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A REVIEW ON AUGER ELECTRON SPECTROSCOPY

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ABSTRACT

The Auger electron energies can be defined as the characteristics of the element from which the electron come. Auger electron Spectroscopy (AES) measures the energies of Auger electrons by identifying surface elemental compositions. This analytical technique has a high spatial resolution and sensitivity. It is a phenomenon which is physical in which electron transition of an electron in an atom filling in an inner-shell vacancy creates the emission of another electron. Electron from higher energy level will fall into vacancy when a core electron is removed leaving a vacancy results in a release of energy. Elemental concentrations are directly proportional to Auger electron peaks. Concentrations are possible measure from the first principles. Auger peaks are influenced by several instrumental factors like sample orientation, energy resolution, primary beam energy and acceptance angle of the analyzer. Methods such as X-rays or bombardment with argon ions and irradiation with electrons can yield an inner shell vacancy. AES is a wide spread method for analysis of thin films, interfaces and surface. It is one of the most commonly employed techniques in surface analysis in determining the composition of sample's surface few layers.

Keywords: Electron Peaks, Analyzer, Resolution, Sensitivity.

INTRODUCTION

Auger electron Spectroscopy (AES) measures the energies of Auger electrons by identifying surface elemental compositions. The Auger electron energies can be defined as the characteristics of the element from which the electron come. Auger electron emission is stimulated when the sample is bombarded with the electron beam. AES is a wide spread method for analysis of thin films, interfaces and surface. It is one of the most commonly employed techniques in surface analysis in determining the composition of sample's surface few layers. It utilizes the low energy electrons emission during Auger process [1].

When electrons with 3-20keV energy are incident upon a conducting sample, they eject the core electrons from atoms present in the sample which results in a photoelectron and an atom with a core hole. When the lower binding energy is dropped into the core hole the atom relaxes. This energy released is converted to emit an electron. This emitted electron is termed as Auger electron after Pierre Auger who first discovered this process of relaxation. After the Auger electron is emitted, a double

ionized state is seen in atom. Thus, the element is identified by the energy of the Auger electron which is unique. These Auger electrons form small peaks in total energy distribution function [2].

AES must be done in UHV conditions. In order to study the buried layers in a sample, combination of AES with a popular method called sputter cleaning is used. When a sample from air is brought into UHV conditions it is coated with carbon and oxygen. This material should to be removed usually by sputtering before investigating the clean surface. Sputtering technique involves the beam of ions usually Ar ions at between 500eV and 5keV at the sample. This process helps in cleaning but can also erode the sample away to reveal the surface beneath. So, this is a destructive technique. This technique is most sensitive to low atomic number elements but cannot detect helium or hydrogen.

Surface study plays a crucial role as any material interacts with external environment with their surfaces. Surface atoms determine the reactivity of the materials in

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chemical reactions like corrosion, catalysis etc.,

History of AES can be dated back to 1922, Lise Meitner, an Austrian physicist, discovered Auger emission process as a side effect in her search for nuclear beta electrons. Later on in 1923, the French physicist Pierre Auger discovered Auger electrons and process. AES is also known as Scanning Auger Microscopy [3].

Auger Theory

Elemental concentrations are directly proportional to Auger electron peaks. Concentrations are possible measure from the first principles. Auger peaks are influenced by several instrumental factors like sample orientation, energy resolution, primary beam energy and acceptance angle of the analyzer. The process of elemental analysis is also influenced by chemical states of elements in the sample. Especially as a function of oxidation state both peak intensity and peak shape vary.

For qualitative analysis sample heterogeneity must be considered. For measurement to be accurate, sample should be homogeneous in the lateral directions relative to the primary beam diameter. The diameter of the primary beam mainly influences the Auger signals that arise from an analytical volume. The meaningful analyses can be made on islands within a sample if the beam is narrower than the scale of heterogeneity. As Auger is highly surface sensitive the analytical volume thickness is small. So, the analyzed surface may not represent the bulk material. For example, thin oxide coating is acquired when metal samples are exposed to air. Quantification of elemental considerations is seen in cases where factors like sensitivity have been measured in the same samples matrix.

Quantification of major and minor elements is required in a typical Auger analysis. The consistent concentration range of Auger analytical detection limits is 1 to 0.01%. In an Auger measurement, the concentration of all elements including the matrix can vary so concentrations should be expressed as percents (CE %) normalized relative to the sum of all others [4].

$$C_E\% = 100\% [C_E / \sum C_X]$$

$$C_E = \text{RSF} [I_E / I_M]$$

C_E = Analyte Concentration

I_E = Analyte Ion Intensity

I_M = Matrix Element Ion intensity

RSF = Measured Relative sensitivity Factor (for the particular element and matrix)

By substituting the right side of the RSF equation for concentrations (CE and CX) in the first equation gives the following equation. This is the most common format for elemental quantification by Auger.

$$CE\% = 100\% [\text{RSF}_E (I_E) / \sum \text{RSF}_X (I_X)]$$

Principle of Auger Effect

It is a phenomenon which is physical in which electron transition of an electron in an atom filling in an

inner-shell vacancy creates the emission of another electron. Electron from higher energy level will fall into vacancy when a core electron is removed leaving a vacancy results in a release of energy. Some times energy is released in the form of an emitted photon; this energy can also be transferred to another electron which is ejected from the atom. This second electron which is ejected is called an Auger electron.

The ejection of the kinetic energy of the Auger electron corresponds to the difference between the ionization energy and initial electronic transition for the electron shell from which the Auger electron was ejected. The chemical environment in which the atom was located and the type of atom are also the factors which control the energy levels. By bombarding a sample with either energetic electrons or X-rays involves the emission of Auger electrons in AES and measures the intensity of Auger electrons as a function of the Auger electron energy. The information about the environment of atom and the emitting atoms identity is determined by the resulting spectra [5].

The Auger process

It has three steps involved. They are:

- 1) Atomic Ionization
- 2) Electron emission
- 3) Analyzing emitted Auger electrons

Ionization

Initiation of Auger process is done by creating a core hole which is typically carried out by sample exposure to a beam of high energy electrons typically with primary energy of 2-10 keV range. These electrons have sufficient capacity to ionize all levels of the lighter elements and higher core levels of the heavier elements. In few studies, using soft x-rays ($h\nu = 1000-2000\text{eV}$) the initial ionization process is carried out. In this method the acronym XAES is used sometimes. But the above change in the ionization method has no significant effect on the final Auger spectrum.

Relaxation and Auger Emission

After the removal of the core hole electron, the ionized atom which is in a highly excited state will rapidly relax back to lower energy state by any one of the following two routes:

1. Auger emission
2. X-ray fluorescence

When one electron from its higher level falls to fill an initial core hole which is in the k-shell liberates energy and this energy liberated during this process is transferred to a second electron and a fraction of this energy is used in overcoming the binding energy of the second electron and the remaining is retained as kinetic energy by the emitted Auger electron. The final stage of the atom is a doubly-ionized state with core holes in the L shells.

Kinetic energy can be determined by:

$$KE = (E_k - E_{L1}) - E_{L23}$$

The mechanism of initial core formation doesn't affect the kinetic energy of the Auger electron.

The above equation can be rewritten as:

$$KE = E_k - (E_{L1} + E_{L23})$$

From the above expression, it is clear that the latter two energy terms could be interchanged without any effect. So it is very difficult to say which electron occupies the initial core hole and which is ejected as an Auger electron.

So Auger transition is therefore characterized mainly by the following two:

1. Location of the final two holes and
2. Location of the initial hole

Fine structure in high resolution spectra may occur when there is a existence of different electronic states of the final doubly- ionized atom.

Working of AES

First with the help of electronic gun sample is irradiated with electrons. By using an electron spectrometer the energy of the emitted secondary electrons is measured. His whole process should be carried out in a UHV environment because the kinetic energy range of free path of electrons is limited to 20 to 2500 eV which makes the technique surface sensitive [6].

Requirements of Sample

The following parameters are to be studied:

Size

Particles with 1 micrometer diameter can be analyzed. Even 1.5cm in diameter and 0.5cm in height is also not uncommon. Specific instrument controls the maximum sample size.

Form

It should be solid in state like organic materials, ceramics with low vapor pressure i.e. <10-8 torr. Sample cooling helps in handling high vapor pressure materials. Liquid samples can also be analyzed by applying a thin film on conductive substrate or by sample cooling.

Topography of the Sample

Usually flat surfaces should be preferred. But rough surfaces can also be studied in averaged over large areas i.e. 0.5mm in diameter or in small selected areas which are equal to 1 micrometer.

Preparation of Samples

They should be free from oils, high vapor pressure materials and also fingerprints.

Analysis Time

Spectrum is estimated to obtain in 1 to 5 minutes. Depending on the required resolution, high-resolution

acquisition takes anywhere from 5 to 25min per region of the spectrum.

INSTRUMENTATION OF AUGER

It consists of both parallel electron detector and field emission electron source and the parallel detector simultaneously record electrons at eight separate energies. The major essential parts of an AES spectrometer are as follows:

1. UHV environment
2. Electron gun
3. Electron energy analyzer
4. Electron detector
5. Data recording, processing and output system.

UHV ENVIRONMENT

Every second on a surface an equivalent of one monolayer of gas impinges in vacuum of 10^{-6} torr, so surface analysis needs UHV environment. For highly reactive surface materials which show high sticking coefficient for most residual gases the contamination of the specimen surface is critical. In order to minimize the influence of residual gases in measurement of surface analysis, the background pressure is reduced [7].

Until 1960, production and measurement of UHV and constructing an apparatus which is suitable for operating in UHV environment remained as two main back draws which inhibited the advancements in surface analysis techniques. Later in early 1960s, standardized stainless steel hardware replaced the glass enclosures. Thus UHV environment is easily achieved by pumping suitable combinations of ion, turbo molecular, cryo and oil diffusion pumps into stainless steel chambers. Temperature in these chambers can also be raised upto 200°C to achieve UHV conditions.

ION GUNS

An AES system is commonly equipped with an argon ion beam. The Ar^+ ion beam is used to sputter the sample surface. The energy of the Ar^+ ions ranges from 0.1 to 5 keV. The ion gun is employed for:

- (1) Cleaning the sample surface, and
- (2) Depth profiling

Nature of an electron gun depends on several factors like desired spatial resolution, speed of analysis which requires high beam current and any changes to sample surface by beam induction.

Beam currents in the range of 10^{-9} to 5×10^{-6} is normally used in AES. Conditions where special resolution is less important high currents may be used to give high sensitivity and speed, where as low current gives high spatial resolution. Sometimes high current can cause surface damage so it should be avoided.

The two critical components of electric gun are focusing forming lens and electron source. In many cases thermionic electron source is used but if highest special

resolution is needed then brighter field emission source is used. As the field emission sources have certain problems like stability and cost so therefore has limited use.

SOURCES

AES has primary electron beam columns which are identical to electron microscope.

There are three types. They are:

Tungsten cathode source

It consists of a wire filament which is bent in the shape of a hairpin. By resistive heating the filament operates at $\sim 2700\text{K}$. These are widely used as they are inexpensive and also reliable. As the densities tungsten cathode current is only about $1.75\text{A}/\text{cm}^2$ lateral resolution is limited.

Lanthanum hexaboride (LaB6)

It can provide higher current densities as it has a greater emissivity and lower work function than tungsten. Current densities of $\sim 100\text{A}/\text{cm}^2$ are available at 2000K . Narrower electron beam is provided by higher current densities which is useful for analyzing smaller features.

Field emission electron source

It has very sharp tungsten points where electrical fields can be $>10\text{E}7\text{V}/\text{cm}$. Electrons at these fields can directly tunnel through the barrier and they leave the emitter with near zero work functions. These provide the brightest beams i.e. $\text{IE}3$ to $\text{IE}6\text{A}/\text{cm}^2$. Extremely clean tips help in obtaining the lower work functions. Work function can increase and electron emission can reduce with a single atom on the tip. Continuous heating i.e. $\sim 2000\text{K}$ and ultrahigh vacuum keeps the electron beam stable and tips clean. Small feature Auger analysis is provided by electron beam as narrow as 10nm . The Auger instrument columns may consist of both electrostatic and magnetic lenses for beam focusing. It also consists of octopole lenses for beam shaping and quadrupole deflectors for beam steering [8].

ELECTRON ENERGY ANALYZER

Electron energy analyzer disperses the secondary emitted electrons from sample based on their energies. It can be electrostatic or magnetic. As stray magnetic fields influence electrons, these fields are cancelled within the enclosed volume of the analyzer. Mu metal shielding helps in accomplishing this task. These are almost used in all commercial spectrometers nowadays as they have relative ease of stray magnetic field cancellation.

The Cylindrical Mirror Analyzer (CMA)

It consists of two coaxial cylinders with a ground potential applied to inner cylinder and negative potential (V) applied to the outer cylinder. The detector and the

samples are located along the common axis of the cylinders. CMA comprises a full cone about the spectrometer axis as it has very large acceptance angle. This helps in reduction of the signal on specimen topography and enhancement of sensitivity. By incorporating the electron gun into the electronic cylinder, system design can be made a compact unit. As CMA has a superior signal-to-noise capacity it is mostly used in modern AES systems.

Concentric Hemispherical Analyzer (CHA)

It has two hemispherical concentric shells with inner and outer radius. Between the two surfaces such that inner sphere is positive and outer sphere is negative, a potential difference V should be applied. There is an equipotential surface of radius r_0 between the two spheres. At a distance of r_0 from the centre of curvature lies the entrance and exit slits. The lens system which is present takes the electrons from the sample and then injects electrons into the analyzer. So the effective solid angle can be compared with that of a CMA. Its main advantage when compared CHA over CMA is to have ability to vary analyzer resolution electro statically without changing physical apertures and to have better access to sample. Sensitivity can be achieved by a large entrance angle. In order to reach a compromise between resolution and sensitivity it is most common practice to choose α such that $\alpha = w/2r_0$ [9].

DETECTORS

Single Channel Detector (SCD)

It is used in conventional instrumentation. It can be termed as electrostatic device which uses continuous dynode surface. To establish a condition for electron multiplication in needs only two electrical connections. Series of pulses that are fed into a pulse discriminator or amplifier and a computer adds to the output of this detector. Most important advantage is it remains unaltered or not damaged in spite of prolonged exposure to air for a long time. Even at zero kinetic energy, it counts electrons with a high efficiency and with the background is $0.1\text{count}/\text{sec}$ or lower. Only single drawback is saturation effect is seen during high count rate.

Multichannel Detector (MCD)

At the output of the analyzer a multiple detection system can be added. Across the whole analyzer output slit plane the system can be in the form of parallel, multiple, position-sensitive or equivalent detector chains. Such arrangements can be analyzed by methods like TV cameras, charged couple devices, discrete anodes, resistive anodes or phosphor screens.

Electron Multiplier

It consists of dynodes which are series of electrodes. Resistor string connects each electrode. The

potentials of dynode differ in equal steps along the chain. When a particle like high energy neutral, high energy photon, ion or electron strikes the first present dynode then it produces secondary electrons. Then to the next dynode secondary electrons are accelerated. So a cascade of secondary electron occurs. Again the secondary electrons are accelerated to the next dynode. And finally the cascade is collected at the anode. Detector has a dual micro channel plate which consists of a hole in the centre to accommodate the primary beam. The current finally resulting is electronically amplified and measured. The every electron as it arrives at the detector is measured like that of pulse counting mode. The eight concentric ring anodes present detect the electrons arriving with eight separate energies.

Data Recording

On a smooth background of secondary electrons, Auger electrons appear as peaks. Mains peaks are readily identified and visible if the specimen surface is clean. It is difficult to discern the smaller peaks and peaks those are caused by the trace elements which are present on the surface from the back ground, as it is normally sloping and also increases the gain of the electron detection system and by applying zero offset every time doesn't add an advantage. Therefore in differential form Auger spectra are recorded. The detailed structure which is not directly visible in the undifferentiated spectrum can be revealed in differential mode in order to enhance the system gain. The negative going feature is at high energy side of the peak in differential spectrum of Auger peak. The point of maximum slope of the parent peak is represented by the minimum of above mentioned feature. So energy of the transition can be defined by the above feature. The differential Auger spectrum has following advantages in the case of systems that record data in mode of analog. They are:

1. Direct peaks are not as sharp as differential peaks, thus it provides a reproducible energy reference point and
2. Amplifier can be easily changed as the background is zero away from the peaks.
3. Convenient measurement of peak intensity is done by the peak to peak height.

Now-a-days, in commercial instruments data is acquired digitally and can be presented in digital or analog mode. Computer controls the AES instruments generally. Acquiring and storing data are two major functions of the computer control system. Lock-in amplifier is used by the conventional Auger electron spectrometer. A sinusoidal modulation of frequency 10 to 20 kHz is superimposed by the oscillator in the amplifier on the potential applied to the outer cylinder of the analyzer. From the multiplier high voltage the AC component of the signal is decoupled and in the lock-in amplifier it is detected. Then to an X-Y recorder the output is fed. In order to yield a compromise between resolution and sensitivity, amplitude of the modulation has to be chosen.

Data is collected in the $N(E)$ versus E integral mode in a typical modern AES. Using a computer software, the data is mathematically differentiated to yield $dN(E)/dE$ versus E Auger spectra. In the constant retardation ratio (CRR), the CMA or CHA is used which helps in determining the resolution. Therefore, optimum modulation voltage will change while scanning a spectrum. High modulation is required for high energy peaks. While recording a spectrum, a constant voltage modulation may be used. Modulation is made proportional to analyzer energy by some instruments so that modulation to analyzer ratio is constant. So while comparing Auger spectra from different instruments this point should be considered [10-12].

Working of AES

Limited mean free path of electrons in the kinetic energy range 20 to 3000eV causes high surface sensitivity of AES. Auger electrons which lose the energy from the process like core excitations, plasma losses, interband transitions are removed from the observed Auger peaks and they contribute to nearly uniform background on which the Auger peaks are super imposed. Temperature doesn't affect the Auger yield. As incident electron beam energy, E_p is independent of the Auger electron escape depth and Auger transition probability, ionization cross section of the initial core level completely governs the dependence of the Auger peak amplitude. During the initial passage of incident electrons through the escape depth region which is of 5 to 25 angstrom thickness, the primary ionization occurs. When the binding energy is substantially less than incident beam energy, the backscattered primary electrons contribute to Auger yield.

Production of Auger Electrons

Methods such as X-rays or bombardment with argon ions and irradiation with electrons can yield an inner shell vacancy. For analytical purpose Auger lines are usually produced by electron impact which produces an intense beam that can be brought into a fine focus. Better peak to background ratios and less radiation damage are two assets of X-ray irradiation.

High Energy Satellite Lines

These are observed in Auger spectra of solids. Due to Plasmon gains the presence of these structures are interpreted. Resonance absorption or initial multiple ionization can be the reason for high-energy lines arising. An Auger satellite in solids is still under active considerations.

Energy loss characteristics

A characteristic energy loss is seen in electrons ejected from a solid which can be due to Plasmon losses. In photoelectron spectroscopy, it is much more difficult to disentangle peaks from characteristic energy from normal

Auger spectrum because these Auger spectra is usually very complex and very often not well resolved and these are spread over a considerable range of energies. Characteristic loss peaks nature is also altered by surface contamination [13-15].

Charging in non-conducting samples

Serious problem in AES is charging which occurs as a result of impinging beam of electrons on a non conductor. This charging and resulting non uniform surface potential can prevent good Auger spectra. By choosing proper angle of incidence and energy of primary electron beam, this problem can be overcome.

SERVICE AND MAINTENANCE

Calibration of the energy analyzer and routine adjustment are required. Maintenance is required for vacuum pumps. Only vacuum compatible samples are to be loaded. Plug in cards facilitate the detection of malfunctioning electronic components. The detector is expensive to replace. Electron gun filament must be replaced often. Majority of the replacements should be carried out by the trained personnel due to the design of the modules. The ion gun focusing components and the electron optics are not normally aligned. So they require regular attention to make sure that operation of the instrument is proper.

APPLICATIONS

Qualitative Identification of Elements

By recording the AES spectra over a wide range i.e. usually from 0 to 2000eV, elements from an unknown sample can be identified. Then the elemental values are compared with the kinetic energies of the intense peaks present in the spectrum.

Chemical Effects

Chemical state of the surface is studied by the AES peaks involving valence electrons. A simple peak shift, a peak shape change or both can represent the chemical shift. In AES interpretation is limited by beam-induced effects. Under electron irradiation many compounds like oxides are readily decomposed so the limitation of current density must be maintained.

Quantitative Analysis

Depending on the density of the material ionization of atoms occurs to a depth of 1 to 2 micrometers when the incident electron beam penetrates with both inelastic and elastic scattering. Therefore, the average concentration of the element and how it gets distributed within the first few layers of atom of the surface affects the AES signal observed. Because of strong inelastic scattering the electrons originating from the outer surface layer is greater than subsurface originating electrons.

Depth Profiling

It is a destructive technique. Here ion sputtering erodes the sample. It provides a convenient way of analyzing the composition of thin surface layers. Ion accelerated in the ion gun bombards the sample of energy in the range of 1 to 4KeV. Small amount of energy is transferred to the surface atoms when the energetic ion strikes the sample which causes them to leave the sample. Surface is exposed with ion beam for known time to remove a uniform layer of the sample. Layer removed can be calculated under controlled conditions. By giving the depth distribution of different species in the sample, the residual surface is analyzed. Inert gases mostly argon are commonly used as ion sources [16-19].

Other Uses

- Qualitative and quantitative determination surface elements
- Chemical states of elements determination
- To determine the chemical reactivity of states
- Elemental map of the Auger electrons in the system.
- Inert gas sputtering depth profiling
- To analyze the atomic concentration of elements
- Fingerprinting spectral analysis
- Deposited materials interface analysis.
- Analysis of chemisorptions of gases on metal surfaces

ADVANTAGES OF AES

- Rapid analysis
- Subsurface or surface analysis can be performed with ease
- Availability of chemical information in some cases
- For except hydrogen and helium it is sensitive to all light elements.
- High spatial resolution
- Reliable semi quantitative analysis

DISADVANTAGES OF AES

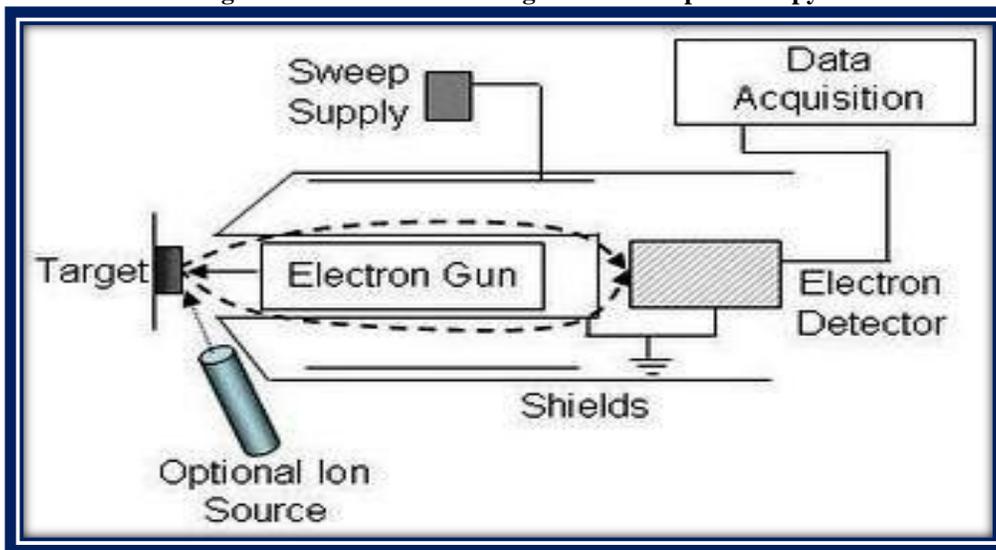
- Incident beam of electrons may damage the surface
- Extensive work is required for precise quantitative analysis
- Due to surface charging it is difficult to study insulators
- Ion sputtering or sectioning is destructive in the case of depth profiling.
- Modest sensitivity (0.1 to 1 atom %)

LIMITATIONS OF AES

- Quantification is not easy
- Only solid samples can be analyzed
- Samples which decompose under electron beam irradiation cannot be studied.
- Semiconducting and conducting sample analysis

- Special procedures are necessary for non-conducting samples.
- Three monolayer's depth sampling
- Auger spatial resolution is of the order 0.2 micrometers or less and a function of analysis time.
- The order of sensitivity is 0.3% [20].

Fig 1. Instrumentation of Auger Electron Spectroscopy



CONCLUSION

Auger electron spectroscopy has a great spatial resolution which is higher or equal to 100nm. It is a very useful technique in analyzing the surface layers composition to a depth of 2nm for all the elements except hydrogen and helium. It is used in studies like surface

segregation as in the stress- corrosion problems solving. This technique is very widely used in the studies of grain boundaries of alloys and metals and especially with the samples which are susceptible to brittle grain boundary fractures.

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