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Technical Program May 6 – May 9
Education Program May 4 – May 9
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Integration of a Mid IR Tunable Quantum Cascade Laser Based Reflectance Spectrometer in a Roll to Roll Sputter Coater

Mike Simmons, Intellivation LLC., Loveland, CO

The integration of a Mid-Infrared (Mid IR) Tunable Quantum Cascade Laser (QCL) based reflectance spectrometer into a Roll-to-Roll (R2R) Sputter Coater offers enhanced spectral resolution and tunability, allowing for precise and selective analysis of materials during the coating process. Integrating this advanced spectrometer into a R2R Sputter Vacuum Coater enables real-time monitoring and control of thin film deposition, facilitating improved process optimization. The ability to monitor reflection across the IR wavelength contributes to the development of a much wider range of functional layer materials and provides a versatile and efficient manufacturing platform for high-throughput production of coated materials with tailored properties, offering new possibilities for applications in various industries.

https://www.svc.org

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INTELLIVATION
ENGINEERED SOLUTIONS

Integration of a Mid-IR Tunable Quantum Cascade Laser based Reflectance Spectrometer in a Roll to Roll Sputter Coater

MIKE SIMMONS
INTELLIVATION LLC

COMPANY

Engineering & Process Centric Equipment Company

- Founded in 2009, Celebrated 15 years.
- Innovation Center
- Located in Loveland, Colorado, USA

Production Roll to Roll Coating Equipment

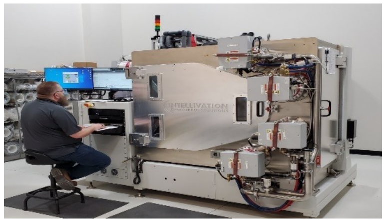
- Innovative R2R Systems up to 2 Meters Wide
- Advanced Process Technology & Monitoring Technologies
  - Reactive Sputtering Control
  - Magnetrons & Magnetics
  - Substrate Pre-Treatment
  - In-Situ Monitoring

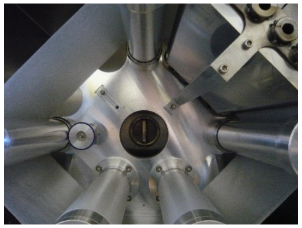
Vertically Integrated

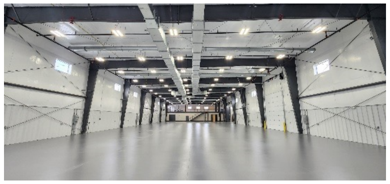
- >50,000 ft^2 Purpose-Built Facility
- Engineering, Machining, Fabrication, Assembly and Testing

In-House Application & Development Lab

- Process & Product Development
- Technology, Component & Process Demonstration



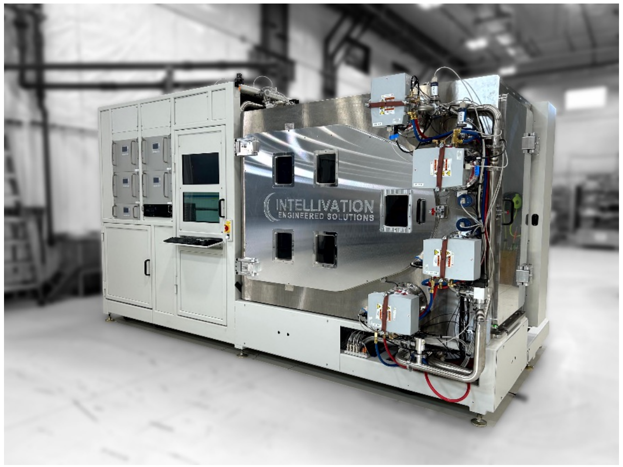


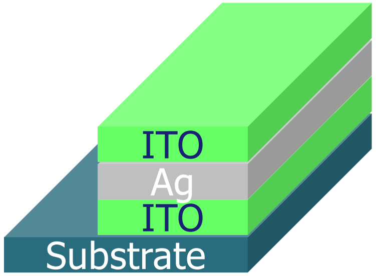


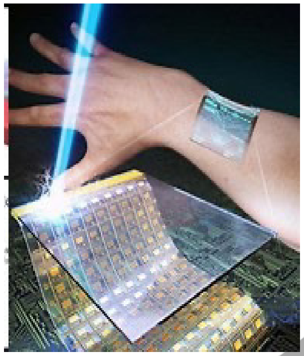
THIN FILM DEVELOPMENT APPLICATION LAB

Intellivation Application Lab

- **Process Demonstration and Development**
  - Complex multi-layer coatings
  - Metals, oxides, nitrides, oxynitrides
- **Interchangeable Deposition Modules**
  - Single or Dual Rotatable & Planar Magnetrons
  - Power Supplies: Bipolar Pulsed DC, DC, RF, HIPIMS
- **Many Target Materials Available On Site**
- **Rapid Coating Development**
- **Intellivation Process Know-how and Support**
- **Modeling and Design of Multi-Layer Coatings**
- **In-house Characterization Lab**

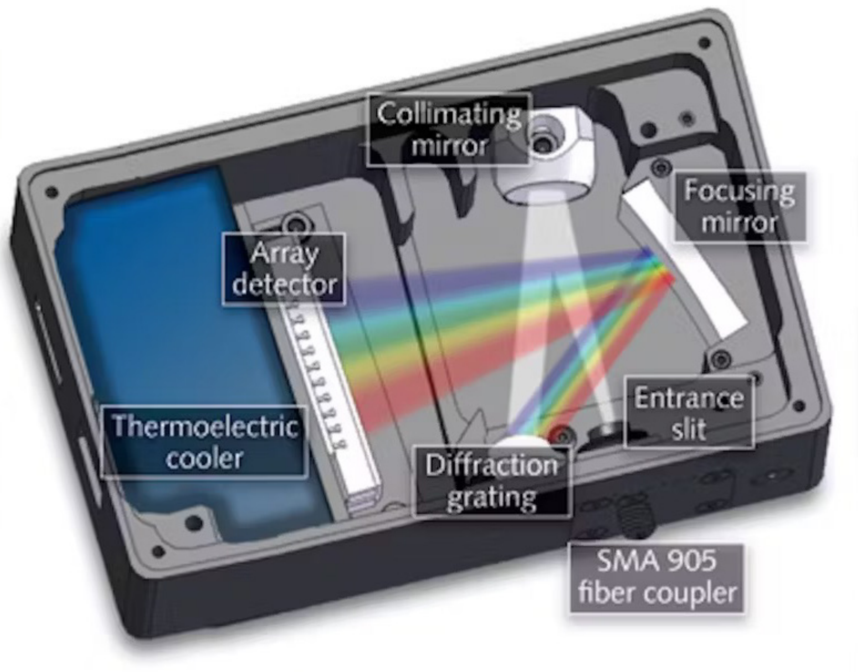






IN-SITU REFLECTANCE MONITORING OF UV-VIS THIN FILM OPTICAL COATINGS IN R2R COATER

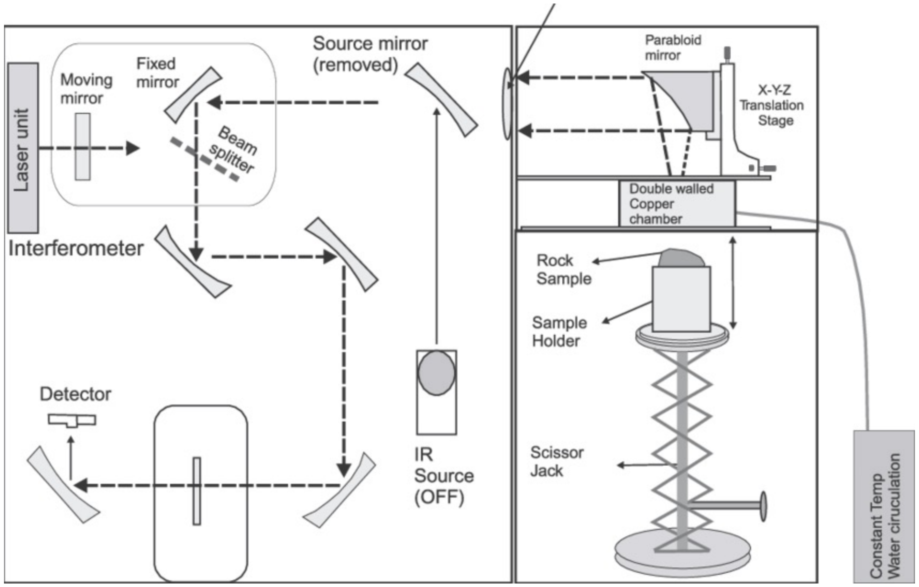
- **Benefits of In-Situ Monitoring**
  - Decreases Process Development Time
  - Increases Production Yield
  - Rapid Materials Development
- **In-Situ UV-VIS and NIR Optical Reflectance Metrology**
  - UV-VIS and NIR Spectroscopy is routine with COTS
    - Fiberoptics
    - High Spectral Power Light Sources
    - MEMS Optical Gratings and
    - CCD/CMOS Linear Array Detectors



IN-SITU REFLECTANCE MONITORING OF MID INFRARED THIN FILM OPTICAL COATINGS IN R2R COATER

Mid-IR Reflectance Presents a Challenge for In-Situ Metrology

- FTIR (Fourier Transform Infra Red) Optical Benches and Light Sources are challenging to integrate into R2R production equipment
  - Physical Size
  - Heat Dissipation
- Thermal Light Sources used for this traditional optical method have very low Spectral Power Densities requiring 100s of scans and requiring integration of 10s of minutes to achieve acceptable Signal to Noise Ratio.
- **FTIR measurement time is too slow for on-line measurement**



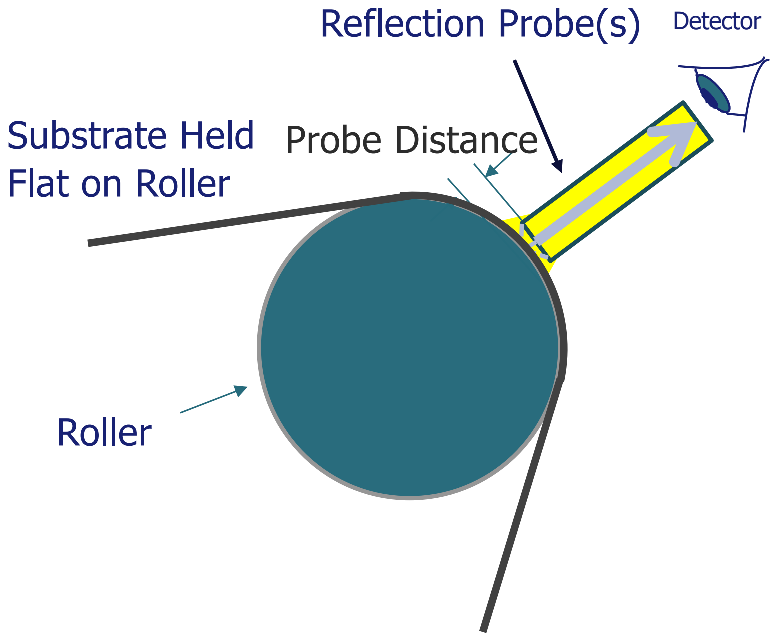
BENEFITS OF CROSS-SUBSTRATE IN-SITU MEASUREMENT

- **Accelerates Development**
  - Real time or fast vs. vent, remove, cut, measure
- **Process Tuning and Optimization**
  - Reactive deposition process
  - Complicated multilayer coating performance
- **Incorporation into Historian System**
  - Automatic integration into the data logger on the coating tool
- **May completely eliminate the need for ex-situ measurements**
  - For well-characterized materials

ALTERNATIVE SOLUTION
MID-IR SPECTROSCOPY TUNABLE QUANTUM CASCADE LASER LIGHT SOURCES

- **Scanning Quantum Cascade Lasers (sQCL) are Capable of Wide Tuning Ranges**
  - Ranges: 2 to 13 um
  - Spectral Power Density is 1 million times greater than thermal light sources used in FTIR measurements
- **Demonstration**
  - sQCL
  - Custom Fiber Optics for Reflection Measurement in Vacuum
  - Customized Signal Detection

PHYSICAL SETUP OF REFLECTION PROBES



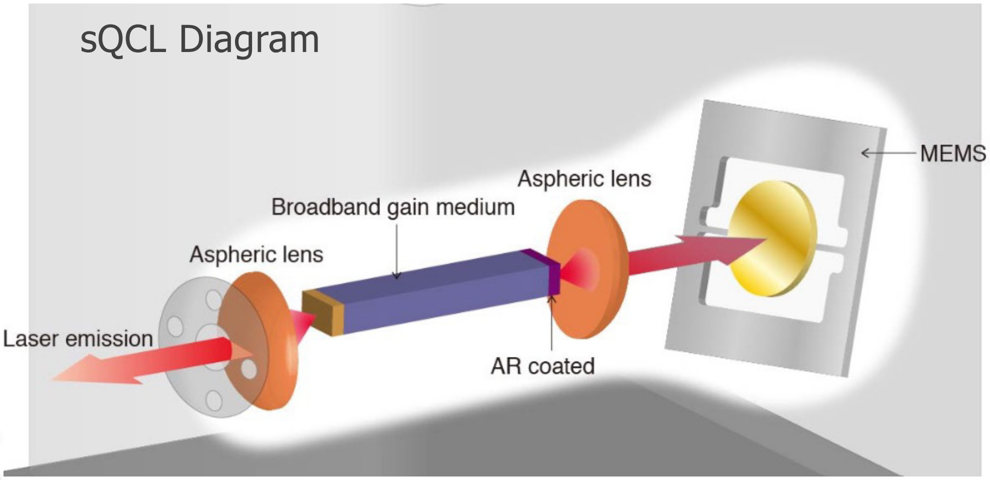
Comparing Light Sources

- **VIS**
  - Broadband through visible range
  - Tungsten Halogen
  - Transmitted from light source via quartz optical fibers
  - Reflection Probe for UV-VIS
- **sQCL**
  - Variable single wavelength
    - Think of a multi-color LED
  - Delivered from laser via optical fibers selected for high transmission in Mid-IR
  - Reflection Probe for Mid-IR
  - Customized opto-mechanical alignment hardware

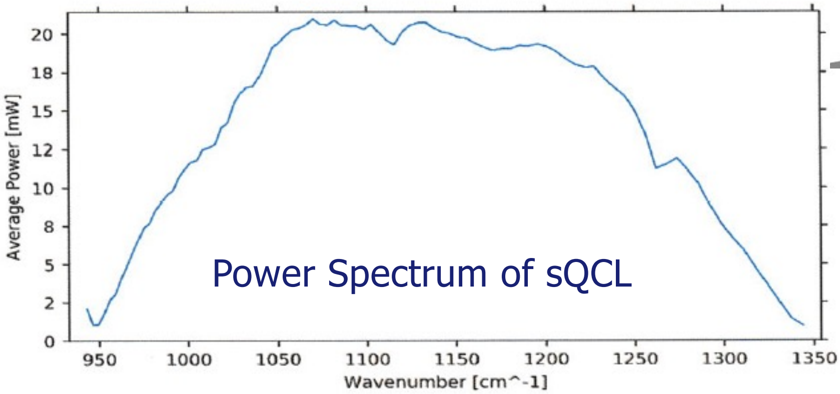
sQCL – SCANNING QUANTUM CASCADE LASER

- **LASER**
  - Light Amplification by Stimulated Emission of Radiation
- **Angle of grating selects wavelengths emitted by laser**

- **The gain medium is pulse pumped by a second light source timed with grating position**
  - Light is pumped in



sQCL Diagram

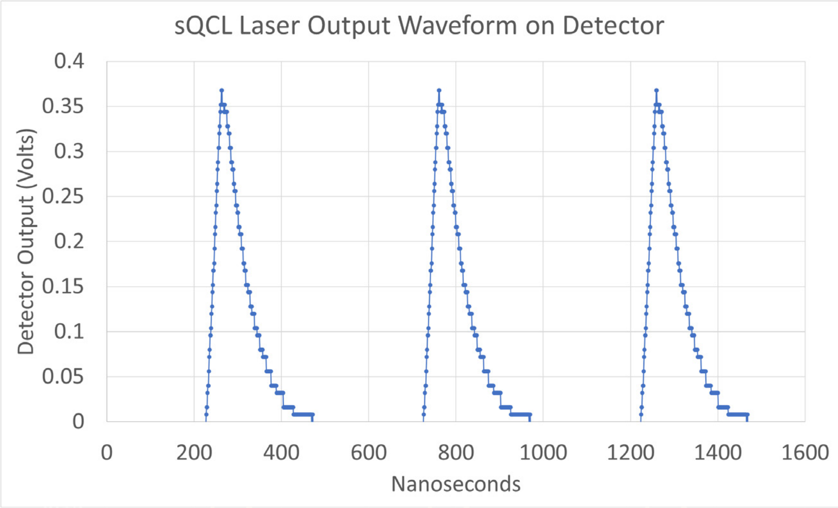


Power Spectrum of sQCL

- **20mW peak power**

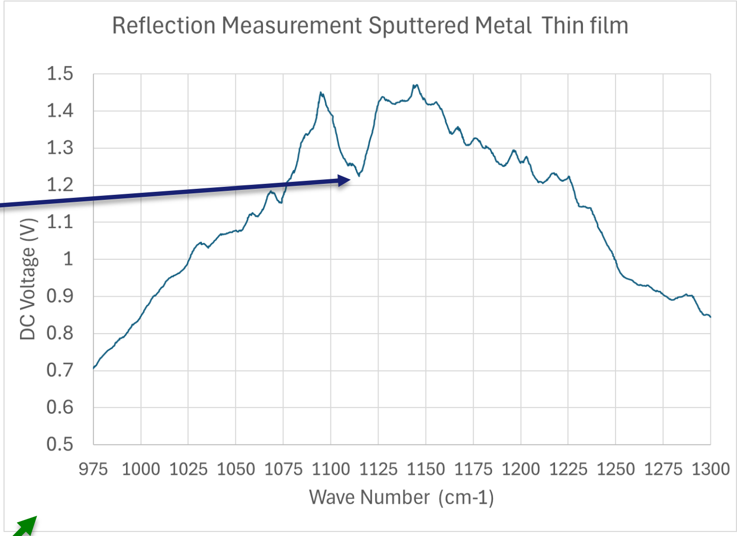
- **Suitable detector also required**

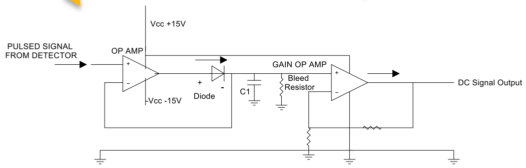
sQCL – Scanning Quantum Cascade Laser



Laser Pulses at Programmable Widths and Periods At Each Wave Number giving a temporal output that is transformed to WN/time

Is this the result of a metal oxygen vibrational mode?
See next slide

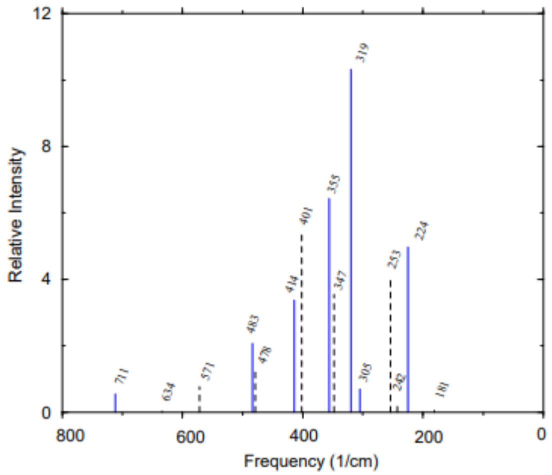




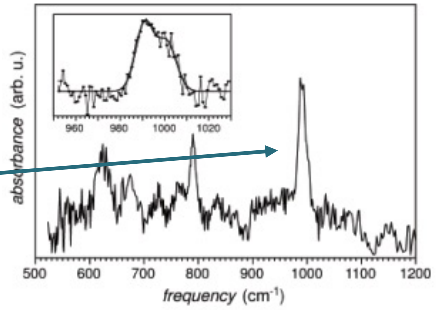
Spectral Response converted to DC Signal for 20 second scan

MID-IR SPECTRA OF METAL OXIDE

Vibrational Absorption Bands of a selected Metal Oxide

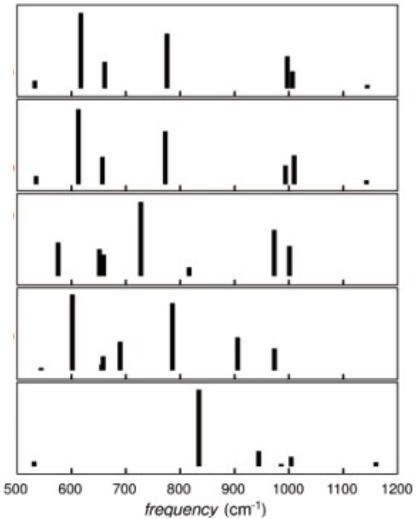


Example of a Metal Oxide with Overlap (absorption @ (1100 cm-1)

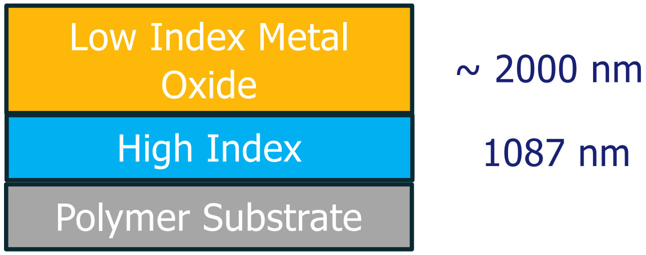


Mid-IR Spectral Region

- Can Overlap Vibrational Modes in some materials
- Review of Literature shows no expected overlap with thin film interference fringes.



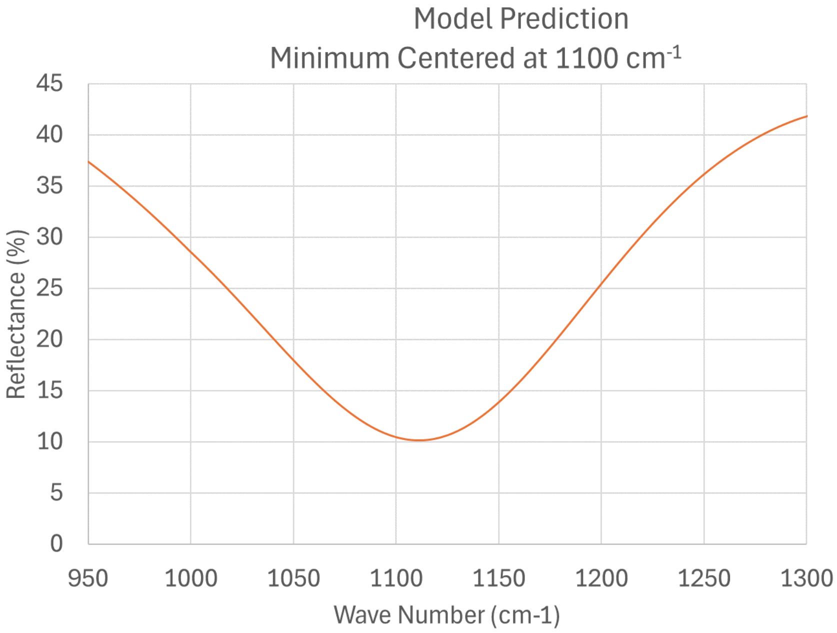
DEMONSTRATION WITH METAL OXIDE COATING



Requirements for This Coating Demonstration

- Reactively sputter a Two Layer Coating including a Low Index Metal Oxide to form a 2-layer AR Coating
- Hundreds of passes for deposition of thick optical coatings
- Reactive Sputtering Control for Stable High-Rate Deposition of Dielectric Thin Films over Multiple hours/days of operation
- Superior drum chilling to prevent heat damage of sensitive polymer substrates
- Integration of both UV-VIS and Mid-IR Reflection Measurements

THEORETICAL MODEL



Model of 2-layer AR Coat at 1125 cm-1

Prediction Assumptions

- We extrapolated Optical Dispersion to higher wavelengths from literature values
- Changes in index from model will adjust interference shape, amplitude and spectral location of the Reflectance minimum

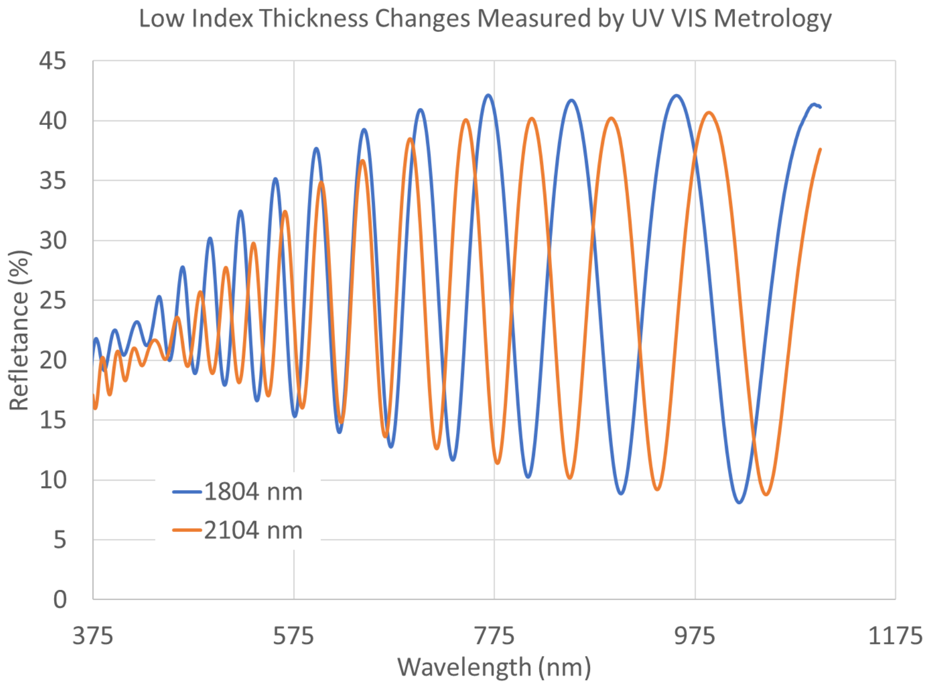
IN-SITU UV-VIS TO VALIDATE LAYER THICKNESS

- **Two thicknesses of the metal oxide coating were deposited and reflectance measured**

- **Determined physical thickness from spectra**
  - 1804 nm and 2104 nm

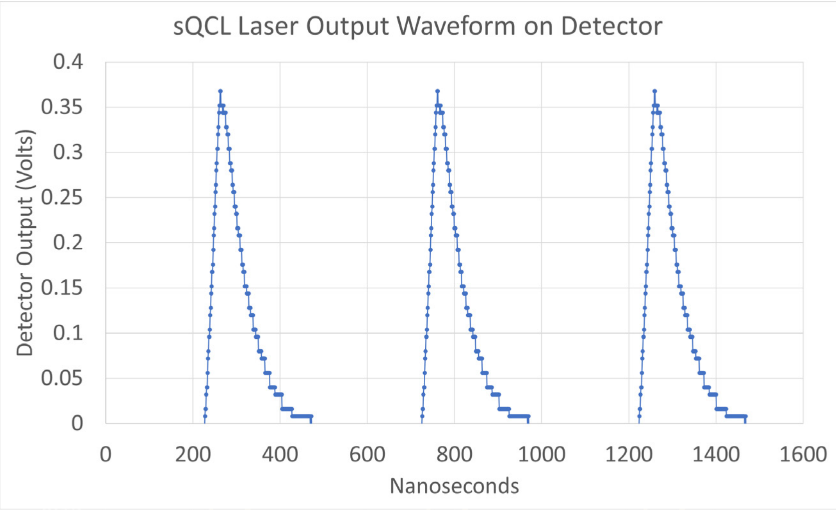
- **Reflectance data alone requires good knowledge of n and k for the film**
  - The exact n and k produced by the process

- **In-Situ Transmission measurements are helpful and also possible**



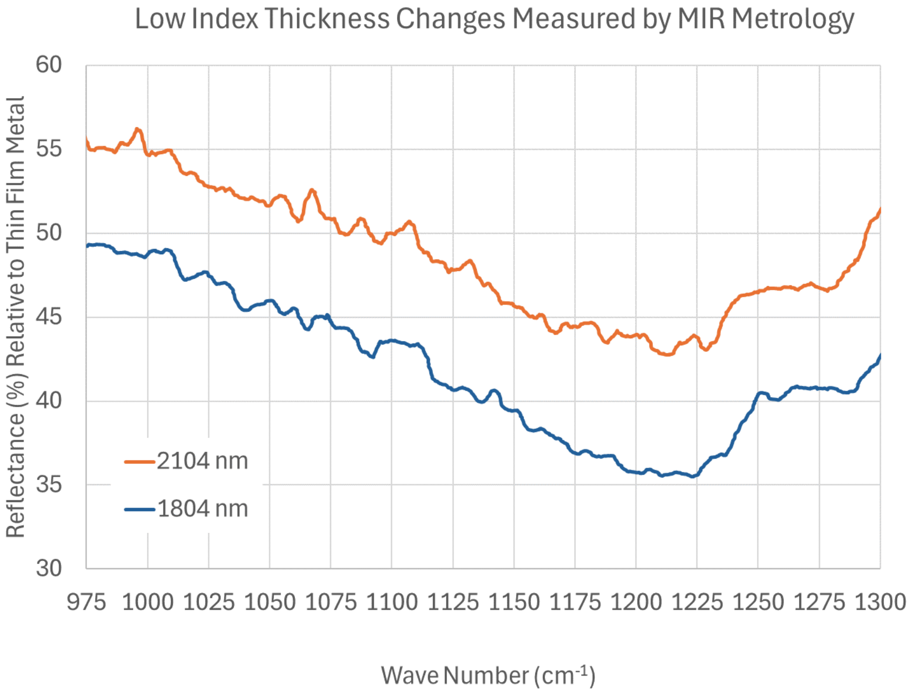
MID-IR sQCL MEASUREMENT DATA

- **sQCL emits Mid-IR pulses**
- **Detector detects reflected Mid-IR as a pulse**
- **Process that signal into wave number spectrum in order to see how the coating performed**



MID-IR sQCL MEASUREMENT DATA

- **Wave number spectral shape vs. target design**
- **Reflection data shows that 1804 nm is a better optical match (lower reflectance) than 2104 nm**
- **Minimum of Reflection curve in the Mid-IR spectra is shifted to 1200 cm-1 (from expected 1100 cm-1) which suggests that the extrapolated index is lower compared to this observation**



SUMMARY

- **Demonstrated Successful Integration of sQCL for Mid-IR Reflectance Measurement into R2R Coater**
- **Large Signal to Noise ratio at rapid scan rates is very encouraging for meaningful in-situ measurements**

  Note: Signal acquisition algorithm is being improved to remove noise
- **Reflection minimum spectral location of coating is consistent with expectations from optical models**
- **Separate off-line characterization of materials in the Mid-IR spectral range of interest needs to be added to optical models before making functional coatings**