COVALENT METROLOGY

Welcome

COVALENT METROLOGY

FAST CHARACTERIZATION OF NANOMETER THIN TO THICK COATINGS USING PULSED-RF GLOW DISCHARGE OPTICAL EMISSION SPECTROMETRY

SPEAKER:

Philippe Hunault
Technical Sales Elemental Analysis Specialist,
HORIBA Scientific

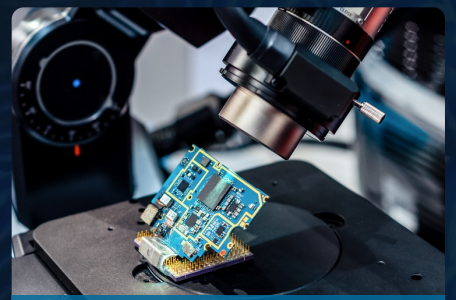
December 2, 2021 | 11am PT

HORIBA
Explore the future

COVALENT METROLOGY

COVALENT METROLOGY

Silicon Valley-based analytical labs and platform delivering quality data and expert analysis for advanced materials and device innovation



Comprehensive Solutions Stack

50+ cutting-edge instruments, offering 100+ Techniques

Analytical Services

Advanced Modeling

Method Development

Temp. Staffing Solutions

Affordable and Fast

Fast Turnaround Times, No Expedite Fees

Volume Savings

Instant Access to Data and Reports in Secure Portal

Flexible Business Model

Custom Consulting Solutions and Certified Onsite Support

Training and Certification on Instrumentation

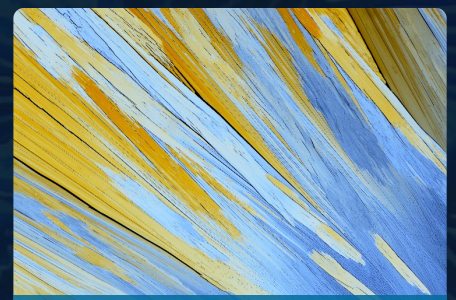
Co-op and Tool-Share Opportunities

Laboratory Audits

Rich Network of Partnerships

Partner to World's Leading Instrument Manufacturers and Labs

Expanding Instrumentation, Lab Connections and Learning



Who We Are, Who We Serve

500+ Clients, 40-60 Added / Quarter

50+ People, 14 PhDs

Cutting-edge Analytical Capabilities

Lab Location: Sunnyvale, CA

Covalent Technical Groups and Organization

COVALENT METROLOGY

PCBA, Semiconductor, and Electronic Device Metrology & Failure Analysis

- DPA / Mechanical Cross-section
- Dye & Pry Test
- EBIC / OBIC failure analysis
- Hot Spot Detection
- IR Imaging / Emission Microscopy
- NIR Imaging
- Root-Cause Failure Analysis

Electron Microscopy and Scanning Probe Microscopy

- AFM & Advanced AFM Modes (EFM, KPFM, MFM, PFM)
- Scanning Acoustic Microscopy (SAM)
- SEM (+ EDS)
- FIB-SEM (+ EDS)
- S/TEM (+ EDS / + EELS)
- Nano-indent / Nano-scratch

Optical Microscopy & Spectroscopy

- Chromatic Aberration
- Digital Optical Microscopy
- FTIR and ATR-FTIR
- Laser Scanning Confocal Microscopy
- Spectral Ellipsometry
- UV-Vis-NIR Spectroscopy
- White Light Interferometry

X-Ray Characterization

- X-Ray Diffraction (XRD)
- X-Ray Reflectometry (XRR)
- Micron-spot ED-XRF
- WDXRF
- Micro-computed X-ray Tomography (Micro-CT)
- 2D X-ray Inspection & X-ray Radiography

Elemental / Chemical Composition Analysis

- EPMA
- GD-OES
- GC-MS
- ICP-MS and LA-ICP-MS
- Raman Microscopy & Spectroscopy
- NMR (1D or 2D; solid / liquid)

Particle Analysis

- Dynamic Light Scattering (DLS)
- Laser Diffraction Particle Size Analysis (PSA)
- Particle Zeta Potential

Material Property Characterization

- DSC
- DMA & TMA
- Rheometry
- TGA
- Surface Zeta Potential
- Porometry / Porosity
- Gas Adsorption
- Gas Pycnometry
- Foam Density
- Tap Density

Surface Spectroscopy Analysis

- Dynamic-SIMS
- ToF-SIMS (Static-SIMS)
- Ion Scattering Spectroscopy (ISS)
- Ultraviolet Photoelectron Spectroscopy (UPS)
- X-ray Photoelectron Spectroscopy (XPS)

Covalent Partners

HORIBA Scientific

- **HORIBA Scientific North American Demonstration Lab** opened in August at Covalent's Silicon Valley headquarters
  - Installed **HORIBA GD Profiler 2 Glow Discharge Optical Emission Spectrometer**
    - Designed for high-speed, quantitative analysis of all elements of interest, including nitrogen, oxygen, hydrogen and chlorine
    - An ideal tool for thin and thick films characterization and process studies
  - Showcases HORIBA's top-of-line spectroscopy solutions
  - Bolsters mutual efforts to develop new methods for chemical analysis applications

Other Covalent Partners

Rigaku

ThermoFisher SCIENTIFIC

J.A. Woollam

KEYENCE

Anton Paar

PVA TePla

Asylum Research an Oxford Instruments company

TA Instruments

Digital Surf

TELEDYNE CETAC TECHNOLOGIES Everywhereyoulook™

Philippe Hunault

Technical Sales Elemental Analysis Specialist,
HORIBA Scientific

- Received Instrumentation Engineer degree in France in 1979
- Involved in Alpha, Beta and Gamma Spectroscopy at the beginning of his career
- Joined HORIBA Jobin Yvon in 1985 as the Glow Discharge Optical Emission Spectrometry product manager
- After promoting GD-OES worldwide for 12 years, joined the HORIBA team in the United States
- Spent the past 17 years helping to promote HORIBA Elemental analyzers in North America

HORIBA

Scientific

Covalent – HORIBA Webinar
December 2, 2021
Tutorial on GD-OES

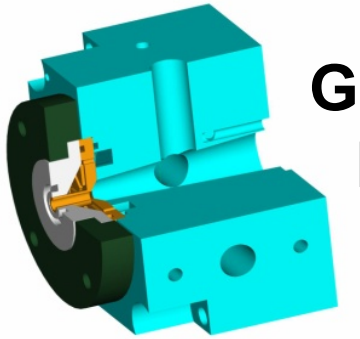
Fast Characterization of nm Thin to Thick Coatings using Pulsed-Rf Glow Discharge Optical Emission Spectrometry

Philippe Hunault , HORIBA Scientific, Piscataway, NJ

Topics of the Presentation

- Introduction to Rf GD-OES
- GD PROFILER 2 Instrument
- Various Applications
  - ✓ Hard Disk, DVD, Media
  - ✓ Li Ion Batteries
  - ✓ Photovoltaic
  - ✓ Semi Conductor
  - ✓ Thin and Thick Oxides
- DIP the latest innovation in GD-OES technique…
- Conclusion

From the sample… …to the Result



Glow Discharge Light Source

Spectrometer

(Optical Emission Spectrometer / OES)

Signal Processing

Data Processing

Chemical Composition

Introduction to Rf GD-OES

Excitation Mechanism

Anode - Cathode

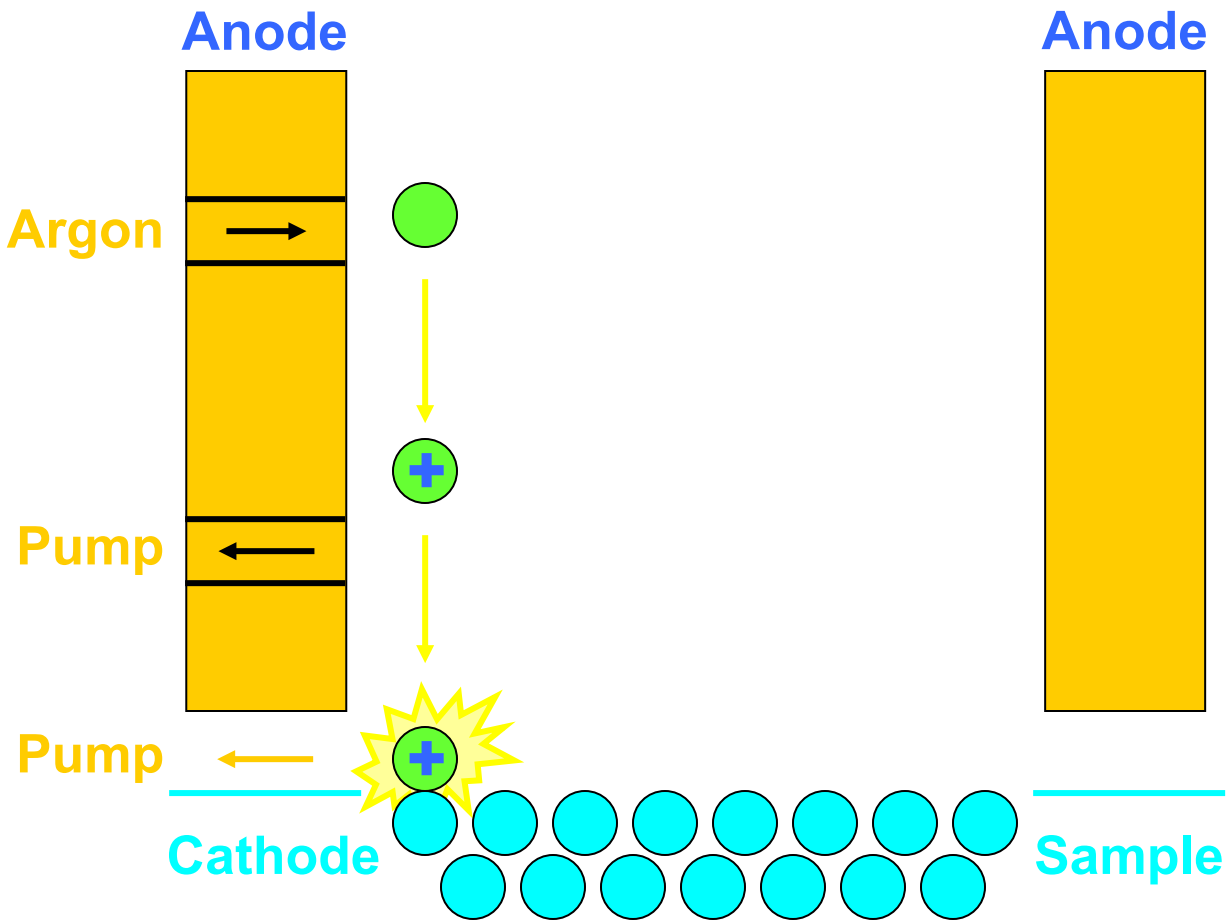
Cathode = Sample

Vacuum

Argon Filling

HV Applied
Ar Ionization

Ionic Bombardment



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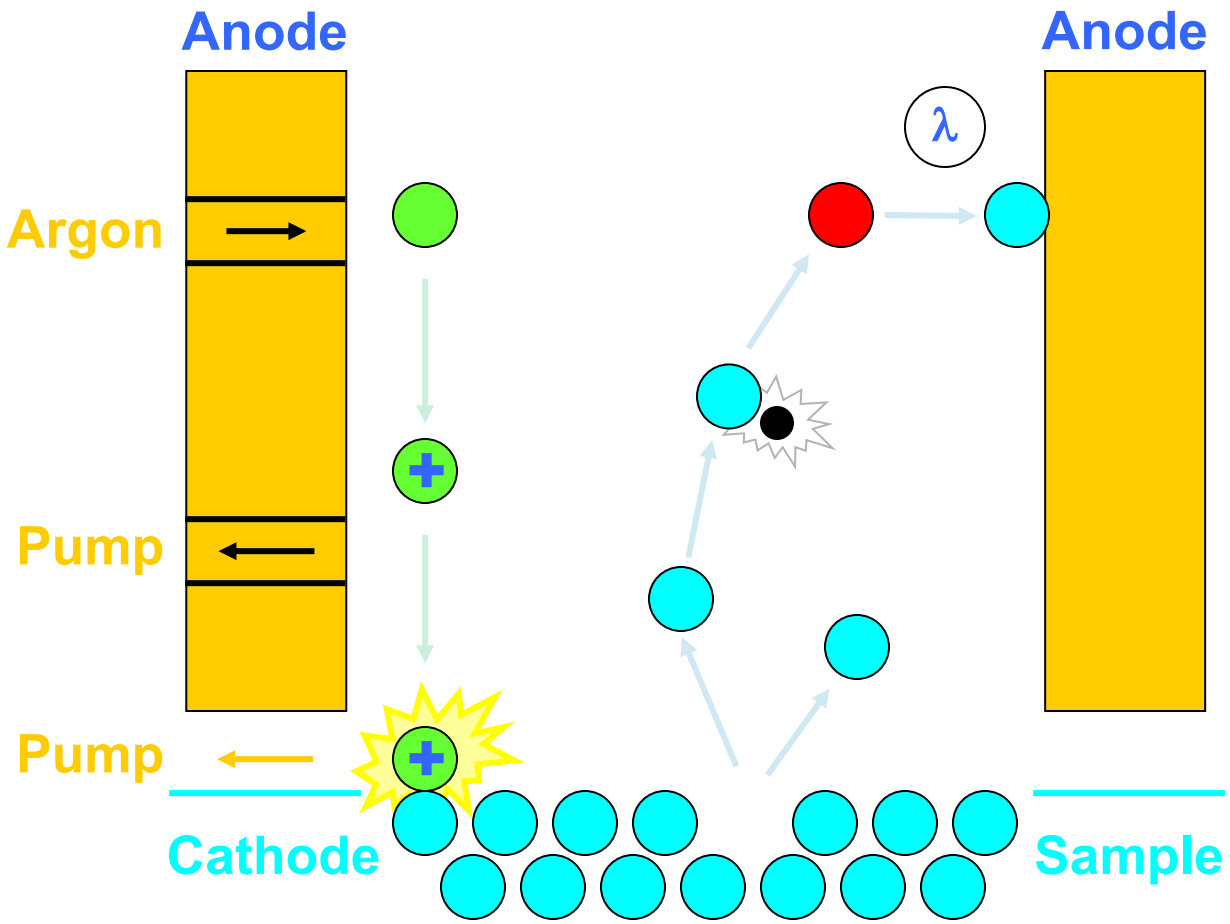
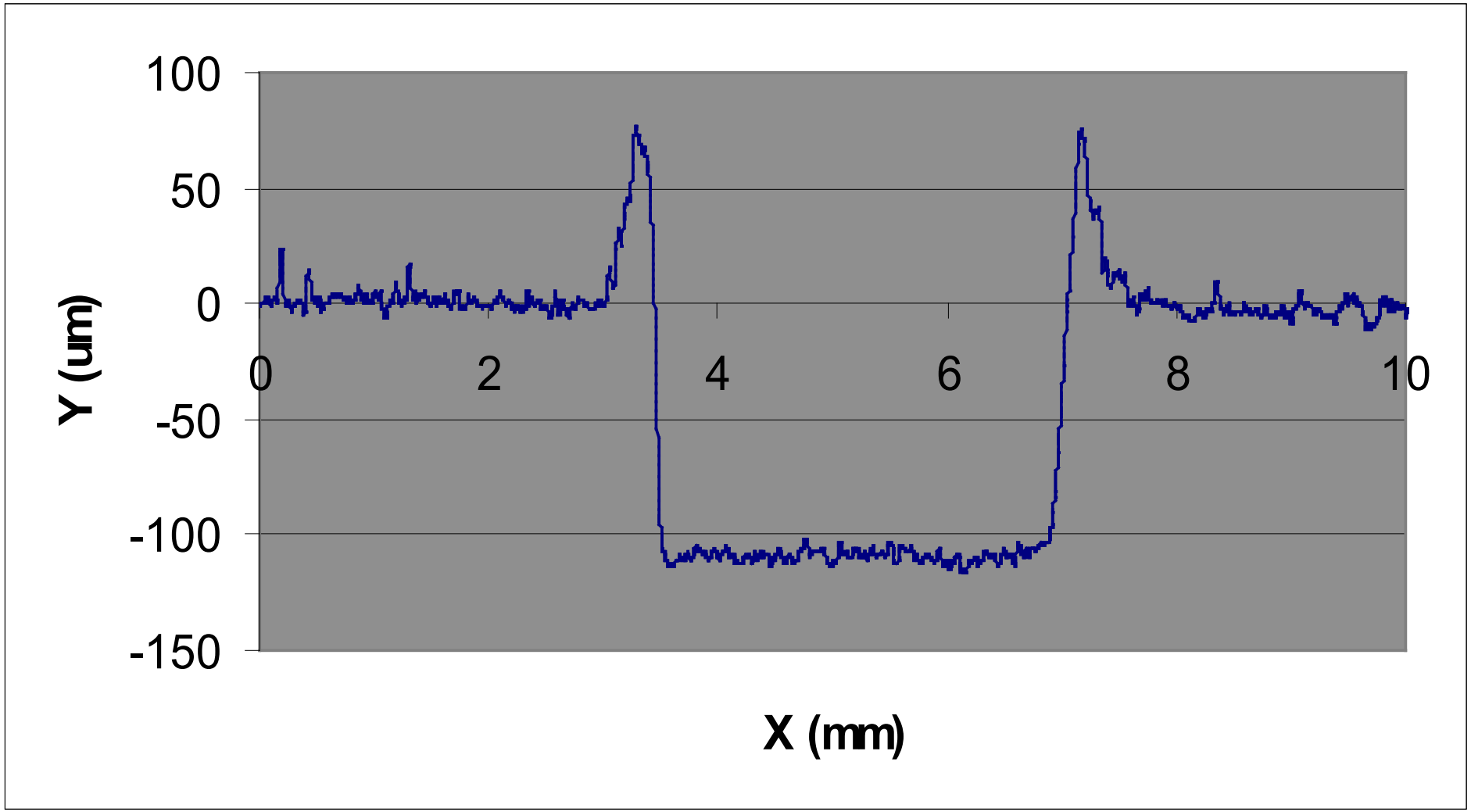


Sample Sputtering
Atoms Ejected

Atoms
Excitation

Light Emission
Wavelength

Typical Rf Crater in Steel



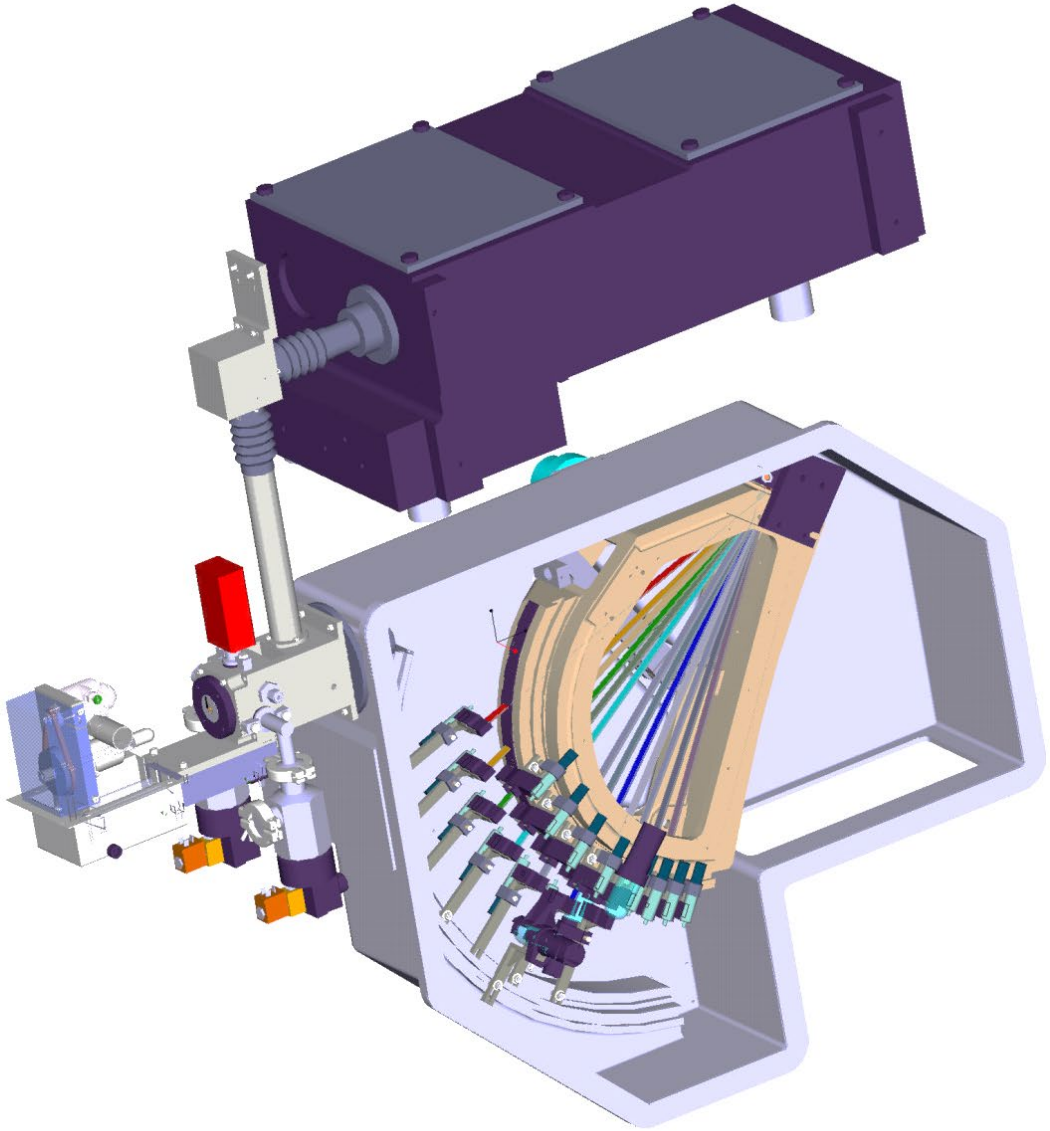
Operating Conditions

Argon Pressure: 1-10 Torr

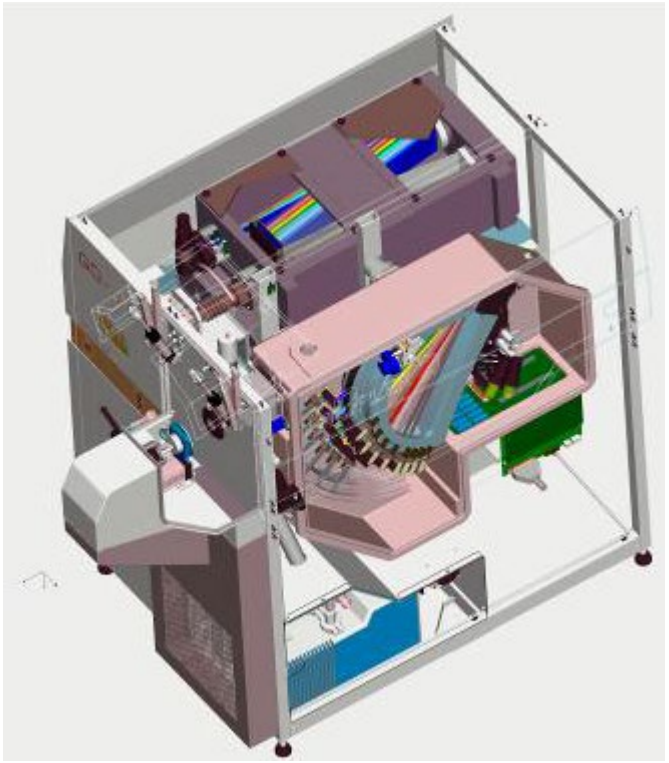
RF Power: 20-80 W

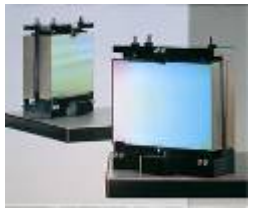
⇒ **Fast sputtering rate**
10-150 nm/s
(microns/min)

GD Profiler 2 Optics



GD Profiler 2



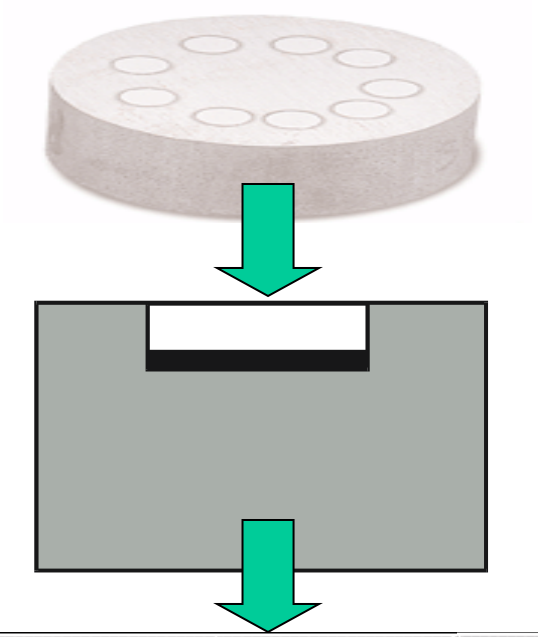


All elements measurable

Spectral range from H 121nm to Li 670nm & K 766nm

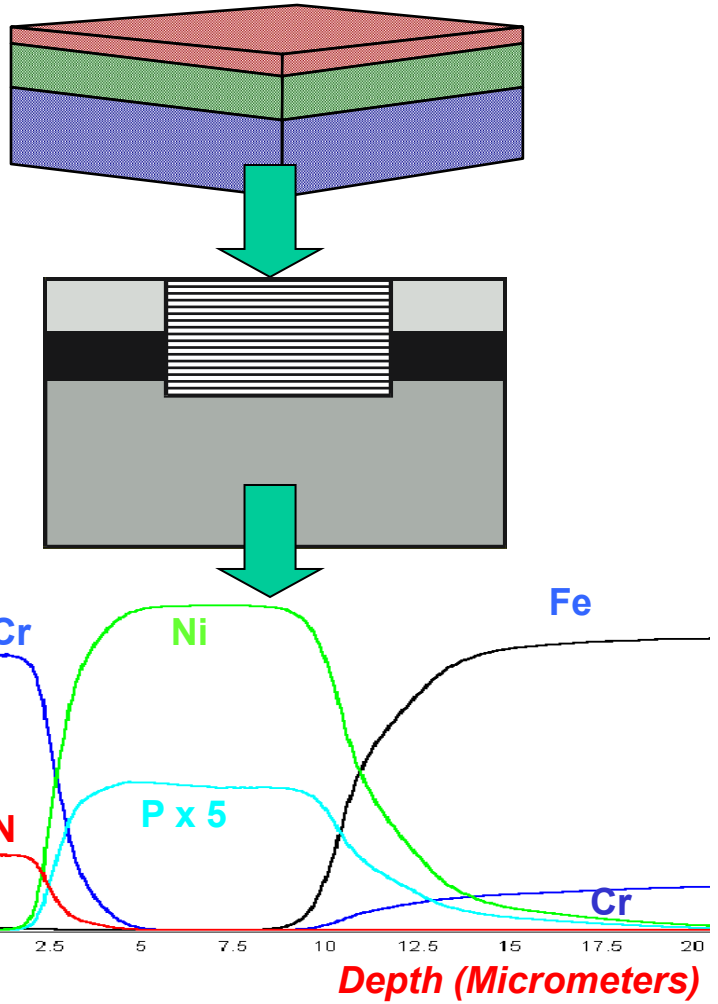
GD-OES Applications

Bulk Analysis

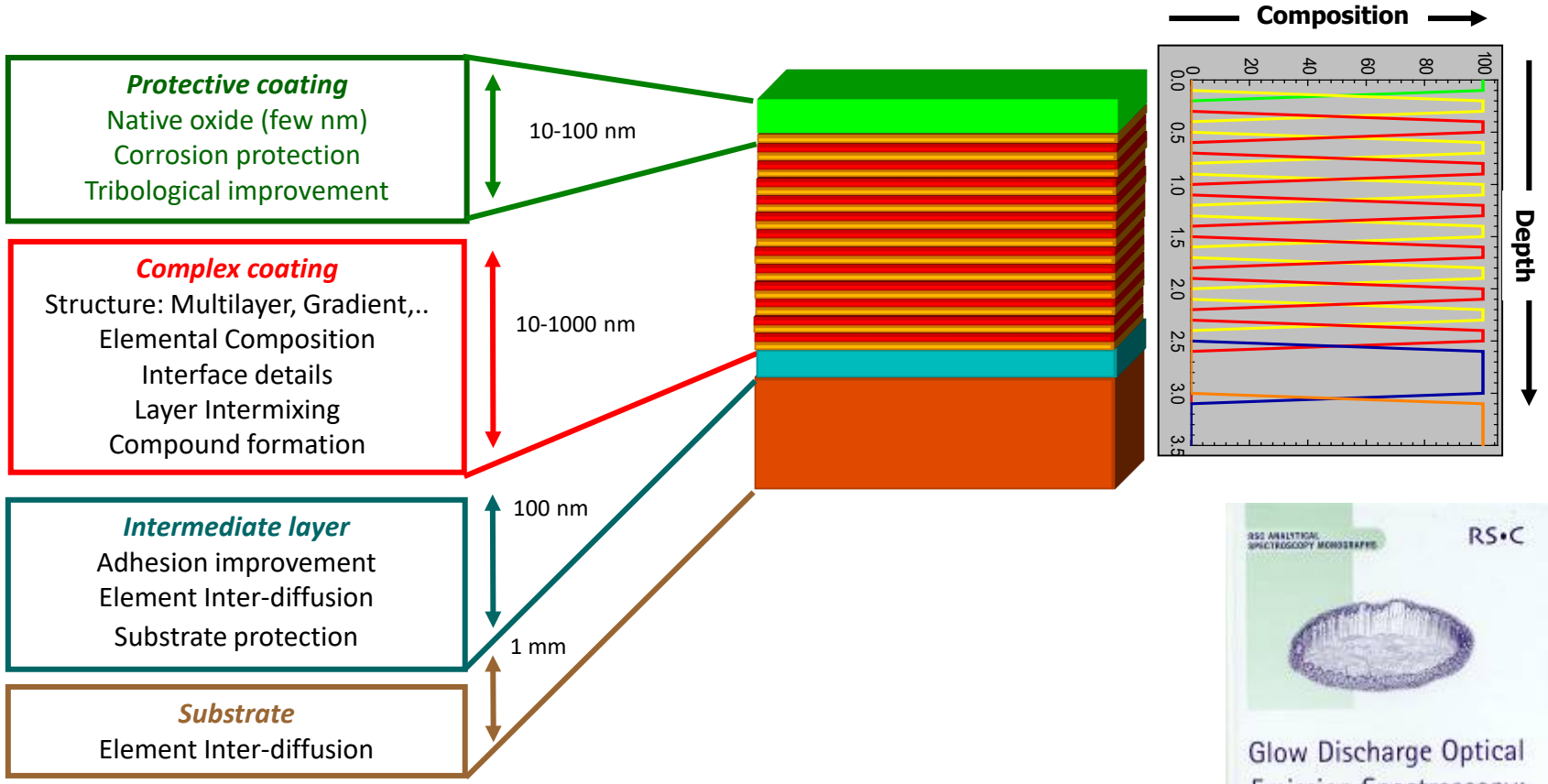


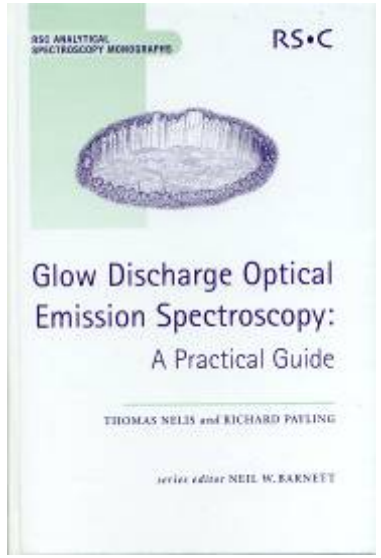
	Burn 1	Burn 2	AVG
C	0.159	0.160	0.160
P	0.00469	0.00470	0.00470
S	0.00451	0.00466	0.00460
Si	0.0112	0.0113	0.0112
Mn	0.0287	0.0286	0.0286

Depth Profiling



Variety of Applications



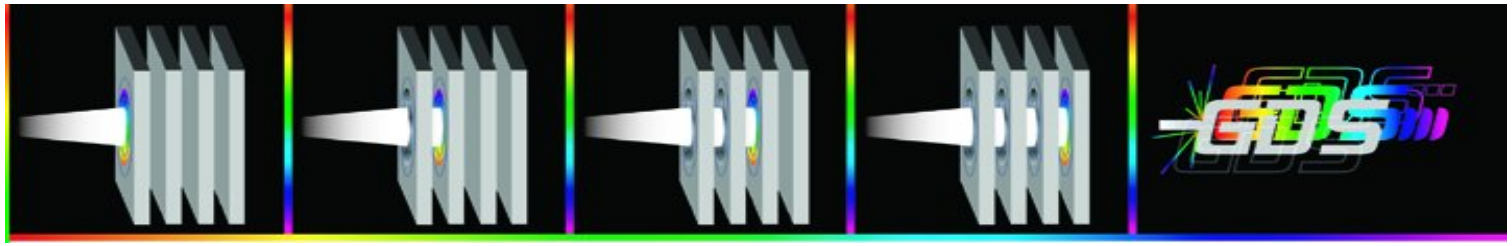


Thin/thick films

Applications of Depth Profiling by GD-OES

- Steel Industry (Galvanization, Anti corrosion coating…)
- Automobile (Engine, Body, Windscreen…)
- Aeronautic (Light materials, Ti, special alloys…)
- Li ion Batteries
- Semi Conductors (Wafers…)
- Photo Voltaic, Solar Cel (CIGS, CdTe, Ga As…)
- Heat Treatment (C, N at the surface)
- Plating, Electro plating…
- PVD, CVD coating (TiN, CrN, …)
- Glass (coating on glass)
- Organic coatings
- New Materials
- Failures and Defects Analysis
- Process Control

Glow Discharge – Optical Emission Spectroscopy



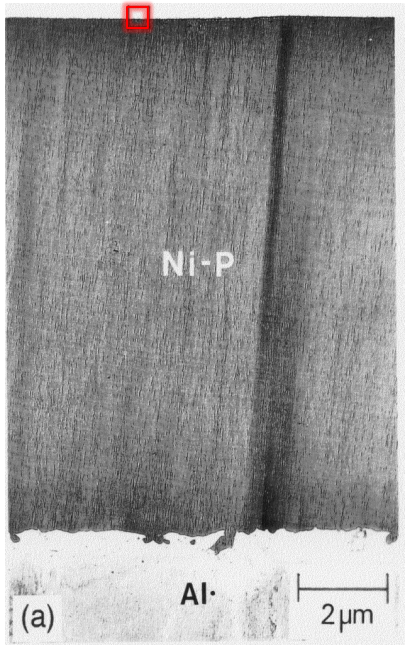
- Direct Elemental Analysis of Solid Samples (no sample prep.)
- Bulk analysis and Depth Profile Analysis
- Conductive and non conductive materials/layers
- Detection Limits 1-10ppm
- Concentration Dynamic: LOD – 100%
- Depth Dynamic: below nm to 150 Microns+
- In-depth resolution: below nm depending on roughness
- Simultaneous analysis of almost all elements including O, N, H, Cl, F, Li…
- Fast sputtering rate – Rapid analysis
- Easy and fast sample mounting (sample NOT in vacuum)
- Affordable price compared to SIMS, XPS…

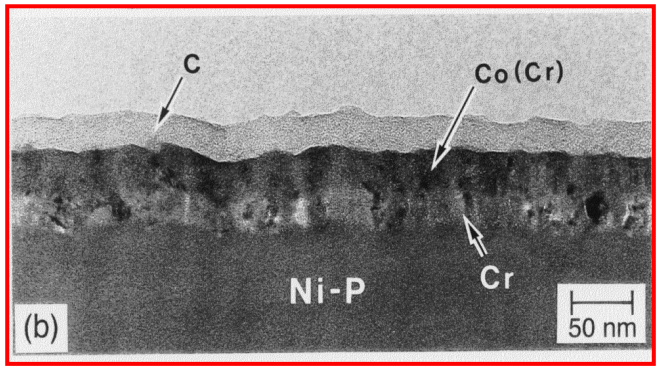
Pulsed Rf - GD-OES Applications

Hard Discs, DVD…
Thin & Thick Layers

Hard Disc Analysis

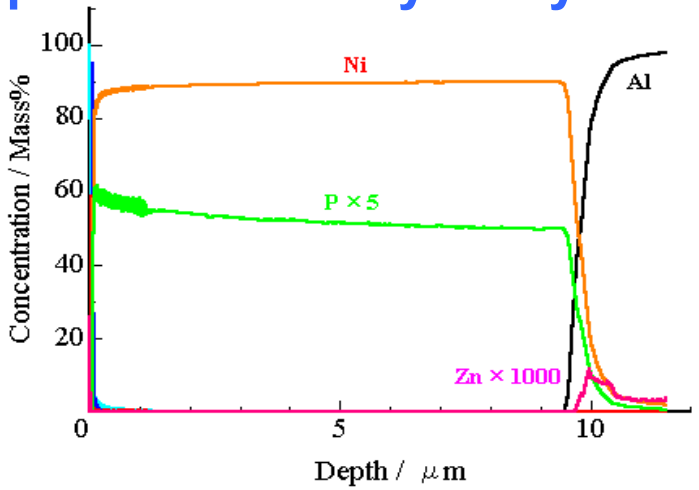
TEM Picture of the sample



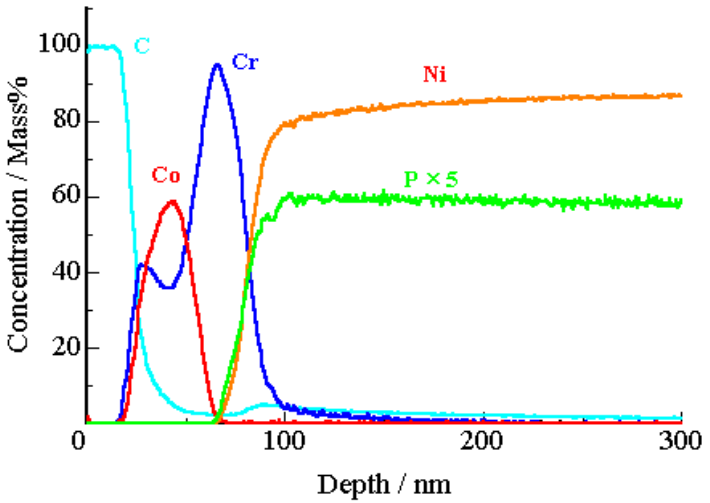


Zoom at the surface

Depth Profile Analysis by GD-OES

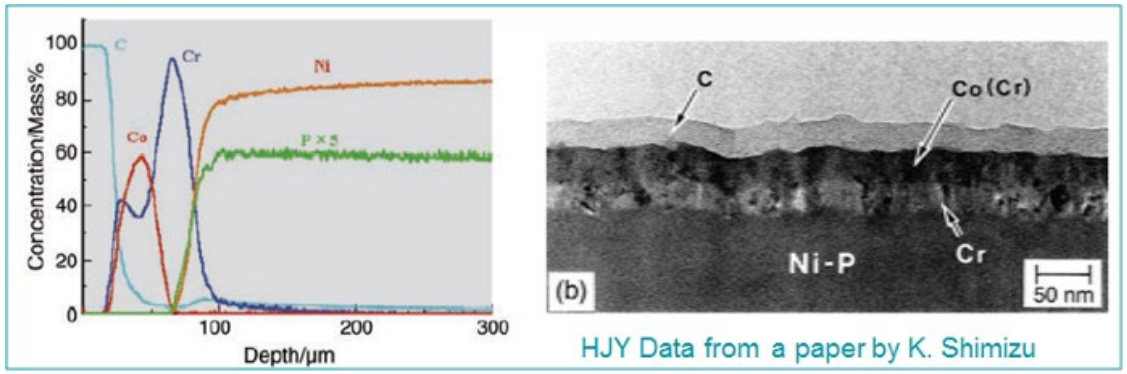


Zoom at the surface



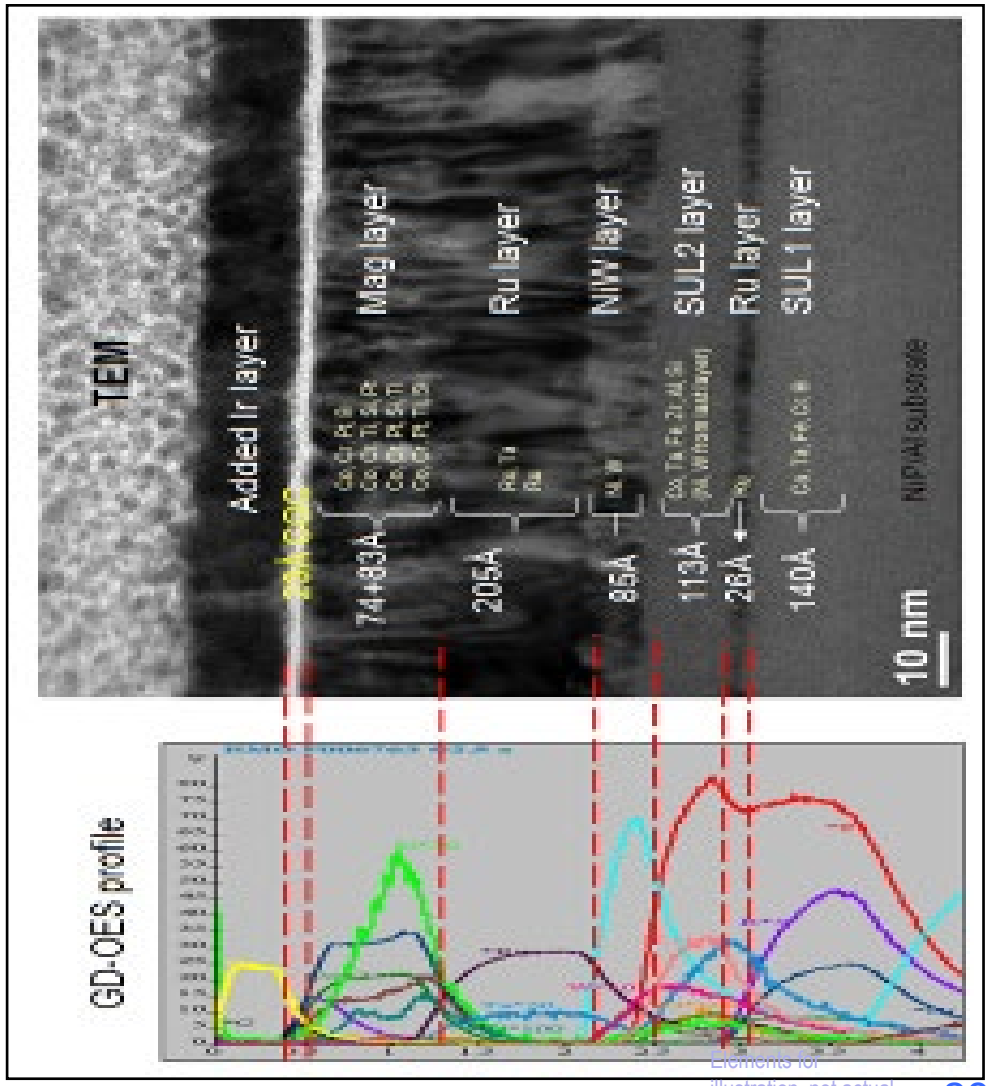
Hard Discs Revolution – From 1st GD Application to Now

- 1st generation longitudinal media before 1996
  - 4 layers, each layer about > 20 nm thick



- 7th generation perpendicular media in 2012
  - As many as 18 layers, total thickness < 100 nm
- Thinnest layer < 1 nm

Seagate



Elements for illustration, not actual

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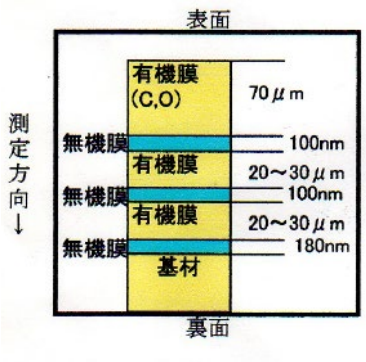
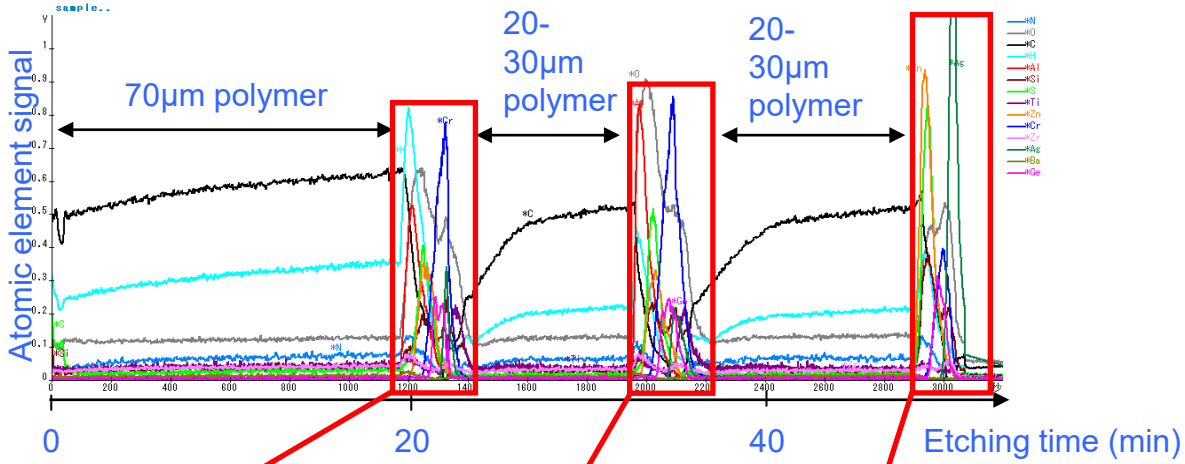
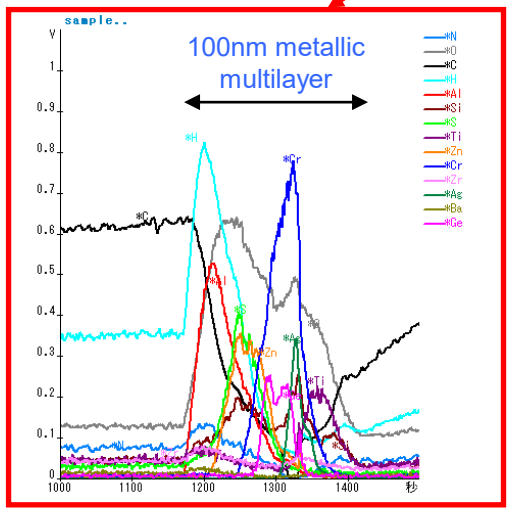
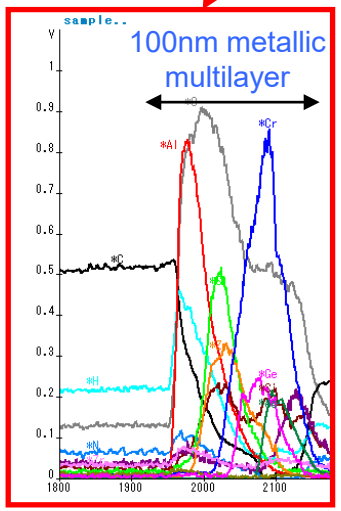
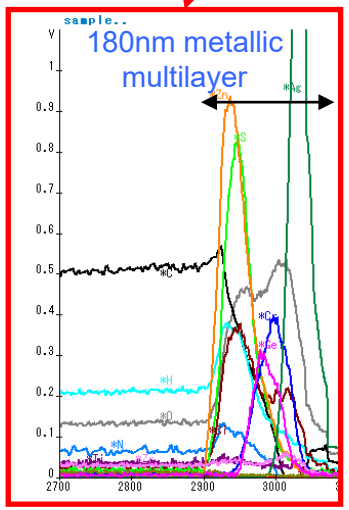
DVD – Ultra Fast Sputtering UFS

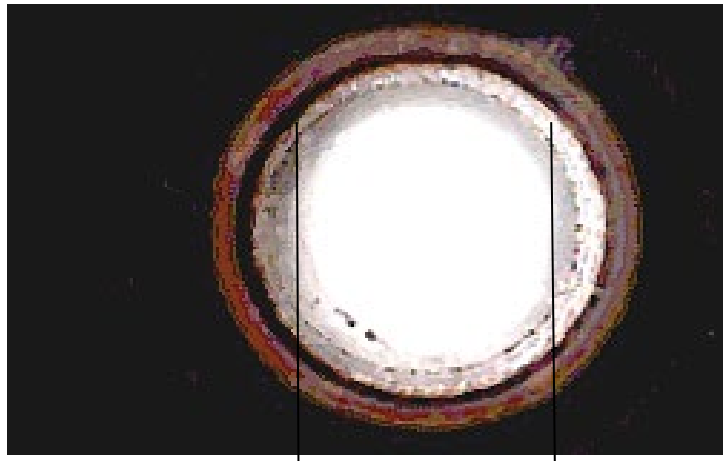


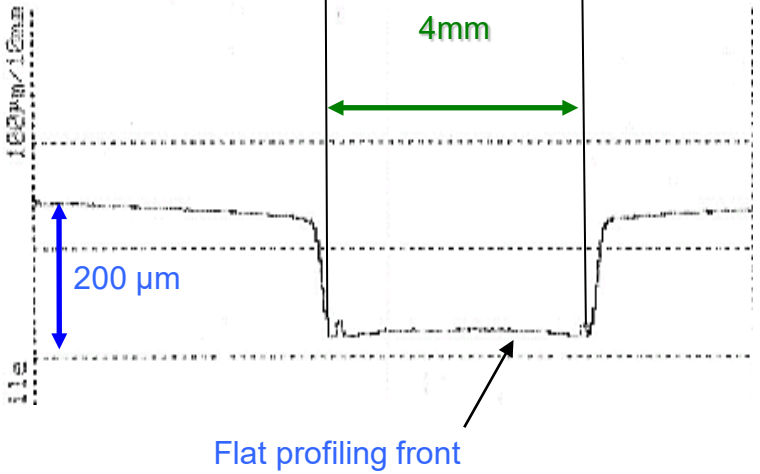




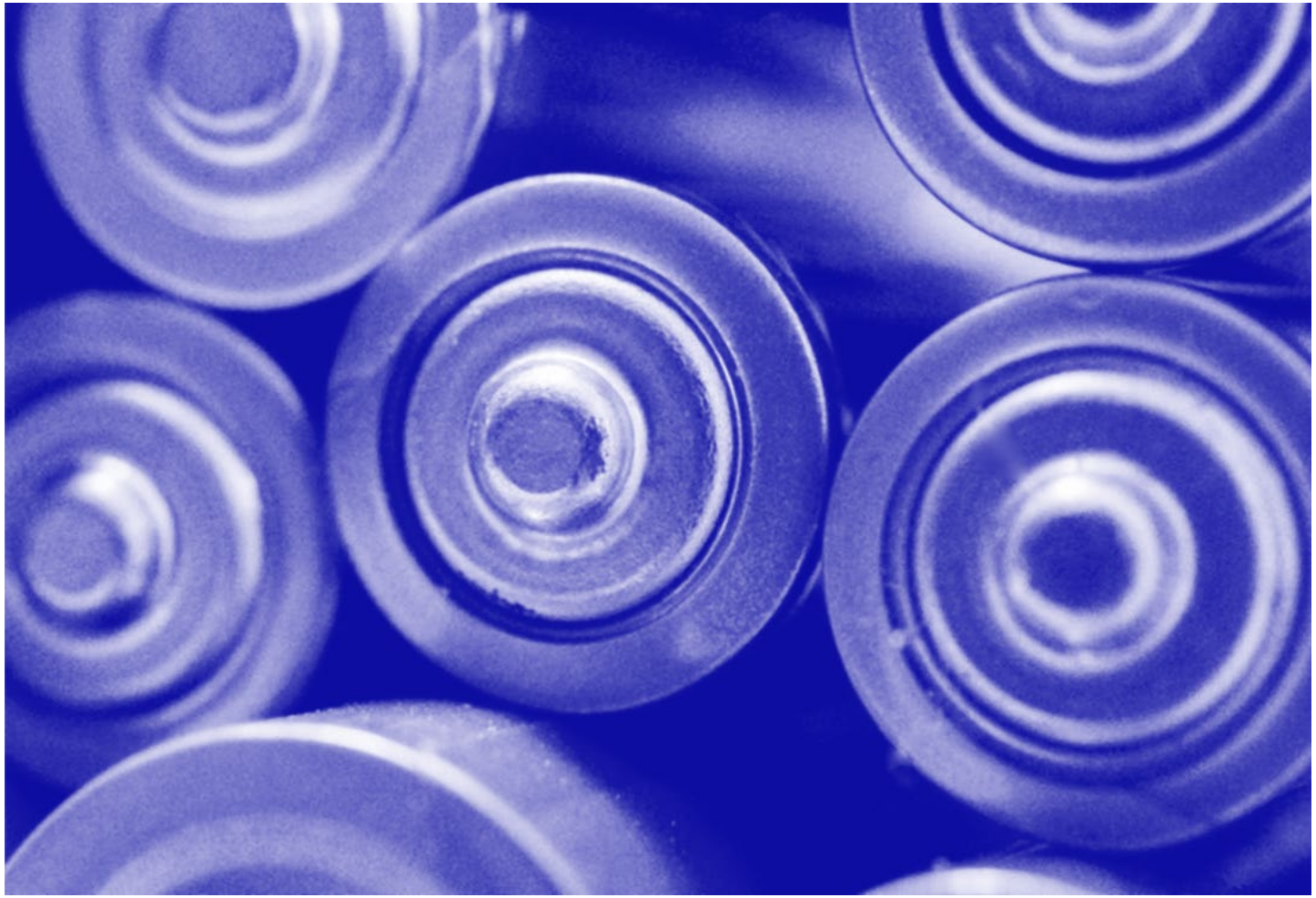




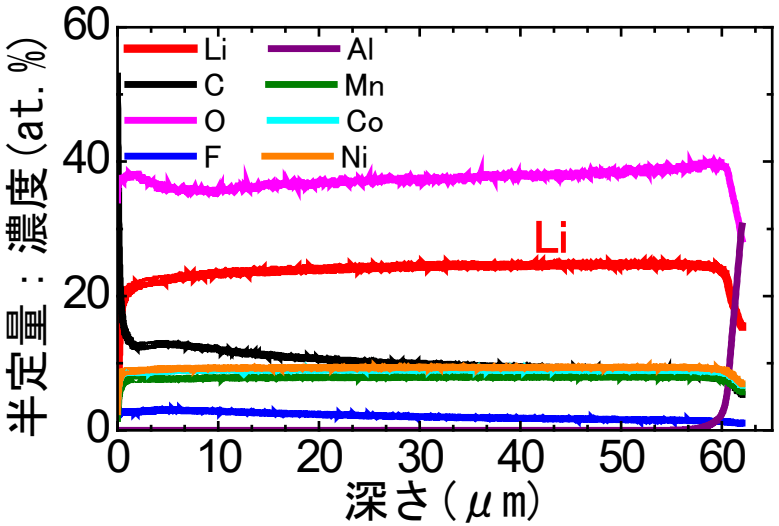




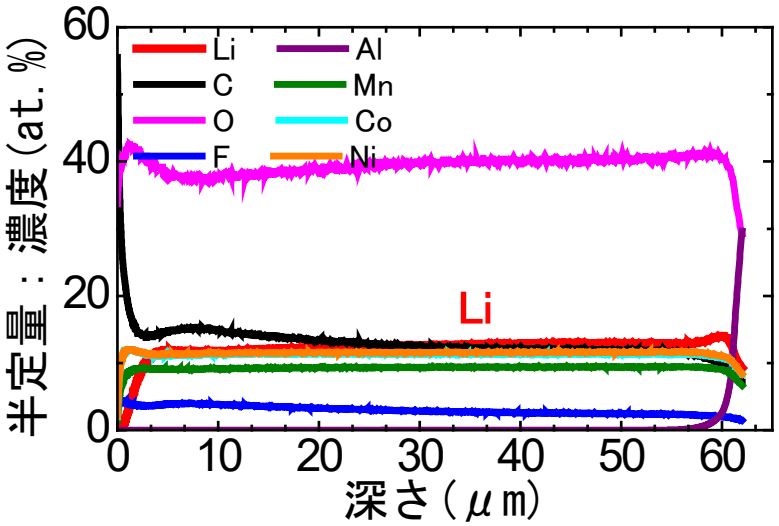
Li Ion Batteries



Depth Profile – Li Ion Battery Electrode



正極 SOC:0%

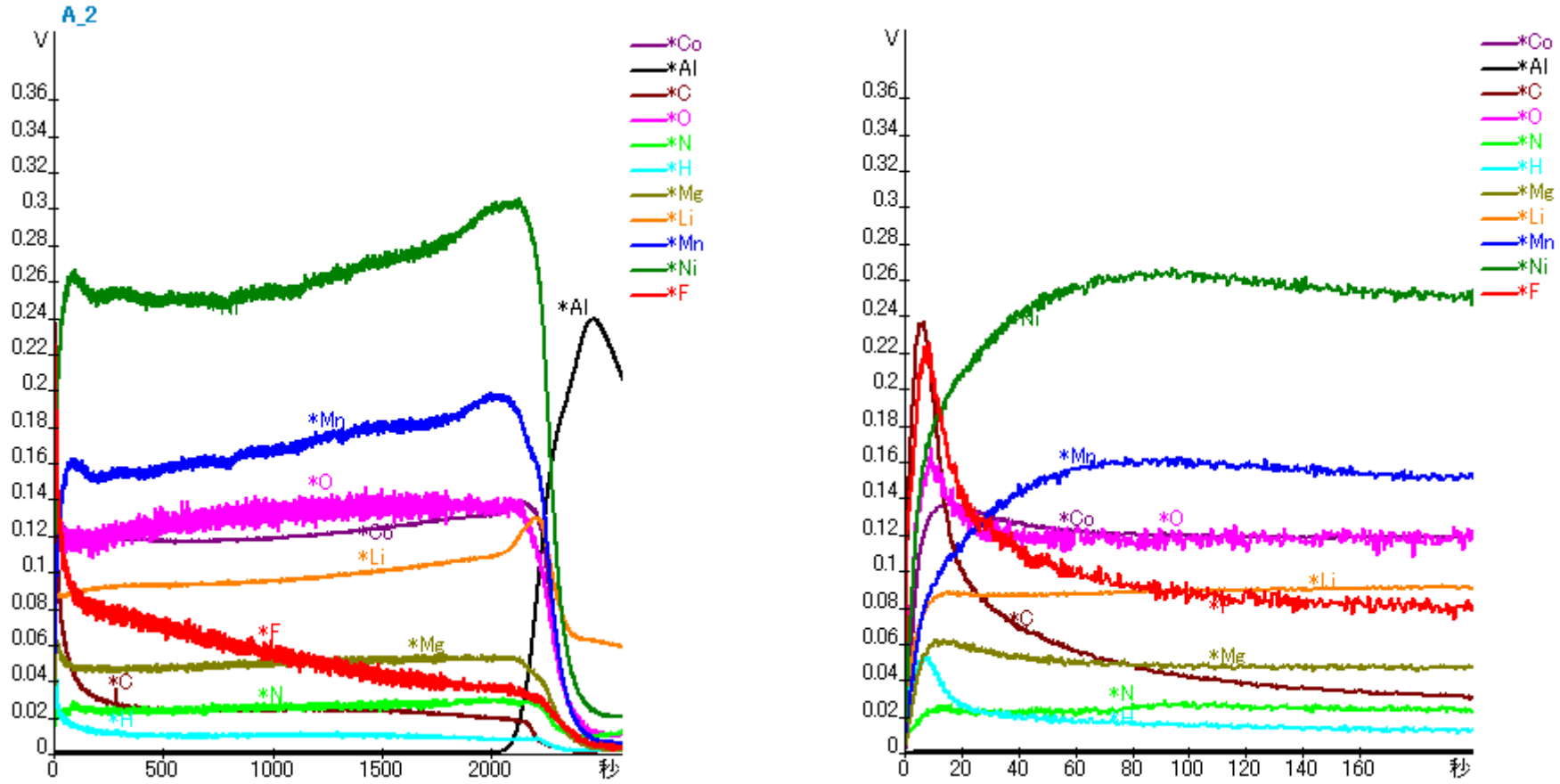


正極 SOC:100%

Comparison of 2 samples with different
State of Charge: 0% and 100%

Ref: Kobelco GD day

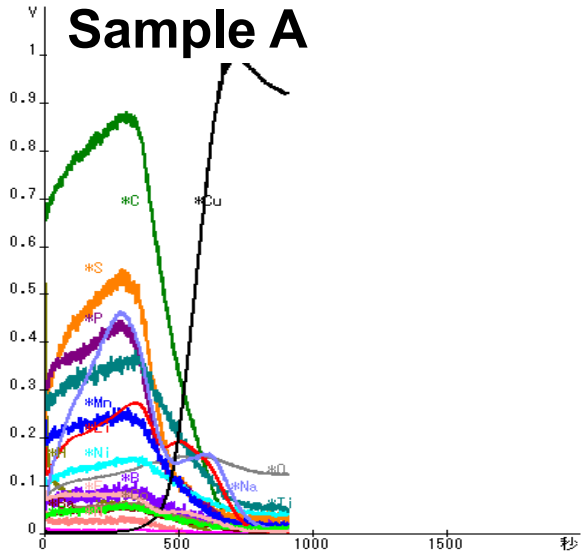
Depth Profile on Li Ion Battery Electrode

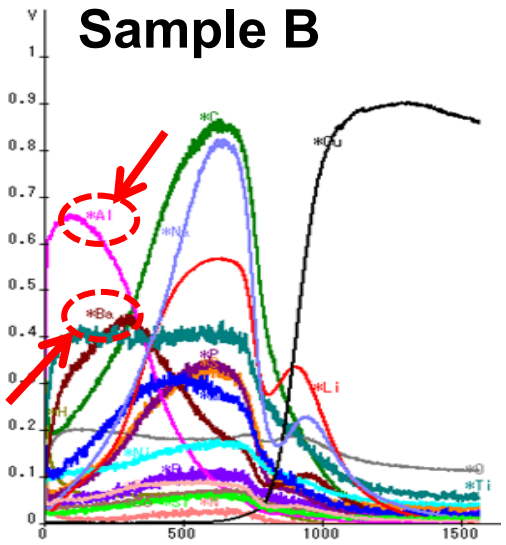


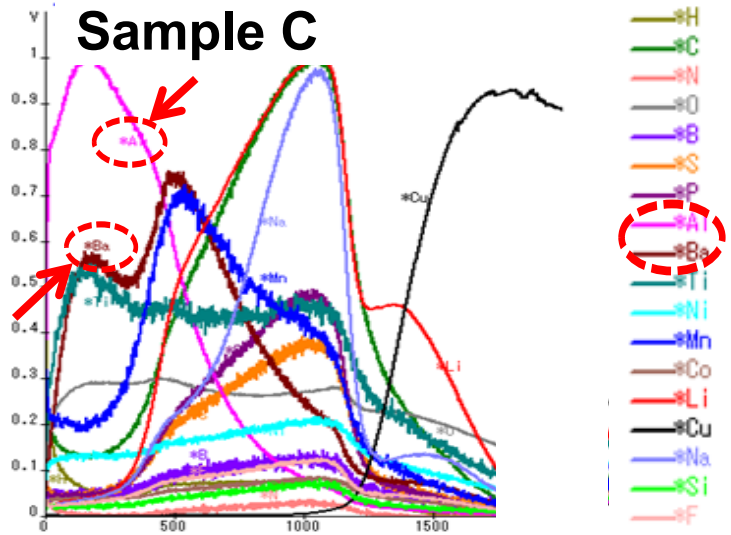
Uses Ne as main plasma gas to excite F along with other elements

Li Ion Batteries

- Degradation evaluation of cathode material
  - Dissolution of anode collector
  - Possible reaction with cathode active material

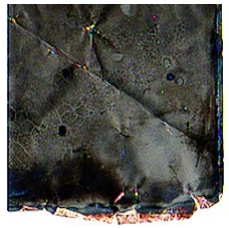






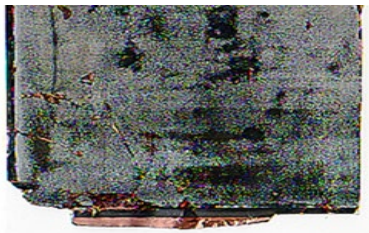
Sample A

No damage



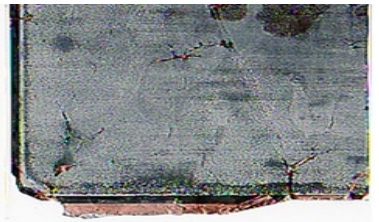
Sample B

Moderate damage



Sample C

Most damage

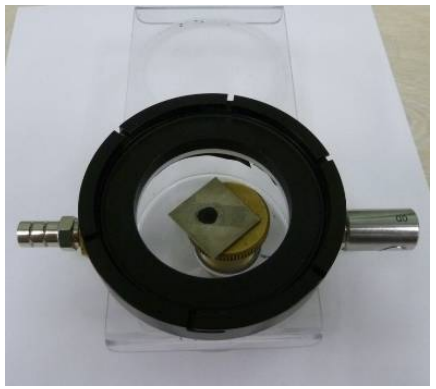


HORIBA Scientific

Accessory: Li bell

- For flammable and air sensitive samples
- Preparation inside glove box (under inert atmosphere)

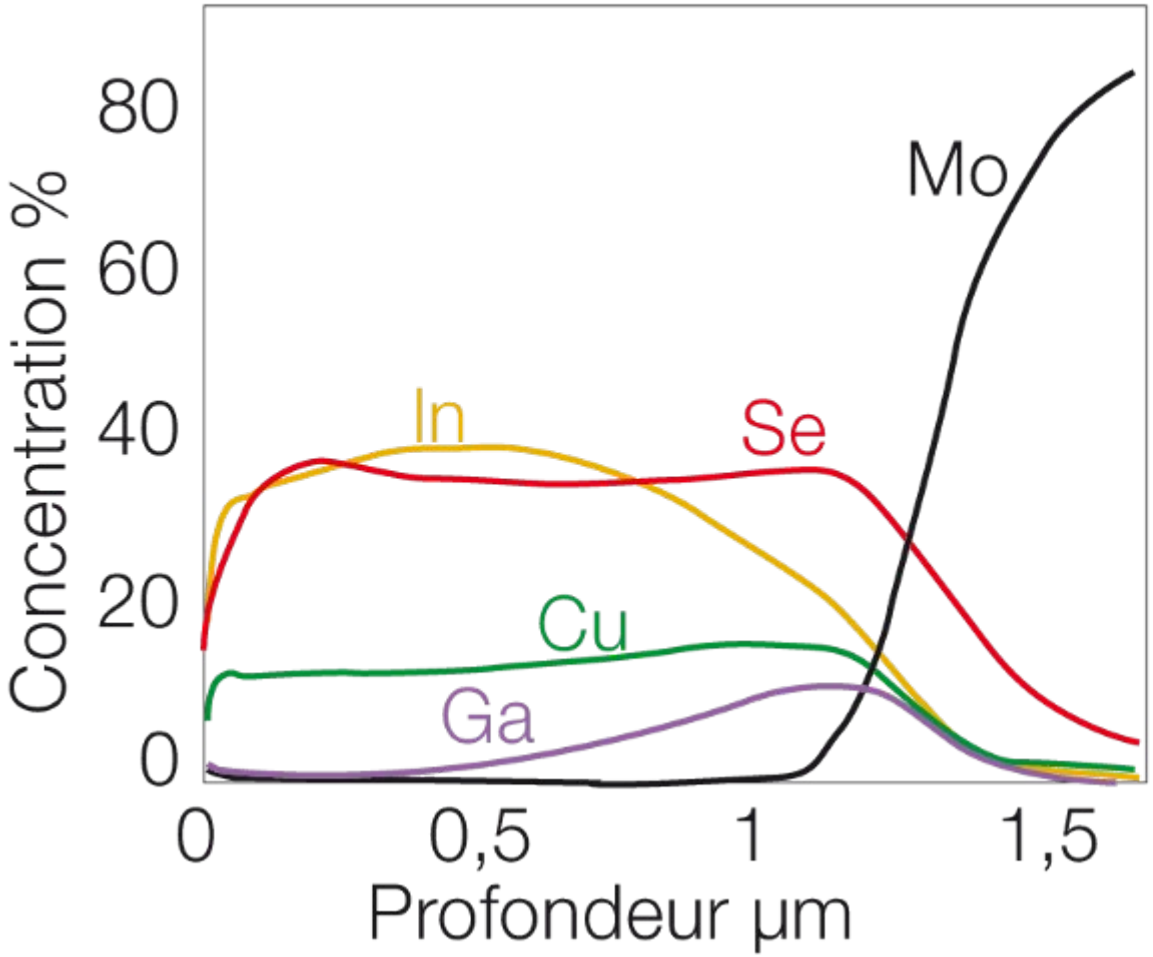
« Li bell »
Transfer chamber to the GD instrument

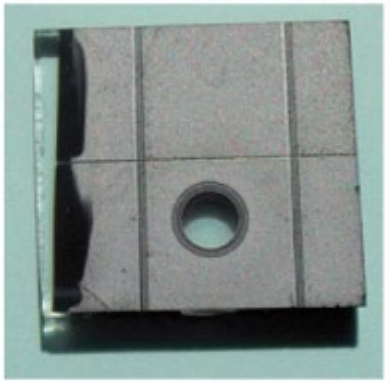


Patent : P 2001-197308

Photovoltaics, Solar Cells

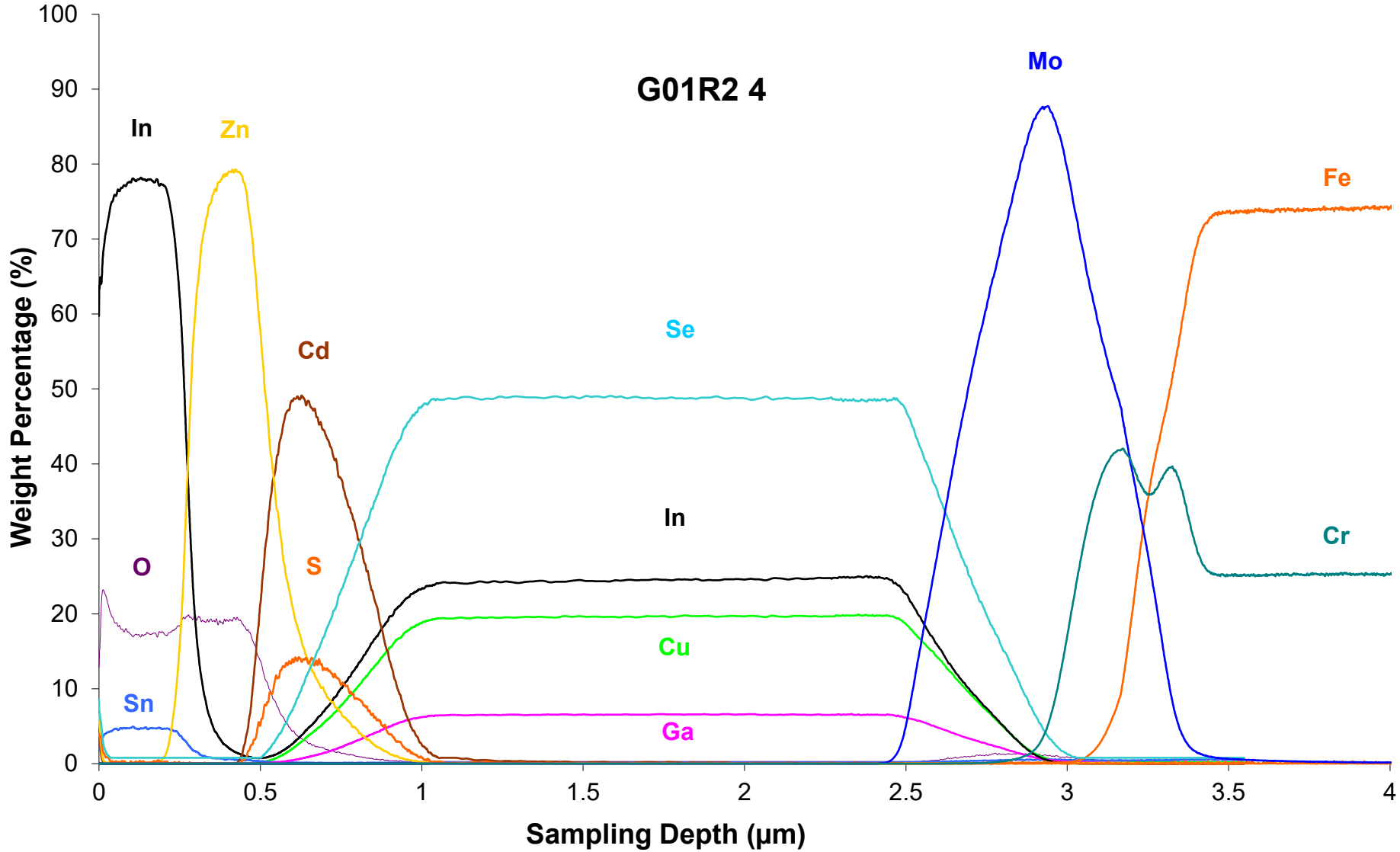
CIGS: Quantified Depth Profile with pulsed Rf GD-OES



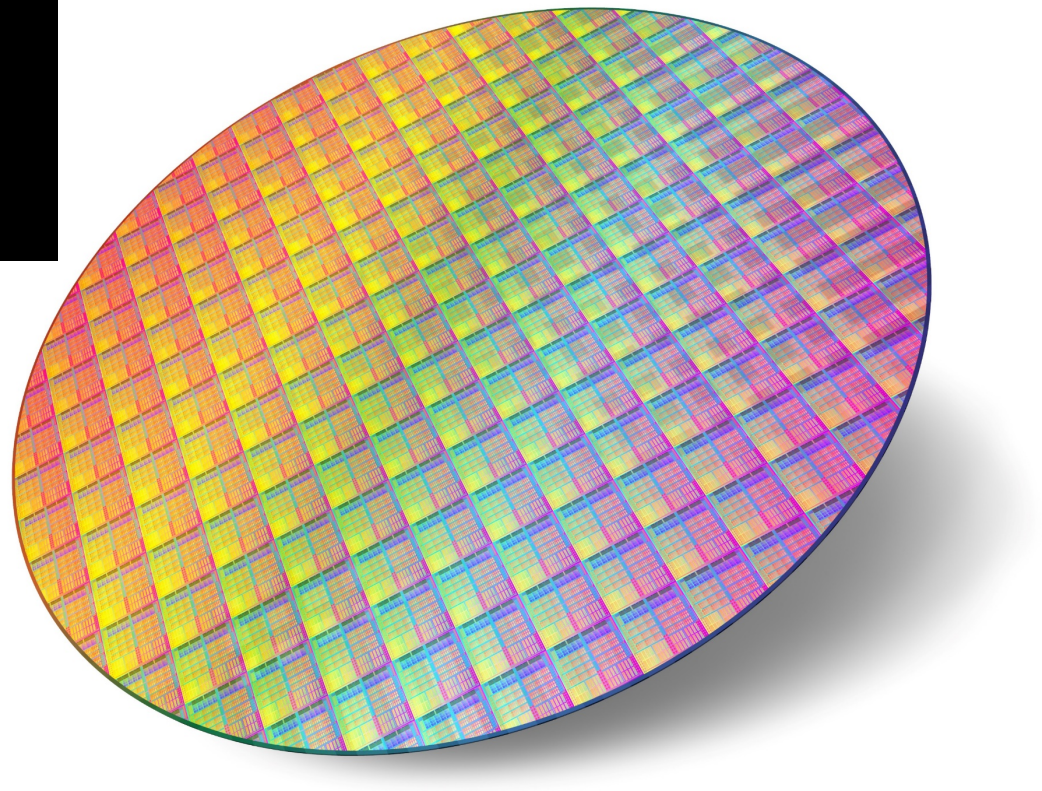


Analysis time: 2 minutes

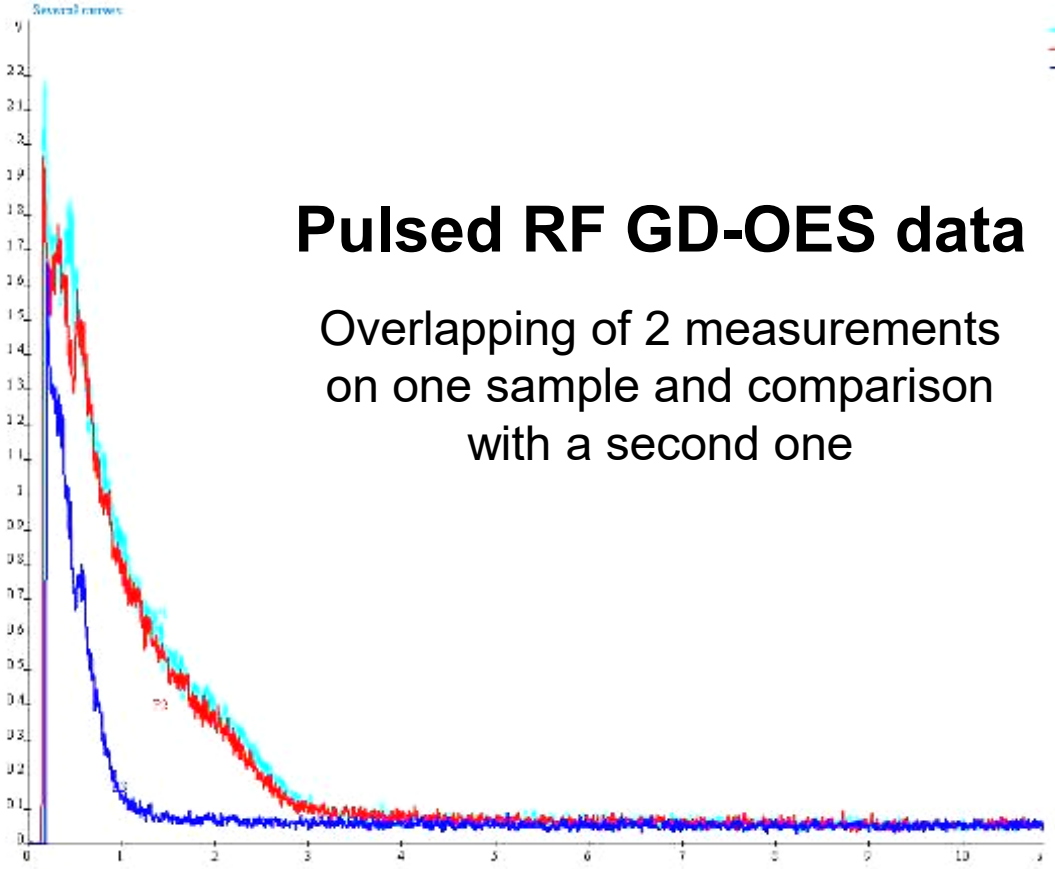
CIGS Application



Semiconductors, Wafers, LEDs



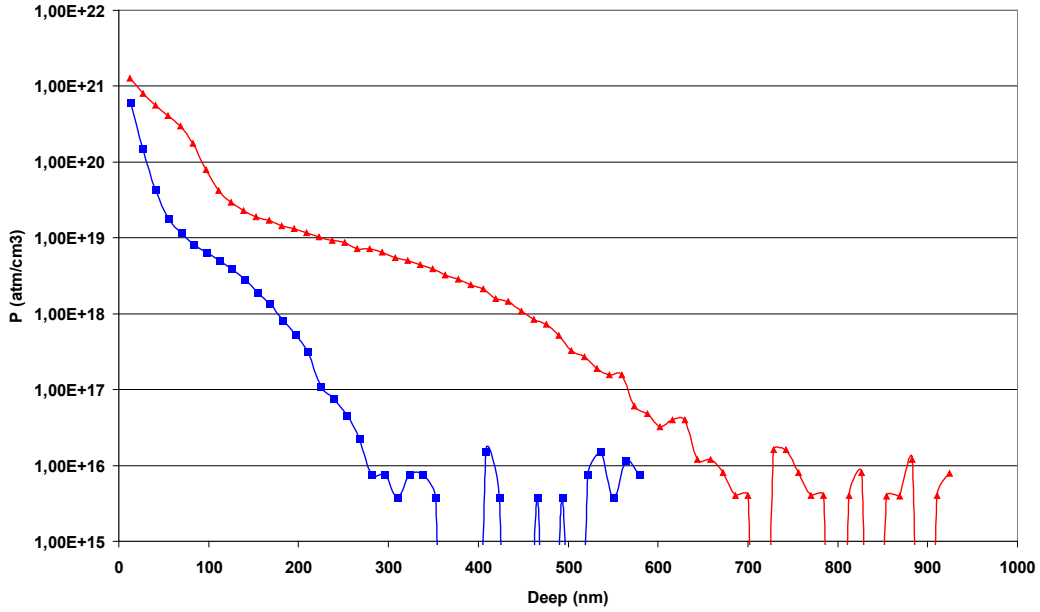
P Doping in Si



Pulsed RF GD-OES data

Overlapping of 2 measurements on one sample and comparison with a second one

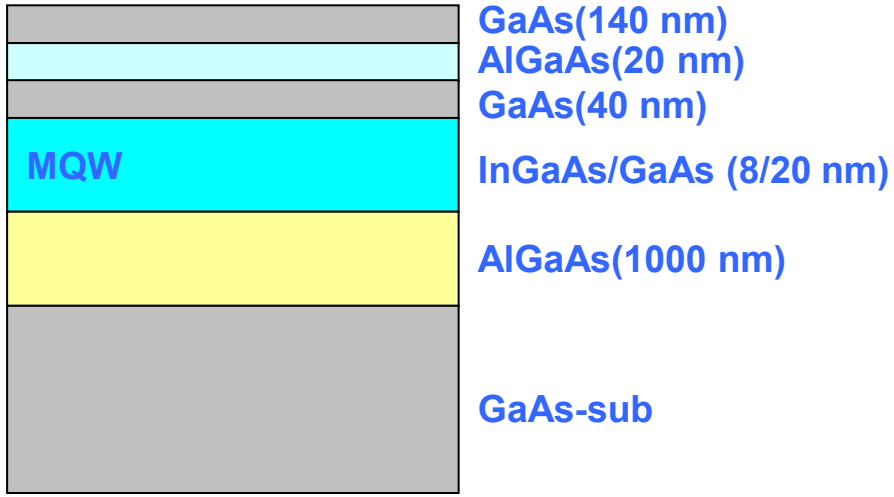
SIMS Comparison

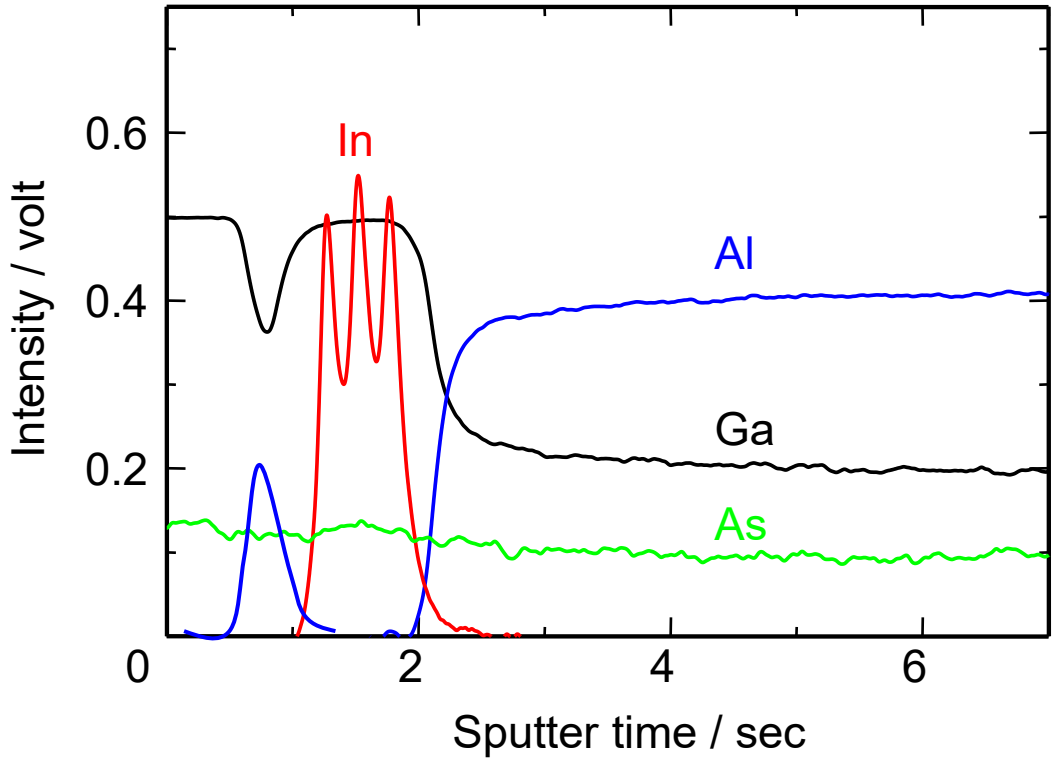


5 seconds vs 2 hours !

Blue LED Structure

Sample Structure

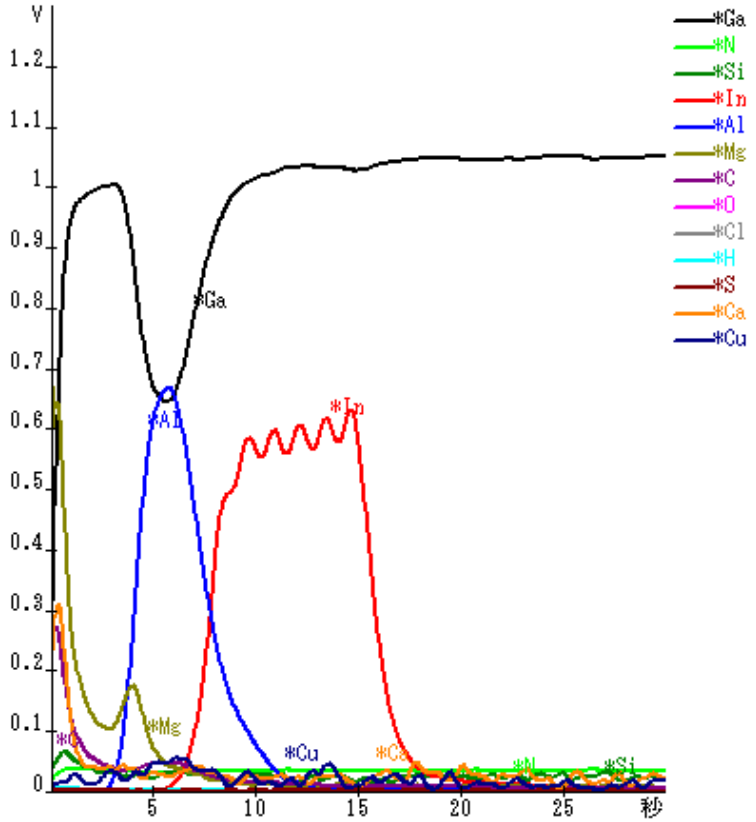




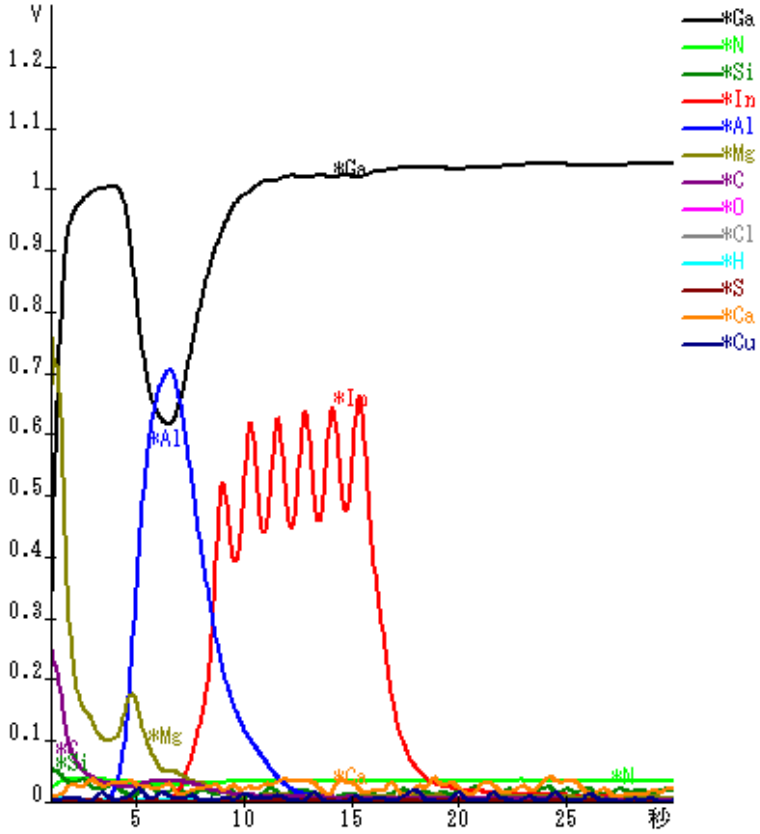
Rf GD-OES provides a result in only 10 seconds.

Blue LED

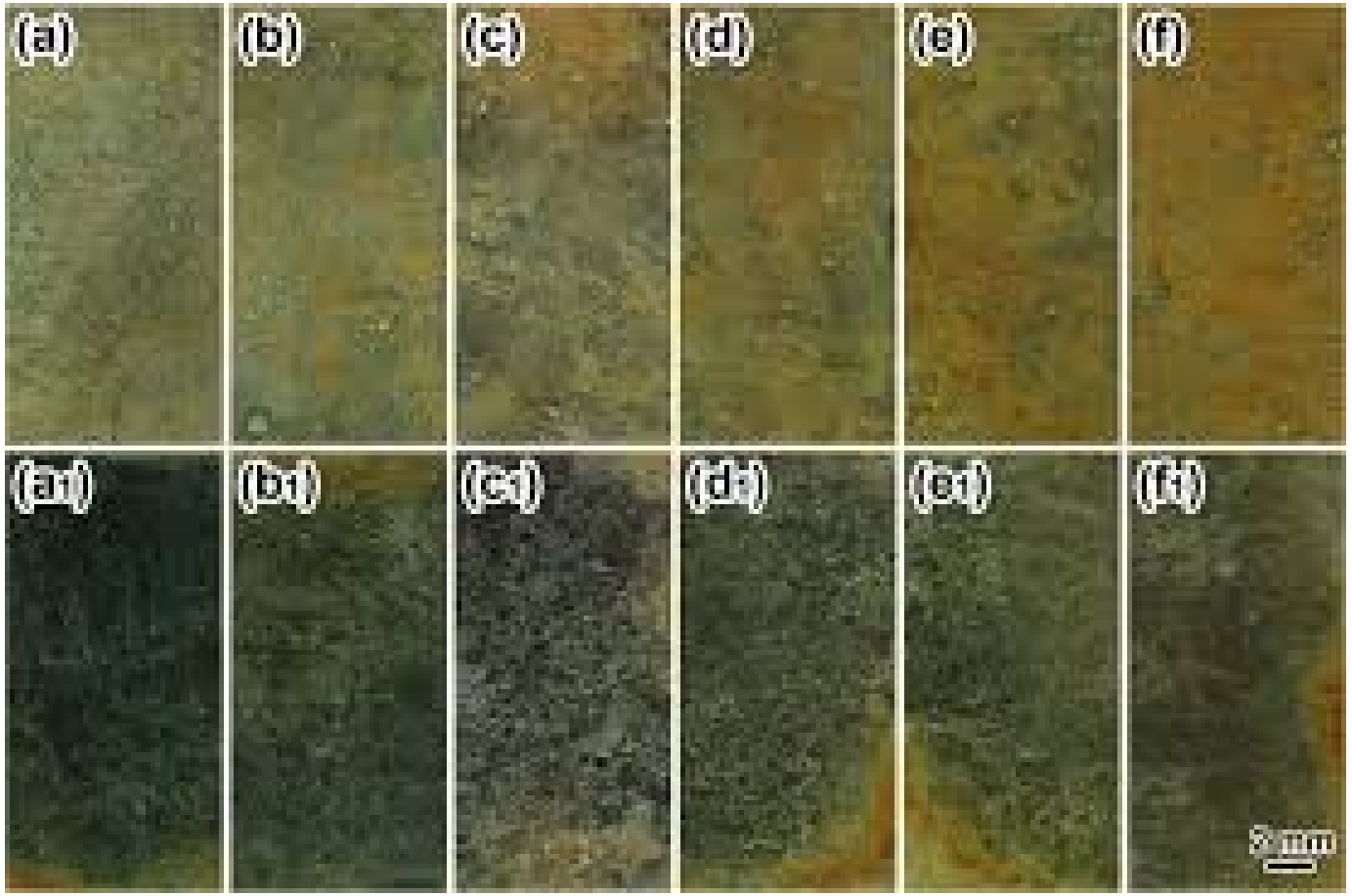
Bad LED



Good LED

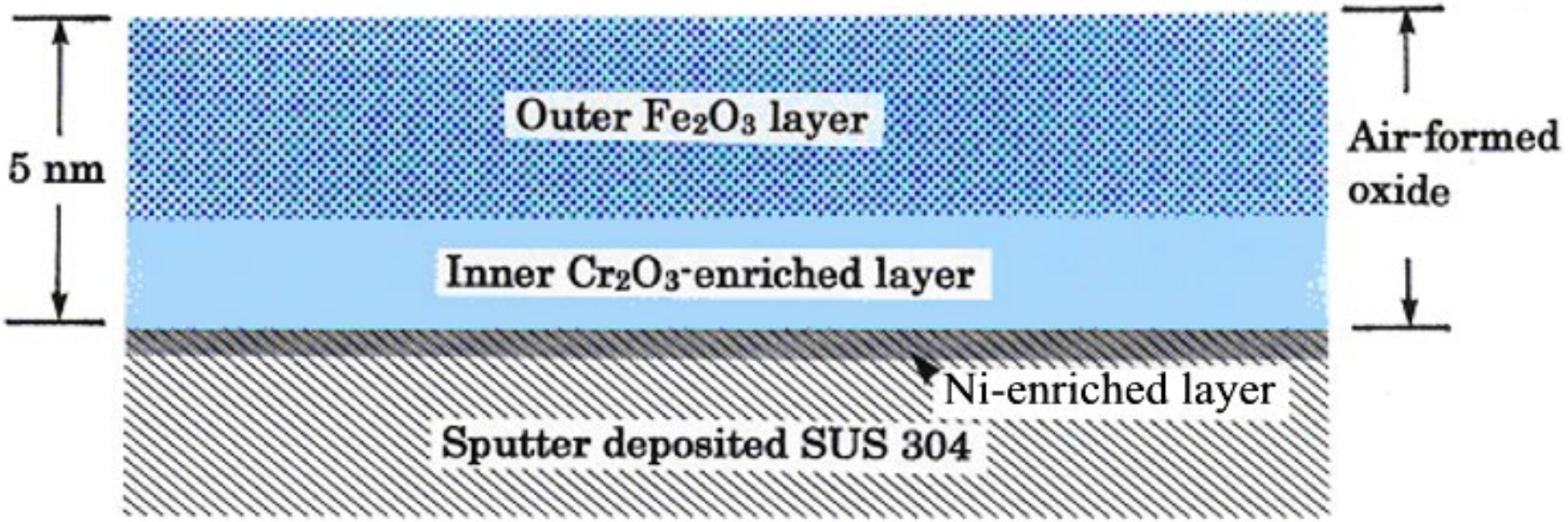


Thin and Thick Oxides:
From nm to several microns

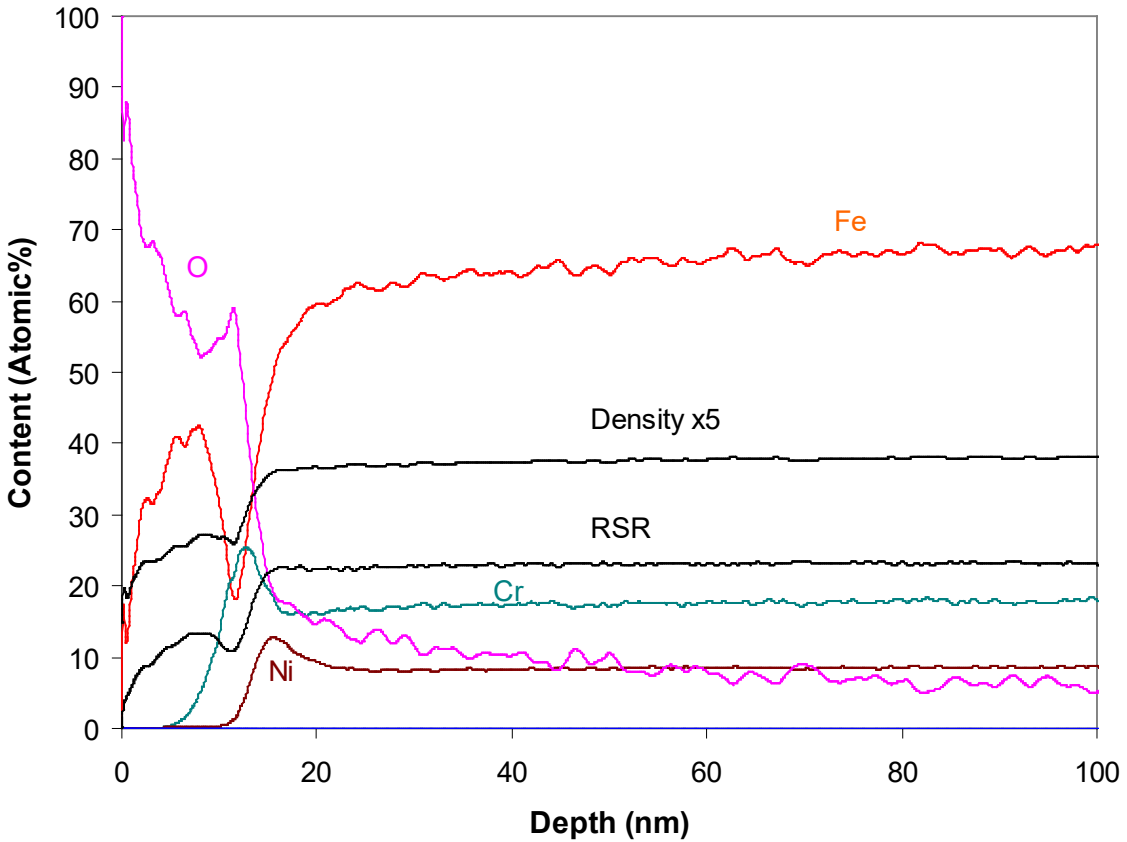


Thin Oxide Layers

(Stainless Steel)

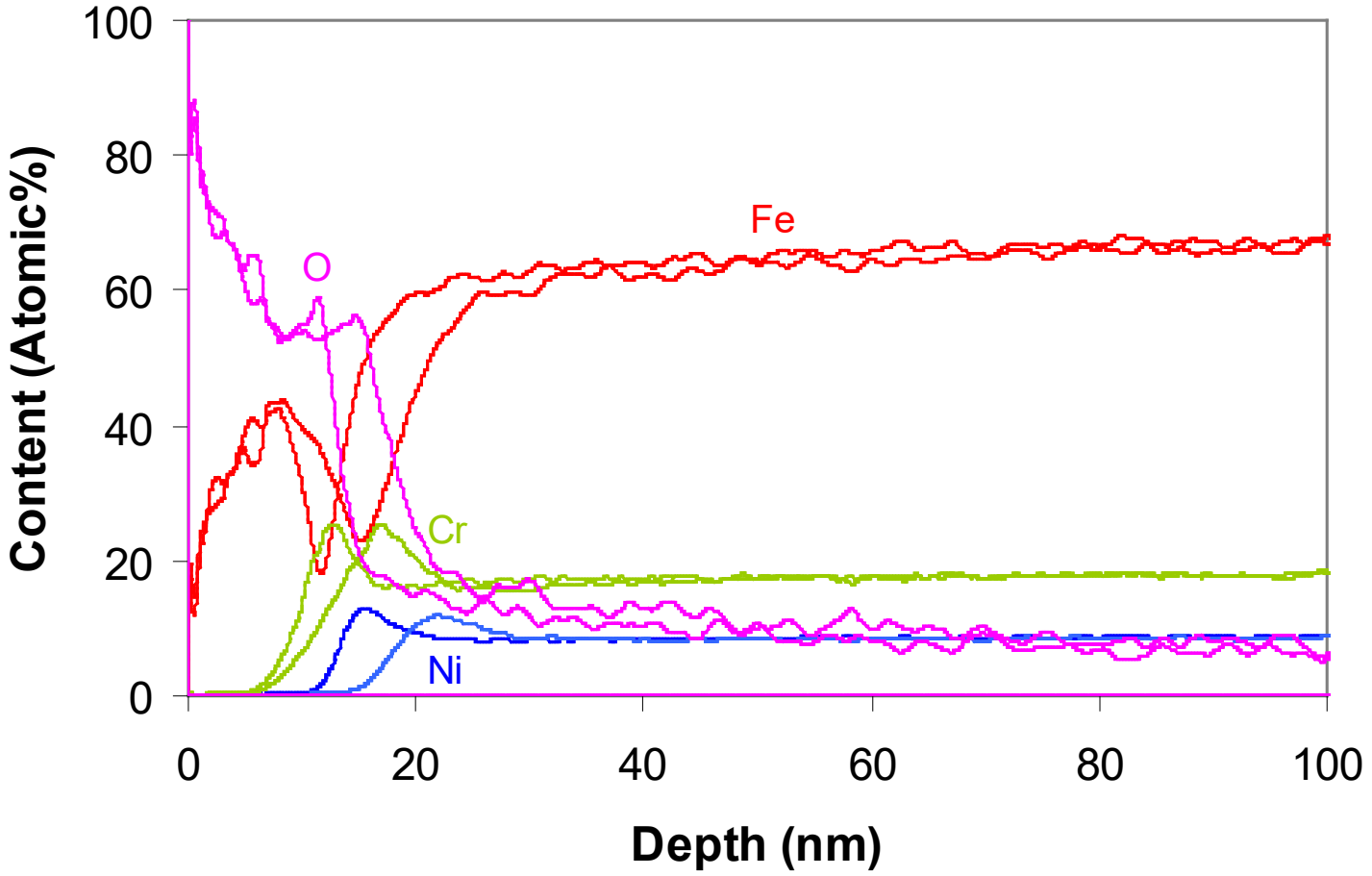


Stainless Steel, polished: Quantified



400 °C

Stainless Steel, polished: Quantified

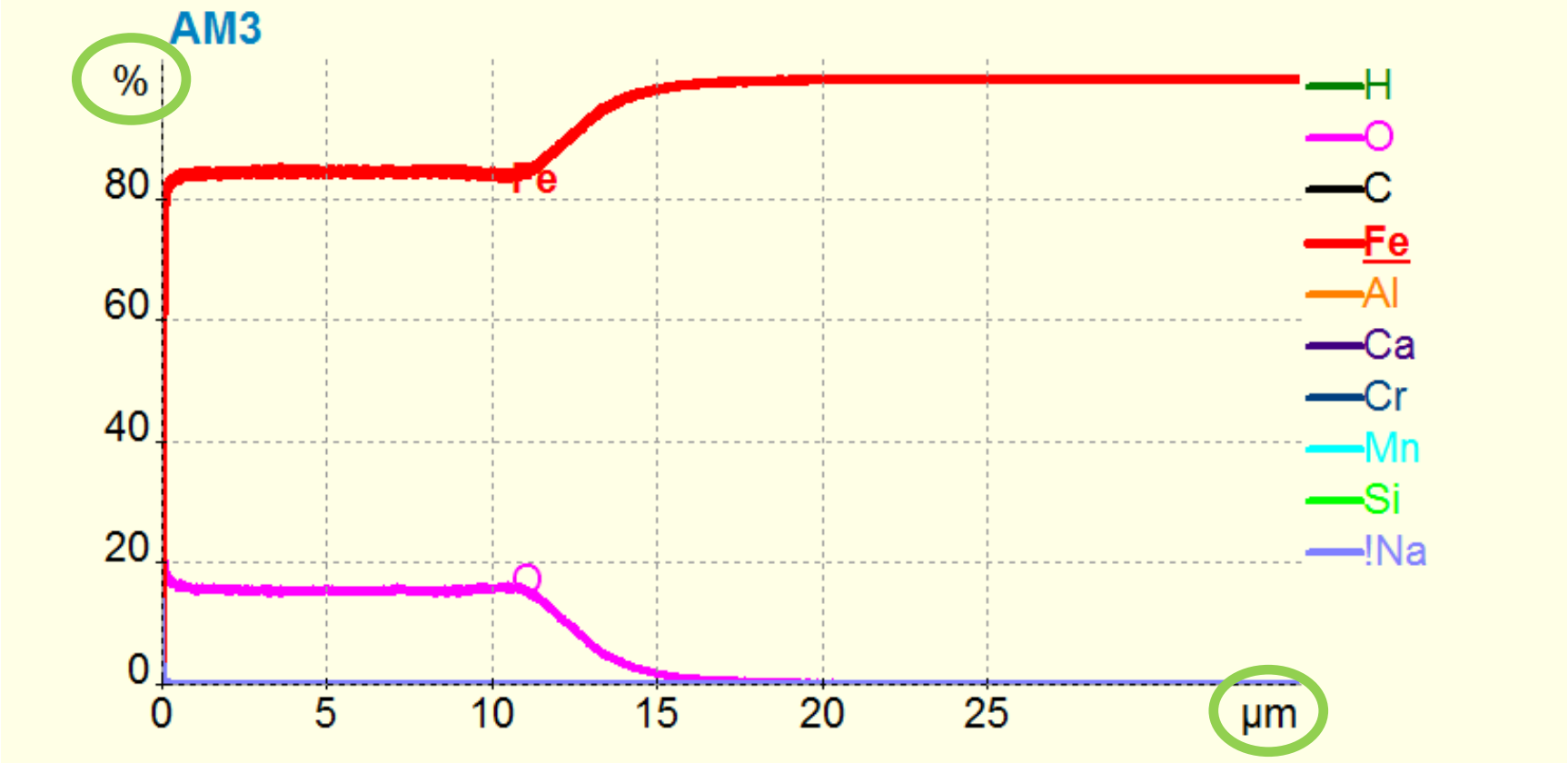


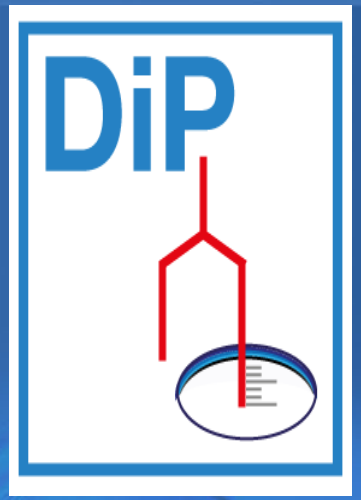
400 °C

&

500 °C

Thick Iron Oxide





Differential Interferometry Profiling

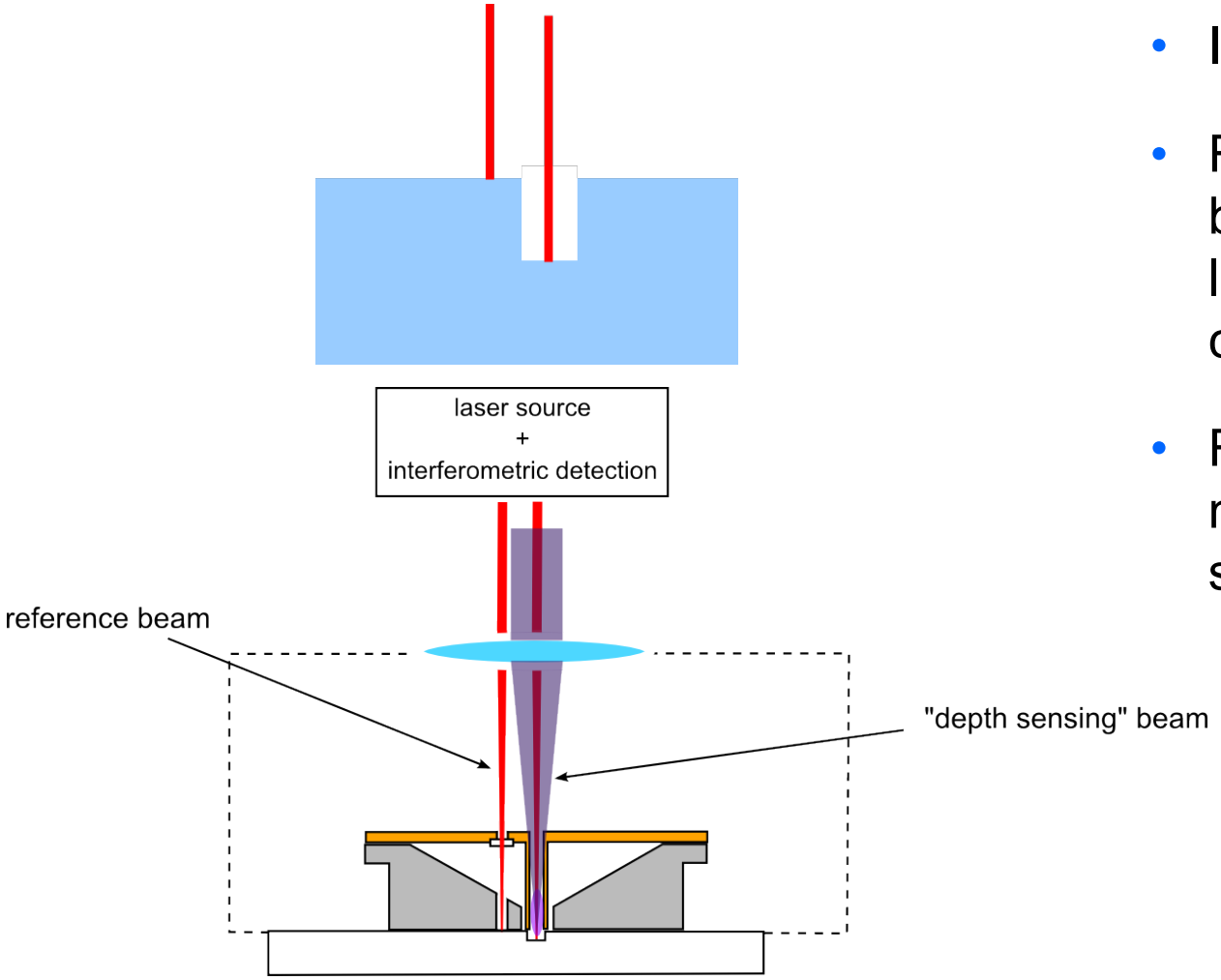
A new function in GDOES:

Direct layer thickness and crater depth measurement

What is DIP?

- Direct sputter rate, layer thickness and crater depth measurement
- Real Time
- No calibration
- Online interferometer built in the GD instrument
- An additional tool for GD-OES calibration

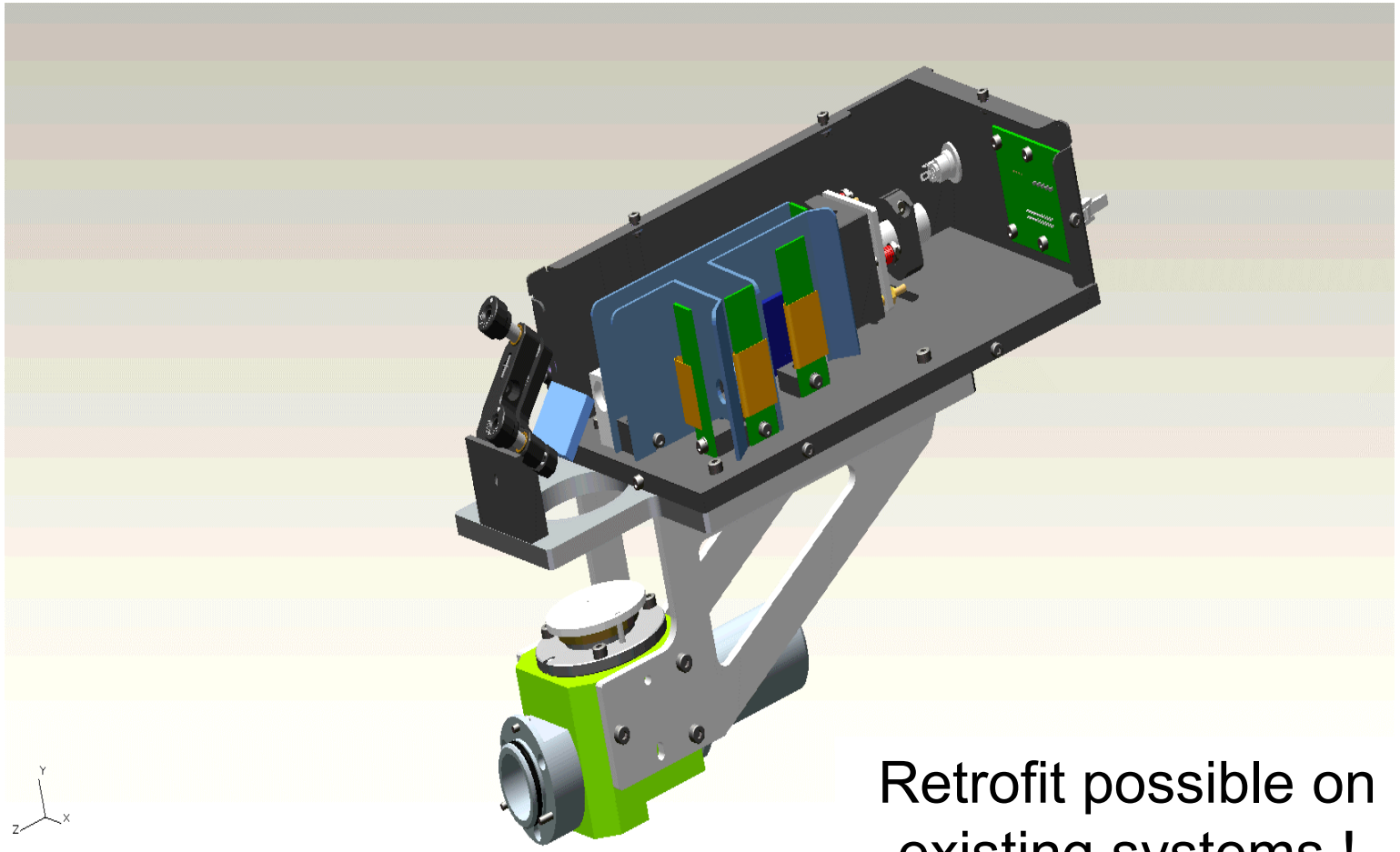
Technical Solution



- Interferometric method
- Relative depth measurement between the crater and a reference location on the sample nearby the crater.
- Red diode laser (~635nm +/- 5nm), no emission line of interest in this spectral region)

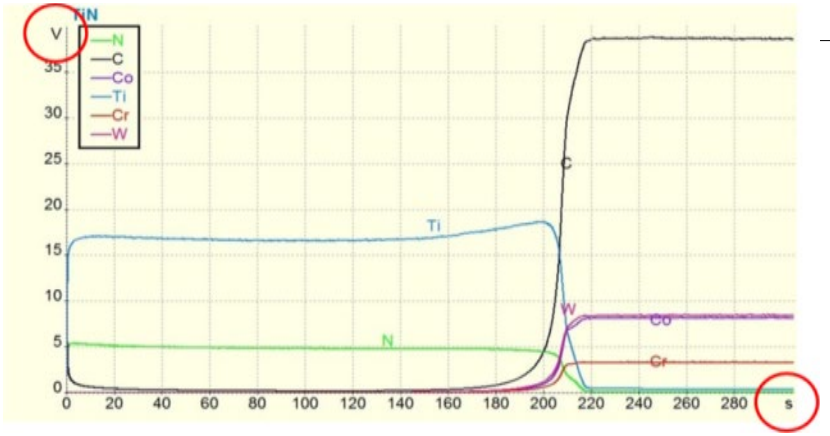
Modified Anode / Cathode

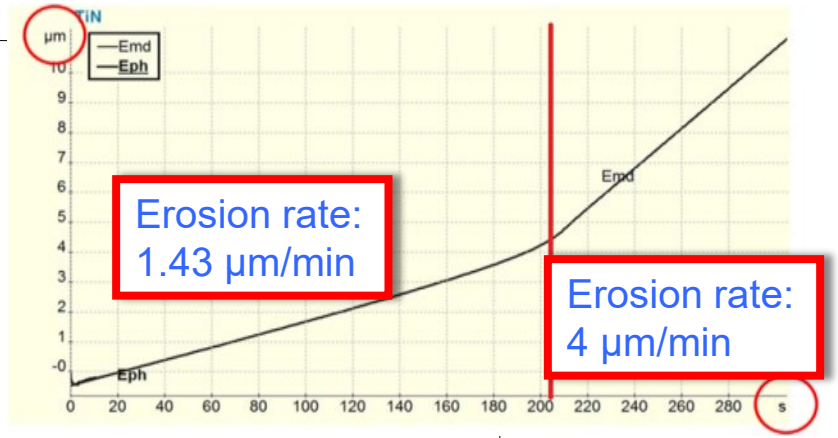
Smart and Compact Design



Retrofit possible on existing systems !

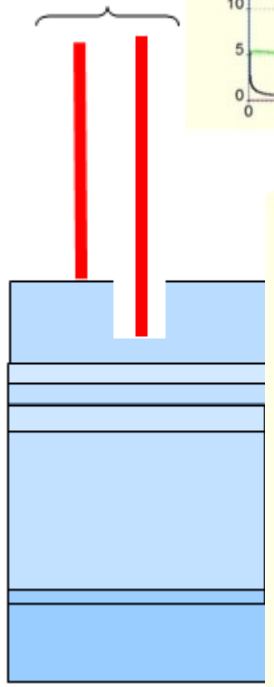
DiP Application: TiN Coating on WC

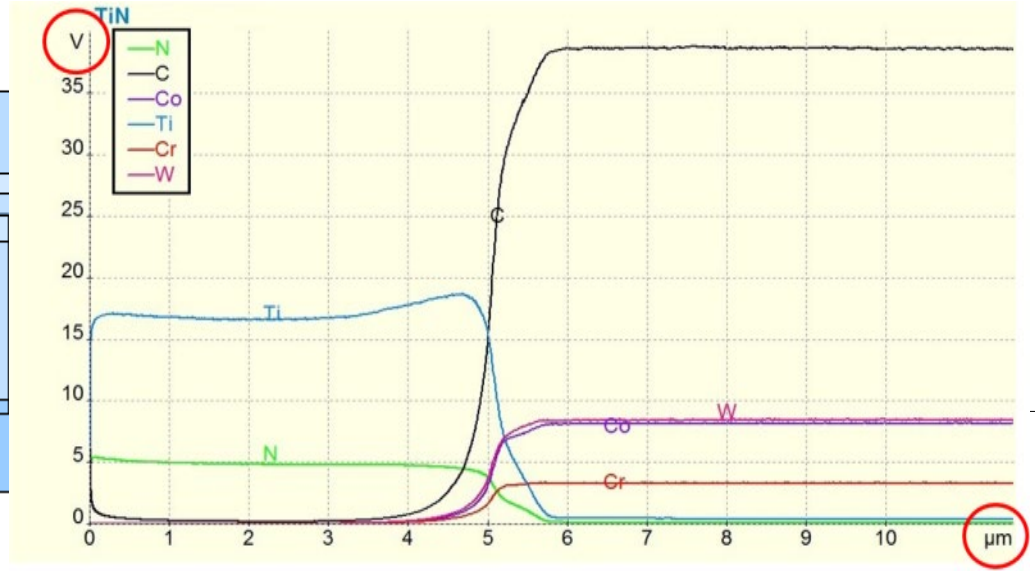




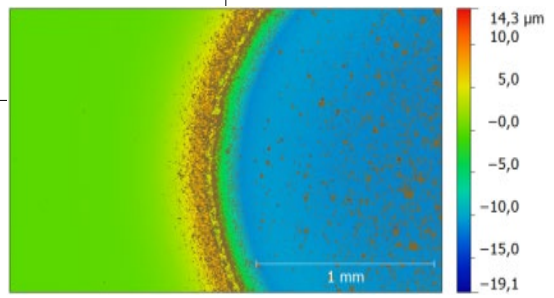
Erosion rate:
1.43 µm/min

Erosion rate:
4 µm/min





Total depth:
- 11.6 µm by DIP
- 11.45 µm by profilometer



Conclusion

- Pulsed Rf GD-OES for conductive and non-conductive materials
- Unique patented features are essential to obtain valuable depth profile on a large variety of materials (Pulsed Rf, UFS, Li Bell…)
- Handling strategies and operating procedures have been developed for numerous applications
- Layers from nm to 150 microns and more…
- From ppm to 100%
- Complementary to SIMS, XPS…
- Affordable, robust, and fast

Thank you

JOBIN YVON Technology

Coming Up...

COVALENT METROLOGY

ADVENTURES IN WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE (WDXRF): FLEXIBLE ELEMENT ANALYSIS FOR THIN FILMS PIONEERS

SPEAKER:

Meredith Beebe
Semiconductor X-ray Metrology Specialist, Rigaku

January 27, 2022 | 11am PT

Rigaku

COVALENT METROLOGY

LASER ABLATION INDUCTIVELY COUPLED PLASMA MASS SPECTROSCOPY: NOT JUST ROCKS

SPEAKER:

Lucas Smith
Director of Business Development for the Americas, Teledyne CETAC

February 10, 2022 | 11am PT

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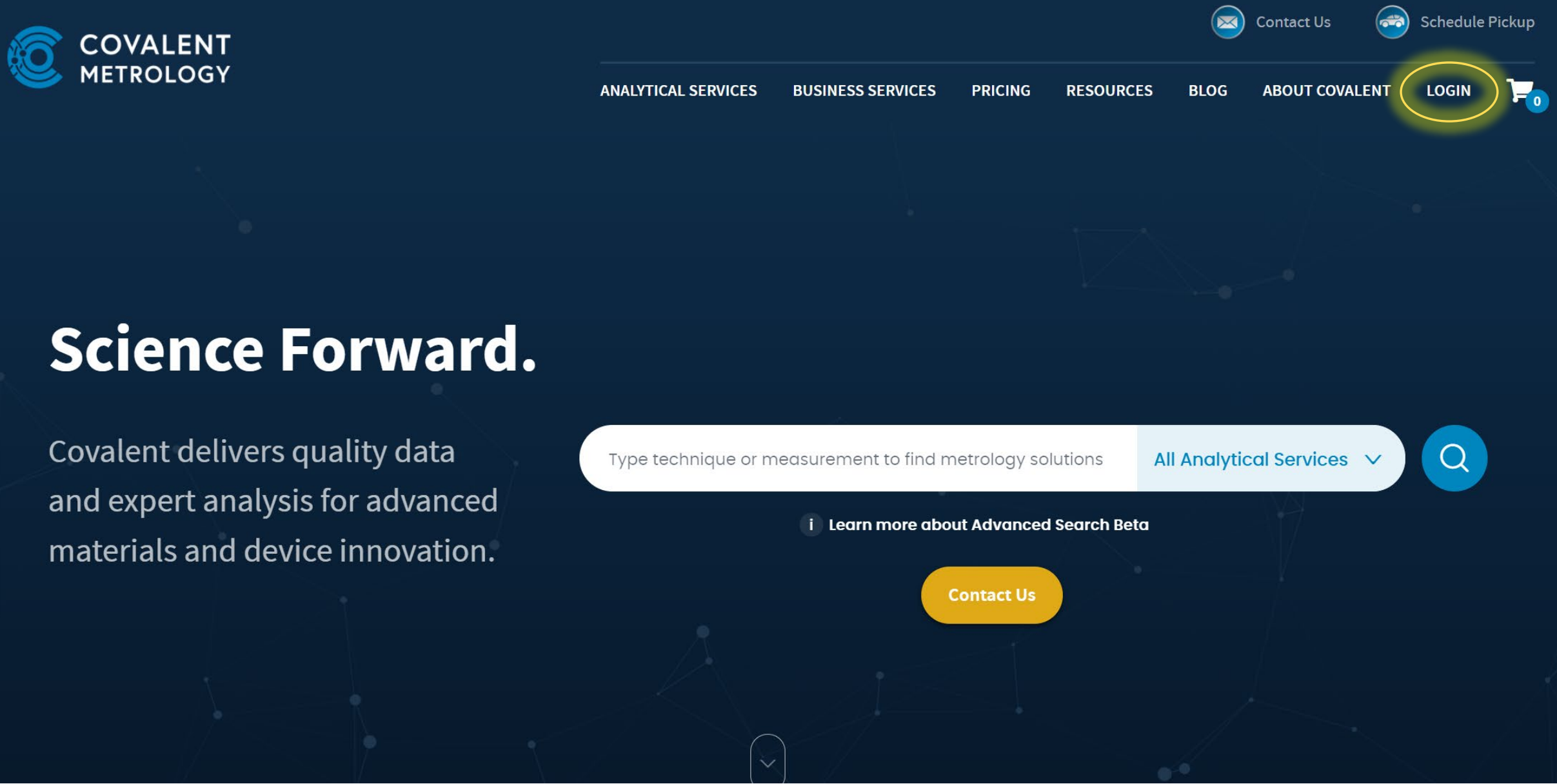

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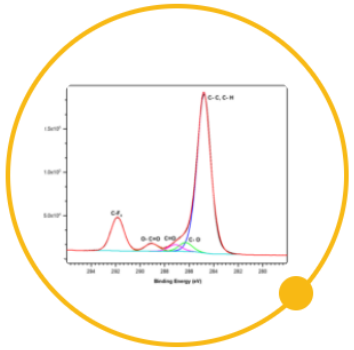

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Q & A Session

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Thank you