



Application Note #1547

Nanoindentation at the Elastic Response Limit: Revealing Elastic Microstructures in a Monolithic Glass

The high sensitivity of nanoindentation and atomic force microscopy allows for the assessment of subtle displacements down to the sub-nanometer regime, enabling non-destructive testing of mechanical properties (such as mechanical moduli) at the elastic response limit. For the small indentation depths relevant in most nanomechanical testing scenarios, the Hertzian model assumption of a flat sample plane for the tip-sample interaction is not applicable, leading to inaccurate modulus amplitude calculations if surface roughness is ignored during the analysis. This application note addresses the real-life situation of non-flat surface conditions in a monolithic metallic glass¹ and how to correct for curvature-induced effects on the indentation modulus by using an equivalent radius. In doing so, an elastic microstructure is revealed in the metallic glass despite the existence of only a single amorphous phase, demonstrating the ability of nanoindentation and atomic force microscopy to access materials properties that remain hidden to other methods.

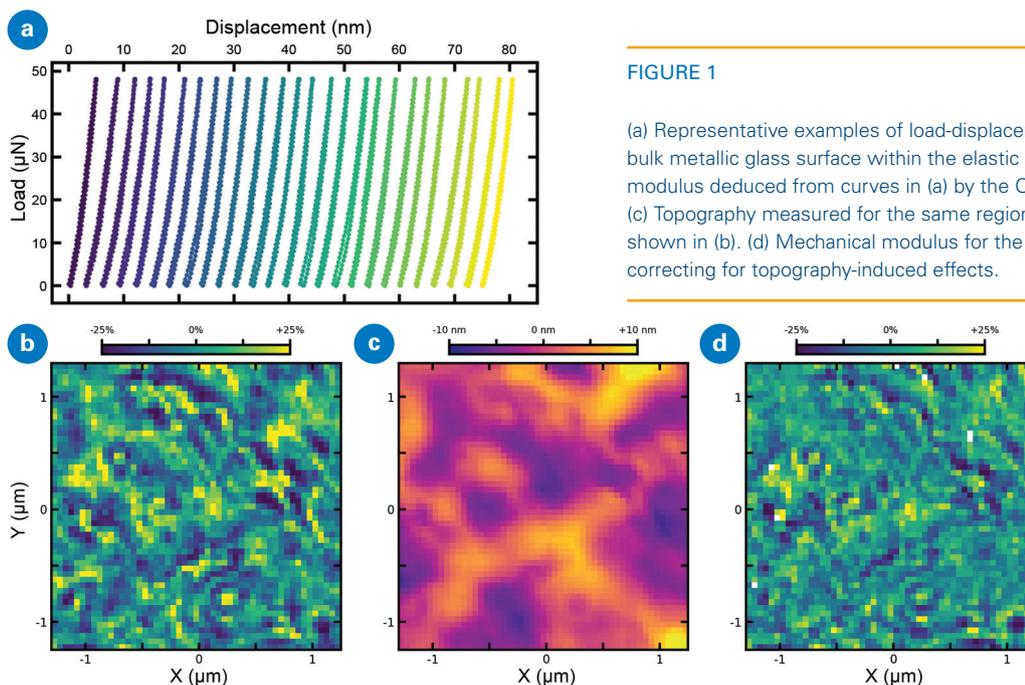


FIGURE 1

(a) Representative examples of load-displacement curves measured on a bulk metallic glass surface within the elastic response limit. (b) Mechanical modulus deduced from curves in (a) by the Oliver-Pharr method. (c) Topography measured for the same region on the sample surface as shown in (b). (d) Mechanical modulus for the corresponding area after correcting for topography-induced effects.

Mechanical Mapping

Spatially resolved nanomechanical measurements were conducted using a Hysitron TI 980 TriboIndenter® with a cono-spherical tip of 890 nm. A well-polished surface of a bulk metallic glass was characterized using accelerated property mapping (XPM™)² over a lateral range of 2.5 μm × 2.5 μm by probing mechanical modulus (Figure 1b) and topography (Figure 1c). Representative curves of local load-displacement (measured at 50 nm indentation distance) are displayed in Figure 1a.

Topography Correction

Although height variations over the width of a single indent may only be in the single nanometer regime, the indentation depth is of the same order of magnitude, resulting in a distinct correlation between modulus and topography.^{1,3} To account for such curvature-induced effects on the elastic modulus, an equivalent radius is determined for each point of the mapped area.⁴ Hence, the local curvature of the measured surface is considered as the inverse of the pair of independent local surface radii, i.e., radii of curvature, (R_x^s, R_y^s) . Together with the tip radius, R_{tip} , these are the basis for determining the equivalent radius, $R_{eq} = \sqrt{R_x R_y}$ by $\frac{1}{R_{eq}} = \frac{1}{R_x} + \frac{1}{R_y} + \frac{1}{R_{tip}}$, resulting in Equation 1.

$$\text{Equation 1: } R_{eq} = R_{tip} \left[1 + R_{tip} \left(\frac{R_x^s + R_y^s}{R_x^s R_y^s} + \frac{(R_{tip})^2}{R_x^s R_y^s} \right) \right]^{1/2}$$

The independent equivalent radii, (R_x, R_y) , behave as visualized in Figure 2. They are smaller than the tip radius if the local surface curvature is positive (e.g., at a local maximum on the surface), equal to the tip radius if the curvature is zero (where the sample surface is flat, i.e., with constant slope), and larger than the tip radius if the curvature is negative. As a special case, the equivalent radii approach infinity towards curvatures of $-1/R_{tip}$. Here, surface dips with curvatures below that limit lead to multiple contacts between tip and sample surface, effectively resulting in measured curvatures equal to $-1/R_{tip}$ that do not reflect the true curvature ($< -1/R_{tip}$) of the surface.

Results

To determine the equivalent radius from measured topography data, the independent radii of curvature, (R_x^s, R_y^s) , are related to the Gaussian and mean curvature, K^s and H^s .^{1,4}

$$\text{Equation 2: } R_{eq} = R_{tip} \left[1 + 2H^s R_{tip} + K^s (R_{tip})^2 \right]^{1/2}$$

These are defined by first and second derivatives of the surface topography and can be assessed by a point-wise analysis of the data.

$$\text{Equation 3: } K^s = \frac{\frac{\partial^2 f}{\partial x^2} \frac{\partial^2 f}{\partial y^2} - \left(\frac{\partial^2 f}{\partial x \partial y} \right)^2}{\left(1 + \left(\frac{\partial f}{\partial x} \right)^2 + \left(\frac{\partial f}{\partial y} \right)^2 \right)^2}$$

$$\text{Equation 4: } H^s = \frac{\left(1 + \left(\frac{\partial f}{\partial y} \right)^2 \right) \frac{\partial^2 f}{\partial x^2} - 2 \frac{\partial f}{\partial x} \frac{\partial f}{\partial y} \frac{\partial^2 f}{\partial x \partial y} + \left(1 + \left(\frac{\partial f}{\partial x} \right)^2 \right) \frac{\partial^2 f}{\partial y^2}}{2 \left(1 + \left(\frac{\partial f}{\partial x} \right)^2 + \left(\frac{\partial f}{\partial y} \right)^2 \right)^{3/2}}$$

Based on the equivalent radius, the corrected elastic modulus, E_c , is calculated from the measured modulus, E_r , and the tip radius, by assuming Hertzian contact mechanics via $E_c = \frac{\sqrt{R_{tip}}}{\sqrt{R_{eq}}} E_r$, as plotted in Figure 1d.

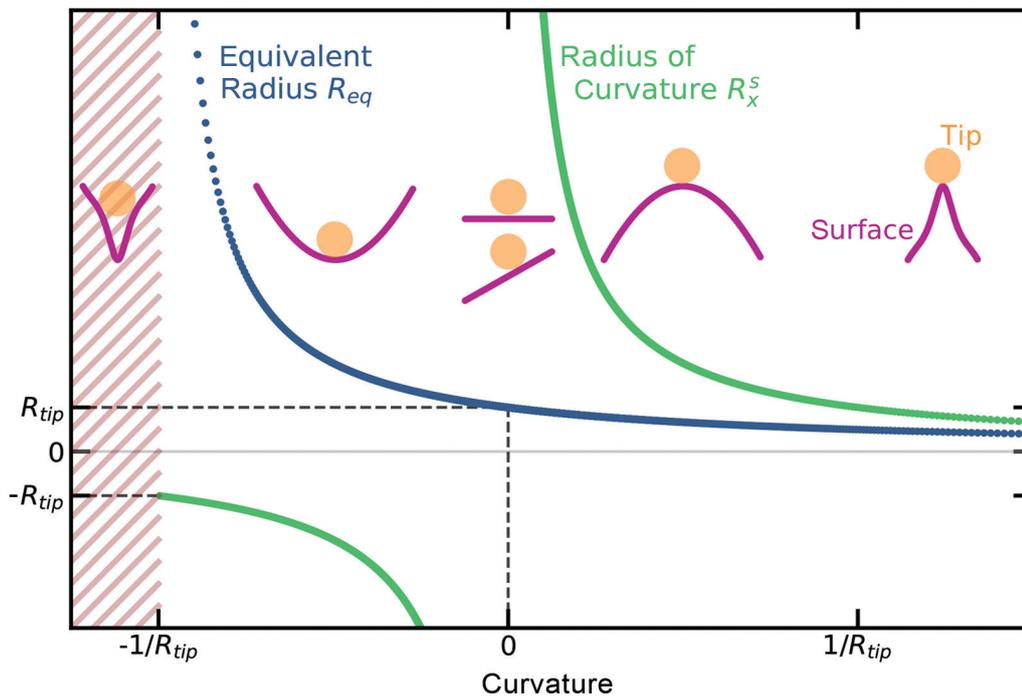


FIGURE 2

Equivalent radius and radius of curvature as a function of curvature with schematic representations of the interplay between surface curvature and tip.

Conclusions

High-throughput, spatially resolved nanoindentation can reveal nanoelastic fluctuations in single-phase solids, as demonstrated here for a monolithic glass. Such high-resolution measurements are very sensitive to long-wavelength topography modulations with amplitudes of only a few nanometers. A method described in References 1 and 4 yields a significant reduction of the influence of topography-related effects on the elastic modulus. Scenarios connected to imaginary solutions for the equivalent radius are avoided when using the same tip for topography and modulus measurements. Diverging values of the equivalent radius are expected at sites where the radii of curvature approach the negative tip radius.

Acknowledgements

Data courtesy of B. Riechers, R. Rashidi, and R. Maass, BAM, Berlin, Germany.

Authors

Dr. Birte Riechers, Federal Institute of Materials Research and Testing (BAM), Germany

Prof. Dr. Robert Maass, Federal Institute of Materials Research and Testing (BAM), Germany

Dr. Douglas Stauffer, Bruker (douglas.stauffer@bruker.com)

References

1. Riechers et al., *Materials & Design* **229**, 111929 (2023).
2. Hintsala et al., *JOM* **70**, 494-503 (2018).
3. Tsai et al., *Acta Materialia* **139**, 11-20 (2017).
4. Campbell et al., *International Journal of Mechanical Sciences* **161**, 105015 (2019).

Bruker Nano Surfaces and Metrology

Minneapolis, MN • USA
 Phone +1.952.835.6366
 productinfo@bruker.com

