Fabrication of Kiln Furniture with Variable Geometry Especially for Technical Ceramics

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1. Summary

Subject is the production and the properties of Kiln furniture of variable geometry, mainly used as transport boxes - so called sintertrays - in the fields of technical ceramics.

By using a self flowing castable basing on high alumina raw materials, kiln furniture of various geometries may be casted. In the present example, small boxes with dimensions of 50mm are produced, including ventilation drain at the sides and outlet drains in the bottom. The bonding of the castings is hydraulic, later - due to the sintering process- it is ceramic bonding. In the present example, the raw material is Al_2O_3 with a grain size of up to 1mm. Other raw materials at almost any grain size are possible.

2. Basics

The demand on kiln furniture used for sintering in technical ceramics are various. In former times, the sense of kiln furniture was to stack and to protect the sintered parts in the kiln. Massive, mostly clay bonded SiC- plates were used. The thickness of the plates was selected high enough to assure a safe firing of the sintered parts. The utilization of clay-bonded SiC was preferred due to its good cold- and hot strength at low costs. To avoid any pollution by black SiC particles to the mostly light colored sintered parts, the plates were coated by a slurry. Because of this treatment, the ratio of sintered parts to kiln furniture was 1:8, with concluding high energy consumption.

Fig. 1: Filled Sintertrays of different geometries

The use of these kiln furniture for the production of technical ceramics - especially for small parts - is of disadvantage caused by cost pressure at high quality demands. The need for light weighted kiln furniture at simultaneously high strength and high temperature stability is large. Further demands aim on customized geometries for optimum filling of the burning chamber to protect the sintered parts from deformation.

A modern forming method for technical ceramics is the ceramic injection molding (CIM), which enables to produce parts with wall thicknesses of 0,5-30mm in large numbers. The so produced parts comprise another challenge for the used kiln furniture, because after they are formed, they pass the process of binder removal. At the end of this process, their strength is quite low. In order to provide the best general framework it would be ideal, if the injection molded parts could be transferred immediately into their kiln furniture, which they only leave after the firing process.

The so called sintertrays offer this possibility: these box-shaped kiln furniture were produced specially for injection molded pieces and may be adjusted to their geometries and demands. Here, the focus is mainly on high dimensional stability, optimized Design, low mass and good handling regarding charging with the injection molded pieces and placement in the kiln.

Fig. 2: Casted body and form

All these demands show that a simple rack with plates will not be sufficient. The production and the use of specially designed kiln furniture can only be profitable, if the production process is adjusted to these demands: pressing as forming method has the advantage to be able to produce a high number of pieces within shortest time. The high number of pieces as necessary to cover the high costs of the forms, but is not required by the market. So, this forming process cannot be regarded as cost-efficient. The production by casting in plastic forms offers advantages in this case: the costs of the form itself are low, demands on changing geometries can be fulfilled fast.

In the following, the development and production of such customized kiln furniture are described.

3. Production of Sintertrays

3.1 Raw Materials

The production of sintertrays needs high refractory and quality controlled raw materials. The initial control of the raw materials includes humidity, loss of ignition, a chemical analysis, a grain size distribution by laser-granulometer and determination of the free specific surface area according to BET. The measurement of the free specific surface area is exactly reproducible and it correlates directly with the sintering activity of the powder.

Because the sintertrays are casted, the composition of the recipe needs to be adjusted to the demands of a refractory castable. The grain size distribution is adjusted to enable a self flowing processing of the mass. This self flowing property is achieved by a maximum of fine grains < 45 um. The grain size distribution of larger grain sizes has less importance on the self flowing properties of the mass, but has influence on porosity, mechanical properties and thermal resistance of the sintered material.

The chemical composition of the batch consists of more than 99% of Al_2O_3. The bonding of the casted bodies is hydraulic, which is transferred into ceramic bonding by sintering. The castable can therefor be considered as NCC (no cement castables).

3.2 Processing Technique

After weighting, the raw materials are mixed first in dry condition and then with liquid. The homogenization is achieved by standard mixing machines. After mixing the mass with the liquid, it stays processable for app. 20 minutes.

This quite short time is accepted, because binding should be enforced. During the development of this castable, the binding time could be reduced from initially 8 hours down to 2 hours. For this, the initial castable, which was a LCC (low cement castable) was modified to a NCC. Because CaO was avoided in the batch, the strength of the casted bodies decreased (from 2,4 MPa to 1,6 MPa after 12 hours; drying at 110°C) but the binding properties could be controlled much more. Drying-cracks could be reduced to almost zero. Another effect of the cement. containing bonding, the slow dehydration under occurrence of reversible CAH-phases and following conversion from CA and CA_2 to the stable CA_6 will be described in detail in the chapter “Drying, Firing, Processing”.

The amount of casting mass and its binding time is well adjusted to the number of forms. This means that with quite few number of forms many sintertrays can be produced due to short binding time.

Because the costs of the form itself are in the calculation of the entire piece, the low number of forms reduces the sintertrays endprice.

3.3 Forming

The used forms consist of PVC and were produced by CNC molding cutter. By the usage of PVC instead of e.g. steel or aluminum, material costs can be reduced considerably. In addition, metal abrasion may be excluded.

The forms were designed as multi part forms. The sintertray is casted on a solid bottom and will be enclosed by an exterior form. The bottom part is equipped with a centered notch, providing the sintertray with a small partition wall. This partition wall means a big challenge to the forming process, because during forming and firing it may not be deformed, and its tolerance is in small limits. During later use, the partition wall is needed to stack the injection molded pieces (ferrules) safely in all the tray.

3.4 Drying, Firing, Processing

The drying process consists of several steps. In the first step, the casted sintertray dry in their forms. This step takes two hours, after the casted parts may be removed after that time. Their strength in this stage is enough to handle the pieces safely. Investigations with test bars showed up a bending strength of 1,6 MPa.

After drying at 110°C for at least12 hours the sintertrays are ready for firing at 1600°C in a electric chamber kiln.

During the sintering process no new phases take place in the batch, allowing to operate with a linear firing curve. In former cement containing castables, dehydration and new phases had to be considered during the definition of the firing curve. The Heating process therefor took 50% more time. This effect is impressively shown in the following picture: above 1400°C the thermal expansion is reversed to a shrinkage and above 1550°C it is again reversed to thermal expansion.

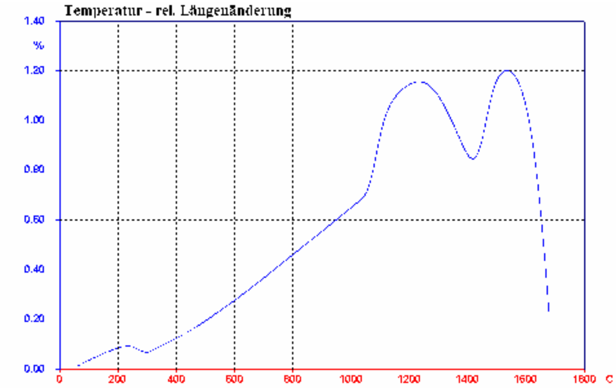

Fig. 3: Thermal Expansion of a cured LCC

These effects show good correlation to the phase transition from CA_2 to CA_6. These quite fast and strong changes between thermal expansion and shrinkage strongly stress the microstructure, cracks may conclude. Another problem is the intense shrinkage of the material above 1600°C in the area of the sintering temperature: a difference in temperature of 5K – which may easily appear also in an electric chamber kiln – has a considerable impact on the shrinkage of the pieces.

Regarding the high demands in allowable variation of the sintertray´s dimension, this behavior cannot be accepted.

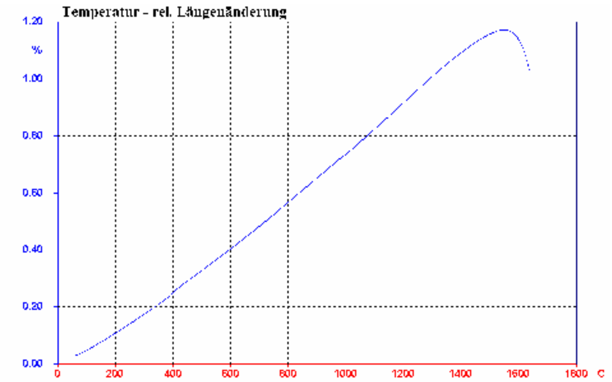

Fig. 4: Thermal expansion of a cured NCC

This figure shows the thermal expansion of the modified batch, which increases constantly until sintering process starts. These effects at the sintering temperature are quite acceptable.

The following grinding is made by diamond-trimmed wheels. Processing here mainly means, that the oversized height of the sintertrays is grinded to the final dimension. All other dimensions are fix due to the form and are kept during firing.

3.5 Quality Assurance

Quality assurance begins with the first analysis of the raw materials , as described in chapter 3.1. The firing of the pieces is controlled by PTCR (Process Temperature Control Rings). Other than Seger cone, the temperature gradient in the kiln and the effective energy can be assessed objectively. The rings shrink in a defined manner and will be measured with a slide gauge after the firing process. Each diameter is a direct indicator for a certain kiln temperature. This offers to set up a detailed temperature profile of the kiln with a variation of ± 1K.

The final control is layed out as 100% control, in which the sintertrays are measured at defined points both inside and outside.

4. Use

4.1 Ceramic Injection Molding

The injection molded pieces are ZrO_2 connection plugs for fiberglass, produced by high pressure ceramic injection molding (CIM), so called ferrules. Because ferrules are produced in millions of items, CIM is a profitable forming method. In addition, the demands on precise dimensions are high. To produce millions of items under these conditions, the production process needs to be mostly automated. This includes special customized kiln furnitures such as the described sintertrays.

The produced ferrules are automatically removed from the injection molding machine into a four-piece metal box. When this box is filled, all ferrules are charged into four sintertrays. After the injection molding the pieces have good mechanical strength due to their binder content of about 15%. This ensures safe handling. In the following processing steps, the binder is removed, which strongly decreases the strength of the pieces. It therefor is essential for the parts, to avoid any mechanical stress, even to avoid touching them.

4.2 Binder Removal (aqueous and thermal)

When placed into the sintertrays, the injection molded pieces are charged into a water bath and stored for 24 hours. The water dissolves about half of the binder content, so that the binder leaves a framework, which tightens the ceramic pieces.

The second step in binder removal is a thermal one. The aqueous debinded pieces are heated up to 300°C, the remaining binder is oxidized. After this step, the binder content is reduced down to less than 1%, the remaining binder is removed completely during the firing process.

4.3 Sintering

The sintering is done is electrically operated chamber-kilns in stacks of up to 20 layers of sintertrays. Other kiln furnitures than sintertrays are not usable.

Fig. 5: Chamber-kiln with sintertrays

5. Summarized Properties

5.1 Bending Strength of the dried parts

The bending strength of the dried parts was determined after 12 hours drying time at 110°C by 3-point-bending-test. A safe handling of the trays is provided with app. 1MPa , the measured value is 1,6 MPa.

5.2 Cold Bending Strength (CBS) and statical Modulus of Elasticity (MOE)

The CBS was determined with the 3-point-bending-test as well. The static E-Modulus was measured by the value of bending.

The measured values of 18,2 MPa are enough to ensure a safe transport of the die casted parts and to protect them during firing.

The static E-Modulus of 43,3 MPa is typical for alumina materials.

5.3 Porosity

The porosity is an important feature to predict the resistance of thermal fatigue. An extreme sold material has no possibility to reduce inner tension by present cavity. Porous materials can do that. The determination of porosity is done by immersion weighing, the values are at 25,8%, which allows the conclusion of a good resistance to thermal fatigue.

5.4 Dynamic Modulus of Elasticity

The dynamic modulus of elasticity also can give information on the resistance to thermal fatigue of a material: very solid, compact and dense materials have a high dynamic modulus of elasticity. It is reciprocal to the resistance to thermal fatigue. To measure the dynamic modulus of elasticity indestructible, the resonance method is a standard testing method. Measured values of 86 GPa are typical for alumina materials and indicate a considerable resistance to thermal fatigue.

5.5 Thermal Expansion

The importance of this testing has been described previously. In the following, the measured values are listed:

Thermal expansion:

-1000°C: 0,73 %
-1200°C: 0,91%
-1400°C: 1,10%

Thermal expansion coefficient 20-1000°C: 7,3 $*10^{-6}$ K^{-1}

5.6 Resistance to Thermal Fatigue

According to the following empiric formula, it is possible to predict the resistance to thermal fatigue:

$$TWB \sim \frac{\sigma \cdot \lambda}{E \cdot \alpha}$$

The coefficient should be high to indicate a good resistance to thermal fatigue. The experimental evidence can be done by shock testing with water. After 10 shocks with water, the material did not show up any kind of cracks, so that it can be regarded as resistant towards thermal fatigue.

6. Postscript

All developments and investigations described previously have been done at Werkstoffzentrum Rheinbach GmbH, Rheinbach.