Thin Film Characterisation Methods

(1) Hardness/adhesion

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 or interface failure.

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features

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

Limitations/constraints

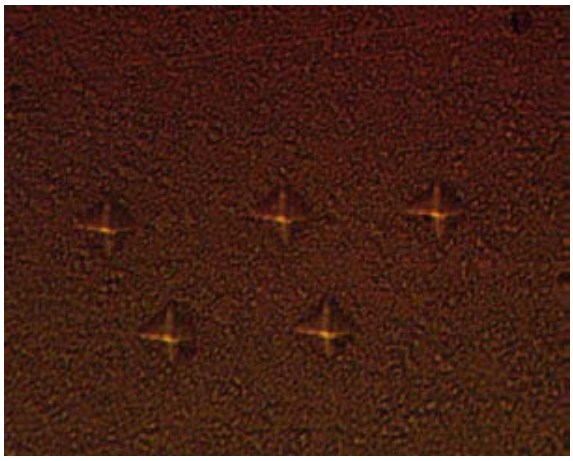
- Microhardness indentation load 5 g > L > 400 g
- Critical load of failure Load 0 < L < 200g
- For hardness determination the indentation depth must be 1/10 or lower of the film thickness

Materials

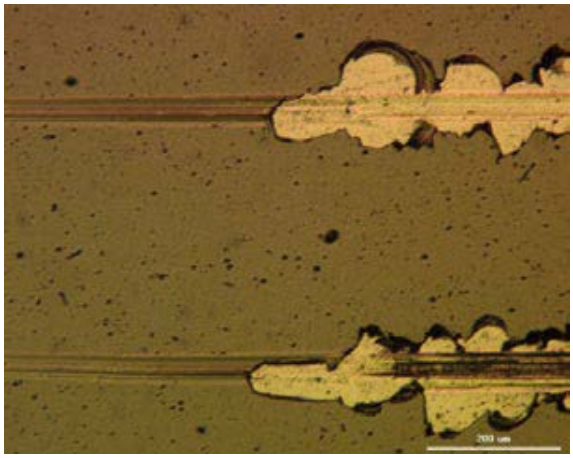
Metals, alloys, ceramic, oxides, nitrides, carbides, glass

(1) Hardness/adhesion *(continued)*

Typical structures and results



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



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

Karlsruhe Nano Micro Facility (KNMF)
Karlsruhe Institute of Technology (KIT)
Hermann-von-Helmholtz-Platz 1
76344 Eggenstein-Leopoldshafen
Germany

User Office
Phone: +49 (721) 608-23123
Fax: +49 (721) 608-26273
Email: knmf-useroffice@kit.edu
Web: www.knmf.kit.edu

Thin Film Characterisation Methods

(2) Nanoindentation

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.

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features

- Force range: 10mN – 10N
- Optical system max magnification 2000x
- Min distance between indents < 250nm
- Recording of depth profiles of hardness and elastic modulus possible after special sample preparation by nanogrinding

Materials

- Polymer, metal, ceramic, glass, silicon, organic

Limitations/constraints

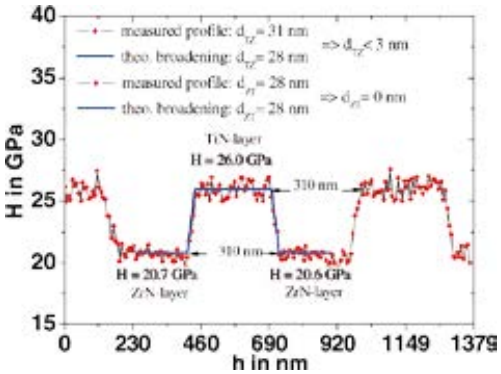
- The surface roughness is a crucial parameter and should be < 10nm
- Indentor geometry Vickers
- Depth resolution 0.3nm
- For thin films on substrates the substrate influence on the hardness can be neglected if the indentation depth is < 1/10 of the film thickness
- For thin films on substrates there is practically no minimum indentation depth to fully exclude the substrate influence on the reduced elastic modulus

(2) Nanoindentation *(continued)*

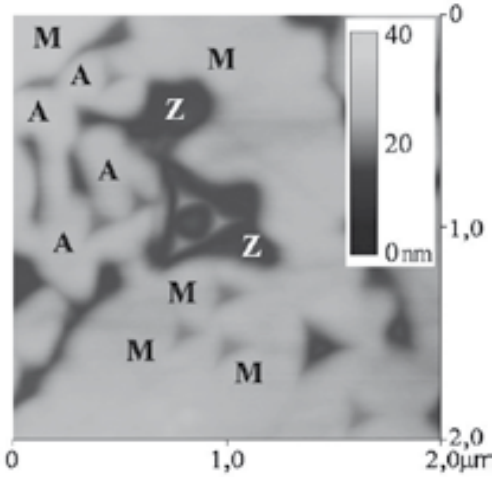
Thin Film Characterisation Methods

(1) Hardness/adhesion
(2) Nanoindentation
(3) Plasma diagnostics and particle flux analysis
(4) Raman spectroscopy
(5) Film thickness
(6) Magnetometer (VSM) and high frequency permeameter
(7) X-ray diffraction XRD/XRR

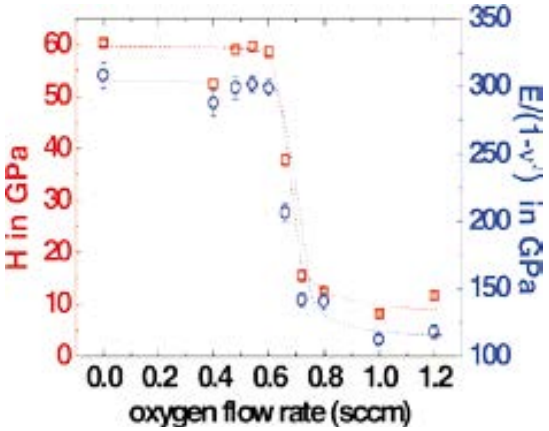
Typical structures and results



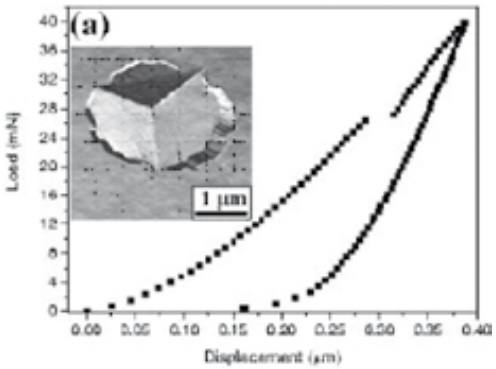
Hardness depth profile of a 20-layer TiN/ZrN nanolaminated composite coating recorded at 2 mN load by small-angle cross section method (SACS)



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)



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



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

Karlsruhe Nano Micro Facility (KNMF)
Karlsruhe Institute of Technology (KIT)
Hermann-von-Helmholtz-Platz 1
76344 Eggenstein-Leopoldshafen
Germany

User Office
Phone: +49 (721) 608-23123
Fax: +49 (721) 608-26273
Email: knmf-useroffice@kit.edu
Web: www.knmf.kit.edu

(3) Plasma diagnostics and particle flux analysis

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).

Thin Film Characterisation Methods

(1) Hardness/adhesion
(2) Nanoindentation
(3) Plasma diagnostics and particle flux analysis
(4) Raman spectroscopy
(5) Film thickness
(6) Magnetometer (VSM) and high frequency permeameter
(7) X-ray diffraction XRD/XRR

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

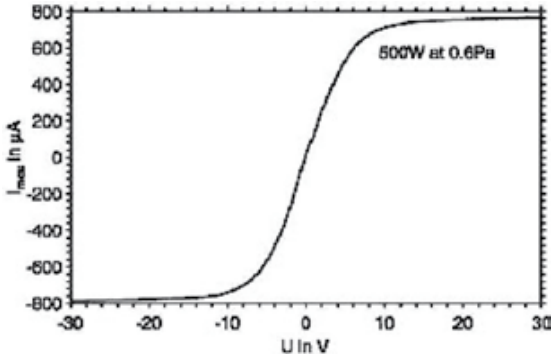
Features

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

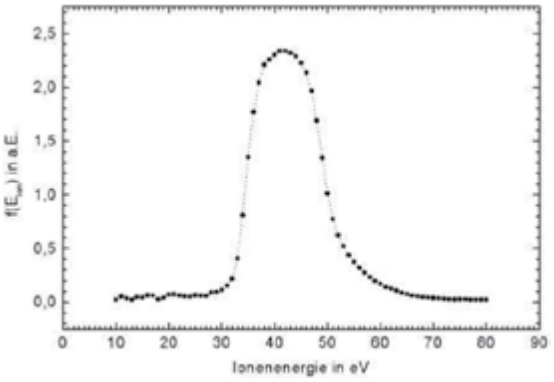
Limitations/constraints

- Height: 50 mm, diameter: 80 mm
- Temperature range: RT–200 °C
- KF40 flange utilizable
- The whole system is transportable

Typical results



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



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

(3) Plasma diagnostics and particle flux analysis *(continued)*

Typical setups

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

Micropole mass spectrometer mounted on KF16 flange

Thin Film Characterisation Methods

(1) Hardness/adhesion
(2) Nanoindentation
(3) Plasma diagnostics and particle flux analysis
(4) Raman spectroscopy
(5) Film thickness
(6) Magnetometer (VSM) and high frequency permeameter
(7) X-ray diffraction XRD/XRR

Karlsruhe Nano Micro Facility (KNMF)
Karlsruhe Institute of Technology (KIT)
Hermann-von-Helmholtz-Platz 1
76344 Eggenstein-Leopoldshafen
Germany

User Office
Phone: +49 (721) 608-23123
Fax: +49 (721) 608-26273
Email: knmf-useroffice@kit.edu
Web: www.knmf.kit.edu

(4) Raman spectroscopy

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.

Thin Film Characterisation Methods

(1) Hardness/adhesion
(2) Nanoindentation
(3) Plasma diagnostics and particle flux analysis
(4) Raman spectroscopy
(5) Film thickness
(6) Magnetometer (VSM) and high frequency permeameter
(7) X-ray diffraction XRD/XRR

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

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

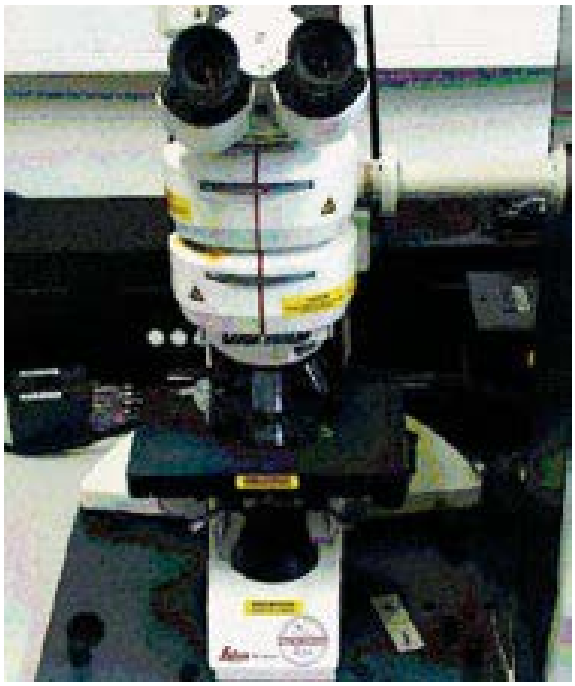
Limitations/constraints

- 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
- Typical sample: flat, thin film, fine powder

Materials

Metal, ceramic, glass, silicon, polymer

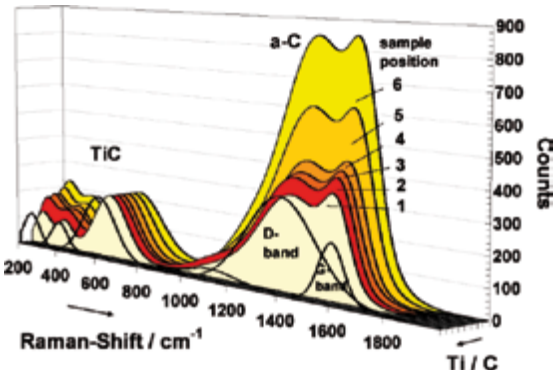
Typical setup



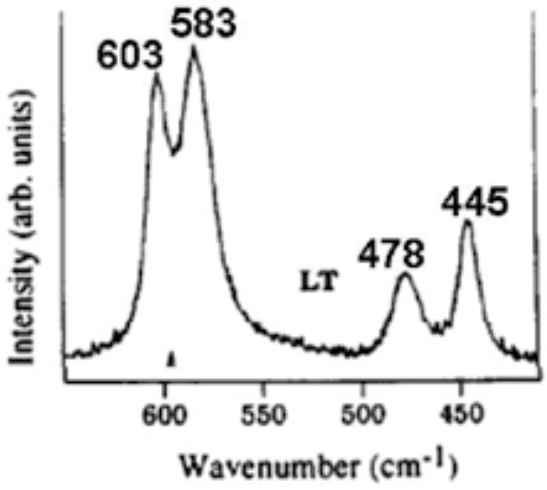
Optical arrangement for Micro Raman spectroscopy

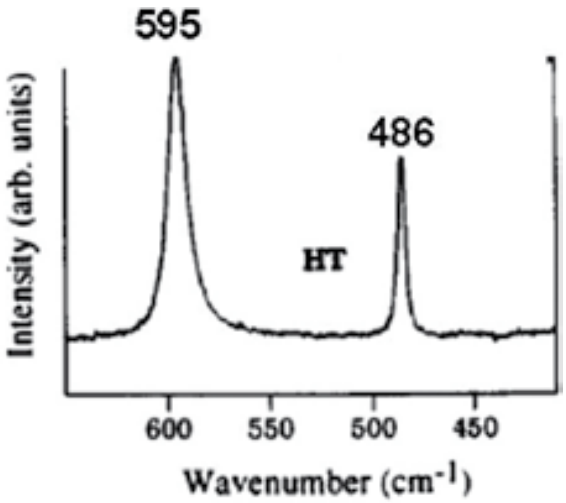
(4) Raman spectroscopy *(continued)*

Typical results



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





Characterisation of $LiCoO_2$ phases

Thin Film Characterisation Methods

(1) Hardness/adhesion
(2) Nanoindentation
(3) Plasma diagnostics and particle flux analysis
(4) Raman spectroscopy
(5) Film thickness
(6) Magnetometer (VSM) and high frequency permeameter
(7) X-ray diffraction XRD/XRR

Karlsruhe Nano Micro Facility (KNMF)
Karlsruhe Institute of Technology (KIT)
Hermann-von-Helmholtz-Platz 1
76344 Eggenstein-Leopoldshafen
Germany

User Office
Phone: +49 (721) 608-23123
Fax: +49 (721) 608-26273
Email: knmf-useroffice@kit.edu
Web: www.knmf.kit.edu

(5) Film thickness

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.

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features

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

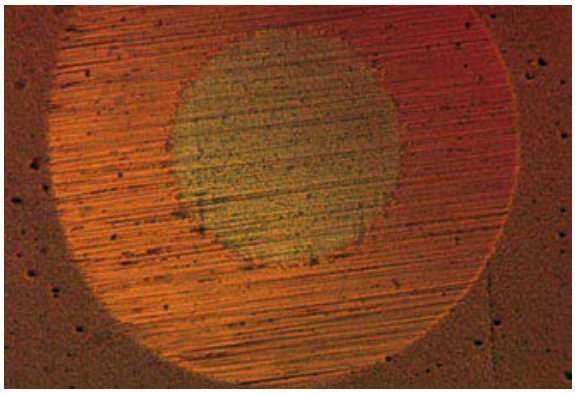
Limitations/constraints

- 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
- Resolution and accuracy depends on roughness

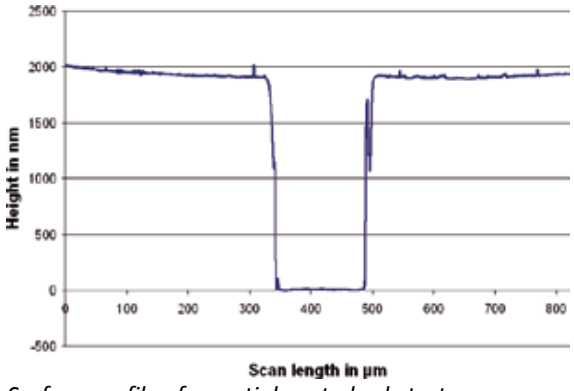
Materials

Metal, ceramic, glass, polymer

Typical results



A calotte polished into a coated substrate



Surface profile of a partial coated substrate

(6) Magnetometer (VSM) and high frequency permeameter

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 30 kHz < f < 6 GHz.

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features

- Vibrating sample magnetometer for thin film samples (max field 600 mT)
- High frequency permeameter 30 kHz < f < 6 GHz

Limitations/constraints

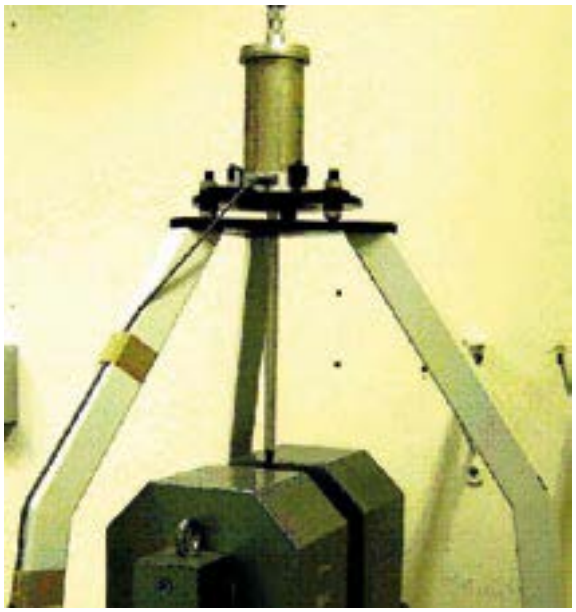
- Vibrating sample magnetometer: Min film thickness min 50 nm
- Vibrating sample magnetometer typical sample: flat, thin film, size 5 x 5 x 0.5 mm^3
- High frequency permeameter, sample size 5 x 5 x 0.3 mm^3
-

Materials

Ferromagnetic thin films on silicon, ceramic, glass

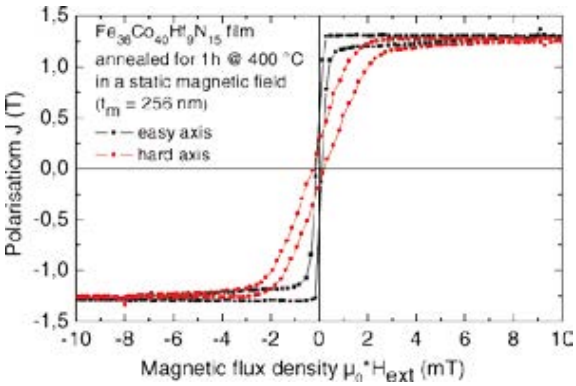
(6) Magnetometer (VSM) and high frequency permeameter *(continued)*

Typical setups and results

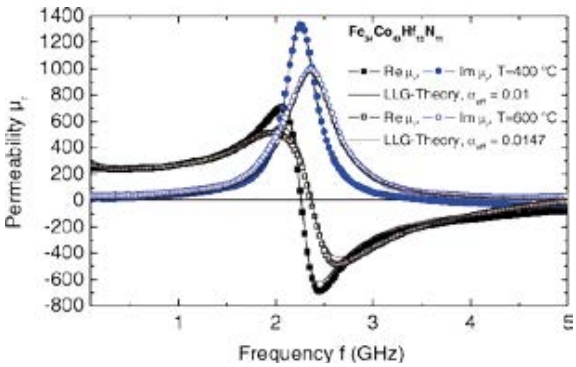


Vibrating sample magnetometer (VSM)

The high frequency permeameter consists of a strip-line measuring head on an attached vector network analyzer



Magnetic hysteresis of a thin ferromagnetic thin film with uniaxial anisotropy (easy axis of magnetisation (black) and hard axis of magnetisation (red)



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

Karlsruhe Nano Micro Facility (KNMF)
Karlsruhe Institute of Technology (KIT)
Hermann-von-Helmholtz-Platz 1
76344 Eggenstein-Leopoldshafen
Germany

User Office
Phone: +49 (721) 608-23123
Fax: +49 (721) 608-26273
Email: knmf-useroffice@kit.edu
Web: www.knmf.kit.edu

Thin Film Characterisation Methods

(7) 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.

Contact

Dr. Harald Leiste
Phone +49 721 22541-22889, fax +49 721 608-22567, email harald.leiste@kit.edu
Institute for Applied Materials (IAM-AWP) - www.iam.kit.edu/awp/english

Features

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

Limitations/constraints

- Resolution 1/10000°
- Max. sample load 5 kg
- Min. beam Ø 2mm
- XRR typical sample: flat, thin film, size 20x20x1mm^3
- Min sample size 5x5mm^2
- Max sample size Ø 150mm x 20mm

Materials

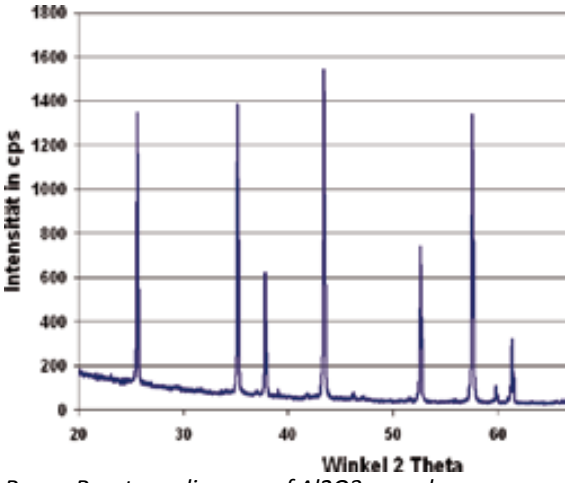
Metal, ceramic, bulk, powder, thin films

Typical setup

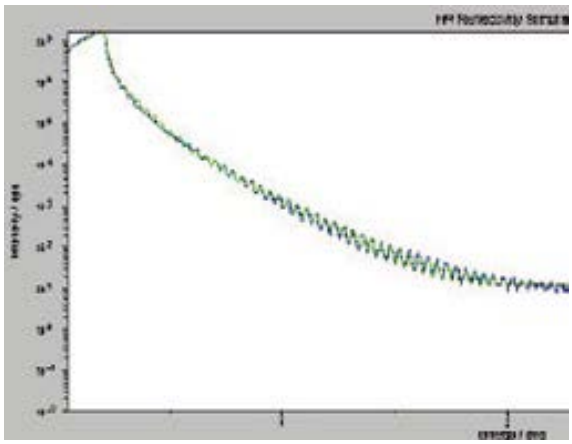
4 circle goniometer for XRD characterization (Seifert 3003 HR)

(7) X-ray diffraction XRD/XRR
(continued)

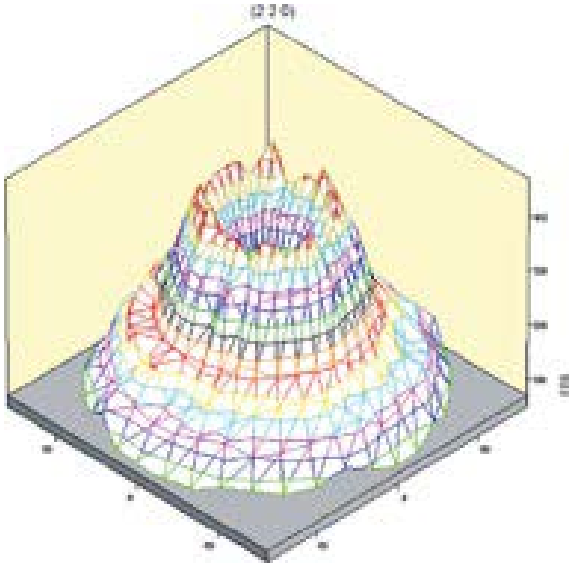
Typical results



Bragg-Brentano diagram of Al2O3-sample



XRR-diagram of a thin film for density determination



Texture diagram of TiN thin film

Karlsruhe Nano Micro Facility (KNMF)
Karlsruhe Institute of Technology (KIT)
Hermann-von-Helmholtz-Platz 1
76344 Eggenstein-Leopoldshafen
Germany

User Office
Phone: +49 (721) 608-23123
Fax: +49 (721) 608-26273
Email: knmf-useroffice@kit.edu
Web: www.knmf.kit.edu