



The GILDA
CRG at ESRF

Application of LTDs to Synchrotron Radiation



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Layout



- Introduction & comparison
- Present & near future applications
 - X-ray Absorption Spectroscopy
 - X-Ray Fluorescence analysis
- Future applications
 - X-ray emission spectroscopy
 - Resonant Inelastic X-ray scattering
 - X-ray Raman Scattering
- Conclusion



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+ and - of LTD detectors



- + Low energy resolution
 - + Absence of geometrical constraints (scattering angles, beam size, ..)
 - + No critical mechanical alignment
 - + In *vacuum* operation
-
- Low count rate
 - Reduced solid angle
 - Applicability to hard X-rays ?

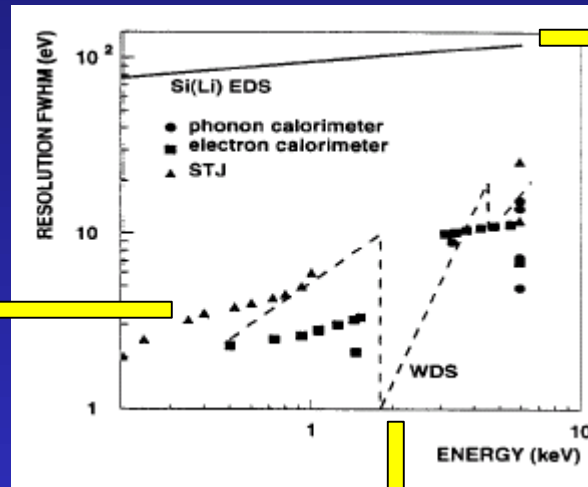


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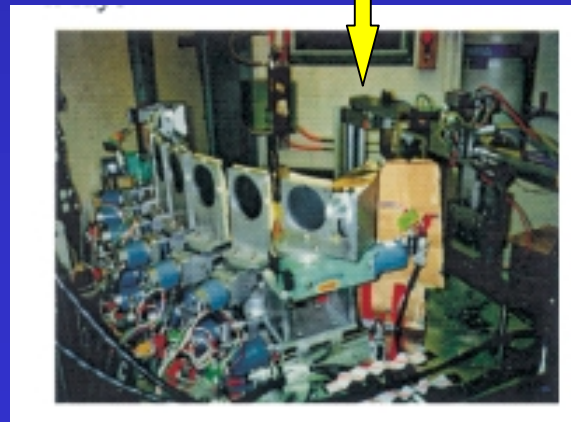
Energy-resolving x-ray detectors



LTD (STJ)



Semiconductor
Solid State



Crystal
analyzers



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Energy-resolving x-ray detectors



Type	ΔE (eV)	E (keV)	Count Rate	Solid angle
HP-Ge, Si:Li	150-300	0.3-120	10^5 cps	10^{-2} sr
STJ *	10	0 - 1?	10^4 cps	10^{-4} sr
TES *	20	0-10	10^3 cps	10^{-4} sr
Bragg/ Grating	0.2	0.1-10	10^6 cps	10^{-3} sr

* Couple high resolution to no geometrical constraints

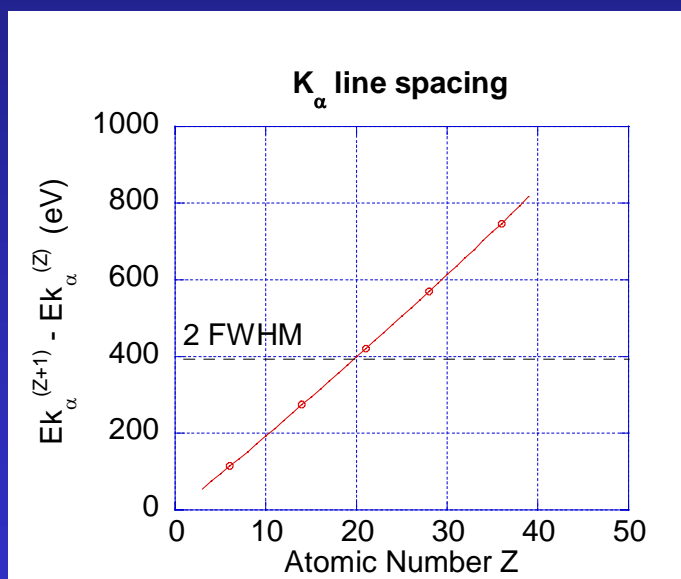


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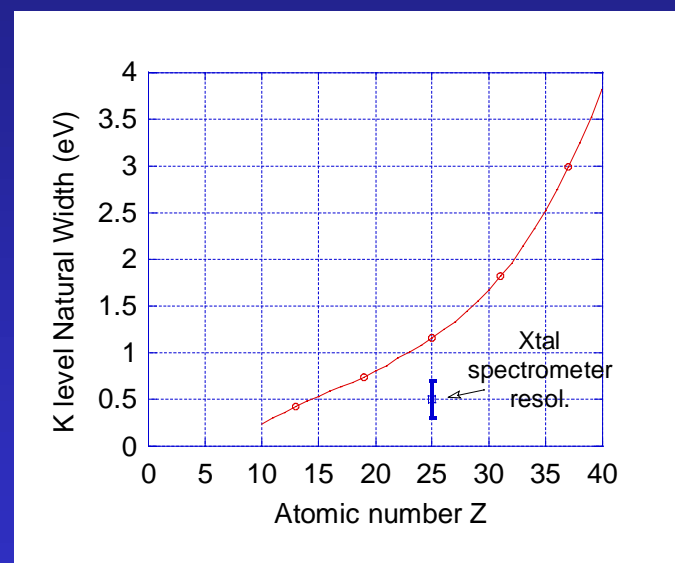
Application fields



Physical parameters defining the energy resolution



- X-ray absorption spectroscopy
- X-ray fluorescence analysis



- X-ray emission spectroscopy
- Resonant Raman Scattering
- Non resonant Raman scattering



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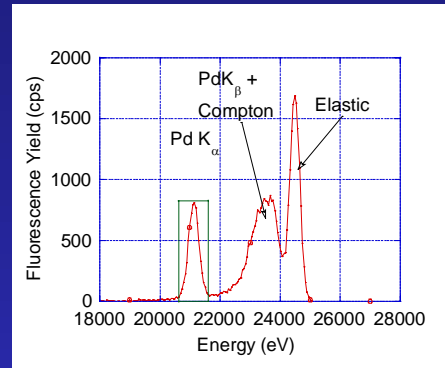


Present & near future applications

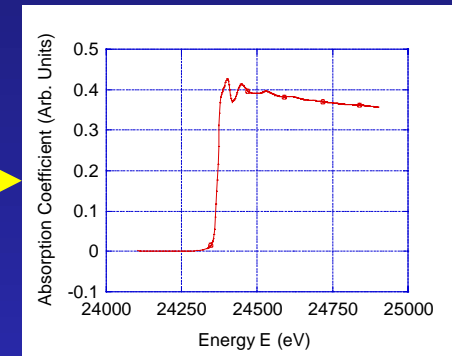


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X-ray Absorption Spectroscopy



X-ray spectrum
coming from the
sample



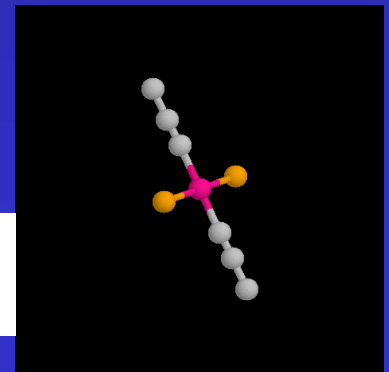
EXAFS spectrum

Sample

Monochromatic X-rays
of energy E

$$w \propto \sum_f \left| \langle f | \exp(i\hat{\epsilon} \cdot \vec{r}) | i \rangle \right|^2 \delta[E_f - E_i - h\nu]$$

F. d'Acapito LTD-10 Conference



Local Structure



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Fluorescence flux



Detector:

Collection solid angle= 10^{-3} sr

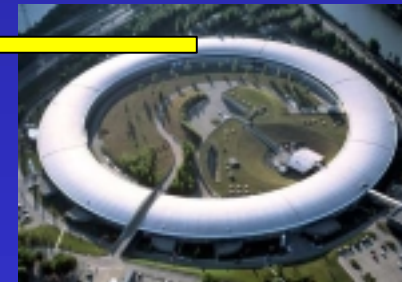
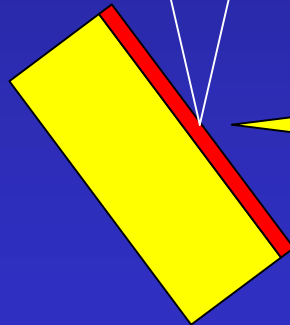


K fluorescence
Flux: 10^4 ph/s

(Neglecting
elastic +
compton)

Sample:

concentration= 10^{15} Mn / cm^2



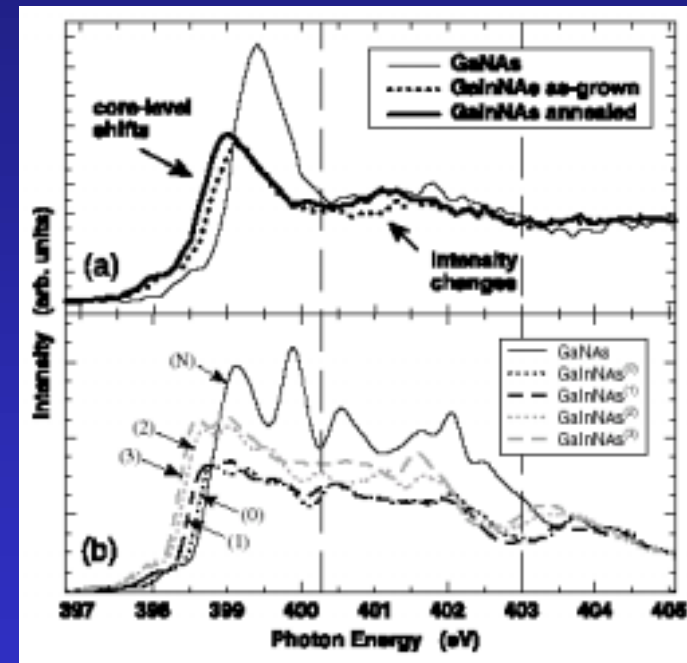
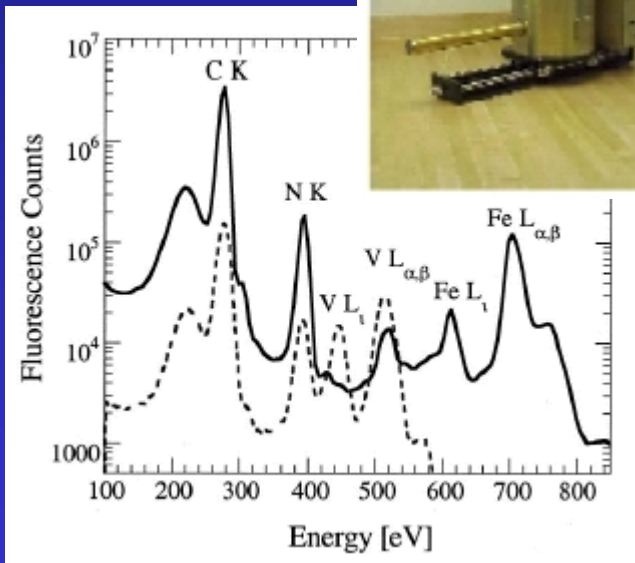
X-ray beam:

Intensity= 10^{12} ph/s @ 6540 eV



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Soft X-ray region - XAS



XAS experiment (InGaAsN at the N k edge)

Lordi et al. PRL **90** (2003), 145505

STJ detector for soft x-ray fluorescence
(Friedrich et al. RSI **73** (2002), 1629.)



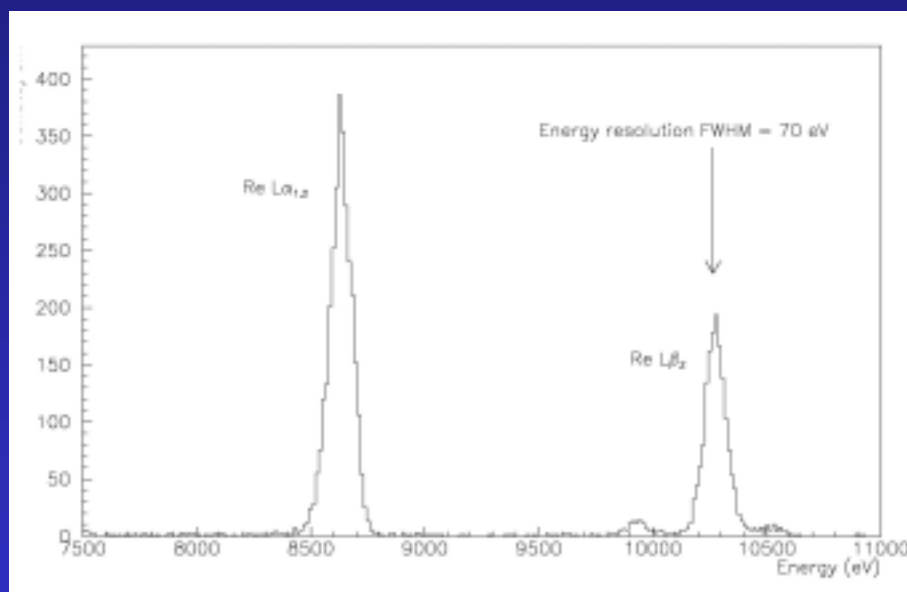
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Hard X-ray region - fluorescence



TES detector

300*400*25 μm^3 Sn absorber
TES sensor an Al/Ag multilayer
Count rate \approx 100cps
 $\Omega \approx 10^{-8}$ sr

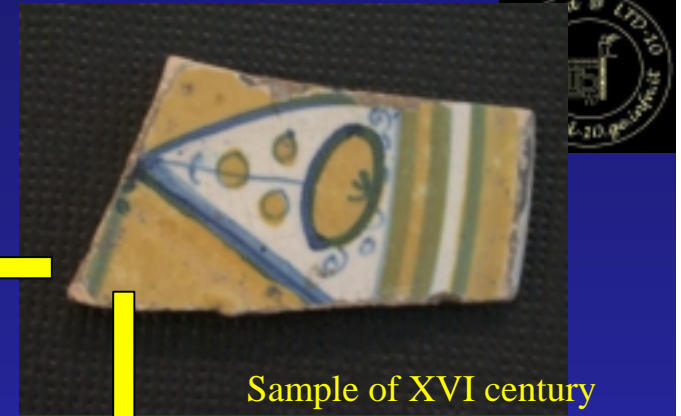
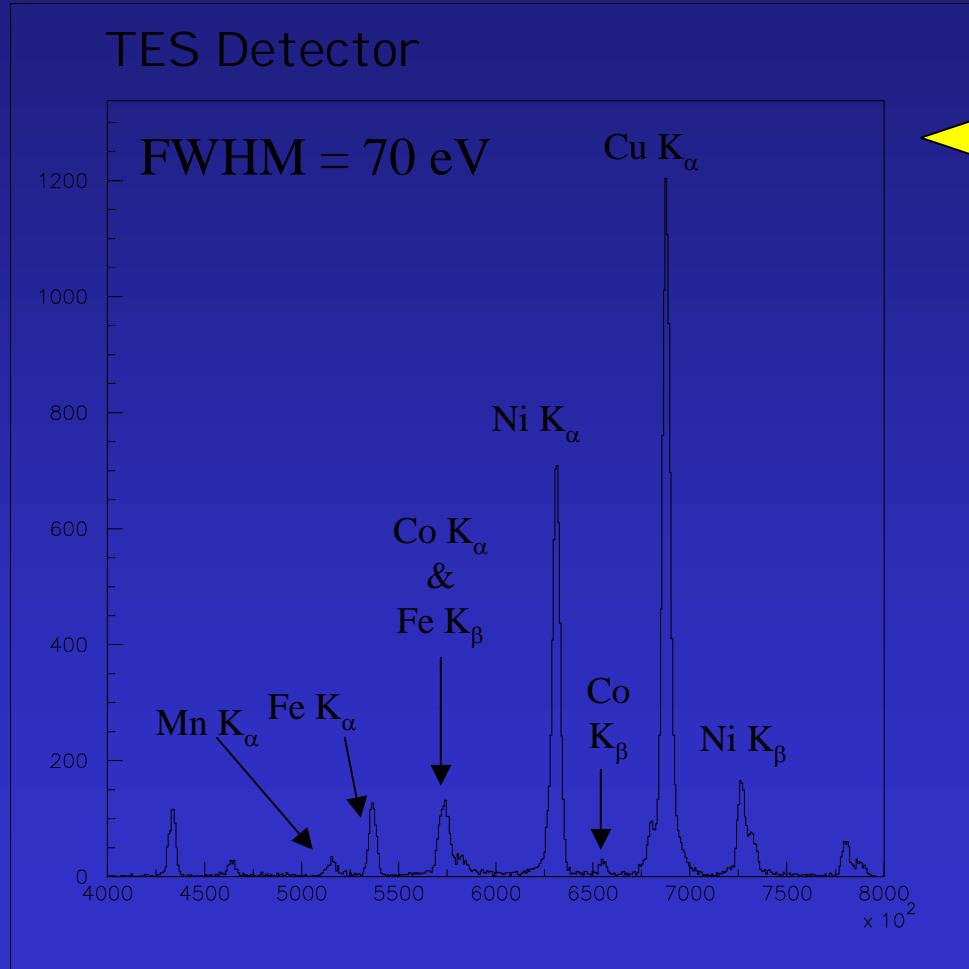


Fluorescence spectrum of a Re foil
F. Gatti et al SRN 3 (2003), in press.
See also Poster F03

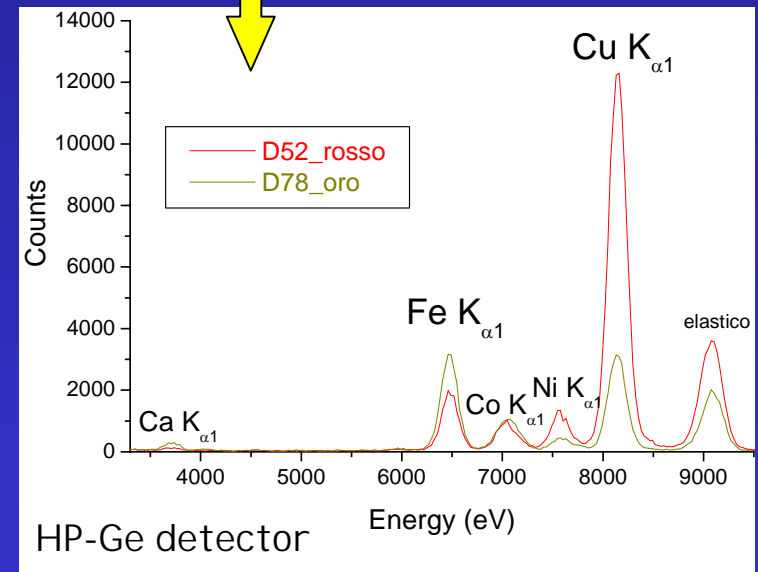


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Fluorescence analysis



Sample of XVI century
italian pottery.



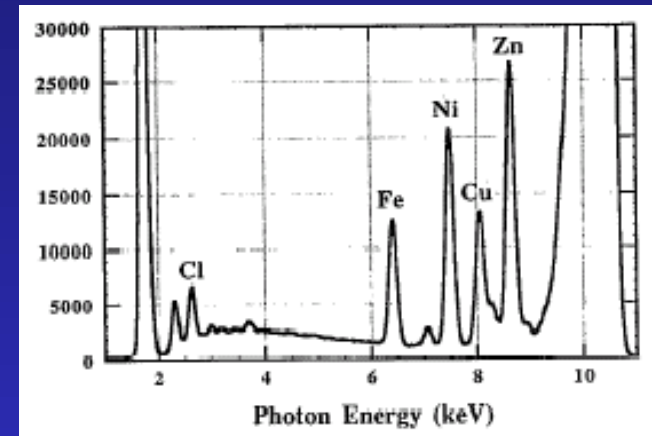
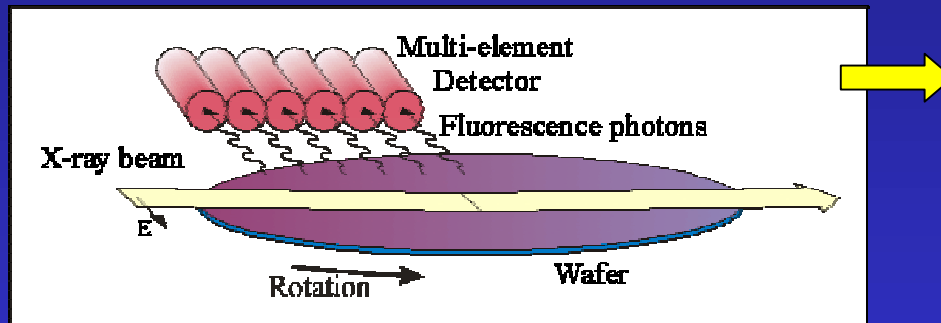


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Wafer surface impurity analysis



- Control of **industrial processes**
- Synch. Radiation source
- Total reflection condition
- Detection limit: $10^8 - 10^{12}$ at/cm²



Intentionally contaminated Si wafer.
Measurement done with a Si:Li
detector, $\Delta E=140$ eV.

P. Pianetta et al. RSI **66** (1995), 1293

High resolution desirable
High count rate ($>10^5$ cps)



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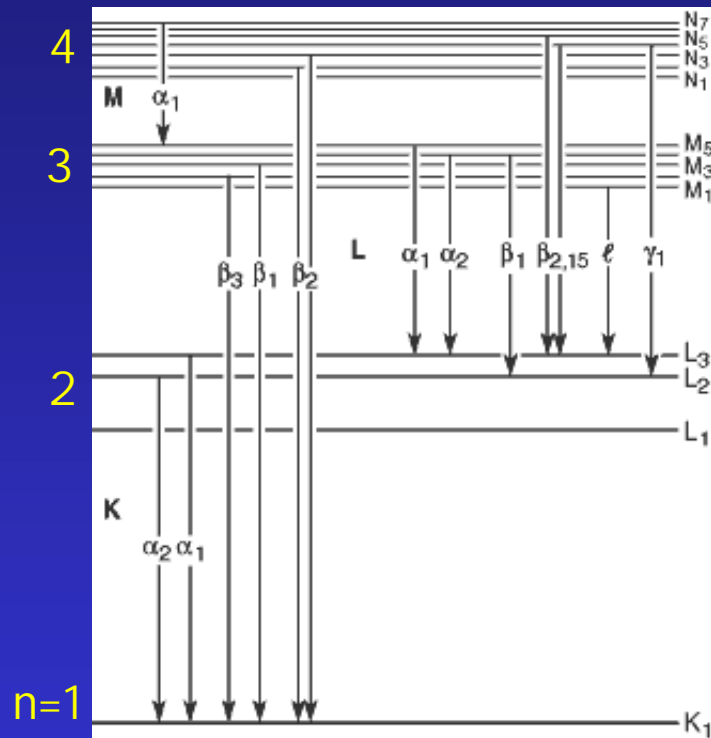


Future applications



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High resolution x-ray emission spectroscopy



Levels of x-ray fluorescence transitions

- K_α and (much more) K_β lines exhibit variations linked to the chemical environment
- Variations are greater than the natural linewidth of the K state
- The achievement of energy resolution of the order of 1-2 eV could give access to the several experimental techniques techniques

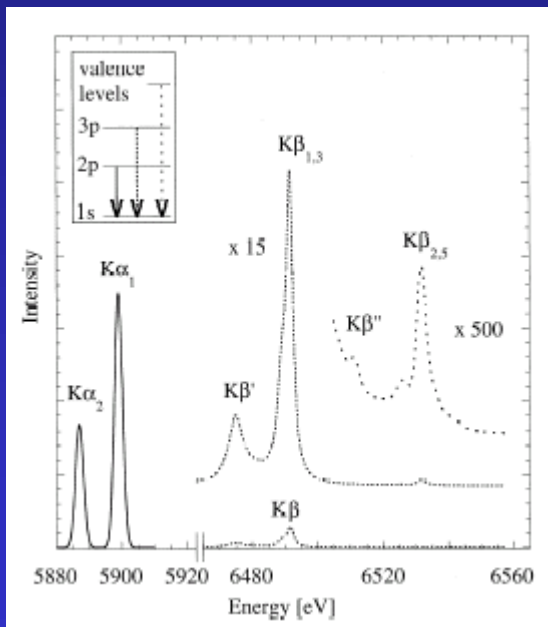


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Mn K fluorescence



U. Bergmann et al. CPL **302** (1999), 119.



Emission from Mn
excited above the K
edge

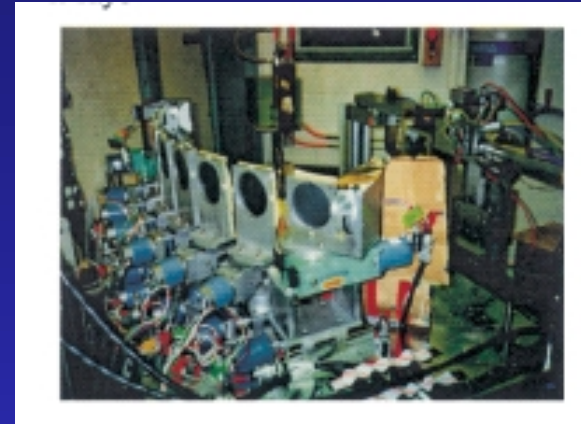
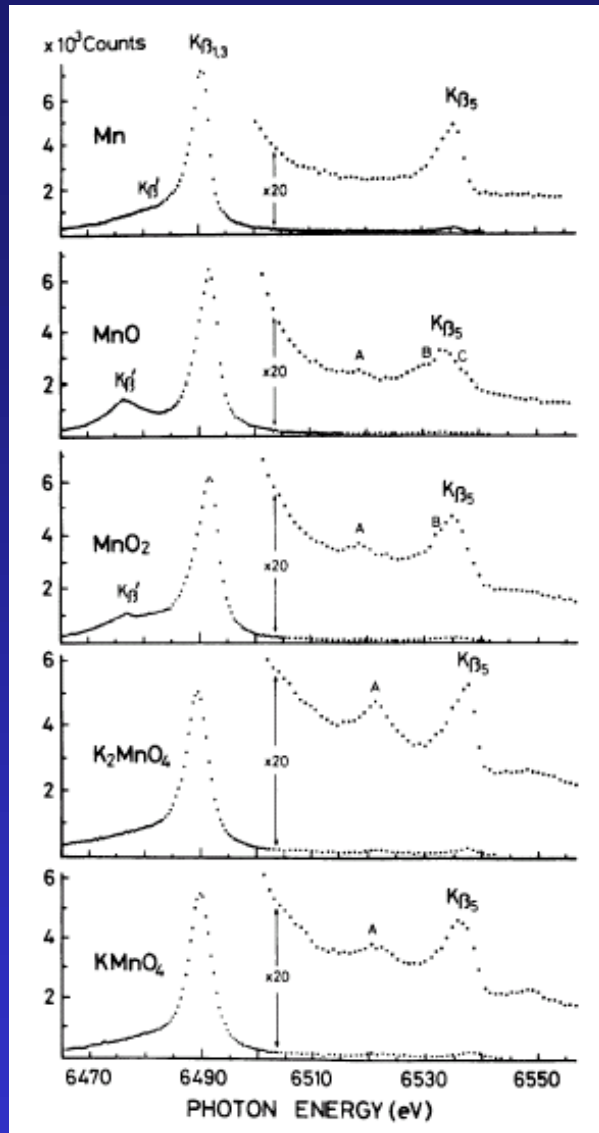
$K_{\alpha 1}$ and $K_{\alpha 2}$ transition from 2p levels: fair chemical sensitivity

$K_{\beta 1,3}$ and $K_{\beta'}$ transition from the 3p states

$K_{\beta 2,5}$ and $K_{\beta''}$ transitions from the valence band

Chemical sensitivity!

Mn K_{β} fluorescence



- Simple chemical characterization
- Data collection currently done with Crystal analyzers
- Possible use of LTD detectors if $\Delta E = \text{few eV}$
- Advantage in using LTD detectors: parallel acquisition, no mechanical movement

Variation of the K_{β} lines for various Mn compounds. Excitation well above the edge. (Tsutsumi et al. PRB **13** (1976), 930.)

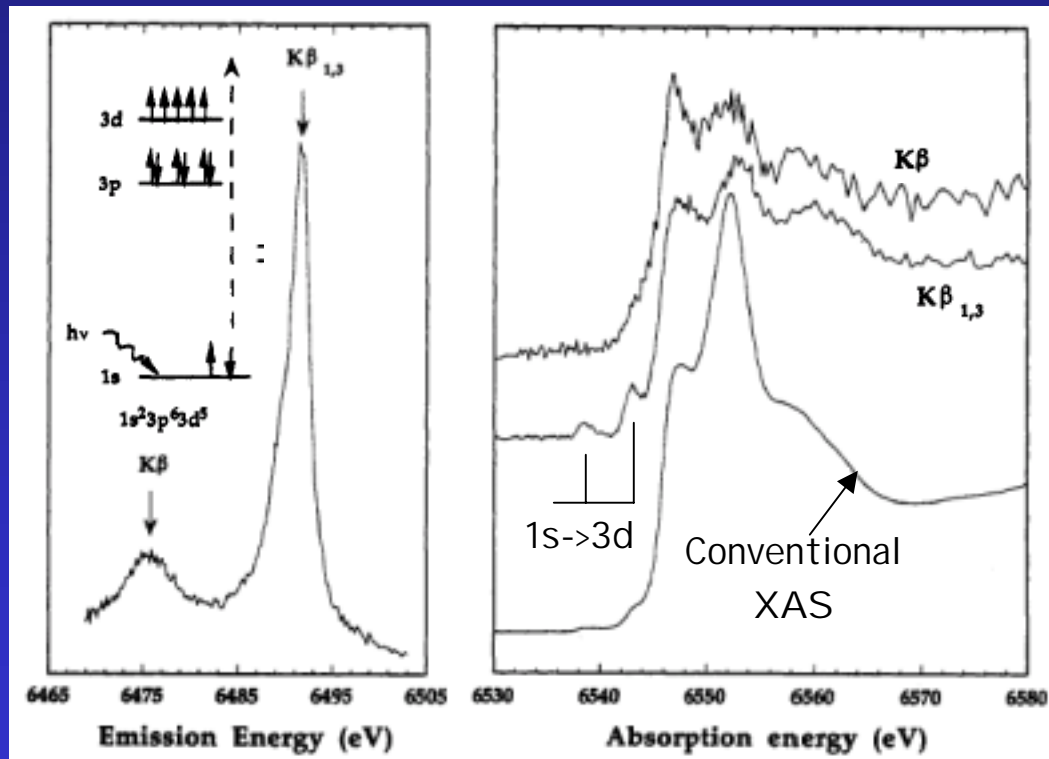


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Spin selective XAS



G. Peng et al. JACS 116 (1994), 2919



- In Mn^{II} compounds it can be shown that $K_{\beta 1,3}$ originates from a spin down electron, K_{β} from a spin up electron

- XAS spectra can be collected by separately monitoring the two lines

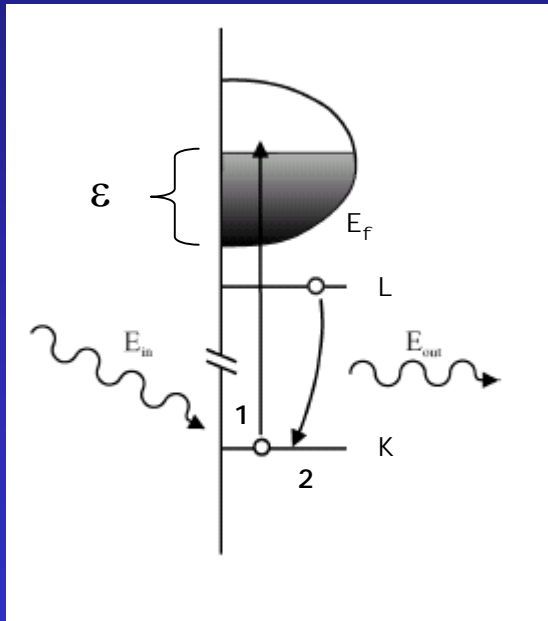
- Differences are observed that reflect the different DOS for the two spin configurations.

- Peak separation 16 eV at 6500 eV.



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Resonant Inelastic X-ray Scattering (RIXS)



- 1) Promotion of a K electron above E_f by photon absorption (virtual state, no energy conservation).
 - 2) Filling of the K hole with an L electron
 - 3) Emission of a photon
-
- The energy of the global process is conserved $E_{in} - E_{out} = \epsilon - E_L$
 - The process is resonant, i.e. enhanced near the (K) edge
 - Access to the electronic structure

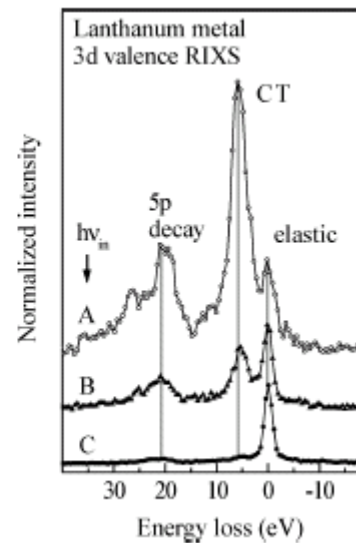
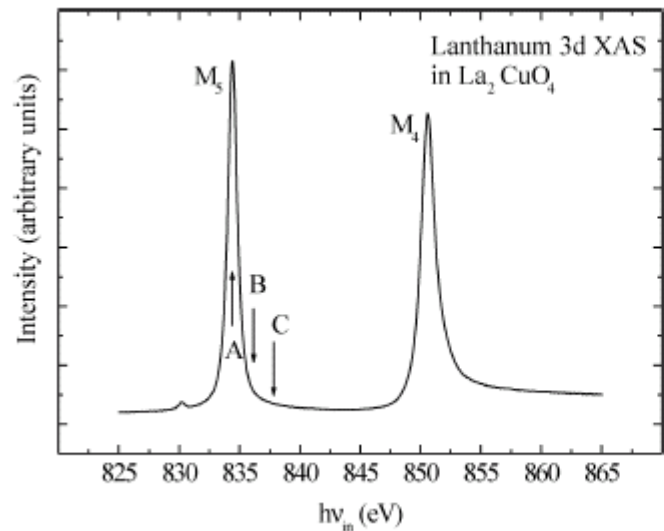


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RIXS



Dallera & Grioni STUC 14 (2003), 57



- Data collected with grating spectrometer

- Evidence of a Charge Transfer peak for an excitation energy on the white line

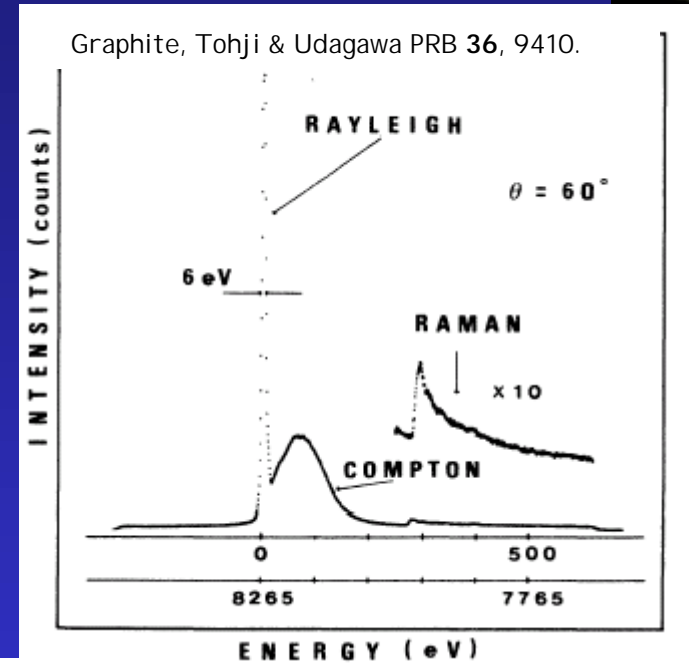
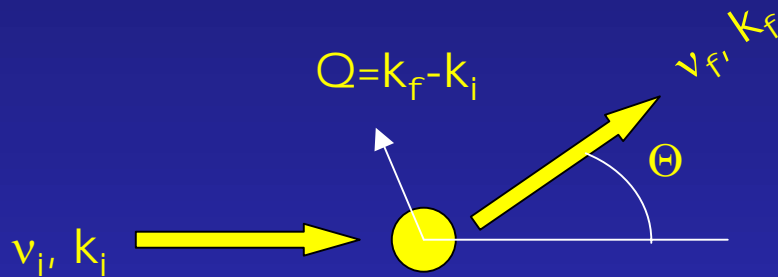
- The CT peak disappears for higher excitation energies

Peak separations 5-20 eV at 835 eV



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(Non resonant) X-ray Raman Scattering, XRS



Transition probability w

$$w \propto (1 + \cos^2 \Theta) \left| \langle f | \exp[i\vec{Q} \cdot \vec{r}] | i \rangle \right|^2 \delta[E_f - E_i - h(\nu_i - \nu_f)]$$

Same operator as XAS

$\langle f |, \langle i |, E_f, E_i =$ electronic states & related energies

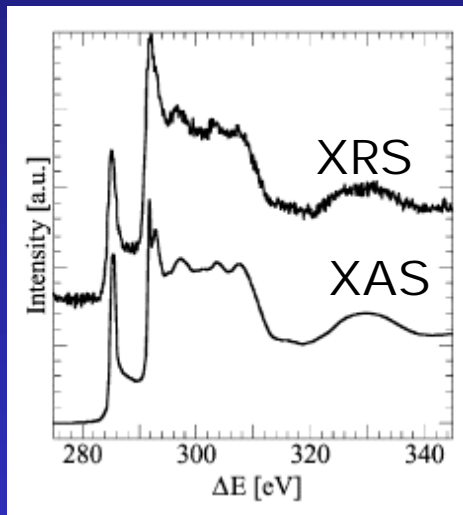


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XRS



C K edge, graphite 6 keV



U. Bergmann et al.
Microchem. J.
71 (2002) 221

- Useful for obtaining **bulk information on light atoms**: experiment is done at high energy.
- **1 eV energy resolution** is OK for data collection.
- Signal **significantly smaller** than Elastic / Compton



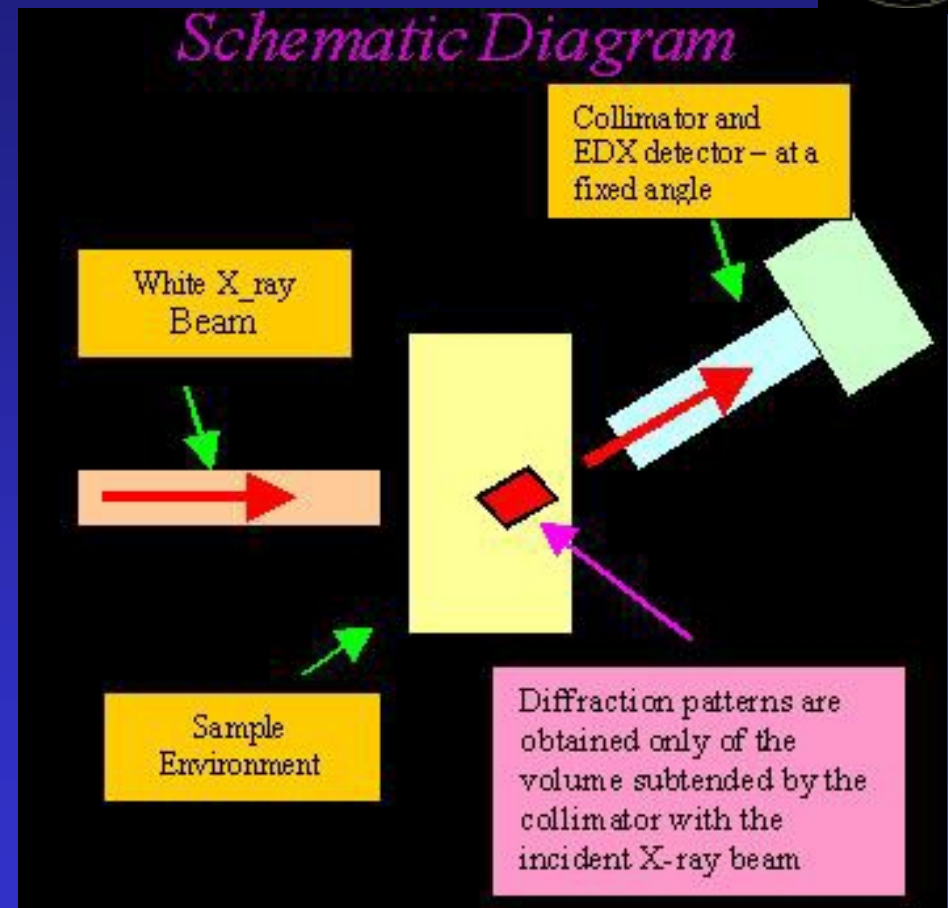
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Energy Dispersive X-ray Diffraction (EDX)



Bragg law: $\lambda = 2d \sin \Theta$

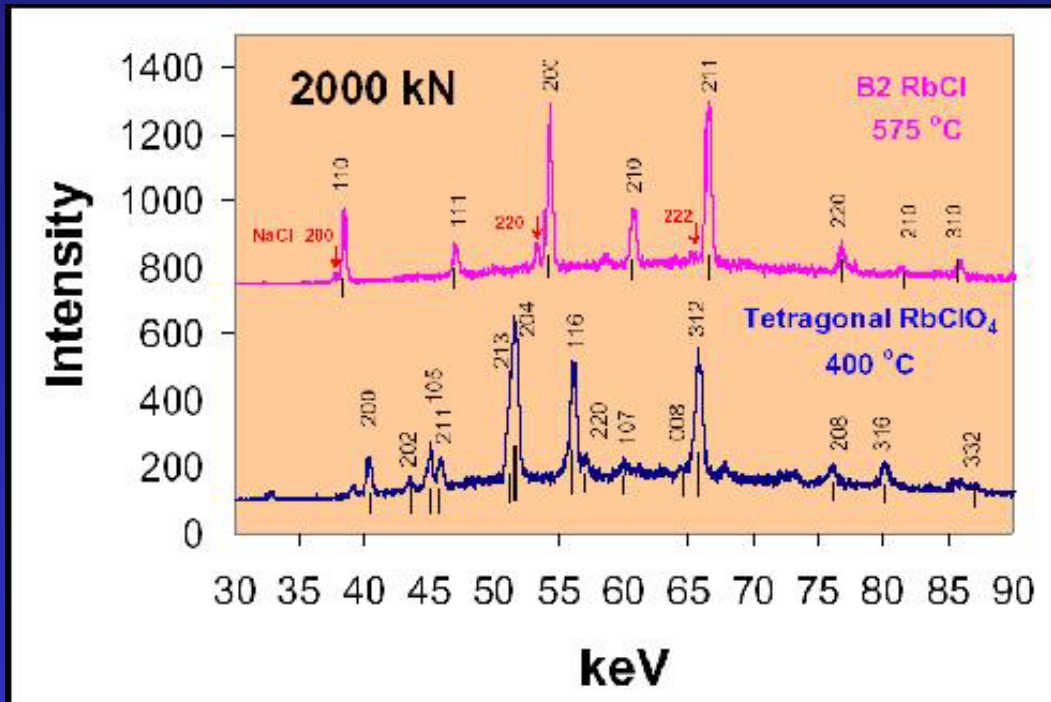
- Normally λ is fixed and Θ is varied
- In EDX Θ is fixed and λ is varied





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EDX



Typical EDX spectra

Used in the early days of SR for experiments needing small sized beams (High Pressure).

Limitations

- Energy resolution
- Count Rate

Application of LTD detectors to small beams



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Conclusion



- High resolution LT-detectors match the requirements for X-ray absorption spectroscopy & fluorescence analysis



Rise the maximum count rate
Extend to hard X-rays

- Possible use for X-ray emission spectroscopy & Raman scattering



Improve the energy resolution down to 1 eV.

- EDX ?