

Depth Profiling with and without Sputtering

TANAKA, Akihiro

ULVAC-PHI, Incorporated 2500 Hagisono Chigasaki 253 Japan

Introduction: Depth profiling is one of the most frequently and widely used techniques of surface analysis. Sputter depth profiling is used for deep depth profiling and non-sputter depth profiling techniques are used for shallow information range near inelastic mean free path of emitted electrons from atoms. I will summarize present status and problems of depth profiling methods by Auger electron spectroscopy and X-ray photoelectron spectroscopy, here.

Sputter Depth Profiling: Sputter depth profiling is a most widely used technique for depth profiling. It was thought that there were several limitations to make a high resolution depth profile. (1) Sputter ions immerse into sample and make knocking on the atoms. As this phenomenon is caused by ion energy, it is thought that the affected range is constant when sputtering a uniform material. (2) Sputter ions hit areas randomly. It can make Poisson's distribution of sputtered depth if lateral distribution of sputtering possibility is kept constant. This fact would cause a profile broadening proportional to square root of sputtered depth. (3) Inhomogeneity of sputtering in the observed area makes a profile broadening proportional to depth. (4) Sample surface is not uniform if the sample does not have a single crystal nature. This can cause difference of sputtering rate corresponding to areas. Difference of crystallographic direction is an example for polycrystalline samples. (5) Sputtering yields of elements are usually different from others, and it causes concentrating of an element of the system.

For example, electron beam to excite the sample sometimes causes sample reduction and desorption of species, and it can also be a reason of different sputtering yield. These problems are coming from sputtering and measurement natures.

Sample rotation introduced by Zalar[1] reduces sample roughening caused by phenomena (2), (3), and (4). Temperature sometimes reduces or enhances the sample roughening by phenomena like recrystallization. InP system is an example easily roughened by sputtering. Simultaneous use of sample cooling and rotation can be effective for keeping surface flat[2].

Sputtering rate is another item to be considered from practical point of view. We use higher energies when we want to get high sputtering rate, and we use

lower energies when we want to get sharp interface results. When a user wants to optimize sputtering rate and sharp interface resolution, the most important thing to do is alignment of ion gun, centering to the analysis point.

Angle Dependent Analysis: Angle dependent analysis of peak signals is mostly utilized and used for XPS. When we try an angle dependent analysis, we usually assume exponential signal decay of electrons and a flat layer structure. Analyzer acceptance angle is also neglected frequently. The latest analyzers tend to have wide acceptance angles for getting higher sensitivities. As there are many assumptions like them, angle dependent analysis tends to be used only for qualitative explanation like "Top surface signal is relatively high at shallow take off angles and substrate signal is relatively high at high take off angles".

Some instruments have a magnetic lens under the sample. The lens focuses electrons from the sample to an area definition aperture. This mechanism achieves higher sensitivity with higher solid angle, but sacrifices quantitative possibility of angle dependent analysis and gives limitation to samples as the samples should not give big aberration to the lens work.

When the effective thickness of the surface layer is several times larger than the escape depth of substrate electron peaks, background increase following to the peaks is obviously large. This background added to the peak makes big distortion to the total spectral shape. This fact means that quantitative background analysis is important to analyze the peak intensities of variable take off angles.

Depth distribution analysis will be required for precise quantification. Both analyzer acceptance angle and elastic scattering of electrons in the surface region affect to the signal intensities. Substrate signals for very shallow take off angle observation can have a magnitude from a simple exponential decay.

Energy Dependent Analysis: Different Auger transitions give us different peak energies, and different inelastic mean free paths of electrons. If the thickness of the material which we want to analyze is located under some surface layers or existing as a top surface layer, peak intensity ratio is different from the clean bulk material. When the material is a substrate and has some layers on the surface, low energy Auger peaks have smaller peaks as the intensities decayed when passing through the layer. If the material is a layer on the top surface, low energy Auger peaks are relatively larger than those from clean bulk materials.

We can do the analysis using Auger peaks only at the Auger peak energies. On the other hand, we can have continuous analysis for energy using synchrotron radiation photoelectron spectroscopy. Synchrotron radiation is a very powerful tool,

but a very limited facility.

We can use a multi anode system to get variable peak energies for a photoelectron transition. We can get the signals of some elements located at the deeper positions when using anode for higher energy photons.

However, there are 3 problems to use a variable photon energy system. Firstly, excitation cross sections are not widely known for many materials except Mg K α and Al K α X-rays. If we want to use another anode, we need to measure relative sensitivity factors for the elements or uniform compounds for quantitative analysis. Secondly, peak broadening will occur when using high energy anodes without a monochromator. Some examples are reported with broadened photon energy widths about 2 eV[3]. These values are not sufficient for chemical analysis compared with Mg K α (width is 0.7eV) or Al K α (width is 0.85 eV). Thirdly, if we want to use small pass energies to get an energy resolution near photon energy width, it gives us lower sensitivity at high energy region.

Spectral Shape Analysis: Spectral deformation can be clearly observed when the photoelectrons and/or Auger electrons pass through a layer. The peak decays and the background following to the peak increases according to a depth distribution function(ddf) and an energy loss function. As the change of the peak and the background is opposite, the spectral deformation is very obvious.

If the energy loss function is known precisely for the sample, the spectral shapes corresponding to the surface structures can be calculated precisely. Spectral shapes and surface structures can have a one-to-one correspondence. This fact means a possibility to understand the surface structure with spectral shape analysis.

Some Requirements to the Instruments: In order to realize precise spectral analysis with background analysis, the analysis instrument should be characterized precisely. One of the most important character is the transmission function of the analyzer and another is mechanical elimination of trouble electrons which are inelastically-scattered and generated in the analyzer. Sometimes, it looks like eliminated using mathematical calculation. However, it is not easy to prove the instrumental response is same for whole energy region. If we can assume to have a whole transmission characteristics of an analyzer, maintaining the analyzer condition for a long period is not easy either.

There can be several ways for transmission correction. One of the easiest way is a relative way to get the functions of sensitivity ratio corresponding to electron energies with assuming trouble electron-free condition. This method can al-

low to compare the instruments. However, it is not possible to get the 'so-called' true spectrum directly from measurements of the sample using the instrument itself. This correction method is valid after getting 'so-called' true spectra at some self-consistent instruments. Another way is a self-consistent way for the instruments of which the transmission characteristics is defined as only one simple function of retarding ratio. Double Pass Cylindrical Mirror Analyzer is a candidate of standards for this purpose as transmission is known as $1/E$ [4]. E is kinetic energy of electron.

Summary: I summarized depth profiling methods with and without sputtering. Sputter depth profiling is most widely used for many sample systems. However, sputtering is a vigorous treatment for many samples and we should take care before depth profiling. System alignment procedure should be clarified for each system to be used and is necessary for achieving sharp interface resolution and reasonable experiment time.

Inelastic mean free path of electrons is another possibility of depth analysis with electron spectroscopy. SR can be a good source for this purpose, but is not easy to get in a laboratory.

Good instrument characteristics are required for spectral analysis itself and for angle dependent analysis of spectra. One is trouble electron-free and the other is a capability of transmission correction. Depth distribution function is also required for quantification with both spectral analysis and angle dependent analysis. DDF must be defined for each sample system to be analyzed. A big database of ddfs not only for simple systems is required for precise quantification.

Both trouble electron free and known transmission function are two important requirements for standard analyzers.

Reference

- [1] A.Zalar, *Thin Solid Films*, **124**, 223-230 (1985)
- [2] T.Ogiwara and S.Tanuma, in press on *J. Surf. Anal.*, **1** (1995)
- [3] J.C.Riviere, *Instrumentation in Practical Surface Analysis Second Edition* (Eds.D.Briggs and M.P.Seah), John Wiley and Sons, (1990), p. 52
- [4] P.W.Palmberg, *J. Vac. Sci. Technol.*, **12**(1), 379-384 (1975)