New Developments in Depth Profiling using AES

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Some alternative methods to common, routine AES depth profiling are outlined, that are based on the specific possibilities of electron beam excited electron spectroscopy with high energy resolution. These methods are (i) simultaneous recording of high and low energy Auger peak of an element during profiling for the determination of the sputtered depth, (ii) determination of the mixing length by angle resolved AES (ARAES), (iii) elastic peak electron spectroscopy (EPES) for improved depth resolution, and (iv) reflection electron energy loss spectroscopy (REELS) for determination of the variation of the electron inelastic mean free path while sputtering through an interface.

INTRODUCTION

When I first met Tetsu Sekine in the late eighties, we were discussing future developments in AES. Later, when he had developed the new JEOL JAMP instrument with hemispherical analyzer, he cordially invited me to test the new equipment. For about one week we were working, and he wrote up some of the results in a common publication in *JEOL NEWS*[1]. In that paper, he clearly pointed out the advantages of the hemispherical analyzer for AES as compared to the cylindrical mirror analyzer. In particular the higher energy resolution –combined with high sensitivity - proved to be advantageous for our work on chemical state depth profiling on Ni_3Al samples. The open mind and the gentle attitude of Tetsu Sekine, as well as the devotion to his work are unforgettable for me. In his work, he was constantly looking for better alternatives to common approaches. Indeed, this is one of the keys of scientific and technological progress. Keeping that in mind, I try to show a few alternative methods beyond routine depth profiling using AES.

ALTERNATIVE METHODS IN AES SPUTTER DEPTH PROFILING

Intrinsic estimation of the depth scale in AES depth profiling

As emphasized in the recently published ISO Technical Report, ISO/TR 15969:2000[2], the measurement of the sputtered depth is one of the key tasks in quantitative depth profiling. AES sputter depth profiling offers the unique chance to estimate the depth scale from the measured interface profiles, if the high and the low energy peak of an element are simultaneously monitored in the same profile[3]. Many elements (e.g. Si, Al, Cu, Ni, Fe, Cr, Ta, Nb, W,...) posess high and low energy peaks with similar sensitivity, for example Ni (LMM 848 eV and MVV 60 eV). Because these peaks have different electron attenuation lengths (λ_2^0 =1.01 and λ_1^0= 0.30 nm[4], respectively, corresponding to an electron escape depth of λ_2 = 0.747 and λ_1 = 0.225 nm for a CMA oriented perpendicular to the sample surface, where $\lambda_{1,2} = \lambda_{1,2}^0 \cos 42.3^0$), the corresponding profiles are shifted by a characteristic length, $\Delta\lambda$, depending on the values of mixing length w and escape depth λ, as given by the MRI-model[3]. For negligible influence of *w*, the shift is given by $\Delta\lambda = \ln 2\,(\lambda_2 - \lambda_1)$[5]. An example gives Fig. 1, where the AES depth profile of the low and

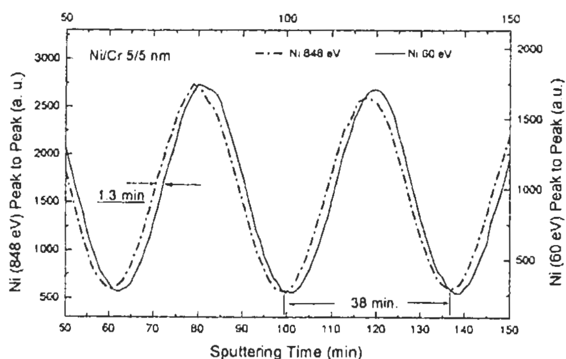

Fig. 1. Mutual shift of the high (848 eV) and low (60 eV) Ni Auger peak determined in the AES depth profile of a Ni/Cr multilayer with 5 nm thickness per single layer[4].

high energy peak of Ni is shown for a 5/5 nm Ni/Cr multilayer structure[6]. The shift of the profiles, normalized to the same amplitude, of about 1.3 min., is clearly recognized in the sputtering time scale that is equivalent to about $\Delta\lambda$ = 0.36 nm. This means, the average sputtering rate is 0.28 nm/min., and the double layer thickness corresponding to 38 min. sputtering time is 10.5 nm, close to the nominal thickness of 10 nm. It should be emphasized that this intrinsic method of sputtered depth determination is not a high precision method, but because of its simplicity -without the necessity of additional methods- it gives at least a valuable estimation in case of unknown samples.

Angle Resolved AES for Determination of the Mixing Length

A quantitative description of a measured depth profile in terms of the original depth distribution of composition, as, for example, with the Mixing-Roughness-Information depth (MRI)-model[3,7,8], depends on three fundamental parameters that can be theoretically predicted or measured. A key parameter is the mixing length *w*. This parameter is usually determined with the help of reference samples[8]. However, it can also be measured independently by angle resolved AES (ARAES). When using a concentric hemisphere analyzer (CHA) for AES, emission angle dependent determination of layer thicknesses are possible as in angle resolved XPS (ARXPS)[9]. A precondition for such a determination is a surface layer of different composition with respect to the bulk beneath. Of course this condition is met in sputter depth profiling, if the profiling is stopped when sputtering through an interface. Such an experiment was carried out earlier on the AlAs/GaAs interface of a multilayer structure[10]. The Al (1396 eV) and the As (1227 eV) signals were measured, that have negligible difference of their attenuation lengths. In the region of decreasing Al-signal, only the mixing zone contains Al. Assuming constant concentration in that zone, the ratio of the Al and As signal intensity, $I(\mathrm{Al})/I(\mathrm{As})$, is given by:

$$I(\mathrm{Al})/I(\mathrm{As})=\mathrm{const}*[1-\exp[-(w/(\lambda \cos\Theta))]] \qquad (1)$$

Where w is the mixing length, λ is the Auger electron attenuation length (here: 2.32 nm[10]), and Θ is the Auger electron emission angle (to the normal to the surface). Figure 2 shows an example for a depth profile obtained with 500 eV Ar^+ sputtering under 80 deg. incidence angle. The

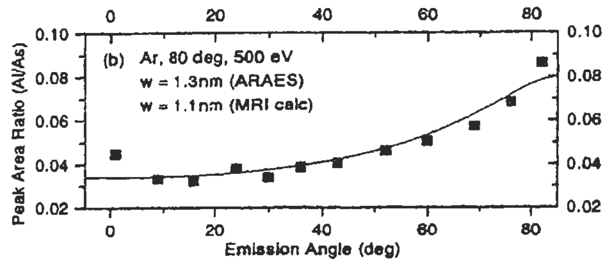

Fig.2. Angle resolved AES applied to the mixing layer of an AlAs/GaAs interface. The full drawn line is the fit of the maesured points (full squares) according to eq.(1)[8].

shape of the curve then determines the value of the mixing length, *w*, given in the inset, together with the one derived from the MRI fit to the profile of this reference sample. Both values are in good agreement.

Depth Profiling using Elastic Peak Electron Spectroscopy (EPES)

Depth profiling using elastic peak electron spectroscopy (EPES) is an alternative to AES depth profiling[11]. Recently, it was successfully applied to a Ge/Si multilayer thin film structure, and an improved depth resolution was

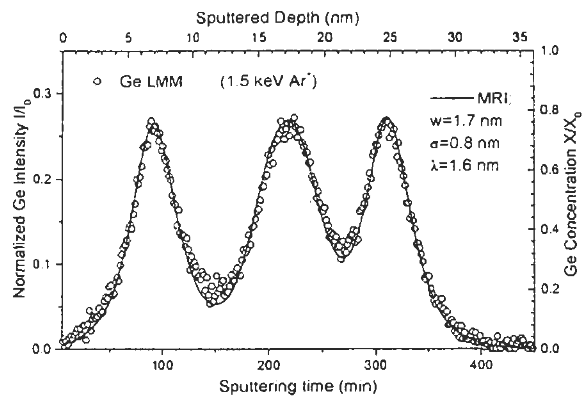

Fig. 3a. AES depth profile of Ge (1147 eV) in a Ge/Si multilayer[10].

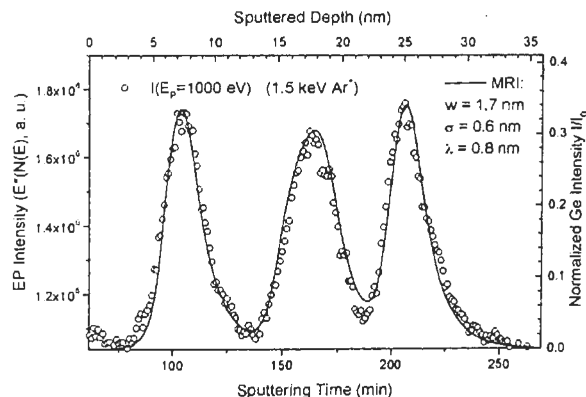

Fig. 3b. EPES depth profile of the same sample as in Fig. 3b[10].

demonstrated, as seen in Fig. 3. Figure 3a shows the AES depth profile of Ge (1147 eV) of a multilayer Ge/Si structure[12], and Fig. 3b the EPES depth profile of the same structure obtained with the same sputtering conditions ((1.5 keV Ar+ ions, 74 deg. incidence angle), using a primary beam energy of 1000 eV. The better depth resolution of the latter is obvious.

Quantitative evaluation of the measured profile with the MRI model (see the respective MRI parameters given in the inset of Figs. 3a ,b) has shown that the depth resolution function in EPES depth profiling is only determined by the effective information depth, and is therefore only a function of the primary electron energy, the incidence angle and the emission angle of the elastic peak electrons[12]. EPES at the same energy as an AES peak gives a considerably higher depth resolution, because the attenuation length for both the incoming and the emitted electrons has to be taken into account. Further advantages of EPES as compared to AES depth profiling are its independence on chemical bonding and the generally higher signal to noise ratio resulting in a higher precision for profile evaluation. This was shown for the determination of small interdiffusion lengths (of the order of 1 nm) with the relatively high precision of better than 0.2 nm[12].

Sputter Depth profiling using Reflection Electron Energy Loss Spectroscopy (REELS)

REELS is often applied to determine the inelastic mean free path (IMFP) of the electrons[13]. The use of this feature in depth profiling is fairly new. At the QSA-12 conference in 2002, its application for determination of the change of the IMFP when sputtering through an Fe/Si interface was demonstrated[14]. The sample consisted of a 150 nm thick Fe layer sputter deposited on a smooth silicon substrate. Sputter depth profiling was performed with 3 keV Ar^+ ions at about 15 deg. incidence angle. Following AES depth profiles, REELS measurements were performed after different sputtering times with 3 keV Ar^+ ions to obtain a REELS depth profile.

Figure 4a shows the development of the REELS spectra when sputtering through the Fe/Si interface, from pure Fe (back) to pure Si (front). These REELS spectra can first be decomposed in the two elemental components to get the average composition at any point at the interface profile. Then, using the theory of Yubero et al.[13], the parameters for each REELS spectrum, notably the IMFP, are determined.

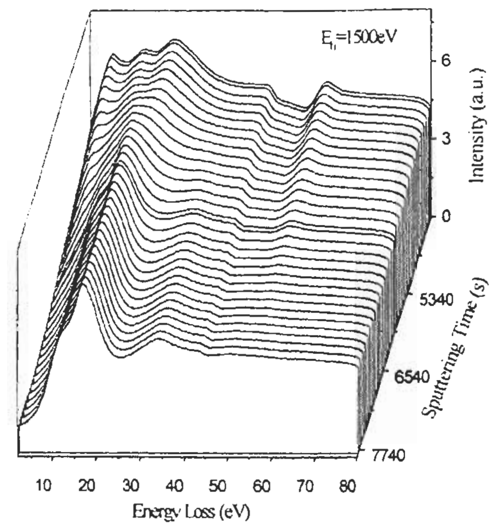

Fig. 4a. REELS depth profile showing the change from Fe (back) to Si (front) loss pectrum when sputtering through an Fe/Si interface[12].

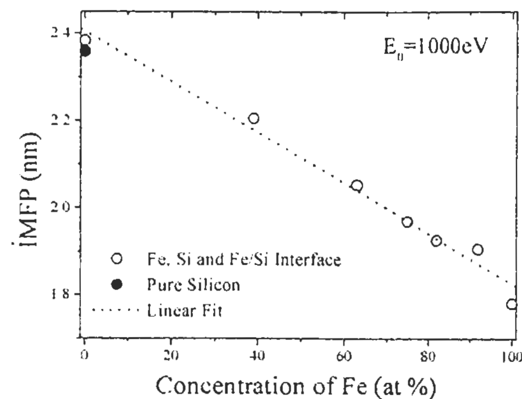

Fig. 4b. Variation of the IMFP as a function of the concentration of Fe when sputtering through an Fe/Si interface [12].

In that way, the plot in Fig. 4b is obtained, that shows the IMFP as a function of the composition when sputtering through the Fe/Si interface. The dependence is approximately linear. Such a linear composition dependence can be incorporated in the MRI model for a more exact quantitative profile evaluation[15].

CONCLUSION

There are quite a few alternative methods to the usual AES depth profiling, that are useful to determine additional important parameters, or to improve the quality of a profile in certain cases. Four main examples of such methods were given, that can be performed with any standard Auger spectrometer. Only the second example, namely the determination of the mixing length by ARAES, needs a concentric hemispheres analyzer or a double pass ana-

lyzer with a slit-drum device. The first example, intrinsic depth scale determination using high and low energy peaks of the same element, is restricted to elements from the second row of the periodic table onward and is hardly possible for some elements with high energy peaks of too little sensitivity (e.g. Ag, Sn, Sb). The more sophisticated EPES depth profiling is restricted to purely binary systems, but because the primary energy can be practically arbitrarily chosen, it offers the unique possibility of decreasing the effective attenuation length to negligible values. Finally, REELS depth profiling has the capability of providing quantitative depth profiles by internal standardization and in addition to yield the electronic parameters including the IMFP, a key parameter in any quantification, as well as its change when sputtering through an interface.

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