Resistant heated rotary furnaces for heat treatment of rare earth minerals and quartz sand

by **Peter Wübben**

The rotary tube furnace combines, in most cases, today's expectations of a modern continuous heat treatment furnace - high throughput, energy efficiency, easy automation and a reliable reproducibility.
Nowadays a number of applications can be realized by the use of high performance materials for the rotary tube. The various operational possibilities of rotary tube furnaces were recently extended by applications for the production of rare earth minerals and high purity quartz sand.

The increasing demand for raw materials for the production of glass, quartz and ceramics for electronic or medical purposes leads to higher requirements with regard to purity, homogeneity and surface properties of the initial powders. Caused by more stricter environment constraints resp. high disposal costs it becomes more interesting to recycle waste materials (i.e. from steel works) internally which requires an intermediate processing. By a thermal treatment organic and inorganic impurities can be decomposed or directly vaporized. For all applications is valid: optimum homogeneous properties will be reached only if every particle of a powder or granulate has received the same treatment. This applies to a temperature-time-curve as well as to the surrounding atmosphere i.e. the oxygen content during calcination.

The simplest method for thermal treatment is the filling of the initial powder into ceramic or metallic boxes resp. crucible and the processing in a batch furnace. However this procedure has a major disadvantages: The weight resp. thermal mass of the containers usually is higher than the product. Due to this a high energy consumption and long cycling time resulting from heating up and cooling down is pre-programmed.

At bulk materials the diffusion of reaction gas is strongly hindered - sometimes amplified by sinter crusts - which limits the filling height to a few centimeters and results in long holding times. The additional effort for loading and unloading reduces the productivity considerably. Furthermore it cannot be excluded that powder properties will vary from the bottom to the surface of the fill. Theoretically this problem can be minimized by cyclic vacuum pumping. But this will increase the effort and consequently the investment and operating costs of a furnace plant. In the case of sensitive products there will be the danger of evaporation or decomposition. Problems also can arise from the contamination of the product by the material of the transport container.

In spite of the above mentioned difficulties a major part of the powder will react at the same time and following disposal systems (i.e. gas scrubber or thermal post combustion) have to be designed for high and very small flow rates. The solution for all these problems is a furnace with a continuous transport system without containers where all particles will be equally mixed to have an exposition against temperature and atmosphere which in average is homogenous: a rotary furnace.

Rotary furnaces can directly or indirectly heated. In the case of direct heating usually the rotating tube will be heated directly internally by a flame from a burner. The temperature impact on the rotating tube can be minimized at an adequate thermal insulation which is advantageous. The thermal insulation can be designed for an outer low temperature. This gives many choices for the material selection of the rotary tube and the abilities for the mechanical bearing, drive etc. are nearly not limited. This enables the manufacturing of extremely large furnace dimensions as used in the concrete industry. A disadvantage of direct heating is the mixing of the flue

Fig. 1:
Rotary furnace with vibrating conveyor and thermal post combustion, up to 1,500 °C

gases from the combustion with process gases released by the heat treatment. In the worst case the flue gas reacts with the product. In the simplest case this leads to bigger diameters of exhaust/process gas ducts.

Contrary to the direct heating the indirect heating is effected by heating the rotating tube from the outside. The heat transfer to the product occurs by the heated wall of the rotating tube. The heating is made electrically or by burners. In opposite to electric heating, gas/oil fired rotary furnaces require a more complex infrastructure and higher investment cost. For use in laboratories or operation as pilot plant with small throughput the gas/oil heating has a lower limit which results from the necessary low heating power. Electrical heated rotary furnaces can be controlled even at low heating power very accurately regarding the temperature and require at permanent operation a few spare parts only.

For tests, technical centers and small production furnaces - the throughput (approx. 1 to 300 dm^3/hour) will be given by the holding time and specific filling degree of the tube - electrical heated rotary furnaces are the best choice. The use of ceramic, plasma sprayed rotary tubes is limited to a diameter of Dmax= 600 mm and to a length Lheated = 5 mtrs. At metallic tubes this dimensions can be higher at temperatures less than 600 °C. **Fig. 1** shows an electric heated rotary furnace for laboratories with a ceramic tube as used at the heat treatment of high purity quartz sand.

MOTION OF PRODUCTS WITH A WATER-LIKE FLOW BEHAVIOUR IN ROTARY KILNS

The motion of the product in the kiln tube is the decisive factor in the transfer of heat from the heated wall of the kiln tube to the product. Seven different flow patterns are differentiated in the motion of bulk products with water-like flow behaviour (**Fig. 2**).

1. Pure slip: The friction between tube wall and bulk material is so low that the good stands in the tube with a time constant angle without any movement and mixture.
2. Oscillation: Caused by the somewhat higher number of turns there is a continuous change between static friction and slide friction on the tube wall. A mixing does not happen. For this reason the first two modes have no practical meaning.
3. Periodic fall: At the change-over from phase 2 to 3 a mixing of the bulk material can be observed at the surface and boundary faces. The surface of the good is designated by two surfaces with an obtuse angle between.
4. Unroll: This angle increases by increasing number of turns. Finally one single layer is created with a material transport at the surface which is continued at the underside.
5. Over-slope: The top edge of the bulk material heightens with increasing number of turns and is rounded continuously by the down slide. The mixing becomes more intensive.
6. Wave rollover: Across the total cross sectional area a big rollover is taking place which is able to grind down the bulk material. This kind of transport happens quit often if fixtures mounted in the tube.
7. Centrifugation: Only at sufficient static friction between tube wall and bulk material the typical centrifugation process can be observed at which the total good sticks

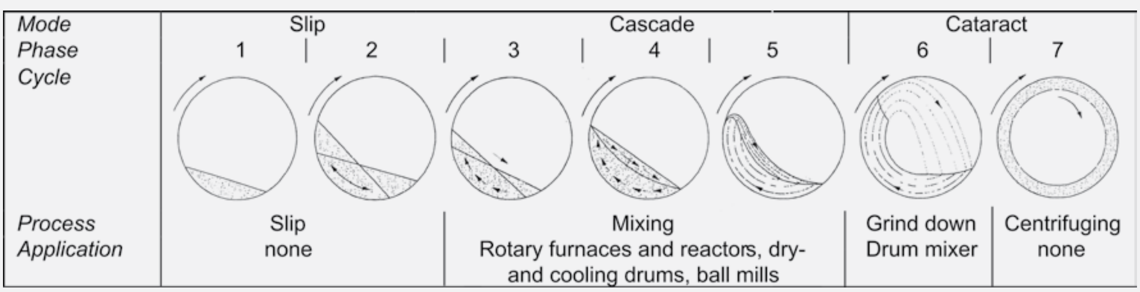

Fig. 2:
Types of flow behaviour in rotary furnace

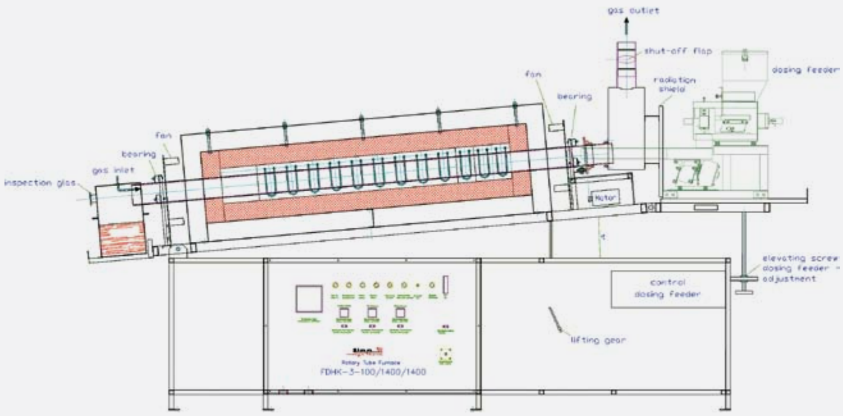

Fig. 3: Layout of high temperature rotary furnace

Fig. 4: Gas and vacuum sealed microwave rotary furnace for CVD process

on the tube wall. This unwanted condition can be converted into condition 6 by using a scraper. This scraper (ceramic or metal tube) can serve at the same time as a protection tube for thermocouples.

FRAME AND MECHANICAL CONSTRUCTION

Charging

The dosage and feed of a rotary tube is realized by vibration conveyors, screw conveyors or belt conveyors. Agitators require beside a frequency control in most cases additional flexible mechanical fixtures for the adjustment of accurate flow rates. At screw conveyors increased abrasion can lead to problems. For continuous charging at protection gas atmosphere double flap sluices are used.

Transport and drive

The rotary drive usually happens via a frequency controlled alternating current motor. The residence time of the product in the tube can steplessly adjusted by the number of turns and the slope of the rotary tube. The number of turns mostly is in the range 1 to 10 min-1.

The furnace itself is built on a tilting frame. The tilting angle at small furnaces can be adjusted manually with a crank handle in a range 0 to 10° (**Fig. 3**). At bigger furnaces in most cases a hydraulic lifting system is used which allows a slope of 0 to 5°. A furnace housing made of stainless steel is advisable because of furnace use in an often chemical polluted environment. The rotating tube should have heads at both ends at operation with normal air too. Particularly at high temperature - even at a small tilting angle - strong natural draught can occur. This air flow is noticeable at a strong radial temperature gradient in the tube. At a temperature control with a thermocouple inside the tube an overheating of the tube wall can occur, which will lead to caking of the bulk material at the tube wall. At a temperature control with a thermocouple between furnace insulation and outer tube wall the normal working temperature in the tube will not be reached. Dependent on tube geometry, furnace slope and temperature the radial temperature gradient can be more than 100 °C at missing end heads.

It is important, that the rotary tube easily can be changed without damaging the furnace insulation heating elements. At smaller rotary tubes it is helpful to insert a long light weighted plastic tube into the rotary tube and use it as a carrier. At bigger rotary tubes the furnace housing must be separable resp. must have a lid to insert the rotary tube from top.

Cooling zone

At small plants an active cooling at the discharge end mostly is required for the protection of the tube bearing, because the product transports only small quantities of heat to the outside. A cooling with air blowers usually is sufficient. Higher cooling rates can be achieved with water cooling and circulating air. At high product discharge rates the rotary tube can be extended and externally behind the furnace housing directly cooled with water sprinkling from nozzles on the tube. The cooling zone length depends on product flow rate, particle size and required product exit temperature.

Product discharge

At the discharge end of the rotary tube resp. cooling zone the product can be collected in a vessel (at small systems water cooled) or transported continuously to following plants.

At Microwave- and Microwave-hybrid rotary furnaces (**Fig. 4**) quartz glass or ceramic tubes will be used. If a metal tube has to be used the Microwave feed has to done from the front sides of the tube. At Microwave-

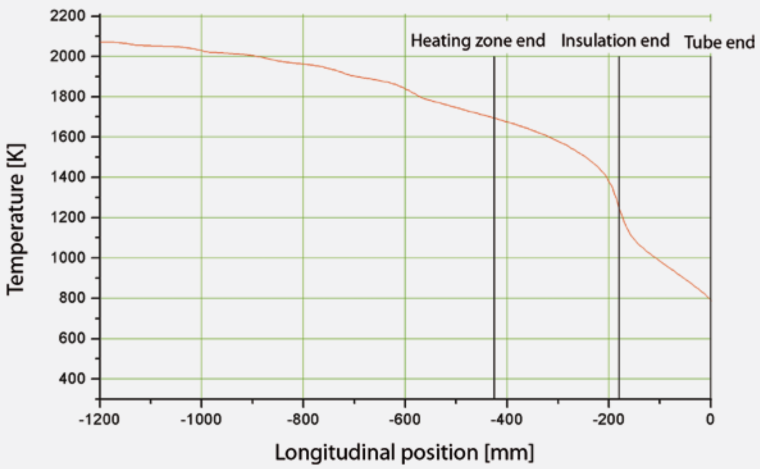

Fig. 5: Temperature course at the end of rotary tube

hybrid rotary furnaces additionally a conventionally heat source is installed which usually is a resistant heating.

Rotary tube

Dependent on temperature and processed material different types of rotary tubes can be used. Possible abrasion, pollution of the treated good and reactions (chemical or "backing" to the tube walls) have to be considered. This problem occurs more often at fine powders which is why a pre-granulation for critical substances may be necessary. In the case of powders which tend to stick it might be useful to mount a chain or a scraper.

Metallic rotary tube materials

Common materials are 1.4841 (AISI 314) up to 1,050 °C, 1.4828 (AISI 309) up to 950 °C, 2.4851 (Inconel 601or alloy 601) up to 1,150 °C and newly APM (FeCrAl) up to 1300 °C dependent on application and chemical impact. Metal tubes are mechanical tough, allow high heating up and cooling down rates and internal fixtures are relatively simple to realise. The low hardness can lead however to pollution of the processed material caused by abrasion and can contain critical alloy components i.e. Ni, Cr.

Special alloys are used at the heat treatment of acid containing products as rare earths minerals. For this application the material 1.4562 (N08031) is well-proven up for temperatures up to 700 °C.

Quartz glass/Fused Silica rotary tubes

The maximal application temperature for a frequent continuous operation with rotary tubes made of quartz is 1,050 °C. The tubes can be used even at higher temperatures of up to approx. 1,450 °C. At temperatures above 1,050 °C the formation of Cristobalite (polymorph of silica) takes place and leads at cooling down below 600 °C to the destruction of the rotary tube. A prediction regarding the allowed number of cycles or life time of a quartz tube is difficult to make because of a strong dependency on many factors i.e. atmosphere, air humidity, wall thickness etc. Quartz glass as preferred material of the semiconductor industry is available in high purity quality. A contamination is excluded if the quartz itself is not degraded. Furthermore the thermal shock behaviour of quartz is extremely good.

Ceramic rotary tubes

Ceramic tubes have a high abrasion resistance. Tube materials are mostly uncritical. High application temperatures up to 1,700 °C are possible with ceramic tubes. The maximal tube dimensions are very limited with slurry casted clay. Plasma sprayed tube allow dimensions up to 600 mm diameter. Compared to metal tube the price of plasma sprayed tubes is very high which often leads to multiple furnaces with smaller diameter tubes.

The most commonly used material is Al_2O_3. Dependent on temperature the Al_2O_3 content is between 60 to 99.7 wt.-%. For special application, i.e. heat treatment of high purity quartz sand, rotary tubes made of high purity Al_2O_3 will be used. Even for these materials many different qualities are available due to different impurities which result from different production methods.

In special cases the use of SiC tubes is useful. The high thermal conductivity enables a good heat transfer at high product flow rates und allows high heating-up rates. For alumina-oxide tube the maximal heating-up rate at dense sintered qualities is 120 to 360 K/hr up 1,200 °C; above this temperature 180 to 360 K/hr. Porous qualities allow up to 400 K/hr. The high sensibility against local temperature gradients has to be considered at the furnace construction and feed of high amounts of cold product. At bigger rotary tubes it is recommended to secure measures for minimising the temperature distribution at the tube end (**Fig. 5**) by a model calculation (**Fig. 6**):

Heating

At the common structural forms of rotary furnaces the heating elements mostly are exposed to the ambient air. For this reason materials are used which are known from normal furnaces. FeCrAl (APM®) up to 1,400 °C, $MOSi_2$ up to 1850 °C heating element temperature. The maximal temperature in the rotary tube is about 50 to 100 °C lower.

The heating mostly has multiple zones because in the rotary tube different reactions can take place:

- The heating up phase with drying of the good which requires a high energy input

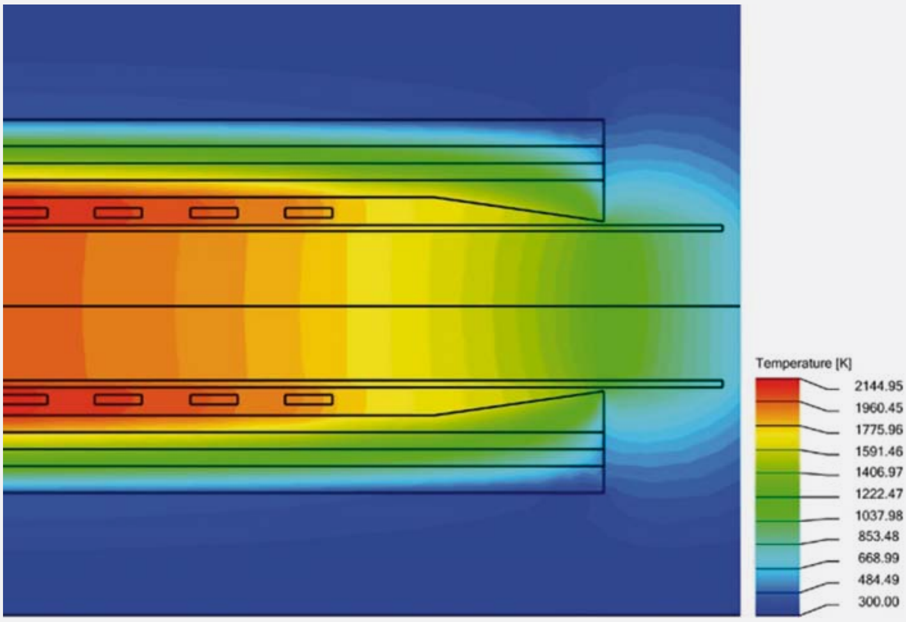

Fig. 6: Model calculation

- A zone with exothermic or endothermic reactions which needs energy input or cooling
- A holding zone for sintering processes or other reactions which need time where heating losses have to balanced.

Three zones are usually sufficient for smaller plants. Thus a high temperature uniformity can be achieved. For bigger furnaces or if accurate temperature profiles are required the number of heating zones have to be increased accordingly. For economical and technical reasons different heating element types may be necessary in the different zones; i.e. adverse operating conditions are in the temperature range 600 to 900 °C for high temperature elements made of $MOSi_2$. For these elements the later tilting angle of the furnace has to be taken into account because these elements can operate at temperatures above 1,300 °C only in a vertical ± 7° installation position.

Design

The usual filling degree (amount of filling) of rotary furnace is approx. 10 % of rotary tube volume. Dependent on the kind and shape of the processed material (powder, grains, granulates etc.) the filling degree may deviate strongly. The filling degree can be increased by the installation of chicanes. Decisive for the required furnace size is the necessary residence time of the product in the tube at working temperature. Usually pre-tests are necessary to determine this time because compared to normal batch furnaces the heat transfer in a rotary furnace is much better and reaction times are much shorter. The reason for this is the better exposition of the bulk material to the furnace atmosphere and the shorter heating-up time caused by the improved heat transfer.

For design calculation different formulas are available which take into account the residence time behaviour in a rotary furnace, number of turns of the rotating tube and bulk material properties. These formulas can be used reasonably only if practical experience with different bulk materials is available. Particularly at bulk materials which change at higher temperatures drastically there flow properties (i.e. getting pasty) reliable values for the residence time can only be obtained by tests.

CONCLUSION

Rotary tube furnaces can be used at many continuous processes for the heat treatment of bulk material. They also find application in the development of new processes and materials. The design according to scientific formulas only is not possible and needs long-time practical experience of many years which has to be to underpinned from case to case by tests.

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