Federal Institute for Materials Research and Testing

CERTIFIED REFERENCE MATERIAL BAM-L101

TiO_2/SiO_2 multilayer on BK7 glass

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Certified quantity:
The batch value for the total layer thickness d_t of TiO_2/SiO_2 multilayer coatings on BK7 glass substrates.

Certified quantity	Certified value, nm	Uncertainty, nm k = 2
d_t	**964**	**24**

Informative values:

A: Surface topography and roughness
The roughness in micrometer range was evaluated by white light interference microscopy (WLIM). The area investigated was (70 x 70) µm², the determined statistical roughness values are

roughness average R_a = 4.6 nm, and
mean roughness depth R_z = 53.8 nm.

B: Single layer thicknesses
Spectroscopic ellipsometry (SE) was used for the determination of d_t. Based on a model, individual layer thicknesses of the multilayer stack have been derived. The following table provides these data from layer 1 on the substrate to layer 10 on top of the layer stack.

	layer no										
	1	2	3	4	5	6	7	8	9	10	sum
thickness/ nm	93,6	91,5	92,1	106,2	93,5	92,4	93,7	101,7	91,4	108,4	964,5

C: Optical properties and depth profiling
In addition, the reference material has been characterised by optical transmission spectroscopy and depth profiling techniques as given in the certification report.

DESCRIPTION OF THE SAMPLE

TiO_2/SiO_2 multilayer coatings (5 x TiO_2/SiO_2) with nominal individual layer thicknesses of 100 nm and 1 µm nominal total layer thickness have been deposited on polished Schott BK7 glass substrates, squares of (50 x 50) mm^2 size and 1.1 mm thick, using an electron beam evaporation process. The deposition sequence starts with TiO_2 (layer on the substrate) and ends up with SiO_2 (top layer of layer stack). To improve adhesion, the substrate has been coated with a 60 nm SiO_2 inter-layer which is not part of the certified layer system.

INSTRUCTION FOR USE

TiO_2/SiO_2 multilayer coatings are intended to be used for the evaluation and calibration of depth measurements and depth resolution of surface analytical methods (e.g. glow discharge optical emission spectroscopy, GD-OES) and for the evaluation and calibration of metallographic preparation methods (e.g. cross-sectioning, ball grinding).

For the batch, a mean value of d_t valid for the central surface area of a size of (45 x 45) mm^2 was certified. This was done for a surface topography as measured by WLIM.

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 film of adsorbed water of some nanometer thickness that may be considered for evaluation.

ANALYTICAL METHODS USED

The determination of the certified value was performed by means of a validated and calibrated spectroscopic ellipsometry measurement according to PAS 1022: "Reference Procedure for Determination of Optical and Dielectric Material Properties and Thickness of Thin Films by Ellipsometry", Beuth Verlag Berlin 2004. The certification procedure was conducted in a laboratory of BAM accredited according to DIN EN ISO/IEC 17025 (DAP-PL-2614.08).

DATA EVALUATION

1. All samples of the batch have been tested non-destructively by means of spectroscopic ellipsometry.
2. For batch reference samples, destructive testing has been performed as well. Preparation method was focused ion beam (FIB) slice sectioning, after which transmission electron microscopy (TEM) imaging and diffraction pattern measurement have been performed.
3. The total layer thickness d_t of the TiO_2/SiO_2 multilayer stack was measured at the centre point of each sample by ellipsometry.
4. Measurements at cross-sections near the centre point of batch reference samples using validated and calibrated preparation and measurement methods (SEM, TEM) were performed.
5. An overall statistical treatment was performed on the batch to determine the batch homogeneity of d_t.
6. The total uncertainty of d_t includes the following components: standard deviation of the mean batch value (52 samples), additional standard deviation from intra-sample inhomogeneity, and additional standard deviation from the model uncertainty. According to the certification report, this results in a total uncertainty for d_t of 24 nm.

SHELF LIFE

Provided the sample is stored and handled appropriately, the certification will remain valid for 24 months from the date of shipment.

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