Simulator System for Development and Optimization of Thermal Processes

Introduction

Does not matter how the ceramic products are formed or shaped all of them must be submitted to some kind of thermal processing in order to obtain the properties required by certain application. This phase will make or break the final product and for this reason it is the most important stage in the manufacturing process. In order to develop an optimum process the scientist or engineer must know how the body behaves during heating, being drying or firing. During heating the ceramic product undergoes physical and chemical transformations that effects the way how the product is dried or fired.

The physical and chemical transformations have as outcome dimensional changes (expansion or shrinkage), release or absorption of energy, decomposition of organic and inorganic compounds and a multitude of chemical reaction. One can not develop optimum and cost effective firing process if this complex process is not understood.

Dilatometwer and STA apparatuses are standard methods used to decipher what occurs in the ceramic body during heating or cooling. Changes in the dimensions are investigated

dilatometers, DTA(differential thermal analysis) apparatuses to quantify the energy released (exothermic) or absorbed (endothermic) due to different transformations either physical or chemical, TGA (thermal gravimetric analysis) apparatuses to define the weight loss or gain during heating or cooling.

The data obtained helped the engineers to understand the phenomena related to thermal processing of the ceramic composition. It was an important advancement in the development of optimized thermal processes.

Anyhow the above studies were done on small samples, milligrams or centimeters taken from ceramic products. The data obtained were useful but did not reveal what happened in the full size product, did not correlate all the phenomena with heat and mass transfer and thus provided only limited information, fact that lengthens the development process.

Simulator System

CTB built a complex sophisticated research kiln that overcomes these shortcomings and offers scientists and engineers at tool for in depths studies of ceramic bodies during heat treatment from room temperature to 1600°C and if required even higher.

Main features of the equipment are:

- the studies can be done at full size prototypes up to 30 kg and with dimensions 500·500·500mm^3
- heating/cooling rates of 10 K/min can be simulated
- during heat treatment diverse atmospheres can be simulated and controlled (oxidizing, reducing , special gases, N_2, inert or generated by fosil fuel combustion)
- the high renewal of the chamber atmosphere allows to study the effect of flow on ceramic physical and chemical
- continuous dimensional or weight (loss or gain) changes are measured with very high accuracy. Dimensional changes are measured on four locations on the product. The equipment has 12 thermocouples, which can be inserted in the product in order to define the exothermic or endothermic reactions and correlate them with dimensional or weight changes.
- real time display of all variables measured (temperatures, composition of atmosphere during heating and cooling, exchange rates per hour, pressure, equipment parameters etc.)
- easy configuration of the conditions of the thermal process in a recipe excel spread sheet
- a program that can perform heating according to the desired weight loss, dimensional changes and heat transfer in the product is available
- data can be easily retrieved at the end of the experiments
- safe operation.

Experimentals

Following examples explain how the simulator can be used to study the behavior of the ceramic composition during heat treatment and obtain useful information for process development.

Dimensional changes

The ceramic product shrinks or expands during heating or cooling fact that generates stresses, which can cause cracks or deformation in the final product. Knowing the temperatures ranges related to dimensional changes the engineer will devise heating or cooling rates to minimize them and thus produce a crack free product with required dimensions and shape. The CTB equipment can measure the dimensional changes of the ceramic product on four locations and correlate to the temperature of the product mea-

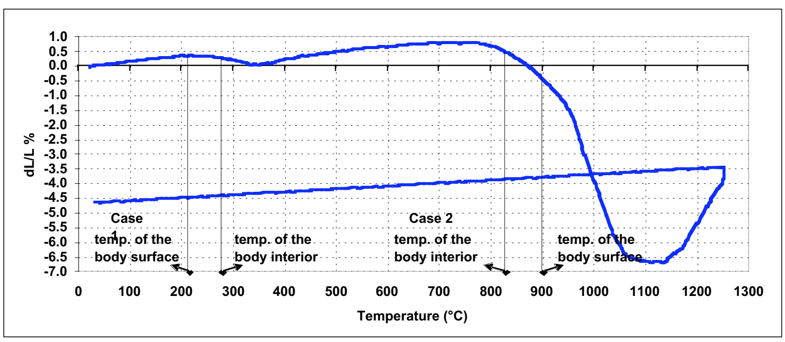

Fig. 1 Dimensional changes of green body during heat treatment

sured by 12 thermocouples. Some thermocouples can be inserted very close to the location where the dimensional changes are measured and thus there is a more precise correlation of temperature and dimensional changes. The data are recorded per location and can be displayed separately or combined.

In Fig. 1 are presented two cases that can occur during heating of the ceramic body.

Case 1 : The temperature of the interior in some ceramic products that contain organic additives can become higher than the temperature of the surface of the ceramic product due to exothermic reaction generated by the burn out of the organic component in relatively high O2 in the kiln atmosphere. The heat generated inside of the product by the combustion of the organic component is not easily transferred to the surface and thus the temperature inside the product will exceed the temperature of the surface. In this case the inside of the body shrinks more then the outside and due to the resistance of the outside to the shrinkage the inside is placed on tension fact that can produce cracks.

*Case 2:*The temperature of the surface of the ceramic body is higher than the temperature of the interior of the ceramic body as during an endothermic reaction generated by the removal of chemical bound water, decomposition of different compounds, etc. The required heat for the reaction is not very easy transferred from the surface to the interior of the product and thus the temperature inside the product falls behind. In this case the surface of the product shrinks more then the inside and due to the resistance of the inside the surface is placed on tension fact that can produce cracks.The engineer can solve these problems by reducing the piece thermal gradients in these regions and thus reducing the thermal stresses in the ceramic body.

Weight Losses

During heat treatment the ceramic body changes the weight. The Fig. 2 exemplifies one of the cases that can be studied with the CTB equipment. In the temperature region 200…300°C and 800…900°C the ceramic body undergoes rapid loss of weight due to different reactions as removal of organic components, decomposition of inorganic constituents, removal of chemical bound water, burnout of pore formers, etc. If these reactions are not controlled and the evolution of gases are to rapid, they can produce defects in the body. Based on the above information the engineer can control the reaction through heating rates or firing environment. This function can automatically be done with the CTB priority program that can perform the desired weight loss as a function of the heating rates and heating environment.

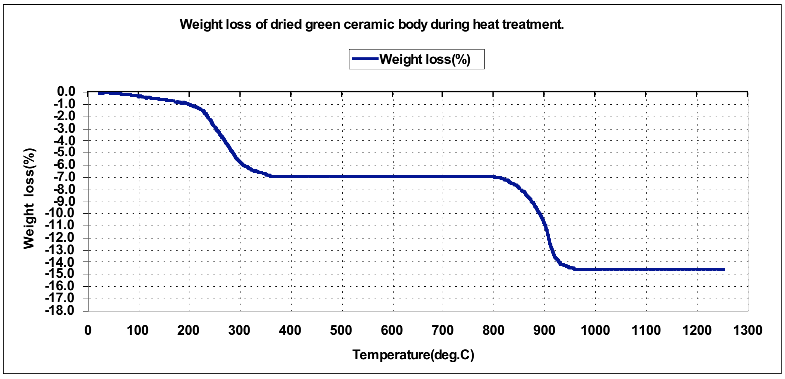

Fig. 2 Weight loss of dried green body during heat treatment

Endothermic and Exothermic Reactions

During heating and cooling physical and chemical transformations that release heat (exothermic reaction) or absorb heat (endothermic reaction) take place in the ceramic product. These reactions affect the properties and defects in the final product.

Knowing the temperature ranges where the reactions occur is very important in developing thermal processes. The reactions are defined by measuring the temperature of the product during heat treatment. The Fig.3 exemplifies the information that the CTB apparatus provides.

Combining all the information in one chart (Fig. 4) , the engineer has a complete picture of all the phenomena that take place in a full size ceramic product during heating and cooling. He can study the effect of heating or cooling rates, different firing atmospheres and flows on product properties and defects in order to

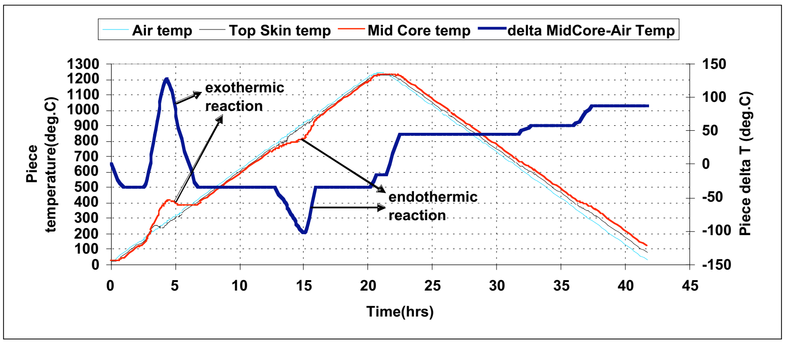

Fig. 3 Temperature distribution in green body during heat treatment

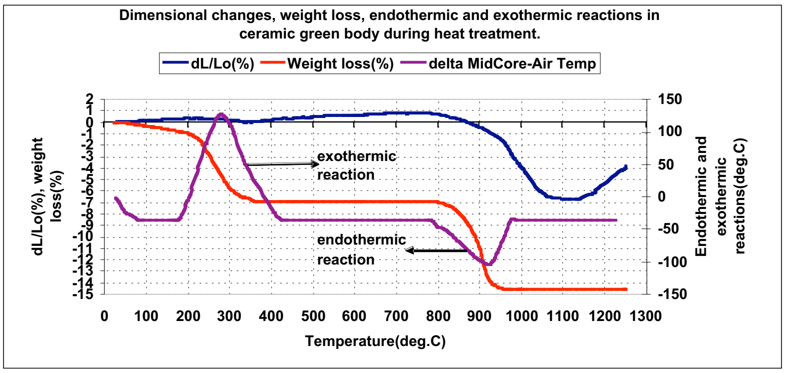

Fig. 4 Dimensional changes, weight loss, endothermic and exothermic reactions in green body during heat treatment

define the optimum parameters for a cost effective firing process. The advantage of using CTB simulator is that all the phenomena are related to mass and heat transfer in a full size piece.

The CTB firing simulator can be used to study drying process on a full size piece as a function of temperature and relative humidity. The data provided are weight loss and dimensional changes correlated to temperature and relative humidity of the environment.

All these data help the engineer to develop optimum and cost effective drying process.

The equipment can be programmed to correlate the moisture removal to the drying parameters as relative humidity and temperature of the heating.

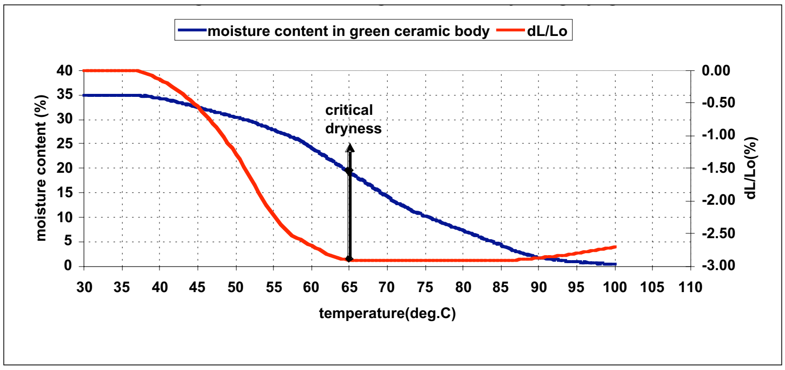

Fig. 5 Weight loss and dl/l_0 in wet green ceramic body during drying

The Fig. 5 exemplifies the behavior of wet green body during drying. The equipment can be programmed to correlate the moisture removal to the drying parameters as relative humidity and temperature of the heating medium.

Also the CTB apparatus can be used for quenching studies of ceramic compositions under diverse heating conditions.