Ma10: Auger- and Electron Energy Loss Spectroscopy

1 Introduction

Electron spectroscopies, namely Auger electron- and electron energy loss spectroscopy are utilized to determine the KLL spectrum and the energies of the surface and bulk plasmons of aluminium, respectively. In addition, the surface sensitivity of the experimental methods are demonstrated *via* adsorption of oxygen on a copper surface.

1.1 Auger Electron Spectroscopy (AES)

Auger electron spectroscopy is a standard analysis technique in surface and interface physics [1, 2]. It is used predominantly to check the cleanliness of a freshly prepared surface under ultrahigh vacuum (UHV) conditions. Other important fields of application includes studies of film growth and surface-chemical composition (elemental analysis) as well as depth profiling of the concentration of particular chemical elements. Beside these applications AES is important for a fundamental understanding of the electronic structure of atoms, the interaction of electrons with solids or interfaces and relaxation processes within the solid.

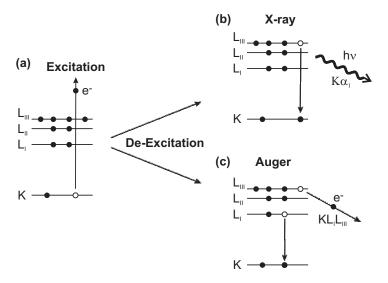


Figure 1: Explanation of the Auger process on the basis of atomic-level schemes. (a) A primary electron produces an initial hole in a core level. Processes for de-excitation of atomic core holes: (b) Emission of X-ray radiation and (c) emission of an Auger electron.

AES is an electron core-level spectroscopy. Thereby a transition can be excited by photons, electrons or even by ion bombardment. Here high energy electrons ($\approx 5~\rm keV$) are used for excitation. The Auger process results in secondary electrons of relatively sharply-defined energy. As with all other electron spectroscopies, AES is surface sensitive due to the limited escape depth of electrons. According to the universal curve of electron mean free path in solid matter, the observation of Auger electrons with a kinetic energy around 1000 eV means an observation depth of about 15 Å. Typical probing depths in AES are in the range of 10–30 Å.

The mechanism of the Auger process is schematically shown in Figure 1. An atom which has been ionized in one of the inner (core) states (Fig. 1 (a)) may return to its electronic ground state *via* one of the following processes:

- (i) An electron of an energetically higher level "jumps" into the core hole, the energy thereby released is emitted as a quantum of characteristic X-radiation (Fig. 1 (b)).
- (ii) The core hole is filled by an outer electron but the available energy is transmitted in an radiationless process to a second electron which may then leave the atom with a characteristic kinetic energy (Fig. 1 (c)). This process is called Auger effect.

1.2 Electron Energy Loss Spectroscopy (EELS)

Electron energy loss spectroscopy (EELS) refers, in a broad sense, to every type of electron spectroscopy in which inelastic electron scattering is used to study excitations of surfaces or adsorbed layers [1, 2]. EELS relies upon the use of a monochromatic, collimated electron beam that is energy analyzed after it has scattered from the surface. The angular distribution of the scattered electrons contains further information, for

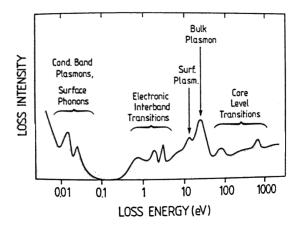


Figure 2: Quantitative overview of the major excitation mechanisms which can contribute to an electron energy loss spectrum over a wide loss energy range. For investigating the different loss energy regimes, in particular below and above 1 eV, different experimental setups are required [2].

instance the orientation of adsorbates with respect to the surface. In principle rotational, vibrational and electronic transitions can be observed in ELLS. Since many different solid excitations can be studied (see Fig. 2) over a wide range from some meV to more than 10^3 eV, different experimental equipment is necessary to provide the required energy resolution at very small and very high excitation energies. The study of vibrations by electron energy loss is often called high-resolution electron energy loss spectroscopy (HREELS) to differentiate it from the study of electronic transitions.

2 Experimental setup

A ultrahigh-vacuum (UHV) chamber with a base pressure of $\approx 10^{-10}$ mbar, equipped with a cylindrical mirror analyzer (CMA) is used for the AES and EELS experiments. Note, that the UHV technique is very sensitive and expensive. Therefore working with this experimental setup requires carefulness and accurateness! The vacuum is generated

Cylindrical Mirror Analyzer (CMA)

Outer cylinder Inner cylinder Sample Electron gun Detector (channeltron) Rear aperture

Figure 3: Schematic cross section of a cylindrical mirror analyzer (CMA).

by a turbo molecular and membrane pump as well as an ion pump, the pressure is measured with a cold cathode ionization gauge. The kinetic energy of the emitted (Auger) electrons are analyzed by means of a CMA (see Figure 3), which consist of two coaxial cylinders. In order to improve the signal-to-noise ratio a lock-in amplifier is used. Accordingly the first derivative of the energy distribution of the electrons (dN(E)/dE) is measured. The CMA exhibits an internal electron gun for the excitation (ionization) of plasmons or core level electrons. The electron gun power supply yields both the current for the thermionic cathode (the electrons are generated via thermionic emission) and the acceleration (ATTENTION: up to 5 kV!), focusing and deflection voltages. The electrons are detected with a channeltron which is a continuous version of a dynode channel type secondary electron multiplier. The channeltron is also operated with high voltage (up to 3 kV). A sudden pressure increase in the chamber during operation may destroy the channeltron. Therefore before doing operations which result into a pressure

rise, e.g., cleaning the sample or gas inlet via a dosing valve, turn off the high voltage for the channeltron. The same should be applied for the emission current and acceleration voltage of the electron gun.

3 Questions and Tasks

3.1 Questions

The following questions are very important in order to understand the experimental methods and results as well as to carry out the experiments. Therefore these questions have to be taken into account for the preparation of the experiment (introductory part) and the report (see references [1, 2, 3, 4, 5]).

(1):

• What is the working principle of a cylindrical mirror analyzer (CMA)?

(2):

• How does a look-in amplifier operate?

(3):

- What is the nomenclature for Auger transitions?
- How does the energy term of the Hamilton operator, which describes the spin-orbit coupling looks like?
- How do the different types of coupling, i.e., jj-coupling and LS- (or Russell-Sounders) coupling come off?
- Which muliplet terms are expected for KLL transitions?
- What are the results for jj- and LS-coupling, respectively?
- What is valid for aluminium?

(4):

• Which Auger transitions are experimentally important? Note, that in standard experimental setups electron energies up to 3 keV can be measured.

(5):

- What is the energy balance in an Auger process?
- How can one calculate the energy of the Auger electrons?

(6):

- How does the so-called universal curve of electron mean free path in metals look?
- What are the consequences for the surface sensitivity of electron spectroscopies in general?

(7):

- What is a plasmon?
- How much energy is required to excite a plasmon?
- What happens at the surface?

(8):

• What is 1 Langmuir?

3.2 Tasks

- (1) Measurement and quantitative analysis of the Auger spectrum of a poly-crystalline aluminium surface. Identification of the characteristic emission bands, which are due to contaminations of the surface (oxygen, carbon etc.) via the comparison with reference spectra (see Ref. [4]). Purification of the surface with the file (mounted on a wobble stick in the UHV chamber) and investigation of the sample cleanliness. Repeating of the cleaning procedure until no contamination are observed.
- (2) Measurement of the aluminium KLL-spectrum and assignment of the observed peaks (multiplet terms). Quantitative comparison with calculated energy positions of the multiplet components (see Ref. [6]).
- (3) Investigation of the plasmon loss structures of aluminum (see Ref. [7, 8]). Use two different primary electron energies $E_p = 1000$ eV and $E_p = 200$ eV for the measurement of the plasmon losses. Thereby the experimental parameter, i.e., position of the analyzer, position of the electron beam, sample position, and modulation amplitude of the look-in amplifier have to be optimized in order to achieve the best possible resolution. For the optimization of the resolution the copper sample should be used.

The measurement with $E_p = 1000 \text{ eV}$ is in comparison to $E_p = 1000 \text{ eV}$ relatively bulk sensitive (why?), therefore $E_p = 1000 \text{ eV}$ should be used to observe bulk plasmons (Note, one should find a series of plasmon losses). What is the influence of surface contaminations on these measurements?

At E_p = 200 eV the measurement is much more surface sensitive, hence it is possible to resolve surface plasmons when the width of the elastic peak is < 2.2 eV. Purification of the surface should lead to an intensity increase of the surface plasmons.

From the loss spectra the plasmon energy of the bulk and surface plasmons, respectively, have to be determined. These values should be compared with the theoretical values calculated by means of the formula for the free electron gas.

(4) Investigation of the oxygen adsorption on the copper surface. Dose 0.1 Langmuir on the clean copper surface and measure the Auger spectrum. What is your conclusion concerning the surface sensitively of Auger electron spectroscopy?

References

- [1] G. Ertl and J. Küppers, Low Energy Electrons And Surface Chemistry, VCH, Weinheim 1985.
- [2] H. Lüth, Solid surfaces, interfaces and thin films, Springer, Berlin, 2001.

- [3] D. Briggs and M.P. Seah, *Pratical Surface Analysis*, Salle and SauerInder 1990, Volume 1 (Auger and X-ray Photoelectron Spectroscopy).
- [4] P.W. Palmberg, G.E. Riach, R.E. Weber, and N.C. Macdonald, *Handbook of Auger Electron Spectroscopy*, published by Physical Electronics Industries Inc.
- [5] A. Zangwill, *Physics at Surfaces*, Cambridge University Press, Cambridge 1988.
- [6] G. Dufour, J.-M. Mariot, P.-E. Nilsson-Jatko, and R. Karnatak, *Physica Scripta* 13, 370 (1976).
- [7] C. Kittel, Einführung in die Festkörperphysik, Oldenbourg Verlag, München, Wien 2006.
- [8] D. Massignon, F. Pellerin, J.M. Fontaine, C. LeGressus, and T. Ichinokawa, *Journal of Applied Physics*, **51**(1), 808 (1980).