Graphitisation up to 3000 °C

High-tech Applications | Heating Methods | Temperature Measurement and Uniformity | Atmospheres and Vacuum

Chamber furnace HTK 400 GR/22 up to 2200 °C with fully automatic control by PLC

Chamber furnace HTK 8 GR/22 up to 2200 °C with semi-automatic control

Chamber furnace HTK 25 GR/30 up to 3000 °C with fully automatic control by PLC and pyrolysis package

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Fig. 1: Graphite / Carbon in High-Tech Applications

Graphitisation at up to 3000 °C – What is it good for?

Graphite is a soft, slippery, greyish-black substance. It has a metallic luster and is opaque to light. Graphite is a good conductor of heat and electricity. Often graphite is simply named carbon. Under inert gas or vacuum graphite is **extremely temperature-resistant** which makes it an interesting material for high temperature applications.

By heating graphite up to 3000 °C its properties are optimized. As a result, heat treatment of graphite is a growing market and **graphite has become an important material** for a variety of applications all over the world. Graphite is often part of composite materials.

In the **automotive industry**, for example, graphite is used for manufacturing brakes, brake linings, clutch facings, engine parts, friction components, mechanical seals and also as a substitute for steel or aluminum in car frames.

The latest most notable field of application are the **lithium-ion batteries** of laptops, small electronic devices, tools and electrical cars. Another area is the production of alkaline batteries.

Less known by end users, however crucial to heavy duty industrial applications is the manufacturing of **diamond tools / special ceramics** or the use as additive in anti-corrosive paints.

A more glamorous application is the transition of purified graphite to **artificial diamonds**. A lot dirtier but more important for the world's industry is the use as roughing electrodes for **aluminium production**.

Unusual but very famous applications are in carbon brake disks in **Formula One racing cars**, or as part of so-called reinforced carbon which is used for the nose cone and wing leading edges of the **Space Shuttle orbiter** to resist enormous temperatures during re-entrance into the earth's atmosphere.

Heat treatment and the change of material properties

Graphitization can be defined as the structural change from highly disordered or defective carbon atom structures towards a perfect three-dimensional crystal of **pure graphite**. Ideally, graphite is arranged in layers, each of these layers is a separate supra-molecule called graphene (see fig. 2).

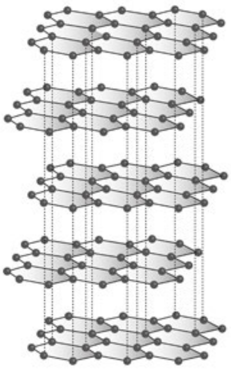


Fig. 2: Cut out of carbon atom layers (aka graphene) in graphite

The ordering process is initiated by heat treatment up to 3000 °C under inert atmosphere. The original carbon material consists of multiple small domains of graphene molecules called basic structural units (BSU). During heat treatment those small domains grow, all differences in orientation of the layers are eliminated and large **straight layers are formed** (see fig. 3 and 4).

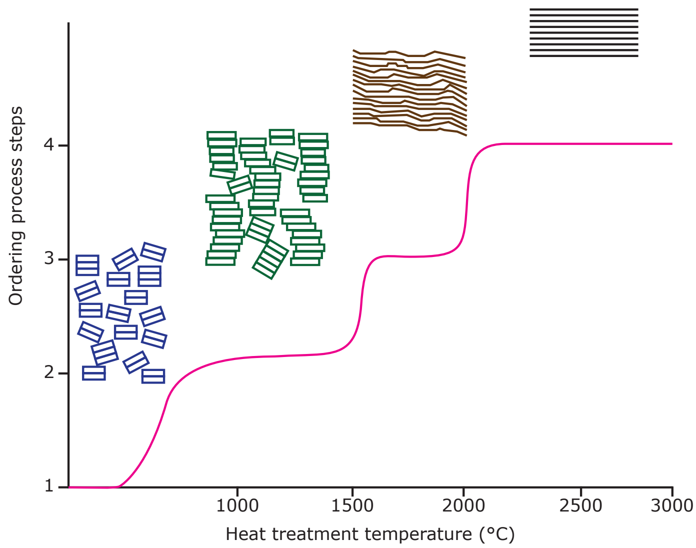


Fig. 3: Representation of the graphitisation process and of ordering the BSU (basic structural units) of the graphite with increasing temperature

After carbonisation the carbon atoms are in chaotic order and therefore don't have ideal properties. **Increasing the temperature allows the atoms to move to more ideal positions** (see fig. 3) and eventually, at very high temperatures, form ideal graphite with superior properties. The absolute temperatures for graphitisation are debatable.

The initial stage of graphitisation takes place between 1900 °C and 2000 °C and leads to interlayer distances > 3.42 Å (see fig. 4). After complete straightening the layer distances are further reduced.

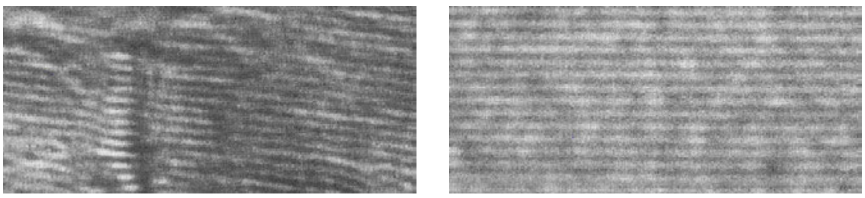


Fig. 4: Electron microscope pictures: left graphite layers before heat treatment with disordered layers and right after heat treatment up to 2200 °C with completely straightened layers

After final heat treatment in a Carbolite Gero furnace at up to 3000 °C the **graphite properties are almost ideal, as well as homogeneous and very reproducible** making it a perfect starting material for several industrial applications (see above).

Heating Methods on the Way to Graphitisation up to 3000 °C

There are two general ways for graphitisation in Carbolite Gero furnaces. Either the starting material already consists solely of carbon atoms and is very clean with minor impurities. In this case a graphite furnace of the LHTG range or the HTK range up to 3000 °C is suitable.

Fig. 5: Overview of Carbolite Gero furnaces suitable for carbonisation and graphitisation

Heating methods (may need further equipment)	Temperature ranges and their related atmospheres and vacuum ability		
	up to 1100 °C	up to 2200 °C	up to 3000 °C
GLO chamber furnaces with inconel retort (CrFeAl heating elements)	N_2, Ar, H_2, vacuum, oxgygen or air		
HTK graphite vacuum chamber furnace (Gr heating elements and graphite felt)	N_2, Ar, H_2, vacuum (no oxgygen or air)		Ar

However, if the starting material consists of organic matter of unknown composition or contains a large amount of impurities, it is recommended to **pre-carbonize the sample in a low temperature hot wall furnace of the GLO range up to 1100 °C** to purify the sample under inert gas atmosphere until it is suitable to be heat treated in a more sensitive high temperature graphite furnace (see fig. 6 and 7).

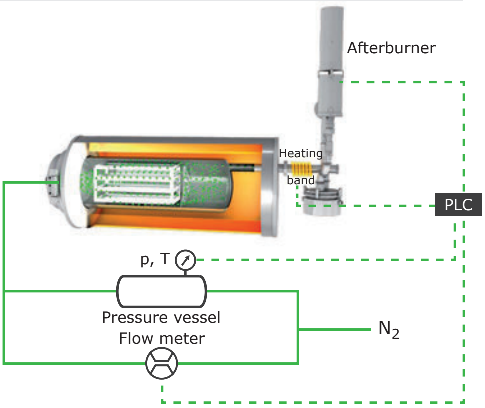


Fig. 6: Side view of a schematic drawing of a GLO hot wall furnace equipped with an afterburner for debinding and pyrolysis up to 1100 °C

Fig. 7: GLO 120/11 Furnace with debinding and pyrolysis equipment

After purification in a GLO furnace at up to 1100 °C the sample can be moved into a graphite furnace for final graphitisation at up to 3000 °C.

For samples with only small impurities and low contamination of non-carbon atoms Carbolite Gero offers **specialized cold wall HTK graphite furnaces with dedicated debinding equipment**, where carbonisation and graphitisation are carried out in one heat treatment step. Those furnaces are equipped with a retort and an intelligent gas guidance system which assures that the impurities are safely discharged from the furnace into the afterburner.

During the first step of heat treatment by slowly ramping up to 800 °C the inert gas enters the retort through small holes. Once the gas enters the retort the only way out of the furnace is through the central gas outlet which is located directly inside the retort. All volatile gases are forced to exit the furnace directly through the heated gas outlet (see fig. 8 and 10). **Hence, contamination of the furnace is prevented which means reduced production costs**.

Fig. 8: Schematic view into a retort in a HTK furnace with central gas outlet trough the bottom of the retort

Fig. 9: HTK 25 GR/30 furnace up to 3000 °C with debinding equipment

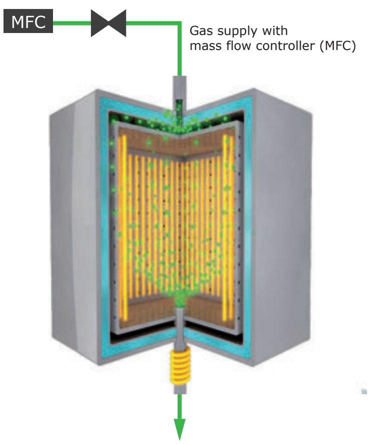


Fig. 10: Schematic view into a cold wall chamber furnace HTK equipped with a retort, gas guidance system and heated gas outlet leading to the afterburner

The volatile organic matter is safely combusted in an active afterburner (see fig. 9). Usually, debinding is carried out in controlled inert gas atmosphere conditions at temperatures below 800 °C with several dwell time steps for debinding different kinds of impurities. **After completed carbonisation the furnace will automatically ramp up to 3000 °C (depending on the furnace model) for final graphitisation**. See figure 12 for a typical debinding process with several dwell times below 800 °C combined with a subsequent graphitisation step up to 2800 °C with fast ramping after complete debinding.

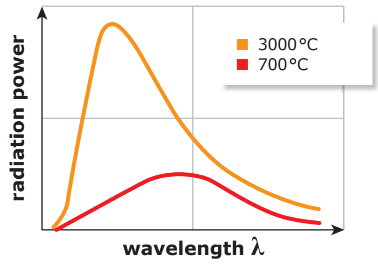


Fig. 11: Schematic comparison of the radiation intensities at 700 °C and 3000 °C inside a Carbolite Gero graphite furnace measurable with a pyrometer

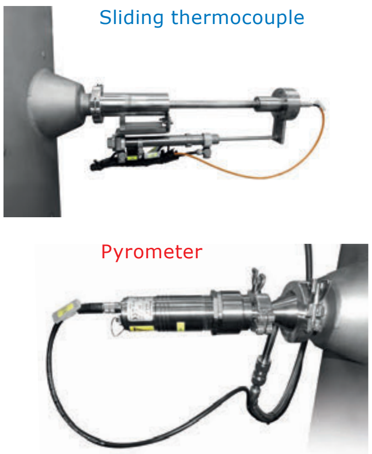
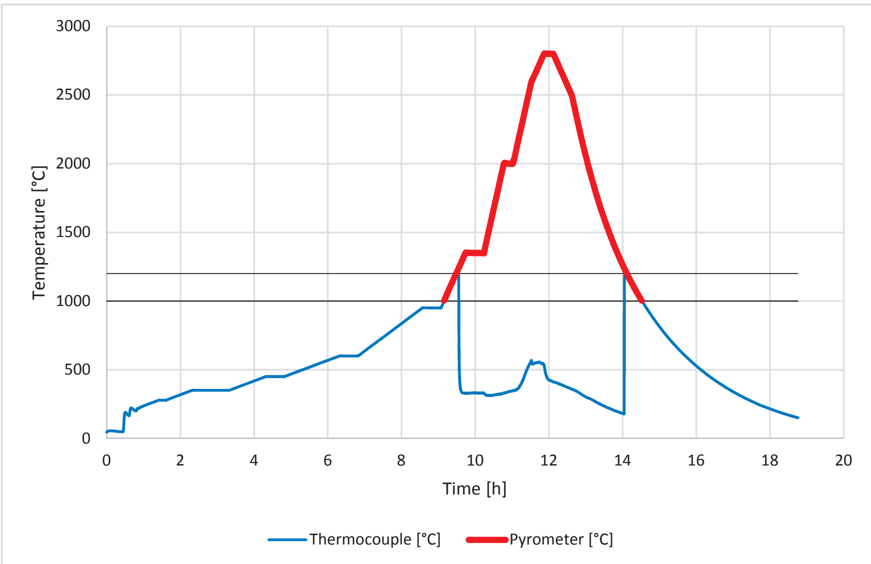


Fig. 12: Temperatures measured by a sliding thermocouple (blue) and a pyrometer (red) during a graphitisation run up to 2800 °C. Simultaneous temperature measurement between 1000 °C and 1200 °C.

Advanced temperature measurement up to 3000 °C

For graphitisation a precise temperature measurement at low as well as high temperatures is necessary. But since a pyrometer is not able to accurately measure low temperatures, and thermocouples are not able to measure high temperatures, CARBOLITE GERO offers an optional sliding thermocouple type S which is located in the hot zone of the furnace to control and monitor the temperature in a range from room temperature up to 1200 °C (blue temperature values in figure 12).

At 1200 °C the sliding thermocouple is moving out of the hot zone and the pyrometer (red temperature values in figure 12) takes over temperature control. Additionally, a rough calibration and proof of the accuracy of the pyrometer is possible by comparing the temperatures measured by the sliding thermocouple and the pyrometer in the overlapping temperature range between 1000 °C and 1200 °C.



After a short period of dwelling at highest temperature of 2800 °C, the furnace naturally cools down until the door can be opened at a temperature below 200 °C.

A long dwell time at high temperatures above 2800 °C is not necessary for graphitisation, at those temperatures the graphitisation process is extremely quick.

High quality heating elements and insulation for superior temperature uniformity

For highest quality in graphitisation all materials in direct contact with the customer's graphite parts need to be as clean as possible. **The hot zones of all CARBOLITE GERO graphite furnaces are constructed exclusively of pure graphite materials**.

Within the water-cooled stainless steel vessel all graphite parts are designed for the highest mechanical stability. Surrounding the heating elements Carbolite Gero fitted high quality graphite felt insulation for highest possible sample temperature uniformity (see fig. 13 and 17). An optional graphite retort can be specified to protect the heating elements from wear in case of sample outgassing (see above).

For the top loader furnace LHTG (see fig. 14) a circular **mantle heater in a perfectly symmetrically meander** is fitted. This kind of arrangement is located around the sample like a belt, while the upper roof and lower bottom of the usable space is tightly sealed with graphite felt as insulation.

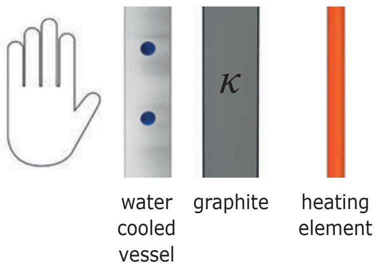


Fig. 13: Schematic heating concept of graphite heating elements and graphite felt insulation within the water-cooled furnace vessel

Fig. 14: Top loader furnace LHTG up to 3000 °C;

Fig. 15: View from above onto the circular heater of the LHTG furnaces, here with central gas outlet in the middle of the bottom

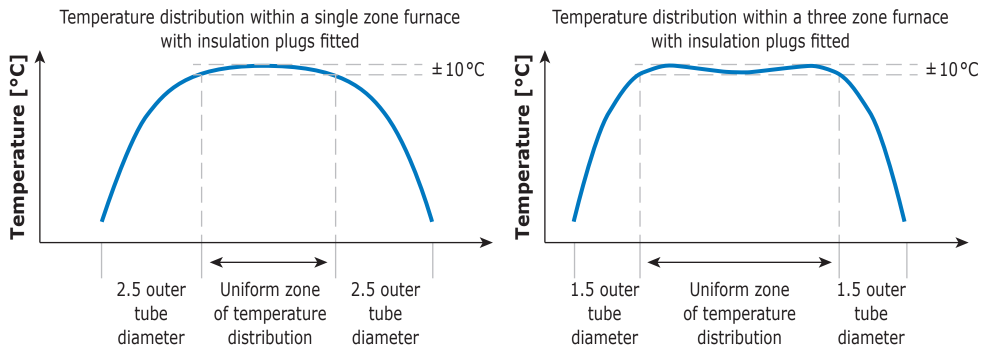


Fig. 16: General difference of the uniformity between a single and three zone furnace

For the front loader HTK Carbolite Gero offers door and back wall heater in a three zone arrangement as an option to increase the uniformity (see fig. 18). Each of the additional zones has either a thermocouple or a pyrometer for separate temperature control. Usually the door and back wall zones are set to slightly higher temperatures during heat treatment to improve the uniformity even further (see fig. 16).

frame 1
water cooled vessel 2
heating cassette 3
groove for the sealing 4
front door 5
graphite insulation 6

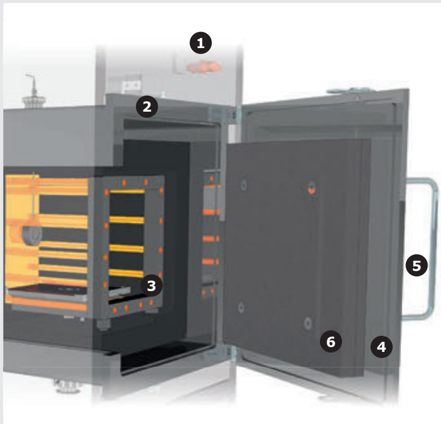


Fig. 17: View into a graphite HTK chamber furnace

Fig. 18: Door heater of an HTK furnace

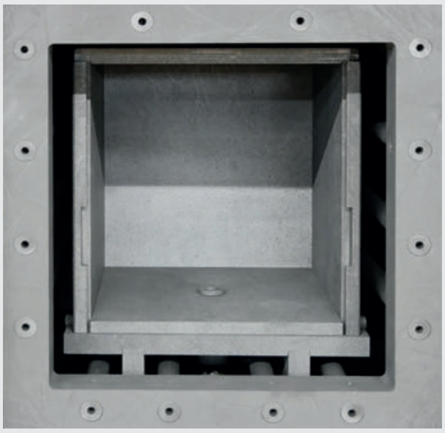


Fig. 19: HTK 8 mantle heater with opened retort

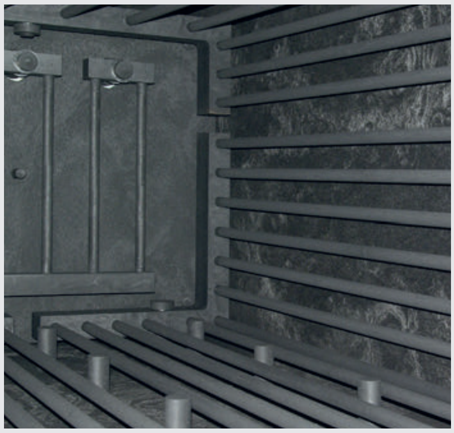


Fig. 20: View into HTK 400 mantle heater with back wall heating and several graphite pillars at the bottom supporting the movable retort for convenient loading and unloading of the furnace

In case of the front loader furnaces of the HTK range the mantle heater heats highly symmetrically from four sides (left, right, below and above) as a standard. For small volumes one heating zone surrounding the sample is provided. However, for larger furnaces multiple heating zones are available (see fig. 16).

Controlled Atmosphere, Partial Pressure and Vacuum in Carbolite Gero furnaces during Heat Treatment

Carbolite Gero offers all kinds of atmospheres to match the customer's needs during the graphitisation process. **However at highest temperatures above 2200 °C, it is strongly recommended to use Argon at 1 atm only.**

In general the definition of heat treatment in controlled atmosphere is to maintain the purity of the used inert (N_2 or Argon) or reactive gas (e.g. H_2, CO, CO_2 and many others on request) which flows through the furnace (see fig. 21).

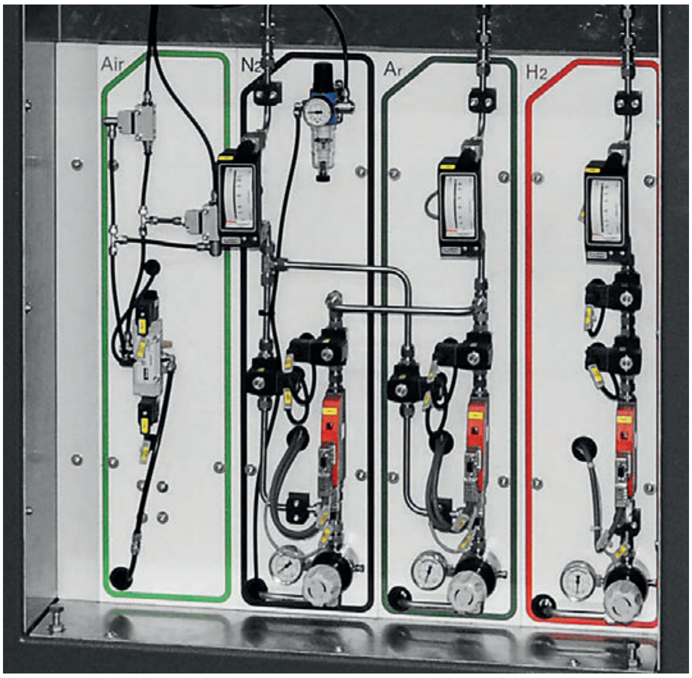


Fig. 21: Fully automated multiple gas controls by MFC at a Carbolite Gero graphite chamber furnace, air is only used for pneumatic valves

The gas is purged inside the furnace, generating a gentle overpressure, and is released again. Prior to heat treatment, the furnace is evacuated with a vacuum pump. Afterwards it is purged with inert gas, hence **the gas purity is maintained**. To exchange the atmosphere it is also possible to simply purge the furnace with inert gas without prior evacuation. This is a reasonable solution for tube furnaces. However, for a chamber furnace with graphite heating elements and graphite felt insulation, evacuation is the only possible solution.

The definition of the **partial pressure mode** is to have a defined inert gas flow at a defined lower pressure inside the furnace (see fig. 22).

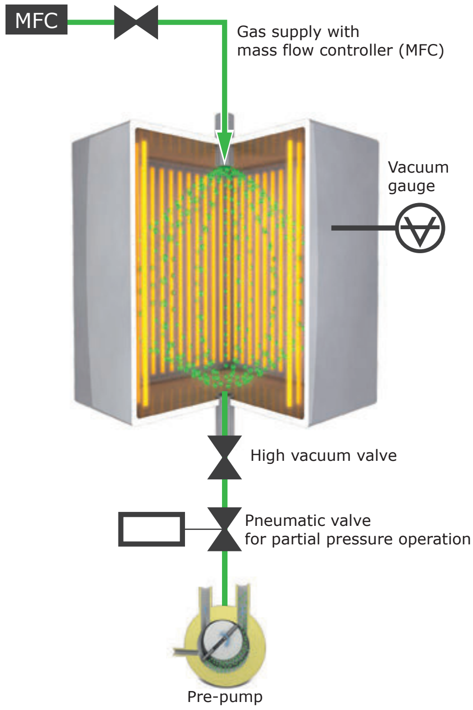


Fig. 22: Schematic drawing of the inert gas partial pressure equipment fully automatically controlled by a PLC

The incoming inert gas flow controlled by a MFC and the pressure can be adjusted by the user in the program table. A pneumatic valve with valve position indicator in front of the vacuum pump is automatically opening and closing precisely to maintain the required lower pressure inside the furnace. **The pressure can be set between 10 and 1000 mbar** (see fig. 23). Usually, single or double stage rotary vane pumps are used for partial pressure control of the inert gas.

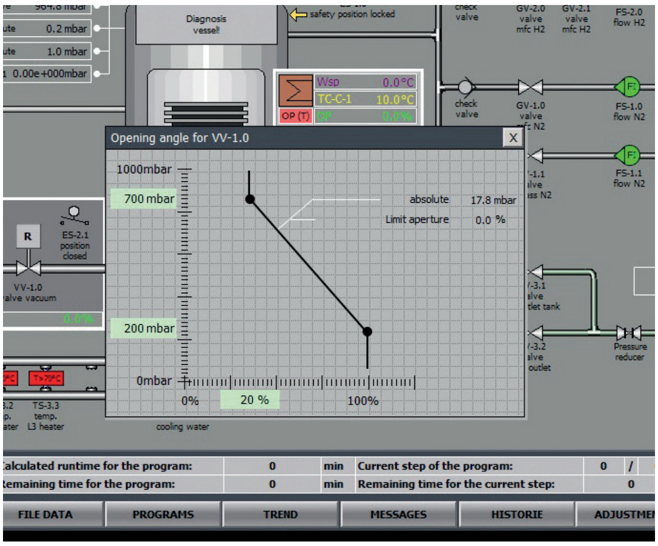


Fig. 23: The PLC software adjusts the opening angle of the pneumatic driven ball valve and the MFC so that the desired lower pressure in the furnace is maintained constantly

The **optional vacuum pumps** are classified with a defined end pressure. This is measured in compliance with the PNEUROP standard. Here, the pressure at the pump's closed flange is measured. Attaching the pump to a vacuum recipient like a furnace changes the situation. The working pressure which is achieved after a certain time depends on many factors: The leakage rate of the vacuum recipient, the desorption rate of the inner surfaces, possible outgassing from the sample or other devices. The leakage rate of the recipient is measured and defined by CARBOLITE GERO. All sealings are carefully chosen to provide lowest possible desorption rates. The vacuum devices are cleaned prior to assembly. What cannot be controlled is the outgassing from the customer's sample, the cleanness in the laboratory as well as the humidity of the ambient air.

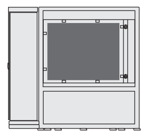
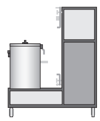
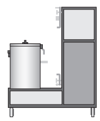
CARBOLITE GERO designs the pumping units in such a way, that for clean, cold, dry and empty (ccde) conditions inside the furnace, the specified working vacuum is achieved in a reasonable time. For ccde conditions, the working vacuum is given for each pumping unit in the above section. A high vacuum furnace should always be flooded with inert gas. The time the furnace is open and the ambient air affects the interior should be reduced to a minimum.

Conclusion

With all these options on offer CARBOLITE GERO is an excellent partner for graphitisation solutions up to 3000 °C. The materials used and the intelligent design of the furnaces in combination with more than 30 years of experience in the development of high temperature furnaces make our furnaces the ideal choice for your graphitisation projects.

Please contact us for a tailor-made solution for your heat treatment needs.
See a brief selection of our 3000 °C graphite furnaces in the following table.

CARBOLITE GERO 3000 °C graphite furnace selection

	Graphite		
			
Model	HTK 80 GR/30-1G	LHTG 100-200/30-1G	LHTG 200-300/30-1G
External dimensions			
***H x W x D* [mm]**	2500 x 2400 x 2500	1800 x 1600 x 1000	1800 x 1600 x 1000
Transport weight			
Complete system [kg]	4000	1000	1500
Usable space			
Volume [l]	80	1.5	9.4
***H x W x D*, usable space without retort [mm]**	400 x 400 x 500	–	–
***Ø x H*, usable space without retort [mm]**	–	100 x 200	200 x 300
***H x W x D*, usable space with retort [mm]**	380 x 380 x 480	–	–
***Ø x H*, usable space with retort [mm]**		90 x 200	180 x 300
Thermal values			
T_{max}, vacuum [°C]	2200	2200	2200
T_{max}, atmospheric pressure [°C]	3000	3000	3000
ΔT, between 500 °C and 2200 °C [K] (according to DIN 17052)	± 10	± 10	± 10
Max. heat-up rate [K/min]	10	20	20
Cooling time [h]	8	5	7
Connecting values			
Power [kW]	250	40	85
Voltage [V]	400 (3P)	400 (3P)	400 (3P)
Current [A]	3 x 362	3 x 310	3 x 85
Series fuse [A]	3 x 400	3 x 125	3 x 100
Vacuum (option)			
Leakage rate (clean, cold and empty) [mbar l/s]	$< 5 \times 10^{-3}$		
Vacuum range depending on the pumping unit	rough or fine vacuum		
Cooling water required			
Flow [l/min]	200	30	75
Max. inlet temperature [°C]	23	23	23
Gas supply			
Nitrogen or Argon flow, others on request [l/h]	200 – 2000	50 – 500	50 – 500
Controller			
Manual operation	Eurotherm with KP 300 panel		
Automatic operation	Siemens		