ELEMENT GD: Applications overview

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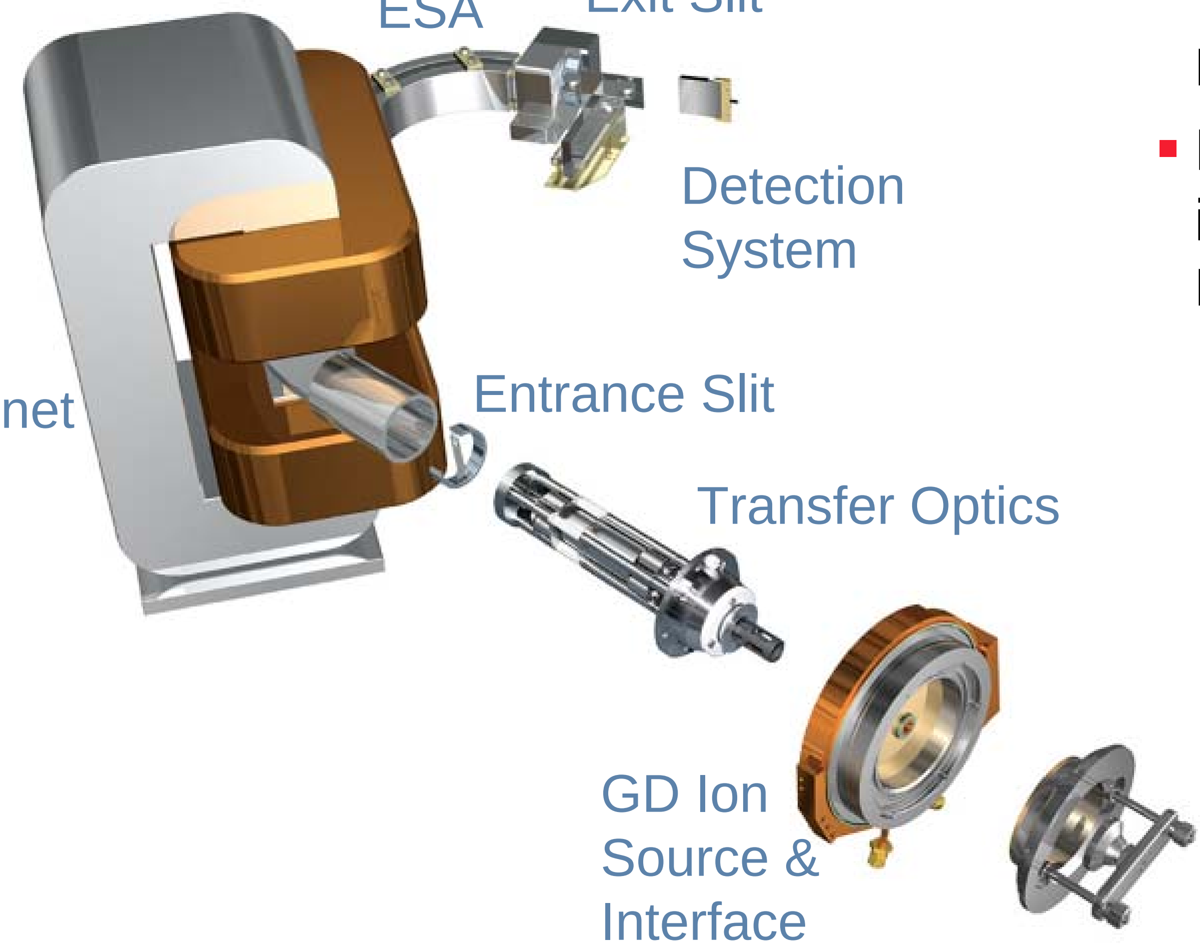
Outline

- Introduction ELEMENT GD
- Application examples
- Current investigations
- Conclusions

Components of the Element GD

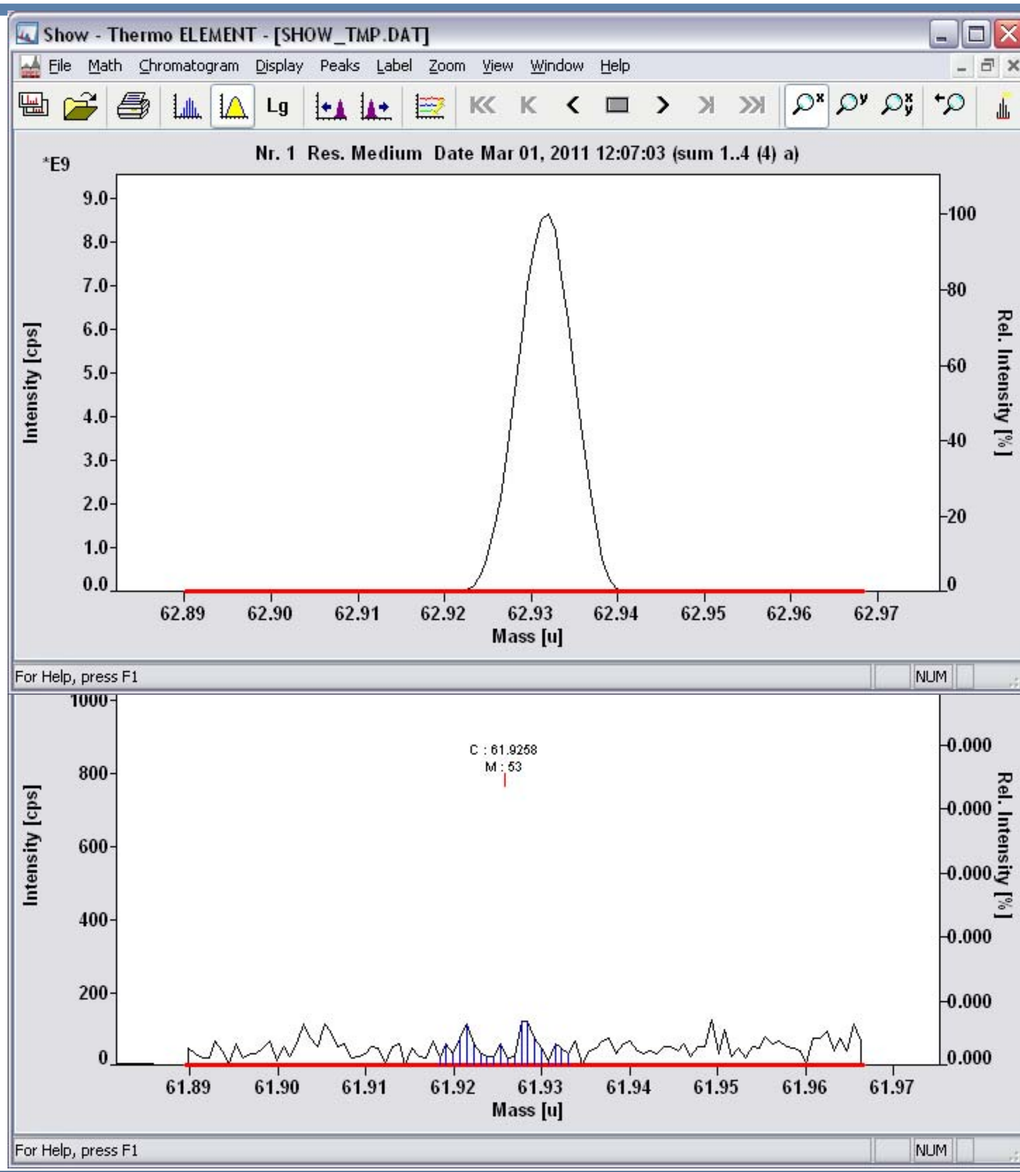


- Similar to 'Grimm Type' DC source:
  Pump down in 10s
- High power, fast flow ion source:
  High sensitivity

Abundance Sensitivity

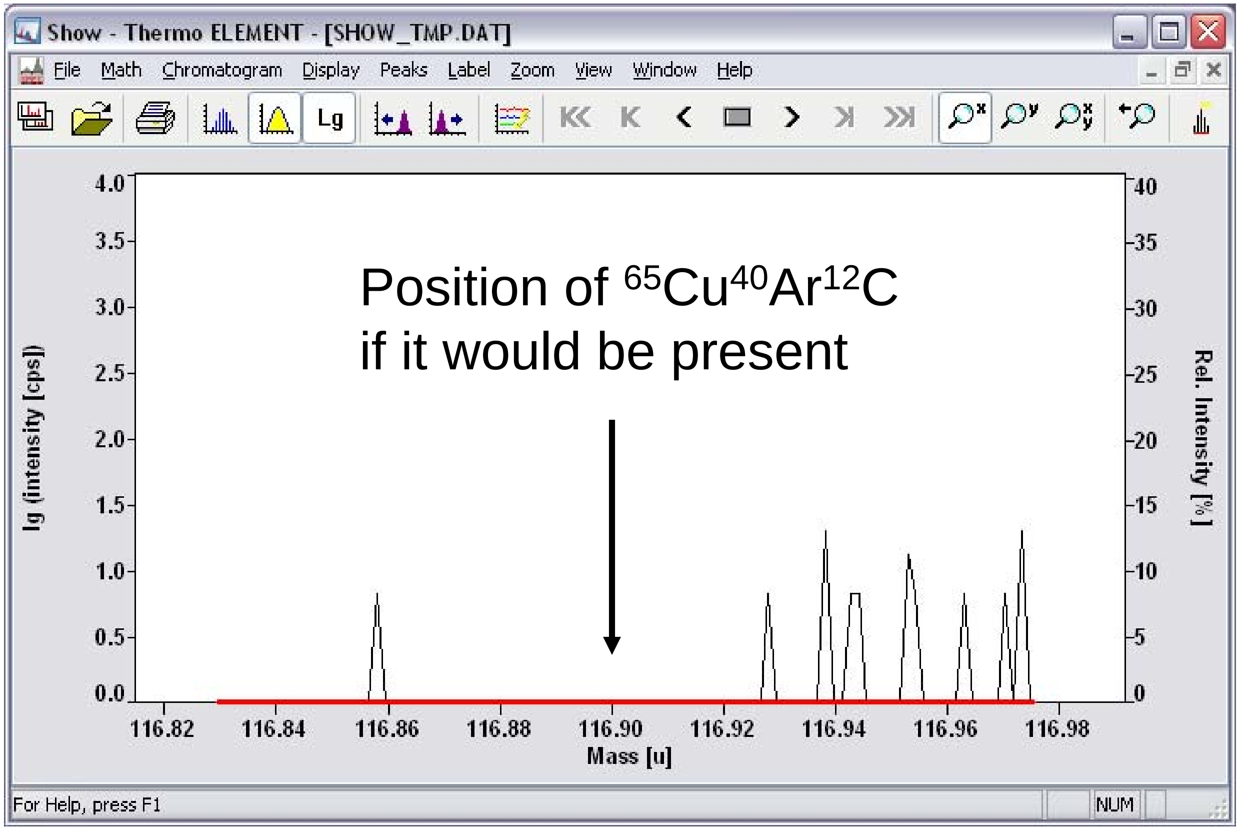
ELEMENT GD:
~ 50 cps on 62
~ 9 x 10^9 cps on 63

abundance sensitivity on m-1 is ca. 6 ppb



Interferences: trimers without cryo-cooling

Example for possible trimeric interference: CuArC on mass 117

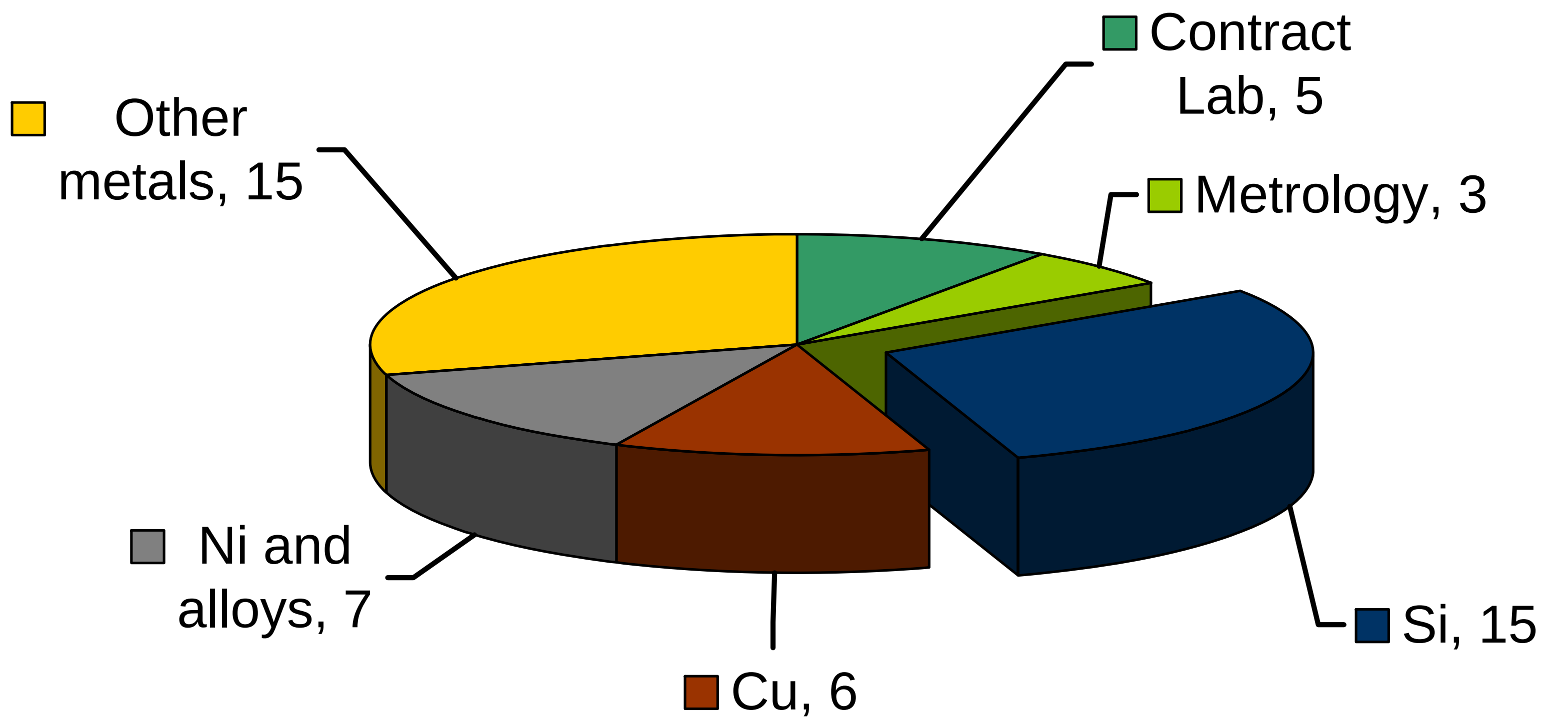


Default 15 ˚C Peltier setting

By design, the fast flow source gives low levels of interferences

Maximum trimeric interference expected is $^{40}Ar_3^+$: ~3000cps (MR) compared to 1 x 10^{10} cps for the matrix

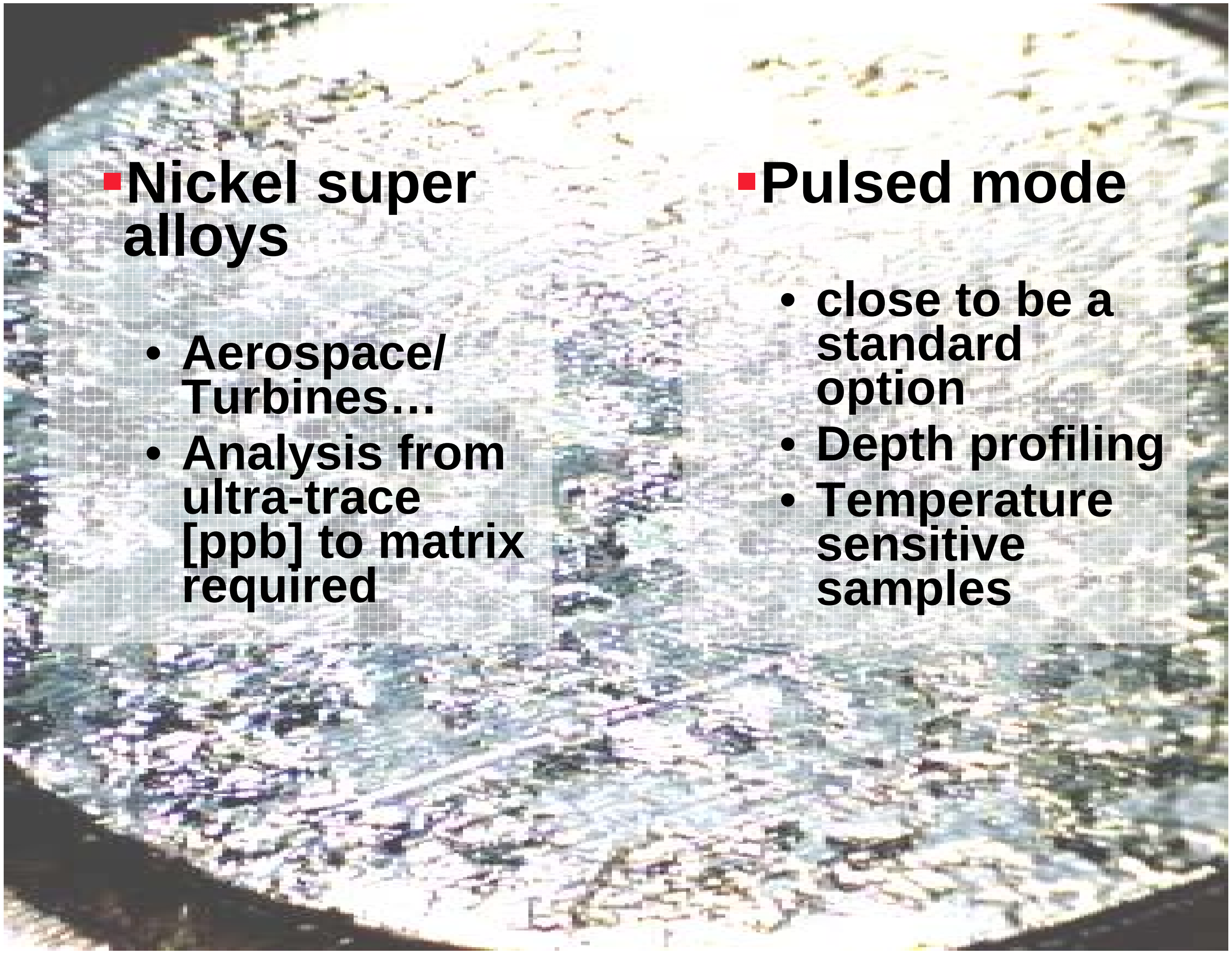
ELEMENT GD by application (2012)



> 50 ELEMENT GD by 2012

Based on technology of ELEMENT ICP-MS with >550 units

Application examples



- **Nickel super alloys**
  - **Aerospace/ Turbines…**
  - **Analysis from ultra-trace [ppb] to matrix required**

- **Pulsed mode**
  - **close to be a standard option**
  - **Depth profiling**
  - **Temperature sensitive samples**

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Ni Alloys

Challenges

- Reliable and routine determination from matrix to ultratrace elements
- Most important: soft metals at ppm/sub-ppm level strongly deteriorate alloy quality (e.g. Bi < 0.3ppm)
- Low level Sulfur

Low Level S analysis in Ni alloys

Sulfur conc. [ppm]

Spot	1	2	3	AVG	STD	RSD
Date	Mar 04 2011	Mar 18 2011	Mar 22 2011	n=3 spots	n=3 spots	n=3 spots
Sample 1	**0.060**	**0.058**	**0.065**	**0.061**	**0.003**	**5.7%**
Sample 2	0.348	0.343	0.355	0.35	0.01	1.7%
Sample 3	4.03	3.93	3.90	4.0	0.1	1.7%

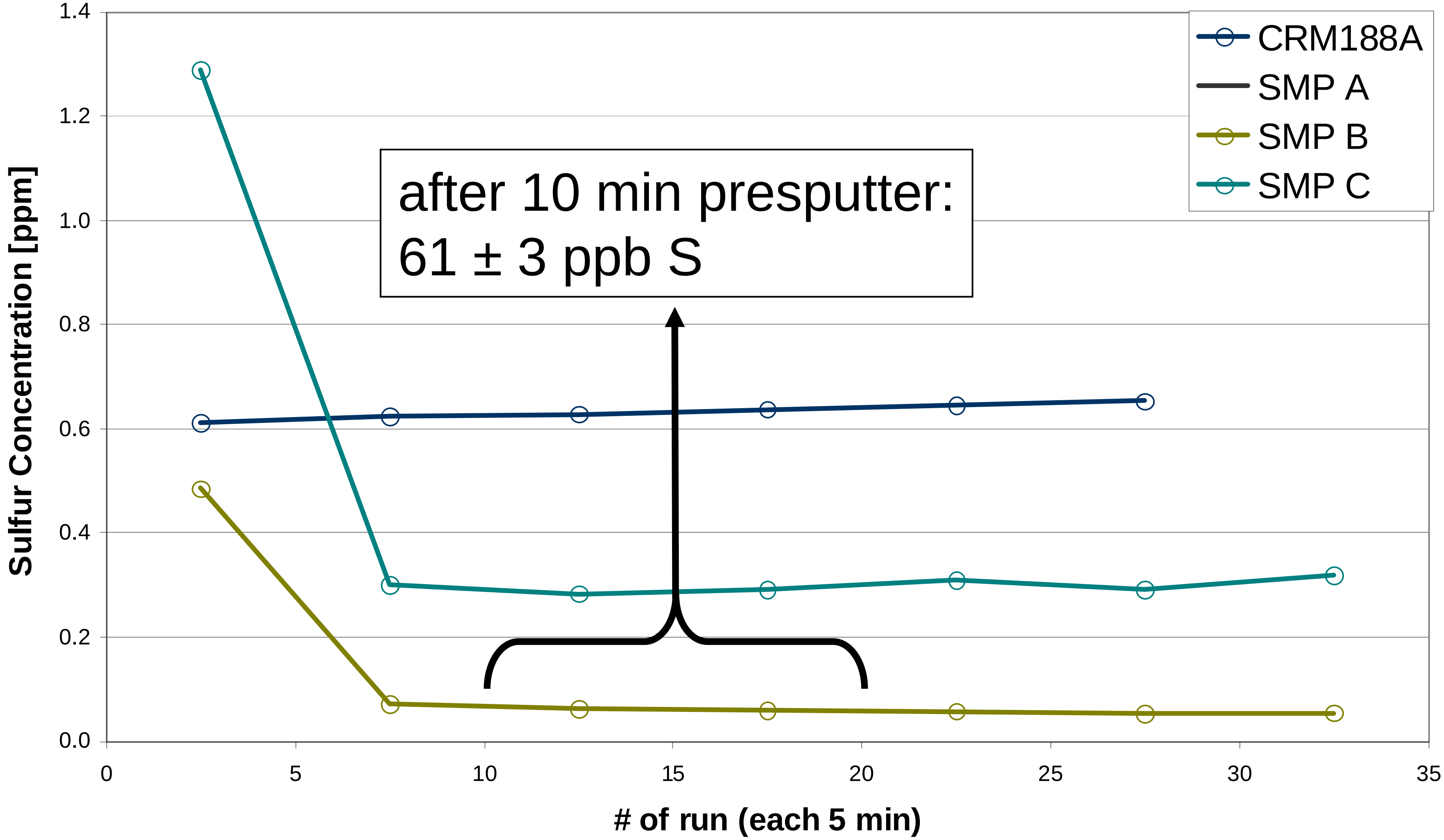
Calibrated with NIST 1249: 6.4 ± 1.0 ppm Sulfur certified

Note:

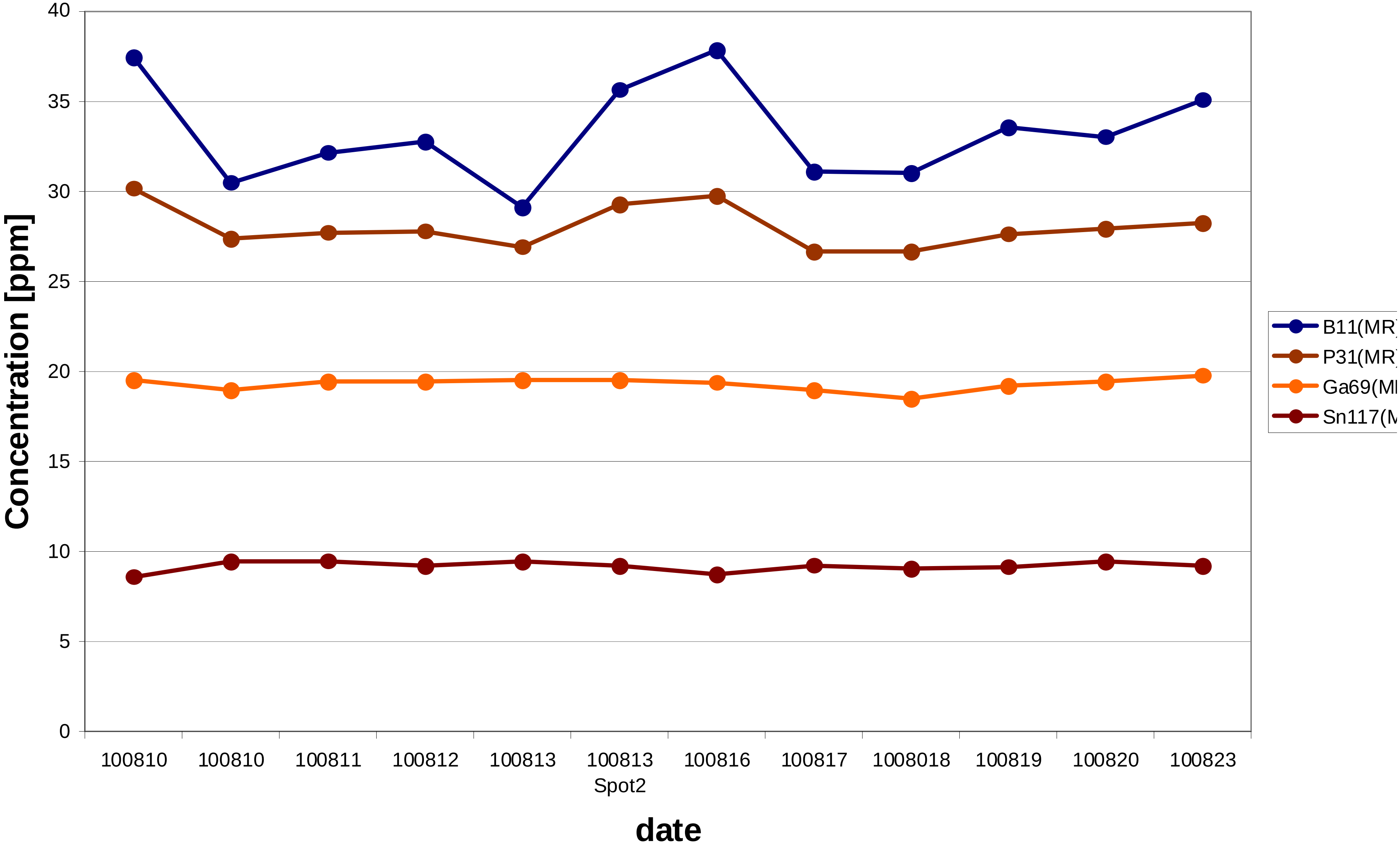
A sulfur retaining gas cartridge now comes as standard on all new instruments.

Reason: sulfur released by stainless steel gas capillaries

Presputter behaviour of S in Ni alloys



Longterm stability: trace metals in Ni alloys



Current investigations

- Pulsed DC source
- He addition as discharge gas

Pulsed DC source

Microsecond pulsed glow discharge in fast flow Grimm type sources for mass spectrometry

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Paper from EU project work on ELEMENT GD

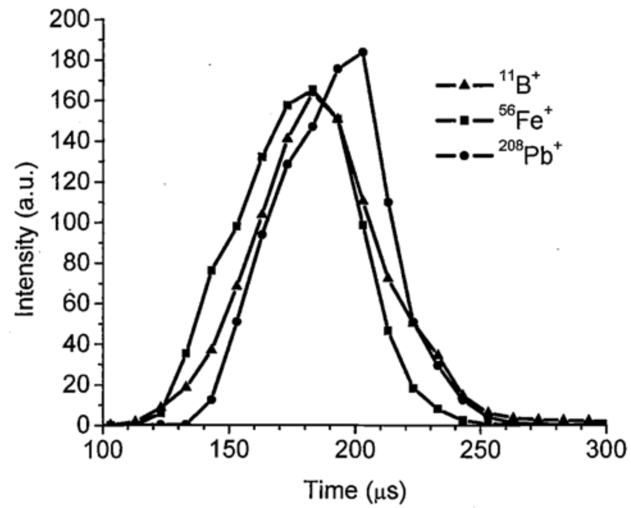
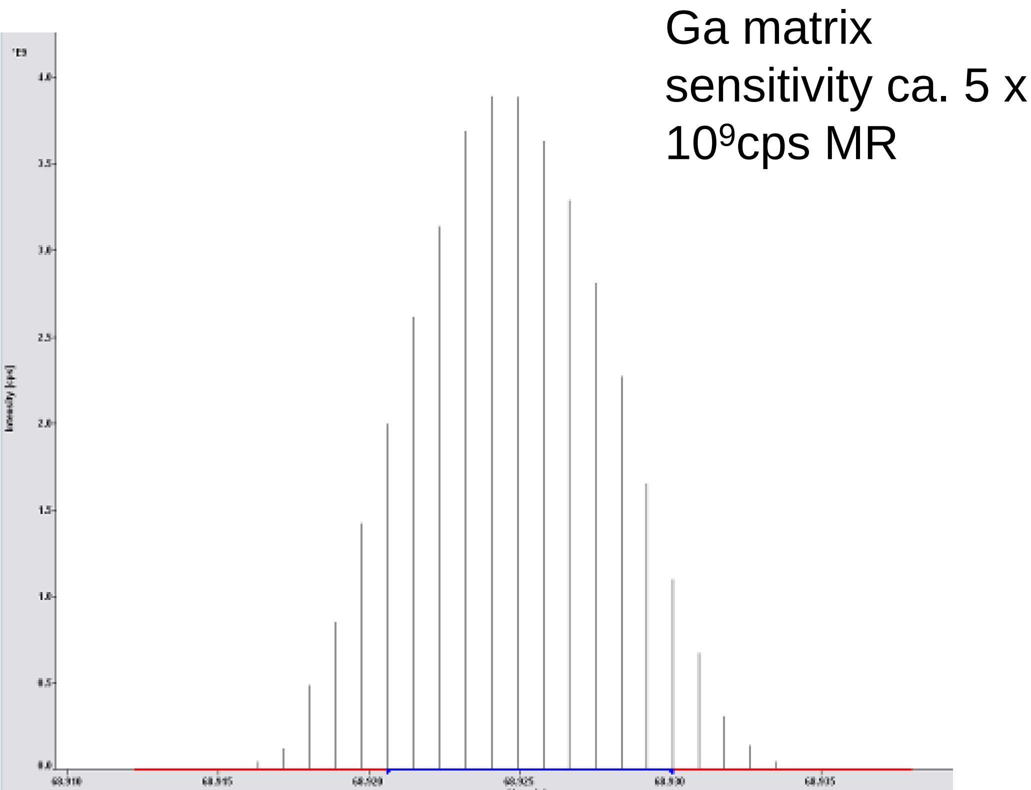


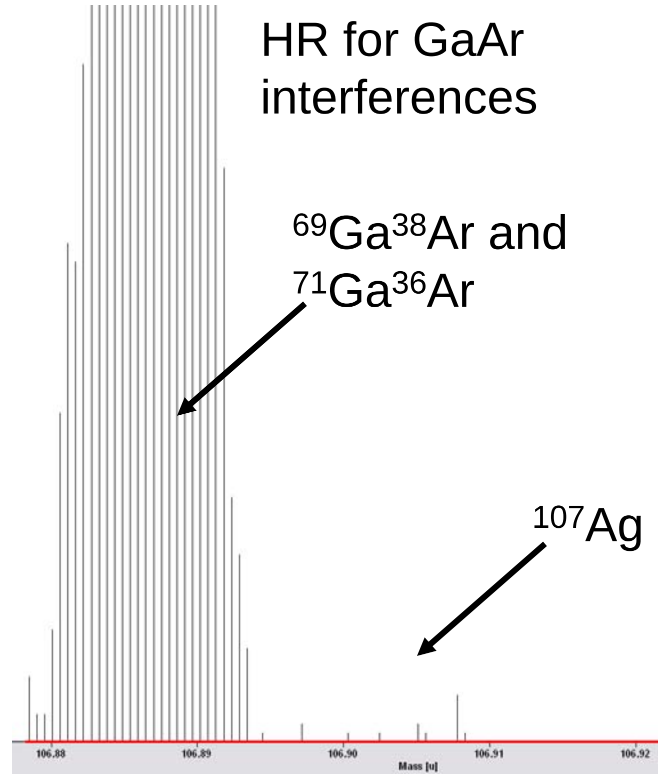
Fig. 4 Dynamics of different sample ions extracted from PGD in ELEMENT GD. PGD Parameters: $L = 430$ sccm, $U = 1.2$ kV, $\tau = 62$ µs, $f = 500$ Hz. Copper sample.

Pulsed DC-source: Gallium analysis

Pulsed mode with 1kHz and 50 µs puls duration running at ca. 3W

Continuous DC would require ca. 50mA, 30W – results in local melting of Ga sample





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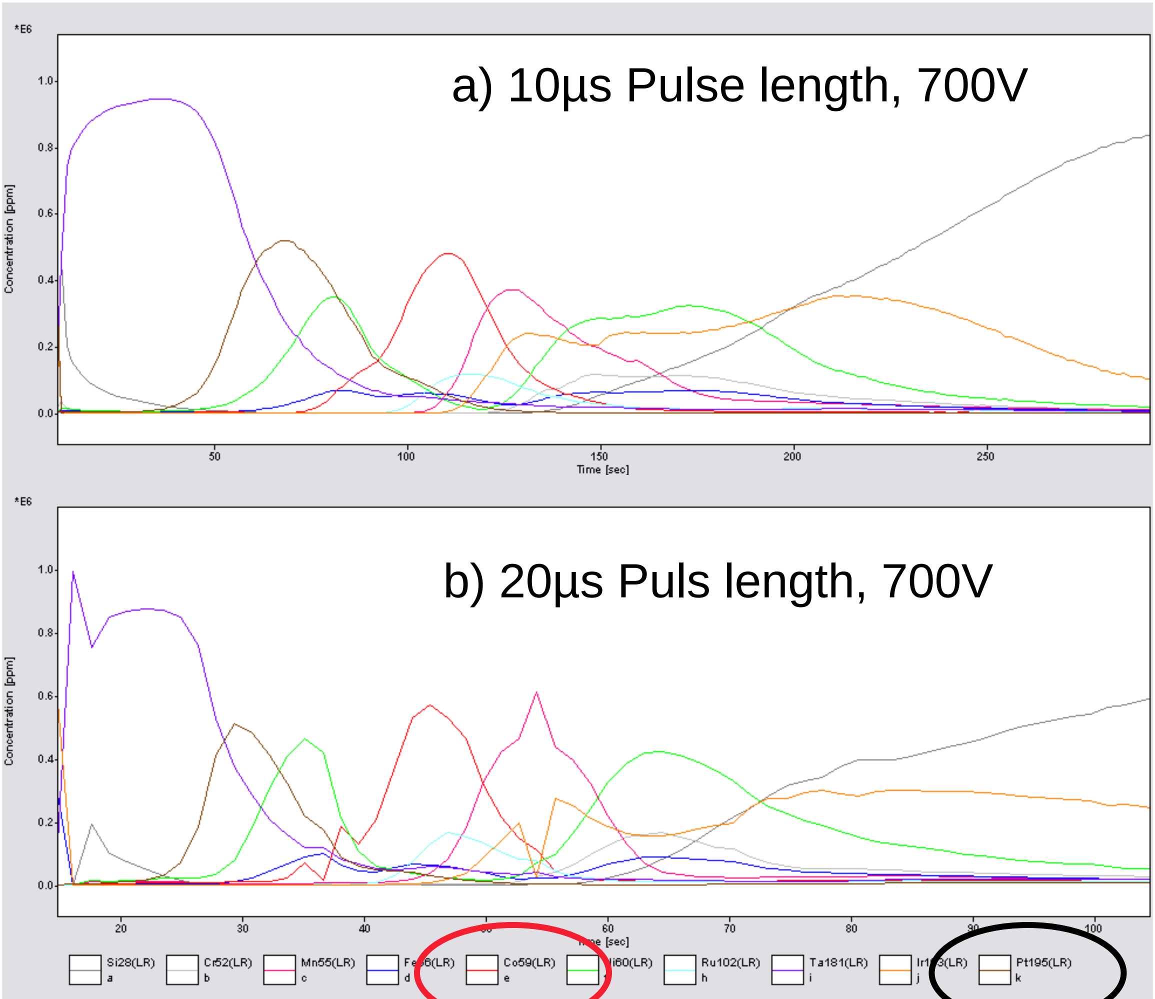
Thin layers



Harddrive:

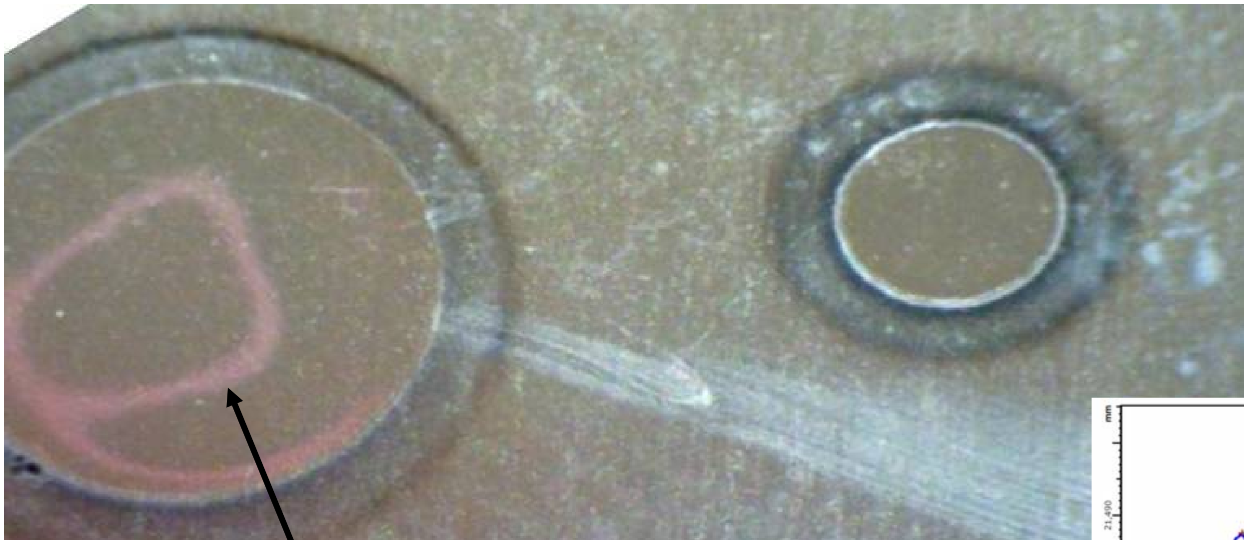
Total thickness ~ 25nm

Longer pulse = higher duty cycle = higher sputter rate

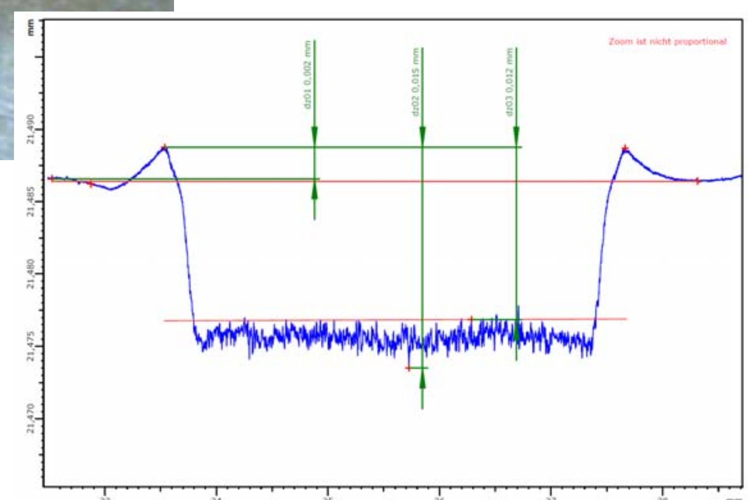
Pt ~ 8 Ångstrøm
Co ~ 10 Ångstrøm

Ni/Cu/Ni layers

Smaller anode diameter gives much improved crater shape

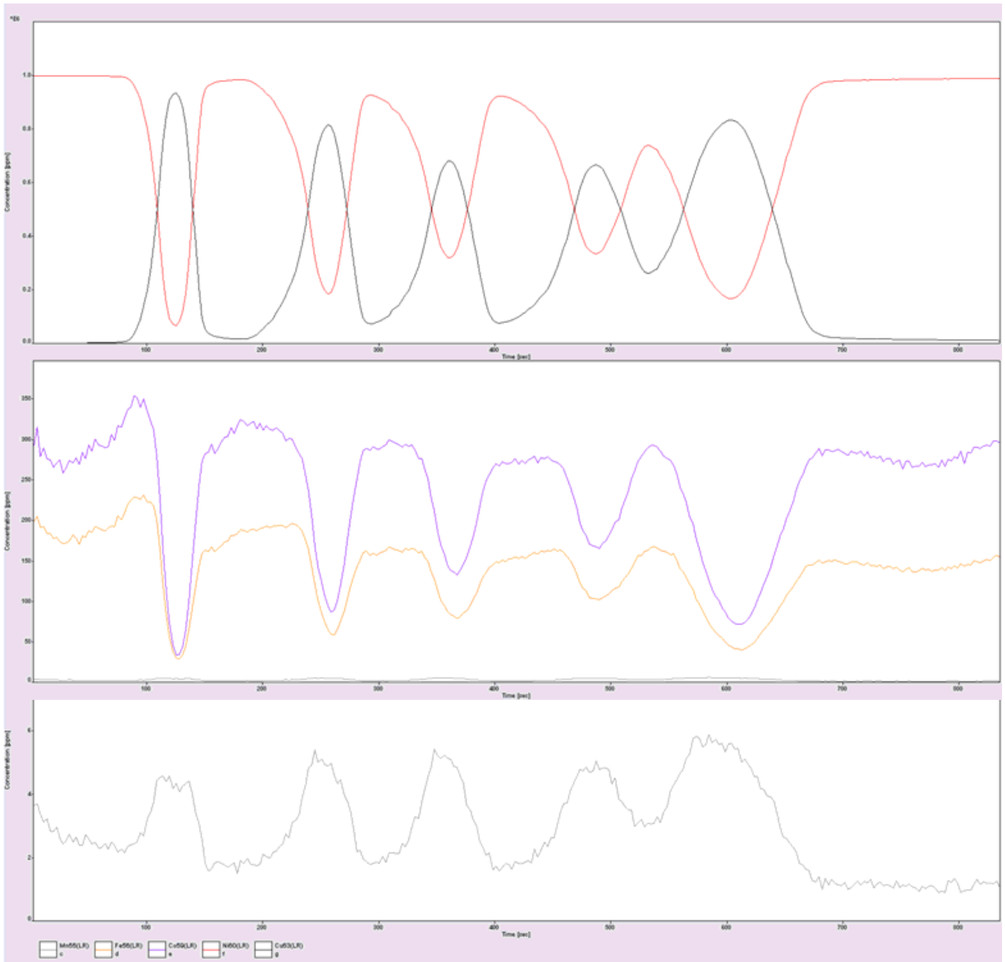


mixing of layers



Trace element determination

Ni/Cu/Ni multilayer system



Matrix components in the % range

High ppm range

(~10-500ppm Fe, Co)

Low ppm range

(~2-6ppm Mn)

Addition of Helium to Argon plasma

Helium addition can be useful for certain applications:

-better ionization of e.g. P, S and other high IP elements

-better transport of sputtered aerosol to the mass analyzer

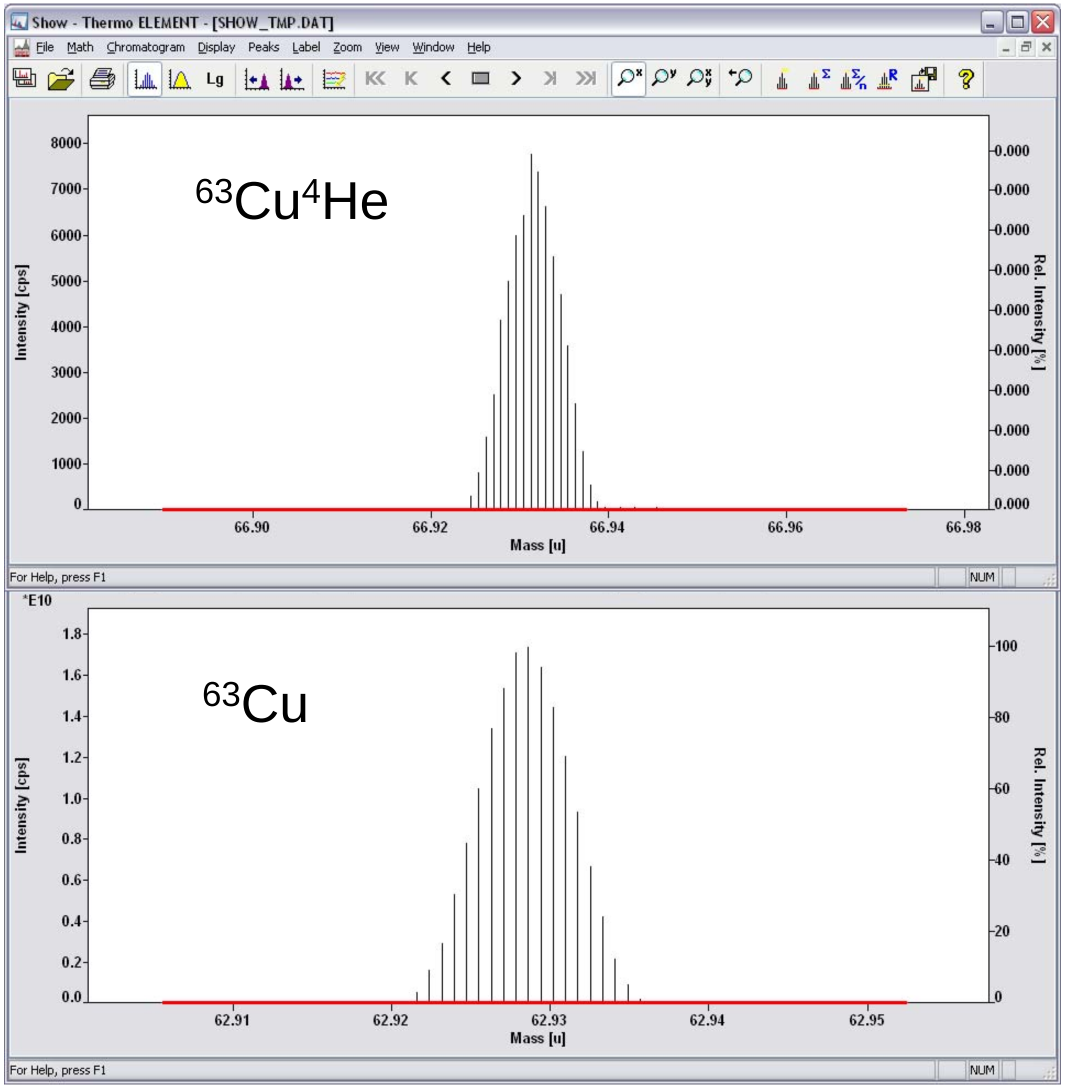
-reduced arcing from unfavourable sample geometries

-signal enhancement when using secondary electrode for non-conductors

Are new interferences generated by another discharge gas?

Does this influence the sensitivity factors?

Interference level of Matrix + He



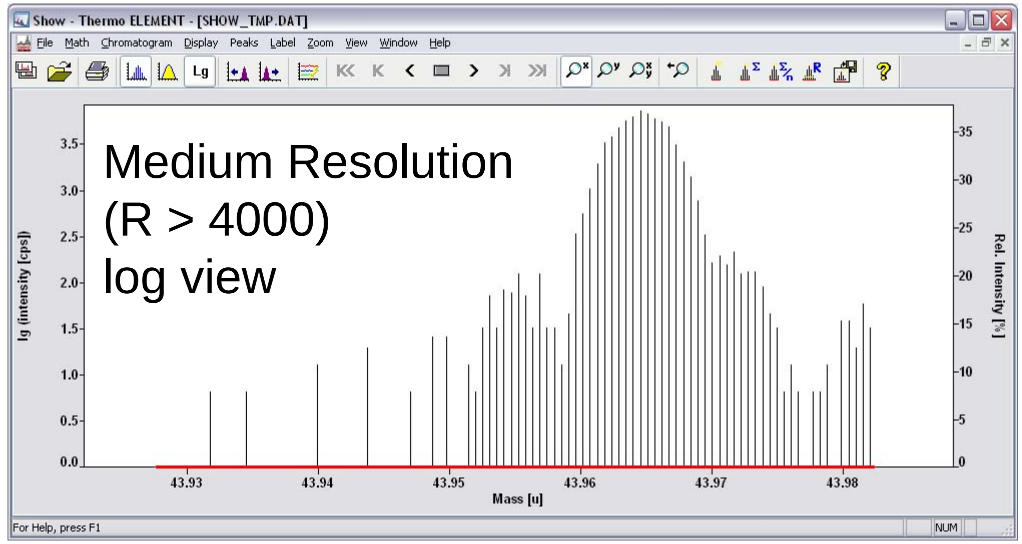
He formation rate ~0.4 ppm

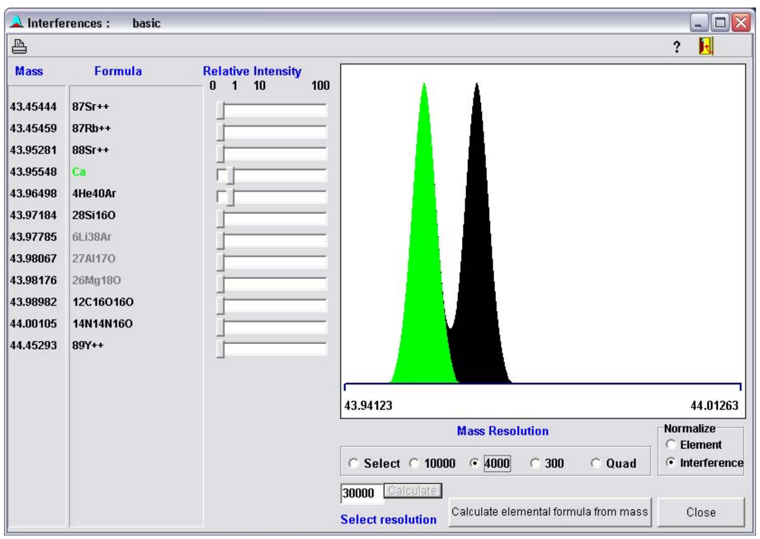
$^{63}Cu^{4}He$ intensity ca. 8000 cps

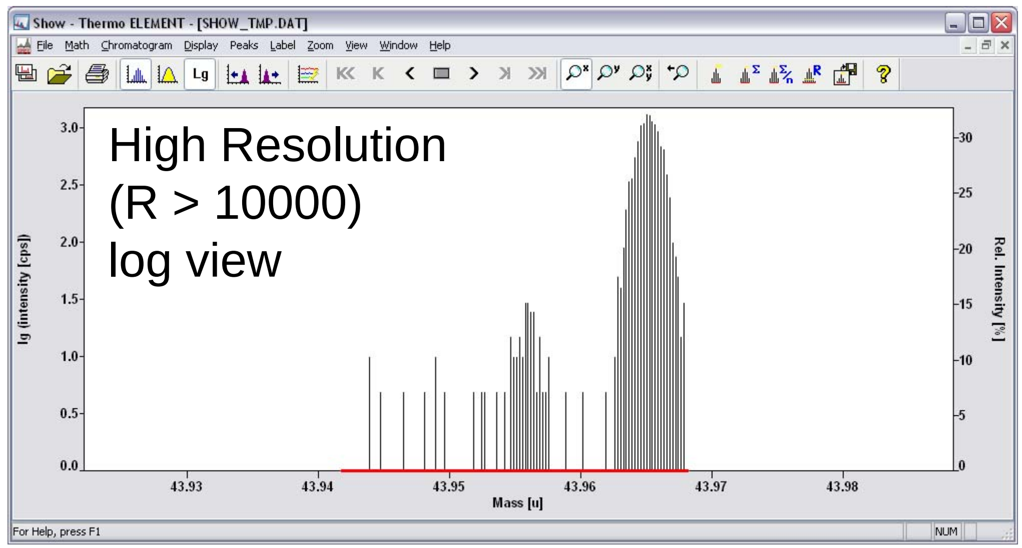
^{63}Cu ca 1.8 x 10^{10} cps

He addition - ArHe

- HeAr interference generated can be resolved in high resolution (or one can use ^{43}Ca)







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Effect of Helium addition on RSF

Flat sample Cu CRM BAM385:

with/without Helium addition, using the same STD RSF table

Element	Certificate BAM 385	Pure Argon	Pure Ar plus He addition
Al	28.6+-2.5	27	25
P	12.9 ± 1.0	10	17
S	31.3 ± 1.5	36	53
Cr	9.81 ± 0.20	6.1	5.8
Mn	10.1 ± 0.2	7.9	5.7
Fe	45.4 ± 1.4	57	35
Co	6.93 ± 0.15	8.5	4
Ni	11.9 ± 0.8	16	9
Zn	58 ± 4	74	80
As	11.4 ± 0.8	15	15
Se	7.2 ± 0.5	8.3	8.8
Ag	28.6+-0.8	31	38
Cd	5.8 ± 0.3	6	5.5
Sn	18.0 ± 0.9	12	6
Sb	19.1 ± 0.8	27	21
Te	10.0 ± 0.4	11	11
Pb	11.3 ± 0.5	11	4
Bi	5.81 ± 0.17	4.9	3.2

- increased sensitivity for light elements like P and S is a known effect of He addition
- for heavy elements some suppression visible
- work in progress to optimize gas flows for routine analyses

Conclusions

- ELEMENT GD is a routine tool
- Direct analysis, fast surface removal, low risk of sample contamination, small influence of sample preparation
- High purity analysis
- Ca. 2-3 samples per hour for full scan analyses at ppb level, ca. 5 samples per hour at 100 ppb level
- Pulsed DC source being finalized – investigate depth profiling
- Helium addition under investigation