

ELECTRON SPECTROSCOPY OF SOLID SURFACES*

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Electron spectroscopic techniques such as Auger electron spectroscopy (AES), X-ray induced photoelectron spectroscopy (XPS) and Elastic peak electron spectroscopy (EPES) are powerful methods for the measurements of chemical composition and bonding among atoms found in the analyzed near surface region of a few nanometer range (AES, XPS) and for determination of a basic electron transport parameter the inelastic mean free path of signal electrons (EPES). Analytical applications of AES and XPS require knowledge of the reliable theoretical model relating the measured signal intensity to the number of atoms emitting the monitored Auger electrons and photoelectrons. The present review will address briefly (i) typical procedures of quantitative analysis commonly used in AES and XPS, (ii) electron elastic scattering effects, (iii) surface sensitivity of the methods used and (iv) applications of the EPES for determination of the inelastic mean free path values and their energy dependencies.

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1 Introduction

At present, the Auger and photoelectron spectroscopic techniques are used as almost universal methods for qualitative and quantitative analysis of near surface regions of solids and to obtain important information about chemical bonding among atoms found in the analyzed volume of samples.

As shown in Fig. 1, experiments consist in irradiating the studied solid surface with the primary beam of electrons (AES, EPES) or photons (XPS) and recording the energy distribution of electrons emitted from the surface. In the case of the EPES method, the studied surface is bombarded by a primary electron beam at a given kinetic energy as for the AES and a peak of elastically reflected electrons at approximately the same kinetic energy is recorded. The kinetic energy of emitted electrons is obviously smaller than or equals to the exciting energy. Although photoelectrons excited by commonly used laboratory X-rays (Mg K α -1254 eV or Al K α -1487 eV) are created in the sample within the layer of the thickness of several thousand nm, they can reach the surface without energy loss only when emitted within first few monolayers due to

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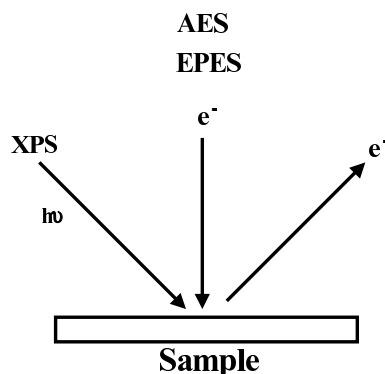


Fig. 1. Schematic picture illustrating AES, XPS and EPES techniques: for AES and EPES methods the primary electron beam at a given kinetic energy is used while for XPS it is the beam of monochromatic photons. Electrons emitted from a sample surface enter to the electron energy analyzer.

strong inelastic scattering of electrons at these kinetic energies. From this reason, XPS (AES or EPES as well) becomes a surface sensitive method providing the qualitative and quantitative information on first few atomic layers.

Line intensities or peaks areas of the photoelectron or Auger electron peaks are of interest in quantitative analysis. The intensity of a given line depends on a number of factors including properties of atoms (for example, cross-sections, Auger electron yield, initial anisotropy of electron emission), properties of a sample (atomic density, backscattering factor, electron escape depth) and properties of a spectrometer (spectrometer transmission, intensity of exciting radiation). The basic equations for quantitative analysis were derived within the so-called Straight Line Approximation (SLA) model of electron transport in a near surface region of a sample [1]. The SLA model, described in the Section 2, neglects electron elastic scattering in the analyzing volume. Several experiments reveal, however, the importance of electron elastic scattering for obtaining realistic quantitative information [2, 3]. Due to a complexity of analytical description of the electron scattering problem, first attempts were made applying Monte Carlo simulations of electron motion in a solid [2, 3].

A depth of analysis or a surface sensitivity of the above mentioned electron spectroscopic methods is described by the mean escape depth (MED) which is related to the key parameter of electron transport in solids called the inelastic mean free path (IMFP) of signal electrons. According to ASTM definitions, the IMFP is defined as an average distance that an electron travels between successive inelastic collisions [4]. The calculated IMFPs for selected elemental solids [5], inorganic compounds [6] and organic compounds [7] have been published by Tanuma *et al.* in the electron energy range 50-2000 eV. An expression for energy dependence of the IMFPs called the TPP-2M predictive formula can be applied to any multicomponent specimen if the parameters characterising the specimen are known: the density, the band gap energy, and the number of valence electrons [7]. The so-called G1 predictive formula of Gries [8] valid for any category of materials in the range 200-2000 eV requires less complex set of input parameters. The IMFP values for selected elements and inorganic compounds in the energy range 200-10000

eV have been also calculated by Ashley and Tung [9] and Kwei and Chen [10].

2 Quantitative information in AES/XPS

Although Auger transitions are in principle more complex processes involving three electrons, formalism describing (electron-induced) AES and XPS quantitatively is surprisingly similar. The main difference between the two techniques is in the excitation beam: electrons for AES and photons for XPS. As a consequence, it is easy simply to replace the ionization cross section, the Auger electron yield and the backscattering factor in an equation describing Auger line intensity by the photoelectric cross section and one can describe the photoelectron current. For the sake of simplicity, let us show below basic formulas describing photoelectron current.

In 1974, Fadley *et al.* [1] published an extensive theoretical background for quantitative analysis in XPS. With minor modifications, the formalism proposed by these authors is commonly used until present time. The formalism is based on the SLA model which neglects elastic scattering of photoelectrons. Other assumptions are following: (i) the solid surface should be ideally flat, (ii) the analyzed sample should be amorphous or polycrystalline, (iii) in-depth composition within an analyzed volume should be uniform, (iv) the X-ray refraction and reflection are neglected and (v) the X-ray attenuation within the analyzed volume is negligible. Under the above assumptions, the photoelectron current dI , emitted from the layer with a thickness dz at a depth z , is expressed by the formula

$$dI = CI_0AMx \frac{d\sigma}{d\Omega} \exp \left[-\frac{z}{\lambda_i \cos \alpha} \right] dz \quad (1)$$

where the constant C comprises the instrumental factors, I_0 is the flux of X-rays, A is the analysed area, M is the total atomic density, x is the atom fraction of a given element, λ_i is the IMFP of photoelectrons, α is the angle between the surface normal and the direction of analysis (emission angle) and $d\sigma/d\Omega$ is the differential photoelectric cross-section [11]

$$\frac{d\sigma}{d\Omega} = \frac{\sigma_t}{4\pi} \left\{ 1 - \frac{\beta}{4}(3 \cos^2 \Theta - 1) \right\} \quad (2)$$

where σ_t is the total photoelectric cross-section, β is the asymmetry parameter and Θ is the angle between X-ray propagation direction and direction of outgoing photoelectron.

In the typical experimental configuration of XPS, a large area specimen is exposed to a broad beam of X-rays irradiating a much larger area than the area being analysed. Hence, we may assume that the analysed area depends on the detection angle according to

$$A = A_0 / \cos \alpha \quad (3)$$

where A_0 is the area seen by the analyser at the normal direction of analysis. Integrating Eq. (1) over all depths with account of Eqs. (2) and (3), we obtain

$$I = CI_0A_0Mx \frac{d\sigma}{d\Omega} \lambda_i. \quad (4)$$

Eq. (4), valid for homogeneous semiinfinite solids, forms a base for calculations of the surface composition. The formalism can be easily extended for a model of a thin layer on a flat substrate [1].

There are two different experimental procedures used for the quantitative analysis: analysis using standard materials and the so-called relative sensitivity factor approach. Let us briefly discussed both approaches.

2.1 Standard materials approach

This approach is now the most reliable in quantitative applications. The standards and samples are measured using the same experimental geometry, under the same parameters used for recorded spectra. An obvious selection for a standard is the clean surface of the element present in the studied sample. The simplest experimental procedure of quantitative analysis consists in measurements of a given peak intensity for a sample and for the standard. Then, Eq. (4) can be modified as follows

$$\frac{I}{I^s} = \frac{M\lambda_i}{M^s\lambda_i^s} x. \quad (5)$$

The described simple procedure of quantitative analysis provides the surface concentration of one element. To obtain the total composition, we should use several standards corresponding to all elements present in the analysing volume of the sample. Eq. (5) for the j -th element can be written

$$x_j = \frac{I_j M_j^s \lambda_j^s}{I_j^s M \lambda_j} \quad (6)$$

and the sum of x_j should be normalized to 1. For simplicity, let us limit the formalism to the two component solids, ab . Then, we derive the following couple of equations

$$\frac{x_a}{x_b} = \frac{x_a^s I_a I_b^s \lambda_b \lambda_a^s}{x_b^s I_b I_a^s \lambda_a \lambda_b^s}, \quad x_b + x_a = 1. \quad (7)$$

Resulting experimental uncertainty in determination of concentrations of atoms in the analysing volume by the standard method can be as low as 5%.

2.2 Relative sensitivity factor approach

Current industrial samples are frequently far from the ideal ones. They have usually rough and contaminated surfaces or can be also in a form of a powder. In such a case it is difficult to find proper standards. The only method of quantitative analysis which can be used in such cases is the relative sensitivity factor approach. The corresponding formalism is very simple. The signal intensity, I_i , due to any elemental constituent of the sample is proportional to concentration

$$I_i = S_i x_i \quad (8)$$

where S_i is the sensitivity factor. The concentration of i -th element is calculated from

$$x_i = \frac{I_i/S_i}{\sum_k I_k/S_k}. \quad (9)$$

There are tabulations of the sensitivity factors available in the literature [12] and also the manufacturers of the XPS spectrometers usually recommended a set of sensitivity factors for a given

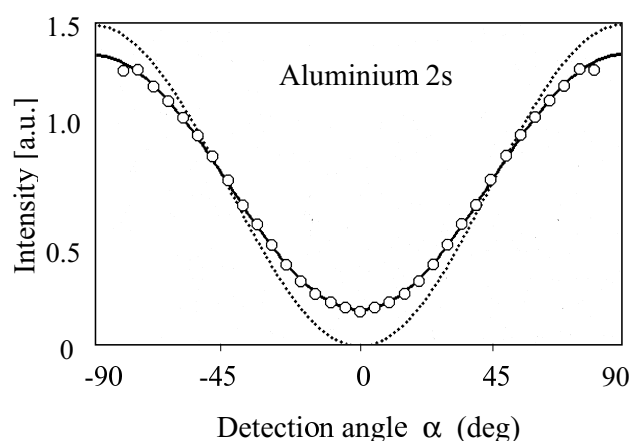


Fig. 2. The Al 2s photoelectron intensity dependence on detection angle α measured with respect to the surface normal. Photon beam excitation direction is parallel to the surface normal. The geometry is the same as shown in Fig. 3. Dots represent the SLA model calculation, Circles and a solid line is the result of Monte Carlo calculations properly describing electron elastic and inelastic scattering processes near the solid surface.

type of the spectrometer. However, the relative sensitivity factor approach is generally less precise than the above described method applying the standards. Although the Round Robin analysis of the same set of samples in several laboratories organized in 1991 [13] resulted in reasonable accuracy of the analysis, it is recommended to use the sensitivity factors measured at the same apparatus. In such a case all instrumental effects including the particular experimental geometry are properly accounted.

An alternative way may be a modification of the approach to perform the analysis of complex materials with a good accuracy. These modifications consist in different methods of introducing corrections accounting for the characteristics of the instrument [14], properties of the sample and the experimental geometry used [15, 16] and elastic scattering effects [17].

3 Electron elastic scattering effects

Applying the SLA model where elastic scattering is neglected, the angular distribution of photoelectrons leaving a sample is proportional to the respective differential photoelectric cross section, i.e. the initial angular distribution (see Eqs. (1) and (2)). As a result, no photoelectron current is expected in emission directions corresponding to zero values of the differential photoelectric cross section. This is, for instance, the case of s-photoelectrons of relatively light elements, whose asymmetry parameter is equal to 2, and the emission directions parallel or antiparallel to that of X-ray propagation. This SLA model prediction is, however, in contradiction with experimental data [2, 3]. As shown in Fig. 2, there is a noticeable photoelectron current in the emission directions forbidden by the dipole transition rules [3]. The geometry applied here

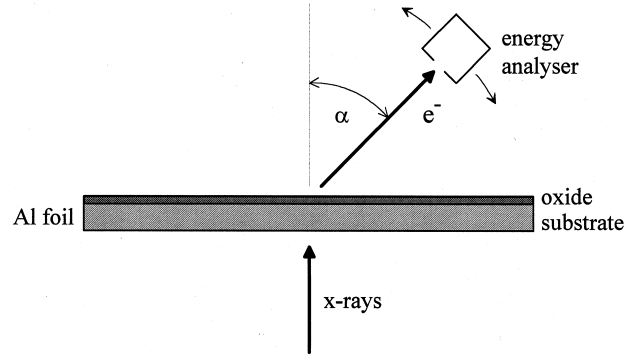


Fig. 3. Schematic representation of the experimental configuration. X-rays are incident on the bottom side of the film/substrate target along the surface normal. The foil should be thin enough to provide good X-ray transmission and should be sufficiently thick with respect to the mean escape depth of photoelectrons leaving the upper side of the sample in emission directions specified by the polar angle α .

is shown in Fig. 3.

The appearance of the current in those directions is explained by multiple elastic scattering of photoelectrons on their way out of the target. Indeed, due to elastic collisions, some of the electrons can change their directions of motion and modify considerably the initial angular distribution. Effects of electron elastic scattering on the measured photoelectron peak intensity can be studied also by the so-called emission depth distribution function (DDF). This function [4] describes contributions dI to the total photoelectron current I arising from different depths z in a solid

$$\Phi(z) = \frac{dI/dz}{I}. \quad (10)$$

From Eqs. (1), (2) and (10) we obtain the following exponential dependence valid within the SLA model

$$\Phi(z) = (\lambda_i \cos \alpha)^{-1} \exp \left(-\frac{z}{\lambda_i \cos \alpha} \right). \quad (11)$$

Due to elastic scattering of photoelectrons the DDF can deviate substantially from Eq. (11), as has been shown by Monte Carlo simulations. Particularly, for photoelectrons with the asymmetry parameters equal 2 leaving a solid surface at emission angles parallel or antiparallel to the X-ray propagation direction, the DDF can reach a maximum at the depth less than the corresponding IMFP value [3]. The same behavior has been found very recently also by an analytical approach based on solving the Boltzmann kinetic equation in the so-called transport approximation [18].

Due to difficulties, experimental determination of the escape probability of photoelectrons leaving a solid surface as a function of depth of origin was not available until recently. The DDF can be measured for a sample containing a thin layer of certain material (marker) buried inside the sample. Due to strong inelastic scattering of electrons and, hence, their small sampling depth

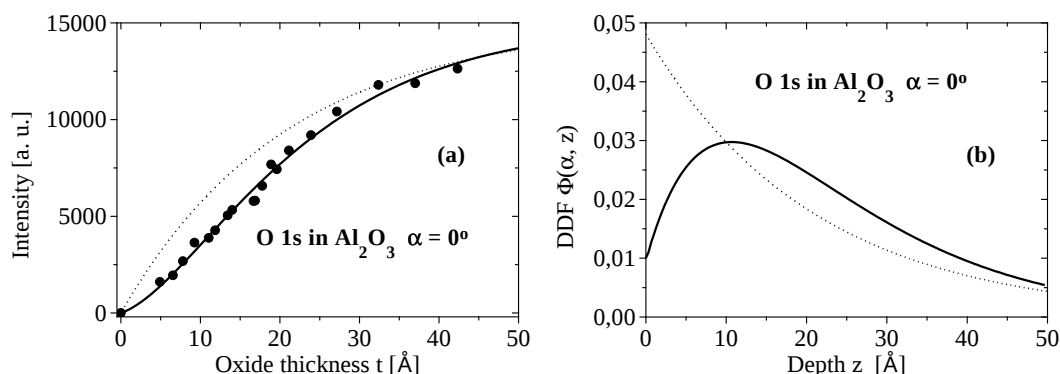


Fig. 4. (a) Oxide thickness dependence of the O 1s photoelectron line intensity emitted from aluminum oxide film grown on aluminum foil. Photoelectrons are collected at the polar emission angle $\alpha = 0^\circ$. The filled circles are the experimental data, the solid curve is the result of curve fitting of the experimental data. For comparison, the same dependence is calculated within the SLA model. (b) Emission depth distribution function of O 1s photoelectrons in aluminum oxide film for the emission angle $\alpha = 0^\circ$. Solid line - experimental data, dotted line - results of the SLA model. The data are normalized so that the integral of the DDFs provide unity.

(in nanometer scale) there are considerable difficulties in growing a well defined and uniform marker layer buried beneath the sample surface at different depths despite expected weak signal from the marker.

Recently, we have succeeded in determining the DDF experimentally. A novel method for the DDF determination based on a surface oxidation has been developed. Experimental assessment of the DDF relies on well defined growth of overlayer homogeneous in thickness on a flat substrate. First results for photoelectrons leaving Al and Cu oxide surfaces have been reported [18–22]. More recently, The DDFs have been determined for silver sulphide grown on silver foil [23]. Similar procedure has been used by other group for experimental determination of the DDF on iron oxide/iron system [24].

The procedure for determining the escape probability of photoelectrons consists of

- step by step film growth (by surface oxidation of Al and Cu substrates or sulphidization of silver foil),
- recording selected photoelectron lines from a film and a substrate,
- in-situ film thickness estimation,
- plotting intensity vs. thickness dependencies, and finally
- evaluating the DDF by differentiation of the intensity vs. thickness plots.

Applications of the above method for the DDF determination are illustrated in Figs. 4 - 7 for 3 different materials: aluminium oxide, copper oxide and silver sulphide. The experimental

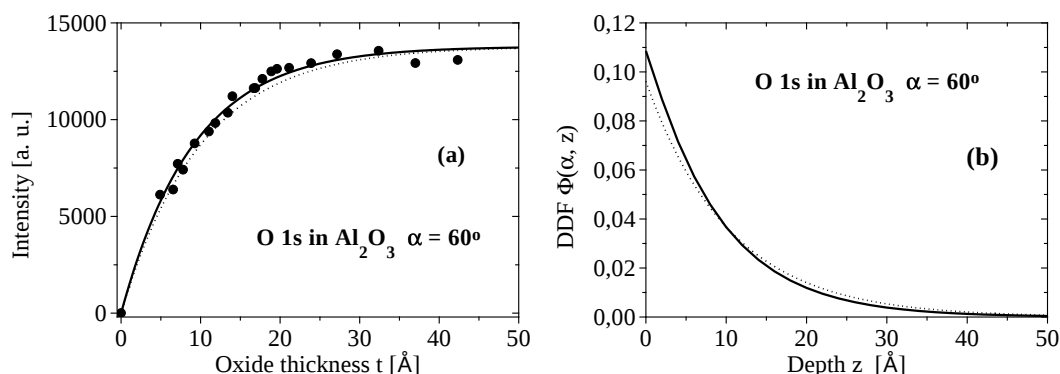


Fig. 5. (a) Oxide thickness dependence of the O 1s photoelectron line intensity emitted from aluminum oxide film grown on aluminum foil. Photoelectrons are collected at the polar emission angle $\alpha = 60^\circ$. The filled circles are the experimental data, the solid curve is the result of curve fitting of the experimental data. For comparison, the same dependence is calculated within the SLA model. (b) Emission depth distribution function of O 1s photoelectrons in aluminum oxide film for the emission angle $\alpha = 60^\circ$. Solid line - experimental data, dotted line - results of the SLA model. The data are normalized so that the integral of the DDFs provide unity.

geometry is documented in Fig. 3. In Figs. 4a and 5a, the intensity plots for O 1s photoelectrons leaving the aluminium oxide at emission angles 0° and 60° are presented and compared to the corresponding SLA results. In Figs. 4b and 5b there are shown the DDFs measured at emission angles 0° and 60° for O 1s photoelectrons and compared to those calculated by the SLA model [21]. In Fig. 6 there is an example of the measured DDF for Al 2s photoelectrons escaping the aluminium oxide surface at emission angle 0° [18]. The experimental data are compared to the results of Monte Carlo calculations, analytical theory (both properly describing the electron scattering problem) and to the SLA model calculation. Fig. 7 summarises measured DDFs for S 2s and S 2p photoelectrons leaving the silver sulphide surface at 0° emission angle [23]. From the above measured and calculated DDFs the following conclusions can be drawn:

- (i) Measured escape probabilities of photoelectrons as a function of depth of origin differ noticeably from the SLA model predictions. Particularly, this is valid for photoelectrons with $\beta = 2$ leaving a surface at electron emission direction parallel or antiparallel to the photon propagation direction.
- (ii) There is a good agreement between measured DDFs and those calculated by using Monte Carlo and analytical theory properly describing electron scattering processes.
- (iii) Large differences observed in the measured DDF shapes from a single exponential function are caused by electron elastic scattering processes.

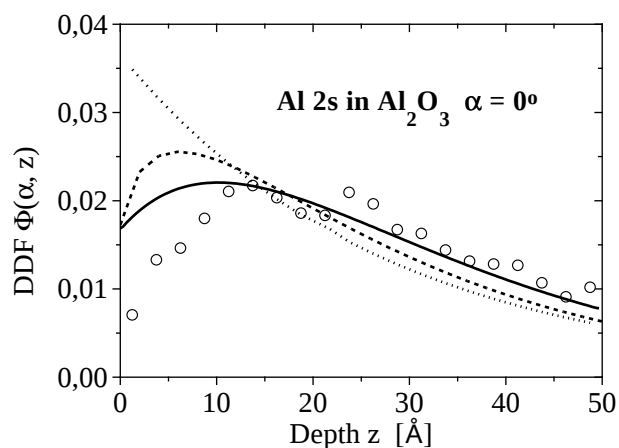


Fig. 6. The emission depth distribution function for Al 2s photoelectrons leaving aluminum oxide surface at emission angle $\alpha = 0^\circ$. Solid line - experimental results, circles - Monte Carlo calculations, dashed line - analytical solution of the Boltzmann kinetic equation in a transport approximation, dotted line - the SLA results.

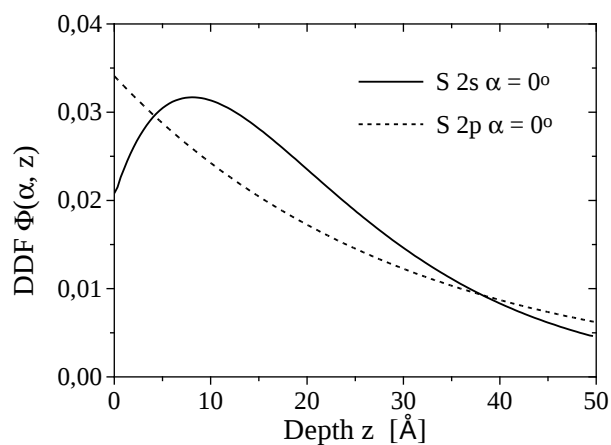


Fig. 7. The measured DDFs for S 2s and S 2p photoelectrons escaping the silver sulphide surface at the emission angle $\alpha = 0^\circ$.

4 Surface sensitivity

The surface sensitivity or the depth resolution of surface sensitive electron spectroscopic methods is determined by the thickness of the material that an emitted electron can transverse without undergoing an inelastic event. This quantity is, however, influenced to some extent also by

electron elastic scattering events. As a measure of the surface sensitivity can be considered the mean escape depth (*MED*) of electrons. The *MED* has been defined by American Society for Testing and Materials Committee [4] as the average depth normal to the surface from which the specified particle or radiation escape. This average mean escape depth, *MED*, is given by

$$MED = \frac{\int_0^{\infty} z \Phi(z, \alpha) dz}{\int_0^{\infty} \Phi(z, \alpha) dz}. \quad (12)$$

Generally, the measured *MED* values are expected to be different in comparison with those calculated within the SLA approach. Disregarding for elastic scattering effects may lead to over- or underestimating the sampled volume in the near surface region [25]. Analytically, an expression for the *MED* has been derived by solving the Boltzmann kinetic equation in a transport approximation [25]

$$MED = \lambda_i \lambda_{tr} (\lambda_i + \lambda_{tr})^{-1} (\cos \alpha + S) \quad (13)$$

where λ_{tr} is the transport mean free path available in the literature [26] and the quantity S is determined by the ratio

$$\begin{aligned} S &= \frac{S_1}{S_2}, \\ S_1 &= \frac{\omega}{2(1-\omega)} \int_0^1 \mu H(\mu, \omega) d\mu, \\ S_2 &= (1-\omega)^{-1/2} - \frac{\beta}{4} \left[\frac{3 \cos^2 \Theta - 1}{H(\cos \alpha, \omega)} \right]. \end{aligned} \quad (14)$$

Here, $\omega = \lambda_i (\lambda_i + \lambda_{tr})^{-1}$ is the single scattering albedo and $H(\mu, \omega)$ is the H - function of Chandrasekhar [27]. It should be noted that when we consider the electron inelastic scattering only, Eq. (13) yields

$$MED = \lambda_i \cos \alpha. \quad (15)$$

The same result is received within the SLA model. In any case, the knowledge of the reliable IMFP values is of key importance in surface sensitive electron spectroscopy.

5 Determination of the IMFPs using the EPES method

The experimental method for the IMFP values determination by using the EPES method has been already applied for selected metals [28–32], inorganic [30, 33–35] and organic compounds [15, 36, 37]. Compilation of methods for the IMFPs determination, including the calculated and experimental values for selected elemental solids, inorganic and organic compounds has been recently published by Powell and Jablonski [38]. It has been shown that there is a considerable scatter of published experimental IMFP values. Furthermore, deviations are observed between the experimental and calculated IMFPs. The authors discuss the sources of possible statistical

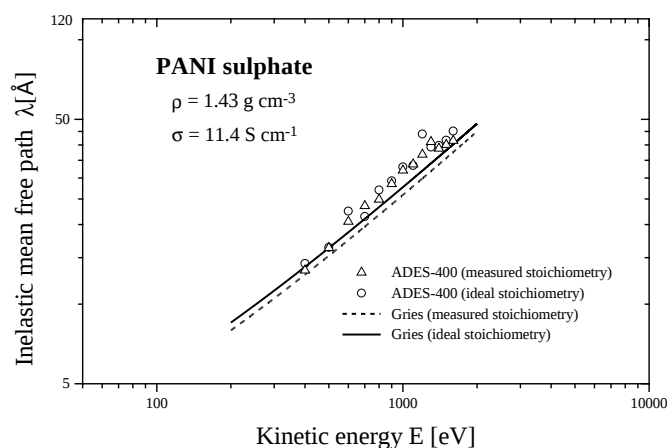


Fig. 8. Energy dependence on the IMFP for the polyaniline (emeraldine) sulfate sample. Triangulars and circles - experimental IMFPs obtain using EPES. Dashed line - IMFPs resulting from the G1 formula of Gries [8] calculated for the polyaniline sample characterized by the measured stoichiometry and the measured density. Dotted-dashed line: IMFPs resulting from the G1 predictive formula of Gries calculated for the polyaniline sample characterized by the ideal stoichiometry and the measured density.

and systematical errors in the measured and calculated values. From the all compiled averaged measured and calculated IMFPs Powell and Jablonski suggested to determine the so-called recommended IMFP values [38].

The EPES method requires the measurements of the elastic backscattering probability from an investigated material and from a standard material. Then, the IMFP value is calculated using a Monte Carlo algorithm simulating the electron transport in a solid [28]. The procedure of IMFPs determination requires calculations of the calibration curve, i.e. the dependence of the backscattering probability (or their ratio) on the assumed IMFP values. The Monte Carlo algorithm applied in the present work has been already described in details elsewhere [28].

The electron trajectories exhibiting multiple elastic scattering processes are simulated assuming homogeneous distribution of atoms in a solid characterised by ideally flat surface. To simulate the elastic scattering processes of electrons, the differential elastic cross-sections were taken from the NIST database [39]. The following set of input parameters is needed for calculating:

- (i) the stoichiometry and density of the studied material,
- (ii) the IMFP value for the studied material,
- (iii) the geometry of analysis (the angle of primary electron beam, the emission angle of backscattered electrons and the solid angle of the analyser).

The calculated ratio of electron elastic backscattering probabilities (I/I_s) from the investigated sample (I) and the standard (I_s), for varying range of input IMFPs creates the so-called

calibration curve. A set of calculated ratios, I/I_s , versus assumed set of IMFP values for the sample, λ_i , is approximated by the following equation

$$\frac{l}{l_s} = \sum_{i=1}^4 a_i (\lambda_i)^{1/2} \quad (16)$$

where a_i are the fitting coefficients. From comparison of the above Eq. (16) and the measured values, the IMFP value for the investigated material can be found.

One important application of the EPES method for obtaining the IMFP values in wide kinetic energy range is illustrated in Fig. 8. As a measured sample the polyaniline (emeraldine) sulphate was used [15]. Experimental IMFPs are compared with those calculated on the base of G1 predictive formula of Gries [8]. There is a good agreement between the measured and calculated data.

6 Conclusions

In recent years much progress has been made in understanding of electron transport near solid surfaces at kinetic energy important for electron spectroscopy. It is now clear that some of the assumptions made in the common formalism may not be valid in certain experimental configurations or in a certain range of energies. Particularly, neglecting the electron elastic collisions may sometimes dramatically affect the calculated signal intensity. Moreover, there are experimental and theoretical proofs that the attenuation of the electrons in solids may be nonexponential. This may influence the depth of analysis and, hence, the reliability of the quantitative information itself.

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