Low Pressure High Temperature Furnace
LPHT-20

User manual

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1. Introduction

LPHT-20 (LPHT-20V) is a laboratory furnace with graphite hot zone designed for rapid annealing of samples of materials at temperatures up to 2100°C in vacuum or in inert gas. A common application of the furnace is low-pressure, high-temperature (LPHT) color treatment of diamond. For instance, the furnaces are used for the reduction (eventual elimination) of brown color of CVD-grown synthetic diamonds.

2. Installation

LPHT-20 furnaces are delivered in ready to use plug-and-play condition.

WARNING! The chamber of the furnace is delivered in evacuated state. If so, DO NOT try to open the chamber lid before the furnace is hooked up to the electrical power line and the chamber is vented.

1. After unpacking, examine the furnace for visible damages. If any are found, do not hook up the furnace to the electrical power line and inform High T Technologies. If leak of vacuum oil is noticed, open right side and rear cover panels of the frame, and check oil level in the roughing vacuum pump. Add vacuum oil if needed. Use only high-quality vacuum oil (e.g., PK-001-106-T, of Pfeiffer Vacuum, Inc.). Contact High T Technologies LLC for instructions.

2. Connect frame of the furnace to the ground.

WARNING! Before connecting the furnace to the power line, make sure that all toggles and switches on the front panel are in OFF position (toggles down), the heater potentiometer knob on the heater control unit (orange box) is turned counterclockwise to its limit (position 0) and the main switch/breaker on the power supply is OFF (down position). See Fig. 1.

3. Hook up the furnace to 208-240 V, 30 A power line (plug is provided).

4. Connect furnace to the water-cooling line with water pressure of 1 atm. The use of demineralized water with high electrical resistivity is recommended.

5. Connect the exhaust outlet of the roughing pump to exhaust system.

6. (Optional) Connect the ventilation outlet to argon or nitrogen cylinder via low pressure regulator. The ventilation gas pressure must be slightly above the atmospheric pressure (e.g., 1050 mbar absolute pressure).

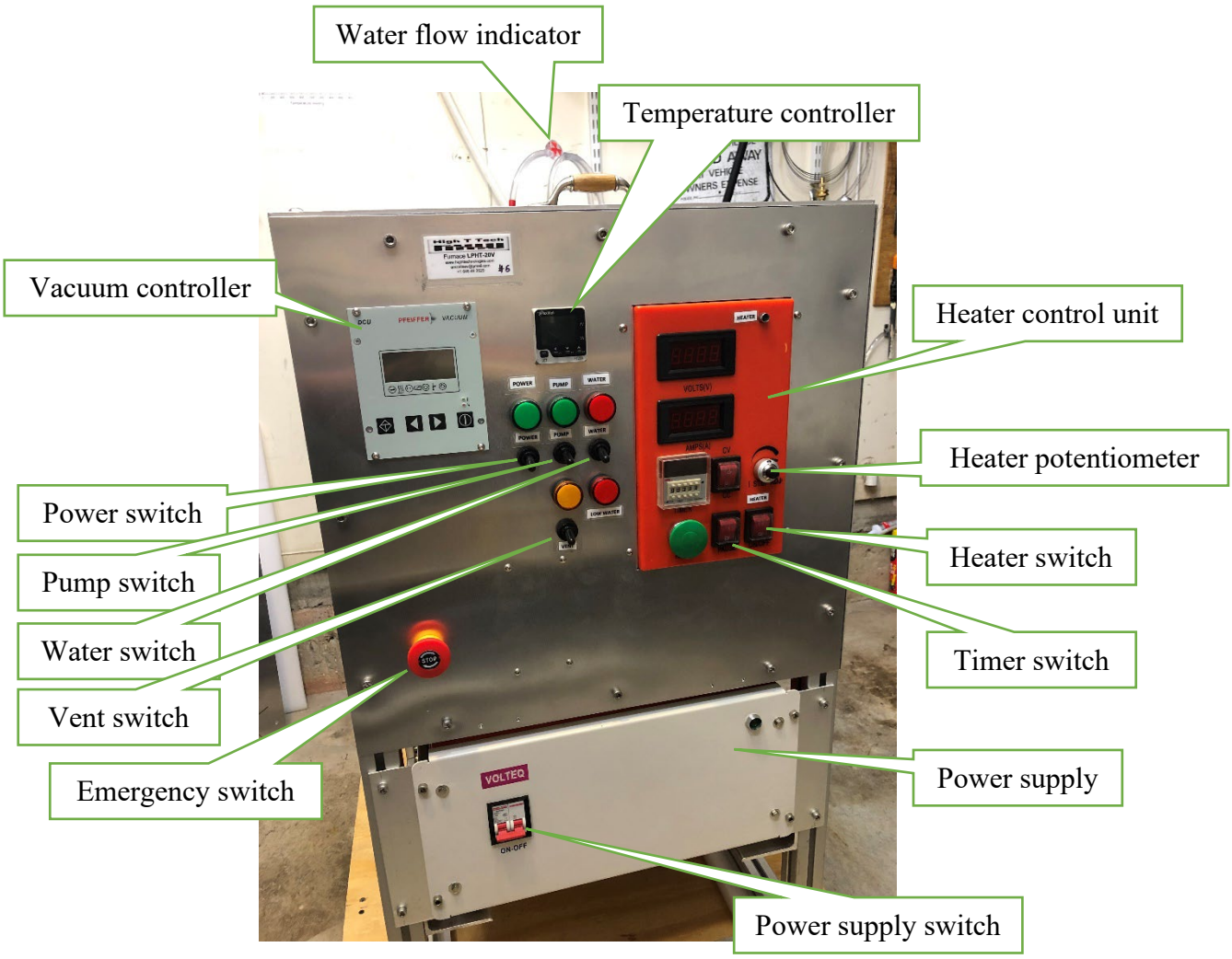

Fig. 1. Front panel of LPHT-20V furnace**.**

3. Test run

Switching furnace on

1. Make sure that the EMERGENCY SWITCH is disengaged (rotate the red knob clockwise to release the switch) and the HEATER switch on the orange control panel is off (position down).

2. Switch on POWER switch (toggle up).

3. Switch on VENT switch (toggle up). Once the chamber is filled with air (vent gas), switch off the VENT switch and open the chamber lid (Fig. 2). Examine the furnace interior for any visible damages. Use flashlight. Make sure that the container visually does not touch the surrounding filament (Fig. 2b). If damages are noticed, DO NOT switch heater. Contact High T Technologies.

4. Check the internal surface of the lid and the sealing O-ring (Fig. 2c). If needed, clean them with lint-free, alcohol-soaked tissue. Some traces of carbon deposit on the lid surface normal a normal condition. DO NOT scratch the lid with sharp hard objects.

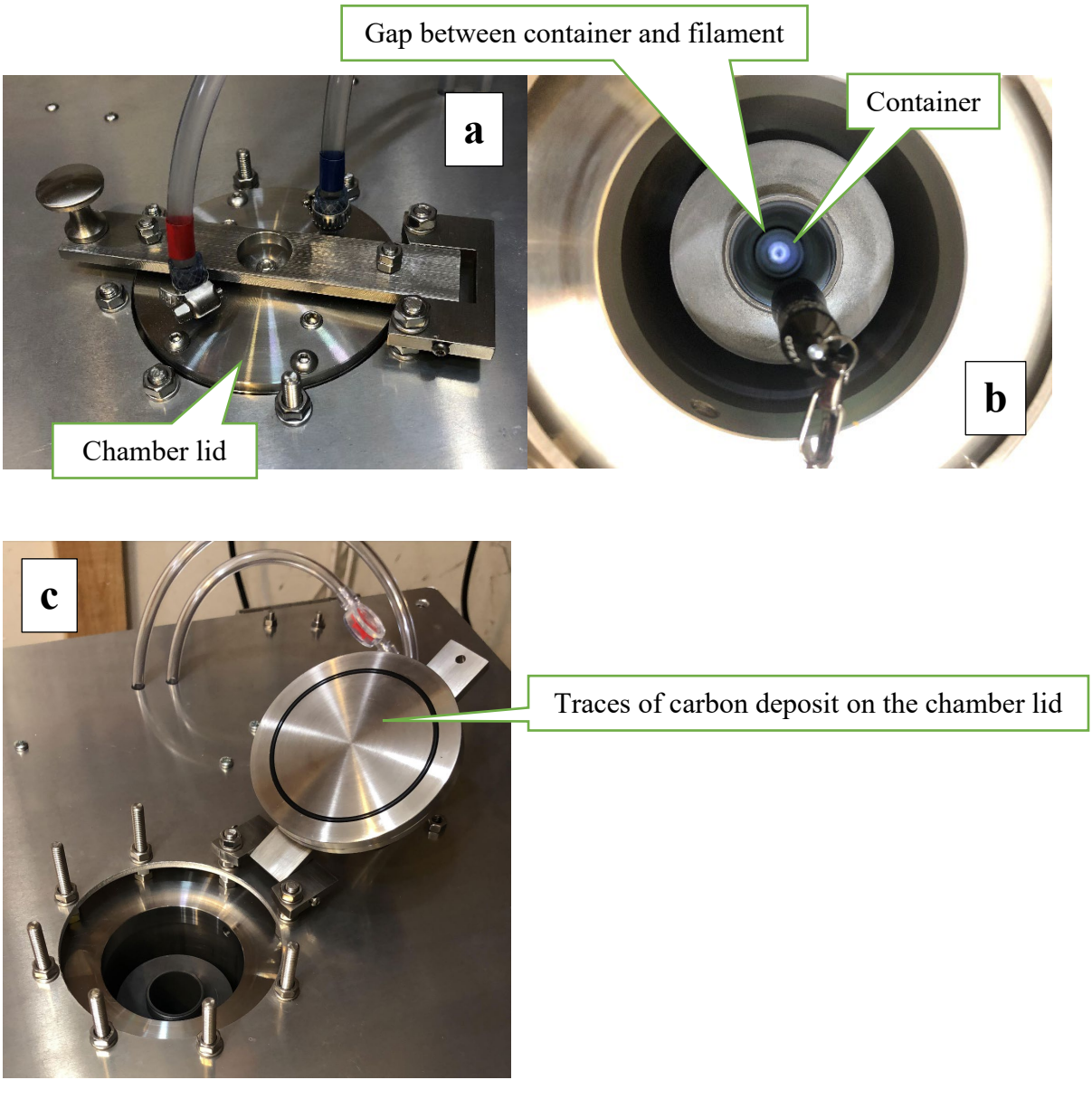

Fig. 2. Chamber lid and the view of the furnace interior.

5. Close the lid and make sure it is in its proper position. Switch on PUMP switch (toggle up).

ATTENTION! Roughing pump will not work if the VENT switch is in ON position. Make sure that the VENT switch is OFF.

6. Check the vacuum level on the display of the vacuum controller. In a few minutes it must be in the range of 10^{-1} hPa (mbar) (Fig. 3).

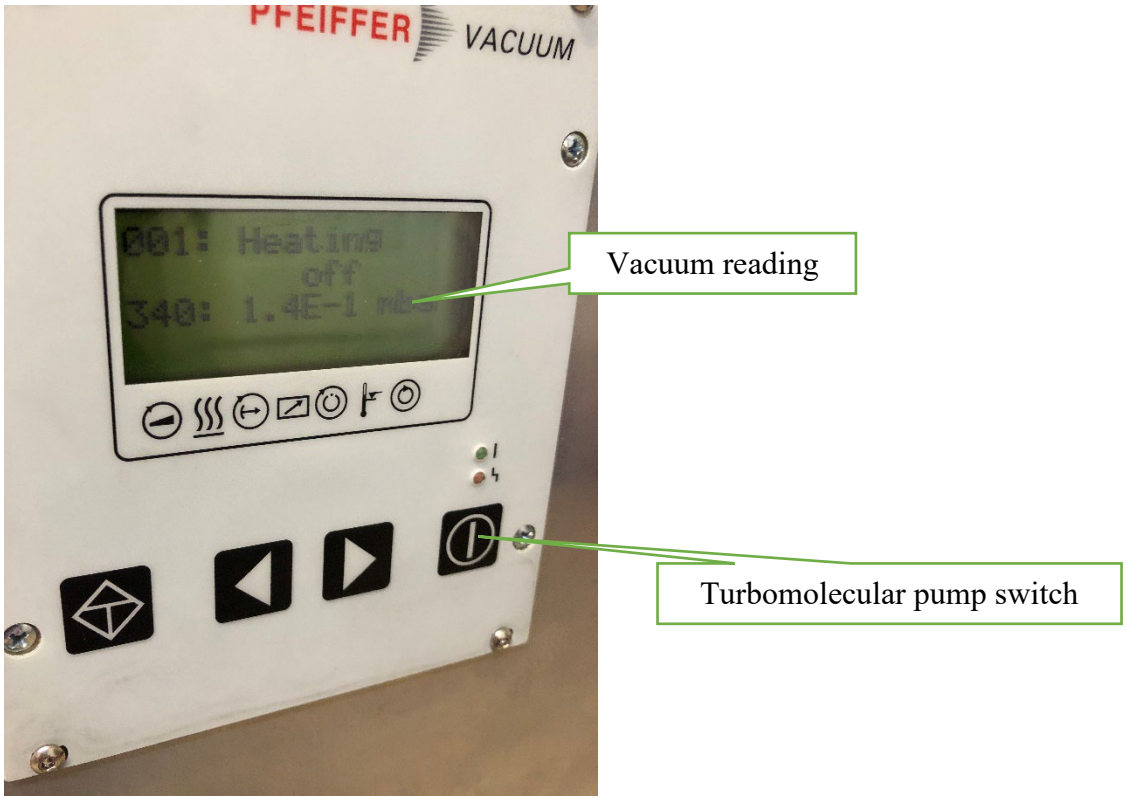

Fig. 3. Front panel of vacuum controller DCU100/DCU110.

7. Switch on turbomolecular pump (press the right button on the vacuum controller). Check the rotation speed (frequency) of the turbomolecular pump and the drive current. In a few minutes, the actual frequency of the pump must be at 1500 Hz (normal operation) and the drive current below 0.5A. Check vacuum level. In 10-15 minutes, vacuum must be in the range of 10^{-4} mbar (for the models equipped with vacuum gauge PCR280), or close to 10^{-3} mbar (for the models equipped with vacuum gauge TPR280 or equivalent).

8. Switch on WATER switch. If the red-light LOW WATER remains on, increase water pressure/flow. Make sure that water is running freely and the wheel of the water flow indicator on the chamber lid (Fig. 4) is spinning.

9. Once the vacuum level and the water flow are OK, put the switch TIMER in the position PAUSE, position down (for models equipped with timer) and switch on the main ON/OFF switch of the power supply. After that switch on the HEATER switch on the orange control panel.

WARNING! Switch on and off the main ON/OFF switch of the power supply only when the HEATER switch on the orange control panel is off.

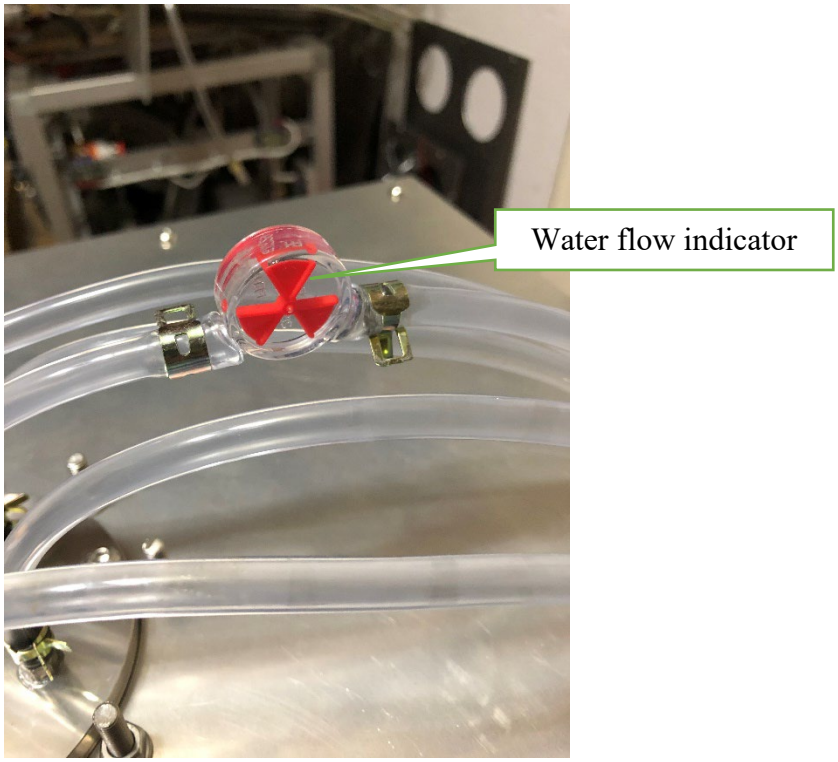

Fig. 4. Water flow indicator.

11. Slowly rotate the heater potentiometer clockwise to increase current to about 50 A. For this current, the voltage on the heater must be in the range from 6 to 8 V. **If the voltage is substantially lower than 6 V, or substantially higher than 8 V, switch of the POWER switch and contact High T Technologies!**

12. Watch the increase in temperature on the display of the temperature controller. Check vacuum. Pressure in the chamber must remain below 10^{-2} hPa. Increase current to 100A. In 10 minutes, the temperature reading must be about 1270.

NOTE: The temperature reading of the temperature controller is NOT the actual temperature inside the sample container. For the actual temperature see section "5. Temperature" below.

Switching furnace off

1. Reduce heating current to zero (heater potentiometer in position 0)

2. Switch off turbomolecular pump (press right button on the vacuum controller). In a minute switch off the PUMP switch. **DO NOT switch on the VENT switch!**

3. Wait until the temperature reading is below 300 and the frequency of the turbomolecular pump is below 200 Hz, switch on the switch VENT and wait until chamber is filled with vent gas.

4. Open the chamber lid. Examine the internal surface of the lid and the chamber interior. A minor carbon deposit may be seen on the lid surface. If you see a heavy deposition, contact High T Technologies for instructions.

Now the furnace is ready for the use.

4. Regular operation

1. Put samples inside the sample container either directly into the graphite container or placing them in a graphite vial (provided). Use long tip tweezers (provided).

!!! WARNING !!! Be very careful when manipulating with tweezers inside the furnace. All internal graphite parts are very fragile!!!

2. Close the lid and start the heating process as described above. Set the current corresponding to the desired temperature (Fig. 5, 6) and check temperature on the display of the temperature controller.

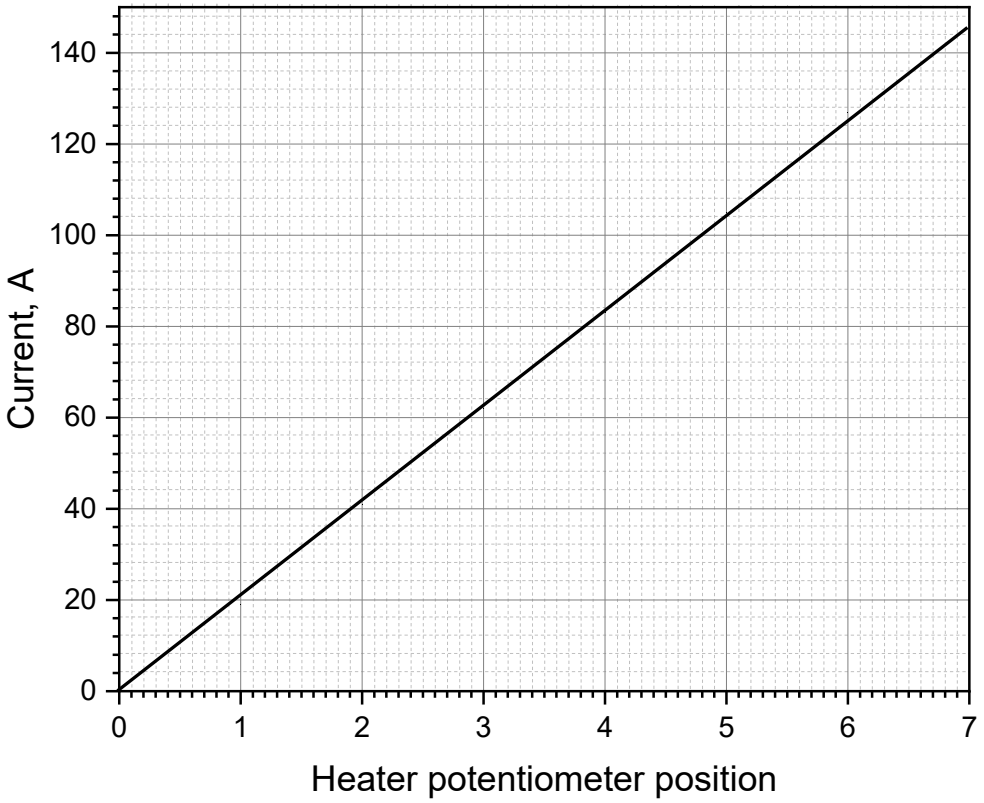

Fig. 5. Drive current in the heater versus position of the heater potentiometer.

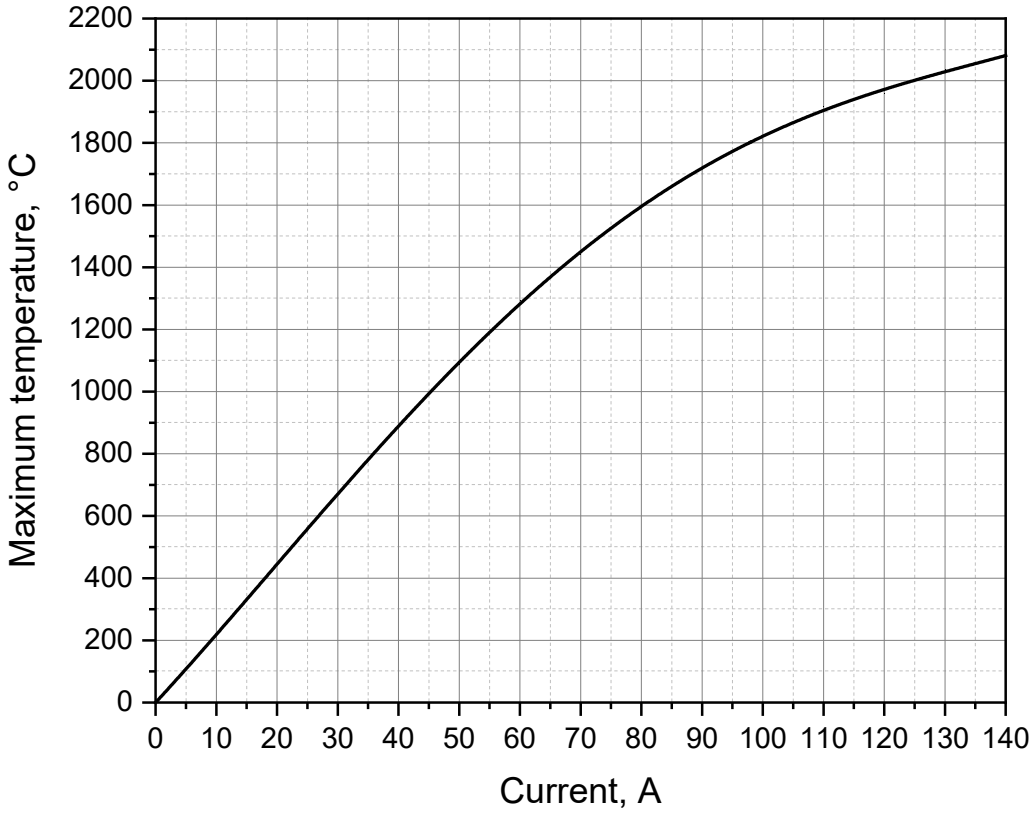

Fig. 6. Dependence of the maximum temperature in the sample container versus current in the heater as measured in vacuum of 1e-3 mbar. The maximum temperature may vary depending on the residual gas content and the pressure level in the chamber. Normally, the maximum temperature is reached in about 5-7 minutes.

WARNING!!! When working with diamond samples, do not increase current over 130A. At this current, temperature in the furnace may exceed 2100°C and the samples will be graphitized completely!

WARNING!!! Never increase current over 150A. At this current, temperature in the furnace would exceed 2300°C and it may cause destruction of thermocouple, filament and thermal shield!

5. Temperature and heating kinetics

Furnace LPHT-20 is equipped with a custom-made thermocouple. The thermal electromotive force of this thermocouple is proportional to that of the standard W-Re thermocouple. However, the actual reading is lower. Every thermocouple is calibrated individually in the temperature range from 600°C to 2000°C by melting of pure metals Al, Au, Cu, Ni, Pt and Rh. The representative calibration curve of thermocouple is shown in Fig. 7. The accuracy of the temperature measurement is about ±20°C.

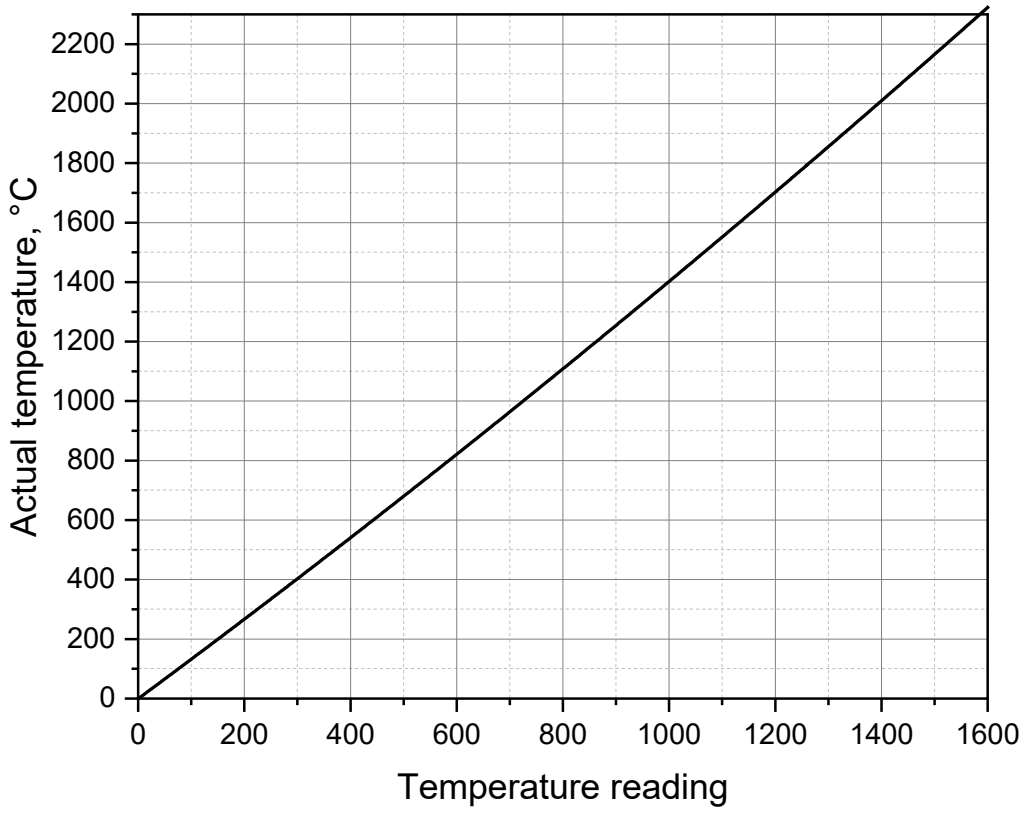

Fig. 7. Correspondence of the reading of temperature controller to the actual temperature in sample container.

The current in the heater determines the maximum temperature achieved in the sample container. In many cases, if the exact temperature is not required, the annealing process can be performed just setting the corresponding current and the heating time. Once current is set, it takes several minutes to achieve the maximum temperature. A typical increase in temperature for current 130 A is shown in Fig. 8.

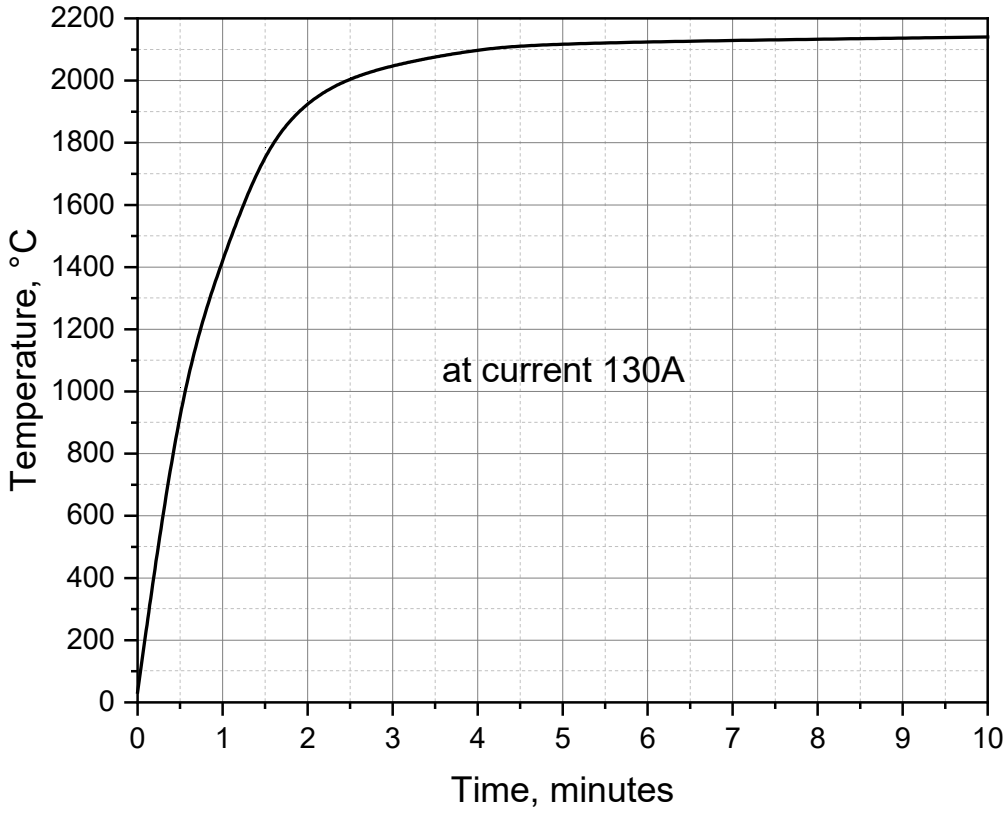

Fig. 8. Increase in the temperature inside sample container at a current of 130 A. Temperature reaches maximum in about 5 minutes.

6. Short-time operation

Speed of the heating-up strongly depends on the current: the high current, the faster heating-up. This feature is used for short-time (pulse) annealing. The process of the fastest heating at a current of 157 A is shown in Fig. 9.

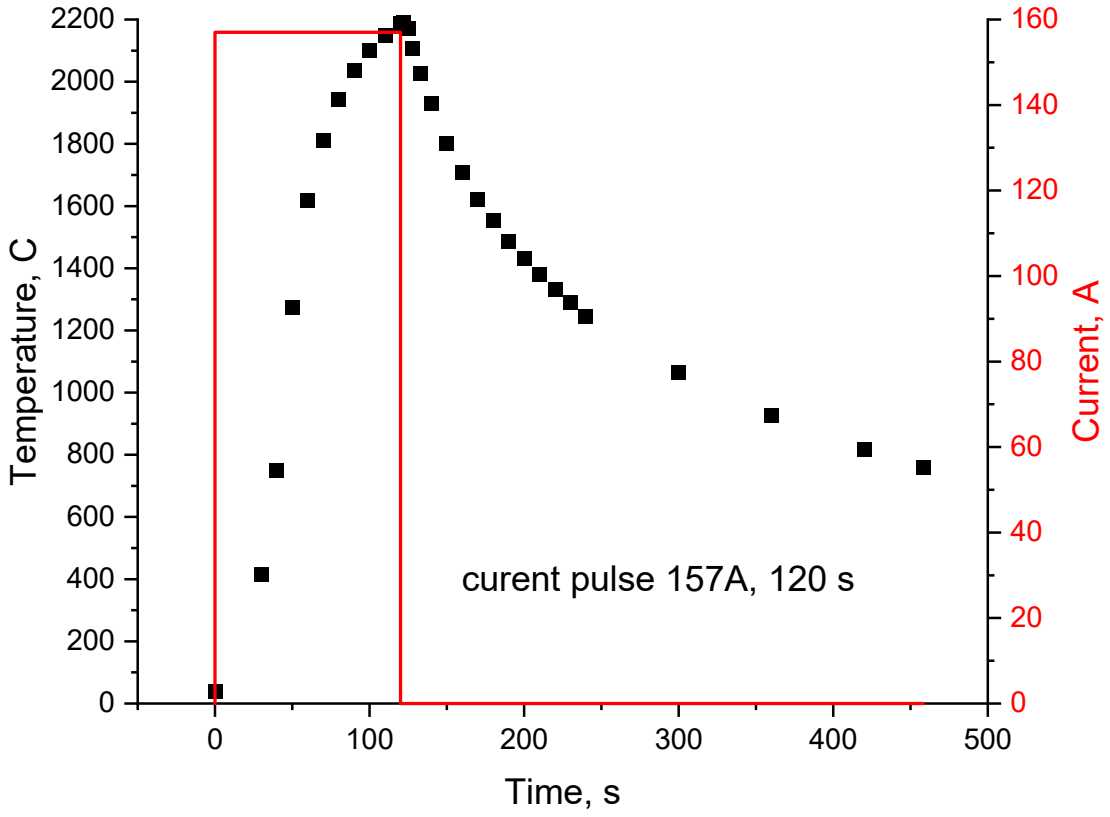

Fig. 9. Pulse annealing at a temperature of 2000±100^{o}C for 50 seconds. The process is performed with current pulse 157A for 2 minutes.

An example of a short time heating at temperature 1900-2000^{o}C for 3 minutes is shown is Fig. 10. The process was performed with current pulse 125A for 5 minutes.

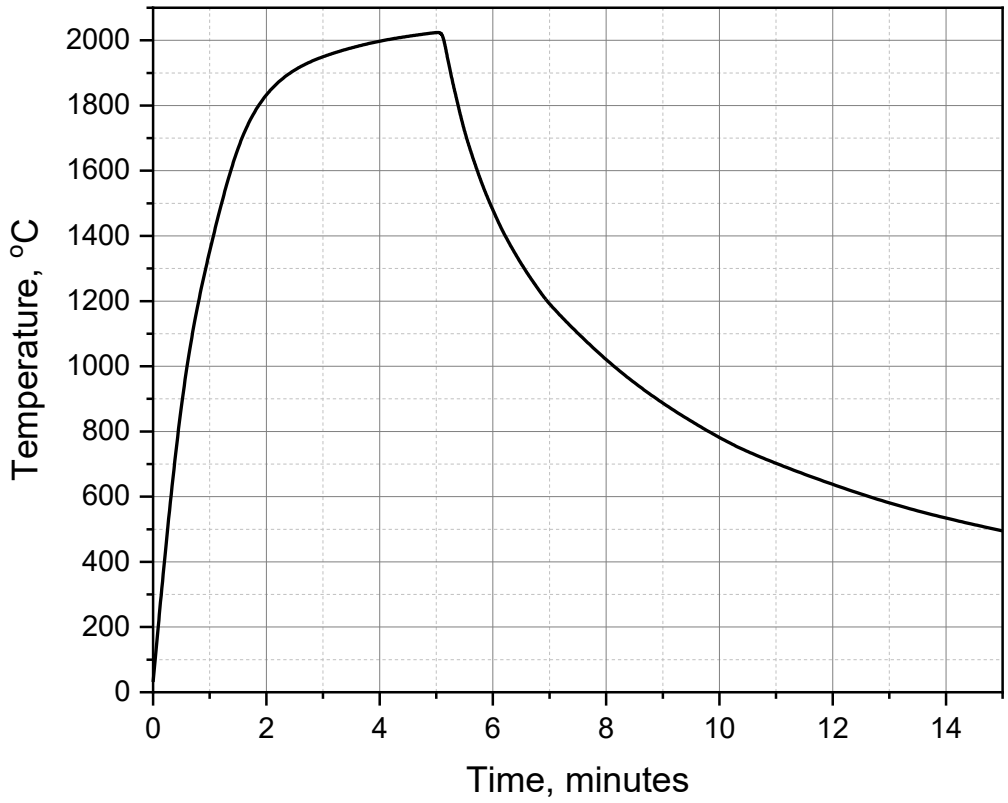

Fig. 10. Temperature vs. time plot of 5-minute heating process at a current 125A. This regime is usually used for removal of brown color of CVD diamonds.

7. Maintenance

1. Keep sample container clean. Remove debris chipped from the annealed samples via soft plastic tube attached to vacuum cleaner.
Be careful! Remember, the graphite parts are very fragile.

2. Always check the internal surface of the chamber lid for excessive deposits and the sealing o-ring for damages. If needed, remove the deposit and contaminations from the lid and o-ring with acetone/alcohol and soft lint-free tissue.

3. From time to time check the level of vacuum oil in the roughing pump.

4. Change oil in the roughing pump every 6-12 months.

In case of any issues and concerns, please contact High T Technologies for help: info.highttechnologies@gmail.com.

7. Specifications

Sample container size:	diameter 20 mm, height 20 mm
Matrial of hot parts:	graphite
Temperature range:	from 400°C to 2100°C
Temperature ramp up/down:	2 to 10 minutes* (depending of temperature)
Treatment time:	1 min to several hours* (depending on temperature)
Base vacuum:	5e-5 hPa
Vacuum during heating:	5e-5 to 1e-2 hPa* (depending on temperature and time)
Control:	semi-automatic
Input power:	one phase 208-240V, 30A
Cooling:	demineralized water at a pressure of 1 atm, 10 l/min
Weight:	40 kg
Dimensions:	550 mm (width), 620 mm (depth), 100 mm (height)

*Consult High T Technologies, LLC (info.highttechnologies@gmail.com)