Heat treatment at low pressures

High temperature vacuum furnaces are used for heat treatment of samples at low pressure (down to 10^{-6} mbar) and under partial gas atmospheres. In the bottom loader version the bottom is lowered and driven out of the furnace. It then pivots forward to permit easy loading. This design makes the useable volume accessible from all sides and allows for exact placement of the sample thermocouples which measure the temperature of the sample.

CARBOLITE GERO's HTBL series is available for working spaces with volumes from 50 l to 200 l and for temperatures up to 2200°C. The furnaces are designed for operation under vacuum. Any pressure between 10 and 1000 mbar can be controlled via an integrated pressure control with a precision of approximately ± 2 mbar. The pressure control works independently of the supplied gas volume which can be defined for every step of the process. At low pressure, for example, high as well as low gas volumes can be supplied. The pressure control compensates the resulting vacillations.

The furnaces are additionally equipped with redundant thermocouples and optical pyrometers to ensure high process stability. The controller carries out plausibility checks independently and, if the thermocouples and optical pyrometers deliver improbable measurement results, switches to the redundant measuring unit. The thermocouples are used from room temperature up to 1200°C. Above that temperature all thermocouples are automatically removed from the furnace for protection, and the system automatically switches to operation with optical pyrometers. These have a measuring range from 350°C to 2200°C. By using 4 independent optical pyrometers per furnace, the sample batch can be closely monitored and the temperature profile can be recorded. In combination with a Siemens controller S7-300 (SPS) it is possible to reliably program, monitor and control exacting thermal processes. The process data are visualized on the integrated PC monitor (WIN CC).

FEATURES

- Water-cooled container with useable volumes up to 200 l
- Metallic and graphite versions available, depending on required atmosphere, maximum temperature and cross contamination with sample
- High vacuum up to 5×10^{-6} mbar
- Hydrogen partial pressure between 10 and 1000 mbar possible
- Ultrapure process chamber
- Sample accessible from all sides

Application examples HTBL

Hardening, annealing, tempering, quenching, brazing, degassing, pyrolysis, siliconisation, carbonisation, rapid prototyping, sintering, debinding, synthesis, sublimation, drying

Synergies between research and industry

Together with the German Aerospace Centre "Deutsche Zentrum für Luft- und Raumfahrt (DLR)" in Stuttgart, Germany, CARBOLITE GERO has built two high temperature vacuum furnaces for industrial production of carbon/carbon-silicon/carbon components through pyrolysis and siliconisation for the Norwegian customer Nammo Raufoss AS.

The furnaces are used for running high temperature processes in the production of thermally and mechanically heavily strained functional components made of fibre-reinforced ceramics.

MATERIAL

The finished C/C-SiC material consists of load-bearing C/C bundles embedded in a SiC matrix. Typical characteristics of C/C-SiC materials are:

- High temperature and thermal shock resistance
- Damage-tolerant (not brittle)
- High mechanical strength at high temperatures
- Low density (1.9 g/cm³)
- Very high abrasion and corrosion resistance
- Very low thermal expansion
- Inner protection against oxidation

HTBL 50 GR

METHOD

1. After the carbon fibres are mixed with (mostly phenolic) resin, the components are pressed into a rough form and are polymerized at 190°C.

2. The following step involves pyrolizing/carbonizing the components in a protective gas or vacuum furnace. By applying heat under nitrogen the exhaust gases from pyrolysis are removed from the component and are completely combusted in an exhaust gas burner. The separate heating from the gas supply to the burner helps to avoid undesired accumulations. The atmosphere is changed from nitrogen to vacuum at approximately 900°C and carbonization is carried out up to a maximum temperature of 1650°C.

3. After cooling down, the open-porous C/C (carbon/carbon) components produced by pyrolysis are removed from the furnace for quality control.

4. The C/C components are then introduced into a siliconization furnace and stacked in crucibles on the base plate. In addition to the component the required amount of granulated silicon for the siliconization process is also weighed in the crucible. A retort is mounted above the crucible to protect the furnace from the silicon vapor which is produced at working temperature. The actual siliconization process takes place at temperatures above the melting point of silicon (approx. 1650°C). The process is executed under vacuum; the liquid silicon infiltrates the porous C/C component exclusively through capillary forces. Thus the component is fully saturated with liquid silicon which reacts to SiC with a small part of the carbon at the inside contact surfaces.

DISTINCTIVE FEATURES

The special bottom loader design with additional floor heating ensures optimum temperature distribution inside the furnaces. By using a graphite retort the gas supply can be adjusted in a way to avoid contaminations to a large extent. Even the gas outlets do not require cleaning. All pyrolysis products are combusted. Thanks to the fully automatic process operational activities are limited to loading and unloading the furnace.