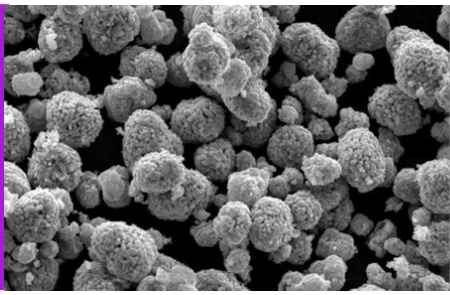
The Importance of Nanomechanical Properties to Battery Materials Performance



Introduction

Batteries are widely used power sources that utilize electrochemical cells for powering various devices. Battery performance is impacted by intrinsic mechanisms of battery failure and degradation, which depend on the electrochemical and nanomechanical interactions of the constituent materials. Tremendous efforts have been made in industry towards the next generation high energy density and high C-rate (a measure of discharge rate) batteries, where new materials, manufacturing processes and integrations are considered in order to optimize battery performance across a wider temperature range[1]. To improve yield rates and scale manufacturing growth, it is crucial to understand batteries' failure and the corresponding root causes. Typical battery failure modes fall into the categories of mechanical failure, thermal failure and electrical failure.

Battery Cell Manufacturing

A typical battery manufacturing process includes the electrode manufacturing, mixing, coating, calendaring, slitting, electrode making, stacking and packing, as shown in Figure 1. Throughout the process, nanoindentation techniques can be used for any of the following measurements:

- Compressive strength of cathode slurry particles (diameter 1-20µm)
- Complex modulus of binder materials
- Indentation hardness and modulus of the composite cathode coating
- Indentation hardness and modulus of the solid electrolyte
- Indentation hardness and modulus of the anode coating
- Fracture toughness and indentation cracking of stacked layers

Nanoindentation techniques enable battery studies on micro-length scales, where nanomechanical properties are evaluated on both the electrode and particle level. For instance, these techniques accomplish the following:

- Facilitate the investigation of emerging battery materials behavior on the length scale of defects and failure mechanisms by offering indentation hardness, modulus, quantification of fracture toughness, etc.
- Boost battery reliability evaluation after cyclic charging and discharging to prevent failure.
- Enhance the battery thermal safety and thermal management by optimizing the components/materials performance at wider temperature ranges.

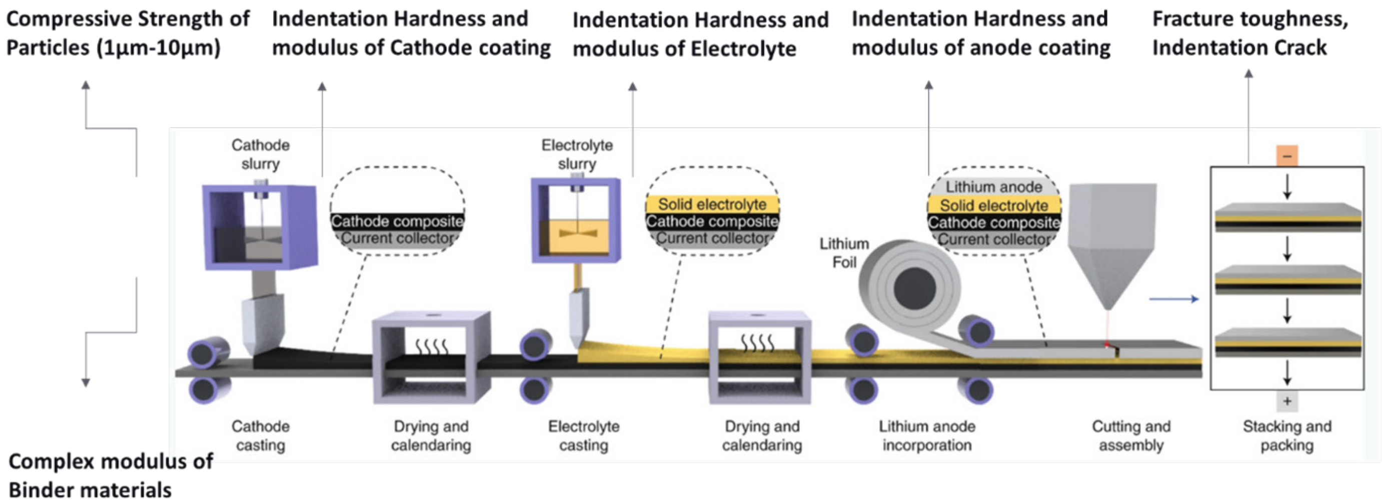


Figure 1. Schematic of large-scale manufacturing of all-solid-state-batteries (ASSBs). Polymer and solid electrolyte composites allow for good mechanical processability as well as decreased separator thickness layers to increase cell-level energy density. *Image credit: Tan et al. [2]*

Measurement of Cathode Load Response

Many test methods are available for the KLA Instruments™ nanoindentation systems. To apply a mechanical load to a commercially available composite graphite on copper foil on glass, a test method was developed that controlled actuation, sensing, data acquisition, parameter calculation and presentation of results. Figure 2 (top) shows the measured elastic modulus as a function of depth, with a SEM image of the $LiNiCoMnO_2$ surface. Figure 2 (bottom) shows the measured hardness of the cathode layer as a function of depth, with a schematic of the actuator. Different testing modes are available to accommodate significant surface roughness.

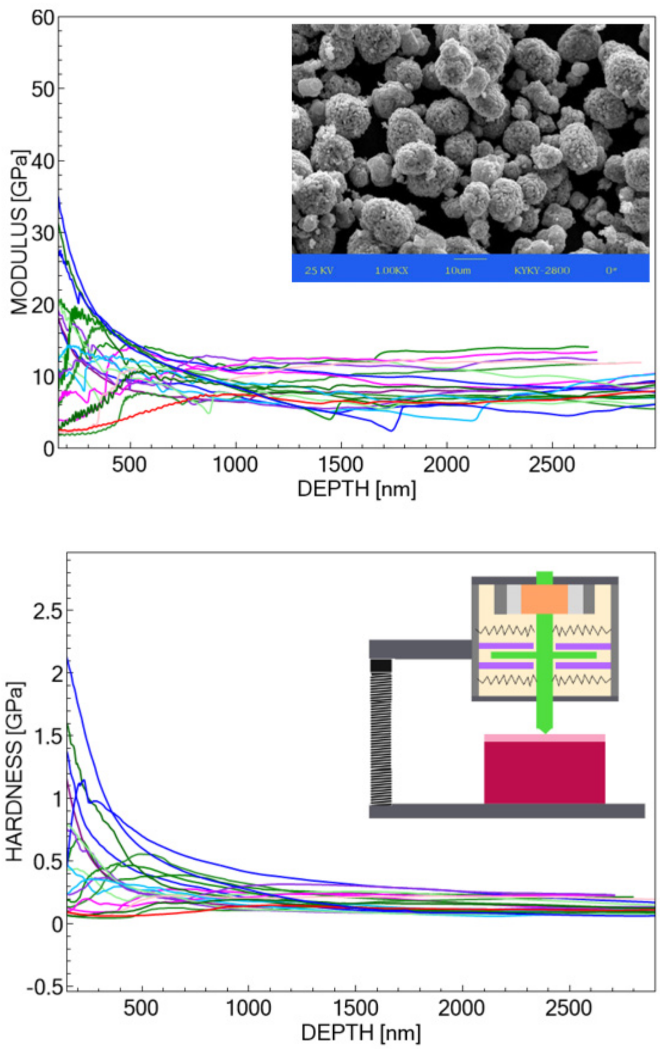


Figure 2. Cathode material modulus (top) and hardness (bottom) as a function of indentation penetration depth, with a SEM image of the $LiNiCoMnO_2$ surface and the KLA actuator schematic. *SEM image credit: MTI Corporation.*

High Speed Nanomechanical Mapping of Composite Cathodes

Nanomechanical properties on a surface can be mapped using NanoBlitz 3D, where an array of indents is specified to measure the properties at 1 second per data point. Figure 3 shows the modulus (top) and hardness (bottom) maps of a lithium-ion battery cathode, composed of a mixture of active particles of lithium nickel manganese cobalt oxide ($LiNiCoMnO_2$ as 5:2:3). Figure 4 shows the NanoBlitz 3D mapping area outlined on the cathode coating surface. Compared to the conventional CSM method, the use of NanoBlitz 3D nanoindentation testing is extremely useful for its higher signal-to-noise ration and its speed in identifying the single-phase mechanical properties and their spatial distribution.

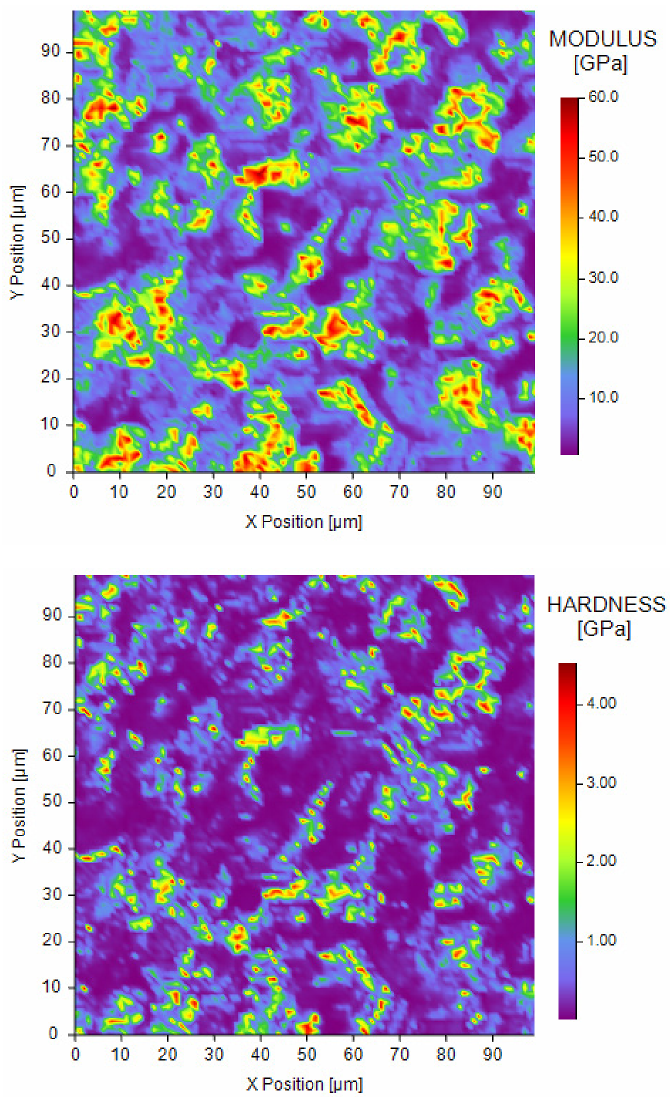


Figure 3. Nanoindentation mapping of elastic modulus (top) and hardness (bottom) on a cathode coating.

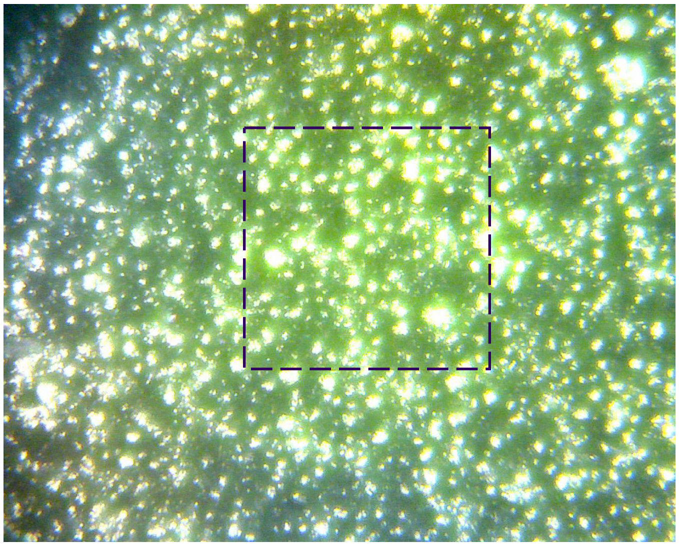


Figure 4. The NanoBlitz 3D test area is shown on the cathode surface.

In Situ Compression on Individual Particles

The often-seen cracking of particles during manufacturing leads to detrimental side reactions. The KLA Instruments InSEM® in situ nanoindentation system enables in situ observation of particle cracking and measurement of compressive strength of particles and mechanical properties of composite coatings. Figure 5 shows the InSEM video images prior to (left) and following (right) particle crush measurement. Figure 6 shows the corresponding load-compression response of particles (top) and rupture stress (bottom).

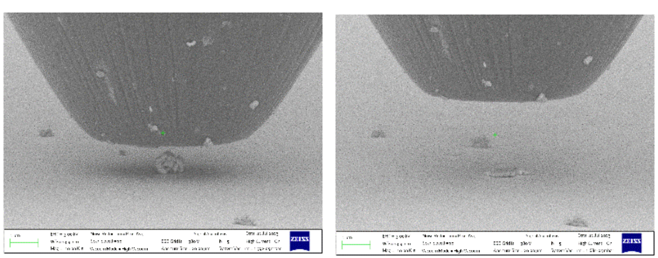


Figure 5. InSEM video image of particle compression prior to (left) and following (right) particle crush measurement.

Measurement of Viscoelastic Response of Binders

The KLA Instruments ProbeDMA™ technique can be used to perform local dynamic mechanical analysis (DMA) testing on binder materials. ProbeDMA has the advantage of quantifying local mechanical properties by targeting specific surface locations with the nanoindenter. Figure 7 compares the measured storage modulus of binder materials under dry and wet conditions, where data was collected at a fixed frequency of 10Hz.

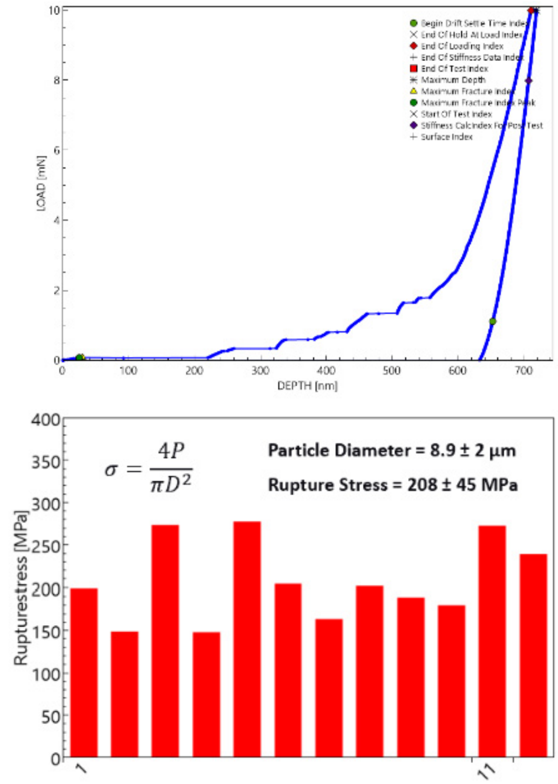


Figure 6. Load-compression response for in situ particle compression (top) and measurements of rupture stress (bottom).

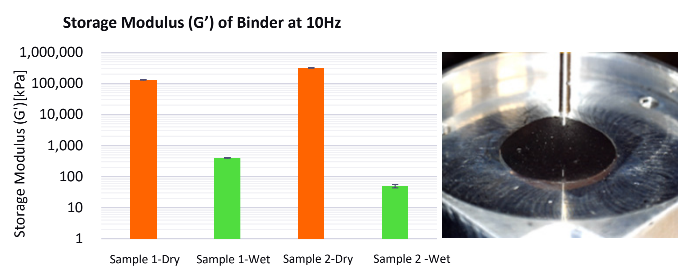


Figure 7. Storage modulus of the binder under dry and wet conditions (left). Nanoindentation measurement in electrolyte solution (right). *Image credit: Toyo Corporation.*

Alternative Battery Materials Testing Environments

Although nanoindentation experiments are typically performed under ambient conditions, some battery materials require nanomechanical testing on liquids or at elevated temperatures that may be best carried out inside a glove box. The KLA Instruments iMicro and iNano® systems are well-suited to these environments due to their compact size. Figure 8 shows a glove box setup enclosing a Nano Indenter® G200 system for testing in an electrolyte fluid cell, where the glove box can be backfilled with argon gas to evaluate nanomechanical properties under argon and fluid environments.

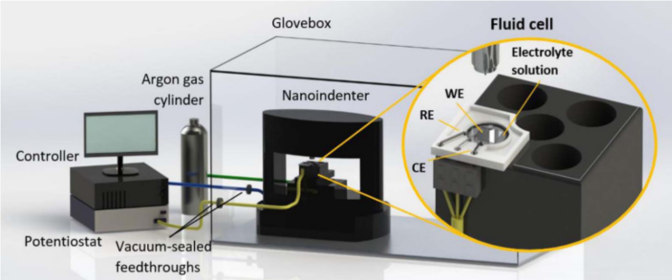


Figure 8. Example of a glove box environment for the Nano Indenter® G200 system, used to measure load response under argon and fluid environments. *Image credit: Scalco de Vansconcelos et al.[3].*

The Versatility and Importance of Nanoindentation to Battery Materials Manufacturing and Research

Battery cell manufacturing includes multiple process steps that affect layer uniformity and interface adhesion. Battery materials research comprises a wide array of applications and environments to test material behavior and performance, under ambient, fluid, gas, and vacuum conditions. If you are interested in learning more about how KLA Instruments nanoindentation can deliver critical nanomechanical behavior data for your battery materials research or manufacturing, please visit our website or contact us today.

References

[1] Fan et al., "Tailoring inorganic-polymer composites for the mass production of solid-state batteries," National Review of Materials 6, 1003-1019 (2021).

[2] Tan et al., "From nanoscale interface characterization to sustainable energy storage using all-solid-state batteries," Nature Nanotechnology 15, 1070-180 (2020).

[3] Scalco de Vasconcelos et al., "Operando nanoindentation: a new platform to measure the mechanical properties of electrodes during electrochemical reactions," Journal of the Electrochemical Society 164 (14), A3840-A3847 (2017).

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