Material Characterization Techniques

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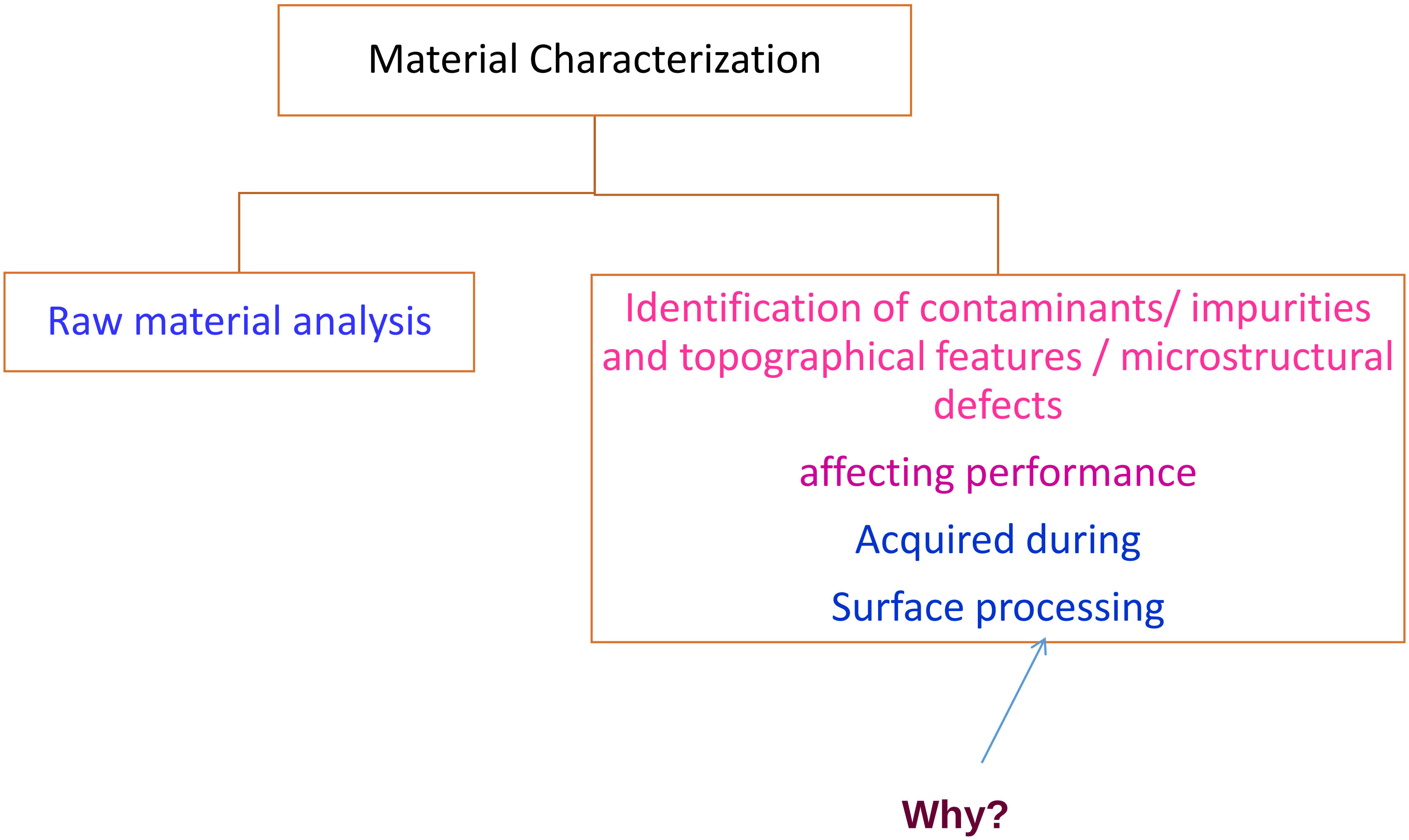
18 July 2017

Material Characterization - Definition

- “Characterization” word describes those features of composition and structure (including defects) of a material that are significant for a particular preparation, study of properties, or use, and suffice for reproduction of the material.

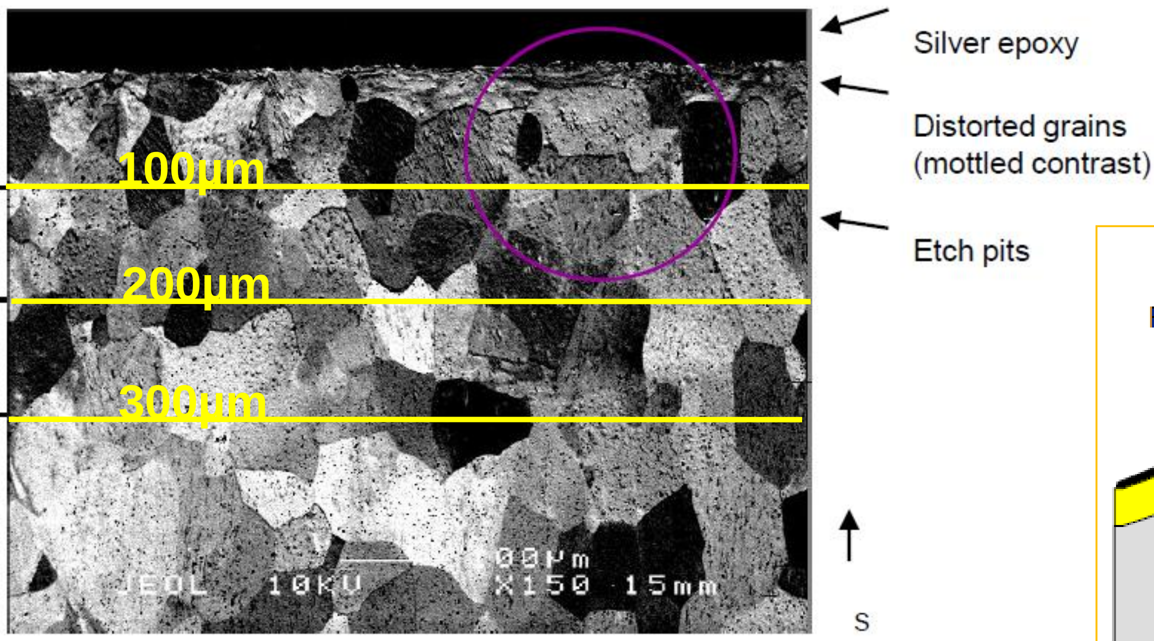
- *Its relevance to SRF cavity Technology……*

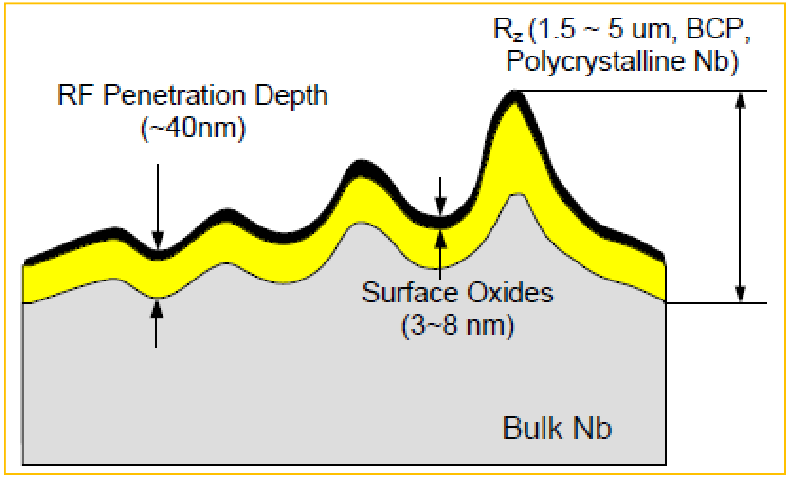
Material characterization for SRF cavity - Categorization



Why Surface processing

1. Surface gets damaged and contaminated during deep drawing operation.
2. RF penetration depth is very shallow.





Ref – C Z Antoine, R Crooks – SRF 2009 poster

Surface processing - Purpose

- Hence the requirement of removal of damage layer through surface processing techniques to improve the cavity performance.

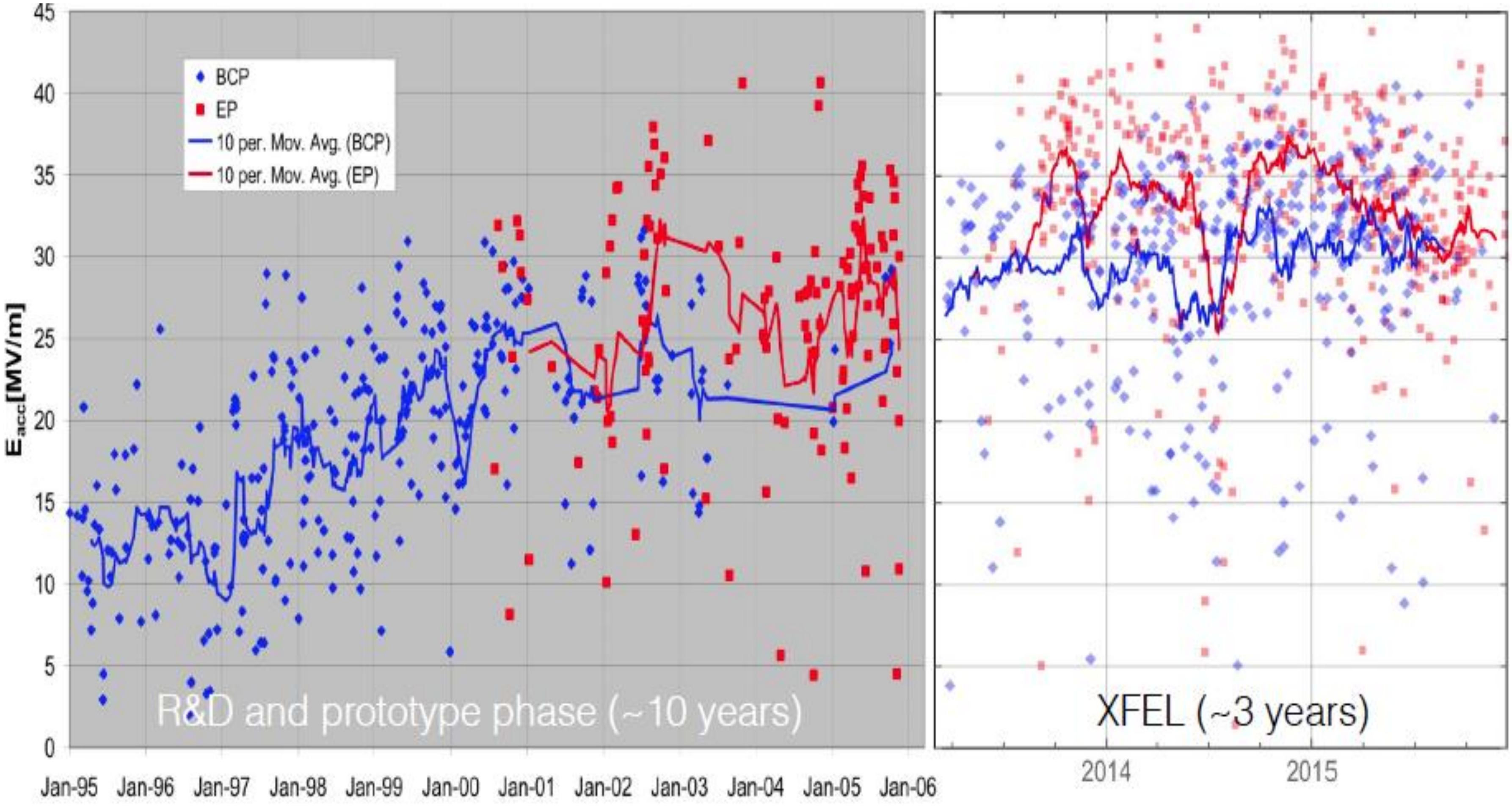
- Performance of SRF cavities is defined by two parameters

  Quality factor (Q_0) & Accelerating gradient (E_{acc})

Quality factor (Q_0) & Accelerating gradient (E_{acc}).

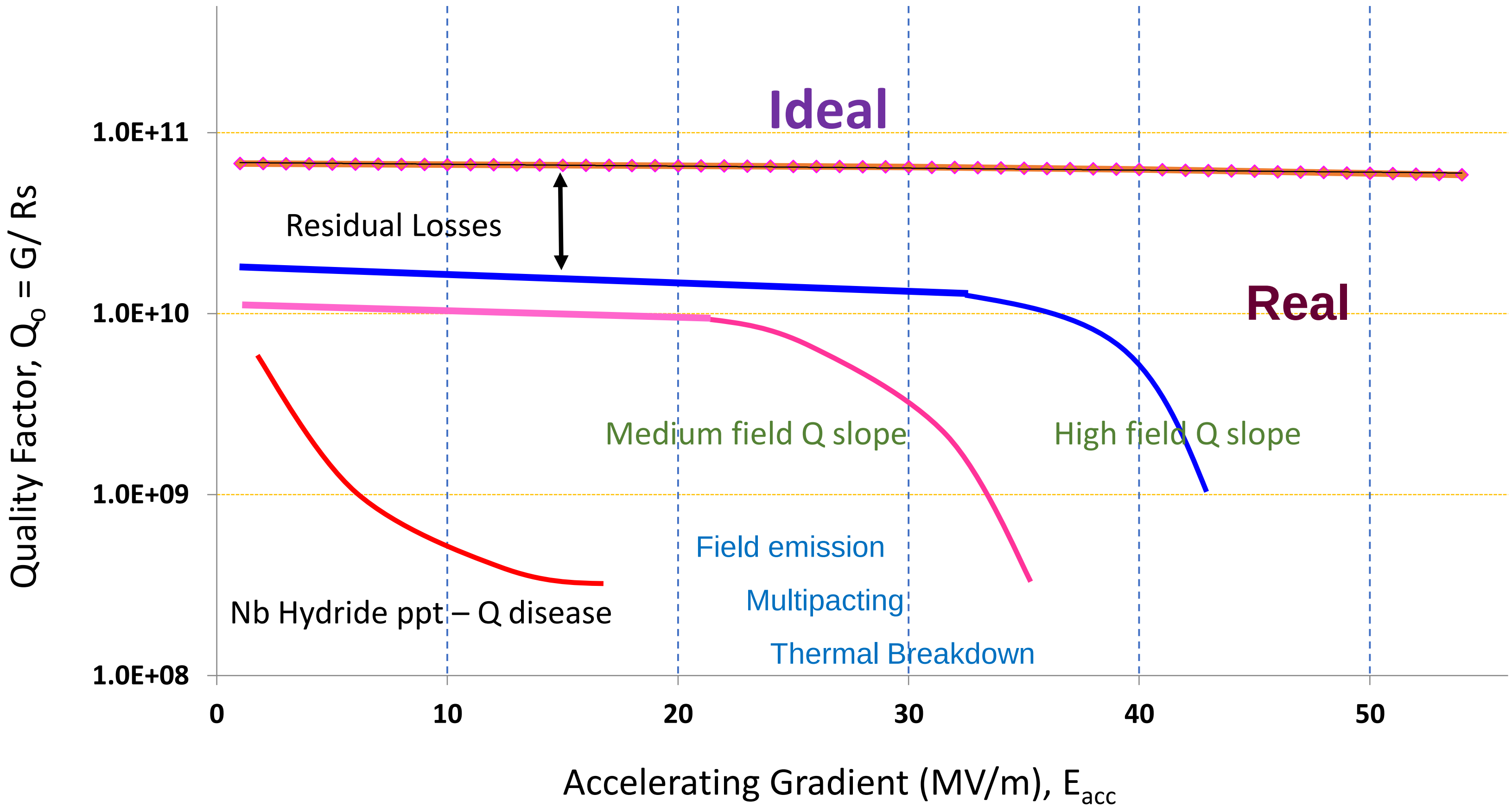
- RF power dissipates through the walls of the cavity due to the existence of a finite surface resistance (R_s).
- Q_0= Geometry factor/R_s
- Higher the Q_0 lesser is the power consumption. (R_s = 10nΩ then Q = 2.5e10)
- R_s depends on many factors and results in strong degradation of Q_0.
- **Hence the target is to minimize R_s.**

- Higher acceleration gradient is equivalent to shorter the Superconducting linac structure.
- E_{acc} = f (Critical magnetic field (H_c) of the superconducting Nb)
- E_{acc} ~55MV/m
- Since Hc is fixed, all cavities should reach E_{acc} before quenching.
- **To target 55MV/m ……BUT……**



Timeline of XFEL cavity performance at DESY

Ideal vs Real performance



Steady progress can be made after limiting phenomena are understood and effective cures are developed

Sources of limitation for Q (Rs) and Eacc (Hc)

- Impurities
  - Surface contaminations like residues, dust, particles, bacteria -leads to FE, MP, residual resistance ↑ – Limits Q and E
  - Sub surface impurities like Oxides - Affects FE, residual resistance ↑- Limits Q and E
  - Inside penetration depth like acidic impurities, Hydrides, Carbon, Inclusions - Limits Q and E
  - Bulk impurities – thermal conductivity ↓ – Limits E
- Surface topography
  - Roughness – Limits E
  - Pits, cracks, facets, grain boundary steps etc. – Limits E
- Microstructural defects
  - Dislocations, grain boundaries, vacancies –residual resistance ↑, hydrogen trapping ↑ - Limits Q and E

Characterization techniques - categories

- Surface, sub surface contaminations and in depth impurity distribution
  - Secondary ion mass spectrometry (SIMS)
  - X-ray photo electron spectroscopy (XPS/ESCA)
- Topographical and microstructural defects
  - Laser scanning confocal microscopy (LSCM)
  - Scanning and transmission electron microscopy (SEM & TEM)

Secondary ion mass spectrometry

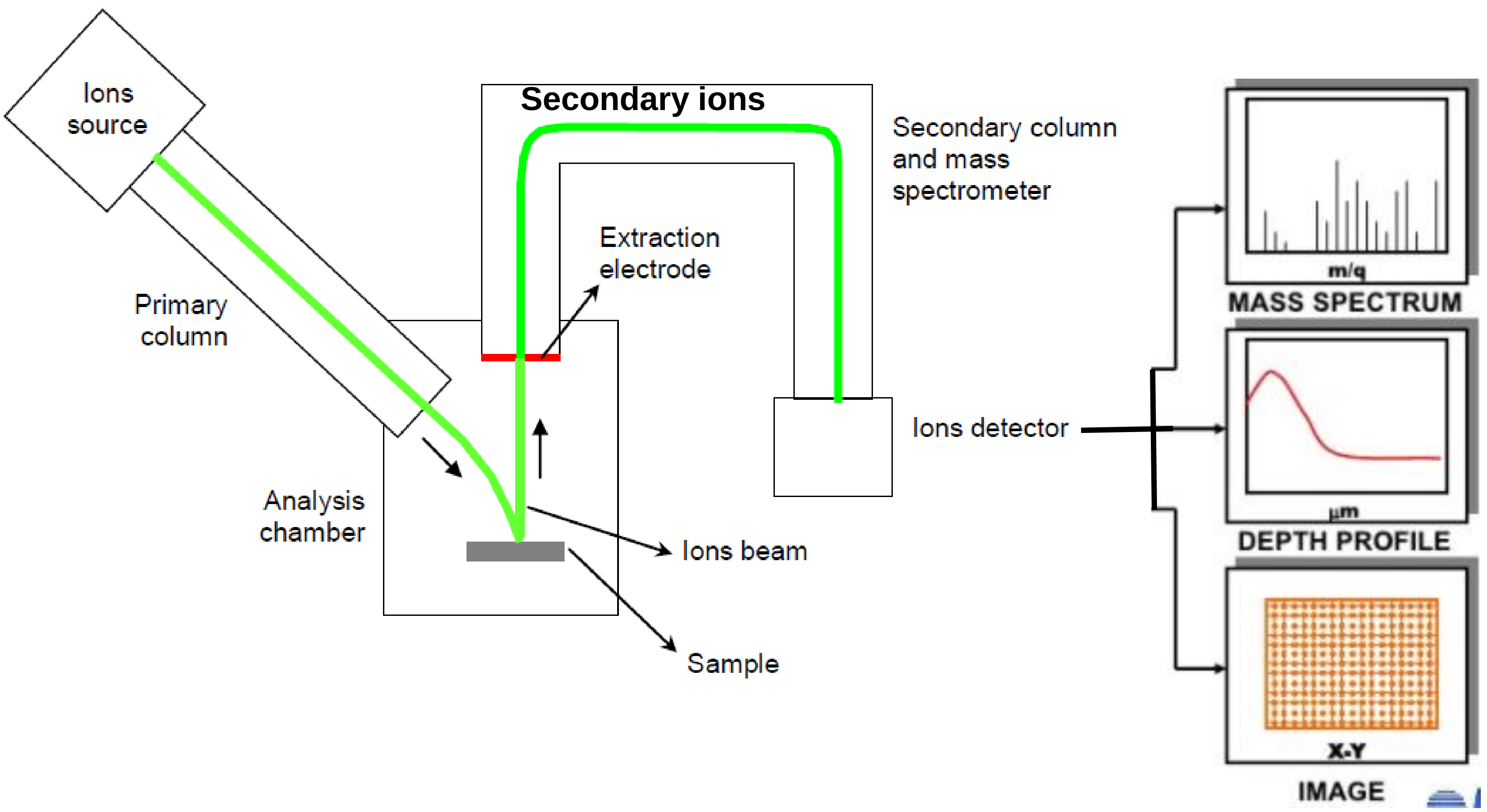
Advantages

- All elements can be detected.
- Detection limit upto ppm range.
- Very good lateral resolution ~ 100nm
- High depth resolution ~ 1nm
- Parallel detection of all elements at a single spot.

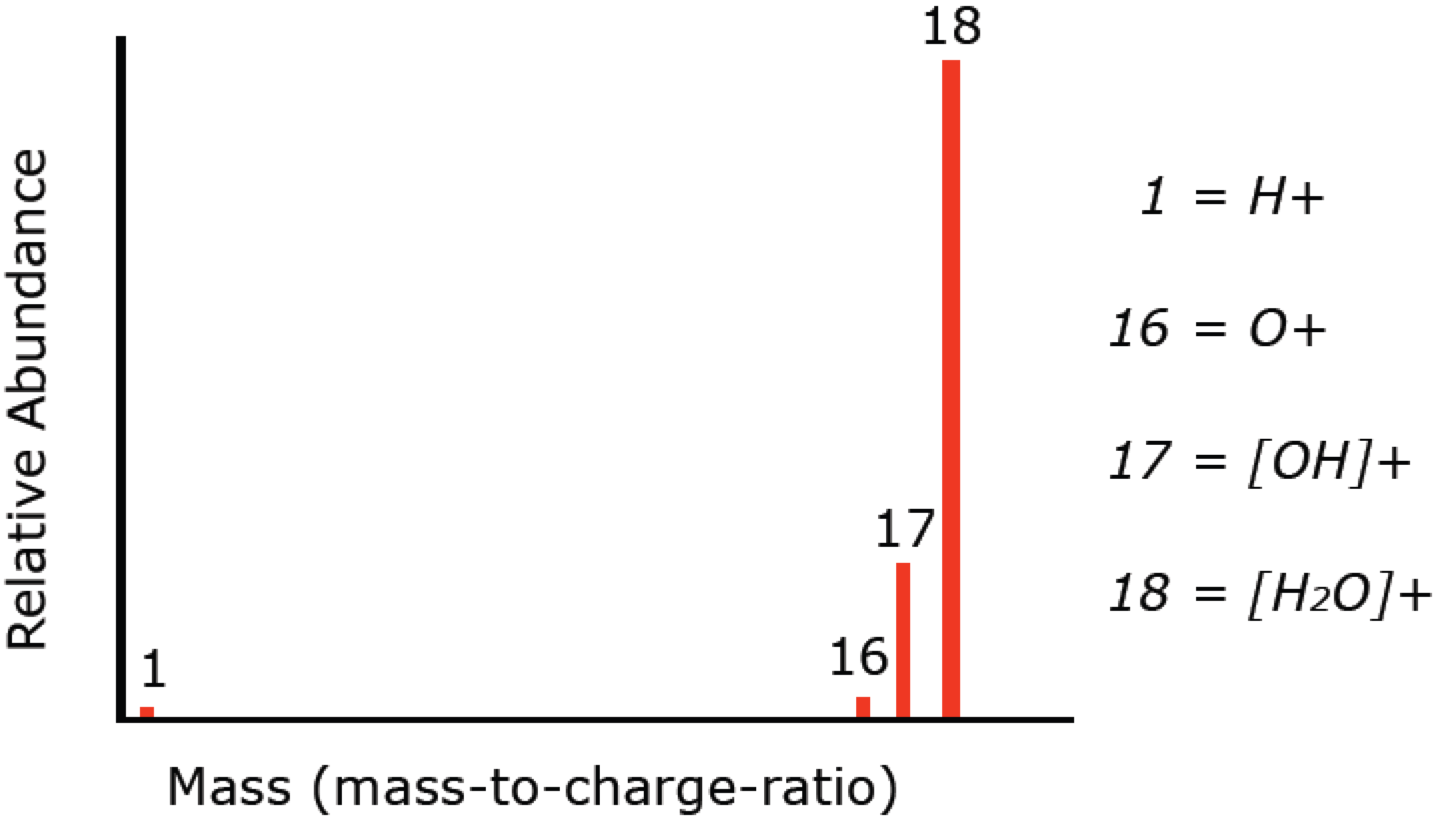
Purpose

- To develop understanding of impurity distribution near the top layer (~100 - 200 nm) of niobium by 2-D, 3-D ion mapping of the impurities after various processing treatments.

Secondary ion mass spectrometry - Principle



Simple mass spectrum of ice



Processing related impurities - Surface spectroscopy

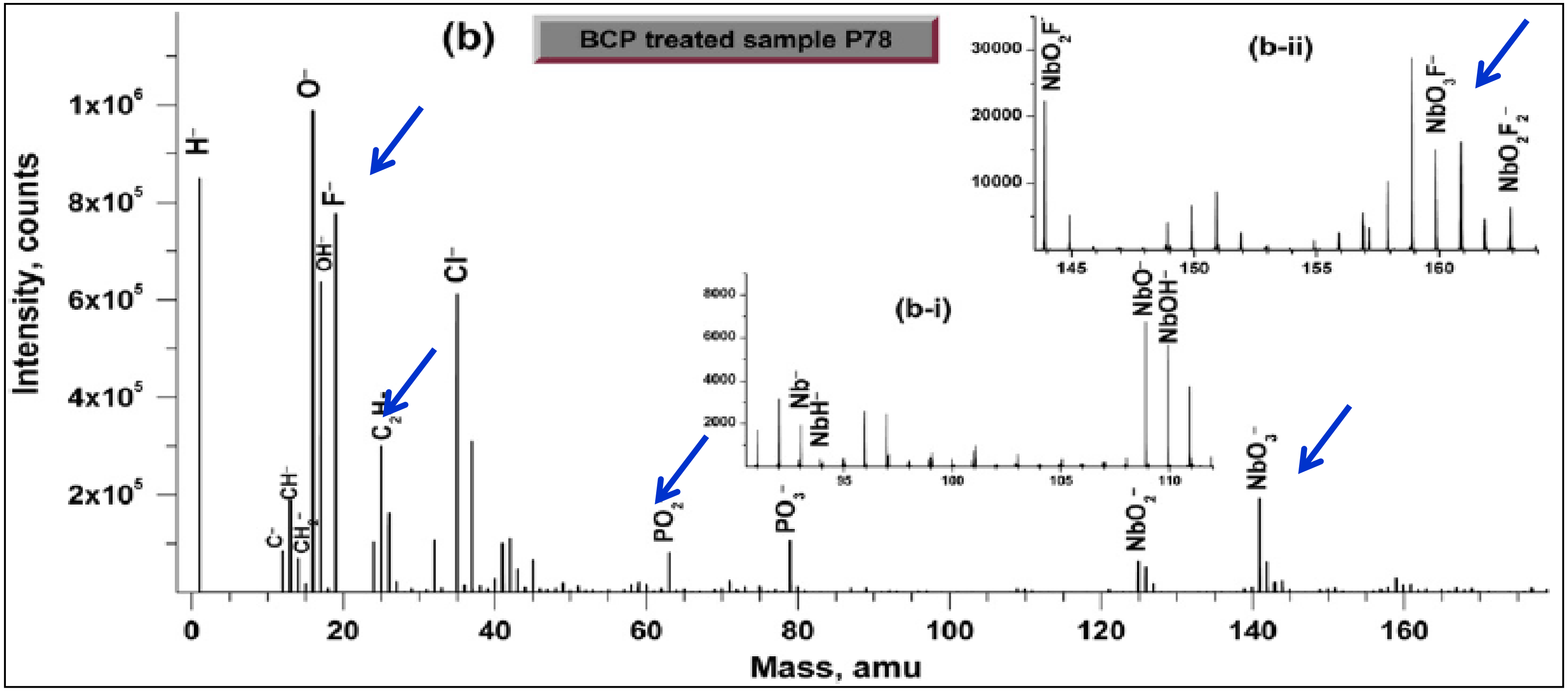


Figure shows mass spectrum of top surface of BCP treated Nb

Ref – A. Bose, S. C. Joshi – SST Journal, July 2015

Processing related impurities - Surface imaging

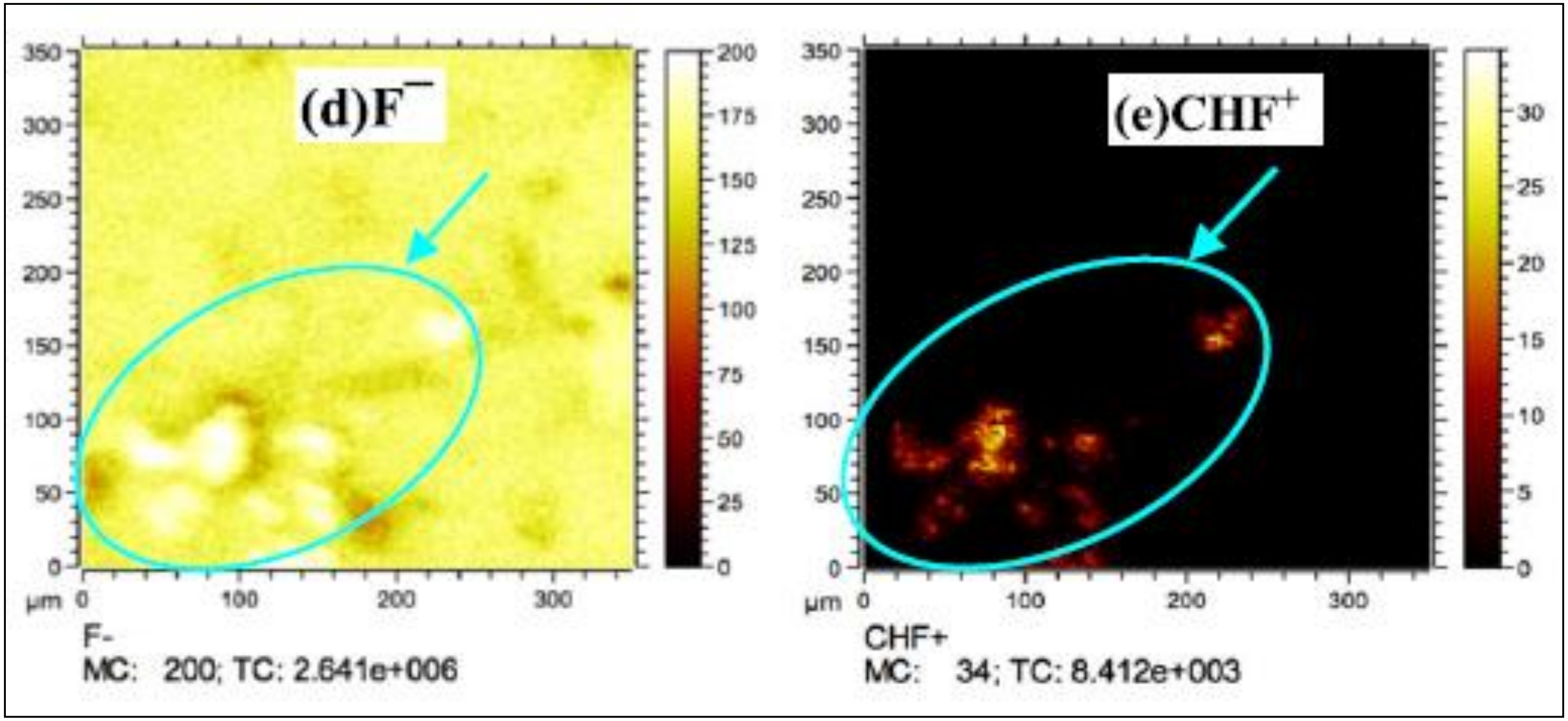
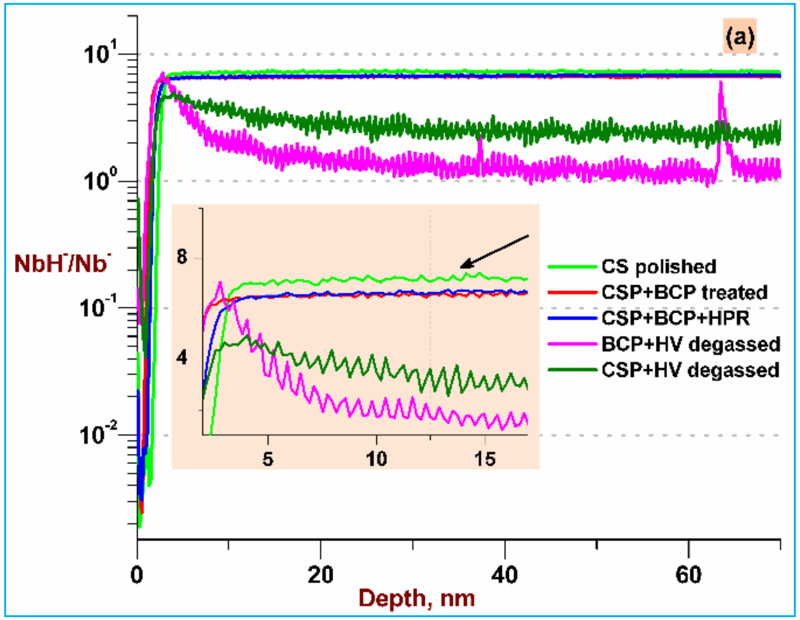
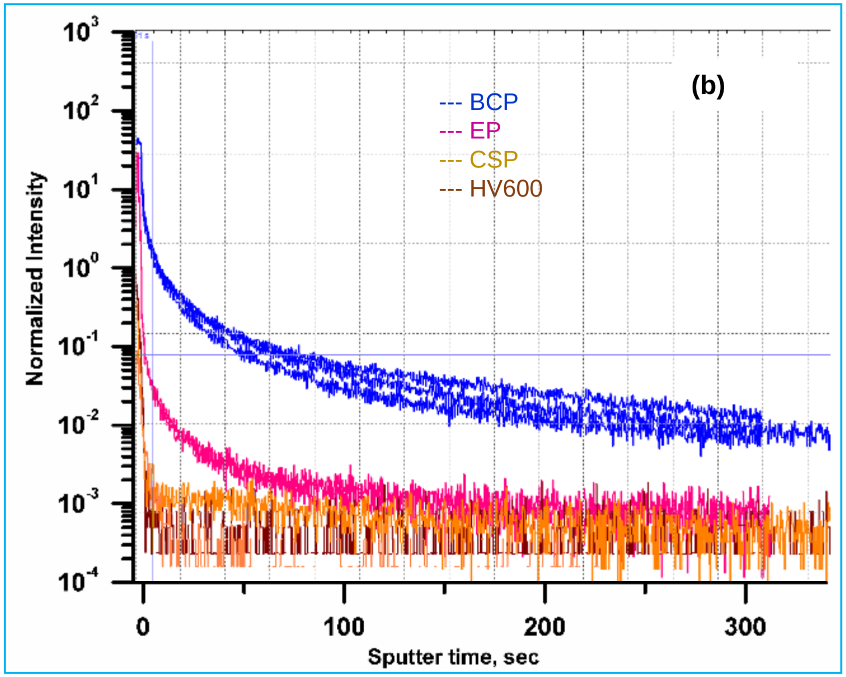


Figure shows the importance of ultrasonic rinsing after chemical cleaning

Ref – A. Bose, S. C. Joshi – SST Journal, July 2015

Processing related impurities – Depth profile of Hydrogen & Fluorine



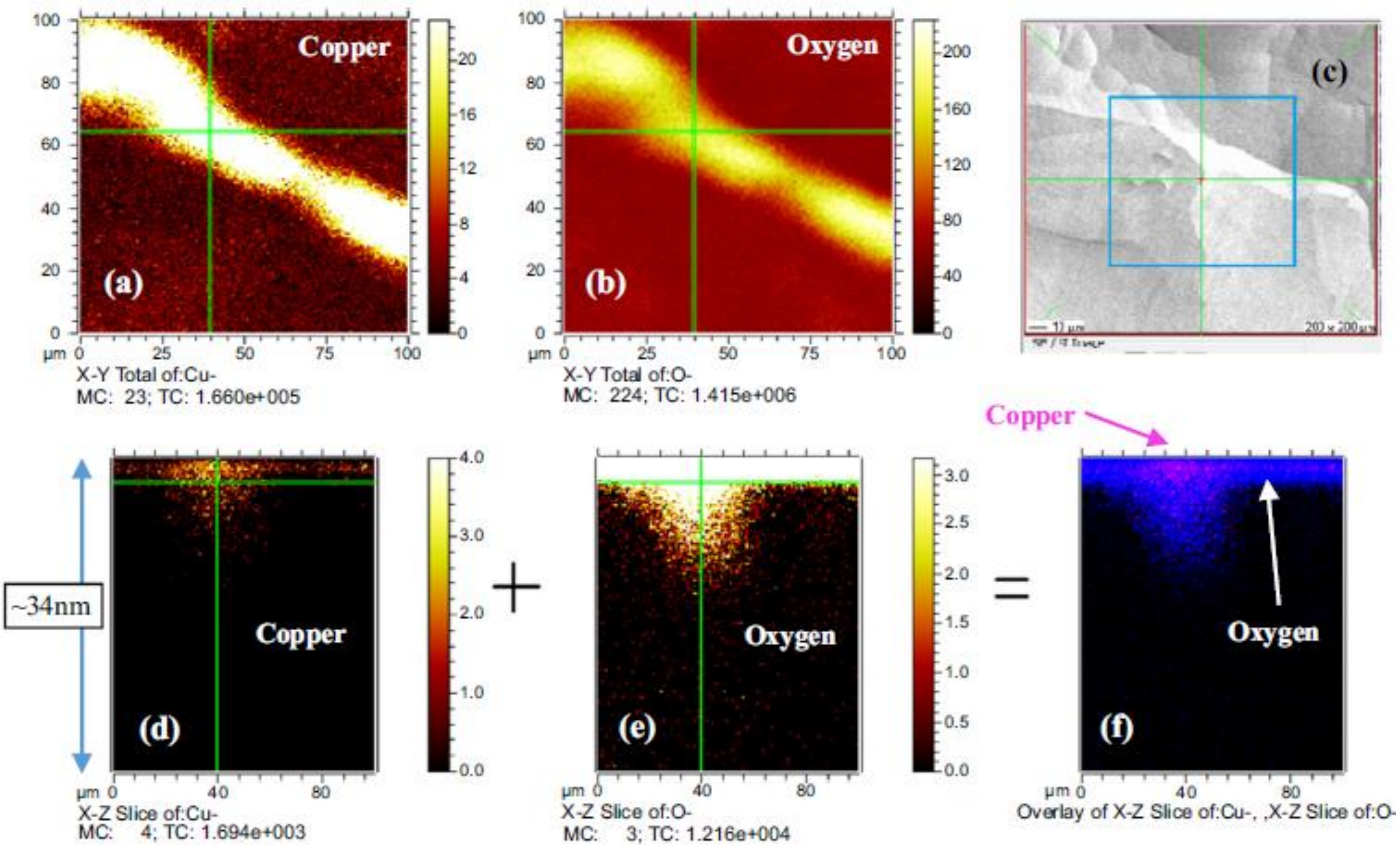


Variation of (a)Hydrogen, and (b) Fluorine with depth after various processing

CS Polish – Colloidal silica polishing
BCP – Buffer chemical polishing
HPR – High pressure rinse
HV – High vacuum 600 C degassing

Ref – A. Bose, S. C. Joshi – SST Journal, July 2015

Processing related impurities – 2d & 3D ion imaging - Effect of HPR



Ref – A. Bose, S. C. Joshi – SST Journal, July 2015

X-ray photo electron spectroscopy (XPS)

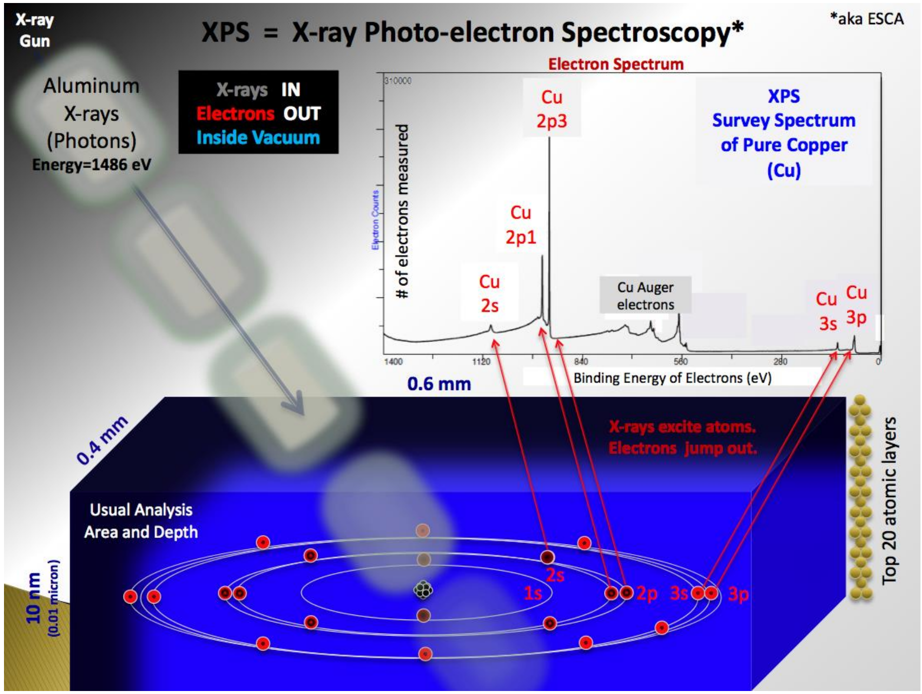
Advantages

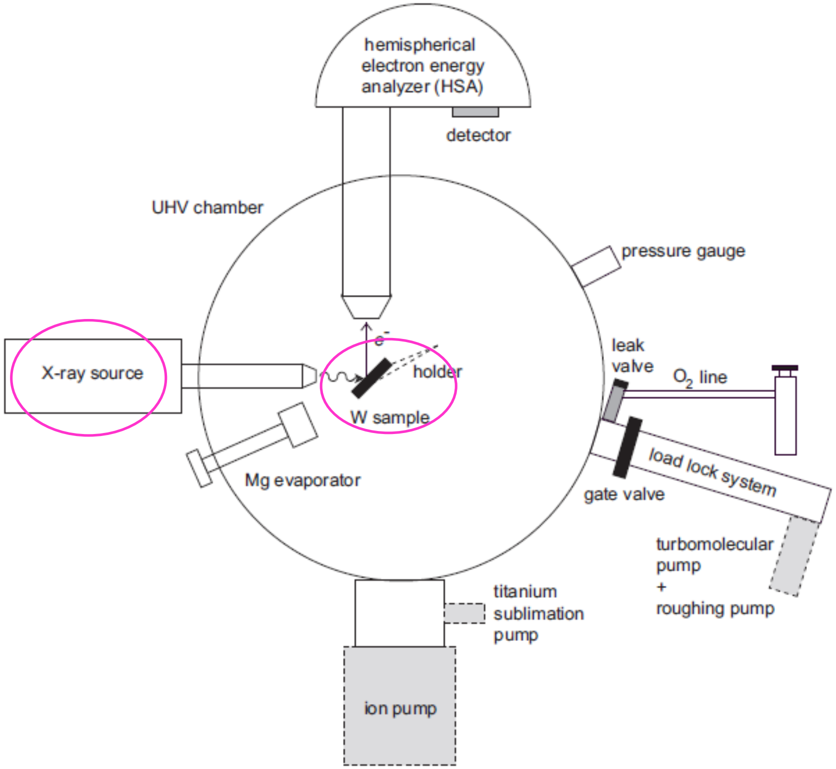
- All elements except H, He can be detected.
- Chemical state identification.
- Compositional analysis.

Purpose

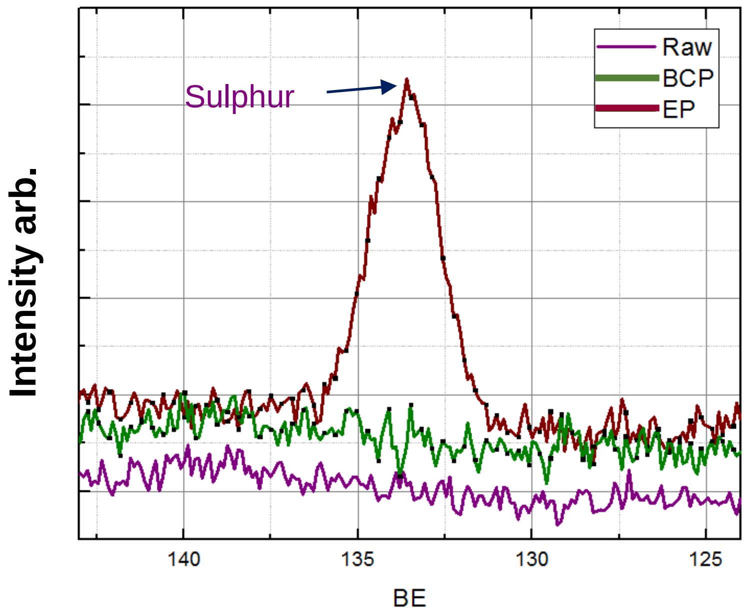
- Nb – oxide layer analysis
- Surface residue/ contamination analysis
- Compositional analysis upto ~10nm

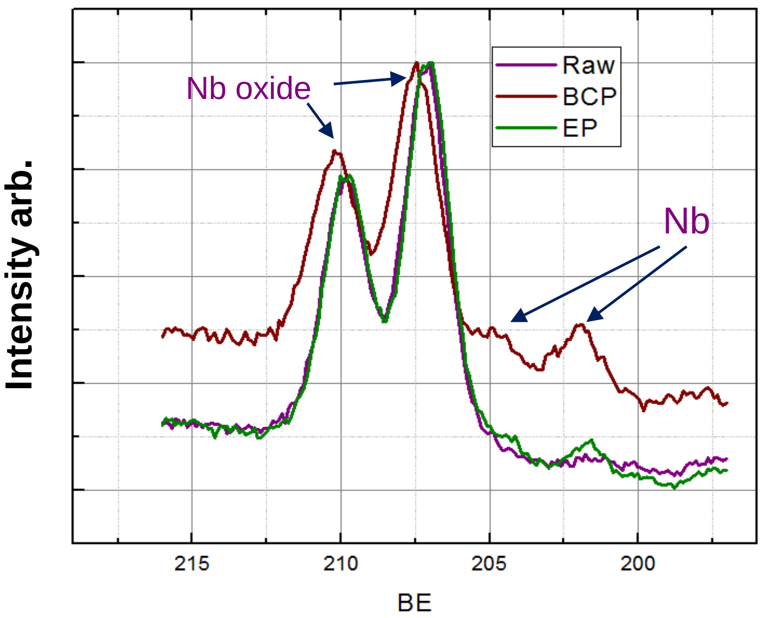
$$E_K = h\nu - E_B - \phi$$





Processing related impurities – Surface contamination & oxide layer





Effect of various processing conditions on Sulphur and thickness of oxide layer

Surface analysis

- Compositional Depth profiling
  - Secondary ion mass spectrometry – Lateral and 3D distribution of elements
  - X-ray photo electron spectroscopy – Oxidation state and Compositional analysis
  - ***Auger electron spectroscopy – Compositional analysis***
  - ***Elastic recoil detection analysis – Light element distribution on surface***
  - ***Total reflection X-ray fluorescence (TXRF) - Compositional analysis***

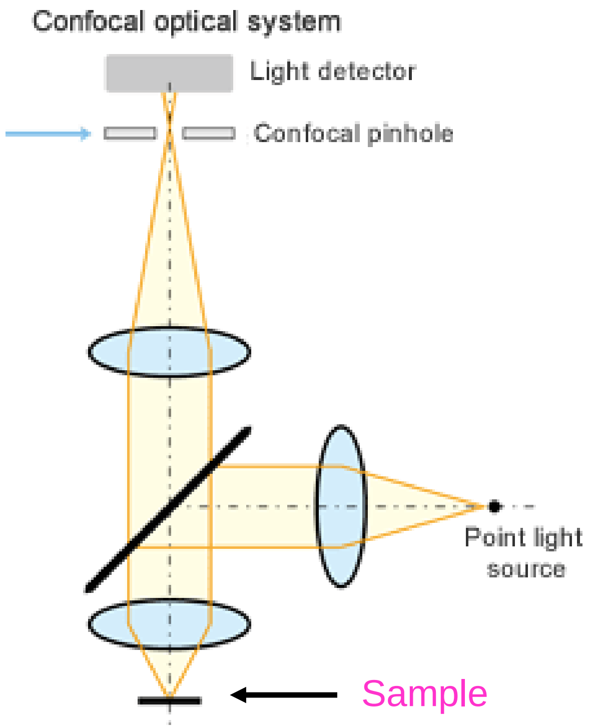
Topographical and microstructural defects

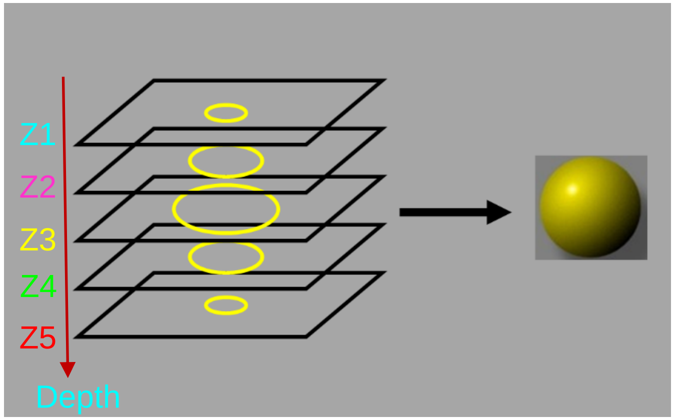
- Laser scanning confocal microscopy (LSCM)
- Scanning and transmission electron microscopy (SEM & TEM)

Laser scanning confocal microscopy (LSCM)

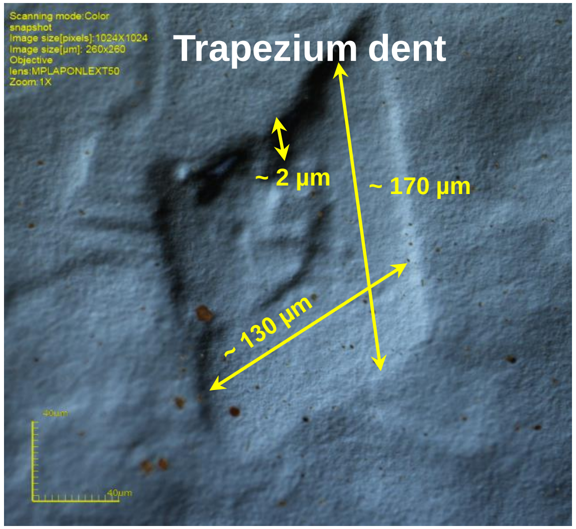
Purpose

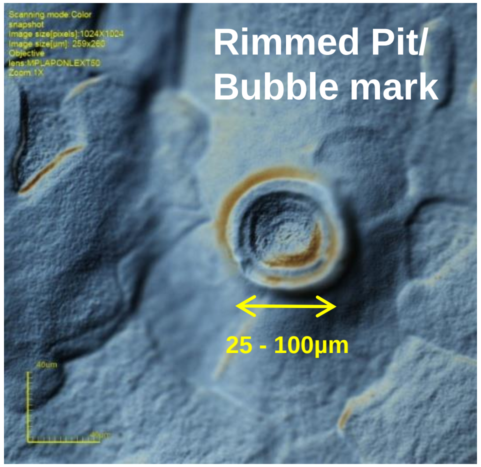
- Roughness estimation after chemical, mechanical and electropolishing treatments
- 3D imaging of topographical defects arising from processing treatments
- EBW bead imaging using replica technique
- It has all the features of optical microscope too.

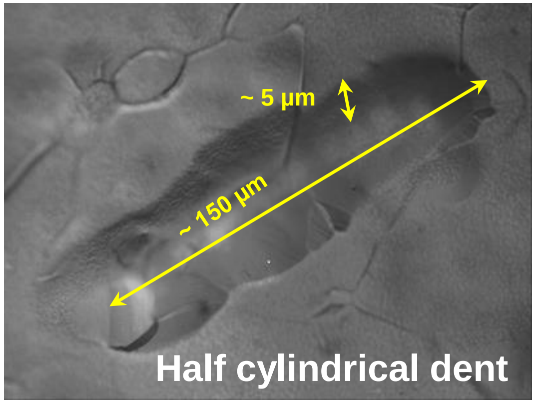


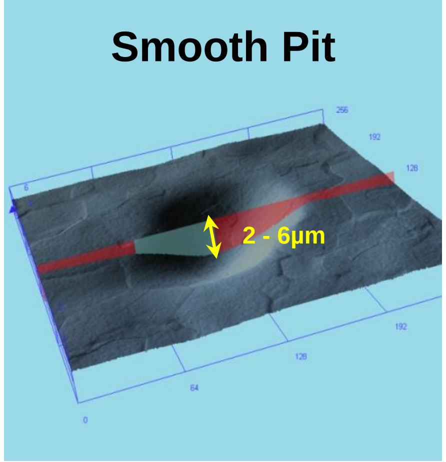


Few defects from EP treated samples





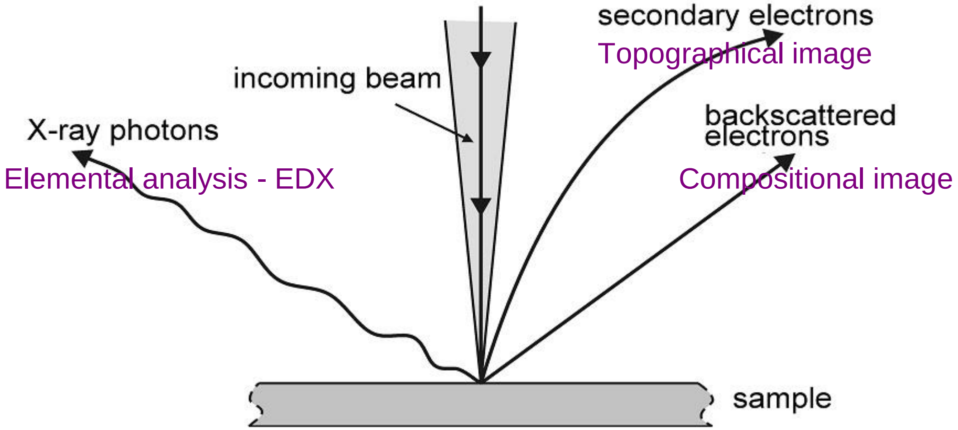


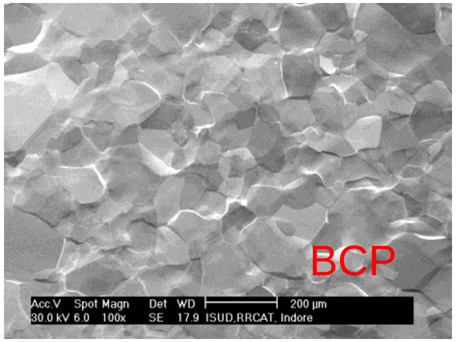


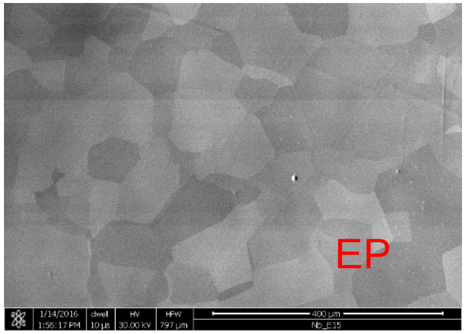
Scanning electron microscopy (SEM)

Purpose

- General image with high depth of field (3D effect)
- Extremely useful for small foreign particle analysis like field emitters
    - Shape
    - size
    - Composition
- Electron back scatter diffraction
    - For crystallographic texture
    - Dislocation density





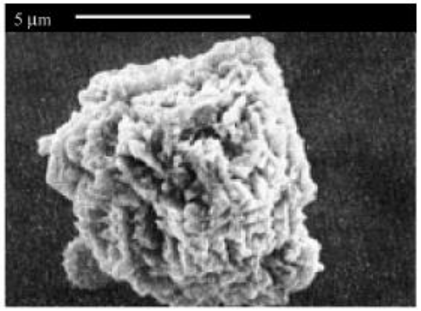


Advantages

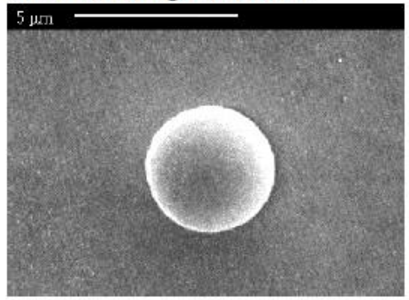
- Features as small as ~ 5nm can be imaged
- Compositional image resolution ~1µm

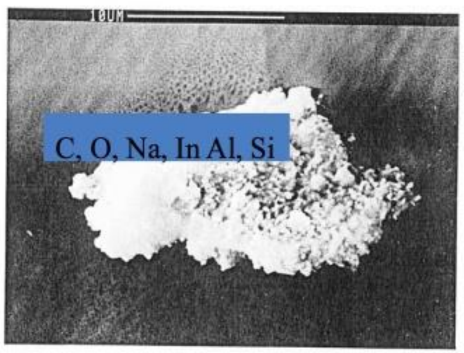
Field emitters

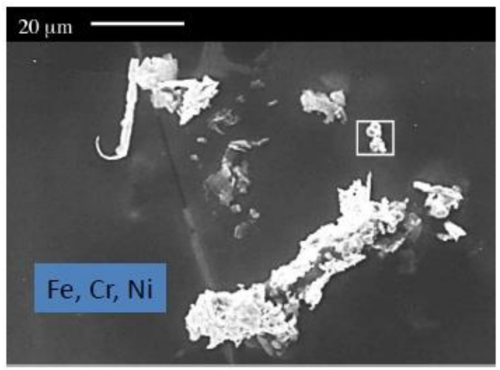
Ni

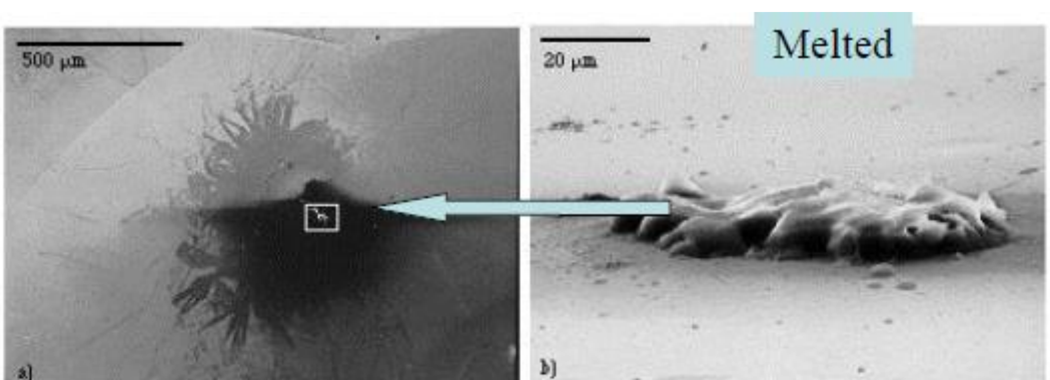


Smooth nickel particles emit less or emit at higher fields.

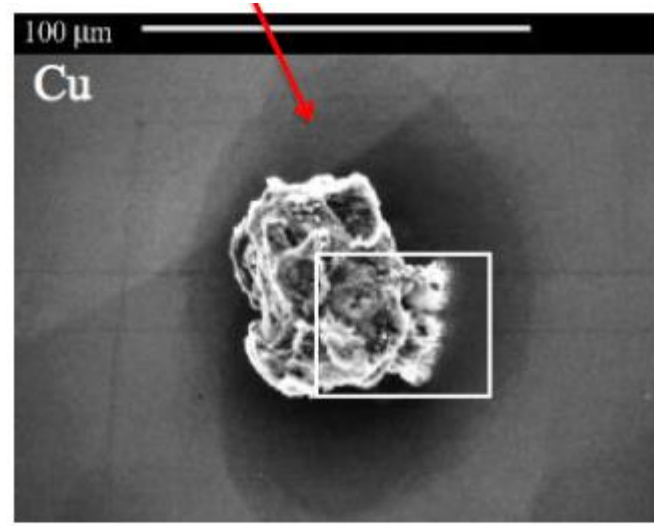


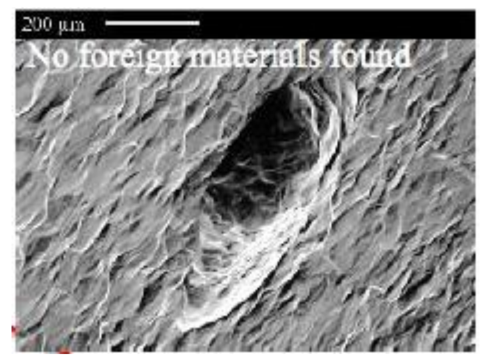






Normal defects- quench cavities

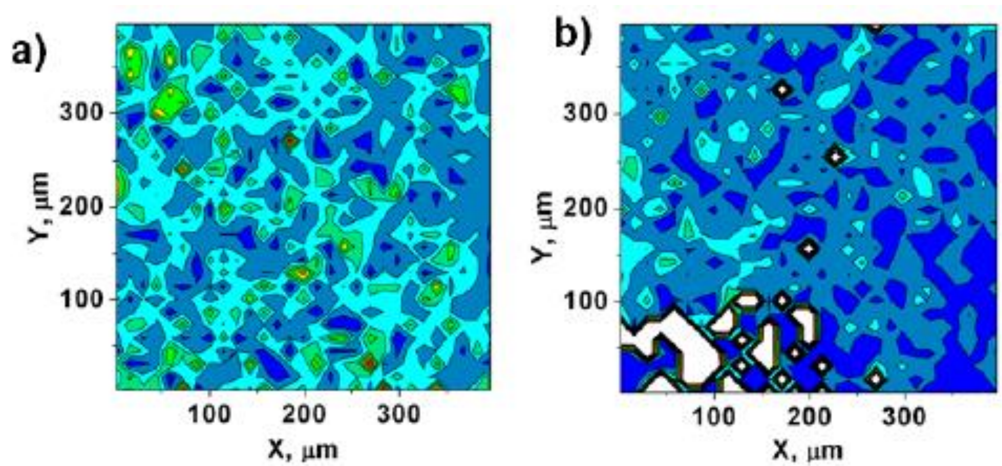




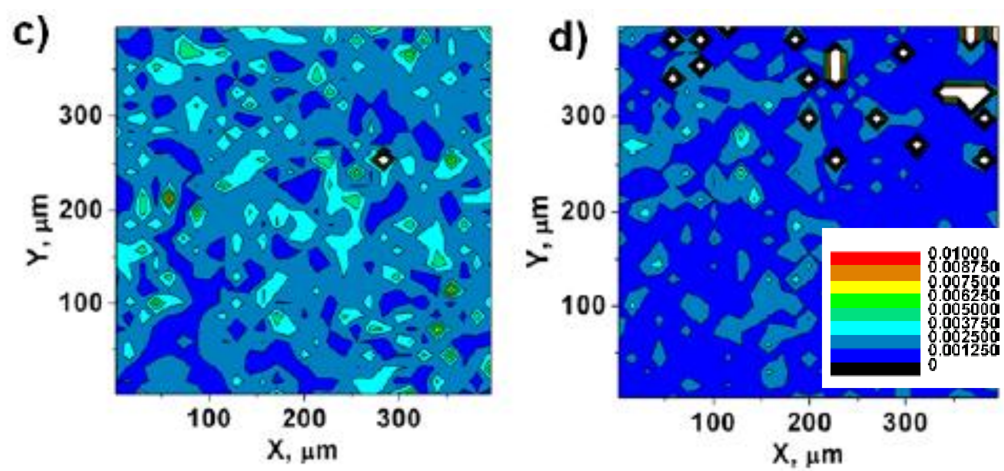
Ref – H. Padamsee, RF Superconductivity

EBSD of hot and cold spots – Mapping Dislocation density

Samples cut out from processed and tested cavity



Dislocation density images of **Hot spot samples** (a) Before bake, (b) After bake



Dislocation density images of **Cold spot samples** (c) Before bake, (d) After bake

Ref - A. Romanenko, Proceedings of SRF2009, Berlin, Germany

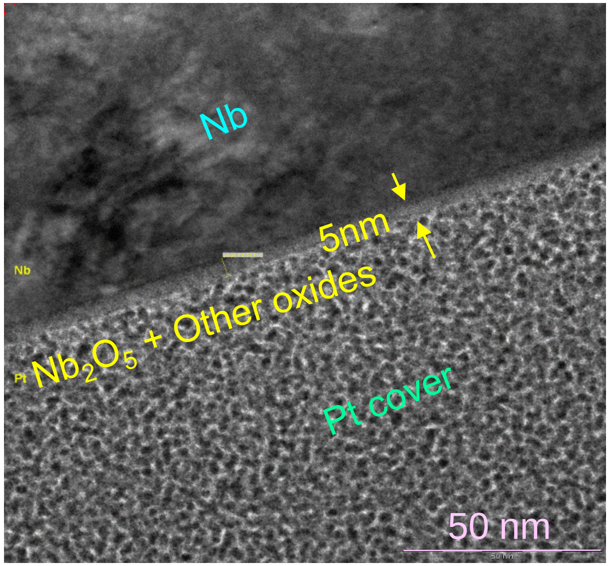
Transmission electron Microscopy (TEM)

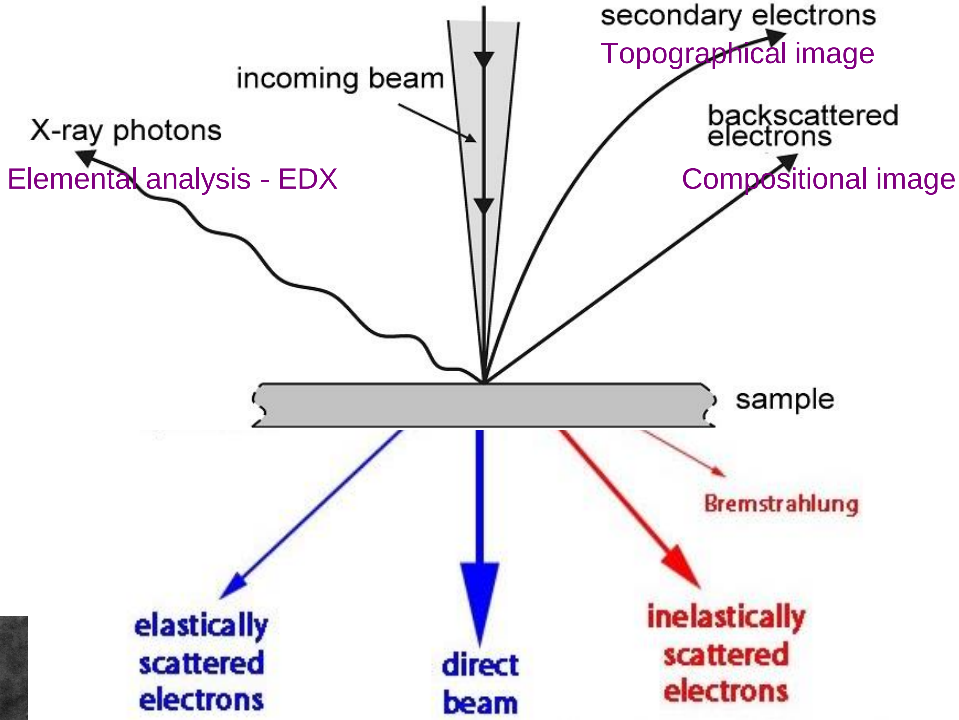
Purpose

- Imaging of defects like dislocations
- Oxide thickness measurement
- Imaging and mapping of hydride precipitates

Advantages

- Features as small as ~ 0.12nm can be resolved in HR TEM mode
- Crystal structure of phases can be detected





modified from Williams & Carter (1996) Fig. 1.3

Topographical and microstructural defects – Other relevant techniques

- Defects and microstructure – requires proper lateral resolution (XY)
  - Optical Microscopy (XY resolution > 200nm)
  - Scanning electron microscopy (XY resolution > 1nm)
  - ***Visual inspection (XY resolution > 100 micron)***
  - ***Radiography, ultrasonic and eddy current testing***
- Roughness and topography – requires lateral and vertical resolution
  - 3 D Laser scanning confocal microscopy (Z resolution >1nm, XY resolution~ 120nm)
  - ***Stylus (Z resolution >0.1nm, XY resolution~ 1nm)***
  - ***Atomic force microscopy (Z resolution ~1nm, XY resolution > 1nm)***
  - ***White light interferometry (Z resolution ~20nm, XY resolution > 120nm)***
- Crystal structure
  - Transmission electron microscopy (Nano-size defect identification)
  - ***X-ray diffraction (Crystal structure, Phase identification, Strain)***

Raw material Analysis for Nb

- Residual resistivity measurement (RRR) – For indirect measurement of purity/ thermal conductivity
- Bulk impurity analysis
- Mechanical property testing
- Microstructural characterization

Residual resistivity ratio (RRR)

Typical RRR level required for cavity applications > 300

Measurement Technique: 4-point probe method $$RRR = \frac{\rho_{300K}}{\rho_{10K}}$$

Higher RRR signifies

- Higher purity in the material
- Lower Density of defects like grain boundaries, dislocations, vacancies etc.

Purpose of high RRR Nb

- High thermal conductivity
- Better cavity performance
- Easy fabricability
- Lower annealing temperature

Bulk impurity analysis

Typical chemical composition of Nb for cavity applications

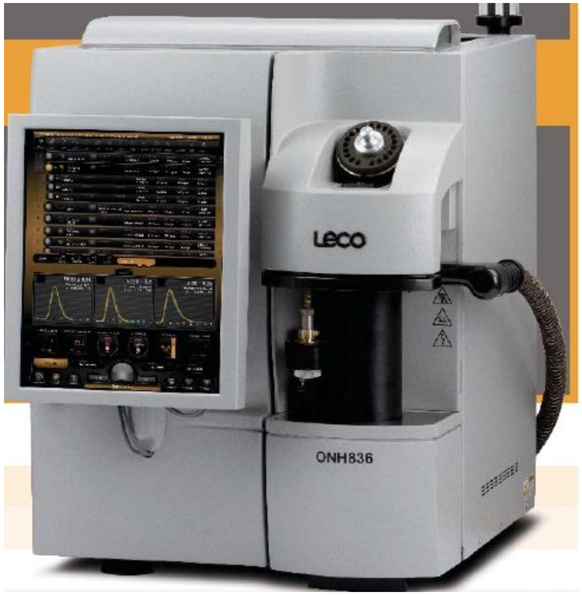
Chemical Composition(Wt.ppm) I: Ingot Analysis P: Product Analysis												
Content	Ta	W	Ti	Fe	Si	Mo	Ni	Any other (Individually)	H	N	O	C
guaranteed	≤500	≤70	≤40	≤30	≤30	≤50	≤30	≤30	≤3	≤20	≤40	≤20
Result (I)	140	11	<5	5	10	<10	<5	<30	2	7	8	5

Preferred Analysis Instrument

Inductively Coupled Plasma- Optical Emission Spectroscopy (ICP-OES)

Preferred Analysis Instrument

Inert gas fusion (IGF)



Analysis Principle

ICP - OES

- the sample in liquid form is nebulized through hot plasma which that excites the electrons of samples to higher levels.
- These electrons during de-excitation releases photon of wavelength specific to the elements present. These are analyzed and its intensity measured to quantify the elements.

IGF

- Samples are heated in graphite crucibles to extremely high temperature that releases the gases.
- These gases are carried to separate chambers where they are quantified based on thermal conductivity measurements and/or infra red absorbance measurements.

Advantages

ICP - OES

- Detection limits – upto parts per billion
- Multielement analysis in the same sample.
- Minimum chemical and matrix interferences.

IGF

- Detection limits – upto parts per million
- Specifically addresses interstitial elements
- Minimum chemical and matrix interferences.

Other relevant techniques - Bulk elemental analysis

- Major and Minor elemental analysis (Major > 10wt%; Minor: 0.1 - 10 wt%)
  - X-ray fluorescence (XRF)
  - Energy dispersive x-ray (EDX) analysis
  - Optical Emission Spectrometry (OES)
  - Spark source mass spectrometry (SSMS)
  - Etc.

- Trace and Ultra trace elemental analysis (Trace: 0.001wt% - 0.1 wt%; Ultra trace < 0.001wt%)
  - Secondary ion mass spectrometry (SIMS)
  - Glow discharge mass spectrometry (GDMS)
  - Total reflection X-ray fluorescence (TXRF)
  - etc.

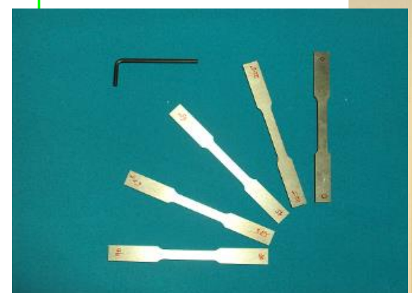
Mechanical property & Microstructural analysis

Typical mechanical and microstructural properties of Nb for cavity applications

Properties	Yield strength	Tensile strength	Elongation	Hardness, Hv	Grain size (ASTM)
ASTM B393	> 50 MPa	> 95 MPa	30%	60 - 75	5; none <4

Instrument

Universal testing machine (UTM)



Technique

- Prepare dogbone shaped samples as per ASTM E8 standards
- Place the samples within the grips as shown along with extensometers.
- Pull the samples at a specified rate of < 0.004 mm-/mm-min upto YS and < 0.04 mm/mm-min beyond YS.

Other measurements of relevance

- Strain hardening exponent $n = \frac{d(\log \sigma)}{d(\log \varepsilon)}$
- Plastic strain ratio $R = \frac{\varepsilon_w}{\varepsilon_t}$

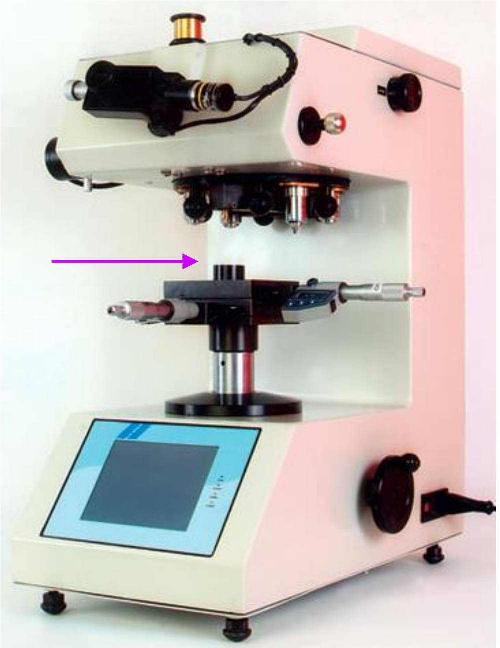
Hardness Measurement

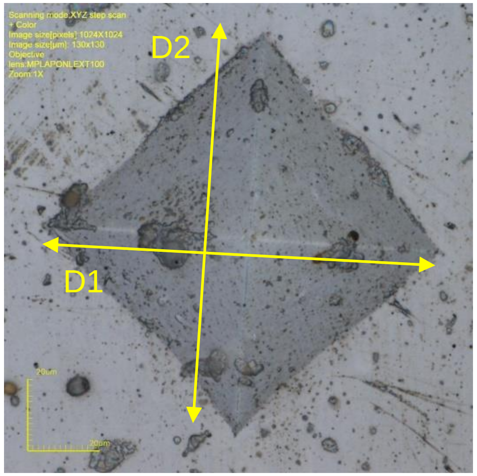
Instrument for Hardness measurement

Vickers Hardness Tester

Technique

- Prepare mirror finish samples and testing as per ASTM E92 standards
- Place the samples as shown in photo and make an indentation on sample with a specified load (< 100gms)
- Measure the diagonals of the indentation and calculate hardness as per the formula



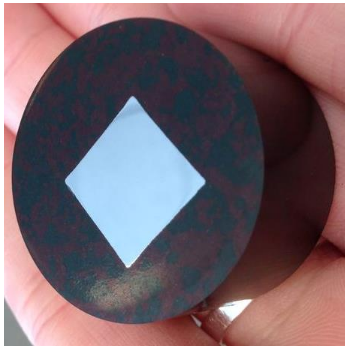


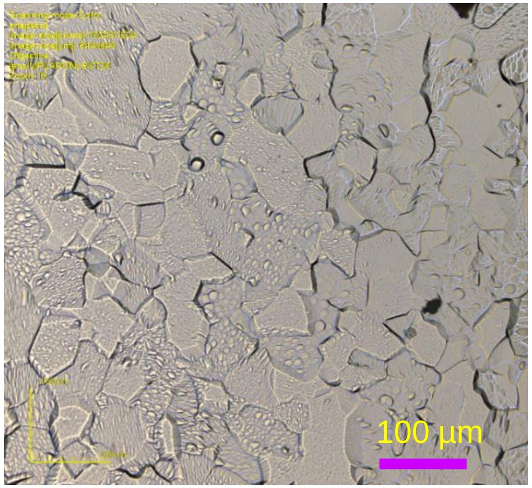
$$HV = 1.854 \frac{P}{d2}$$

Microstructural characterization

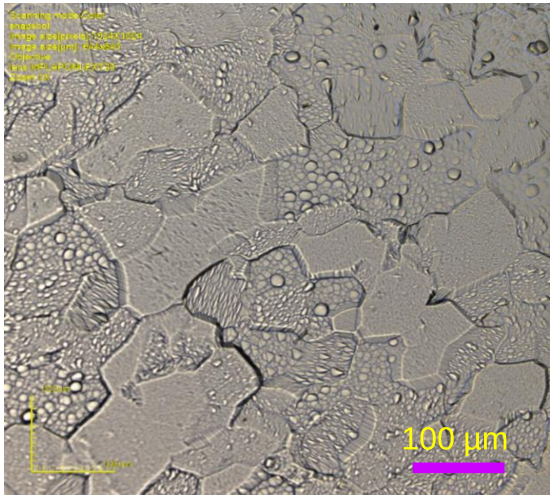
Process requires a proper metallography set up that includes

- Cut samples
- Mounting
- Polishing
- Etching
- Optical microscope



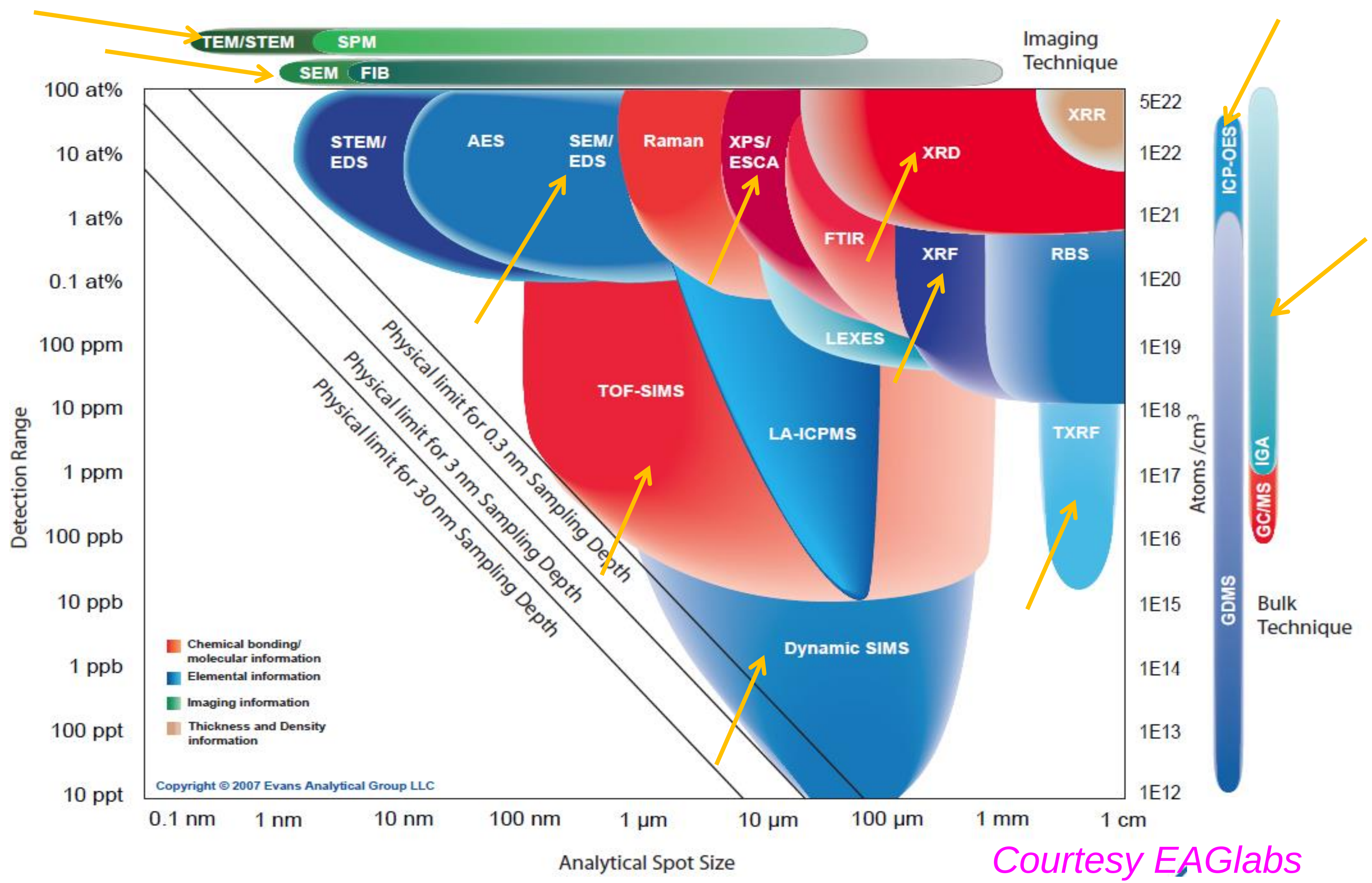


ASTM Size - 5



ASTM Size ~ 4

Comparison of all the major characterization techniques



Courtesy EAGlabs

Thank you.