A Framework for Quantitative Depth Profiling

S. Hofmann
Max-Planck-Institut für Metallforschung, Inst. für Werkstoffwissenschaft
D-70174 Stuttgart, Germany

In scientific common sense, to be quantitative means putting error bars to data. Therefore accuracy and precision have to be defined within a certain concept of measurement and evaluation and, based on the gathered knowledge, the experimental approach has to be optimized in order to minimize the errors as far as possible.
There are numerous papers and review articles dealing with quantitative depth profiling. For about twenty years, many researchers have more and more disclosed the various parameters that influence the error, i.e. the deviation of a measured depth profile from the true in depth distribution. Theoretically modeling these deviations helped to understand optimized profiling conditions and therefore to work with them . The various approaches to quantitative depth profiling agree within a general framework but often differ with respect to the degree of comprehensiveness and sophistication.

Common to all sputter depth profiling work are the following considerations: As a final analytical result, the in depth distribution of elemental composition (=concentration as a function of depth) is required, based on the measured raw data which usually consist of elemental signal intensities as a function of the sputtering time. Therefore the following three fundamental procedures are necessary /1/:
(1) Conversion of the measured sputtering time into sputtered depth ($z = f(t)$)
(2) Conversion of the measured signal intensities I into concentrations X ($X = f(I)$)
(3) Conversion of the measured profile shape into the exact shape of the original in depth distribution. This means corrections of errors or artefacts introduced by ion beam/sample interactions. These distortions limit the precision of a measured profile and are represented by the resolved depth which is called depth resolution Δz.

It should be noted that the above three problems are closely coupled and can only be separated in a first order approximation. From a conceptual viewpoint, most desirable is a quantitative deconvolution, i.e. a mathematical procedure which is able to directly convert the measured profile into a quantified in depth distribution of composition. Much less complicated and easier to perform are the three tasks outlined above. They can be favourably subdivided in stages of different complexity between the coarse,

simple quantitation procedures with elemental sensitivity factors only and the more sophisticated ones, depending on both the required accuracy and the available additional anformation. These stages are outlined in the following:

1. Quantification of the Sputtered Depth $z = f(t)$

This task means to find an appropriate value of the sputtering rate $\dot{z} = dz/dt$ which in general is a function of the composition and therefore may vary through the profile. Only if the sputtering rates of the elements are not too different, $\dot{z}$ = constant and $z = \dot{z} \cdot t$ applies to convert t in units of depth z. Of course there is always a change in the sputtering rate at the beginning due to initial build up of the altered layer until sputter equilibrium is attained. According to increasing accuracy and requirements in experimental procedures, three practical approaches to obtain z can be differentiated:

1.1 Sputter profiling of reference samples with known thickness using identical sputtering conditions: At present, tantalum pentoxide layers of 30 and 100 nm thickness are available as certified reference materials. After a calibration run, the depth scale is determined in equivalent tantalum pentoxide sputtering rate thicknesses.

1.2 Measurement of the ion sputtering crater depth after profiling by mechanical stylus, optical interference or atomic force microscopy methods to get the depth/time ratio.

1.3 In situ monitoring of the instantaneous crater depth: This can be done either directly e.g. by laser interferometry, or indirectly by determination of the sputtered matter e.g. by X-ray analysis, by microbalance or by quantitative SNMS.

Whereas methods 1.1 and 1.2 only give average values of z, the methods in 1.3 can disclose the instantaneous z as a function of the sputtered depth.

2. Quantification of the Elemental Signal Intensity, $X = f(I)$

Here it is necessary to differentiate between analysis of the sputtered matter as in SIMS, SNMS etc. and analysis of the remaining surface as in AES, XPS, ISS etc. The difference is mainly with respect to preferential sputtering. Quantification of both methods depends on surface topography, concentration changes within the altered layer, and on the information depth of the respective analysis method. Again, according to the allowed neglections, we can differentiate between several approaches:

2.1 Quantification using relative elememtal sensitivity factors: In its simple form this approach is often used in electron spectroscopies because matrix factors are relatively small and typical errors are of the order of 20% or less in constant composition regions.

2.2 Quantification including matrix effect corrections: In electron spectroscopies, matrix effects in electron escape depths and in primary electron backscattering in AES have to be considered to improve accuracy as well as their change in steep concentration gradients.

2.3 Quantification including sputtering induced effects: Topographical and compositional changes in the surface region are caused by sputtering and influence analysis during profiling. In particular, the most fundamental effects of surface roughness, atomic mixing and information depth have to be considered together to correctly evaluate profiling data. This refers directly to the next point.

3. Quantification of the Profile Shape: Depth Resolution Δz and Deconvolution

Basically, the considerations in points 1.3 and 2.3 lead to the concept of depth resolution as a quality figure of the precision of a profile. Both a simple and a more precise approach are generally performed:

3.1 Depth resolution Δz and error function approach: Broadening of the measured profile is expressed in terms of an error function description of a rectangular depth distribution. The definition $\Delta z = 2\,\sigma$ with one parameter only, the standard deviation σ, can be used in any case as a first order approximation to profile broadening and to deconvolution procedures.

3.2 Quantification by the resolution function: In general, profile shape changes are not symmetric as assumed in 3.1, and the combined influence of all parameters results in a resolution function $g(z'-z)$ which requires nmore than one parameter for its description. Knowledge of this function and its dependence on the sputtering time enables deconvolution of measured profiles by suitable mathematical methods. Easier to perform is the calculation or simulation of measured profiles by assumption of a true in depth distribution to which the appropriate resolution function is applied. Recent advances in modeling measured profiles of GaAs/AlAs superlattice structures using a resolution function based on the three parameters atomic mixing, surface roughness and information depth appear very promising with respect to the obtained precision in the atomic monolayer regime /2/.

The procedures 1-3 briefly described above only establish a framework of quantitative profiling which is somewhat modified by different authors. Numerous examples of various degrees of sophistication in the literature show that our knowledge on how to quantify profiles has much increased during the past twenty years but still needs further effort to fully exploit the ultimate high resolution analytical possibilities of depth profiling.

References:

/1/ S. Hofmann, Compositional Depth Profiling by Sputtering, Progr. in Surface Science 36 (1991) 35 - 87

/2/ S. Hofmann, Surf. Interface Anal. 21 (1994) 673