

Electron Probe MicroAnalysis for Nuclear Science

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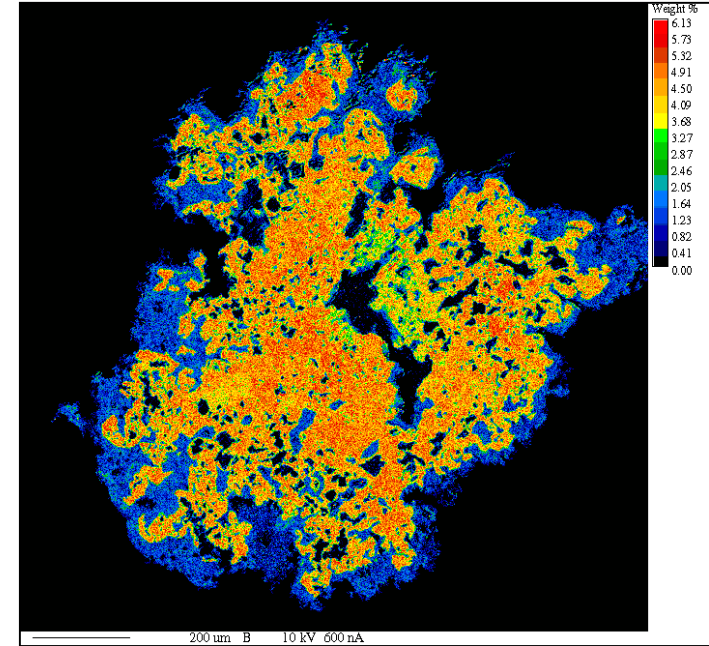
Thanks to **Pranesh Sengupta** from BARC, Mumbai for sharing his experience with us.

- EPMA Introduction
 - *Brief EPMA Intro*
 - *SXFive TACTIS and Skaphia*
 - *Wavelength Dispersive Spectrometry*
- Waste management work at BARC on non-shielded EPMA
- Conclusion

The World Leader
in Elemental and Isotopic
Micro & Nanoanalysis



- Determine concentration of element
- Show distribution of these elements in solid specimen



- Direct measurement
- Non destructive technique
- True quantitative analysis for wide materials needs
Covers the whole periodic table from Lithium
- 100ppm (0.01wt%) detection limit (heavy elements)
- 300 to 500ppm (0.05wt%) detection limit (light elements)

Field of view: **1.3mm x 1.3mm**

Stage Motion

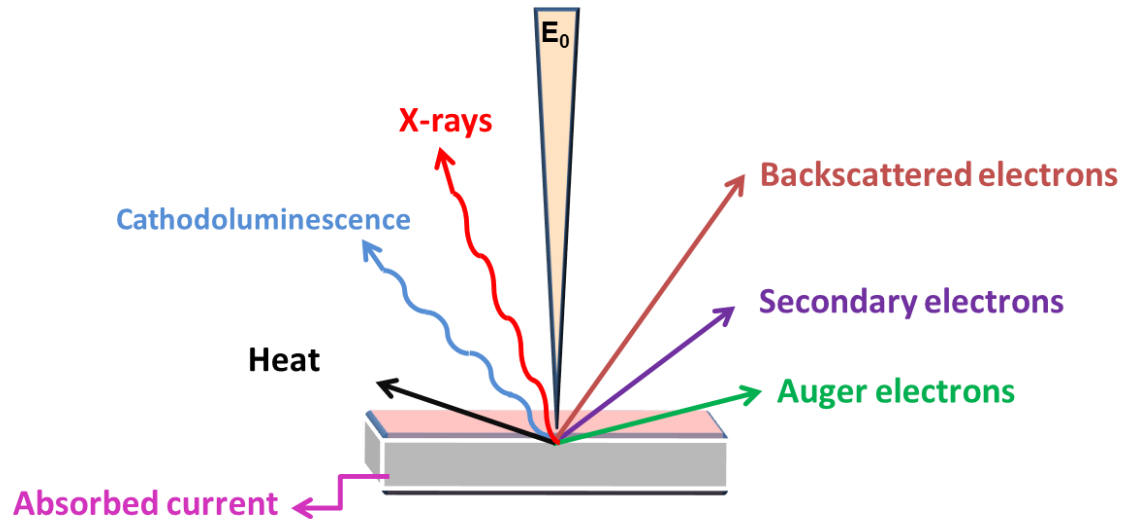
Acquisition time: **7h**

10 keV, 600 nA

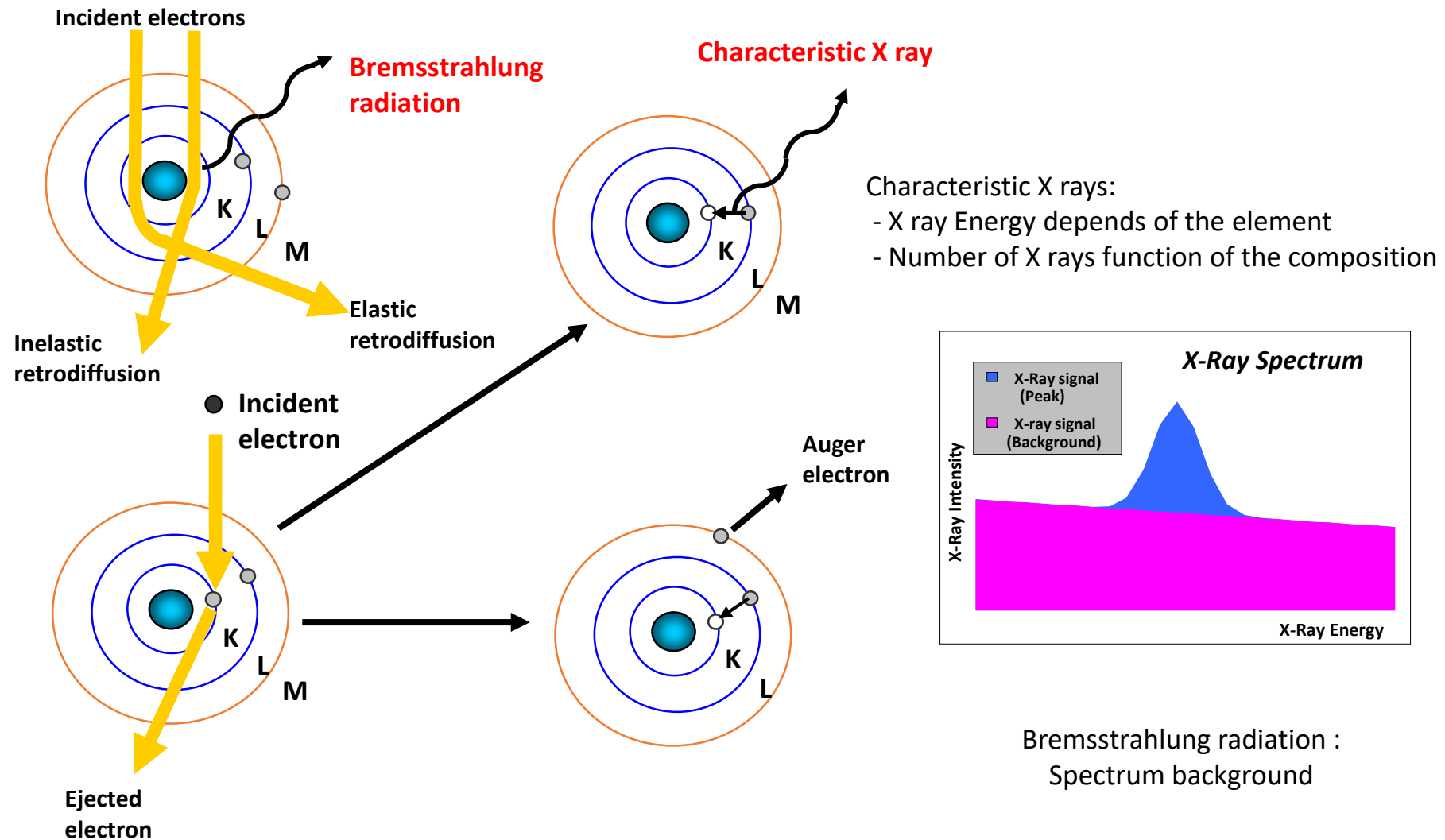
650 x 650 pixels

100 ms per step

By courtesy of
Piotr Dzierzanowski
Academy of Sciences Warsaw, Poland



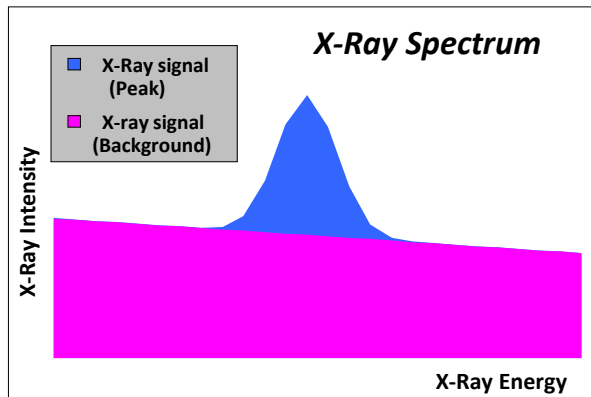
- *Inelastic interactions generate:*
 - *Secondary electrons (SE): $E_K < 50\text{eV}$ (convention)*
 - *Cathodoluminescence (CL) (light emission): $\sim\text{eV}$*
 - *Continuum x-ray radiation (Bremsstrahlung): $10\text{eV}-100\text{keV}$*
 - *Inner-shell ionization (characteristic X-rays, Auger electrons): $10\text{eV}-100\text{keV}$*
- *Elastic interactions:*
 - *Backscattering electrons (BSE): $50\text{eV} < E_K < E_0$*



Why make a shielded EPMA?

Characteristic X rays:

- X ray Energy depends of the element
- Number of X rays function of the composition



Bremsstrahlung radiation :
Spectrum background

A radioactive material triggers
X-ray generation **without the electron beam ON**

→ Specimen activity creates additional background

→ CAMECA shields EPMA for these specific applications

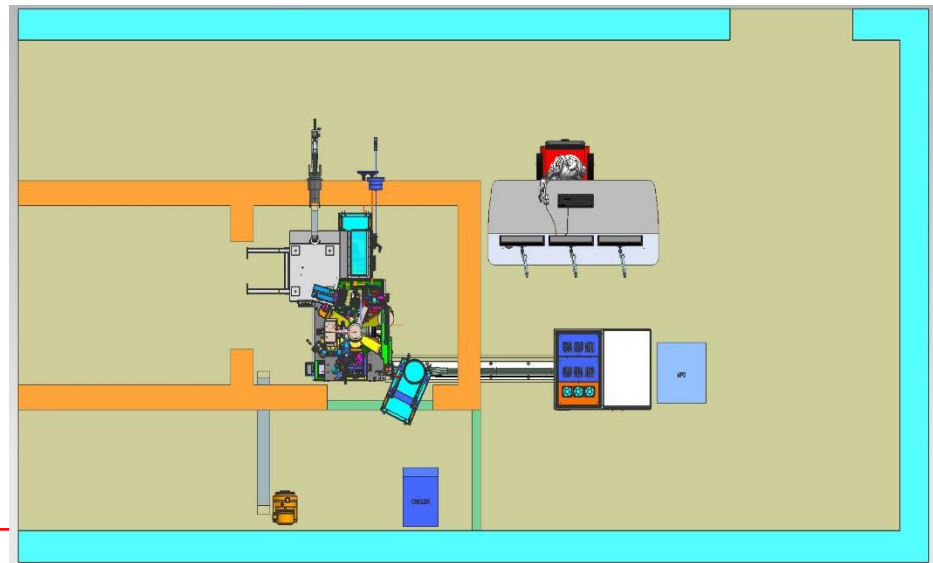
Standard EPMA: SXFiveTACTIS

- Standard sample
- Low activity samples

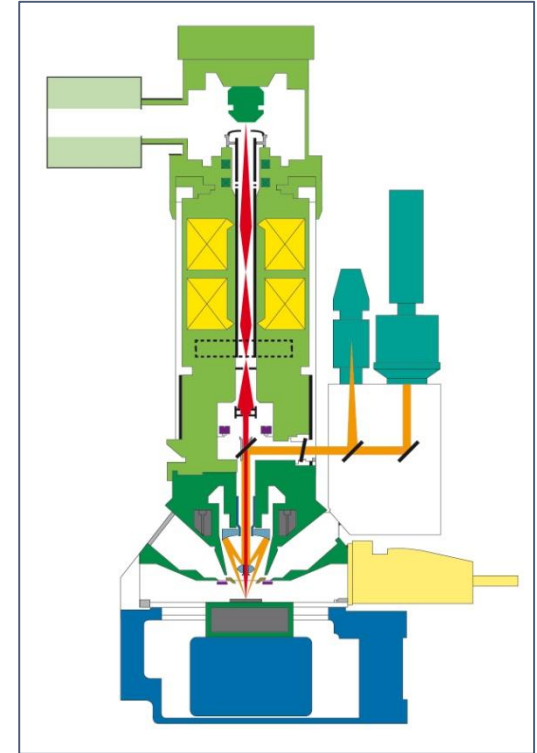
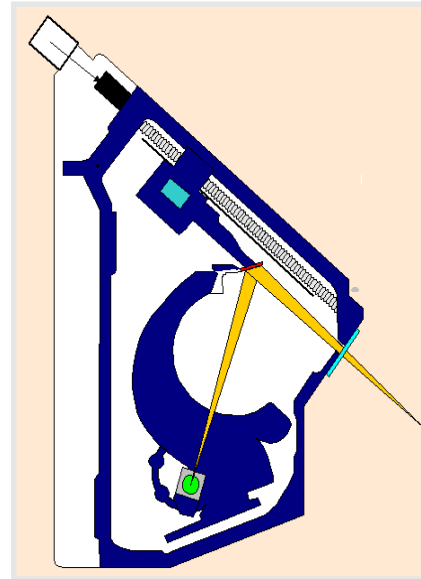


Shielded EPMA: SKAPHIA

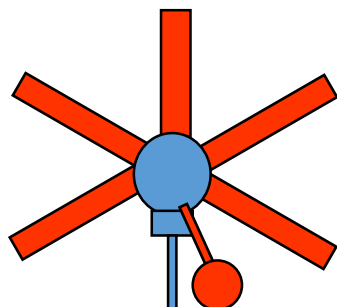
- HOT samples (max of 111 GBq at an energy of 0.75 MeV.)



- **Electron Column**
- **X Ray Spectrometer**
- **Vacuum Chamber**
- **Automation System**

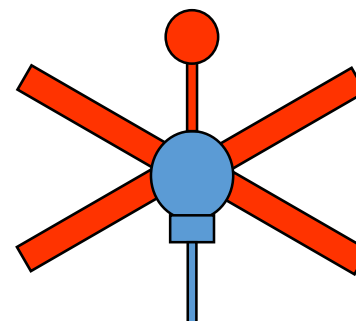


5 WDS for TACTIS and 4 WDS for SKPAHIA : optimized quantifications



TACTIS

Configuration with 5 vertical WDS and 1 EDS



SKAPHIA

Configuration with 4 vertical WDS and 1 EDS

Each spectrometer / channel can host up to 2 to 4 crystals, acting as energy filter

Large filters allow faster collection time

	2d (nm)	Shape	Counter pressure	10	20	30	40	50	60	70	80	90
TAP	2.576	J	LP	9F 15P		25Mn 41Nb			57La 80Hg			
LTAP	2.576	JS	LP	9F 15P		25Mn 41Nb			57La 80Hg			
Ext.TAP Ext LTAP	2.576	J JS	LP	9F 15P		25Mn 41Nb			57La 80Hg			
PET	0.8762	J	LP or HP (better)		14Si 25Mn		38Sr 65Tb				73Ta	
LPET	0.8762	JS	LP or HP (better)		14Si 25Mn		38Sr 65Tb				73Ta	
LiF	0.4027	J	LP or HP (better)			21Sc 37Rb			52Te			
LLiF	0.4027	JS	LP or HP (better)			21Sc 37Rb			52Te			
(L)PC0	4.5	J	LP	7N 11Na								
(L)PC1	6.0	J	LP	9C 9F								
(L)PC2	10.0	J	LP	5B 9O								
(L)PC3	20.0	J	LP	4B 5B								
L Boron	14.5	J	LP	5B								
L Nitrogen	8	J	LP	7N								

Advantages of CAMECA High Intensity crystals:

- P/B ratio is the same for small and large crystals
- No limitation in the analytical range
- No loss of spectral resolution thanks to the Johansson geometry

Crystals:

L: large

J: Johansson geometry

JS: Johansson geometry

AMETEK
MATERIALS ANALYSIS DIVISION

Counter Pressure:

LP: low pressure

HP: high pressure

Horizontal spectrometers: LP only

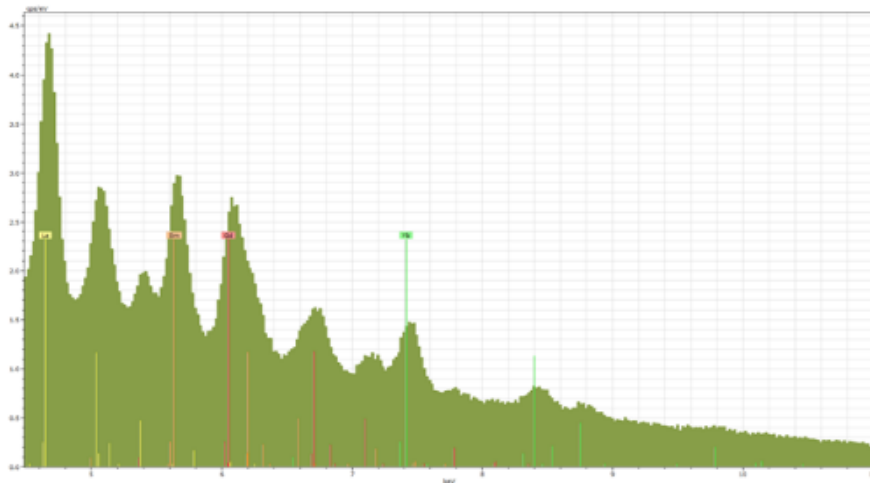
K lines

L lines

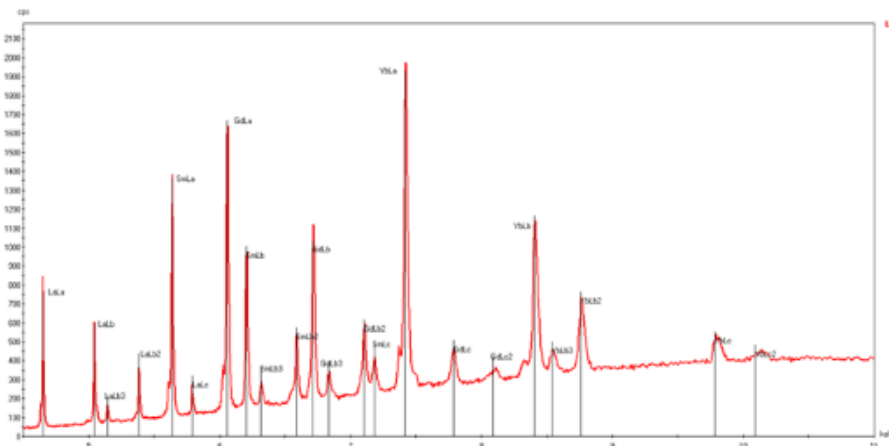
M lines

EDS versus WDS

A different league in terms of energy resolution



EDS: 121V energy resolution on the Mn



WDS: 20eV on the Mn



24 eV difference peak to peak

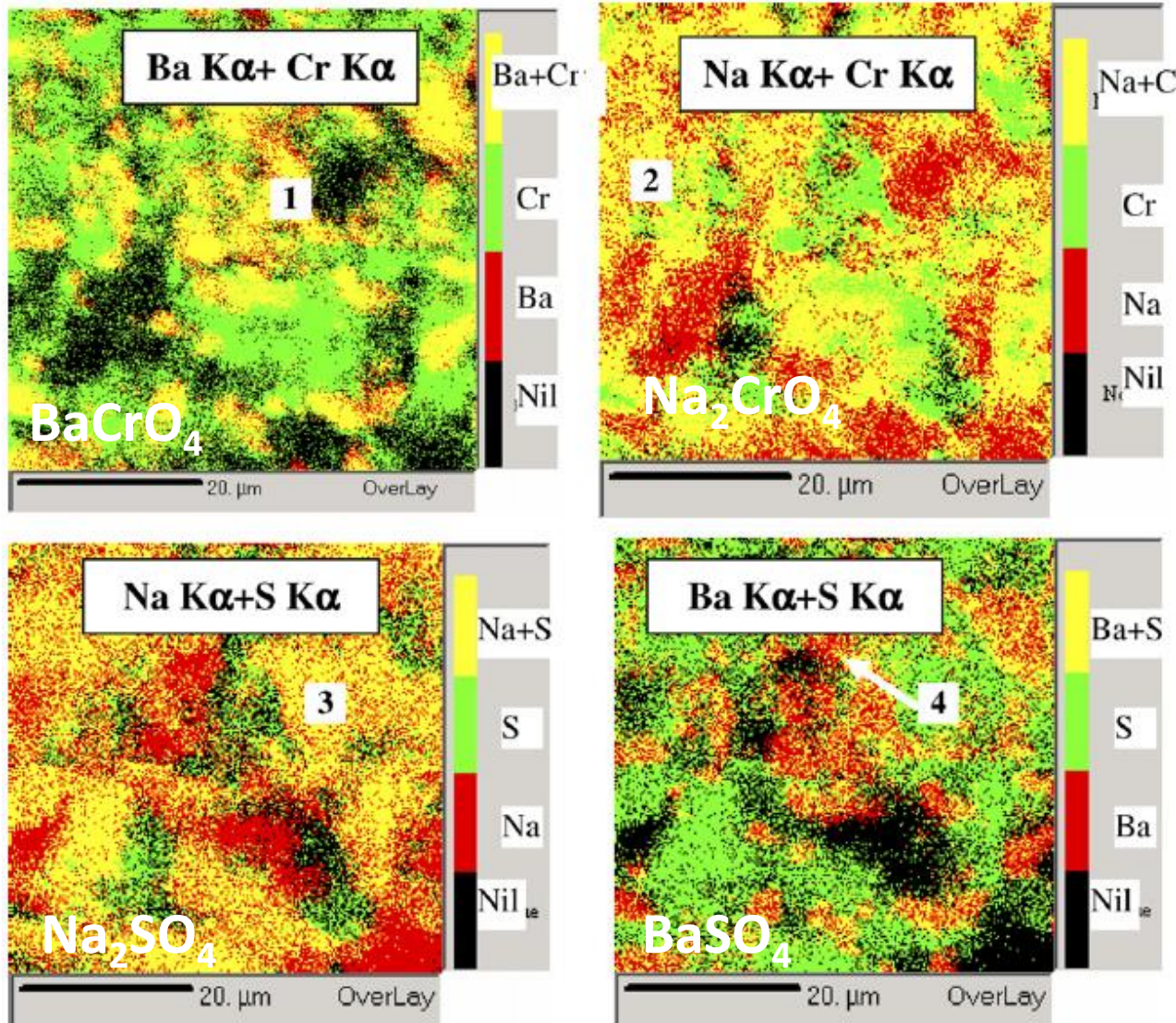
57	58	59	60	61	62	63	64	65	66	67	68	69	70
La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb
88	90	91	92	93	94	95	96	97	98	99	100	101	102
Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No

In irradiated nuclear fuel analysis, the most important elements to take into consideration are **U** and **Pu** as well as desintegration elements such as americium (**Am**), curium (**Cm**), and to a lesser extent, xenon (**Xe**) and Rare Earth Elements (REE).

- **High level nuclear liquid waste**
 - Select adequate Barium borosilicate glass for sulfate
- Selection of suitable glass matrix is challenging: EPMA is part of the suite of instruments to analyze and select the matrix.
- Homogeneity / chemical durability / pouring temperature
- Use simulated waste
- EPMA: assess chemical homogeneity of the glass sample

*Barium Borosilicate glass – a potential matrix for immobilization of sulfate bearing high-level radioactive liquid waste. C.P. Kaushik et al., **Journal of Nuclear Materials** 358 (2006) 129-138*

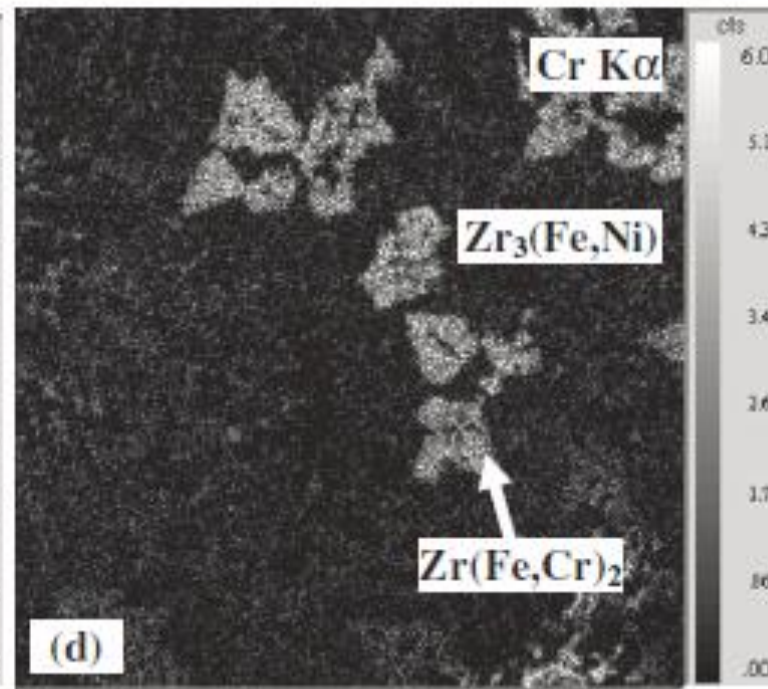
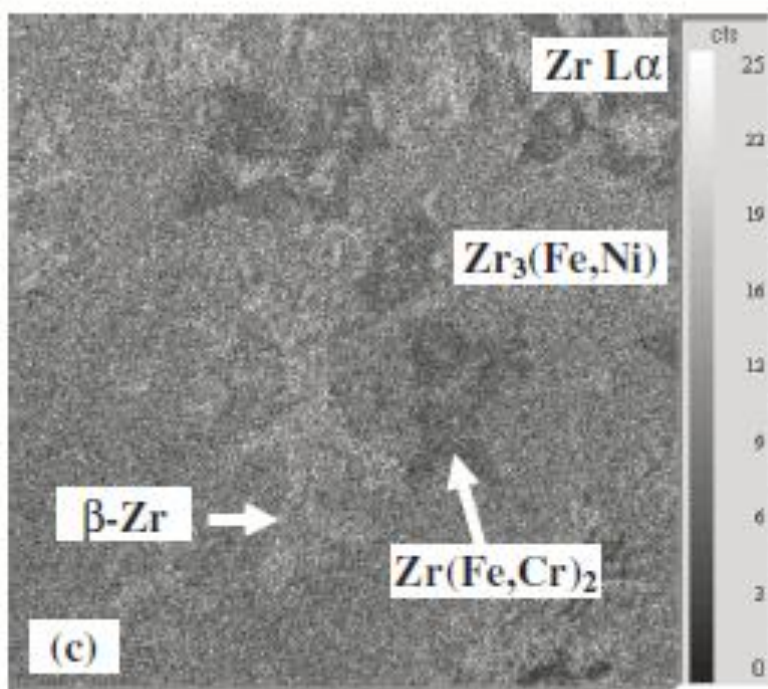
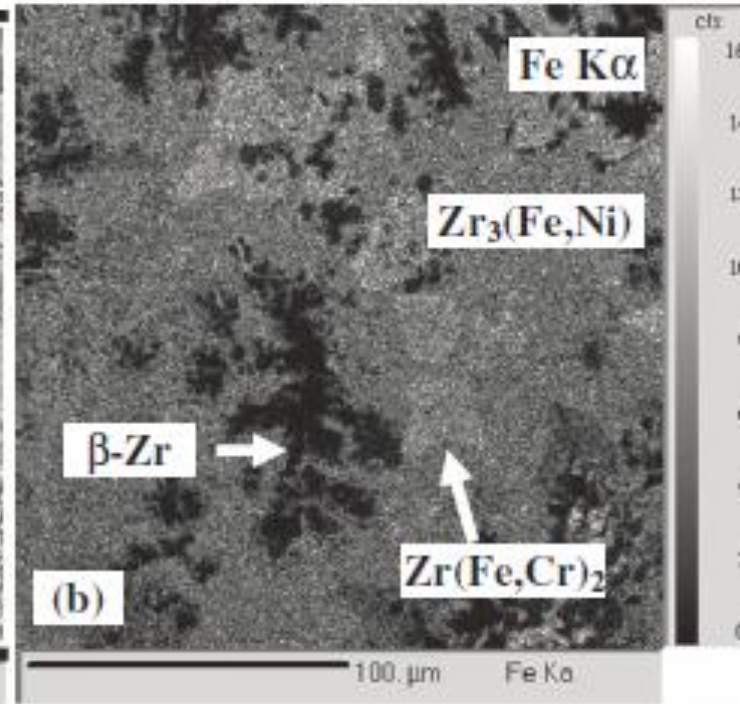
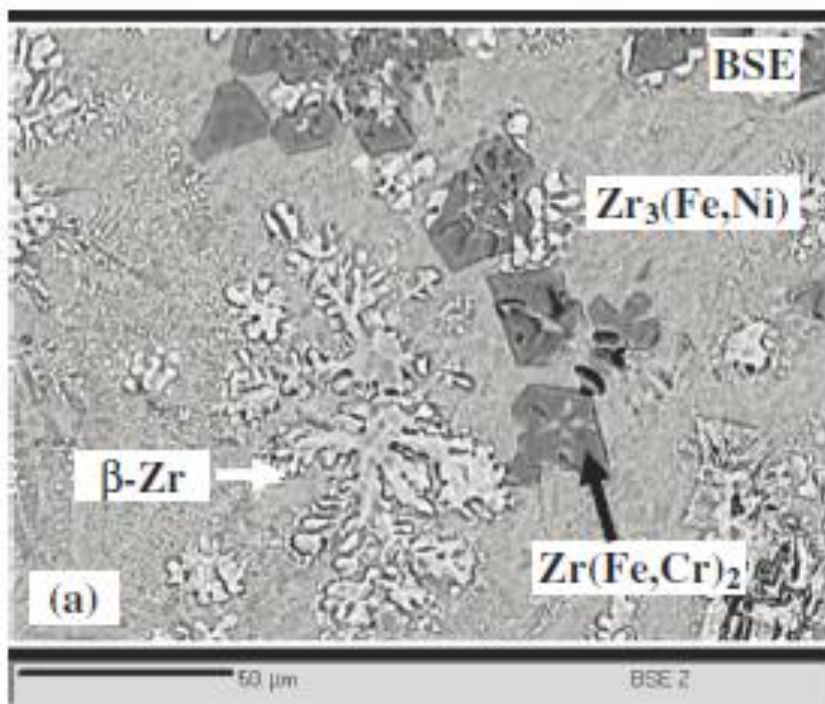
Phase identification in BBS glass



Elemental mapping
+
Overlay
→ Phase
identification
and distribution

- **High level nuclear metallic solid waste**
 - Target Deep geological repository
 - India chose the melting route
 - Evaluation of alloy composition identification for immobilization of radioactive Zr and stainless steel.
 - Considered Zr-Fe binary phase diagram as a pseudo binary to represent representative of Zircaloy-2-stainless steel 304 L.
 - Preparation and investigations of several binary / ternary / quaternary alloys for cross checking.

*Metallurgical characterizations of Fe-Cr-Ni-Zr base alloys developed for geological disposal of radioactive hulls, N. Das et al, **Journal of Nuclear Materials**, 420 (2012) 559–574*



- EPMA: Quantification of elements in a solid specimen: can be used in the characterization suite of instruments for nuclear projects on hot and cold sample
- 2D elemental and phase mappings
- Rely on WDS for steady quantification
- Main strength is trace analysis measurements
- India is a strong customer of EPMA instruments with roughly 25 active systems
 - On going deliveries at BARC and IGCAR.

Thank You

and

Dhanyavad !!!