SURFACE AND INTERFACE ANALYSIS
Surf. Interface Anal. 2004; **36**: 1102–1105
Published online in Wiley InterScience (www.interscience.wiley.com). DOI: 10.1002/sia.1850

Instrument for thin film diagnostics by UV spectroscopic reflectometry

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Received 6 August 2003; Revised 28 November 2003; Accepted 1 December 2003

This paper describes the principles, design and testing of an instrument for the UV/Vis-optical-reflectance monitoring of homogeneity of the optical properties of thin films developed in our group. A 200 μm reflection probe, miniature fibre optic spectrometer and a home-built computer controlled x–y mapping stage together with original data fitting software are the principal parts of the instrument. The results achieved show the usability of this instrument for the post-production testing of samples of various optical thin films prepared by different technologies. By this instrument the gradients and other inhomogeneities of thickness and spectral optical constants (n, k) of thin films can be monitored. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: UV spectroscopic reflectometry; surface homogeneity

INTRODUCTION

Reflectometry together with ellipsometry are common methods for measuring optical properties of thin films because they are accurate, non-destructive, and require virtually no special sample preparation. At present, spectroscopic reflectometry and ellipsometry are the two most common optical methods. Spectroscopic reflectometry measures the light intensity within a wavelength interval reflected from a thin film. In this method, the incident light direction is perpendicular to the sample. Spectroscopic ellipsometry is a similar technique, except that it measures reflectance at an oblique light incidence for two different polarizations. In general, reflectometry is much simpler, faster and less expensive than ellipsometry, but it is limited to the measurement of less-complex structures. In surface homogeneity measurements, the simplicity and speed of reflectometry is a big advantage as thousands of points needs to be measured over the sample area. In our Institute, an instrument working on the principles of spectroscopic reflectometry equipped with a mapping stage allowing surface homogeneity measurements has been developed. This type of instrument is a useful tool for the post-production monitoring of transparent thin-film samples prepared by various deposition techniques—in our case by ion beam assisted deposition (IBAD).

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Contract/grant sponsor: Czech Grant Agency; Contract/grant number: GA102/02/0506.
Contract/grant sponsor: Ministry of Education, Czech Republic; Contract/grant number: ME 334, SEZ:J22/9826200002.

MEASUREMENT PRINCIPLES OF SPECTRAL REFLECTANCE

The basic principle of reflectometry is very simple—the spectral intensity of a light beam is measured before and after its reflection on the sample under study.[1] The ratio of the intensity of the reflected and incoming beam is termed the *absolute reflectance*. It is generally very difficult to directly measure the intensity of the light beam before it strikes the sample. Because of that, we use the relative reflectance measurements—the intensity of a light beam reflected from the sample under study is divided by the intensity of the same light beam reflected from a known (standard) sample. The ratio of the reflected intensity from the unknown sample to the reflected intensity from the standard sample is termed the *relative reflectance*, and represents the reflectance of the unknown sample relative to the standard sample.

The absolute reflectance of the unknown sample can always be calculated from its relative reflectance as long as the absolute reflectance of the standard sample is known. We use a single-crystal silicon wafer as the standard sample.

DETERMINING FILM PROPERTIES FROM SPECTRAL REFLECTANCE

The amplitudes and periodicity of the reflectance spectrum of a thin film are determined by the film thickness, optical constants (index of refraction n, extinction coefficient k), and other properties; for instance, interface roughness.[2] A mathematical dispersion model describing n and k over a range of wavelengths by using only a few adjustable parameters is generally used—for example, the Cauchy function:

$$n(\lambda) = A + \frac{B}{\lambda^2} + \frac{C}{\lambda^4}, k(\lambda) = D + \frac{E}{\lambda^2} + \frac{F}{\lambda^4}.$$

For dielectric materials such as silicon oxide it is justifiable to use this function both in the visible and UV range (the extinction coefficient k can be omitted for most of these materials). For semiconductors, for example a single-crystal silicon substrate in the UV range, dispersion behaviour is more complicated and light absorption may no longer be neglected.

Optical film properties are determined by calculating the reflectance spectra from the trial values of thickness and model parameters in the Cauchy function.[3] We use a fitting program for adjusting these values until the calculated reflectance matches the measured reflectance.

EXPERIMENTAL

Experimental set-up

Reflectivity is measured by a 200 μm reflection probe (Avantes FCR 7-UV-200). It consists of six illuminating fibres surrounding one central reading fibre; the diameter of each of these fibres is 200 microns. A combined deuterium–halogen light source (Avantes DH 2000) is used as the light source. Detection of reflected light in the ultraviolet/visible range (200–900 nm) is provided by a miniature fiber optic spectrometer (Avantes S2000). Samples are placed on a computer controlled x–y mapping stage that has been constructed by our group (see Fig. 1). Samples up to 3 inches in diameter can be easily positioned with a maximum resolution of 50 microns.

Data-fitting software

Our home-built data analysis software SPEKTRA3 allows the calculation of reflectivity using various dispersion functions (Cauchy, Urbach[1]), as well as tabulated values of n and k. Minimization of the sum of squares between theoretical and experimental reflectivity values was done by the well-proven Marquardt–Levenberg method.[4] This very effective algorithm can quickly calculate optical parameters in thousands of points over a sample surface. This is particularly important for homogeneity measurements on big samples. Each parameter can be set as 'fixed' or 'free'. If, for instance, our interest is aimed only to the thickness of a multilayer of well-known material, the parameters of n and k dispersion relations can be pre-set to known values and only thicknesses of particular layers are calculated. Optionally, a Hanson–Krogh solver[5] can be used. This method allows the setting of lower and upper limits for model parameters, which is useful for the acceleration of a fitting procedure.

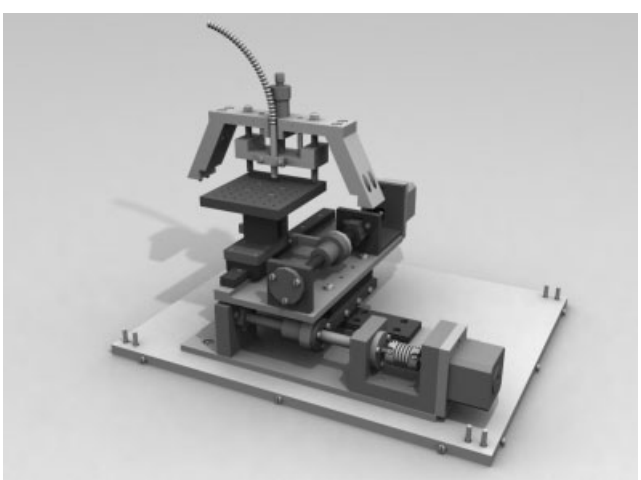

Figure 1. x–y mapping stage with a reflection probe.

RESULTS AND DISCUSSION

Silicon oxide thin films prepared by ion beam assisted deposition (IBAD) on Si (111) substrates were chosen as testing samples. In a deposition chamber five samples were placed into a line in such a way that the deposition rate was the lowest over the sample #1 and highest over the sample #5. One of the substrates was left without depositing the thin film and was used as a standard for reflectivity measurements.

The samples had been measured over a 10 × 10 mm area with a resolution of 200 μm, which represents 50 × 50 testing points. To achieve a maximum signal, the reflection probe was kept 3 mm above the sample surface and the integration time of the S2000 spectrometer was set to 4 milliseconds. The scanning time over the whole sample area was approximately 10 minutes. At each testing point the spectral reflectivity of the sample in the wavelength interval ranging from 200 to 800 nm was measured.

An experimental spectral-reflectivity curve in one testing point of the sample #5 together with its best fit, is shown in Fig. 2. There is a very good agreement between the experimental curve and the fit. The best fit gives us the optimized searched parameters of the Cauchy function (and thus n and k) and the film thickness as well. In the case of a silicon substrate we have used tabulated data for n and k from the UV–Vis range.[6] Comparative reflectivity measurements by another spectrophotometer (Varian CARY 5E) with different analysis software also were made and the results showed the perfect match to the experimental curve in the figure (not plotted for better readability) as well as a very good match in the calculated optical properties.

The optimized parameters used in the best fit for the given testing point were as follows: $d = 266$ nm, $A = 1.485$,

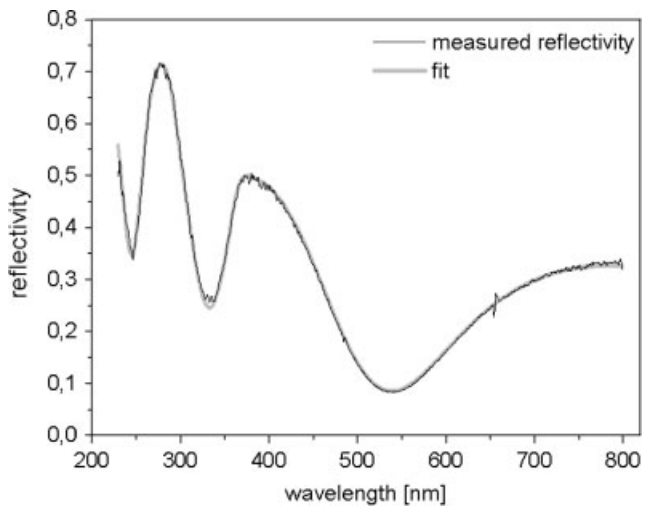

Figure 2. Spectral reflectivity in one testing point of the sample #5.

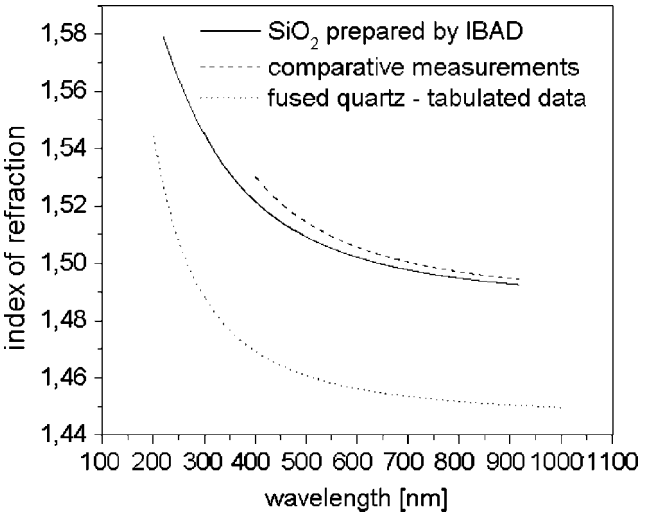

Figure 3. Refractive index of the sample #5 in comparison with tabulated data for fused quartz.

$B = 6400\,\mathrm{nm}^2$, $C = -9 \times 10^7\,\mathrm{nm}^4$, where d is the layer thickness and A, B, and C are parameters in the Cauchy function:

$$n(\lambda) = A + \frac{B}{\lambda^2} + \frac{C}{\lambda^4}.$$

The sum of squares for this fit was 8×10^{-2}. A plot of refraction index as a function of the wavelengths calculated from the Cauchy formulae by using the optimized parameters is shown in Fig. 3. Additionally, the index of refraction data obtained from comparative measurements of the same sample in the visible range by the spectrophotometer Varian CARY 5E and tabulated data of fused quartz are shown here.[7] The values of the index of refraction calculated from the measurements carried out by the fibre optic spectrometer correspond well to those obtained by the comparative measurements. The refractive index of the deposited sample is in the whole wavelength range higher than the one tabulated for fused quartz. Higher values of refractive index indicate that the Si–O film prepared by IBAD is non-stoichiometric, i.e. it can be assigned as SiO_x, where $x < 2$. This is in agreement with the literature,[8,9] where the value of the refractive index $n = 1.55$ of a non-stoichiometric SiO_2 at $\lambda = 550$ nm is reported.

The fitting calculations have been done for each testing point of the sample surface and the resultant maps of film thicknesses and refractive indices are plotted in Figs 4 and 5, respectively. The calculation time for all 2500 testing points was approx. 20 minutes, when a PC equipped with the AMD Athlon 1.5 GHz processor was used. Both in the thickness and refractive index maps some gradients of the relevant data are visible. The thickness map shows a gradient over one sample axis, which is in accordance with estimated deposition rates given by the geometry of the deposition configuration.

The maps might thus serve as a source of valuable information for optimization of the quality and reproducibility of the deposition process.

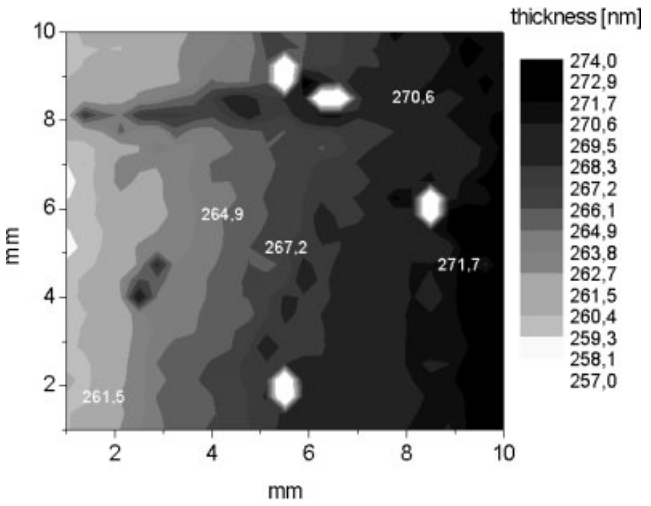

Figure 4. Map of thicknesses of the sample #5.

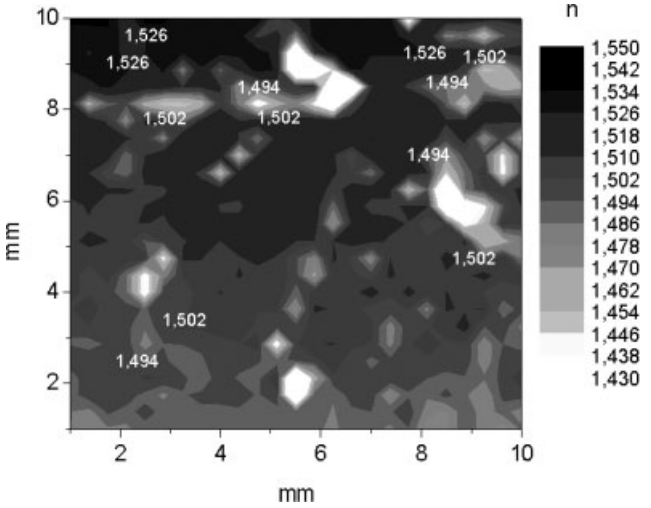

Figure 5. Map of refractive indices of the sample #5.

CONCLUSIONS

In our group an instrument for the UV/Vis-optical-reflectance monitoring of the homogeneity of the optical properties of thin films has been developed, constructed and tested. The results achieved show the usability of this instrument for the post-production testing of samples of various optical thin films prepared by different technologies.

The gradients and other inhomogeneities of thickness and spectral optical constants (n, k) of thin films can be monitored using this instrument. The main goal of the project was to gain experience in the field of software analysis of reflectance spectra. The results of this study will be directly used in the development of a more sophisticated instrument for the *in situ* real-time monitoring of homogeneities of optical properties of thin films grown by various deposition techniques.[3]

Acknowledgements

This work was supported by the Czech Grant Agency (grant no. GA102/02/0506) and the Ministry of Education (ME 334, SEZ:J22/9826200002).

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