APPLICATION NOTE

Nanoindentation of ZnO Crystals

Using *in-situ* SPM imaging with the Closed Loop Scanner on the TI 950 TriboIndenter®

Zinc oxide (ZnO), a semiconducting and piezoelectric material by nature, has been widely utilized in numerous electronic applications that exist today. Micron-scale ZnO crystals have an exciting potential application where a network of crystals within a textile matrix could provide self-sustaining energy via wind, body movement, or other forms of mechanical energy by harnessing the piezoelectric potential of the crystals.

For these applications to be realized, mechanical integrity of the ZnO crystals must be optimized. Mechanical optimization can be achieved by testing mechanical properties of crystals processed under different growth conditions. In this study, a Hysitron **TI 950 TriboIndenter** equipped with the **Closed Loop Scanner** option was used to investigate the mechanical properties of small-scale ZnO crystals grown on glass substrates under varying growth conditions.

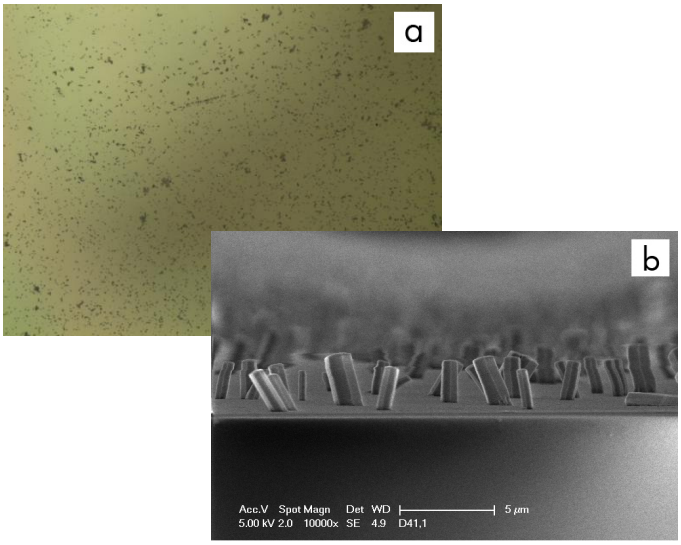


Figure 1. **a)** 20× optical microscope image of ZnO crystals grown on a glass substrate. Nanoindentation of the relatively small individual crystals requires use of *in-situ* SPM imaging. **b)** SEM image of same ZnO crystals on glass substrate. *In-situ* SPM imaging of these ZnO crystals is possible using forces less than 500 nN; imaging with larger forces causes the crystals to break loose from the substrate.

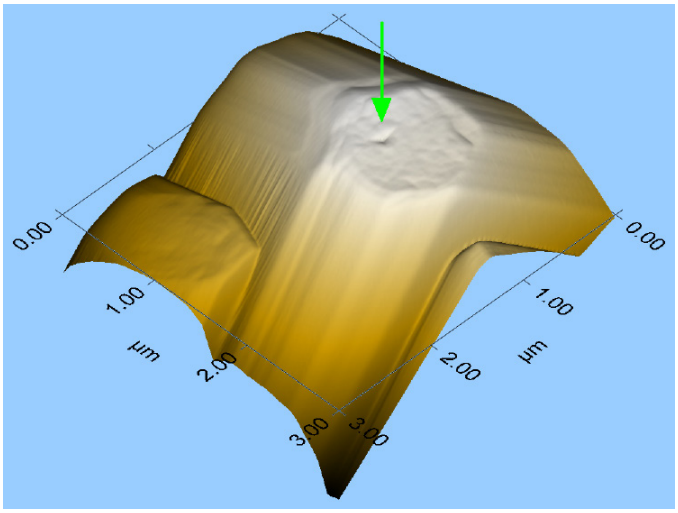


Figure 2. 3-D topographical *in-situ* SPM image of a single nanoindentation test performed on an individual ZnO crystal. Post-imaging using the same probe used for indentation allows for quick verification of indent execution and location.

In-Situ SPM Imaging

Figure 1 shows optical and SEM images of the ZnO crystals on the glass substrate. Indentation positioning directly via the optical image would be difficult due to the small size of the crystals (<3 µm). *In-situ* SPM imaging was used to position the tip and is critical for accurate indent placement and test verification as demonstrated in Figure 2. The imaging force was kept below 500 nN to ensure the high-aspect ratio crystals were not damaged.

Nanoindentation was performed on three different crystal types grown under varying conditions (A, B, and C). Single indents were performed on five individual crystal

faces (growth face) for each crystal type. A diamond 90° cube corner probe was utilized for indentation and SPM imaging.

Figure 3 shows reduced modulus data from indents performed on the three crystal types. Results indicate that growth condition C produced crystals with the highest mechanical properties of the three crystal samples.

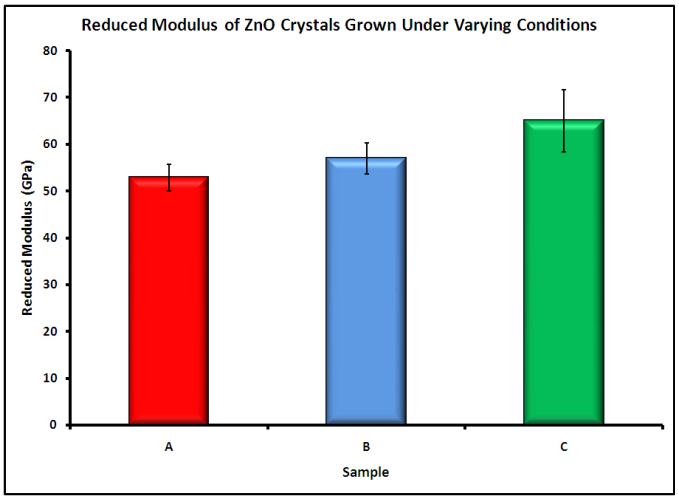
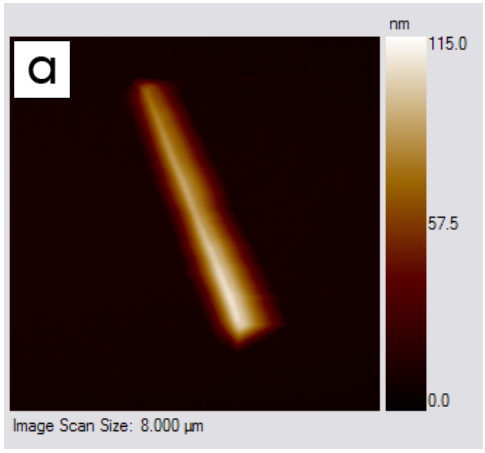


Figure 3. Bar graph of reduced modulus from nanoindentation tests performed on growth faces of ZnO crystals grown under three varying conditions (A,B, and C). Results clearly show different modulus values for each crystal type.

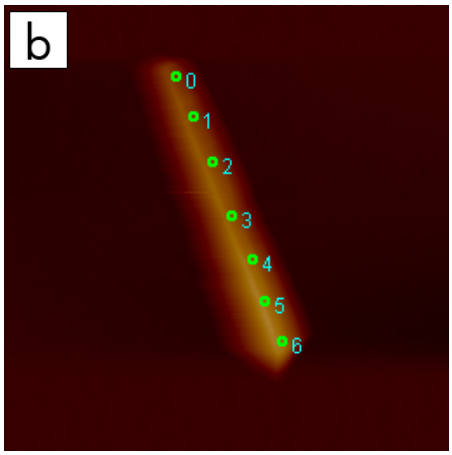
In addition, several nanoindentation tests were performed on the side of a ZnO crystal as shown in Figure 4. Indents were precisely placed within the 150 nm width of the crystal face by utilizing the increased accuracy exhibited by the **Closed Loop Scanner** option and using Click-Script Piezo Automation, an automated point-and-click feature that allows users to easily define individual indentation locations on an image generated using *in-situ* SPM imaging.

Conclusion

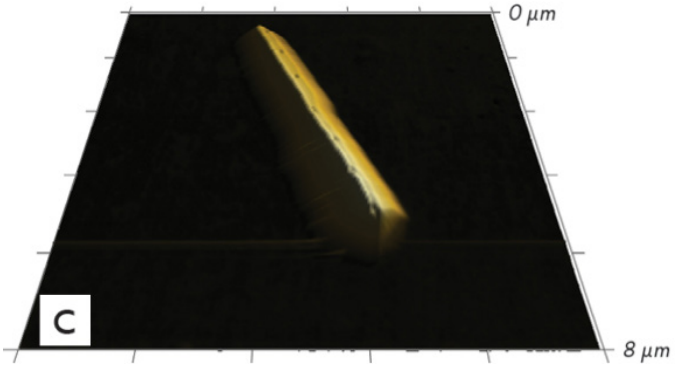
Quasistatic nanoindentation was used to examine the mechanical properties of ZnO crystals grown on glass substrates. Execution and verification of high-precision indentation was made possible by utilizing the **Closed Loop Scanner** option on the **TI 950 TriboIndenter**.



2-D Topographical In-situ SPM Pre-image



User-defined Nanoindentation Test Locations



3-D Topographical In-situ SPM Post-image

Figure 4. **a)** 2-D topographical *in-situ* SPM image of ZnO crystal on side prior to nanoindentation. Crystal face width is approximately 150 nm wide. **b)** SPM image with user-defined nanoindentation test locations on ZnO crystal side. **c)** 8 3-D topographical *in-situ* SPM image after execution of user-defined nanoindentation tests. Precision of nanoindentation tests performed within 150 nm crystal face width made possible by low hysteresis exhibited by the **Closed Loop Scanner** option.

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