Federal Institute for Materials Research and Testing

# CERTIFIED REFERENCE MATERIAL
# BAM-L104 / 050

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## TiC single layer on 100Cr6 steel

**Certified property:**

Layer thickness $d_{CRM}$ of TiC coatings on 100Cr6 steel

| Certified quantity | Certified value, µm | Uncertainty, µm (k = 2) |
|---|---|---|
| $d_{CRM}$ | 2.57 | 0.17 |

**Informative values:**

***A: Surface topography and roughness***

The evaluation of roughness according to DIN EN ISO 4287/4288 by a mechanical profilometer (evaluation length 4 mm) 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.
($R_a$ - roughness average, $R_z$ - mean roughness depth)

The evaluation of roughness by WLI (White Light Interferometry), evaluation area (70 x 70 ) µm², gives $R_a$ = 0.02 µm and $R_z$ = 1.3 µm.

The evaluation of roughness by AFM (Atomic Force Microscopy), evaluation area (5 x 5) µm², gives $R_a$= 10 nm and $R_z$ = 80 nm.

***B: Mechanical properties***

The determination of $H_{IT}$ ($H_{IT}$-indentation hardness) and $E_{IT}$ ($E_{IT}$-indentation modulus) by instrumented indentation tests ($F_{max}$ = 10 mN, $h_{max}$ = 160 nm, 100 indentations averaged over an area of (20 x 20) $mm^2$) according to DIN EN ISO 14577 gives $H_{IT}$ = (30 ± 8) GPa and $E_{IT}$ = (334 ± 72) GPa.

***DESCRIPTION OF THE SAMPLE***

TiC coatings with a nominal layer thickness of 2.5 µm have been deposited on polished 100Cr6 steel substrates (discs of 30 mm in diameter and 4 mm thickness) using a PVD UBM sputtering process. To improve adhesion, a Cr inter-layer with a layer thickness of approximately 100 nm was used.

## *INSTRUCTION FOR USE*

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

## *ANALYTICAL METHODS USED*

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 performed in a laboratory of BAM accredited according to DIN EN ISO/IEC 17025 (DAP-PL-2614.08).

## DATA EVALUATION

1. For all batch reference samples, destructive testing has been performed. 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).
2. The average layer thickness $d_a$ of TiC and the standard uncertainty $u(d_a)$ were calculated from 5 measurements at different points along the cross-section of the batch reference samples using validated and calibrated preparation and measurement methods described before.
3. For the calculation of the certified layer thickness $d_{CRM}$, the difference $(d_{SE,SiO_2} - d_{SEM,SiO_2})$ was taken as constant correction ($\Delta_{corr} = 0.03$ µm): $d_{CRM} = d_a + \Delta_{corr}$.
4. All samples of the mounting levels with the highest respectively lowest deposition rate have been tested non-destructively by means of surface acoustic wave (SAW) technique. So, a fingerprint of layer thickness, Young´s modulus, Poisson's ratio and density was taken. The upper bound of the standard uncertainty of the layer thickness within mounting level with the highest deposition rate was taken as constant $u(d_{SAW}) = 0.06$ µm.
5. The average layer thickness $d_a$ determined for the batch reference samples of each mounting level was identified as the certified value of coating thickness $d_{CRM}$ for samples of identical mounting levels.

## *SHELF LIFE*

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

## *LEGAL NOTICE*

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