
بسم الله الرحمن الرحیم

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Materials Characterization Methods

Second Session

(Introduction to Methods of Materials Characterization and Analysis)

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Introduction to Materials Characterization Methods

- ✓ The word analysis means not just the chemical analysis, and its more complete meaning is characterization.
- ✓ In the past, the word analysis was used as the chemical analysis of substances because the act of characterization of materials was made only through chemical analysis or decomposition.
- ✓ Nowadays, the characterization of materials is performed in a variety of methods, so the term analysis is not only related to the concept of chemical analysis.

- ✓ In general, if the goal is the chemical analysis of materials, we can use the word chemical analysis instead of the word analysis, and if the purpose of identification is with the help of other methods, we can refer to the desired method along with the word analysis.

- ✓ A better word instead of analysis for the general meaning of identification and investigation is the word **characterization**, which means identification and description of materials.

Importance and Necessity of Materials Characterization Science

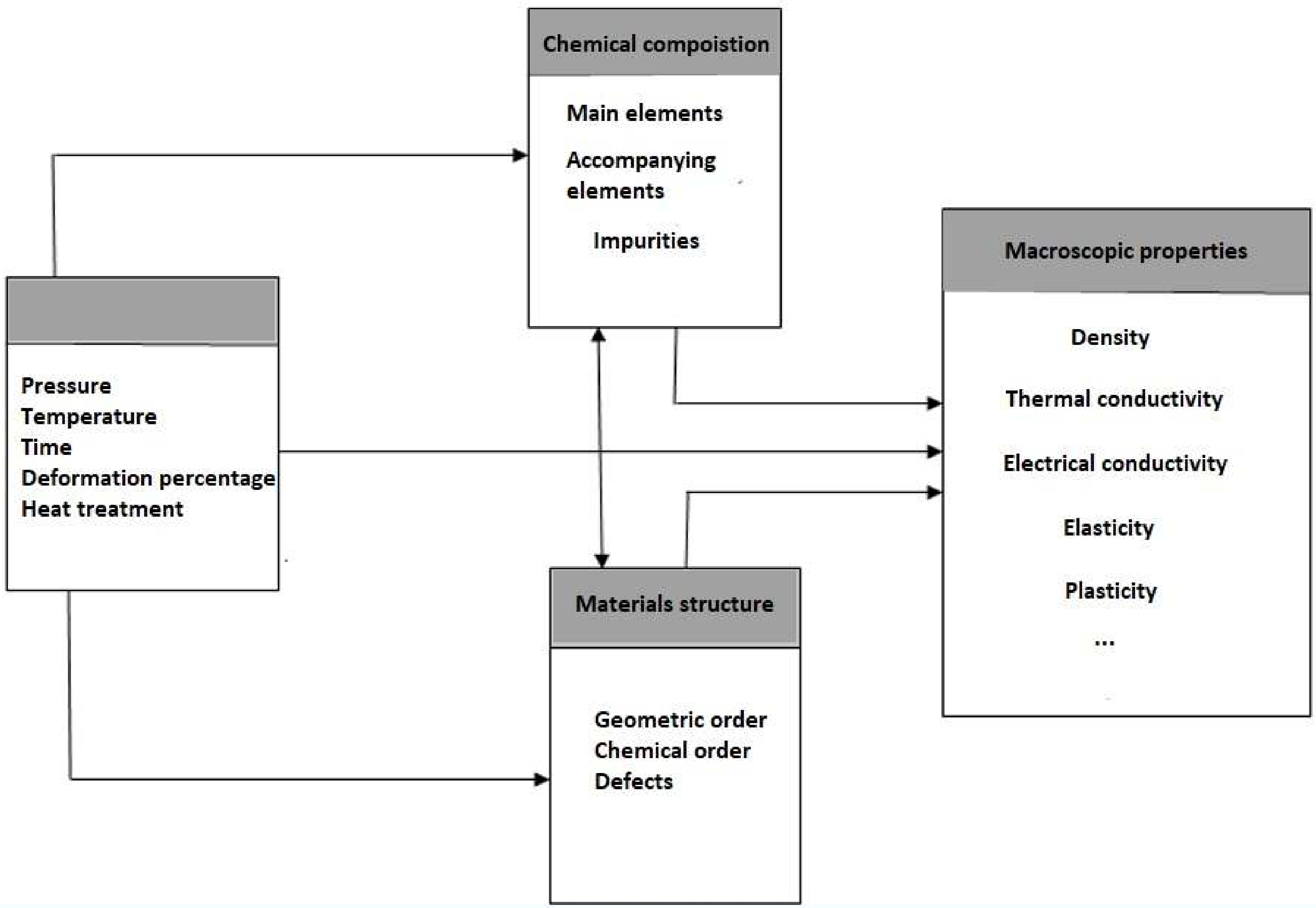
- ✓ Methods of materials analysis are used for characterization and study of raw materials, production processes and microstructure. These methods do not directly examined the properties of matter (mechanical, chemical, physical, etc.), but are directly associated with these properties. These properties are affected by the three themes.
- ✓ On the other hand, these factors affect one another, so it is necessary to be able to perform analysis and characterization operations with the help of certain methods and equipment. For example, in the characterization of the raw materials, the type and amount of impurities, the shape and distribution of the particles and the crystal structure are effective and they affect the quality of materials.

Importance and Necessity of Materials Characterization Science

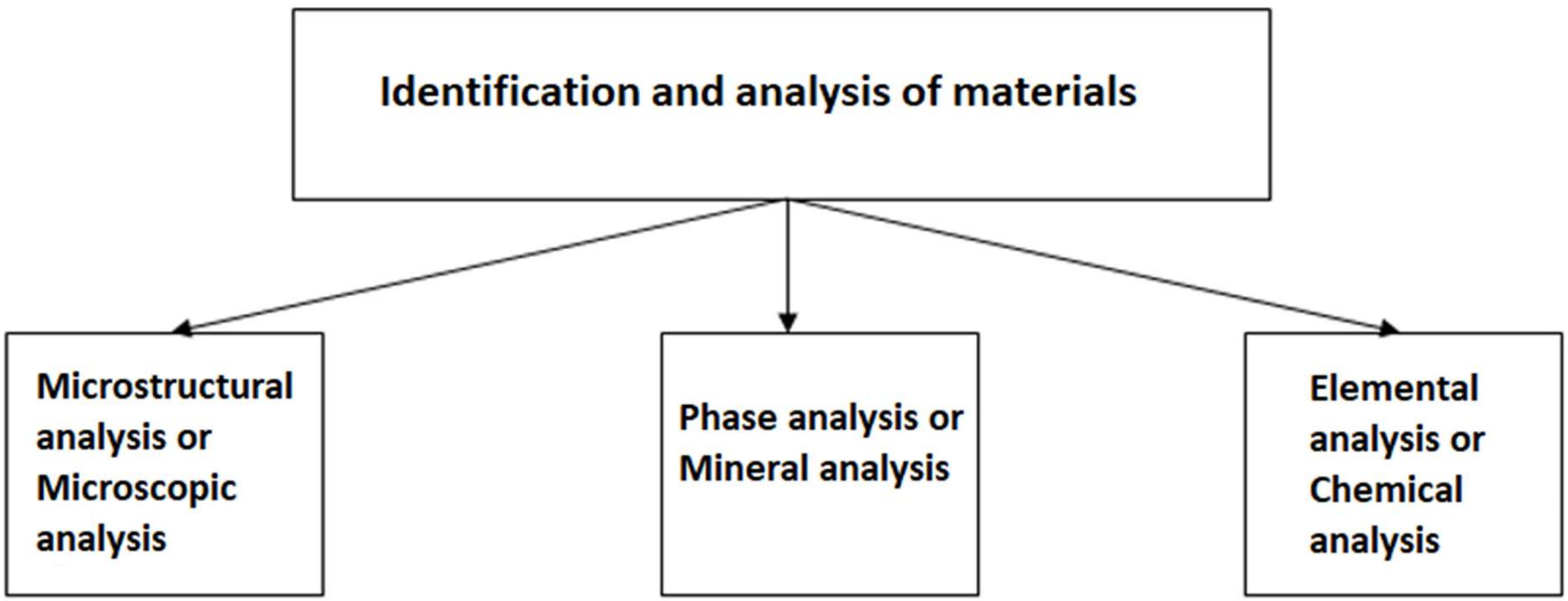
- ✓ In the microstructure or macrostructure structure, the type of phases, shape, size, and distribution were investigated.

- ✓ It is necessary to control the production process and ensure the stability of the final product features as well as the application of analysis methods and characterization at different production stages.

Dependent of Properties of Materials from Raw Materials, Manufacturing Process and Microstructure



Classification to Materials Characterization and Analysis Methods



Classification to Materials Characterization and Analysis Methods

- ✓ There is another type of analysis that explores the issue from another perspective. This analysis is a surface analysis.

- ✓ In cases, the surface of the solid material does not have the same chemical state with the sample bulk. For example, in the presence of catalysts and corrosion-resistant coatings, the sample surface plays a more important role and the chemical composition of the surface can be different from the internal parts of the sample.

- ✓ With this view, the characterization and analysis methods are divided into six broad categories.

Classification to Materials Characterization and Analysis Methods

1. ATOMIC SPECTROSCOPY BASED METHODS

- ✓ Auger Electron Spectroscopy (AES)
- ✓ Atomic Absorption Spectroscopy (AAS)

2. X-RAY BASED METHODS

- ✓ X-Ray Diffraction (XRD)
- ✓ X-Ray Fluorescence (XRF)

3. ELECTRON BEAM BASED METHODS

- ✓ Scanning Electron Microscopy (SEM)
- ✓ Transmission Electron Microscopy (TEM)

4. THERMAL ANALYSIS METHODS

- ✓ Termo-Gravimetric Analyze (TGA)
- ✓ Differential Scanning Calorimetry (DSC)
- ✓ Simultaneous Thermal Analysis (STA)
- ✓ Differential Thermal Analyze (DTA)
- ✓ Thermo-Mechanical Analyze (TMA)

5. SURFACE ANALYSIS METHODS:

- ✓ Scanning Tunneling Microscope (STM)
- ✓ Atomic Force Microscopy (AFM)
- ✓ X-Ray Photoelectron Spectroscopy (XPS)
- ✓ Magnetic Force Microscopy (MFM)
- ✓ Auger Electron Spectroscopy (AES)
- ✓ Spational Light Modulators (SLMS)

6. ANALYSIS METHODS OF CARBON, HYDROGEN, NITROGEN, OXYGEN AND SULFUR

- ✓ CHNOS

Methods of Chemical Analysis of Engineering Materials

Ceramics, glass Elementary / General			
Qualitatively		Quantitative	
High / low	Minor / Very Minor	High / low	Minor / Very Minor
FTIR (3,4)	FTIR(3,4)	AAS	AAS
IC (3)	IC(3)	FTIR(3,4)	FTIR (3,4)
ICP-AES	ICP-AES	IC(3)	IC (3)
IR (3,4)	IR(3,4)	ICP-AES	ICP-AES
OES	NAA	IR(3,4)	IR(3,4)
RS (3,4)	OES	OES	NAA
SSMS	SSMS	RS(3,4)	OES
XRS		SSMS	SSMS
		XRS	

Methods of Chemical Analysis of Engineering Materials

Metals and alloys Elementary /General			
Qualitive		Quantitive	
High/ low	Minor/ Very minor	High/ low	Minor/ Very minor
IC (3)	ESR (3)	AAS	AAS
ICP-AES	FTIR (5)	IC (3)	COMB(3)
NAA	IC (3)	ICP-AES	ESR (3)
OES	ICP-AES	IGF (3)	FTIR (5)
SSMS	IR (5)	NAA	IC (3)
XRS	NAA	OES	ICP-AES
	OES	SSMS	IGF (3)
	SSMS	XRS	IR (5)
			NAA
			OES
			SSMS

Methods of Chemical Analysis of Engineering Materials

Polymers and plastics General / macroanalysis							
Elementary				Molecular / composition			
Qualitive		Quantitive		Qualitive		Quantitive	
High/ low	Minor/ Very minor	High/ low	Minor/ Very minor	High/ low	Minor/ Very minor	High/ low	Minor/ Very minor
COMB (3)	COMB (3)	COMB (3)	COMB(3)	EFG	FTIR	EFG	FTIR
EFG	ESR (3)	EFG	ESR (3)	FTIR	IC (3)	FTIR	IC(3)
ESR (3)	IC (3)	ESR (3)	IC (3)	IC (3)	IR	IC(3)	IR
IC (3)	MFS (3)	IC (3)	MFS (3)	IR	LC (4)	IR	LC(4)
MFS (3)	NAA (3)	MFS (3)	NAA (3)	LC (4)	MFS (3)	LC (4)	MFS(3)
NAA (3)	UV/VIS	NAA (3)	UV/VIS	MFS (3)	UV/VIS	MFS (3)	UV/VIS
NMR (3)		UV/VIS		NMR (3)		RS	
UV/VIS		XRS (3)		RS		UV/VIS	
XRS (3)				UV/VIS			
				XRD (4)			

- The numbers 1 to 6 mentioned in the tables indicate the following:
- 1. The concentration range is defined as follows: High: 10 wt.%, Low: 0.1 to 10 wt.%, Minor: 0.0001 to 0.1 wt.%, Very Minor: Less than 0.0001 wt.%
- 2. Wet chemical analysis, electrochemical, UV/VIS and MFS are commonly used to perform a number of general analyzes.
- 3. They can be used for a limited number of elements.
- 4. Under certain conditions
- 5. Semiconductors only
- 6. In inorganic solids, most methods (techniques) can be applied to any type of residue after combustion.

General Analysis Methods

1- Atomic Absorption Spectrometry (AAS)

Applications:

Quantitative analysis about 70 elements. The study range is from ppm (one part per million) to ppb (one part per billion).

Limitations:

This method cannot be used directly for sulfur, carbon and nitrogen analysis, and is essentially a single element.

General Analysis Methods

2- High-Temperature Combustion (COMB)

Applications:

Determining carbon and sulfur in metals and organic materials.

Limitations:

- The sample must be homogeneous.
- It's a destructive technique.

General Analysis Methods

3- Elemental and Functional-Group Analysis (EFG)

Applications:

- Identification of organic compounds and determine their formulas.
- Determine the composition of a mixture.
- Determining purity.

4- Electron Spin Resonance (ESR)

Applications:

- Identification of the elements of the transition metals group.
- Identification of the capacity of ions of transition elements.

Limitations:

The sample should contain a high concentration of paramagnetic centers (transition ions, free foundations, defect centers, etc.).

General Analysis Methods

5- Ion Chromatography (IC)

Applications:

Quantitative and qualitative analysis of the wide range of organic and organic anions and some cations in aqueous solutions. The range of examination below ppm for a number of ions and ppb under ideal conditions.

Limitations:

It must be ionic in solution and soluble in water.

6- Inductively Coupled Plasma Atomic-Emission Spectroscopy (ICP-AES)

Applications:

Multi-element analysis simultaneously (quantitative and qualitative) for more than 70 elements in large, low, and minor values. The study range is from ppm to ppb.

Limitations:

It does not have the ability to analyze noble gases. The halogens and some non-metals require a spectrometer and a vacuum optics. Its sensitivity is week for alkali metals, especially rubidium, and it cannot determine the cesium.

General Analysis Methods

7- Inert-Gas Fusion (IGF)

Applications:

Quantitative analysis of oxygen, nitrogen and hydrogen in metallic and non-metallic materials.

Limitations:

- The metals with the low boiling point require precautions.
- For materials with stable oxides or nitrides, the flux should be added.
- It is a destructive technique.

8- Infrared Spectroscopy (IR)

Applications:

- Identifying and determining the structure of organic and organic materials.
- A quantitative determination of molecular creators in the blends.

Limitations:

- Low information about elements.
- The molecule must have a suitable dipole in one of their domain vibrations.

General Analysis Methods

9- Fourier-Transform Infrared Spectroscopy (FT-IR)

This method is in fact a IR spettroscopy computer technique that provides information on the full-infrared spectrum of the sample.

10- Liquid Chromatography (LC)

Applications:

Isolation and quantitative analysis of constituents in organic and inorganic mixtures. Analysis of organic and inorganic compounds to detect impurities.

Limitations:

Solids should dissolve easily in the solvent. Clear identification of a particular constituent is difficult and may require further analysis by IR or solid spectrometry.

General Analysis Methods

11- Molecular Fluorescence Spectroscopy (MFS)

Applications:

Quantitative and qualitative chemical analysis. The detection range for highly fluorescent chemical samples can be wide from nanomolar to sub-picomolar depending on the type of device used.

Limitations:

The desired molecules (or atoms) should exhibite natural fluorescence or be paired with other chemical species or changed by physical or chemical reactions.

12- Neutron Activation Analysis (NAA)

Applications:

Non-destructive measurement of partial percentage of element for each substance. Extremely high sensitivity for quantitative analysis 10^{-12} g/g but destructively.

Limitations:

Some elements are not detectable (except by irradiation).A high-intensity neutron source is required. Intense radioactivity is created in one or more major elements that can cause the presence of some or all of the minor elements to disappear.

General Analysis Methods

13- Nuclear Magnetic Resonance Spectroscopy (NMR)

This method is also known as magnetic resonance imaging.

Applications:

Quantitative analysis of specific ingredients and subgroups.

14- Optical Emission Spectroscopy (OES)

Applications:

Quantitative determination of constituent elements in high and small percentages. Quantitative analysis of the element.

Limitations:

Determining some elements such as nitrogen, oxygen, hydrogen, halogens and noble gases is difficult or impossible.

15- Raman Spectroscopy (RS)

Applications:

Molecular analysis of bulk samples.

Limitations:

Its sensitivity to small amounts is weak without amplification.
Requires concentrations higher than about 1 to 5 percent.
Fluorescence samples or impurities may interfere with Ramen's characteristic.

16- Spark-Source Mass Spectroscopy (SSMS)

Applications:

- Quantitative and qualitative analysis of inorganic elements.
- Measurement of minor impurities in materials.
- Check the composition of alloys.
- The range for most materials is ppb.

Limitations:

- It is not usually used to measure gaseous elements.
- Chemical preparation of the sample can cause major contamination.

17- Ultraviolet Visible Absorption Spectroscopy (UV/VIS)

Applications:

Quantitative and qualitative analysis, especially for organic compounds.

Limitations:

The specified material must absorb the radiation from 200-800 nm or can be converted in such a way that it can absorb the radiation in this range. Additional steps are often required to eliminate or calculate interferences caused by organic species that absorb radiation close to the breakdown wavelength.

18- X-Ray Diffraction (XRD)

Applications:

Identification of compounds using powder and single crystal methods.

Limitations:

The sample of analysis must be crystalline.

19- X-Ray Spectrometry (XRS)

Applications:

- Determining the elementary quantity and quality.
- The range of study used to determine the bulk with respect to X-ray energy and the composition of the sample field is from a few ppm to tens of ppm.

Limitations:

- Not suitable for elements with atomic number less than 11.
- Elements up to atomic number 6 may be determined using special equipment.

SYNCHROTRON

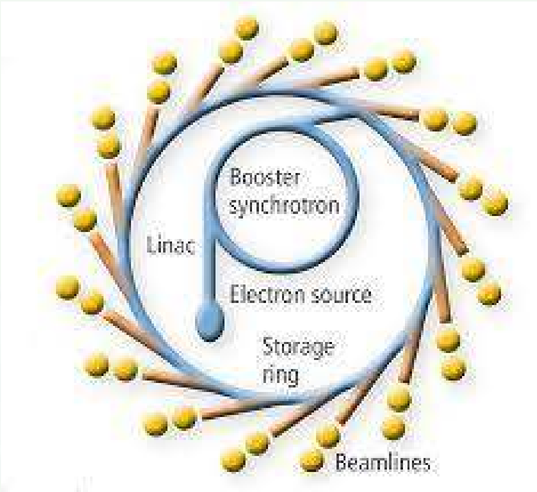
- ✓ A synchrotron is a type of circular particle accelerator. It works by accelerating charged particles (electrons) through sequences of magnets until they reach almost the speed of light.

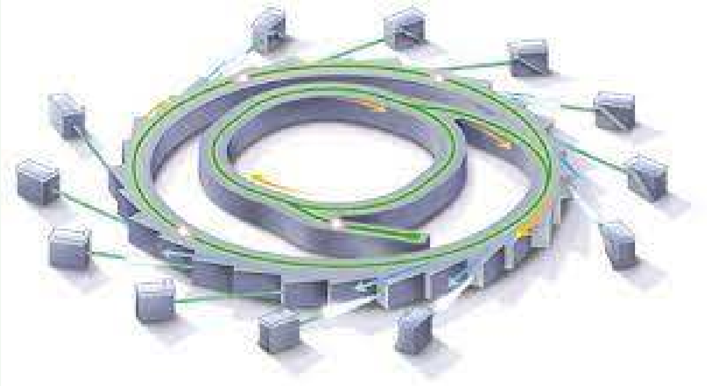
- ✓ With these high-speed particles and synchrotron radiation, the characterization and analysis of different materials, imaging, study of molecular and cellular structures, etc. can be studied with much higher accuracy than the accuracy of conventional devices.

SYNCHROTRON

- ✓ Today, synchrotron radiation is widely used in industrial research and one of the main users of synchrotron laboratories in the world are industrial companies.
- ✓ Oil and gas, materials and metallurgy, food, cosmetics, pharmaceuticals, mining, agriculture, environment and archeology are some of the many applications of synchrotron radiation.

SYNCHROTRON





Company Logo

SYNCHROTRON

Company Logo

SYNCHROTRON

Some Methods Used in Synchrotron

- ✓ SMALL ANGLE X-RAY SCATTERING/DIFFRACTION (SAXS)
- ✓ WIDE ANGLE X-RAY SCATTERING/DIFFRACTION (WAXS)
- ✓ EXTENDED X-RAY ABSORPTION FINE STRUCTURE (EXAFS)
- ✓ X-RAY ABSORPTION FINE STRUCTURE SPECTROSCOPY (XAFS)
- ✓ X-RAY ABSORPTION NEAR EDGE STRUCTURE (XANES) AND QUICK XANES
- ✓ NEAR ATMOSPHERIC PRESSURE PHOTO-EMISSION (NAPP)
- ✓ HIGH RESOLUTION POWDER DIFFRACTION
- ✓ PHOTOEMISSION SPECTROSCOPY (PEEM)
- ✓ MICRO-COMPUTED TOMOGRAPHY
- ✓ X-RAY MAGNETIC CIRCULAR DICHROISM (XMCD)
- ✓ SOFT X-RAY SPECTROSCOPY/MICROSCOPY
- ✓ X-RAY REFLECTIVITY (XRR)

SYNCHROTRON

Some Methods Used in Synchrotron

- ✓ GRAZING INCIDENCE X-RAY DIFFRACTION (GIXD)
- ✓ SURFACE X-RAY DIFFRACTION (SXRD)
- ✓ GRAZING INCIDENCE SMALL ANGLE X-RAY SCATTERING (GISAXS)
- ✓ MACROMOLECULAR CRYSTALLOGRAPHY (MX)
- ✓ HIGH-ENERGY X-RAYS OR HEX-RAYS
- ✓ SPRING-8 ANGSTROM COMPACT FREE ELECTRON LASER
- ✓ SURFACE-EXTENDED X-RAY ABSORPTION FINE STRUCTURE (SEXAFS)
- ✓ ANGLE-RESOLVED PHOTOEMISSION SPECTROSCOPY (ARPES) AND NANO-ARPES
- ✓ LIGAND K-EDGE SPECTROSCOPY
- ✓ X-RAY ABSORPTION SPECTROSCOPY (XAS)

SYNCHROTRON

Some Methods Used in Synchrotron

- ✓ X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)
- ✓ NEAR EDGE X-RAY ABSORPTION FINE STRUCTURES (NEXAFS)
- ✓ X-RAY FLUORESCENCE (XRF)
- ✓ HAXPES: HARD X-RAY PHOTOELECTRON SPECTROSCOPY
- ✓ NEXAFS: NEAR EDGE X-RAY ABSORPTION FINE STRUCTURES
- ✓ XSW: X-RAY STANDING WAVES
- ✓ PHD: PHOTOELECTRON DIFFRACTION
- ✓ EDD: ENERGY DISPERSIVE DIFFRACTION
- ✓ PCI: PHASE CONTRAST IMAGING
- ✓ USAXS: ULTRA SMALL ANGLE X-RAY SCATTERING
- ✓ IMAGING MICROFOCUS SPECTROSCOPY INFRARED