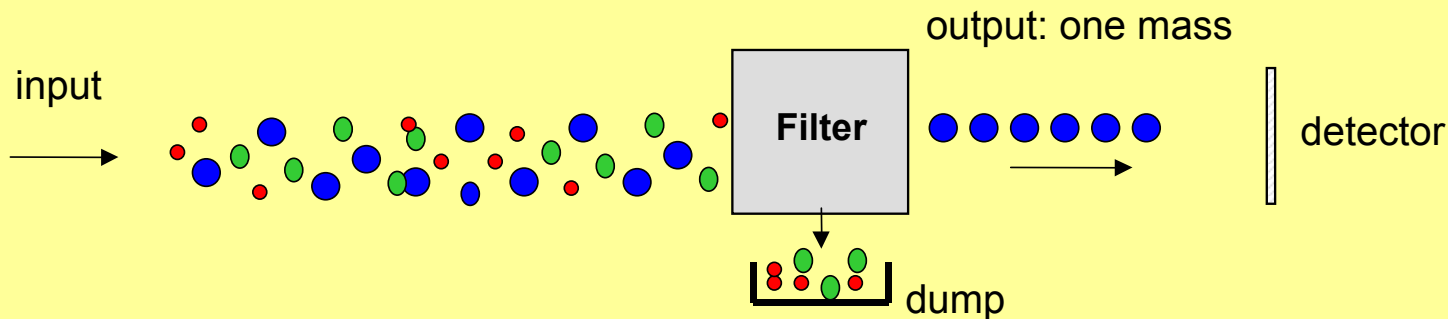


Zwei Beispiele von Aktivitäten in Thun:

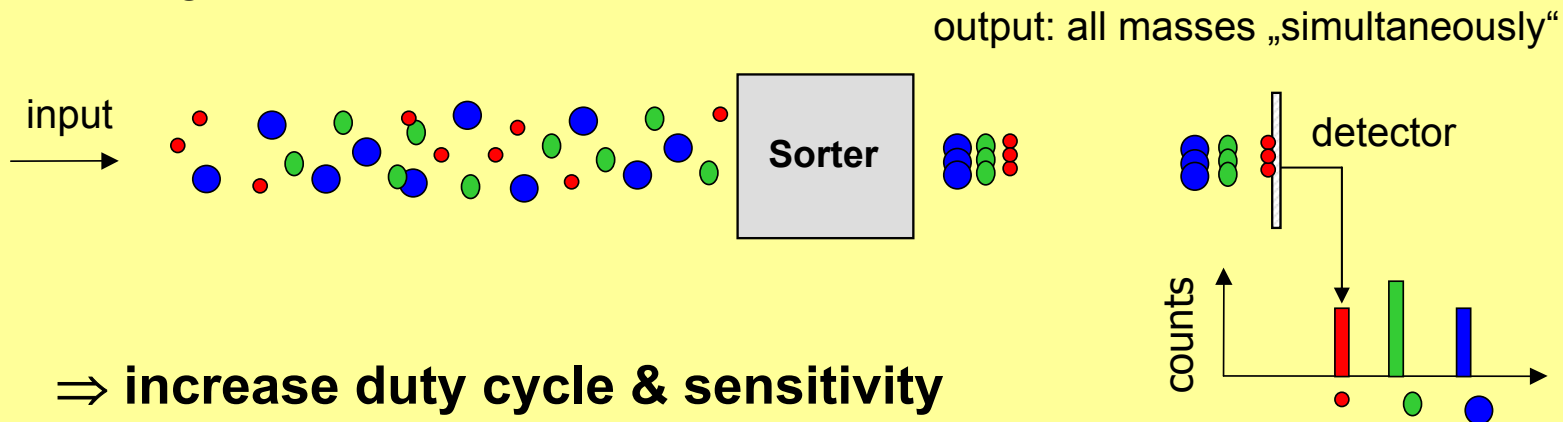
- Entwicklung einer GD-TOFMS
- Analyse von Blei Zirkonat-Titanat (PZT) Schichten mit gepulster GD-OES

Why TOF: it's not a filter

Quadrupole MS, Sector MS



TOF

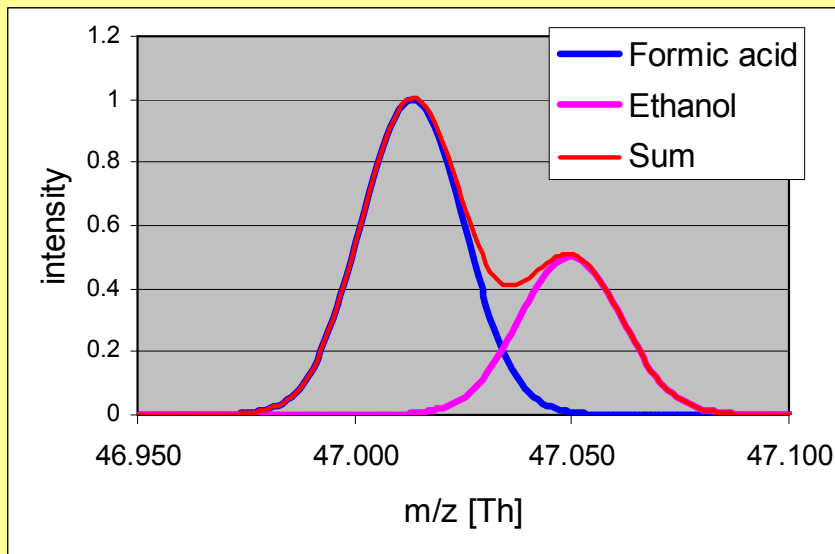


Why TOF: mass resolution

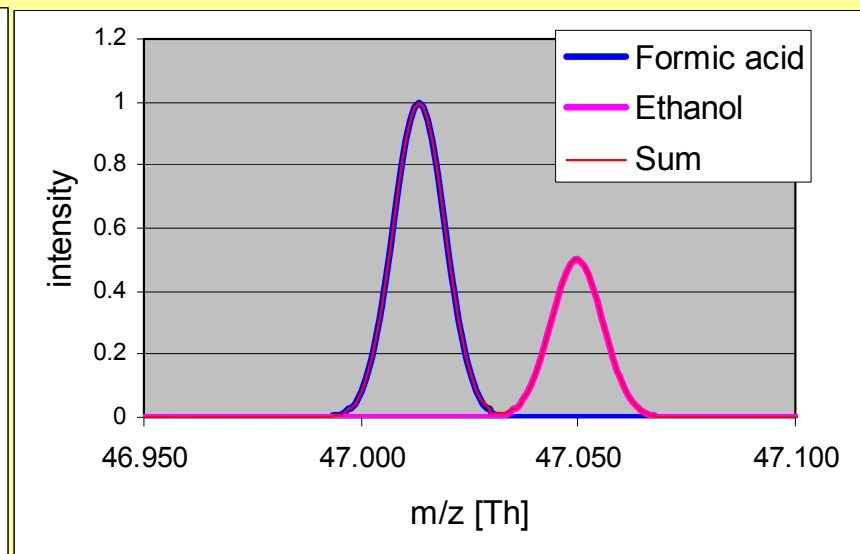
Definition:

mass resolution: $R = m/\Delta m$ ($\Delta m = \text{FWHM}$)

TOF resolution 1700

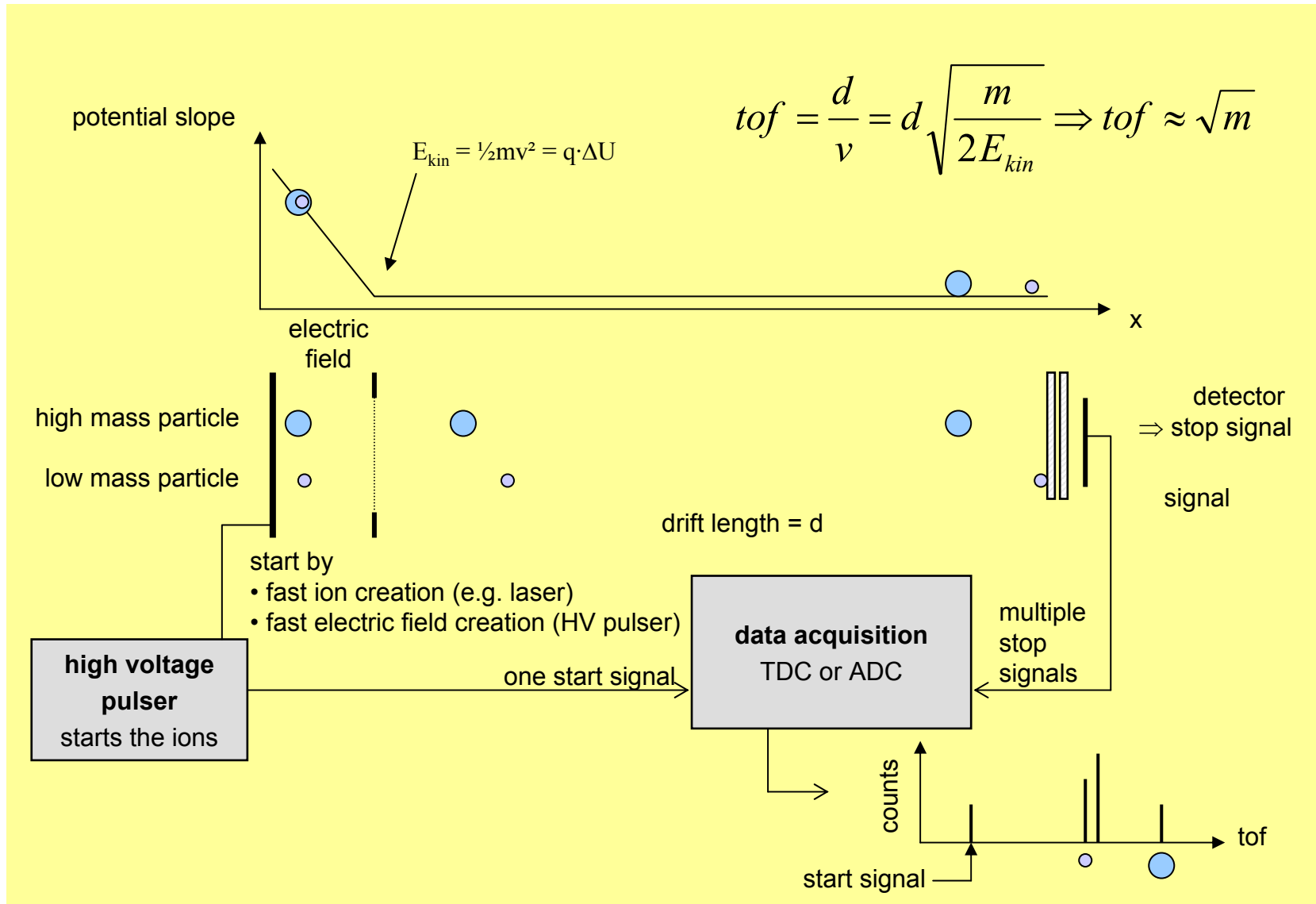


TOF resolution 3400

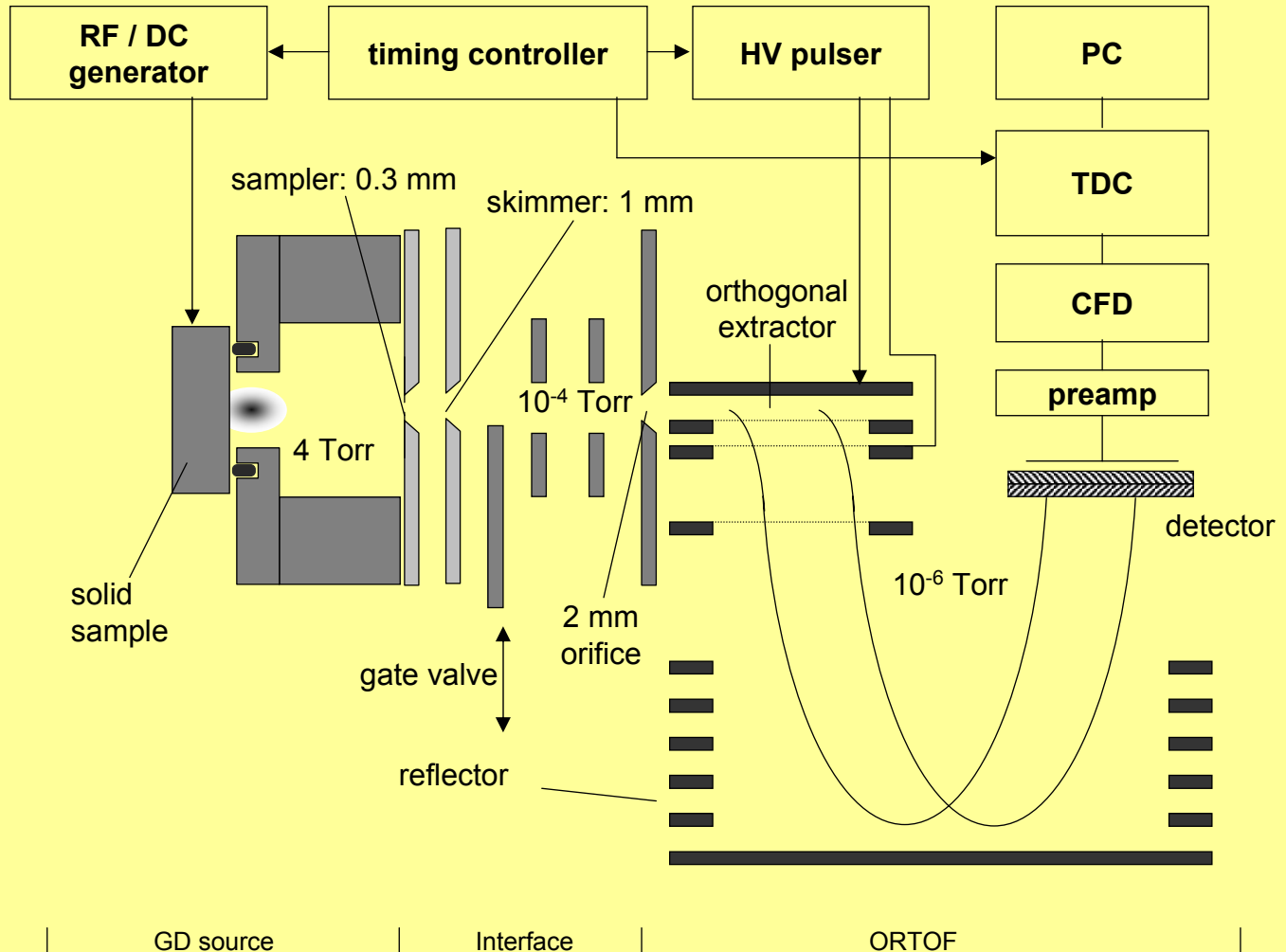


⇒ reduce interferences

Intro: basic principle of mass separation



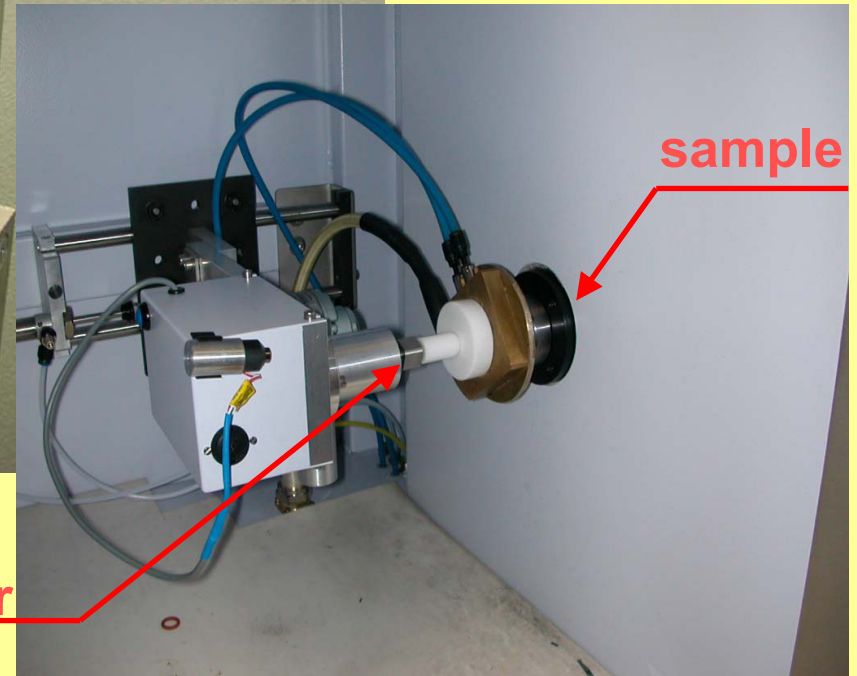
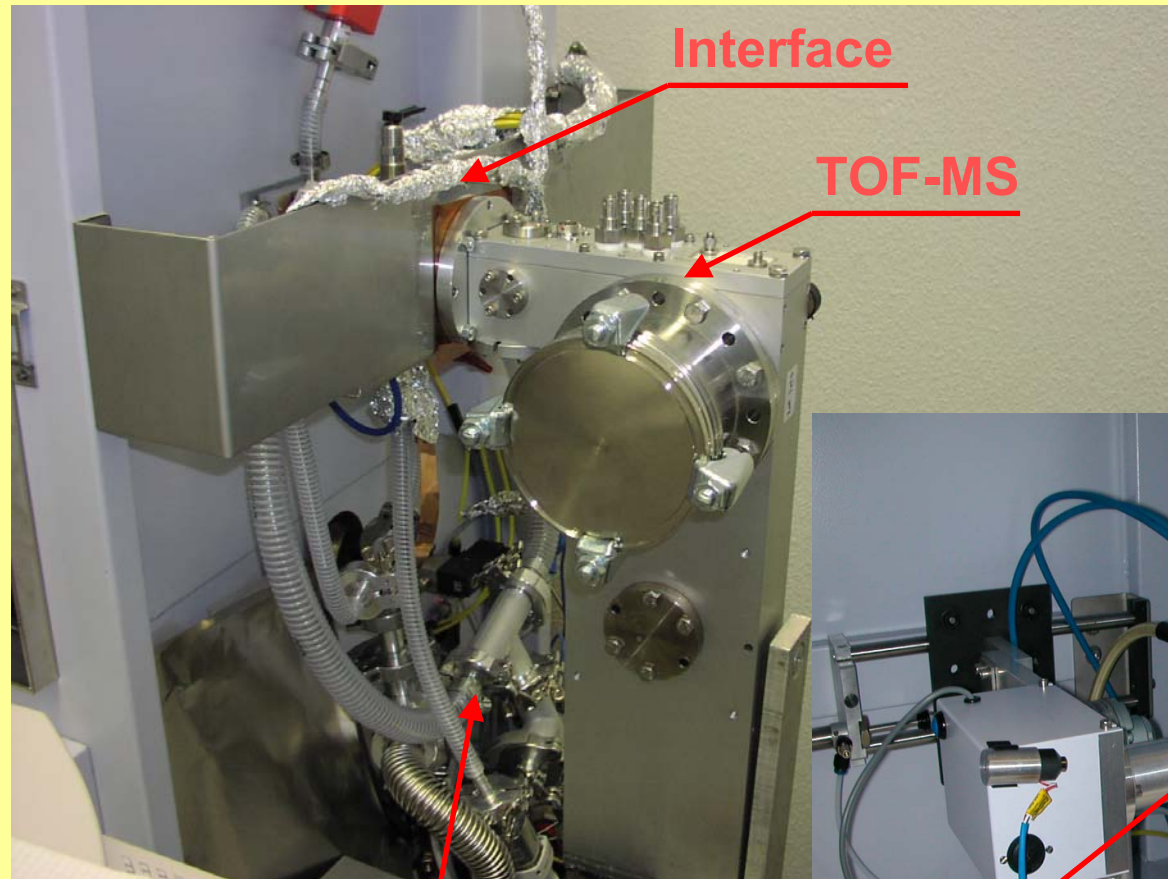
EMPA GD-TOF-MS Scheme



GD-TOF-MS (experimental setup)



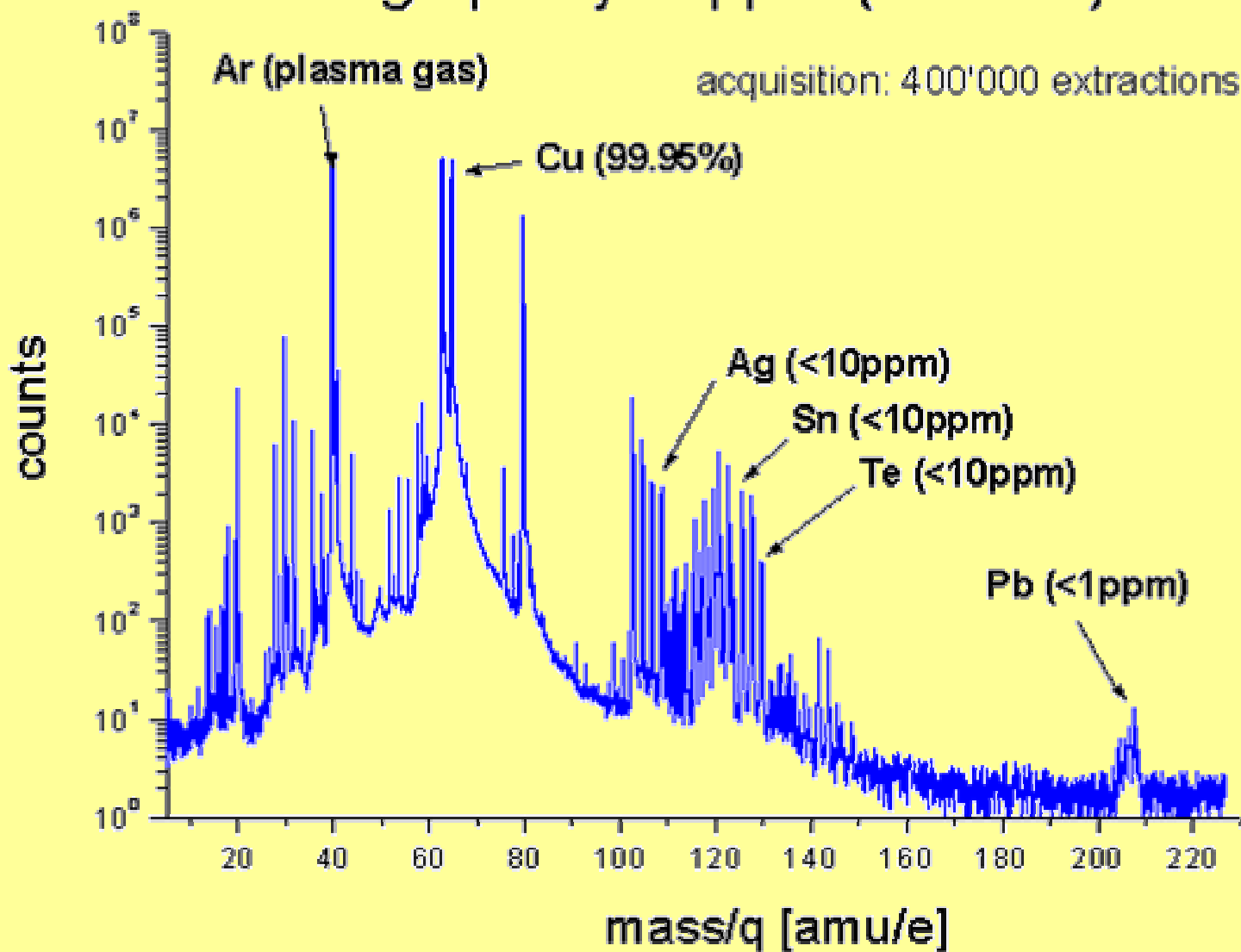
GD-TOF-MS (zoomed view)



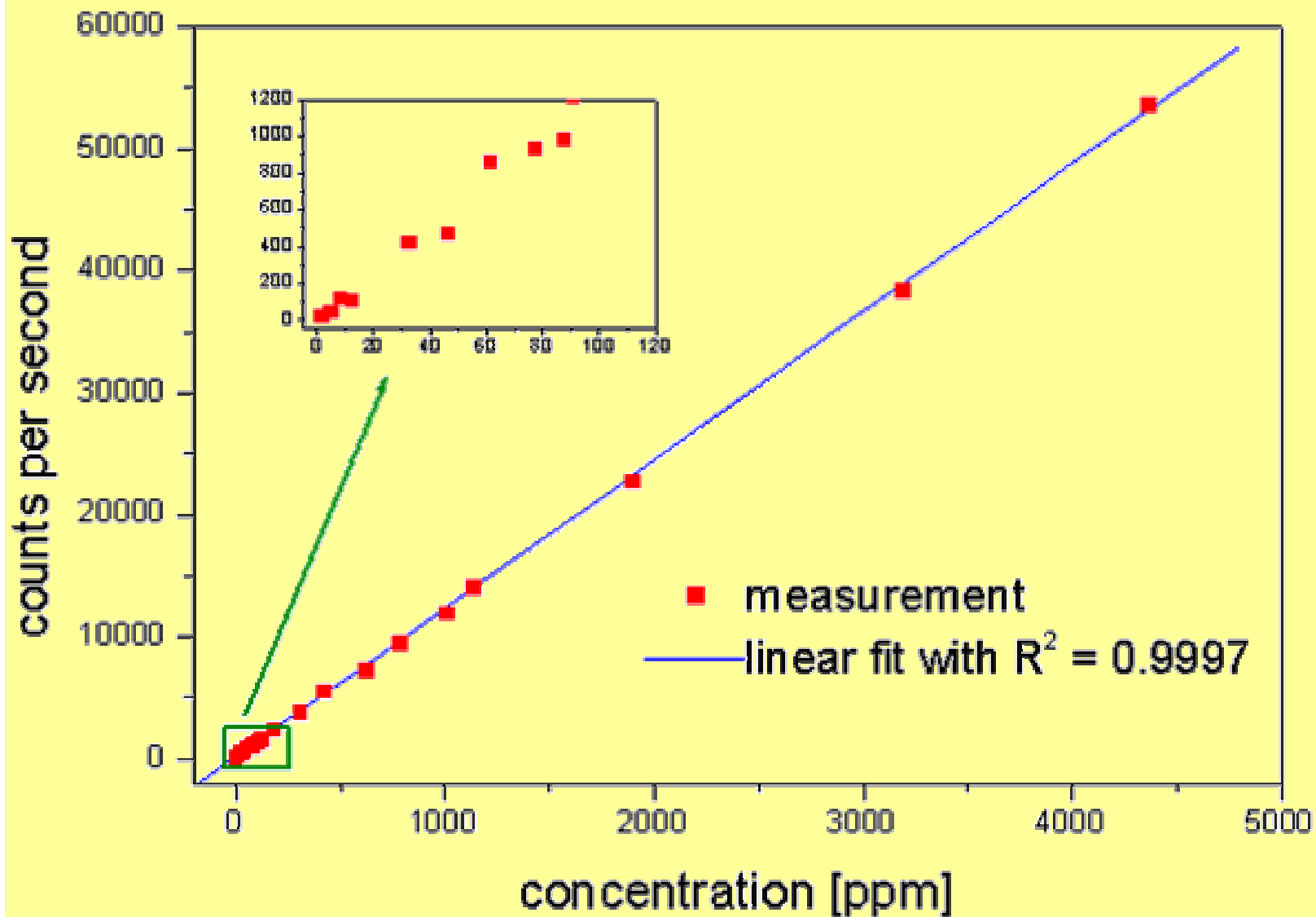
Vacuum system

Sample holder

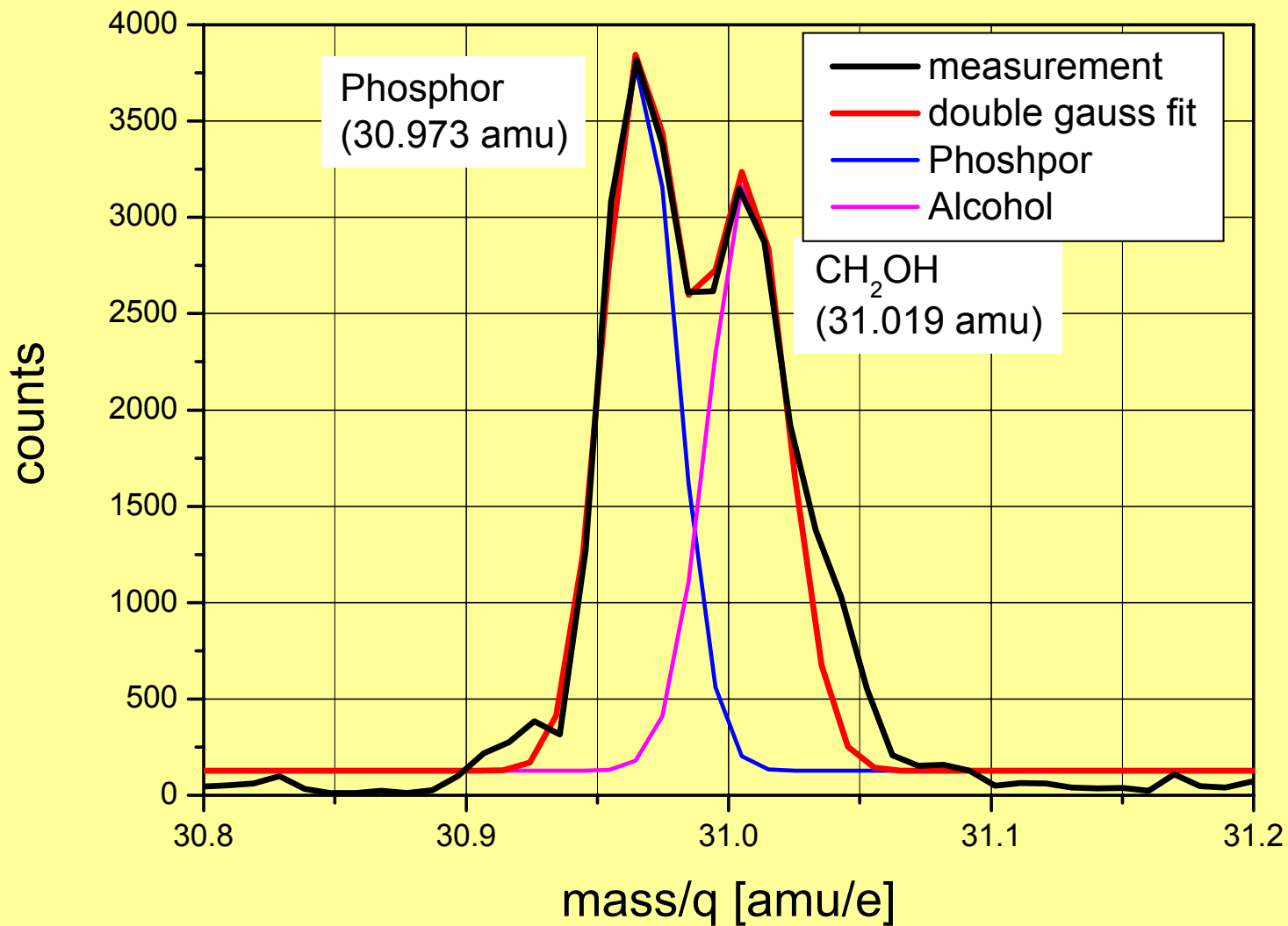
High purity copper (99.95%)



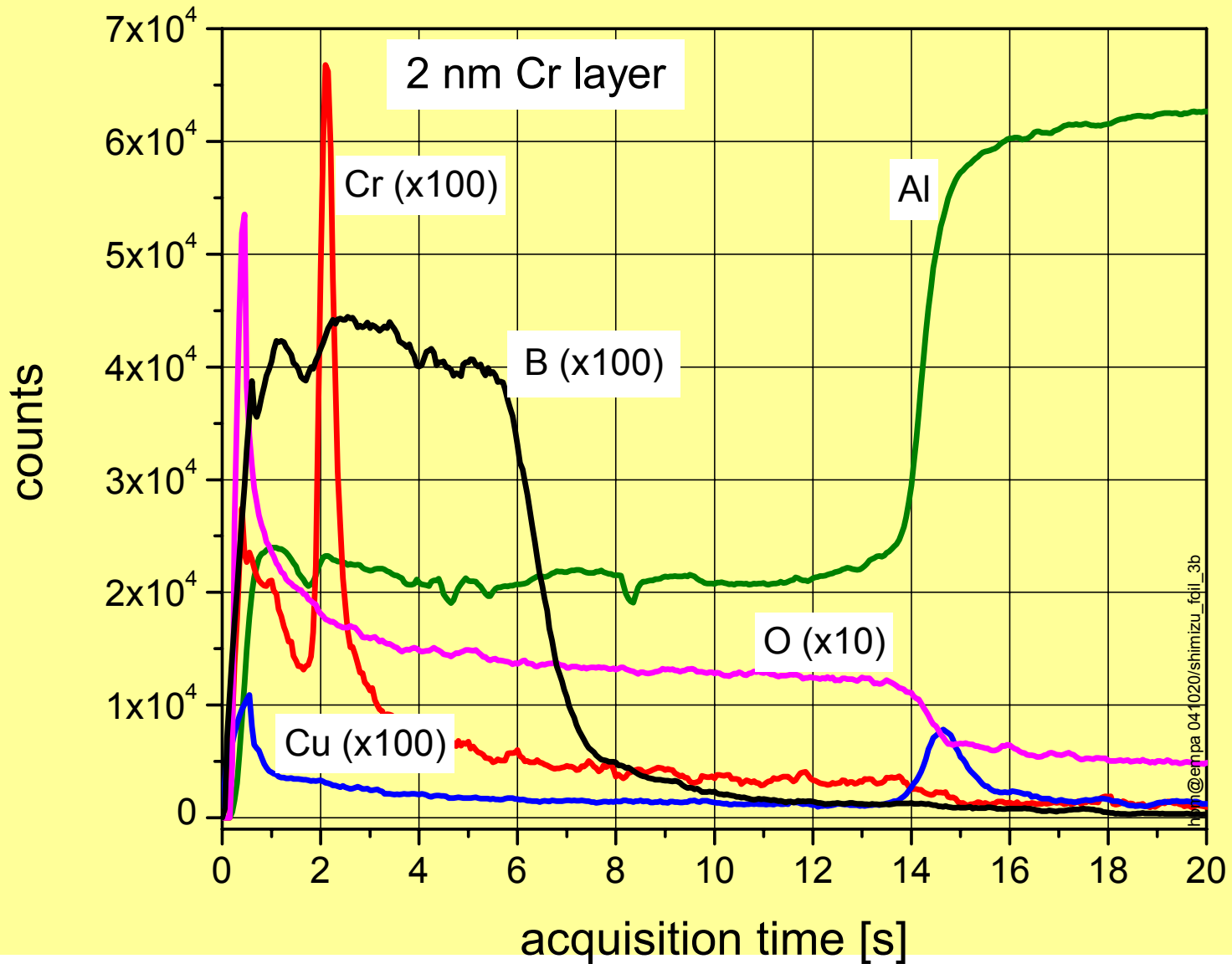
Calibration curve for Sn in Cu



separation of P and alcohol



Depth profile of Al_2O_3 with a chromium marker of 2 nm



Probleme welche noch gelöst werden müssen:

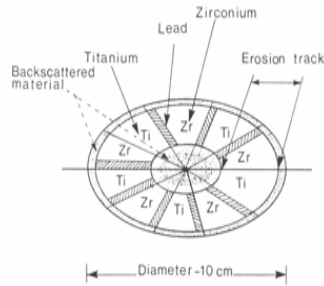
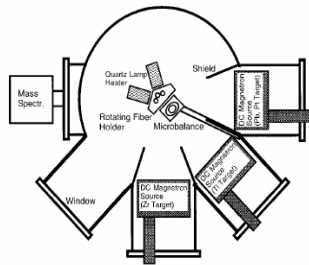
- **Die Kraterform: entweder sind die Intensitäten sehr hoch aber die Kraterform ist nicht ideal oder die Kraterform ist brauchbar aber dafür sind die Intensitäten eher tief.**
- **Der dynamische Bereich des Detektors ist noch nicht befriedigend. Obwohl wir bereits mehr als 7 Größenordnungen an Intensität erreichen wären 9 bis 10 wünschenswert..**

Rapid depth profiling of lead zirconate titanate (PZT) thin films by pulsed glow-discharge optical emission spectroscopy

EMPA Materials Science and Technology
aFeuerwerkerstr. 39, CH-3602 Thun, Switzerland
bLerchenfeldstr. 5, CH-9014 St. Gallen, Switzerland

Piezoceramic thin films

- Application: FRAM, MEMS
Mass production (industrial production line): **single target**



- New: **DC pulsed**



Application fields for thin PZT coatings

Ferroelectric-RAM (FRAM):

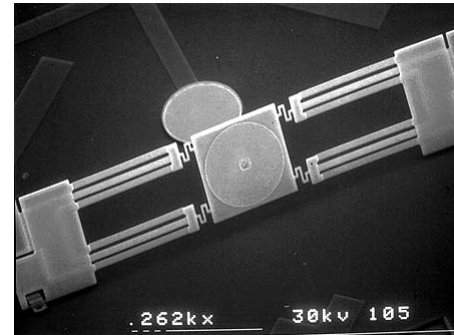
non-volatile memory
designed to replace flash memory
faster, less power, more easily intergrated with other
circuits on a chip than flash)



<http://www.pbliz.com>

Micro-Electromechanical Systems (MEMS):

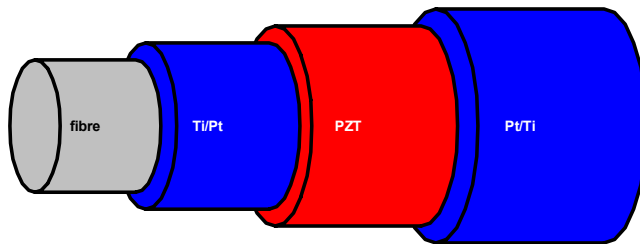
sensors and actuators



<http://www.semiconductor-technology.com>

PZT coated optical fibers used as textile sensors:

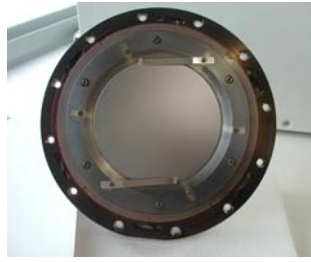
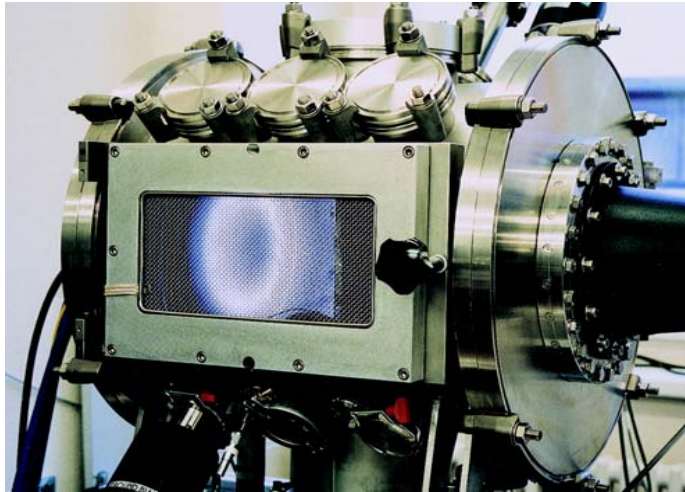
wearable computing, built-in functional textiles



Fox, G.R. et al., J. Vac. Sci. Technol. A **14** (1996) 800

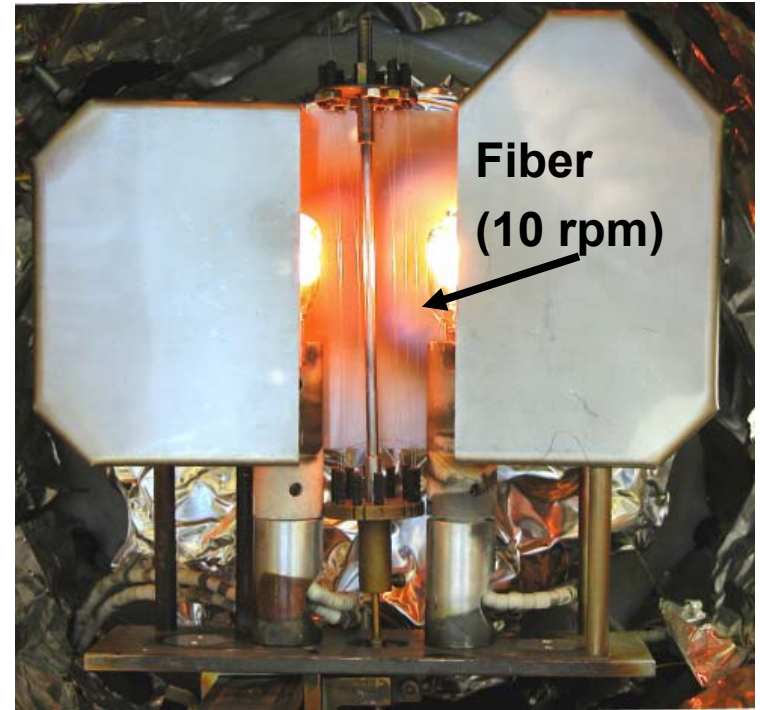
Process parameters

Wafer coating



DC pulsed, 250 W, 35 kHz, 9 μ s,
 pO_2 : 0.45, T_{dep} : 490°C, t_{dep} : 8.5h,
d: 0.9 μ m -> ,as deposited'

Fiber coating & Rapid thermal annealing (RTA)



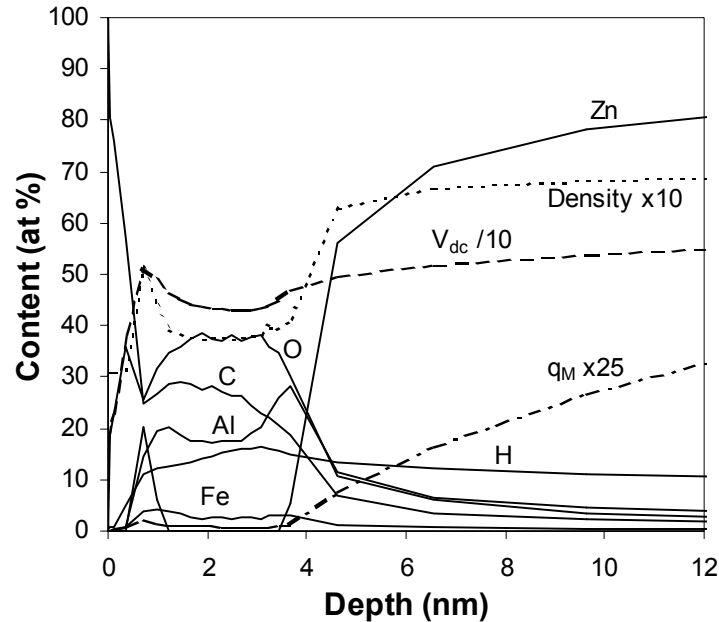
T_{anneal} : 550, 600, 650°C (18°C s⁻¹)
 T_{anneal} : 700°C (23°C s⁻¹)

Glow Discharge-Optical Emission Spectroscopy

GD-OES depth profiling: advantages & drawbacks

Example:

Al_2O_3 thin film

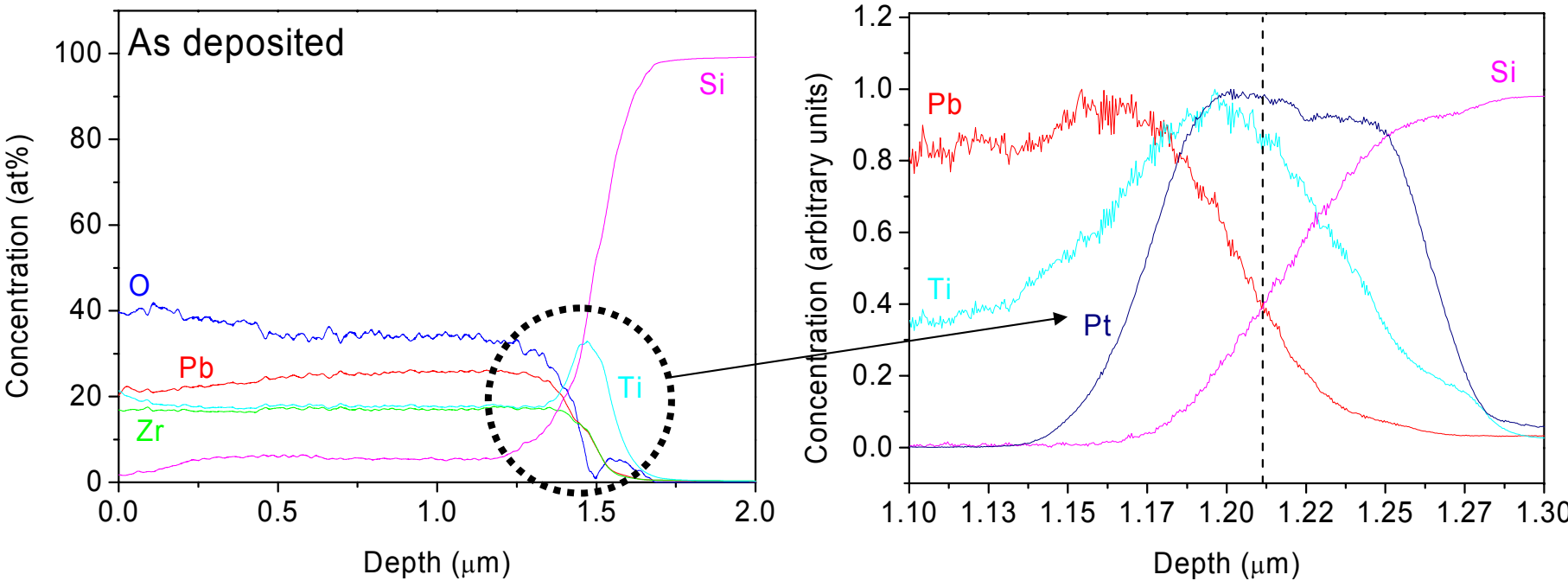


GD-OES:

- Fast ($\mu\text{m}/\text{sec}$) and cheap method for depth profiling
- H can be measured
- Surface sensitivity: down to a few nm
- Destructive
- Calibration samples needed (here: Fe & bulk PZT)

Glow Discharge-Optical Emission Spectroscopy

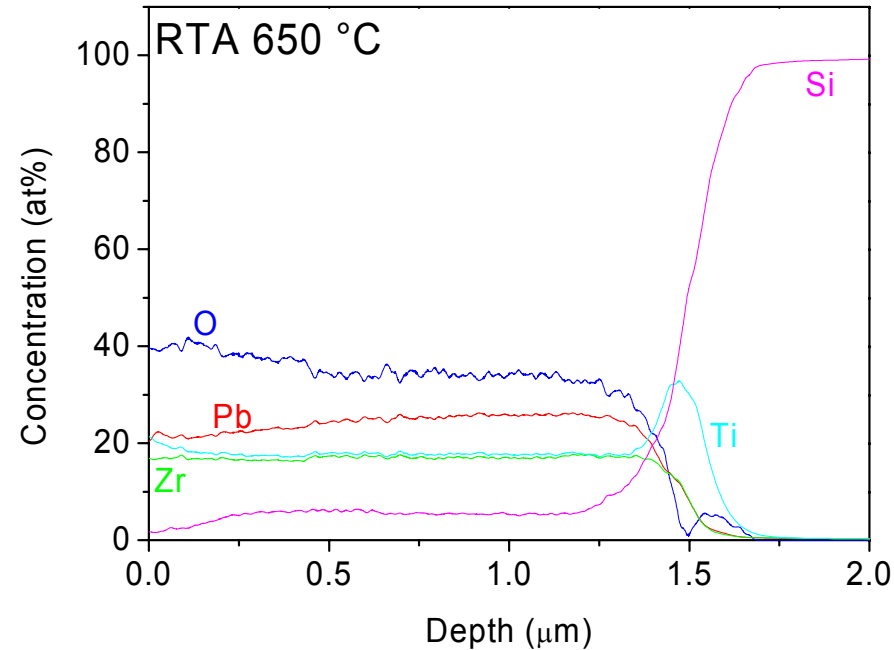
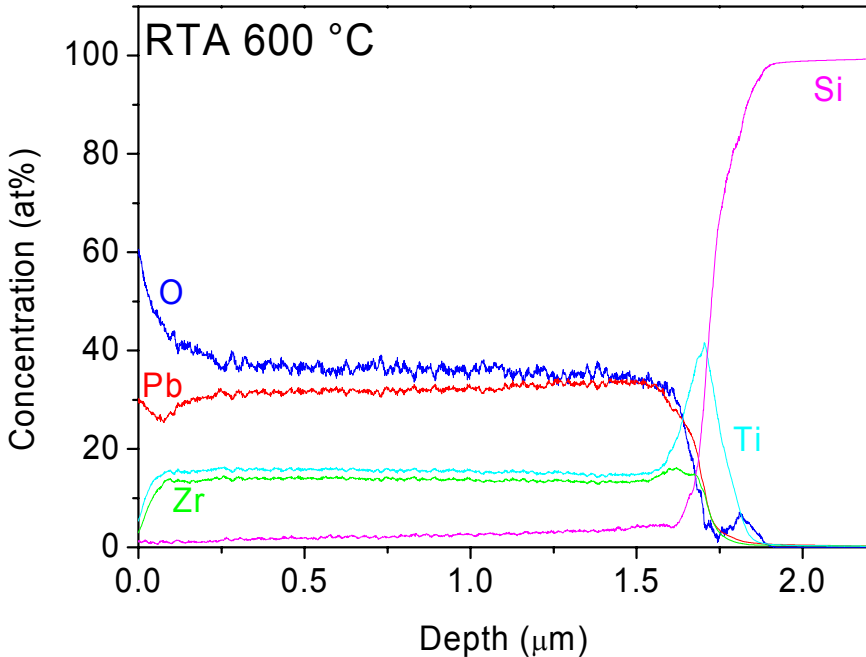
Depth profiling of 'as deposited' PZT samples



Sample	Pb (at%)	Zr (at %)	Ti (at%)	O (at %)
PZT nominal	20	10.4	9.6	60.0
as deposited	16.0	5.6	7.5	43.7

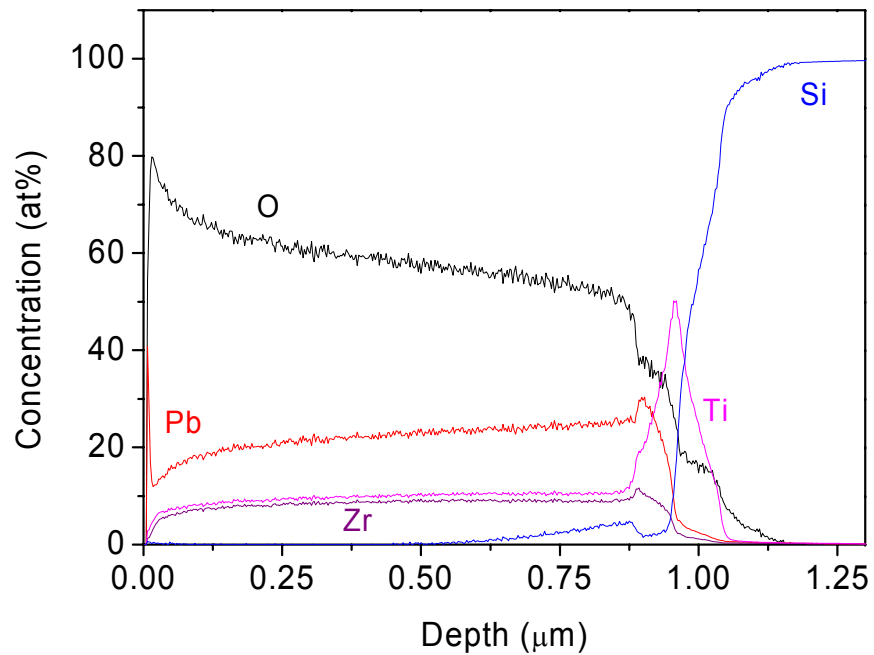
Glow Discharge-Optical Emission Spectroscopy

Depth profiling of 'RTA-treated' PZT samples



Sample	Pb (at%)	Zr (at %)	Ti (at%)	O (at %)
PZT nominal	20	10.4	9.6	60.0
RTA 600 °C	22.3	9.8	12.6	25.8
RTA 650 °C	13.9	10.0	11.8	19.6

Sample Batch2: Quantitative spectra



Sample	Pb (at%)	Zr (at%)	Ti (at%)	O (at%)	Pb/(Zr+Ti)
PZT nominal	20.0	10.4	9.6	60	1.0
PZT low oxygen	31.7	13.5	15.3	37.3	1.1
PZT high oxygen	21.5	8.9	9.4	59.8	1.2

Schlussfolgerungen

- Ein DC gepulster reaktiver Sputterprozess von einem einzelnen metallischen Target mit der Zusammensetzung Pb (55at%), Zr (22.5at%), and Ti (22.5at%) hat sich erfolgreich durchgesetzt.
- Multikristalline PZT Dünnschichten (Perovskite Typ) wurden auf Si-Wafer und auf optische Fasern abgelagert.
- Die GD-OES bewies, dass sie ein effizientes Hilfsmittel zur Bestimmung der Schichtzusammensetzung ist. Die Elementverteilung ist durch die ganze Schicht hindurch uniform. Erste XPS und GD-OES Resultate zeigten einen Sauerstoffmangel an. Deshalb musste ein Sputterprozess mit höherer Sauerstoffflussrate ins Auge gefasst werden.