Thin Film Characterisation Methods

Technologies
- Hardness/Adhesion
- Nanoindentation
- Plasma Diagnostics and Particle Flux Analysis
- Raman Spectroscopy
- Film Thickness
- Magnetometer (VSM) and High Frequency Permeameter
- X-ray Diffraction XRD/XRR

Hardness / Adhesion

Short technology description

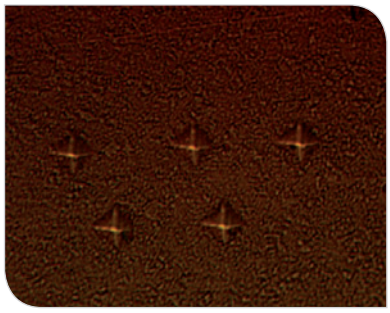
The film hardness can be determined by Vickers indentation with a diamond tip. The geometry of the indentation is measured by an attached optical microscope and delivers the dimensions of indentation and therefore the value of hardness.
A diamond stylus (Rockwell geometry) is scratching with linear increasing load over the coated surface. The load value at which the film begins to fail can be defined. This value is a criterion for the film adhesion. In order to optimize the substrate-film system the origin of the failure can be evaluated by the type of failure, film failure ore interface failure.

Special features
- Vickers hardness material independent
- Scratch test – for brittle thin film material

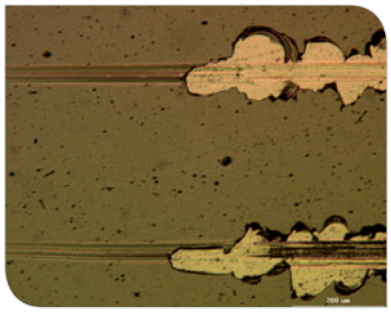
Limitations/constrains
- Microhardness indentation load 5 g > L > 400 g
- Critical load of failure Load 0 < L < 200g

Material examples
- For hardness determination the indentation depth must be 1/10 or lower of the film thickness

Typical samples



Indentation for the determination of the hardness (load 50 g)



Trace of the scratch test for the determination of the critical load of failure

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Technology

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Characterisation

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Material class

Silicon	Polymer	Metal	Ceramic
		X	X
Glass	Organic	Other	
X			

Nanoindentation

Short technology description

Nanoindentation is the most important method for the nanoscale measurement of mechanical properties (hardness, elastic modulus, fracture toughness) of surfaces, thin films and small volumes of material. A pyramidal diamond tip is pressed into the specimen as an indenter and removed again after reaching a maximum load. During this process, the load and the penetration depth of the indenter are recorded. This load/penetration depth curve represents a "finger-print" of the mechanical properties averaged over a certain volume area that increases with increasing load. The curve fitting allows the extraction of the mechanical properties.

Special features

- Force range: 0.5 µN–10 mN
- Spatial resolution of indents < 50nm
- In-situ imaging on a maximum scan range of 100 µm x 100 µm + automated measurement of up to 100 indents possible by combination of Dimension 3100 AFM and Hysitron TriboScanner
- Recording of depth profiles of hardness and elastic modulus possible by using small-angle cross section method (SACS) after special sample preparation by nanogrinding

Limitations/constrains

- Problems associated with the „pile-up" or „sink-in" of the material on the edges of the indent during the indentation process remain a problem that is still under investigation
- International standard only for metals available: ISO 14577 1–3 (2002)
- Thermal drift, zero point correction, machine compliance determination
- 1.5 – 2* tip radius < film thickness

Design rules

- For thin films on substrates the substrate influence on the hardness can be neglected if the indentation depth is < 1/10 of the film thickness
- For thin films on substrates there is practically no minimum indentation depth to fully exclude the substrate influence on the reduced elastic modulus

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Material class

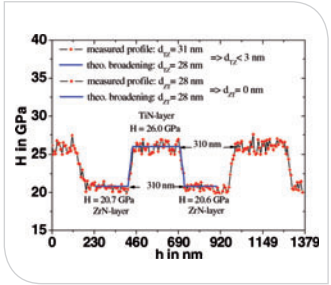
Silicon	Polymer	Metal	Ceramic
X	X	X	X

Glass	Organic	Other
X	X	

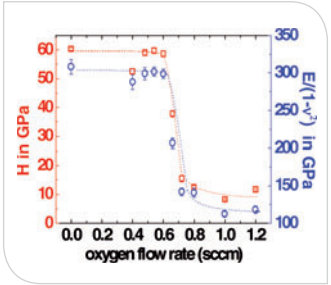
Further publications

[1] C. Ziebert, S. Ulrich, Hard multilayer coatings containing TiN and/or ZrN – A review and recent progress in their nanoscale characterization, Journal of Vacuum Science & Technology A 24(3) 554–583 (2006)
[2] J. Ye, S. Ulrich, C. Ziebert, M. Stüber, Stress reduction of cubic boron nitride films by oxygen addition, Thin Solid Films 517, 1151–1155 (2008)
[3] C. Ziebert, K.-H. Zum Gahr, Ortsaufgelöste Messung mikrotribologischer Eigenschaften mehrphasiger Oxidkeramiken mit der Rastersondenmikroskopie, Materialwissenschaft und Werkstofftechnik 35, 785–793 (2004)
[4] J.-K. Park, C. Ziebert, M. Stüber, Y.-J. Baik, Improvement of Hardness and Toughness of TiAlN Coating by Nanoscale Multilayered Structurization with Si_3N_4, Plasma Processes and Polymers, 4, S902–S905 (2007)
[5] C. Ziebert, J. Ye, K. Sell, S. Ulrich, High-resolution depth profiling of mechanical properties of thick cubic boron nitride coatings, Surface & Coatings Technology 200, 6454–6458 (2006)

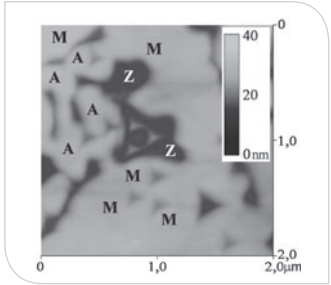
Typical samples



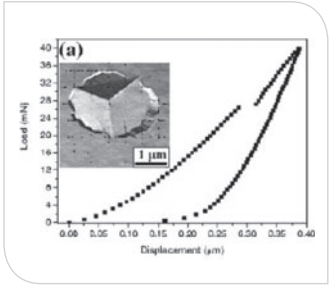
Hardness depth profile of a 20-layer TiN/ZrN nanolaminated composite coating recorded at 2 mN load by small-angle cross section method (SACS) [1]



Dependence of hardness and reduced elastic modulus of 100 nm thick c-BN:O films on the oxygen flow rate measured at 700 µN load [2]



AFM topography image of eutectic Al_2O_3-ZrO_2 region in a laser-modified alumina ceramic showing nanoindents (maximum load: 1 mN; M = Al_2O_3 matrix, A = Al_2O_3 lamella, Z = ZrO_2 lamella) [3]



Load–displacement curve and AFM image (insert) of the cracking of a TiAlN single-layered coating for the calculation of fracture toughness [4]

Plasma Diagnostics and Particle Flux Analysis

Short technology description

Low pressure plasmas can be characterized by electrical single and double probes and optical emission spectroscopy. The particle fluxes onto the substrates can be determined by Faraday cup as well as retarding field analyzer. Based on the measurements the following physical quantities are calculated: electron temperature Te, plasma density ne, ion and electron current density jion and je, plasma potential Upl, ion energy Eion, energy distribution of ions f(Eion).

Special features

- Faraday cup/retarding field analyzer: 0–1000 eV
- Single- and double probe: plane and cylindrical
- Optical emission spectrometer: 200 – 800 nm

Limitations/constrains

- Height: 50 mm, diameter: 80 mm
- Temperature range: 0 °C–200 °C
- KF40 flange utilizable

Design rules

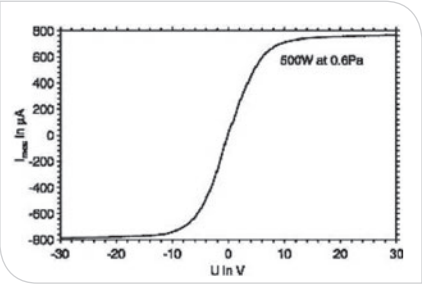
- The hole system is transportable

Further publications

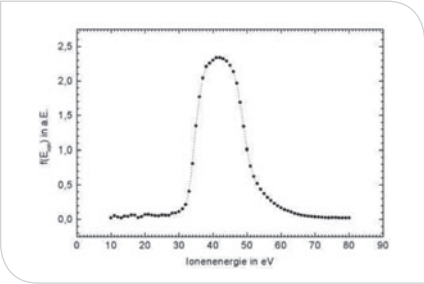
[6] Kratzsch, S. Ulrich, H. Leiste, M. Stüber, H. Holleck, Surf. Coat. Technol. 116-119 (1999) 949-955

[7] T. Nguyen, S. Ulrich, J. Bsul, S. Beauvais, W. Burger, A. Albers, M. Stüber, J. Ye, Diam. Relat. Mater. (2009) in press, online available

Typical samples

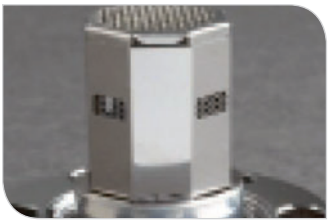


Current–voltage-characteristic of a double probe measurement (r.f. magnetron argon plasma)



Energy distribution of Ar-ions during film growth by magnetron sputtering

Retarding field analyzer for measurement of plasma potential, ion energy and energy distribution ions



Micropole mass spectrometer mounted on KF16 flange

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Material class

Silicon	Polymer	Metal	Ceramic
X	X	X	X
Glass	**Organic**	**Other**	
X			

Raman Spectroscopy

Short technology description

By laser excited Stokes Raman scattering the chemical bonding and microstructure of compact, powder and thin film materials are evaluated.
In solid state physics, Raman spectroscopy has become an important method to investigate carbon species and a-C: metal nanocomposites. Raman spectroscopy is also suitable for the microscopic examination of minerals, polymers and ceramics, but also of cell and proteins. Recently, the Raman spectroscopy could be used for measuring concentration profiles within micro channels.

Special features

- Backscattering geometry
- Excitation with Argon-Ion-Laser (514.5 nm)
- Excitation with Helium-Cadmium-Laser (325 nm)
- Holographic grating 2400 grooves mm^{-1}
- UV-enhanced CCD detector with thermo-electric cooling

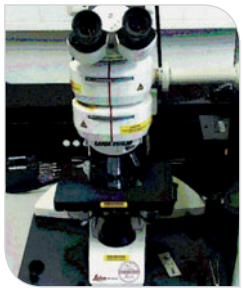
Limitations/constrains

- Spectral resolution 1–2 cm^{-1}
- Spectral range (Raman shift) < 200–4000 cm^{-1}
- Spatial resolution 2 µm lateral (x 50 objective)
- Confocal mode possible

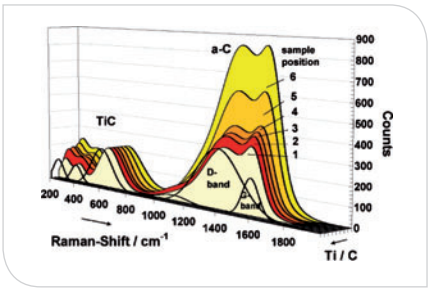
Design rules

- Typical sample: flat, thin film, fine powder

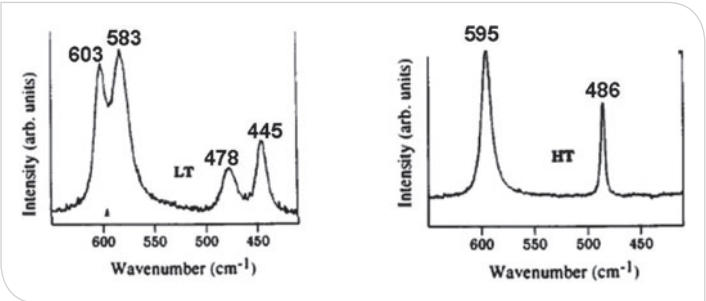
Typical samples



Optical arrangement for Micro Raman spectroscopy



Raman spectra of six samples with different Ti:C ratios deposited using -150 V substrate bias voltage



Characterisation of $LiCoO_2$ phases

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Material class

Silicon	Polymer	Metal	Ceramic
X	X	X	X

Glass	Organic	Other
X		

Film Thickness

Short technology description

The film thickness can be determined by calo-test, where a spherical calotte will be polished into the surface. The sectional view diameters provide, together with the geometry of the grinding sphere, the value of the film thickness.
By the use of a surface profilometer (Type Tencor P10), the roughness, waviness, step size of uncoated/coated regions and curvature of bending can be measured by the stylus method with a diamond tip.

Special features

- Calo-test is a local destructive methode
- Roughness values like Ra,Rt, Rz will be given

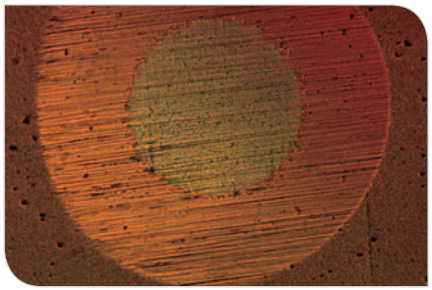
Limitations/constrains

- Calo-test film thickness 500 nm < t < 5 µm; min. film area 1mm
- Profilometer film thickness 20 nm < t < 5 µm; max. scan length 150 mm

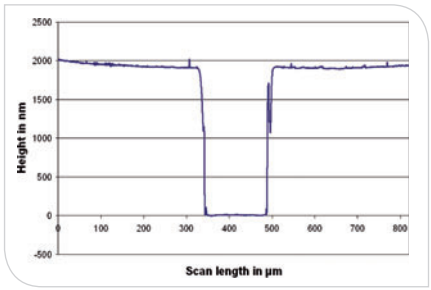
Design rules

- Resolution and accuracy depends on roughness

Typical samples



A calotte polished into a coated substrate



Surface profile of a partial coated substrate

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Material class

Silicon	Polymer	Metal	Ceramic
		X	X
Glass	Organic	Other	
X			

Magnetometer (VSM) and High Frequency Permeameter

Short technology description: For the characterisation of ferromagnetic thin films properties especially the determination of magnetic hysteresis behaviour a vibrating sample magnetometer can be used to determine the saturation magnetisation Ms, the coercivity Hc, permeability μ and anisotropy Ha.
The high frequency permeability with real and imaginary (damping) part can be determined by the use of a coplanar measuring head in a frequency range of 0.05 < f < 5GHz.

Special features

- Vibrating sample magnetometer for thin film samples (max field 20mT)
- High frequency permeameter 0.05 < f < 5GHz

Limitations/constrains

- Vibrating sample magnetometer: Min film thickness min 20nm

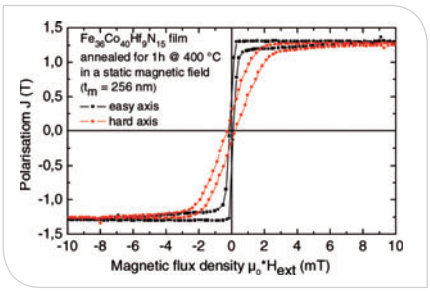
Design rules

- Vibrating sample magnetome-ter typical sample: flat, thin film, size 5 x 5 x 0,5 mm³
- High frequency permeameter, sample size 5 x 5 x 0,5 mm³

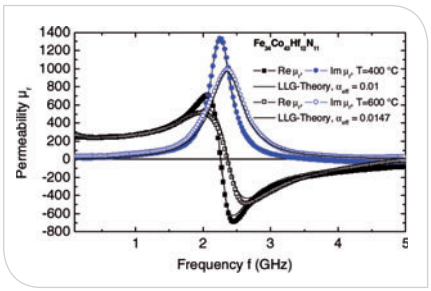
Typical samples

Vibrating sample magneto-meter

The high frequency permeameter consists of a coplanar measuring head on an attached vector network analyzer



Magnetic hysteresis of a thin ferromagnetic thin film with uniaxial anisotropy (weak (black) and strong (red) direction)



Frequency dependent permeability; real part (black) and imaginary part (blue)

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Material class

Silicon	Polymer	Metal	Ceramic
		X	X
Glass	**Organic**	**Other**	
X			

X-Ray Diffraction XRD/XRR

By means of X-ray radiation (Cu-cathode) the microstructure of powder, compact and thin film material can be measured and evaluated. Different geometries, and circle movements (Bragg-Brentano-geometry, GID, texture, X-ray reflectivity) can be realized. It can be decided if the material is amorphous or crystalline, and the lattice parameters can be determined from the line positions.

Special features

- Bragg-Brentano
- Texture
- XRR-characterisation
- Cu-X-ray tube
- Optional monochromator
- Compact material, thin films, powder samples

Limitations/constrains

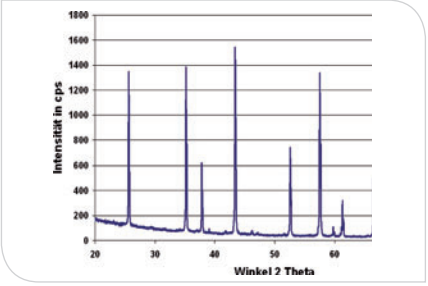
- Resolution 1/10000°
- Max. sample load 5 kg
- Min. beam Ø2mm

Design rules

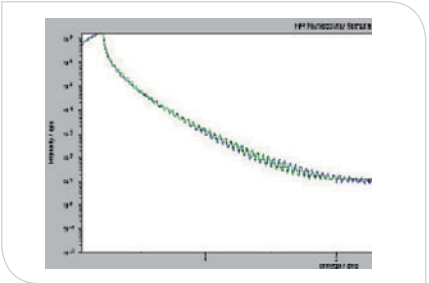
- XRR typical sample: flat, thin film, size 20x20x1mm^3
- Min sample size 5x5mm^2
- Max sample size Ø150mm x 20mm

Typical samples

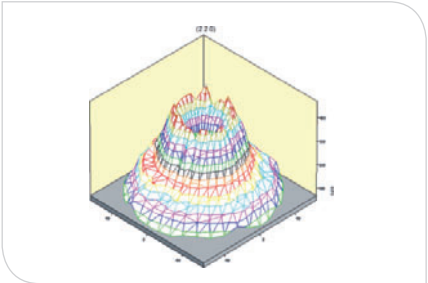
4 circle goniometer for XRD characterisation (Seifert 3003 HR)



Bragg-Brentano diagram of Al_2O_3-sample



XRR-diagram of a thin film for density determination



Texture diagram of TiN thin film

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Material class

Silicon	Polymer	Metal	Ceramic
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Glass	**Organic**	**Other**	
X			