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Simulation and measurement of AES depth profiles; a case study of the C/Ta/C/Si system

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ABSTRACT

A multilayer sample (C (23.3 nm)/Ta (26.5 nm)/C (22.7 nm)/Si substrate) was submitted to AES depth profiling by Ar^+ ions of energy 1 keV and angles of incidence of 72°, 78°, and 82°. The shapes of the as-measured depth profiles were strongly different emphasizing that the ion-bombardment conditions strongly affects the shapes of measured depth profiles. We simulated the depth profile measured at an angle of incidence of 72° by calculating the backscattering factor, applying attenuation lengths available in the literature, and simulating the ion-bombardment-induced specimen alteration with a TRIDYN simulation and a trial and error method. The good agreement between the calculated and measured depth profiles justified the method applied.

1. Introduction

Auger electron spectroscopy (AES) depth profiling is a widely used method for more than 30 years for analyzing thin-film structures. Its principle is well known; the surface layers are removed by repetitive ion bombardment and current concentration of the instantaneous surface is determined by AES. The method provides the Auger intensities vs. sputtering time curve. Knowing the sputtering rate, the sputtering time can be converted to depth providing the so-called depth profile. The depth profile is generally used to reveal the in-depth concentration profiles of the sample studied, but it can also be used to reveal the ion-bombardment-induced alteration if the sample structure is known in advance. In both cases, the challenge is to find all effects which contribute to the distortion of the "ideal" profile.

Several papers were devoted to technical problems of depth profiling [1–4]. Part of the problem is connected to the material-removal process. It is well known that ion bombardment (depending on the material, projectile type, projectile energy, etc.) introduces alteration to the material especially at interfaces (e.g. ion mixing), which are frequently the target of the study. Without knowing the alteration introduced, one cannot convert the sputtering time to depth, and thus cannot reconstruct the in-depth distribution of concentrations of the sample.

The other part of the problems is connected to AES itself. The Auger current of element i is proportional to $\int I_0 N_i r \exp(-z/\lambda \cos\alpha)\, dz$ [5], where I_0 is the primary current, N_i is concentration of element i in-depth z, r is the backscattering factor (BF), λ is the inelastic mean free path (IMFP) and α is the emission angle. When AES depth profiling is applied to reveal in-depth concentration distributions, obviously, N_i, λ, and r depend on z. The dependencies of the two latter parameters are different, however. The IMFP depends on the local composition of the uppermost layer. On the other hand, r depends on the structure of the material studied for depths (depending on the energy of the Auger line) of hundred of nms, since the primary-electron beam, penetrates deeply into the bulk of the solid and the backscattered current also contributes to the excitation process [6]. Thus, during depth profiling r continuously changes depending on the structure of the remaining material.

Considering these problems, it seems evident that the determination of the concentration distribution of the elements from the measured Auger depth profile, that is, to invert the integral for all depths is in general not possible. On the other hand, reasonable methods are known for the calculation of the attenuation length of Auger electrons [7] and the BF for any structure [8,9] (which made it necessary to introduce a new definition for the BF [10]), and ion mixing can also be modeled [11,12]. Using these ingredients, one can simulate (assuming an initial structure) the AES depth profile. By comparing the simulated profile with the measured AES depth profile the initial structure of the material can be estimated.

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Menyhard [13] long ago proposed simulation of the AES depth profile based on the calculation of ion mixing using the TRIDYN simulation. That method does not account for the variation of the IMFP with composition and for the dependence of the BF on the current bulk structure. The MRI model developed by Hofmann considers the above parameters but is based on particular assumptions for either the BF or ion mixing [2,14].

In this paper we will present a simulation of the AES depth profile based on calculations of ion-bombardment-induced alteration, attenuation lengths, and BFs. The simulated depth profile will be compared with the measured profile.

A layered system, C/Ta/C/Si substrate, made of elements with different BFs, was AES depth profiled. Ion mixing was found to be very different on the two C/Ta and Ta/C interfaces, and this type of sample is thus ideal for checking the model for calculation of ion mixing. To simulate the depth profile we use BFs calculated by the Zommer et al. algorithm [8,9], attenuation lengths for the mixed regions from the available NIST database [7] with Tanuma et al. IMFPs [15], and ion mixing is modeled by applying TRIDYN simulation [16] and a trial and error method [17]. We have found good agreement between the simulated and measured AES depth profiles, which supports the validity of our approach.

2. Experimental

Ta/C layers were sputter-deposited to HF etched Si(1 1 1) substrate in a stainless steel UHV chamber equipped with 2 in. diameter unbalanced DC magnetrons. The base pressure of the system was 1×10^{-8} mbar, and the Ar working-gas pressure was 2.5×10^{-3} mbar. The applied power was 100 W and 150 W using Ta (99.95%) and C (99.999%) Kurt J. Lesker targets with deposition rates of 0.26 nm/s and 0.23 nm/s, respectively.

Cross sectional transmission electron microscopy (XTEM) and high resolution electron microscopy (HREM) analysis were carried out with a Philips CM20 200 kV analytical electron microscope. The sample preparation method, which is described in detail by Barna et al. [18], consisted of mechanical thinning and polishing to a thickness of 50 μm, and followed by grazing-incidence (85°) ion-beam thinning with 10 kV Ar^+ ions. The ion-beam thinning was finished with 3 keV Ar^+ bombardment.

AES depth profiling was carried out in our dedicated system, equipped with a differentially pumped TELETWIN (made by Technoorg Linda) ion gun [19]. Ar^+ ions were used for sputtering. The ion-beam shape is Gaussian (FWHM = 0.3 mm) and the typical ion current is about 3 μA. The bombarding ion current was kept constant during the depth-profiling measurements. The energy of the ions was 1 keV with angles of incidence (with respect to the surface normal) of 72°, 78°, and 82°. The specimen was rotated during ion bombardment with a rate of 3 rev/min.

AIST-NT SmartSPM 1000 atomic force microscope (AFM) in semicontact mode was used for ex situ measurement of the ion-bombardment-induced surface morphology. The scanned area was 1 μm × 1 μm. The root means square (RMS) values of height deviations were used as a parameter describing surface roughness. The surface roughness was determined on sample ion bombarded at an angle of incidence of 72° by terminating the AES depth profiling in the vicinity of the Ta/C1 and C2/Ta interfaces.

A STAIB DESA 100 cylindrical mirror type analyzer (with preretardation) recorded the Auger spectra. The energy and angle of incidence (with respect the surface normal) of the primary-electron beam were 5 keV and 54°, respectively. The primary-electron current was 20 nA with a beam diameter of 45 μm. The electron and ion beams were aligned to hit the same point. The angle between the axis of the analyzer and the sample normal was 3°. The acceptance angle of the analyzer is 26 ± 6°.

The AES depth profile was collected in an alternate sputtering mode (computer controlled). This procedure consists of two steps. First, the ion sputtering takes place with rotated specimen. Then the ion bombardment is interrupted. This is followed by stopping the sample always in the same position. The Auger analysis takes place on a fixed specimen. Then the whole process is repeated. The following Auger peaks were measured in counting mode: Si (92 eV), Ta (181 eV), C (272 eV), with energy resolutions of 1 eV, 2 eV, 4 eV, respectively. The measured $N(E)$ curves (recoded with energy steps of 0.3 eV, 0.3 eV, and 0.5 eV for resolutions of 1 eV, 2 eV, and 4 eV, respectively) were numerically smoothed and differentiated by applying the Savitzky–Golay routine with 11 points. The peak-to-peak amplitude was considered as the measure of Auger intensity. The following Auger peaks were measured in counting mode: Si (92 eV), Ta (181 eV), C (272 eV), with energy resolutions of 1 eV, 2 eV, 4 eV, respectively.

The measured sputtering time should be converted to depth (removed layer thickness) to provide useful information on the layer structure. This is not a simple task if the difference in the sputtering yields of the two materials is large and the interface broadening is not negligible. In the present case, the relative sputtering yields are strongly different (Y_C/Y_{Ta} are 2.7, 4.6, and 5.9 for angles of incidences of 72°, 78°, and 82°, respectively). In addition, the Ta/C interface in the AES depth profile is rather broad [20]. Thus we will not convert the sputtering time to depth for the comparison of the simulation and measurement; rather we will compare the time (or fluence) vs. Auger intensity curves.

3. Calculations

3.1. Backscattering factor

The main features for the BF calculations for a layered sample were introduced in Refs. [8–10]. The MC model presented in [8,9] allows one to obtain the BF for a given layer of material for a given transition in a layered sample. The layer of interest can be buried inside a matrix material or deposited at the surface. Additionally, the layers do not have to be uniform with depth, since in such cases every non-uniform layer can be approximated by an appropriate set of thin layers of varying compositions.

Let us present the main concepts of the BF calculations. We derive at first the relevant defining formula. We assume, as in the common formalism for AES, that the Auger current, $dJ_A(\alpha, z)$, originating in a very thin layer dz at a depth z in a layer of thickness t at depth d, and leaving the sample surface in direction at emission angle α can be expressed as

$$dJ_A(\alpha, z) = rI_0P_AN\sigma(E_p)\sec\theta_0\varphi(z, \alpha)dz \quad (1)$$

In contrast with the common formalism, the probability of Auger-electron escape from a sample is not required to be an exponential function of depth, but is expressed here generally by $\varphi(z, \alpha)$ the emission depth distribution function (EMDDF) that is determined by MC calculations. Other symbols have the usual meaning: r is the BF, I_0 is the primary beam current, P_A is the probability that the Auger transition follows the ionization, N is the atomic density in the layer, $\sigma(E_p)$ is the ionization cross section of the level responsible for the selected Auger transition at the incident energy E_p, θ_0 is the incident angle of the primary beam, and α is the emission angle of the Auger electron with respect to the surface normal. Some constants in Eq. (1) are omitted for clarity. The same current, $dJ_A(\alpha, z)$, can be expressed in a different way as the product of two functions:

$$dJ_A(\alpha, z) = \Psi(z, E_p, \theta_0)\varphi(z, \alpha)dz \quad (2)$$