Non-ambient X-ray Diffraction and Nanostructure Analysis
At Anton Paar we have a close relationship with our customers, are constantly searching for answers and pushing back physical and technical boundaries. We enjoy the challenge of research and development and take pride in our achievements. The result: customers receive instruments produced to the highest level of technical perfection, tailored to meet their specific requirements.

**Key factors in our success:**

- Highly motivated employees working on their own initiative
- More than 20 % of annual turnover invested in R&D
- Long-term investment and cultivation of key technologies
- An international sales and service network
- Efficient and respectful use of both natural and human resources
- A flat decision-making structure consisting of responsible teams
- A charitable foundation as the owner
- Thorough and continuous training of employees and apprentices

Anton Paar’s long tradition as a manufacturer of scientific instruments for X-ray studies has been characterized by innovation and the continuous integration of new technologies in our design concepts. Following our company philosophy, all improvements were made with the customer in mind, whether that be providing novel technical solutions or making the instruments easier to use.

Our large selection of non-ambient attachments for X-ray diffraction and our innovative small-angle X-ray scattering equipment are the result of this process.

Modern materials science demands sophisticated tools - we have them for you!
Non-ambient XRD attachments

- **DHS 900 | DHS 1100**
  For direct sample heating up to 900 °C | 1100 °C on four-circle goniometers

- **DCS 350**
  For XRD studies on four-circle goniometers in the temperature range of -100 °C to 350 °C

- **HTK 1200N**
  For XRD studies with environmental heating up to 1200 °C
  With **HTK 1200N Capillary Extension** for diffraction in transmission geometry up to 1000 °C

- **HTK 16N | HTK 2000N**
  For direct sample heating up to 1600 °C | 2300 °C

- **XRK 900**
  For XRD measurements of solid state and solid state-gas reactions up to 900 °C and 10 bar

- **HPC 900**
  For XRD measurements in various gases up to 100 bar and 900 °C

- **TTK 450**
  For XRD investigations in the temperature range of -193 °C to 450 °C

- **CHC plus+**
  For XRD studies under different temperature and humidity conditions

Mechanical testing

- **TS 600**
  Tensile stage for in-situ X-ray investigations of stress/strain phenomena at ambient conditions

Small-angle X-ray scattering

- **SAXSess mc² system**
  For SAXS studies of solid and liquid samples in the temperature range of -150 °C to 300 °C and in different atmospheres
A Unique Heating Attachment for Four-circle Goniometers

DHS 1100

The DHS 1100 is an advanced heating attachment for in-situ diffraction studies on four-circle goniometers up to 1100 °C. It fits all common four-circle goniometers, replacing the standard sample holder.

The instrument is extremely compact and lightweight. The design of the heating plate guarantees a high temperature uniformity and good position stability at elevated temperatures.

The unique dome-shaped X-ray window made of graphite allows the analysis of samples under vacuum and under inert gas conditions to avoid oxidation or other chemical reactions of the sample at high temperatures.

Extensive cooling of the dome and the DHS 1100 housing is achieved by using compressed air.

The unique design of the DHS 1100 is registered and provides all the features our customers have in mind - compactness, safety and high performance.

Typical applications

- Temperature-induced phase transition investigations
- Texture measurements
- Stress analysis
- Profile analysis
- Grazing incidence investigations
- High resolution studies
- Investigation of layered structures

Technical data

- Temperature range: 25 to 1100 °C
- Atmospheres: air, inert gas, vacuum (10^{-1} mbar)
- Diameter/Height/Weight: 128 mm/51 mm/450 g
- X-ray geometry: reflection
Low-temperature Attachment for
Four-circle Goniometers and XYZ Stages

DCS 350

The DCS 350 is a novel attachment for in-situ X-ray diffraction studies between –100 °C and 350 °C on four-circle goniometers and XYZ stages. The clever design of the instrument provides for a high temperature uniformity and good position stability of the sample over the whole temperature range.

The X-ray transparent dome allows investigations to be carried out under vacuum or under inert gas conditions to prevent chemical reactions of the sample at high temperatures or condensation at low temperatures. Samples are fixed with springs and are easily exchangeable.

The DCS 350 housing is temperature-controlled with water to avoid condensation at low temperatures. The layout of all supply hoses provides the best possible flexibility.

The combination of a liquid nitrogen flow control unit and a temperature control unit guarantees short cooling and heating cycles. High-precision temperature measurement is performed in the sample holder.

With the DCS 350 Domed Cooling Stage, Anton Paar offers another valuable analytical tool for materials scientists!

Typical applications

- Temperature-induced phase transition investigations
- Texture measurements
- Profile analysis
- Stress analysis
- Investigation of layered structures

Technical data

Temperature range: -100 °C to 350 °C
Atmospheres: air, inert gas, vacuum (10⁻¹ mbar)
Diameter/Height/Weight: 115 mm/65 mm/850 g
X-ray geometry: reflection
Environmental Heating for Homogeneous Sample Temperature

HTK 1200N | HTK 1200N Capillary

The HTK 1200N has been the attachment of choice for in-situ XRD studies on flat samples up to 1200 °C for many years. The novel capillary extension turns this well-known oven chamber into a powerful capillary heater.

Due to its environmental heater, there is virtually no temperature gradient in the sample, even in samples of up to 5 mm thickness.

The sample spinning option provides highly random grain orientation, which is necessary for good diffraction data quality and subsequent profile fitting routines.

The temperature sensor is located right underneath the sample in a protective ceramic sample holder, guaranteeing reliable and repeatable temperature measurement.

The alumina sample carriers can be easily exchanged and can accommodate various sample forms like powder samples, bulk samples as well as thin layers. With the capillary extension a variety of capillary holders and capillaries can be used, depending on the specific properties of a particular sample.

The HTK 1200N is the first “two-in-one” attachment for combined reflection and transmission high-temperature XRD!

Typical applications

- Structure determination
- Coefficients of thermal expansion
- Investigation of phase diagrams
- Studies of chemical reactions
- Dynamic structure changes
- Lattice parameter measurements

Technical data

- Temperature range: 25 to 1200 °C
- Atmospheres: air, inert gas, vacuum (10^{-4} mbar)
- X-ray geometry: reflection and transmission
Filament Heating
up to 2300 °C

HTK 16N | HTK 2000N

HTK 16N and HTK 2000N are strip-heater type chambers for X-ray studies with direct sample heating at very high temperatures.

Investigations can be carried out under vacuum or in various gases depending on the experiment and the used heating filament (Pt, Ta, W, C or others on request).

Pre-stressing of the heating strip with a sophisticated linear stage guarantees high stability of the sample position over the complete temperature range. Integrated alignment slits allow exact height alignment of the strip at all temperatures.

The new graphite heating filament with inert sample support platelets offers the advantages of better temperature homogeneity in the sample and a higher chemical resistance.

The front cover of the chamber has a bayonet catch for quick and easy sample exchange.

HTK 16N and HTK 2000N are well-proven instruments for many of our customers’ applications providing high-grade materials, superior design and simple handling.

Typical applications

- Structure analysis
- Mineralogical studies
- Investigations of chemical reactions
- Aging processes
- Annealing
- Crystallographic characterization

Technical data

Temperature range: 25 to 1600 °C (HTK 16N)
25 to 2300 °C (HTK 2000N)

Atmospheres: air/inert gas, up to 1600°C
vacuum (10^{-4} mbar), up to 2300°C

X-ray geometry: reflection
The XRK 900 is a well-proven reactor chamber for X-ray diffraction experiments up to 900 °C and 10 bar. Its robust and sophisticated design allows to perform studies of solid state and solid state/gas reactions at high temperatures and pressures.

The special arrangement of the electrical heater inside the furnace guarantees the absence of temperature gradients in the sample. Two thermocouples reliably measure and control the sample temperature.

For solid state/gas reactions, defined atmospheric conditions are an important precondition. The design permits homogeneous flushing with reaction gas as well as gas flow through the sample. The housing can be heated up to 150 °C to prevent condensation of reaction products.

The sample spinning option provides highly random grain orientation, necessary for good diffraction data quality and subsequent profile fitting routines. Different sample holders made of stainless steel or ceramics are available.

The XRK 900 is a unique tool for in-situ XRD investigations of solid state reactions - unmatched in robustness and performance.

Typical applications

- Dynamic structure changes
- Studies of solid state reactions
- Simultaneous investigation of structural and catalytic parameters of catalysts
- Analysis of materials which are unstable under ambient conditions
- Kinetic investigations of solid state reaction processes

Technical data

- Temperature range: 25 to 900 °C
- Pressure range: 1 mbar to 10 bar
- Atmospheres: air, inert gas, reactive gases, vacuum (1 mbar)
- X-ray geometry: reflection
High-Pressure Chamber for Solid-Gas Interaction

HPC 900

The HPC 900 is a novel chamber for X-ray diffraction experiments up to 100 bar and 900 °C. It allows users to perform studies of solid state and solid state-gas reactions in various gases including hydrogen.

HPC 900 High-Pressure Chamber features a ‘two-shell’ design. The compact inner shell is the pressure container, housing the sample, the heater and the reactive gas. The outer shell ensures that no hazardous gas escapes into the environment even in case of leakage from the inner part.

Although HPC 900 is designed as a high pressure vessel, it provides easy sample mounting without any tools. The sample cup can easily be detached from the sample holder, and cups made of different materials can be used.

The sophisticated furnace of HPC 900 creates excellent temperature uniformity around the sample. The temperature sensor is located directly underneath the sample inside the sample holder, which guarantees reliable measurement and control of the sample temperature.

HPC 900 High-Pressure Chamber enables entirely new studies in many scientific fields, among them hydrogen storage, fuel cell research and catalysis.

Typical applications

- In-situ study of crystal structure and chemical composition during
  - solid-gas reactions
  - gas absorption and desorption
  - temperature changes
- Investigation of materials for
  - hydrogen purification, hydrogen storage and fuel cells
  - solid state membranes
  - catalysts

Technical data

Temperature range: 25 to 900 °C
Pressure range: 1 mbar to 100 bar
Atmospheres: air, inert gas, hydrogen, various other gases
X-ray geometry: reflection
Low-temperature XRD Studies between −193 °C and 450 °C

TTK 450

The TTK 450 Low-Temperature-Chamber is a versatile sample stage for X-ray diffraction studies in the temperature range from −193 °C to 450 °C. Measurements may be carried out either under vacuum, air or inert gas conditions.

For work at low temperatures, liquid nitrogen is transferred by insulated hoses from a Dewar vessel to the chamber. The hoses are connected with a specially designed two-pipe ball connector which can be turned by approx. 180°, even if the apparatus is frozen.

The flow rate of liquid nitrogen can be automatically controlled by using the LNC Liquid Nitrogen Controller. This precise control unit in combination with the TCU 110 temperature control unit guarantees short cooling and heating cycles.

The TTK 450 sample holder is easily accessible. No realignment is necessary for high and low temperatures or for different sample holders.

The TTK 450 is compact in design and easy to operate - a well-established tool for XRD studies in the low temperature range!

Typical applications

- In-situ characterization of the crystal structure of pharmaceutical substances and food ingredients
- Changes in chemical composition during solid-solid and solid-gas reactions
- Accurate determination of coefficients for thermal lattice expansion
- Investigation of polymer materials
- Structure studies of samples with melting points near or below ambient temperature

Technical data

- Temperature range: -193 °C to 450 °C (liquid nitrogen cooling)
  -10 °C to 450 °C (compressed air cooling)
- Atmospheres: air, inert gas, vacuum (10⁻² mbar)
- X-ray geometry: reflection
CHC plus+ is a unique combination of the multi-purpose CHC Cryo & Humidity Chamber and an advanced relative humidity (RH) generator for in-situ X-ray diffraction studies at low and high temperatures as well as under controlled humidity conditions.

The gas humidifier is mounted directly on the CHC plus+ chamber and the humidity is controlled with a calibrated RH sensor located inside CHC close to the sample.

The chamber housing is temperature-controlled with a water bath.

This set-up together with the excellent control performance of the RH generator provides uniform and well defined humidity conditions around the sample.

All types of experiments can be done in one go without removing the sample. Easy sample preparation without the need for realignment after sample exchange considerably speeds up measurement preparations.

The large temperature range combined with the possibility to control the humidity around the sample make CHC plus+ the ideal tool for XRD studies of temperature- and humidity-induced changes of crystal structures.

**Typical applications**

- Temperature- and humidity-induced changes in pharmaceutical substances and food ingredients
- Polymorphism in APIs
- Hydration/Dehydration of zeolites and clay minerals
- Hardening processes in building materials

**Technical data**

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**TS 600**

The TS 600 Tensile Stage is the first commercial sample stage designed especially for in-situ X-ray investigations of structural changes in materials under mechanical load. It allows to study stress/strain phenomena in fibers, foils and thin films.

Because of its compactness and low weight, the instrument can be used on synchrotrons and laboratory X-ray diffractometers alike. And what’s more, it can be operated in transmission and reflection mode.

Two load cells are available for TS 600: a low-range, high-resolution one up to 5 N and a full-range load cell from 1 to 600 N. The cells can be easily exchanged and the cell type is automatically detected by the instrument.

TS 600 comes with user-friendly software for stage control and data acquisition. Elongation and force-controlled straining makes it possible to program complex load profiles, including cyclic straining. The data can be viewed online in different representations and exported in various formats.

**Typical applications**

- Stress/strain studies of fibers, foils and thin films
- In-situ structure analysis under mechanical load

**Technical data**

- Force range: 0.05 to 5 N (5N load cell), 1 to 600 N (600N load cell)
- Straining speed: 0.05 to 5 mm/min
- Diameter/Height/Weight: 155 mm/49 mm/1.3 kg
- X-ray geometry: reflection and transmission
Nanostructure Analysis

**SAXSess mc²**

SAXSess mc² is a high performance small- and wide-angle X-ray scattering system to analyze nanostructures in all different kinds of samples, from liquids (e.g. colloids, protein solutions) to solids (e.g. polymer films, nanocomposites).

Its compact design in combination with powerful X-ray sources ensures high-intensity and short measuring times. The unique block collimator together with advanced detection systems results in an outstanding overall resolution.

The TrueSWAXS feature allows the user to simultaneously derive information of the nanostructure and the phase state of a sample in a single (SWAXS) experiment. Dedicated software packages for quick and easy data processing and evaluation ensure that the user gets the most information out of the SAXS data.

A wide variety of temperature-controlled sample holders is available for studying almost any type of sample. High-throughput experiments are possible with dedicated autosamplers. The multi-purpose VarioStage is used for multi-directional sample positioning for nanography and to study the sample’s orientation. The precise GI-SAXS stage is applied for GI-SAXS and reflection studies of nanostructured thin-film samples.

The SAXSess mc² system: modular, compact and powerful!

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**Typical applications**

- Size and size distribution information
- Structure and mass determination
- Characterization of crystalline/amorphous phase ratio
- Pore analysis and surface-to-volume ratio
- Degree of association and degree of coiling
- Characterization of aggregation number and interaction behavior
- Analysis of (micro)emulsions
- Nanostructure analysis of surfaces

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**Technical data**

- q range: 0.03 nm⁻¹ to 28 nm⁻¹
- Typical measuring time: 1 to 30 minutes
- Sample volume: > 7 μl (typically 100 μl)
- Temperature range: -150 °C to 300 °C
- Atmospheres: vacuum, air, inert gas, humidity
Instruments for:
Density & concentration measurement
Rheometry & viscometry
Sample Preparation
Microwave synthesis
Colloid science
X-ray structure analysis
Refractometry
Polarimetry
High-precision temperature measurement