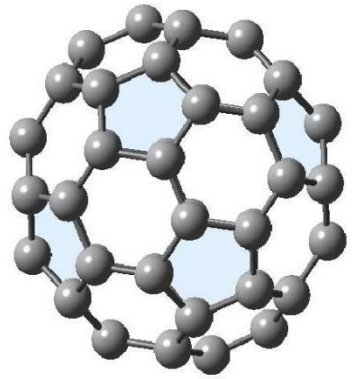
NANO – LECTURE 6



Nano Science and Nanotechnology

Characterization of Nanomaterials Part: 2

STM, UV-VIS Plasmon, XRD, SAXS

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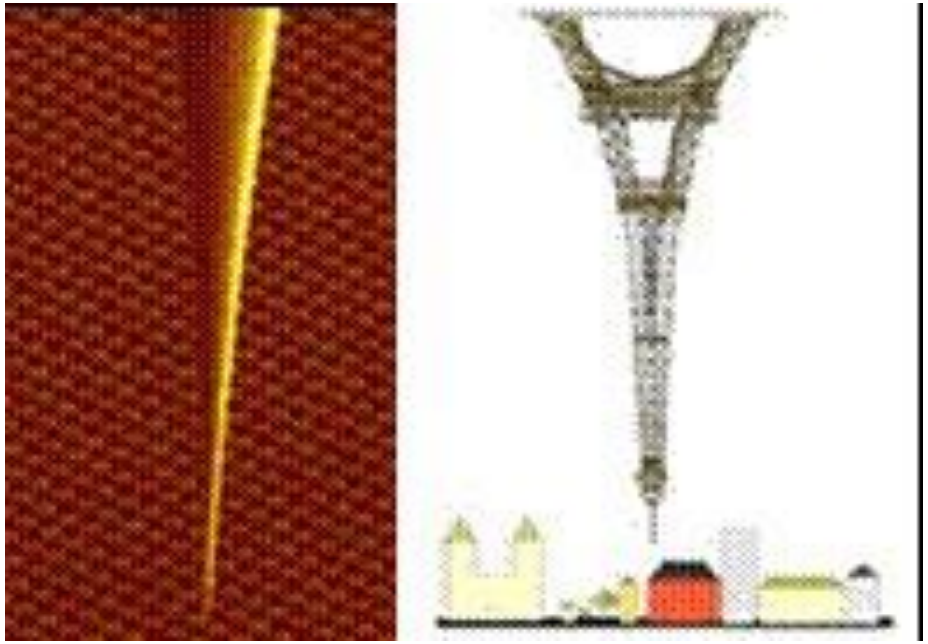
The different aspects of crystallite information that can be investigated using SEM are listed below:

- ***Topography***: The surface features of an object or 'how it looks.
- ***Morphology***: The shape, size and arrangement of the particles making up the object that are lying on the surface.
- ***Composition***: The elements and compounds that the sample is composed of and their relative ratios, in areas ~ 1 micrometer in diameter and depth.
- ***Crystallographic information***: The arrangement of atoms in the specimen and their degree of order; only useful on single-crystal particles >20 micrometres.

Scanning Tunnelling Microscope (STM)

- The STM is a fundamental tool in nanoscience and nanotechnologies.
- It is used in both industrial and fundamental research to obtain atomic-scale images of metal and semiconducting surfaces.
- It provides a three-dimensional profile of the surface roughness, allowing the observation of surface defects and the determination of the size and conformation of molecules and aggregates.
- Another astonishing property of the STM is that it can be used to manipulate (move!) individual atoms, trigger chemical reactions, as well as performing electronic spectroscopy.

Scanning Tunnelling Microscope (STM)



Left: 3 mm tip positioned 0.1 nm above the surface; right: macroscopic analogy: a 300 m high Eiffel Tower located 0.01 mm above the city

Uv-Visible Plasmon Absorption And Emission

- **Metal nanoparticles**, in particular gold and silver, are characterized by a plasmon resonance absorption that gives rise to intensely coloured solutions.
- The absorption band is due to electrons confined at the particle surface that collectively oscillate at a specific frequency, commonly referred to as the surface **plasmon resonance frequency**.
- As examples, the plasmon band of a 20 nm silver (Ag) particle is centred at 395 nm, resulting in a yellow solution, while a 20 nm gold (Au) particle absorbs at 520 nm resulting in a red solution.
- The **plasmon absorption effect** occurs for particles up to approximately 50 nm in diameter and scales with particle volume.
- Absorption can be in the visible and UV area of the spectrum.

X-RAY DIFFRACTION (XRD)

- XRD is extensively used to study the crystal structure of solids, defects and stresses.
- In XRD, a beam of X-rays, with wavelength ranging from 0.07 to 0.2 nm, is diffracted by the crystalline specimen according to **Bragg's law: $\lambda = 2d \sin \vartheta$**
- where d is the interplanar distance and λ is the wavelength of the X-rays.
- The intensity of the diffracted beam is measured as a function of the diffraction angle (2ϑ) and the specimen's orientation.
- The diffraction pattern can be used to identify the crystalline phases and their structural characteristics.
- XRD is non-destructive and does not require detailed sample preparation.

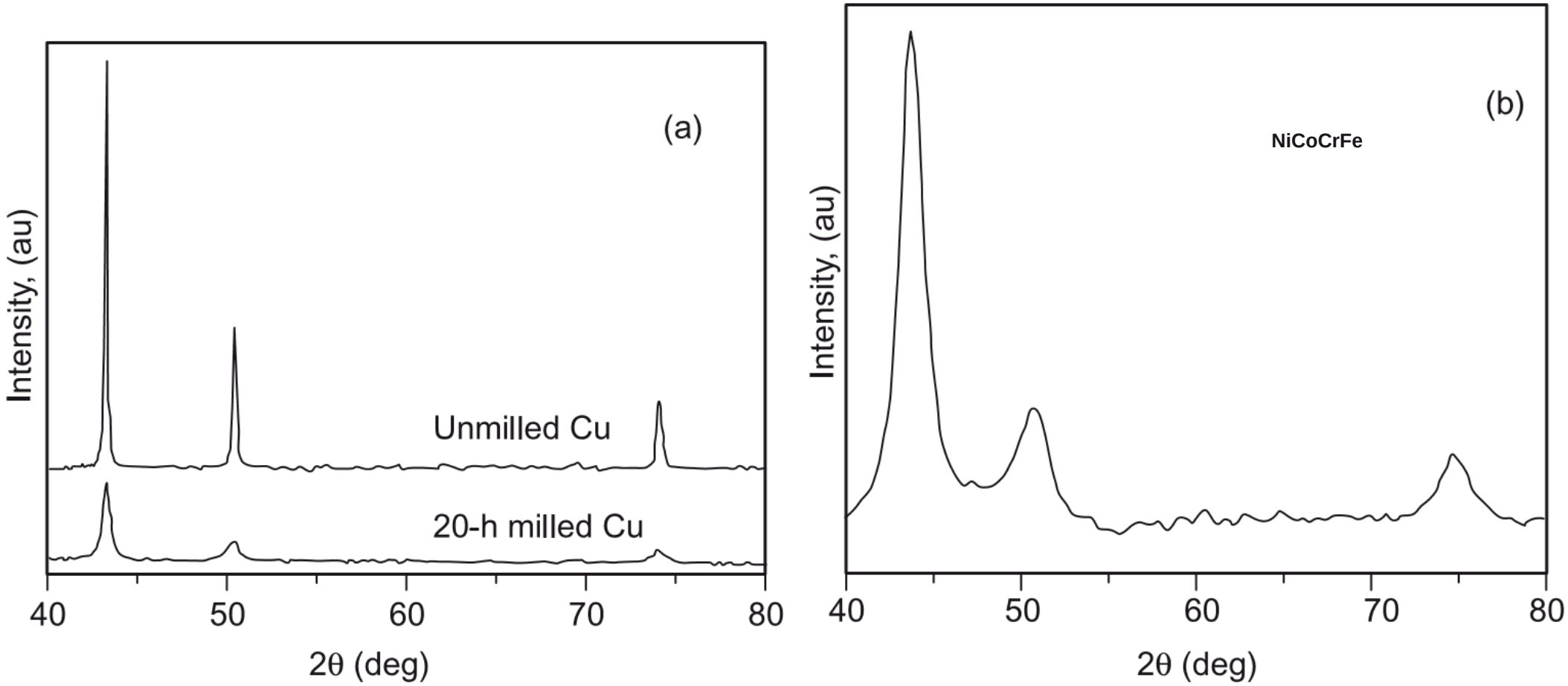
X-RAY DIFFRACTION (XRD)

- A shift in the X-ray peak positions indicates a change in *d*-spacing caused by a change in lattice constants.
- Peak broadening can be due to the fine crystallite size, which is independent of sin ϑ.
- In the absence of inhomogeneous strains, the crystallite size, *D*, can be estimated from the peak broadening using Scherrer's formula:
- Scherrer's formula: $D = K\lambda / B \cos \vartheta_B$

X-RAY DIFFRACTION (XRD)

- Scherrer's formula: $D = K\lambda / B \cos \vartheta_B$
- where λ is the X-ray wavelength, B is the full width at half maximum (FWHM) height of a diffraction peak, ϑ_B is the diffraction angle, and K is Scherrer's constant, which is of the order of unity for a spherical crystal.
- It is also important to note that X-ray diffraction provides only an average crystallite size.
- The thickness of epitaxial and highly textured thin films can also be determined using XRD.

XRD patterns of nanocrystalline (a) copper and (b) NiCoCrFe



SMALL ANGLE X-RAY SCATTERING (SAXS)

- SAXS is another powerful tool for characterizing nanostructured materials.
- Strong diffraction peaks result from constructive interference of X-rays scattered from ordered arrays of atoms and molecules.
- A variety of information can be obtained from the angular distribution of scattered intensity at low angles.
- Fluctuations in electron density over lengths in the order of l0 nm or larger are sufficient to produce appreciable scattered X-ray intensities at angles of $2\theta < 5°$.

SMALL ANGLE X-RAY SCATTERING (SAXS)

- The amount and angular distribution of scattered intensity provides information about the size of very small particles or their surface area per unit volume, regardless of whether the sample or particles are crystalline or amorphous.
- Since SAXS is very effective in measuring inhomogeneity in the region of 1–100 nm, it has been widely used in the characterization of nanocrystals.
- SAXS has been employed to characterize a wide spectrum of materials including biological structures, metallic and non-metallic specimens, composites and mesoporous materials.

Thanks for Watching