Ultimate Depth Resolution in Sputter Profiling

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Abstract

Progress in both understanding the various causes of degradation of depth profiles and advanced instrumental performance has led to much improvement of the depth resolution in sputter profiling, characterized by one parameter, Δz. In general, the depth resolution function is non-Gaussian and often asymmetric. Therefore more than one parameter is needed to precisely describe the depth resolution function, which is necessary to reconstruct the original depth distribution of composition from the measured profile. For many systems it is possible to approach the physical limits of ultra high depth resolution, which are mainly determined by the three fundamental parameters atomic mixing, surface roughness and information depth. It is pointed out how each one has to be optimized, i.e. made as small as possible in order to attain the physical limits of the depth resolution. Some recently determined experimental resolution functions are discussed in terms of the so-called MRI model. The physical limit of any of these parameters appears to be around 1-2 monolayers, their random superposition yields about 3 monolayers or roughly 0.8-1 nm for the ultimate depth resolution, and values of about 1.5 nm have already been obtained.

1. Introduction

Sputtering in combination with any of the most popular surface analysis techniques AES, XPS and SIMS has become the most frequently and routinely applied method to obtain depth profiles which- in most cases- more or less exactly resemble the original distribution of elemental composition with depth (for review, see e.g. [1]). Precision and accuracy of depth profiling, i.e. the deviation between the in depth distribution and the measured profile is usually given by a certain depth resolution Δz which describes the apparent broadening of the depth distribution as represented in the measured profile.

The definition of Δz adopted by IUPAC and by the ASTM E-42 committee on surface chemical analysis [2] states that "depth resolution is the depth range over which a signal increases (or decreases) by a specified amount when profiling an ideally sharp interface between two media. By convention the depth resolution corresponds to the distance over which a 16% to 84% (or 84% to 16%) change in signal is measured". If the resulting shape of such an interface profile can be approximated by an error function, this definition means $\Delta z = 2\sigma$ where σs is the standard deviation of the corresponding Gaussian resolution function [1].

2. Depth Resolution (DR) and Deconvolution of Profiles

For the experimental researcher, the depth resolution simply describes a depth region over which the concentration distribution is averaged. Provided that the basic relations between intensity and concentration and between sputtering time and depth are linear, this means for example that with a DR of Δz=3 nm (about 10 monolayers) we should not be able to differentiate e.g. between an elemental concentration of 100 % in the first 5 monolayers and 0 % in the second 5 monolayers or vice versa or that of 50 % for all of the 10 monolayers. In fact the shape of the respective measured profile will look slightly different and an exact knowledge of the depth resolution function (DRF) allows profile deconvolution with a precision in the monolayer regime [3], as shown below.

Sputter depth profiling is the transformation of a real world compositional distribution into an image of it, namely the measured depth profile. This transformation is described by the convolution integral which is governed by the depth resolution function (DRF) g(z-z') [1], by which the mole fraction X(z') of the respective element at depth z' is convoluted to yield the measured and normalized intensity I(z) at the sputtered depth z.

Deconvolution means the reverse operation necessary to directly obtain X(z') which of course is possible if the DRF g(z-z') and I(z) are known. However, it was shown earlier that usual mathematical approximation and measurement errors tend to "roughen" the

retrieved X(z') curve, making it somewhat ambiguous [1]. Therefore, it is customary to solve the convolution integral by first assuming X(z'), then to get a respective I(z) and to compare this calculated ("simulated") profile with the measured one until an optimum fit is obtained by trial and error [4]. In any case, the DRF g(z-z') has to be known either from experiment or from theory.

3. Depth Resolution Function: Experimental Determination and Model Calculations

According to the convolution integral, a very thin layer ("delta layer") with X(z) = 0 in its vicinity directly gives I(z) = g(z-z'). Because the maximum normalized signal intensity of a single layer decreases considerably with its thickness [1] this method to experimentally determine the DRF is particularly useful in SIMS with its high detection sensitivity [5]. Although it was already shown in 1977 that this approach can also be used in AES profiling [6], the step function approach is more useful here. From the convolution integral it follows that for a step function distribution (X(z) = 1 or 0) the DRF g(z-z') is given by the absolute value of the measured (and normalized) profile I(z) differentiated after z [7,8]. Likewise, the depth z can be replaced by the sputtering time t if the sputtering rate, dz/dt, is known and constant. The result of such a procedure applied to the measurements in Fig. 1a is shown in Fig. 1b by the open points. Fig 1a shows the AES sputter depth profile of Al in a GaAs/AlAs (8.8/9.9nm) multilayer (performed with Ar^+ ions of 600 eV energy at 80 deg. incidence angle) using both the low (Al1, 68 eV) and high energy (Al2, 1396 eV) Auger peaks of aluminium.

Recently, a model for the theoretical description of the DRF useful for both SIMS and AES profiling was developed based on a simple model for the mixing influence [7,8] by extending it to include surface roughness and information depth (called MRI-model from Mixing - Roughness - Information Depth [1,3,5], given by the mixing length, w, the Gaussian roughness standard deviation, σ, and the electron (AES, XPS) or ion (SIMS) escape depth, λ). Note that these parameters also include the observed shift of the measured interface location with respect to the original one (see Fig. 1).

Comparison with the theoretically determined DRFs calculated with the MRI-parameters for

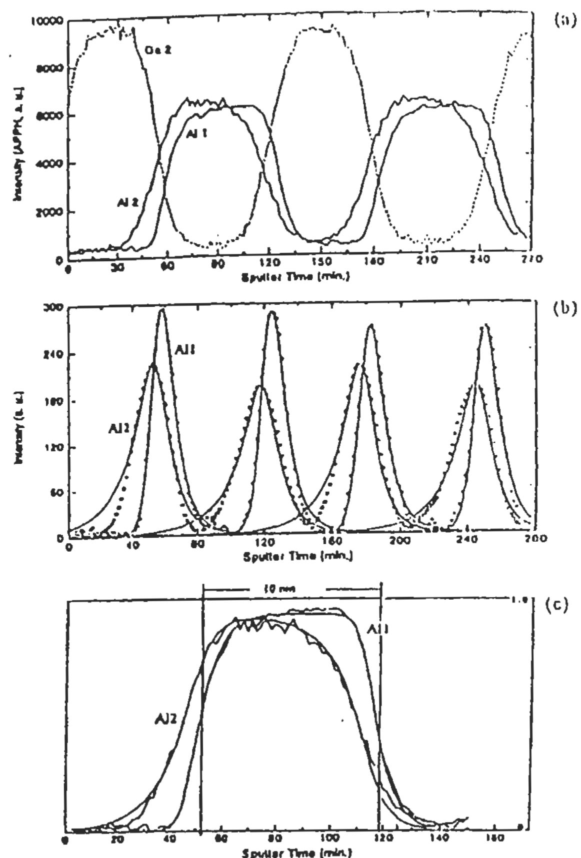


Fig. 1 a) Low (Al1) and high (Al2) energy AES sputter depth profile of Al in a GaAs/AlAs (8.8/9.9 nm) multilayer using 0.6 keV Ar^+ ions and 80 deg. incidence angle.(from refs.3,8)
b) Experimental depth resolution functions (DRFs) for Al1 and Al2 obtained from a) by differentiation (open points) and fitted by calculated DRFs (full drawn lines) using the MRI model with w=10nm, σ=0.6nm, λ(Al1)=0.4nm and λ(Al2)=1.7nm.
c) Profile simulation of a) with the parameters in given in b) and assuming a rectangular distribution of Al with depth [3].

optimum fit (full drawn lines) shows very good agreement (Fig. 1b). As seen by the large difference of the DRFs for which only λ differs (by 1.3 nm, i.e. about 4.5 monolayers), a difference of about 1-2 monolayers in the original concentration - depth distribution can already be detected. Fig. 1c shows the result of the measured profile simulation with these parameters assuming a sharp rectangular concentration distribution of Al in the AlAs layer sandwiched between two GaAs layers [3,8].

4. Optimizing the Experimental DRF: Towards Ultimate Depth Resolution

For a suitable sample which is laterally

homogeneous and provided that instrumentally caused artefacts (e.g. non-constant ion beam intensity in analyzed area etc.) are negligible, the main parameters are contained in the MRI-model: Ion beam induced atomic mixing, roughness and the information depth. To attain the ultimate possible depth resolution, each parameter has to be optimized, i.e. made as small as possible.

Atomic mixing in the collisional cascade is the fundamental effect of sputtering induced surface composition changes. At the practical limit of about 150-200 eV, only about 1-2 atomic monolayers are expected for the "mixing" zone. To achieve such a low mixing length, molecular ions of larger mass can be employed at somewhat higher energies[9]. The "mixing" effect can be further reduced to one monolayer using reactive ions if the energy approaches the sputter limit and "chemical" sputtering becomes prevalent. The mixing length w can be furthermore diminished by sputtering with reactive ions if "swelling" of the matrix lattice occurs due to compound formation.

Preferential sputtering, radiation enhanced diffusion and interfacial segregation sometimes play a preponderant role and may change the DRF in a way which is difficult to predict. In a two component system, preferential sputtering can be directly included and predictions compare fairly well to experiments[5].

Fig. 2 shows an example of the DRF for different sputter coefficient ratios r = ż(delta layer element)/ż(matrix). It is recognized that preferential sputtering of the delta layer component decreases w (w→w/r) [5] and vice versa..

Sputtering induced surface roughness can be drastically reduced by sample rotation[10]. In favourable cases, i. e. for optimum adjustment and with high enough rotation speed and high incidence angle the physical limit of about 2 monolayers can be attained as measured by AFM[11].

The information depth is inherently given by the analysis method. It is about 1-2 monolayers in SIMS (given by the depth of origin of secondary particles) and about 1-10 monolayers in AES, depending on the escape depth and therefore on the kinetic energy and on the emission angle of the Auger electrons (see the marked difference in Fig. 1 between the profiles and DRFs for the Al 68 and Al 1396 eV intensity).

The ultimate depth resolution is given by the physical limits of the three parameters mixing length (w), roughness (σ) and information depth (λ) [12]. According to the brief discussion above, the limiting values are estimated to w = 1-2 ML, σ = 1 ML and λ =1 ML or, in nm units (1ML= 0.28 nm) w = 0.3-

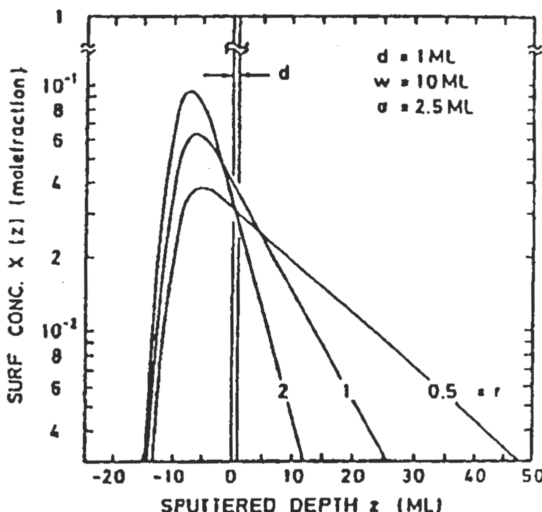


Fig. 2 MRI-calculation of the (SIMS) sputtering profile of a delta marker layer of one monolayer (ML) thickness and different marker/matrix sputtering rate ratios r = 0.5; 1; 2. Other MRI-parameters are σ (roughness) = 2.5 ML, w (mixing) =10 ML, λ(info)=0.[5] (Note that the DRF becomes nonlinear with concentration if r differs from unity)

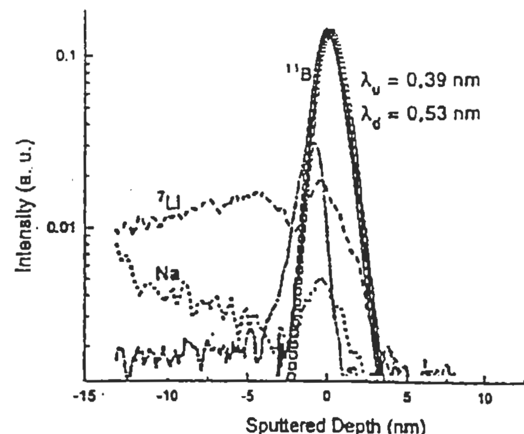
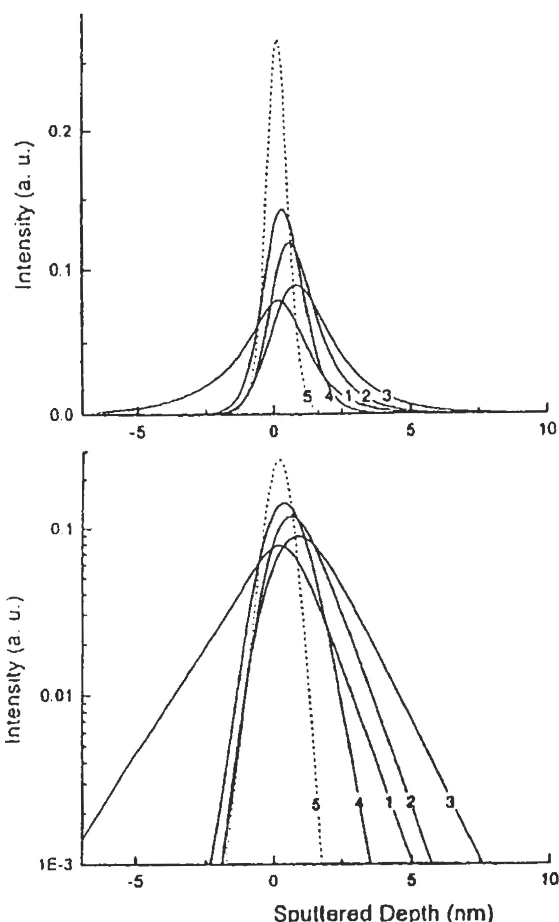


Fig. 3 ToF-SIMS profile of a B delta layer in Si after Iltgen et al. [9] (full line), fitted with the MRI-model with w= 0.53 nm, σ=0.6 nm, λ= 0.39 nm (open squares).

0.6 nm, σ = λ = 0.3 nm. Recently, Iltgen et al. [9] succeeded in coming close to these limits with a ToF-SIMS profile of a delta layer of B in Si using SF5+ primary ions of 600 eV energy.



MRI-Parameter (nm)

DRF	w	σ	λ	Ref.
1	1.0	0.6	1.7	3
2	1.0	0.6	0.4	3
3	1.39	0.8	0.3	5,11
4	0.53	0.6	0.39	9
5	0.3	0.3	0.3	limit

Fig. 4 Experimental DRFs (curves 1,2: AES; curves 3,4: SIMS) fitted by MRI-calculations with the parameters shown in the attached table, and a purely theoretical DRF (curve 5) representing the ultimate depth resolution limit (corresponding to Δz - 0.9 nm). Upper curves: linear and lower curves: logarithmic intensity scale.

Fig. 3 shows the measured profile together with the fitted MRI-calculated DRF, yielding w = 0.53 nm , σ = 0.6 nm, λ = 0.39 nm (Note that the values for w and λ are equal to the characteristic trailing and leading edges [3]). Because w and λ are already optimized almost to their physical limit, the roughness parameter (which contains straggling of the mixing zone and original layer sharpness, [3]) mainly determines the depth resolution. In Gaussian approximation, the adding up in quadrature of the three contributions gives about Δz = 1.6 nm in fairly well agreement to the value of about 1.4 nm derived from the FWHM of the profile in Fig. 3 and is comparable to the best values reported for optimized conditions in AES depth profiling of Ta2O5/Ta[13] and of GaAs/AlAs [14] interfaces. Fig. 4 shows some experimental DRFs (curves 1-4) fitted by MRI-calculations with the parameters given in the inset table and a purely theoretical DRF (curve 5) representing the ultimate depth resolution limit (corresponding to Δz = 0.9 nm). The set of curves is shown with linear and logarithmic intensity scale, as it is customary in AES and SIMS profiling, respectively. As shown here and e.g. by Kitada et al. [15], the MRI model is capable to reproduce measured DRFs very well with the additional advantage to get an analytical description based on physically meaningful parameters.

5. Conclusion

By careful optimization of all depth profiling parameters, ultra-high depth resolution can be attained. It is determined by the physical limits of sputtering induced changes of composition and topography and of the information depth of the analysis method, basically described by the three parameters atomic mixing, surface roughness and information depth. The depth resolution function is shown to be well represented by three respective functions, two exponentials and one Gaussian, mathematically coupled in the so called MRI model according to their physical relevance. Experimentally, the width of the atomic mixing zone is minimized by using a low primary ion energy (typically below 500eV) and glancing ion incidence. Roughness is diminished by using glancing ion incidence and sample rotation. The information depth depends on the analysis method. In SIMS, it is given by the depth of origin of the detected particle and is generally confined to the first 1-2 monolayers, whereas in AES it is determined by the electron escape depth. The physical limit of any of the three parameters appears to be around one monolayer, their random superposition yields about 3 monolayers or roughly Δz = 0.8-1 nm for the ultimate depth resolution. If the experimental resolution function is known with the high accuracy of about one monolayer or better, features of about this size can be resolved /1/.On the other hand, this accuracy requires high quality standard samples for measurement of the resolution function of atomically sharp

delta layers and /or interfaces.

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