Covalent Academy Episode 23 Q&A

Nanoindentation with the UNHT3 Indentation Tester: Nano-mechanical Analysis with a Big Impact

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Q: I believe you said that one needs to know the Poisson's ratio of the film in order to calculate the modulus? How hard is it to get this value for thin films? Is Poisson's ratio for thin films affected by residual stress?

The Poisson ratio generally ranges from 0.0 to 0.5 and is often set at a default of 0.3. Precise values for Poisson's ratio can be found for most materials pretty easily with a simple web search. Yes, there are references from the literature that both Young's Modulus and Poisson's ratio can be effected by residual stress. Of course attention to these details improves the accuracy of your results.

Q: If hardness is a function of depth, what is the optimum depth to measure and report hardness?

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Hardness is not always a function of depth, but it can be. If hardness varies as a function of depth, one way to measure this is with "Sinus" mode – sinusoidally loading and unloading as the overall load progressively increases. By measuring the unloading curve at each of the sinusoidal load/unload events, the hardness can be calculated as the indenter moves into the surface.

Q: Would you please elaborate on how to measure the coating thickness with nanoindentation directly (without using cross sectional analysis or any other instruments)?

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Nanoindentation is not a preferred method for coating thickness measurements. However, at the interface between the two materials, the hardness will likely change. Using Sinus mode this can often be detected. Depending on the relative hardness of the two materials (hard on soft or soft on hard) the compliance of one of the films may adversely influence the validity of the measurement. This is why X-ray or optical techniques are typically used for thin film coating thickness measurements.

Q: How appropriate is this technique for softer materials like elastomers?

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This technique is often used to measure very soft materials like elastomers or even hydrogels.

Q: What is the nanoindenter tip made of? Can it be replaced?

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Typically the indenter tip is made of diamond and is prefabricated for use in the indentation system. Alternative materials or geometries are not impossible but generally not something that Anton Paar does.

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Q: What is a Berkovich Indenter? What are some alternative indenter tips? How do I select the correct indenter tip?

A Berkovich indenter is a 3-sided pyramidal diamond indenter cut along the diamond crystal planes to insure precise and repeatable geometries. Remember that the projected area calculation is dependent on knowing the exact indenter geometery – $Hardness = \frac{F_{max}}{A_{projected}}$. So poor tip calibration has a big impact on the accuracy of your hardness calculation. From the equation, it's clear that other geometries, like Vickers, and others can be used, but for instrumented indentation the Berkovich tip is the standard.

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Q: What is the reduced modulus?

The reduced modulus describes the elastic deformation that occurs when one material is pressed into another. Both materials experience an elastic deformation and the reduced modulus is the combination of these relative deformations – actually the inverse sum. $\frac{1}{E^*} = \frac{1-\upsilon^2}{E} + \frac{1-(\upsilon')^2}{E'}$; where E* is the reduced modulus, E is the modulus of the material being indented, and E' is the modulus of the indenter. υ and υ' are the Poisson ratio of the material under test and the indenter respectively.

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Q: What are the advantages of nanoindentation over conventional hardness testing methods?

Although nanoindentation can be used to measure bulk hardness properties, it is usually used to measure the hardness of thin films or other engineered surfaces. In these cases, the hardness on interest is geometrically localized in terms of depth (thin film) or laterally (material grains). Historic indentation techniques, like Vickers or Rockwell, use relatively large indenters and penetrate deep into the surface. Those techniques are preferred for bulk hardness measurement of homogeneous and isotropic materials.

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Q: I understand mounting the sample is important. Can you please explain why? What are some common techniques?

If the sample is not well mounted, it will not be strongly mechanically coupled to the stage. As a result, on a microscopic (or maybe macroscopic) level it will act like a spring. The indentation is then a convolution of the spring like mounting and the sample itself causing inaccuracies in the data. Often samples are mounted to a carrier with super glue or something similar and then mounted to the instrument using a vice. Removing the influence of the sample, the pure hardness and modulus of the part under test can be measured.

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Q: What do you do differently if you are measuring a hard material on a softer substrate?

In an effort to avoid substrate effects of the softer underlayment, one must be sure to indent less than 10% of the film thickness – even less is better.

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Q: I understand the 10% of depth is a rule of thumb…When is it not applicable and what should you be looking for?

This rule of the thumb is not applicable when the film thickness is so thin that it is mechanically impossible for the indentation system to indent <10% of the film thickness – think gate oxide on Si. Substrate effects are rarely obvious. So, if this is a concern, check your results against published values for these materials. If the results are not similar, be concerned and contact the instrument maker for help.

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Q: How do you know the results are valid from looking at the data only?

Bad data will often exhibit substantial noise in the loading and unloading curve and may have peculiar "pop-in" or "pop-out" artifacts as well. Creep in the data is another common cause of poor results. Anton Paar resolves this with a reference indenter to reduce the mechanical path length from indenter to sample.

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Q: For converting from reduced modulus to Young's modulus, how is Poisson's ratio determined? Can it be done via nano-indentation?

Poisson's ratio is often taken from the literature. If the hardness and modulus of the sample under test are known, Poisson's ratio can be back calculated from the equation shown above (question 7).

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Q: Are you able to determine the hardness of several thin films deposited on top one another? How?

This is generally done in a serial process. That is, a film is deposited on a substrate and the hardness is measured being careful not to exceed the 10% rule. The next layer is applied, and the hardness of that film is measured, and so on. It is true that Sinus mode can be used to measure hardness as a function of indentation depth. Often it will show differences at material interfaces, but the exact contact mechanics becomes quite complicated particularly if there are multiple films.