Auger Electron Spectroscopy (AES)

Silvia Natividad, Gabriel Gonzalez and Arena Holguin

1. Introduction

Auger Electron Spectroscopy (Auger spectroscopy or AES) was developed in the late 1960's , deriving its name from the effect first observed by Pierre Auger, a French Physicist, in the mid-1920's. It is a surface specific technique utilizing the emission of low energy electrons in the Auger process and is one of the most commonly employed surface analytical techniques for determining the composition of the surface layers of a sample.

The principal advantages of AES over other surface analysis methods are excellent spatial resolution (< 1 μm), surface sensitivity (~20 Å), and detection of light elements. Detection limits for most elements range from about 0.01 to 0.1 at%.

Auger electrons have relatively low kinetic energy, which limits their escape depth. Any Auger electrons emitted from an interaction below the surface will lose energy through additional scattering reactions along its path to the surface. Auger electrons emitted at a depth greater than about 2 - 3 nm will not have sufficient energy to escape the surface and reach the detector. Thus, the analysis volume for AES extends only to a depth of about 0.5 – 5 nm (See figure 1). Analysis depth is not affected by the energy of the primary electron energy.
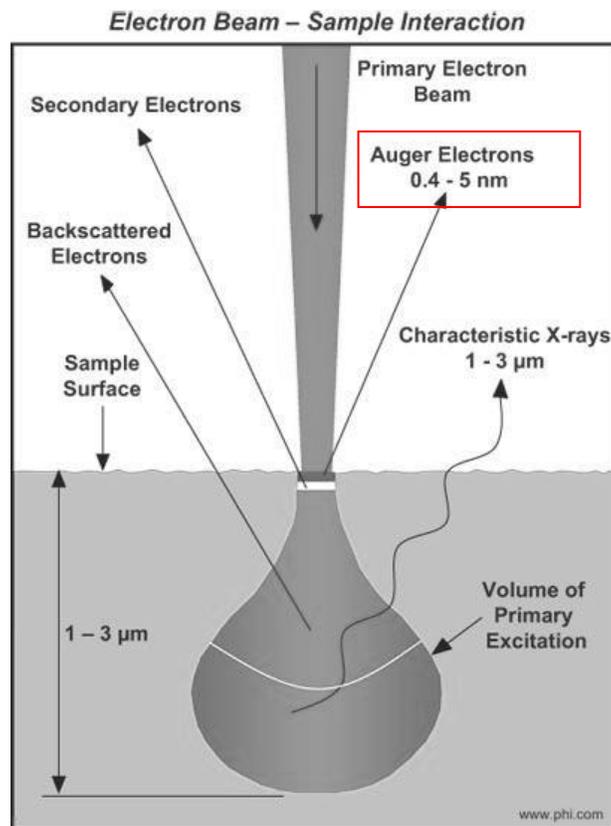

Figure 1 – Depth of Auger

On the other hand, AES cannot detect H or He. It does not do nondestructive depth profiling. It uses an electron beam as a probe, which can be destructive to some samples. It requires the sample to be put into and to be compatible with high vacuum. Some nonconducting samples charge under electron beam probing and cannot be analyzed. The sputtering process can alter the surface composition and thereby give misleading results. It does turn out to be the technique of choice, in its area, much of the time. The purpose of this article is to make clear what it can and cannot do and how to get the most information from it[1].

Auger spectroscopy involves three basic steps:

1. Atomic ionization (by removal of a core electron) as shown in figure 2. The Auger process is initiated by creation of a core hole – this is typically carried out by exposing the sample to a beam of high energy electrons (typically in the range 2 – 10 keV). Such electrons have sufficient energy to ionize all levels of the lighter elements, and higher core levels of the heavier elements. In some studies, the initial ionization process is instead carried out using soft x-rays ($h\nu = 1000 – 2000$ eV). In this case, the acronym XAES is sometimes used. This change in the method of ionization has no significant effect on the final Auger spectrum.
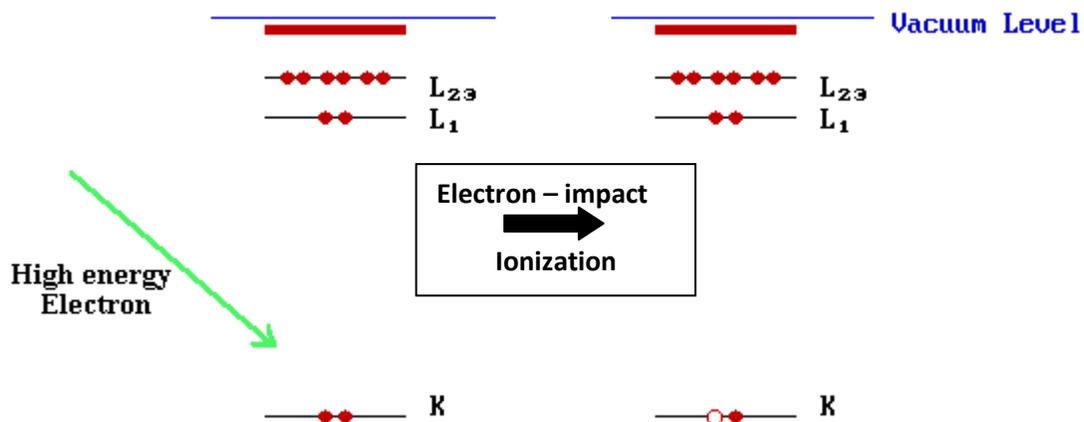

Figure 2 – Atomic ionization

2. Electron emission (the Auger process). The ionized atom that remains after the removal of the core hole electron is, of course, in a highly excited state and will rapidly relax back to a lower energy state by X-ray fluorescence, or Auger emission. In the figure 3 below, one electron falls from a higher level to fill an initial core hole in the K-shell and the energy liberated in this process is simultaneously transferred to a second electron; a fraction of this energy is required to overcome the binding energy of this second electron, the remainder is retained by this emitted Auger electron as kinetic energy.
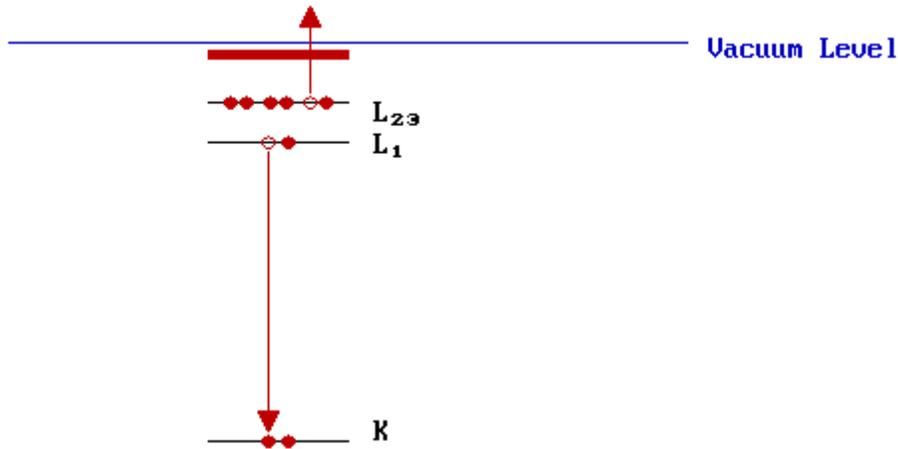
Figure 3 - Final state is a doubly-ionized atom with core holes in the L_1 and $L_{2,3}$ shells.

KE of the Auger electron from the binding energies of the various levels involved can be estimated as: **KE = (E_K - E_{L1}) - E_{L23}**
Auger Spectroscopy is based upon the measurement of the kinetic energies of the emitted electrons. Each element in a sample being studied will give rise to a characteristic spectrum of peaks at various kinetic energies.

2. The Physics of AES

Auger Electron Spectroscopy is a surface analysis technique that uses an incident electron beam generated from a tungsten or lanthanum hexaboride filament to eject Auger electrons from a sample for data acquisition. The process begins by ejecting a core electron from a lower energy level K, L, or M with the incident electron beam. An electron at a higher energy level then falls down to fill in the hole left behind by the initially ejected electron. The radiation energy produced by the electron that fell is then used by an Auger electron in order to be emitted from the sample. The electron transitions in the process of emitting an Auger electron are labeled according to the sequence of energy levels that are used. Figure 4 shows an example of a KL_1L_{23} transition where the initial electron is ejected from the K energy level, the second electron falls from the L_1 level, and the Auger electron is emitted from the L_{23} energy level. Thus, the Kinetic Energy in an Auger process can be calculated by subtracting the Energy of the two higher energy levels from the lowest energy levels. For the given example, the Kinetic Energy would be calculated as $KE=E_K-E_{L1}-E_{L23}$. Additionally, since AES is a three electron process, it can only detect elements with an atomic number, z, greater than or equal to 3.

The Auger electrons move outward in all directions from the sample surface and some enter the Cylindrical Mirror Analyzer (CMA.) A high vacuum chamber encloses the CMA and also shields it from any external magnetic fields. The CMA has an outer and inner cylinder in which the former has negative charge relative to the latter. The electrons scattered from the sample flow

through the space between the inner and outer cylinder. The negative potential between the two cylinders bends the electrons, which have the right velocity, toward the center axis of the CMA where they are sent through two apertures and into the electron multiplier which amplifies the signal for data acquisition. A schematic diagram of the general instrumentation is shown in Figure 5. (Whan) (Lopez, 2011)
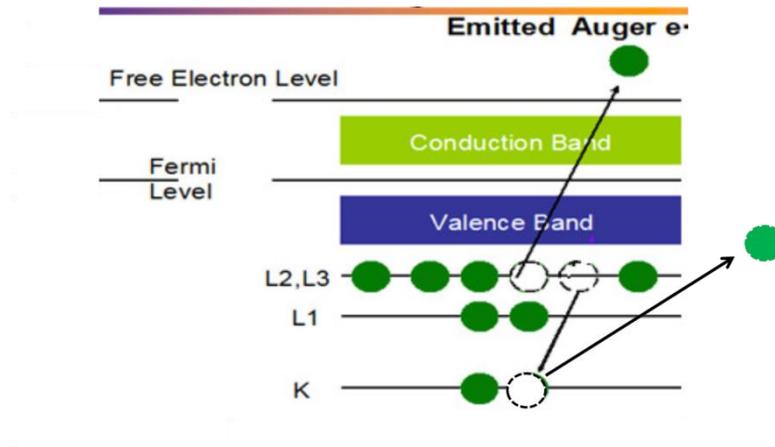

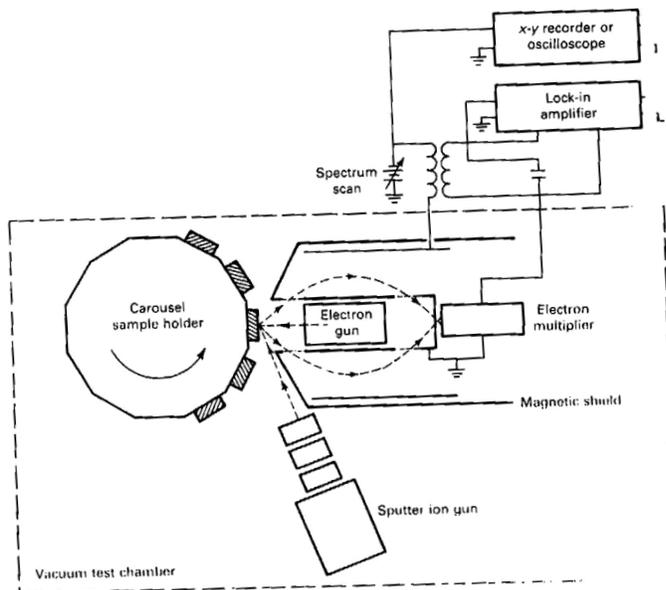
Figure 4. Auger Electron Transition (Lopez, 2011)

Figure 5. General diagram of AES instrumentation (Whan)

3. Auger electron spectroscopy of super-doped Si:Mn thin films   (Abe, 1999)

Example 1:

a. Sample Preparation

Two types of substrates were used, one was made of phosphorus doped n-type Si(001) and the other was oxidized Si(001) (SiO_2). An excimer laser with a KrF source was used for evaporation of Mn in a Knudsen cell at temperatures of 780-850C. The Mn condensed onto the surface of the substrates which were held at 400C. Surface cleaning of the samples was done by Ar-ion sputtering at 3 kV for 1.5 minutes.

b. Experiment Procedure

The thin film samples were analyzed using an Auger Valence Electron Spectroscopy (AVES) AVES ULVAC-PHI model SAM 650. A micron-sized electron beam from a LaB cathode was used at a primary electron energy of 2 keV and the Auger spectra was numerically averaged from 5 to 20 scans. The derivative Auger spectra, dN(E)/dE, was obtained from the derivation of raw N(E) integrated spectra..
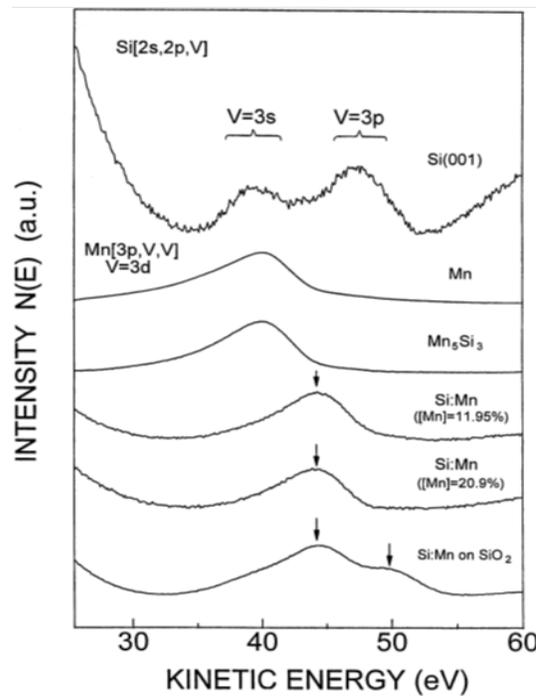
c. Experimental Data

Fig. 6. AVES integrated spectra due to Si[2s,2p,V] (V=3s,3p) and Mn[3p,V,V] (V=3d) transitions for Si(001) simple crystal, pure Mn, Mn_5Si_3 compound, Si:Mn thin films on Si(001) substrate, and Si:Mn thin film on SiO_2/Si(001) substrate. (Abe, 1999)

d. Experimental Analysis

AES spectra for pure Si, Mn, and Mn_5Si_3 were used as refereces for the spectra obtained from Si:Mn thin films. The peaks at 40 and 45 eV in the pure Si spectra are due to the 3s and 3p states respectively; this analysis was verified through comparison with another study done by Nakayama et al. The Auger peaks for Mn and Mn_5Si_3 at 40 eV are due to the Mn [3p,3d,3d] transitions. The main peak for the Si:Mn thin films appeared to shift to a higher kinetic energy with respect to the reference spectra. The Mn:Si thin film grown on the Silica substrate also shifted to a higher energy and a new peak appeared at 50 eV. The peak shifts for the Si:Mn spectra demonstrate the formation of Si-Mn bonds. The presence of the new peak in the spectra for the thin film grown on Silica suggests a hole is formed by the Mn [3p,3d,3d] Auger transition process.

4. Compositional Defect Review on the 300mm Whole Wafer SMART-Tool[TM] and 200 mm Whole SMART-200[TM] Differentiates Particles that look alike but Originate from Different Sources. (EAG – Evans Analytical Group) [2]

Example 2:

In order to effectively increase yields by eliminating defects sources, it is necessary to correctly identify particulate defects that occur during device processing. This is complicated by reduced design rules and new processes which result in smaller critical defects, and in particulate defects which may look alike but often have different composition and originate from very different sources. Thus, imaging alone does not uniquely identify the defect type or the defect source. However, measuring the particle composition provides accurate identification, and leads to more reliable determination of the particle source. The inherent spatial resolution limits of EDS make it a less desirable method for analyzing submicron defects like these. Auger measurements with the **SMART-Tool[TM] and SMART-200[TM]** are ideally suited for composition defect identification of small, thin and complex defects on 200mm and 300mm wafers respectively. This information leads to identification of different sources for defects that may look alike, resulting in faster and more comprehensive yield improvement.
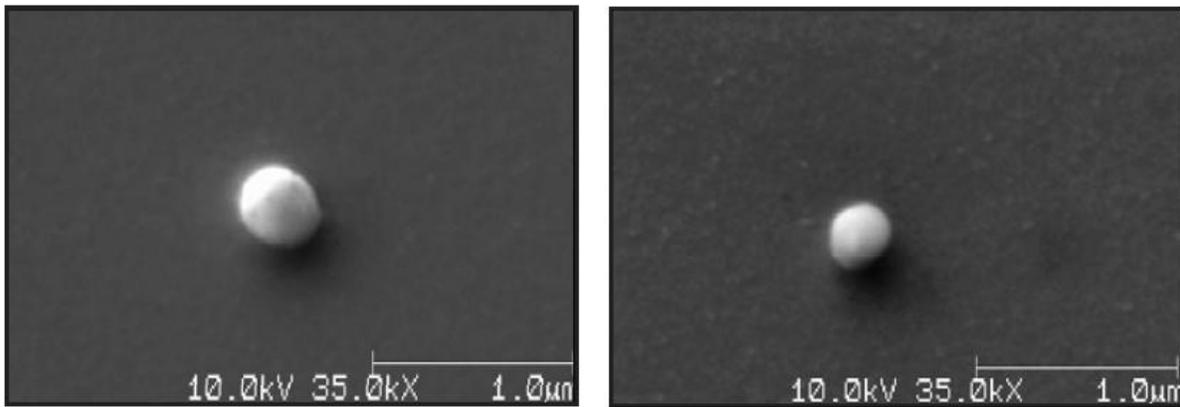
SEM images of two defects do not discriminate between defect types, nor suggest their source.

SEM images: Same size, same shape, same brightness.

a. W Silicide CVD Process

Optical inspection after W silicide CVD (WF_6+$SiCl_2H_2$) revealed multiple defects. SEM images of two particles on this wafer show that they have the same shape. Defect classification by morphology would indicate that these defects are therefore the same type, and have a single defect source. This classification does not indicate a specific, probable defect source that could be eliminated to increase yield. Energy Dispersive X-ray spectroscopy (EDS) was also unable to determine the composition or source of these defects since the particle sizes are much smaller that the excitation and analysis of EDS.

c. Auger composition measurements.

Auger compositional measurements identified one defect as unreacted W, and the other defect as a stainless steel particle with a thin SiO_2 coating. This information identifies two different defect types with two different defect sources. The unreacted W particle originates from a process chemistry problem. The stainless steel particle originates from the process toll hardware. Additionally, the thin Si oxide coating on the steel particle shows that it has also been through an oxide deposition tool, indicating cross-contamination between process tool.

Auger Measurements
> Different Composition
> Different Defect Types
> Different Defect Sources

Auger compositional measurements of two defects reveals two different defect types, and indicate probable defect sources.
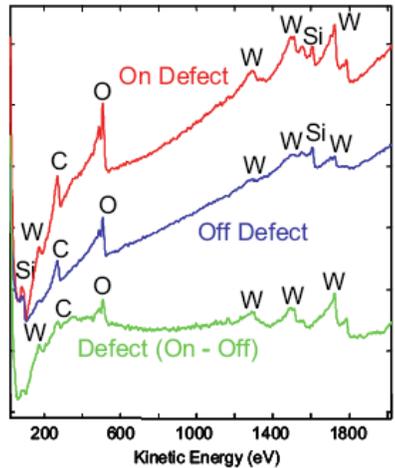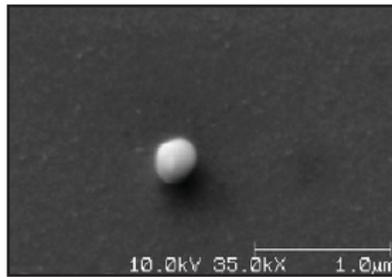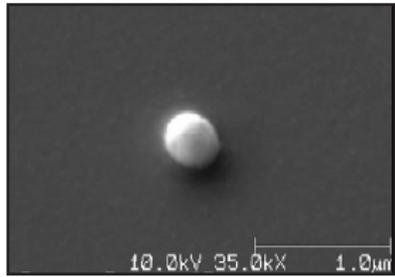
Unreacted Tungsten Particle
Process Chemistry
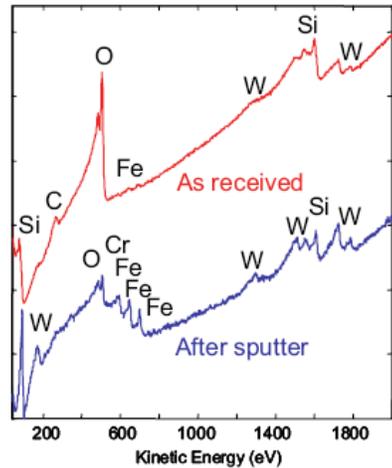
Stainless Steel Particle with SiO₂ Coating
Process Tool Hardware

References

Abe, S. (1999). Auger electron spectroscopy of super-doped Si:Mn thin films. Applied Surface Science(142), 537-532.

Lopez, J. (2011, October). Auger Electron Spectroscopy. The University of Texas at El Paso.

Whan, R. E. (n.d.). ASM Materials Characterization (Vol. 10). Metals Park, Ohio: American Society for Metals.

Grant, John T.; David Briggs (2003). Surface Analysis by Auger and X-ray Photoelectron Spectroscopy. Chichester: IM Publications. ISBN 1-901019-04-7.

http://www.eaglabs.com/files/appnotes/AN429.pdf