SVC Topics

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PVD Processes—Auger Electron Spectroscopy (AES)

It is often desirable to analyze the chemical composition of the surface of a substrate, or of a deposited film. This can be accomplished with surface-sensitive analytical techniques using electrons, ions or photons as the probing species.

One of the most common surface analytical techniques for determining the elemental chemical composition is Auger electron spectroscopy (AES), which uses high-energy (5–30 keV) electrons as the probing species, and low-energy (100–2500 eV) electrons, which have an energy characteristic of the target atoms, as the detected species.

Structure of an Atom

An atom consists of a nucleus containing protons and neutrons in nearly equal numbers, surrounded by electrons in specific energy ranges called *shells* or *orbitals* (Fig. 1). The shells are indicated with letters (K, L, M, N ...) as measured from the nucleus outward. For an uncharged atom of an element, there are as many electrons as there are protons. The number of protons in the nucleus determines the atomic number, or "Z" of the material. The total mass of the atom is the sum of the masses of the protons and neutrons, and is given in atomic mass units* (amu) of the material. Isotopes of the elements have different masses because of the differing number of neutrons in the nucleus.

Excited Energy Levels

All the shells, except for the innermost shell, are subdivided into several energy levels (s, p, d, ...). The inner atomic shells can be filled to the specific number of electrons that they can contain (2, 8, 18 ...). The outermost or valence shell can be "full" or not, depending on the number of electrons available. For some materials, shells below the valence level may not be full.

There are energy levels outside the valence shell to which electrons can be excited. Electrons that are excited to these levels will usually return rapidly to the lower-energy state with the release of energy in the form of a photon of a specific energy. This gives rise to a characteristic emission spectrum, such as the yellow light seen from a sodium vapor lamp.

Electrons can remain in certain excited energy levels, called *metastable states*, until they collide with another atom or a surface. Electrons can be excited to such an extent that they leave the atom, causing the atom to become a positively charged ion. Electrons can be excited thermally by an energetic photon, or by collision with an energetic electron. Atoms can also accept an extra electron and become a negatively charged ion.

Figure 1 shows what happens when an energetic electron collides with an atom. The collision can scatter the impinging electron, excite an atomic shell electron to cause ionization, or excite an electron into an excited energy level. When an electron is excited from its orbital, it leaves behind a vacancy that can be filled by an electron from a more-outer shell. The energy released by this transition can appear as an X-ray having a characteristic energy, or by a radiationless process, called an Auger transition, which gives an electron with a characteristic energy known as an Auger electron. This electron will have energies of a few tens to a few thousand electron volts, depending on the relative position of the energy levels involved.

The ejected Auger electron is identified by the shell that had the vacancy, the energy level that provided the electron to fill the vacancy, and the level from which the Auger electron originated. A KLL Auger electron,

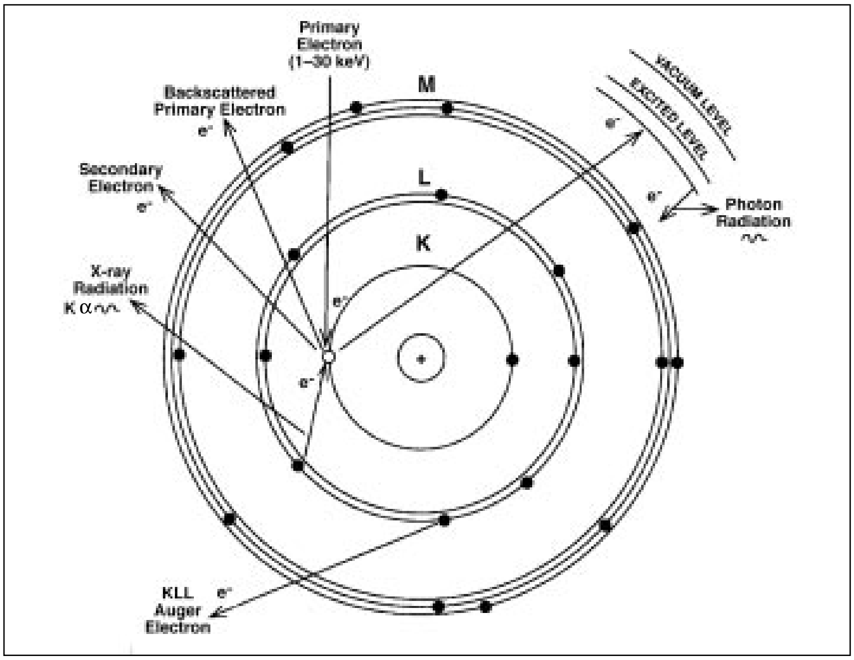

Fig. 1—The interaction of an energetic electron with an atom.

* *Atomic mass unit (amu) is defined as 1/12 of the mass of the C^{12} isotope i.e. = 1.66×10^{-24} g.*

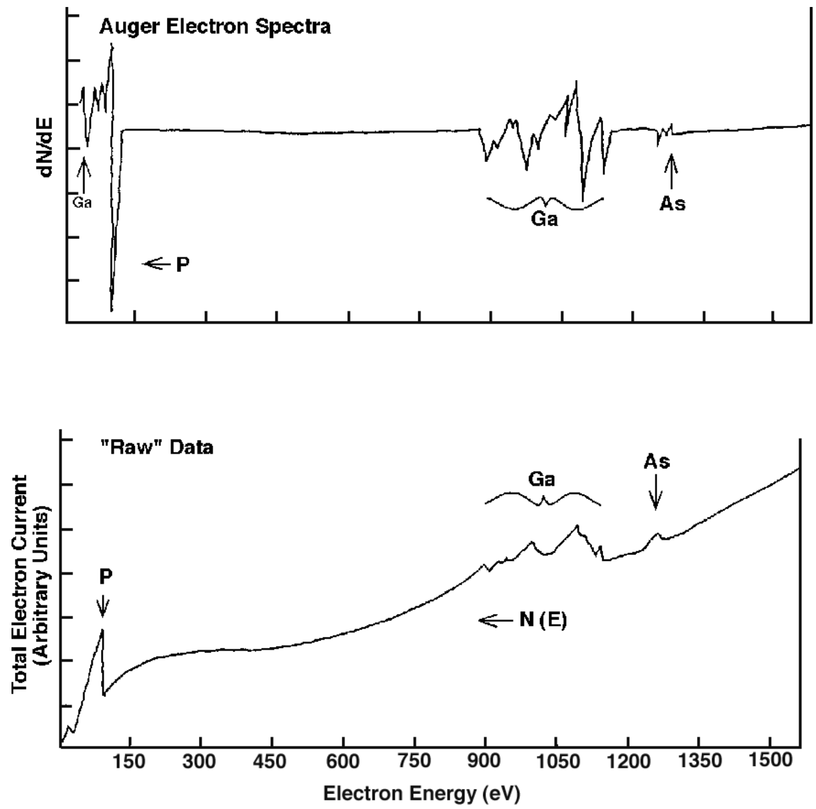

Fig. 2—The electron current collected as a function of the electron energy (raw data) and the Auger electron spectra determined by differentiating the raw data curve.

therefore, originated from the L energy level when another electron from the L level filled a vacancy in the K level. Aluminum, for example, has three principal KLL Auger electrons, with the primary one at about 1400 eV. Solid lithium has one principal KLL Auger electron at about 30 eV; lead has five principal MNN Auger electrons, with the primary one at about 2180 eV.

Auger Electron Spectroscopy

Auger electron spectroscopy uses these characteristic electron energies to identify the atom from which the electron came, as well as the intensity, to give an indication of the number of atoms involved. The Auger electron spectra are generated by striking the atom with an electron beam that has an energy in the range of 1000–30,000 electron volts. The number of the ejected electrons (current) is determined as a function of the energy of the ejected electron using an electrostatic electron energy analyzer, such as a cylindrical mirror analyzer.

The electron energy spectra will show a continuum of backscattered electrons with peaks at specific energies, caused by the Auger electrons. The positions (energies) of these peaks is the Auger spectrum, which is characteristic of the atomic structure. Figure 2 shows the "raw" electron energy spectrum of gallium phosphide, with some arsenic present, and the Auger spectrum, with the background spectrum eliminated by differentiation (dN/dE).

Auger electrons are not emitted by helium and hydrogen, and the sensitivity of the AES technique increases with atomic number. The detection sensitivity ranges from about 10 atomic percent for solid lithium to 0.01 atomic percent for uranium. Energetic electrons rapidly lose energy when moving through a solid, so the characteristic energy of the Auger electrons is only preserved if the electrons escape from the first few monolayers (<10Å) of the surface (escape depth). AES, therefore, is a very surface-sensitive analytical tool. For in-depth analysis, the surface can be sequentially sputter-etched and analyzed by AES to obtain a compositional profile, which is often given as a function of sputtering time.

AES can detect the presence of specific elements; however, to quantify the amount requires calibration standards that are close to the composition of the sample. With calibration, the composition can be established to about ±10 percent. Where there is a mixture of several materials, some of the Auger peaks can overlap, but the whole spectrum can be deconvoluted to give individual Auger spectra. Electron beams can be focused to small diameters to give high lateral resolution, so AES can be used to identify the elemental composition of very small (submicron) particles, as well as extended surfaces.

The secondary electrons emitted by the probing electron bombardment can be used to visualize the surface in the same manner as scanning electron microscopy (SEM). The probing electron beam, therefore, can be scanned over the surface to give an SEM micrograph of the morphology and an Auger analysis map of the surface composition. In some cases, chemical bonding information can be obtained from the Auger spectra, but this type of information is more easily obtained from X-ray photoelectron spectroscopy (XPS) analysis.

Use of AES in PVD Processing

In PVD processing, AES is used to establish the reproducibility of the chemistry of the surface of the as-received substrate material, to determine the effect of surface preparation on the substrate surface chemistry and to identify inorganic particulate contamination on a surface.

AES is also beneficial in analyzing materials used for system fabrication. SEMATECH, for example, has issued standards for the surface chemistry, as determined by AES, for electropolished stainless steel tubing use in gas distribution systems for silicon device processing. *P&SF*

References

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