Covalent Academy Episode 38 Q&A

Pressing Ahead: Unveiling Material Insights through Dynamic Nanoindentation

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01

Q: Can you explain in a bit more detail how stress / strain measurements work on the nano-indentation tool? Why is Sinus Mode necessary for collecting these measurements?

Answer [Paraphrased from the Live Broadcast]:

You need a dynamic profile to produce a stress/strain curve. This is because you need to measure stiffness as a function of depth (as with standard quasi-static load conditions), while also being able to separate the stress measurement under variable strain conditions. The Sinus mode on the STeP6 system also gives you more control over the applied strain via nonstandard tips that improve the resolution of stress / strain measurements.

02

Q: For depth profiling, what might be the approximate maximum penetration depth one could expect? (For example: in a soft polymer sample.)

Answer [Paraphrased from the Live Broadcast]:

The maximal range of the actuator in the UNHT3 probe is about 100 microns; this is as far as one could expect the probe to penetrate in air.

03

Q: What kind of information can be obtained with dynamic nanoindentation when performed on hard composite materials, such as asphalt, rock, or glass?

Answer [Paraphrased from the Live Broadcast]:

It will come down to the grain sizes of the different phases in the composite. With nanoindentation, if we can isolate an area with only one phase, nanoindentation enables us to probe the mechanical properties of this specific phase. Repeating this for each phase gives us a robust mechanical analysis of all the components in the composite.

Regarding the hardness of the sample(s): typically, very hard materials are not a cause for concern. The indenter tips are made of Diamond, which is exceptionally hard. Furthermore, at Covalent we routinely test ceramics / ceramic coatings, as well as Diamond-Like-Coatings (DLCs) with these indenter tips, and have proven their efficacy even for comparably hard material samples.

04

Q: In the battery industry, it is often helpful or even critical to be able to measure mechanical properties (like Fracture Toughness, Hardness, or Tensile Strength) of raw materials in powder form. Can you confirm: does nanoindentation work on powders or microparticles? If so: how can it be used for these kinds of applications?

Answer [Paraphrased from the Live Broadcast]:

If you need to understand the fundamental mechanical properties of a powder material (like its elastic/storage modulus, stiffness, hardness, or even fracture toughness), then nano-indentation works really well to perform these measurements on single particles/grains within the sample. We actually run this kind of analysis regularly for customers researching battery materials!

There two main considerations to keep in mind when running nanoindentation on powders: first regarding sample preparation, and then regarding the choice of tip used in the actual technique.

When prepping samples at Covalent, we will typically either dry-cast the powders on an appropriately chosen substrate, like a pure-Silicon wafer, or will ‘pot’ the powder in an epoxy and then sand/grind the resulting epoxy-sample chuck to expose individual grains for measurement.

This latter approach is sometimes helpful for softer materials to provide some ambient rigidity around the target particle/grain for the indenter measurement.

When we’re proceeding with the actual nanoindentation measurement, we want to make sure to use a Flat-Punch indenter tip. This provides significantly finer control of the applied stress and distributes it more uniformly over a larger surface area (relative to the Berkovich or Spherical tips). In tandem, these features of the Flat-Punch indenter tip help to ensure that the powder grain/particle does not splinter or disintegrate, preserving its full size and shape.

Unfortunately, as Tensile Strength measurements specifically require a pulling force, we are unable to measure this directly with a nano-indenter system. While many applications do not truly require direct measurement of Tensile Strength, we know there are a few that do. For those cases: there’s actually a Nano-Pull-Test method which has started to gain some traction in recent experimental research papers; however, this not something we are equipped to run at Covalent at this time.

05

Q: Just how important is the tip geometry to a nanoindentation measurement? (You mentioned that Covalent has options like the standard Berkovich tip or the Sphero-Conical / Flat-Punch tips for viscoelastic measurements.) How does tip geometry factor into calculations of parameters like tan(∂)?

Answer [Paraphrased from the Live Broadcast]:

Tip area is factored into the calculations of *Storage Modulus (E')* and *Loss Modulus (E'').* The ratio of these two values gives us the *tan(∂)* for the sample material:

$$\tan\delta = \frac{E''}{E'}$$

Thus, for viscoelastic measurements, it's particularly important that we use a consistent tip shape (and thus tip area function) for all trials, as this will affect the tip area function the system factors into its output modulus values.

On the other hand, *tan(∂)* is agnostic to tip geometry, as the tip area function is cancelled out when one divides the Loss and Storage moduli.

This makes it possible to compare *tan(∂)* measurements from nanoindentation with *tan(∂)* values collected through other test methods, such as bulk Dynamic Mechanical Analysis (DMA).

Actually, *tan(∂)* has been used for exactly this purpose in many past studies to contrast the results of nanoindentation against classical mechanical analysis on polymer samples. These studies confirmed that the *tan(∂)* results from nanoindentation usually fall within 5-10% variance of the *tan(∂)* values obtained through tensile or compression DMA tests.