SPACE RESEARCH & PLANETARY SCIENCES
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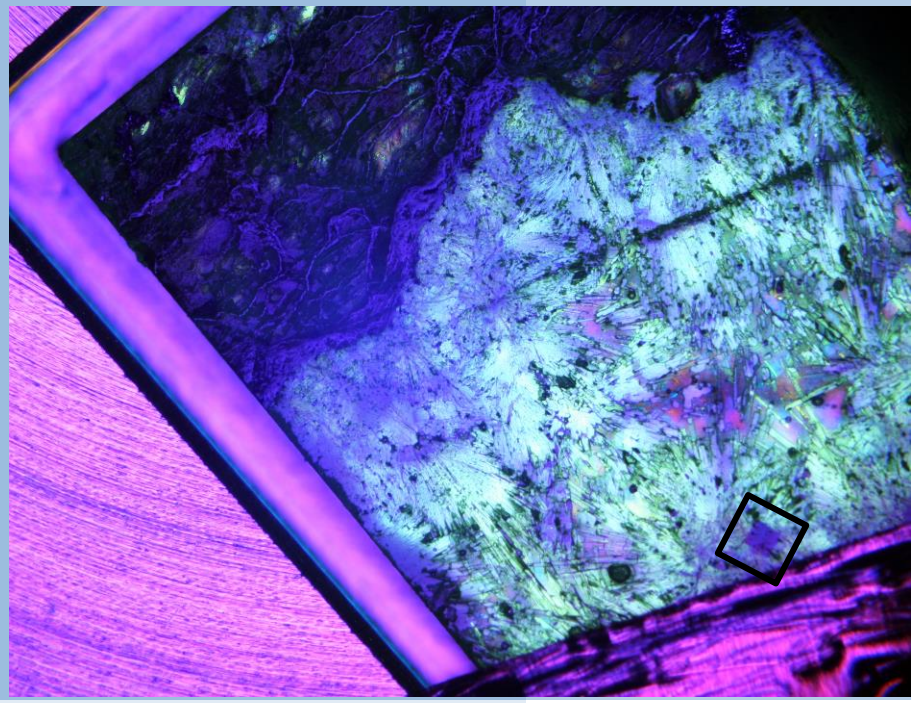
HEMS 2015

Towards detection of life in space exploration missions by using a miniature laser ablation ionization mass spectrometer

Andreas Riedo
P. Moreno-García, V. Grimaudo, M.B. Neuland, M. Tulej, P. Broekmann and P. Wurz

Physics Institute, Space Research and Planetary Sciences University of Bern, Switzerland

Department Chemistry and Biochemistry, Interfacial Electrochemistry Group, University of Bern, Switzerland



Introduction

Space research and Planetary Sciences

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» **In-situ measurements on extra terrestrial material**

(chemical composition on-site, isotope analysis, bio-signatures)

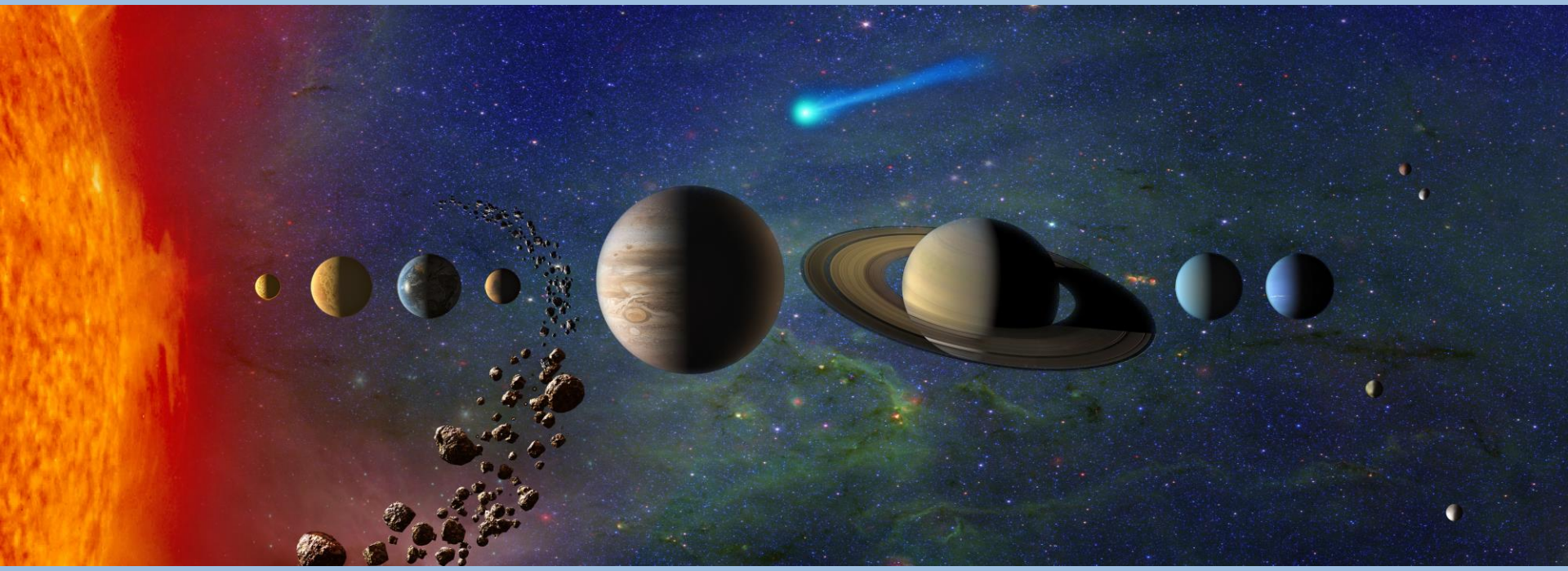

Image credit: NASA

Introduction
Bio-Signatures in Space Research

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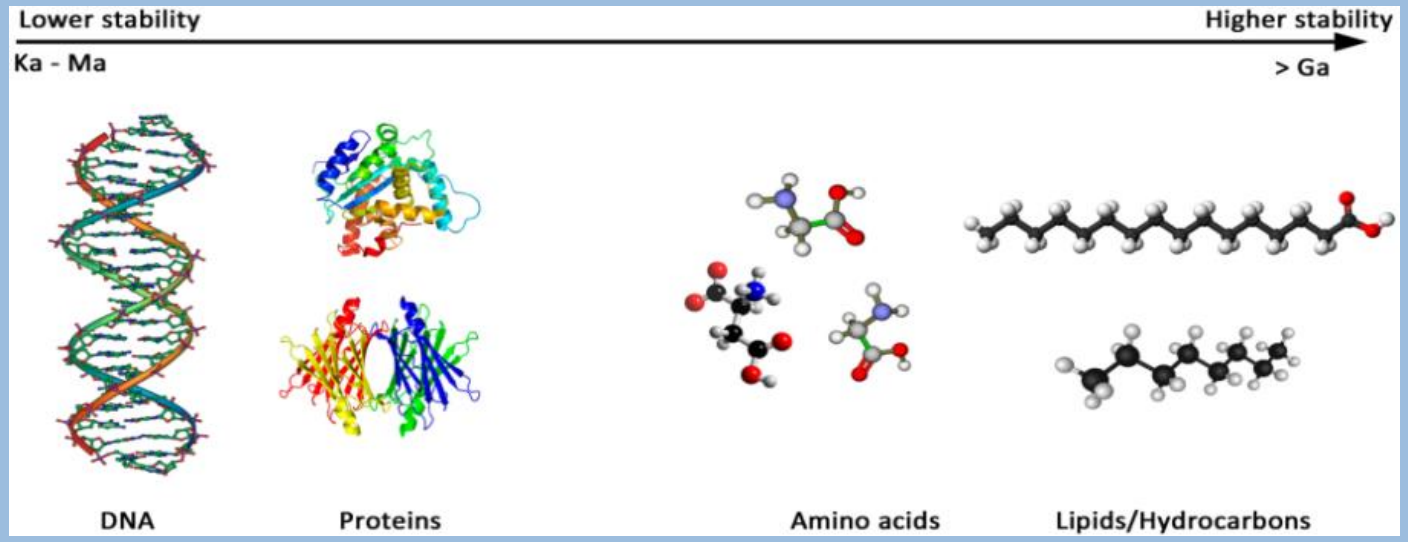
» **Various bio-signatures exists**
(e.g. DNA, proteins, amino acids, lipids/hydrocarbons, fractionated elemental composition within solid material)

Image credit: NASA

Introduction
Bio-Signatures in Space Research

» **Life time of bio-markers depends on environment!**
(temperature, UV and ionizing radiation, etc.)



J.W. Aerts, W.F.M Röling, A. Elsaesser and P. Ehrenfreund, Life, 4, 535 – 565, 2014

» Sub-surface environment is of high interest!

Introduction
Current and Future Space Mission

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- Need for advanced instruments capable of the detection of bio-signatures at mircometre scale
- Simple(er) sample preparation and measurement procedures
- Searches for geochemical signature:
  - habitable environments
  - chemical composition indicative of biology or metabolic processes
- Microorganism of interest: endoliths and epiliths
  - Well preserved, embedded in vein or vesicle filling mineral phases e.g., carbonates or quartz

Laser Mass Spectrometer – LMS
Capabilities (in respect of Astrobiology)

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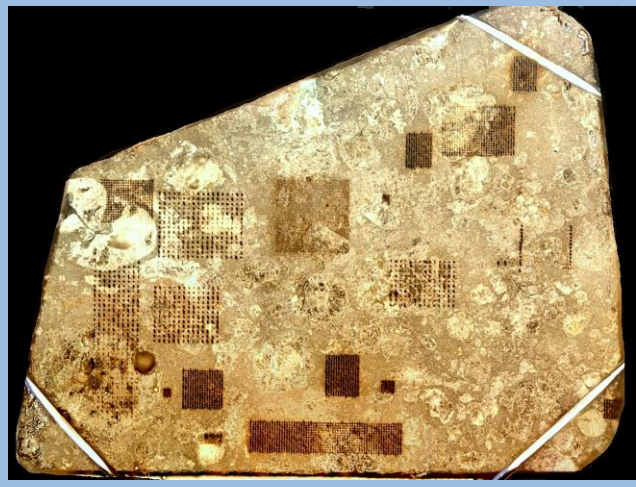
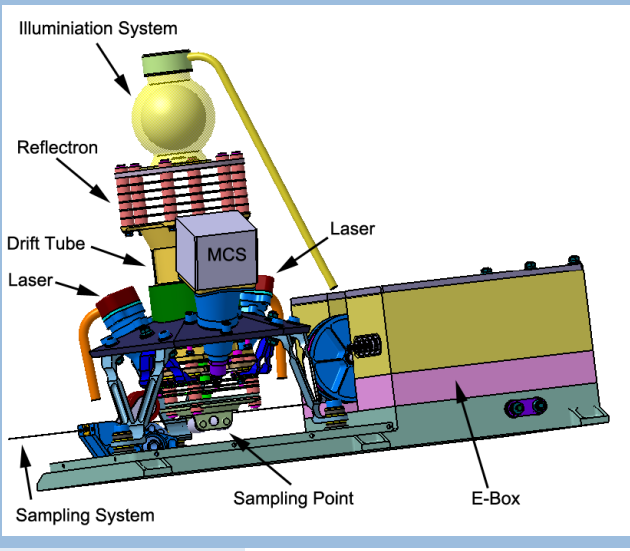
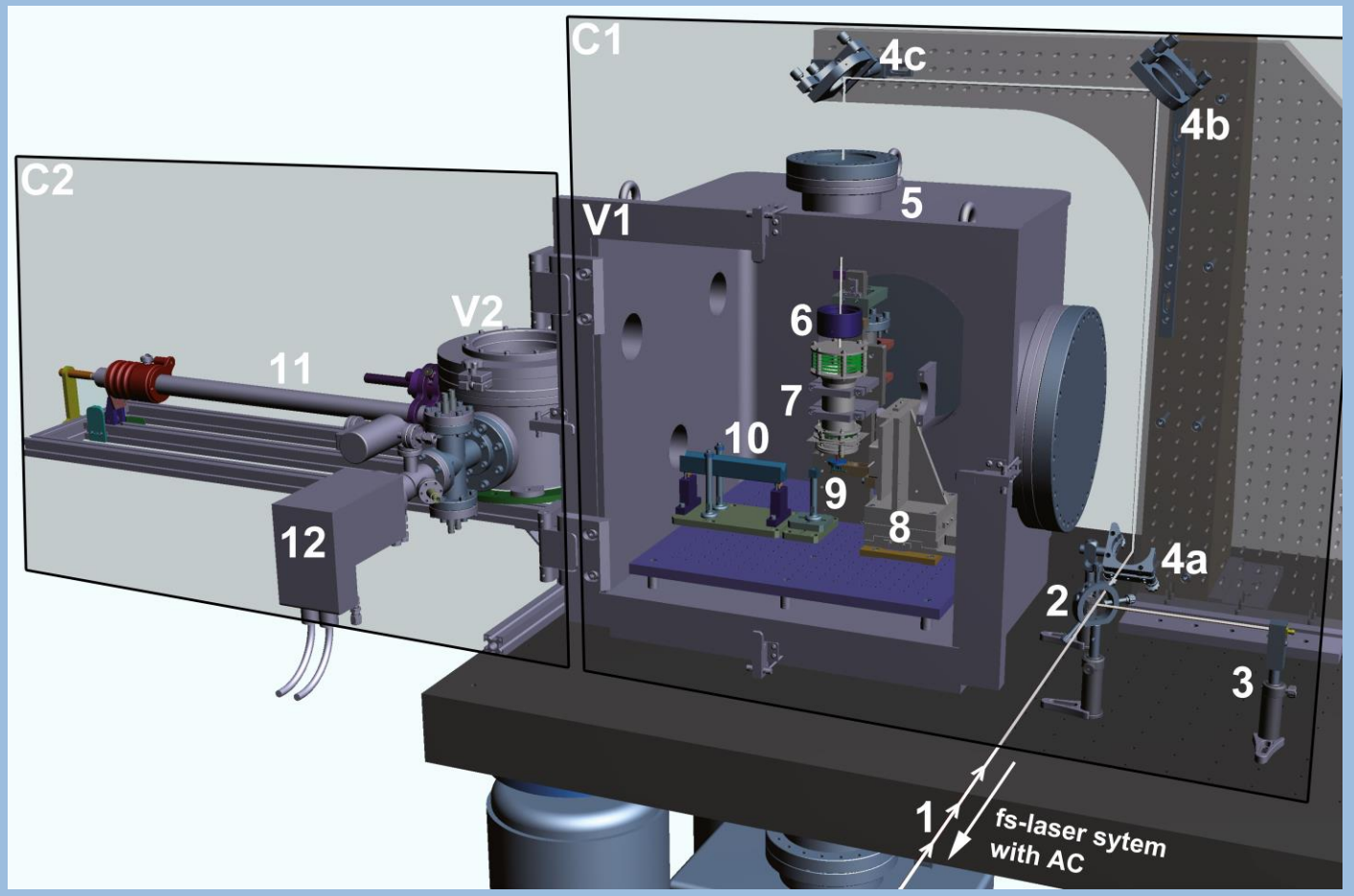
> **Quantitative elemental and isotope analysis (ablation mode)**

- Elements: host and fossil composition
- Isotope: possible fractionation processes (*Riedo et al., J. Mass Spectrom., 2013, Riedo et al., J. Anal. At. Spectrom., 2013, Riedo et al., Planet Space. Sci., 2013*)
- Chemical maps of heterogeneous material surfaces (*Neuland et al., Planet. Space Sci., 2014*)
- Depth profiling: quantitative analysis of redistribution of elements across the surface thickness with nm-resolution (*Grimaudo et al., Anal. Chem., 2015*)
- LMS combined with optical microscopy (CAMAM):surface and microstructure morphology, texture *(Tulej et al., Geostand. Geoanal. Res., 2014)*



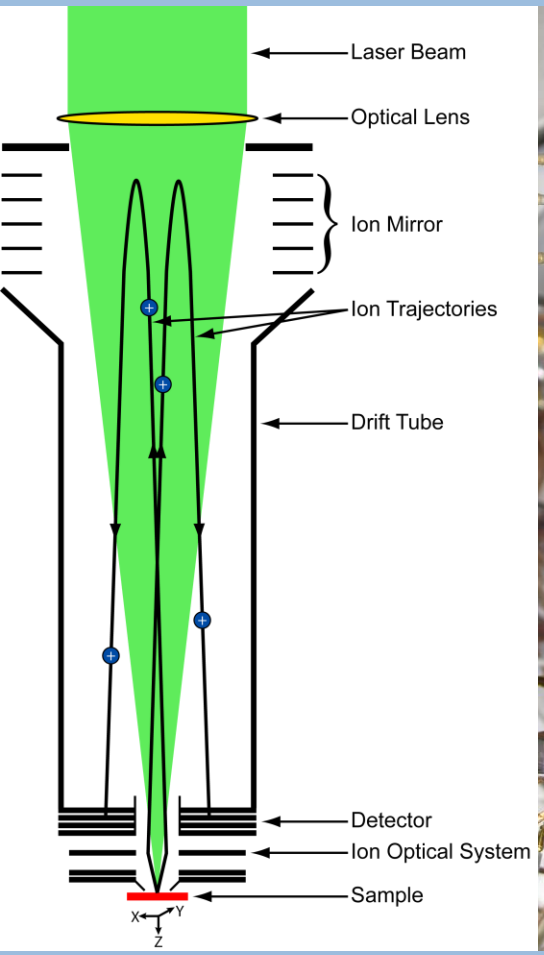


Laser Mass Spectrometer – LMS
Instrumental Setup for R&D

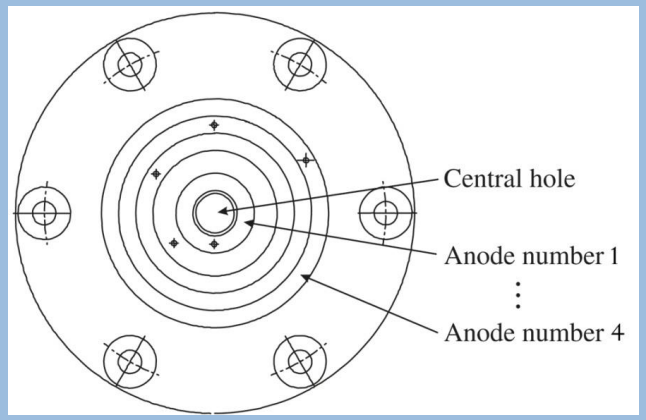


Riedo et al., J. Anal. At. Spectrom., 2013

Laser Mass Spectrometer – LMS
Instrument Design & Principle of Operation



- Dimensions of 160mm x Ø 60mm, ~2kV, ~2kg, ~15W (flight design)
- Ion Trajectory Simulations by SIMION
- Ring anode detector
- 2x 8-bit high speed digitizer with on-board processing ADC cards, each with 2 channels



Rohner et al., Meas. Sci. Technol., 2003; Riedo et al., J. Mass Spectrom., 2013

Laser Mass Spectrometer – LMS
Laser Ablation Ion Source & Mass Analyzer Characteristics

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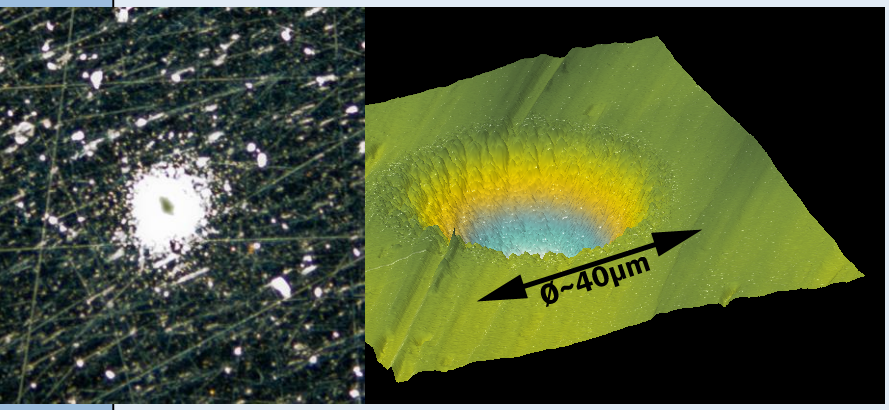
» **Ion Source:** pulsed laser system

fs-laser system

— **IR (775nm)**
~190fs pulse width
≤ 1kHz repetition rate
~5-40µm crater diameter
< 5TW/cm^2



R-TOF characteristics

» Mass calibration: $m(t) = k_0(t-t_0)^2$

» Spectra collected within ~13 µs

» Mass resolution: 500-1000
(desorption mode ~≤ 1500)

» Dynamic range ≥ 10^8

» High detection sensitivity down the 10ppb

Riedo et al., J. Mass Spectrom., 2013,
Riedo et al., J. Anal. At. Spectrom., 2013

Research in Semiconductor Industry
Case Study for Depth Profiling using LMS

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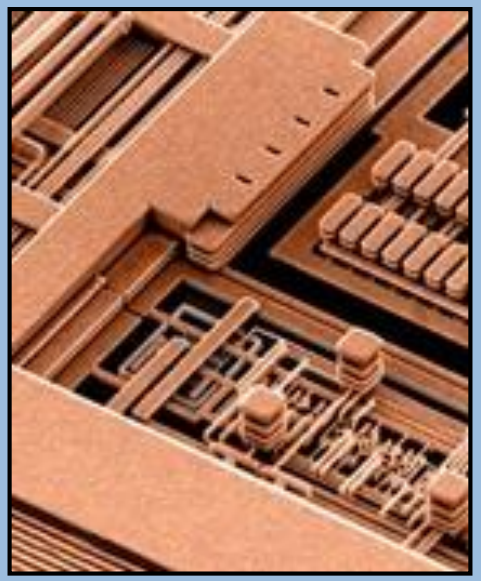
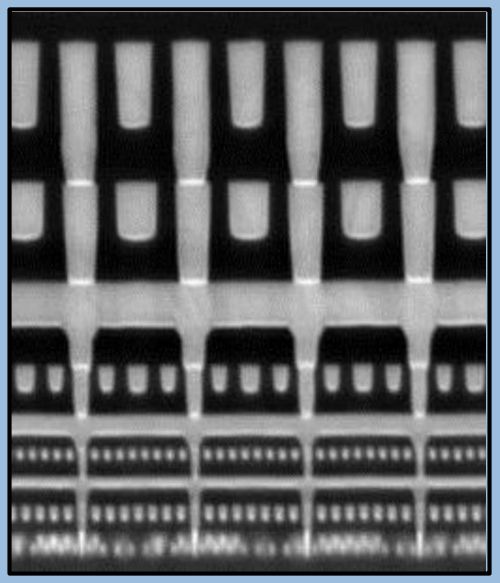
top view



side view



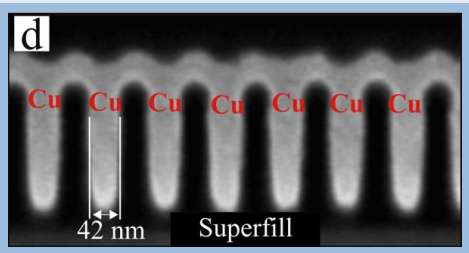
Why copper ?

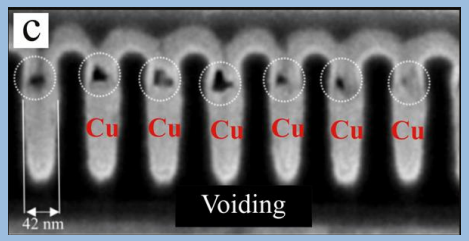
- ✓ Lower resistance
- ✓ Higher allowed current density
- ✓ Increased scalability

>1 billion interconnects on a chip

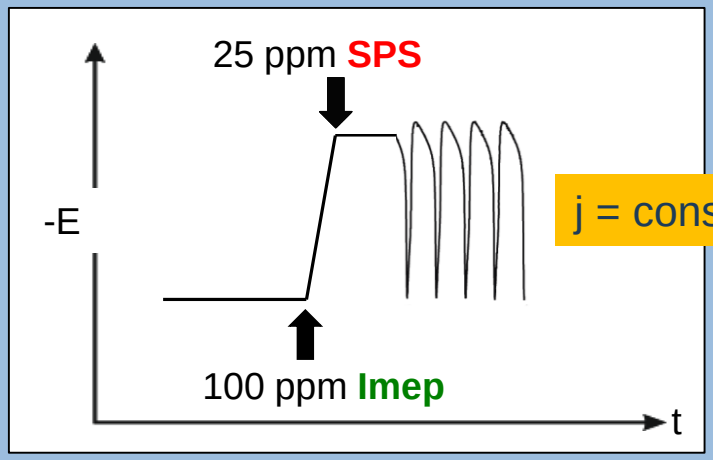
10 nm features

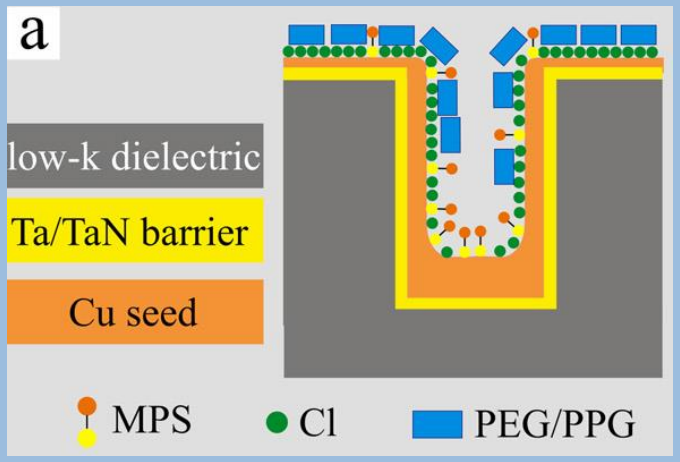
Research in Semiconductor Industry
Super conformal Fill

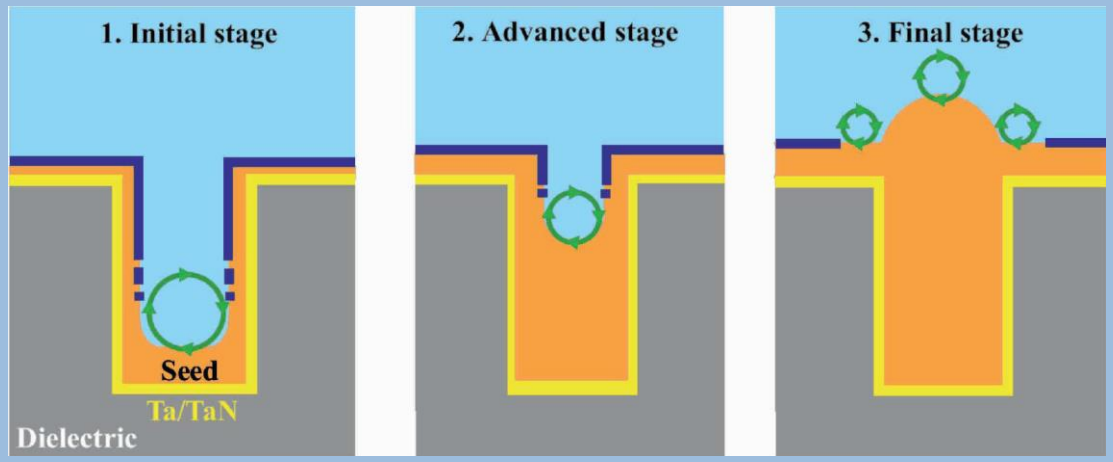




Galvanostatic copper deposition:







P. Broekmann et al., Electrochim. Acta, 2011; T. P. Moffat et al., IBM J. Res. & Dev. 2005; R. Akolkar et al., J. Electrochem. Soc. 2011

Research in Semiconductor Industry
Classical procedure

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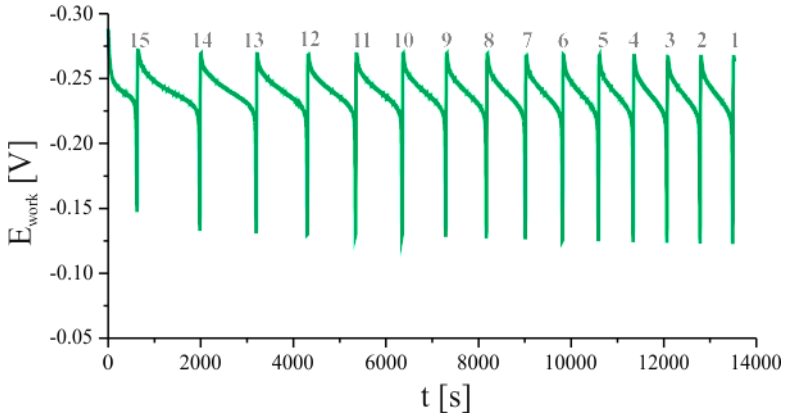
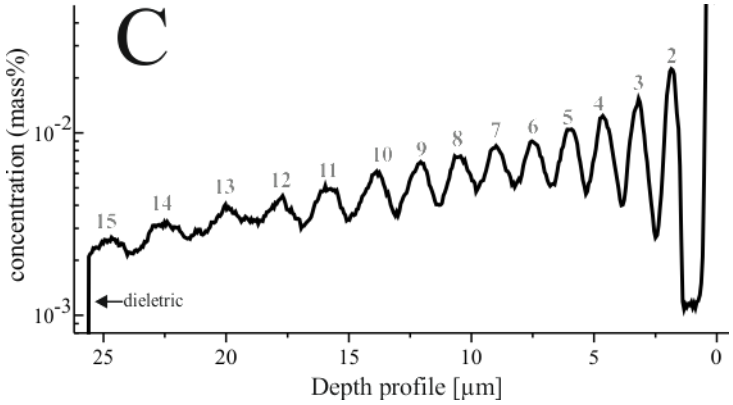
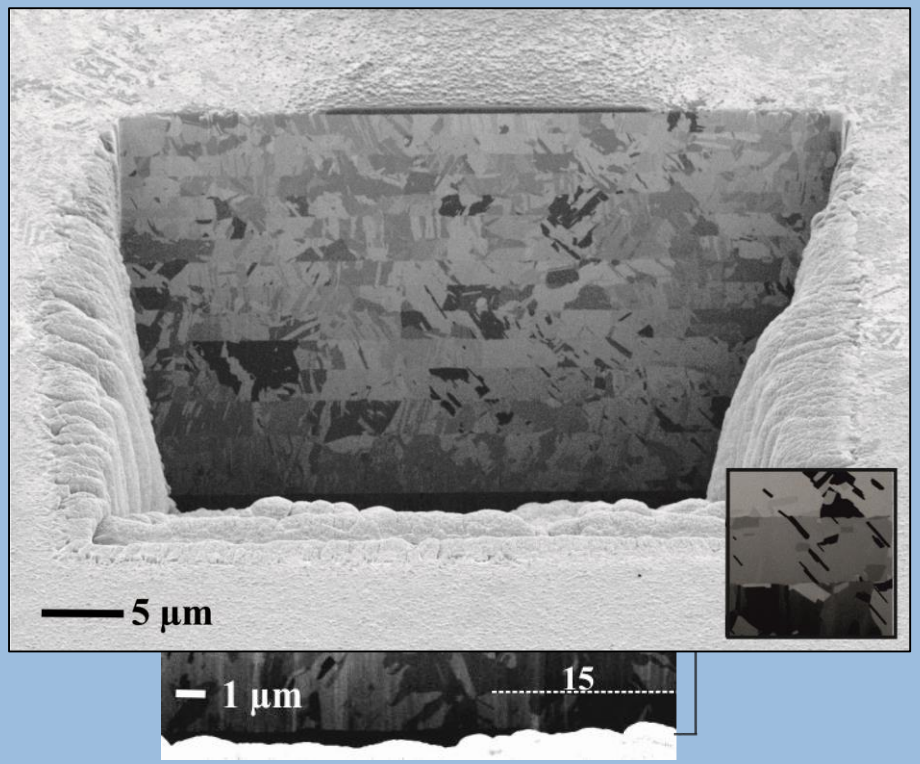
Potential transient



Secondary Ion Mass Spectrometry (SIMS)



Focused Ion Beam (FIB) - cut



N.T.M. Hai, J. Odermatt, V. Grimaudo, K.W. Krämer, A. Fluegel, M. Arnold, D. Mayer, and Peter Broekmann, ***J. Phys. Chem. C 116 (2012) 6913***

P. Wurz et al., Conference Proceedings of Symposium on Surface Science, Sputtering of clean and oxidized Cr and Ta Metal Targets using SNMS and SIMS, (1990) 181-185

Depth Profiling using LMS
Depth Resolution – Mean Ablation Rate

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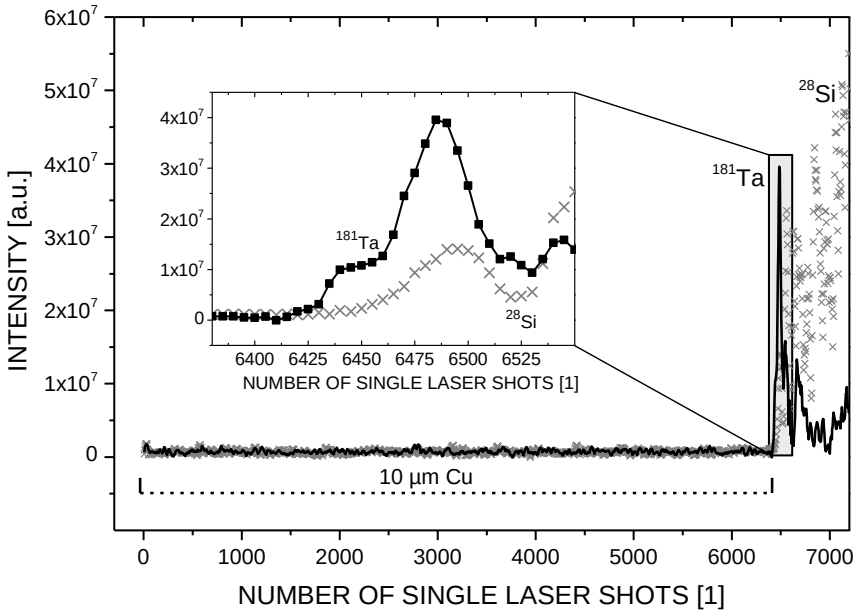
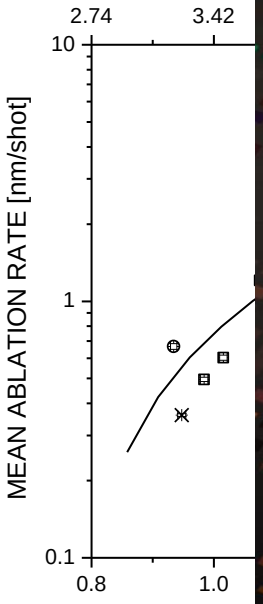
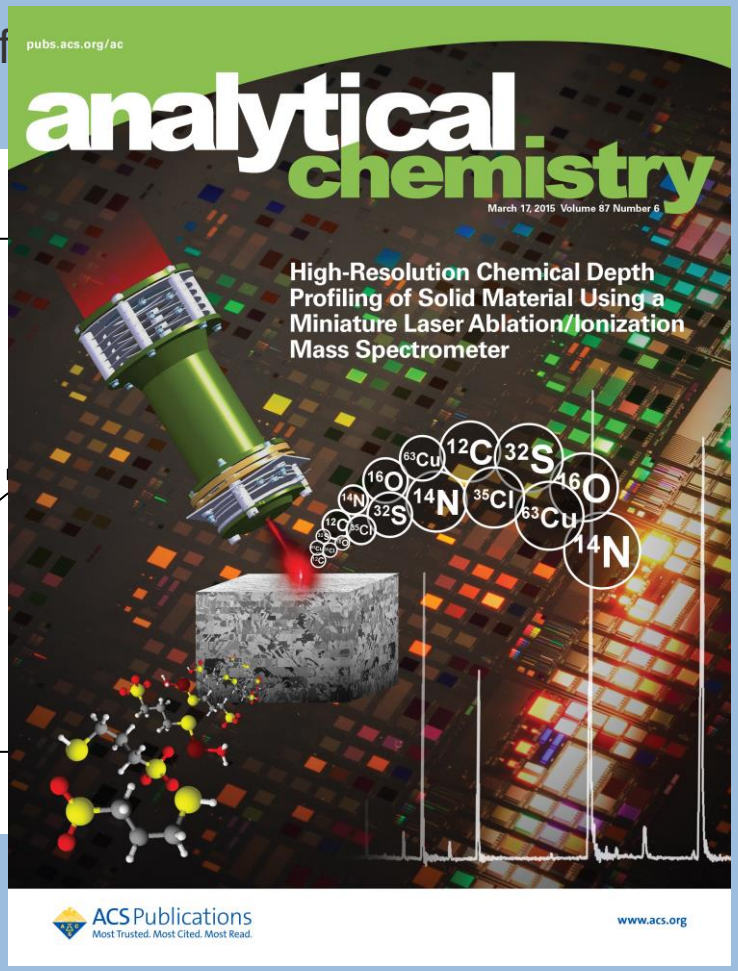
» Investigations on different Cu samples with def
substrate







V. Grimaudo et al., Anal. Chem., 87, 2037–2041, 2015

Depth Profiling using LMS
Depth Resolution – Multi Layer Samples

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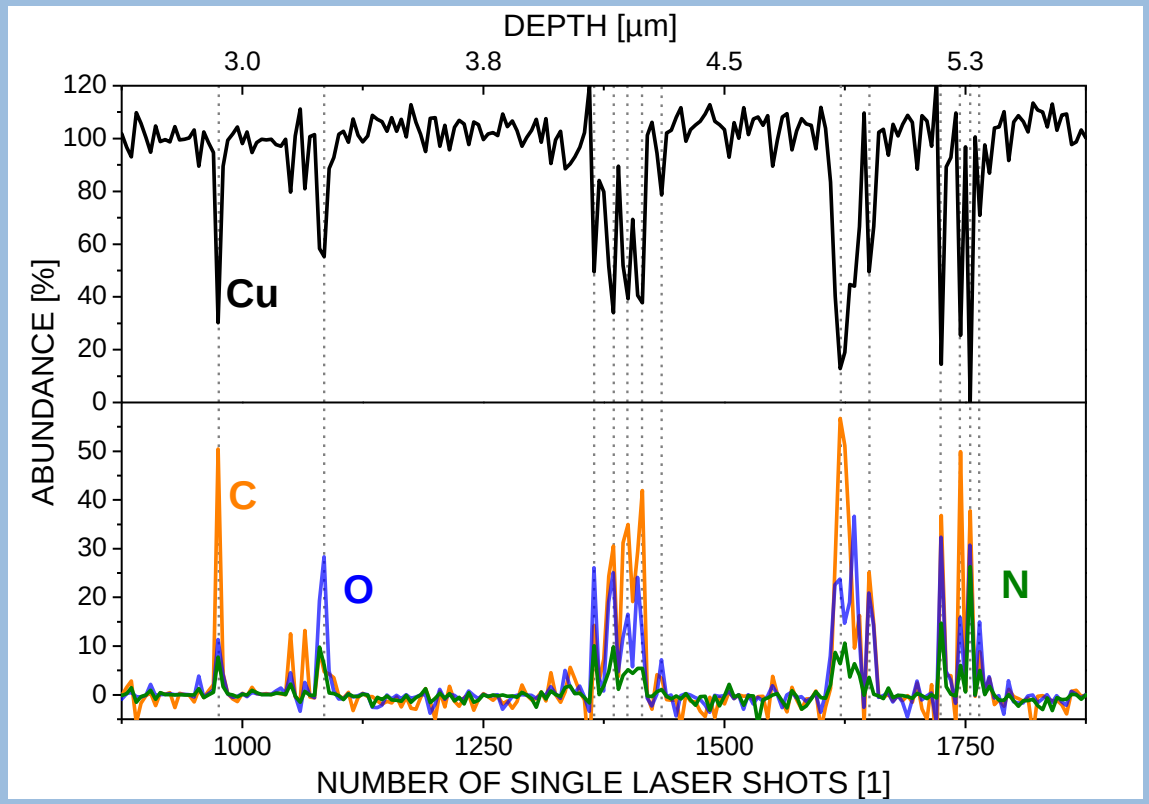
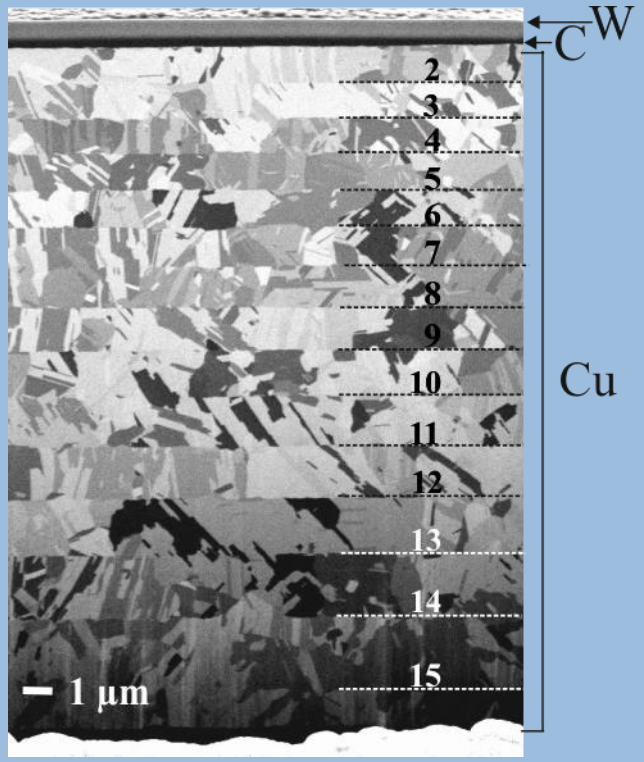
Anti-correlation of Cu and contaminants





V. Grimaudo et al., Anal. Chem., 87, 2037–2041, 2015

Depth Profiling using LMS
Depth Resolution – Multi Layer Samples

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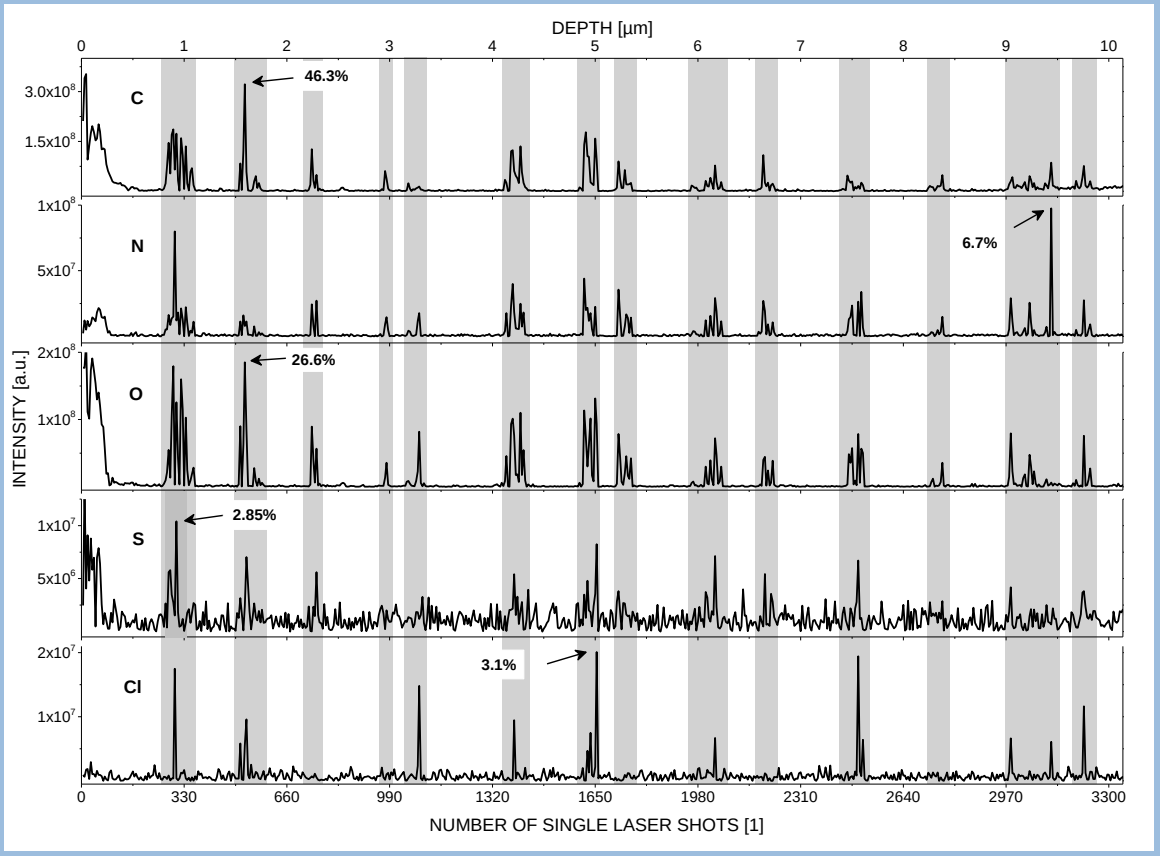
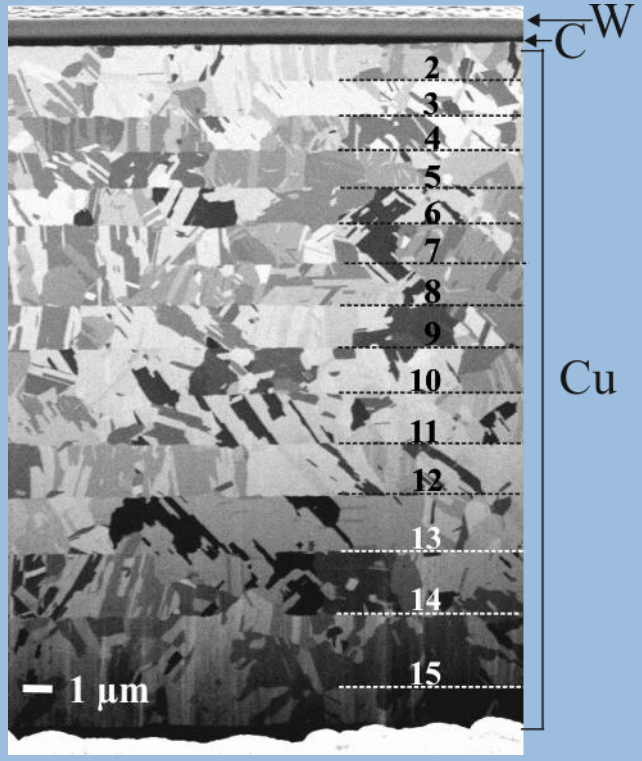
Oscillation of C, O, N in Phase

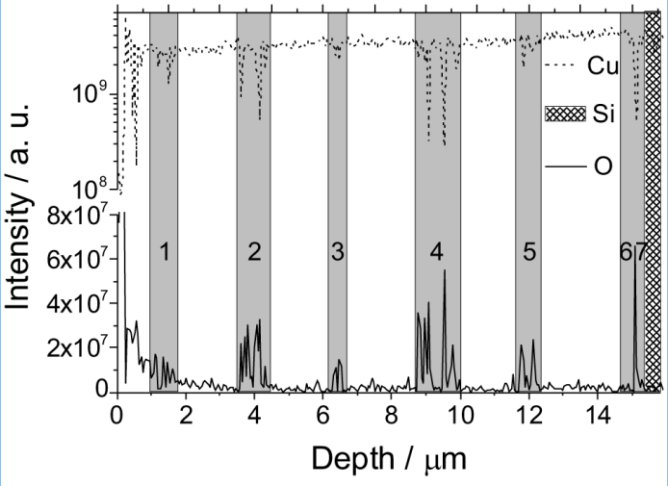


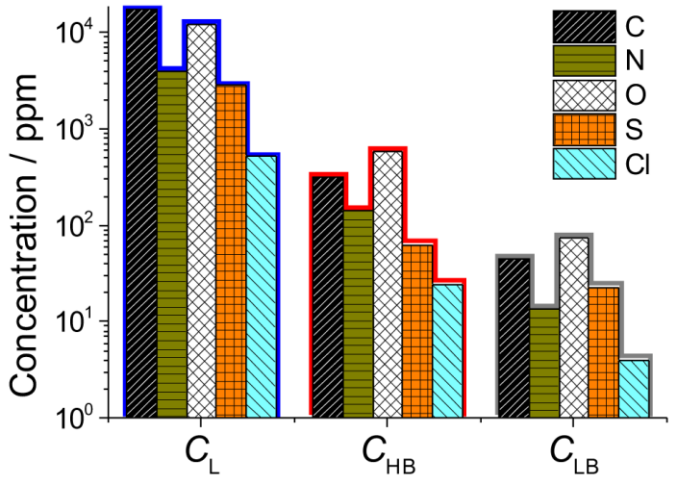


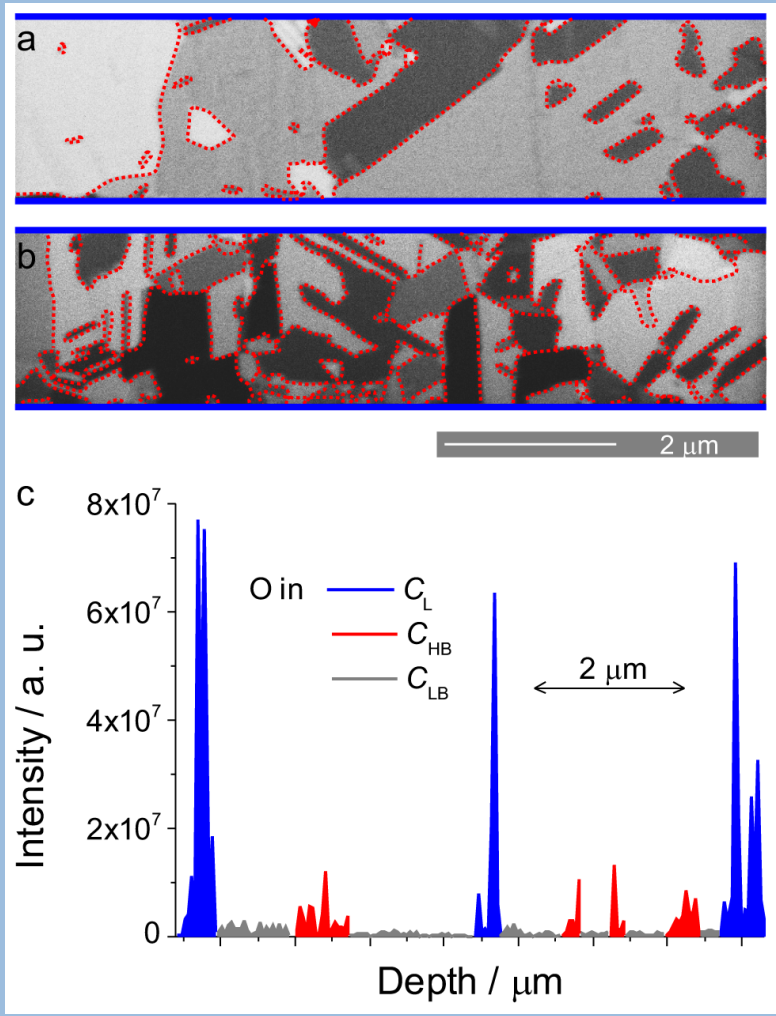
V. Grimaudo et al., Anal. Chem., 87, 2037–2041, 2015

Current Studies - Semiconductor Industry

Incorporation studies between transition layers (Fundamental)



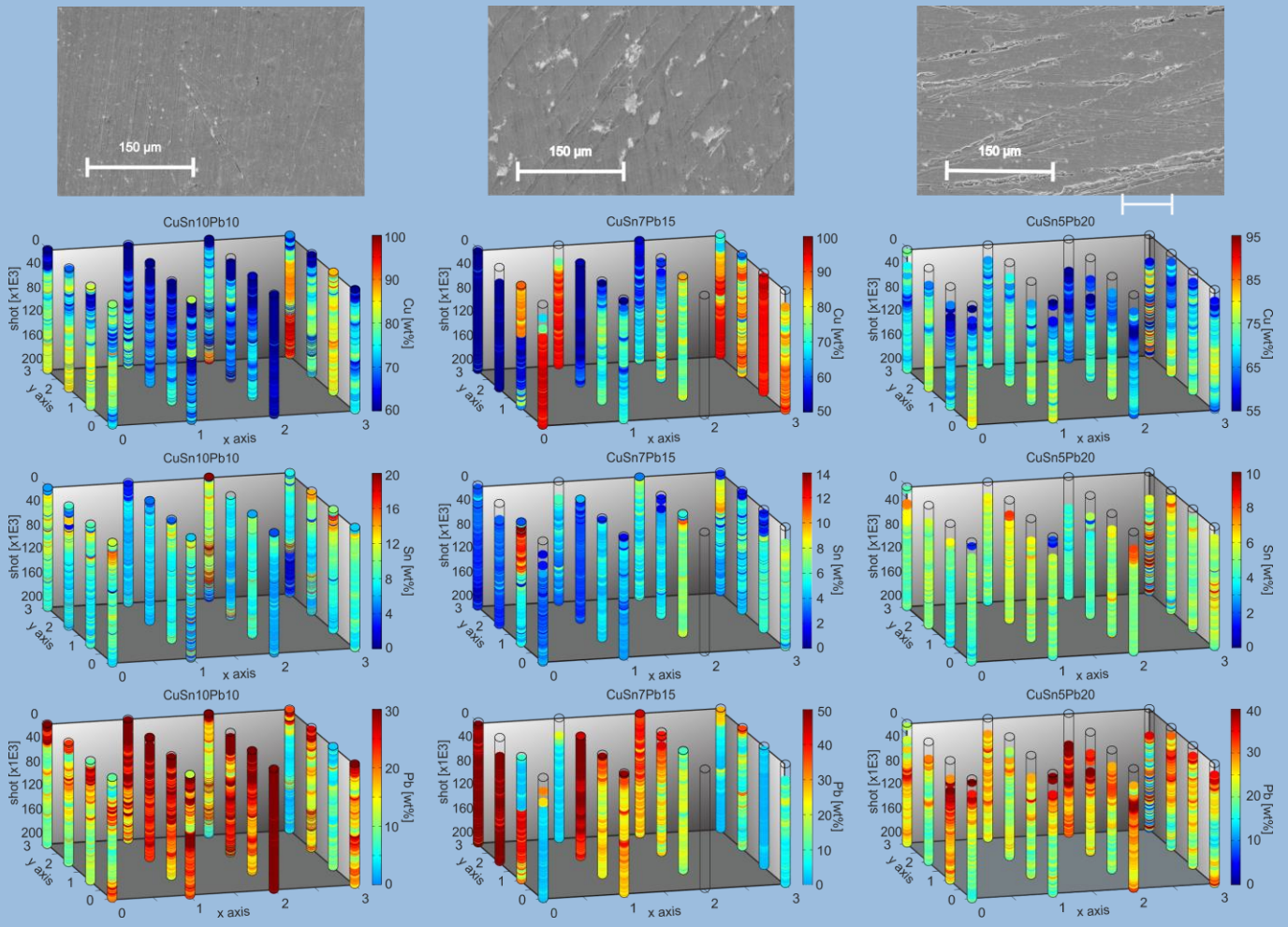


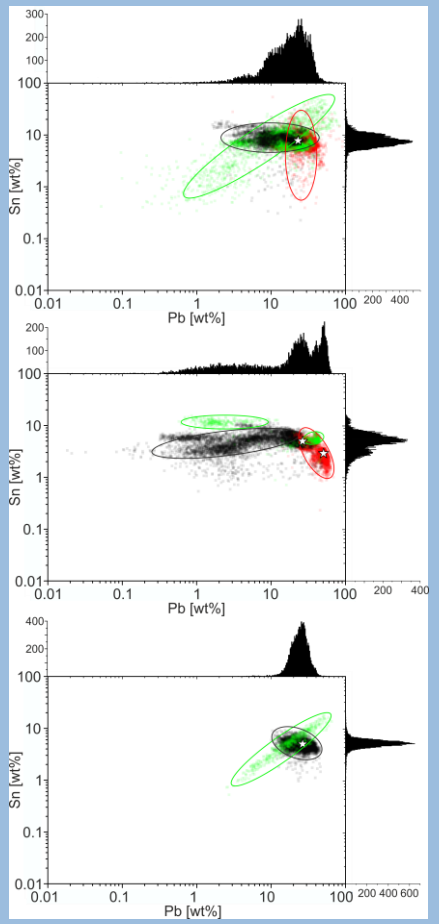


P. Moreno-García et al., Electrochimica Acta, 2015, submitted

Current Studies - Pharmaceutical Industry

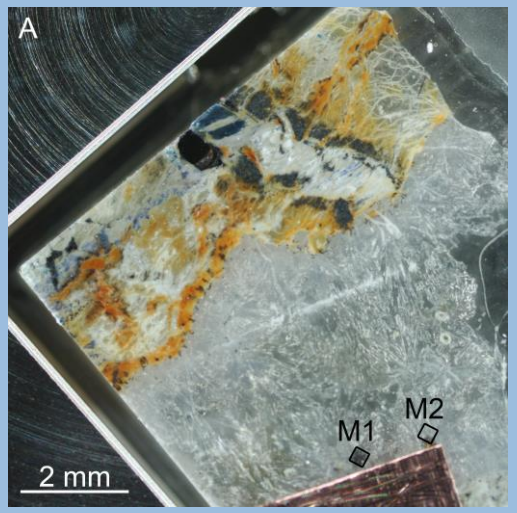
3D chemical analysis of alloy samples (Application)

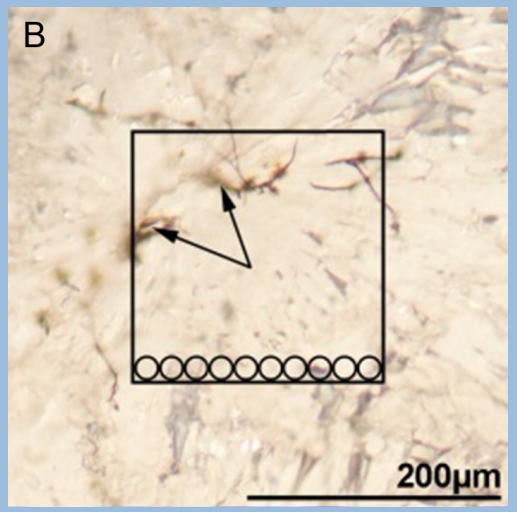


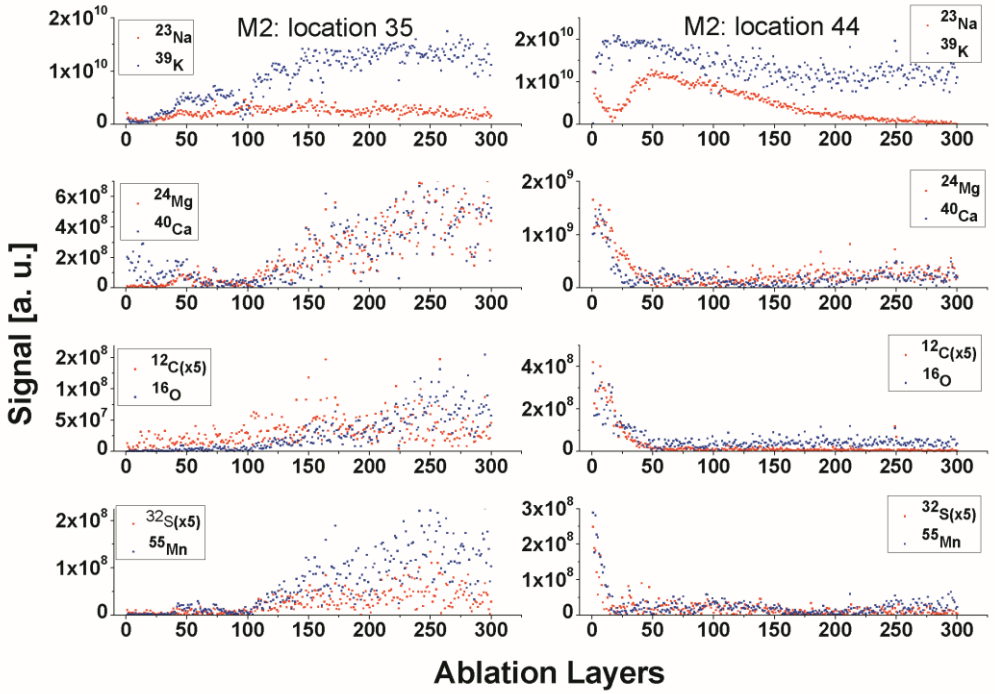


V. Grimaudo et al., Anal. Chem., 2015, to be submitted.

Current Studies – Astrobiology
Chemical Analysis of micro-sized Fossil (HIGHLIGHT)







M. Tulej et al., Astrobiol., 15, 669–682, 2015

Conclusions and Outlook

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- **Figure of merit of current LMS:**
  - High dynamic range of at least eight orders of magnitude
  - Detection sensitivity down to few ppb's
  - Mass resolution (m/Δm) in the range of 500 – 1000
  - Quantitative elemental and isotope analysis
  - Quantitative depth profiling -> 3D elemental imaging
- **Applications:**
  - Studies of heterogeneous materials with high spatial resolution
  - Quantitative elemental analysis of microstructures down to the trace element level (sub-ppm)
  - Element correlation studies (insight to host and sample mineralogy)
- **Outlook:**
  - fs system operated at UV radiation
  - Increase of lateral resolution by implementation of new optical system

Q & A

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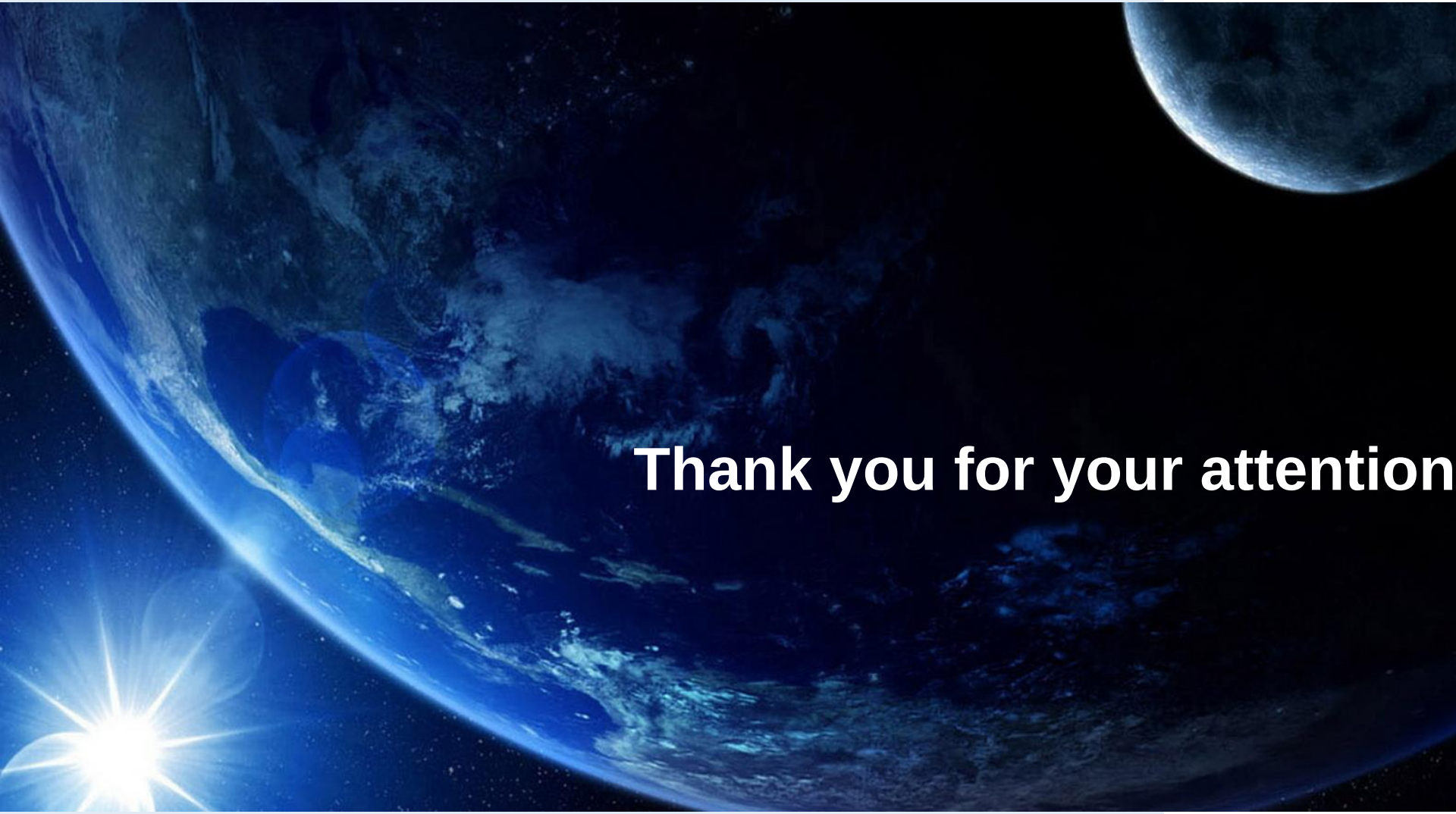
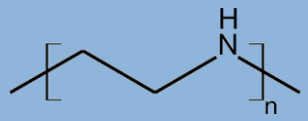


Additives

PEG

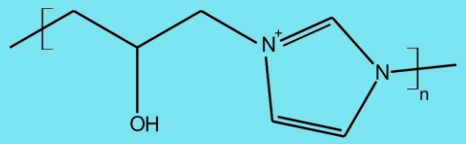
Polyethylene glycol

PEI



Polyethyleneimine

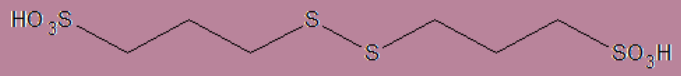
Imep



Polymerizates of imidazole and epichlorohydrin

Additives

SPS



Bis-(sodium-sulfopropyl)-disulfide

P. Broekmann et al., Electrochim. Acta, 56 (2011) 4724-4734; T. P. Moffat et al., IBM J. Res. & Dev. 49 (2005) 19-36; R. Akolkar et al., J. Electrochem. Soc. 151 (2011) C702-C711