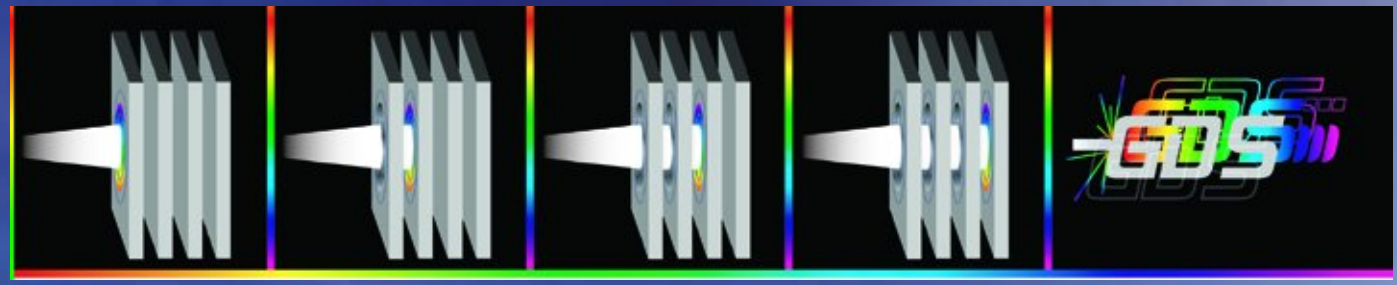
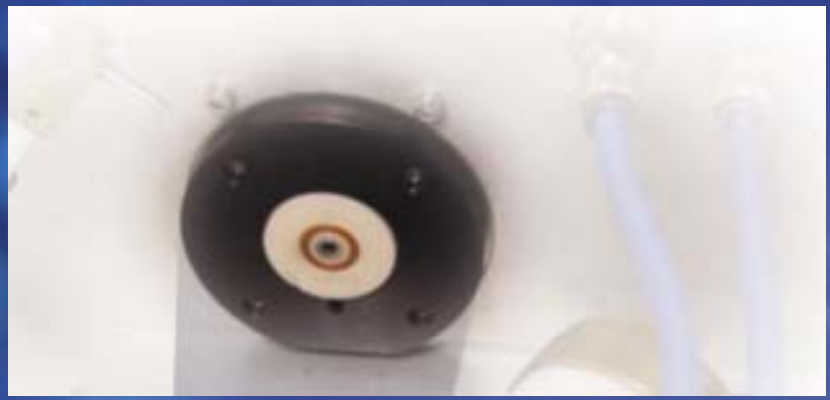
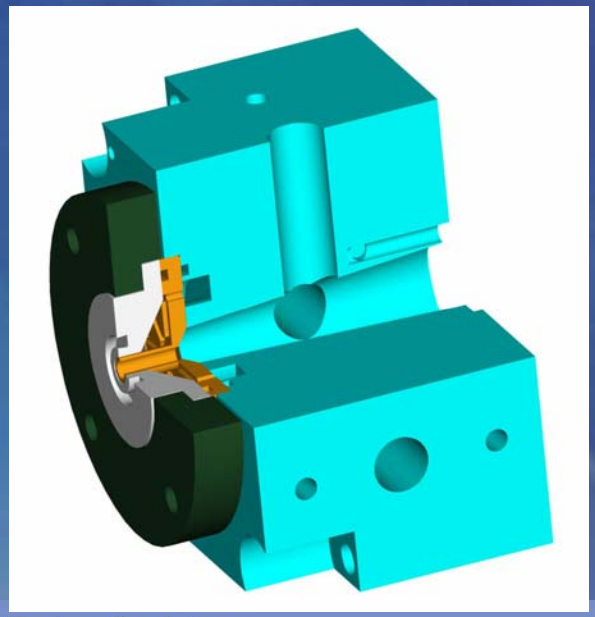


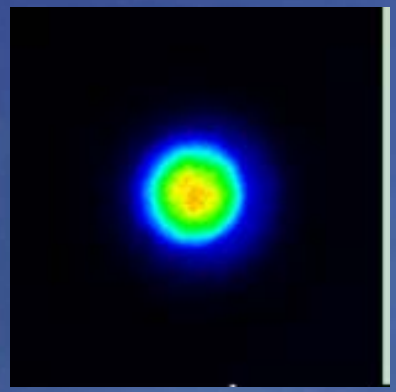
New developments in RF–GD-OES extend the range of applications

Christophe Deraed and Patrick Chapon
Jobin Yvon

Glow Discharge Source







External mounting of the sample

Primary vacuum (double pumping)

Fast sputtering rate (1-5 µm/mn)

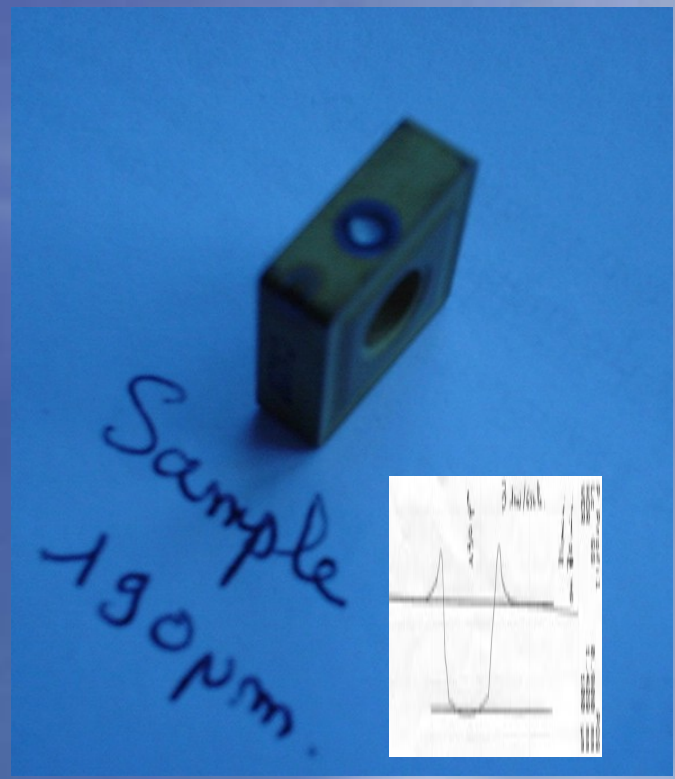
Measure all elements (including H, O, N, Cl, C and F)

Conductive and non conductive layer

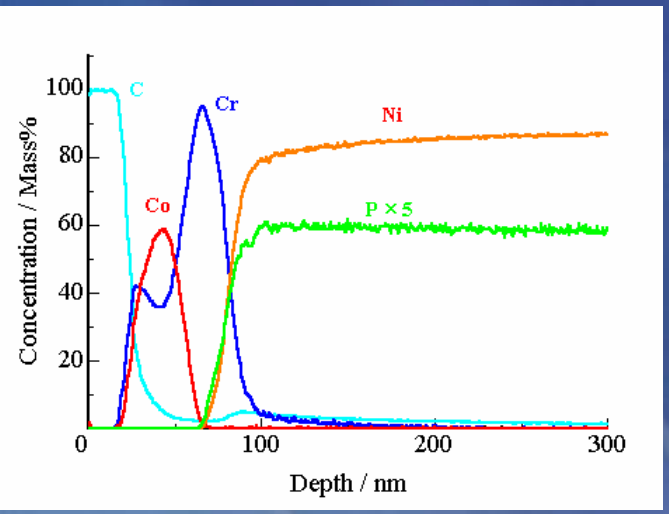
Ease of use

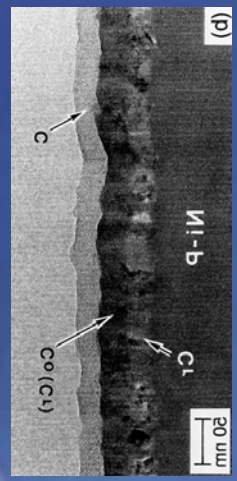
Range of applications

Cutting tool. 2mm Anode, deep crater



Surface layers of a hard disk (X scale in nm)





Practical benefits of recent advances in theory and practice

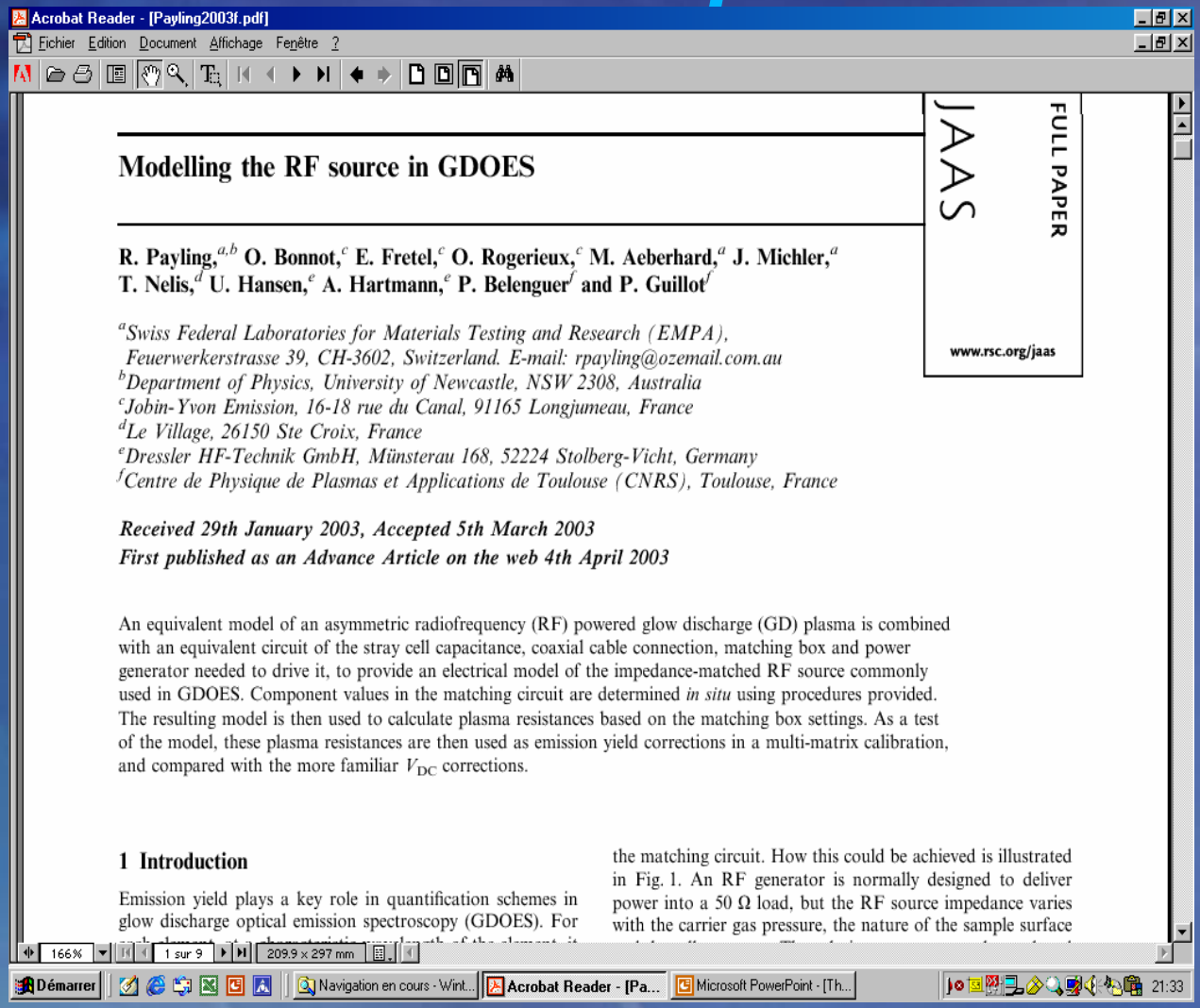
- Understanding the GD RF plasma.
- >> Running efficiently large samples.

- Development of a pulse RF source.
- >>Applications to fragile materials.

- Emphasis on accessories
- >> Application to small and/or odd samples (tubes etc)

- New ideas and approches in Quantification.
- >> Improvements of CDP for ultra thin layers

1) Understanding the GD RF plasma



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JAAS FULL PAPER

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Modelling the RF source in GDOES

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An equivalent model of an asymmetric radiofrequency (RF) powered glow discharge (GD) plasma is combined with an equivalent circuit of the stray cell capacitance, coaxial cable connection, matching box and power generator needed to drive it, to provide an electrical model of the impedance-matched RF source commonly used in GDOES. Component values in the matching circuit are determined *in situ* using procedures provided. The resulting model is then used to calculate plasma resistances based on the matching box settings. As a test of the model, these plasma resistances are then used as emission yield corrections in a multi-matrix calibration, and compared with the more familiar V_{DC} corrections.

1 Introduction

Emission yield plays a key role in quantification schemes in glow discharge optical emission spectroscopy (GDOES). For

the matching circuit. How this could be achieved is illustrated in Fig. 1. An RF generator is normally designed to deliver power into a 50 Ω load, but the RF source impedance varies with the carrier gas pressure, the nature of the sample surface

166% 1 sur 9 209.9 x 297 mm

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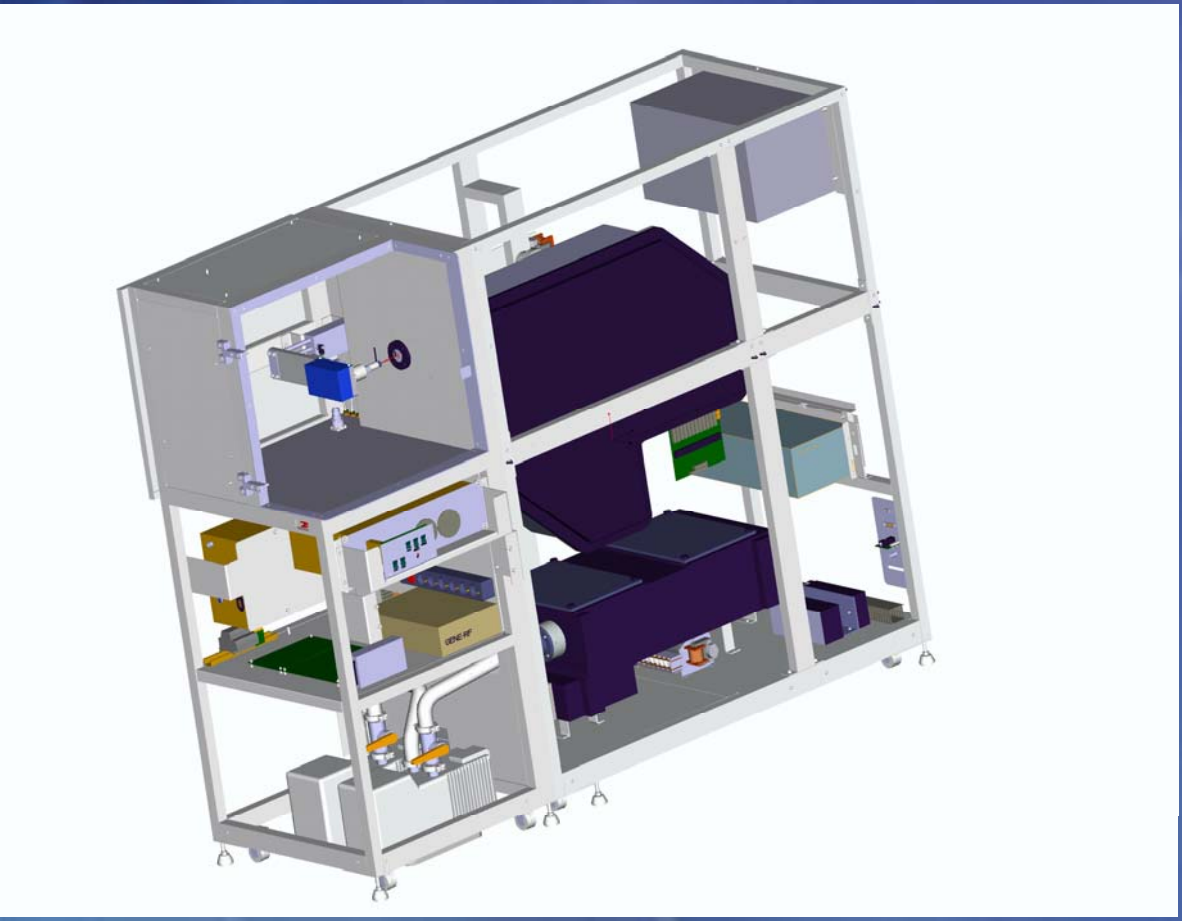
- Extensive work done in cooperation with other researchers
- Recent paper published (JAAS 2003 – special edition on GD)

Achievements and practical benefits of the work

- The RF circuitry is fully described
- Changes in impedance are understood and can be either monitored and/or minimized
- This approach is validated by the characterisation of the RF plasma
- Benefits are the improvement of the lamp design and of the quantification.

GD-Profiler design



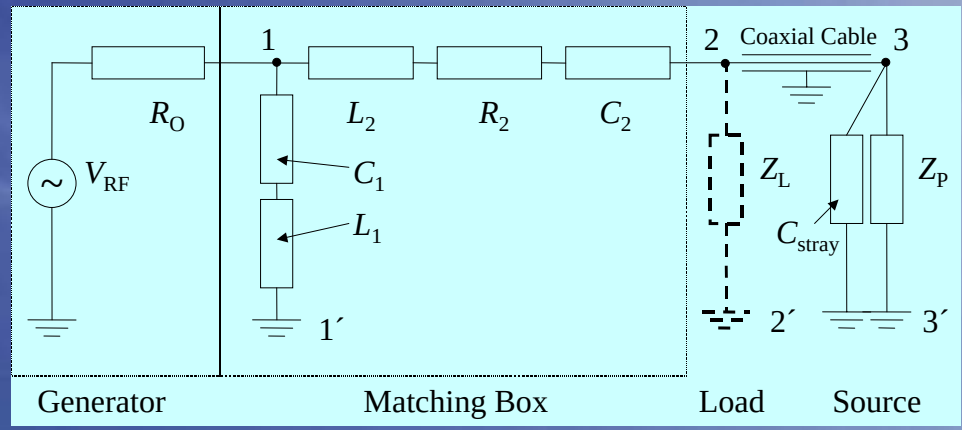
- Large sample compartment
- Centrelite for precise positionning

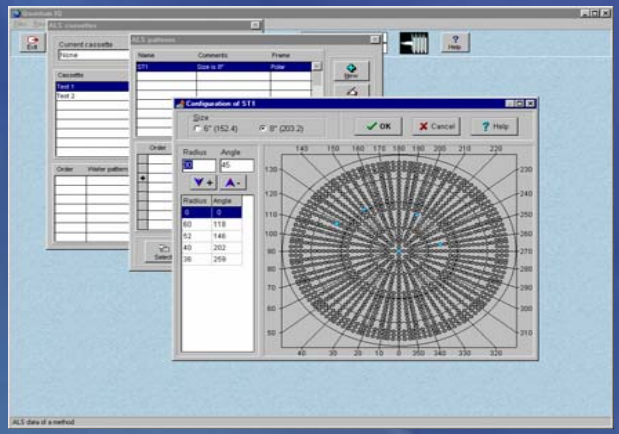
Samples over 50cm in diameter

- Large wafers

Large samples : handling and analysis

Modelisation of the lamp





Software screen for the analysis of wafers

JY Horiba patent for large wafers analysis

No signal variation due to sample positionning

B deposited on wafers

Document1 - Microsoft Word

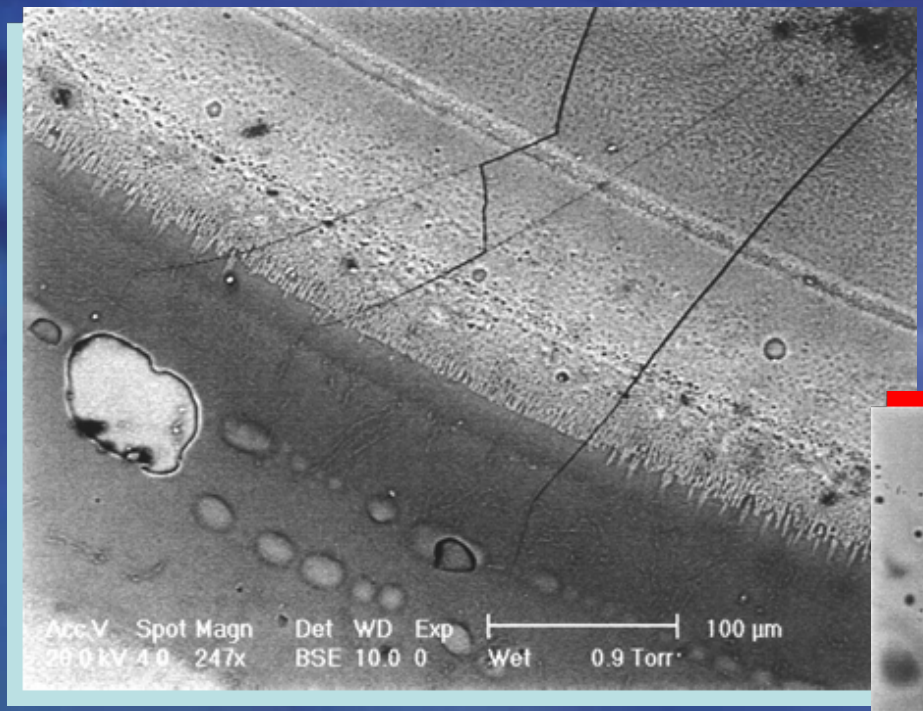
Table 1 rf-GDOES results for B in BSG

Slot	TMB	GDOES Center			Edge			Average		Ratio Center/ Edge
		Mean	SD	RSD	Mean	SD	RSD	Mean	SD	
1	85	12.67	0.15	1.17	11.85	0.24	2.05	12.26	0.20	1.07
2	85	12.13	0.09	0.75	11.92	0.11	0.96	12.02	0.10	1.02
3	135	18.27	0.31	1.67	17.40	0.45	2.61	17.83	0.39	1.05
4	135	17.85	0.26	1.46	17.28	0.04	0.26	17.57	0.19	1.03
5	100	15.40	0.07	0.43	15.24	0.30	1.98	15.32	0.22	1.01
6	100	15.27	0.22	1.44	14.27	0.22	1.57	14.77	0.22	1.07
			mean=	1.15			1.57			1.04

2) Development of a pulse RF source

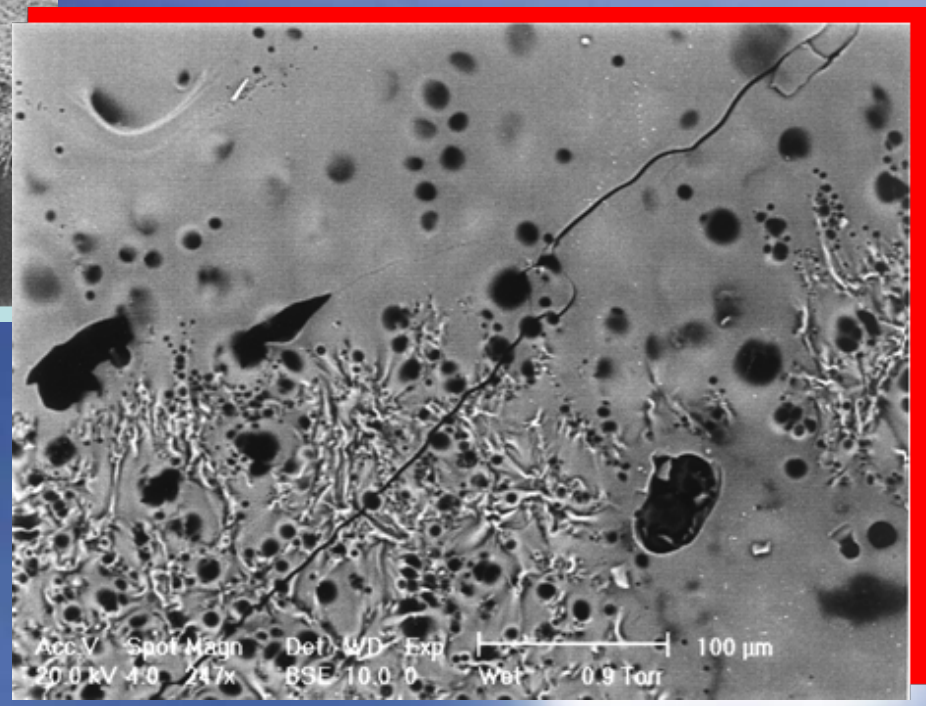
- RF can analyze conductors and non conductors
- Non conductors have a poor thermal dissipation
- Risks of overheating and cracks of some materials when RF is continuously applied
- Pulsed RF extends the range of applications to thermal sensitive materials

Effect of the thermal constraint on thin glasses



Outer

Inner

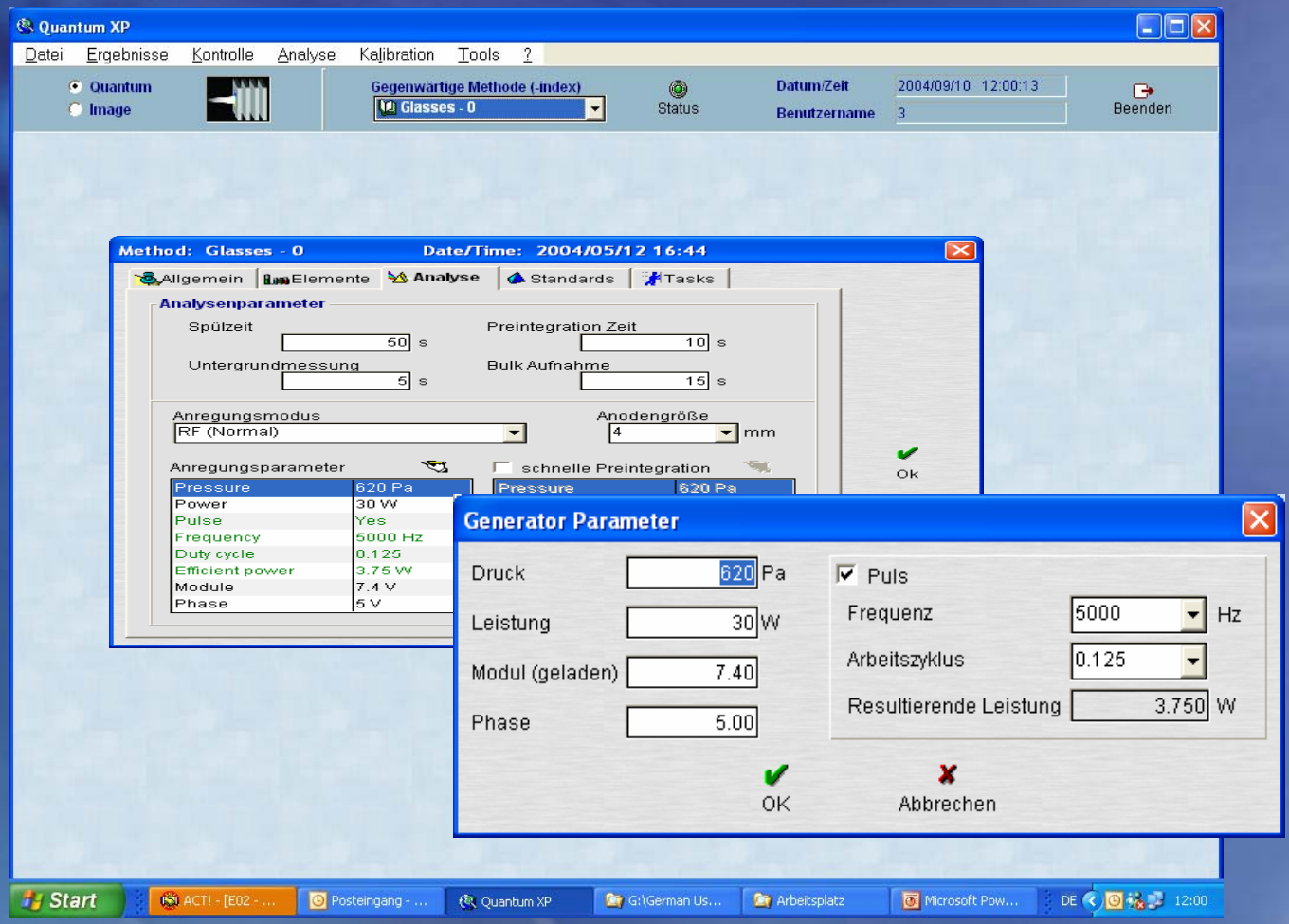


JY new RF source

- Can operate in normal mode and in pulsed mode.
- All modes are computer controlled
- Simple method parameters to select

- Benefits : extended ranges of applications

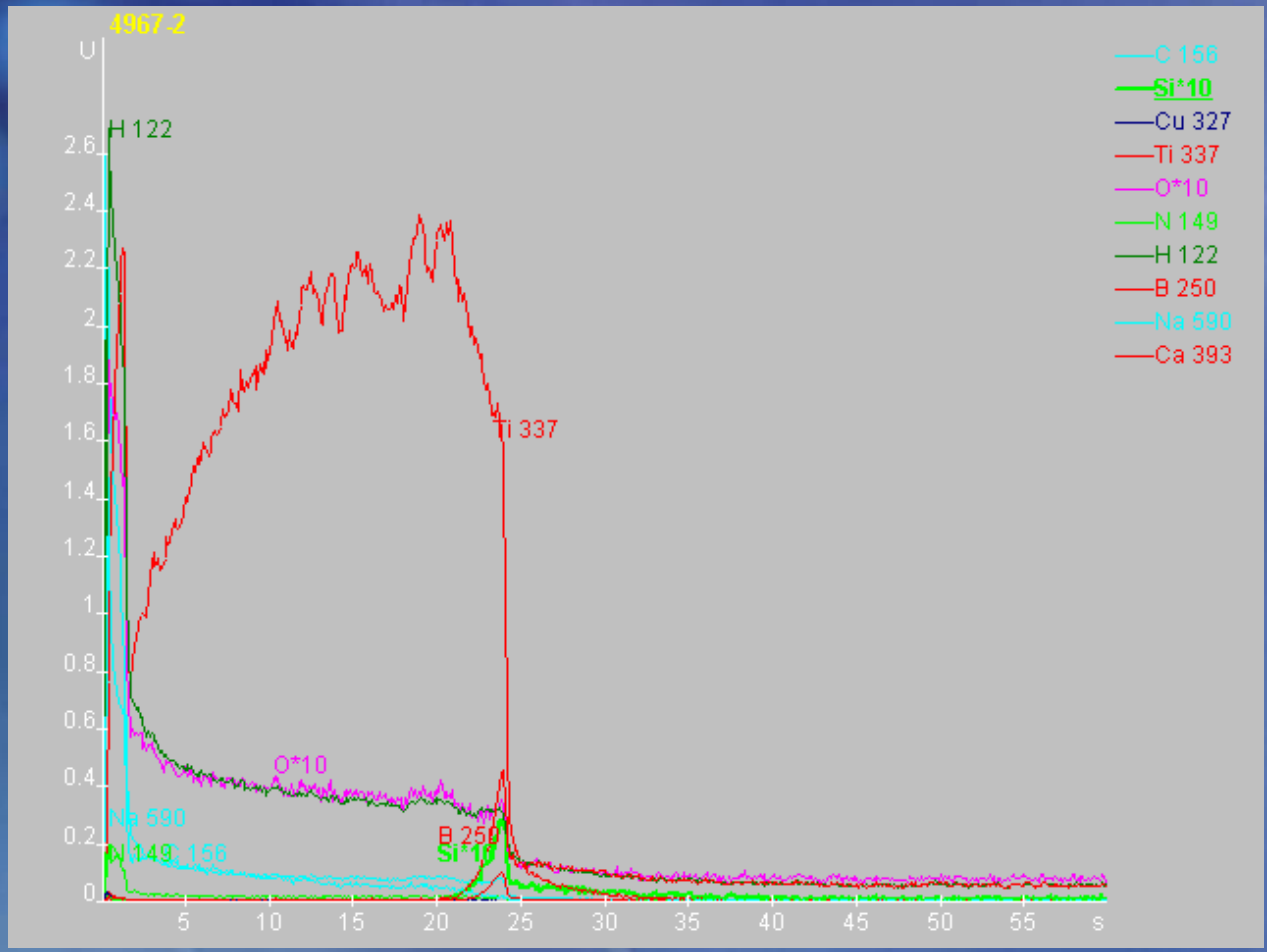
Operation in pulse mode : software



Max power 300W in pulse mode

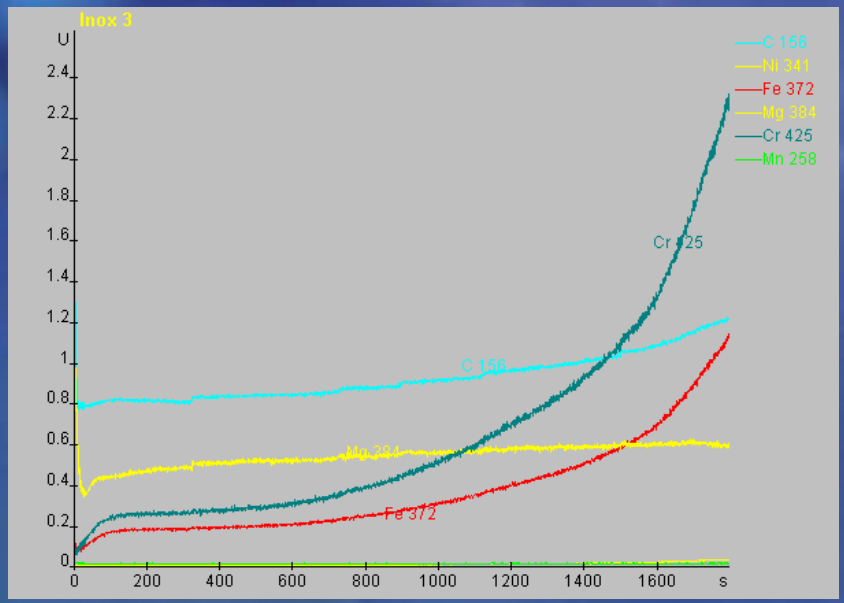
Pulse frequency and duty cycle are computer controlled

Pulsing: Coated glass

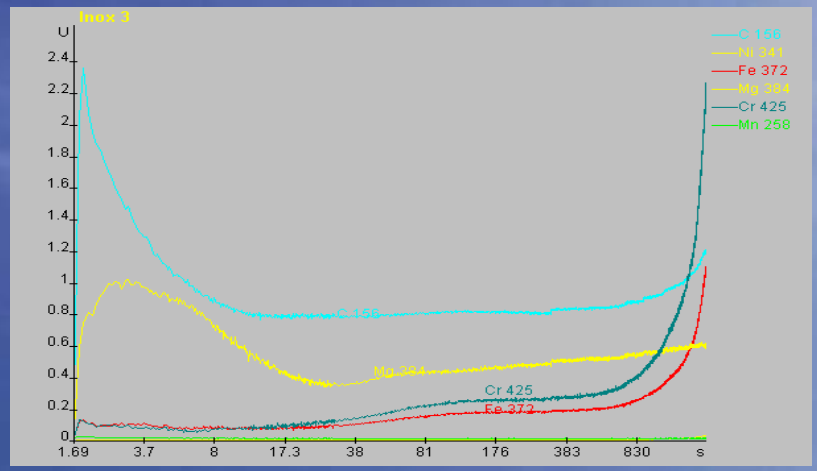


This example shows the result done on a spectacle glass lens, slightly curved with a Ti2O3 based, anti-reflection coating. The sample immediately melts when using non pulsed GD.

Sample : rubber on stainless steel. Normal RF



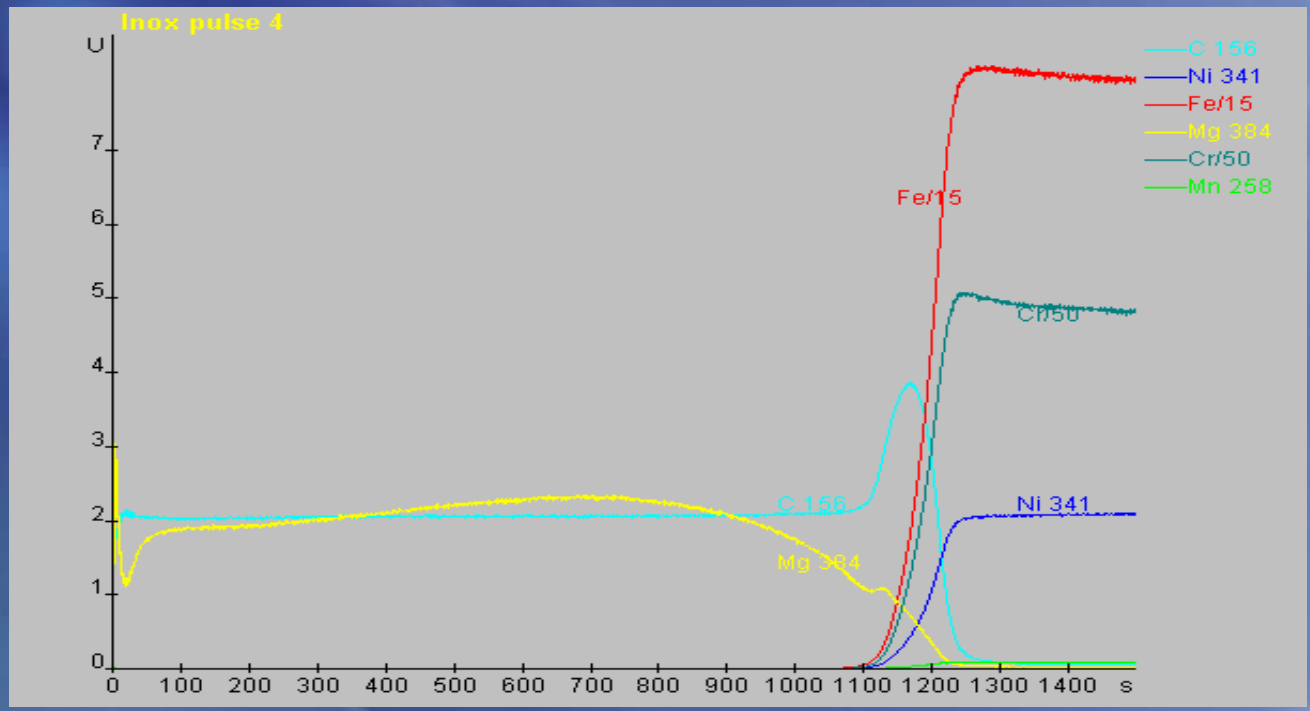
Intensities (a.u.) versus time



(Same with X log scale)

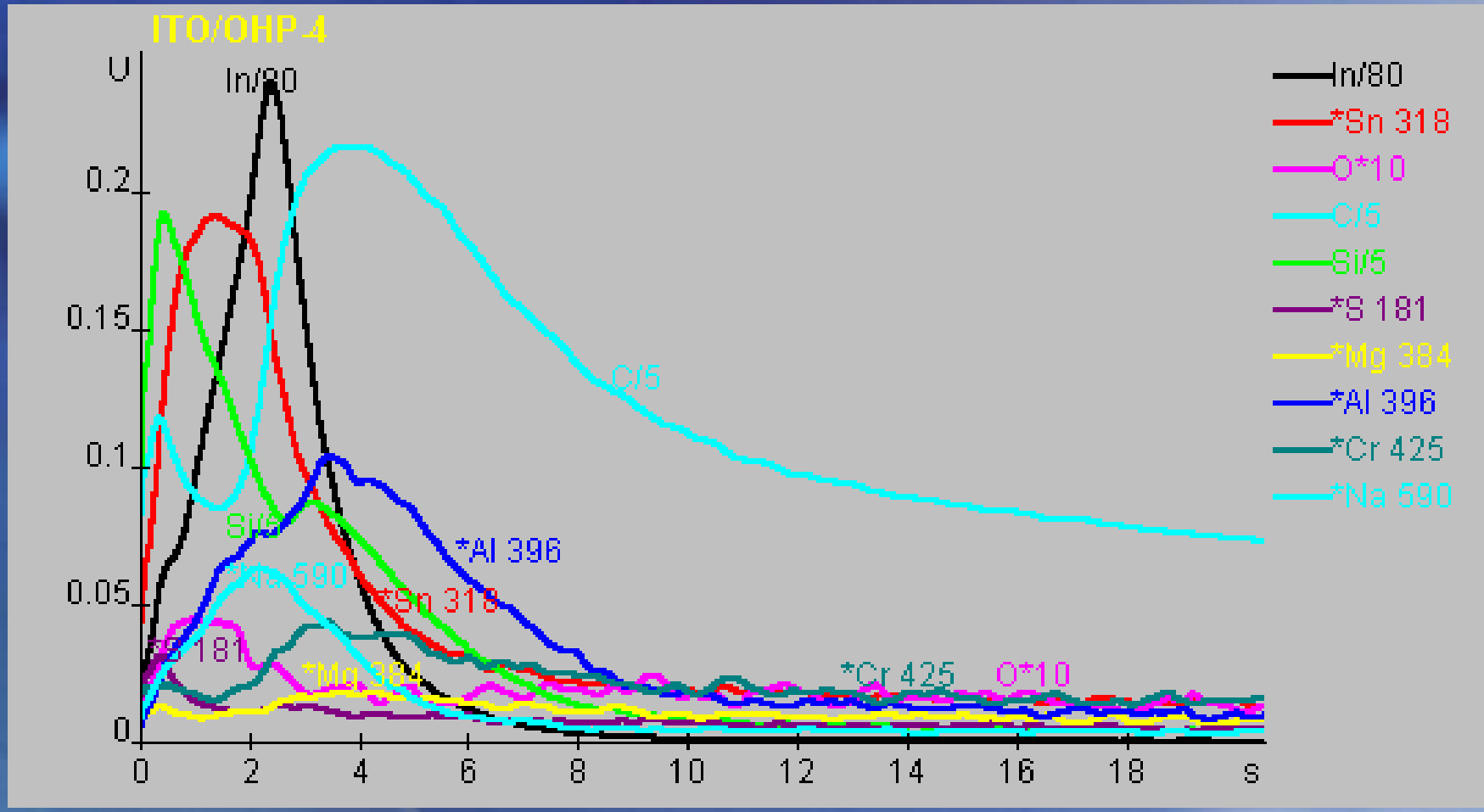
Fe and Cr signals are recorded from the beginning indicating that the outer layers are melted

Same Sample : rubber on stainless steel. Pulsed RF

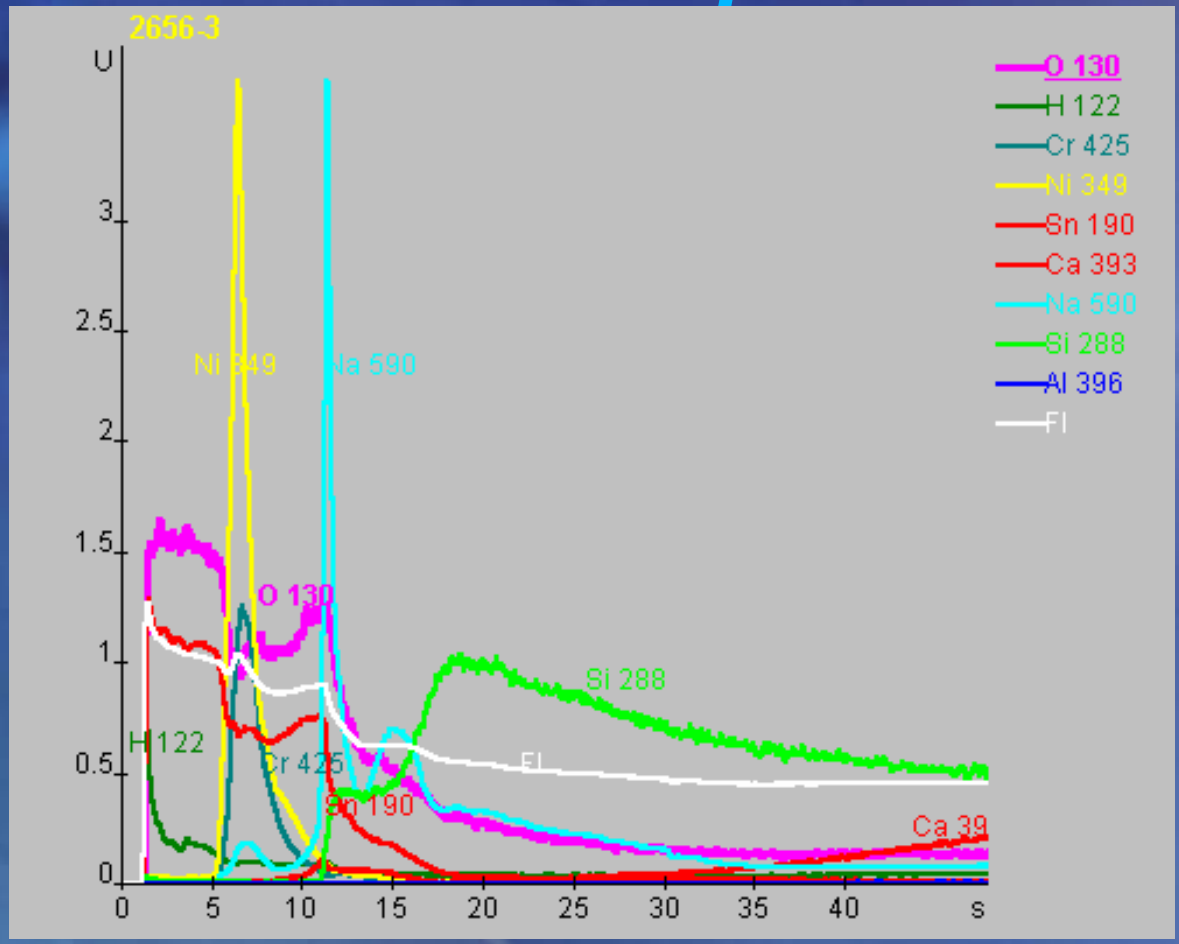


Correct analysis of the sample

Low melting point layers



Thin coatings on fragile glasses with pulsed RF

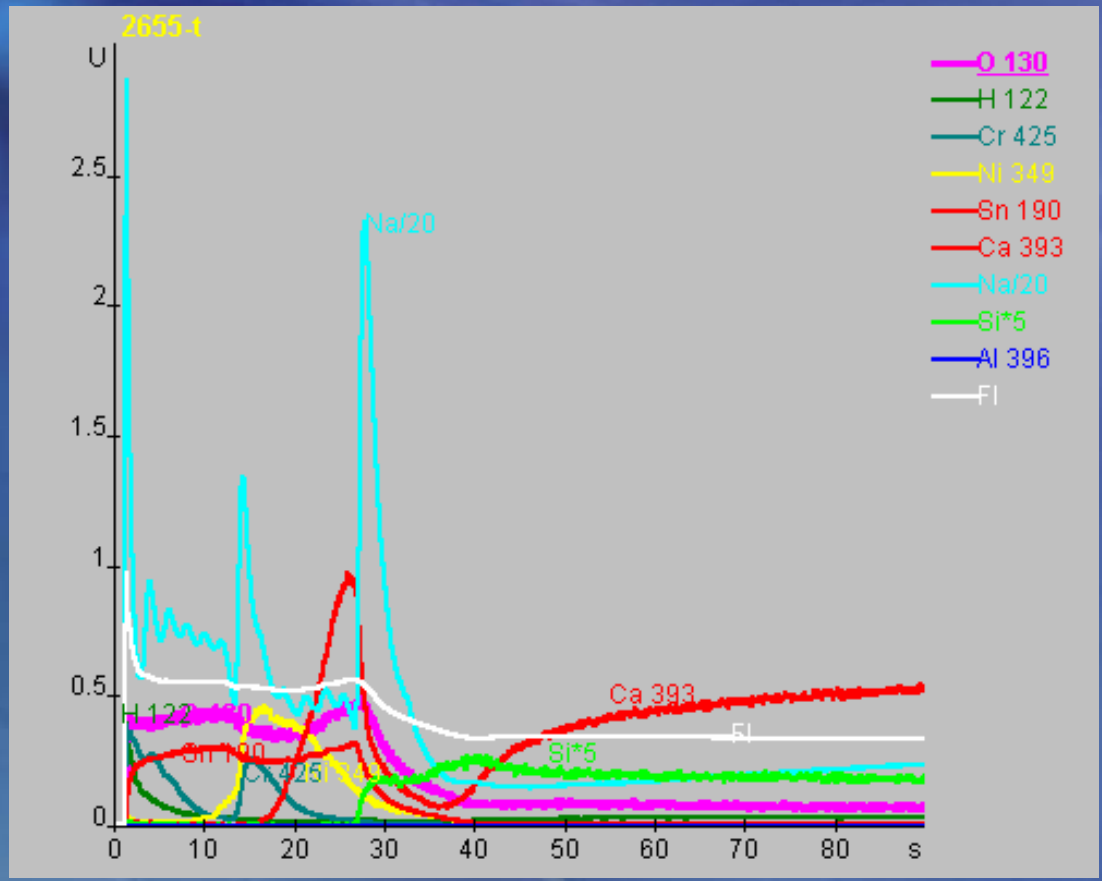


SnO2/Ni-Cr/SnO2 on glass

Original sample

Recent application done at ITU. Paper will be presented

Thin coatings on fragile glasses



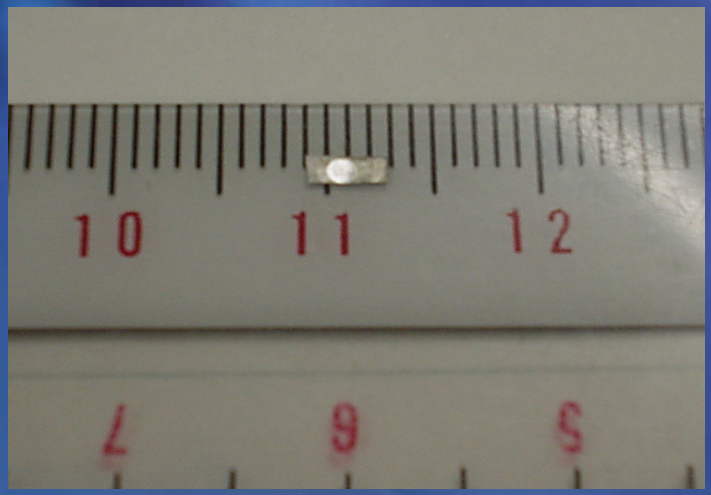
Heat treated sample

Migration of Na

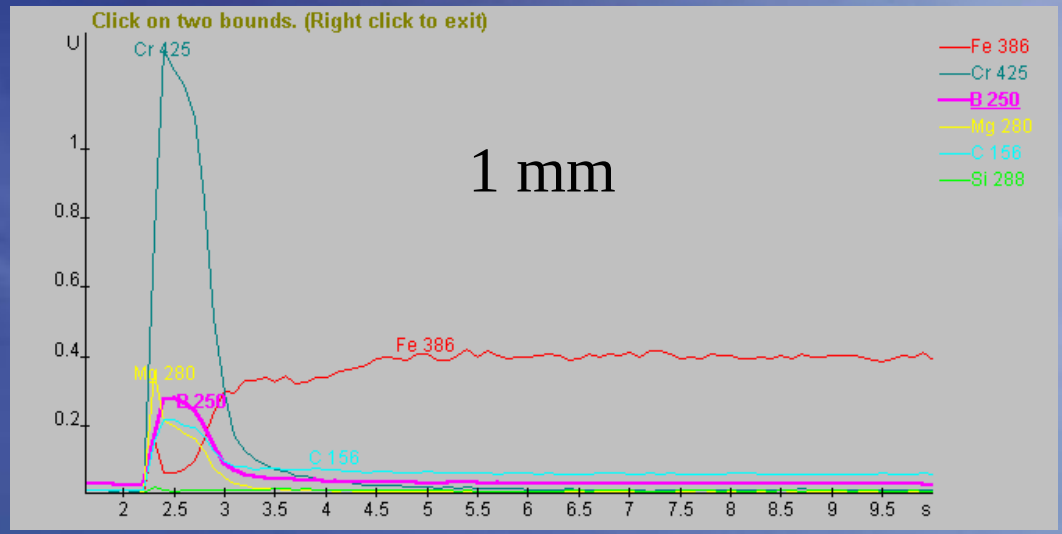
Migration of Cr

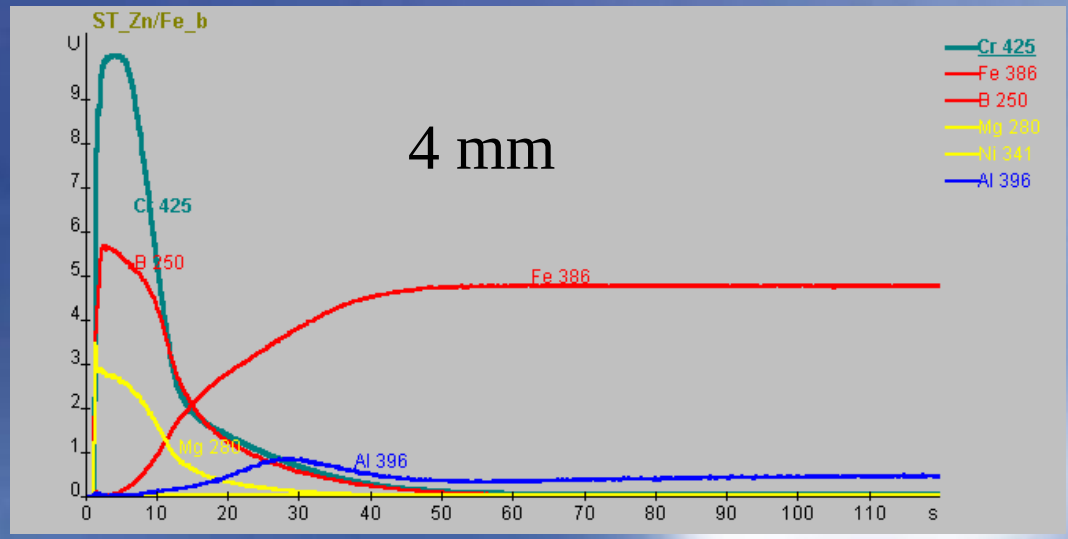
Diffusion of Sn

3) Small samples



1mm anode



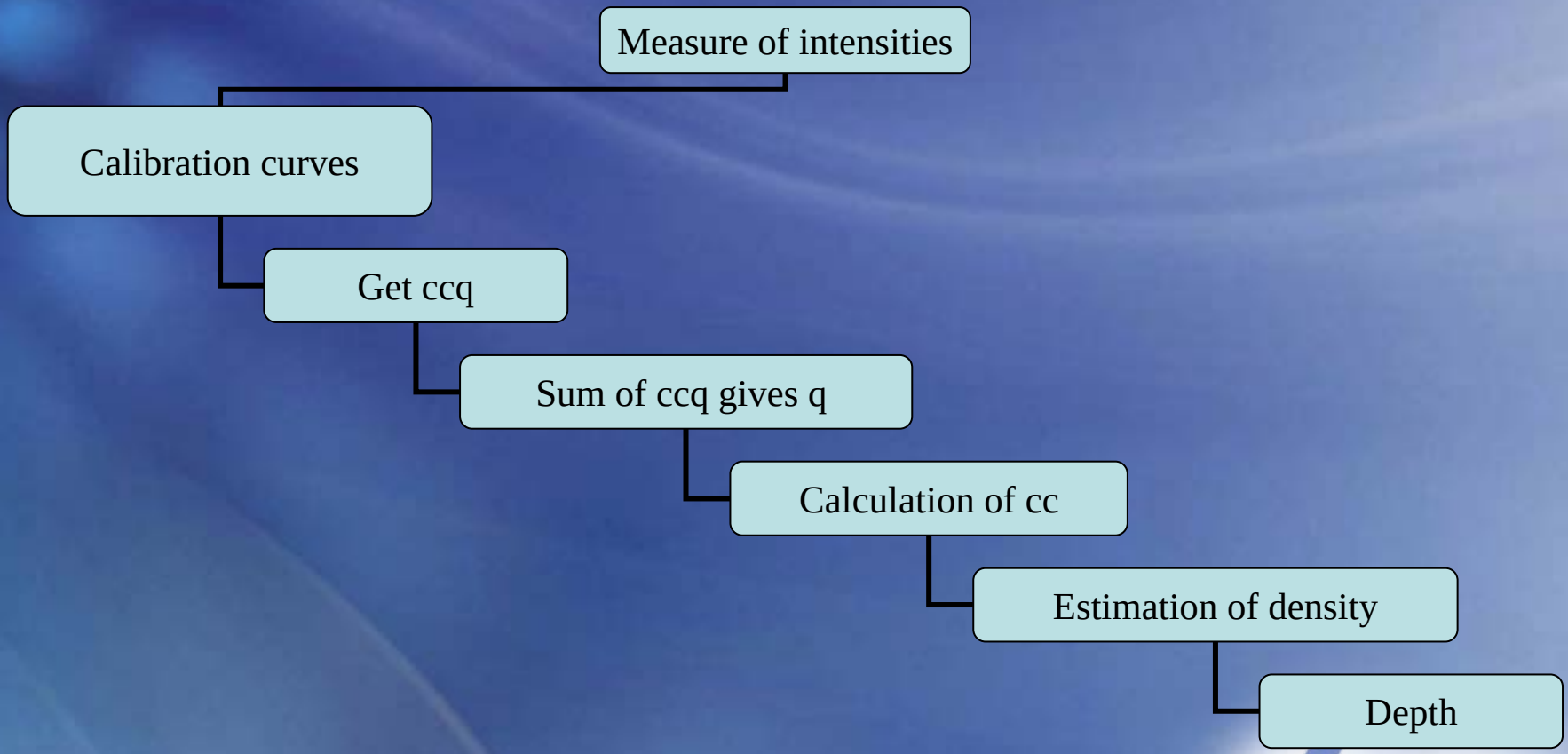


Odd shape samples

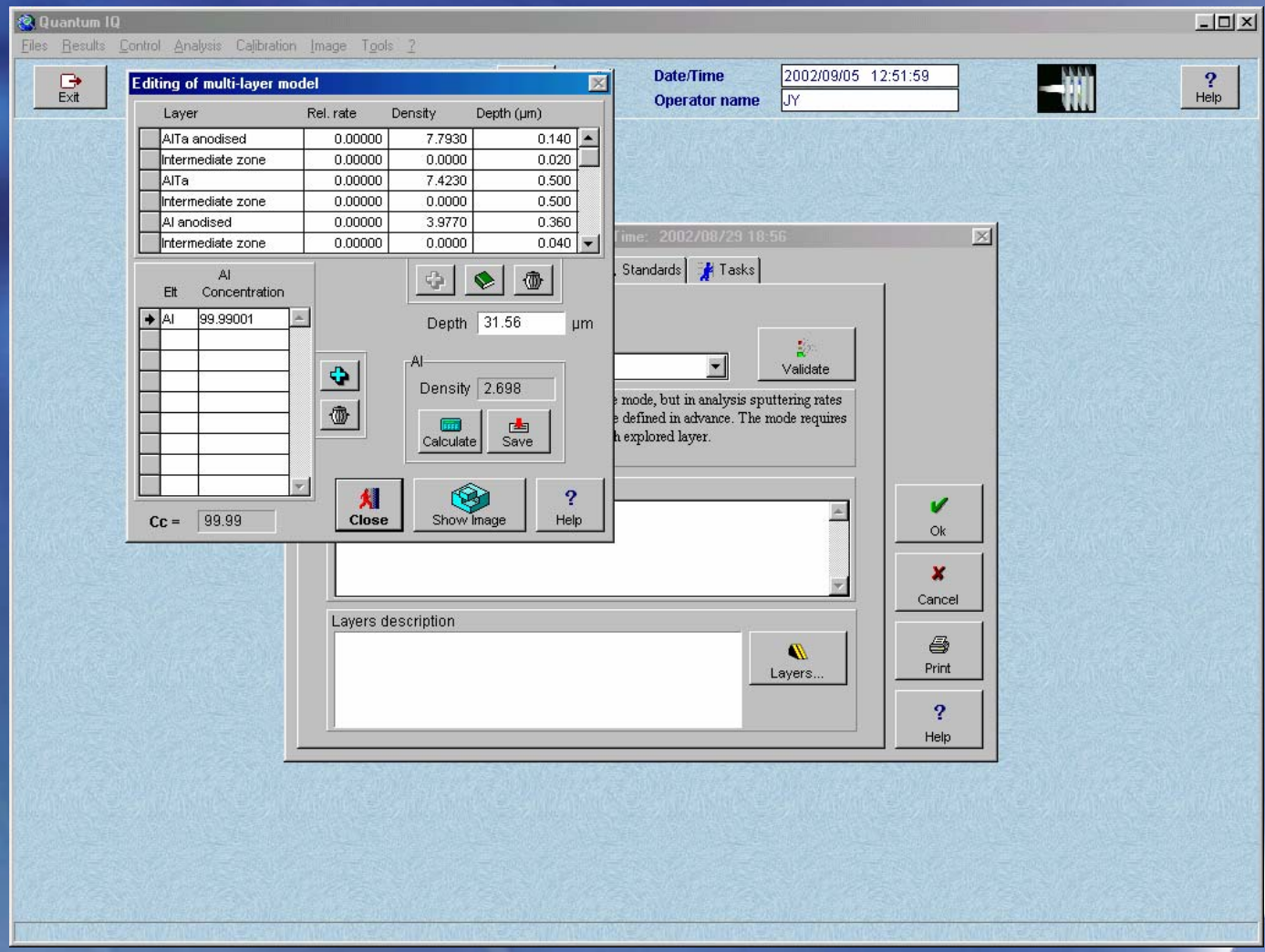
2 mm spots

Special sample holder

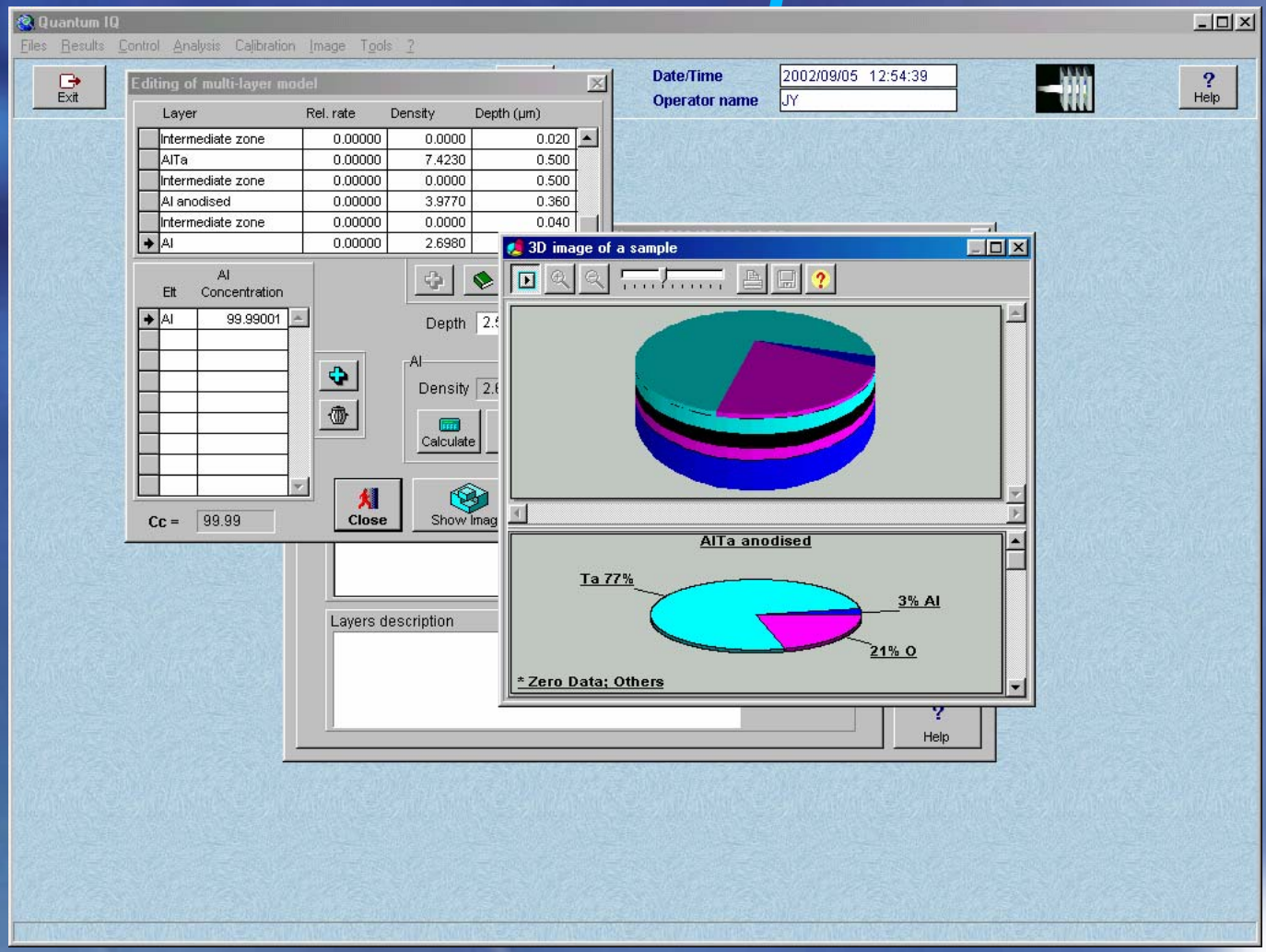
CDP : the quantification chain



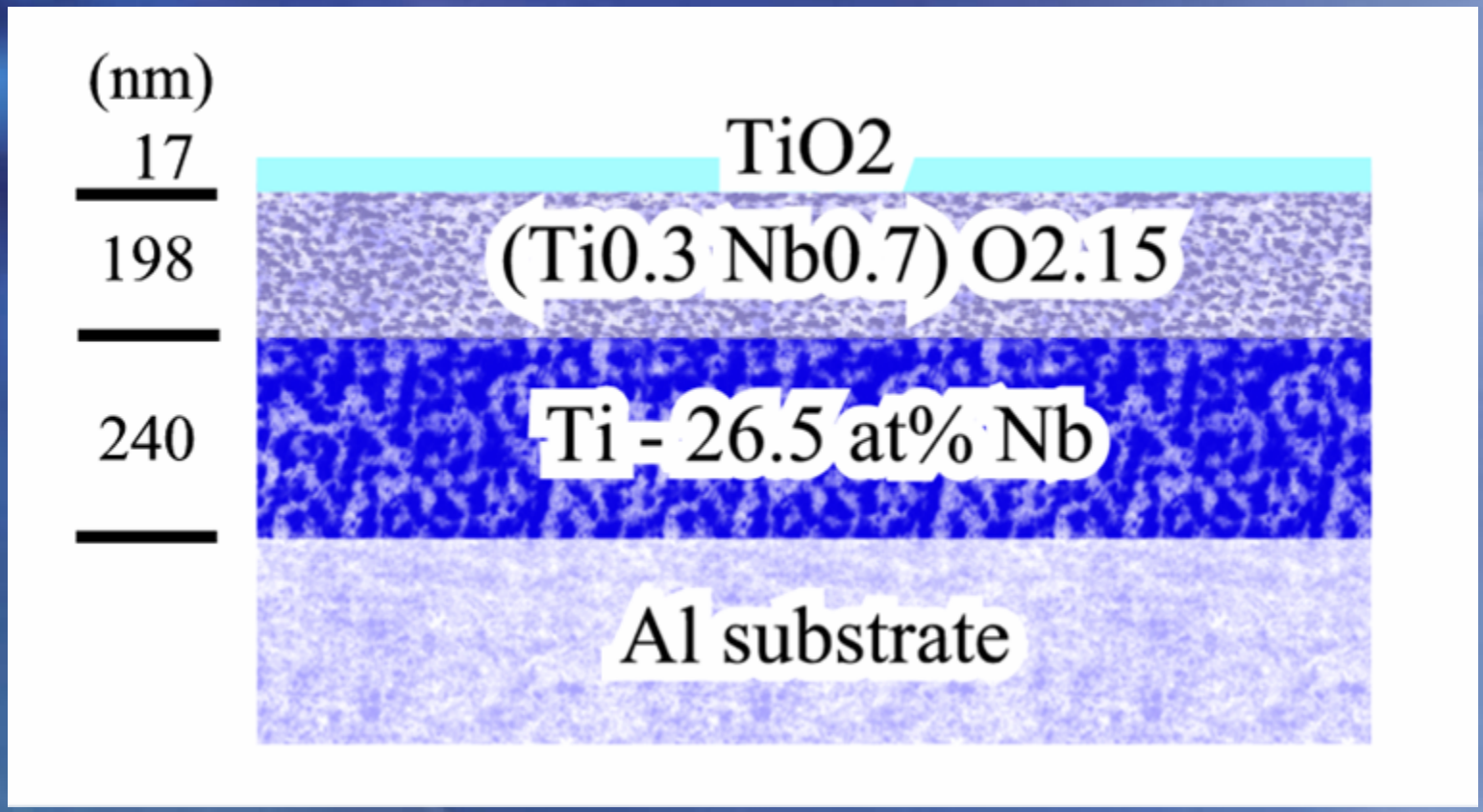
4) Layer mode : entering known information into the model



Layer mode 3D display of the sample

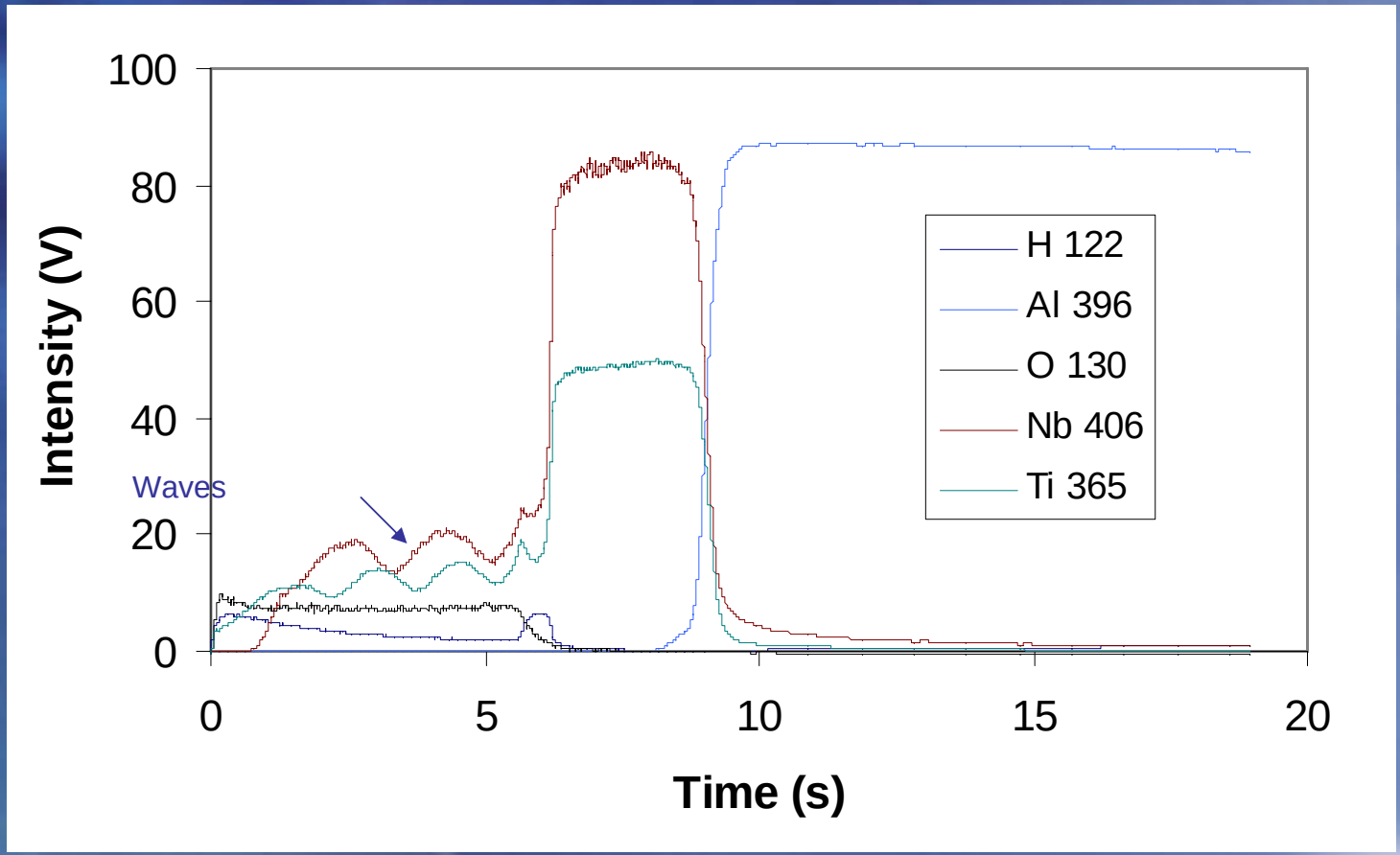


Example : Ti-Nb Layers on Al



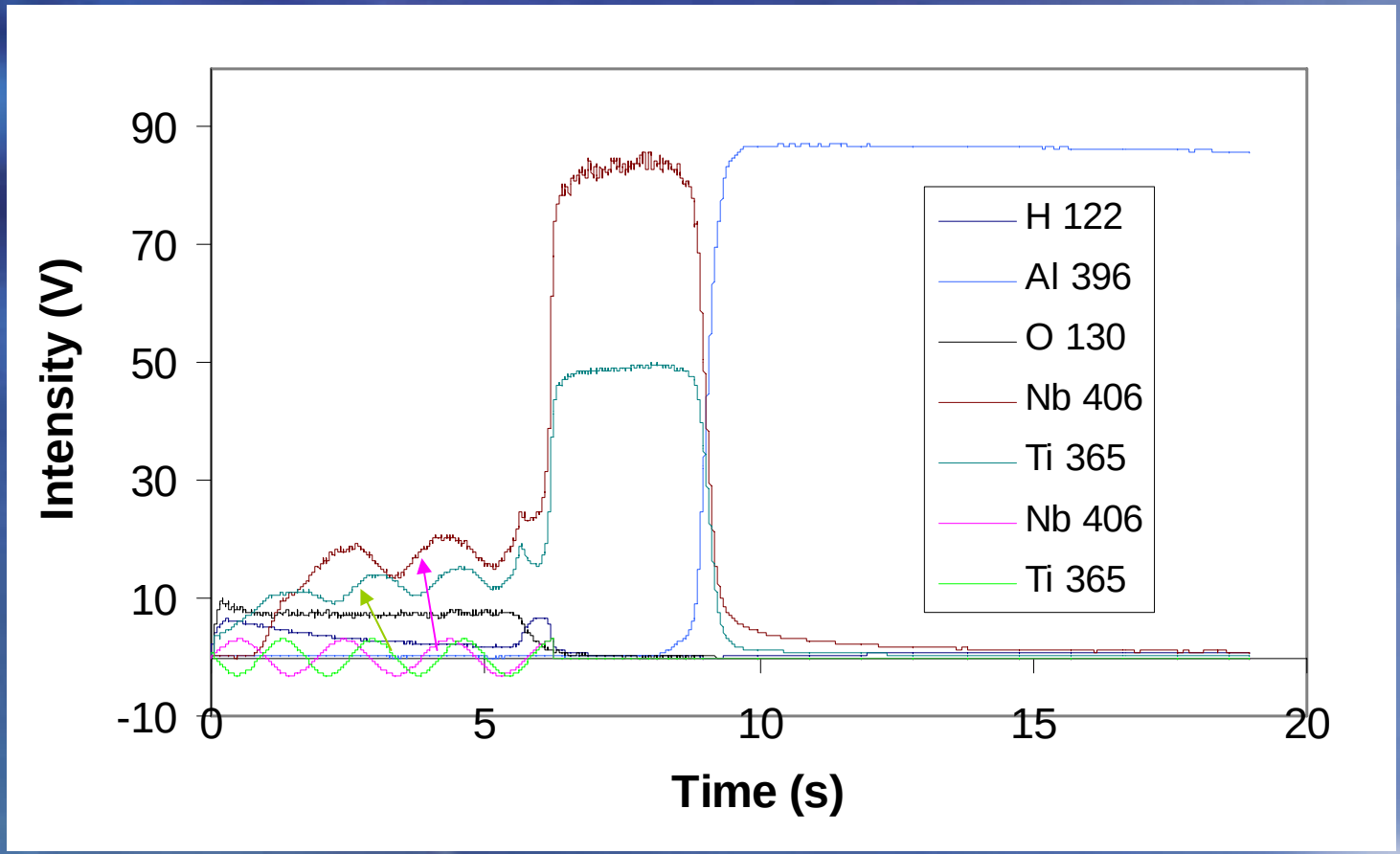
Determined by Rutherford Backscattering

Qualitative Depth Profile

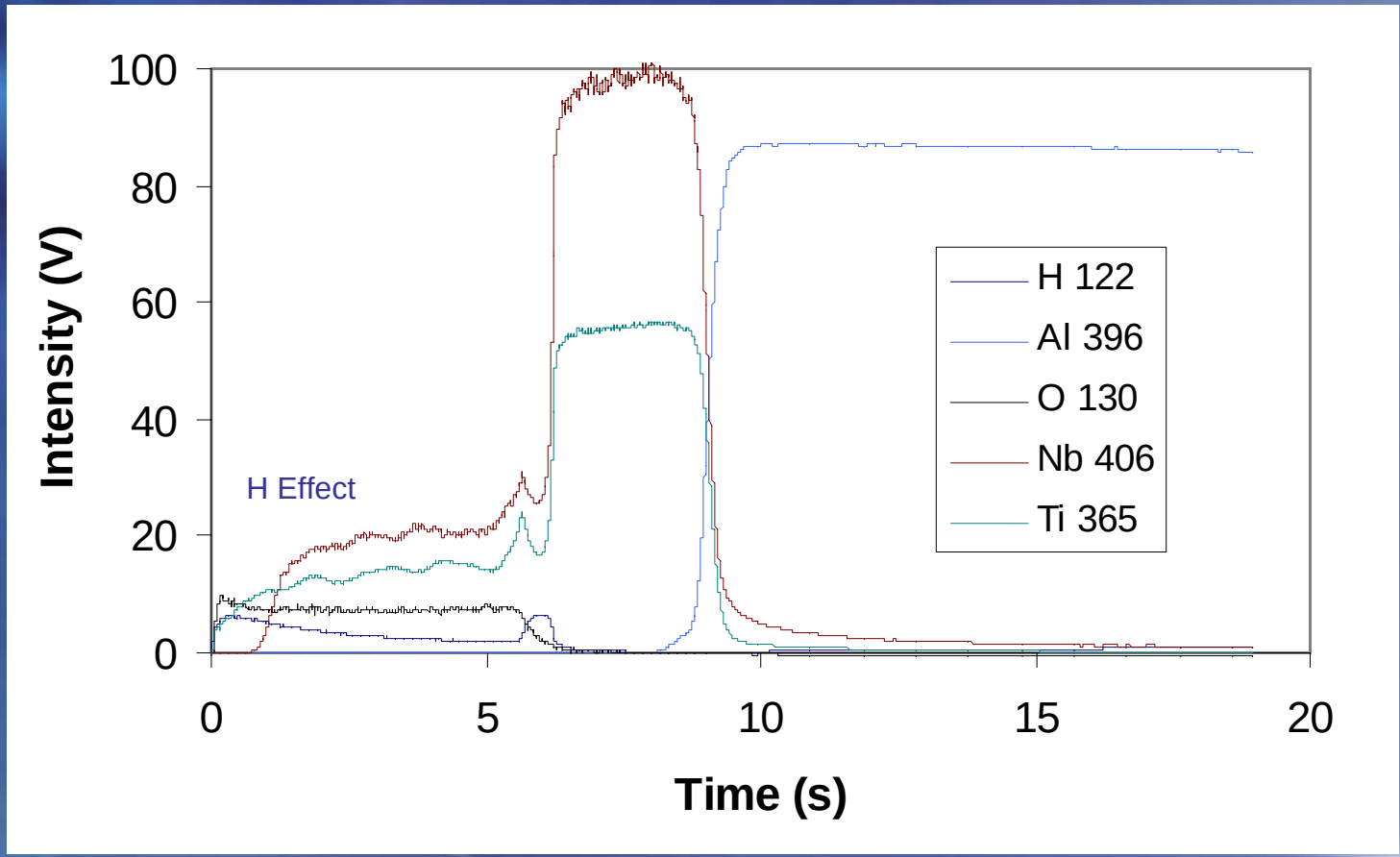


Note: 455 nm in 9 s is about 50 nm s^{-1}

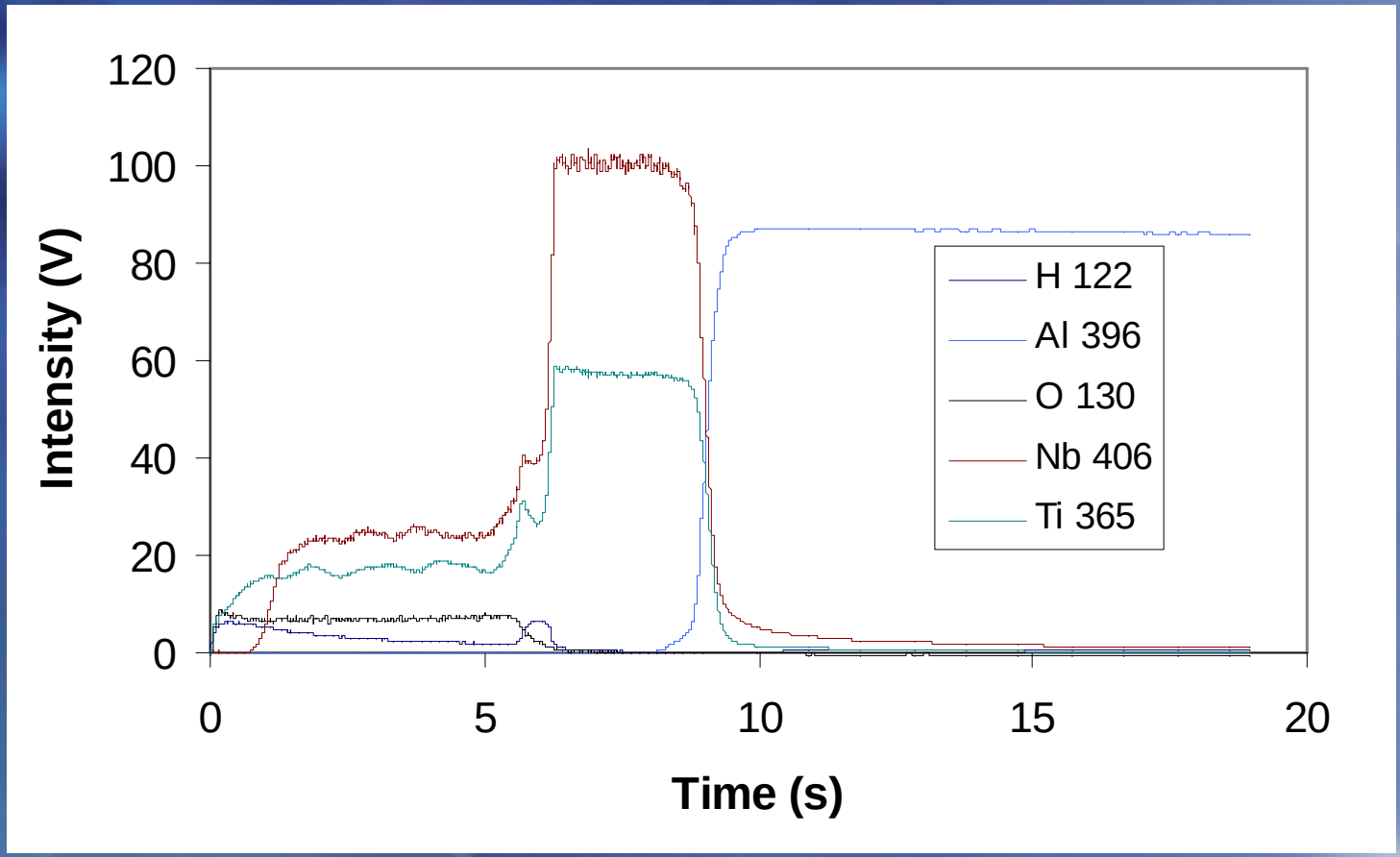
Wave Corrected Depth Profile



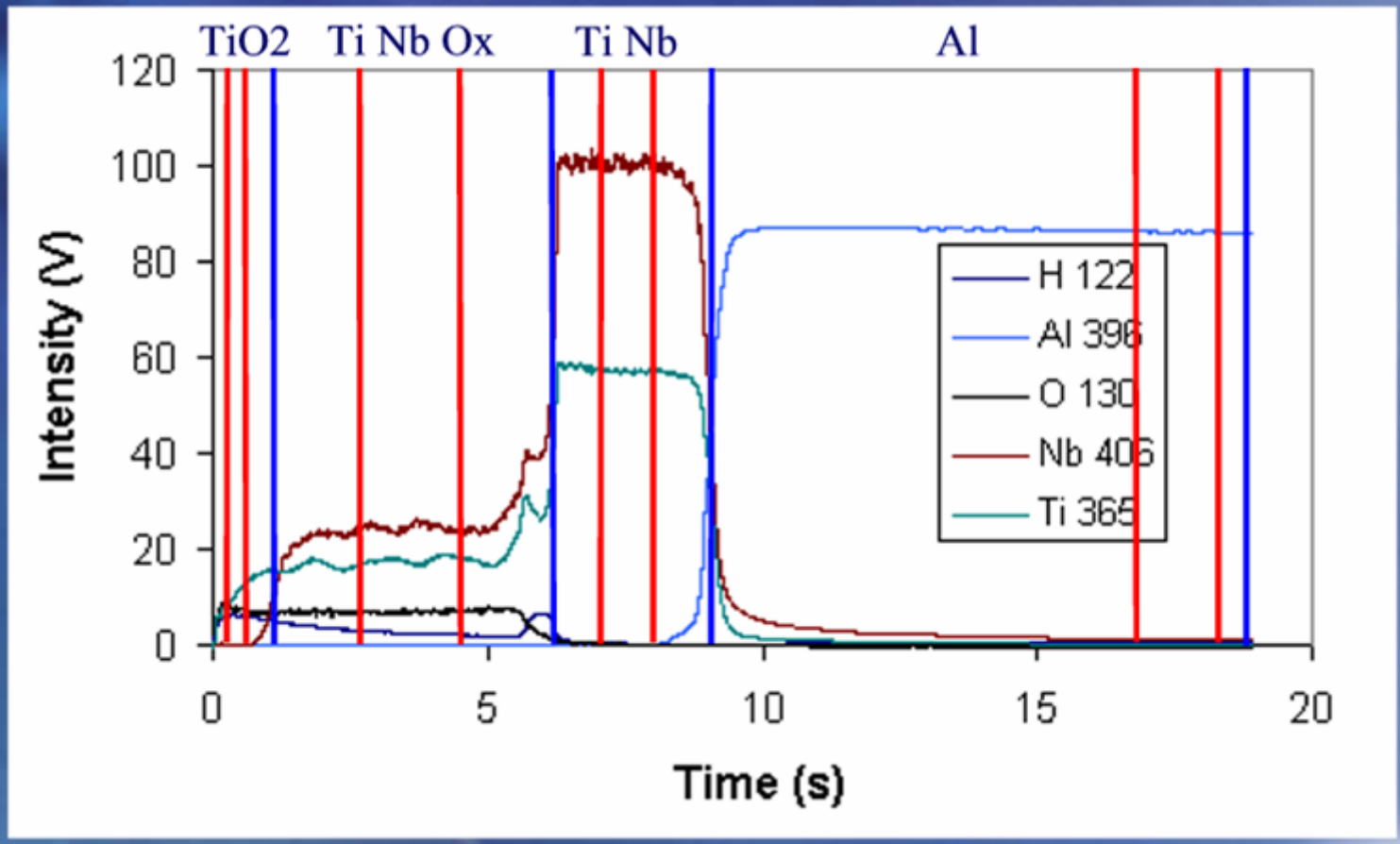
Wave Corrected Depth Profile



H Corrected Depth Profile



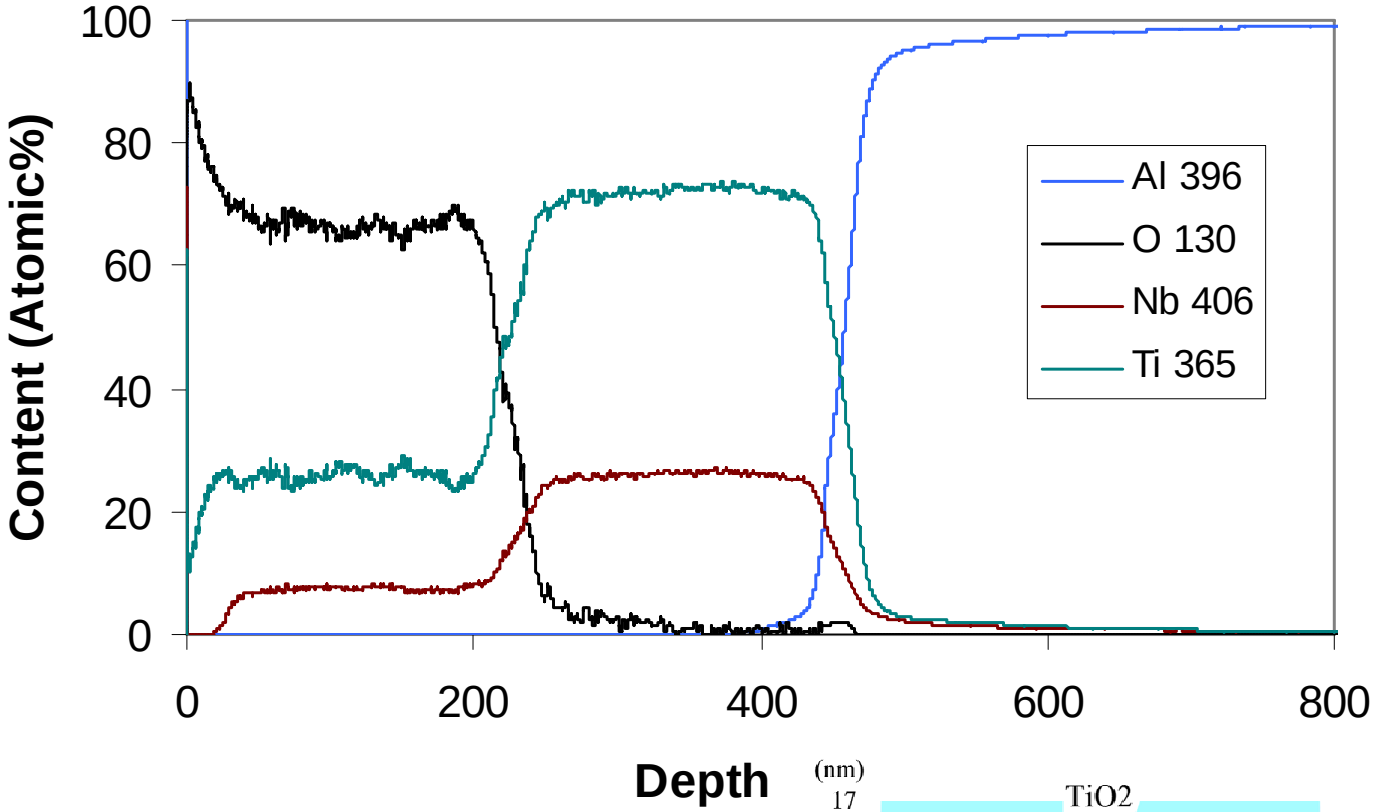
Qualitative Depth Profile

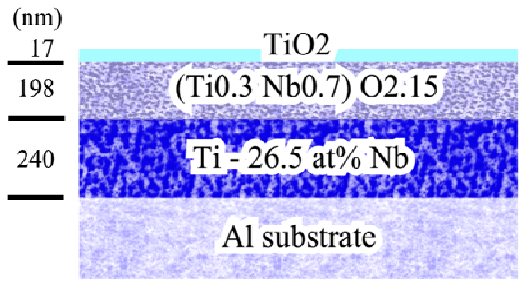


Blue: erosion rate

Red: mean intensity

Compositional Depth Profile

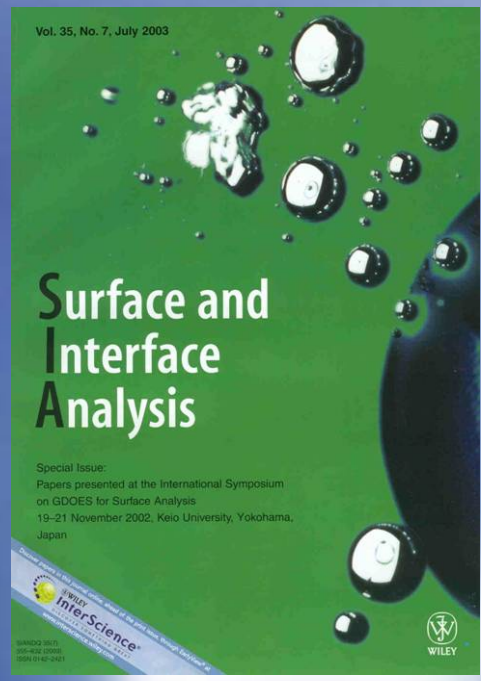




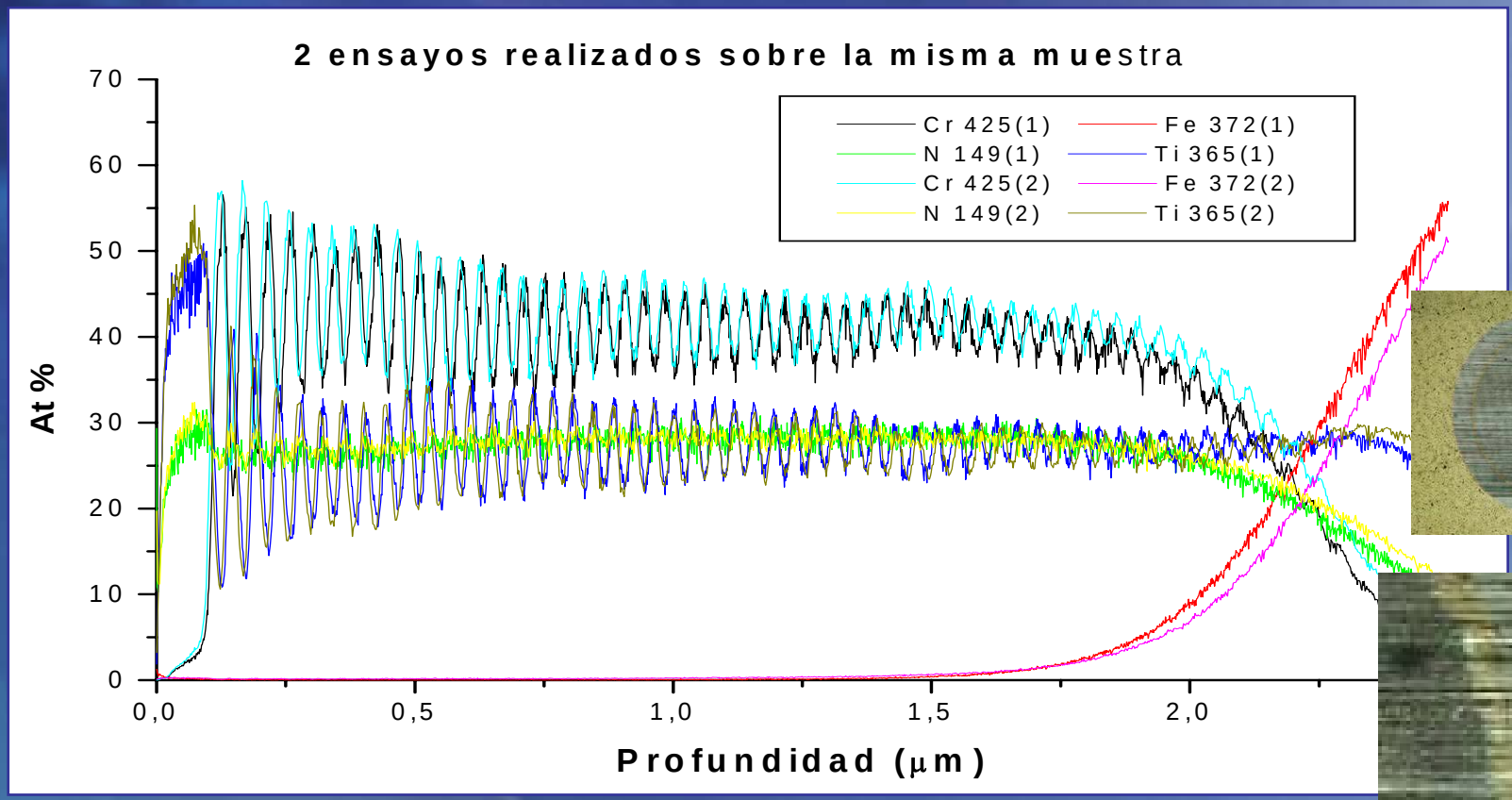
More on thin layers

- ISO TC201 SC8.
- First norm published : Introduction to use
- Second norm finished : Zn coatings
- Start of a new work on thin oxides on metals

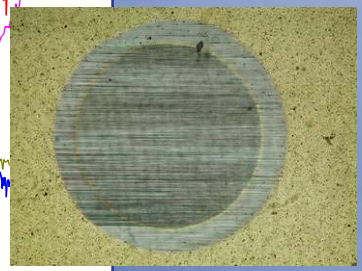
- Special edition of Surface and Interface Analysis (Vol 45, 7) based on papers presented at the first international symposium on GD-OES for Surface Analysis

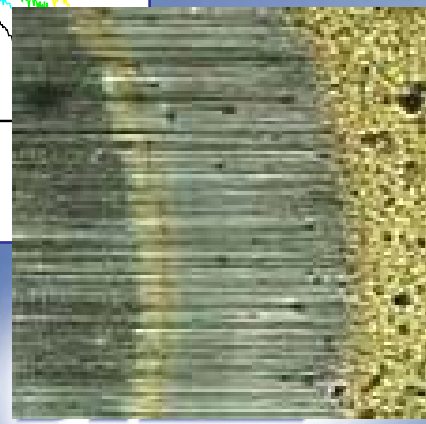


Repeatability of the JY GD



107 layers

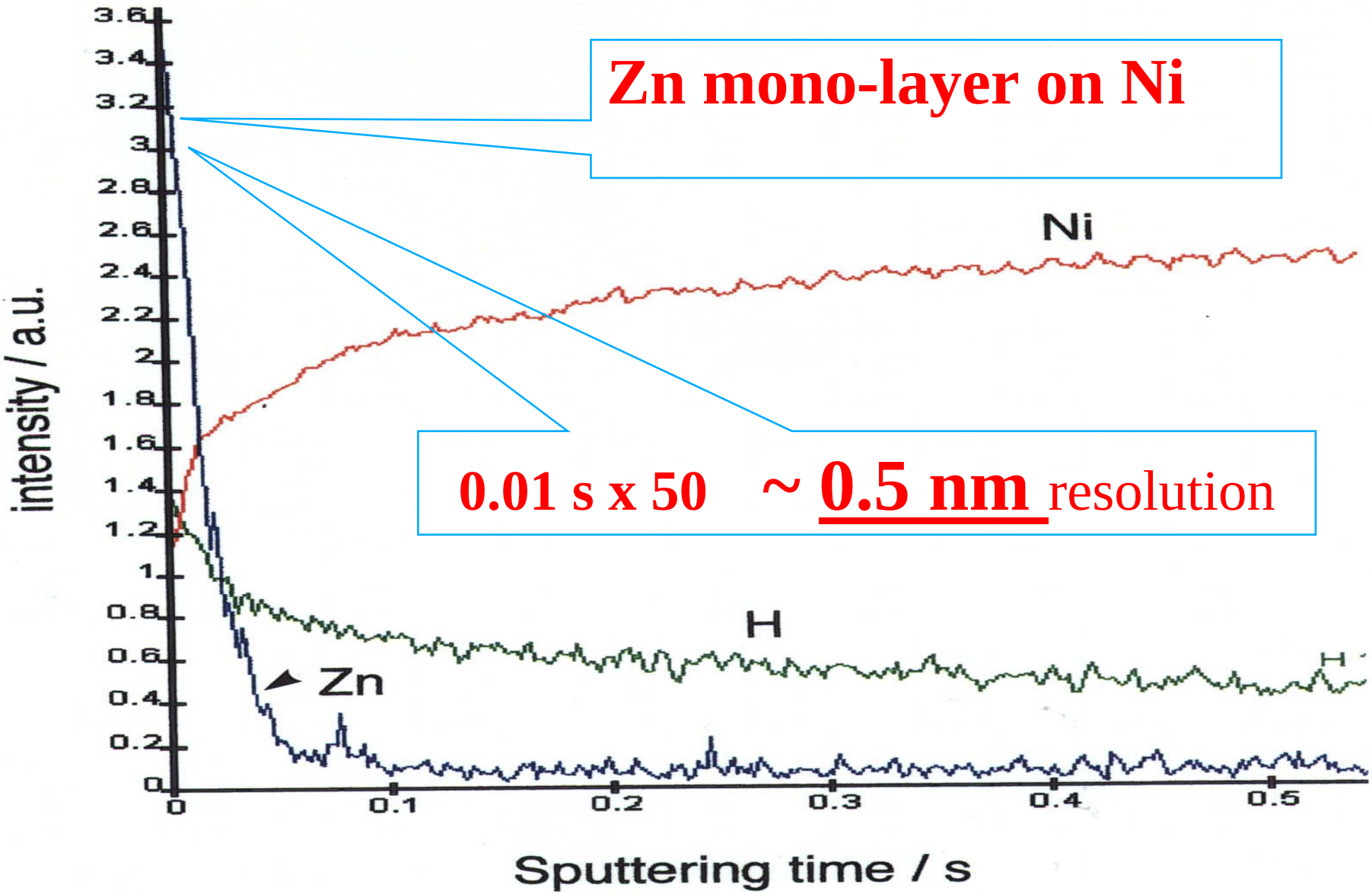




Zn Monolayer on Ni

- Ni: substrate, electropolished, high purity
- Zn: single atomic layer
  - deposited electrochemically
  - under potential deposition (UPD) method

Surface analysis – by GDS



Conclusions

- Recent advances in theory and practice extend the range of RF GD-OES applications :

- **Analysis of large samples**
- **Fragile samples in pulse mode**
- **Odd shape samples**
- **Layer mode offers simplified accurate CDP**
-

GD-PROFILER