Anton Paar

Measure,
what is measurable,
and make measurable
that which is not.

Galileo Galilei (1564–1642)

Instruction Manual

DHS 1100

Domed Hot Stage
Version PANalytical

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Contents

1 About the Instruction Manual 7
2 Safety Instructions 8
2.1 General Safety Instructions 8
2.1.1 Liability 8
2.1.2 Installation and Use 8
2.1.3 Maintenance and Service 9
2.1.4 Disposal 9
2.1.5 Returns 9
2.1.6 Precautions for Highly Inflammable Samples and Cleaning Agents 9
2.2 Radiation Safety 10
3 Overview 11
3.1 Description of the Instrument 11
3.2 Design of the Instrument 13
3.2.1 Dome 16
3.2.2 Housing and Internal Parts 17
3.2.3 Connection device 19
4 Supplied Items 20
5 Installation 21
5.1 Installation requirements 21
5.2 Unpacking the Instrument 21
5.3 Mounting the DHS 1100 on the Goniometer 22
5.4 Connecting the Cooling Air Supply 22
5.5 Connecting the Gas Supply/Vacuum Equipment 23
5.6 Connecting the Temperature Control Unit 23
5.6.1 Connecting the Heater Cable 23
5.6.2 Connecting the Temperature Sensor Cable 24
6 Alignment 25
7 Operation 26
7.1 Preparing the Instrument 26
7.2 Mounting the Sample 27
7.2.1 Temperature Homogeneity of the Sample 28
7.3 Mounting and Removing the Dome 28
7.4 Performing a Measurement 29
7.4.1 Before Starting 29
7.4.2 Creating the Sample Environment 30
7.4.2.1 Operation with Air 30
7.4.2.2 Operation with Non-Reactive Gases 30
7.4.2.3 Operation under Vacuum 31
7.4.2.4 Reactive, Explosive, and Poisonous Gases 33

7.4.3 Setting the Sample Temperature and Heating Rate 33
7.4.4 Recording the X-ray Scan 34
8 Maintenance 35
8.1 Checking the Dome 35
8.2 Replacing the O-rings 35
8.3 Technical Support 35
Appendix A: Technical Specifications 36
Appendix B: Temperature Control Data 39
Appendix C: Diffraction Patterns 42
Appendix D: Warranty 44
Appendix E: Accessories 45
Index 47

1 About the Instruction Manual

Thank you for buying this instrument from Anton Paar GmbH.

This instruction manual informs you about the installation and the safe handling and use of the instrument. Make sure that the instruction manual is easily accessible to all personnel involved with the instrument. Pay special attention to the safety instructions and warnings in this manual and on the instrument.

Symbols in this Instruction Manual

This instruction manual features the following symbols:

Warning:
This symbol calls attention to **the risk of accidental injuries or damage to goods**. Do not proceed until the indicated conditions for averting this threat are fully understood and met.

Caution:
This symbol calls attention to **the risk of instrument damage or measurement errors**. Do not proceed until the indicated conditions for averting this threat are fully understood and met.

Information:
This symbol calls attention to any **additional information** of use to the operator.

Radiation hazard:
This symbol calls attention to **hazard** caused by exposure to **dangerous radiation**. Do not proceed until the indicated conditions for averting this threat are fully understood and met.

Extreme temperatures:
This symbol calls attention to **hazard** caused by **extremely hot or cold parts** of the instrument. Do not proceed until the indicated conditions for averting this threat are fully understood and met.

2 Safety Instructions

- Read this instruction manual before using DHS 1100.
- Follow all information and instructions contained in this instruction manual to ensure the correct use and safe functioning of DHS 1100.

2.1 General Safety Instructions

2.1.1 Liability

- This instruction manual does not claim to address all safety issues associated with the use of the instrument and samples. It is your responsibility to establish health and safety practices and determine the applicability of regulatory limitations.
- Operate DHS 1100 with TCU 200 Temperature Control Unit from Anton Paar GmbH only.
- Anton Paar GmbH only warrants the proper functioning of DHS 1100 if no adjustments have been made to the mechanics, electronics, and firmware.
- Only use DHS 1100 for the purpose described in this instruction manual. Anton Paar GmbH is not liable for damages caused by incorrect use of DHS 1100.

2.1.2 Installation and Use

- DHS 1100 is **not** an explosion-proof instrument and therefore must not be operated in areas with risk of explosion.
- It is the responsibility of the customer to provide all the set-up (tubings, valves,..) necessary for proper work with the DHS 1100.
- The installation procedure should only be carried out by authorized personnel who are familiar with the installation instructions.
- Do not use any accessories or spare parts other than those supplied or approved by Anton Paar GmbH.
- Make sure all operators are trained to use the instrument safely and correctly before starting any applicable operations.
- In case of damage or malfunction, do not continue operating DHS 1100. Do not operate the instrument under conditions which could result in damage to goods and/or injuries and loss of life.
- Check DHS 1100 for chemical resistance to the samples and cleaning agents.

- Do not operate DHS 1100 with explosive or poisonous gases. Do not measure samples that evaporate explosive or poisonous gases.
- The results delivered by DHS 1100 not only depend on the correct function of the instrument, but also on various other factors. We therefore recommend to have the results checked (e.g. plausibility tested) by skilled personnel before consequential actions are taken based on the results.

2.1.3 Maintenance and Service

- Service and repair procedures may only be carried out by authorized personnel or by Anton Paar GmbH.

2.1.4 Disposal

- Concerning the disposal of DHS 1100, observe the legal requirements in your country.

2.1.5 Returns

- For repairs send the **cleaned** DHS 1100 unit to the local representative of your diffractometer manufacturer or to Anton Paar GmbH in Graz, Austria. If the stage cannot be cleaned from residues, material safety data sheets for all contaminations must be sent together with the instrument.

2.1.6 Precautions for Highly Inflammable Samples and Cleaning Agents

- Observe and adhere to your national safety regulations for handling the measured samples (e.g. use of safety goggles, gloves, respiratory protection etc.).
- Only store the minimum required amount of sample, cleaning agents and other inflammable materials near the DHS 1100.
- Do not spill sample material or cleaning agents or leave their containers uncovered. Immediately remove spilled sample material and cleaning agents.
- Make sure that the setup location is sufficiently ventilated. The environment of DHS 1100 must be kept free of inflammable gases and vapors. Keep a fire extinction unit within easy reach of the instrument.
- Ensure sufficient supervision of DHS 1100 during operation.

2.2 Radiation Safety

- The DHS 1100 represents an open system and is allowed to be operated only on diffractometers equipped with a radiation enclosure.
- Before starting the experiment, the user has to make sure that the radiation protection equipment corresponds to the local requirements.

3 Overview

3.1 Description of the Instrument

The DHS 1100 Domed Hot Stage is a heating attachment for four-circle X-ray diffractometers. It is designed for X-ray diffraction experiments in reflection geometry with sample temperatures from 25 °C to 1100 °C.

The unique design of the instrument provides:

- Sample temperatures from 25 °C to 1100 °C
- High temperature uniformity across the sample
- Easy sample mounting
- Compact design, low weight and high flexibility
- Versatile applications

The special, dome-shaped X-ray window of the DHS 1100 sample stage combines two important properties: gas-tight enclosure of the sample and X-ray transparency over the complete half space above the sample. Due to the gas tightness, samples can be investigated in vacuum or inert gas to avoid oxidation or other chemical reactions of the sample at high temperatures.

Due to the design of dome, heater and sample fixing, very low angles of incidence can be reached and the following types of X-ray measurements can be carried out:

- pole figures
- grazing incidence diffraction
- grazing incidence in-plane diffraction

Cooling of the dome and the housing of the DHS 1100 attachment is achieved by using compressed-air. The cooling air supply is controlled with the TCU 200 temperature control unit. The TCU 200 monitors if sufficient cooling air is available and automatically turns on the cooling air if the heater temperature exceeds 200 °C.

Below 200 °C heater temperature, the instrument can be run without dome and without cooling air.

The main applications for the DHS 1100 are to investigate the temperature dependence of

- crystalline textures
- crystal stress and strain
- temperature-induced phase transitions
- structural properties of thin films

To ensure reliable operation of the DHS 1100, the instrument must be operated with:

- 25000 TCU 200 TEMPERATURE CONTROL UNIT FOR DHS 1100

In order to avoid contamination of the DHS 1100 by the cooling air Anton Paar GmbH provides an air service unit with pressure regulator and air filters as an accessory:

- 6931 AIR SERVICE UNIT

The TCU 200 and the air service unit are described in separate instruction manuals.

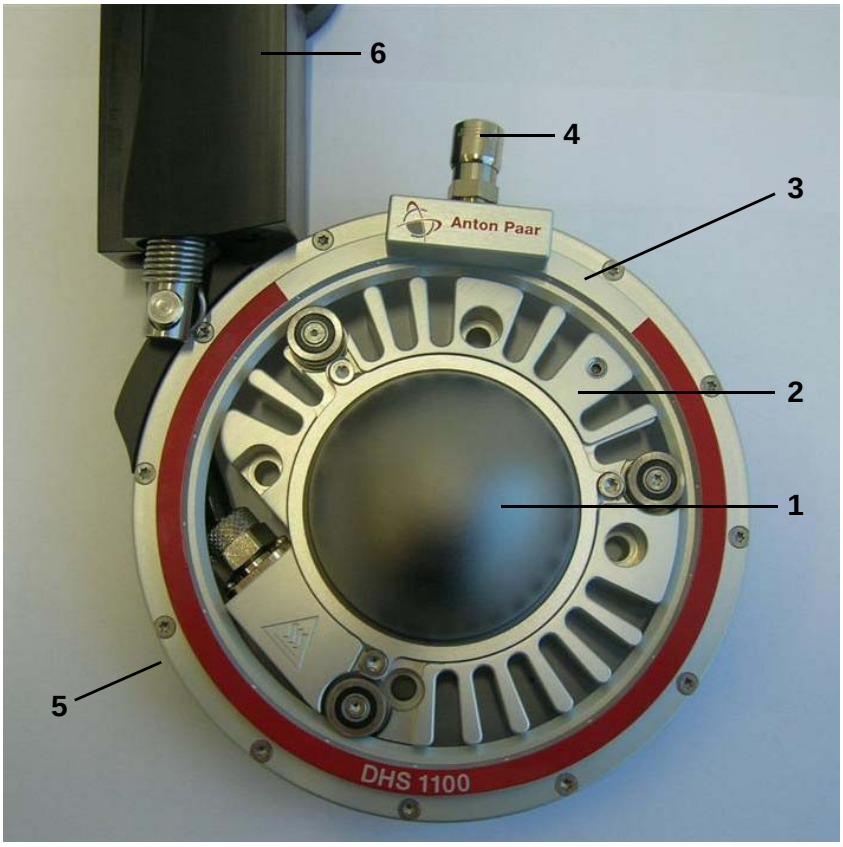
Information:
Refer to *Appendix E: Accessories* and the *TCU 200 Instruction Manual* for detailed information.

3.2 Design of the Instrument

Depending on the type of diffractometer (**PANalytical X'Pert Pro** or **PANalytical Empyrean**), there are two different models of DHS 1100 Domed Hot Stage. Functionality and operating conditions are the same for both DHS 1100 models.

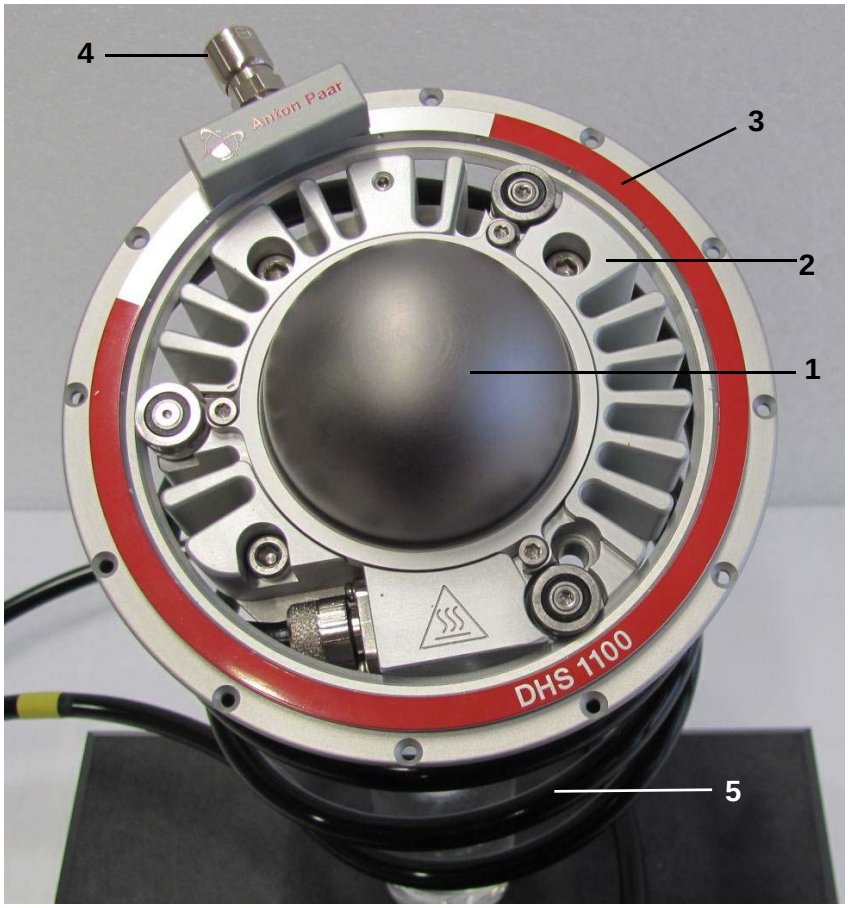
The DHS 1100 for **PANalytical X'Pert Pro** consists of:

1. Dome
2. Housing
3. Turnable cooling ring with outlets for cooling air
4. Quick coupling connection for cooling air supply
5. Hose guide ring beneath the instrument (see next page)
6. Supply hose guidance



The DHS 1100 for **PANalytical Empyrean** consists of:

1. Dome
2. Housing
3. Turnable cooling ring with outlets for cooling air
4. Quick coupling connection for cooling air supply
5. Spiral-shaped supply hose



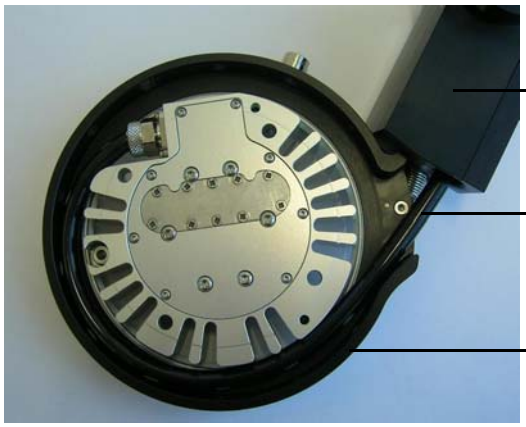
Caution:
Do not disconnect the supply hose from the DHS 1100 housing, because the connecting wires from the thermocouple and the heater can be damaged.

The DHS 1100 has an air cooling system, which has the advantages that only one hose is needed for the cooling agent and that no liquid can leak inside the cabinet in case of a defect.
The cooling gas, preferably clean compressed-air, is distributed around the chamber inside the turnable cooling ring (3). Small outlets in the ring direct the gas onto the housing and the dome. The cooling ring is turnable so that the connector for the cooling air hose (4) can be placed in a position where it does not interfere with the incident and diffracted X-ray beams.

The cooling air supply is controlled with the TCU 200 temperature control unit, which automatically turns on the air cooling when the heater temperature reaches 200 °C.

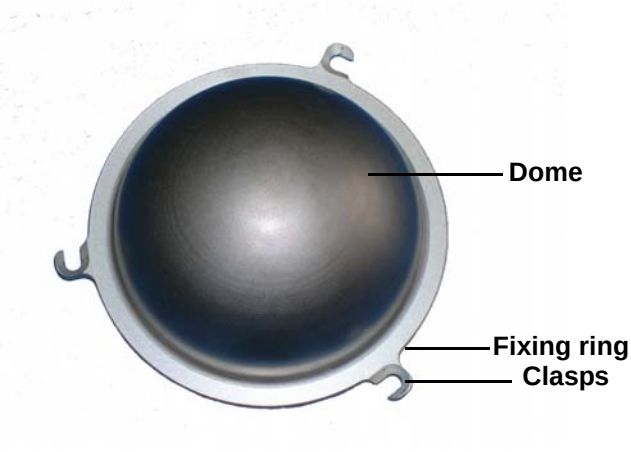
In order to achieve the maximum movability of the DHS 1100 on the goniometer the number of hoses to the instrument is kept to a minimum. Besides the hose for the cooling air there is only one additional hose which contains the electrical wires for thermocouple and heater and is used to evacuate the dome or to fill it with gas. The shape and length of the supply hose differs for the two types of DHS 1100. As a consequence, the minimum pressure (vacuum) underneath the dome is type dependent (refer to *Appendix A: Technical Specifications).*

1. The **supply hose** for DHS 1100 **PANalytical X'Pert Pro** is lead through a hose guide ring underneath the instrument to avoid damage of the supply hose.



2. The **supply hose** for DHS 1100 **PANalytical Empyrean** is spiral-shaped and does not have a hose guide ring.

3.2.1 Dome



The dome of the DHS 1100 is made of graphite, which exhibits an excellent combination of good mechanical properties, high temperature resistance and excellent X-ray transmission.

The dome is fixed to the housing of the DHS 1100 by a fixing ring made of aluminum with three clasps.

Vacuum tightness is guaranteed by an O-ring in the housing.

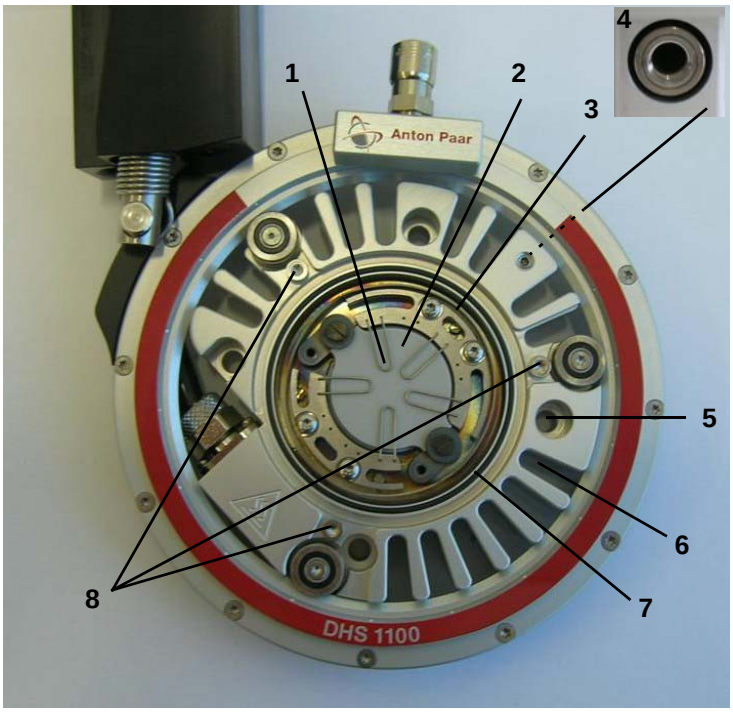
Caution:

- Always lift the dome by the fixing ring. Do not touch the graphite dome.
- Make sure that the cooling air is on to guarantee sufficient cooling of the DHS 1100 housing and the dome during operation at temperatures higher than 200 °C.
- Never touch the dome during operation, especially when it is evacuated or at elevated temperatures!
- If the inner surface of the dome has become coated with evaporated sample components, clean it with a dry cloth.

Information:

- For a diffractogram of graphite, please refer to *Appendix C: Diffraction Patterns*.
- Anton Paar GmbH gives no warranty for the dome if it is not handled according to our instructions.
- The dome is also available as spare part, refer to *chapter Appendix E: Accessories*.

3.2.2 Housing and Internal Parts



1. Springs for fixing the sample
2. Sample plate
3. Temperature shieldings
4. Pressure relief valve
5. Bore holes for mounting the DHS 1100
6. Cooling fins
7. O-ring for the dome
8. Fixing screws for the dome

The housing of the DHS 1100 is made of anodized aluminum and is therefore very light. It contains cooling fins which provide sufficient heat transport from the heated sample plate of the DHS 1100 to the surroundings.

The sample plate of the DHS 1100 is made of aluminum nitride (AlN), a ceramics with excellent temperature conductivity and good chemical resistance. In case of damage, the sample plate can be easily replaced by a new one.

The springs for fixing the sample are made of Inconel and can be loosened very easily by using tweezers.

Information:

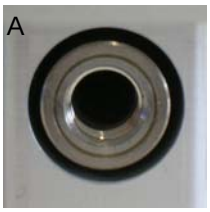
- For a diffractogram of aluminum nitride and Inconel, please refer to *Appendix C: Diffraction Patterns*.

The heater is located just underneath the AlN heating plate. The design and the excellent temperature conductivity of the AlN plate guarantee high temperature uniformity across the heating plate. The thermocouple is located just underneath the middle of the heating stage.

The connecting wires from the thermocouple and the heater run within the black hose (supply hose) to the connection device described in *chapter 3.2.3 Connection device*.

Caution:
Do not disconnect the supply hose from the DHS 1100 housing because the connecting wires from the thermocouple and the heater can be damaged.

The housing of the DHS 1100 contains a bore hole for various connectors (A). The following connectors are supplied with DHS 1100:



1. Blind plug
2. Feed-through adapter
3. Pressure relief valve
4. Hose connector

A blind plug (B.1) is provided for measurements under vacuum.

The feed-through adapter (B.2) can be used together with the hose connector (B.4) for additional gas discharge.

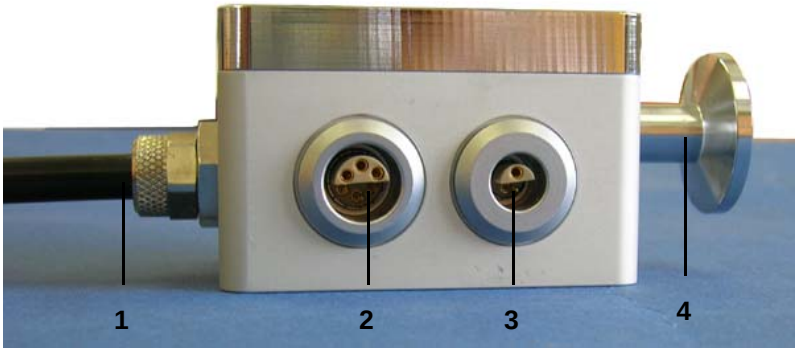
The pressure relief valve (B.3) is a safety device that opens if an overpressure of 0.35 ± 0.05 bar relative pressure is generated in the dome.

Information:
Additional information for replacing the connectors can be found in *chapter 7.4.2.3: Operation under Vacuum.*

Inside the DHS 1100 housing is a protective thermoswitch to avoid overheating of the instrument in case the air cooling system fails. The thermoswitch interrupts the power supply to the DHS 1100 if the temperature of the housing exceeds 115 °C. An error message is displayed on the TCU 200 temperature control unit (for details refer to the *TCU 200 Instruction Manual*).

3.2.3 Connection device

The connection device terminates the supply hose of the DHS 1100, which contains the electrical wires of the instrument and which is used to evacuate the instrument or to fill it with gas. It is a vacuum-tight interface box with connectors for the temperature sensor and the heater inside the DHS 1100 and a flange to connect a vacuum pump or the gas supply.



1. Supply hose with wires to thermocouple and heater
2. Connector for thermocouple (6 poles)
3. Connector for heating (2 poles)
4. Flange for gas supply/vacuum pump (DN16KF)

Caution:
Place the connection device for gas/vacuum and temperature control in a way that the hose is not caught by any parts of the goniometer.

4 Supplied Items

The DHS 1100 was tested and packed carefully before shipment. However, damage may occur during transport.

1. Keep the packaging material (box, foam piece, transport protection) for possible returns or questions from the transport and insurance company.
2. Check the delivery for completion by comparing the supplied parts to those listed in table 4.1.
3. If a part is missing, contact the local representative of your diffractometer manufacturer or Anton Paar GmbH in Graz, Austria.
4. If a part is damaged, contact the transport company and either the local representative of your diffractometer manufacturer or Anton Paar GmbH in Graz, Austria.

Table 4.1: Supplied Parts

Pcs.	Article Description	Cat. No.
1	DHS 1100 Domed Hot Stage - for **PANalytical X'Pert Pro** or - for **PANalytical Empyrean**	 25011 96450
1	Instruction Manual	---
1	Accessory box (content specified on label)	---

Information:

Always use the original packing material when shipping the instrument or parts of it. Store the packing material carefully for later use!

5 Installation

5.1 Installation requirements

Electrical power and computer interface

- Operation of TCU 200 requires 230 V AC / 50...60 Hz.
- For remote control of TCU 200, a PC with RS 232 C serial interface is required.

Compressed-air for sample cooling

For operation of DHS 1100 with compressed-air cooling, the main requirements for the compressed-air supply are:

- Cooling air pressure: min. 2 bar rel. / max. 4 bar rel. (rel. = relative to atmospheric pressure)
- Air flow rate at 2 bar: 0.12 m^3/min
- The air must be free of oil and dust.

Information:
We recommend to use the Air Service Unit by Anton Paar GmbH (refer to *Appendix E: Accessories*).

5.2 Unpacking the Instrument

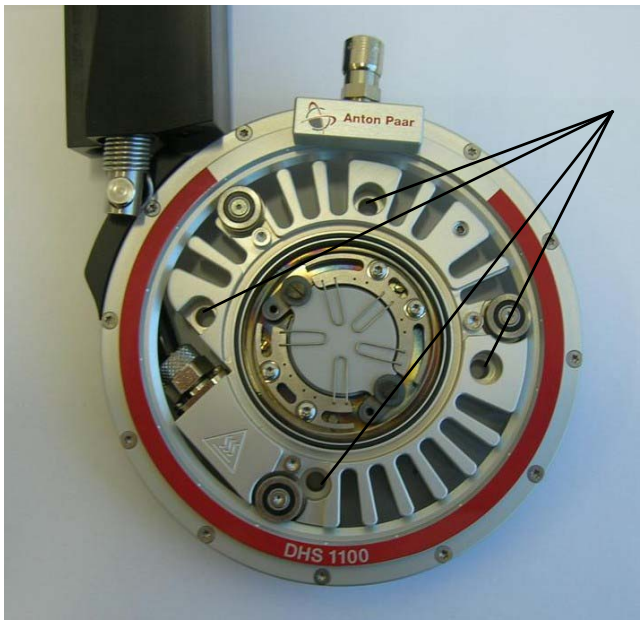
Caution:
- Unpack the system with care
- Do not drop the instrument.
- The graphite dome is a fragile item, handle it with particular care.
- Always lift the dome by the fixing ring, do not touch the graphite dome.

Radiation:
The DHS 1100 represents an open system and therefore is only allowed to be operated on goniometers equipped with a radiation enclosure.

Information:
- Anton Paar GmbH gives no warranty for the dome if it is not handled according to our instructions.
- If the dome is already broken when unpacking DHS 1100, please take pictures that show explicit the broken dome with the original packing material. Otherwise no warranty can be accepted by Anton Paar.

5.3 Mounting the DHS 1100 on the Goniometer

The DHS 1100 is mounted directly on the goniometer via the four bore holes, as shown below.



Boreholes for mounting DHS 1100

For detailed information on the installation refer to the *Instruction Manual* of your *Diffractometer*.

5.4 Connecting the Cooling Air Supply

Cooling of the dome and the housing of the DHS 1100 heating attachment is achieved by using clean compressed-air. If no compressed-air supply is available in the laboratory, a small compressor can be used to generate the cooling air.

Information:
A special Air Service Unit can be used to clean the cooling gas from dust and oil (see also *Appendix E: Accessories*).

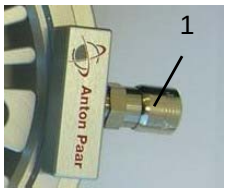
Caution:
Do not connect the DHS 1100 directly to the cooling air supply.
The cooling air must be supplied through the TCU 200 temperature control unit.

A black hose for the cooling air is supplied with the TCU 200. Install this supply hose as follows:

1. Feed the cooling air hose through the cabinet of the diffractometer.

2. Connect the end outside the cabinet to the cooling air outlet connector on the rear panel of the TCU 200 (refer to the *TCU 200 Instruction Manual*).

3. Connect the other end of the hose inside the cabinet to the appropriate quick coupling connector (1) on the turnable ring of the DHS 1100.



4. Connect the transparent hose, that is aslo supplied with the TCU 200 to the cooling air inlet connector on the rear panel of the TCU 200 and to the cooling air supply (refer to the *TCU 200 Instruction Manual* for more information).

5.5 Connecting the Gas Supply/Vacuum Equipment

The graphite dome allows the investigation not only in air, but also under vacuum or in inert gas to avoid oxidation or other chemical reactions of the sample at high temperatures.

Feed an appropriate gas/vacuum hose through the cabinet of the goniometer and connect it to the flange (DN16KF) on the connection device of the DHS 1100.

The connection device for the gas supply/vacuum equipment is described in *chapter 3.2.3 Connection device*.

Information:
For further information on operation of the DHS 1100 under vacuum or various gases, please refer to *chapter 7.4.2 Creating the Sample Environment*.

5.6 Connecting the Temperature Control Unit

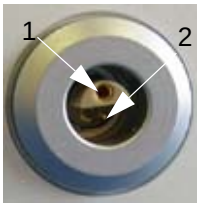
The connector for temperature measurement and the connector for the heater are located in the connection device for gas/vacuum and temperature control, described in *chapter 3.2.3 Connection device*.

Warning:
Switch off the TCU 200 and disconnect the cable for the mains supply before disconnecting or connecting the cables to the connection device.

5.6.1 Connecting the Heater Cable

The heater cable is fixed to the rear of the TCU 200. Feed the cable through the enclosure of your diffractometer and connect it to the connection device of the DHS 1100.

Connector for heating



- Heating voltage:
  max. 26 VAC
- Resistance between heater pins:
  approx. 2 Ω

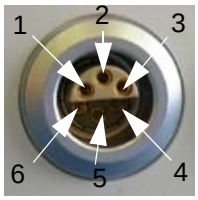
5.6.2 Connecting the Temperature Sensor Cable

The temperature sensor cable is fixed to the rear of the TCU 200. It contains the wires for the type S thermocouple measuring the sample temperature inside the DHS 1100 and the wires for the thermoswitch which protects the DHS 1100 housing from being overheated. Feed the cable through the enclosure of your diffractometer and connect it to the connection device of the DHS 1100.

Connector for thermocouple

PIN assignment:

PIN	
1	thermocouple (+) pole
2	shield
3	thermocouple (–) pole
4	DHS 1100 housing thermoswitch
5	NC
6	DHS 1100 housing thermoswitch



6 Alignment

The standard alignment of the DHS 1100 is carried out at room temperature.

Radiation:
The DHS 1100 represents an open system and therefore is only allowed to be operated on goniometers equipped with a radiation enclosure.

For very accurate measurements, e.g $\theta/2\theta$ scans at 2θ angles < 60°, it may be necessary to repeat the alignment at the temperature of the measurement to compensate the thermal height expansion of sample stage and sample. Data for the thermal expansion of the sample stage are shown in *Appendix A: Technical Specifications*.

To align the sample height, proceed as follows:

1. Mount the sample as described in *chapter 7.2 Mounting the Sample*.
2. Perform the standard alignment of the sample, refering to the *Diffractometer Instruction Manual*.

7 Operation

Measurements with the DHS 1100 are performed in the same way as when using the standard sample stage for the diffractometer.

7.1 Preparing the Instrument

Before starting to work with the DHS 1100 make sure that the following safety requirements are fulfilled:

Radiation:
The DHS 1100 represents an open system and must be operated on goniometers equipped with a radiation enclosure only.

Hot Surface:
During operation and even after turning off the heating, the sample stage and the dome can be hot. Make sure that all parts of the DHS 1100 are below 50 °C before touching the instrument.

Warning:
Make sure that no inflammable material is lying below or near the DHS 1100 during operation.

In the temperature range between room temperature and 200 °C it is possible to work without the cooling gas and the graphite dome. At temperatures above 200 °C the dome has to be mounted and the air cooling must be on to guarantee sufficient heat removal from the dome and the housing of the DHS 1100.

Caution:
Never touch the dome during operation, especially when it is evacuated or at elevated temperatures, because this may easily damage the dome.

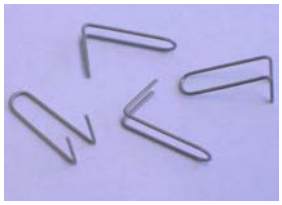
For information on the temperatures of the dome, the housing and the goniometer after operation at 1100 °C for 1 hour, refer to *Appendix A: Technical Specifications*.

7.2 Mounting the Sample

The DHS 1100 heating attachment is preferably used for flat and/or flake-shaped samples with a maximum diameter of 25 mm and a maximum thickness of 2 mm. The **recommended sample diameter** is 15–20 mm and the **recommended sample thickness** is ≤1 mm. This ensures good clamping of the sample to the heating plate and maximum temperature homogeneity across the sample.

Caution:
- Make sure that the sample components do not react with the sample plate at elevated temperatures.
- Check the melting point of the sample **before** the experiment and choose the temperature of the investigation correspondingly.

The sample is fixed on the sample holder with springs. The springs are supplied in the accessory box and can be easily exchanged by pulling them out of the small bore holes. Depending on the sample size, use either the large or the small springs for fixation.



Hot Surface:
Before mounting the sample make sure that all parts of the DHS 1100 are at room temperature.

Caution:
Use a pair of tweezers for fixing the sample on the sample stage.
- Carefully mount the sample with the fixing springs and do **not** bend or deform the springs.
- Used fixing springs can be bend and used for further measurements or replaced (refer to *Appendix E: Accessories*).

To **mount the sample**, proceed as follows:

1. Loosen the fixing springs by using tweezers.

2. Place the sample in the middle of the heating plate to make sure that the X-ray beam hits the sample. If the X-ray beam hits the heating plate or the springs, diffraction and fluorescence from the heating plate or the springs may be detected. In this case mention notice *Appendix C: Diffraction Patterns*.

3. Insert and push down the springs to fix the sample. Make sure that the sample has very tight contact to the heating plate to guarantee optimum heat transfer. Fix the sample tightly so that it does not move during the measurement (upon movement of the goniometer).

7.2.1 Temperature Homogeneity of the Sample

Be aware that, depending on the sample thickness and the thermal conductivity of the sample, the temperature of the heating plate will be different from the temperature on top of the sample.

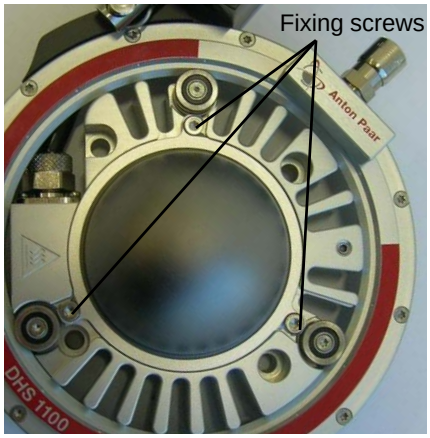
Heat transfer to the sample and temperature homogeneity of the sample surface is much better in inert gas atmosphere than in vacuum.

7.3 Mounting and Removing the Dome

Caution:
- Always check the dome before you mount it. In case of a defect do not mount it.
- The graphite dome is a fragile item, handle it with particular care.
- Always lift the dome by the fixing ring, do not touch the graphite dome.
- Always make sure that all parts of the instrument are cooler than 50 °C before you start to remove the dome.

To **mount the dome**, carry out the following steps:

1. Make sure the O-ring and the contact surface of the dome are free from dust.
2. Place the dome on the O-ring.
3. Turn the dome until the three clasps rest underneath the fixing screws.
4. Fix the 3 screws using the screw driver from the accessory box.
5. Check whether the dome is mounted firmly and cannot fall off the instrument due to the movement on the goniometer.



To **remove the dome**, carry out the following steps:

1. Turn off the heater on the TCU 200 temperature control unit (refer to the *TCU 200 Instruction Manual*).
2. Make sure all parts of the DHS 1100 are cooler than 50 °C.
3. Loosen the 3 screws using the screw driver from the accessory box.
4. Take the dome by the fixing ring and turn it until the three clasps are free.
5. Lift the dome off the housing.

7.4 Performing a Measurement

After having mounted and aligned the sample you are now ready to start with your measurements (for sample mounting and alignment refer to *chapter 7.2 Mounting the Sample* and *chapter 6 Alignment*).

If the sample stage temperature does not exceed 200 °C during the entire measurement, the measurement can be carried out without dome and cooling air. Above 200 °C, dome and cooling air are necessary.

7.4.1 Before Starting

Before you start the measurement:

- Check the dome. In case of any defect do **not** use it.
- Make sure that the dome is mounted correctly and the three fixing screws provide tight connection between dome and the DHS 1100 housing.
- Make sure that the pressure relief valve is not blocked or closed with a blind plug.
- Make sure that the temperature of your experiment is below the melting point of the sample and that no sample components react with the sample stage.
- Make sure that the goniometer is operated in a way so that it **always** moves back to its starting position. This is important to avoid that the hoses wind up. If changes in the diffractometer software are necessary, contact the diffractometer manufacturer.

Information:
We recommend to move the goniometer with the DHS 1100 on it over the complete range of angles intended for the measurement to ensure that the hoses are not caught by any parts of the diffractometer.

- If the DHS 1100 is heated to more than 200 °C, make sure that cooling air is supplied to the TCU 200 and that the cooling air hose is connected to the DHS 1100 (refer to *chapter 5.4 Connecting the Cooling Air Supply*).

7.4.2 Creating the Sample Environment

With DHS 1100 sample can be investigated in vacuum or different gas atmospheres.

Caution:
- The temperature control of the DHS 1100 is optimized for **stationary atmospheres** around the sample. Do not measure with a gas flow onto the sample or through the dome.
- The atmosphere inside the dome has an influence on the maximum achievable sample heating/cooling rates and on the temperature offset between sample plate and probed sample surface.
- When the DHS 1100 is filled with **helium**, the **maximum operating temperature** is **1000 °C** due to the large heat transfer from the heating plate to the dome.

7.4.2.1 Operation with Air

No special measures must be taken if the sample is measured in air. The sample stage can be heated up to the maximum temperature of 1100 °C.

7.4.2.2 Operation with Non-Reactive Gases

In order to prevent oxidation of samples, the DHS 1100 can be filled with non-reactive gases like helium or nitrogen. We recommend not to use argon because of its high X-ray absorption.

Caution:
All pressure values in this section are relative to atmospheric pressure.

The **pressure relief valve** in the DHS 1100 housing is set to 0.35 +/- 0.05 bar. This is the maximum pressure which can be achieved inside the dome.

Caution:
- Use a suitable pressure reducer/regulator with a range from 0 to ≤ 5 bar and an accuracy of +/- 0.1 bar to control the gas pressure supplied to the DHS 1100.
- Make sure the pressure relief valve is not blocked or closed with a blind plug.
- Avoid pressure bursts, because they can destroy the dome.
- For flushing the instrument with gas, never apply more than 1 bar pressure. This can destroy the dome.

To **fill the sample stage with gas**, proceed as follows:

1. Feed a suitable gas hose through the cabinet of the diffractometer and connect it to the flange (DN16KF) on the connection device of the DHS 1100 (refer to *chapter 5.5 Connecting the Gas Supply/Vacuum Equipment*).

2. Connect the other end of the gas hose to the pressure reducer/regulator of your gas supply.

3. In order to remove the air from the sample stage, apply a pressure of approx. 0.5 bar. This opens the pressure relief valve and the instrument is flushed with the non-reactive gas.

4. After a few minutes, depending on the permitted amount of residual air, reduce the pressure to 0.2 - 0.3 bar to close the pressure relief valve and create a stationary atmosphere.

5. To remove the gas from the DHS 1100, close the gas supply, slowly loosen the fixing screws of the dome to allow the gas to escape and take off the dome.

The feed-through adapter can be used together with the hose connector (for hoses with an inner diameter of 4 mm) for additional gas discharge after removing the pressure relief valve or the blind plug.

7.4.2.3 Operation under Vacuum

With a standard vacuum pump with 4.8 m^3/h capacity, the following vacuum levels can be achieved inside the dome of the DHS 1100:

- ≤ 1 mbar with DHS 1100 for PANalytical X'Pert Pro
- ≤ 3 mbar with DHS 1100 for PANalytical Empyrean

When applying vacuum to the instrument, the pressure relief valve is automatically closed. However, in order to prevent possible gas leackage through the pressure relief valve, we recommend to mount the blind plug, which is supplied in the DHS 1100 accessory box (see also *chapter 3.2.2 Housing and Internal Parts*).

To **replace the pressure relief valve with the blind plug**, proceed as follows:

1. Loosen the Allen screw on top of DHS 1100 with a 2 mm Allen key (1).
2. Then insert a screw (M4, not supplied by Anton Paar) into the embedded valve to pull it out of the DHS 1100 (2).

1

2

3. Insert the blind plug and tighten the Allen screw.

To **evacuate the sample stage**, proceed as follows:

1. Feed an appropriate vacuum hose through the cabinet of the diffractometer and connect it to the flange (DN16KF) on the connection device of the DHS 1100 (refer to *chapter 5.5 Connecting the Gas Supply/Vacuum Equipment*).
2. Connect the vacuum hose to a suitable vacuum pump, e.g. a rotary vane pump.

   Between vacuum pump and DHS 1100 connection device you should have
   - a valve to close the vacuum pump
   - a valve to vent the DHS 1100 sample stage
   - a pressure gauge
3. To apply vacuum, close the venting valve and open the valve to the vacuum pump.

Information:

With a vacuum pump with capacity 4.8 m^3/h it takes approx. 30 min to reach:

- ≤ 1 mbar with DHS 1100 for **PANalytical X'Pert Pro**
- ≤ 3 mbar with DHS 1100 for **PANalytical Empyrean**

Information:
Anton Paar GmbH offers a suitable vacuum equipment for the DHS 1100. All required components for the installation are included in the delivery. Please refer to *Appendix E: Accessories*.

7.4.2.4 Reactive, Explosive, and Poisonous Gases

Warning:
Do **not** operate the DHS 1100 with reactive, explosive or poisonous gases or with gases which form explosive mixtures with air.
In particular, do **not** operate DHS 1100 with hydrogen.

7.4.3 Setting the Sample Temperature and Heating Rate

Sample temperature and heating/cooling rate are controlled by the TCU 200 temperature control unit (TCU).

There are two ways of defining the desired sample temperature and heating/cooling rate:

I. Sample temperature and heating/cooling rate can be set manually by using the keypad on the front panel of the TCU. Refer to the *TCU 200 Instruction Manual* for detailed information.

II. Sample temperature and heating/cooling rate can be defined in the diffractometer control software. Refer to the *Diffractometer Instruction Manual* for detailed information.

The **maximum heating and cooling rate** which can be set in the TCU (parameter SPR) is 500 °C/min. Be aware that this is not the maximum heating or cooling rate for the sample plate which you can actually achieve with the instrument. The achievable heating and cooling rates for the sample plate depend on the operating conditions (gas type, temperature range). Information about the physical range of heating and cooling rates is given in *Appendix B: Temperature Control Data*. In order to avoid excessive load on the heater, do not use heating rates > 300 °C/min.

Use the following **setpoint limits for heating rate and cooling rate**:
maximum heating rate: 300 °C/min
maximum cooling rate: 500 °C/min

Temperature control of TCU 200 has been optimized in such a way that it can be used for all specified sample environments without any adjustments to the internal control parameters. Due to the different thermal properties of the specified gases, a temperature overshoot of a few °C may occur under certain operating conditions. The temperature overshoot can be reduced by reducing the heating rate.

7.4.4 Recording the X-ray Scan

Before you start the scan make sure that the connector on the turnable cooling ring does not block the incident or diffracted beam.

Refer to the *Diffractometer Instruction Manual* for information about recording an X-ray scan.

8 Maintenance

8.1 Checking the Dome

The dome should be regularly checked, depending on the operating hours, the usual operating temperatures and the applied atmospheres.

Caution:

- Always lift the dome by the fixing ring.
- Never touch the dome during operation especially when it is evacuated or at elevated temperatures!
- Check the dome for cracks and coatings on the inner surface.
- If the inner surface of the dome has become coated with evaporated sample components, clean it with a dry cloth.
- If cracks are observed, replace the dome (refer to *Appendix E: Accessories*).

8.2 Replacing the O-rings

In general, commercially available O-rings made of Viton are used.

While detaching an O-ring, make sure that the sealing surfaces remain undamaged.

Before fitting a new O-ring, clean the sealing surfaces with a suitable solvent.

8.3 Technical Support

If you need technical support, please contact the local service organization of your diffractometer manufacturer or Anton Paar GmbH in Graz, Austria.

Contact details of Anton Paar GmbH:

Anton Paar GmbH	Tel:	+43 316 257-0
Anton-Paar-Strasse 20	Fax:	+43 316 257-257
A-8054 Graz	E-mail:	info@anton-paar.com
AUSTRIA / Europe	Web:	www.anton-paar.com

Appendix A: Technical Specifications

Temperature range:	
- Air, N_2, vacuum	25 to 1100 °C
- Helium	25 to 1000 °C
Heating rate:	max. 300 °C/min
Atmospheres:	
- Gases	Air, N_2, inert gas
- Overpressure inside dome	max. 0.35 ± 0.05 bar rel.
Vacuum:	
- for DHS 1100 PANalytical X'Pert Pro	≤ 1 mbar
- for DHS 1100 PANalytical Empyrean	≤ 3 mbar
Angular range:	
2 Theta (2Θ)	0 to 166°
Omega (ω)	0 to 83°
Psi (ψ)	0 to 85°
Phi (φ)	0 to 360°
Dimensions/Weight:	
Diameter	135 mm
Weight (without adapter)	460 g
Sample holder:	
Sample holder diameter	29 mm
Sample diameter	max. 25 mm
Sample thickness	max. 2 mm
Temperature sensor	thermocouple Pt–Pt10Rh (type S)
Height above housing base plate	22 mm
Parallelism to housing base plate	≤ 0.05 mm
Thermal height expansion	see graph below
Sample fixing	Inconel springs
Dome:	
Diameter	63 mm
Wall thickness	0.25 mm
X-ray transmission	CrKα 40%
(primary + diffracted beam)	CuKα 65%
	MoKα 95%
Cooling requirements:	
Cooling air pressure	min. 2 bar rel. / max. 4 bar rel.

Air flow rate at 2 bar	0.12 m³/min
Gas hose:	
material	polyamide
inner x outer diameter	4 x 6 mm

List of materials:

Sample plate	aluminum nitride
Sample fixing springs	Inconel
Thermocouple	Pt–Pt10Rh
Heater	Kanthal
Dome	graphite
Housing	aluminum, Inconel
Supply hose	polyamide, PTFE
Connection device box	aluminum, Cr-plated brass
Control unit:	
Type	TCU 200
Electrical requirements	230 V AC ± 10% / 50...60 Hz
Overvoltage category	II according to EN 61010
Ambient conditions:	
Ambient temperature	5 °C to 35 °C
Atmospheric humidity	max. 80 % relative, not condensing
Contamination class	2 according to EN 61010

The DHS 1100 and the TCU 200 have been designed for indoor use at an altitude up to 2000 m only. Protect the instruments from moisture!

Disposal

The instrument should be disposed of in accordance with the local regulations.

The DHS 1100 in combination with TCU 200 temperature control unit complies with the following EU guidelines:

Electromagnetic compatibility (89/336/EEC + amendments 92/31/EEC, 93/68/EEC)

Electrical equipment for measurement, control and laboratory use - EMC requirements:	EN 61326:1997+ A1+A2+A3:2003

Low Voltage Directive (73/23/EEC + amendment 93/68/EEC)

Safety requirements for electrical equipment for measurement, control and laboratory use	
Part 1: General requirements	EN 61010-1:2001
Part 2-010: Particular requirements for laboratory equipment for the heating of materials	IEC 61010-2-10:2003

Appendix B: Temperature Control Data

Fig. B - 1 Maximum heating rate for different atmospheres

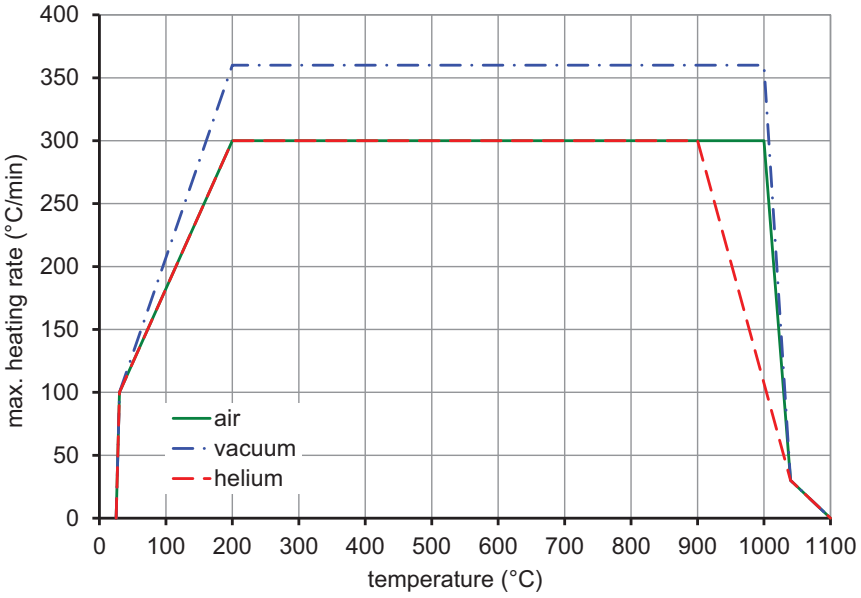


Fig. B - 2 Maximum cooling rate for different atmospheres

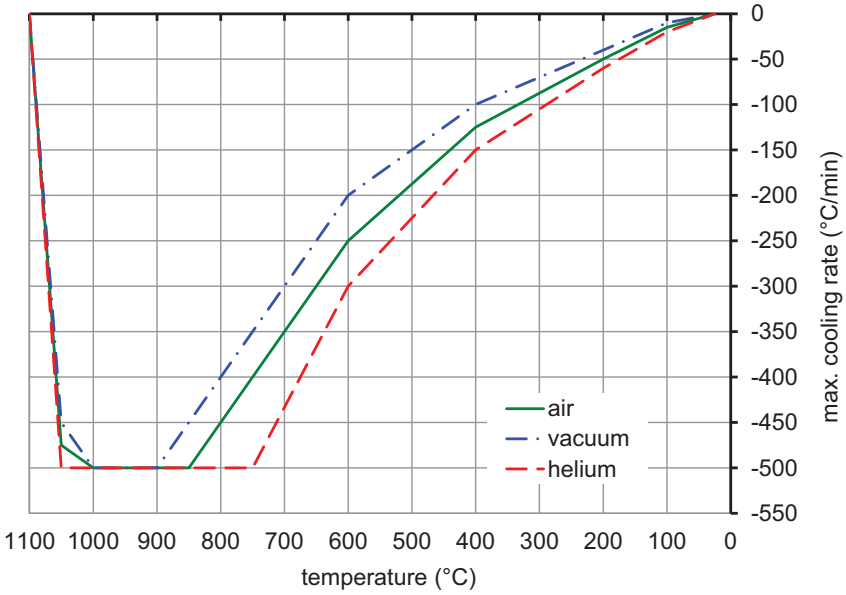


Fig. B - 3 Thermal height expansion at five different sites on the heating plate

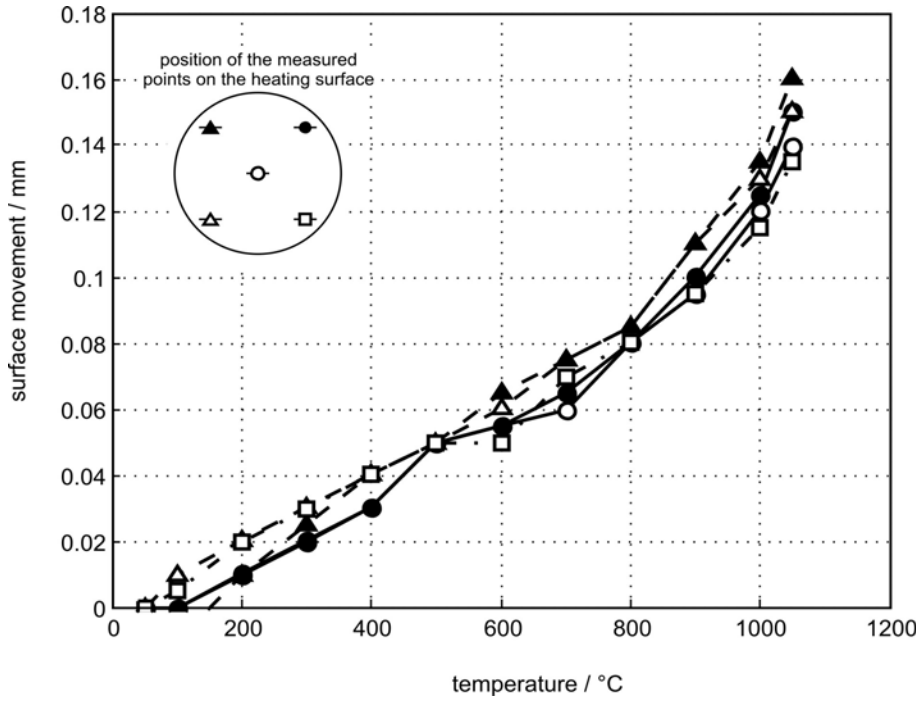


Fig. B - 4 Temperature distribution across the heating plate

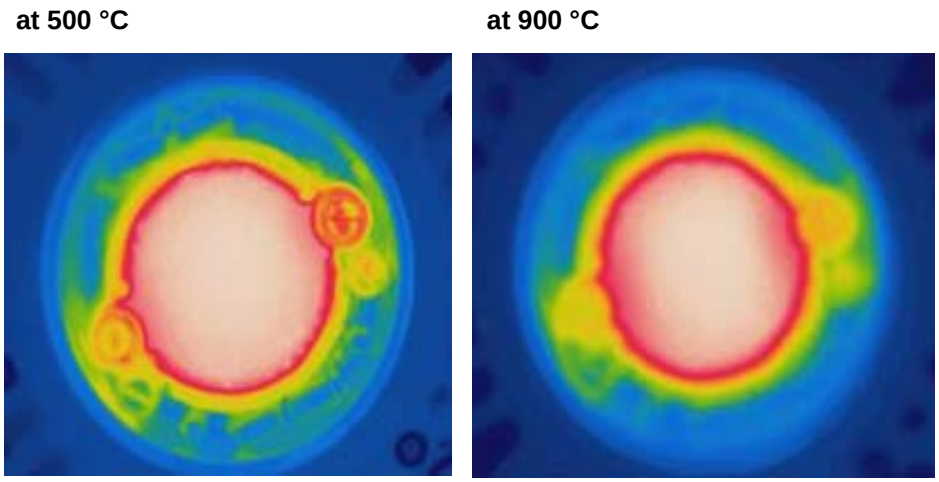


Fig. B - 5 Temperature at dome, housing and goniometer after operation for 1 hour depending on the set temperature

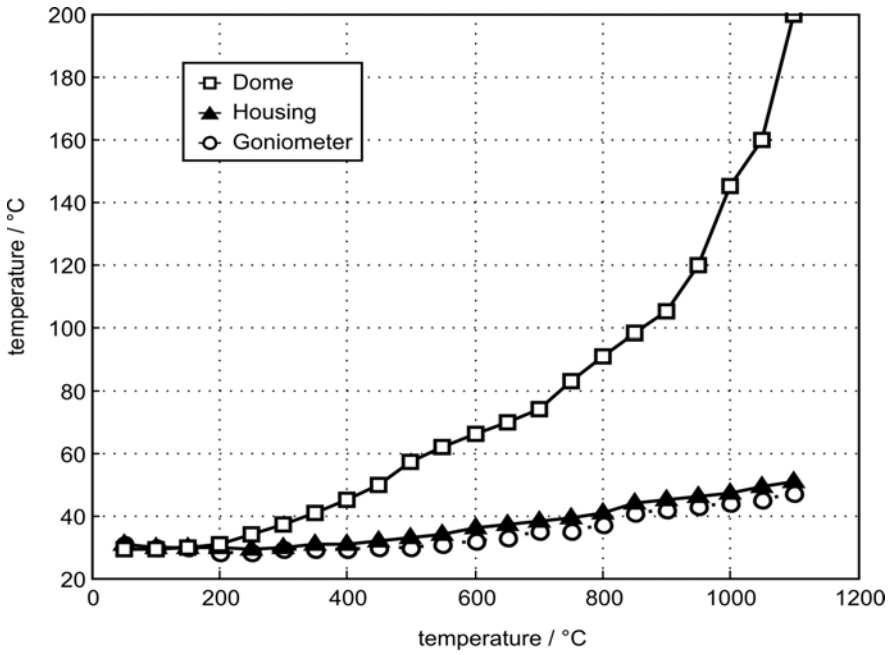
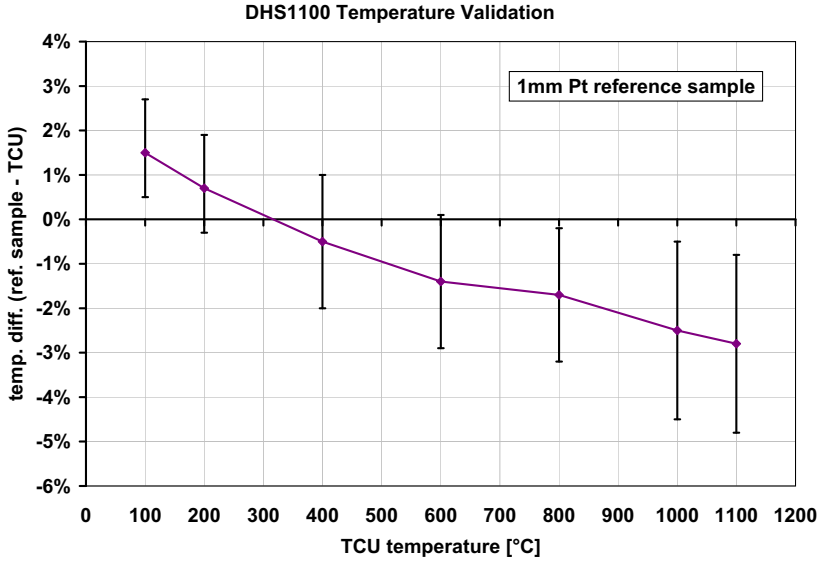


Fig. B - 6 Sample surface temperature



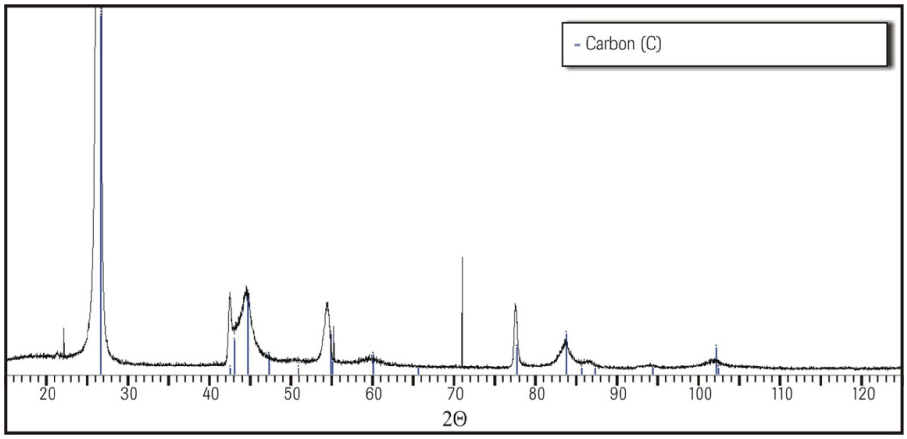
A 1 mm thick 10 x 10 mm platinum plate with a type S thermocouple spot- welded to the surface was used as a reference sample to give an indication of the temperature offset between the stage temperature displayed on the TCU 200 Temperature Control Unit and the surface of a sample scanned with the X-ray beam. The measurements were done in air.

The temperature deviation for your actual sample will depend on the sample thickness, the thermal contact between sample stage and sample, the thermal properties of the sample and the sample environment (gas/vacuum).

Appendix C: Diffraction Patterns

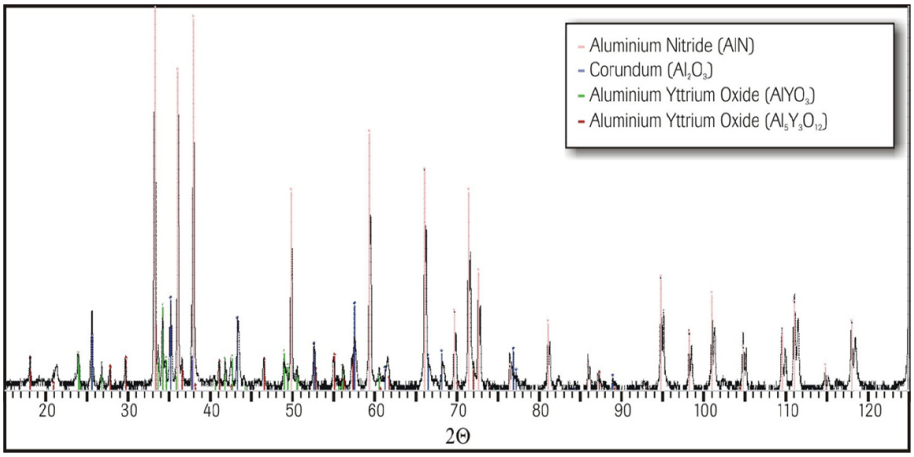
All diffraction patterns were measured with CuKα radiation in Bragg-Brentano geometry.

- **Graphite**



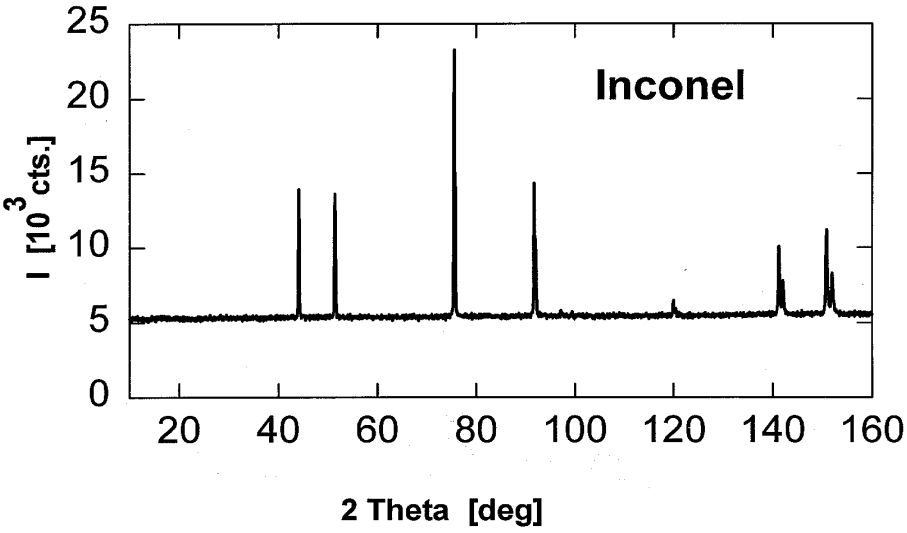
Set temperature 900 °C

- **Aluminum nitride**



Set temperature 500 °C

- **Inconel**



Set temperature 25 °C

Appendix D: Warranty

The warranty regulations for the DHS 1100 are in accordance with the "General Terms of Delivery" of the Austrian Electrical and Electronic Industry.

Anton Paar GmbH gives no warranty for the dome if it is not handled according to our instructions (see also *chapter 3.2.1 Dome* and *chapter 5.1 Installation requirements*).

Appendix E: Accessories

Accessories and spare parts for the DHS 1100

24998	GRAPHITE DOME
22010	DHS 1100 HOT PLATE
6931	AIR SERVICE UNIT
8415	FILTER CARTRIDGE (0.01µm PORE SIZE)
7626	O-RING SET
7624	SET OF SPRINGS FOR LARGE SAMPLES
7625	SET OF SPRINGS FOR SMALL SAMPLES
92920	SPARE PARTS PACKAGE DHS 1100 (2)

Option

58974	VACUUM EQUIPMENT

Index

A

Accessories 45
Air cooling 16, 22, 26
Air service unit 12, 21, 22
Alignment 25
Ambient conditions 37
Angular range 36
Anton Paar contact details 35
Applications 11
Atmospheres 30, 36

B

Blind plug 18, 32

C

Connection device 19, 23
Connectors
  cooling air 13, 14, 23
  gas supply / vacuum 19
  heater 19, 24
  thermocouple 19, 24
Control unit 23, 37
Cooling
  rate 33
  ring 13, 14
  system 14
Cooling air 22, 29, 30
  flow rate 37
  requirements 36
Cooling air supply 15, 22
Cooling rate
  limit 33, 39

D

Damage in transit 21
Diffractograms 42
  aluminium nitride 17
  aluminum nitride 42
  graphite 16, 42
  Inconel 17, 43
Dimensions 36
Disposal 37
Dome 13, 14, 16, 21, 26, 28, 29, 36
  mounting 28
  removal 29
  replacement 35

E

Electromagnetic compatibility 37
Evacuation 32

F

Feed-through 18, 31
Feed-through adapter 18
Fixing ring, dome 16
Fixing screws, dome 17
Fixing springs 17, 27

G

Gas/vacuum supply 23
Gases 30
  air 30
  explosive 33
  non-reactive 30
  poisonous 33
  reactive 33
Goniometer 26, 29

H

Heater 17, 24
Heating plate 40
Heating rate 33, 36
  limit 33, 39
Hose connector 18
Hose guide ring 15
Hot surface 7, 26, 27
Housing 13, 17, 26

I

Inflammable material 26
Installation 22

M

Materials, list of 37
Measurement 29
Missing part 20
Mounting holes 17, 22

O

Operating temperature 30, 36
Operation 12, 25
Original packing 20

O-ring, dome 17, 28
O-ring, replacement 35
Overheating protection 18
Overpressure 18, 36

P

PIN assignment 24
Pressure regulator 12
Pressure relief valve 17, 18, 29, 31

R

Radiation 21, 25, 26
Radiation hazard 7

S

Sample
- environment 30
- holder 29, 36
- mounting 27
- plate 17
- size 27
- stage 17
- temperature 33, 41
- thickness 27, 28
- types 27

Sample size
- maximum 27, 36
- recommended 27

Spare parts 45
Spiral-shaped supply hose 14
Supplied items 20
Supply hose 13, 14, 15, 19

T

TCU 200 22, 23, 33
Technical support 35
Temperature
- deviation 41
- distribution 40
- homogeneity 28
- offset 28
- overshoot 33
- sensor 36

Temperature control unit 23, 33
Thermal height expansion 40
Thermocouple 36
Thermoswitch 18

U

Unpacking 21

V

Vacuum 31

W

Warning 23, 26, 33
Warranty 44
Weight 36

X

X-ray scan 34
X-ray transmission 36