8.2 Protecting the heating strip

Anton Paar GmbH offers different heating strips for HTK 2000 (see *section 6.1 General information about heating strips*).

When performing high-temperature diffraction studies, an important issue is the reactivity of the heating strip. High-temperature reactions and sample melting can reduce the lifetime of the heating strip or even destroy it.

Caution:

- Sample components might react with the heating strip and destroy it.
- **Before** starting an experiment, **check the chemical resistance of the heating strip** for the respective sample in the applied atmosphere up to the desired maximum temperature.
- Refer to *Appendix F: Platinum Heating Strip* for more information on chemical properties and handling of platinum heating strips.
- The **tungsten heating** strip can only be **used in a vacuum** of approx. 10^{-4} mbar.

If no information about possible reactions between sample and strip are available, we recommend to perform an initial test with the sample on a piece of foil made of the strip material (e.g. platinum foil). The sample should be heated in a conventional furnace under the same conditions which will be used for the diffraction experiment.

Barrier substrates

If there is a reaction between the sample and the heating strip or if sample components may melt during the experiment, we recommend to use a barrier substrate between heating strip and sample.

If the expected reaction between sample and heating strip is weak, a piece of thin foil (e.g. 0.05 mm Pt-foil wrapped around the Pt heating strip) is often sufficient to protect the heating strip.

Caution:

- Make sure that the foil does not short circuit the wires of the thermocouple.
- Above 1450 °C the Pt foil will melt onto the Pt heating strip and cannot be removed easily.

If the expected reaction between sample and heating strip is strong, an inert barrier substrate such as alumina is required.

Caution:
Such barrier substrates are rather thick (usually 0.3 – 0.5 mm) and have limited thermal contact with the heating strip. **Expect large temperature deviations between the displayed temperature (TCU) and the real temperature of the sample surface.** We strongly recommend to do temperature validation of heating strip plus barrier substrate before measuring the first sample (see *chapter 8.8 Sample temperature*).

8.3 Applying a sample

Warning: DANGER OF BURNING!
Make sure that the heater of TCU 2000/20 is turned off and that the heating strip is at room temperature when applying the sample. In case of indications that TCU 2000/20 does not display the correct temperature, check the temperature with a suitable external temperature probe.

In general, several tests are necessary to determine the most suitable way of sample preparation. It is strongly dependent on the sample, temperature range and goal of the experiment. Therefore, the following instructions should be taken as suggestions only.

Caution:

- Sample components might react with the heating strip and destroy it.
- **Before** starting an experiment, **check the chemical resistance of the heating strip** for the respective sample in the applied atmosphere up to the desired maximum temperature.
- Refer to *Appendix F: Platinum Heating Strip* for more information on chemical properties and handling of platinum heating strips.

8.3.1 Powder samples

The powder to be investigated should be as fine as possible (grain size approx. 10 µm).

Prepare and apply the sample as follows:

1. Mix the powder with a suitable liquid in a small glass vessel or mortar to create a slurry.
2. Suck up the slurry with a pipette.

3. Apply a thin film of slurry to the heating strip.
   The sample should cover the complete width of the strip and should be approx. 10 mm long. The thickness of the sample should not exceed 0.1 mm.

4. Wait until the sample is dry or slowly heat up the sample to evaporate the solvent.

Information:
- Do not heat fast, because rapidly evaporating liquid creates holes in the sample.
- Make sure that the sample completely covers the heating strip. Holes in the sample can cause large background signal from the heating strip.

8.3.2 Bulk samples

Bulk sample are usually difficult to measure because of large and irreproducible deviations between the temperature displayed on the TCU and the temperature of the sample surface scanned with the X-ray beam (see *section 8.8 Sample temperature*).

Main properties which determine the temperature gradient are:

- thermal conductivity and thickness of the sample
- flatness and smoothness of the sample surface
- flatness and smoothness of the heating strip surface

Information:
- If possible, do not measure bulk samples in vacuum. Heat transfer in vacuum is usually very bad. Use inert gas (He) instead of vacuum.
- Bulk samples ofter bend upon heating, which changes the thermal contact and the temperature gradients in an undefined way.

8.4 Operating HTK 2000 with gas

HTK 2000 allows to heat the sample in air and various other gases.

Caution:
- Bear in mind that the gas inside the chamber expands when heated and that the maximum permissible pressure inside HTK 2000 is 1 bar rel..Take appropriate measures (e.g. relief valve) to release overpressure during your experiment.
- The tungsten heating filament can only be used in a vacuum of approx. 10^{-4} mbar.
- Make sure that the heating strip which is mounted can be heated in inert gas (see *Appendix A: Technical Specifications*) up to the desired maximum temperature for your experiment (e.g. Tantalum only up to 400°C).

8.4.1 Operation with air

Caution:
- Make sure that the heating strip which is mounted can be heated in air (see *Appendix A: Technical Specifications*) up to the desired maximum temperature for your experiment (e.g. Tantalum only up to 400°C).
- The tungsten heating filament can only be used in a vacuum of approx. 10^{-4} mbar.
- If the heating strip has a temperature limit < 1600 °C for operation in air, set the appropriate temperature limit for TCU 2000/20 in the control software of your diffractometer (see the instruction manual for your diffractometer for delailed information).

There are no special requirements for the operation with air inside the chamber.

Leave the vacuum flange in the lid or the gas supply hose open to allow release of gas during heating in order to avoid overpressure inside the chamber.

8.4.2 Operation with gases other than air

Warning:
HTK 2000 **must not** be used with hazardous (e.g. explosive or poisonous) gases.

Standard gases (apart from air) for measurements with HTK 2000 are inert gases and nitrogen. Other non-hazardous gases can be used, provided the gas itself and possible reaction products of the gas with the sample material do not damage the heating strip and the chamber **in the complete temperature range of interest**.

Caution:
- Check the chemical resistance of the heating strip and the materials inside the chamber against your sample and the applied atmosphere up to the required maximum temperature BEFORE the experiment.
- The tungsten heating filament can only be used in a vacuum of approx. 10^{-4} mbar.
- Bear in mind that the gas inside the chamber expands when heated and that the maximum permissible pressure inside HTK 2000 is 1 bar rel. Use a pressure relief valve to release overpressure during your experiment.
- Use a suitable pressure regulator for the pressure range 0–1 bar rel. to supply the gas.

Gas can be supplied to and extracted from the HTK 2000 through the vacuum flange on the lid and through the adapter. If you need oxygen-free sample environment, combine the gas supply system with a vacuum equipment.

A typical gas supply system consists of:

- gas supply with pressure reducer and pressure regulator