Anton Paar

Non-ambient X-ray Diffraction and Nanostructure Analysis

Sample Stages
Overview

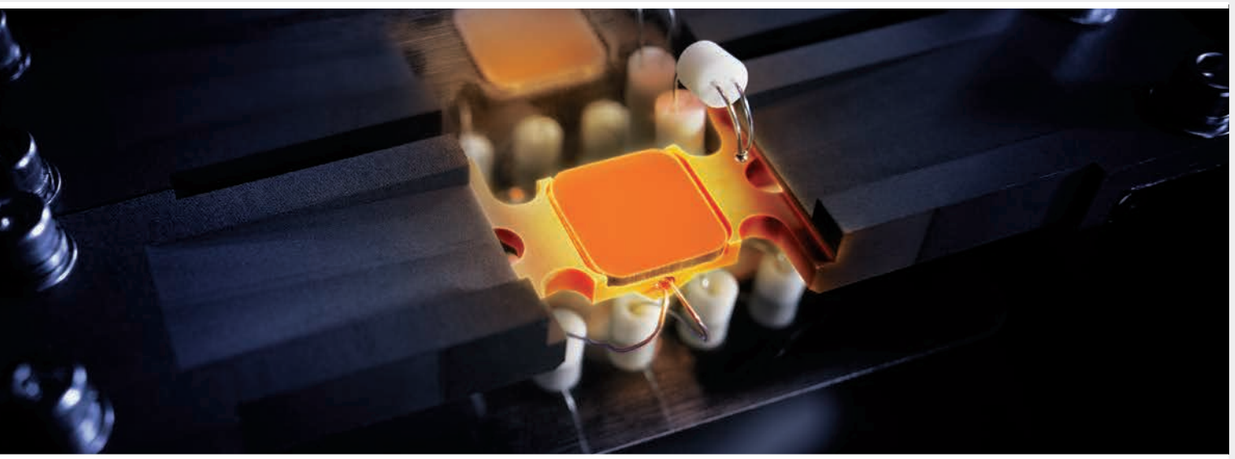
Setting the Pace in Non-ambient XRD

Non-ambient X-ray diffraction (XRD) has become an indispensable technique to understand the influence of temperature, atmosphere or pressure on materials of any kind. Besides its relevance for conducting research, this knowledge is essential for optimizing technical processes and performing quality control in industry.

The focus of interest in an XRD experiment is the sample and this deserves sample stages optimized for the specific application. Sample stages from Anton Paar ensure a precisely controlled sample environment in terms of temperature, atmosphere and pressure.
Automatic sample height compensation also guarantees the correct geometrical position of the sample in the X-ray beam throughout the experiment. Use of high-quality materials avoids unwanted chemical reactions between sample and stage components and ensures the long lifetime of the equipment.

Due to their popularity Anton Paar stages fit to all commercial diffractometers and are fully integrated solutions in their software and hardware for ease of use.

Best in performance, best in results - non-ambient attachments by Anton Paar.



	HTK 1200N | HTK 16N | HTK 2000N | DHS 1100	High temperatures
	TTK 600 | DCS 500 | BTS 150/500	Low to medium temperatures
	XRK 900 | HPC 900	High temperatures & high pressures
	CHC plus^{+} | MHC-trans	Humidity
	TS 600 | SAXSpace, SAXSpoint 2.0	Stress/strain | SAXS

Heating and cooling technology

Temperature is the most important parameter in non-ambient XRD. Depending on the sample type, the temperature and atmosphere two different methods of heating are applied:

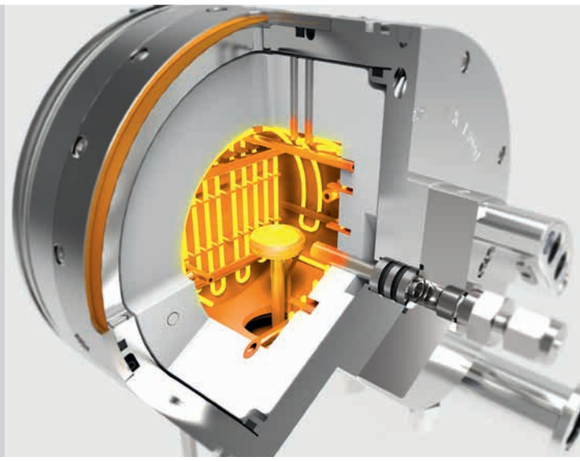
With **radiation heating** the sample is placed on a sample holder which is surrounded by the heater. This results in a homogeneous temperature distribution measured by a thermocouple close to the sample. The sample holder is made of an inert material to avoid unwanted chemical reactions and is often rotated to improve data statistics.

To reach very high temperatures **direct heating** is applied to the sample which is placed on a heating strip or heating plate usually made of metal. The direct contact between sample and heater allows fast temperature changes. Chemical reactions are avoided by using inert sample carriers.

Radiation heating

- Homogeneous sample temperature
- Sample holders made of inert material, no reaction with sample
- Sample rotation for better statistics
- Operation in oxidizing or reducing atmospheres, vacuum
- For any sample type

Unbeaten temperature homogeneity!



Direct heating

- Highest sample temperatures
- Fast temperature changes
- Sample holders made of inert material available
- Operation in oxidizing or reducing atmospheres, vacuum
- Particularly for fine powders

For high temperatures!

Heating and cooling

- Low and medium sample temperatures
- Easy handling
- Operation in air, inert gas, vacuum
- Lowest temperatures in vacuum
- For any sample type

Multi-purpose instruments!

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.

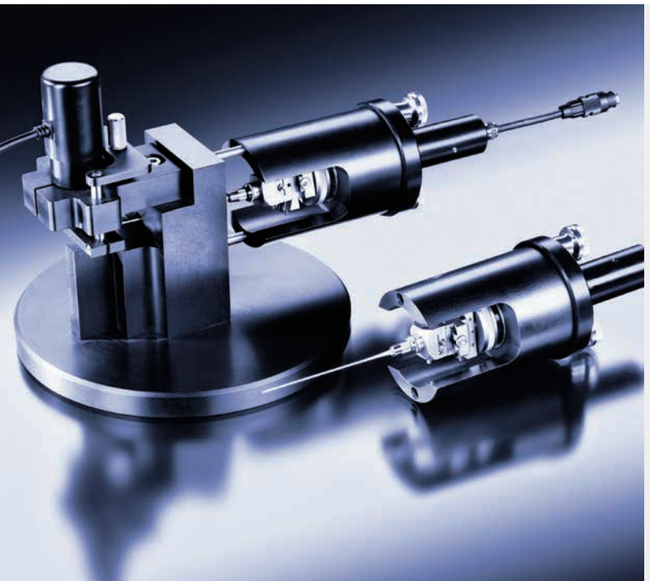
The sample spinning option provides highly random grain orientation, which is necessary for good diffraction data quality and subsequent profile fitting routines.

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 °C 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).

The design of the chambers is optimized for minimum temperature gradients in the sample. A thermocouple, spot-welded onto the heating strip, provides reliable and accurate temperature measurement and control under all operating conditions.

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 graphite heating filament with inert sample support plate 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 °C to 1600 °C (HTK 16N) 25 °C 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

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. Due to the compact design with a minimum of supply hoses, the DHS 1100 does not restrict the movements of the goniometer.

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.

The replaceable sample holder plate of the DHS 1100 is made of aluminum nitride (AlN), a material with outstanding thermal conductivity and high chemical resistance.

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 you need: 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 °C 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 XRD Studies between -190 °C and 600 °C

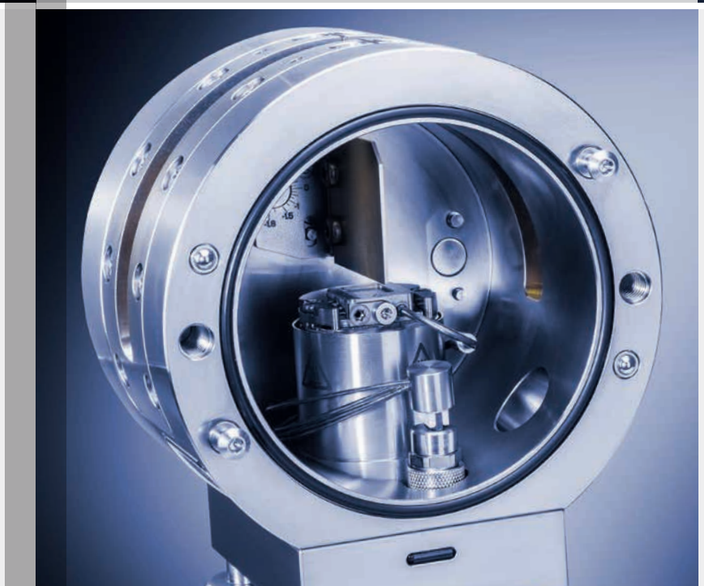
TTK 600

The TTK 600 Low-Temperature Chamber is a versatile sample stage for X-ray diffraction studies in the temperature range from -190 °C to 600 °C. Measurements may be carried out under vacuum, air or inert gas conditions.

Three types of sample holders ensure maximum flexibility concerning sample types and measuring geometries: the standard sample holder allows studies in reflection geometry. The optional capillary and transmission sample holders are used to investigate powders, foils and paste samples in transmission geometry.

The sample temperature is measured with an accurate Pt100 sensor right underneath the sample. The heat transfer between heater and all sample holders is optimized by design. The capillary and the transmission sample holders both work with additional convection heaters to enable fast temperature changes and improved temperature distribution. The CCU Combined Control Unit guarantees maximum temperature stability and economical use of liquid nitrogen or compressed air, respectively.
The different sample holders can be easily exchanged without Z-alignment. Air-sensitive samples can be safely transferred into TTK 600 by using an antechamber.

TTK 600 is a unique all-rounder 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

Technical data

Temperature range:	-190 °C to 600 °C (liquid-nitrogen cooling) -10 °C to 600 °C (compressed-air cooling)
Atmospheres:	air, inert gas, vacuum (10^{-4} mbar)
X-ray geometry:	reflection and transmission

Low-temperature Attachment for Four-circle Goniometers and XYZ Stages

DCS 500

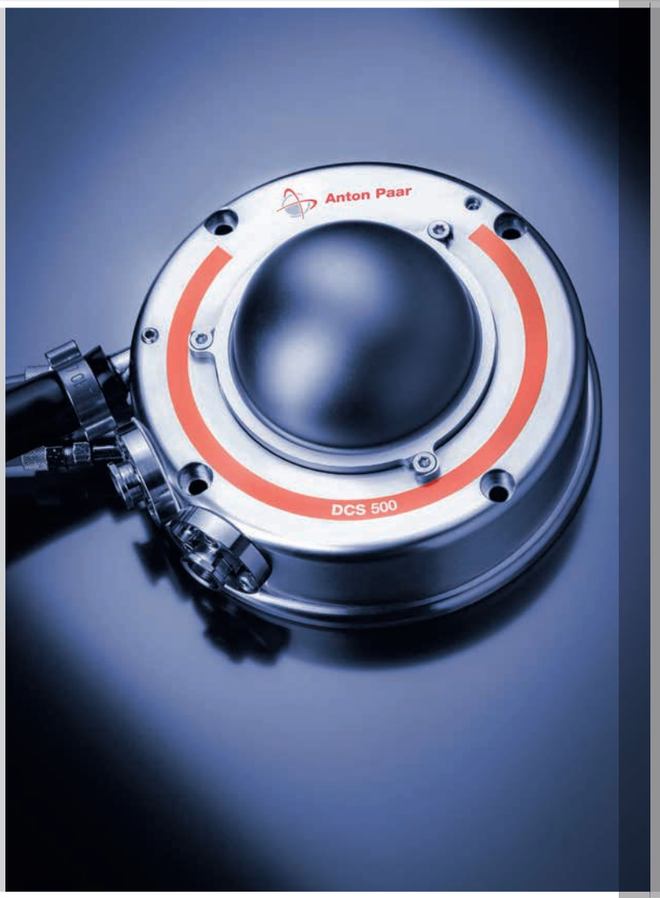
The DCS 500 is a novel attachment for in-situ X-ray diffraction studies between -180 °C and 500 °C on four-circle goniometers and XYZ stages. The clever design of the instrument provides for a high temperature uniformity and good position stability of the sample over the whole temperature range.

The X-ray transparent dome, made of graphite, allows samples to be investigated in controlled atmospheres. Vacuum or inert gas prevent chemical reactions of the sample at high temperatures or condensation at low temperatures. Cooling or heating of the dome is not necessary.

The DCS 500 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, induced by the underpressure of a venturi nozzle, and a temperature control unit guarantees short cooling and heating cycles. High-precision temperature measurement is performed in the sample holder.

With the DCS 500 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:	-180 °C to 500 °C
Atmospheres:	air, inert gas, vacuum (10^{-1} mbar)
Diameter/height/weight:	115 mm/65 mm/850 g
X-ray geometry:	reflection

In-situ XRD Studies on Benchtop Diffractometers

BTS 150/500

The BTS 150/500 Benchtop Heating Stages are the first commercial non-ambient stages for benchtop diffractometers and extend their applicability to in-situ XRD studies. Measurements can be performed between -10 °C and 150 °C with BTS 150 and from ambient to 500 °C with BTS 500.

Both instruments are extremely compact in design to fit into the restricted space of typical benchtop diffractometers. The control electronics are integrated in the heating stages and provide easy operation.

Samples are heated by a Peltier (BTS 150) or a resistance heater (BTS 500). Excellent insulation and air cooling avoid heat transfer to any components of the benchtop diffractometer. The Pt100 temperature sensor is located close to the sample and guarantees reliable and repeatable temperature measurements.

The BTS 150/500 sample holders are easily accessible. Studies may be carried out either under vacuum, air or inert gas conditions. The instrumental design guarantees minimum thermal expansion of the sample holder and therefore a correct geometrical position of the sample in the X-ray beam throughout the experiment.

The unique design of BTS 150/500 is patented and provides all the features required by benchtop diffraction applications: compactness, reliability and ease of use!

Typical applications

- In-situ phase characterization
- Structure determination
- Phase properties (cell parameters, crystallite size, lattice strain)
- Dynamic structure changes
- Rietveld analysis

Technical data

Temperature range:	-10 °C to 150 °C (BTS 150) ambient to 500 °C (BTS 500)
Atmospheres:	air, inert gas, vacuum (10^{-1} mbar)
X-ray geometry:	reflection

In-situ XRD Investigations of Solid State Reactions

XRK 900

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 you to perform studies of solid state and solid state-gas reactions at high temperatures and pressures.

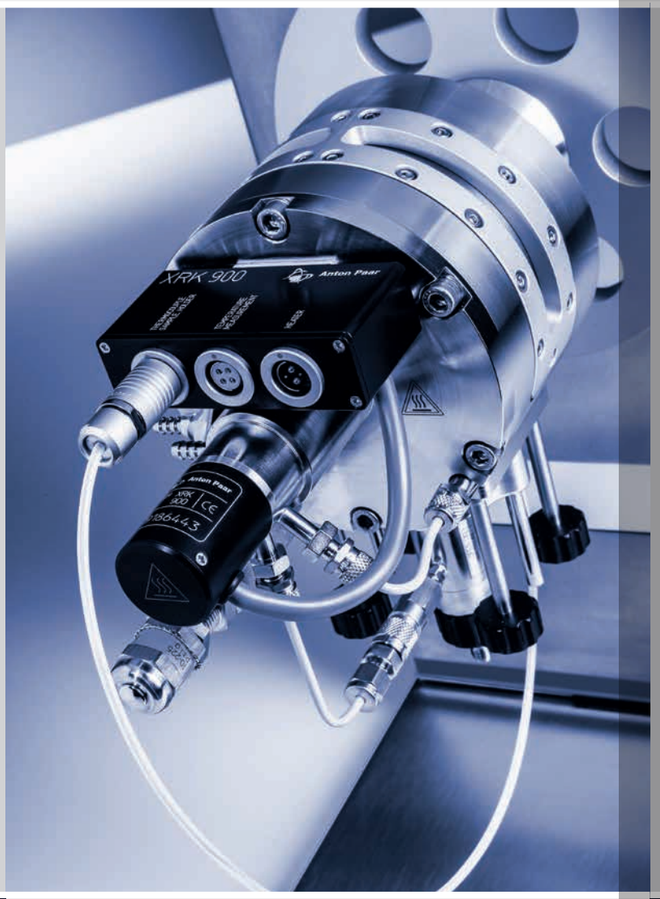
The special arrangement of the electrical heater inside the furnace guarantees the absence of temperature gradients in the sample. Two thermocouples reliably measure and control the sample temperature.

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 °C 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, temperature changes, gas absorption and desorption
- Investigation of materials for hydrogen and carbon dioxide storage, fuel cells, solid state membranes, catalysts

Technical data

Temperature range:	25 °C to 900 °C
Pressure range:	1 bar to 100 bar
Atmospheres:	hydrogen, carbon dioxide, oxygen, nitrogen, air, inert gas, other gases on request
X-ray geometry:	reflection

Studies Under Controlled Temperature and Relative Humidity Conditions

CHC plus+

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 setup 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

RH range:	5 to 95 %RH from 10 °C to 60 °C 5 to 70 %RH at 80 °C
Temperature range:	-180 °C to 400 °C (vacuum) -120 °C to 300 °C (dry air)
Atmospheres:	(humid) air, inert gas, nitrogen, vacuum (10^{-2} mbar)
X-ray geometry:	reflection

Multi-sample Chamber for In-situ Transmission XRD under Controlled Humidity

MHC-trans

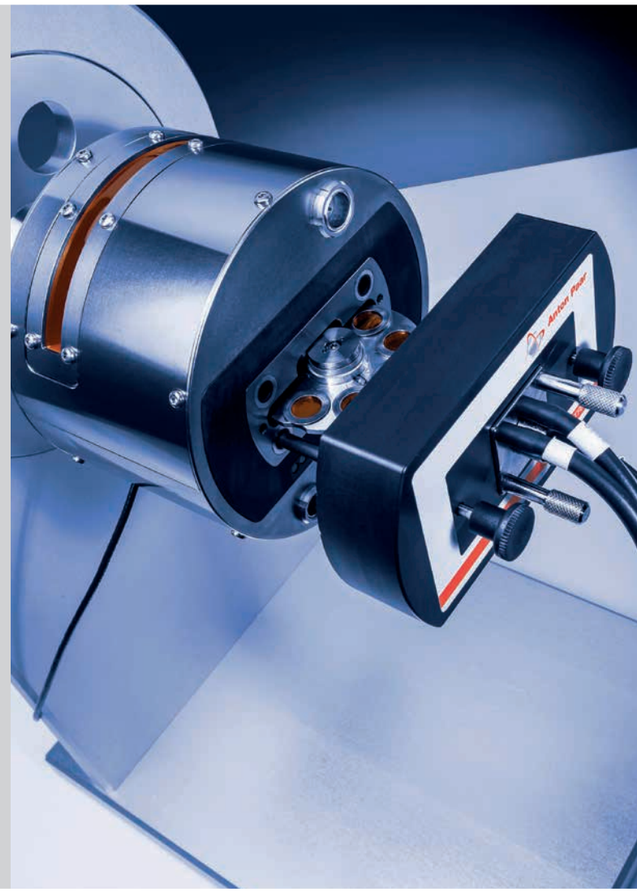
MHC-trans Multi-sample Humidity Chamber is the first multi-sample stage for in-situ powder XRD with perfect control of sample temperature and humidity. Due to the integrated sample changer you can investigate up to 8 samples simultaneously. All samples are kept under the same conditions and each sample can be moved into the X-ray beam to record a diffractogram.

MHC-trans has been designed for diffraction in transmission geometry with focusing beam optics to achieve higher resolution, better signal-to-noise ratio and less sensitivity to volume changes of the sample. MHC-trans can be operated with the X-ray source above or below the sample stage.

MHC-trans features accurate and uniform temperature control of the complete sample compartment. The device for humid gas generation and the humidity sensor are integrated in the sample chamber, providing a humidity control range of 5 %RH to 95 %RH.

To provide easy integration in the diffractometer hardware and software, the TCU 60M temperature control unit and the MHG humidity generator can be remote controlled via one common interface on TCU 60M.

MHC-trans is the first non-ambient sample changer which allows you to boost your in-situ XRD.



Typical applications

- Temperature- and humidity-induced phase changes in pharmaceutical substances and food ingredients
- Polymorphism in APIs
- Hydration/dehydration of crystalline materials

Technical data

Humidity control:	5 to 95 %RH from 10 °C to 60 °C, up to 80 °C with max. 70 %RH
Temperature range:	-10 °C to 150 °C
No. of samples:	max. 8
Atmospheres:	(humid) air/nitrogen, inert gas
X-ray geometry:	transmission

In-situ Studies of Mechanical Material Properties

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 you to study stress/strain phenomena in fibers, foils and thin films.

Because of its compactness and low weight, the instrument can be used on both synchrotrons and laboratory X-ray diffractometers. 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 N 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.

TS 600 — gain new insights into the world of strain and stress!

Typical applications

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

Technical data

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

Nanostructure Analysis

SAXSpace / SAXSpoint 2.0

SAXSpace and SAXSpoint 2.0 are high-performance small- and wide-angle X-ray scattering systems to analyze nanostructures in all different kinds of samples, from liquids (e.g. colloids, protein solutions) to solids (e.g. polymers, nanocomposites).

Their compact design in combination with powerful X-ray sources ensures high intensity and short measuring times. Their unique, scatterless collimation setups combined with advanced detection technology result in outstanding resolution and performance of both systems.

The TrueSWAXS feature allows you to simultaneously derive information on 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 you get 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 multipurpose 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 on nanostructured thin-film samples.

SAXS systems from Anton Paar: versatile, compact and brilliant!

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 nanostructured surfaces

Technical data

q range:	0.02 nm^{-1} to 40.7 nm^{-1}
Typ. meas. time:	1 to 30 minutes
Sample volume:	> 8 µL (typically 50 µL)
Temperature range:	-150 °C to 500 °C
Atmospheres:	vacuum, air, inert gas, humidity

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