ELECTRON PROBE MICRO ANALYSER (EPMA)

The **Central Research Facility (CRF)** of Indian School of Mines, Dhanbad has installed Fifth Generation Electron Probe Micro Analyser "**SX Five"** from **CAMECA,** France equipped with five Wave length dispersive Spectrometers, BSE Detectors, SE Detectors, cathodoluminance and sophisticated visible light optics providing image magnification ranging from 40 to 400,000.

SPECIFICATIONS:

SX- Five with W Column:

Maximum Accelerating Voltage: 30KV
Beam Diameter in Analytical Mode: 600 nm @ 15 KV, 100nA
: 400 nm @ 10 KV, 10nA
Maximum Beam Current: Up to 10 uA
Beam Stability: +-0.5% per hour @ 20kV, 20nA

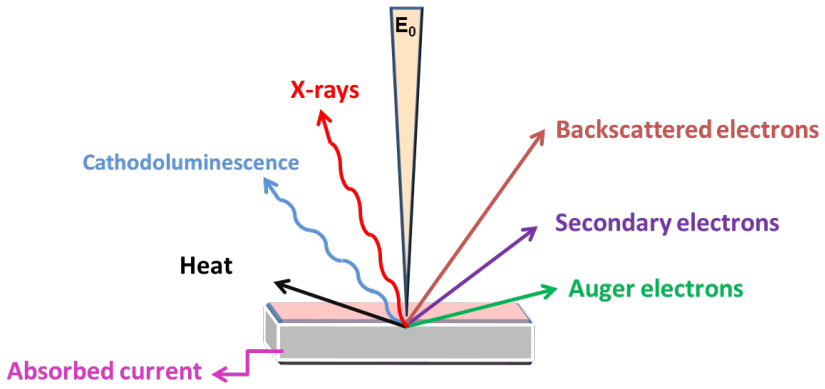
SX- Five with LaB6 Column:

Maximum Accelerating Voltage: 30KV
Beam Diameter in Analytical Mode: 100 nm @ 20 KV, 10nA
: 200 nm @ 10 KV, 10nA
Beam Current: Up to 1 uA
Stability: +-0.2% per hour @ 20kV, 10nA

Maximum Beam

BASIC PRINCIPLE:

EPMA works by bombarding a micro-volume of a sample with a focused electron beam (typical energy = 5-30 keV) and collecting the X-ray photons thereby emitted by the various elemental species. Because the wavelengths of these X-rays are characteristic of the emitting species, the sample composition can be easily identified by recording WDS spectra (Wavelength Dispersive Spectroscopy). WDS spectrometers are based on the Bragg's law and use various moveable, shaped monocrystals as monochromators.



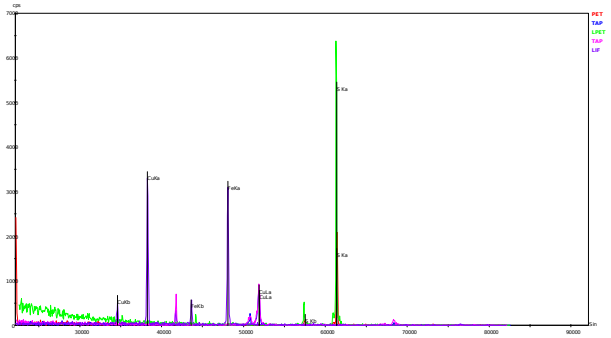
- EPMA is a fully qualitative and quantitative method of **non-destructive elemental analysis of micron-sized volumes** at the surface of materials, with sensitivity at the level of ppm. Routine quantification to 1% reproducibility is obtained over several days. It is the most precise and accurate micro-analysis technique available and **all elements from B to U and above** can be analyzed.
- EPMA is fully compatible with routine analysis sessions, with easy and direct interpretation of the results.

- EPMA instruments are equipped with a complete kit of built-in microscopy tools that allow **simultaneous X-ray (WDS), SEM and BSE imaging**, plus sophisticated visible light optics; they provide very flexible sample inspection with image magnification ranging from 40 to 400,000.
- Determination of thickness and elemental composition from nm to mm thick layers in stratified materials is possible.
- EPMA provides much better results than standard SEM/EDS systems. Because of the internal properties of WDS, the general sensitivity, analysis of light elements and risks of erroneous interpretation of qualitative spectra are all superior with EPMA. Spectral resolution and detector dead time are much better than EDS (Energy Dispersive Spectroscopy).
- The excitation beam regulation system and sophisticated sample stage capabilities guarantee that this technique provides outstanding stability and measurement repeatability.

USES:

1.Qualitative analysis: WDS Spectrum-To find out the elements present

Wavelength Dispersive Spectrometry is acknowledged as the method of choice for high precision quantitative microanalysis. The SX Five spectrometers have the highest resolution and, as a result, high peak to background ratios, thereby providing high sensitivities. The absolute spectral positioning is provided by optical encoders attached to the system. Only one peak measurement is needed to calibrate the entire spectrometer range.



2. Quantitative analysis: Accurate Quantification of Chemical composition of any solid Material

The SXFive deliver the true quantitative analysis. The quantitative data can be displayed such as wt%, oxide wt%, cations. See the table below for example:

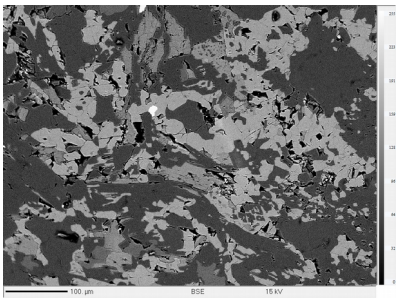
Na_2O	Al_2O_3	F	MgO	SiO_2	K2O	CaO	Cl	TiO_2	FeO	MnO	P_2O_5	Cr_2O_3	Total
-0.04	20.62	0.01	6.84	39.12	0.01	6.07	0.05	0.04	25.62	1.04	-0.04	0.02	99.33
2.31	31.53	0.07	0.02	48.81	0.02	16.38	0.02	0.01	0.16	0.05	0.06	0.01	99.3
-0.01	1.75	0.02	21.02	52.96	0.02	0.34	0.02	0.06	24.24	0.38	0.09	0.02	100.89

3. BSE Imaging: (Phase Identification, Mineral Identification) Backscattered electrons are high energy primary electrons scattered in such a direction (>90°) that they leave the target entirely. Most BSE have energies slightly lower than that of the primary electron beam i.e. PE scattered elastically. By using these emission as a video signal we can produce BSE Image.

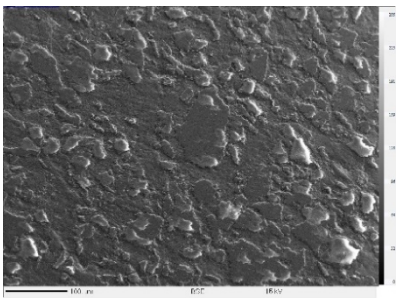
4. SE Imaging (Use: Topography,): Secondary electrons are inelastically scattered primary electrons and produced as a result of interactions between the beam electrons and weakly bound conduction

electrons and outer shell valence band electrons in semiconductors or insulators. By using these emission as a video signal we can produce SE Image.

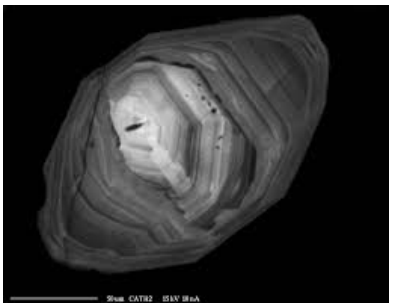
5. Cathodoluminance Imaging (Use: Reveals the defects and impurities in materials**):** Cathodoluminescence (CL) (non-metal valence shell phenomenon leading to light emission). By using these emission as a video signal we can produce Cathodoluminance Image.



BSE Image

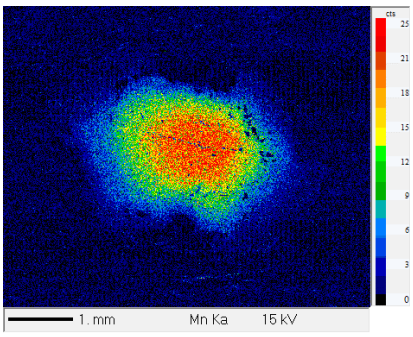


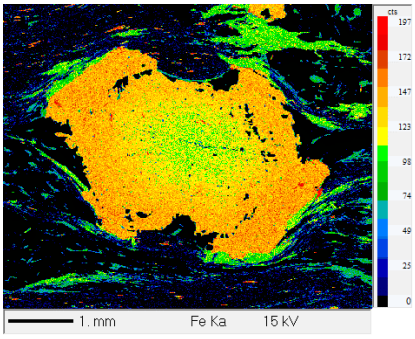
SE Image

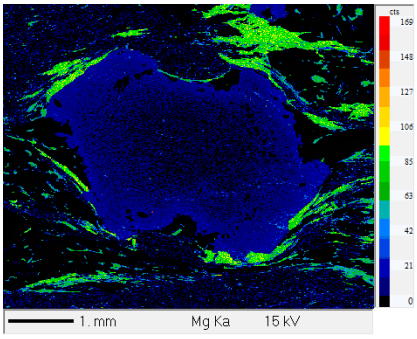


CL Image

6. X-Ray Mapping: Elemental distribution: With the help of X-Ray Mapping one can see the distribution the particular element in desired area of interest and segregation of impurities in metal alloy samples.



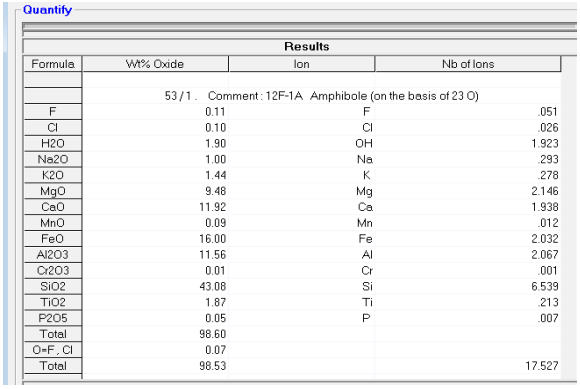




7. Mapping Quant: The Mapping Quant option performs the true quantification of X-Ray Mappings. Based on the images Acquisition on peak and backgrounds, the mapping quant procedure requires from the user the selection of the calibration for each measured element. The raw x-ray data will be processed in a true quantification procedure with a correction matrix applied pixel by pixel to the whole image. The results are quantified X ray maps displayed in weight %, Atomic% or Oxide%. After quantification, the measurement concentration at each pixel or for a set of pixels is available.

8. Geo Quant : Cation Calculation

With the help of Geo quant Software it is easy to do the cation calculation. Software does it automatically after providing the kind the mineral.



Quantify

Results

Formula	Wt% Oxide	Ion	Nb of Ions
	53 / 1 . Comment : 12F-1A Amphibole (on the basis of 23 O)		
F	0.11	F	.051
Cl	0.10	Cl	.026
H2O	1.90	OH	1.923
Na2O	1.00	Na	.293
K2O	1.44	K	.278
MgO	9.48	Mg	2.146
CaO	11.92	Ca	1.938
MnO	0.09	Mn	.012
FeO	16.00	Fe	2.032
Al2O3	11.56	Al	2.067
Cr2O3	0.01	Cr	.001
SiO2	43.08	Si	6.539
TiO2	1.87	Ti	.213
P2O5	0.05	P	.007
Total	98.60		
O=F, Cl	0.07		
Total	98.53		17.527

9. Age Quant : Monazite Dating

Monazite (LREE phosphate) is remarkable robust mineral for geochronology, and is commonly measured by conventional isotopic methods. However, multiple ages are often recorded within a single monazite grain and on a spatial scale smaller than can be resolved by even small-spot isotopic instruments. Because monazite crystalline with significant Th and U and negligible original Pb, it is possible to use nondestructive EPMA analysis for accurate in situ measurement of Th, U and Pb in order to determine its age of crystallization. Given below are some examples of different ages of standard samples analysed with this EPMA:

PbO	ThO_2	UO_2	Total	Age (Ma)	Age err (±)
0.23	15.94	1.3	100.61	276	27
0.24	15.83	1.3	100.13	287	28
0.34	6.37	0.22	99.48	1089	42
0.29	4.73	0.25	100.7	1180	52
0.44	7.24	0.41	100.5	1181	48

MAIN APPLICATIONS IN VARIOUS FIELDS:

1. **Geology**
   - Crystal chemistry
   - Pressure, Temperature of equilibration
   - Mineral prospecting
   - Geochronology
2. **Metallurgy**
   - Trace analysis : C, N, ...
   - Segregation
   - Phase equilibrium
   - Non-destructive thin film analysis
3. **Semi conductor industry**
   - Defect analysis
   - Particle analysis
   - Thin Film analysis
   - Forensic sciences

4. Particle analysis