A quantitative evaluation of the depth resolution of AES depth profiling of Cu/Ni multilayer thin films using the MRI model.

X.L. Yan [1], Y. Liu [2], H.C. Swart [1], J.Y. Wang [2], J.J. Terblans [1]*
[1] Department of Physics, University of the Free State, PO Box 339, Bloemfontein, 9300, South Africa
[2] Department of Physics, Shantou University, 243 Daxue Road, Shantou, 515063 Guangdong, China

E-mail: terblansjj@ufs.ac.za

Abstract: Depth profiles of as-deposited Cu/Ni multilayer thin films were investigated using Auger electron spectroscopy (AES) in combination with Ar^+ ion sputtering. The Cu/Ni multilayer structures were deposited on a SiO_2 substrate by means of electron beam evaporation in a high vacuum. The measured AES depth profiles of the as-deposited Cu/Ni multilayer was quantitatively fitted by the MRI model assuming that the roughness parameter has linearly increased with the sputtered depth. The roughness values extracted from the depth profiling data fits, agreed well with those measured by atomic force microscopy (AFM). The depth-dependent depth resolution upon depth profiling of the Cu/Ni polycrystalline multilayer was quantitatively evaluated accordingly.

1. Introduction

Auger electron spectroscopy (AES) in combination with ion beam sputtering is widely used for the determination of the composition-depth profiles of thin films. The quality of depth profiling can be characterized by the so-called depth resolution Δz, which defines the depth range to which a certain composition has to be assigned (see section 3) [1]. The measured depth profile is always influenced by factors such as atomic mixing, sputter-induced surface roughness, information depth, preferential sputtering, segregation, etc. [2]. Often the measured depth profile as compared to the true concentration-depth profile is described by a so-called depth resolution function (DRF), which can be experimentally determined or theoretically estimated. The Mixing-Roughness-Information depth (MRI) model is a theoretical description of the DRF taking into account atom mixing, sputter-induced surface roughness and information depth. The model developed by Hofmann was first given in reference [3]. Since then, numerous extensions were developed to enable its applicability for preferential sputtering [4], non-stationary mixing, information depth as well as for non-Gaussian roughness [5,6], and the analytical depth resolution function for thin delta layer [7]. Profile reconstruction from measured depth profile data can be obtained by the MRI model, where the original in-depth distribution can be obtained by a least square approach optimizing the fit of the calculated profile to the measured data.

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* To whom any correspondence should be addressed.

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The aim of this study is to test the MRI model on a Cu/Ni polycrystalline multilayer structure from measured AES depth profiling data with assuming a linear roughness with sputter depth to fit. The fitting roughness values are compared with those measured by atomic force microscopy (AFM). The depth-dependent depth resolution upon depth profiling of the Cu/Ni polycrystalline multilayer are quantitatively evaluated accordingly.

2. Experimental

The Ni/Cu multilayer structures composed of four pairs of Ni and Cu layers were prepared by electron beam physical vapour deposition onto passivated silicon (100) substrates (SiO_2) at a base pressure of $<7\times10^{-6}$ Torr, resulting in polycrystalline multilayer thin films. The layer thickness was monitored by an Inficom XTC thin film monitor as Cu(8 nm)/Ni(8 nm)/Cu(11 nm)/Ni(11 nm)/Cu(15 nm)/Ni(12 nm)/Cu(13 nm)/Ni(14 nm)/SiO_2. The topographies measured by AFM with scanning area 3×3 μm^2 are shown in Fig.1(a) for the surface of the as-deposited sample and in Fig.1 (b) for the crater centre for sample sputtered at the depth of 100 nm (substrate SiO_2 surface), respectively. The corresponding root mean square (RMS) roughness values were determined as 0.91 nm and 5.54 nm, respectively.

The AES depth profiles were measured using a PHI 600 SAM without sample rotation at a base pressure < 10^{-9} Torr. A static primary electron beam of 10 keV and beam current of 200 nA with beam size diameter of 9.2 μm was used. Ion sputtered was performed with 2 keV Ar^+ ions at an incidence angle of 60° to the normal of the sample surface, the beam current density was 0.127 A/m^2 with a raster area 2×2 mm^2. The Auger peak-to-peak heights (APPHs) were recorded as a function of sputtering time for Ni(680-740 eV), Cu(880-989 eV), C(240-295 eV), O(480-510 eV) and Si(70-105 eV). In order to apply the MRI model, the APPH-sputter time profile was converted into normalised APPH depth profiles. The Cu APPH was normalized to the maximum of the Cu 922 eV APPH in the as-prepared sample. The time scale was converted into the depth scale using the average sputtering rate of 0.05nm/s determined by depth profiling data with the known thicknesses of the Cu/Ni layers.

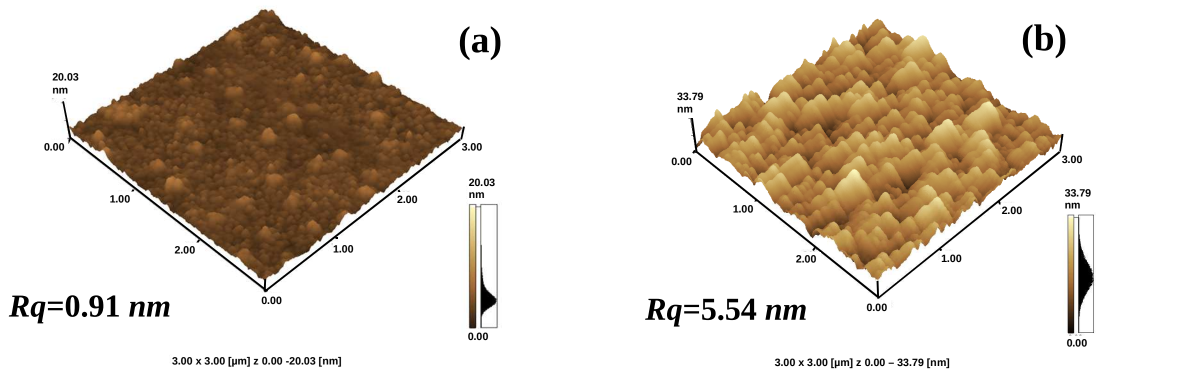

Figure 1. AFM images of (a) the surface of the as-deposited sample and (b) the crater centre for sample sputtered at the depth of 100 nm (substrate SiO_2 surface). The corresponding root mean square (RMS) *Rq* roughness values were determined as 0.91 and 5.54 nm, respectively.

3. Depth resolution and MRI model

The depth resolution Δz is defined conventionally as the depth range over which a 16-84% (or 84-16%) change in signal by a specified amount when profiling an ideally sharp interface between two media (see Fig.2) [8]. This definition has a physical meaning only for a Gaussian shape of the depth resolution function in Fig. 2. However, if the concentration saturation levels of 100% and 0% for the signal analyzed no longer occur, for example, in case of sputter depth profiling of a thin multi-layered film, the determination of Δz defined above is no longer possible. Using the MRI model for the calculation of sputter depth profiles, it is shown that, for the same Δz, different resolution functions can be obtained with different full width at half-maximum (FWHM) values [9].

The depth resolution function in the MRI model takes into account three effects: atom mixing, escapes depth of Auger electrons, surface roughness and is described as [3]:

Mixing length (w): $g_w = \frac{1}{w}\exp\left[-\left(z - z_0 + w\right)/w\right]$ (1);

Roughness (σ): $g_\sigma = \frac{1}{\sqrt{2\pi}\sigma}\exp\left[-(z - z_0)^2/2\sigma^2\right]$ (2);

Information depth (λ): $g_\lambda = \frac{1}{\lambda}\exp\left[-\left(z_0 - z\right)/\lambda\right]$ (3).

where w is atomic mixing length, z is sputter depth, z_0 is the running depth parameter, σ is the surface roughness and λ is the information depth parameter.

Based on the above-discussed refinements of the DRF in terms of symmetric (Gaussian functions) and asymmetric (non-Gaussian functions), it is necessary to clarify the contribution to the depth resolution Δz 16-84%. According to this MRI model, a symmetric contribution to the depth resolution function originates from the intrinsic roughness and the surface roughening by ion sputtering, which both are described by a Gaussian smearing function (see Eq.(2)), characterized by its standard deviation: the surface roughness parameter σ ($\Delta z_\sigma^2 = 2\sigma^2 = 2\sigma_i^2 + 2\sigma_s^2$, where σ_i is the intrinsic roughness, σ_s is the sputter induced roughness.). For the asymmetric broadening functions, the atomic mixing is described by an exponential function (see Eq.(1)) and this exponential function is characterized by the atomic mixing parameter w (Δz_w=1.668w); the information depth of the Auger electrons (for AES) is also described by an exponential function (see Eq.(3)) and this exponential function is characterized by the information depth parameter λ (Δz_λ=1.668λ). Thus, three parameters (σ, w and λ) suffice to characterize the total smearing, Δz (see Ref.[1]). Values for these three parameters may be obtained experimentally and/or calculated theoretically (see the section 4).

According to the above-discussion in the MRI model, the total "symmetric" and "asymmetric" contribution to the depth resolution, Δz, can approximately be written as [10]:

$$\Delta z = \left(\left(2\sigma\right)^2 + \left(1.668w\right)^2 + (1.668\lambda)^2\right)^{1/2} \quad (4).$$

Fitting the MRI model to experimental sputtering-depth profiles in principle leads to values for σ, w and λ in Eqs. (1-3). Then Δz can be calculated using Eq.(4).

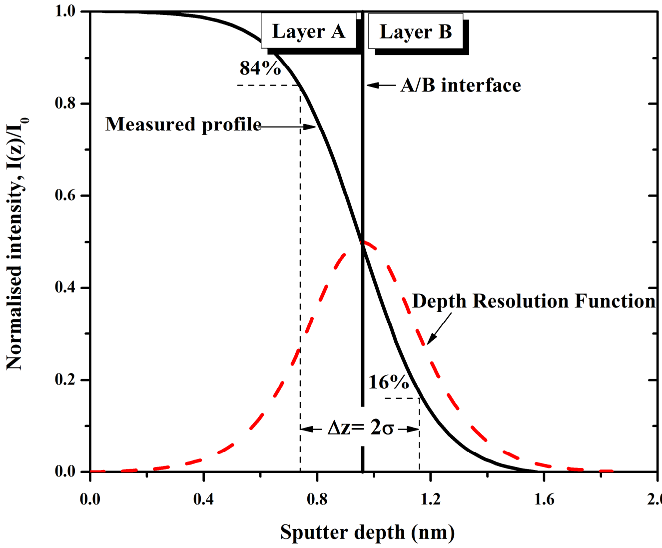

Figure 2. Schematic definition of the depth resolution, Δ*z* (84-16%), at a sharp interface in the broadening profile and a Gaussian resolution function (dashed line).

4. Results and discussion

The measured Cu depth profiling data of the as-deposited Cu/Ni multilayer is shown in Fig. 3 as open circles. A close examination of Fig. 3 revealed that the measured maximum/minimum value of each sublayer decreased/increased with the sputtered depth. This effect is related to the development of the ion bombardment induced roughness for polycrystalline material upon stationary depth profiling because of the dependence of ion sputtering yield on the orientation of the crystal. The measured Cu depth profiling data for the as-deposited sample was fitted and shown as a solid line in Fig. 3 by the MRI model assuming simply that the roughness parameter increased linearly with the sputtered depth and taking the other two MRI parameters into account. The atomic mixing length *w* was estimated as 1.0 nm by the TRIM code [11] and the Auger electron escape depth λ (effective attenuation length times cos(θ), where θ is the angle of emission of the detected electrons) for Cu (922 eV) was calculated as 0.8 nm [12]. The fitted roughness parameter was obtained as $\sigma = (0.9+0.049\times z)$ nm, which agrees very well with the measured RMS *Rq* values of 0.91 nm and 5.54 nm by AFM at the depth of $z = 0$ nm and $z = 100$ nm as indicated in Fig.1(a) and (b) , respectively.

Using the aforementioned fitted parameters in the MRI model, the depth resolution Δ*z*, as given by Eq. (4), for the as-deposited sample have been calculated as a function of the sputter depth in Fig.4 and is shown as a solid line. The fitted roughness σ for the as-deposited sample agrees very well with the *Rq* roughness values measured by AFM at the depth of $z = 0$ nm and $z = 100$ nm as closed squares denoted in Fig.4 and its corresponding twice value 2σ (contribution in depth resolution, see the first term of right side in Eq. (4)) is presented in Fig.4 as dashed lines.

As is shown in Fig.4, when the sputter depth increased, the values of depth resolution Δ*z* was closer to the values of roughness 2σ.This result implies that the roughness contribution is the dominant factor degradation the depth resolution at the deeper sputtered depth. We should also note that the MRI fitting roughness parameter σ=5.0 nm at the depth *z*=100 nm is larger than the other two parameters (*w*=1.0 nm and λ=0.8 nm).

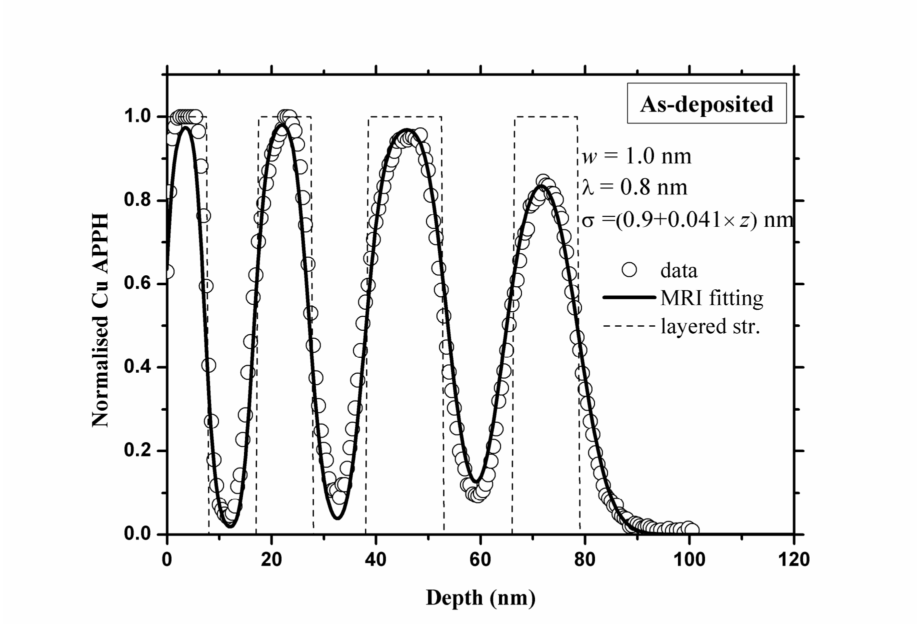

Figure 3. The normalised Cu depth profiling data and the best fit to the measured data by the MRI model for the as-deposited sample. The dashed line drawn represents the as-deposited Cu sublayer structure.

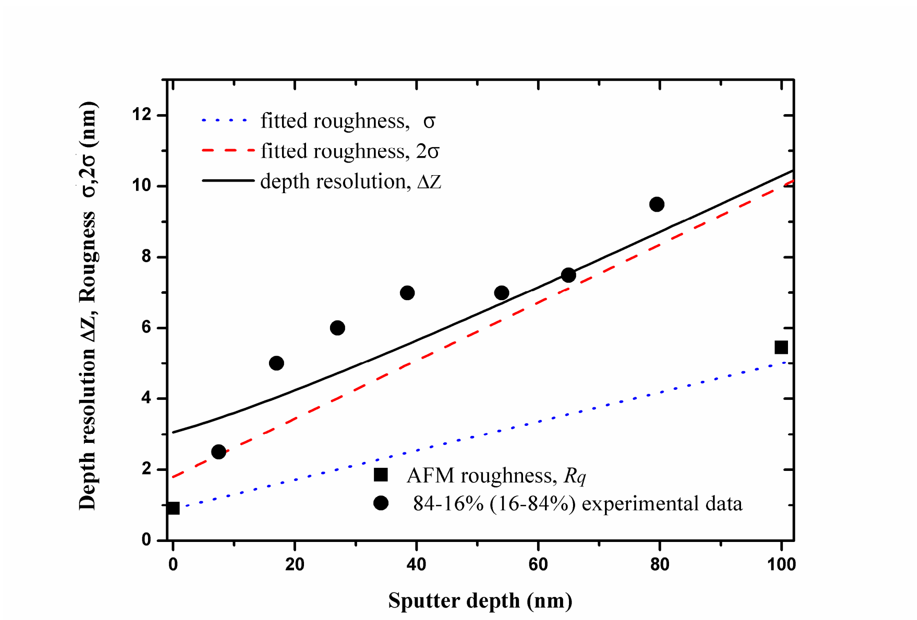

Figure 4. The depth resolution Δz (solid line) and the fitted roughness parameter σ and its double value 2σ (dotted and dashed lines, respectively) as a function of the sputter depth for the as-deposited sample. The closed circles and squares represent the values of the depth resolution by 84-16%(16-84%) method and the *Rq* roughness values determined by AFM (see Fig.1), respectively.

In additional, the values of the depth resolution in the MRI calculation are in accordance satisfactorily with the ones in the experimental data by the 84-16% (16-84%) method denoted as closed circles in Fig.4. This confirms further the capability of the MRI model with the linear roughness in the Cu/Ni polycrystalline multilayer structure. The calculated Δz/2 value of 4.1 nm at the last Cu/Ni interface (corresponding to a depth of 78 nm) for the as-deposited sample is less than the thickness of the last sublayer Cu/Ni under the present measurement conditions.

5. Conclusions

The application and capability of the MRI model to evaluate different contributions to the depth resolution were demonstrated for the Cu/Ni polycrystalline multilayer structures by fitting the measured AES depth profiling data. The roughness parameter was assumed linearly increased with the sputtered depth. And the roughness values extracted from the MRI model fitting agreed well with those measured by AFM. The depth-dependent depth resolution upon depth profiling was quantitatively evaluated accordingly.

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