EnerTHERM – a Joint Effort for Energy and Cost Efficient Heat Treatments

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Energy efficiency is the key parameter controlling the worldwide efforts to reduce CO_2 emissions. More and more companies become aware of the benefits of energy management systems which are defined in DIN EN ISO 50001. Recording energy flow within production and implementing measures for a systematic reduction of energy consumption usually pays off within few years. Additionally political incentive schemes for improving energy efficiency can be used like the exemption from the cost allocations under the German Renewable Energies Act (EEG-Umlage).

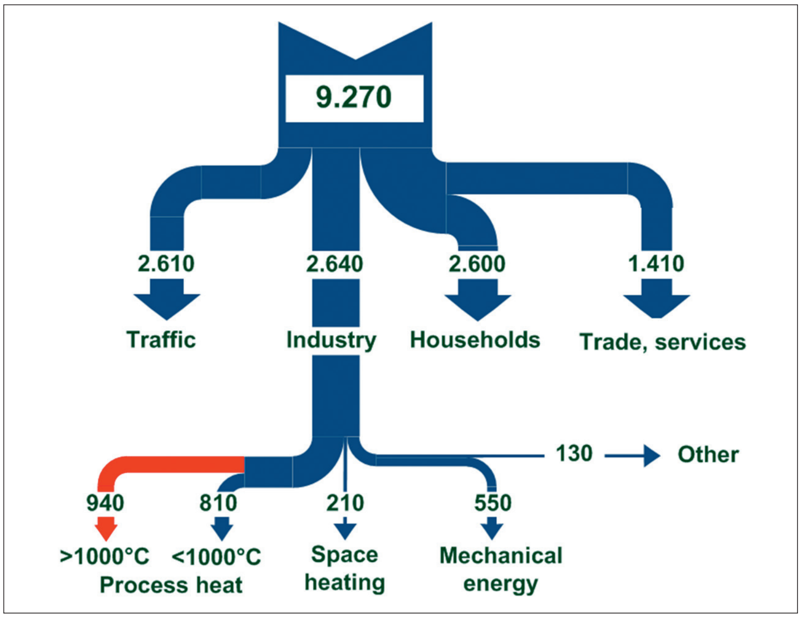

Fig. 1 Energy consumption in Germany 2013 in PJ (1 PJ = 10^{15} J) [1, 2]

Introduction

Within manufacturing industry as much as two third of energy are used for heating processes, half of it for high temperature processes with temperatures higher than 1000 °C (Fig. 1). This offers a very large potential for energy efficiency measures. In heating processes, energy efficiency is closely coupled to material efficiency and product quality, which is an additional motivation for careful optimization of these processes. Moreover energy savings and reduction in duration of heating cycles are closely correlated, the later allowing for higher throughput and further economic benefits. Ceramic industry can profit twice from energy efficiency measures. On the one hand its own heating processes are improved, i.e. mainly: drying, debinding and sintering. On the other hand new ceramic products are developed which are required to improve energy efficiency in other industries, e.g.: heat exchangers, refractory slabs and gas distribution systems.

EnerTHERM

The team of Fraunhofer Center for High Temperature Materials and Design (HTL) has investigated energy efficiency of industrial heating processes for more than a decade. In a joint project, finished in 2012, they demonstrated that considerable energy savings can be obtained in the production of various technical ceramics [3]. Important methods are a systematic optimization of heating parameters and a careful control of green compact quality. Both topics were published in previous contributions to this journal [4, 5].

Recently a new large project, EnerTHERM, was started at HTL [6] extending the scope of previous research. EnerTHERM addresses high temperature materials, process parameters of heating cycles and furnace design in a holistic approach to realize the full potential of industrial heat treatments. Aim is a significant improvement of energy efficiency, material efficiency and product quality. Within the project know how is generated which is transferred to industry in numerous follow-up-projects. Industrial partners are from ceramic and refractory industry, producers of thermoprocessing equipment and metal industry.

Methodology

The performance of heating operations depends on processes comprising the micron scale of individual grains, the centimeter and meter scale of components and the multi meter scale of production furnaces. Heat is transferred via conduction, convection and radiation from the furnace to the charge and from the outside to the interior of the charge and the individual components generating a complex temperature field. The local temperature causes thermally activat-

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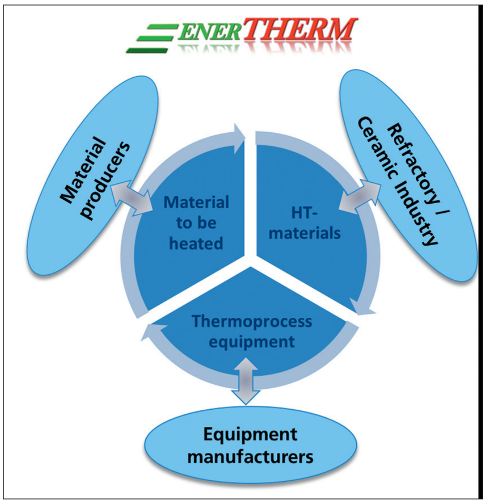

Fig. 2 Holistic approach of project EnerTHERM

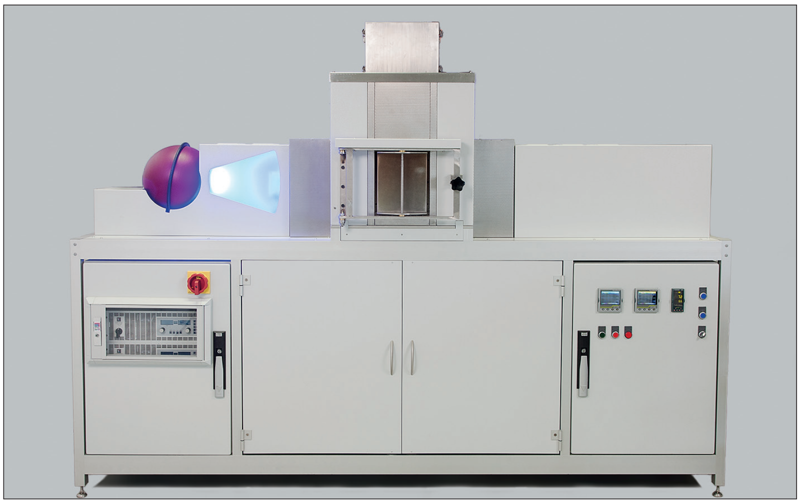

Fig. 3 New thermooptical measuring device, TOM_air, for studying debinding and sintering processes up to 1750 °C

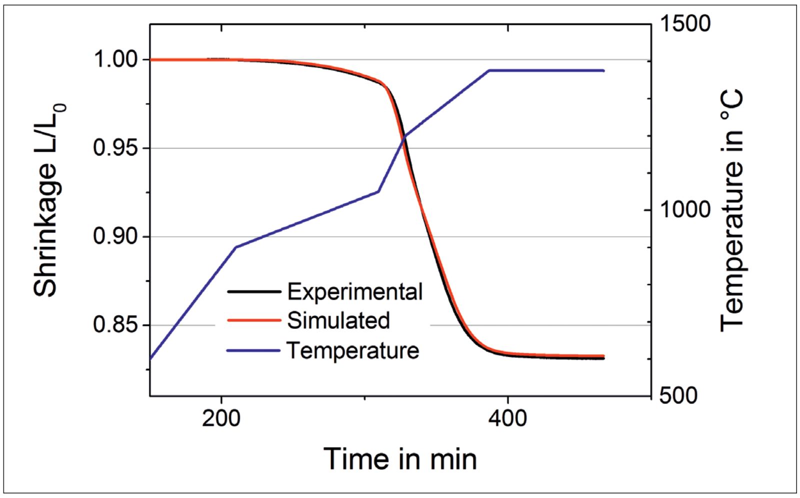

Fig. 4 Demonstration of predictive capability of kinetic field approach using an arbitrary time-temperature cycle for sintering an alumina green sample

ed reactions of the material: drying, pyrolysis, binder burn out, dehydration, solid state reactions, phase transformations, sintering. All of these processes are governed by the thermodynamic minimization of free energy. Many processes cause volume changes of the charge or emission of reaction gases. Some also affect local temperature distribution. Thermomechanical stresses arise which are detrimental for product quality. On the other hand throughput and energy efficiency require short temperature cycles. Short temperature cycles increase temperature gradients and thermomechanical stresses in the furnace and the charge.

The trade-off between cycling time and product quality is solved by a systematic approach in EnerTHERM considering the bottom up view from the material and component side on the one hand and the top down view from the furnace side on the other hand. Thereby another trade-off is resolved: in principle energy losses of production furnaces can be reduced using thicker isolation layers at the outside. But depreciation cost and overall CO_2 footprint of the heating process increase considering the cost and the energy for production of additional refractory materials. Another disadvantage of thicker isolations occurs in batch furnaces: prolonged cooling cycles. The interaction between furnace design, energy consumption and temperature fields is considered in EnerTHERM.

The project relies on three pillars (Fig. 2):

- The in situ measurement of material changes during the heat treatment
- The investigation of temperatures, atmospheres and energy flow in production furnaces
- The development of high temperature materials and components.

In addition, computer simulations are developed. They combine the pillars covering micro scale as well as macro scale processes.

In situ measuring

The material response during the heat treatment is measured in lab furnaces which are specially designed to reproduce exactly the atmosphere of the production furnace and to measure in situ those quantities required for a simulation of the heat treatment. These lab furnaces have been named Thermooptical Measuring (TOM) devices. Atmospheres comprise gas fired kilns, electrical heated furnaces operating in air, inert gas, Hydrogen and other reducing gases as well as vacuum and overpressure. It was shown in previous work that atmosphere has an essential impact on material response during the heat treatment [3]. So, four different TOM devices are designed for the realization of different furnace atmospheres. Three additional TOM devices are constructed to realize different gas flow and to allow up scaling of sample or stack size from 3 cm in the standard set up to 30 cm in the largest furnace.

The most important in situ measuring quantities are weight loss and shrinkage. In addition thermal conductivity, elastic and vis-

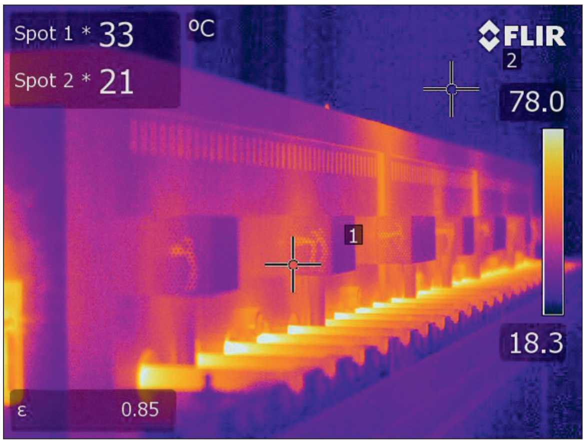

Fig. 5 Thermographic image of a roller kiln

Fig. 6 Set-up of a small batch furnace for alumina sintering

cous properties and acoustic emission are required to consider heat flow, mechanical stresses and crack formation during the heat treatment. Special high temperature sensors were developed to obtain these data with sufficient accuracy and reproducibility. The basic measuring principle was already described previously [7].

Within project EnerTHERM resolution and reproducibility of the in situ measuring methods were further improved. E.g., with optical dimension measurement a resolution of 0,1 µm was achieved in a new device named TOM_air (Fig. 3). Together with a very high reproducibility this allows the precise measurement of sintering kinetics. The well-known kinetic field technique [8] has been further improved to accurately predict sintering shrinkage for arbitrary time-temperature-cycles once a basic set of experimental data has been measured. As an example Fig. 4 shows the shrinkage of an alumina green sample during an arbitrary time-temperature cycle. The difference between simulation by the kinetic field technique and the experimental data can hardly be seen.

Besides optical dimension measurement TOM_air was equipped with microphones to detect acoustic emissions during the heat treatment. The simultaneous use of four microphones enables the exact localization of the sound source. So even low crack signals from the sample can be filtered out from the noisy lab background. The same technique is used in another new TOM device, named TOM_pyr, especially designed for the investigation of debinding processes. Here the acoustic emission measurement is combined with in situ weight measurement in controlled atmosphere and controlled gas flow.

Investigation of production furnaces

Data on temperature fields, atmospheres and energy flow are obtained from production furnaces. For that, existing methods, like thermography (Fig. 5), are validated and – if necessary – improved. For the measurement of temperature fields process temperature control (PTCR) rings are used. PTCR rings are placed at different positions in the furnace and indicate thermal energy by the shrinkage of the ring diameter after the heat treatment. The accuracy and the range of application of this technique have been significantly improved within EnerTHERM. For that, new compositions of the rings for measuring at temperatures above 1800 °C were developed. Careful calibration of the PTCR rings was established utilizing the optical detection of melting points of eutectic mixtures in the TOM devices. The kinetic field technique mentioned in the previous paragraph was utilized to obtain accurate temperatures for non-standard time-temperature cycles. The latter is important in the control of production furnaces which can rarely be adapted to the requirements of the producers of PTCR rings.

For the measurement of energy flow in production furnaces, thermal material properties have to be measured at high temperatures. Another TOM device, named TOM_wave was designed for that purpose. Thermal diffusivity is measured in TOM_wave by the well-known laser-flash technique. A new inverse simulation of the temperature distribution enables the investigation of large samples with a volume of some tens of cubic centimeters. This is essential in the investigation of refractories: their heterogeneous microstructures require large probe volumes to achieve reproducible measuring results.

Another technique, available with TOM_wave, is the thermal shock measurement at high temperatures. Whereas customary methods of thermal shock measurement use ambient temperature as the lower temperature reference, thermal loads in practical use of many high temperature components occur at higher temperature ranges. Therefore, the lower reference temperature is raised to the real level in the furnace of TOM_wave and the thermal shock is applied by additional fast laser heating or subsequent fast cooling. The before mentioned acoustic measuring technique is used for a sensitive detection of cracks during thermal shock testing. Whereas thermal diffusivity is needed in the simulation of the production furnaces thermal shock behaviour is important in lifetime predictions of the refractories enabling better decisions on alternative kiln furniture.

Exemplary, the optimization of energy efficiency and throughput is shown for alumina sintering in a small batch furnace (Fig. 6). Different holding times between 0,5–4 h were tested. The respective holding temperature, required to achieve 99 % of theoretical density, was calculated by the kinetic field technique. The power consumption was recorded using current clamps. The microstructure of the sintered samples was investigated by electron microscopy.

Fig. 7 shows the temperature cycles and shrinkage curves. Energy consumption is

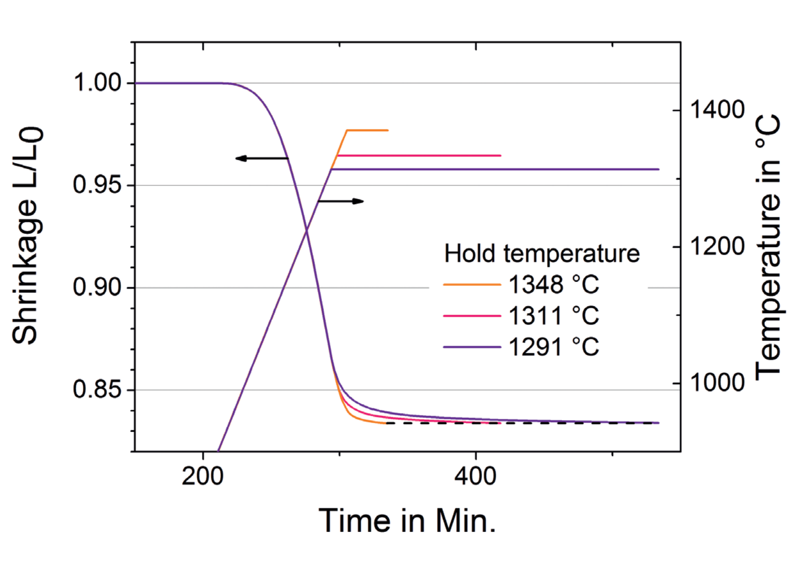

Fig. 7 **Time-temperature cycles and shrinkage curves used in the batch furnaces shown in Fig. 6**

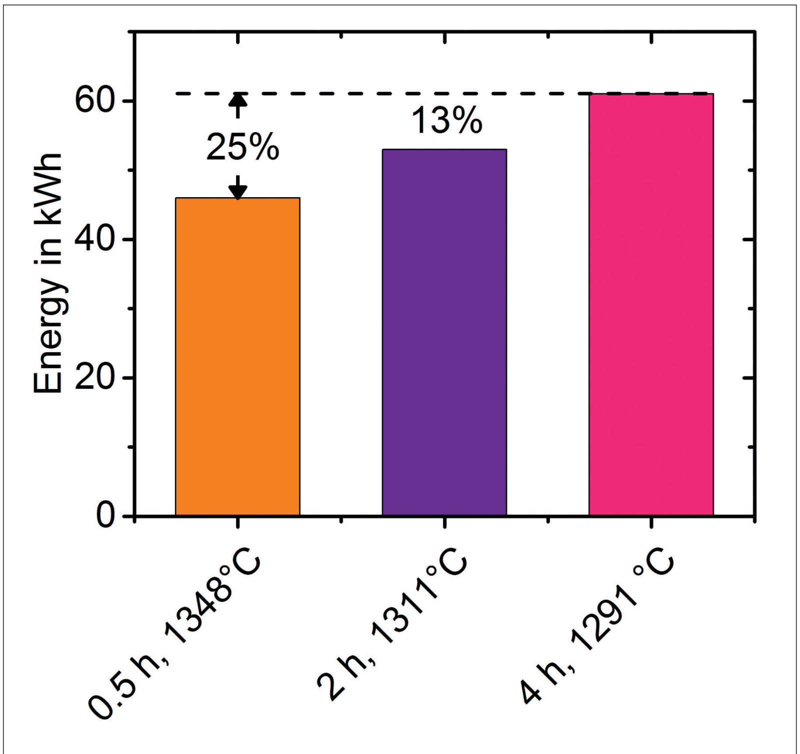

Fig. 8 **Energy consumption during the heating cycles shown in Fig. 7**

significantly lower at higher holding temperature (Fig. 8) and throughput is considerably improved, whereas final density and microstructure of the different batches are similar.

Outlook

The third pillar (compare section "Methodology") comprises special fiber reinforced ceramics, materials with low heat capacity, high temperature coatings and joining methods for connecting components at high temperatures. Details will be reported in a later publication. On the micron scale the simulation of the structure of multiphase ceramics and the calculation of its macroscopic material properties has made large progress. Thermal, mechanical and electrical properties can be simulated for a large variety of ceramic microstructures with sufficient accuracy [9]. The microstructure simulation supports the experimental development of new ceramics and optimized variants of customary ceramics. Recently sintering was included in the generation of microstructures on the computer establishing models which consider interface energies [10]. It was shown, that metastable states exist during sintering which cause differential sintering and deteriorate homogeneity and reliability of sintered products. On the other hand, parameters for inherent homogeneous sintering processes were identified. So a large potential for the improvement of industrial heating processes has been identified regarding energy and cost efficiency and microstructure homogeneity and product quality as well.

Results of project EnerTHERM will be presented at an industry workshop at 17 June 2015 at Fraunhofer Center HTL in Bayreuth.

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