Federal Institute for Materials Research and Testing

CERTIFIED REFERENCE MATERIAL
BAM-L100 / 001

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Ti/Al multilayer on 100Cr6 steel

Certified property:
Total layer thickness d_{CRM} of Ti/Al multilayer coatings on 100Cr6 steel

Certified quantity	Certified value, µm	Uncertainty, µm (k = 2)
d_{CRM}	1.63	0.15

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= 0.01 µm and R_z = 0.19 µm.

The evaluation of roughness by AFM (Atomic Force Microscopy), evaluation area (5 x 5) µm², gives R_a= 7.0 nm and R_z = 57.1 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} = 1 mN, h_{max} = 130 nm, 100 indentations averaged over an area of (20 x 20) mm^2) according to DIN EN ISO 14577 gives H_{IT} = (2.9 ± 0.4) GPa and E_{IT} = (108.6 ± 22.8) GPa.

DESCRIPTION OF THE SAMPLE

Ti/Al multilayer coatings (5 x 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 the layer stack).

INSTRUCTION FOR USE

Ti/Al multilayer 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, an individual value of the total layer thickness, valid for the central surface area of 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. 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.
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).
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 standard uncertainty of the total layer thickness data $u(d_t)$ measured by SEM was calculated from the difference of the maximum $d_{\max}$ and the minimum $d_{\min}$ of the total layer thickness: $u(d_t) = (d_{\max} - d_{\min}) / 2\sqrt{3}$.
5. For the calculation of the certified total layer thickness, the difference $(d_{SE,SiO_2} - d_{SEM,SiO_2})$ was taken as constant correction ($\Delta_{corr} = 0.03$ µm): $d_{CRM} = d_t + \Delta_{corr}$.
6. The total layer thickness d_t determined for the batch reference samples of each mounting level was identified as the certified value of the total layer 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.

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Berlin, 27. January 2004

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