

# High-Throughput Material Screening by nanoDMA® III

## Fast and Accurate Assessment of Mechanical Properties

### Introduction

The mechanical reliability of thin film coatings is an integral part of today’s growing industries. The fast paced development of single layer and multi-layer coatings requires high-throughput and accurate mechanical property screening. Hysitron has developed **nanoDMA® III** with *CMX* routines (Continuous Measurement of X; X = hardness, storage modulus, loss modulus, tan-delta, etc.) to provide a fast and accurate way to measure mechanical properties as a function of depth into the material’s surface. *CMX* offers a direct and continuous measurement of dynamic contact stiffness during any portion of an indentation test and allows a true observation of small volume deformation with reduced sensitivity to thermal drift. The accuracy and efficiency gains of this new capability are demonstrated by performing mechanical property screening of combinatorially deposited Transparent Conducting Oxide (TCO) thin films.



Figure 1: Combinatorial library, spread of composition of indium-zinc-tin-oxide (In-Zn-Sn-O) thin film by co-sputtering of indium oxide, zinc oxide, and tin oxide targets on 2”x2” glass substrate.

### Procedure

TCO’s consist of mixed metal oxide combinations that exhibit low optical absorption in the visible light range and low resistivity through the combined effects of electronic mobility and carrier concentration. These materials find application in photovoltaics, displays, transparent thin film transistors and electrochromic devices. Exploration of new TCO’s for photovoltaic applications focuses mainly on increasing the conductivity while maintaining optical transparency. Combinatorial synthesis of these materials allows for the rapid fabrication of ‘libraries’ that span a range of compositions on a single substrate. Figure 1 shows the combinatorial deposition of TCO thin film on a 2” x 2” substrate. The thin film was probed at 44 positions representing 44 different compositions for the electrical, optical, structural, and mechanical properties.

The reduced elastic modulus and hardness of these In-Zn-Sn-O thin films were measured using **nanoDMA III** with *CMX* routines and compared with quasistatic indentation. Quasistatic indentations were performed in displacement-control mode over contact depths ranging from 15 nm to 100 nm. Dynamic testing with *CMX* was performed at a constant strain rate over the same contact depth range as used for the quasistatic indentations with a frequency of 220 Hz. To minimize the substrate effect during indentation, only the top 5-10% of total film thickness (350±20 nm) was used as a contact depth for data analysis.

## Results

The reduced modulus and hardness measured with quasistatic and dynamic indentations are shown in Figure 2 (a) and (b), respectively. The total test time using **nanoDMA III** with *CMX* took 11 hours to screen all 44 areas with 4 dynamic indentations per area, while measuring similar data over the required depth range using quasistatic indentation took ~200 hours (more than one week). In summary, *CMX* is more than one order of magnitude faster compared to conventional quasistatic indentation. The value of reduced modulus and hardness obtained from quasistatic indentation was  $134.0 \pm 2.7$  GPa and  $11.7 \pm 0.2$  GPa, while the average value obtained over the four dynamic indentations was  $133.9 \pm 2.8$  and  $11.3 \pm 0.4$  GPa, respectively. The results reveal good agreement in reduced modulus and hardness between those observed under quasistatic and dynamic indentation testing, demonstrating the correlation between dynamic and quasistatic measurements.

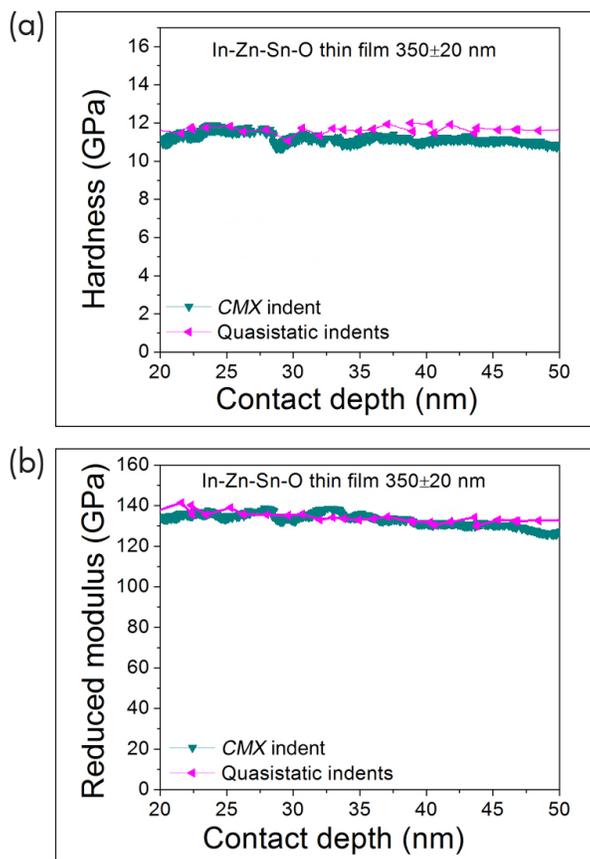


Figure 2: (a) Hardness and (b) reduced modulus with contact depth in indium-zinc-tin oxide (In-Zn-Sn-O) thin film measured with quasistatic indentation and dynamic indentation.

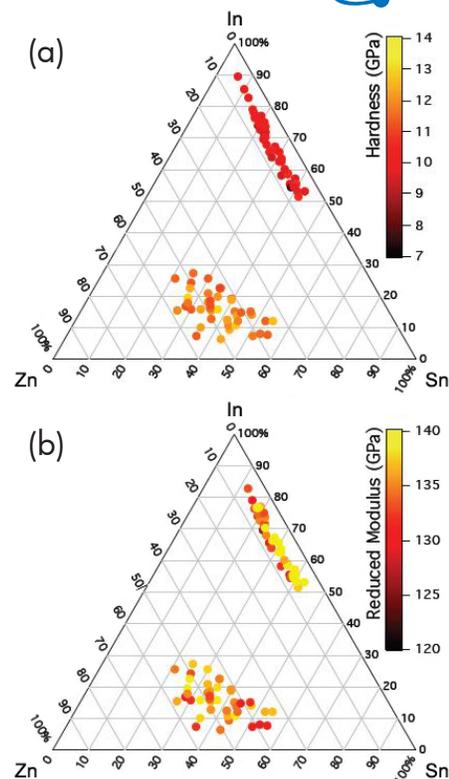


Figure 3: (a) Hardness and (b) reduced modulus of two indium-zinc-tin-oxide (In-Zn-Sn-O) thin film combinatorial libraries deposited on glass substrate using RF magnetron co-sputtering from three different ceramic targets of  $\text{In}_2\text{O}_3$ ,  $\text{ZnO}$ , and  $\text{SnO}_2$ .

## Conclusion

The mechanical properties of two different combinatorial libraries can be referenced to composition to determine the composition-structure-property relationship as shown in Figure 3 (a) and (b). The triangle shows how the mechanical properties are changing with the different cationic composition In-Zn-Sn-O thin films. The reduced modulus varies by 7-12% and does not show dependence with the composition (error  $\approx 5\%$ ), while the hardness varies by 15-25% and appears to be higher for lower indium content (error  $\approx 5\%$ ). These results indicate that **nanoDMA III** with *CMX* is a fast and accurate way to screen the mechanical properties in a system that is well-matched to next generation materials development.

The In-Zn-Sn-O samples were provided by the National Renewable Energy Laboratory. Further details can be found in the paper "Optimizing amorphous indium zinc oxide film growth for low residual stress and high electrical conductivity", M. Kumar, A.K. Sigdel, T. Gennett, J.J. Berry, J.D. Perkins, D.S. Ginley, and C.E. Packard, Applied Surface Science, 283, 65, doi:10.1016/j.apsusc.2013.06.019 (2013).

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