Determination of the Be–Auger-electron attenuation length in Be using 160-keV protons

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We report the first results of a method for determining the inelastic attenuation length of low-energy electrons in the surface region of a solid from the yield of characteristic Auger electrons excited by proton bombardment. Samples of evaporated beryllium were bombarded by 160-keV protons, and the attenuation length of 100-eV electrons in Be was determined to be 6.1 Å.

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During the past decade, there has been increasing interest in electron-spectroscopic techniques [particularly Auger-electron spectroscopy (AES) and x-ray photoelectron spectroscopy (XPS)] for the analysis of the surface and near-surface regions of solid materials.[1] One of the principal problems associated with these techniques has been the limited availability of accurate electron attenuation lengths needed for the conversion of observed intensities to elemental concentrations.[2]

A commonly used technique for determining electron attenuation lengths is to monitor the Auger-electron current (caused by electron-beam bombardment) or the x-ray photoelectron current emitted from a substrate material as successive overlayers of another material are evaporated onto the substrate surface. Two problems inherent in this technique are (1) that assumptions have to be made concerning the structure of the overlayer itself and (2) that there appear to be significant sources of systematic error when short attenuation lengths ($\lesssim 10$ Å) are to be measured.[3]

In this paper we report the initial results obtained using a technique suggested by Musket and Bauer[4] to determine electron attenuation lengths in semi-infinite solids using proton-beam-excited Auger-electron emission. The yield of noninelastically scattered Auger electrons is measured with an external detector, and the electron attenuation length (at the characteristic Auger-electron energy) is determined with the use of a simple model to describe electron transport in the solid. In essence, proton bombardment can provide a calculable source of Auger electrons in the sample of interest. The principal advantage of the method is that an attenuation length can be derived with an experimental configuration and a data-analysis model similar to those now used in AES and XPS experiments.

We report here a measurement of the yield of 100-eV Be Auger electrons excited by 160-keV protons and the derived value of the attenuation length in Be for electrons of the Auger-electron energy. The observed Auger-electron signal originated predominantly ($\gtrsim 99\%$) from a surface region of depth 30 Å, whereas the range of 160-keV protons in Be is about 10^4 Å. Under these conditions, the use of proton excitation rather than conventional electron excitation is advantageous in that the transport of protons through the surface region is relatively uncomplicated, with essentially undeflected trajectories, little proton energy loss, and little proton backscattering.

A schematic outline of the experimental configuration is shown in Fig. 1. Samples of Be of at least several hundred angstroms thickness were prepared by evaporation onto an Fe substrate with the substrate surface facing the tungsten-filament evaporator. The samples were bombarded by 160-keV protons[5] at an angle of 70° to the sample normal, and the Auger electrons were detected with a four-grid retarding-field analyzer (RFA). This analyzer was oriented with its axis at 20° to the sample normal (in the plane of incidence) and was operated in the conventional manner.

Figure 2 shows a typical Be Auger spectrum where the first energy derivative of the collected current is plotted as a function of analyzer retarding voltage (i.e., the electron energy distribution). The Auger peak of interest is superimposed on a background of low-energy secondary electrons and also overlaps with structure that is predominantly associated with inelastic scattering of the Auger electrons. To determine the total noninelastically scattered Auger-electron current,

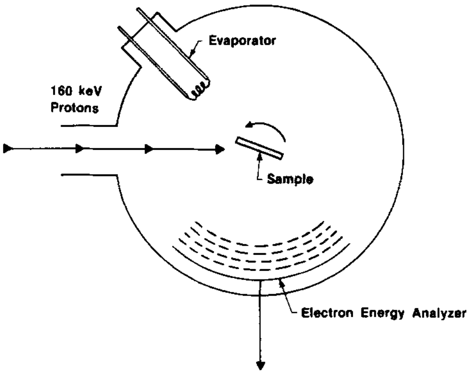

FIG. 1. Schematic outline of experimental arrangement.

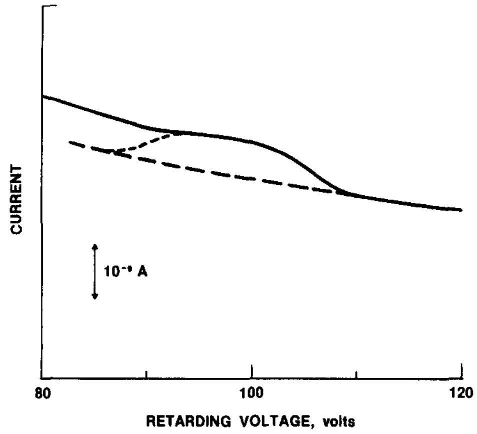

FIG. 2. Typical experimentally measured Auger-electron spectrum for beryllium under the following conditions: $E_p = 160$ keV, $I_p = 1.2$ μA, $k = 2$ V. The long-dashed line shows the extrapolated secondary electron background and the short-dashed line indicates the Auger-electron peak shape used to determine the total characteristic Auger-electron current as described in the text.

the secondary-electron background at energies higher than the Auger-electron feature has been extrapolated to lower energies in the manner indicated by the long-dashed line; the background has, in essence, been assumed to be a smooth curve over the range of interest. The predominant modes of inelastic scattering are surface- and volume-plasmon excitation,[6] and the energies and line shapes for these excitations are such that there is little distortion due to inelastic scattering of the measured Auger-electron feature for electron energies higher than about 100 eV. For this preliminary investigation, we have assumed the Auger-electron feature to be a Gaussian peak, with amplitude and width determined from the high-energy half of the observed feature, as represented by the short-dashed line in Fig. 2. We have used Taylor's formula[7] to determine the total noninelastically scattered Auger-electron current

$$I_m = 2.5\sqrt{2} I_e \sigma / k, \qquad (1)$$

where I_e is the measured current at the peak maximum, σ is the parameter describing the width of the Gaussian-shaped peak (FWHM $= 2.36\sigma$), and k is the amplitude of the modulation voltage applied to the analyzer. This value of I_m has been decreased by 5% to account for the effect of inelastic scattering on the measured current at the peak maximum and increased by 12% to account for expected asymmetry of the Auger peak.[8]

The yield of characteristic Auger electrons $Y = I_m/I_p$, where I_p is the proton current to the sample, can now be used to determine the inelastic attenuation length in the sample at the average Auger-electron energy.[4] The rate of K-shell ionization by protons in an incremental thickness dz at depth z in the solid is

$$R_0 = N\sigma_i I_p \, dz / \cos\beta,$$

where σ_i is the cross section for ionization at the proton energy E_p, N is the number density of atoms in the solid, and β is the angle of incidence of the proton beam with respect to the sample normal. If the fluorescent yield is small, then we can write $R_0 \approx R_A$, the rate of generation of characteristic Auger electrons.

The probability of an Auger electron emerging from the solid without inelastic scattering in a solid angle $d\Omega$ at an angle θ to the sample normal is

$$P = \exp(-z/\lambda \cos\theta)\, d\Omega / 4\pi,$$

where λ is the total inelastic mean free path or attenuation length in the sample at the Auger-electron energy. The total Auger-electron current emitted in the direction θ is then

$$I_A(\theta) = \int R_A P = N\sigma_i I_p \lambda \cos\theta \, d\Omega / 4\pi \cos\beta,$$

where we performed the integration with respect to z from zero to an effective upper limit of infinity. The total Auger-electron current collected by the external analyzer is obtained by an integration over the appropriate solid angle, and is

$$I_m = GTN\sigma_i I_p \lambda, \qquad (2)$$

where $T = 0.47$ is the overall collection efficiency of the RFA determined in an independent calibration of the apparatus.[8] The factor $G = 0.376$ is determined from the angular integration for our geometry [corresponding to collection of electrons from 4.2° (limit set by an internal gun) to 48° with respect to the RFA axis].

The Be Auger electron yield of $Y = I_m/I_p = 8.0 \times 10^{-3}$ was obtained as the average result for seven separate experimental measurements, most of which were obtained immediately after evaporation of a fresh Be film. This value has an imprecision (probable error) of ±10% and lower and upper bounds of 6.2×10^{-3} and 13.4×10^{-3}, respectively; these bounds have been estimated from the principal sources of error (associated with background location, peak asymmetry, sample roughness, and the possible existence of satellite Auger-electron intensity).[8]

We have estimated[8] a value of $\sigma_K = 6 \times 10^{-18}$ cm^2 for Be with $E_p = 160$ keV from the measurements of Terasawa *et al.*[9] and of Toburen,[10] and from the calculations of Garcia *et al.*[11] using the binary-encounter approximation, of Khandelwal *et al.*[12,13] using the plane-wave Born approximation, and of Basbas *et al.*[13] using a modified plane-wave Born approximation. This value of σ_K is believed to have an uncertainty of ±25%. The parameter $N = 1.24 \times 10^{23}$ atoms cm^{-3} was based on the bulk Be density.

Equation (2) has been evaluated to yield $\lambda = 6.1$ Å for 100-eV electrons in Be. We estimate that the lower and upper bounds of λ are 2.6 and 9.4 Å, respectively, as determined by combining the estimates of systematic error in Y, σ_K, N, G, and T with the imprecision (probable error) of ±10%. While the systematic errors of the present measurements are large, it is believed that they can be substantially reduced in future work. Our result for λ is consistent with the value of 8.6 Å for 110-eV electrons in Be measured by Seah[14] (uncertainties unknown) and with the value of 3.9 Å calculated for 100-eV electrons in bulk Be from a theory developed recently by Penn[15]; this theory was developed for electron energies greater than about 200 eV and may not be valid for lower energies.

We point out that the analysis leading to Eq. (2) is based on constant electron attenuation per unit path length in a homogeneous medium. This model may be invalid when the attenuation length of interest is comparable to a lattice constant and when surface-plasmon excitation may be a significant source of attenuation.[8] Thus, although the attenuation lengths obtained by the present method may not correspond to the values characteristic of the bulk material (although there is, in fact, reasonable agreement), the derived attenuation lengths are nevertheless appropriate for use in quantitative Auger-electron and x-ray photoelectron experiments.

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High-resolution topographical images of small metal particles

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A technique to obtain high-resolution topographical information of particles in the Angstrom-size with a transmission electron microscope is described. This consists of obtaining dark-field images of particles which are tilted away from the Bragg angle and in a N-beam diffracting condition. The effective extinction distance is then substantially reduced and topographical analysis of the small crystallities can be obtained from Pendellösung fringes.

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The possibility of topographical analysis of small particles (10–300 Å in diameter) is of considerable importance in the study of nucleation and growth phenomena. Moodie and Warble[1] and Cullis and Maher[2] have described some methods to obtain topographical information in transmission electron microscopy. However those techniques are not adequate for small epitaxial particles. We describe here an alternative method which allows high-resolution topographical information to be obtained in a level which is not available from other techniques. The method consists of imagining in the dark-field mode particles which have been tilted away from the Bragg condition by 2°–3°. The diffraction conditions must be such that several reflections are simultaneously excited (including systematic and nonsystematic reflections). These conditions produce an effective extinction distance whose value is much lower than that of the normal two-beam Bragg value.[3] The images of the particles appear as concentric contours of Pendellösung fringes which gives direct information about their shape.

To illustrate the effect we used gold epitaxial particles. Samples were grown on a vacuum-cleaved NaCl substrate at 250 °C. Gold films were backed with a carbon layer and then stripped from the substrate and mounted on grids. Observations were carried out in a Jeol 100-C microscope using a top entry goniometer stage. The diffraction conditions used can be observed in Fig. 1(a). Dark-field images were formed using the