APPLICATION OF ELECTRON SPECTROSCOPIES FOR THIN FILM ANALYSIS

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SUMMARY

The appreciation and interpretation of the characteristics of thin solid films and their modification for a particular purpose requires detailed characterization. Thin film analysis can be characterized by their capability for providing information from a millimeter to nanometer scale of the spatial distribution of physical or chemical properties. In the strict sense, the sampled volume is in the cubic micrometer (μm^3) range or less.

Many surface analysis methods have been used for thin film analysis because the film can be "sliced" into numerous "very thin" surfaces by ion sputtering. Analytical features in the last 40 years include (1) the lowering of the detection limit, (2) to improving the capability of elemental imaging on a nm scale with lateral resolution down to single atomic scale, and (3) having sophisticated methods of data acquisition, processing and computer graphics with minicomputer as a powerful tool.

Electron spectroscopies which are using electrons as information carrier in thin film analysis provide some important advantages. Among them two features are of extremely importance: (1) the mean free path of the electrons with a conventionally used primary energy *Ep* is in the nm range, (2) electrons causes no contamination to a vacuum chamber after use for thin-film analysis.

Auger Electron Spectroscopy (AES) and its derivative, the Scanning Auger Microprobe (SAM), are widely used for thin film analysis. They are using Auger electron emission which is accompanied with a radiationless process in bringing an electron-excited atom to a lower energy state with the characteristic energy as a "finger print" of the atom. The electron energy analyzer conventionally used is a cylindrical mirror analyzer (CMA) with an incidence angle of 42.3° which satisfied the second-order focusing condition. But we have found that this parameter is not really optimized. The optimal operation parameters were derived by numerical method for various cases and the corresponding angular abberation is only 25% of that obtained by the "42.3°-method". New AES instruments with these CMA have been manufactured by a factory in Shenyang.

Electron Energy Loss Spectroscopy (EELS) is based on the energy loss in order to produce core level ionization, gives information about empty states. EELS can be acquisited on an AES machine with some slight modifications. Therefore in a single equipment with a combination of AES and EELS can give a complimentary knowledge of DOS. For an illustration, EELS has been used for studying Y-Ba-Cu-O superconductive films and experimental results shown that in such an oxide system the coordination between Cu and O plays an important role in superconductivity.

Total Current Spectroscopy (TCS) differs from AES by scanning the energy Ep of primary electrons and measuring the total secondary electron current which will vary rapidly at some proper *Ep* values. With this method it was found that in a high-Tc oxide the 3d-electrons in Cu play the most important role in superconductive phase.

EELS is not only used for DOS study. Even more important, the spectra recorded above the edge of an element with modulation in the differential inelastic electron scattering cross-section can give reliable surface structural information. This method is an analogy to the principle of Extended X-ray Absorption Fine Structure (EXAFS) and thus is termed as the Surface Extended Energy Loss Fine Structure (SEELFS). SEELFS is the only method which can be used on highly disordered systems such as amorphous film surfaces. Some boron-contained metallic glasses have been tested.

Appearance Potential Spectroscopy (APS) is the only surface spectroscopy to be obtained without an electron energy analyzer, thus dramatically simplified the equipment. Only recently a preliminary experiment has shown that APS can also be used as a scanning probe to obtain an *elemental map* similar to an Auger map but without CMA. The vacuum chamber is much smaller and less expensive as compared to a SAM. It is expected that in the near future a conventional scanning electron microscope (SEM) operated under UHV can be easily improved to give both topographical and elemental micrographs.

Problems raised for thin-film analysis with surface spectroscopy are mainly the reliability of quantitative analysis and the reliability of depth profiling. The former suffers from the matrix effects. For instance, in AES the backscattering of primary electrons is the most important matrix effect because it enhances the Auger yield. A method is described for determining the backscattering factor during AES analysis where the conventionally ignored information provided by the background was used for this purpose. Moreover, since both the intensity of Auger electrons and the energy distribution of backscattered electrons were obtained under the same experimental conditions, the accuracy of quantitative analysis might be further improved. For the depth profiling, the phenomena of ion mixing may cause some significant errors and should be empirically determined if high accuracy is needed for a thin film analysis.

Multi-method synergism seems important especially for the characterization of some important analytical works. For instance, Rutherford Backscattering (RBS) is a good non-destructive technique for the reference of depth profiling. High-energy (1 - 5 MeV), low Z primary ions (mainly He+) are used for RBS since at these energies the nuclear stopping power is negligible compared to the electronic stopping power and sputtering does not occur. Quantitative determination of the concentration is obtained with a high accuracy using databank of RBS cross-section or by empirical calibration and interpolation for different elements. The number of particles backscattered with a certain particle energy is proportional to the number of scattering centers of the corresponding mass number in a depth layer dz at the corresponding depth z. An example is shown for the thin film analysis of a new photochromatic complex for high-density optical and electrical recording media.