

COVALENT METROLOGY

Covalent Academy Episode 40 Q&A

R&D Applications of Spectroscopic Ellipsometry (SE):

Advanced Insights for Process Control and Property Monitoring

Presented By:

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Q: Can you please explain a bit more about how porosity measurements can be conducted with Ellipsometry?

Answer paraphrased from the live broadcast:

The idea here is that when you have a porous material, you can think of your sample as a mixture of your solid material with air. (For example, porous Silica can be thought of as Silicon oxide with some air mixing in.)

When we do porosity measurements, we flow in water vapor or Thallium vapor to fill in the pores. Depending on the pore sizes, you'll need to apply different pressure to saturate the pores. This is a basic absorption/desorption chemical process. Based on calculations with the applied pressure, you can determine the pore size and distribution by comparing measurements with the vapor to those taken in ambient air conditions.

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Q: Can spectroscopic ellipsometry be used to measure thickness of thin films and organic polymers? If so: what are the optical requirements of the substrate?

Answer paraphrased from the live broadcast:

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Yes! Organic polymers are typically easier to measure than some of the other kinds of samples we've discussed. Most traditional polymers, such as polystyrene, PMMA, or polycarbonate, are transparent in the visible range. These can all be easily measured with ellipsometry.

Many organic polymers have a similar refractive index to glass substrates, so in these cases you will get improved optical contrast if you use something like a Silicon substrate (which has a much higher refractive index). That will give you more accurate layer thickness.

However, if that's not an option, you can usually get better optical contrast in the UV range, so it is still possible to use ellipsometry to make thickness measurements.

Q: Can you share some examples of the types of information that can be extracted from structural properties of polycrystalline / single-orientation / or grown materials, etc.?

Answer paraphrased from the live broadcast:

This is a great question, but, unfortunately, I don't think we have the answer right now, at least for grain direction, due to that polycrystalline samples can have multiple, random orientations.

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This becomes an issue given the scale of the beam spot. Ellipsometry uses a few millimeters up to 10mm spot size, and you can capture a variety of orientations within the spot when testing polycrystalline samples.

Ellipsometry is probably also not going to be able to give you grain size or relative grain size differences in these samples. We've seen some applications that wanted to evaluate anisotropy / isotropy of materials, but ultimately had better results with SEM and TEM than ellipsometry due to the increased randomness in orientation at the scale of the ellipsometry beam spot.

In order to measure anything, you'd thus need to have a large range order for ellipsometry to provide the sensitivity needed.

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Q: Does J.A. Woollam have software support for OCD measurements? Alternatively, can you recommend a software partner who might provide tools to analyze this kind of data to extract device dimensions?

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Answer paraphrased from the live broadcast:
[J.A. Woollam] does not provide OCD (critical dimension) measurement software, but we do have a business relationship with Onto Innovation / Onto Semiconductor, and I believe they have OCD software that is open source or available for purchase for outside users.

This software is also compatible with all our software and tools.

Q: Can Spectroscopic Ellipsometry detect the coverage of a self-assembled monolayer?

Answer paraphrased from the live broadcast:
Yes, we can do this kind of analysis with ellipsometry! One of the examples I showed earlier on in this webinar was a self-assembled monolayer (SAM). For these kinds of samples, coverage measurement is definitely doable. Even in ALD samples, we've shown that J.A. Woollam's ellipsometers have the sensitivity to measure film thickness in angstrom or nanometer range and to map these measurements across a surface.

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For self-assembled monolayer applications, the question is less about whether it *can* be done, but rather: *how should you set up your experiment correctly*?

Ellipsometry is very sensitive to surface conditions! Exposing the reference substrate to ambient air for any length of time can lead to contamination or absorption of stray particulates which will significantly skew your measurements. Thus, it's critical to make measurements both before and immediately after the monolayer assembly, preferably at the same location on the sample.

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Q: What is the thickness limit for measurements?

Answer paraphrased from the live broadcast:
For very thin samples, it depends on the optical contrast of the film against the substrate.

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For very thick films, non-uniformity of the material can contribute to optical depolarization that will skew your measurements.

Generally, we estimate that ellipsometry can be used to analyze films from several nanometers up to tens of microns thick; however, on both ends of that range, there are specialized techniques and experimental setups involved to achieve accurate thickness measurements.

Q: What is the maximum thickness acceptable for a metal sample for it to be measured with spectroscopic ellipsometry?

Answer paraphrased from the live broadcast:
For metal thin films, absorption effects are present that will impact your measurements.

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When you use ellipsometry to measure thickness, you need the light to be able to 'see' both the top and bottom of the film. In metal samples, light is absorbed before it is able to get back to the detector.

This usually limits the thickness of metal films to about 100 nm for ellipsometry measurements, since in thicker films the light will not be able to reach the bottom of the film

Q: How well can ellipsometry handle samples with multilayer stacks? In particular, how well does it handle very many layers (e.g. 5-10)?

Answer paraphrased from the live broadcast:
This is a pretty common question we [at J.A. Woollam] are asked. Actually, it doesn't matter so much how many layers you have. (We've seen some applications with as many as 37…even 64 stacked layers!) It really comes down to what kind of materials comprise the layers you're looking at and in particular whether those materials are intrinsically **optically consistent.**

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For instance, consider a layer of Silicon oxide: we know the optical constants for Silicon oxide and these don't change much. On the other hand, if you have a layer of Silicon nitride

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(as was shown in one of the examples in this webinar), then the optical properties of that layer could vary quite a bit.

If you have layers like this with less optically consistent materials, then it's typically recommended that you prepare and analyze separate samples with a single layer of those materials before beginning to model the full multilayer stack. You can then input the measured optical constants of your single-layer samples as you begin modeling the multilayer stack.

The final computed optical constants for those layers may still differ in the multilayer stack from the values you measured in the separately tested, isolated layers. However, in addition to providing a good starting place for your model, the measurements on the single-layer samples will also give you more control over which parameters or specific layers you allow to vary as the model is refined.

Some applications, such as OLEDs (like ITO and PDO), can exhibit this tendency to have significantly different optical properties when measured in the full layer sack versus as separate, single-layer samples. For those kinds of applications, it can be useful to prepare samples with partial layer stacks as well.

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Imagine you have a stack of 3 layers (A, B, and C) on your substrate. If you observe that the optical properties of each layer measured separately are very different from the measured values when they are all stacked, you could try to do some intermediary tests with 2 layers at a time (e.g. A and B on the substrate; and then B and C on the substrate). Taking these measurements can allow you to build-up to a more accurate multilayer model.

Another thing to keep in mind is the **optical contrast** between the layers in your sample. If there is insufficient optical contrast between your materials, it can be hard to differentiate them and you won't be able to get accurate thickness measurements for individual layers. That said, the refractive index of materials can vary across the EM spectrum.

Consider a simple example with two layers: a photoresist on an anti-reflection coating. As both of these are organic, they have similar refractive indices in the visible range. Their differences lie in the UV spectrum. If you have an instrument that can detect in the UV range, you'll be able to differentiate the layers and compute their respective thicknesses.

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