THE FIRING OF RED-EARTHENWARE TILES BY RADIATION-HEATED ROLLER KILNS

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The firing of ceramic tiles is highly energy-consuming and strongly helps rising costs: therefore, great technological efforts were carried out by ceramic factories in recent years to develope fast-firing roller kilns. Nevertheless, red earthenware tiles, which are greatly disadvantaged by defect-enhancing dry-milling technologies, need generally more prolonged firing times to minimize the dishomogeinity of the mixtures and the presence of raw materials rich in organic carbon and sulphur content. These pollutants are the first cause of the appearance of black-core and pin-holes in the products.

The introduction of a new firing system, which employs innovative methods of heat transmission and distribution, permitted a dramatic enhancement of the fired products properties. Previous works by the authors investigated the clay-type raw materials used in this process[1], a theoretical model[2] and the properties of bricks fired by this system[3].

The present works deals with the control of the technological parameters of red-earthenware sintered tiles: a comparison between the data obtained by traditional and by radiation firing is reported.

a) Thermal analysis investigation of firing process

The thermal analysis (TG, DTG and DTA) of raw materials shows the phenomena occurring in the products during heating, as weigth-losses and henthalphy variations. These methods, widely used at present in the ceramic laboratories, exhibit water, carbon dioxide and organic materials losses during heating, yielding in first approximation the content of kaolinite and of carbonates in the material: a first firing pattern can be

[1]. M. Poppi, B. Venturelli, D. Minichelli, F. Ricciardiello, "Tiles & Bricks Internat.", 7(5), 337-38 (1991)
[2]. D. Minichelli, F.Genel Ricciardiello, "Tiles & Bricks Internat.", 10(5), 329-32 (1994).
[3]. M.G. Parasporo, M. Poppi, F. Genel Ricciardiello, D. Minichelli, "Tiles & Bricks Internat.", 11(2), 99-101 (1995).

obtained by these informations, considering only the mineralogical and chemico-physical characteristics of the materials and not the technological ones (milling, pressing and drying). A typical DTA curve for a red clayish material show that an optimized firing cycle must have some plateless zones at prefixed temperatures, while exist several thermal zones where the ranging up of temperature is scarcely influent on the chemico-physical events that occurr in the mixtures.

The machine investigated by the authors was projected to move evenly from one step to the following in very short times, while heat transmission is performed by radiation in the moduli at constant temperatures; through this method, not only heat loss due to convenction phenomena could be avoided, but a significative increase in the final product characteristics was reached.

b) <u>Theoretical guidelines of radiation-heating process</u>

The ceramic firing process can be generally described with the following four steps:

- Drying,
- Degassing,
- Sintering,
- Cooling.

The best experimental conditions for each of these steps were obtained on the basis of a trial-and-error method, with the aim of obtaining products with low porosity and good mechanical properties.

In the drying step water is easily removed by the applied thermal gradient, while degassing must be effected at the lowest possible temperature, because yet at 500°C recrystallization phenomena and grain size increasing can be observed.

The "secret" of the process is to reach the sintering temperature starting from grains as smaller as possible, effect that can be obtained with a fast passage from the degassing temperature to the sintering one. In this case, densification will be faster giving smaller grains and holes in the sintered mass, thus enhancing all the mechanical properties.

Not only heating phases, but also cooling can give a better product: a material quenched from sintering temperatures up to the quartz inversion one (600°C), show near doubled bending strength values.

c) <u>Characterization of fired products</u>

To test the validity of the proposed model, a series of determinations were made on red-earthenware tiles fired in different conditions; in addition, several determinations, both chemico-physical and technological, were then performed on these samples (Optical Microscopy, X-ray Diffraction, Differential thermal analysis, Porosity determination, Thermal expansion analysis).

d) <u>Results</u>

The tests carried on the specimens showed that, as can be seen from mycroscopycal, XRD and porosity determinations, samples obtained after radiation firing and fast cooling, exhibited a greater part of glass phases, smaller grains and good distributed pores as respect to samples traditionally fired (tunnel kilns). In other samples, fired by radiation and then reheated in muffle at 1000°C, textures quite similar to that showed by the traditionally fired sample could be observed: this is a sign that radiation-firing system does not reach equilibrium conditions. Moreover, in the not-quenched specimens, a content of hematite-type phase as greater as the samples attained equilibrium, was observed: this fact is strictly connected to the color of the product, which is a function of the firing cycle[4].

The presence of a greater part of glass-type phases was also confirmed by observation of TEA curves, while DSC tests showed, in the quenched samples, a little endothermic peak near 500°C which is probably referred to recrystallization phenomena: this peak was totally absent in the refired samples.

Bending-strength tests made on the fired tiles showed values largely greater for quenched samples as respect to the ones fired in tunnel kilns; this fact was confirmed by all the determinations of mechanical resistance performed in the industrial practice.

e) Control of time-depending technological properties

As previously described, tiles obtained from radiation-heating process markly showed that products did not attain equilibrium. It is therefore possible to suggest a decrease, during the time, of their technological properties; specific controls appeared then necessary. To obtain such informations, an artificial process of ageing was made on the tiles. From a theoretical point of view, it is correct to postulate that a heating at 150°C for 200 h is equal to a period of 10 years at 50°C; this hypothesis can be suggested considering the kinetics of the phenomena occurring in solid phase and suggesting that no nucleation effects can happen at this temperature, as confirmed by DSC data.

To test the correctness of our hypothesis, indentation tests were perfomed both on normal and on artificially aged samples, with the aim to evidence the behavior of the samples when subjected to a sudden impact.

The results of impact tests showed a behavior of the aged samples strictly similar to the ones of the not-aged samples, being observed variations in the Vickers hardness not over 10%. In addition, by optical observation of the cracks induced by the impacts on the samples, a substantial similarity of brittleness in all the types of specimen could be observed: this fact points to a similar chance of brittle fractures in all the samples.

CONCLUSIONS

On the basis of the obtained results, the following conclusions can be pointed out:

- After the same processes performed on the powders (mixture types, milling, pressing), radiation firing yields sintered products with a bending-strength as much higher as faster is the cooling step (from 30% up to the double);
- The color exhibited by the tiles is a function of the firing process; a partially reduced kiln atmosphere yields a darker product;

[4]. A. DE PRETIS, D. MINICHELLI, "Proc. 7th Euroclay Conference", Greifswald 91, 269-73 (1991).

- The sintered products, owing to very short cycles, did not attain equilibrium; further thermal and mechanical treatments can change their characteristics;
- Variations with time of the properties seem to be highly unlikely;
- Raw materials normally unsuited for production, owing to a high impurities content, can be advantageously used by radiation-heating process.