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Analytical Methods

Analytical Resolution vs. Detection Limit

Imaging Techniques

AFM
TEM/STEM
EBIC
FIB
OP
SEM
RTX

STEM/ EELS
STEM/ EDS
Auger
SEM/ EDS
Raman
XPS/ ESCA
XRD
XRR
FTIR
XRF
RBS
TOF-SIMS
LA-ICPMS
TXRF
Dynamic SIMS

Physical limit for 0.3nm sampling depth
Physical limit for 3nm sampling depth
Physical limit for 30nm sampling depth

Chemical bonding/ molecular information
Elemental information
Imaging information
Thickness and density information only (no composition information)
Physical properties

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Detection Range Atoms/cm³

5E22, 1E22, 1E21, 1E20, 1E19, 1E18, 1E17, 1E16, 1E15, 1E14, 1E13, 1E12

100 at%, 10 at%, 1 at%, 0.1 at%, 100 ppm, 10 ppm, 1 ppm, 100 ppb, 10 ppb, 1 ppb, 100 ppt, 10 ppt

0.1 nm, 1 nm, 10 nm, 100 nm, 1 µm, 10 µm, 100 µm, 1 mm, 1 cm

Analytical Spot Size

ICP techniques
GC-MS, LC-MS
IGA
IC
TGA/DTA/DSC
NMR
GPC
GDMS

Bulk Techniques

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Picking a technique

- Many techniques are available and you should pick the technique that is most appropriate for your problem, not just the most convenient
- Questions you need answers for
  - What is it that you want to measure?
  - Do you need images/micrographs?
  - What size are the features?
  - Do you need elemental information?
    - Which elements?
  - Do you need chemical bonding information?
  - What is the concentration?
  - Do you need information in depth?
  - Do you need quantitative information or qualitative?
    - Do you have standards?

When do you use it?

- Auger
  - When you need elemental information (>1% concentration) on um to 10's of nm size objects (both surface and depth information available). Excellent for surveys
- XPS
  - When you need elemental or bonding information (>0.1%) on large (>10 um) features (both surface and depth information available). Excellent for surveys.
- Static SIMS
  - When you need ppm detection limits on sub-micron (>200nm) size areas. Gives both elemental and bonding information. Excellent for surveys.
- SPM
  - When you need quantitative topographic information on your sample (i. e. roughness) with nm scale lateral resolution. Are also a large # of associated techniques
- Atom Probe
  - When you need 3D elemental identification with atomic resolution on < 200nm sized areas
- XRD
  - When you need orientation or strain information. Also film thickness and density can be measured with XRR

When do you use it

- Backscattering (RBS, ISS, MEIS)
  - When you need elemental information (>10ppm) on large areas (mm). Becomes more surface sensitive as you go to lower energies (ISS>MEIS>RBS), but less depth measured.
- SEM/EDS
  - When you need micrographs of your sample (down to few nm). Can get elemental information when you use EDS. EDS good for surveys
- TEM
  - When you need micrographs of your sample (sub-nm). Can get elemental information when you use EDS or EELS.
- Raman
  - When you need non destructive molecular information on micron sized areas
- GDOES
  - When you need elemental depth compositional depth profiling of up to 100um depth
- FIB
  - When you need to modify materials on the nm scale. If it is a DB FIB you can also get high resolution images or EDS/EBSD maps. Also used for sample preparation for HR TEM analysis

ANALYTICAL TECHNIQUE	TYPICAL APPLICATIONS	ELEMENTS DETECTED	DETECTION LIMITS	IMAGING/ MAPPING	LATERAL RESOLUTION/ PROBE SIZE
Accelerator Techniques Rutherford Backscattering Spectrometry Nuclear Reaction Analysis, Hydrogen Forward Scattering Spectrometry Channeling	Thin film composition/thickness, quantitative dose measurement, quantitation without standards, hydrogen in thin films, defects and lattice locations	B - U (RBS) B, N, O, C (NRA) ^{1}H, ^{2}H (HFS)	RBS 0.001 – 10at% (Z dependent) NRA > 1×10^{15} at/cm^2 HFS 0.1at%	No	≥1mm
AFM Atomic Force Microscopy	Roughness, 3-dimensional imaging of surfaces, magnetic field, grain size	n/a	n/a	Yes	1.5 – 5nm
Auger Auger Electron Spectroscopy	Surface, particle, defect analysis, and large and small area depth profiling	Li - U	0.1 – 1at% submonolayer	Yes	≥10nm (Field Emission)
EBSD Electron Backscatter Diffraction	Grain size, grain orientation, grain misalignment, % crystallinity	n/a	n/a	Yes	1nm imaging; ~80nm minimum grain size
FIB/SEM Imaging Dual Beam and Single Beam	In-situ cross section preparation and imaging	n/a	n/a	Yes	1nm
FTIR Fourier Transform Infrared Spectroscopy	Identification of polymers and organics; contamination identification, particles	Molecular functional groups	0.1 – 1wt%	No	≥15µm
Raman Raman Spectroscopy	Identification of organics and inorganics; particle identification, stress measurement, carbon phase identification	Chemical and molecular information	1wt%	Yes	1µm
SEM (with EDS) Scanning Electron Microscopy Energy Dispersive X-ray Spectroscopy	Imaging, elemental identification	B - U	0.1 %	Yes	1nm imaging 0.5µm EDS
STEM (with EDS and EELS) Scanning Transmission Electron Microscopy Energy Dispersive X-ray Spectroscopy Electron Energy Loss Spectroscopy	Imaging, Z-contrast, elemental mapping elemental identification, EELS line scans, lattice imaging, Bright Field and Dark Field imaging	B - U	1%	Yes	0.25nm
TEM (with EDS and EELS) Transmission Electron Microscopy Energy Dispersive X-ray Spectroscopy Electron Energy Loss Spectroscopy	Imaging, elemental identification, crystallographic information, lattice imaging	B - U	0.5%	Yes	0.19nm
XPS/ESCA X-ray Photoelectron Spectroscopy/ Electron Spectroscopy for Chemical Analysis	Surface analysis of organic and inorganic materials, depth profiling	Li - U Chemical bonding	0.01 – 1at% submonolayer	Yes	10µm – 2mm
XRD X-ray Diffraction	Identification of crystal phases, crystal orientation and crystal quality, % crystallinity	H - U	1at%	No	15µm
XRR X-ray Reflectivity	Determination of film density, roughness and thickness	n/a	n/a	Yes	5mm

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