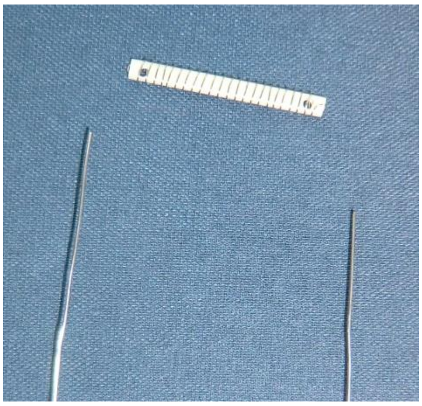
INTERNAL HEATER

Short description of use of internal heater

ProboStat™ internal heater considerations

Parts description



The internal heater consists of Ø0.25mm^2 Pt10Rh wire wrapped around a flat alumina capillary tube. The piece has two small holes lengthwise, as well as two bigger holes through. Two wires (Ø 0.5mm^2 Pt wire) are each threaded through the hole on the ends and wrapped tightly. The thinner wire has higher resistance and can be heated by sending current through it.

Setup assembly

Once the desired assembly with the sample is ready, the internal heater is placed on top of the spring load assembly, and the two minicontacts are connected to any available pins on the bottom. It is very important to take note to which pins the heater is connected to and to connect the power source to corresponding contacts on the ProboStat. Do not use contacts for shields or for K type thermocouples, as these may cause damage to the sample, to the cell or to the measurement instrumentation. Refer to ProboStat wiring schematics to identify pins and BNC sockets.

Depending on the sample size and correspondingly the length of the roof bar support the distance between the heater and the sample is normally between 5 and 10 cm.

In addition to ProboStat, internal heater, furnace and measurement instrumentation an adjustable power supply is required. The power source should have range of 0 to at least 20W and it may be manual or programmable.

Foreknowledge

Heat dynamics for each setup, temperature region, furnace type and currently used atmosphere are always different. Heat gets conducted, convected and radiated differently for each part and area in the setup. It is necessary to study the temperature behavior of the setup at the target temperatures before turning on the internal heater.

The internal heater location tries to eliminate heat radiation to the sample and thermocouples. Two ways of heat transfer (from the internal heater to the sample and thermocouples) must be considered: conduction through the roof bar support & other solids, and convection through the atmosphere. Conduction through the solids dominates at low temperatures (and vacuum) and atmospheric transfer at higher temperatures; heat conductivity of alumina is inversely proportional to the temperature.

The standard method of placing thermocouple tips between the sample and the roof- and floor supports is vulnerable to the solids conduction of heat (roof hotter, floor colder than sample), so it is recommended to use this only above 300 °C, where the solid part temperature equilibrium is result of atmosphere temperature around the solid, and not result from heat conduction in the solids.

Having thermocouple tips very close but not touching the sample is a setup vulnerable to the heat conduction of solids, and in addition to heat radiation from the enclosing tube heated by the furnace. This can be clearly seen as temperature noise during furnace heating, especially with quartz outer tube. Turning off (once at desired temperature) the furnace heating eliminates the radiation problem.

Sintering the thermocouple tips directly to the side of the sample with help of Pt ink is less vulnerable but more labor-intensive method. Combined with vacuum and turning off the furnace eliminates radiation and atmosphere effects.

The power for the heater is highly dependent of conditions and desired effect. The delay between internal heater action and thermocouple response also varies greatly depending on the conditions. Reaching a new thermodynamic equilibrium takes minutes in the best case, and much longer in the worst case. Using software that not only logs the temperatures but also indicates the rate of change can be very useful when estimating heating power effects and the timing of measurements (such as Omega software from NorECs).

The temperature of the wire is not the temperature in the cell, but much higher. Firstly, this is to be taken into consideration at higher furnace temperatures and/or vacuum, as not to melt the heating wire and secondly, the resistance of the wire is a function of the wire temperature. If power source is controlled in constant voltage mode, changing resistances may cause the total heating power to drift. If the power supply is used in constant current mode, the heating power remains stable, but the maximum voltage should be limited in a way that in case of open circuit conditions the voltage may not rise higher than 40 V; the threshold of dangerous voltage.

Moving the cell up and down in respect of the furnace is useful method for varying the sample average temperature and the steepness of the temperature gradient over the sample, but this method varies them both. The internal heater adds additional control which, when skillfully used, can achieve steeper gradients with same average sample temperature.

Internal heater operation

To operate internal heater one should have adjustable DC power source that allows limiting both Voltage and Current. Before starting the current limit should be turned to 0 Amps and the Voltage limit should be turned to the maximum, but not higher than 20V. Connect the power supply to the cell to the BNC contacts that connect to the pins where the internal heater is connected to. Turn on the power supply and confirm immediately that the applied current is 0 Amps. Then increase the applied current to for example 1 Amps, and wait to see a feedback from one of the thermocouples. Observe with increased amperes and long enough waits, while reading a feedback from various thermocouples to study the thermodynamic behavior of the setup.

General wire behavior

Name	Resistance Ω / meter	Applied current	Effect in room temperature
K+ and K- wires	3.6 and 1.3	Do not apply current	
S+ and S- wires	0.19 and 0.17	5 A	Slightly warm to touch
PtRh10% 0.25mm diam.	4.1	3 A	Faint red glow <500°C
		4 A	Very strong red glow ~750°C
		5 A	Yellow glow >1000°C