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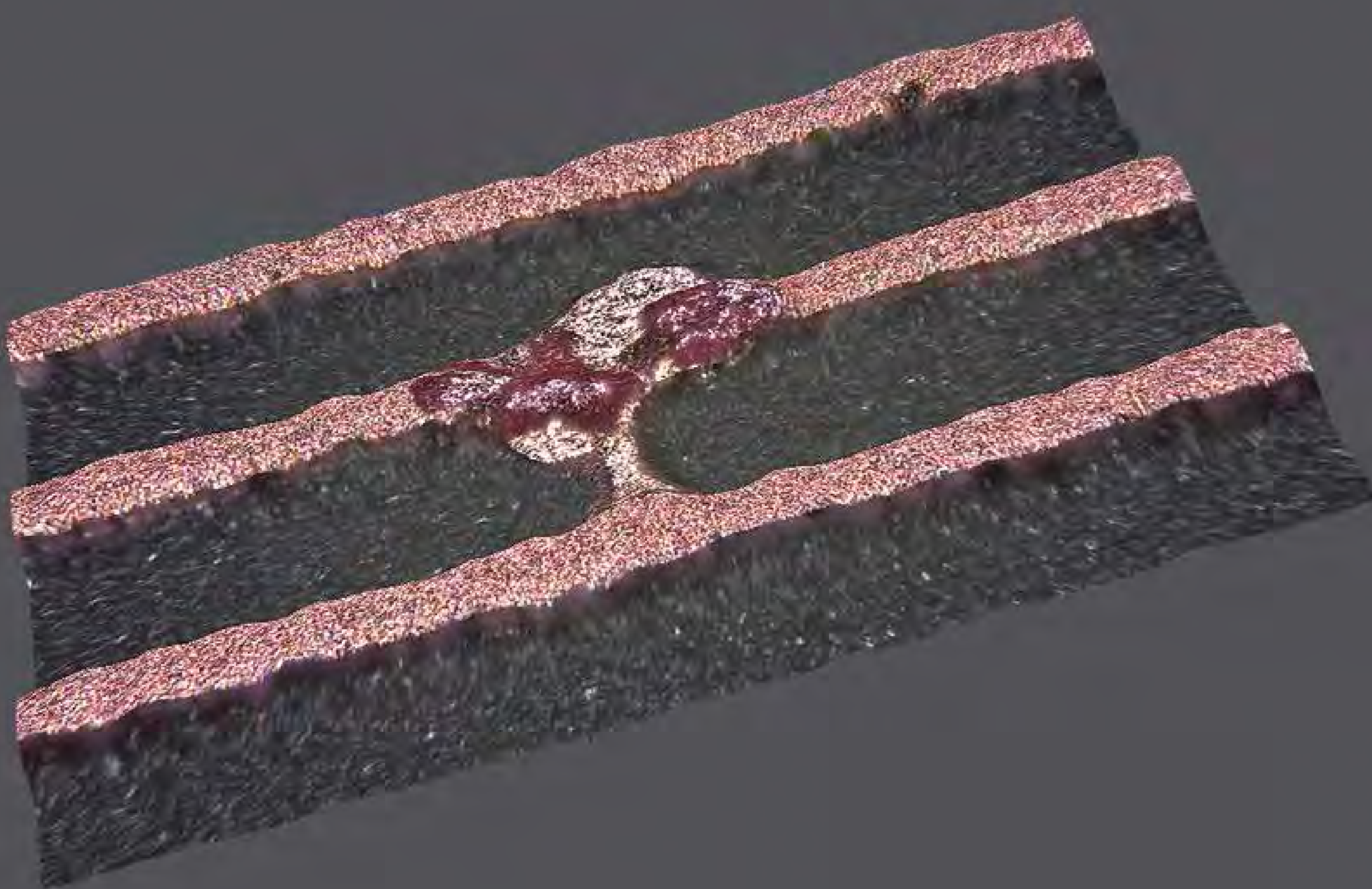
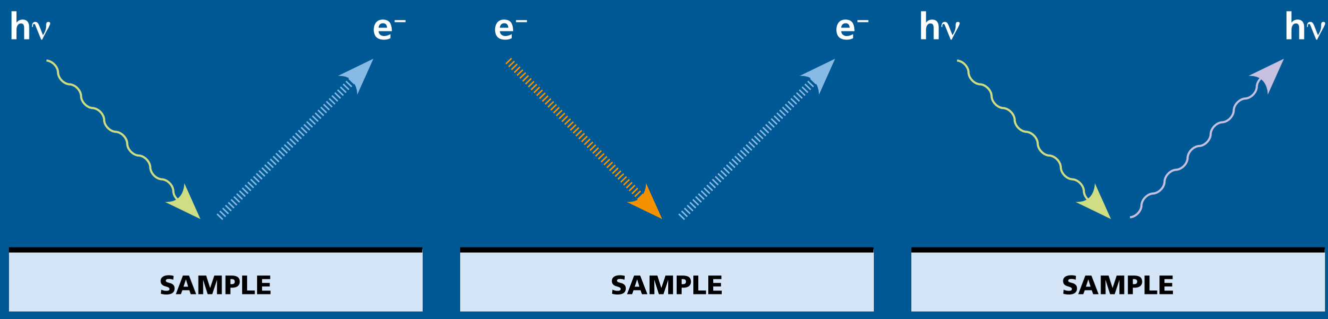
FRAUNHOFER INSTITUTE FOR INTERFACIAL ENGINEERING AND BIOTECHNOLOGY IGB

SURFACE ANALYTICS

PROCESS ANALYTICS, FAULT ANALYSES, PRODUCT DEVELOPMENT, QUALITY CONTROL





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SURFACE AND INTERFACE ANALYTICS

The surface of every material or product interacts with the outside world. Therefore the chemical, physical and biological properties of the surface substantially determine the specific applications and frequently also the service life of a component.

To an increasing extent innovative products are finished with a tailor-made surface design, for which the processes and surface composition have to be controlled down into the submicroscopic or atomic range. This is because adhesion, wettability, wear and corrosion are affected even by the smallest contaminations.

Only modern methods of surface analytics provide access to this information.

Due to their large surface-to-volume ratio, micro- and nanoparticles often have their own characteristic properties and are used as a separate material or as a constituent of surface coatings. The characterization of the particles is therefore of great importance and requires appropriate methods of analysis.

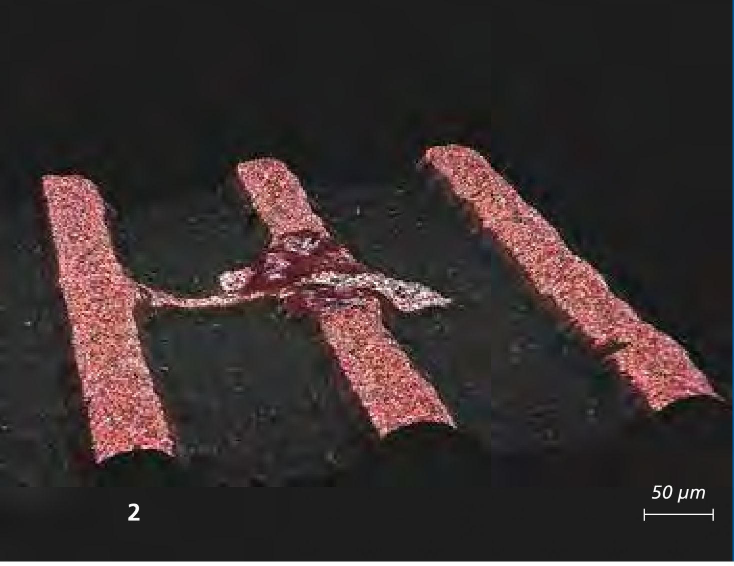
Surface analytics at the Fraunhofer IGB has a wide range of highly specialized methods, procedures and types of equipment at its disposal.

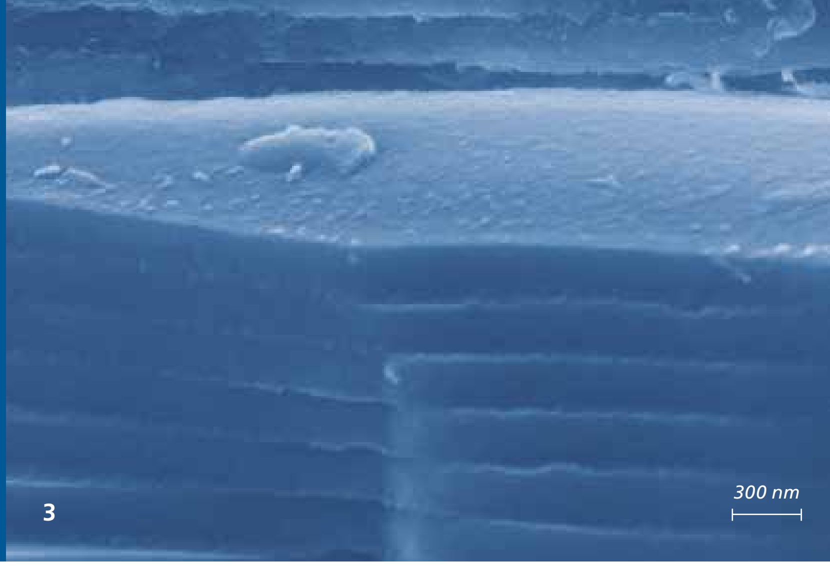
We characterize the following properties of surfaces and interfaces, e.g. ultra-thin layers, powders, particles and membranes:

- chemical characteristics (e.g. elemental surface composition)
- physical characteristics (specific surface, film/layer thickness, wettability)
- morphological characteristics (e.g. roughness and topography) and
- the antibacterial effect of surfaces.

In cooperation with our micro- and cell biologists we also determine biological properties such as biocompatibility, cell adhesion or the antimicrobial finishing.

Both the material surface and in particular the process media utilized play a vital role in optimizing processes and products, so our central chemical analytics are equipped with all the widely used methods of characterizing and analyzing liquid media.





APPLICATIONS AND THE ECONOMIC BENEFITS

Surface-analytical procedures provide a detailed insight into the chemical and structural composition of the material surfaces and the contamination layers on them – whether in the form of a film or particles – which are no longer visible to the unaided eye. That is why modern industrial practice is no longer conceivable without methods for the precise characterization of product surfaces.

These methods provide crucial information for:

- fault and damage analytics in production and processing
- quality control and monitoring of process steps, and
- product development and process optimization.

Fault and damage analytics

If problems occur in the process sequence that become noticeable as a result of the faulty performance or malfunctioning of the product, the cause has to be identified and eliminated as quickly as possible. Frequently this is like looking for a needle in a haystack. Surface-analytical methods can give crucial information here about the origin of undesirable contaminations or about changes in process parameters.

Monitoring the process steps

The early use of analytics to monitor and optimize production steps is more efficient and more cost-effective than the subsequent elimination of faults. Often sophisticated and high-end analytics can only be used during process development, as these methods cannot always be integrated into practical operations. Later on monitoring can be carried out by simpler means.

Optimization of products

"Think small" – the trend towards miniaturization is being accepted more and more widely in the key branches. As a result, in the automotive sector, metal-working, the production of textiles, the production of polymers, pharmaceuticals and medical engineering – material surfaces have long since turned out to be a microcosm of the possibilities. In this world modern ingenious nano- and biotechnological elements, sophisticated coatings and advanced finishing of inexpensive bulk materials define the innovation of products. But the times are over when you could literally examine the problem under your own microscope. The target-oriented development of products with innovative surface characteristics requires the systematic use of surface-analytical methods.

1 *Principles of surface-analytical methods.*
2 *A case of damage: a defective conductor track.*
3 *Product optimization: development of barrier layers.*

CHEMICAL SURFACE ANALYTICS

For the analysis of the elemental composition or the bonding states on material surfaces we use the appropriate method or combinations of them depending on the type of sample and the matter in question. Due to the information depth of approx. 10 nm only the outermost surface is analyzed. However, depth profiles can be achieved in combination with sputter removal.

Electron spectroscopy for chemical analysis (ESCA)

With ESCA we analyze the chemical nature e.g. of particle contaminations, such as the dirtying of a spectacle lens, by means of imaging and small-spot analysis (see diagram on the right).

Infrared (IR) microscopy and spectroscopy

IR spectroscopy and microscopy offer various possibilities for measurement. For example, with ultra-thin films measurements can be made in the transmission and reflection mode or by means of attenuated total reflection (ATR). With spectroscopy middle-range IR (MIR) and near IR (NIR) are available. Organic compounds can be identified by comparison with data banks or reference substances.

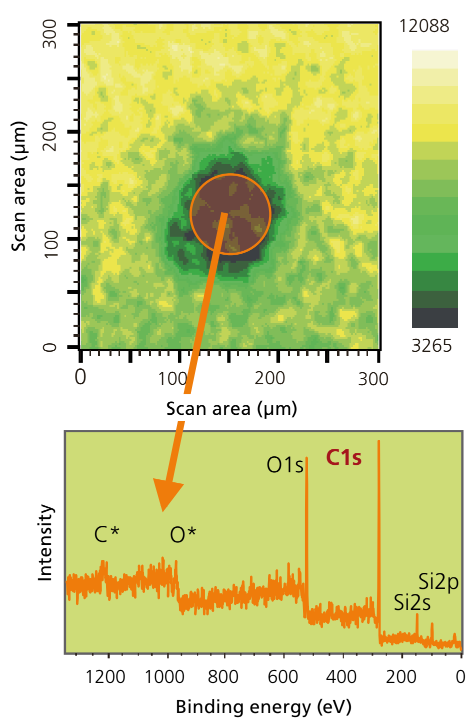
1 *ESCA.*

2 *Raman spectroscopy of a tablet.*

Energy dispersive X-ray microanalysis (EDX)

With energy dispersive X-ray microanalysis the elemental composition of a surface can be imaged using SEM. In addition to two-dimensional and spot measurements, elemental mapping images can also be created.

ESCA image and spectroscopy of 100 µm particles on a spectacle lens.



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Confocal Raman spectroscopy

Confocal micro-Raman spectroscopy permits the chemical analysis of material surfaces of smaller than 1 µm local resolution. Mapping can determine the distribution of a substance, for example the active ingredient in a tablet.

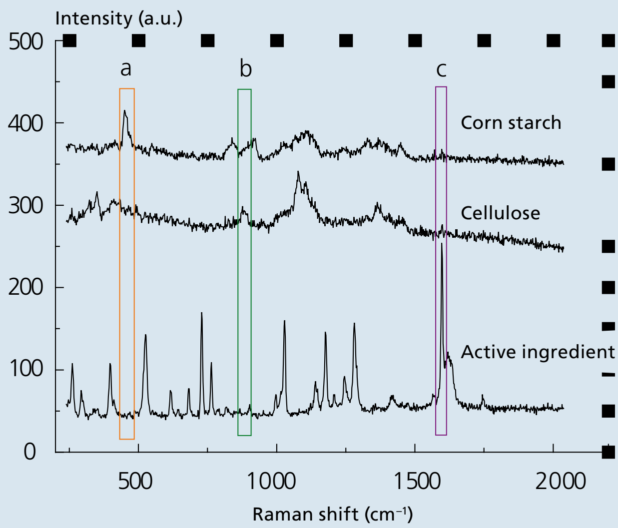
Fluorescence microscopy

Using fluorescence light is absorbed by the sample and immediately after that light with a longer wavelength is emitted. Inherent fluorescence or substances labeled with suitable fluorescent dyes can be detected.

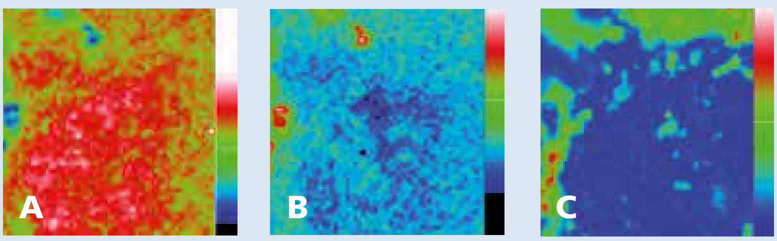
UV / VIS spectroscopy

UV/VIS spectroscopy can be used, for example, to detect unsaturated organic substances. To identify contaminations on surfaces these are rinsed off with a suitable solvent and measured in solution.

Verification of the active ingredient of a tablet by means of Raman spectroscopy and Raman mapping.



Intensity distribution of the tablet ingredients in a 120 µm² area of the tablet.



A: Intensity distribution in the range 440–480 cm^{-1}, characteristic of cornstarch.

B: Intensity distribution in the range 856–900 cm^{-1}, characteristic of cellulose.

C: Intensity distribution in the range 1584–1607 cm^{-1}, A B C characteristic of the active ingredient.

1

TOPOGRAPHY AND MORPHOLOGY

Depending on the dimension of the topography to be profiled we use 3D light microscopy, a scanning electron microscopy (SEM) or special scanning techniques.

3D light microscopy

With 3D light microscopy three-dimensional images can be generated with a high level of depth definition and then viewed three-dimensionally. The choice of the lenses permits a 5–5000-fold magnification.

Scanning electron microscopy (SEM)

Scanning electron microscopy is the most versatile of the microscopic methods of investigation. Its advantages are:

- wide magnification range
- definition with a large depth of field
- maximum resolution 1 nm

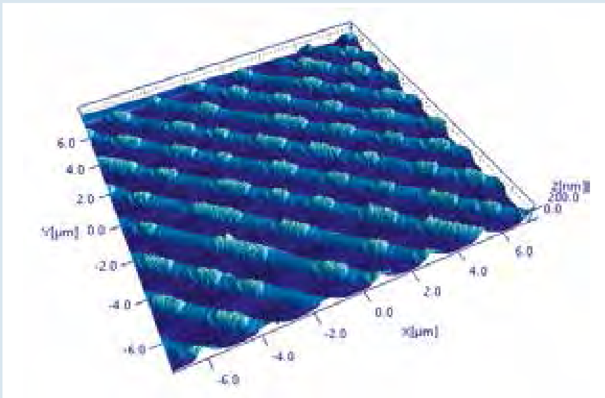
1 *Image of the particle size distribution of microparticles by means of light microscopy.*

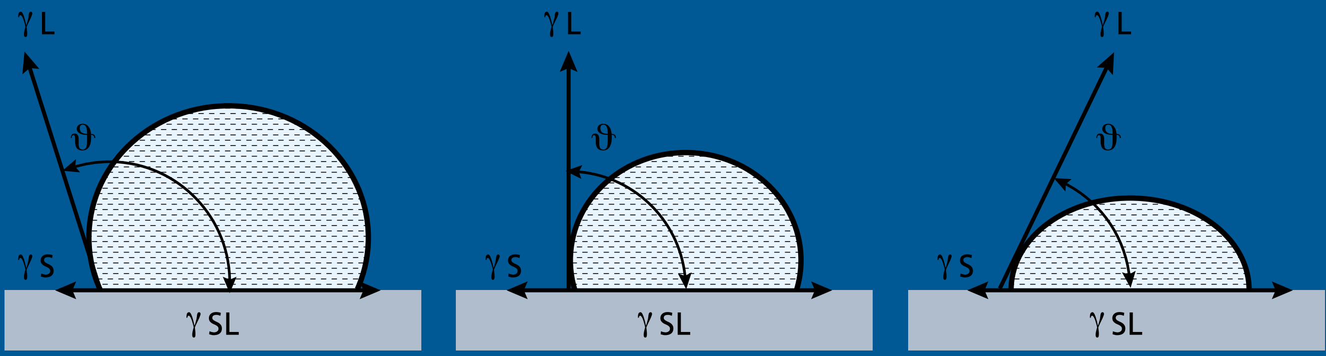
2 *Diagram showing the contact angle measurement.*

Atomic force microscopy (AFM)

With atomic force microscopy the microstructure of surfaces can be imaged down to the nanometer range and, for example, the plasma-technical increase in the microroughness of synthetic materials can be detected. Average roughness values can be calculated by evaluating the image. Besides profiling the topography, various modes also give information about the material contrast.

AFM-Image of a CD-ROM surface.





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SURFACE TENSION, WETTABILITY AND TENSIOMETRY

Interface and surface tensions provide information about the wettability of surfaces and are important material properties in printing, painting and coating.

Contact angle measurements

The relatively simple measurement of the wetting properties of material surfaces by means of contact angle measurements can provide important information about the cleanliness or bonding properties of the product. The polar and nonpolar fractions of the surface energy can be profiled by varying the measuring liquid. Determining the hysteresis from the advancing and receding contact angle provides information about inhomogeneities or roughness of the surface.

Measuring dynamic surface tension of liquids

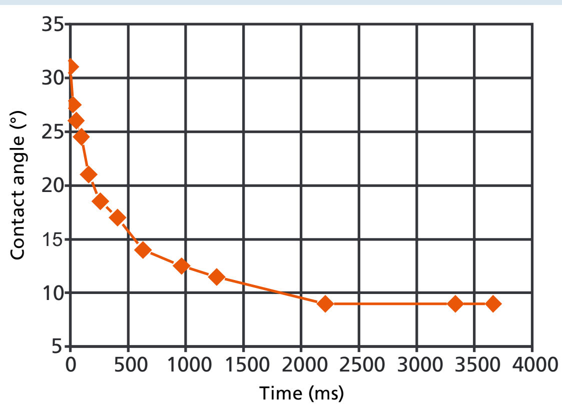
Various methods are available for measuring the dynamic surface tension of liquids.

- Wilhelmy plate method
- Bubble pressure tensiometry
- Drop volume tensiometry

Wetting characteristics of powders

- Observation of the contact angle using the method of the sessile drop on pellets
- Imbibition method:
  Here the quantity of liquid absorbed by scattered powder is measured in relation to the time. A calibration is carried out by means of complete wetting. The contact angle of the powder is then determined using incomplete wetting liquids.

Dynamic development of the contact angle in rapid wetting processes.



SPECIAL METHODS

The Fraunhofer IGB has further types of analytical equipment at its disposal. Should you be looking for a specific method that we do not have at the institute, have a word with us about it. Through our extensive network with other Fraunhofer Institutes, university institutes, major research centers and private analytics providers we can find the suitable method or combination of methods for the your investigation.

Spectroscopic ellipsometry

Spectroscopic ellipsometry is an optical method of investigation for measuring the optical indices n and k and the thickness of thin films, for example oxide layers or polymer films.

Electron spin resonance (ESR)

With electron spin resonance (ESR) radicals can be detected and characterized. Due to the high sensitivity, decay curves of the density of radicals on material surfaces can also be detected after a plasma treatment. This procedure can also be used for larger surface areas in the mapping mode.

MALDI-TOF / TOF mass spectrometry

This instrument determines the molecular mass (ms mode) and also the PSD fragments (post source decay PSD, ms / ms mode). Typically it is utilized for protein analytics. In individual cases it can also be used for the analysis of other polymers.

1 *MALDI-TOF / TOF-MS.*
2 *Molecularly imprinted nanoparticles.*
3 *The functionalization of chip surfaces.*


2


3

CHARACTERIZATION OF POLYMERS AND PARTICLES

Dynamic light scattering (DLS)

With dynamic light scattering, also called image correlation spectroscopy (ICS), the size distribution of particles from 1 nm down to the micrometer range can be analyzed precisely and nondestructively.

Differential scanning calorimetry (DSC)

This method measures the quantity of heat required for the physical or chemical conversion of a substance or the resulting amount of heat.

Thermogravimetry (TG)

Thermogravimetry measures the mass or the change in mass of a sample depending on the temperature and/or time. Changes in the mass occur in evaporation, decomposition, chemical reactions, and magnetic or electric conversions.

Simultaneous thermal analysis (STA)

Simultaneous thermal analysis is a combination of the two methods DSC and TG. It permits the simultaneous measurement of energetic effects and changes in the mass of one and the same sample.

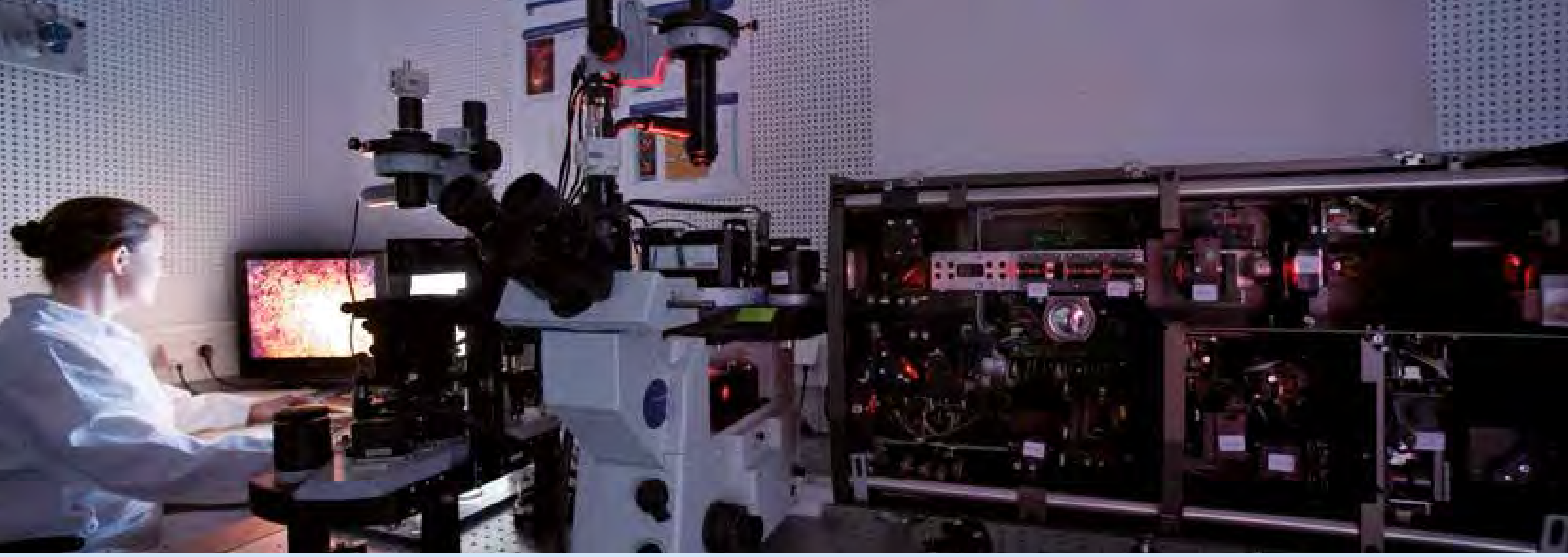
Gel permeation chromatography (GPC)

Gel permeation chromatography (GPC) is is a type of size exclusion chromatography (SEC) and a standard method for determining molar mass distributions in polymer samples.

Volumetric sorption analysis

Various material sizes can be determined with this method:

- Specific surface (BET, Langmuir)
- Pore radius distribution
- Micropore and mesopore analysis
- Water vapor sorption
- Chemisorption analysis



TECHNICAL RESOURCES AT THE FRAUNHOFER IGB

Chemical composition analysis

- Electron spectroscopy for chemical analysis (ESCA), multipoint, imaging, valence band, depth profile, angle-dependent
- Energy-dispersive X-ray microanalysis (EDX)
- MIR and NIR spectroscopy, diffuse reflexion, transmission, grazing incidence, attenuated total reflection (micro)-ATR
- Infrared microscopy (transmission, ATR, reflexion)
- (Fourier Transformation) Infrared spectroscopy / microscopy (FT-IR)
- Confocal fluorescence spectroscopy
- Confocal micro-Raman spectroscopy, also combined with atomic force microscopy

Topography / morphology

- Light microscopy (LM), also 3D
- High resolution field emission scanning electron microscopy (FE-SEM)
- Atomic force microscopy (AFM)

Further methods

- Spectroscopic ellipsometry
- Electron spin resonance (ESR)
- UV/VIS spectroscopy
- MALDI-TOF/TOF mass spectrometry
- Quartz oscillator microbalance with dissipation monitoring (QCM-D)

Surface tension / wettability

- Static and dynamic contact angle measurements, various tensiometers e.g. according to Wilhelmy, du Noüy, dynamic drop-volume or bubble-pressure
- Adsorption / desorption curves (BET surfaces, pore radius distribution)
- Gel permeation chromatography (GPC)

Characterization of polymers and particles

- Dynamic light scattering (DLS)
- Zeta potential
- Particle size distribution
- Adsorption / desorption isotherms (BET surfaces, e.g. with IGA)
- Thermogravimetric analyses (TGA)
- Dynamic difference calorimetry (DSC)
- Microcalorimetry (µ range)
- Gel permeation chromatography (GPC)

Membrane characterization

- Permeability, MWCO determination
- Porometry
- Post mortem analyses

Analyses of liquids and process media

- We have a wide range of equipment in our chemical analytics group such as HPLC, ICP-AES, GC-MS, AAS

"FOR MANY YEARS WE HAVE BEEN WORKING WITH THE FRAUNHOFER IGB. THE SPEEDY, CONFIDENTIAL AND COMPETENT HANDLING OF THE JOBS AND ORDERS IS VERY IMPORTANT FOR US."

Dirk Meyer, Dipl.-Ing.: MAHLE Kleinmotoren-Komponenten GmbH & Co. KG

WE CHARACTERIZE FOR YOU

Surfaces and interfaces of

- work pieces, solids
- layers
- membranes
- powders
- liquids

from all kinds of materials e.g.

- polymers
- ceramics and glasses
- metals
- composite materials

Using these analyses we can answer questions about the following parameters:

- wettability
- adsorption
- corrosion
- film thickness
- adhesion
- purity
- roughness
- chemical composition
- chemical functions
- topography

Competence

Experienced specialists use modern equipment to provide you with a wide range of know-how. The results obtained in the analyses help your innovations to achieve market success.

Accreditation

The Fraunhofer IGB is a modern service provider in the field of surface and interface analytics. In order to guarantee the best possible quality for our customers, we have introduced a quality management system (QMS) with a range of accredited analytical methods.

Confidentiality and reliability

We guarantee absolute confidentiality as well as a speedy and reliable handling of jobs and projects.

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Combined expertise by networking

The Fraunhofer Cleaning Technology Alliance deals with the cleaning of surfaces, especially with special procedures. Surface-analytical methods are used to monitor cleaning efficacy, quality assurance and also damage analytics. Thus a wide range of analytical methods are available through the Alliance partners.

www.allianz-reinigungstechnik.de

Polymeric surfaces are increasingly being equipped with specific functions in order to provide new or enhanced properties for the product. The analytical competence required for the characterization, evaluation and process optimization of polymeric materials is constantly being extended by the Fraunhofer Polymeric Surfaces POLO® Alliance.

www.polo.fraunhofer.de

Developments in the field of nanotechnology require innovative surface-analytical methods to optimize processes and already implemented material properties. In some cases existing methods have to be further developed and modified to meet the latest requirements. Here we are partners in the Fraunhofer Nanotechnology Alliance.

www.nano.fraunhofer.de

Fraunhofer IGB brief profile

The Fraunhofer IGB develops and optimizes processes and products in the fields of medicine, pharmacy, chemistry, the environment and energy. We combine the highest scientific standards with professional know-how in our competence areas of Interfacial Engineering and Materials Science, Molecular Biotechnology, Physical Process Technology, Environmental Biotechnology and Bioprocess Engineering, as well as Cell and Tissue Engineering – always with a view to economic efficiency and sustainability.

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