile strength, MPa	530
Modulus (initial loading), GPa	65
Modulus (later loading), GPa	85
Elongation to failure, pct	2.3

tends to find small grains as well as large ones, whereas the manual intercept procedure seems to favor large grains. The values of average grain diameter measured by the different techniques differed over a range of a factor of 2. A typical engineering stress-strain curve for the Cu film material is shown in Figure 2.

The Berkovich indenter geometry used by almost all of the participants in this study has a projected area A given approximately by $A = 24.5 \cdot h^2$, where h is the penetration depth. For the depth of 300 nm used here, the corresponding value of the area is 2.16 μm^2. Comparing this size against the scale bar shown in the micrograph of the specimen material shown in Figure 1, it is clear that the indenter will be in contact with multiple grains of the specimen material at the depth where the hardness is recorded. This is consistent with the low values of standard deviations generally reported by the participants. The multiple indentations, typically 25, made in the course of each complete measurement each sampled multiple grains, allowing an averaging effect.

The strength listed in Table I can be checked for consistency with the strength of other copper films, using a Hall–Petch plot, as shown in Figure 3. This plot is based on the idea that the strength of a pure, polycrystalline metal depends on its grain size; both theoretically and experimentally, the strength should increase linearly with the reciprocal of the square root of the grain size.[16] Figure 3 shows yield strength values for several Cu films, as determined from microtensile tests.[21] This figure shows that the measured yield strength value for material of the current study follows the trend of other copper films, though it may be a little to the high side.

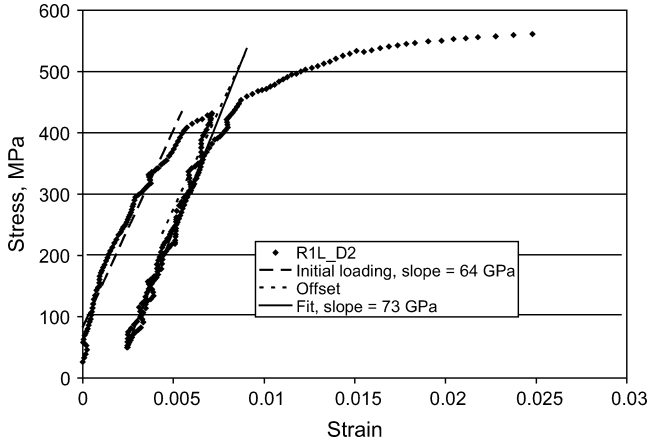

Fig. 2—Stress-strain curve for the Cu film of this study. The specimen was loaded, then unloaded to obtain a modulus value, and then loaded to failure.

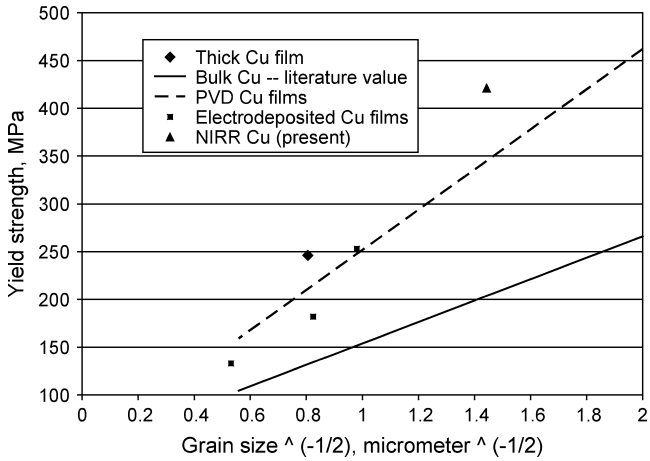

Fig. 3—A plot of yield strength against the reciprocal of the square root of the grain size (Hall–Petch plot). The material of the current study (NIRR for nanoindentation round robin) falls within the trend of other copper films, though it may be a little on the high side.

B. *Nanoindentation Results*

The nanoindentation results for the hardness of the specimen film, as reported by all participants in this study, are shown in Figure 4. The indentation modulus results for all participants are shown in Figure 5. While the average reported uncertainty (1 standard deviation) among the individual participants was 4 pct of the average hardness, the interlaboratory standard deviation of the hardness values was 15 pct. Similarly, the average reported uncertainty of the modulus was 5 pct of the average value, but the interlaboratory standard deviation of the modulus was 19 pct.

The procedures for nanoindentation testing were recently reviewed by Oliver and Pharr.[2] The vast majority of the participants in this study used commercial instrumentation

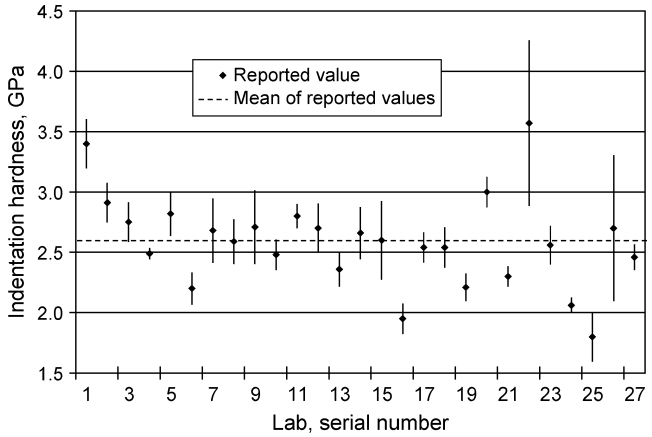

Fig. 4—Reported results for indentation hardness of the round robin specimen material, a Cu film on a silicon substrate. The error bars at each data point span a total range of four times the standard deviation reported for multiple indentations. The average of the reported hardness values is 2.59 GPa.

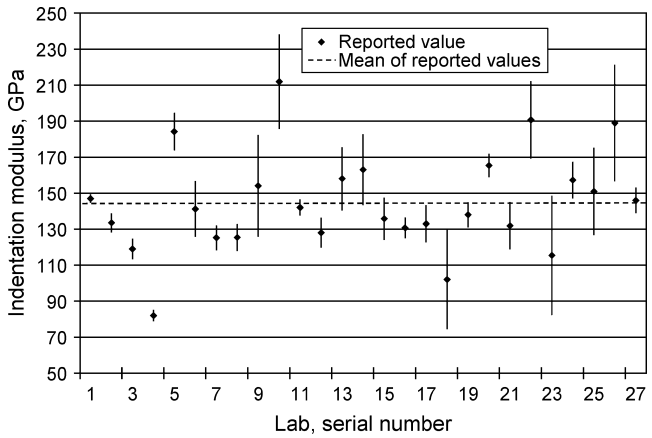

Fig. 5—Reported results for indentation modulus of the round robin specimen material, a Cu film on a silicon substrate. The error bars at each data point span a total range of 4 times the standard deviation reported for multiple indentations. The average of the reported modulus values is 144.5 GPa.

designed specifically for nanoindentation. All of these instruments implement computer-controlled data acquisition and data reduction procedures that implement the Oliver and Pharr method. Most of the participants indicated that their results were based on the software by the manufacturer of their instrument. The remainder of the participants indicated that they had followed procedures based on Oliver and Pharr. The indentation hardness value is simple in concept; it is given by $H = F/A$, where F is the force imposed to drive the tip into the specimen surface, and A is the projection in a plane parallel to the specimen surface of the contact area, which is the area of the tip that is in contact with the specimen material while the force F is applied. The subtleties of the method for measuring H result from the difficulty of obtaining A. The method of Oliver and Pharr consists mainly of procedures for obtaining A from hardness measurements. The zero-order approximation is the ideal area of the indenter tip; for the Berkovich tip used in 25 of the 27 measurements reported here, this area is given by $A = 24.5\ h^2$.[1] The depth h and the indenter geometry related to the numerical factor are both subject to variability that is addressed by the test method. For example, comparison of experimental hardness as a function of penetration depth against expected values provides information about possible deviations of the actual tip profile from the ideal value.

C. *Repeatability and Reproducibility*

ASTM E 691 recommends procedures for calculating repeatability and reproducibility for inclusion in standard test procedures. The larger the numerical values of these quantities, the more uncertain are the results obtained. The values found using ASTM E691 calculation procedures and all the data reported by the participants in this study are listed in Table II. The calculation uses a combination of squared deviations of the measured values; the reproducibility is larger than twice the standard deviation because E691 is aiming for a 95 pct confidence level.

D. *Other Experimental Variables*

The experimental variables listed previously in Section I–B, including make and model of testing machine, indenter type, tip condition, *etc.,* were tested for statistically significant effects on the measured values of hardness and indentation modulus of the Cu film using an analysis of variance techniques. Because the data were too sparse for a full analysis, the variables were considered one at a time as single factors. The F-test was used to check the statistical significance of the variations among the different ''treatments'' relative to the variations within treatments. For each variable, the hypothesis tested was that the means were the same among the treatments. The results are shown in Table III. It can be seen that if the mount type has no effect on the hardness value, the probability of observing the current results is 0.28. A p value of 0.05 or smaller indicates that an experimental variable is significant. By this test, no statistically significant effects were identified.

Figure 6 shows a graphical display of the data for the different mount types. Hot mount means the specimen chips were attached to a metal block with a hot-melt bonding agent such as wax; cold mount means that the chips were attached with a room-temperature–cured bonding agent such as cyanoacrylate; the remainder, including those who specified no bonding agent and those who did not specify their mounting technique, were grouped as ''none or no response.'' Data for the various types appear to overlap and to have generally similar scatter. All of the experimental variables were plotted in the same way, and all the plots resembled Figure 6, with overlap of the distributions of reported values among the different groups and similar scatter for all groups.

The repeatability and reproducibility values listed in Table II are surprisingly large, at least to the authors of the current study. They are larger than might have been expected by extrapolating the values reported in the previous studies discussed in Section I.[8,9] Where does the variability come from? Since no statistically significant effects at the current measurement errors were found among the particular group of experimental variables addressed in the reports of the participants, this set of variables can be excluded as the source of the variability; these

Table II. Repeatability and Reproducibility of Hardness and Modulus Measured by Instrumented Indentation, Calculated According to ASTM E 691

Cu Film on Substrate, 27 Data Sets	
Hardness, GPa	
Average	2.59
Repeatability	0.34
Reproducibility	1.13
Indentation modulus, GPa	
Average	144.5
Repeatability	22.9
Reproducibility	81.7
Uncoated Silicon Substrate, 18 Data Sets	
Hardness, GPa	
Average	11.6
Repeatability	1.1
Reproducibility	4.5
Indentation modulus, GPa	
Average	147.6
Repeatability	9.5
Reproducibility	49.0

Table III. Results of Statistical Tests for Equality of Means for the Effects of Each Experimental Variable

Experimental variable	p value
Mount type	0.28
Time since instrument calibration for force and displacement	0.85
Tip condition	0.24
Apparatus type	0.43
Time since tip area calibration	0.84
Analysis method	0.57

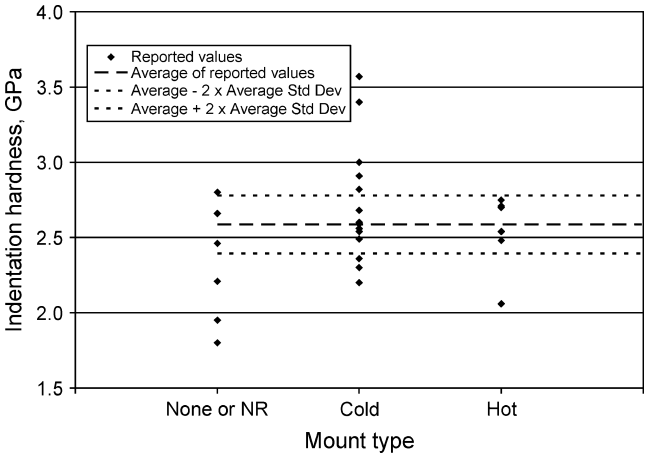

Fig. 6—Reported hardness values plotted against mount type. The group "None or NR" includes participants who used no bonding agent and those who did not provide a response to the question about mount type.

include instrument manufacturer, analysis procedure, tip type, and reported qualitative tip condition. Of course, all the participants were reputable laboratories with knowledgeable users of research-grade instruments.

What about the specimen materials as a source of variability? Present day commercially obtained silicon wafers are highly standardized and controlled single-crystal materials, and the data scatter for the uncoated silicon were similar to that for the Cu film. In addition, the results were examined for dependence on distance of the chip from the center of the wafer (Figure 7) and day of measurement (Figure 8). Such variations could have come from the deposition process or from aging effects in the Cu film. No statistically significant effects of these possible covariant variables were found.

Since the present specimens were distributed in the form of small chips, it was necessary for each researcher to mount the specimen in the testing machine in some way. The participants were asked to indicate whether they used a hot-mount procedure, such as with wax, or a cold-mount procedure, such as with a room-temperature-cured adhesive. This question did not anticipate the possibility that some investigators may have simply placed their specimen chip on a hard surface within their instrument with no bonding agent. A few respondents did not make their procedure clear. The available data were checked, but no statistically significant effect of the specimen mounting was found.

This study was designed to evaluate the repeatability and reproducibility that can be expected by a "typical customer" who requests nanoindentation hardness and modulus values from a "typical provider" for a thin film of a type typically studied and used for microelectronics applications. It was assumed that the customer would not attempt to specify the details of the measurement procedure. Certain requests were made of the participants: provide data at a penetration depth of 300 nm (0.2 × film thickness), provide data averaged over at least 10 indentations, and use specific values for the Poisson's ratio. (The Poisson's ratio enters the data reduction process for extraction of the indentation modulus).

A previous standardization effort for modulus by nanoindentation was reported by Jennett and Meneve.[8] This study differed from the present effort in several respects; in that study,[8] test procedures were controlled much more tightly; fewer laboratories were involved; reference specimens, as well as test specimens, were distributed and tested by the participants; and hard films on hard substrates were used. These authors did not report ASTM-style repeatability and reproducibility results, but their reported standard deviation among laboratories was much smaller than was found here. Jennett and Bushby[9] reported another extensive interlaboratory study involving ten European laboratories and four coating-substrate materials systems. The emphasis in this study was on development of a protocol for conducting the tests and analyzing the data, in order to reach estimates of the "true" coating properties. The referenced proceedings[9] include one plot of hardness data for a soft film on a hard substrate, gold on glass. The scatter in the hardness values that appear on the plot at a penetration depth of 0.2 × film thickness seems roughly comparable to the scatter reported here.

All of the indentation tips used in a previous study[8] were characterized by traceably calibrated metrological atomic force microscopy. No such checks were used for the apparatus used in the current study. Variations in indentation tip shapes, not adequately compensated by presently used procedures for determination of the tip area function, cannot be excluded as the source of the variations of the

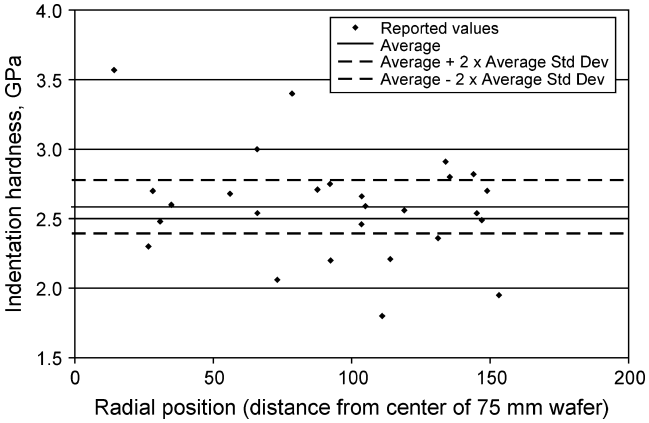

Fig. 7—Indentation hardness plotted against radial position of the respective specimen chip. These data show that there is no statistically significant effect of the location of the chip on the measured indentation hardness.

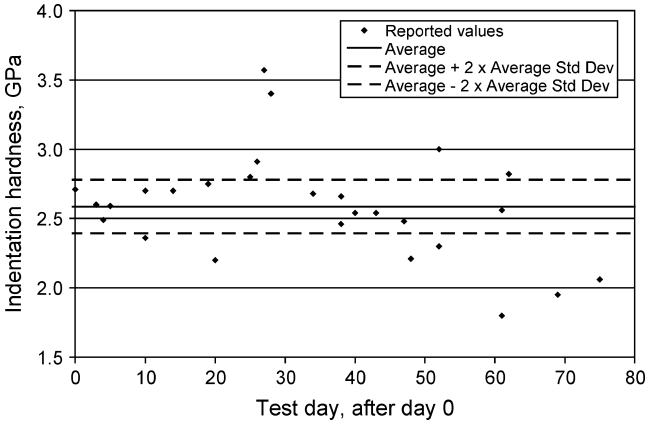

Fig. 8—Reported indentation hardness values plotted against test day, counted from an arbitrarily selected day 0. The data plotted here show that there are no statistically significant effects of specimen aging.

results of the current study. Example calculations were made based on the first set of explicit area function coefficients discussed in Reference 2 and the penetration depth and average hardness and modulus obtained here; the coefficient of the dominant term in the area function, denoted C_0, has a value of 24.65. An error of 28 pct in C_0 raises the apparent hardness from 2.6 to 3.6 GPa. This same error in C_0 raises the apparent modulus by 25 GPa. This example shows that errors of the order of 28 pct in C_0, or even larger errors in the higher-order terms of the hardness function, would be needed to explain the scatter found in the present intercomparison, if the tip area function were the actual source of the scatter.

E. *True Values?*

Mechanical properties determined from tensile tests, including yield and ultimate strength and Young's modulus, are used in structural design. The customary view at present is that nanoindentation results are useful in themselves, so their relationship to tensile results is not a serious issue. Tabor[17] estimated that the hardness should be equal to 3 times the tensile stress at a strain of 8 pct. However, the present specimen material reaches a tensile strain of only 2.3 pct (Table I). The best that can be said is that the extrapolation of the stress-strain curve (Figure 2) is not inconsistent with a stress of 860 MPa at a strain of 8 pct. The particular technique used for these microtensile tests has a history of producing low values for Young's modulus,[22] so the discrepancy between the modulus value listed in Table I and the value from indentation in Table II should not be considered problematic. The indentation value is slightly higher than the accepted polycrystalline average value for copper of 128 GPa.

The experimental procedures that contribute the most variation to the hardness values are candidates for attention in future efforts to improve the repeatability of nanoindentation measurements. Within the mount types, the scatter for cold mount was highest. The variation for labs with the longest times since force and displacement calibration was higher than the variation for those with more recent calibrations. Further, the variation among labs with new Berkovich tips was larger than that among labs with used and heavily used tips.

III. CONCLUSIONS

In an interlaboratory study to determine the precision of nanoindentation for measuring hardness and elastic modulus of metal thin films on silicon substrates, the interlaboratory standard deviation of the hardness values was 15 pct of the average hardness value, while the average reported uncertainty (1 standard deviation) among the individual participants was 4 pct of the average. Similarly, the interlaboratory standard deviation of the modulus was 19 pct, while the average reported uncertainty was 5 pct of the interlaboratory average value.

The study did not identify a likely source of the uncertainty. This study included sufficient data to examine, one at a time, the effects of several important variables, such as the manufacturers of the different instruments, the test and analysis procedures used, the condition of the indentation tip, and the time since instrument calibration. No statistically significant effects of these variables were identified. The calibration procedures for these instruments rely on certain specimens, including quartz and aluminum single crystals. Typically, each laboratory has a set of these specimens. These may be supplied by the instrument manufacturer or may be obtained elsewhere. It is widely assumed that these specimens are sufficiently consistent that instruments calibrated using these specimens in the usual way will perform identically. The current study may bring this assumption into question. Variations in indentation tip shapes, not adequately compensated by presently used procedures for determination of the tip area function, cannot be excluded as the source of the variations of the results of the current study.

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