Influence of instrumental factors on Auger quantification applied to dispenser cathodes

JEAN-MICHEL ROQUAIS

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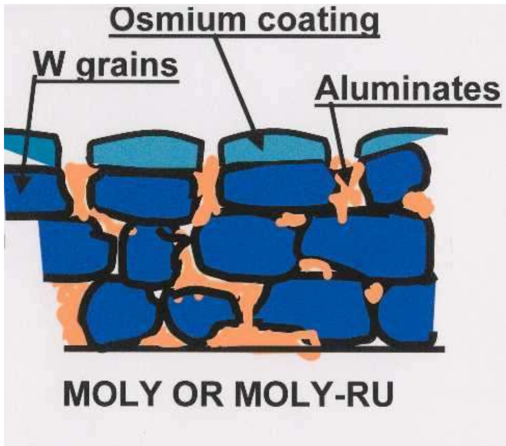
OUTLINE

- **Description of the cathodes**
- **Purpose of Auger measurements**
- **Auger systems set-up & typical spectra**
- **Comparison PHI CMA vs. SPECS HSA**
  - Angular differences and effects on signal intensity
  - Choice of acquisition mode of HSA (FRR & FAT)
- **Comparison between experiment and calculation**
- **Conclusion**

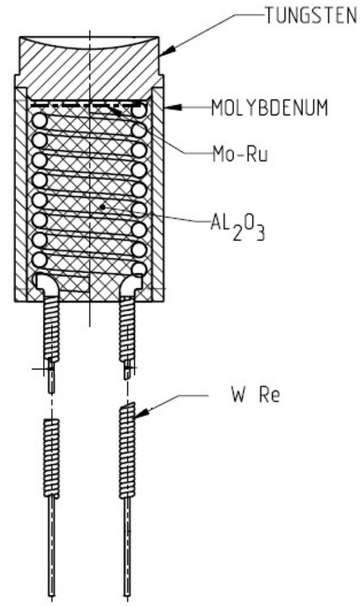
Description of the cathodes

M-type

- The cathode porous matrix is impregnated with 4-1-1 Ba, Ca aluminate



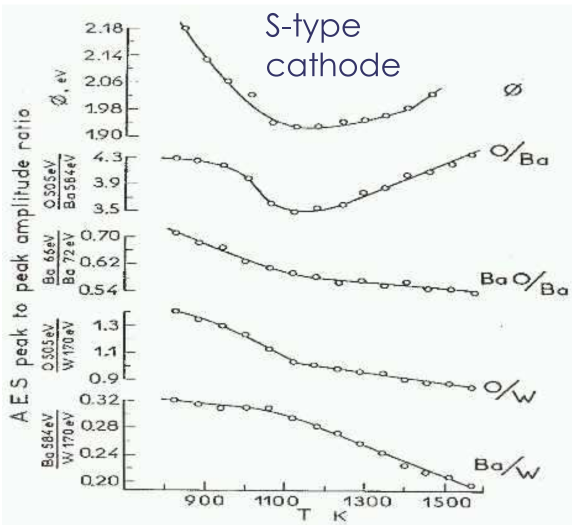
Cross-section

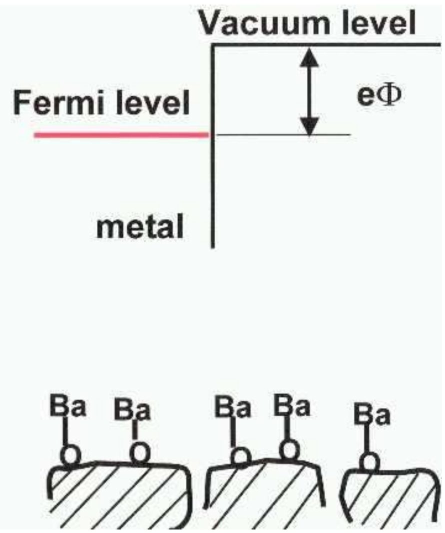


Purpose of Auger measurements

Link between surface composition and cathode emission

- *See D. Brion, J.C. Tonnerre & A.M. Shroff Applications of Surface Science 16 (1983) 55-72*
- Ba/W proportional to Ba coverage θ of the surface (fraction of monolayer)

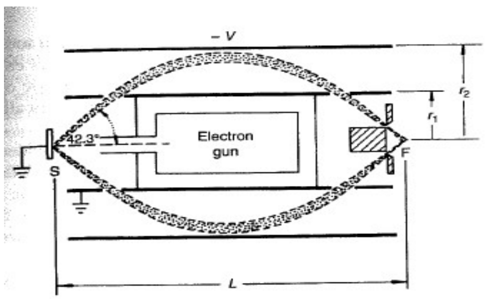




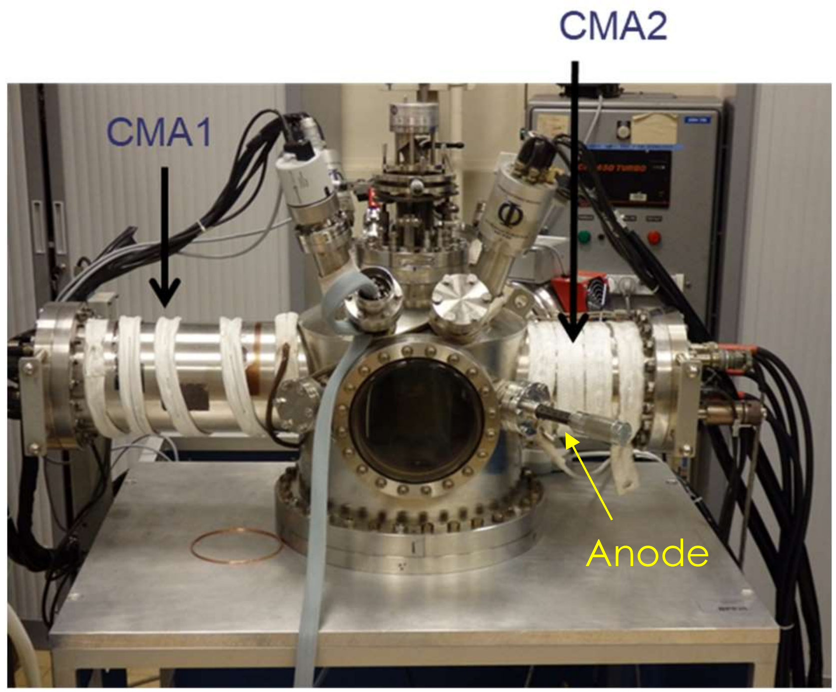
Auger systems set-up & typical spectra

PHI 545 from PHI

- CMA(Cylindrical Mirror Analyzer)



- e gun co-axial with the cylinders
- Anode dedicated to measure electronic emission of the cathode under test



Auger systems set-up & typical spectra

Phoibos HSA from SPECS GmbH

- H.S.A: Hemispherical Sector Analyzer

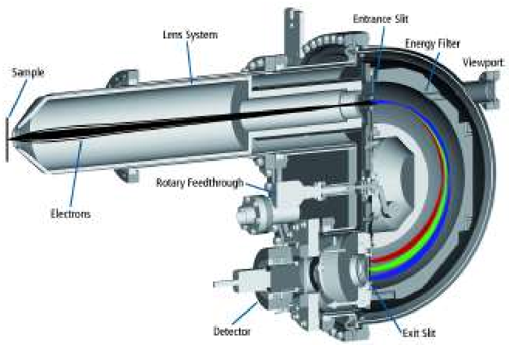
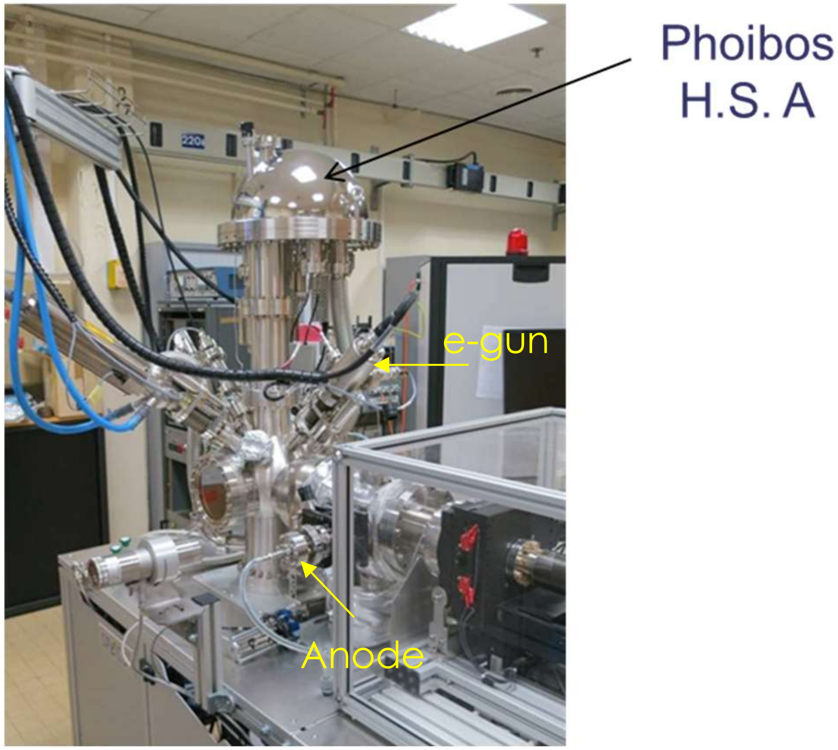


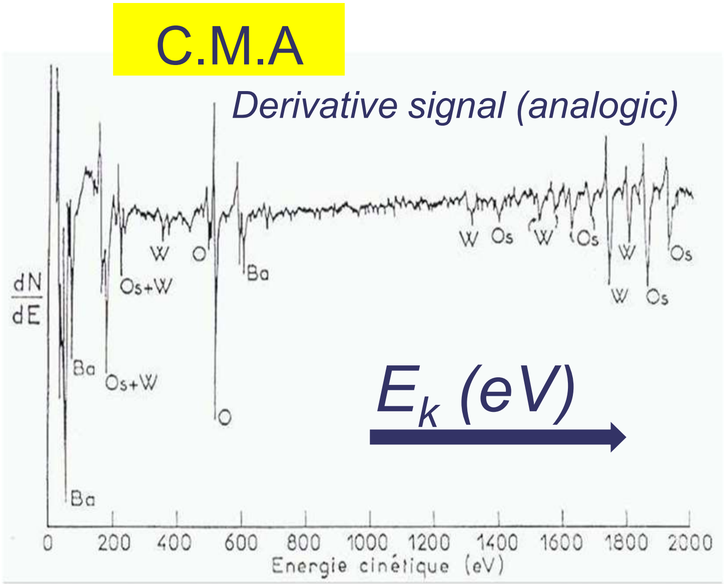
Figure 40: Cut-out view of PHOIBOS 100/150 analyzer

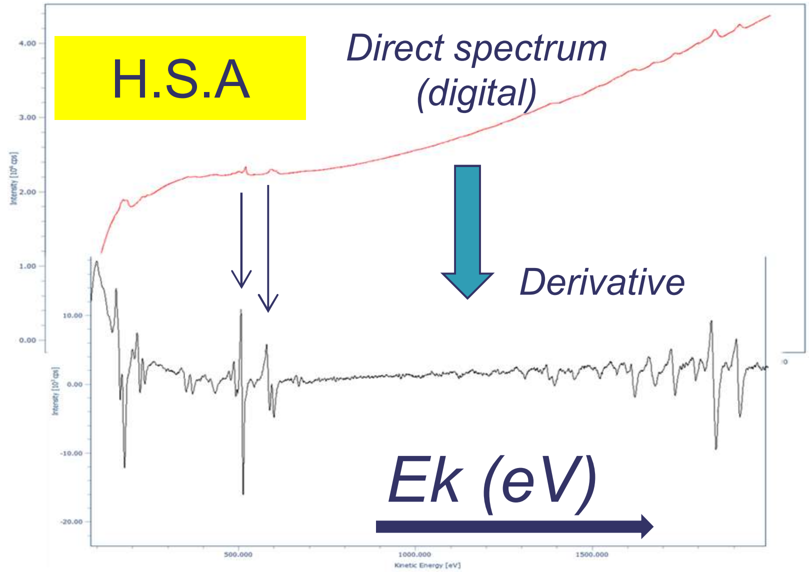
- e gun for Auger, X-Ray source for XPS
- Anode to measure electronic emission



Auger systems set-up & typical spectra

Typical spectrum on cathode surface acquired at 1000 °CB

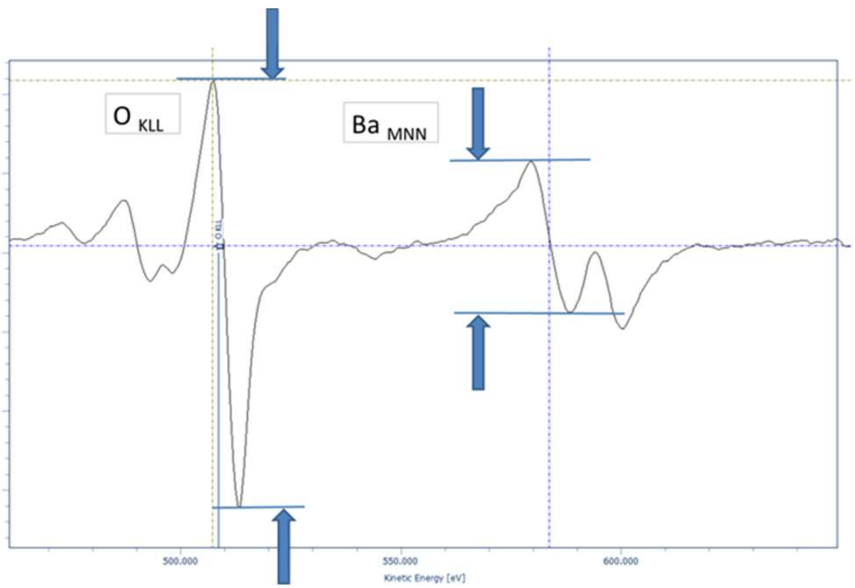


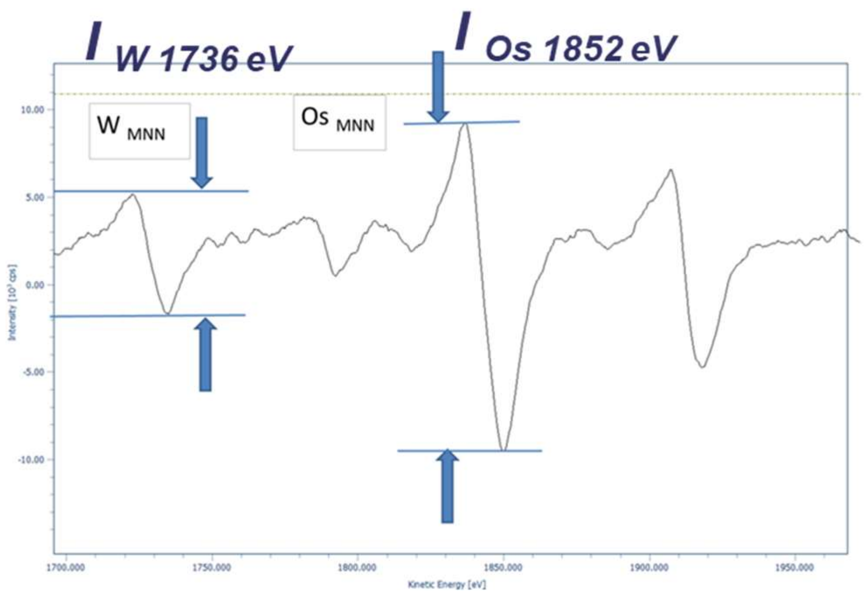


THALES

Auger systems set-up & typical spectra

Quantification





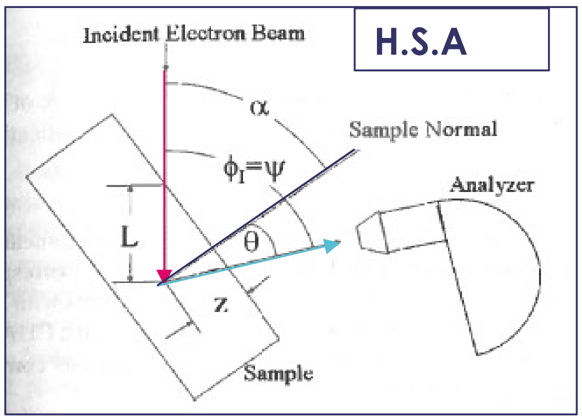
$$O/Ba = I_{O\ 510\ eV} / I_{Ba\ 584\ eV}$$

$$Ba/{}^{*}W_{h} = I_{Ba\ 584\ eV} / (I_{W\ 1736\ eV} + I_{Os\ 1852\ eV}/S_{rel.})$$

Comparison PHI CMA/ SPECS HSA/ angular differences

Geometry of Auger systems *

	Electron Incidence angle α with respect to the sample surface normal $\vec{n}$	Electron emission angle θ with respect to the sample surface normal $\vec{n}$
PHI 545 (CMA/ Single pass)	30 °	θ $_{avg.}$ ~ 50 °
SPECS (HSA)	48 °	0 °



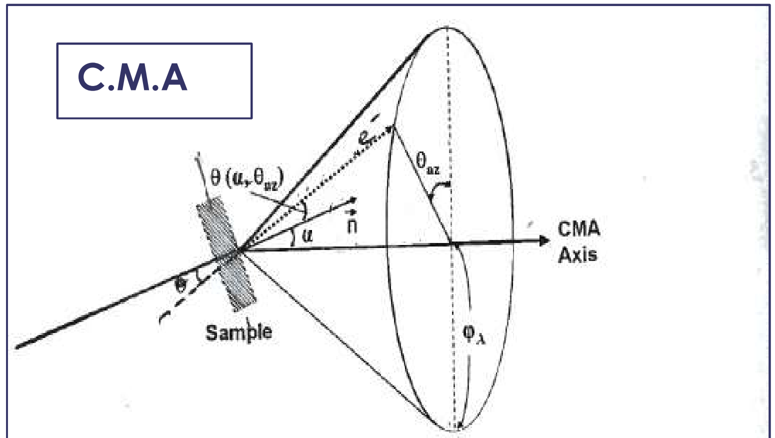


Fig. 5.4 Schematic view of the cone of the emitted electrons which are detected in a CMA with angle $\varphi_A = 42.3°$ cone angle. The emission angle to the sample surface normal n is $\theta(\alpha, \theta_{az})$. For a concentric electron gun, the sample tilt angle α is equal to the electron incidence angle. Changing the azimuth angle θ_{az} in a double pass CMA with a movable drum with a slit (6° opening angle) cuts out an emission angle θ given by (5.3). (Adapted from S. Hofmann and J.Y. Wang [5.11])

*** Siegfried Hofmann "Auger-and X-Ray Photoelectron Spectroscopy in Materials Science" Springer 49**

Comparison PHI CMA/ SPECS HSA/ angular effect on intensities

Approximation of thin overlayer A on substrate B (from *Hofmann § 4.4.3.2):

- E_A: kinetic energy of signal from A
- E_B: kinetic energy of signal from B
- S_A, S_B : elemental sensitivity factors (not instrument linked)
- *$\lambda_{A,E(A)}$: attenuation length of signal of A at E_A*
- *θ*: electron emission angle

***Auger intensities ratio I_A/I_B* depends on incident angle α and emission angle θ**

$$\frac{I_A}{I_B} = \frac{S_A}{S_B} * \frac{1+r_{B,U(A)}}{1+r_{A,U(A)}} * \left[\exp\left[\frac{d}{\lambda_{A,E(A)}\cos\theta}\left(\frac{EA}{EB}\right)^{0,75}\right] - \exp\left[\frac{-d}{\lambda_{A,E(A)}\cos\theta}\left(1-\left(\frac{EA}{EB}\right)^{0,75}\right)\right]\right]$$

Cancels at instruments comparison

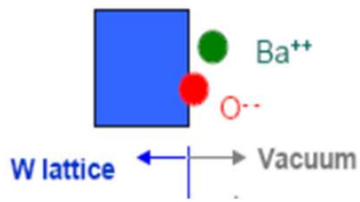
α influence
Backscattered term close for SPECS and PHI systems

θ influence

Comparison PHI CMA/ SPECS HSA/ angular effect on intensities

Approximation of thin overlayer A on substrate B (from *Hofmann § 4.4.3.2):

- Surface layer A = O-Ba monolayer
- Substrate B = W-Os
- *λ_{590} ~ 4,8 mono-layers

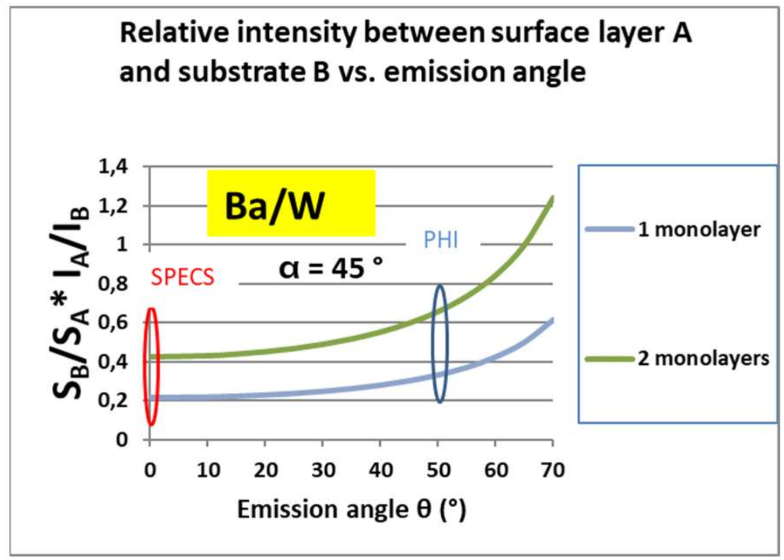


Emission angles: $\theta_{SPECS} \neq \theta_{PHI}$

➔ $I_A/I_{B\,(SPECS)} \neq I_A/I_{B\,(PHI)}$

Calculation for cathode leads to:

Ba/W $_{(\theta = 50°/PHI)}$ = 1,56 * Ba/W $_{(\theta = 0°/SPECS)}$



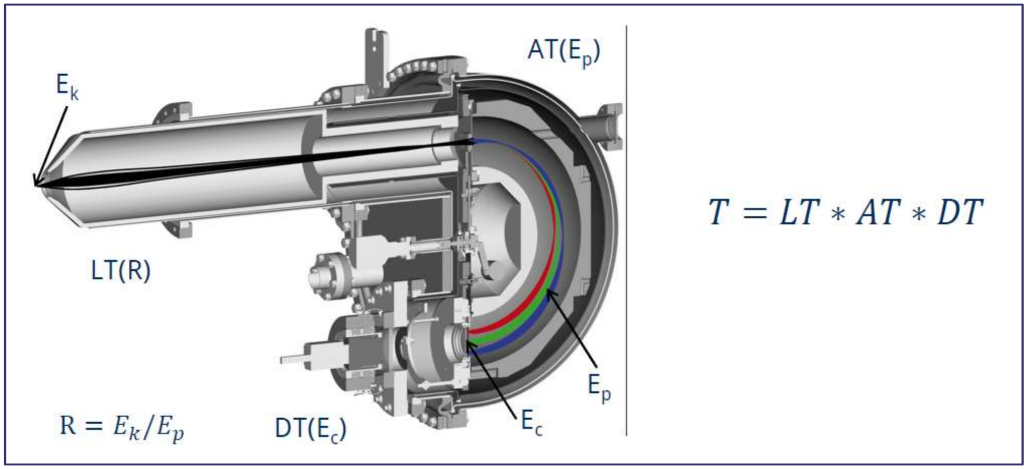
* G. A. Haas et al. Applied Surface Science 24 (1985) 430-446

Choice of acquisition mode of HSA

Signal intensity *I* is proportional to analyzer transmission function *T*

H.S.A: 3 sections determine *T*

- **Lens section** (= retarding section)
- **Analyzer** (hemispheres)
- **Detector** (MultiChannel Plates)



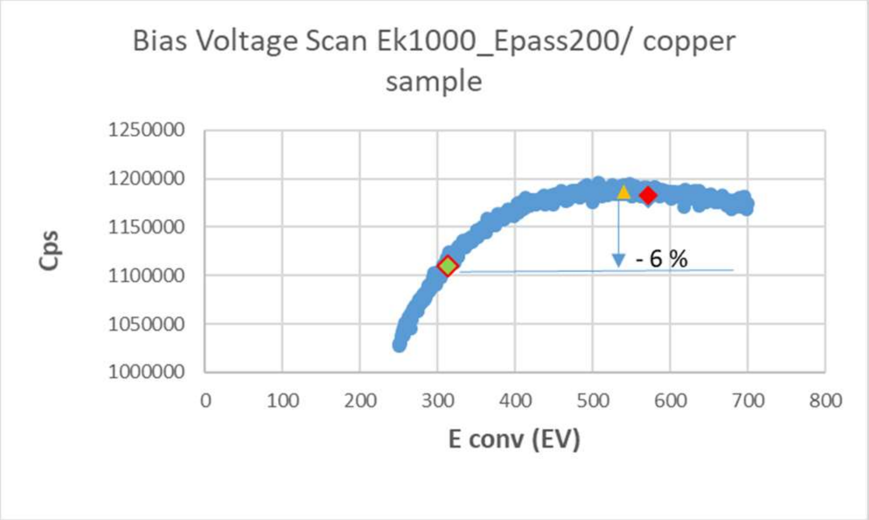
Electron kinetic energy

- E_k at Lens section entrance
- Pass energy E_{pass} = E_k/R at hemispheres entrance
- Conversion energy **Ec** = E_{bias} + E_{pass} (a bias voltage is applied between hemispheres exit and detector)

Choice of acquisition mode of HSA

Detector efficiency (Signal intensity vs. E conversion)

- E conv. = $E_{bias} + E_{pass}$
- In FRR mode (Fixed Retarding Ratio) E_{pass} = E k/R
- DT(E_c) not constant and E_{conv}. varies with E_k ➔ DT(E_k) not constant



		FRR5	
	Ekin (eV)	Epass (eV)	**Econv (eV)**
Ba584	584	116,8	**316,8**
W1736	1736	347,2	**547,2**
Os1850	1850	370	**570**

Transmission function *T*

FRR vs. FAT

- FAT (Fixed Analyzer Transmission): E_{pass} is constant
- E_{pass} chosen to match quantification results in FRR

	Advantage	Drawback
FRR mode	• $\Delta E/E$ = cste like for the CMA (PHI)	**Detector ageing**: If drift of detector gain ➔impact on quantification
FAT mode	No influence of detector on quantification when only Auger peak ratios are under consideration	Need to verify Lens Transmission on a regular basis (check applied voltages)

Comparison between experiment and calculation

Determination of « transposition » factor between PHI and SPECS

- Comparison of cathodes from the same material batches
- The ratios are average values of 5 spectra/ cath.

Results

- O/Ba ratio is the same in PHI and SPECS
- Ba/Wh ratio shift between SPECS and PHI
- The experimental transposition factor matches the calculated one
- The difference of e^- emission angle (cosθ factor) explains mainly the shift

		O/Ba (Measured)	Ba/Wh (Measured)	
PHI	Avg. 15 Cath.	**2,93**	**0,341**	
SPECS /FAT	Avg. 9 Cath.	**2,83**	**0,207**	**X 1,65**
Experimental transposition factor				
PHI/SPECS FAT		**1**	**1,65**	
Theoritical transposition SPECS/PHI :				
Cosθ (SPECS)/ Cosθ (PHI)			**1,56**	

Comparison between experiment and calculation

Statistical results

- Following transposition tests, comparison of 29 cath. in SPECS vs. 29 cath. In PHI.
- Equivalent results are obtained in SPECS and PHI
- Typical J0 is 11A/cm² at 1000 °CB.

Statistical confirmation		O/Ba (Measured)	Ba/Wh (Measured)
PHI	Avg. 29 Cath.	**2.88**	**0,345**
SPECS/FAT	Avg. 29 Cath.	**2,98**	*** 0,341**
*** Experimental transposition factor applied**			

Conclusion

Transposition from PHI to SPECS system was completed succesfully:

- Acquisition mode « FAT » was retained for quantification (not impacted by detector efficiency)
- A transposition factor between PHI and SPECS systems has been calculated based on differences in systems geometries
- The transposition factor has been confirmed by comparative measurements on cathodes in both Auger systems

After completion of transposition, statistical results on 29 cathodes assess the transposition factor

Acknowledgements

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Thank you for your attention !