Calibration of Electron Spectrometers in a Wide Energy Range

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1. Significance and use of wide energy range electron spectroscopies

Electron spectroscopic methods are very powerful tools for providing information on the analytical composition and on the electronic structure of surface layers as well as of overlayer-substrate interfaces. The information depth of the analysis is strongly dependent on the energy of the escaping electrons and to encompass surface or near surface regions of a larger (several monolayer) thickness, electron lines, excited from the specimen in a wider kinetic energy range, are necessary to be detected with spectrometers having a good resolution and sensitivity and calibrated by methods applicable in this energy range. In this report the current methods and problems of calibration of electron spectrometers in the 1.5-10 keV energy range are reviewed, focusing on high energy XPS and X-ray excited Auger spectroscopy.

The increasing use of these techniques is motivated by many practical applications of great technological importance, e.g. by the nondestructive analysis of polymer/steel interfaces for monitoring changes influencing the strength of the adhesive bonding. This way of obtaining information on chemical states at buried interfaces can be easily combined with other related nondestructive methods like depth profiling by using different kinetic energy peaks of the same elements or by angle-resolved methods.

On the other hand, Auger parameters involving deep core levels and presented in chemical plots are enhancing the possibilities for identification of chemical states and can be used for gaining an insight into initial and final state contributions to chemical shifts in different atomic environment and for determining charge transfer in alloys. High resolution Auger spectroscopy of deep (KLL) core transitions is applicable for controlling the validity of different angular momentum coupling models for elements in the low medium atomic number region and for selecting among (often contradictory) theoretical approaches used for calculation of Auger transition energies and probabilities. Strong chemical and solid state effects on high energy core-valence Auger lineshapes can be identified and the local electronic structure of the atomic environment can be studied bysuch experiments.

2. Some problems of energy calibration of high energy electron spectrometers

Avoiding the need for relativistic corrections high energy electrons are decelerated below 1 keV in front of the analyzer and for ensuring optimum intensity, while keeping the overall energy resolution high, wide spectrometer slits are used. Unlike the case of the energy region below 1.5

keV, for high energy electrons, the width of the spectrometer function is usually comparable to the inherent width of the photo- and Auger lines, even using a very high energy resolution. Therefore for precise calibration a detailed knowledge of the shape of the spectrometer function is needed. The methods for obtaining the spectrometer response function in this energy range include procedures based on photoemission, internal conversion and elastic backscattering experiments, these procedures, their comparison and related problems will be discussed in detail.

A different important field of specific problems is the correction for inelastic background evaluating high energy electron spectra, because of the larger number of scattering events in the sample. Obtaining the energy loss function of the sample from optical data through the dielectric theory or from high resolution measurements of energy loss spectra of high energy reflected electrons, the part of the photoinduced spectra attributable to electrons scattered inelastically can be modeled by Monte Carlo simulations. Various approaches for testing the validity of the assumptions on the scattering process will be discussed, as well as the possibilities for separation of the unresolved satellite part of the spectra from the inelastic background using the results of the simulation.

3. Methods of energy calibration in the high energy region

High energy calibrating lines can be excited most effectively by X-rays. Use of photolines requires characteristic radiation while Auger lines can be induced by continuum photon spectra as well. In many cases high energy Auger lines are produced by the bremsstrahlung part of the X-ray spectrum from a low atomic number anode, so, the only possibility for energy calibration is using known energy Auger transitions from standard specimens. Currently recommended data available for high energy calibration, i.e. energy data of characteristic X-rays in the 1.5-10 keV region as well as the applicable binding energy data will be presented. Possibilities of using radioactive samples (internal conversion lines) for energy calibration will also be discussed. Regarding high energy Auger transitions, the available choice of high precision data for calibration is rather narrow. Discussing the necessary experimental conditions as well, the recent result of our high resolution KLL and KLM Auger measurements for V, Cr, Mn and Fe (4.4-6.4 keV) will be given, in comparison with previous results obtained by radioactive samples and with theoretical predictions. The possible use of these data for energy calibration purposes will be discussed.

4. Efficiency calibration

For quantitative applications the efficiency of the spectrometer should be determined in the energy range of operation. Different methods for determination of the relative and absolute efficiency function of electron spectrometers in a wide energy range, will be discussed, including internal conversion electron spectroscopic measurements on radioactive samples and measurements of yields of electrons scattered from a metallic specimen irradiated by a primary electron beam.