Energy Despersive X-ray (EDX)
&
Scanning Probe Microscopy (SPM)

Energy Despersive X-ray (EDX)

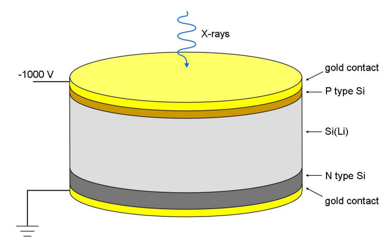
In EDX an electron from an outer shell of an atom (e.g. the 2s shell) lowers its energy to fill the hole in a lower shell (e.g. the 1s shell) which results in the emission of an x-ray. These emitted x-ray are characteristic of the particular atom undergoing emission. Therefore, by looking at the x-ray spectral lines of an atom one could identify that specific atom.

Majority of EDX systems are interfaced to SEM, where they use the same electron beam source to excite x-rays from the specimen under study.

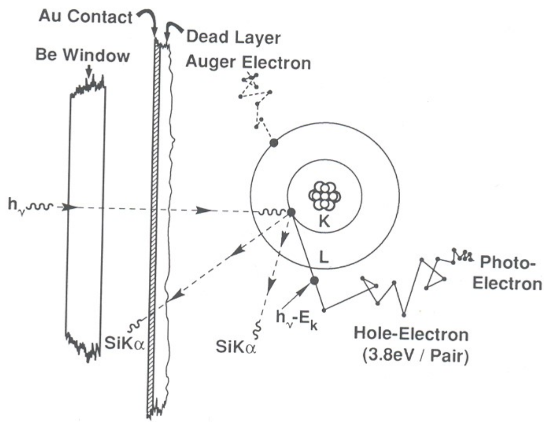
The most important part of the EDX spectrometer is the detector (also called “Crystal”). Most energy dispersive X-ray spectrometers employ an Si(Li) detector.

Detection of X-rays: Si(Li) detector

This consists of a 3–5 mm thick silicon junction diode with a bias of -1000 V across it. The central part is a lithium-drifted silicon crystal. When an X-ray photon passes through this crystal, electron-hole pairs are produced, and this causes a voltage pulse. To obtain sufficiently low conductivity, the detector must be maintained at low temperature using liquid-nitrogen.



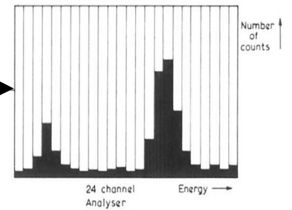
X-ray Detection Process in the Si(Li) Detector



The Detection Process

- When x-ray photons are captured by the detection crystal they create electron-hole pairs. These electron-hole pairs are formed charge pulse by the applied bias and they are further converted to voltage pulse by a charge-to-voltage converter (preamplifier).
- The signals further amplified and shaped by a linear amplifier and finally passed to a computer x-ray analyzer (CXA), where the data is displayed as a histogran (energy).

X-ray → voltage pulse → pulse processor →



Artifacts of the Detection process

Six types of major artifacts may possibly be generated during the detecting process:

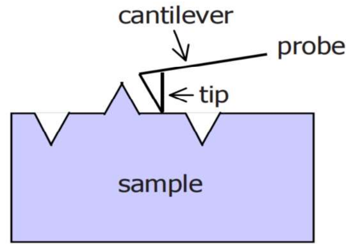
1. Peak Broadening
2. Peak distortion
3. Silicon x-ray escape peaks
4. Sum peaks
5. Silicon and gold absorption edges
6. Silicon internal fluorescence peak

Scanning Probe Microscopy (SPM)

Scanning Probe Microscopy (SPM)

This method employs the concept of scanning an extremely sharp tip (3~50 nm radius of curvature) across the object surface.

The tip is mounted on a flexible cantilever, allowing the tip to follow the surface profile. When the tip moves in the proximity of the object under investigation, forces of interaction between the tip and the surface influence the movement of the cantilever. These movements are detected by selective sensors.



Schematic of an AFM tip scanning over the surface of a sample

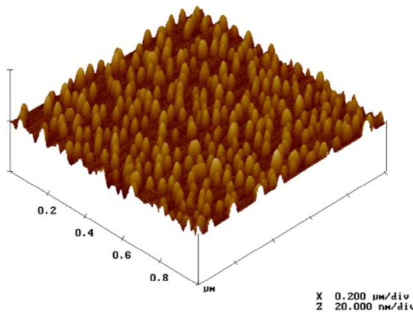
Major Types of SPM:

- **Atomic Force Microscopy (AFM)** measures the interaction force between the tip and the surface. The tip may be dragged across the surface, or may vibrate as it moves. The interaction force will depend on the nature of the sample, the probe tip and the distance between them.
- **Scanning Tunneling Microscopy (STM)** measures a weak electrical current flowing between tip and sample as they are held a very short distance apart.
- **Magnetic Force Microscopy (MFM)**
- **Scanning Near-field Optical Microscopy (SNOM)**
- **Many others..................**

Atomic Force Microscopy (AFM)

The AFM is capable of reconstructing the surface morphology of the materials with atomic scale precision.

- AFM has a very high resolution to the order of fractions of a nanometer.
- An AFM can measure the strength of the force, create an image of the surface, and manipulate the atoms on the surface of the specimen, depending on the situation.

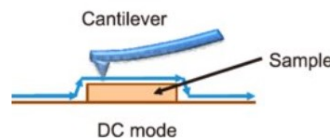


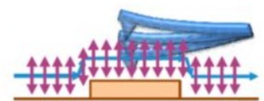
3D AFM image of the surface of a sample consisting of InAs quantum dots grown on top of a GaAs/InP substrate.

Types of AFM Measurement

There are several different subtypes of this microscope, including:

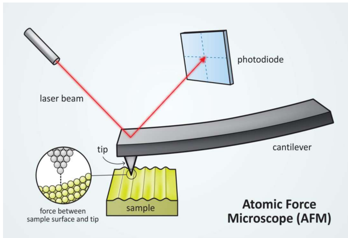
- **Contact/DC AFM**
- **Non-contact/AC AFM**
- **Tapping AFM**





Working of an AFM

A pointed LASER beam is focused onto the cantilever and made reflect onto a detector, which acts as a sensor. The cantilever bending is measured to determine the distance of the sample from the probe.

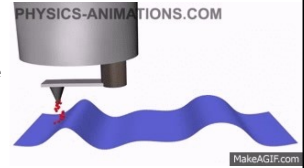


FM can be used for conductive or non-conductive samples, giving it a wider range of use. It can be used in contact mode, also known as DC mode, or tapping mode, also known as AC mode.

Scanning Tunneling Microscopy (STM)

STM measures the electrical current (tunneling) between the tip and the specimen.

- An extremely fine conducting probe is held close to the sample. Electrons tunnel between the surface and the stylus, producing an electrical signal. It slowly scans across the surface at a distance of only an atom's diameter.
- The stylus is raised and lowered in order to keep the signal constant and maintain the distance.
- Recording the vertical movement of the stylus makes it possible to study the structure of the surface atom by atom.



- A profile of the surface is created, and from that a computer-generated contour map of the surface is produced.

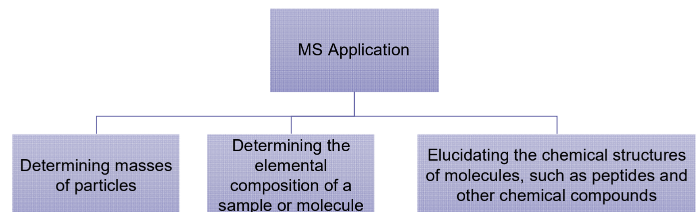
When to Choose AFM and STM?

The choice between AFM and STM depends on the nature of the sample and the type of information desired.

- AFM provides high-resolution topographic images, making it ideal for imaging delicate biological samples, polymers, and insulators.
- STM excels at atomic-scale electronic characterization of flat **conductive surfaces like metals & semiconductors**. STM can map variations in surface conductance.
- STM requires ultra-high vacuum while AFM can operate at ambient pressure or in liquid (for live biological samples).
- AFM has a lower resolution than STM on flat conductive samples.

Secondary Ion Mass Spectrometry (SIMS)

- Is a kind of spectrometry which uses an analytical technique to measure the mass-to-charge ratio of charged particles(ions)
- Mass Spectrometer works by ionizing chemical compounds to generate charged molecules or molecule fragments and measuring their mass-to-charge ratios

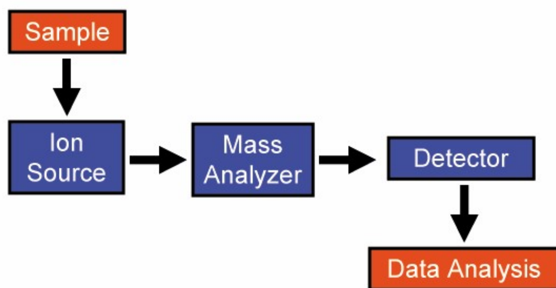


Secondary Ion Mass Spectrometry (SIMS)

General Structure of Mass Spectrometer

- Generally, a typical Mass Spectrometer consists of three parts: an ion source, a mass analyzer and a detector
- The function of the ion source is to produce ions from the sample.
- The function of the Mass Analyzer is to separate ions with different mass-to-charge ratios
- Then the numbers of different ions are detected by the detector
- Finally, the mass spectrum is generated after all the data have been collected

General Structure of Mass Spectrometer....



- The output, i.e. mass spectrum, is an intensity vs. m/z (mass-to-charge ratio) graph

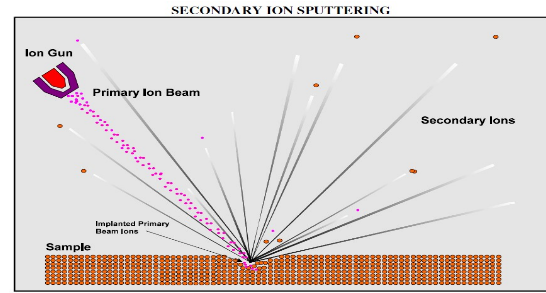
Types of Mass Spectrometers

- Due to the differences in ionization techniques, various analyzers and detectors, the mass spectrometers can be divided into several types.
- **For example:**
  - SIMS(Secondary ion mass spectrometry)
  - ICP-MS (Inductively coupled plasma-mass spectrometry)
  - AMS (Accelerator mass spectrometry)
  - TIMS (Thermal ionization-mass spectrometry)
  - SSMS (Spark source mass spectrometry)
  - IRMS (Isotope ratio mass spectrometry)

Introduction to SIMS

- SIMS is based on the observation that charged particles (Secondary Ions) are ejected from a sample surface when bombarded by a primary beam of heavy particles.

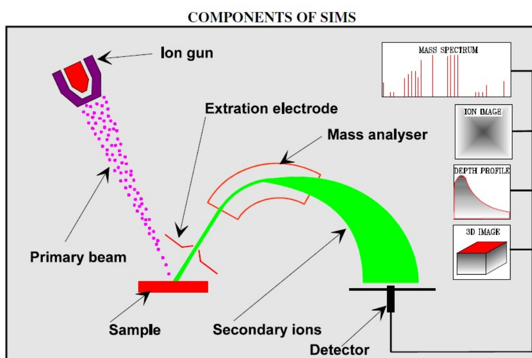
SECONDARY ION SPUTTERING



Secondary ion mass spectrometry (SIMS)

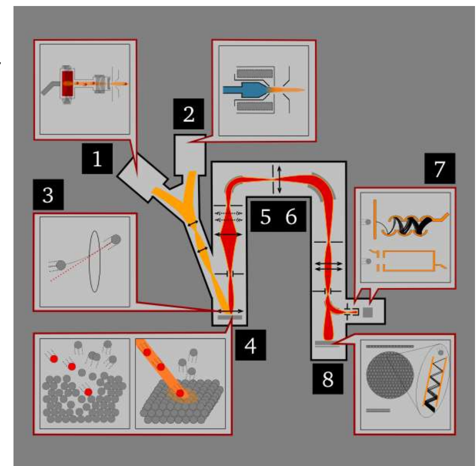
Structure of SIMS

COMPONENTS OF SIMS



How Does SIMS Work?

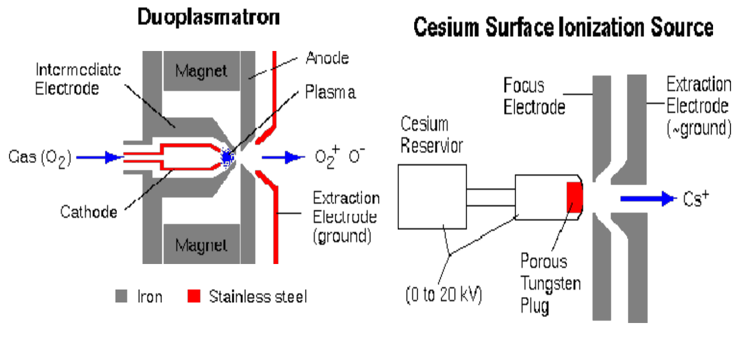
- Typical schematic of a SIMS instrument.



Step 1, 2

High energy ions are supplied by an ion gun

- SIMS Primary Ion Sources:

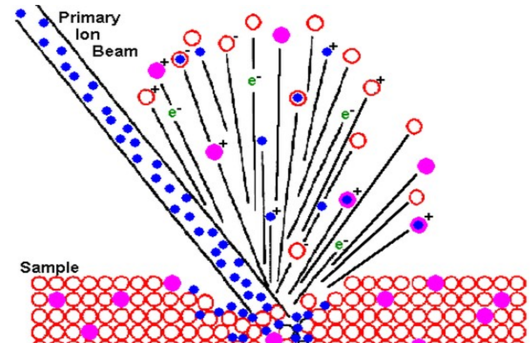


Step 3, 4

Accelerating and focusing the beam onto the target sample

Which ionizes and sputters some atoms off the surface

- This leads to the ejection (or sputtering) of both neutral and charged (+/-) species from the surface.



Step 5, 6

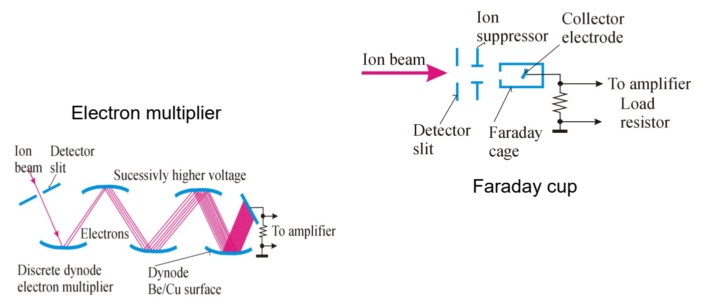
These secondary ions are then collected by ion lenses

- Ions generated by this process form the secondary beam and are subsequently transmitted within a continuous high vacuum environment to a mass spectrometer

Filtered according to atomic mass

Step 7

Then projected onto an electron multiplier (top), Faraday cup (bottom) Ion detectors



Limitations

- Generally does not produce quantitative analyses
- Optical capabilities are typically limited
- Charging may be a problem in some samples
- There is commonly an image shift when changing from positive to negative ion data collection mode;
- Too much data