CERTIFICATION REPORT

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Certified Reference Material

BAM-L100

Ti/Al multilayer on 100Cr6 steel

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Abbreviations and symbols
as used within the report in alphabetical order:

ABS	Arc Bond Sputtering
AES	Auger Electron Spectroscopy
AFM	Atomic Force Microscopy
CRM	Certified Reference Material
cps	counts per second
DAP	Deutsches Akkreditierungssystem Prüfwesen GmbH
DIN	Deutsches Institut für Normung e.V.
d_{SiO_2}	average layer thickness of the SiO_2 reference coating
d_{CRM}	certified total layer thickness
$d_{\max}$	maximum total layer thickness measured by SEM
$d_{\min}$	minimum total layer thickness measured by SEM
d_{tn}	normalised total layer thickness with respect to the average of the total layer thickness of all batch reference samples
d_t	total layer thickness
E_{IT}	indentation modulus (can be correlated to Young's modulus)
EN	Euro-Norm
ESCA	Electron Spectroscopy for Chemical Analysis
F_{Ar}	Argon flow rate
F_{max}	maximum test force
GD-OES	Glow Discharge Optical Emission Spectroscopy
GIXRD	Grazing Incidence X-Ray Diffraction
H_{IT}	indentation hardness (can be correlated to Vickers' hardness)
h_{max}	maximum indentation depth
I_{coil}	magnetic coil current
I_{nx}	relative intensity deviation of identical reflexes with respect to the average of this reflex of all samples
ISO	International Organisation for Standardisation
i_n	absolute intensity of reflexes
P	electrical power
PI_n	relative intensity deviation with respect to the average of the sums of normalised reflex intensities of all samples
PVD	Physical Vapour Deposition
R_a	roughness average
R_{max}	maximum roughness depth
R_q	root man square (RMS) roughness
R_z	mean roughness depth
SE	Spectroscopic Ellipsometry
SEM	Scanning Electron Microscopy
StAA	Standard Arbeitsanweisung (standard testing procedure)
sccm	standard cubic centimeter per minute
T	temperature
t_{Al}	time for deposition of Al-layer
t_{Ti}	time for deposition of Ti-layer

U_{BIAS}	BIAS voltage
UBM	Unbalanced Magnetron
$U(d_{CRM})$	expanded uncertainty of certified total layer thickness (k = 2)
$u(d_t)$	standard uncertainty of total layer thickness
$u(d_{SiO_2})$	standard uncertainty of layer thickness measurement for ideal samples
$u(d_{SEM})$	standard uncertainty of total layer thickness measured by SEM
$u(d_{XRD})$	standard uncertainty of total layer thickness of deposition levels measured by GIXRD
$u_c(d_{CRM})$	combined standard uncertainty of certified total layer thickness
WLI	White Light Interferometry
Δ_{corr}	correction of layer thickness measured for ideal samples by SEM

1. Scope

This report describes the certification of the total layer thickness d_t of Ti/Al multilayer coatings that are useful for the evaluation and calibration of depth measurement and depth resolution of surface analytical methods (e.g. GD-OES, AES, ESCA) and for the evaluation and calibration of metallographic preparation methods (e.g. cross-sectioning, ball grinding).
Ti/Al multilayer coatings (5 double layers of Ti/Al) with a nominal layer thickness (Ti/Al) of (100/250) nm and 1.75 µm nominal total layer thickness have been deposited on polished 100Cr6 steel substrates (discs of 30 mm diameter and 4 mm thickness) using a PVD UBM sputtering process. The deposition sequence starts with Ti (layer on the substrate) and ends up with Al (top layer of layer stack).
For each CRM, a value of the averaged total layer thickness d_{CRM} is certified with respect to deposition levels. The certified value d_{CRM} is valid for the central surface area of a diameter of 25 mm. The determination of the certified value was performed by means of a validated and calibrated scanning electron microscope (SEM) at cross-sections of batch reference samples.
The certification procedure was conducted in a laboratory of BAM accredited according to DIN EN ISO/IEC 17025 [1] (DAP-PL-2614.08). Wherever possible, BAM reference procedures [2] have been used.

2. Design and preparation

2.1 Substrate-related features

100Cr6 steel substrates (discs of 30 mm diameter and 4 mm thickness) have been polished to a mirror finish (R_a = 0.03 µm; R_z = 0.2 µm, according to DIN EN ISO 4287/4288 [3]) using a metallographic standard procedure. The substrate material contains (besides iron) the following elements (weight %) [4]: C: 0.90-1.05 %; Si: 0.15-0.25 %; Mn: 0.25-0.45 %; Cr: 1.35-1.65 %; Cu < 0.3 %; Ni < 0.3 %).

2.2 Coating-related features

Ti/Al multilayer coatings have been prepared by means of PVD UBM sputtering in a commercial deposition system HTC 625 Multilab ABS™ (HAUSER Techno Coating) similar to that described in detail elsewhere [5].

Sputter targets were made from solid Ti- (99.99% purity) and Al-plates (99.95% purity). Ar gas (99.999 % purity) was used as working gas.

To avoid any influence of sample clamps on the homogeneity of the coatings, the substrates were mounted on special substrate holders (Fig. 1). These holders are fixed on a planetary rotational system (Fig. 2) which can rotate around two axes. The frequency of rotation around the first axis was 10 rev/min.

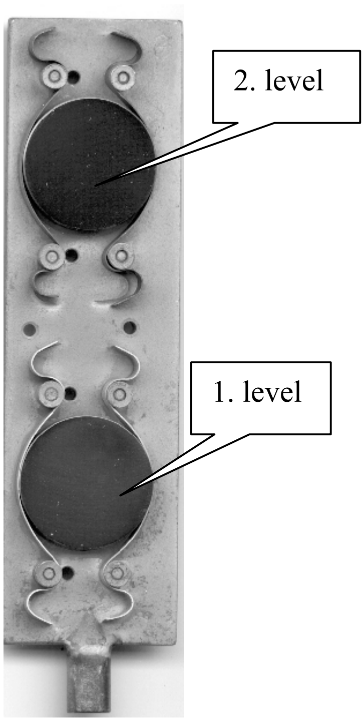


Fig. 1: Lower substrate holder with clamps and mounting levels 1 and 2

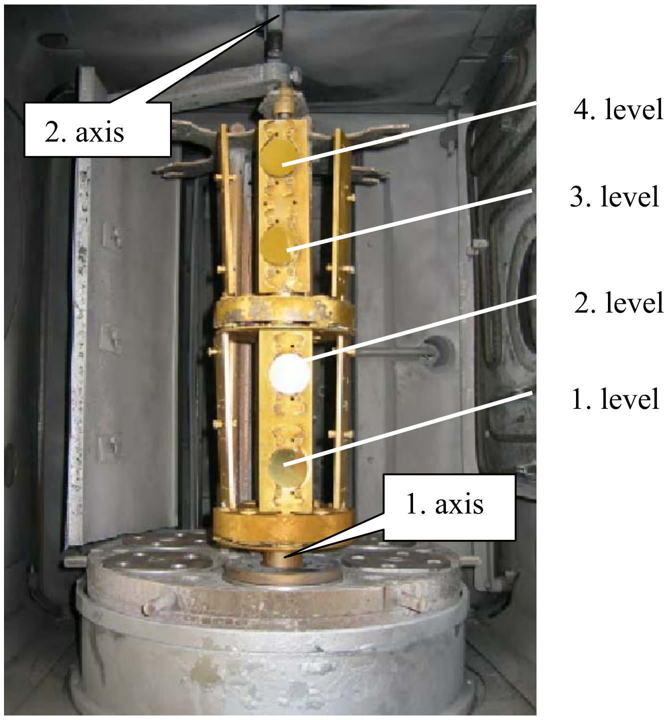


Fig. 2: Planetary rotational system with one pair of upper and lower substrate holders

By rotation around the second axis, the sample holders move automatically in defined positions in front of the Ti- or Al-target with holding times t_{Ti} = 240 s, t_{Al} = 350 s. Argon flow rate (F_{Ar} = 150 sccm), power (P = 3 kW), bias voltage (U_{BIAS} = 60 V), coil current (I_{coil} = 1 A) and temperature (T = 150 °C) were kept constant for Ti- and Al-deposition periods. In this case, and as proved for this set-up, deposition rate and corresponding layer thickness depend only on the mounting level of the substrates in the deposition chamber. Three sets of samples (deposition batches 436, 437, 439) have been prepared, using four mounting levels for substrates (Fig. 2). An example of a complete deposition sequence is provided in the appendix.

Each set of samples includes 16 reference materials (L100/#) for non-destructive testing by GIXRD and 4 batch reference samples (BL100/#) for additional destructive testing (cross-sections) and SEM inspection. Prior to cross-sectioning, batch reference samples (BL100/#) have been additionally coated with a TiN hard coating using PVD UBM sputtering at 150°C. The position of all individual samples on different mounting levels is given in Table 1.

Table 1: Sample positions and mounting levels

batch 436

4. level	L100/004	L100/008	L100/012	L100/016	BL100/580
3. level	L100/003	L100/007	L100/011	L100/015	BL100/559
2. level	L100/002	L100/006	L100/010	L100/014	BL100/527
1. level	L100/001	L100/005	L100/009	L100/013	BL100/506

batch 437

4. level	L100/020	L100/024	L100/028	L100/032	BL100/581
3. level	L100/019	L100/023	L100/027	L100/031	BL100/563
2. level	L100/018	L100/022	L100/026	L100/030	BL100/554
1. level	L100/017	L100/021	L100/025	L100/029	BL100/529

batch 439

4. level	L100/036	L100/040	L100/044	L100/048	BL100/583
3. level	L100/035	L100/039	L100/043	L100/047	BL100/546
2. level	L100/034	L100/038	L100/042	L100/046	BL100/545
1. level	L100/033	L100/037	L100/041	L100/045	BL100/544

3. Certification

3.1 Strategy of certification

The total layer thickness d_t was measured according to StAA-No. VIII.23-5 [6] and StAA-No. VIII.23-PM [7] at cross-sections using a calibrated SEM. The certification strategy was as follows:

1. All reference materials and batch reference samples have been tested non-destructively by means of grazing incidence X-ray diffraction (GIXRD). So, a fingerprint of stoichiometry, microstructure, density and (total) layer thickness(es) was taken. As shown earlier [5] for a VAMAS inter-laboratory comparison, a special evaluation procedure for GIXRD data allows the evaluation of the reproducibility of overall layer quality. Moreover, it has been shown that GIXRD data are sensitively correlated to the mounting level of samples in the deposition chamber.
2. For batch reference samples, destructive testing has been performed additionally. Preparation method (cross-sectioning) and measurement method (SEM) have been validated using reference SiO_2 coatings on Silicon. Prior to destructive testing, the layer thickness of SiO_2 coatings has been determined non-destructively and independently by spectroscopic ellipsometry (SE), BAM reference procedure [2], according to StAA-Nr. VIII.2901-V.2.01 [8]. Further details are given in sec. 3.2 and in the certification report BAM-L101.
3. The total layer thickness d_t of Ti/Al multilayers was measured at cross-sections of batch reference samples using validated and calibrated preparation and measurement methods described before.
4. The total layer thickness d_t determined for the batch reference samples of each mounting level was identified as the certified value of layer thickness d_{CRM} for samples of identical mounting levels.

3.2 Validation of the certification method

The preparation method (cross-sectioning) and the measurement method (SEM) have been additionally validated and re-calibrated using SiO_2 reference coatings on Silicon. An example of a cross-section of a SiO_2 reference coating on Si, coated with a TiN hard coating, is shown in Fig.3.

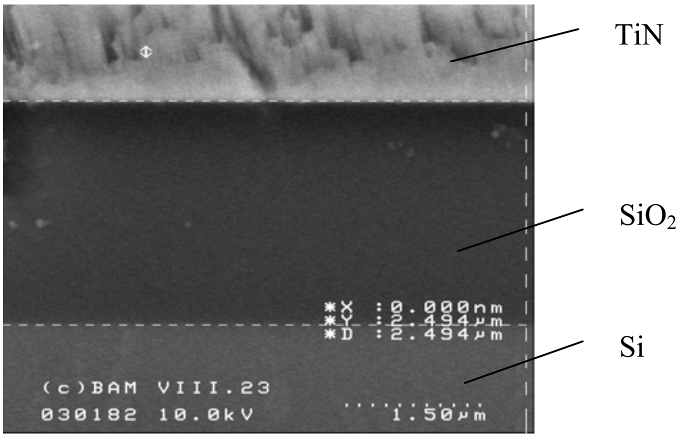


Fig.3: Cross-section of a SiO_2 reference coating on Si with TiN hard top coating

Table 2 provides 12 measurements of the layer thickness of SiO_2 at different points along the cross-section. The evaluation length was 5 µm for each measurement.

Table 2: Thickness of cross-sections of SiO_2 reference coatings (coated with TiN) on Si

measurement - ID	**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 uncertainty $u(d_{SiO_2})$	0.05

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

$$d_{SEM,SiO_2} = (2.48 \pm 0.05)\,\mu\text{m}. \qquad (1)$$

For the validation of the preparation method (cross-sectioning) and the certification method (SEM), this result was compared with the layer thickness of SiO_2 derived from SE using the BAM reference procedure [2] spectroscopic ellipsometry according to StAA-No. VIII.2901-V.2.01 [8]. From the analysis of ellipsometric data, the average layer thickness of the SiO_2 reference coating was derived to

$$d_{SE,SiO_2} = (2.51 \pm 0.01)\,\mu\text{m}. \qquad (2)$$

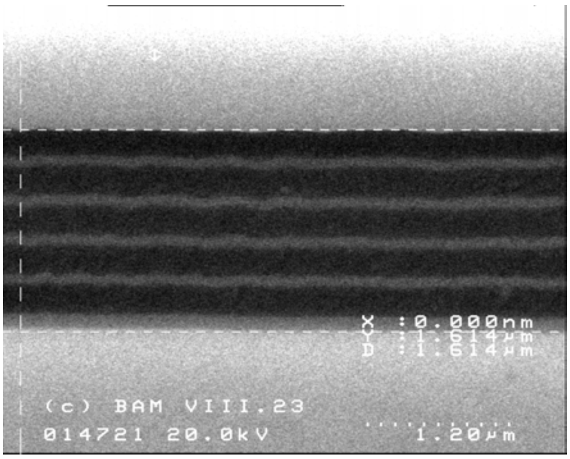
For the calculation of the certified total layer thickness, the difference $(d_{SEM,SiO_2} - d_{SE,SiO_2})$ was taken as constant correction (Δ_{corr} = 0.03 µm) of the TiN layer thickness data measured by SEM.

3.3 Certification measurements

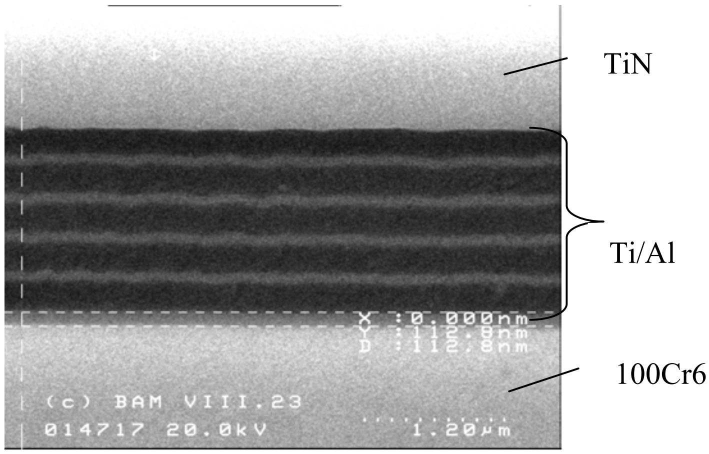
3.3.1 Total layer thickness of batch reference samples

As a result of the standard calibration procedure of the SEM, it was shown that the uncertainty of the thickness measurements $u(d_{SEM})$ is 10 nm for ideal samples. As an example, for the measurement of layer thickness by SEM, a cross-section of a Ti/Al batch reference sample coated with a TiN hard coating is shown in Figs. 4 a-d. The length of the evaluated cross-section was 5 µm.

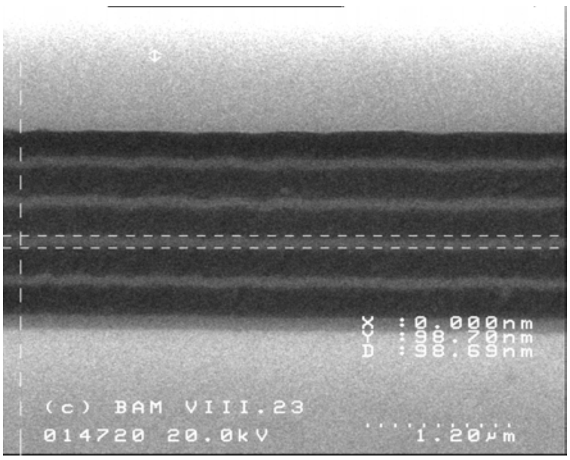
Fig.4: Measurement of the (total) layer thickness of a Ti/Al batch reference sample (coated with TiN hard coating)



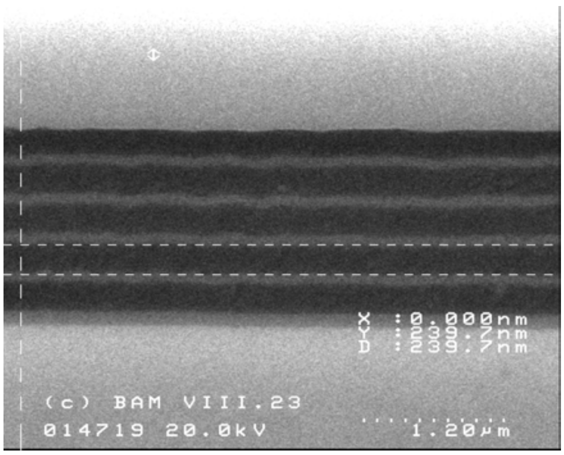
a) measurement of the total layer thickness: d_t = 1.61 µm



b) measurement of the layer thickness of the first Ti-layer: d = 0.11 µm



c) measurement of the layer thickness of the third Ti-layer: d = 0.10 µm



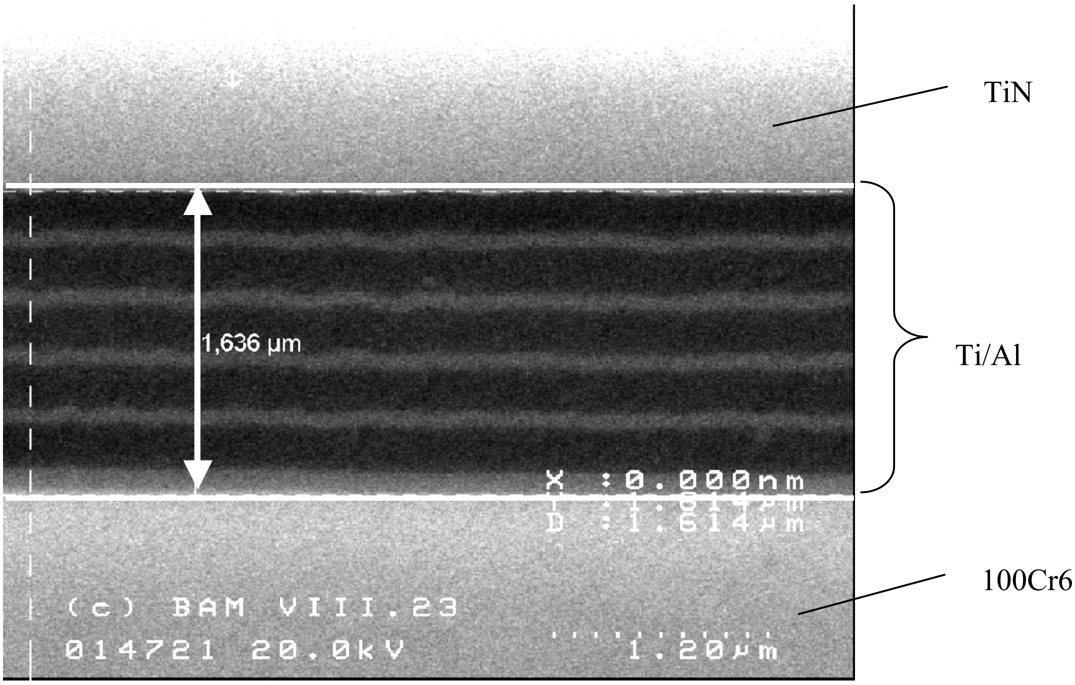
d) measurement of the layer thickness of the second Al-layer: d = 0.24 µm

Because of the high waviness of the individual coating interfaces along the measuring length of 5 µm (Fig. 4), the uncertainty $u(d_t)$ was calculated from the difference of the maximum $d_{\max}$ and minimum $d_{\min}$ of the total layer thickness [9] as follows:

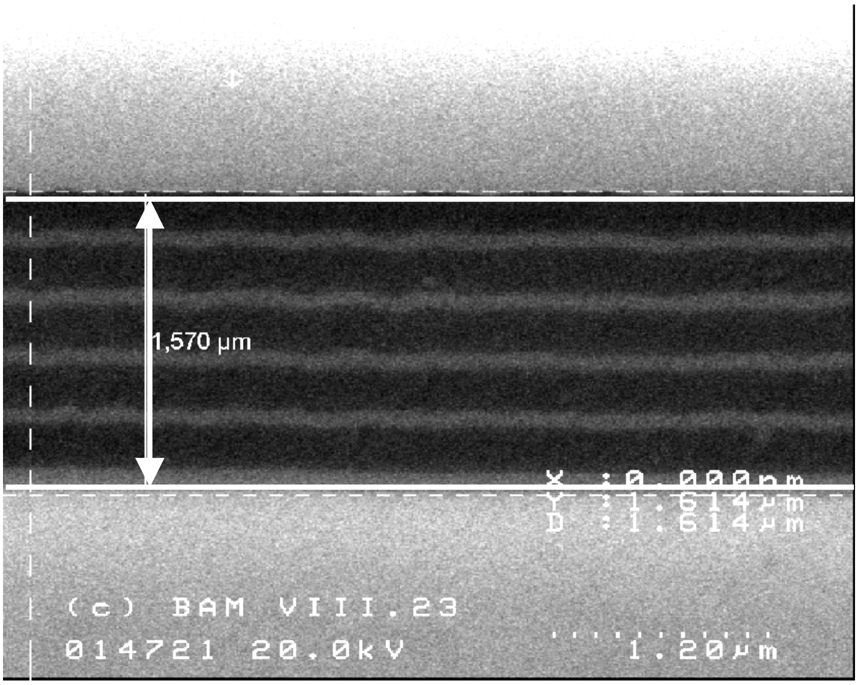
$$u(d_t) = (d_{\max} - d_{\min}) / 2\sqrt{3} \qquad (3)$$

An example of this estimation of $(d_{\max} - d_{\min})$ is shown in Fig. 5. For batch reference samples, $u(d_t)$ was estimated as follows: $u(d_t) = 0.035/\sqrt{3} = 0.02$ µm.

Fig. 5: Estimation of maximum layer thickness scatter $(d_{\max} - d_{\min})$



a) measurement of the maximum of total layer thickness: $d_{\max}$ = 1.64 µm (d_t =1.61 µm)



b) measurement of the minimum of the total layer thickness: $d_{\min}$ = 1.57 µm (d_t =1.61 µm)

3.3.2 Evaluation of batch homogeneity

Batch homogeneity has been evaluated non-destructively for each reference material and all batch reference samples by means of grazing angle X-ray diffraction (GIXRD) in terms of a fingerprint technique for stoichiometry, microstructure, density and (total) layer thickness. As described in more detail earlier [5], normalised intensity ratios were calculated for evaluation of the reproducibility of overall coating quality (Table 3).

Table 3: GIXRD-intensity ratios (batch 436)

level	sample ID	PI_n Ti/Al	i_n Ti (101) (39.69°)	i_n Al (220) (65.05°)	i_n Ti (103) (70.22°)	i_n Al (311) (78.24°)
1	L100/001	0.87	32.0	293	181	92.1
2	L100/002	1.01	58.8	301	151	107.0
3	L100/003	1.12	83.1	275	108	124.0
4	L100/004	0.99	79.5	222	98	97.8
1	L100/005	0.90	54.4	268	119	103.0
2	L100/006	1.06	61.8	315	133	128.0
3	L100/007	1.06	64.5	323	143	118.0
4	L100/008	0.91	59.1	268	112	101.0
1	L100/009	0.97	42.7	301	215	81.4
2	L100/010	1.08	55.6	304	215	91.0
3	L100/011	1.09	64.5	337	174	105.0
4	L100/012	0.91	56.9	283	133	90.1
1	L100/013	0.93	36.1	264	230	70.8
2	L100/014	1.09	56.8	316	178	115.7
3	L100/015	1.08	63.2	300	198	91.0
4	L100/016	0.93	47.4	299	167	91.6
1	BL100/506	0.95	54.5	294	120	109.0
2	BL100/527	1.00	75.6	286	123	125.0
3	BL100/559	1.12	63.2	327	179	110.0
4	BL100/580	0.95	52.8	270	160	91.6

A Seifert X-ray diffractometer XRD 3000 TT was used for GIXRD measurements. Each sample was analysed at an angle of incidence of 2° to the surface with sample rotation. The evaluation area was 1.5 cm². From the absolute intensity of reflexes (i_n in cps), the relative intensity deviation I_{nx} of identical reflexes was calculated with respect to the average of this reflex of all samples (equation 4), and the relative intensity deviation PI_n was calculated with respect to the average of the sums of normalised reflex intensities of all samples (equation 5). The data for each sample of batch 436 are given in Table 3.

$$I_{nx} = \frac{i_{nx} n}{\sum_n i_{nx}} \quad (4)$$

$$PI_n = \frac{\sum_x I_{nx} n}{\sum_n \sum_x I_{nx}} \quad (5)$$

The relative intensity deviations PI_n for each sample are shown in Fig. 6 in dependence on the mounting level for batch 436.

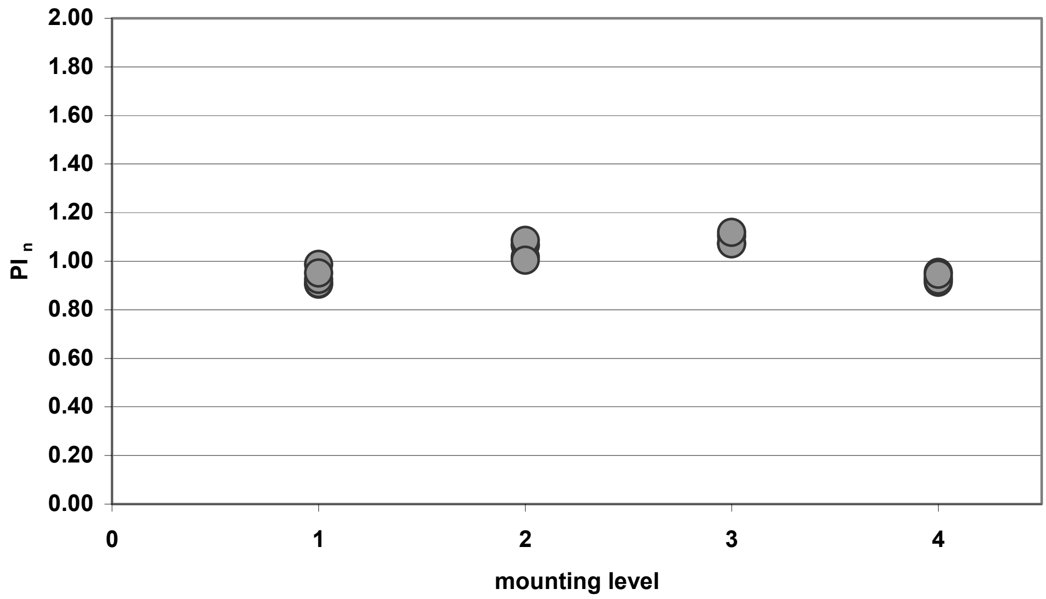


Fig. 6: Relative intensity deviations PI_n in dependence on the mounting level (batch 436)

Correlation coefficients between relative intensity PI_n and relative total layer thickness d_{tn} for batch reference samples are given in Table 4. The relative total layer thickness d_{tn} was normalised with respect to the average of the total layer thickness d_t of all batch reference samples.

Table 4: Correlation between relative intensity PI_n and normalised total layer thickness d_{tn}

batch/sample ID	level	d_{tn}	PI_n	correlation coefficients (d_{tn}, PI_n)
batch 436				**0.79**
BL100/506	1	0.96	0.95	
BL100/527	2	1.07	1.00	
BL100/559	3	1.07	1.12	
BL100/580	4	0.90	0.95	
batch 437				**0.93**
BL100/529	1	0.93	0.97	
BL100/554	2	1.10	1.07	
BL100/563	3	1.10	1.02	
BL100/581	4	0.87	0.94	
batch 439				**0.82**
BL100/544	1	0.93	1.04	
BL100/545	2	1.04	1.13	
BL100/546	3	1.10	1.11	
BL100/547	4	0.93	0.94	

Assuming a linear approximation between relative intensity PI_n and the total layer thickness d_t standard uncertainty of total layer thickness of levels $u(d_{XRD})$ was calculated. The data are given in Table 5.

Table 5: Standard deviations of relative intensity (PI_n) from average value of levels and standard uncertainty of total layer thickness of levels $u(d_{XRD})$

batch/level	average value of PI_n	standard deviation of PI_n	$u(d_{XRD})$ /µm
batch 436			
1	0.96	0.03	0.048
2	1.07	0.04	0.072
3	1.07	0.02	0.036
4	0.90	0.02	0.030
batch 437			
1	0.96	0.05	0.080
2	1.07	0.03	0.057
3	1.07	0.03	0.057
4	0.90	0.02	0.030
batch 439			
1	0.96	0.10	0.160
2	1.07	0.04	0.072
3	1.07	0.05	0.095
4	0.90	0.09	0.144

3.4 Summary of certification results

1. The standard calibration procedure of the SEM Hitachi S4100 (BAM StAA-No. VIII.23-PM1) results in an uncertainty of length measurements of $u(d_{SEM})$ = 10 nm.
2. The additional evaluation of the certification method SEM by means of SE as a standard-free reference method results in a constant correction Δ_{corr} = + 30 nm.
3. From cross-sections of the batch reference samples, the averaged values for the total layer thickness d_t have been derived by SEM. The standard uncertainty $u(d_t)$ was estimated to be 0.02 µm.

4. The mean value of the total layer thickness d_t with the standard uncertainty $u(d_t)$ of batch reference samples has been identified as the certified total layer thickness for reference samples of the same mounting level.
5. Consequently, the certified value for the total layer thickness can be written as follows:

$$d_{CRM} = d_t + \Delta_{corr} \qquad (6)$$

$$u_c(d_{CRM}) = \sqrt{[u(d_t)]^2 + [u(d_{SiO_2})]^2 + [u(d_{SEM})]^2 + [u(d_{XRD})]^2} \qquad (7)$$

$$U(d_{CRM}) = k * u_c(d_{CRM}), \qquad (k = 2) \qquad (8)$$

All certification results are summarised in Table 6.

Table 6: Certification results

sample ID	batch	level	d_{CRM} / µm	$U(d_{CRM})$ / µm, k = 2
L100/001	436	1	1.63	0.15
L100/002	436	2	1.83	0.18
L100/003	436	3	1.83	0.13
L100/004	436	4	1.53	0.12
L100/005	436	1	1.63	0.15
L100/006	436	2	1.83	0.18
L100/007	436	3	1.83	0.13
L100/008	436	4	1.53	0.12
L100/009	436	1	1.63	0.15
L100/010	436	2	1.83	0.18
L100/011	436	3	1.83	0.13
L100/012	436	4	1.53	0.12
L100/013	436	1	1.63	0.15
L100/014	436	2	1.83	0.18
L100/015	436	3	1.83	0.13
L100/016	436	4	1.53	0.12
L100/017	437	1	1.63	0.19
L100/018	437	2	1.93	0.16
L100/019	437	3	1.93	0.16
L100/020	437	4	1.53	0.12
L100/021	437	1	1.63	0.19
L100/022	437	2	1.93	0.16
L100/023	437	3	1.93	0.16
L100/024	437	4	1.53	0.12
L100/025	437	1	1.63	0.19

L100/026	437	2	1.93	0.16
L100/027	437	3	1.93	0.16
L100/028	437	4	1.53	0.12
L100/029	437	1	1.63	0.19
L100/030	437	2	1.93	0.16
L100/031	437	3	1.93	0.16
L100/032	437	4	1.53	0.12
L100/033	439	1	1.63	0.34
L100/034	439	2	1.83	0.18
L100/035	439	3	1.93	0.22
L100/036	439	4	1.63	0.31
L100/037	439	1	1.63	0.34
L100/038	439	2	1.83	0.18
L100/039	439	3	1.93	0.22
L100/040	439	4	1.63	0.31
L100/041	439	1	1.63	0.34
L100/042	439	2	1.83	0.18
L100/043	439	3	1.93	0.22
L100/044	439	4	1.63	0.31
L100/045	439	1	1.63	0.34
L100/046	439	2	1.83	0.18
L100/047	439	3	1.93	0.22
L100/048	439	4	1.63	0.31

4. Stability testing

The stability of the reference material has been evaluated by repeated GIXRD measurements after 600 days. Samples have been stored under ordinary laboratory conditions. One example of these measurements is shown in Fig. 7.

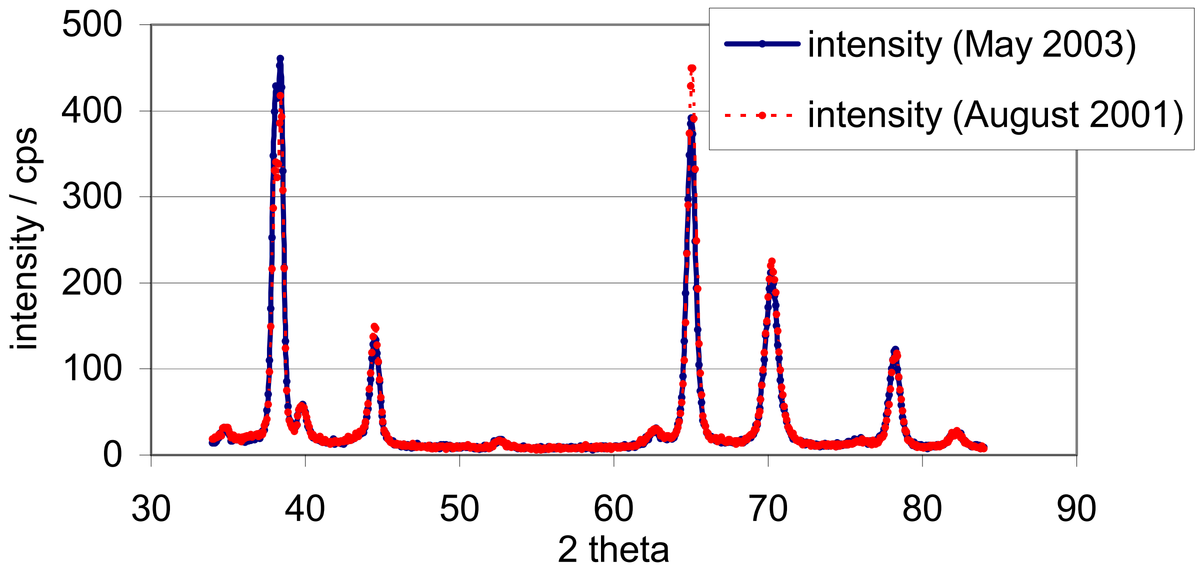


Fig.7: Repeated GIXRD measurements after 600 days of storage

From the agreement of both measurements, i. e. no significant difference in the derived GIXRD ratios, it was concluded that the samples are stable under ordinary laboratory conditions.

5. Instruction for use

For each CRM, a value of the total layer thickness, valid for the central surface area of a diameter of 25 mm, was certified. In case of use, it is a prerequisite to avoid any mechanical (such as scratching) or chemical treatment (such as aggressive agents) of the surface. Storage under ordinary laboratory conditions results in an unavoidable native oxide film of some nanometer film thickness.

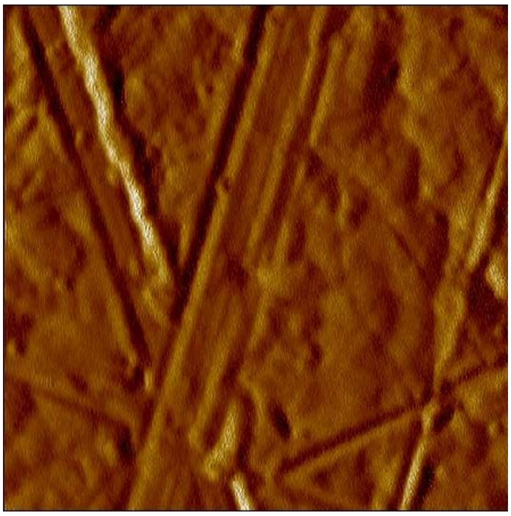
6. Additional information

6.1 Topography and roughness

The surface morphology of Ti/Al coatings is typical of the PVD deposition process [10, 11]. The Ti coatings have a columnar but relatively dense structure with minor defects or droplets, whereas the Al coatings show a dimple fracture morphology which is characteristic of a very ductile material.

The evaluation of roughness according to DIN EN ISO 4288 [3] with a mechanical profilometer has shown that there is no measurable difference in surface roughness between the coated and uncoated samples (R_a = 0.03 µm; R_z = 0.2 µm).

Moreover, the topography of the CRM´s was investigated by AFM for the characterisation of smaller surface areas (evaluation area was (5 x 5) µm^2). An example is given in Fig. 8.



R_a = 7.0 nm

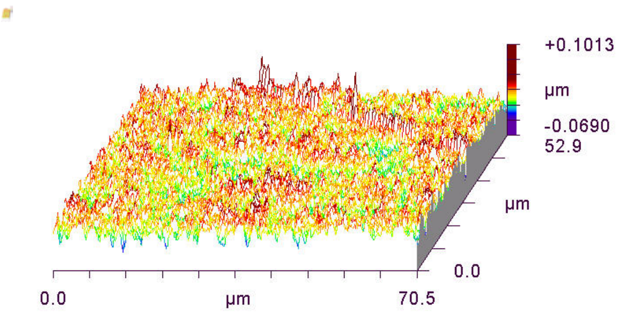
R_{max} = 68.8 nm

R_z = 57.1 nm

area : (5 x 5) µm²

Fig. 8: AFM-topography BAM-L100/007

In addition, WLI (evaluation area was (70 x 50) µm^2) has been used for the characterisation of larger surface areas. An example is shown in Fig. 9. Table 7 summarises the results of WLI measurements.



R_a= 0.01 µm

R_{max} = 0.31 µm

R_z = 0.19 µm

area : (70 x 70) µm²

Fig. 9: WLI-topography BAM-L100/007

Table 7: Summary of the WLI – measurements

no.	sample ID	batch / level	R_z / µm
1	L100/005	436/1	0.11
2	L100/006	436/2	0.13
3	L100/007	436/3	0.13
4	L100/008	436/4	0.23
5	L100/019	437/3	0.13
6	L100/035	439/3	0.38
		average value	**0.19**
		standard deviation	**0.10**

6.2 Mechanical properties

Mechanical properties (H_{IT}, E_{IT}) of BAM- L100/030 were determined by instrumented indentation tests (F_{max} = 1 mN, h_{max} = 130 nm, 100 indentations averaged over an area of (20 x 20) mm^2) according to DIN EN ISO 14577 [12] and StAA VIII.2901-V.1.11 [13] as follows:

$$H_{IT} = (2.9 \pm 0.4)\ \text{GPa}$$
$$E_{IT} = (108.6 \pm 22.8)\ \text{GPa}$$

Because the maximum indentation depth was roughly 1/10 of the total layer thickness, the values H_{IT} and E_{IT} describe the effective plastic-elastic behaviour of the entire multilayer.

7. References

[1] DIN EN ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories (2000-04)

[2] Testing and Chemical Analysis. Catalogue of reference procedures provided by BAM. Berlin, April 2001 (ISSN 1617 – 6634)

[3] DIN EN ISO 4288, Geometrical Product Specifications (GPS) - Surface texture: Profile method - Rules and procedures for the assessment of surface texture (1998-04)

[4] Stahlschlüssel, Verlag Stahlschlüssel Wegst GmbH (1998) ISBN 3-922599-14-1

[5] U. Beck. Th. Fritz. N. Gamer. Th. Wirth, VAMAS project in TWA 2 "Evaluation of multilayer reference coating for quantitative GD-OES ", BAM FB No. 242 (2001), ISBN: 3-89701-713-X

[6] StAA-no. VIII.23-5

[7] StAA-no. VIII.23-PM

[8] StAA-no. VIII.2901-V.2.01

[9] "Guide to the expression of uncertainty in measurement (GUM)", International Organization For Standardization (1993), ISBN: 92-67-10188-9

[10] C. Charrier. P. Jacquot. E.Denisse. J.P.Millet. H. Mazille "Aluminium and Ti/Al multiplayer PVD coatings for enhanced corrosion resistance". Surface and Coating Technology 90 (1997) 29-34

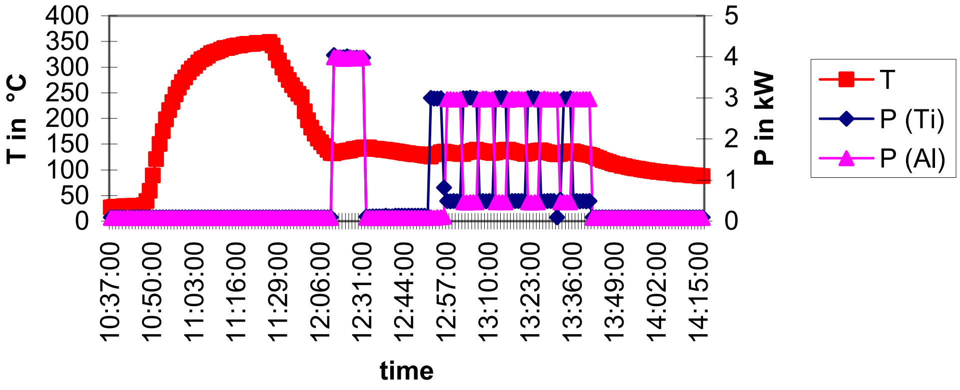
[11] J. Creus. H. Indrissi. H.Mazille. F. Sanchette. P. Jacquot „Corrosion behaviour of Al/Ti coating elaborated by cathodic arc PVD process onto mild steel substrate". Thin Solid Films 346 (1999) 150-154

[12] DIN EN ISO 14577, Metallic materials - Instrumented indentation test for hardness and materials parameters - Part 1 (2003-05)

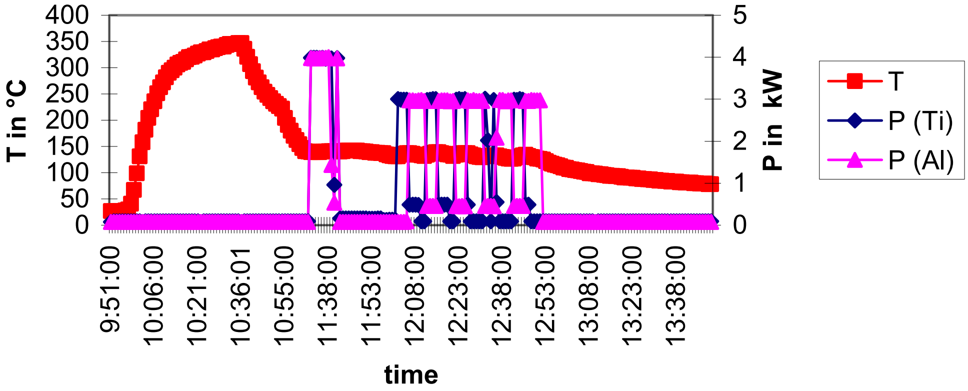
[13] StAA-no. VIII.2901-V.1.11

Appendix: Information on preparation

batch 436 Ti/Al



batch 437 Ti/Al



batch 439 Ti/Al