Characterization methods

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Insitute of Electronic Materials Technology

Elemental Analyzer

The Department of Chemical Synthesis and Graphene Flakes

Facilities:

CHN 628, S 628, O 836 (LECO), determination of mass fraction of: carbon, hydrogen, nitrogen, sulfur and oxygen,

Sample mass:

From 0.3 g

Typical applications:

- Wide-range measurement of oxygen, nitrogen, and hydrogen content of inorganic materials, ferrous and nonferrous alloys, and refractory materials.
- Measurement of carbon, hydrogen, nitrogen, sulphur in organic matrices, from food to fuels.

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Main surface analysis by gas adsorption - BET isotherm

The Department of Chemical Synthesis and Graphene Flakes

Facilities:

Quadrasorb Evo analyzer with FloVac Degasser (Quantachrome Instruments)

Adsorbate gas: Nitrogen

Sample mass:

From 0,2 g

Typical applications:

This kind of analysis is to determine the specific area of microporous and mesoporous materials. The device measures in range from 0.01 m^2/g to no known upper limit (for nitrogen).

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Hall Effect Measurement System

The Department of Chemical Synthesis and Graphene Flakes

Facilities:

Escopia HMS-5300 (Bridge Technology), analysis in temperature 80°K to 350°K &/or 300°K to 573°K

Typical applications:

The systems can be used to characterize various materials including all semiconductors including Si, SiGe, SiC, GaAs, InGaAs, InP, GaN (N Type & P Type can be measured), metal layers, oxides.

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Thermal analysis (TG, DSC) and specific heat (Cp) measurements

The Department of Composites and Ceramic Materials

STA 449 F5 Jupiter TG-DTA/DSC Thermal Analyzer

Facilities:

Differential Scanning Calorimetry (DSC) is conducted. True TGA and DSC measurements can be performed at sample temperatures from ambient to 1600°C. TGA measurements are also possible, even on large or heavy samples.

Parameters:

- Temperature range: RT – 1600°C
- Temperature resolution: 0.001 K
- Heating rate: 0.001 to 50 K/min
- Atmospheres: inert, oxidizing, static, dynamic, vacuum

Sample size:

- Maximum sample load: 35 g (incl. crucible)
- Sample volume: up to 5 cm^3

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Electric measurements

The Department of Composites and Ceramic Materials

SeebTest 1.0 producent PESS

Facilities:

Simultaneous determination of Seebeck Coefficient and Electrical Conductivity. Electrical conductivity and Seebeck coefficient (thermoelectric properties) of semiconductor and metallic materials can be characterized in a single measurement with the 4-probe method in vacuum or inert protective atmosphere. A sample is vertically placed between current electrodes (two-heater system) and compressed by 5N force. The Peltier effect is minimized by using an alternated current.

Sample size:

- Cylindrical (from 5.0 to 13.0 mm, length 6-20mm)
- Rectangular (from 5.0x5.0 to 10.0x10.0 mm, length 6-20mm)
- Other shapes are also possible after consultation

Parameters:

- Temperature range: RT - 550°C with unlimited number of temperature steps
- Seebeck Coefficient range: 5 – 1000 μV/K with accuracy better than 5%
- Electrical conductivity range: 0.1 – 1000000 S/m with accuracy better than 5%
- Atmosphere: vacuum 10 $^{-3}$ mbar, inert, oxidizing

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Thermal conductivity /diffusivity measurements

The Department of Composites and Ceramic Materials

LFA 457 Micro Flash NETZSCH

Facilities:

- LFA 457 Micro Flash NETZSCH equipment is applied for thermal diffusivity measurements. The front side of a plane parallel solid sample is heated by a short laser pulse.
- The temperature rise on the rear surface is measured versus time using an infrared detector.
- Compared with the direct measurements of thermal conductivity, this method has an advantage of a simple test piece configuration, small test piece size, applicability to a wide range of diffusivity values, great accuracy and reproducibility.

Parameters:

- Temperature range: RT – 1100°C
- Thermal Diffusivity range: 0.01 mm^2/s to 1000 mm^2/s
- Thermal Conductivity range: 0.1 W/(m·K) to 2000W/(m·K)

Sample size:

- Rectangular (8.0x8.0 mm or 10.0x10.0 mm)
- Cylindrical (10.0 mm, 12.7 mm, 25.4 mm)

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Dilatometric measurements

The Department of Composites and Ceramic Materials

DIL 402 Expedis NETZSCH

Facilities:

- Dilatometer DIL 402 Select Expedis allows to analysis of length change phenomena of different materials (metals, ceramics, polymers, composites), thus revealing information regarding their behavior during heating or cooling process.
- For preparing a measurement, the defined sample is inserted into a holder and brought into contact with the pushrod. After closing the furnace the temperature rises and the linear thermal expansion can be measured. Thermal expansion of the sample during heating is detected by the displacement system.
- System allows for detection of phase transitions, density change, shrinkage steps, sintering behavior, etc.

Parameters:

- Temperature range: RT - 1100°C
- Measuring range: ±25 mm
- Pushrod load: 0.01- 3.0N
- Atmosphere: Argon

Sample size:

- Rectangular (from 3.0x3.0 to 8.0x8.0 mm, length 12-25mm)
- Cylindrical (from 3.0 to 10.0 mm, length 12-25mm)

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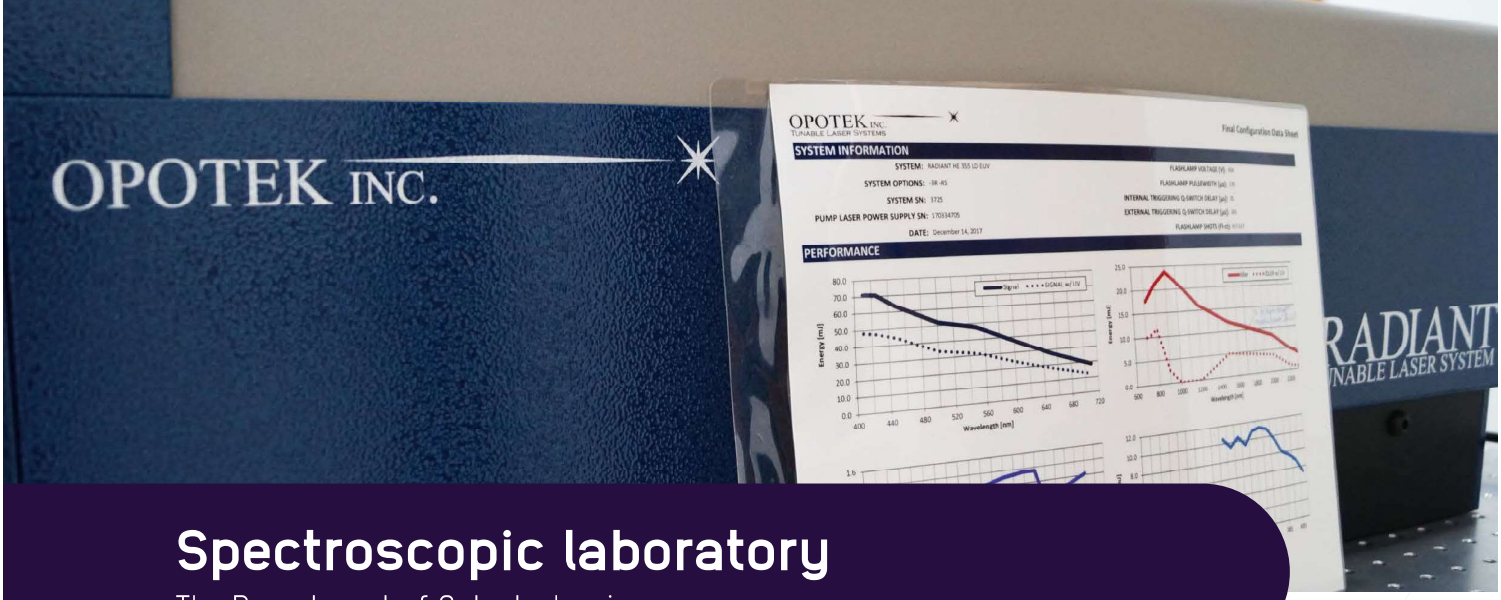

Spectroscopic laboratory

The Department of Optoelectronics

Spectroscopic laboratory

Facilities:

- Nanosecond tunable laser **Opotek Radiant 355 HE EUV,**
- High resolution spectrometer **Princeton Instruments/Acton ARC-SP-750,**
- Helium cryostat **AS Scientific CH-202,**
- Time correlated photon counting Becker&Hickl

Parameters:

- Emission and excitation spectroscopy in the wavelength range 195 nm – 2,5 µm
- Time-resolved analysis of absorption and emission properties of luminescent materials in various forms (powders, fluids, solids) within temperature range 10K-300°C
- Characterization of materials for passive Q-switches
- Determination of laser damage threshold, photometric characterization

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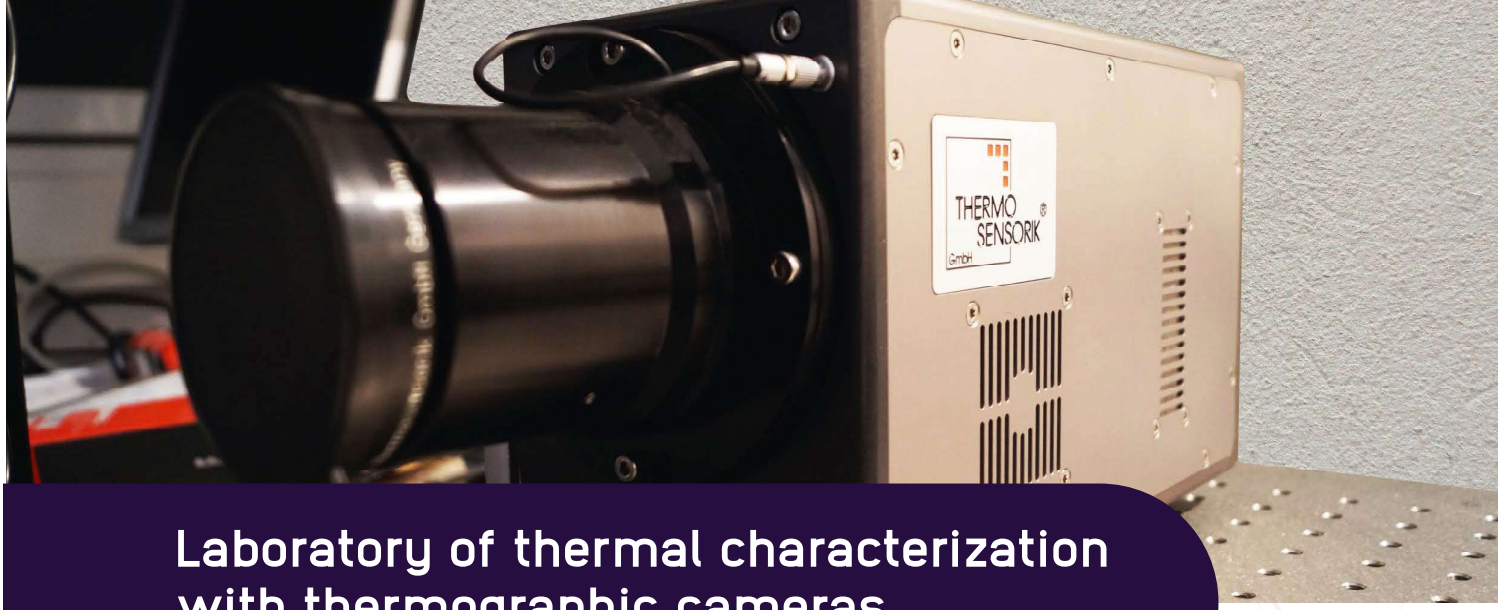

Laboratory of thermal characterization with thermographic cameras

The Department of Optoelectronics

Laboratory of thermal characterization with thermographic cameras

Facilities:

- **Thermosensorik 640SM** sensitive in the spectral range: 1.1 – 5.3 µm, NETD < 20 mK, wide-field and microscopic optics, spatial resolution 3 µm x 3 µm,
- **FLIR T630sc** sensitive in the spectral range 8 – 14 µm, NETD < 40 mK, spatial resolution 15 µm x 15 µm,
- **Chiller Thermotek,** 695 watts cooling capacity; 72 psi/3.8 LPM,
- Pumps,
- Temperature sensors,
- Environmental chamber.

Typical applications:

- Investigations of heat flow in the micro- and milliscale,
- Studies of thermal phenomena in electronic devices,
- Defect recognition,
- Characterization of various cooling systems (micro-channel, immersion).

Sample size:

- minimum 1mm x 1mm
- maximum 20cm x 20 cm

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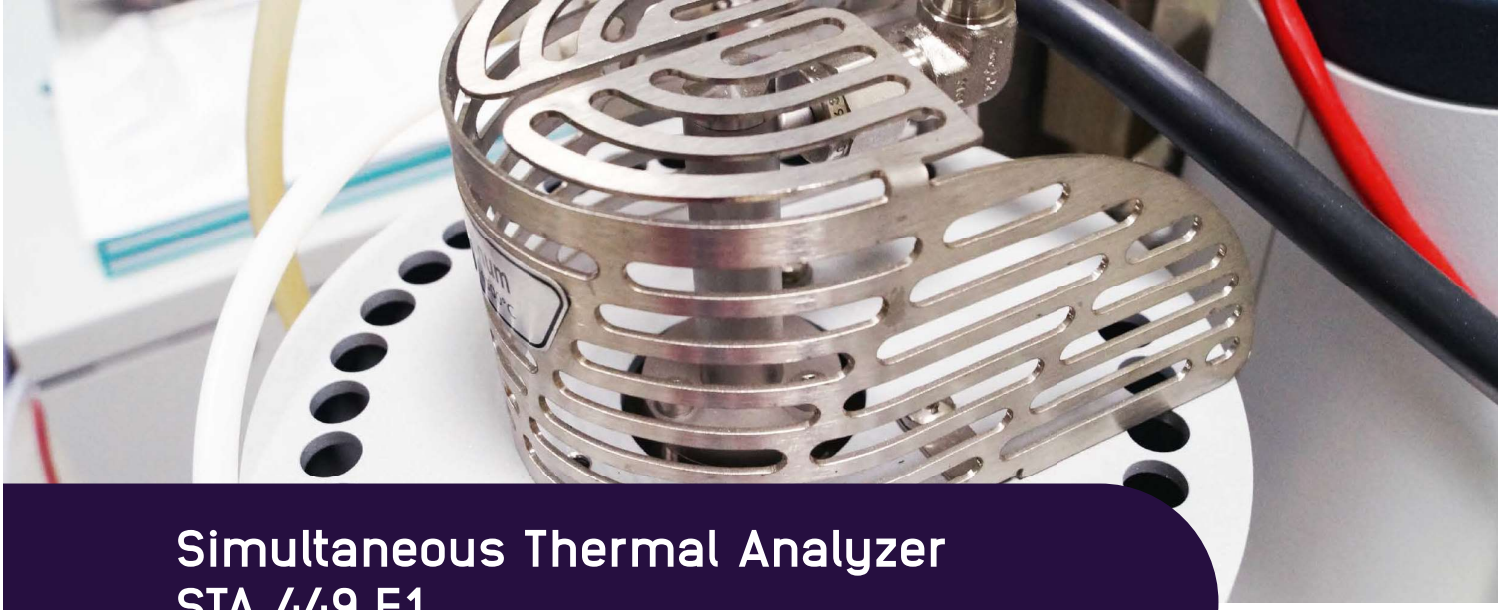

Simultaneous Thermal Analyzer STA 449 F1

The Department of Functional Materials

Simultaneous Thermal Analyzer STA 449 F1

Facilities:

The Thermal Analyzer from Netzsch GmbH is equipped with thermobalance, calorimeter and two distinct furnaces: platinum and graphite. This enables one to cover a wide range of conditions: oxidizing (up to 50% vol. oxygen) up to 1350°C and inert (under the flow of argon) up to 1850°C. High sensitivity of the system makes it possible to detect mass change as small as 0.1µg and heat flow change as low as 0.01mW/mg. As structural and chemical reactions are associated with exchange of heat, the apparatus is qualified for identification of phase transitions.

Typical applications:

- Thermal stability (e.g. nanomaterials, organic-based composites at elevated temperatures).
- Revealment of chemical reactions (e.g. decomposition, oxidation).
- Characteristic temperatures and reaction enthalpies of phase transitions (e.g. Tg, Tx, Tm in glass materials).
- Determination of reaction kinetics (e.g. crystallization rate at isothermic conditions, effectiveness of the sinthering process).

Various types of studies are possible:

- Recording the mass change as a function of temperature - thermogravimetry (TG).
- Quantitative measurement of weight loss rate with respect to time (isothermal) or temperature (dynamic).
- Quantitative registration of thermal effects in differential thermal analysis (DTA) or differential scanning calorimetry (DSC) mode.
- Quantitative measurements of thermal effects (endo- or exothermic) occuring at isothermic conditions.

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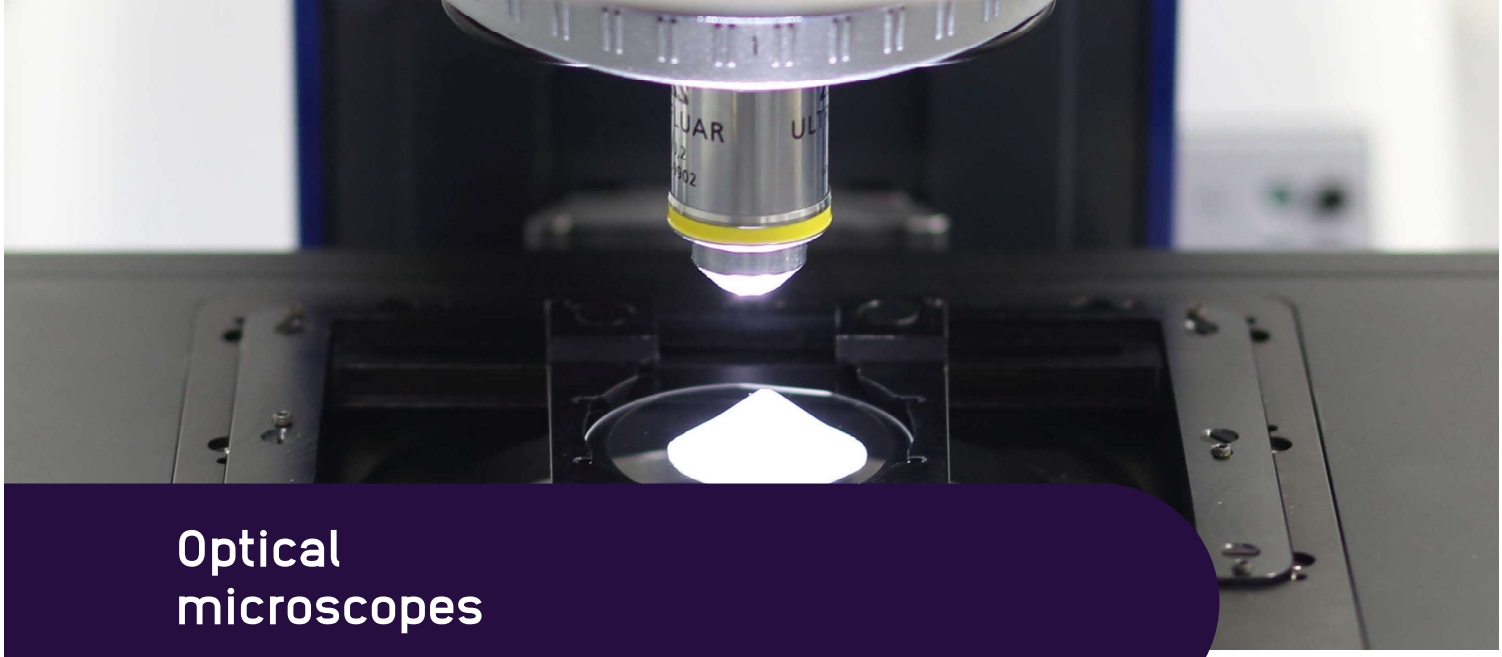

Optical microscopes

The Department of Functional Materials

Optical microscopes

- A manual, nosepiece type microscope which meets the various needs of observation, inspection, research and analysis across a wide range of industrial and scientific fields.
- In LFM ITME, two optical microscopes are available for transmission and reflection imaging of the samples. Both have modular anti-vibration structure with an integrated

Facilities:

They are equipped with the polarizers – analyzers system and optics for differential interference contrast (DIC) and for observation of the surface of the sample in dark field regime. DIC, dark field and the set of polarizers are useful tools for observation of crystal structures, stress, structural defects, surface morphology.

Typical applications:

- Microscopes with PC software facilitate image analysis, perform three-dimensional cross-sectional area, a full description of the quantitative parameters, the size distributions of objects - such as statistical and geometric - and indicators of the shape and uniformity.
- The equipment also includes Linkam heating and cooling stages enable observations at high and low temperatures, up to 1500 °C and as low as -200°C.

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UV-Visible-NIR CRAIC Microspectrophotometer

The Department of Functional Materials

UV-Visible-NIR Microspectrophotometer

Facilities:

- The **UV-Visible-NIR microspectrophotometer** acquires spectra of extremely small sample areas non-destructively. Measurements can be made while light is transmitted through the sample or reflected from it.
- The UV-visible-NIR range is especially important for most substances, even more for colorless ones which absorb in the UV than in the visible and infrared regions.
- Our system covers spectral range from 200 nm to 1000 nm and provides measurements from the area 750x750nm to 150x150µm with two modes (transmission and reflection).
- The system combined with the precise heating (-200°C to 1500°C) stage facilitates observation of temperature dependent processes (phase transitions, energy activations.)

Typical applications:

Microspectrometers are used to identify and quantify microscopic samples ranging from the microfluidic kinetics, matching fibers or paints by a forensic chemist, the qualification of gems or coal by a geologist, the determination of the color of ink or paint by a process chemist or even the analysis of great works of art by conservators.

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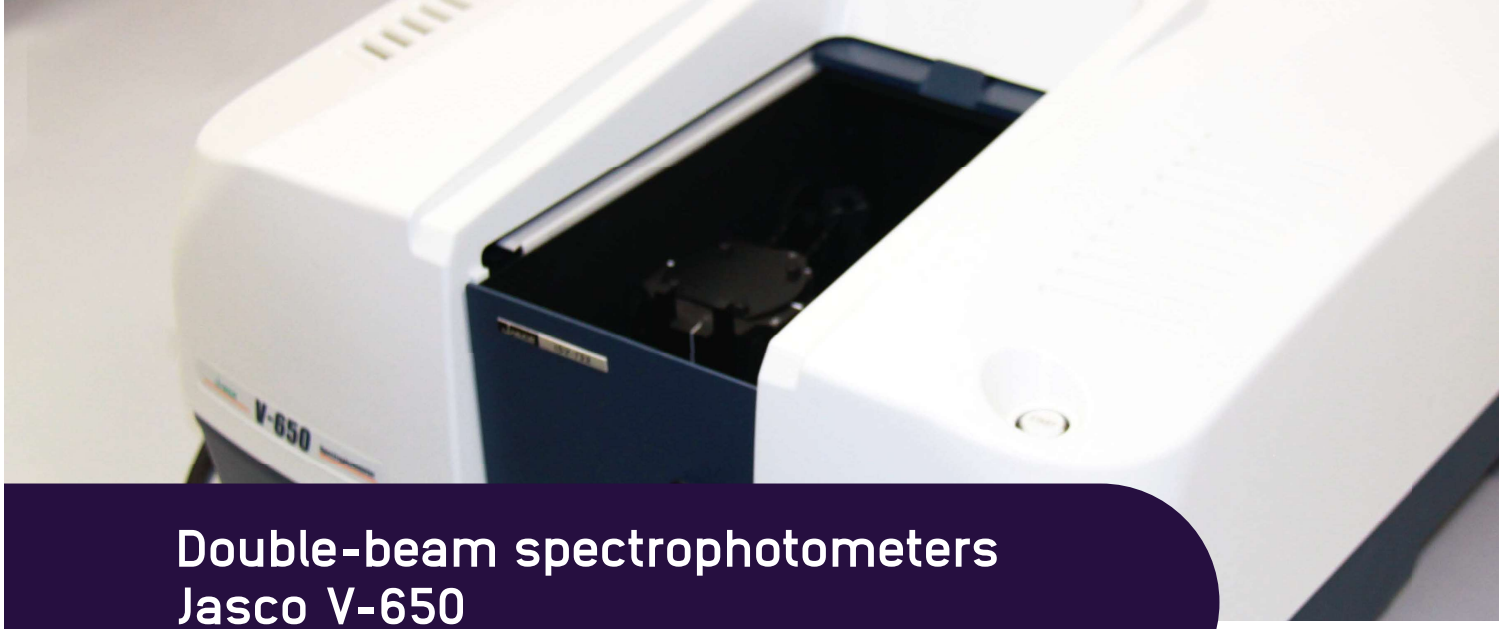

Double-beam spectrophotometers Jasco V-650

The Department of Functional Materials

Double-beam spectrophotometers Jasco V-650

Facilities:

- The high sensitivity of the used detector and high spectral resolution (0.05nm) enable measurements of low concentration samples or samples with very weak optical response in the spectral range 190-900nm. The device examines the transmission and absorbance spectrum of solids and liquids.
- With the proper set of samples, it is possible to define concentrations of solution curves, suspension characterization or characterization of slow chemical processes. Equipped with handling accessories such as integrating spheres, it collects diffuse light transmitted or reflected by the sample.

Typical applications:

- It is possible to characterize ceramics, policrystals, powder materials or unpolished crystals.
- Identification of absorption bands and determination energy gap of the tested materials.

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DIL 402 PC
dilatometer Netzsch GmbH

The Department of Functional Materials

DIL 402 PC
dilatometer Netzsch GmbH

Facilities:

The high temperature DIL 402 PC dilatometer from Netzsch GmbH enables measurements from RT up to 1500°C, owing to the silicon carbide furnace. By using the Pt-PtRh10 thermocouple one can probe not only inert conditions (e.g. in the flow of N_2 or Ar) but also under the flow of air. The scan rates are direction-dependent, i.e. heating rate of 0.1-50K/min and cooling rates of 0.1-99.9K/min are acceptable. The detection limit is as low as 0.01µm.

Typical applications:

- Sintering onset temperature (e.g. the temperature at which the ceramic changes (usually decreases) its volume).
- Revealment of volume-change assisted phase transitions (e.g. involving the change of crystallattice).
- Determination of equilibrium phase diagram for unknown material systems.
- Determination of Temperature-Time-Transition plots (e.g. for optimizing fabrication conditions).

Various types of studies are possible:

- Recording of length change versus temperature (dL scan).
- Calculation of linear expansion coefficient (α).
- Qualitative analysis of phase transitions (1st order, 2nd order, etc.).
- Quantitative analysis of phase transitions (change of volume/expansion coefficient, reaction temperature onset, scale of the temperature inertia).
- Determination of doping effect on the phase transition (temperature, scale, etc.).

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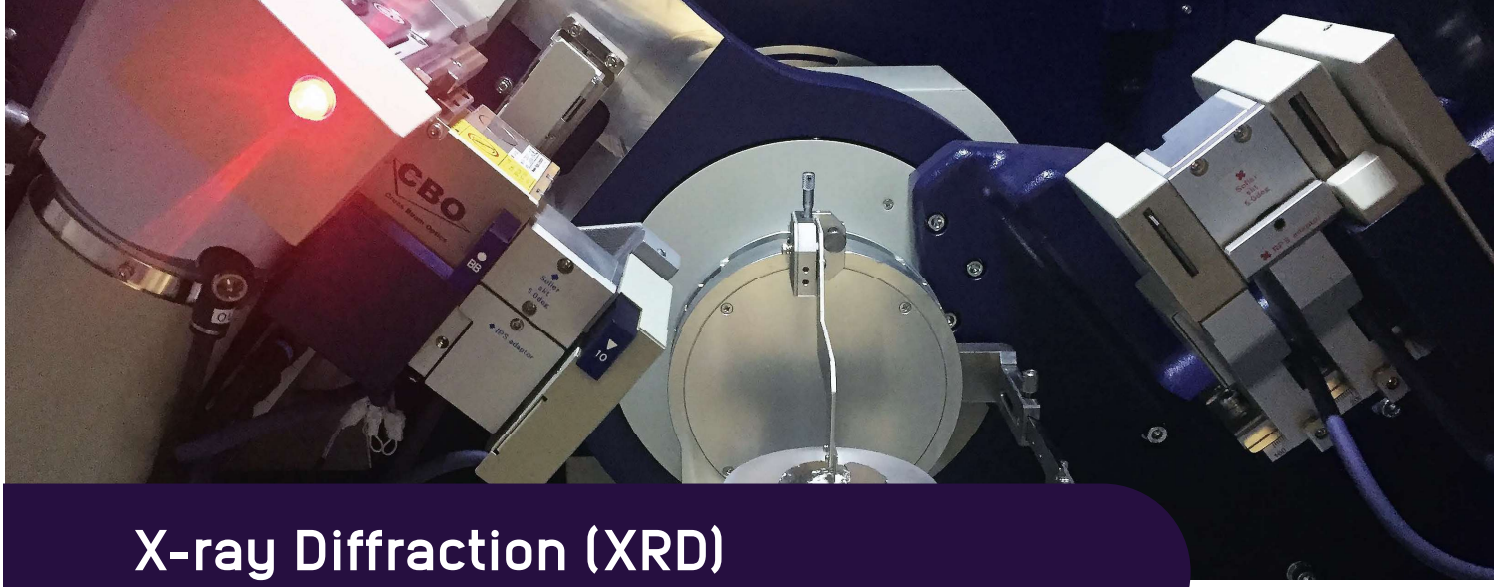

X-ray Diffraction (XRD)

The Laboratory of Structural Research and Materials Characterization

X-ray Diffraction (XRD)

Facilities:

Universal X-ray diffractometer **Rigaku Smartlab 3 kW** is equipped with vertical goniometer and Cu lamp.

The diffractometer is equipped with **Rigaku Cross Beam Optic System** (CBO) enabling either a divergent beam (Bragg Brentano focusing) or a parallel beam without changing the configuration and provides easy mounting of samples.

Both, scintillation and position sensitive D/tex detectors are available.

The SmartLab is equipped with the special Guidance Software which allows users to choose the proper configuration and automatically updates configuration of equipment. Multi-year license ICDD PDF4+ Data Base.

Minimum sample size:

The minimum weight of a powder sample is milligrams order.

Typical applications:

- Qualitative and quantitative phase analysis of polycrystalline materials at different temperatures (25–1100°C).
- Determination of crystal grain size and micro and residual strain.
- Texture analysis.
- In-situ structure determination at high temperatures up to 1100°C:
  - qualitative and quantitative phase analysis of polycrystalline materials,
  - the refinement of structural parameters with Rietveld method,
  - the evaluation of crystalline size and strains,
  - the analysis of textures with pole figure method,
  - the high-temperature in-situ measurements up to 1100°C.

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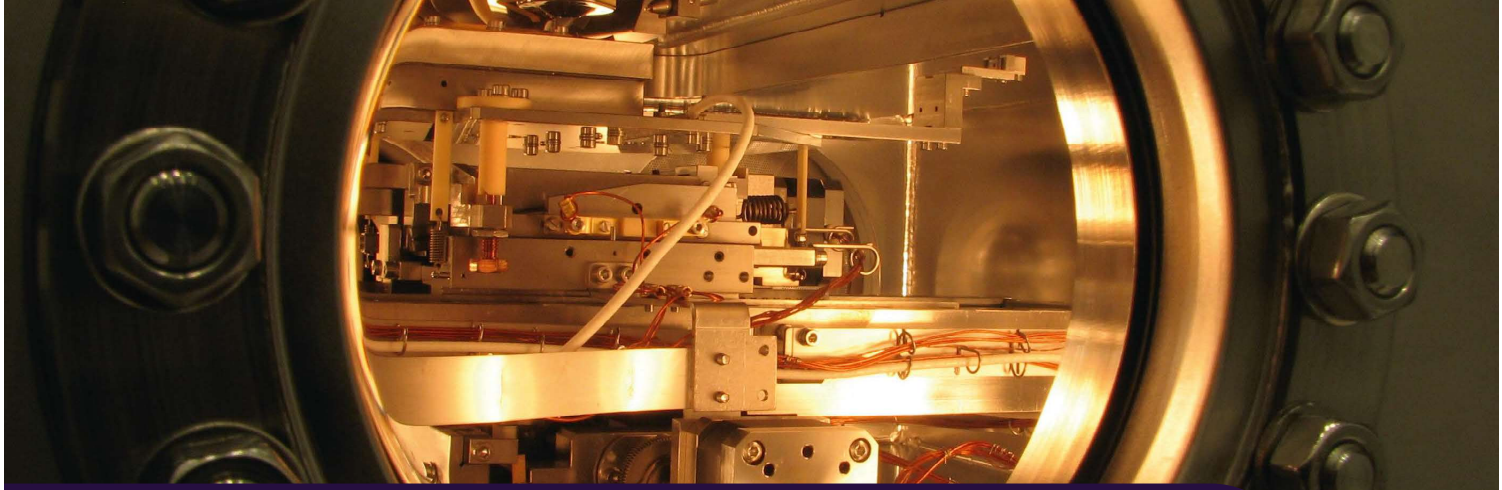

Secondary ion mass spectrometry (SIMS)

The Laboratory of Structural Research and Materials Characterization

Secondary ion mass spectrometry (SIMS)

Facilities:

- CAMECA IMS SC Ultra

Typical applications:

- Precise elemental analysis with sensitivity below 1 ppm.
- Depth profiling with subnanometer resolution.
- Lateral resolution of 1 µm.
- 3D analysis.

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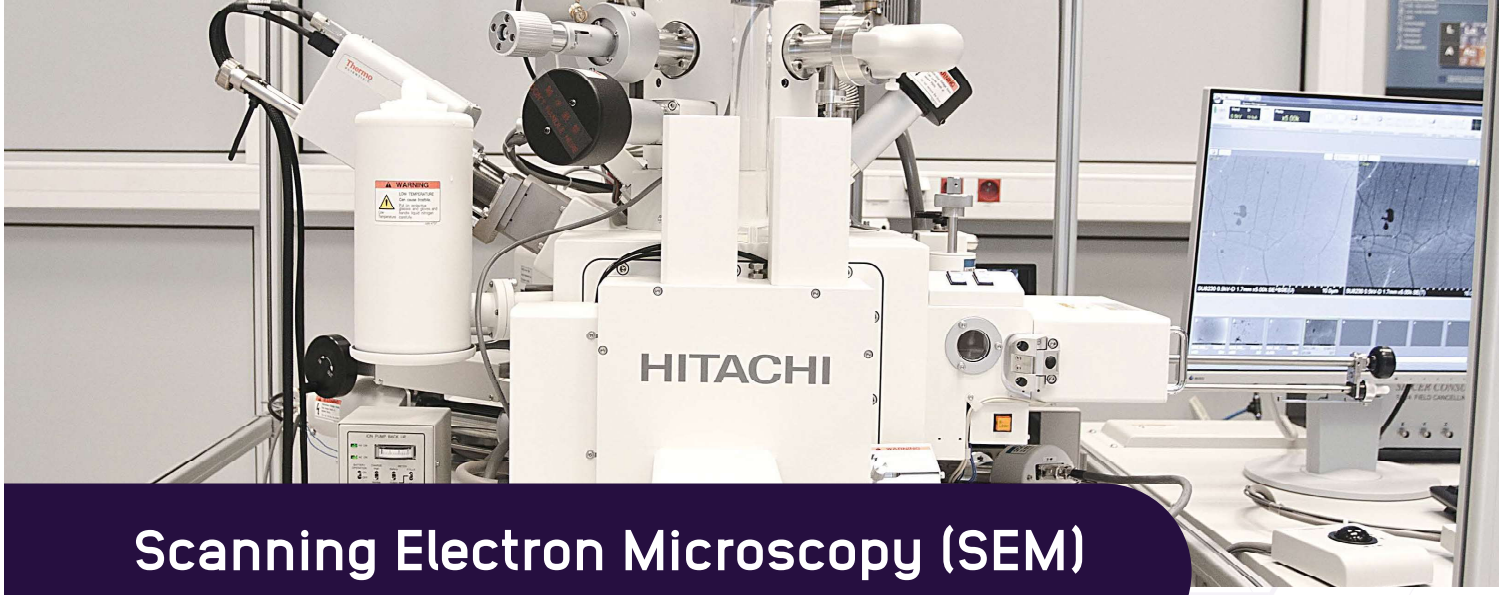

Scanning Electron Microscopy (SEM)

The Laboratory of Structural Research and Materials Characterization

Scanning Electron Microscopy (SEM)

Facilities:

- **Hitachi SU8230** equipped with Energy Dispersive X-Ray Spectroscopy (EDS) system and
- **Zeiss Auriga CrossBeam Workstation** equipped with Energy Dispersive X-Ray Spectroscopy (EDS), Cathodoluminescence (CL), Electron Backscattered Diffraction (EBSD) and Focused Ion Beam (FIB) systems.

Maximum sample size:

Diameter: 200 mm, height: 43 mm

Typical applications:

Surface topography visualization of any type of materials (metals, plastics, glasses and semiconductors) with:

- nanometer resolution,
- visualization of precipitates in multi-phase materials,
- elemental mapping (chemical composition),
- mapping of crystallographic orientation in polycrystalline materials,
- cross-sections of the materials for in-depth analysis of their structure,
- thin specimen preparation for transmission electron microscopy (TEM) analysis.

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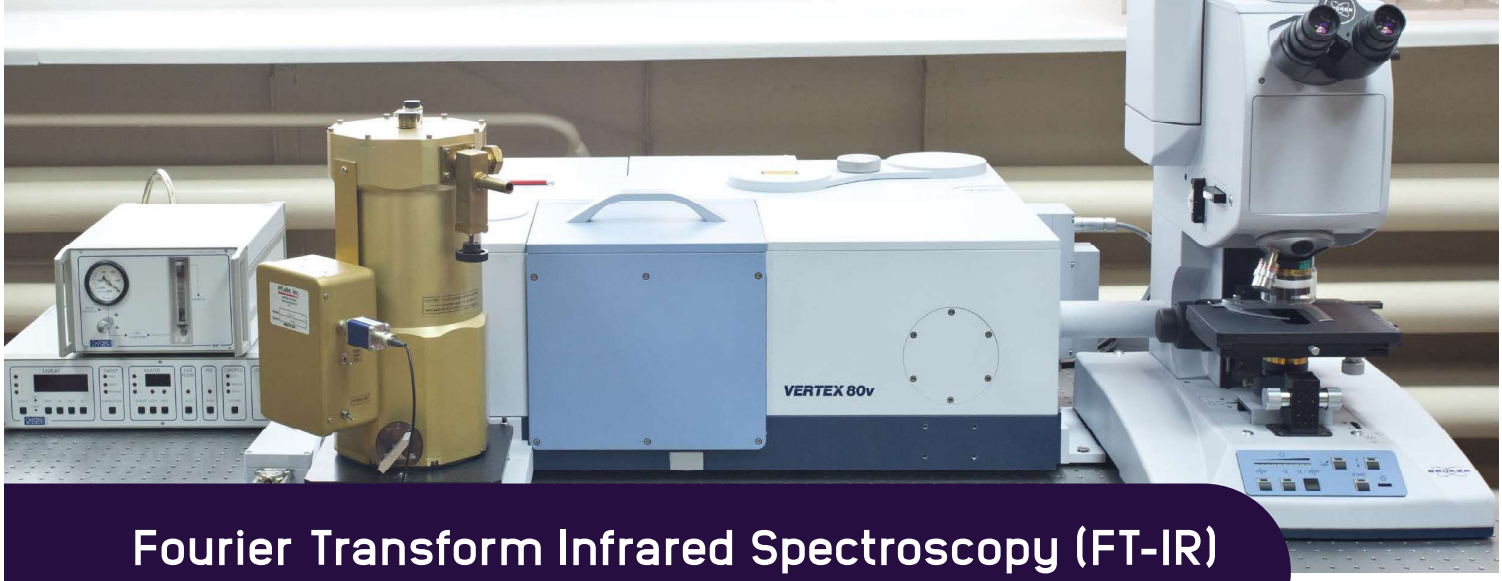

Fourier Transform Infrared Spectroscopy (FT-IR)

The Laboratory of Structural Research and Materials Characterization

Fourier Transform Infrared Spectroscopy (FT-IR)

Facilities:

VERTEX 80v vacuum FT-IR spectrometer

- Spectral range: 25000 – 10 cm^{-1}, spectral resolution 0.06 cm^{-1}.
- Measurement techniques: transmittance, specular reflectance, single reflection ATR and diffuse reflection.
- Equipped with IR microscope and set for transmittance measurement at liquid He temperatures.

Typical applications:

- Detection and characterization of thin and few-layer layers on various substrates.
- Analysis of passivation layers and epi-layers on semiconductors.
- Investigation of nano-structured materials.
- Characterization of optical and highly reflective materials (windows, mirrors).
- Determination of oxygen and carbon contents in silicon wafers.
- Measurements of shallow impurities and defects in semiconductors.
- Investigation of the chemical structure and functionality of materials.

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Fe-57 Mössbauer Spectroscopy

The Laboratory of Structural Research and Materials Characterization

Fe-57 Mössbauer Spectroscopy

Facilities:

- Constant acceleration **Mössbauer spectrometer** with Co-57 source, equipped with a CSW-202N Advanced Research System closed-cycle cryostat with a low vibration interface (Displex DMX-20).

Typical applications:

Qualitative and quantitative phase analysis, and determination of oxidation and magnetic states of iron in iron-containing materials, such as steels, amorphous and crystalline iron alloys and compounds, iron oxides, minerals, and meteorites.

Measurements and samples details:

- Measurements in transmission geometry performed in a wide range of temperatures from RT down to 10 K.
- Conversion electron Mössbauer spectroscopy (CEMS) measurements for phase analysis of a surface layer about 200 nm in depth.
- Samples for transmission measurements: powders or thin absorbers up to about 60 μm in thickness, diameter of 4-12 mm.
- Samples for CEMS measurements must fit into a holder with a diameter of 30 mm and be max. 5 mm thick.

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Raman spectroscopy

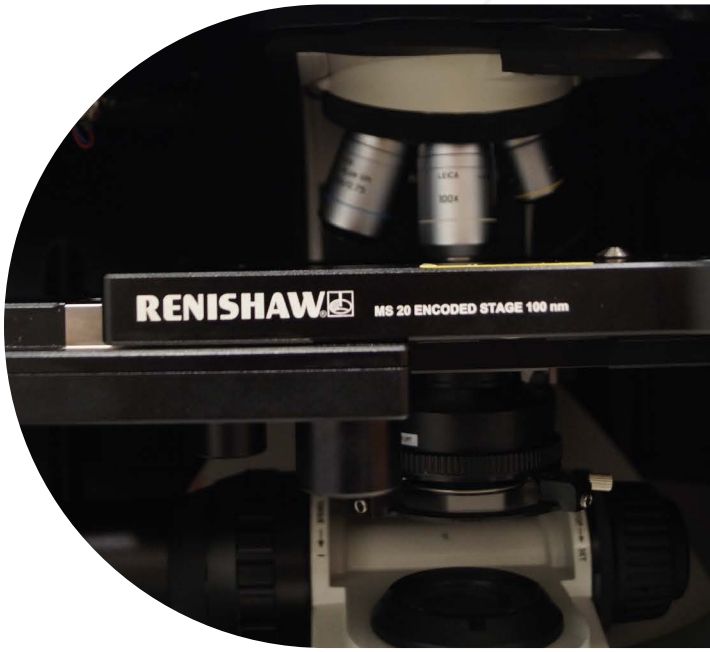
The Department of Graphene and Materials for Electronics

Renishaw inVia confocal Raman Microscope

Parameters:

- Excitation (cw): Lasers 532 nm, 633 nm, 1064 nm, 325 nm
  Configuration: Back-scattering
  Spatial resolution: 0.5 micron
  Spectral resolution: about 1.5 cm^{-1}
  Spectral range (Raman shift): 50 cm^{-1} – 6000 cm^{-1}
  Experimental temperature: ambient

- Sample size: 15 x 15 x 2 cm



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