

# Non-ambient

- Diffraction Solutions

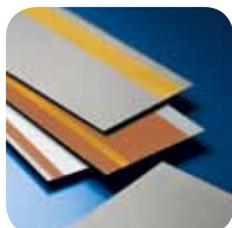
# Non-ambient X-ray diffraction – extract more from your sample



Loose powder



Pharmaceuticals



Coatings and films



Capillary



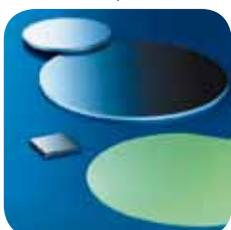
Filters



Small sample amounts



Multilayer nanostructures



Coatings

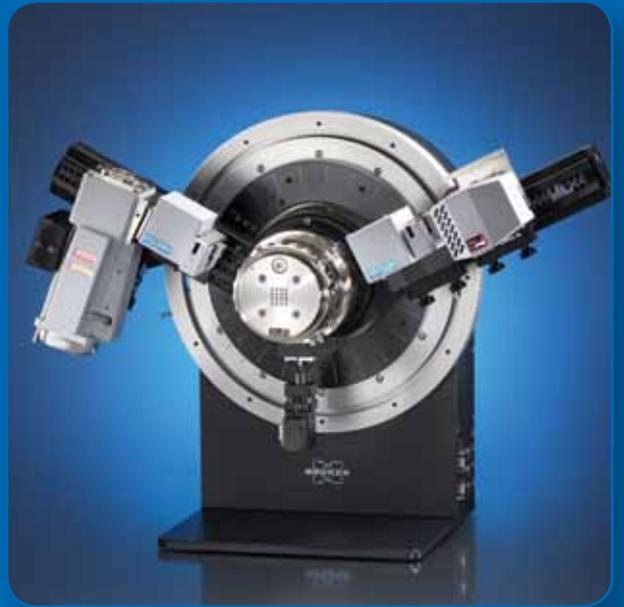
Synthesis of ceramics through sintering at elevated temperatures under controlled atmosphere, steel hardening through elaborate annealing steps, the effect of humidity and temperature on the stability of pharmaceutical compounds... Detailed knowledge of what happens to a sample at non-ambient conditions is essential for optimizing industrial processes, performing quality control, or for conducting research.

Since it all comes down to the formation of new phases, changes in microstructure, or optimization of stress and texture properties... X-ray diffraction is the predestined method to gather that knowledge.

Our state-of-the-art X-ray diffractometers, D8 ADVANCE and D8 DISCOVER, enable fast data acquisition to efficiently study kinetics and thermodynamics at non-ambient conditions. All that is required is an additional, dedicated sample stage to study the effect of parameters like temperature, atmosphere, humidity or pressure. Our large portfolio of in-house stages covers a vast range of non-ambient conditions and fits seamlessly to the DAVINCI design.

As you may expect of an integrated solution, each non-ambient stage is fully software supported: from setting up a measurement to the final data evaluation.

**Bruker AXS – competence in  
all aspects of non-ambient  
diffraction!**



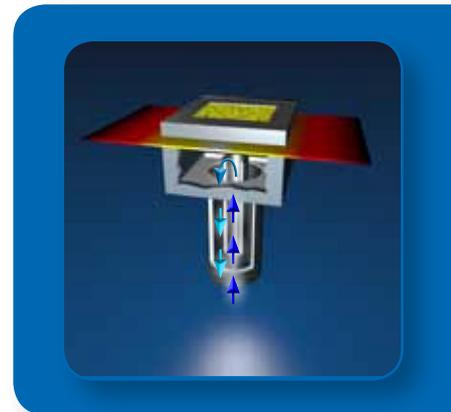
# Non-ambient chambers – different heating/cooling technologies

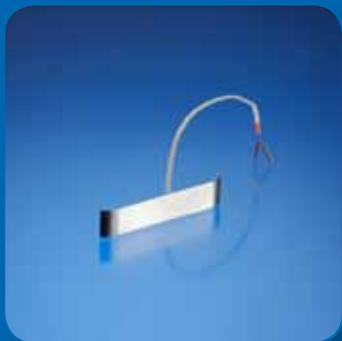
Of all non-ambient parameters, temperature is the one that is most often of interest. Heating the sample can be realized in different ways, depending on the sample type, temperature range, and atmospheric conditions.

With direct heating, the sample is mounted on the heater and is heated by means of thermal conduction. The heater itself is a metallic strip, typically made of tantalum for working in vacuum, or platinum for working in vacuum or oxidative atmosphere. The temperature is measured with a thermocouple welded at the back of the heating strip. Direct heating is mainly used for powder samples, provided they do not react with the heating strip.

Alternatively, the sample can be heated by radiation. In this case the heater surrounds the sample to achieve excellent temperature uniformity. Radiation heating is also appropriate for heating bulk samples and thin films. The temperature is measured with a thermocouple close to the sample. By design, a reaction between the sample and the heater material is avoided.

An actual high-temperature chamber may use one of these methods for heating the sample or even combine them. In addition, a non-ambient chamber may combine sample heating with cooling for extending the temperature range, with humidity, or with other non-ambient conditions.





### Direct heating

- for achieving high temperatures
- easy sample handling
- for fine powder samples
- fast temperature ramping
- for operation in air, inert gas or vacuum (depending on heater material)



### Combined direct and radiation heating

- for achieving highest temperatures
- for powder samples
- fast temperature ramping
- for operation in vacuum



### Radiation heating

- uniform sample temperature
- sample rotation for enhancing crystallite statistics
- for powder, bulk and thin film samples
- inert sample holder, no reaction between sample and heater material
- for operation in air, vacuum or inert gas (depending on heater material)



### Heating and cooling

- low and medium-high temperature range with one setup
- compact design
- for powder and bulk samples
- lowest temperatures achieved in vacuum



# Modular non-ambient chambers – extend your capabilities

Non-ambient conditions can be as diverse as the sample characteristics! That's why our non-ambient chambers are based on a uniquely modular, platform-based design.

A common platform serves as the basis for a series of different non-ambient stages and consists of a chamber lid, a chamber body and a chamber base plate. The water-cooled chamber lid can be easily removed without tools for quick access to the sample. The chamber body has a thin Kapton window to allow X-ray entrance and exit. The water-cooled base plate carries the electrodes and all required connectors. This common chamber platform is mounted on a manual or motorized height alignment adapter that fits directly onto the goniometer.

Starting from the common platform, dedicated temperature chambers can be easily configured by simply adding appropriate sample holders and heaters:

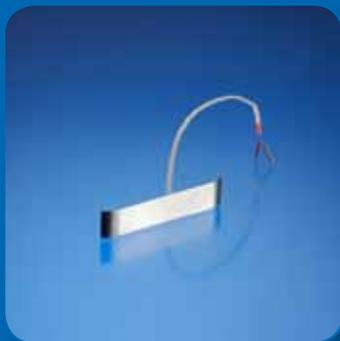
- MTC-HIGHTEMP for fast direct heating
- MTC-HIGHTEMP+ for achieving highest temperatures by combining direct and radiation heating
- MTC-FURNACE for best temperature uniformity by radiation heating
- MTC-LOWTEMP for heating and cooling

Converting one chamber type into another one is straightforward. In just a couple of minutes, you're ready to start a different application.

**Modular chamber design – tailored for today, flexible for the future!**



## Easy configuration



### MTC-HIGHTEMP

- direct heater
- PtRh for use in vacuum and oxidative atmosphere up to 1600°C
- optional Ta for use in high vacuum up to 1500°C
- pre-stressed heating strip for compensating thermal expansion



### MTC-HIGHTEMP+

- combination of direct and radiation heating for optimum uniformity at highest temperatures
- Ta heaters for use in vacuum up to 2000°C
- easy sample preparation – radiation heater can be moved down



### MTC-FURNACE

- radiation heater
- AlCr heater for use in vacuum or oxidative atmosphere up to 1100°C
- optional Ta heater for use in high vacuum up to 1300°C
- chemically inert sample spinner made of  $Al_2O_3$



### MTC-LOWTEMP

- heating and cooling chamber
- Ni-coated Cu sample holder
- down to -180°C in vacuum with  $LN_2$  cooling
- +300°C in air, +450°C in vacuum

# Dedicated non-ambient solutions – experts for your application

Characterization of complex materials under actual-usage conditions is a must in today's materials research and quality control. For example, the temperature dependence of pharmaceuticals and thin multilayer structures is very important for product consistency and long-term stability. In order to produce the appropriate sample environment, dedicated non-ambient stages are required. With the TC-series, Bruker AXS offers a large selection of application optimized non-ambient solutions.



## TC-REFLECTOMETRY

This temperature chamber is specifically designed for X-Ray Reflectometry (XRR) and thin film diffraction at high and low temperatures. It is equipped with a Knife Edge Collimator (KEC) to properly define the irradiated sample area. The sample is mounted on a stable Inconel sample carrier, surrounded by a radiation heater.

- from ambient temperature up to 800°C in vacuum and oxidizing atmosphere
- optionally down to -180°C
- precise alignment of the KEC height, sample height and tilt



## TC-DOME

The dome-type temperature chamber can be mounted directly to a Eulerian cradle or Universal Motion Control (UMC) stage. The X-ray transparent beryllium dome enables a large field of view. This is ideal for texture measurements on thin film samples and for combination with a 2-D detector.

The TC-DOME is equipped with a strip heater for heating powder samples up to 1400°C, or optionally for heating bulk and thin film samples up to 1100°C.

- modular design
- hemispherical Be dome for scatter-free background and high vacuum operation
- optionally down to -180°C



## TC-HUMIDITY

This chamber is for XRD investigations at different humidity levels and elevated temperature. The humid air is blown directly over the sample to guarantee accurate humidity conditions at the sample.

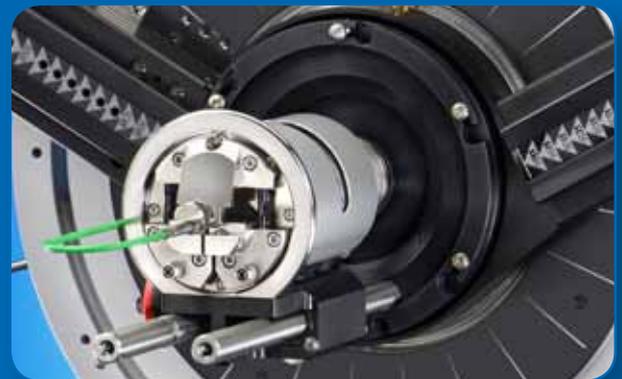
- patented technology
- relative humidity range 5% to 95% RH, dew point  $-5^{\circ}\text{C}$  to  $75^{\circ}\text{C}$
- from ambient temperature up to  $250^{\circ}\text{C}$  in dry atmosphere
- humidity sensor directly at the sample holder
- single "all-in-one" control unit



## TC-TRANSMISSION

The TC-TRANSMISSION is a compact furnace for high temperature XRD investigations in transmission mode. The sample is prepared in a glass capillary, which can be spun during measurement to enhance statistics.

- from ambient temperature up to  $1000^{\circ}\text{C}$
- excellent sample temperature uniformity



## KRYOFLEX II

The KRYOFLEX II generates a highly stabilized low-temperature nitrogen gas-stream to cool single crystals or powder samples prepared in capillaries.

- heat-exchanger principle,  $\text{LN}_2$  evaporation
- temperature range from 80 K to 400 K
- low nitrogen consumption



# Non-ambient XRD – a fully integrated solution



TWIN: Motorized slit

TWIN: Motorized slit



TWIN: Göbel mirror

TWIN: Equatorial Soller



DAVINCI SNAP-LOCK



LYNXEYE 1-D detector



VÅNTEC-1  
1-D detector



VÅNTEC-500  
2-D detector

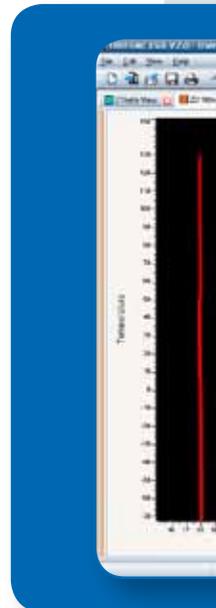
Studies of phase transitions benefit from high intensity and fast data collection; this can be achieved with the focusing Bragg-Brentano geometry in combination with a 1-D linear detector, such as LYNXEYE or VÅNTEC-1. When thermal expansion or compression is of interest, lattice parameters need to be determined very accurately. For such studies, parallel-beam geometry is preferred since this setup is insensitive to sample height displacement. Both geometries can be easily realized thanks to our DAVINCI design. This pioneering diffractometer concept eliminates the problems of awkward configuration and adjustments once and for all.

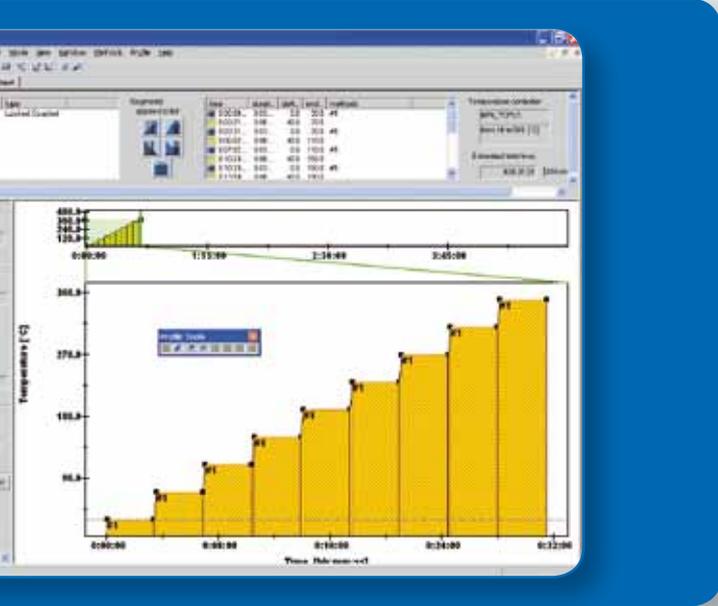
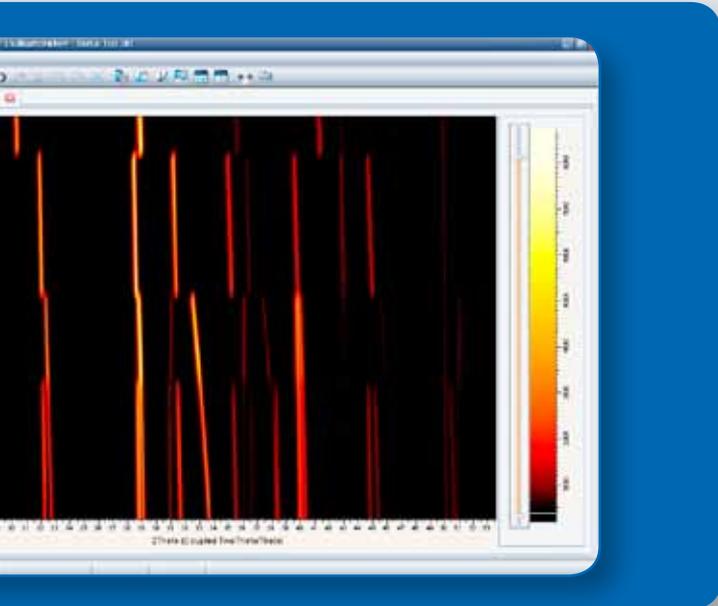
Our non-ambient chambers are an integral part of the DAVINCI design. Exchange with other sample stages is fast and reproducible thanks to the bayonet interface. Each chamber is automatically identified when mounted. A manual or motorized height adjustment brings the sample to the center of the goniometer. The motorized version even enables automatic compensation of the sample height displacement due to thermal expansion.

Inherent to the DAVINCI design, all our in-house temperature chambers are controlled by the same controller TCPU1. This compact controller includes a Eurotherm temperature control unit, a power unit, a cooling-water flow controller and a safety switch to detect a too high temperature at the chamber housing. The controller can be operated manually and, more conveniently, directly by our measurement software.

Complex temperature profiles, synchronized with XRD measurements, can be easily created in a graphical or table form with DIFFRAC.WIZARD. For subsequent data evaluation, the full power of DIFFRAC.SUITE is at your command.

**DAVINCI design – non-ambient conditions under your control!**





## Technical Data

	Modular temperature chambers <sup>1)</sup>				Dedicated temperature chambers				
	MTC-HIGHTEMP	MTC-HIGHTEMP+	MTC-FURNACE	MTC-LOWTEMP	TC-REFLECTOMETRY	TC-TRANSMISSION	KRYOFLEX II	TC-HUMIDITY	TC-DOME
<b>Heating/cooling method</b>	Direct	Combined direct and radiation	Radiation	Direct	Radiation	Radiation	Heat-exchanger, LN <sub>2</sub> evaporation	Direct	Direct
<b>Temperature range</b>	RT to +1600°C	RT to +2000°C	RT to +1100°C	-180°C to +450°C	RT to +800°C	RT to +1000°C	80K to 400K	RT to +250°C	RT to +1400°C
<b>Atmosphere</b>	Vacuum, air, inert gas	Vacuum	Vacuum, air, inert gas	Vacuum, air, inert gas	Vacuum, air, inert gas	Air	Nitrogen	Air	Vacuum, air, inert gas
<b>Sample holder</b>	PtRh (or Ta)	Ta	Al <sub>2</sub> O <sub>3</sub> ceramic	Ni-plated Cu block	Inconel	Glass capillary	Glass capillary	Ni-plated Cu block	PtRh
<b>Heater</b>	PtRh (or Ta)	Ta	AlCr (or Ta)	AlCr	AlCr	Kanthal	–	Heating cartridge	PtRh
<b>Temperature controller</b>	TCPU1	TCPU1 PU1	TCPU1	TCPU1	TCPU1	TCPU1	KRYOFLEX II controller	Humidity controller	TCPU1

<sup>1)</sup> each of these chambers can be easily converted into another configuration

X-ray analysis system with humidified sample US 6 896 247 B2,  
 LYNXEYE turned 90°: EP 1 647 840 A2; EP 1 510 811 B1,  
 Primary TWIN: US 6 665 372, DE 10 141 958,  
 Secondary TWIN: US 7 983 389 B2,  
 MIKROGAP technology, VANTEC-1 and VANTEC-500: US 6 340 819 B1

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