Determination of the Depth Scale in Sputter Depth Profiling (The Sputtered Depth in Depth Profiling)

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One of the fundamental parameters in quantification of sputter depth profiles is the sputtered depth. Usually, the sputtered depth is not known from the measured profile, because only the sputtering time is recorded. Only if the instantaneous sputtering rate (= sputtered depth per unit of time) is known, the depth scale can be derived from the time scale. To determine the average sputtering rate, a certain sputtered depth has to be correlated with a measured sputtering time. In general, the sputtered depth has to be determined by additional measurements that is the topic of a recently published ISO Technical Report (ISO/TR15969). In this work, we focus on the correction of the time scale in terms of a true depth scale in case of preferential sputtering of one component of a binary system with concentration variation, as shown for the example of a Si/Ta multilayer. Using a modified version of the MRI model, it is shown how the correct the time/depth relation for quantification of sputter depth profiles is obtained.

Key words: Depth Profiling, preferential sputtering, Si/Ta multilayer, MRI model.

1. Introduction

The aim of any depth profile analysis is the retrieval of the original in depth distribution of composition. The measured depth profile usually consists of an elemental signal intensity as a function of the sputtering time. Conversion of these raw data to the original in depth distribution of composition, as usually performed by deconvolution or profile reconstruction, includes the following three tasks:

(1) Calibration of the intensity scale (e.g. in counts per second) in units of instantaneous concentration (e.g. atom-%);

(2) Calibration of the sputtering time scale in units of depth (i.e. distance from the original surface;

(3) Estimation of the depth resolution and/or determination of the resolution function.

Calibration of the intensity scale covers the general topic of quantitative surface analysis which is treated in special publications [1]. Specific to depth profiling are the effects of sputtering induced changes of surface topography and composition. The fundamental effect of atomic mixing causes a deviation between the instantaneous surface composition and the original sample composition, that can be taken into account and is the basis for quantification of the concentration scale in depth profiling.

The second fundamental task is calibration of the sputtering time (t) in terms of sputtered depth (z). The similarity of the profile to the original in depth distribution of composition is determined by the depth resolution. Knowledge of the amount of profile broadening enables the establishment of the depth resolution function.

Performance of the tasks (1), (2), (3) is a necessary condition for the reconstruction of the original depth distribution from the measured profiling data. Recently, some simple mathematical models were developed which are capable to quantify depth profiling data [2 - 5]. Among them, the so called MRI (mixing-roughness-information-depth)-model, based on three physically well defined parameters, can take into account even nonlinearities in the sputtering time/depth or the intensity/concentration relationships, and is therefore well suited for quantitative depth profile evaluation [3,6,7].

2. Determination of the correct depth scale

2.1 According to ISO/TR 15969:2001: Surface Chemical Analysis - Depth Profiling - Measurement of Sputtered Depth

Here, our focus is on the depth scale and their quantitative determination in sputter depth profiling. Calibration of the sputtering time (t) in terms of sputtered depth (z) requires knowledge of the instantaneous sputtering rate $\dot{z}$= dz/dt. For constant sputtering rate, the sputtered depth is proportional to the sputtering time, i.e. z (t) = (dz/dt) * t. An estimation of the average ¿ is possible by measurement of the depth of the crater generated by sputtering after a certain sputtering time. An expert committee in the ISO TC 201 (SC4:Depth Profiling) elaborated a technical report on the topic: Surface Chemical Analysis—Depth Profiling –Measurement of Sputtered Depth [8].

The methods described in ISO/TR 15969:2001 are divided in those measuring the crater depth after depth profiling (by mechanical stylus or optical interferometry) and in those comparing a depth profile of a reference sample with interfaces as depth markers.

The crater depth is defined as the average distance (perpendicular to the surface) between the original surface and the region of a crater bottom from which the measured signal is derived. An exact equality of the sputtered depth with the crater depth is only valid under the following conditions: first, primary ion implantation and retention should be negligible. These effects cause an enlargement ("swelling") of the sample in the direction perpendicular to the surface. Whereas for noble gas ion sputtering, primary ion retention is usually of the order of a few atomic percent, for sputter profiling with reactive gases it may change the whole matrix. For example, oxygen primary ions in SIMS profiling of a silicon matrix have been shown to cause formation of a silicon dioxide layer of the thickness of the ion range [9]. This means a swelling of the matrix (and therefore a reduction of the crater depth of some nanometers, if sputtering is performed by primary ions of some keV energy. If the sputtered depth is measured by crater depth measurement outside the analysis chamber, surface reactions (e.g. oxidation) may add to the swelling of the crater bottom, i.e. the crater depth is generally measured as being less than the sputtered depth by typically 2-5 nm [8]. Usually, this value is within the standard deviation of both mechanical stylus and optical interference methods for the determination of the sputtered depth.

A known depth of an interface or the depths of several interfaces can be used to determine the sputtered depth by comparison with the location of the 50 % drop of the plateau value on the sputtering time scale in the sputter profile. Errors involved are (a) the initial change of the sputtering rate, and the usual surface contamination layer, leading to typical errors of the order of 1-5 nm), and (b) a systematic shift of the 50 % plateau intensity to apparently lower depth as compared to the correct interface location [5]. This error is of the order of the signal escape depth (electron (AES, XPS) or ion escape depth (SIMS)) or the atomic mixing length, depending on the larger value. Under typical profiling conditions, the shift is of the order of 1-5 nm. Under favorable conditions, (a) and (b) may compensate and a linear relation between sputtering time and depth without a zero point shift is obtained, as seen in the depth profiling results of Seah et al. [10] for Ta_2O_5/Ta reference samples. Under less favorable conditions and by using the onset of the profile decay as sputtering time marker, Tsong et al. [11] found a positive offset of the zero value on the depth axis when plotting the thickness of SiO_2/Si layers versus the time to the interface.

In multilayer profiling, the above effects (a) and (b) are similar at every interface and therefore they are often negligible when using the interfaces as depth markers.

2.2 Influence of Preferential Sputtering

Due to the dependence of the sputtering rate on the instantaneous surface composition a non-linear time/depth relation may result. In this case, only in situ measurements of the receding surface can provide the correct answer, which are however complicated and generally not available. A first-order correction of the time/depth relation can be made by assumption of a linear dependence of the sputtering rate on the surface composition, as demonstrated for depth profiling results of a Ni/Cr multilayer sample with nitrogen ions [12]. This correction can be considerably improved by application of the Mixing-Roughness-Information depth (MRI)-model in its modified form [13,14].

If the different sputtering rates of the components A, B are $_A$ and ¿$_B$, using the approximation:

$$¿ = ¿_{A}X_{A} + ¿_{B}X_{B} \qquad (1)$$

With the sputtering rate ratio r = ¿$_A$/¿$_B$, the sputtering time scale normalized to the sputtering rate of the pure component B, t_B, is

$$t_{B} = \int_{0}^{z} \frac{1}{\&_{B}\,[X_{A}\,(r-1)+1]}\,dz. \qquad (2)$$

Introducing equation (2) in the MRI formalism yields the sputtering time scale, i.e. the measured profile if the normalized (or quantified) intensity is plotted against the sputtering time. Of course, the latter has also to be modified as shown in [13].

3. A Practical Example: Ta/Si multilayer

As an example, Fig.1 shows the measured depth profile of a Ta/Si multilayer (10 double-layers), consisting of alternating 10.5 nm thick Si layers and 7.5 nm thick Ta layers, depth profiled with 3 keV Ar$^+$ ions at 81 deg incidence angle [13]. The peculiar shape was the reason that a first application of the above correction was applied in the early stage of the MRI model for one layer [13]. Now we have refined the preferential sputtering approximation in the model and apply it to quantitatively evaluate the whole measured multilayer profile.

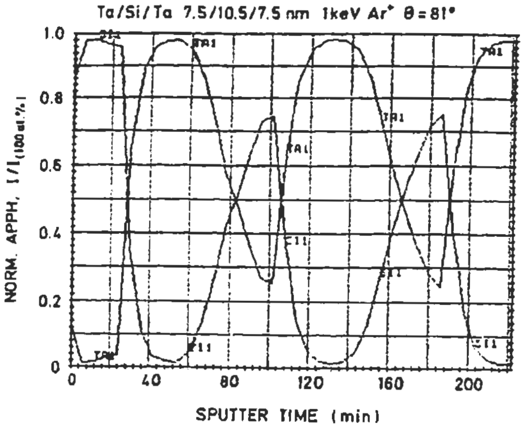

Fig. 1: AES depth profile (3 keV Ar$^+$, 81 deg. inc. angle) of a Ta(10.5nm)/Si(7.5nm) multilayer: Normalized Si (92 eV) and Ta (179 eV) APPH versus the sputtering time.

Fig 2 shows some of the measured points taken from Fig. 1 and plotted together with the MRI calculation of the profile using the MRI parameters: mixing length w= 2.6 nm (from measurements), roughness σ = 1.1 nm, information depth λ = 0.4 nm [13], and sputtering rate ratio r(Si/Ta) = 3.5 for the optimum fit. Note that only σ and r were adjustable parameters.

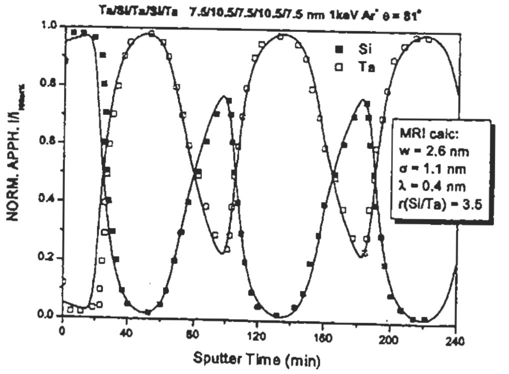

Fig. 2: Comparison of measured profiles (points from Fig.1) with the calculated MRI profile (Parameters see inset and text).

The sputtering rate ratio of 3.5 is in rather good agreement with the ratio of 4.3 for 0.5 and of 2.9 for 3 keV Ar^+ ions found by Hofmann and Stepanova [15].

Fig. 3 shows the plot of the sputtered depth scale versus the sputtering time. The dashed line corresponds to the average sputtering rate.

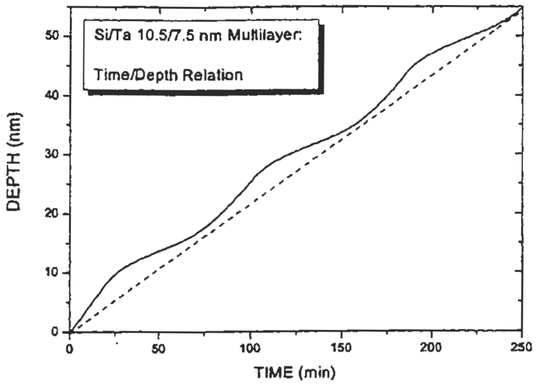

Fig. 3: Depth/Time relation for Fig. 2.

Fig 4 shows the "rectified" plot of the profile in depth scale coordinates, shown together with the in depth distribution of composition.

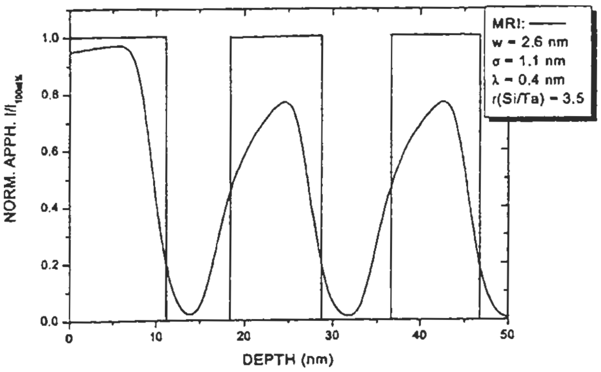

Fig. 4: Depth profile in linear depth coordinates (Fig. 2 recalculated with the instantaneous sputtering rate from Fig. 3).

4. Conclusion

The exact determination of the depth scale in sputter depth profiling is not easy even in case of a constant erosion rate throughout the profile. In general, however, particularly in multilayer profiling, we have different sputtering rated of the components. In that case, the usual methods as described in ISO TCR 15969: 2001 only give an average sputtering rate, which does not represent the nonlinear relation between sputtering time and sputtered depth. Using a modified version of the MRI model, it is shown, for the example of a Ta/Si multilayer, that by quantification of the measured profile, i. e. by reconstruction of the in depth distribution of composition, with an additional parameter, the sputtering rate ratio r(Si/Ta), the sputtering time/depth scale relation can be determined and the depth profile in "true" depth coordinates is disclosed.

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