

ATOMIC FORCE MICROSCOPY-BASED MODES FOR NANOSCALE ELASTIC-PLASTIC CHARACTERIZATION OF MATERIALS USED IN HYBRID BONDING

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ABSTRACT

One of the most promising techniques for integration in advanced packaging is hybrid bonding, which connects dies and wafers without the need for solder bumps. This method enables a higher density of interconnects with a pitch scale of less than 10 micrometers. Additionally, hybrid bonding facilitates 3D stacking, increases bandwidth, and enhances overall system optimization.

However, measuring properties at the length scale of hybrid bonding structures presents significant challenges. These difficulties stem from limited access to the features and structures being measured, the complex adaptation of established macroscale measurement techniques, and the intricacies involved in analyzing these measurements.

The Atomic Force Microscope (AFM) is one of the most versatile tools available for directly measuring various material properties at the nanoscale. It can probe the localized mechanical response of a wide range of materials, including those used in the semiconductor industry. In this work, we demonstrate several AFM mechanical characterization modes applied to SiO₂/Cu hybrid-bonding-ready patterns. By utilizing three AFM modes—Contact-Resonance AFM, Single-Step AFM indentations, and Multi-Step AFM indentations—we effectively map the elastic-plastic response at the nanoscale for these structures. These types of measurements and corresponding data are essential for enhancing predictive manufacturing in hybrid bonding applications.

Although our measurement methods are demonstrated on copper prior to hybrid bonding, they can be applied to any other relevant material systems for which modeling mechanical processes at the nanoscale needs to include the effect of fabrication on material properties, such as temperature and stress.

Key words: multimodal AFM metrology, nanoscale elastic-plastic characterization, materials for hybrid bonding.

INTRODUCTION

The main steps involved in hybrid bonding are relatively straightforward: aligning two mirrored dielectric-copper patterned surfaces, bonding the dielectric components at room temperature, and then fusing the copper pads at elevated temperatures. However, due to size constraints and differences in thermomechanical properties between the dielectric and copper, the heterogeneous integration based on hybrid bonding has not yet reached its full potential or gained widespread market adoption. Therefore, developing measurement methodologies to optimize the hybrid bonding processes is both relevant and timely for the semiconductor industry [1, 2].

To this end, we developed a nanoscale elastic-plastic characterization method performed at room temperature on electroplated copper surfaces arranged in hybrid-bonding-ready patterns [3, 4]. This methodology combines several AFM modes that provide various elastic and plastic characteristics of materials at the nanoscale. With high spatial resolution and in the same selected area of interest, we demonstrated the use of Contact-Resonance AFM along with Single-Step and Multi-Step AFM indentation techniques. This multimodal approach allowed us to characterize the mechanical heterogeneity of the material, quantify nanoscopic yield stress statistics, and derive indentation stress-strain curves. From these measurements, we clarified the mechanisms of early plasticity and determined the elastoplastic constitutive response of polycrystalline copper, including parameters such as elastic modulus, yield stress, and strain-hardening slope. The developed metrology underscores the utility and versatility of using AFM for characterizing nanoscale mechanical properties, which are

crucial for the development of heterogeneous integration in advanced packaging.

CONTACT-RESONANCE AFM

Contact-Resonance Atomic Force Microscopy (CR-AFM) is a dynamic mode of AFM that relates changes in the resonance frequency of the cantilever to the contact stiffness between the tip and the sample [5]. The resonance frequency in this context depends on both the contact geometry—including the contact radius under applied loads—and the elastic moduli of the tip and the sample. By conducting a proper contact mechanics analysis, the measured contact resonance frequencies can be converted into the elastic modulus of the sample being probed [6, 7]. In recent years, significant efforts have been devoted to customizing CR-AFM measurements for various materials and structures relevant to the semiconductor industry [8, 9]. A key feature of CR-AFM is its ability to perform measurements in either point mode or mapping mode, which allows for elastic modulus measurements with a spatial resolution of just a few nanometers. This capability is especially important for characterizing both in-plane and out-of-plane properties of heterogeneous materials.

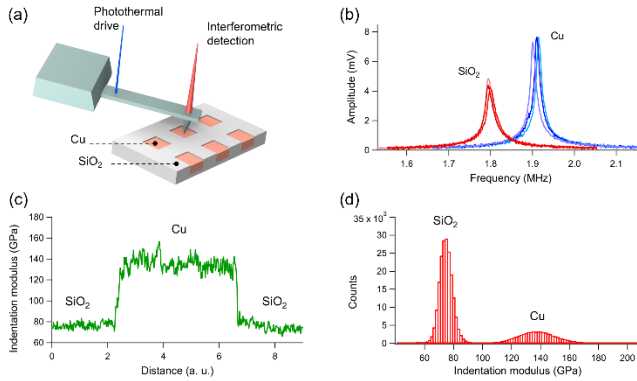


Figure 1. (a) CR-AFM measurements over SiO₂/Cu pads. (b) Frequency sweeps in CR-AFM showing the difference in elastic response between SiO₂ and Cu. (c) Indentation modulus variation across SiO₂ and Cu regions. (d) Histogram of the indentation modulus calculated from a CR-AFM scan over SiO₂/Cu pads.

We recently used CR-AFM to quantify the elastic modulus of SiO₂/Cu patterns from a die that is ready for hybrid bonding. This was done using a Vero AFM (Oxford Instruments/Asylum Research, Santa Barbara, CA, USA) equipped with a photothermal drive, commonly referred to as the blue drive, which actuates the cantilever from its base (see Figure 1a). The system also includes interferometric detection for measuring the vertical deflection of the cantilever. Individual frequency spectra around the CR frequency for both SiO₂ and Cu regions are illustrated in Figure 1b. To account for potential variations surrounding these CR frequencies, we performed a CR map across the SiO₂/Cu patterns in dual-frequency track mode. An example of CR frequency tracking across the SiO₂/Cu region is

displayed in Figure 1c. The CR frequencies obtained from the map were then converted into elastic moduli.

The contact mechanics model employed to analyze these measurements is the Hertz model. The most relevant mechanical parameter, the indentation modulus, is defined as $M = E/(1 - \nu^2)$, where E represents the Young's modulus and ν denotes the Poisson's ratio. For our analysis, we used SiO₂ as a reference material, with a Young's modulus of $E_{\text{SiO}_2} = 72$ GPa and a Poisson's ratio of $\nu_{\text{SiO}_2} = 0.17$. This corresponds to an indentation modulus of $M_{\text{SiO}_2} = 74.1$ GPa. The indentation modulus of the tip, which is made of diamond, is taken as $M_{\text{Tip}} = 1250$ GPa. Through this analysis, we observed a significant variation in the indentation modulus of Cu, with a measured value of $M_{\text{Cu}} = 136.4 \text{ GPa} \pm 11.5 \text{ GPa}$. The uncertainty is reported from the experimental standard deviation of the measurements. The smaller variations in the indentation modulus of SiO₂, $M_{\text{SiO}_2} = 74.1 \text{ GPa} \pm 4.1 \text{ GPa}$, can be attributed to minor fluctuations in the contact area during CR mapping. In contrast, the larger variations seen in the indentation moduli of Cu, as illustrated in Figure 1d, are linked to the three principal crystallographic directions of Cu. The theoretical indentation moduli are 129 GPa, 138 GPa, and 141 GPa for $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$, respectively [10]. This variation indicates the presence of crystallographic heterogeneities within the Cu pads, which are influenced by

SINGLE-STEP AFM INDENTATION

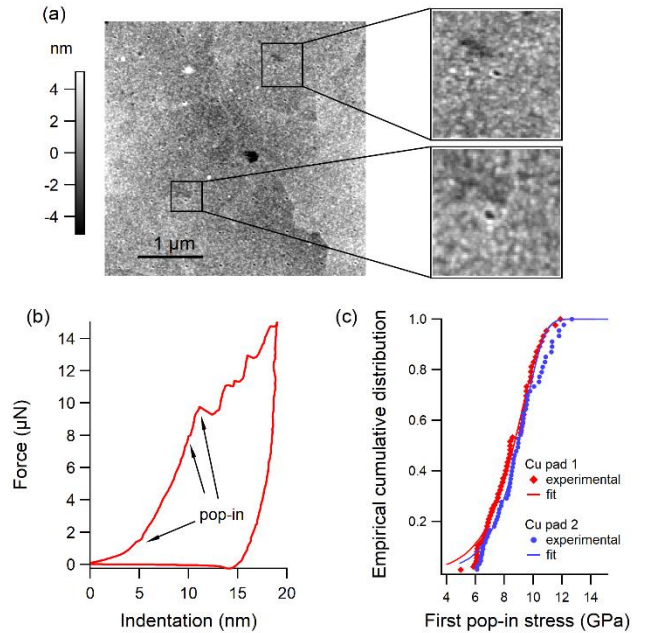


Figure 2. (a) AFM topography image of a Cu pad, over which an array of 8×8 indents was performed. The two insets on the right provide enlarged views of the regions around these indents. (b) Force versus depth response obtained from a Single-Step AFM indentation. (c) The cumulative distribution of the maximum shear stress at the first pop-in event from indents made on two different Cu pads.

the different fabrication processes involved, such as Cu seeding via physical vapor deposition, Cu electroplating, annealing, and chemical mechanical polishing [11].

The indentation moduli of Cu, as determined from CR-AFM measurements, were subsequently used in the analysis of elastic-plastic AFM-based indentations that we conducted on Cu. The Single-Step indents were arranged in a square array, similar to those depicted in Figure 2a. At each location, we performed load-unload tests up to a predefined maximum load. A typical example of a Single-Step AFM indentation test is illustrated in Figure 2b. During the loading phase, the response clearly transitions from the elastic to the plastic regime, marked by a pop-in event.

The maximum shear stress $\tau_{\max}^{\text{pop-in}}$ calculated at the first pop-in on the force versus indentation depth curve relates to the onset of plasticity and can be regarded as the nanoscopic yield stress. Figure 2c shows the empirical cumulative distribution function of these maximum shear stresses observed at the first pop-in events from Single-Step indentations performed across two Cu pads. As can be seen in Figure 2c, these distributions are narrow, ranging from 5 to 12 GPa, with a median maximum shear stress of approximately 8.6 GPa. This value is comparable to the theoretical shear strength of copper, which is generally estimated to be around $G/2\pi$ (where G is the shear modulus of the material; for Cu<111>, this is about 48 GPa). The high values of maximum shear stress, combined with the good fit of the empirical cumulative distribution of the measurements, suggest that the plastic events observed as the first pop-in in the force-depth curves are related to the homogeneous nucleation of dislocations. This process is triggered by the reconfiguration of a small number of atomic bonds, rather than by a larger-scale reconfiguration of the lattice. It is worth noting that this is exclusively due to the ability of AFM to probe extremely small volumes of materials.

MULTI-STEP AFM INDENTATION

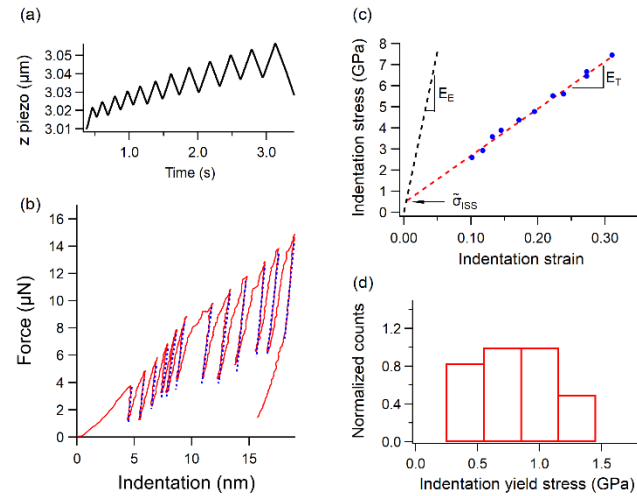


Figure 3. (a) Z-piezo modulation during Multi-Step AFM indentations. (b) Force versus depth response of a Multi-Step AFM indentation on Cu. (c) Indentation stress versus

indentation strain curve in the plastic regime derived from the fits of the partial unloading segments shown in (b). (d) Statistics of the indentation yield stress obtained from measurements similar to those presented in (c).

To gain deeper insights into the material's plastic response, we perform Multi-Step AFM indentations. As illustrated in Figures 3a and 3b, we reach the maximum load through a series of loading and unloading cycles. During these cycles, we partially unload by up to 25 % of the maximum load to ensure consistent contact between the tip and the sample during the unloading process.

Using a methodology developed by Pathak and Kalidindi [13], we fitted the unloading segments of the multi-step indentations (as indicated by the dashed lines in Figure 3b) to derive the constitutive relations for Cu in the form of indentation stress-strain (ISS) curves. In this analysis, the applicability of the Hertz model is extended to examine the partial elastic unloads within the plastic regime. This is achieved by augmenting the physical tip radius with an equivalent tip radius, while retaining the elastic moduli measured by CR-AFM. Both the effective tip radius and elastic moduli were used to calculate a pair of stress-strain values during each of the partial unloading events. A set of indentation stress-strain pairs is shown in Figure 3c (blue markers). As illustrated, these pairs follow a linear relationship defined by a slope E_T , which is smaller than the elastic slope E_E of Cu. Consequently, the slope E_T represents the linear strain-hardening behavior of the Cu pads, also referred to as tangent stiffness. The average value of E_T obtained from tests over on a Cu pad was found to be 20.3 GPa. Additionally, by analyzing the intersection of the plastic deformation path (the dashed red line in Figure 3c) with the 0.2 % offset elastic path (the dashed black line in Figure 3c), a median indentation yield stress of $\tilde{\sigma}_{ISS} = 0.64$ GPa was determined. The statistics for the measured indentation yield stress values are presented in Figure 3d. These results can be compared with yield stress values for copper reported by Niu et al. [14], which range from 0.2 GPa to 0.6 GPa, and by Ramachandramoorthy et al. [15], which are approximately 0.4 GPa, both determined using micropillar compression tests. It is important to note that the copper tested in [14] was a single crystal, while the material in [15] came from micro-additive-manufactured pillars.

CONCLUSIONS

By combining CR-AFM with both Single-Step and Multi-Step AFM indentation tests, we have developed a multimodal AFM-based methodology for characterizing materials at the nanoscale. This approach has allowed us to quantify the statistics and characteristics of incipient plasticity, providing insights into the nanoscale constitutive behavior of copper hybrid-bonding-ready pads. The parameters we obtained—such as Young's modulus, yield stress, and strain-hardening slope—can be effectively utilized in the design and modeling of key hybrid bonding processes, including chemical-mechanical polishing and diffusion bonding.

We have developed a roadmap for integrating an additional AFM-based material property characterization into existing AFM metrology used in the semiconductor industry. The combinatorial AFM methodology presented here focuses on nanoscale mechanical measurements and demonstrates that, in addition to critical dimension metrology (such as topographic measurements), AFM provides a wide range of capabilities for characterizing material properties. Unlike other techniques, AFM does not require additional sample preparation, allows for high-precision positioning, and can be applied in near-line metrology applications.

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The authors declare no competing financial interest.

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REFERENCES

- [1] *International Roadmap for Devices and Systems (IRDSTM) 2023 Edition - IEEE IRDSTM*. <https://irds.ieee.org/editions/2023>.
- [2] J. H. Lau, “Flip Chip, Hybrid Bonding, Fan-In, and Fan-Out Technology”, *Flip Chip, Hybrid Bonding, Fan-In, and Fan-Out Technology*, Springer, 2024.

- [3] N. A. Alderete, P. D. Daharwal, C. V. Ciobanu, and G. Stan, “Probing the Nanoscale Onset of Plasticity in Electroplated Copper for Hybrid Bonding Structures via Multimodal Atomic Force Microscopy”, *ACS Appl. Nano Mater.* 8, 24244–24253, 2025.
- [4] G. Stan, and N. A. Alderete, N. “Elastic-plastic AFM indentation on copper”, *data repository at the National Institute of Standards and Technology*, DOI: 10.18434/mds2-3867, 2025.
- [5] U. Rabe, K. Janser, W. Arnold, “Vibrations of Free and Surface-coupled Atomic Force Microscope Cantilevers: Theory and Experiment”, *Review of Scientific Instruments* 67, 3281–3293, 1996.
- [6] G. Stan, S. W. King, and R. F. Cook, “Nanoscale mapping of contact stiffness and damping by contact resonance atomic force microscopy”, *Nanotechnology* 23, 215703, 2012.
- [7] G. Stan, E. Mays, H. J. Yoo, and S. W. King, “Nanoscale tomographic reconstruction of the subsurface mechanical properties of low-k high-aspect ratio patterns”, *Nanotechnology* 27, 485706, 2016.
- [8] G. Stan, S. W. King, Atomic Force Microscopy for Nanoscale Mechanical Property Characterization”, *Journal of Vacuum Science & Technology B, Nanotechnology and Microelectronics: Materials, Processing, Measurement, and Phenomena*, 38, 60801, 2020.
- [9] G. Stan, C. V. Ciobanu, and S. W. King, “Resolving the Subsurface Structure and Elastic Modulus of Layered Films via Contact Resonance Atomic Force Microscopy”, *ACS Appl. Mater. Interfaces*, 14, 55238–55248, 2022.
- [10] J. J. Vlassak and W. D. Nix, “Measuring the Elastic Properties of Anisotropic Materials by Means of Indentation Experiments”, *J. Mech. Phys. Solids*, 42, 1223–1245, 1994.
- [11] J. D. Messemaeker, N. Jourdan, P. Nolmans, S. W. Kim, And G. Beyer, “Texture Control for Cu Hybrid Bonding Pads Using a PVD Cu Process”, *2019 International Conference on Solid State Devices and Materials, SSDM*, 2019.
- [12] X. Xiao and L. A. Yu, “Unified Statistical Model for Indentation Pop-in: Effects of Indenter Radius and Microstructure Density on the Transition from Homogeneous Nucleation to Heterogeneous Nucleation to Bulk Plasticity”, *Int. J. Plast.*, 141, 102980, 2021.
- [13] S. Pathak and S. R. Kalidindi, “Spherical Nanoindentation Stress–Strain Curves”, *Materials Science and Engineering: R: Reports*, 91, 1–36, 2015.
- [14] R. Niu, X. An, L. Li, Z. Zhang, Y. W. Mai, and X. Liao, “Mechanical Properties and Deformation Behaviors of Submicron-Sized Cu–Al Single Crystals”, *Acta Mater.*, 223, 117460, 2022.
- [15] R. Ramachandramoorthy, S. Kalacska, G. Poras, J. Schwiedrzik, T. E. J. Edwards, X. Maeder, T. Merle, G. Ercolano, W. W. Koelmans, and J. Michler, “Anomalous High Strain Rate Compressive Behavior of Additively Manufactured Copper Micropillars”, *Appl. Mater. Today*, 27, 101415, 2022.