SPOTLIGHT *feature*

Ovens, Furnaces & Heating Equipment

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

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Graphite is a good conductor of both heat and electricity. However, in an inert gas or vacuum, graphite is extremely temperature-resistant which makes it an excellent material for high temperature applications. By heating graphite up to 3000°C its properties are optimised. As a result, heat treatment of graphite is a growing market and graphite has become an important material and it is a component of many composite materials.

In the automotive industry, graphite is used for manufacturing brakes, brake linings, clutch facings, engine parts, friction components, mechanical seals and also as a substitute for steel or aluminium in car frames. Other notable current fields of application are lithium-ion batteries for laptops, small electronic devices and electric cars. It is also used to manufacture alkaline batteries.

Graphite is crucial in the manufacturing of diamond tools/special ceramics, serves as an additive in anti-corrosive paints and is ideal for roughing electrodes in aluminium production.

The most commonly known use of graphite is for carbon brake disks in Formula One racing cars, or as a component of 'reinforced carbon' which is used for the nose cone and leading edges of reusable space launch systems to resist enormous temperatures during re-entry into the earth's atmosphere.

The Change of Material Properties when Heat-Treated

Graphitisation 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.

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 *Figure 1*).

After carbonisation the carbon atoms are in chaotic order and therefore don't have ideal

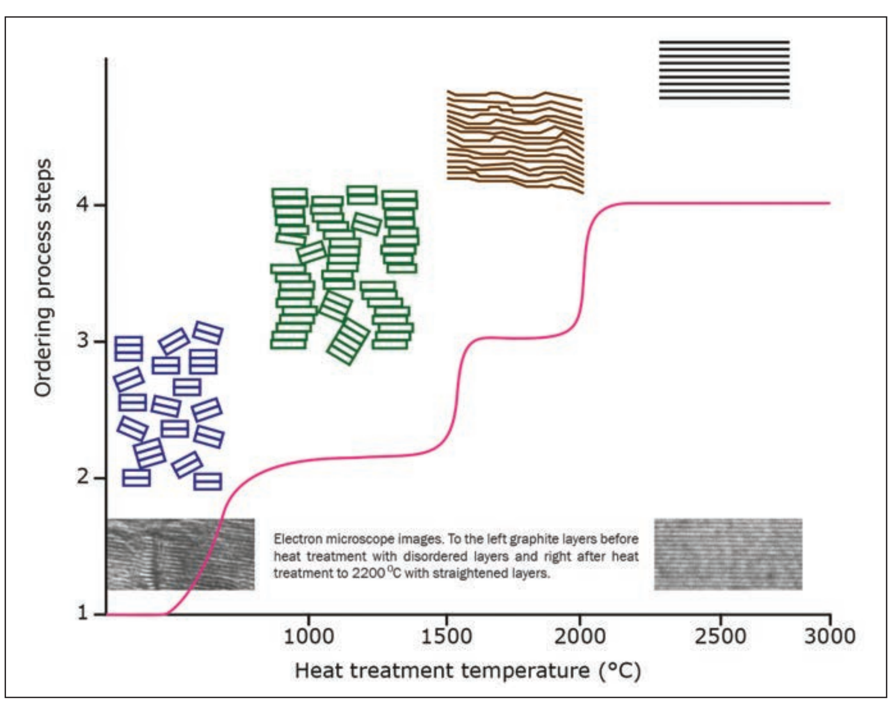

Figure 1

properties. Increasing the temperature allows atoms to move to more ideal positions and eventually, at very high temperatures, they 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. After complete straightening the layer distances are further reduced.

After final heat treatment in a Carbolite Gero furnace at up to 3000°C the graphite properties are almost ideal; uniform and very reproducible making it a perfect starting material for numerous industrial applications.

Heating Methods for Graphitisation up to 3000°C

There are two options for graphitisation in Carbolite Gero furnaces. If the starting material consists solely of carbon atoms and is clean with only minor impurities then a graphite furnace from our LHTG or HTK range is the right choice. However, if the starting material consists of organic matter or contains a large amount of impurities, it is recommended to pre-carbonise the sample in a low temperature hot wall GLO range furnace to 1100°C. This purifies the sample in an inert gas atmosphere until it is suitable to be heat treated to 3000°C in a more sensitive high temperature graphite furnace.

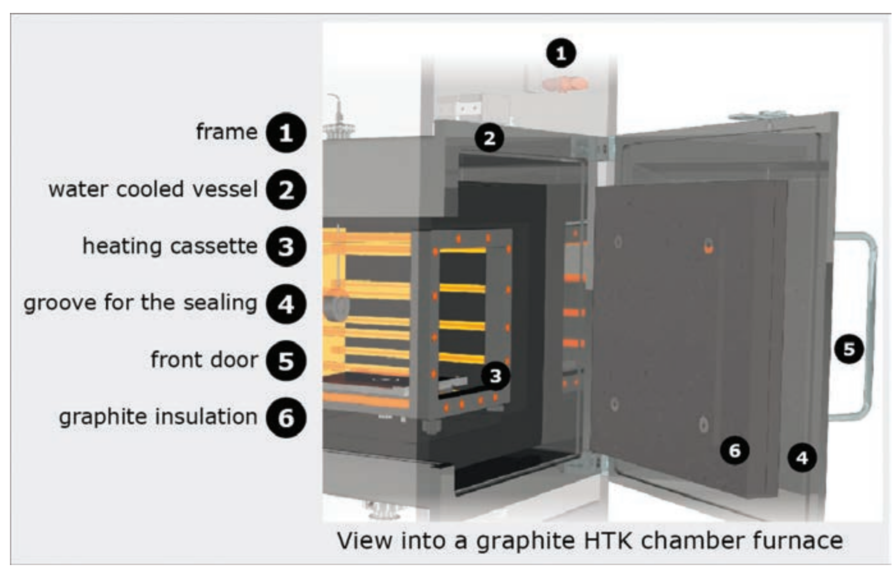

Figure 2

For samples with minor impurities and low contamination our specialised cold wall HTK graphite furnaces (See *Figure 2*) have dedicated debinding equipment, where carbonisation and graphitisation are carried out in one process. These furnaces are equipped with a gas retort and an intelligent gas system to assure volatile organic matter is safely combusted in an active afterburner. Typically, debinding is carried out in an inert gas environment at temperatures below 800°C with several dwell time steps for debinding different kinds of impurities. After completed carbonisation the furnace automatically ramps up to 3000°C for final graphitisation.

Advanced Temperature Measurement to 3000°C

For graphitisation a precise temperature measurement at low as well as high temperatures is necessary. However, a pyrometer is not able to accurately measure low temperatures,

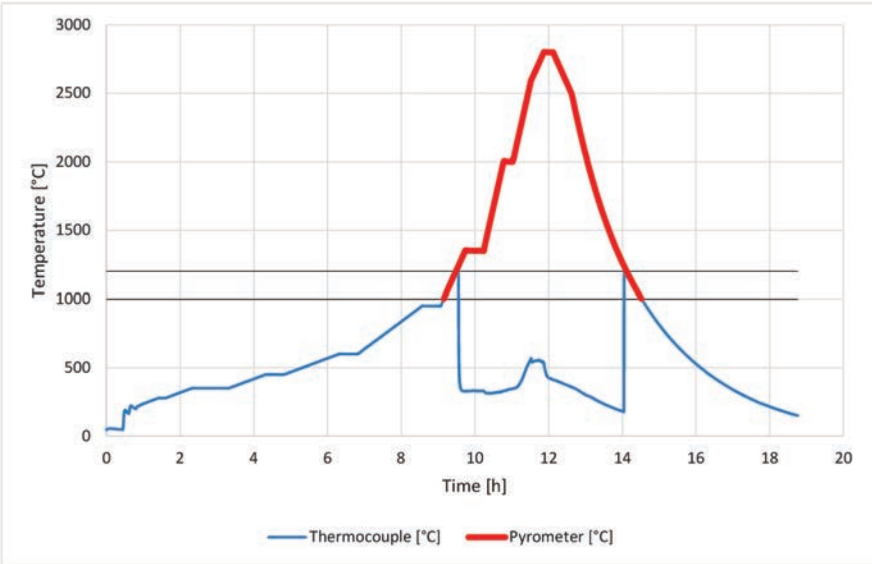

Figure 3

and thermocouples are not able to measure high temperatures. Therefore, Carbolite Gero offers an optional s-type sliding thermocouple located in the hot zone of the furnace to control and monitor the temperature from room temperature up to 1200°C.

At 1200°C the sliding thermocouple is moved out of the hot zone and the pyrometer takes over temperature control (See *Figure 3*). 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 dwell period at highest temperature of 3000°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 as at those temperatures the graphitisation process is extremely rapid.

Quality Heating Elements and Insulation for Outstanding Temperature Uniformity

The hot zones of all Carbolite Gero graphite furnaces are constructed exclusively with pure graphite materials.

Inside the water-cooled stainless steel vessel all graphite parts are optimised for stability. Surrounding the heating elements Carbolite Gero has fitted high quality graphite felt insulation for optimum temperature uniformity. An optional graphite retort can be specified to protect the heating elements from wear if outgassing to an active afterburner.

In top loading LHTG furnaces a circular mantle heater in a symmetrical pattern is fitted and located around the sample like a belt, while the roof and bottom of the space is tightly sealed with graphite felt for insulation. For front loading HTK furnaces, Carbolite Gero offers door and back wall heating in an optional three zone arrangement to increase temperature uniformity. Each additional zone 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 further improve uniformity. Finally, in front loading Carbolite Gero's HTK furnaces, heat is uniform from all four sides.

Controlled Atmosphere, Partial Pressure and Vacuums During Heat Treatment

Carbolite Gero offer atmospheres to match the customer's needs during the graphitisation process. However, above 2200°C it is recommended to use Argon at only 1 atm.

In general the definition of heat treatment in controlled atmosphere is to maintain the purity of the used inert (N_2, Ar) or reactive gas (e.g. H_2, CO, CO_2 and many others on request) which flows through the furnace (See *Figure 4*).

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

Figure 4

The selected gas is fed in to the furnace, generating a gentle overpressure, and then released again. Prior to heat treatment, the furnace is evacuated with a vacuum pump. Afterwards it is purged with inert gas to maintain purity. To change the atmosphere it is 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 felt insulation, evacuation is the only possible solution.

In Partial Pressure mode the incoming inert gas flow is controlled by a Mass Flow Controller and the pressure can be adjusted by the user. A pneumatic valve with a position indicator in front automatically opens and closes precisely to maintain the reduced pressure inside the furnace. This can be set between 10 and 1000 mbar. Usually, single or double stage rotary vane pumps are used for partial pressure control of the inert gas.

Optional vacuum pumps are classified with a defined end pressure. This is in compliance with the PNEUROP standard where 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 specified time depends on many factors: - leakage rate of the vacuum recipient; 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. Seals are chosen to provide lowest possible desorption and all vacuum devices are cleaned prior to assembly. What cannot be controlled is the outgassing from the customer's sample, cleanliness in the laboratory or the humidity of the ambient air.

Carbolite Gero designs the pumping units for clean, cold, dry and empty (CCDE) conditions inside the furnace where a working vacuum is achieved in a reasonable time. For CCDE conditions the working vacuum is specified for every pumping unit and any high vacuum furnace should always be flooded with inert gas and opening times to ambient air minimised for optimal results.

In Conclusion

With so many options on offer, Carbolite Gero is an excellent partner for graphitisation solutions up to 3000°C. The materials used and the exceptional design of our high temperature furnaces with over 30 years of experience makes them the ideal choice for your graphitisation projects.

Please contact Carbolite Gero for a tailor-made solution for your heat treatment needs.