

CERTIFICATION REPORT

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Certified Reference Material

BAM-L101

TiO₂ / SiO₂ multilayer on BK7 glass

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Berlin, February 2005

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Abbreviations and symbols

A, B, C, D	SELLMEIER refractive index model parameters
F, G, H	URBACH optical absorption model parameters
AES	AUGER electron spectroscopy
AOI	angle of incidence
d_{SiO_2}	thickness of the SiO ₂ reference coating
$d_{\text{SE, SiO}_2}$	thickness of the SiO ₂ reference coating, measured by SE
d_t	total layer thickness
ESCA	electron spectroscopy for chemical analysis
FIB	focused ion beam
GD-OES	glow discharge optical emission spectroscopy
k	imaginary part of the refractive index, extinction coefficient
λ	wavelength
MSE	mean squared error (quantity used for ellipsometric fit procedure)
n	real part of the refractive index
Ψ, Δ	ellipsometric quantities
$\Psi_{\text{exp}}, \Delta_{\text{exp}}$	experimentally measured ellipsometric quantities
$\Psi_{\text{th}}, \Delta_{\text{th}}$	theoretically derived ellipsometric quantities
R_a	roughness average
rf	radio frequency
r_p, r_s	amplitude FRESNEL reflection coefficients of the parallel and perpendicular polarization state
R_z	mean roughness depth
$\sigma_{\text{exp}, \Psi}, \sigma_{\text{exp}, \Delta}$	standard deviations of the experimental ellipsometric parameters
SD	standard deviation
SE	spectroscopic ellipsometry
SEM	scanning electron microscopy
SIMS	secondary ion mass spectrometry
SNMS	secondary neutral mass spectrometry
TEM	transmission electron microscopy
ToF	time of flight
$u(d_{\text{SiO}_2})$	uncertainty of d_{SiO_2}

1 Scope

This report describes the certification of the total layer thickness d_t of a TiO_2 / SiO_2 multilayer coating intended to be used for the evaluation and calibration of depth measurement and depth resolution of surface analytical methods, such as glow discharge optical emission spectroscopy (GD-OES), Auger electron spectroscopy (AES), and electron spectroscopy for chemical analysis (ESCA) and for the evaluation and calibration of metallographic preparation methods (e.g. cross-sectioning, ball-grinding).

2 Design and preparation

The preparation of the multilayer system was done in cooperation with Schott Glas, Mainz, Germany. The substrate material is 1 mm thick BK7 glass (Schott). The layer materials have been deposited by means of an electron-beam evaporation process. The layer system consists of 5 double layers of $\text{TiO}_2/\text{SiO}_2$ with a nominal layer thickness of 100 nm. For the improvement of adhesion, an additional SiO_2 layer was applied. This layer is located between the substrate and the first TiO_2 layer. Its thickness is approximately 70 nm. In this report, the total layer thickness of the stack of 10 layers is specified and certified without this interlayer.

3 Certification

3.1 Strategy of certification

The total layer thickness was measured using several methods. All samples have been measured and certified non-destructively by spectroscopic ellipsometry (SE). As this is, for the determination of layer thickness, a model-based method, it had to be validated by transmission electron microscopy (TEM) for selected samples. There are three sources of uncertainty to be considered for the certified quantity:

- batch inhomogeneity (sample to sample),
- intra-sample inhomogeneity, and
- the model-dependent uncertainty of the result from spectroscopic ellipsometry.

Of these individual uncertainty, the latter one is the most important while the first two are less important (see sections 3.2.6 and 3.2.7).

As informative, not certified data, the results of the following methods are provided:

- Scanning electron microscopy (SEM) on a cross-section of a BAM-L101 sample,
- Transmission spectroscopy, including the comparison with a theoretical spectrum of the design determined by ellipsometry,
- Surface analytical methods with depth resolution: glow-discharge optical emission spectroscopy (GD-OES), secondary neutral mass spectroscopy (SNMS), time of flight secondary ion mass spectroscopy (ToF-SIMS),
- Surface topography and roughness analysis with white light interference microscopy (WLIM).

3.2 Spectroscopic ellipsometry

3.2.1 General Remarks

Ellipsometry is a reflection experiment with polarised light. It determines the FRESNEL reflection coefficients r_s and r_p of perpendicular and parallel (with respect to the plane of incidence) polarised light, respectively. From these values, the ellipsometric quantities Ψ and Δ are calculated. They are defined as follows:

$$\frac{r_p(\lambda)}{r_s(\lambda)} = e^{i\Delta(\lambda)} \tan \Psi(\lambda). \quad (1)$$

The ellipsometric quantities are not only dependent on the wavelength but also on the angle of incidence (AOI). By solving the equations of reflectance, the quantities Ψ and Δ can be calculated theoretically for a given multilayer system and AOI. This enables the analysis of complex layered systems with a set of ellipsometric measurements at different AOIs. For the determination of the parameter layer thickness, the theoretical quantities Ψ_{th} and Δ_{th} have to be calculated and then compared to the measured ones (Ψ_{exp} and Δ_{exp}). The mean squared error (MSE) as a measure of the agreement of theoretical and experimental values is calculated by

$$MSE \propto \left(\frac{\Psi_{th} - \Psi_{exp}}{\sigma_{exp, \Psi}} \right)^2 + \left(\frac{\Delta_{th} - \Delta_{exp}}{\sigma_{exp, \Delta}} \right)^2, \quad (2)$$

where the parameters $\sigma_{exp, \Psi}$ and $\sigma_{exp, \Delta}$ are the standard deviations of the parameters Ψ_{exp} and Δ_{exp} .

Using a standard least squares algorithm, the MSE value is minimised by fitting the parameters of the layer system (optical or dielectric constants, layer thicknesses). If the same fitting procedure and identical models have been used, the MSE of several fit procedures can be directly correlated to the quality of the fit^[1].

The SE measurements were performed with a VASE ellipsometer from Woollam / LOT. All measurements were carried out in the 400 nm – 1000 nm spectral range at AOIs of 65°, 70°, and 75°. For some applications, such as rf-GD-OES depth profiling, the thickness of the BK 7 glass substrate has to be in the range of 1 mm. Therefore, the backside reflection of the sample has to be considered when measuring and evaluating ellipsometric measurements of these samples (see below).

3.2.2 Validation of the certification method

Validation with TEM

The total layer thickness as well as the single layer thicknesses was evaluated by TEM. The measurements were done by means of a Jeol 4000 FX transmission electron microscope at a 400 kV voltage. The sample preparation was done with a FEI 200 XP focused ion beam (FIB) sample preparation/microscope device. In a first step, the samples were coated with a layer of Pt. Then a narrow slice was cut out with a focused ion beam. This slice was transferred to the TEM. The transmitted electron beam was projected on negative film slides. The magnification of the electron microscope was calibrated by a reference sample. For the BAM-L101 samples a magnification of 60000 was chosen. The real magnification was calibrated against a MAG*I*CAL calibration standard (Plano) by measuring the well known (111) layer distance of a Silicon single crystal. In the case of BAM-L101, the specified uncertainty value (u_{95}) of the length measurement is 2,6%, resulting in an absolute uncertainty of 25 nm.

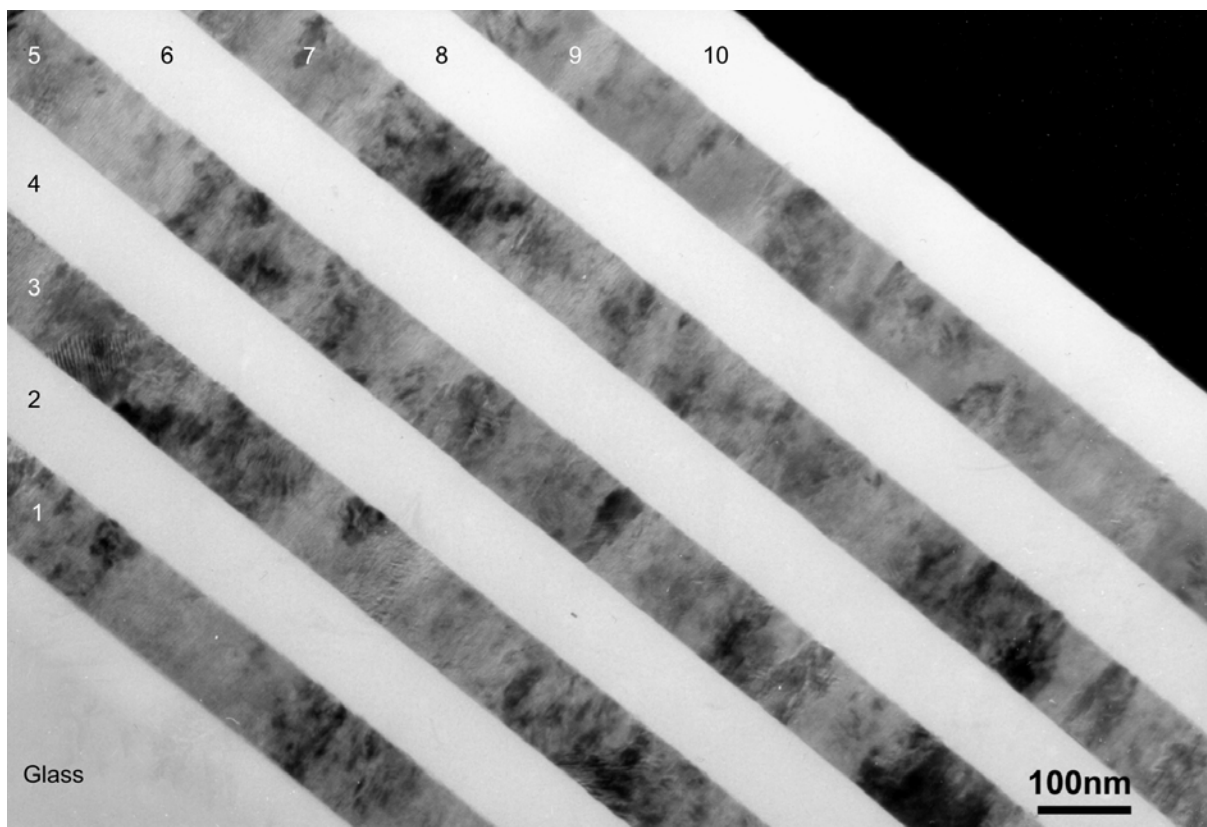


Figure 1: TEM image of BAM-L101 (positive). Odd numbers: TiO₂ layers, even numbers: SiO₂ layers

Figure 1 shows a typical TEM image which was used to determine the layer thicknesses of the of TiO₂ (dark) and SiO₂ (bright). As seen, Silica is amorphous and the Titania is micro-crystalline. The additional adhesive layer of Silica between the substrate and layer no. 1 is also vaguely visible.

Table 1: Layer thickness for BAM-L101 from the TEM image, sample no. 21

	positions					average	SD	u ₉₅
	1	2	3	4	5			
1. TiO ₂	103	103	103	103	103	103	0,0	
2. SiO ₂	92	89	91	89	89	90	0,6	
3. TiO ₂	94	96	94	97	97	95,6	0,7	
4. SiO ₂	97	97	94	97	94	95,8	0,7	
5. TiO ₂	96	96	96	96	96	96	0,0	
6. SiO ₂	96	96	96	96	96	96	0,0	
7. TiO ₂	96	96	96	96	96	96	0,0	
8. SiO ₂	92	94	91	94	96	93,4	0,9	
9. TiO ₂	103	97	97	96	96	97,8	1,3	
10. SiO ₂	94	94	94	94	96	94,4	0,4	
<i>d_t</i>	963	958	952	958	959	958	1,8	24,9

Table 1 and Table 2 show the results of the TEM measurement. The values do not differ more than indicated by the uncertainty of the individual measurements and they are also within the uncertainty interval of the ellipsometric measurement (see following sections, $d_t = 964$ nm with $u_{95} = 24$ nm). The value gained by ellipsometry is therefore supported by the TEM measurements.

Table 2: Layer thickness for BAM-L101 from the TEM image, Sample no. 53

	positions				average	SD	u_{95}
	1	2	3	4			
1. TiO ₂	103	104	103	103	103,3	0,3	
2. SiO ₂	85	85	85	87	85,5	0,5	
3. TiO ₂	96	97	101	97	97,8	1,1	
4. SiO ₂	92	89	85	91	89,3	1,5	
5. TiO ₂	97	94	97	96	96,0	0,7	
6. SiO ₂	94	94	89	94	92,8	1,3	
7. TiO ₂	96	94	96	96	95,5	0,5	
8. SiO ₂	94	94	94	94	94,0	0,0	
9. TiO ₂	96	96	97	96	96,3	0,3	
10. SiO ₂	96	97	96	96	96,3	0,3	
d_t	949	944	943	950	946,5	1,8	24,6

3.2.3 Backside correction effects

As the samples are thin, transparent and smooth on both sides, the backside reflection has to be considered and excluded. There are several experimental techniques to avoid backside reflections to a certain extent. The most important ones are blackening with varnish or soot and roughening. However, all these methods would affect the spectroscopic examination. Therefore, the samples were kept with the backside flat, smooth and untreated. Instead, the backside reflection was taken into account numerically.

Mainly two parameters are important for numerical backside correction: The total number of reflections and the fraction of the secondary beams contributing to the measurement. For the presented measurements, a number of 3 reflections and a fraction of 90% of the primary beam have been chosen.

3.2.4 Ellipsometric raw data

The hardware control of the measurement and the following numerical data evaluation were done with the WVASE32 software package, version 3.441 from Woollam Corp.

Figure 2 shows exemplarily the results of an ellipsometric measurement and the corresponding fit procedure as graphical expression of the accuracy of the thickness determination.

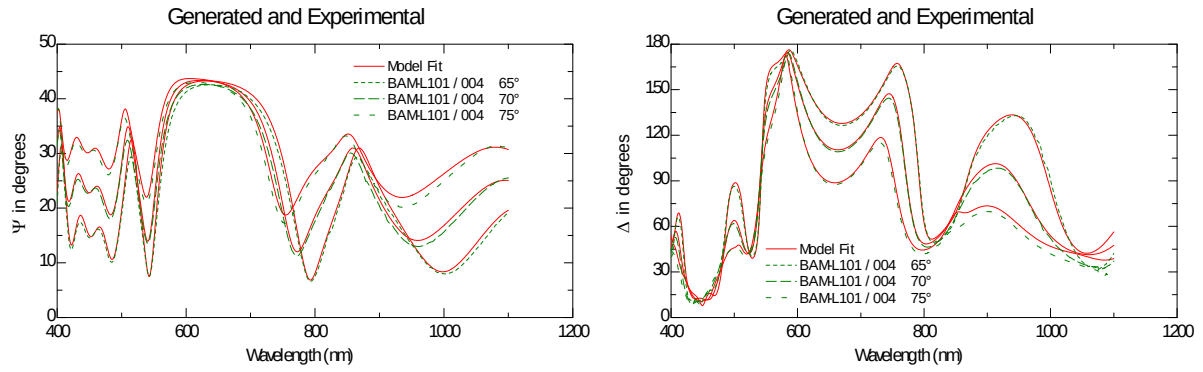


Figure 2: A typical result of an SE measurement (sample no. 4, measured in the centre of the sample)

The graphs in the figure show the Ψ and Δ values of the measurement, experimental spectra in green and the generated (from the best fit) in red colour. This gives a visual impression of the quality of the fit.

3.2.5 Fit model

To obtain the correct layer thickness by ellipsometry, at first, the optical constants of the materials involved have to be determined. In the case of the substrate material (BK7 glass) and of the SiO_2 layer material, the values known from literature could be used^[2,3]. However, for TiO_2 , the values of n and k had to be determined independently. This was done in an iterative way by determining at first the best fit of the layer thicknesses using the literature values of TiO_2 . Then, in a second fit procedure, the n and k values have been calculated with the resulting layer thicknesses. To avoid a point by point fitting of the dielectric constants, a mathematical model was chosen for the layer material as follows^[4,5]:

- For the values of $n(\lambda)$, a SELLMEIER description according to

$$n(\lambda) = \sqrt{A + B \frac{\lambda^2}{\lambda^2 - C^2} + D \frac{\lambda^2}{\lambda^2 - E^2}} \quad (3)$$

- For the values of $k(\lambda)$, an URBACH tail according to

$$k(\lambda) = F \lambda e^{-G(\frac{1}{H} - \frac{1}{\lambda})} \quad (4)$$

Figure 3 represents the graphs of the resulting n and k values.

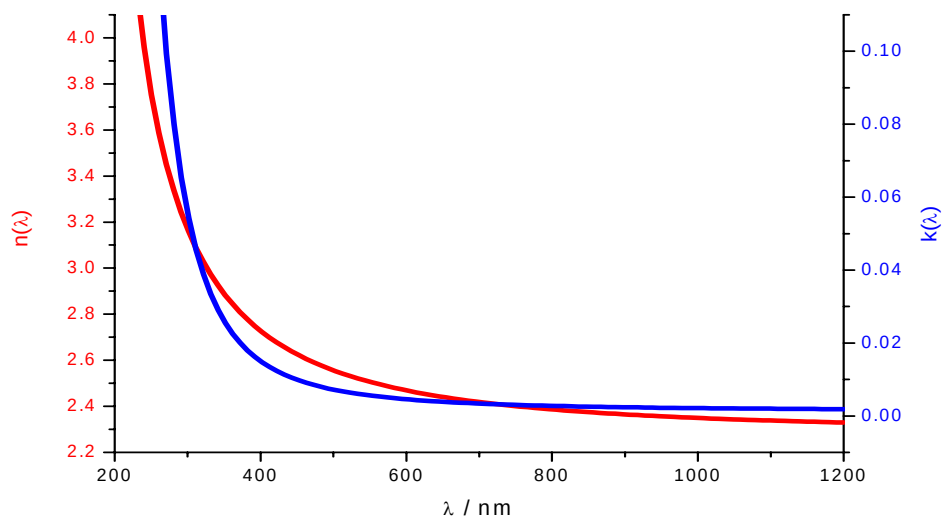


Figure 3: Calculated n and k values of the TiO_2 layer material

3.2.6 Batch homogeneity

The complete batch consists of 54 samples. The certified value is the total layer thickness d_t of the system of 10 layers. Figure 4 shows the results in a graph. Table 3 shows the values of d_t of all samples including the corresponding MSE values. The statistical treatment leading to the uncertainty (u_{95} , $k = 2$) of d_t can be found in Table 5.

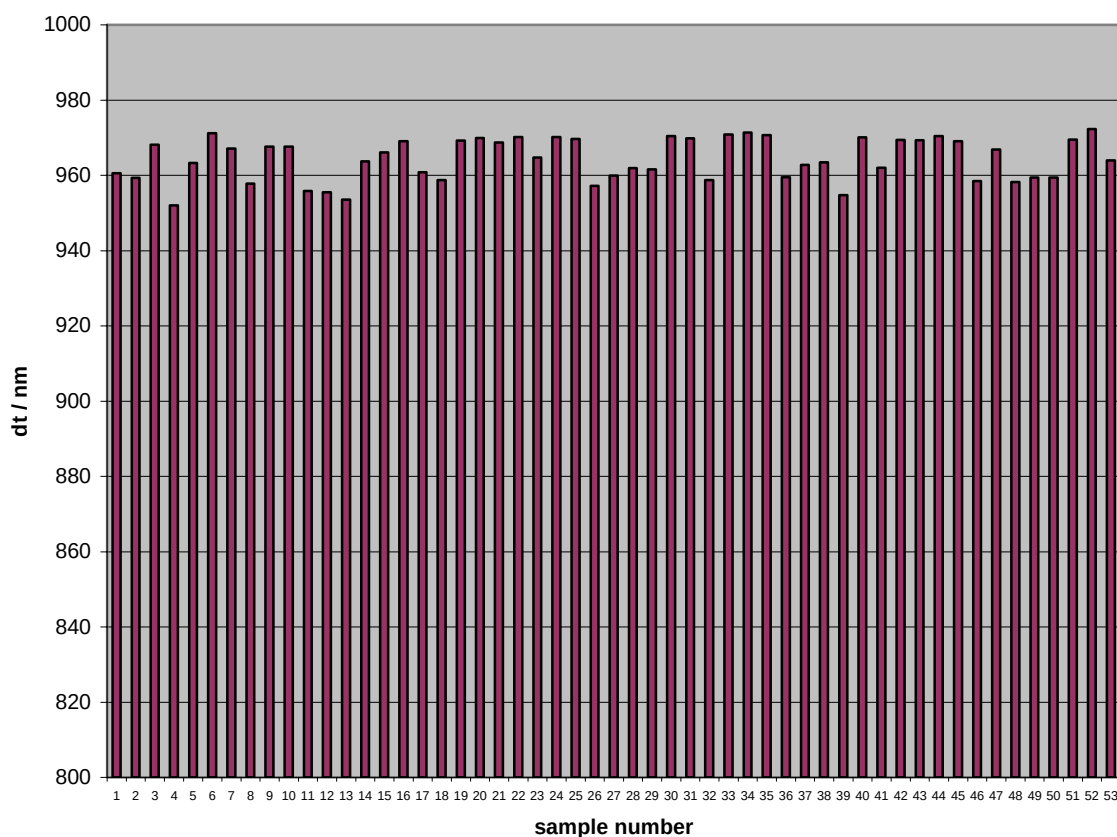


Figure 4: Total layer thickness of all samples measured in the centre of each sample.

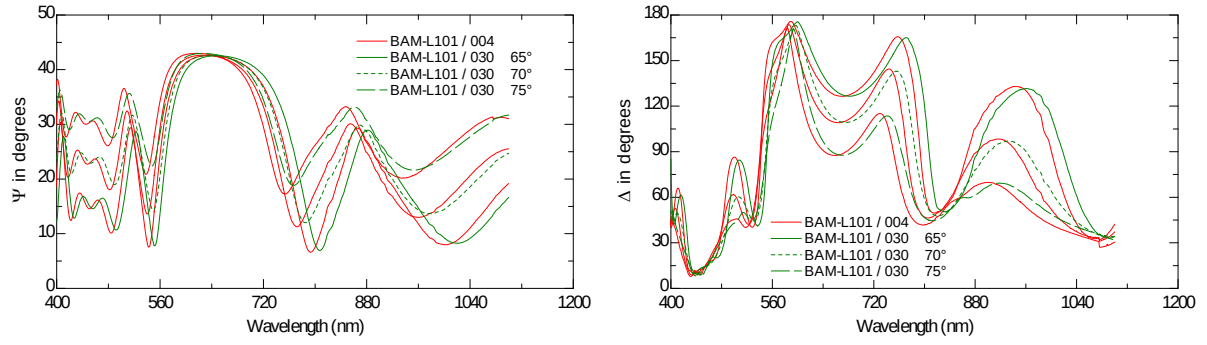


Figure 5: Upper and lower bounds of Ψ and Δ for one batch

To evaluate the batch homogeneity, upper and lower bounds samples with the largest difference in the SE spectra have been chosen. The SE spectra can be seen in Figure 5. The spectra belong to samples no. 4 and 30 with corresponding d_t values of 952 nm and 970 nm, and MSE values of 17 and 15, respectively.

Table 3: Total layer thickness and MSE of all samples according to the SE investigation (the outlier sample No 31 was not included in the batch statistics)

sample no.	d_t / nm	MSE
1	960,6	20,2
2	959,3	15,2
3	968,2	15,8
4	952,0	17,3
5	963,3	14,9
6	971,2	22,2
7	967,1	17,8
8	957,8	19,2
9	967,7	15,5
10	967,7	15,5
11	955,8	20,0
12	955,5	16,3
13	953,6	15,2
14	963,7	15,6
15	966,1	21,7
16	969,1	15,2
17	960,8	16,5
18	958,7	19,8
19	969,3	19,8
20	969,9	17,0
21	968,7	16,6
22	970,2	17,0
23	964,7	19,3
24	970,2	17,1
25	969,7	17,2
26	957,2	17,2
27	959,9	16,3
28	961,9	24,2
29	961,6	15,9
30	970,4	15,6
31	969,9	45,9
32	958,7	15,8
33	970,9	16,1
34	971,4	17,6
35	970,7	16,7
36	959,5	17,3
37	962,8	16,5
38	963,5	14,4
39	954,8	14,9
40	970,1	23,2
41	962,1	15,4
42	969,4	16,2
43	969,3	16,1
44	970,4	21,2
45	969,1	27,0
46	958,5	17,8
47	966,9	17,0
48	958,2	17,5
49	959,4	19,0
50	959,4	19,0
51	969,5	18,5
52	972,3	29,3
53	964,0	32,0

3.2.7 Intra-sample homogeneity

To determine the measurement uncertainty for d_t , it is necessary to investigate how d_t changes over the surface of an individual sample. This enables the evaluation of rim effects in the coating process that may generate inhomogeneities.

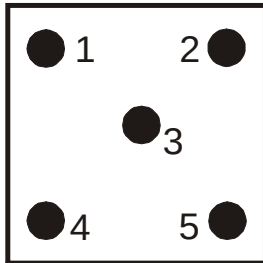


Figure 6: Distribution of the measurement spots on the surface

Figure 6 shows the measurement pattern. For this investigation, sample no. 1 was used. Table 4 shows the results. Again, the inhomogeneity of d_t within an individual sample is much smaller than between different samples of the same batch. As a certification of individual samples is not provided, this source of uncertainty is neglected for the present certification.

Table 4: Comparison of the d_t obtained at 5 different spots on the sample surface

position no.	d_t / nm
1	968,3
2	969,0
3	968,5
4	970,3
5	969,8
average	969,2
SD	0,9

3.3 Determination of the certified value

Table 5: Statistical data of the total layer thickness (all thickness values in nm)

average value of d_t	964,3
batch single value standard deviation of d_t	5,6
number of samples in batch	52
batch average standard deviation of d_t	0,8
standard deviation from inhomogeneity	0,9
model uncertainty (assuming rectangular distribution)	20,0
model standard deviation	11,5
total standard deviation	11,6
total uncertainty u_{95} ($k = 2$)	23,2
d_t rounded acc. DIN1333-2	964
uncertainty of d_t rounded acc. DIN1333-2	24

The standard Deviation (SD) of the total layer thickness over the complete batch of samples is 5,6 nm resulting in a u_{95} value of 11,2 nm with respect to the statistical variance only. How-

ever, as the ellipsometric measurement is model-dependent, a model-related error (optical constants and/or incorrect backside-correction, possible errors due to surface and interface roughness at the 12 interfaces involved) has to be considered in addition. To obtain an estimate for the model uncertainty, it is possible to carry out the fit procedure near the upper and lower bounds of d_t with fixed thicknesses and variable optical constants. This test was carried out and resulted in a maximum span of ± 20 nm for d_t . Assuming a rectangular flat distribution of values in this range, the standard deviation rising from possible model errors becomes 11,5 nm^[6]. Therefore, the value of the certified value calculates as

$$d_t = 964 \text{ nm} \pm 24 \text{ nm (uncertainty } u_{95}, k = 2) \quad (5)$$

according to^[6].

The homogeneity analysis indicates that the samples do not differ beyond that confidence interval. Therefore, this certificate applies to all samples of the batch.

4 Informative values

In this section, additional non-certified values are provided. All these values have been obtained during the certification process with different methods of analysis.

4.1 Single layer thicknesses

Table 6 shows the single layer thicknesses of d_t for all samples. This result is obtained from the ellipsometric fit procedure.

Table 6: Single layer thickness measured by SE

sample no.	thicknesses / nm										
	layer 1	layer 2	layer 3	layer 4	layer 5	layer 6	layer 7	layer 8	layer 9	layer 10	sum
1	93	93	92	104	94	93	94	99	92	107	961
2	93	90	92	104	93	92	93	102	91	109	959
3	95	89	93	109	94	91	94	104	91	109	968
4	92	92	91	104	92	92	93	99	91	107	952
5	95	88	93	108	94	91	94	102	91	109	963
6	93	95	92	106	94	94	94	101	92	109	971
7	94	92	93	107	94	93	94	101	92	108	967
8	92	94	91	104	93	93	93	100	91	107	958
9	95	87	93	110	94	90	94	105	91	110	968
10	95	87	93	110	94	90	94	105	91	110	968
11	92	94	91	103	93	93	93	99	91	107	956
12	93	90	92	105	93	91	93	101	91	107	955
13	93	90	92	104	93	91	93	100	91	107	954
14	95	86	93	108	94	89	94	103	91	110	964
15	93	94	92	105	94	93	94	100	92	108	966
16	96	85	94	112	94	90	94	104	91	109	969
17	93	91	92	105	93	92	93	102	91	108	961
18	92	94	91	103	93	93	93	100	91	107	959
19	93	93	92	107	94	94	94	102	92	109	969
20	94	91	93	108	94	93	94	102	91	109	970
21	94	90	93	109	94	92	94	102	91	109	969
22	94	92	93	108	94	93	94	102	92	109	970
23	93	93	92	105	94	93	94	101	91	108	965
24	94	91	93	108	94	93	94	103	91	109	970
25	95	90	93	109	94	92	94	103	91	109	970
26	93	92	91	104	93	93	93	100	91	107	957

sample no.	thicknesses / nm										
	layer 1	layer 2	layer 3	layer 4	layer 5	layer 6	layer 7	layer 8	layer 9	layer 10	sum
27	93	91	92	105	93	92	93	100	91	108	960
28	92	95	91	105	93	93	94	100	92	107	962
29	93	91	92	105	93	92	93	102	91	108	962
30	95	89	93	109	94	91	94	105	91	110	970
31	92	100	91	103	94	95	94	101	93	107	970
32	93	90	92	106	93	91	93	101	91	108	959
33	94	91	93	109	94	93	94	102	91	109	971
34	94	92	93	108	94	93	94	102	92	109	971
35	94	91	93	108	94	93	94	102	91	109	971
36	93	91	92	106	93	92	93	101	91	108	959
37	94	90	92	107	94	93	94	100	91	108	963
38	95	85	93	110	94	90	94	103	91	109	963
39	93	90	92	105	93	91	93	101	91	107	955
40	93	95	92	106	94	94	94	102	92	108	970
41	94	89	92	107	93	91	93	103	91	108	962
42	95	89	93	109	94	92	94	103	91	109	969
43	95	89	93	109	94	92	94	103	91	109	969
44	93	95	92	106	94	93	94	102	92	109	970
45	93	97	92	105	94	94	94	101	92	107	969
46	93	93	91	104	93	93	93	101	91	108	958
47	94	91	92	107	94	93	94	103	91	108	967
48	93	91	92	105	93	92	93	101	91	107	958
49	93	93	91	103	93	93	93	101	91	108	959
50	93	93	91	103	93	93	93	101	91	108	959
51	94	93	92	107	94	93	94	102	92	109	969
52	93	96	92	106	94	95	94	102	92	108	972
53	92	97	91	103	94	94	94	100	93	107	964
aver- age	94	91	92	106	94	92	94	102	91	108	964
SD	0,9	2,9	0,7	2,2	0,5	1,3	0,5	1,4	0,5	1,5	5,6

4.2 Scanning electron microscopy

Validation of the thickness measurement with scanning electron microscopy

The preparation method (cross-sectioning) and the measurement method (SEM) have been additionally validated and re-calibrated using SiO₂ reference coatings on Silicon. This validation was also used for the reference materials BAM-L100, BAM-L102, BAM-L103, BAM-L104, and BAM-L105. An example of a cross-section of a SiO₂ reference coating on Si, coated with a TiN hard coating, is shown in Figure 7.

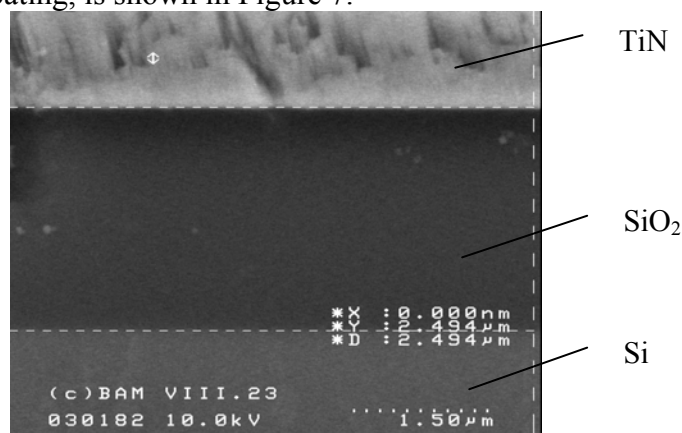


Figure 7: Cross-section of a SiO₂ reference coating on Si with TiN hard top coating

Table 7 provides 12 measurements of the layer thickness of SiO₂ at different locations along the cross-section. The evaluation length was 5 µm for each measurement.

Table 7 Thickness of cross-sections of SiO₂ reference coatings (coated with TiN) on Si

measurement identification number	d/µm
30170	2,57
30172	2,45
30173	2,47
30174	2,45
30175	2,47
30177	2,47
30178	2,46
30179	2,46
30180	2,44
30181	2,54
30182	2,49
30183	2,48
average value	2,48
standard deviation of single values $u(d_{SiO_2})$	0,05

The average thickness d_{SiO_2} of the SiO₂ reference coating was derived to

$$d_{SEM, SiO_2} = (2,48 \pm 0,05) \mu\text{m}. \quad (6)$$

For the validation of the certification method (SE), this result was compared with the SiO₂ thickness derived from SE using the BAM reference procedure^[7] spectroscopic ellipsometry according to the appropriate standard operation procedure^[8]. From the analysis of ellipsometric data, the average thickness of the SiO₂ reference coating was derived to

$$d_{SE, SiO_2} = (2,51 \pm 0,01) \mu\text{m}. \quad (7)$$

Figure 8 shows different SEM images obtained for BAM-L101. For SEM investigation, a Hitachi S4100 scanning electron microscope with a voltage of 10 kV was used. For the measurement, the embedded samples have been ground and polished perpendicular to the surface. As the magnification and the resolution are lower than in case of the TEM, the length measurements have not been considered for the certificate.

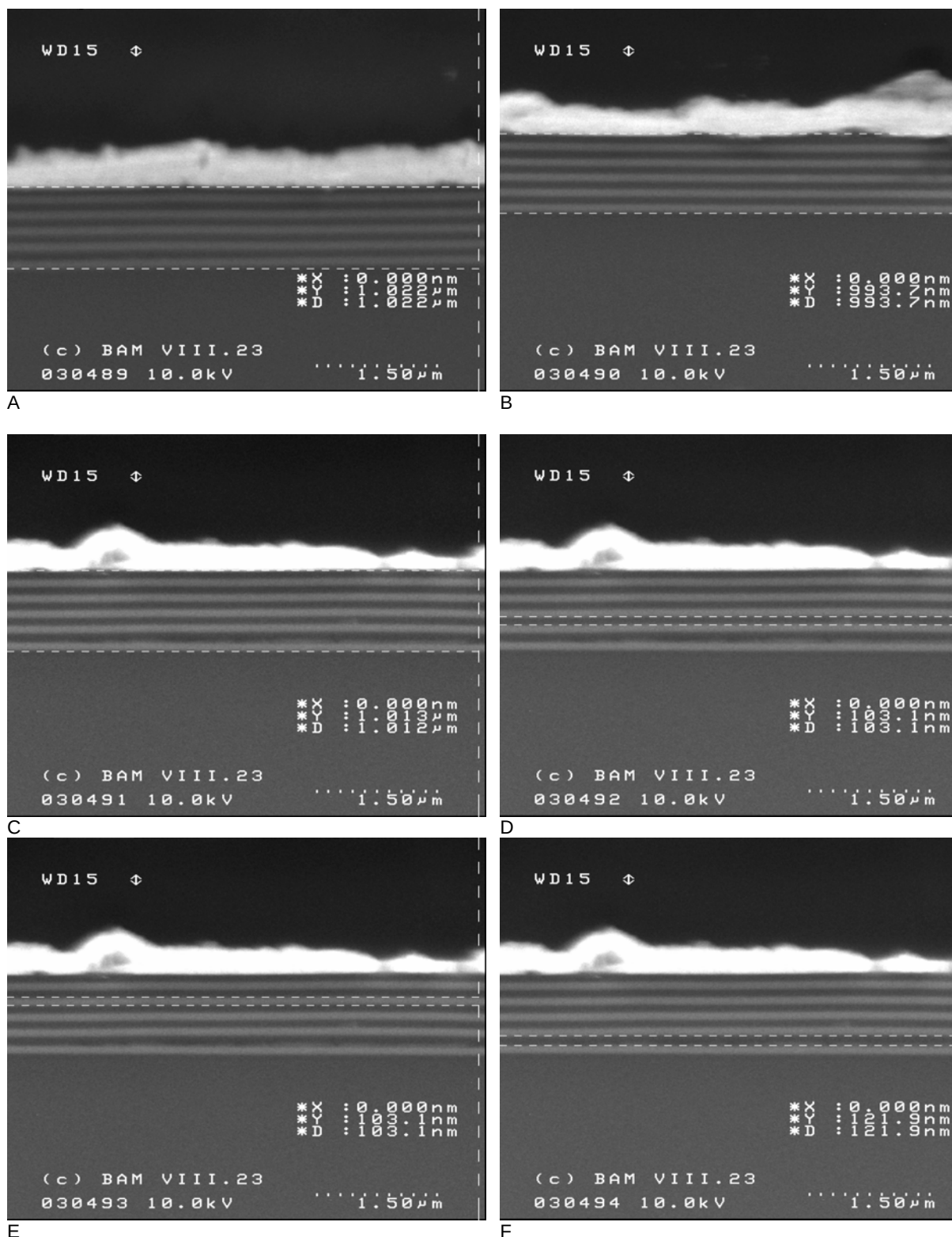


Figure 8: SEM images of BAM-L101: Total layer thickness at different positions (A, B, C), thickness of different single layers at one position (D, E, F).

4.3 Roughness analysis

The surface roughness was evaluated by means of white light interference microscopy (WLIM) of Zygo-LOT. As the determination of roughness values with optical interference measurements was not yet standardised during the certification process, a roughness value can not be given as a certified value. Figure 9 shows the result of the surface topography meas-

urement. The roughness values were obtained with the MetroPro Software as values for the whole measured area of approximately $(70 \times 53) \mu\text{m}^2$. From this analysis, the surface roughness values are determined as follows:

- roughness average: $R_a = 4,6 \text{ nm}$
- mean roughness depth: $R_z = 53,8 \text{ nm}$

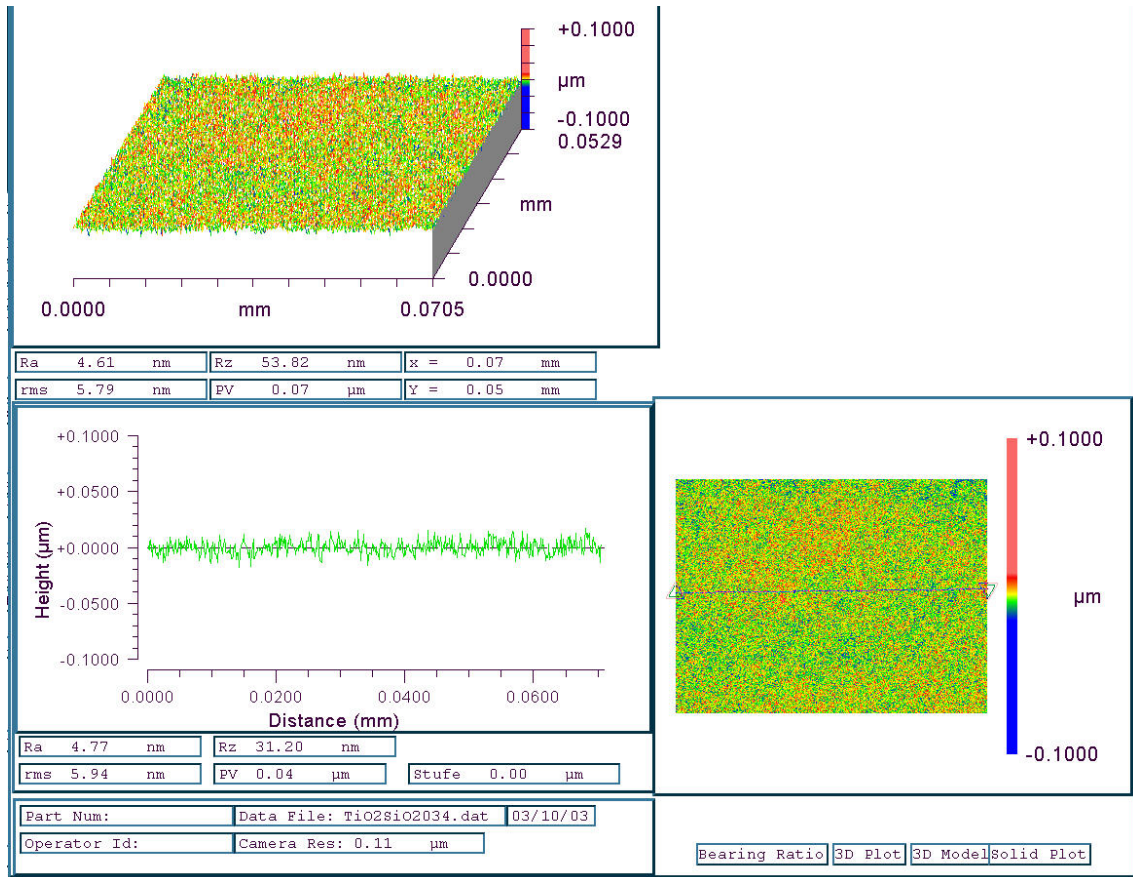


Figure 9: Surface topography of a BAM-L101 sample measured by WLIM.

4.4 Optical transmission

The optical transmission spectrum is a characteristic property of a dielectric layer system and is related to the ellipsometric measurement. The optical transmission was measured using a LAMBDA 900 UV-vis-NIR spectrometer (Perkin-Elmer). In the case of an isotropic layer, it is possible to calculate the theoretical intensity transmission spectrum analogous to the ellipsometric measurement. For this, the OptiLayer software has been employed. Figure 10 shows the results of measurement and of calculation.

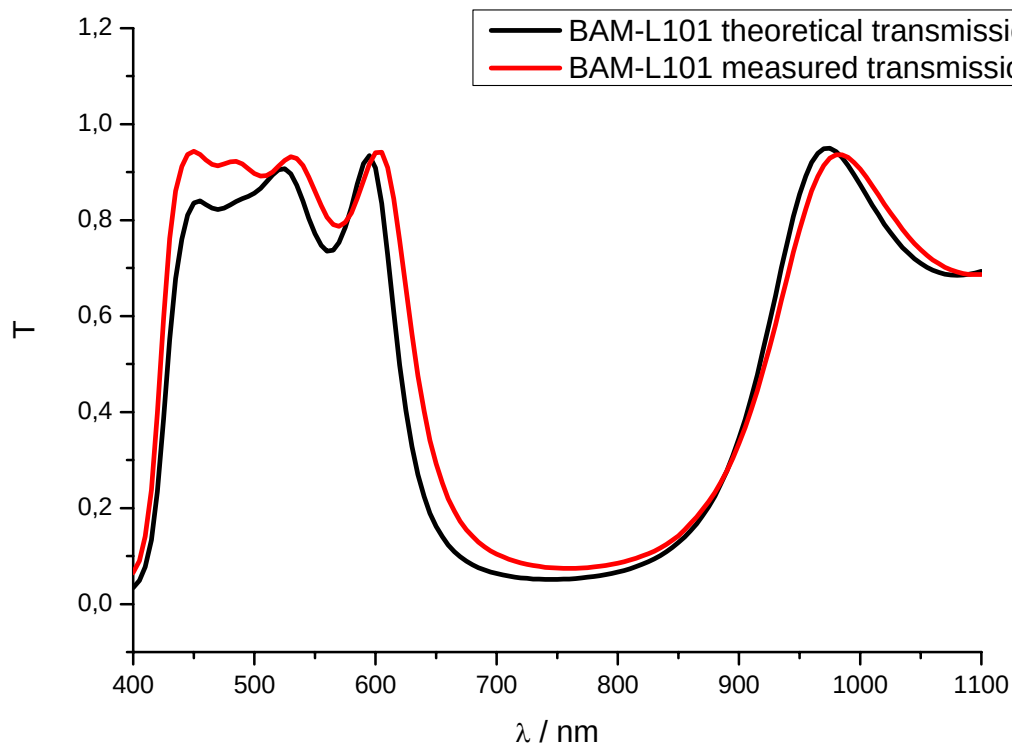


Figure 10: Theoretical and measured transmission of BAM-L101 (sample no. 14)

4.5 Application: depth profiling^[9]

One of the main purposes of the reference material BAM-L101 is the provision of a reference material for depth profiling in surface chemical analysis. There are several methods for surface analytics available that have been employed for testing of BAM-L101 samples.

4.5.1 ToF-SIMS

ToF-SIMS was used to investigate the in-depth the multilayer structure. In a first attempt, the multilayer has been in-depth profiled by sputtering layer-by-layer. Due to the extremely low sputtering rates (and hence long times for analysis), the BAM-L101 specimen was cross-sectioned and ToF-SIMS images of the cross-section reconstructed to depth profiles – as an alternative way to the first approach. The result is given in Figure 11. The measurements have been performed on a TOF.SIMS 4 spectrometer (IONTOF), on sample no. 21. The cross-sectioning technique has the advantage of providing a depth resolution independent of sputter rates in contrast to the other analytical methods presented here. The depth profile shows perfect agreement with the layer design. Every Ti and Si maximum represents a Titania and Silica layer, respectively. The crown glass BK7 of the substrate can be identified by its contents of Na and K. The first Si peak at the substrate represents the additional SiO₂ adhesive layer.

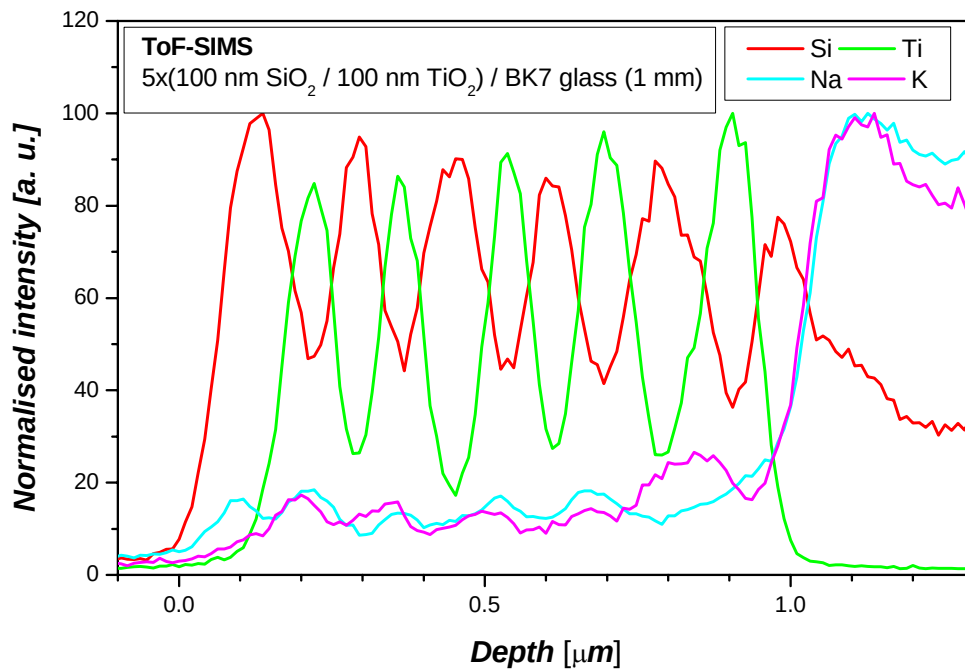


Figure 11: TOF-SIMS measurement of BAM-L101 (line-scan on cross-section)

4.5.2 rf-GDOES

The BAM-L101 multilayer stack has been in-depth profiled with GD-OES. It was possible to investigate such non-conductive layers by applying an rf-excitation. An optimised qualitative RF-GDOES depth profile is shown in Figure 12. A LECO SDP-750 GD-OES spectrometer with a Grimm-type source of 2,5 mm anode tube diameter in conjunction with a free running rf-generator was used for investigation.

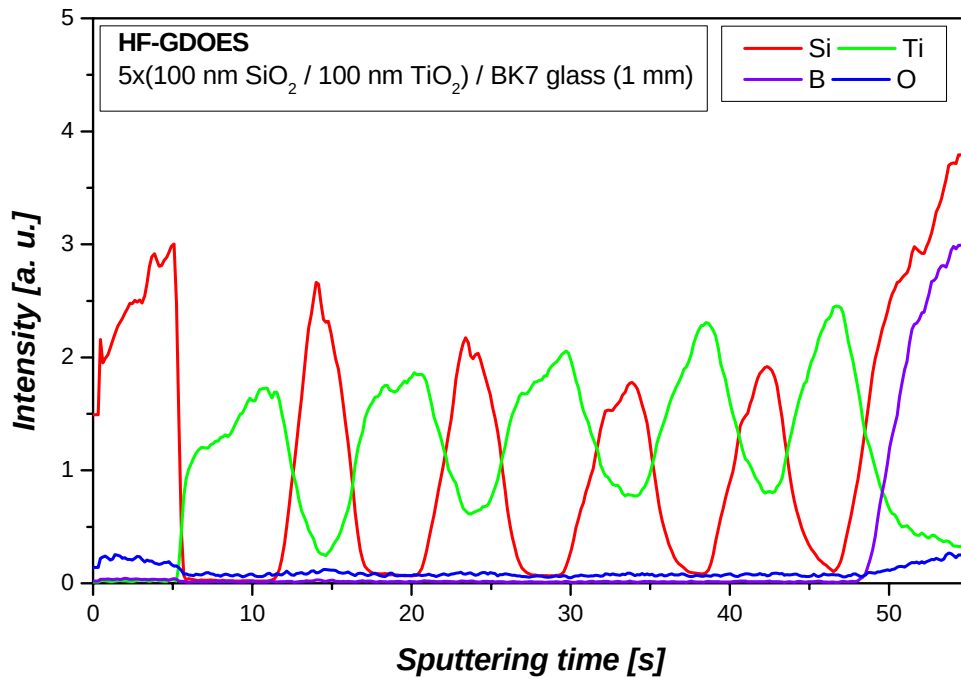


Figure 12: rf-GDOES measurement of BAM-L101 (depth profile)

4.5.3 SNMS

Another analytical technique for depth profiling of thin (non-conductive) layers is SNMS—a method considered to be close to GDS in terms of sample sputtering with noble gas ions and post-ionisation by collisions in a plasma – therefore the name Plasma SNMS. An optimised qualitative depth profile of BAM-L101 is presented in Figure 13. The measurements have been performed on an INA3 instrument , Leybold/SPECS, equipped with a rf-module.

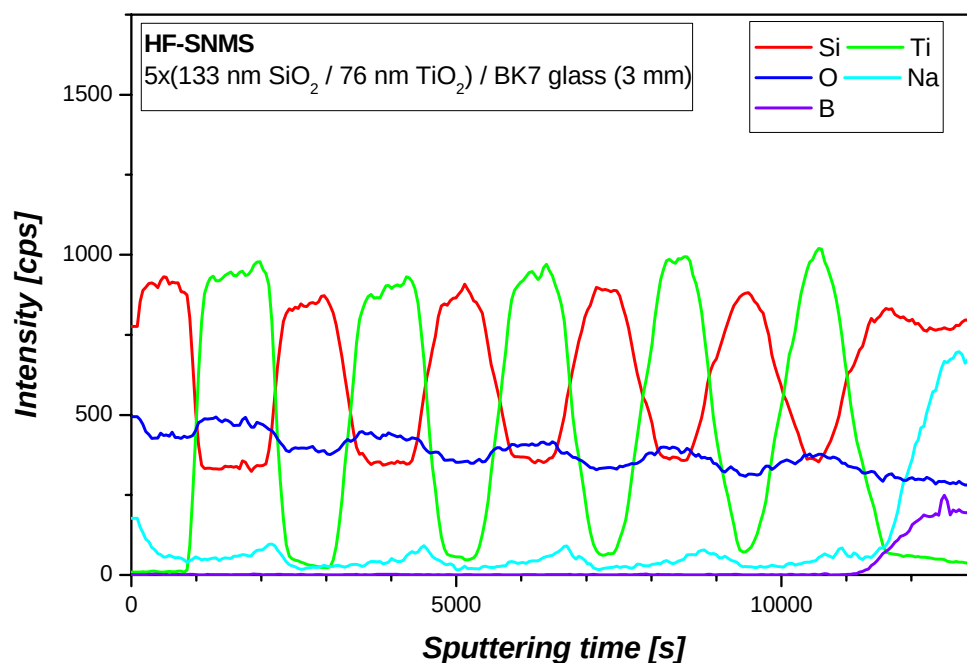


Figure 13: SNMS measurement of BAM-L101 (depth profile)

5 References

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