

Special Electrically Resistant Heated Furnaces

Drying of Ceramic Components

Autor: Roland Waitz, Malte Möller

Many ceramic masses are fabricated in plastic or liquid condition by addition of water. In a drying process this water has to be removed from the ceramic part after shaping. This is often realized by convection (circulation air). The circulating air transports the heat to the material to be dried and at the same time the evaporated humidity, which evaporates from the material transported away.

A continuous airflow and temperature control has to be observed when designing of a circulation air dryer. If not there is the risk of damage and destruction especially with highly variable wall thickness of the ceramic material (drying is effected in common convective dryers from outside to the inside).

There are many design possibilities. The easiest one is a circulation air dryer, which has a detailed controller technology and can be operated with mixed air to decrease the air humidity in a controlled fashion.

Also continuous solutions are possible – as continuous belt furnace or space saving paternoster furnace (picture 1 paternoster furnace)

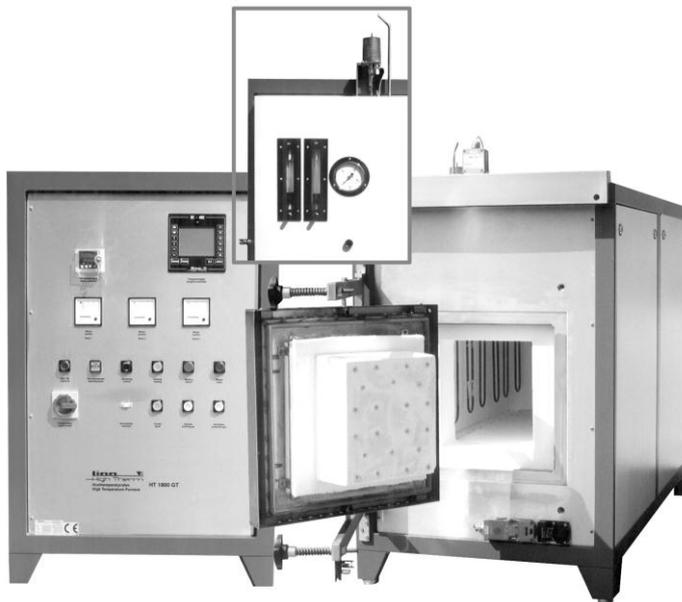


picture 1: paternoster elevator furnace 500 °C (picture Linn High Therm)

Calcination of Ceramic Powders and Granulates

The increasing requirements on ceramics for technical, electronic or medical applications has decreased the demands on the purity, homogeneity, and surface quality of the green powder. Optimal features are only achieved by homogenous mixture treatment. This applies for the temperature time curve as well as for the furnace atmosphere.

With the easiest method, the raw materials are filled in ceramic and metallic cases or crucibles and are treated in a batch-furnace. However this process has serious disadvantages:
high energy consumption, long cycle times
crushing of sinter cake
variation of powder features



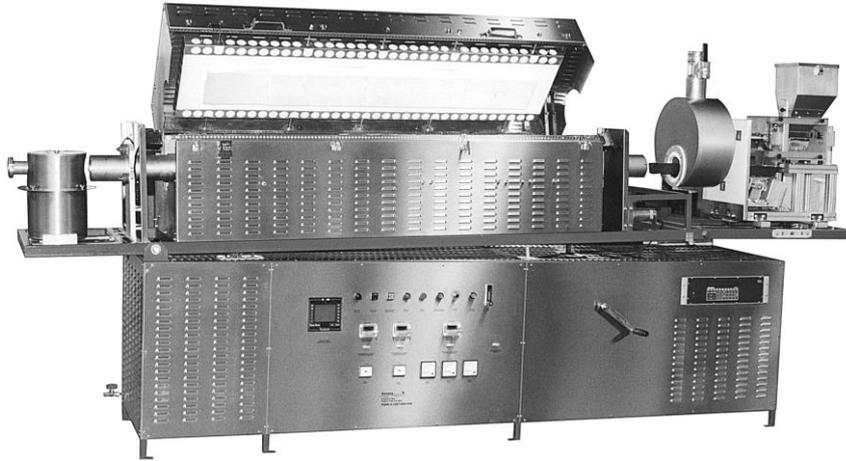
picture 2: protective gas tight high temperature chamber furnace 52 liter / 1820 °C (picture Linn High Therm)

The solution is a furnace with continuous material transport without kiln furniture storage in which the product is mixed at the same time, so that the exposure to temperature and atmosphere is homogenous in the compound: The rotary tube furnace.

When using gas heated rotary tube furnaces the tube is heated from inside contrary to the electrically heated furnaces. Therefore the tube itself serves as heat insulation, it remains relatively cool and is freely accessible from all sides. The selection of material and the possibilities for mechanical bearing, drive etc. is nearly not limited.

Gas heated furnaces, however, require high infra structure input and are not easily controllable regarding the purity of gas atmosphere and gas flow.

Electrically heated rotary tube furnace are the better choice for testing purposes and small production ranges,. The limits are at app. $D_{\max} = 500$ mm and $L_{\text{heated}} = 5$ m, with ceramic plasma sprayed tubes. The maximum sizes could be higher with metallic tubes be at temperatures < 600 °C. (picture 3 high temperature rotary tube furnace)



picture 3 high temperature rotary tube furnace 1600 °C (picture Linn High Therm)

In microwaves and microwave hybrid rotary tube furnaces quartz glass tubes or ceramic tubes are used. If it necessary to use a metallic tube, the microwave can only be applied from the front side of the tube. This application is limited to small on medium power due to the high power density in the reaction space. Microwave hybrid rotary tube furnaces are additionally heated conventional, normally with gas or resistance heating.

Tube

Depending on temperature and sintering material several types of tubes can be used. Possible wear and rear as well as contamination of the sintering material and reaction must be considered.

An additional wiper device has to be installed for powders to avoid “caking”.

Metal Tube

Materials used include 1.4841 for temperatures up to 1050 °C (embrittlement tendency for an exclusive operation at 700-850 °C), 1.4828 for temperatures up to 950 °C, Inconel up to 1150 °C and recently APM[®] (FeCrAl) up to 1300 °C. Metal tubes are mechanically sturdy, allow relatively high heating up and cooling down rates, the components e.g. conveyer and wiper are relatively easy to realize. But the low degree can also effect contamination by abrasion, as the metallic high temperature material contains many and also critical alloy components e.g. Ni.

Quartz glass/fused silica T_{max} for quartz is 1050 °C. The tubes could also be used at higher temperatures. When cooling down below 700 °C, recrystallization, which may lead to destruction of the insert tube. The number of cycles and lifetime is difficult to forecast since they depend extremely on the external parameters like e.g. the air humidity. A wide variety of highly pure quartz glass, the preferred material of the semi-conductor industry, is available on the market. The contamination of the sintering material is almost impossible. Furthermore quartz has an extremely good thermal shock behavior.

Ceramic

Ceramic tubes are highly resistant to wear and tear, the included substances are less critical and they can be operated at high temperatures up to 1750 °C. Large dimensions are realizable with expensive plasma sprayed. Mostly used is Al₂O₃ material according to temperature with increasing Al₂O₃-content between 60-99.7 %. In special cases it is also possible to use

SiC-Tubes. The excellent thermal conductivity of SiC-tubes is responsible for the heat transfer in connection with throughput and allows higher heating up rates. The max. heating up rate for dense sintered qualities of aluminum oxide is 120-360 K/h up to 1200 °C, above it is 180-360 K/h.

Heaters: For common types of construction the heaters are exposed to ambient, therefore common materials in furnace industry are used. FeCrAl (APM[®]) up to 1400 °C, MoSi₂ up to 1850 °C heating conductor temperature. The maximum temperature in the tube should be appr. ca. 50-100 °C lower.

The heating has usually several zones as there are several reactions in the rotary tube. The heating-up rate with drying of powder with relative high energy consumption, one zone, in which could be exothermic or endothermic reactions and a holding and cooling down phase, in which the heating loss should be balanced or even cooled. For good temperature uniformity 3 zones, small aggregates are usually sufficient.

Design: The usual filling factor for rotary tube furnaces is app. 10 % of the tube volume. According to type and shape of the sintering material (powder, granulate) the filling factor can extremely differ.

Charging: The dosage and charging is done by vibration machine or a screw conveyer. Vibration machines mostly require besides a frequency control, additional flexible mechanical inserts to adjust capacity.

Transport and drive: a frequency controlled three-phase motor effects drive. The transit time is continuously adjustable by rotation speed (1-10 min⁻¹) and angle of incidence (1-20 °)

Debinding of Ceramic Components

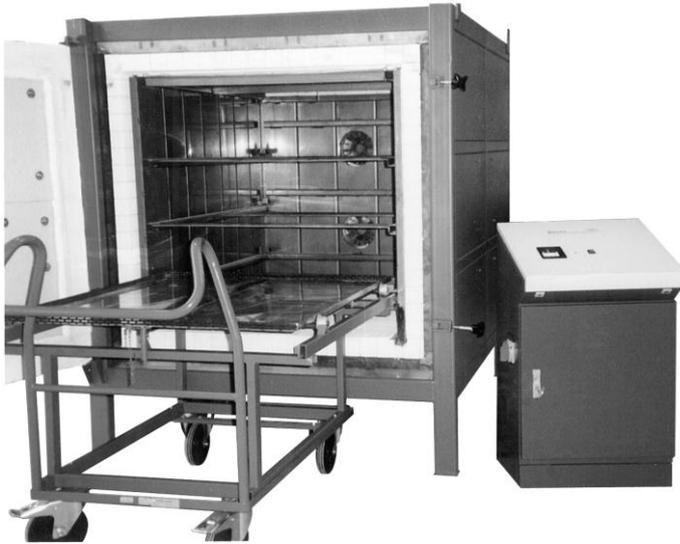
Besides the classic process for small, complex shaped ceramic parts the CIM (Ceramic Injection Moulding) Technology has been established in the last years for mass production. A reason for this was the drastic decrease of debinding times by new organic sintering systems. 3 technologies can be distinguished; thermal, solution and catalytic debinding. With all three mentioned processes approx. 0,5 – 3 % of the binder usually remains in the product to ensure the mechanical stability until sintering.

Thermal Debinding

To organic binder material is added for plastification. The organic has to be removed before sintering. This happens normally in a temperature range between 200 °C and 700 °C.

The oxide ceramics are debinded under normal atmosphere in simple furnaces. But important is a good sealing of the furnaces chamber and the possibility for active exhaust and to introduce in preheated air on demand.

An air circulation assures homogenous temperatures and fast removal of reaction products. (picture 4 air circulation chamber furnace)



picture 4 air circulation chamber furnace 0,6 m³ / 550 °C (picture Linn High Therm)

For non-oxide is a protective gas atmosphere; mostly Nitrogen, Argon or also forming gas with little hydrogen component is necessary. As protective gas furnace a retort furnace is used. As mostly high molecular exhaust gases develop during debinding without oxygen, which condensate again at relatively high temperatures, a cooling trap has to be provided directly on the gas output of the furnace. In the industry a direct afterburning is preferred due to the high disposal costs.

A gas circulation is absolutely necessary due to the condensating exhaust gases. This assures also the necessary temperature conformity in the lower temperature range up to 500 °C. For higher temperatures, e.g. if the debinding is combined with a pre-sintering a multiple zone control is recommended for the furnace due to the main heating transport by radiation.
(picture 5 protective gas tight furnace)



picture 5: protective gas tight chamber furnace 480 litre / 1100 °C (picture Linn High Therm)

Catalytic debinding:

For this technology working temperatures around 120 °C and gas circulation it is possible to debinder also relative big parts in an acceptable period of time. Beside the dosing pump for the catalyzer (smoking nitric acid) is also a gas heated afterburning necessary, which can only be done in closed rooms with smaller units. Large furnaces operate with burning-off devices on the roof, which require a permission.

Sintering of Ceramic Components

Sintering is a process in the ceramic production, in which a conversion from porous green piece with low mechanical stability a dense piece with higher mechanical stability is effected. The sintering is mostly at app. 70-80 % of the melting temperature of the materials.

For sintering following furnace types are suitable:

- chamber furnaces for universal application
- roller kilns for continuous process
- shuttle kilns for easy charging and discharging
- bell type furnaces for difficult superstructures (the charged bottom is stationary).
- elevator furnaces for even temperature distribution by four-sided heating and an economical charging as a changing bottom can be charged parallel to the furnace cycle. As the cooling down of batch in the furnace is effected very slowly in the lower temperature range, but for many ceramics the cooling speed below 500 °C is not critical, a elevator furnace can be discharged comfortably and without danger at 500 °C. So a high charge frequency can be achieved.

An even temperature distribution (± 5 K and better) can be achieved in these furnaces types by multiple zone control.

For sintering at nitrides, carbides and also sometime oxide ceramics the above mentioned furnace types are realized as protective gas and discontinuous vacuum furnaces up to 10^{-5} mbar. Furthermore special materials ceramic like Piezo, capacitor, Bio or transparent ceramic, require special furnace atmospheres, e.g. hydrogen, Nitrogen etc.. Modified model with furnace chambers for overpressure up to 200 bar and temperatures up to over 2000 °C are required for special applications. (picture 6 high pressure chamber furnace)



picture 6: High pressure chamber furnace 25 bar / 1800 °C (picture Linn High Therm)