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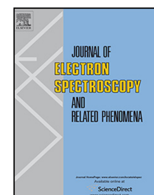

Effective attenuation length of keV photoelectrons in silicon measured by transmission through thin membranes

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ABSTRACT

We use 9 nm and 15 nm thin membranes for determining the effective attenuation length of photoelectrons in silicon. One side of silicon membranes was covered with a thin film of aluminium and exposed to X-rays with energies from 3 to 8 keV. We recorded Al 1*s* and 2*s* photoelectrons that were (a) emitted from the Al film directly and (b) transmitted through the membranes. With the help of the ratio of both yields, we obtained values for the effective attenuation length (EAL) of electrons with kinetic energies up to 7.9 keV in silicon. The experimentally determined EAL values are smaller than obtained from different predictive equations. Using a power law fit $EAL\,(k,p) = kE_{kin}^{p}$ to the experimental and predicted EAL values we find that mainly different is the pre-factor of the power law, *k*, while the exponent, *p*, i.e. the dependence on kinetic energy E_{kin} is represented well. Our study underlines the feasibility of using membranes for investigating surfaces under (near) ambient pressure conditions by photoelectron spectroscopy and points out the advantages of employing hard X-rays.

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1. Introduction

Photoelectron spectroscopy (PES) is a well-established powerful technique providing valuable information about the electronic properties, chemistry and structure of materials. While every photo-excited electron emerging from the sample carries useful information, the so called elastic electrons, which did not lose energy in collisions, are of particular interest. The flux of elastic electrons observed from a specific photo-excited element of a buried object depends on the probability of the photoexcited electrons to escape the material without energy loss. In practical applications (e.g. film thickness determination) a parameter used to describe the probability of escape is the effective attenuation length (EAL) of the electrons. The EAL depends on the electron kinetic energy, the material traversed by the electrons and the used geometry. It is an important parameter for the exact quantitative characterization of the structure of materials by photoelectron spectroscopy (PES). For the exact quantitative structural and chemical characterization of micro- and nano-structures as well as thin films and interfaces, which are presently in the focus of interest, accurate values for EAL are needed as a function of electron kinetic energy.

Of course, the flux I_{pe} of emitted photo electrons (PEs) depends on the photoelectric cross section σ_{PE}, which strongly depends on the specific element and electronic orbital. Values for the cross sections have been calculated comprehensively, e.g., by Scofield [1,2]. Since cross sections decrease dramatically with excitation energy E_γ (roughly, $\sigma_{PE} \sim E_\gamma{}^{-3}$), PES studies were for a long time carried out mostly using moderate photon energies, i.e., within the ultraviolet and up to the soft/tender X-ray range (E_γ <(<) 2 keV). Hence, over the years, most of the EAL data has been collected for photoelectrons with kinetic energies in that range. Comprehensive data are available, e.g. from the National Institute of Standard and Technology (NIST) [3] regarding electron elastic scattering cross sections, the inelastic mean free path (IMFP), the effective attenuation length and other useful parameters.

The EAL of electrons is determined by the inelastic scattering and elastic scattering of the electrons. The inelastic mean-free-path of a photoelectron is the mean distance that an electron travels in a specific material before it undergoes an inelastic collision and is thus lost from the elastic channel. Electrons with kinetic energy E_{kin} suffer energy loss (ΔE), in the order of probability, by plasmon excitation ($\Delta E \approx 10$ eV), phonon excitation ($\Delta E < 1$ eV), excitations of core electrons with binding energy $E_B < E_{kin}$ ($\Delta E = E_B$) and Bremsstrahlung ($\Delta E \leq E_{kin}$). The IMFP depends on the atomic

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numbers Z of the elements in a material but much less than e.g. X-ray attenuation lengths. For electron kinetic energies above about 100 eV and all elements from lithium (Z = 3) to bismuth (Z = 83) the difference in IMFP is less than an order of magnitude [4].

The probability that an electron is able to travel within a material from a point A to B without energy loss is further reduced by elastic scattering. Elastic collisions, which change the direction (k-vector) of the electron without energy transfer, are dominated by scattering by the (screened) Coulomb potential of the heavy core. Differential elastic scattering cross-section can be calculated using Dirac-Hartree-Fock method [5]. Because of elastic scattering in the material electrons do not travel along a straight-line, thus having an increased probability of inelastic scattering. The elastic scattering also modifies the angular distribution of photoelectrons. For electrons escaping the material both effects combined, generally, lead to EAL being shorter than IMFP for electrons escaping at an angle up to about ≈60° with respect to the surface normal whereas EAL may be even longer than IMFP for even larger angles [6]. This non-intuitive fact is a result of the definition of the EAL by the International Organisation for Standardisation: "Parameter when introduced in place of the IMFP into an expression derived for AES and XPS on the assumption that elastic-scattering effects are negligible for a given quantitative application, will correct that expression for elastic-scattering effects" [7].

It is self-evident that the depth from which an electron is escaping from the material depends on the electron emission angle α with the surface normal. For a specific material the mean escape depth (MED) equals EAL if the electron is emitted normal to the surface (α = 0). For various reasons [7], which we do not want to discuss in detail here, the angular dependence is not described by a simple 1/cos(α) relationship for larger α. For some more information on MED, IMFP and EAL of electrons, we suggest consulting a brief review that was recently published by Powell and Tanuma [7].

The drastic decrease in the photoelectric cross sections with photon energy mentioned above can only be compensated by a corresponding boost in photon flux. Highly brilliant third- generation synchrotron light sources are becoming increasingly available and hard X-ray PES (HAXPES) has become continuously more popular over the last decade [8–10] for investigating true bulk properties or deeply buried objects [11]. Extending the analytic power of PES into the (multi-) keV range requires accurate values for the EAL in this energy range as well, which motivated the present study. The availability of more experimental EAL values will also allow testing existing numerical approximations and phenomenological predictions for the EAL in this energy range. To this extent, we are presenting here a novel approach to obtain EAL values in the (multi) keV range by determining the transmission of photoelectrons through thin silicon membranes [12].

We should note already here that for using this approach the measurement of the EAL can be affected by the oxidation of the membrane material and surface contaminations. Oxygen atoms chemically reacting with the membrane increase the number of scattering centres. Therefore, values of the EAL of the silicon membrane obtained without accounting for its oxidation will underestimate the electron EAL in the pure material. Useful for future exploitation of this method, a detailed discussion of the effect of these contaminations is found in Section 3.2. It turns out that the overall uncertainties introduced by these contaminations are small.

The present study is also motivated by another reason. There are numerous studies published for applying the analytical power of PES to investigating surfaces under (near) ambient conditions [13]. However, the contradicting requirements of operating the sample at high pressure while keeping the necessary vacuum conditions for the electron analyser impose serious restrictions on the experimental set up. Using thin membranes for such applications may help. In particular combined with HAXPES, we believe that membranes might be useful for separating the delicate photoelectron analyzer hardware and electronics from harsh environments.

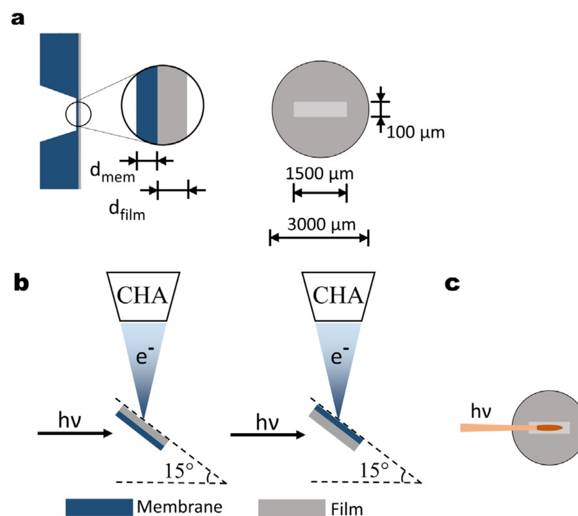

Fig. 1. (a) Schematic of aluminium coated Si membranes where d_{mem} and d_{film} are the thicknesses of the membrane and the Al film respectively. (b) Schematic top views of the two experimental geometries with film (grey) and membrane (dark blue) facing the lens-entrance of the photoelectron analyzer (CHA). Both Al films had been deposited side by side and were thus of the same thickness. (c) Sketch of the $40 \times 40\,\mu m^2$ beam incident at an angle of 15° with respect to the surface plane positioned in the middle of the membrane. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

2. Experimental

The experiments were performed on the I09 beamline at the Diamond Light Source Ltd (DLS). X-ray excitation energies E_γ of 3, 4, 6 and 8 keV were selected by a Si(111) monochromator. Photoelectron spectra were acquired using a VG Scienta EW4000 HAXPES concentric hemisphere analyzer (CHA) equipped with a 70 frame/s CCD camera as electron detector.

For the study we employed commercially available (TEMwindows.com) $0.1 \times 1.5\,mm^2$ size silicon membranes with two thicknesses d_{mem}, i.e. 9 and 15 nm. The membranes are supported on a 0.1 mm thick silicon substrate with a diameter of 3 mm as schematically shown on the right hand side of Fig. 1(a). On the face where the membrane was flush with the substrate, membrane and substrate were covered with aluminium (in vacuum, base pressure 2×10^{-6}) by e-beam evaporation with the substrates held at room temperature. The film thickness, d_{film} = 22 nm, was measured by a profilometer.

For the method which we employed for determining the EAL, it was necessary to have two membranes covered with an Al film of identical thickness. To assure this, two substrates with membranes of the same thickness were deposited simultaneously side by side with Al. With 22 nm the Al film thickness was chosen thin enough such that it was illuminated homogeneously over the whole thickness with the same X-ray intensity even for the lowest X-ray energy used (3 keV), i.e. that absorption of the X-rays in the film could be neglected. As will become clear from the following, slight differences in the thickness of these two silicon membranes would not affect the accuracy of the results. The covered samples were stored under ambient conditions for several weeks prior to the experiments. The structure of the samples which we used is shown schematically in Fig. 1(a).

Such a pair of membranes was mounted side by side on a high precision manipulator in a way that in one case the Al film and in the other case the membrane was facing the photoelectron analyzer as

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