

# Investigation the properties of nanostructured materials

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**Abstract:** Elements have various characteristics that require precise tools and devices to determine each of them, EDS (Energy Dispersive Spectroscopy) system, or (Energy Dispersive X-ray Spectroscopy) EDAX or as a tool for energy dispersive spectroscopy and system ( Wavelength Dispersive X-ray Spectroscopy (WDS) is introduced as wavelength dispersive spectroscopy. These systems are used as peripheral equipment of the scanning electron microscope (SEM). In EDS spectroscopy, by measuring the energy of X-rays emitted from the sample, it is possible to examine the sample quantitatively and qualitatively. In WDS spectroscopy, the emitted X-ray waves are classified and separated based on wavelength and analyzed. Using these methods, quantitative and qualitative analyzes can be performed on the sample, and the type and amount of elements in the sample can be determined.

**Keywords:** nanostructured materials, energy dispersive spectroscopy, energy dispersive X-ray spectroscopy, elements

## 1. INTRODUCTION

The elements have various specifications, and accurate tools and equipment are needed to determine each of them. Microscopic, structural, elemental, bonding, classical identification methods and methods for determining surface characteristics are among the identification methods that are used for this purpose. The basis of some of these objects is the collision of the electron with the substance, the result of which is shown in the form of a picture, spectrum or graph. By examining these results, information is obtained about the dimensions, shape, types of connections and the porosity of the materials.

The collision of an electron with a material includes various interactions, one of which is the excitation of electrons in the internal level of the material. Excited electrons return to the basic state through the production of Auger electrons and X-ray production, and by measuring each of them, some characteristics of the material, such as its shape, size, structure, and chemical composition, can be obtained. Research related to investigating the properties of nanostructured materials, the scanning electron microscope (SEM) is one of the most important and widely used devices that works based on the interaction of electrons with matter, and in most studies, this

microscope is used to determine the size and shape of nanostructured materials. There are different types of identification methods used in electron microscopes, which include Auger Electron Spectroscopy (AES<sup>7</sup>), Wavelength Dispersion Spectroscopy (WDS), Energy Dispersion Spectroscopy (EDS) and Electron energy depletion spectroscopy (EELS<sup>8</sup>). The information obtained from these methods is used for quantitative and qualitative investigation of chemical compounds. In these methods, the detection of elements with high atomic number is easily possible, but there is a problem in the case of elements with low atomic number (especially in small concentrations). In this study, two identification methods, WDS and EDS, have been studied.

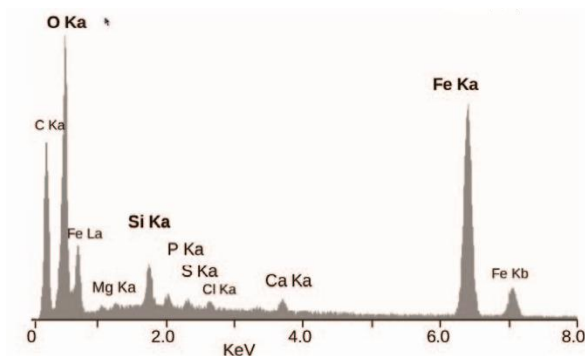


Figure 1: A sample EDS spectrum

## 2-The basis of work of EDS and WDS systems

In the EDS system, the sample is bombarded by an electron beam. Due to the impact of electrons on the sample, some of the electrons of the atom are displaced. In order for the atom to reach the equilibrium state, the electron migrates from the higher levels to the created vacancy and fills the vacancy. To perform this action, the electrons of higher levels, which have more energy, must lose a part of their energy to reach the energy level of the new level and become stable, in which case, the energy is emitted in the form of X-rays.

The amount of energy released depends on the levels from which the electron is separated or migrated to. On the other hand, the atoms of each element emit X-rays (with a unique amount of energy) while moving from one level to another. Therefore, by measuring the amount of X-ray energy released during the electron bombardment of a sample, it is possible to determine the type of atom present in it, and the results are shown as an EDS spectrum (Figure 1). This diagram is drawn based on the amount of receiving X energy from each energy level. Each of the peaks shown in this diagram is assigned to a specific atom. Peaks with a higher height mean a higher concentration of the desired element in the sample.

An important point regarding the EDS spectrum is the difference in the type of X-ray released. For example, if the electron migrates from the L level to the K level, the peak of the released X-ray is called the K-Alpha peak, and the peak caused by the electron going from the M level to the K level is called the K-Beta peak (Figure 2).

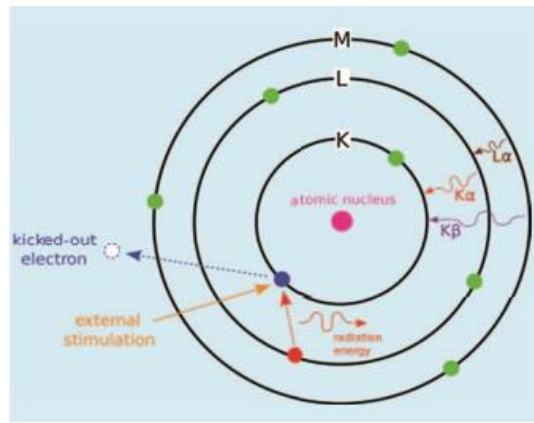


Figure 2: Show different electronic levels

The working method of the WDS system is similar to the EDS system. In this system, an X-ray analyzer crystal is used, which provides the possibility of different wavelengths. This operation is usually performed under vacuum conditions to reduce the absorption of soft radio waves in the air (photons with low energy) and increases the sensitivity to detect light elements (elements between boron and oxygen). This system is very sensitive and accurate for determining the structure and chemical composition of materials. Therefore, like the EDS system, each peak indicates an element.

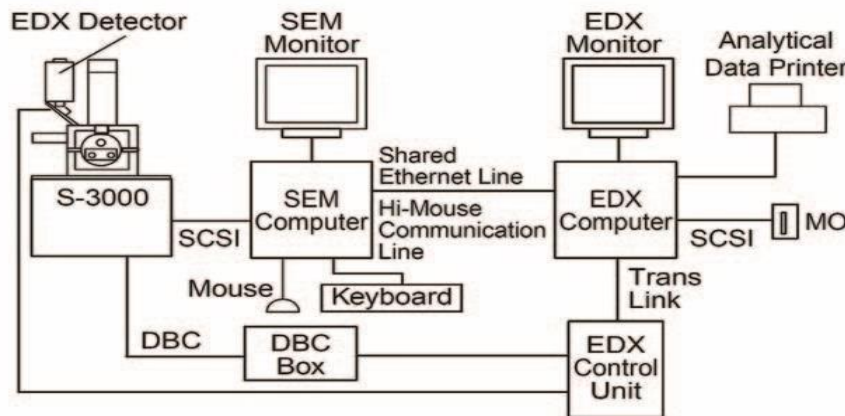


Figure 3- A view of the EDS system

### 3-Application of EDS system

The wavelength of the produced X-ray depends on the type of material under investigation and is a suitable criterion for the chemical identification of the material. The use of the EDS system in the scanning electron microscope requires a sample with a large thickness. Therefore, in this method, the electron beam penetrates in a large part of the desired area and gives an average value that is not suitable for identifying fine structures.

The X-ray energy values obtained from the EDS chart are compared with the standard X-ray energy values of each element to determine the presence of the element in the sample and qualitative identification of the sample. With this method, elements

with atomic numbers ranging from beryllium to uranium can be identified. EDS system cannot detect the presence of elements with atomic number less than 5.

3-1. The following are the limitations of the EDS system:

- Quasi-quantitative measurement of samples that are not flat, polished and uniform cannot be done easily because the unevenness prevents X-ray photons from reaching the hollow parts;
- There is a limitation in the sample size;
- The sample must be identifiable in a vacuum environment;
- For elements with an atomic number less than 5, due to the absorption of low-energy photons by the windows, the sensitivity is low;
- Due to the small spatial angle of detection, the speed is relatively low;
- The existence of problems caused by constant cooling of the detector, which causes the need for constant charging of liquid nitrogen;
- In some examples, disturbing peaks are created, and the detection of these disturbing peaks requires proper analysis or the high power of the analyzing software;
- In qualitative examination, lines less than 100-200 electron-volts cannot be detected. Because in addition to the overlapping of two adjacent peaks, the height of the peaks cannot be easily recognized from the background.

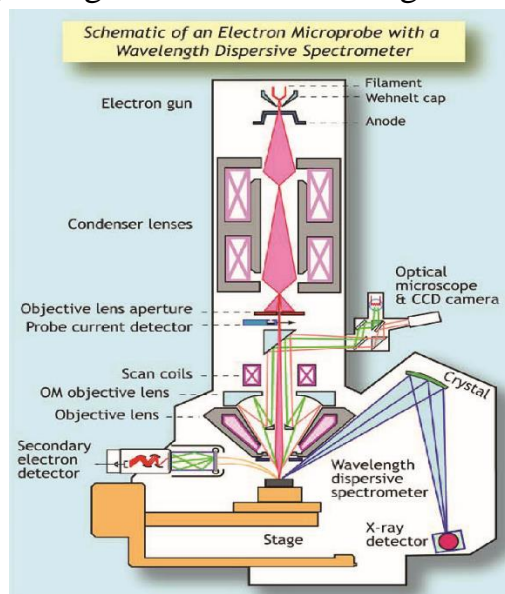


Figure 4-Schematic of an electron microscope with a wavelength refraction spectrometer

#### 4-Application of WDS system

The spectrum obtained from the WDS system at high wavelengths has a suitable quality and its most common application is the quantitative identification of elements. Due to its high separation power, this system is sensitive to very small amounts of elements and is mostly used for the quantitative identification of elements with boron atomic number and higher. This method is used in natural and synthetic solid materials,

including mineral materials, glass, ceramic, metal, etc. The intensity of X-ray with any energy value determines the relative concentration of that element. The use of high voltage shifts the graph to higher energies and makes the peaks with higher energy larger; While the peaks with less energy become smaller.

4-1. Among the limitations of the WDS system, the following can be mentioned:

- Using the WDS system for uneven samples is not suitable, because the unevenness prevents X-ray photons from reaching the hollow parts;
- The WDS system cannot identify elements below the Bohr atomic number;
- Despite the separation of the spectrum of elemental peaks, overlapping peaks can create problems in their identification;
- This system does not have the ability to identify elements in the excited state (2+Fe versus 3+Fe) and another method is used to identify them, such as mass spectrometry;
- This system also does not have the ability to identify isotopes. To identify them, mass spectrometry can be used;
- Due to the need for high vacuum ( $10^{-8}$  torr), this method causes damage to sensitive samples, such as some organic and biological compounds;
- It has a relatively low speed, but the use of 9CCD array detectors along with the above system reduces this problem;
- For the purpose of quantitative identification, it takes a lot of time to gather information for sufficient electronic statistics. If the information is sufficient, it is possible to determine the thickness of the sample and the chemical composition of the elements (in addition to its type and concentration).

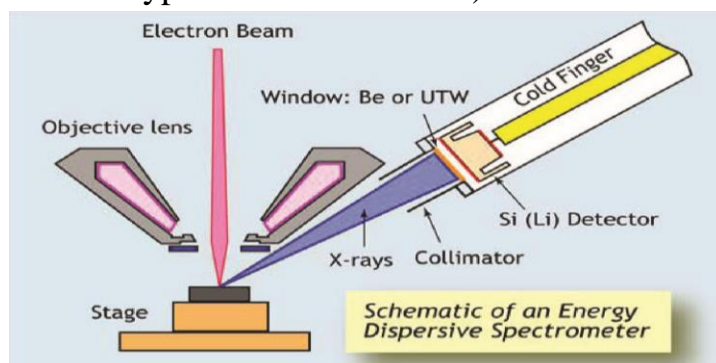


Figure 5- Schema of energy refraction spectrometer

#### 5-Advantages of using EDS and WDS systems

- Versatile use, cheap and widely accessible;
- Ability to use a little for some samples (flat, polished and uniform);
- Quick and easy identification, due to the large spatial angle of the detector and the collection of all rays with different energies at the same time;
- The rarity of the overlap of adjacent peaks and the ability to identify elements with X-ray photon energy close to each other;



- Quantitative measurement of the concentration of elements with high accuracy due to the high ratio of the height of the peak to the background;
  - Reducing the detection limit, if several crystals are used at the same time when identifying elements;
  - High detection speed if the type of element is known, by keeping the angle fixed at a certain value.
  - For high-quality identification in multiple samples, a special device called Electron Probe Microanalysis (EPMA10) has been designed, which in some cases has the ability to install two systems simultaneously on the device.
- #### 6-Comparison of EDS and WDS systems
- Separability in low concentrations with WDS system is better than EDS system (0.05% against 0.1%);
  - The separation of peaks is done better using the WDS system than the EDS system, which has overlapping peaks;
  - The disturbances in the field are less in WDS peaks, which provides the possibility of more accurate quantitative identification;
  - Using the WDS system is more time-consuming than the EDS system and has a higher cost;
  - Using WDS system compared to EDS causes more damage to the sample and container; Because X-rays with more energy are needed.

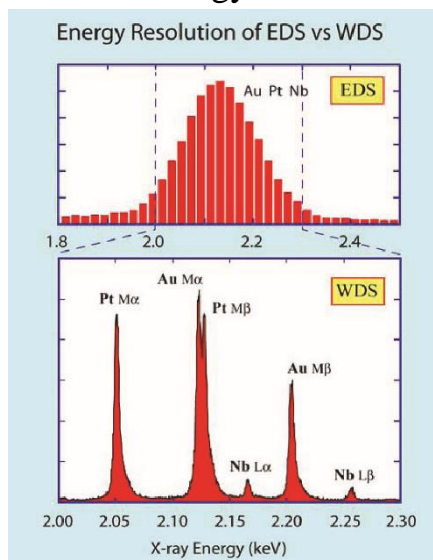


Figure 6- Comparison of EDS and WSD spectra in energy separation

#### 7-CONCLUSION

In the research related to investigating the properties of nanostructured materials, the electron microscope is one of the most important and widely used devices for determining the size and shape of nanostructured materials. By means of this device, the size and shape of particles in the nano scale is obtained, and the type of material and identification method used in this study play an important role. The scanning

electron microscope device works based on the collision of the electron with the sample and its return. Returned electrons from the surface of the sample recorded by different systems form the image of the surface of the sample. Also, this device is used to identify elements that can be used with different resolution and magnification in different samples (mining and geological samples related to nano, biological, metallurgical studies, etc.). In addition, by installing lateral detection equipment such as EDS and WDS, qualitative and quantitative analyzes can be performed to identify the type and amount of sample elements by studying the reflected rays from the sample.

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