Determination of in-depth density profiles of multilayer structures

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We developed and demonstrate an analysis method in which we calibrate the intensity scale of cross-sectional transmission electron microscopy (TEM) using Cu K_α reflectometry. This results in quantitative in-depth density profiles of multilayer structures. Only three free parameters are needed to obtain the calibrated profiles, corresponding to three TEM image intensity levels. Additionally, the optical indices of the two multilayer materials used and the assumption that the layers are laterally homogeneous are used in the model. The power and the general usefulness of the method is demonstrated using experimental data of W/Si and Mo/Si multilayer systems with sharp interfaces as well as multilayers of which the interfaces were deliberately intermixed. © 2005 American Institute of Physics. [DOI: 10.1063/1.1882773]

INTRODUCTION

Multilayer structures are used in a variety of fields, e.g., extreme ultraviolet lithography,¹ x-ray fluorescence analysis,² and vertical cavity surface emitting lasers.³ In view of the optical application of such structures, especially the density profile, including information on layer intermixing, interface roughness and the distribution of the components is crucial. To be able to properly investigate these structures, extensive analysis of the produced structures should be performed. Available analysis tools based on depth profiling using energetic ions, like Auger electron spectroscopy, x-ray photoelectron spectroscopy, as well as secondary ion mass spectroscopy, may induce changes of the original profile. This can be due to intermixing or preferential sputtering.

Microscopic techniques like bright field cross-sectional transmission electron microscopy (CS-TEM) are less destructive, but produce an image that is difficult to translate into a quantitative profile. On the other hand, nondestructive techniques based on reflectivity measurements like ellipsometry and x-ray reflectometry, suffer from the so-called inversion problem or phase problem: it is impossible to unambiguously determine the structure from the measured spectrum.^{4,5} As a solution, these data usually are subsequently processed by fitting an empirically determined model to the measured reflectivity spectrum. The free parameters to be fitted are for example individual layer thicknesses, material concentrations, interface structures, and interlayer thickness. However, the use of such a large amount of free parameters and the implicit information contained in the model reduces the reliability of the fit, even when a perfect match between experiment and fit is found. This is due to the fact that in practice no single model is able to comprehensively

describe all physical phenomena, and that different parameters in the model can have a similar effect on the reflectivity spectrum.

Aschentrup *et al.*⁶ demonstrated a method in which they reduced the number of free parameters by determining the layer thicknesses from CS-TEM images. Although this improves the reliability of the result, it still heavily depends on the implicit information contained in the model used to describe the expected structure. In the method we propose here, we will determine the in-depth density profile without using such a model. For this, we obtain an in-depth density profile from a CS-TEM image. This density scale is then calibrated by fitting a simulated reflectivity spectrum determined from the TEM profile to a measured spectrum. Essential data required for this scale are average density (obtainable from the measured critical angle), and optical contrast between the two materials (obtainable from the measured height and shape of the first order Bragg peak after the average density has been determined). Though this not necessarily results in a perfect fit, the resulting in-depth density profile is considerably more reliable than the alternatives described above.

As a result, more complete information, also including layers with different densities and their position is obtained. Since this method does not use any other, implicit data than the CS-TEM image, the actual reflectometry data, the refractive indices of the two, pure, materials in the multilayer structure and the assumption that the layers are laterally homogeneous, the outcome of the method represents a unique description of the structure.

Several merit functions have been tested to find the optimal fit, and both a logarithmic and a linear fit were used. Additionally we added the option to include a weighting of the fit for instrumental errors. Although the fits were affected

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by the choice of the merit function, the results of the total analysis did not change significantly. This demonstrates the stability of this method.

The power of this method as well as the general applicability of the analysis is demonstrated for several deposited multilayers having different interface structures.

METHOD

CS-TEM images are obtained from the transmission of electrons through a structure of typical 20–100 nm thickness. Because of the symmetry in the deposition setup, we can assume that the structure is homogeneous in the direction of the electron beam. For a high reflective coating, the multilayer has to be composed of materials with a large difference in refractive index. In general this means that one material has a high atomic number Z , whereas the other material has a low Z . According to the scattering contrast theory for amorphous specimens in bright field mode as formulated by Reimer,⁷ we can describe the transmission of electrons T as an exponentially decaying function depending on the product of density ρ and thickness t :

$$\ln(T) = -\beta \frac{Z^\alpha}{A} \rho t, \quad (1)$$

where α and β are aperture- and energy-dependent constants and A is the atomic mass. The transmission through a mixture of a high- Z material and a low- Z material in a depth-dependent ratio of $C_{\text{high}}(z)$: $1 - C_{\text{high}}(z)$, where z is in the direction normal to the surface of the multilayer sample, can thus be described as:

$$\begin{aligned} \ln(T(z)) = & -\beta \frac{(Z_{\text{high}})^\alpha}{A_{\text{high}}} \rho_{\text{high}} t C_{\text{high}}(z) \\ & -\beta \frac{(Z_{\text{low}})^\alpha}{A_{\text{low}}} \rho_{\text{low}} t (1 - C_{\text{high}}(z)). \end{aligned} \quad (2)$$

This can be rewritten as:

$$T(z) = p e^{-k C_{\text{high}}(z)}, \quad (3a)$$

where

$$p = e^{-\beta \frac{(Z_{\text{low}})^\alpha}{A_{\text{low}}} \rho_{\text{low}} t}, \quad (3b)$$

and

$$k = \beta \frac{(Z_{\text{high}})^\alpha}{A_{\text{high}}} \rho_{\text{high}} t - \beta \frac{(Z_{\text{low}})^\alpha}{A_{\text{low}}} \rho_{\text{low}} t. \quad (3c)$$

When we assume that the imaging system (e.g., film, scanner, camera, etc.) has been operated within a linear range, the measured intensity I depends on the transmission T according to

$$I(z) = aT(z) + b, \quad (4)$$

where a is related to the optical quality of the imaging system (e.g., brightness or contrast) and b corresponds to the measured background intensity for which the beam is completely blocked. Using (3a) this results in a dependence of the measured intensity to the concentration of the high- Z material according to

$$C_{\text{High}}(z) = -\frac{\ln\left(\frac{I(z) - b}{ap}\right)}{k}. \quad (5)$$

In case we have areas in the TEM image that correspond to nonintermixed zones we can determine from the TEM image an intensity I_{max} , which corresponds to 0% of high- Z material as well as an intensity I_{min} which corresponds to 100% of the high- Z material, the product of a and p can be replaced by $I_{\text{max}} - b$ and k by $-\ln((I_{\text{min}} - b)/(I_{\text{max}} - b))$, which results into:

$$C_{\text{High}}(z) = \frac{\ln\left(\frac{I(z) - b}{I_{\text{max}} - b}\right)}{\ln\left(\frac{I_{\text{min}} - b}{I_{\text{max}} - b}\right)}. \quad (6)$$

Equation (4) contains, in principle, only *one* free parameter (b), which is to be determined by a fit to $\theta/2\theta$ reflectometry data.

In structures that do not contain layers of 100% of high- Z material or layers containing 0% of high- Z material, the intensities I_{min} or I_{max} cannot be determined directly from the TEM image and have therefore to be introduced as additional free parameters. Even when the structures do contain layers with 100% or 0% of high- Z material, the intensity can be higher than I_{max} or lower than I_{min} , due to noise in the acquired TEM image. This also necessitates introducing these two boundaries as additional free parameters. Intensities that are above I_{max} or below I_{min} are then put equal to the respective boundary.

Thus, these three parameters enable us to convert the depth-dependent intensity of the CS-TEM image into a depth-dependent density of the two materials in the structure. This can be used to calculate the angle dependent reflectivity $R_{\text{calculated}}(\theta)$, e.g. with the matrix method, which results in the reflectivity using the propagation of the electric field in the structure.^{5,8} Using a fitting algorithm we can change the free parameters in such a way to minimize one of the merit functions M as described by

$$M_{\text{linear}} = \int_{\theta_{\text{min}}}^{\theta_{\text{max}}} (R_{\text{measured}}(\theta) - R_{\text{calculated}}(\theta))^2 d\theta, \quad (7a)$$

$$\begin{aligned} M_{\text{weighted linear}} = & \int_{\theta_{\text{min}}}^{\theta_{\text{max}}} ((R_{\text{measured}}(\theta) - R_{\text{calculated}}(\theta)) \\ & \times R_{\text{measured}}(\theta))^2 d\theta, \end{aligned} \quad (7b)$$

$$\begin{aligned} M_{\text{logarithmic}} = & \int_{\theta_{\text{min}}}^{\theta_{\text{max}}} (\log(R_{\text{measured}}(\theta)) \\ & - \log(R_{\text{calculated}}(\theta)))^2 d\theta, \end{aligned} \quad (7c)$$

$$\begin{aligned} M_{\text{weighted logarithmic}} = & \int_{\theta_{\text{min}}}^{\theta_{\text{max}}} ((\log(R_{\text{measured}}(\theta)) \\ & - \log(R_{\text{calculated}}(\theta))) \\ & \times R_{\text{measured}}(\theta))^2 d\theta, \end{aligned} \quad (7d)$$

where $R_{\text{measured}}(\theta)$ is the measured reflectivity curve, θ_{min} is equal to the lowest measurable (grazing) angle, and θ_{max} is the largest angle in the measured range still at a reasonable signal level.

As a fitting algorithm we used CFSQP (C code for Feasible Sequential Quadratic Programming), developed by Craig Lawrence, Jian L. Zhou, and André L. Tits,^{9,10} which is a set of C functions for the minimization of the maximum of a set of smooth objective functions.

REFLECTIVITY MEASUREMENT

We now proceed to determine the minimal required angular range in the Cu K reflectivity measurements. Basically, a reflectometry spectrum $R(q_z)$ is in first order approximation equal to the Fourier transformation of the depth dependent electron density $\rho(z)$ combined with the Fresnel reflectivity $R_F(q_z)$ of a substrate according to:⁴

$$R(q_z) = R_F(q_z) \left| \frac{1}{\rho(\infty)} \int \frac{d\rho(z)}{dz} e^{iq_z z} dz \right|^2, \quad (8)$$

where the wave factor q_z is defined as

$$q_z = 2 \frac{2\pi}{\lambda} \sin \alpha_i, \quad (9)$$

with α_i equalling the grazing angle of incidence and λ the wavelength of the incident radiation. From (8), we can see that low frequency changes in the electron density, as observed in the CS-TEM material density profile, will appear at small grazing angles (low q_z) in the reflectometry measurements. High frequency changes will be seen at larger grazing angles.

We recall that the missing parameters to determine the intensity-to-density scale of the TEM profile are I_{max} , I_{min} , and b which only influence the average density and the maximal optical contrast between the two materials. The average density δ is a constant value, and will thus influence the reflectivity only at very grazing angles. This is also expressed in the approximation for the critical angle θ_c :¹

$$\sin \theta_c = \sqrt{2\delta}. \quad (10)$$

For periodic multilayer structures, the amplitude of the optical contrast between the two materials should be contained within the height and shape of the first order Bragg peak. Therefore, a fit of a reflectivity measurement including the critical angle and the first order Bragg peak is sufficient to acquire the desired calibration of the TEM data. To be able to include layer thickness variations, which cause a deviation of the periodicity of the structure and thus influences reflectivity measurement primarily at larger angles, one should include higher orders in the fitting routine. However, since the layer thickness variations are detectable in the CS-TEM picture, they are in principle already taking into account.

It is noted that an extension of the q -space analysis to much larger q -values then up to the first or second Bragg peaks would not necessarily increase the accuracy of the method. This is due to the fact that TEM basically represents

a 2D projection of a sample with finite thickness. High resolution features, corresponding to higher orders in the Cu K data, are therefore usually not visible. The minimally measured angular range in the reflectivity spectrum should thus at least include the critical angle and the first order Bragg peak. Inclusion of higher angles may add to the accuracy, but most emphasis should be put on the more grazing angles for our TEM intensity scaling purpose.

EXPERIMENT

We prepared all multilayers used for demonstration of the method by e-beam evaporation in an ultrahigh vacuum system (base pressure 10^{-9} mbar). The thickness of the components was controlled by in situ reflection of C K radiation having an angle with the sample surface of 35° . This technique also provides information on the roughness development of the sequential interfaces. The samples consisted of 9, 10, or 50 periods. The material combinations W/Si and Mo/Si were used. W/Si is known to form graded interfaces under ion bombardment,¹¹ whereas Mo/Si is known to form silicides¹² under ion bombardment. For this reason we investigated samples with and without Kr^+ ion bombardment of the high- Z material. Additionally, energetic Kr^+ ions were applied to all Si layers to reduce the interface roughness between the components. The samples were characterized by $\theta/2\theta$ reflectometry at Cu K_α wavelength (0.154 nm) using a Philips X'pert reflectometer. TEM cross-sectional specimen were prepared by sawing and mechanical polishing to $\sim 20 \mu\text{m}$ thickness, followed by ion milling (Gatan PIPS model 691). Putting the specimen in the edge-on position in the TEM was done with the help of the substrate orientation. The photographs were digitized at 1500 dpi (Nikon super coolsan 8000 ED film scanner). Intensity profiles obtained from line scans perpendicular to the multilayer structure were averaged over a length of 10 nm parallel to the multilayer structure.

Although the thickness T of the multilayer stack can be determined from the cross-sectional TEM images, a small optimization (within 1%) allowed us to obtain a much higher precision than is possible using data from the TEM image only (1%). This is a fourth free parameter T in the total model. In principle one can obtain this value also by analysis of position of the higher order Bragg peaks.¹ However, to be able to measure these high orders a multilayer with a high number of periods is required. The error in the determination of the total thickness is then equal to the error in the period thickness multiplied with the number of periods. This value is generally larger than obtainable by the optimization proposed here.

RESULTS

Our first application of the method is on a W/Si multilayer of which only the Si surface was smoothened by energetic ions. From the TEM image [Fig. 1(a)] an intensity profile of the multilayer was extracted and averaged as described above [Fig. 1(b)]. By fitting the calculated reflectivity, determined from the TEM intensity profile, to the measured Cu K_α reflectometry data, as shown in Fig. 2(a) by

FIG. 1. (a) Cross-sectional TEM image of a 9 period W/Si multilayers with period thickness of 5.9 nm. After deposition of each Si layer a smoothing procedure with 300 eV Kr^+ ions was applied. (b) Averaged intensity profile as obtained from the top 10 nm of the cross sectional TEM of (a). Indicated are the levels of $I_{\max}$, $I_{\min}$, and b , determined from the fitting routine.

points, the four free parameters ($I_{\min}$, $I_{\max}$, b , and T) are then determined. The result of the optimization of the merit function M_{linear} [Eq. (7a)] is indicated by a solid line. The density profile resulting from this fitting procedure is shown in Fig. 2(b). Although the fits were affected by the choice of the merit function, the final density profile did not change significantly. Since the linear fit puts more weight on the more grazing angles, which, as discussed above, include information about layer thicknesses and average composition, we preferred this merit function.

The good agreement observed up to the second order diffraction peak demonstrates that period thicknesses as well as individual layer thicknesses and densities are estimated correctly. Small errors in the density profile within each period may cause a deviation of the calculated reflectivity from the measured data beyond the second order Bragg peak. However, the slightly asymmetric shape might still be used to get a good impression of the density profile of a single period, as shown in Fig. 2(c), and this asymmetry can be explained by diffusion of W into Si.

A similar procedure has been conducted for a second W/Si multilayer, where all but one W layer was completely intermixed into Si. This was achieved by depositing a full period of Si and a half period of W, followed by ion bombardment with 1000 eV Kr^+ ions until the extra half period was removed. As a result, most of the W was removed, but a part of the W was driven into the Si. Figure 3(a) shows the density profile of the entire stack, which shows the different

FIG. 2. (a) Measured (thick line), and fitted (thin line) $\theta:2\theta$ reflectivity curves at 0.154 nm ($\text{Cu } K_{\alpha}$). (b) Tungsten concentration profile of the full multilayer stack as obtained from the cross-sectional TEM after calibration of the density and depth scale using the fitting algorithm. (c) Detail of (b): tungsten concentration profile of the fourth period. Indicated are the determined interface widths of the Si-on-W interface (1.4 nm) and the W-on-Si interface (1.9 nm).

first W layer (not intermixed) and the subsequent intermixed periods. The strong asymmetric shape of the density profile of each intermixed period [Fig. 3(b)] indicates an in depth variation of the W concentration caused by intermixing.

As a third example, a Mo/Si multilayer was analyzed. In contrast with the W/Si system, Kr^+ ions of 300 eV were sufficient to perform intermixing of Mo into Si. A striking difference with the W/Si structure is that we do not observe a significantly asymmetric shape within each intermixed period in the density profile [Figs. 4(a) and 4(b)], which suggests that Mo intermixes with Si only in a constant ratio: the Mo concentration of approximately 40% in the intermixed periods corresponds to MoSi_2 (Ref. 13) (Mo content 38%).

FIG. 3. (a) Calibrated tungsten concentration profile of the full multilayer stack. Clearly visible is the difference between the first (deepest) W layer, which is not treated with ions, and the subsequently deposited layers, which are treated with 1000 eV Kr^+ ions. (b) Detail of (a): tungsten concentration profile of the fourth period as determined by the fitting algorithm. Indicated are the widths of the Si-on-W interface (1.0 nm) and the W-on-Si interface (1.3 nm).

As a last example we show data of a 50 period standard Mo/Si multilayer, as generally used in EUV lithography. In this multilayer mirror the Mo layer was not treated with ions. From the CS-TEM image we could determine that small crystallites were present within the Mo layers. Because crystallites appear darker in the CS-TEM image, the minimal intensity in the CS-TEM is too low in this case. However, because $I_{\min}$ is a free parameter in the fitting routine, this will not result in an incorrect determination of the intensity of a pure, amorphous Mo layer. The $\text{Cu } K_{\alpha}$ reflectometry measurement is shown in Fig. 5(a) (thick line, for clarity only a moving average of the measurements is shown; the analysis has been performed on the complete, unaveraged, spectrum). The high reflected intensity of this sample enables more Bragg peaks to be applied in the fitting routine. The results for the linear fit (thin line) as well as the logarithmic fit (dotted line) are shown.

For the linear fit the d -spacing was not estimated perfectly as we can observe from the small difference in position with respect to the measured data of the higher order Bragg peaks. Although we observe a relatively good agreement between the fitted result and measurement up to the second order, for larger angles a deviation is visible. This is in contrast to the result of the logarithmic fit for which the larger angles agree well. These deviations are caused by a wedged shape of the TEM sample, causing a gradual change

FIG. 4. (a) Calibrated molybdenum concentration profile of the full multilayer stack. Also in this figure the difference between the untreated first Mo layer and the subsequently treated Mo layers is visible. (b) Detail of (a): Molybdenum concentration profile of the eighth period as determined by the fitting algorithm. No significant difference is observed between the Si-on-Mo interface and the Mo-on-Si interface.

of the observed density. Although the shape of the two determined profiles is similar [Fig. 5(b)], the logarithmic determined profile (dotted line) shows an apparently incorrect $\text{Mo}_{75}\text{Si}_{25}/\text{Mo}_{35}\text{Si}_{65}$ structure as compared to the Mo/Si structure determined using the linear fit (solid line).

Because the essential information is provided in the more grazing angles, we conclude that the linear fitting results in a better agreement with the measured data. The density profile determined by linear fit clearly shows two interlayers in each period [Fig. 5(b)]. These interlayers were already visible in the original TEM data as can be seen in Fig. 5(c). Using our method we are now able to determine the absolute concentrations at these interlayers. At the Si on Mo interface a Mo concentration of about 40% is observed, whereas on the Mo on Si interface a Mo concentration of about 70% is visible. This is in good agreement with the Mo concentrations in MoSi_2 (35%) and Mo_5Si_3 (69%) found in earlier investigations.¹⁴

DISCUSSION

Because the determined density profiles were mostly insensitive for the choice of the merit function, and thus insensitive for the quality of the fit, these examples already give a good indication that the derived density profiles also correspond with the actual structures. However, as with any TEM

FIG. 5. (a) Measured (thick line), linear fitted (thin line), and logarithmic fitted (dotted line) $\theta:2\theta$ reflectivity curves at 0.154 nm (Cu K_{α}) of a 50 period Mo/Si multilayer (period thickness 7.2 nm). For clarity a 9-point moving average of the data is shown. Best agreement at small angles is visible for the linear fit, and at large angles the logarithmic fit shows a better agreement. The Si layers were smoothed by Kr^{+} ions. (b) Molybdenum concentration profile of the eighth period as determined by the linear fitting algorithm (solid line) and logarithmic algorithm (dotted line). (c) TEM intensity profile of the eighth period used to determine the concentration profile. Indicated are the determined levels of the three missing parameters using the linear fit.

analysis, an improper alignment of the substrate relative to the electron beam would reduce the quality of the images, notably at the interfaces. When we take into account a maximal CS-TEM sample thickness of 100 nm and a misalignment of maximal 1° , a blurring of 1.7 nm can occur. In that case higher order Bragg peaks lose importance in the fitting procedure. A similar effect is caused by the procedure to obtain the intensity profiles by averaging the CS-TEM pictures. Figure 6(a) shows the result of the analysis of the TEM

input profile of the 10-period W/Si multilayer where this profile has been blurred using a 2 nm wide averaging (dotted line). The previously determined profile has been included for reference (solid line). Due to the averaging, details with a scale below 2 nm are removed. The calculated reflectivity for both profiles is shown in Fig. 6(b). Clearly visible is the perfect agreement around the first-order Bragg peak, and the large differences beyond the second order peak. This again confirms our finding that for this method the reflectivity between the critical angle and the first order Bragg peak suffices for analysis, since this range contains the essential data missing in the TEM analysis. The accuracy of the determined profile thus only depends on the resolution of the TEM image.

Typically, the accuracy of the method in determining the d -spacing amounts to less than 1%. Our method is even able to determine the individual interlayer compositions, for example, $MoSi_2$ at the Si-on-Mo interface and Mo_3Si_5 at the Mo-on-Si interface. This result is in contrast to the general assumption^{6,14,15} that the composition of both types of interlayers is equal. This assumption is based on the limitation of the analysis using reflectometry, which is unable to distinguish a Mo_3Si_5 layer from a thinner $MoSi_2$ layer.¹⁴

The wedged shape of the cross section TEM substrate, as for example seen in the 50 period Mo/Si multilayer structure, has the same effect as a low frequency change in concentration profile. According to the Fourier model this error shows in the reflectometry curves at small angles, and thus at high reflectivities. This part is of considerable influence for the fitting procedure. It is especially important to reduce this wedge error as much as possible for thick structures. This can clearly be observed in Fig. 7, which shows the fitted and calculated reflectivity curves for the 50 period Mo/Si multilayer structure between the critical angle and the first order Bragg peak. Adding an extra parameter, i.e. the wedge angle, in the model used for the fitting procedure might result in an improvement. However, each addition of a free parameter in the fitting routine dramatically increases the risk that the resulting concentration profile does not correspond any more with the real profile.

When we compare the result of the linear fitting routine and the logarithmic fitting routine, especially in the case of the 50-period Mo/Si multilayer mirror, we observed that the logarithmic fit resulted in a better agreement at larger angles and a worse agreement at the smaller angles. However, this logarithmic fit would result in an unrealistic molybdenum silicide structure of which the 13.5 nm near normal incidence reflectivity is largely incorrect (<40% vs the experimentally measured 61%). We therefore conclude that the logarithmic fitting routine results in an incorrect determination of the density profile.

In order to demonstrate that the results of the analysis are not depending on the use of *a priori* information, other than the used materials and the lateral homogeneity, even the most obvious, experimentally determined information was excluded, i.e. oxidation of the top Si layer. From simulations using software for modelling the optical properties of multilayer films (IMD) written by Windt¹⁶ we learned that the effect of oxidation of this layer is primarily observed as

FIG. 6. (a) Comparison of the result of the analysis (a) of the W/Si multilayer using the original TEM input profile (solid line) and the 2 nm averaged TEM input profile (dotted line). (b) Comparison of the calculated reflectivity spectra of the original profile (thin solid line) and the averaged profile (dotted line) to the measured spectrum (thick solid line).

changes in reflectivity between the Bragg peaks. This is due to the fact that the optical indices of Si and SiO_2 are very close to each other, as well as very close to the optical index of air. A good fit of the Bragg peaks, as was found in our experimental results, therefore indicates that the determined multilayer structure does agree with the real profile.

An interesting example for demonstrating our method is the standard W/Si multilayer (Figs. 1 and 2). Based on the good fit we can assume that the misalignment of the electron beam is negligible and the sample is not prepared in a

wedged shape. From the asymmetric density profile [Fig. 2(c)] we therefore can conclude that W diffuses into Si during or after deposition.

CONCLUSIONS

We have developed and demonstrated a method to analyze CS-TEM images, based on a description of the electron-beam absorption in the TEM sample. This enabled us to determine the absolute intensity-to-density-conversion-scale of

FIG. 7. Detail of Fig. 5(a). Small angle part of measured and fitted reflectivity curves at 0.154 nm ($\text{Cu } K_\alpha$) of a 50 period Mo/Si multilayer (period thickness 7.2 nm), showing the influence of the wedge-shape of the CS-TEM sample on the calculated reflectivity curve.

CS-TEM images by a fit of essentially only three free parameters. Using this result we can obtain a complete quantitative depth-dependent density profile. As input data we only used the refractive indices of the two materials used in the structure, measured x-ray reflectivity data and the CS-TEM image. The single assumption was that the layers of the multilayer structure are laterally homogeneous. It has to be emphasized that no model containing *a priori* known information, like compositions, thicknesses, and roughnesses of the individual layers and interfaces, was needed to obtain a good agreement with the x-ray reflectometry data used in addition to the TEM. This makes our method more reliable than alternative methods, e.g., standard fits to x-ray reflectivity data.

To achieve subnanometer resolution, proper preparation of the cross-sectional TEM sample as well as perfect alignment in the TEM is essential. The experimental results demonstrate that the method can be used to determine the in-depth density profile, thickness, roughness, and even stoichiometry for each individual layer in the structure.

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