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Characterization of nanolayers by sputter depth profiling

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Abstract

Sputter depth profiling of nanostructures requires quantitative thin film analysis with high accuracy and with optimum resolution. More recently, powerful quantification models, such as the so-called mixing roughness information depth (MRI) model, have increased the accuracy of depth profiling to the sub-monolayer region. Using the MRI model, interdiffusion coefficients at nanolayer interfaces can be determined based on a mean diffusion path length of the order of 1 nm. After summarizing the progress to date, focus is on new developments with respect to experimental improvements such as ultra-low ion energy and glancing incidence, cluster ion sputtering, and improvements in theoretical modeling and quantification of nonlinearities with respect to the intensity scale and to the time scale of a depth profile, and the change of important quantification parameters such as electron escape depth in AES and XPS, mixing length and roughness during profiling, particularly when sputtering through interfaces. Today, the accuracy of quantification of sputter depth profiles of layered nanostructures is typically about 20% of a monolayer, or 0.06 nm on the elemental layer thickness scale.

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1. Introduction

The details of the property/composition/structure relationships of interfaces in layered nanostructures can only be understood if their elemental composition and that of their interfaces are characterized with high spatial resolution, ultimately in atomic monolayer dimensions. Among the various techniques developed for this purpose, surface and interface analysis methods in combination with ion sputtering [1,2] are most frequently applied because they are applicable to almost any kind of solid material and allow the attainment of optimum depth resolution in the atomic monolayer region over a wide depth range up to several micrometers. The in-depth distribution of elemental composition in thin films is conveniently obtained by sputter depth profiling based on surface erosion as a result of energetic particle bombardment. This fact has led to two main analytical branches, depending on the characterization of the removed matter (as in SIMS, SNMS, GDOES), or of the remaining surface (as in AES or XPS). These complimentary methods only show the same result if preferential sputtering of a component can be neglected [1]. Sputter depth profiling is a fast, reliable and universally applicable method to obtain informa-

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tion about the in-depth distribution of composition in thin films [1]. Application to nanolayers means to push the experimental approach to the physical limit of high depth resolution, and to quantify depth profiles with a correct theoretical model to obtain the optimum information about the layer structure and composition on the atomic monolayer scale. Both these aspects will be considered in more detail in the following.

2. Optimizing the experimental approach

The aim of any profiling experiment is to get a sputter depth profile (intensity versus sputtering time) which resembles as closely as possible the original elemental distribution with depth. While it is obvious that the instrumental setup requires correct adjustment of ion beam impact area and of the analysis spot as well as stable operation of the equipment with time, a number of additional conditions have to be considered in order to ensure minimum degradation of the measured profile. The basic conditions are compiled in Table 1. Optimization of sputter depth profiling mainly means selection of instrumental parameters in order to achieve a high depth resolution, Δz, that is a measure of profile broadening. Therefore, establishing optimized profiling conditions means minimizing all the effects which tend to increase Δz. The concept of depth resolution, as adopted by IUPAC [3] and the ASTM E 42 committee [4] (... when profiling an ideally sharp interface between two media ... the depth resolution corresponds to the distance over which a 16–84% (or 84–16%) change in signal is measured), has proved extremely useful to establish the dependence of the depth resolution on different parameters. For example, systematic studies of the dependence of the influence of the angle of incidence of the ions on Δz in a series of experiments on Ni/Cr multilayers [5,6] could finally be explained by the model of Pamler et al. [7], based on the influence of crystal orientation on the sputtering yield and the role of the [1 1 1] film texture to explain the maximum Δz for an ion incidence angle of about 35° from the normal to the sample plain. In practice, sample rotation during sputtering introduced by Zalar in 1985 [8] is the preferred remedy for the crystallite orientation effect and a valuable means, particularly for metallic samples, to obtain high resolution depth profiles.

Since the introduction of sample rotation during profiling, sputtering-induced roughening of more than a few nanometer and its increase with sputtered depth can be avoided. High resolution depth profiling with a depth resolution $\Delta z < 5$ nm is generally achieved, when low-energy ions ($\leq$1 keV Ar^+) are used. Ultra-high depth resolution

Table 1
Optimized profiling conditions

Sample ambient	Low residual reactive gas pressure ($<10^{-8}$ Pa) 'Free' sample mount to prevent redeposition.
Ion beam	Constant uniform intensity (scanning) Low beam energy (<1 keV) High mass ion species (or reactive species) Large incidence angle for smooth sample (>60°) Low incidence angle for rough sample (<60°) Sample rotation
Analyzing conditions	Analyzed area centered in and small against sputtered area Minimized information depth (escape depth in AES, XPS)
Sample characteristics	Smooth, polished surface Non-crystalline, without second phases Components with similar sputtering yield Negligible diffusion and segregation Good electrical and thermal conductivities