

Multiscale Mechanical Characterization of Hybrid-Bonding Copper: From Instrumented Indentation to AFM

Nicolas A. Alderete
Material Measurement Laboratory
National Institute of Standards
and Technology (NIST)
Gaithersburg, MD, USA
nicolas.alderete@nist.gov
ORCID: 0000-0002-2145-8825

Paresh D. Daharwal
Advanced Packaging
Technology and Manufacturing
Intel Corporation
Hillsboro, OR, USA
paresh.daharwal@intel.com

Yvonne B. Gerbig
Material Measurement Laboratory
National Institute of Standards
and Technology (NIST)
Gaithersburg, MD, USA
yvonne.gerbig@nist.gov
ORCID: 0000-0002-8947-4215

Gheorghe Stan
Material Measurement Laboratory
National Institute of Standards
and Technology (NIST)
Gaithersburg, MD, USA
gheorghe.stan@nist.gov
ORCID: 0000-0001-5773-0328

Cristian V. Ciobanu
Department of Mechanical
Engineering and Materials
Science Program
Colorado School of Mines
Golden, CO, USA
cciobanu@mines.edu
ORCID: 0000-0002-8476-4467

Abstract— As transistor dimensions approach their physical scaling limits, advanced packaging has emerged as an effective solution to today’s challenging demands. Hybrid bonding (HB), in particular, has become a disruptive enabler in this space. With interconnect pitches below 10 μm and trending submicron, the influence of length-scale effects cannot be overlooked as the behavior of metals at these scales deviates from that predicted by bulk properties. In this context, understanding, modeling, and optimizing HB-systems for reliability requires knowledge of elastoplastic mechanical properties at the micro- and nanoscale.

Here, we present a framework for multiscale mechanical characterization of polycrystalline, electroplated copper (Cu) surfaces in HB-ready patterns under ambient conditions. At the microscale, we leveraged the instrumented nanoindentation technique (IIT) with spherical probes of 1 μm and 5 μm nominal radii, near the critical dimension of the metal pads. At the nanoscale, we employed contact resonance and indentation via atomic force microscopy (AFM) with spherical probe radii of less than 30 nm, enabling localized probing of nanoscale features. Using single-step indentation we first examined the stochastic, earliest onset of plastic deformation (i.e., incipient plasticity). In complement, we then harness multi-step indentation to derive indentation stress-strain (ISS) curves that reflect later stages of plasticity (i.e., developed plasticity) and from which indentation yield stress is obtained. Our experiments reveal a marked difference in the incipient plasticity of Cu, with AFM approaching the theoretical limit ($\tau_y \approx 8.7$ GPa), and IIT revealing lower strength limits ($\tau_y \approx 0.75$ GPa). ISS curves show an indentation yield stress of ($\sigma_y \approx 0.64$ – 0.83 GPa), comparable to results from micropillar compression. Our findings emphasize the importance of length-scale relevant metrology, which is critical for modeling the response of Cu under different loading scenarios (e.g., nanoscopic contact at bonding, thermal warping). Moreover, our novel multimodal AFM methodology provides an effective approach for elastoplastic characterization aligned with the industry’s drive for smaller pitches, in-situ testing, and the widespread availability of AFM technology. Concurrently, to facilitate the adoption of our framework, we introduce an open-source software for the analysis of micro- and nanoscale spherical indentation experiments.

Keywords— *advanced packaging, hybrid bonding, microscale and nanoscale plasticity, size-effects, atomic force microscopy, nanoindentation.*

I. INTRODUCTION

Hybrid bonding (HB), the vertical stacking of chiplets through direct dielectric-to-dielectric and metal-to-metal interconnections, has established itself as a frontrunner in the race for high-density, low-latency, and efficient integration across memory, logic devices, photonics, and chiplets [1]. The disruptive promise of hybrid bonding, projected to reach a global market size of \$633.9 million by 2032 [2], is tempered by technical challenges primarily related to process yield and product reliability. These challenges can be traced to mechanical and thermomechanical stresses that arise in the materials at a range of spatial and temporal scales, during critical fabrication stages (e.g., chemical-mechanical polishing (CMP), annealing, bonding), handling operations (e.g., pick-and-place), and during normal operation (e.g., thermal and power cycling, vibration) [3], [4]. Under the inevitable presence of stresses (strains), understanding their magnitude, over what lengthscales and features they act, and their resulting effect on the constituent materials becomes critical to ensuring the mechanical reliability of devices (Fig. 1). Accordingly, this necessitates knowledge of accurate, temperature- and lengthscale-dependent, elastoplastic properties of the materials which, with HB-interconnect pitches approaching sub-10 μm dimensions, cannot be presumed to be those of the bulk or macroscale material. Despite mechanical size-effects being known for almost a century now [5], metrology for nanomechanical properties of materials continues to be one of the most overlooked issues in the community, as emphasized by IEEE’s International Roadmap for Devices and Systems (IRDS) [6]. The scarcity of precise material properties, largely due to the challenges associated with near- and atomic-scale measurements, limits the predictive capability of modeling and simulations, thereby hindering our understanding of the underlying physics in HB, as well as other heterogeneous integration approaches [7].

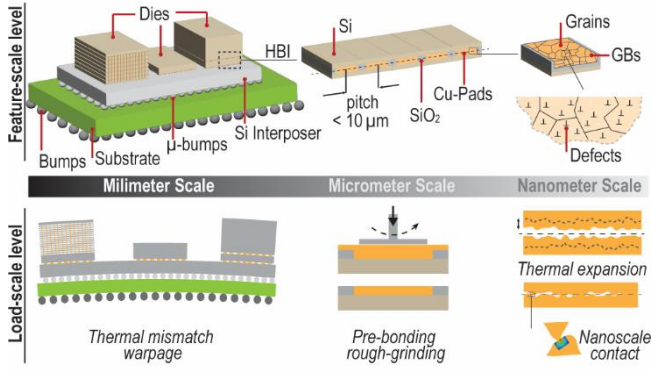


Fig. 1. Overview of spatial multiscales (millimeter-, micrometer, and nano-scale) associated with advanced packaging technologies at the component/feature level (top panel) and the mechanical load level (bottom panel).

To address these knowledge gaps, we built upon our previous work [8] and here report on a multiscale, mechanical characterization framework that quantifies and compares the elastic-plastic response of polycrystalline, electroplated copper (Cu) surfaces in HB-ready patterns (i.e., prior to bonding). Specifically, we investigated two distinct spatial scales, spanning two orders of magnitude and under ambient conditions, with measurements conducted directly on the HB-patterns (Fig. 2a). Using spherical probes with nominal radii of 1 μm and 5 μm , the first lengthscale was probed via the instrumented indentation technique (IIT). With probe radii comparable to the critical feature size of the HB-pattern, IIT provides a window to the average, microscale behavior of the Cu-pads. The second lengthscale examined is enabled by multimodal, interferometric atomic force microscopy (AFM) with spherical probe radii in the order of tens of nanometers [8]. As such, access to the nanoscale behavior of the Cu-pads is gained with localized probing inside individual grains and in the vicinity of grain boundaries. Single-step indentation (SSI) and multi-step indentation (MSI) experiments (i.e., single/multiple load-unload cycles) are performed with both techniques to shed light on incipient plasticity stresses (i.e., the onset of plasticity) and stress-strain behaviors, respectively, which can be used in multiscale and scale-aware modeling. Unlike other techniques, such as micropillar compression, microbeam bending or laser ultrasonic techniques, our measurements do not require extensive sample preparation or specialized setups. Moreover, by being in-situ, they can be utilized in near-line facilities for control check-ups and corrective manufacturing adjustments.

II. METHODS

A. Test Materials

Mechanical tests were conducted on samples cut from wafers featuring hybrid bonding SiO_2/Cu patterns (Intel Corporation, Hillsboro, OR, USA) that had undergone CMP. For IIT experiments with 1 μm nominal tip radius and AFM experiments, square HB patterns were probed, whereas for IIT experiments with the 5 μm nominal tip radius, pillar-like HB patterns were probed (Fig. 2a). In all cases, the electrochemically deposited Cu-pads exhibit polycrystalline features with micrometer-sized grains.

B. Instrumented Nanoindentation

IIT experiments were performed using a Nano Indenter XP (KLA, Milpitas, CA) with sphero-conical sapphire

indenter tips (Micro Star Technologies Inc., Huntsville, TX) of nominal tip curvature radii of 1 μm and 5 μm . The actual radii of the IIT tips were obtained from analyzing indentations on three reference materials: fused silica, c-plane sapphire, and silicon (100), following established calibration procedures [9].

SSI experiments were done under force-control following the imposed loading profile shown in Fig 2c. This profile consisted of a ramp to the maximum load, a dwell segment, an unloading ramp to 10 % of the maximum load, and a hold segment for thermal drift rate determination prior to final unloading. Alternatively, multi-step indentation experiments were performed under force-control, using a cyclic loading profile that consisted of a ramp to the maximum cycle load, a dwell period, a partial unloading ramp to a specified percentage of the maximum cycle load, and a subsequent reloading ramp to a maximum cycle load twice the previous maximum cycle load (Fig. 2e). A total of 10 steps were executed at each indentation site, with the final cycle including an additional hold for thermal drift rate determination. Both experimental modalities were conducted on several Cu pads.

The load-indentation depth (P - h) data from experiments on reference materials and Cu pads were first corrected for thermal drift, machine compliance, and zero-point offset. The data were subsequently analyzed using the in-house developed software SpherIndenter [10] (more details on SpherIndenter can be found in the Results section). The full set of parameters for the IIT experiments is provided in Table I.

TABLE I. EXPERIMENTAL PARAMETERS FOR SSI AND MSI EXPERIMENTS VIA IIT AND AFM

Indentation parameter	IIT		AFM	
	SSI ^a	MSI	SSI	MSI
Avg. tip curvature radius (nm)	5075.4 1848.7	5075.4	12.3	22.0
Maximum test load (μN)	5000 250	1000	18	15
Dwell time (s)	5	5	NA	NA
Hold time thermal drift segment (s)	75	75	NA	NA
Loading/unloading rate ($\mu\text{N/s}$)	200 20	20 50	0.036	0.05
Number of cycles	1	10	1	12
Load increase ^b (%)	NA	200	NA	20
Partial unload ^c (%)	NA	90	NA	25
Sampling rate (Hz)	10	10	500	500
Number of tests	10 22	- 18	126	20

^a. Where two values are presented, the top one corresponds to SSI experiments with the larger tip.

^b. Load increase relative to maximum cycle load of previous cycle.

^c. Partial unloading relative to maximum cycle load.

C. Multimodal Atomic Force Microscopy

AFM experiments were performed using an interferometric, environmental Vero AFM (Oxford Instruments/Asylum Research, Santa Barbara, CA, USA). Exploiting the versatility of AFM, three different modes were leveraged: topographic scanning, contact resonance (CR-AFM), and single- and multi-step indentation. Unlike conventional AFMs that use optical beam deflection, the Vero AFM employs interferometric principles to measure true vertical displacements based on the wavelength of the light. Similarly, the instrument's low noise floor eliminates the need for extra hold segments to correct for thermal drift [11]. Supplementary experimental details on the multimodal

AFM characterization undertaken can be found in more detail on [8].

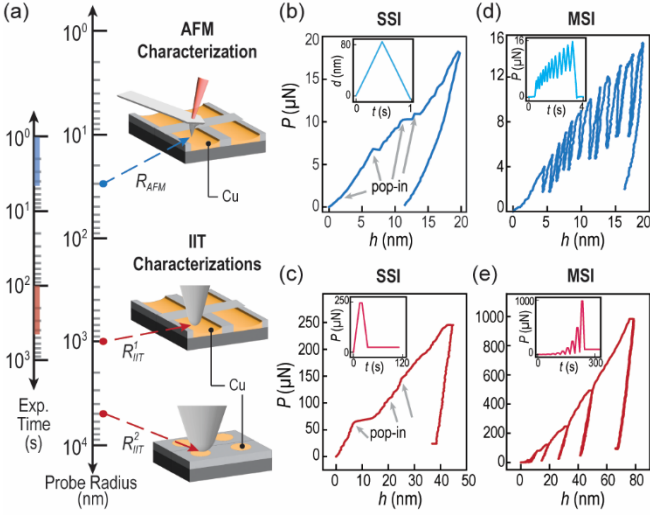


Fig. 2. Indentation experiments via AFM and IIT. (a) Length and time scales of combined AFM-indentation and IIT characterization of hybrid bonding-ready Cu. Characteristic force-indentation depth P - h curves with imposed time-load profiles (insets) for SSI (b) AFM (top) and (c) IIT experiments (bottom). Characteristic force-indentation depth P - h curves with imposed time-load profiles (insets) for MSI (d) AFM (top) and (e) IIT (bottom) experiments.

AFM-indentation measurements were performed using silicon cantilevers with conospherical diamond tips (Adama Innovations Ltd., Dublin, Ireland), with a spring constant k_c in the 508 N/m to 528 N/m range. The shape of the tips was regularly assessed against a test grating (K-TEK Nanotechnology, Wilsonville, OR) to check for wear or damage. Given that AFM tips are susceptible to damage when imaged under an electron beam, the probe tip radii for SSI and MSI were obtained concurrently with fits of the loading and unloading curves. P - h curves were derived from the cantilever deflection voltage (V) and piezo displacement (d) data as $P = k_c \cdot h$ and $h = d - V \cdot S$, respectively, with S the deflection sensitivity of the cantilever in nm/V.

SSI AFM experiments were conducted following the imposed loading profile shown in Fig 2b. This profile included an approach segment, a ramp to maximum piezo-displacement, a full unloading ramp and final retraction. Indentations were performed in a grid pattern on two different copper pads. MSI AFM experiments were performed on a single copper pad under force-control, following a 12-cycle loading profile. Each cycle consisted of a ramp to a maximum cycle load, followed by a partial unloading to 25 % of the maximum cycle load. The latter was chosen to maintain conformal contact between the tip and the sample (Fig. 2d). Topographic imaging of selected areas within the HB-patterns was performed in non-contact AFM mode, both before and after AFM indentations, using the same tip for both operations. The full set of experimental parameters used in AFM-indentations is reported in Table I.

Quantitative nanomechanical mapping of elastic modulus was performed using CR-AFM with DT-NCHR probes (Nanosensors, Neuchâtel, Switzerland). These probes have a resonance frequency in air $f_{l,air}$ around 415 kHz and a spring stiffnesses k_c of approximately 9 N/m. The shift in CR frequency (f) of the AFM cantilever was tracked on both a reference (SiO_2) and an unknown (Cu) sample (Fig. 3a),

through the photothermally excited dual AC mode, with its two frequencies kept apart at a constant difference of 120 kHz. A maximum force of 750 nN was applied between cantilever and samples to establish elastic contact. The mapping was conducted over areas of $25 \mu\text{m} \times 25 \mu\text{m}$ on both SiO_2 and Cu, with a resolution of 512 pixels $\times$ 512 pixels.

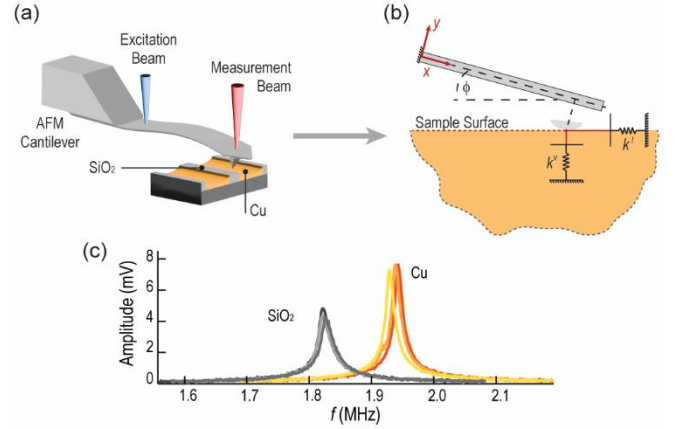


Fig. 3. CR-AFM on SiO_2/Cu patterns. (a) Schematic of CR-AFM measurements on hybrid bonding pads with photothermal excitation of the cantilever; (b) schematic diagram illustrating the analytical model for the system in (a) used to relate the cantilever dynamics to the mechanical properties of the sample; (c) measured CR frequency spectra obtained from sweeps over SiO_2 (reference material) and Cu (unknown material properties) regions.

D. Analysis

SSI AFM and IIT curves were first examined for displacement bursts, also known as pop-ins (Fig. 2b-c), which are manifestations of changes within the material as it is being indented. In the context of plasticity, the first pop-in is of particular importance as it signals the departure from the elastic regime and the onset of plasticity. In this context, the classical Hertz model for elastic contact of a paraboloid with a half-space can be used to model the load-indentation depth (P - h) response up to the first pop-in [12]:

$$P = (4/3)E_{eff} R^{3/2} h^{3/2}, \quad (1)$$

where E_{eff} and R are the effective modulus and the tip curvature radius, respectively. The effective modulus is defined as:

$$E_{eff} = (1/M_{Sample} + 1/M_{Tip})^{-1}, \quad (2)$$

where $M = E/(1-\nu^2)$ is the indentation modulus, with E the Young's modulus and ν the Poisson's ratio. Moreover, under the conditions of Hertzian elastic contact, the maximum shear stress $\tau_{max}^{pop_1}$ in the indented material at the first pop-in load (P^{pop_1}) can be calculated as [12]:

$$\tau_{max}^{pop_1} = (0.31/\pi) (E_{eff}/R)^{2/3} (6P^{pop_1})^{1/3} \quad (3)$$

Consistent with the notion that plasticity in ductile materials occurs due to slippage along crystallographic planes, the resulting shear stress ($\tau_{max}^{pop_1}$) thus obtained represents the yield stress of the material. Due to the stochastic occurrence of pop-ins, a probability distribution is necessary to capture the variability in the data and to extract a robust median value for the incipient yield ($\tau_{50\%}^{pop_1}$). By modeling the empirical cumulative distribution function

(F_{ecdf}) of these events [13], the activated volume (v^*) during the first pop-in event can be estimated:

$$F_{ecdf} = 1 - \exp[-(\eta k_B T / \dot{\tau} v^*) \cdot \exp(v^* \tau_{max}^{pop} / k_B T)] \quad (4)$$

where k_B , T , v^* , η , τ , $\dot{\tau}$ are Boltzmann's constant, temperature, activation volume, defect nucleation rate, and the shear stress and rate, respectively.

For multi-step IIT and AFM indentations, emphasis is placed on analyzing the unloading segments of these indentations. Under the assumption of elastic unloading, the unloading segments (q) can be modeled using the more general Hertz expression:

$$P^{(q)} = (4/3) E_{eff} R_{eff}^{1/2} h^{(q)3/2} \quad (5)$$

where $R_{eff}^{(q)}$ accounts now for the fact that the original surface has been plastically deformed and is no longer flat. Indentation stress-strain curves can be derived from the unloading segments by obtaining indentation stress-strain pairs as [14]:

$$(\epsilon_{ISS}^{(q)}, \sigma_{ISS}^{(q)}) = (4h_t^{(q)} / 3\pi r_c^{(q)}; P_t^{(q)} / \pi r_c^{(q)2}) \quad (6)$$

where $P_t^{(q)}$, $h_t^{(q)}$ are the maximum load and the depth at the maximum load of each segment, respectively. $r_c^{(q)}$ is the radius of contact calculated as $r_c^{(q)} = (3P_t^{(q)} R_{eff}^{(q)} / 4E_{eff})^{1/3}$. If the resulting stress-strain pairs encompass both the elastic and plastic regimes, an indentation yield stress can be determined by computing the intersection of the elastic and plastic indentation curves.

The conversion of CR-AFM images into moduli maps was achieved using a procedure based on a single reference material, which in this case was SiO₂. Essentially, we modeled the dynamics of the physical system by considering the cantilever as a uniform rectangular silicon beam, tilted with respect to the sample surface ($\theta = 12^\circ$), and clamped at its base and spring-coupled at the tip position (Fig. 3b). The elastic tip-sample contact with the sample is represented by vertical and lateral springs, with $k^l = 0.85 \cdot k^v$. These boundary conditions shape the solution of the dispersion equation (i.e., frequency-wavenumber relationship) from the flexural vibration of the system [15] and provide a solution for the vertical contact stiffness (k^v). The contact stiffnesses measured for Cu (k_{Cu}^v) and SiO₂ ($k_{SiO_2}^v$), were then used to determine the indentation modulus of Cu in relation to the indentation modulus of the reference sample ($M_{SiO_2} = 74.1$ GPa) and that of the tip ($M_{Tip} = 1250$ GPa):

$$M_{Cu} = [M_{SiO_2} (k_{Cu}^v / k_{SiO_2}^v)^{3/2}] [1 + (M_{SiO_2} / M_{Tip}) (1 - (k_{Cu}^v / k_{SiO_2}^v)^{3/2})]^{-1}, \quad (7)$$

where the vertical stiffnesses (k_{Cu}^v , $k_{SiO_2}^v$) are functions of the resonant frequencies of the cantilever in contact with each of these materials.

E. Statistical and Regression Analysis

Continuous distributions from data histograms were obtained using Gaussian kernel density estimation. Curve fittings based on the dependencies given by (1,4,5) were performed using the method of nonlinear least-squares.

Stress-strain pairs derived from (5,6) were discarded if the coefficient of determination (R^2) from the fit of (5) was $R^2 < 0.5$. Unless otherwise stated, uncertainties reported herein are expressed as the standard deviation of the experimental measurements.

III. RESULTS AND DISCUSSION

A. Indentation Modulus

The local elastic modulus of the Cu pads was determined using CR-AFM mapping (Fig. 4a). Fig. 4b presents the indentation modulus profile from a randomly selected line scan across adjacent SiO₂ and Cu regions. Additionally, Fig. 4d displays the distribution of indentation moduli obtained from the shaded region in Fig. 4a. The CR-AFM measurements show minimal variation over the SiO₂ regions, with an indentation modulus of $M_{SiO_2} = 74.1 \text{ GPa} \pm 4.1 \text{ GPa}$. Conversely, a higher variation, with $M_{Cu} = 136.4 \text{ GPa} \pm 11.5 \text{ GPa}$, is observed on the area corresponding to the Cu pad. The statistical variability observed in the modulus of SiO₂ is attributed to the inherent fluctuations in the contact area during CR-AFM mapping. In comparison, the variation in the modulus of Cu is due to its polycrystalline structure, as evidenced by the AFM topographic image shown in the inset of Fig. 4c.

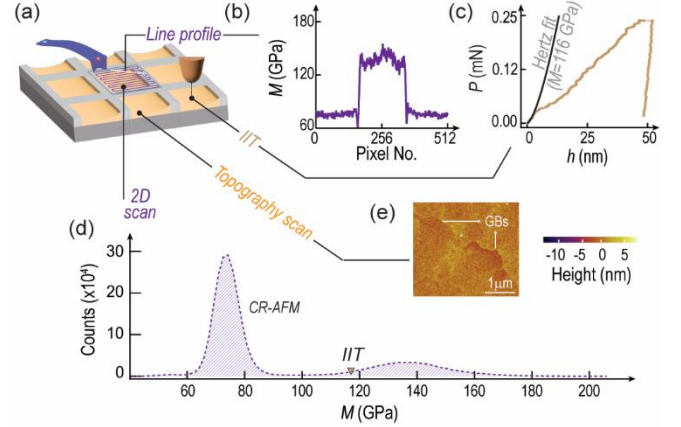


Fig. 4. Indentation modulus characterization via CR-AFM and IIT. (a) Schematic of HB-ready pattern, highlighting line and 2D scans performed with CR-AFM; (b) indentation modulus along a single line scan from (a); (c) IIT indentation modulus obtained by fitting of the elastic portion of the loading curve from SSI experiments with nominal probe radius 1 μm ; (d) distribution of the CR-AFM indentation modulus from the shaded region in (a) and the average IIT indentation modulus; (e) AFM topography of a region within a Cu pad, with two grain boundaries highlighted.

In IIT experiments, the indentation moduli were obtained by fitting the elastic portion of the loading curve using (1-2). The average value for the indentation modulus thus obtained is marked on the histogram of Fig. 4d. Though within the bounds of the statistical distribution from CR-AFM, the average indentation modulus from IIT measurements appears to be biased to the left of the CR-AFM central value (116 GPa compared to 136.4 GPa). This discrepancy can be attributed to the significant difference in probe radii between AFM and IIT experiments. The smaller probes (and contact areas) of AFM indentations provide sensitivity to nanoscale variations, and enable moduli measurement for individual grains, thereby capturing orientation-dependent moduli fluctuations in the polycrystalline Cu. The IIT measurements conducted in this study are insensitive to variations in crystallographic orientation due to the larger sample area probed, and hence reflect orientation-averaged values of indentation modulus.

Noteworthy, the throughput of the two techniques differs considerably, with IIT averaging over tens of indentations, and CR-AFM averaging measurements over the entire scan size (512^2 pixels²).

B. Onset of Plasticity

Force-indentation depth curves from SSI AFM and IIT experiments exhibit shared characteristics despite probing different spatial scales. In all cases, the loading segments present a jagged appearance, consistent with the occurrence of pop-in events; while the unloading segments appear smooth, in line with purely-elastic unloading (Fig. 2a-c). As briefly mentioned in the Analysis section (II.D), the first pop-in event is generally accepted to represent the departure from elastic behavior when indenting metallic materials (provided neither fracture nor phase transformations are present). With AFM and IIT indentation curves both presenting a first pop-in, the question naturally arises as to which one corresponds to the initiation of plastic deformation. The answer is both of them and the key to it resides in the multiscale character of plasticity, which is elicited by the use of nano- and microscale indenter probes in our experiments. Accordingly, the size of the indenter radius determines the interaction between the stress field imposed on the indented material and its underlying structure (e.g., dislocations, defects, grain boundaries). In other words, given an equal density of pre-existing defects, a smaller probe radius pressing on a material has a smaller probability of activating pre-existing defects, while larger probes tend to create larger stress fields which are more likely to encounter existing defects, hence making it easier to initiate plasticity (Fig. 5a). As such, the onset of plasticity can be generally categorized into three deformation stages: a homogeneous dislocation nucleation stage, a heterogeneous dislocation nucleation stage, and a bulk (or developed) plasticity stage [16].

Consistent with the foregoing, our SSI experiments reveal a marked difference between the incipient plastic yield stress obtained with the microscale IIT probes and the nanoscale AFM probes. As shown in Fig. 5b, the empirical cumulative distribution function (ecdf) of the stochastic yield stress at the first pop-ins ($\tau_{max}^{pop_1}$) differs by an order of magnitude between those from IIT and those from AFM experiments. In particular, AFM indentations exhibit a median incipient yield stress of $\tau_{50\%}^{pop_1} = 8.7$ GPa, which is close to the theoretical strength limit of Cu (estimated as $G/2\pi$, with G the shear modulus; for Cu $\langle 111 \rangle$ $G \approx 48$ GPa). In contrast, IIT experiments display a lower median incipient yield stress of $\tau_{50\%}^{pop_1} = 0.75$ GPa, with virtually no difference between the distributions observed for the experiments with the 1 μm and 5 μm nominal tip radii. The ratio of the activation volumes from (4) to the atomic volume of Cu ($1.182 \times 10^{-2} \text{ nm}^3$) are 0.2, 3.55 and 6.2 for AFM and IIT with probes of 1 μm and 5 μm nominal radii, respectively.

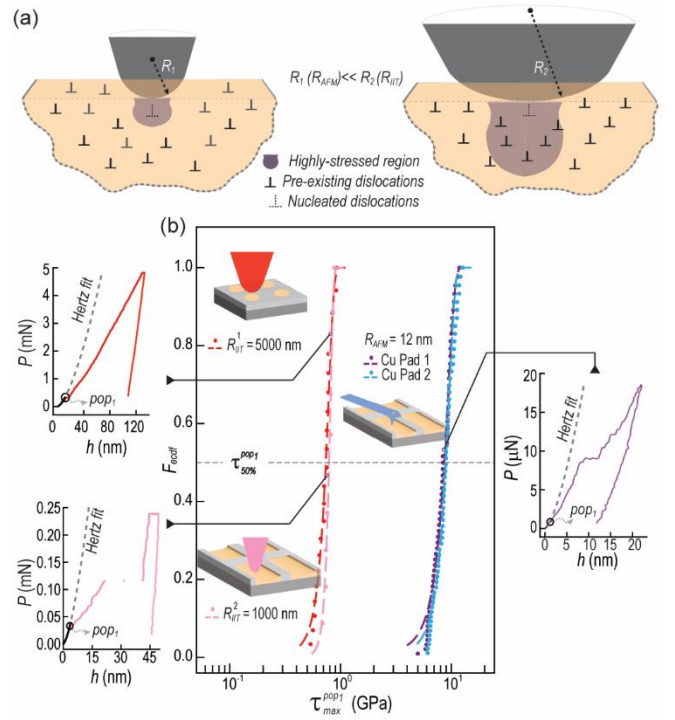


Fig. 5. Incipient plasticity results from SSI via AFM and IIT. (a) Schematic illustration of the origin of size effects in the context of plasticity induced by indentation; (b) empirical cumulative distribution functions for the multiscale incipient yield stress obtained from the analysis of the first pop-in in SSI.

Further dissection of the indentation curves, more specifically focusing on the areas underneath the loading and unloading segments, allows to compute the plasticity index of the tested material. This metric, which measures the ratio between the plastic work and the total work ($\eta = W_p/W_T$), informs on the degree of recoverability of the material following unloading. As such, an index value of $\eta \approx 1$ corresponds to a high level of permanent deformation, while a value of $\eta \approx 0$ indicates highly elastic deformation. In our experiments on square copper pads, the plasticity index from AFM indentations is centered around $\eta \approx 0.6$, while for IIT indentations it is centered at a higher value of $\eta \approx 0.9$ (Fig. 6). The observed difference can be rationalized in terms of the mechanisms underlying recovery, which generally involve repositioning of dislocations, as well as the differing degrees of localized versus distributed deformations elicited by AFM and IIT, respectively.

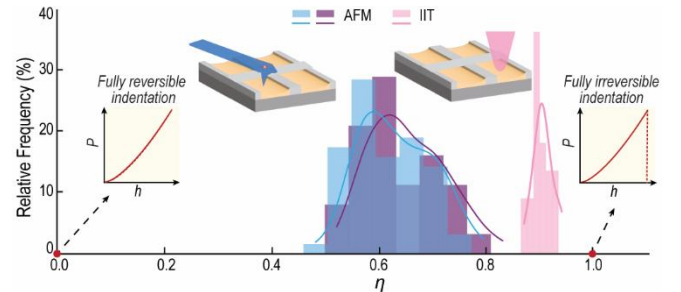


Fig. 6. Statistical distribution of plasticity indices from the analysis of the areas under the loading and unloading segments of the indentation curves obtained on square copper pads.

While our results on incipient yield and recoverability provide valuable insights, we acknowledge the need for additional experiments and multiscale simulations to further

elucidate the underlying mechanisms. Along these lines we highlight the use of transmission electron microscopy for dislocation imaging, and molecular dynamics, discrete dislocation dynamics or crystal plasticity finite elements for computational modelling [17].

C. Indentation Stress-Strain Curves

Our MSI experiments shed light on the material behavior of hybrid bonding Cu considerably after the onset of plasticity (i.e., in the bulk/developed plasticity regime). This is initially apparent upon initial inspection of the MSI force-indentation depth curves. Both the AFM and IIT curves in Fig. 7a-b display a clear hysteresis, characterized by non-overlapping loading and unloading segments, which is a hallmark of inelastic deformation. In conformity with this, the indentation stress-strain pairs derived from the analysis of unloading segments will correspond to the developed (bulk) plasticity regime. Moreover, the associated constitutive parameters, namely the median indentation yield stress ($\sigma_{0.2\%}^{YS}$) and the plastic hardening slope (E_T) will reflect more homogenized plastic characteristics commensurate with a collective movement of dislocations (i.e., unlike local nucleation-type events).

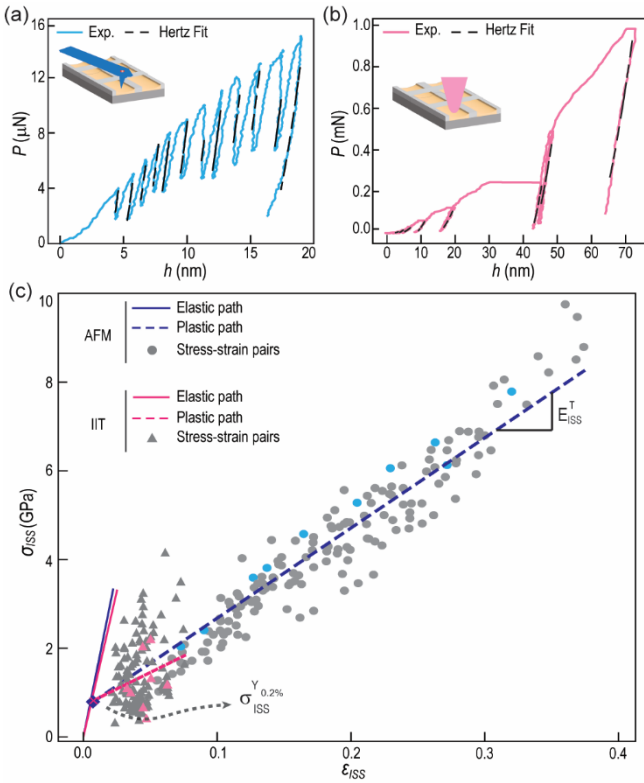


Fig. 7. MSI experiments for derivation of indentation stress strain curves. (a) MSI AFM curve with Hertzian fits on the unloading segments; (b) MSI IIT curve with Hertzian fits on the unloading segments; (c) combined ISS curve from the analysis of MSI AFM and IIT experiments.

Fig. 7c presents the ISS pairs obtained from MSI experiments performed via AFM and IIT. Pairs from IIT fall within the lower end of the indentation strain range analyzed, while those from AFM occupy the upper range. Linear regression applied to each dataset produces estimates for the hardening slope and the indentation yield stress: $E_{IIT}^T=14.8$ GPa and $\sigma_{IIT}^{Y_{0.2\%}}=0.83$ GPa from IIT experiments, and $E_{AFM}^T=20.3$ GPa and $\sigma_{AFM}^{Y_{0.2\%}}=0.64$ GPa from AFM experiments. We note that the pairs from the IIT experiments

exhibit a wider spread compared to those from AFM. This variability is attributed to the occurrence of large pop-ins throughout the different cycles of the MSI tests (see large pop-in in Fig. 7b) [14].

The indentation yield stresses obtained in this study are consistent with recently reported values (0.2 GPa – 0.6 GPa) from micropillar compression tests on various types of copper [18]. However, it is important to note some key differences between micropillar and indentation experiments that are relevant to this comparison. Among these, is the fact that indentation stress-strain curves reduce a complex triaxial state of stress to a uniaxial one, whereas micropillar compressions naturally impose a uniaxial stress state. Furthermore, the localized deformation imposed in our small-probe IIT and AFM experiments generates large strain gradients, which are known to contribute to enhanced hardening [19].

As previously mentioned, additional experiments and simulations are deemed critical to gaining a better understanding of the mechanics underlying these phenomena.

D. SpherIndent Analysis Software

The incipient and developed plasticity characterizations detailed in the preceding sections, are uniquely facilitated by the use of spherical probes for indentation. Unlike indenter probes with flat or pyramidal geometries, spherical and sphero-conical indenter probes generate stress fields of a non-self-similar nature, meaning the induced indentation stresses increase gradually with applied load (and indentation depth). In SSI experiments, this allows for clear observation of the transition between elastic and plastic regimes, whereas in MSI experiments, it enables the application of a formalism to obtain stress-strain curves.

Despite these merits, accessible (i.e., free, open and user-friendly) and comprehensive software tools capable of performing calibration, conditioning, and analysis of spherical indentation data are currently lacking. This, in turn, curbs the adoption of indentation-based elastoplastic metrology.

Against this background, we developed SpherIndent [10], which is a Python-based, open-source software (OSS) for calibration, analysis and visualization of spherical indentation experiments (Fig. 8) for users of different proficiency levels. For the non-specialist, the software allows a plug-and-play modality, whereas for more advanced users its modular, OSS-character allows flexible customization. Furthermore, SpherIndent has visualization tools tailored to each particular experiment type (i.e., SSI, MSI) and leverages Python's pickle files for efficient, serialized data storage and management. The SpherIndent software includes:

- i. calibration routines to determine machine compliance based on indentation of reference materials,
- ii. calibration routines to determine the tip geometry (i.e., tip radius and cone angle) via tip-area function calculation or tip image analysis,
- iii. data conditioning routines to eliminate thermal drift, compute zero-point offset, and correct for machine compliance effects,
- iv. analysis routines to extract various material metrics of interest (e.g., Hertz and Oliver-Pharr elastic moduli, dissipation indices, maximum shear stress at first pop-

in, stress-strain pairs) from SII and MSI indentation tests.

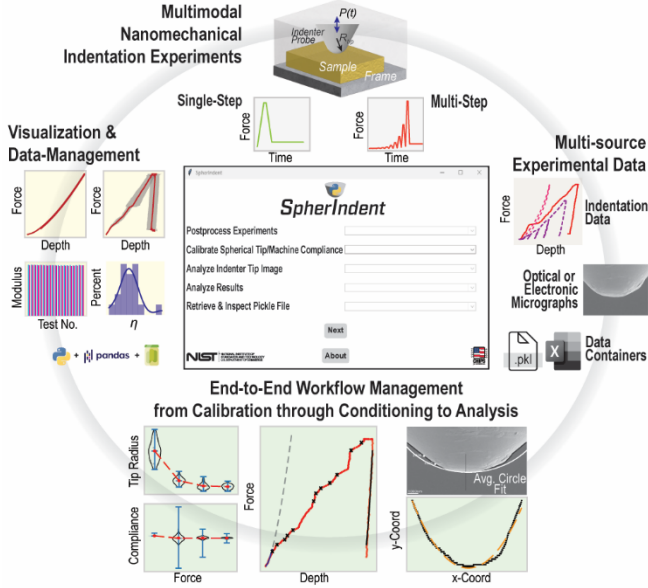


Fig. 8. Overview of SpherIndNet: a Python-based, open-source, user-friendly software for calibration, analysis and visualization of multimodal and multiscale spherical indentation experiments.

The different capabilities of this software, appropriately tailored to the particulars of each experimental technique (e.g., AFM does not require thermal drift correction), were leveraged to produce the diverse set of indentation results presented here.

E. Associated Data and Software

The data underlying this manuscript are publicly available in NIST Public Data Repository [20]. The current version of SpherIndNet can be found in the NIST GitHub repository [21].

IV. CONCLUSIONS

This study underscores the importance of length-scale relevant metrology for understanding the mechanical behavior of materials at small scales. The initiation of plastic deformation at the component level (Cu-pads) has been shown to occur at significantly lower stress levels than at the sub-component level (individual grains within the Cu-pads). This difference is linked to the local interaction of the stress fields induced in the material with the available material defects. Beyond the incipient plasticity stage, we arrive at a yield stresses that are reflective of less local events and pertaining to the bulk plasticity stage. These observations carry important implications for the allowable (critical) stress levels to consider when performing stress analyses for reliability and design purposes [22] since most failure modes (e.g., mechanical and thermomechanical fatigue [23], interfacial fracture and debonding [24]) involve elastoplastic phenomena operating at a variety of lengthscales. While our measurements were conducted on Cu, the underlying principles are expected to be relevant and equally applicable to the development of novel, nanoscale interconnect metallization approaches, an area of ongoing research and development.

Although the current interconnect dimensions bring IIT to operate near its spatial limits, we have demonstrated the potential of using multimodal AFM for elastic-plastic nanomechanical mapping. Given that AFM is already

extensively used for in-situ critical dimension metrology, the experimental protocols introduced here could be implemented to maximize the information output from the instrument, thus reducing metrology cycle times (i.e., obviating multiple instruments and dedicated sample preparation). Both aspects are well-aligned with pressing metrology demands from the sector, and the future vision of metrology as outlined in various technology roadmaps [6], [25]. The presented SpherIndNet software was conceived and developed to further bolster the application of the protocols herein introduced.

This study lays the groundwork for further research on a number of critical avenues. Leveraging the high-temperature capabilities of modern equipment [26], the effect of temperature on the mechanical properties of advanced packaging materials can be investigated emulating in-operando conditions. Additionally, the high-throughput capabilities offered by AFM and showcased in this work can be expanded along the lines of data-driven and big-data-generating experiments [27]. Lastly, we emphasize the need for a more comprehensive approach that incorporates micro- and nanostructural imaging together with simulations to gain deeper mechanistic insights.

ACKNOWLEDGMENT

This work was performed with funding from the CHIPS Metrology Program, part of CHIPS for America, National Institute of Standards and Technology, U.S. Department of Commerce.

Certain commercial equipment, software, instruments, and materials are identified in this document. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products identified are necessarily the best available for the purpose.

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