PILOT-SCALE STUDY OF THE INFLUENCE OF FIRING CYCLE VARIABLES ON SINGLE-FIRE TILE CURVATURE

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ABSTRACT

Lack of planarity in ceramic tiles basically arises in firing. This is due to the characteristics of the firing cycles used, as tiles are made of layers of different materials, and because of the conveying system.

Numerous studies can be found on this issue in the literature, but the absence of appropriate facilities have limited the studies performed to laboratory tests, more or less complicated mathematical modelling, and very few industrial-scale observations.

A study has been undertaken in the present work on a semi-industrial scale of the influence of firing variables on ceramic tile curvature. This has been possible as a result of using a specially designed batch pilot kiln.

1. BACKGROUND

One of a ceramic tile's most valued characteristics is its flatness. This is an indispensable requirement for these materials, whose ultimate purpose is to cover walls and floors to improve their hygienic and aesthetic quality. However, different factors in the ceramic tile manufacturing process can affect a tile's flat shape. This is particularly the case when tiles are formed by pressing in a typical single-fire manufacturing process.

In single-fire tile manufacture, green ceramic bodies undergo various operations [1] in the successive production stages, which can affect tile flatness. This is due to the nature of the body and to the operations the tile undergoes. Studies abound in the literature [4-12] on the factors that affect tile flatness during firing. Indeed, industrial experience [7] shows how this last manufacturing process stage exerts the most decisive influence on flatness.

However, the studies published to date on this issue relate to work done on a laboratory scale, in which at most, limited observation took place in industrial conditions. This constraint was not accidental, but rather a condition imposed by actual industrial production needs. These needs determine the extent to which the variable involved can be acted upon, and hence the possibility of performing studies with the extent and rigor needed to achieve a detailed, accurate understanding, and quantify the effect of firing variables on tile end curvature.

Pilot-scale kilns are therefore needed to enable studying the various issues relating to the firing process in detail. It was recently established that using a batch pilot kiln [5] enabled reproducing industrial firing conditions with great reliability.

A pilot-scale kiln of this kind was used in the present study to study the influence of the effect of the most significant firing stage variables on single-fire tile curvature.

2. AIM AND SCOPE OF THE STUDY

The study had two objectives. First, showing that the available batch pilot kiln was a sufficiently accurate tool to study tile curvature during firing.

Secondly, after verifying the first objective, establishing the influence of the firing cycle on tile curvature. This part of the study focused particularly on the firing cycle stages that are generally recognised as being the most effective for regulating curvature. That is, preheating in the 750-1000°C temperature range; firing, which corresponds to the peak temperature zone typically ranging from 1110 to 1140°C, and finally, the range of temperatures near the glaze-fitting temperature during cooling.

3. MATERIALS, FACILITIES AND APPARATUS, AND EXPERIMENTAL PROCEDURE

3.1. MATERIALS

Two types of single fire body were used in the study: a redware floor tile and a redware wall tile body. Their expansion curves are presented in Figs. 1 and 2. An engobe and two glazes (a transparent and an opaque glaze) were also used.

The green floor tile body measured 350x 350 mm, with a green bulk density of 2100 kg/m³. The green wall tile body measured 330x450 mm, with a green bulk density of 1950 kg/m³. Both types of body were industrially processed.

With regard to the glazes, the most important information as far as this study is concerned relates to glaze thermal expansion curves. Fig. 3 depicts the respective glaze thermal expansion curves.

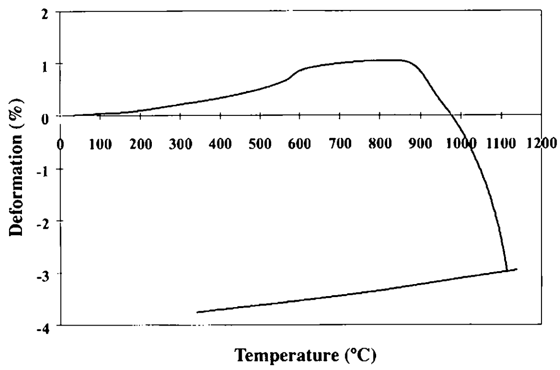

Figure 1. Thermal expansion curve of the floor tile body.

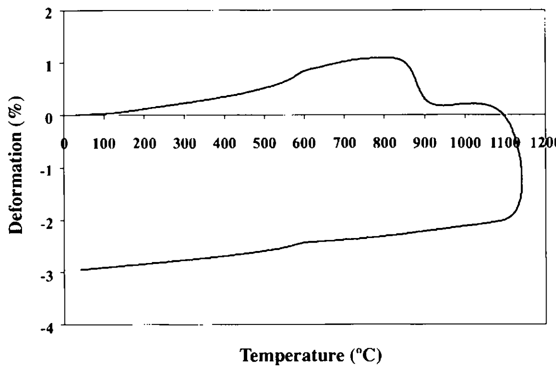

Figure 2. Thermal expansion curve of the wall tile body.

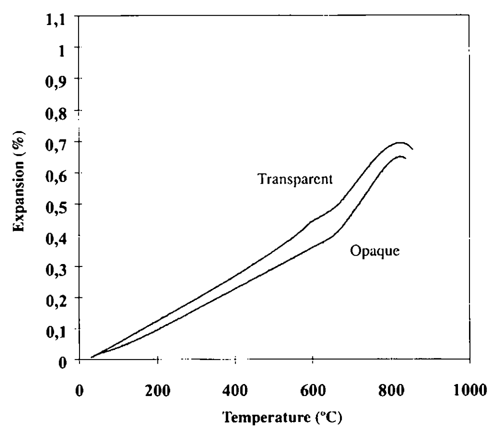

Figure 3. Glaze dilatometric curves.

3.2. FACILITIES AND APPARATUS

3.2.1. Laboratory apparatus

The thermal expansion curves were measured on an absolute dilatometer, calibrated with a sapphire standard. The experimental procedure involved in this measuring technique has been widely described in the literature [12].

Tile curvature and the profiles of each tile side were measured on a 3-dimensional measuring system, with a 20-mm spacing between measuring points.

3.2.2. Pilot-scale facilities. Glazing plant

The glazes and engobe were milled in a batch ball mill, with a nominal charge of 150 kg dry solid. Engobe and glaze application was performed in a glazing line fitted with two 500-mm diameter bells and a wetting chamber, with adjustable glazing rate regulation. An electric heater near the glazing line kept the ceramic bodies at the desired temperature.

Tile glazing was conducted using standard industrial density, viscosity and layer thickness.

3.2.3. Pilot-scale facilities. Kiln

The tiles were fired in a batch pilot kiln. The kiln is fitted with eight high-speed burners, operating intermittently. The tiles are conveyed on ceramic rollers by a meshed system, and enter and exit the kiln simulating the movement of an industrial continuous kiln.

The tiles are fed into the kiln manually through an opening at one end. The combustion gas collector and gas exhaust stack are located at the other end.

The inner kiln useful surface in the roller plane is 2 m^2. Total kiln length, including fan and electrical panel is 6 m and kiln height, excluding the stack, is 2.2 m

Refractory fibre blocks were used as insulating material in building the kiln. The material has good thermal insulation characteristics while also exhibiting low inertia, allowing fast thermal cycles to be run. Further detail on kiln characteristics and operation can be found elsewhere [5].

3.3. EXPERIMENTAL PROCEDURE

As is well known, the usual way to modify tile curvature during firing is by creating a temperature gradient between the top and bottom surface. It is also known from industrial experience, that the effect of a given temperature gradient on tile curvature also depends on the existing tile surface temperature when the gradient is produced.

To evaluate the influence of both these factors, the experiments set out in the points below were programmed. The firings run to study the preheating and firing stages were performed on bodies without an engobe or glaze. Glazed bodies were only used in the experiments conducted on the cooling stage. This approach was adopted to allow distinguishing between the effect of firing and glaze fitting [11 and 12].

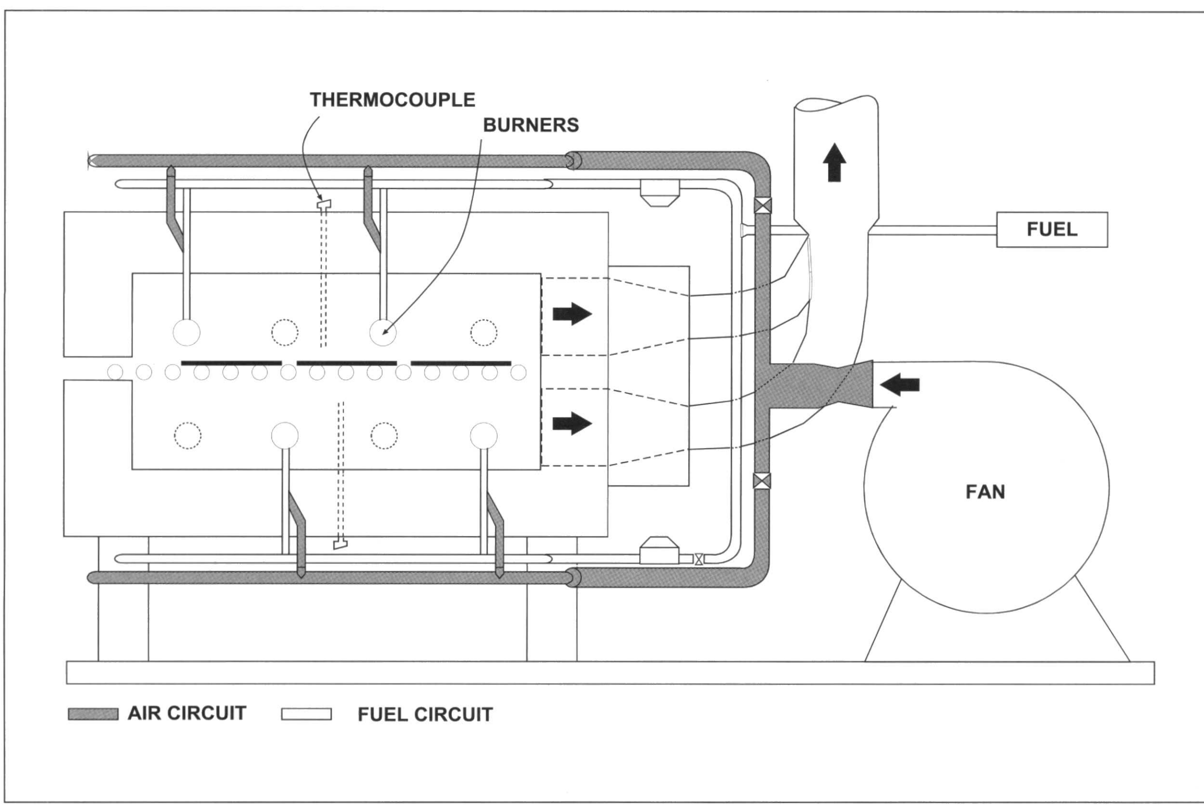

Figure 4. Schematic illustration of the pilot kiln.

3.3.1. Preheating stage experiments

To assess the influence of the thermal gradient on both bodies, thermal cycles similar to those depicted in Fig. 5 were programmed (in which only the preheating stage is shown). The difference in temperature between both chambers was kept practically steady at 150°C while heat-treatment lasted, and cooling started rapidly after attaining the desired maximum temperature in each chamber. Table 1 presents the pairs of maximum values reached in the bottom chamber (bottom temperature) and in the top chamber (top temperature) for each tested thermal cycle.

FLOOR TILE				WALL TILE			
Cycle	Bottom temperature (°C)	Top temperature (°C)	Mean temperature (°C)	Cycle	Bottom temperature (°C)	Top temperature (°C)	Mean temperature (°C)
GP3	1000	850	925	PP2	900	750	825
GP6	1050	900	975	PP3	950	800	875
GP4	1100	950	1025	PP4	1000	850	925
GP5	1150	1000	1075	PP5	1050	900	975
				PP6	700	850	775
GP7	850	1000	925	PP7	750	900	825
GP8	900	1050	975	PP8	800	950	875
GP9	950	1100	1025	PP9	850	1000	925
GP10	1000	1150	1075	PP10	900	1050	975

Table 1. *Variations in the preheating cycles. Floor and wall tile.*

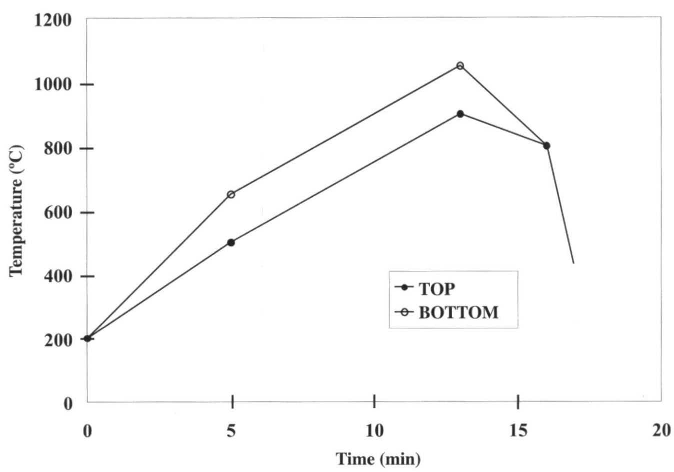

Figure 5. *Thermal cycle used in the preheating stage. Floor and wall tile.*

3.3.2. Firing stage experiments

Experiments were carried out by modifying the maximum firing temperature zone of the thermal cycles shown in Figs. 6 and 7, corresponding respectively to the original floor and wall tile schedules. In the case of the floor tile, the peak temperature reached in the bottom chamber was 1138°C and in the top chamber 1129°C. The maximum temperatures for the wall tile were practically the same in both chambers at 1100°C.

Table 2 details the temperature differences (bottom temperature minus top temperature) found in the original floor and wall tile cycles. The duration of the firing stage was not varied.

FLOOR TILE				WALL TILE			
Cycle	Bottom temperature (°C)	Top temperature (°C)	Temperature difference (°C)	Cycle	Bottom temperature (°C)	Top temperature (°C)	Temperature difference (°C)
GC1	1128	1119	9	PC2	1130	1130	-
GC2	1138	1129	9	PC3	1120	1120	-
GC3	1148	1139	9	PC4	1140	1140	-
GC11	1078	1109	-31	PC6	1080	1120	-40
GC10	1088	1099	-11	PC5	1090	1110	-20
GC9	1098	1089	9	PC7	1100	1100	-
GC12	1108	1079	29	PC8	1110	1090	20
GC13	1118	1069	49	PC9	1120	1080	40

Table 2. Variations in the firing stage cycles. Floor and wall tile.

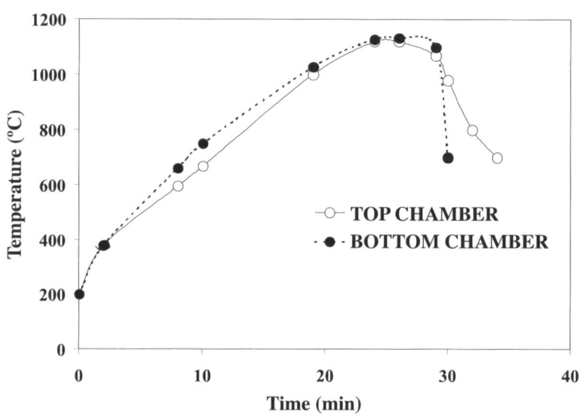

Figure 6. Floor tile thermal cycle.

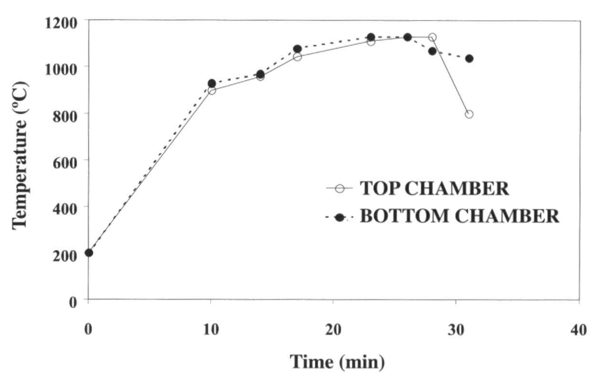

Figure 7. Wall tile thermal cycle.

3.3.3. Cooling stage experiments

Engobed and glazed bodies were used in these experiments, with each of the two glazes mentioned above (transparent and opaque), to assess the influence of glaze fit. The cooling stage of the floor and wall tile cycles shown in Figs. 6 and 7 were also altered. A large temperature difference was thus produced between the top and bottom kiln chambers, when tile temperatures were close to estimated glaze fitting temperature [12].

Table 3 sets out the changes made in the original thermal cycles for each tested glaze. The temperature difference (bottom temperature minus top temperature) was kept approximately in the range 1000-650°C.

THERMAL CYCLE Floor and wall tile	Glaze	Temperature difference (°C)
EGB1	Opaque	-78
EGB2	Opaque	-
EGB3	Opaque	114
EGC1	Transparent	-78
EGC2	Transparent	-
EGC3	Transparent	114

Table 3. Variations in cooling stage cycles. Floor and wall tile.

4. RESULTS AND DISCUSSION

4.1. RESULTS OF THE PREHEATING ZONE EXPERIMENTS

The studied bottom temperatures in this stage were close to those at which both bodies achieve maximum thermal expansion and start the shrinkage step, as the dilatometric curves show.

In the zone with the highest studied range of temperatures, the dilatometric behaviour of both bodies was quite different. As was to be expected, the wall tile bodies exhibited a zone between about 930 and 1030°C, in which the body underwent slight expansion, whereas the floor tile body exhibited continuous shrinkage.

After conducting the programmed firings, the value was determined of the centre deflection of each tile side as a measure of the curvature, which had a positive sign when curvature was convex and a negative sign when it was concave.

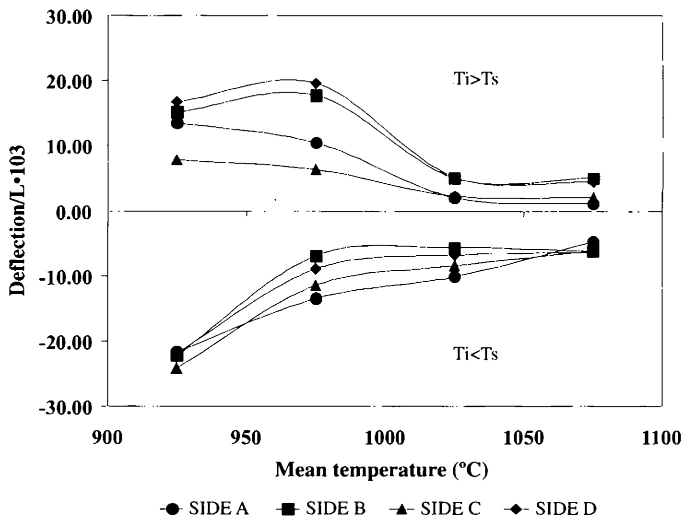

Figure 8. *Floor tile curvature. Preheating.*

Fig. 8 plots the data obtained with the floor tile body when the temperature in the bottom region (Ti) was higher than in the top (Ts) and vice versa. The figure plots the quotient of deflection to starting length of each side versus the arithmetic mean of top and bottom temperatures.

The floor tiles exhibited a large curvature, which was concave or convex according to the sign of the thermal gradient at mean temperatures ranging from about 925 to 1000°C. Curvature was found to peak at around 975°C when bottom chamber temperature was higher than that of the top chamber.

At mean temperatures exceeding 1000°C, curvature (concave or convex) decreased notably. This was due to the evolution of the material's elastoplastic characteristics with temperature (4 and 10), and the fact that the tiles are conveyed on rollers, supporting their own weight.

In all the range of tested temperatures, the sides parallel to the direction of advance (sides B and D) present a more convex deformation (or respectively less concave deformation) than sides A and C (Fig. 8). This asymmetry in tile deformation has also been observed industrially. This fact, determined by the conveying system, has been theoretically discussed elsewhere [6]. The conveying system therefore involves a non-uniform distribution of tile support points in each of the two directions of the tile sides.

To illustrate the foregoing, Figs. 9 and 10 plot the profile of floor tile side B when these tiles underwent the different tested thermal cycles (GP3-GP10).

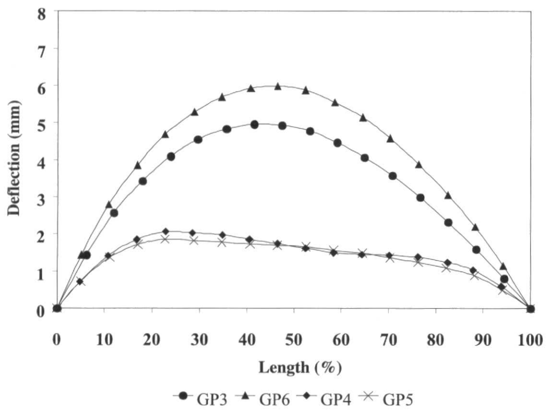

Figure 9. Floor tile profiles. Preheating.

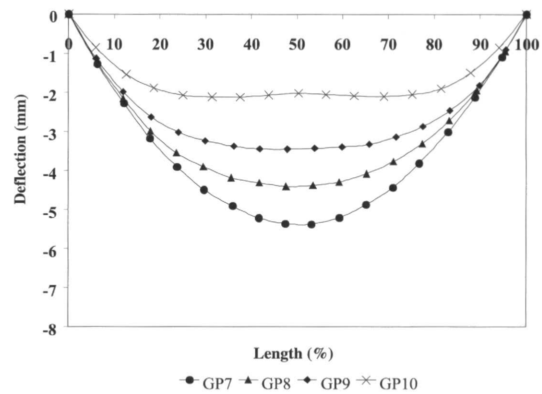

Figure 10. Floor tile profiles. Preheating

The wall tiles (Fig. 11) exhibited certain qualitative differences in behaviour compared to floor tiles in the preheating stage. When the bottom chamber temperature was higher than that of the top chamber (Ti > Ts) the curvature was convex in the mean temperature range of around 825 to 920°C. However, curvature was slightly concave between 925 and 950°C. This effect could be basically due to the expansion that this material undergoes in this temperature range, as the corresponding thermal expansion curve shows. The wall tiles also exhibited a great difference in curvature between sides A and C and sides B and D. Thus, when curvature was convex, sides B and D exhibited a larger curvature than sides A and C in the range 825-900°C. However, there was a small temperature range close to 925°C in which side A and C curvature was greater than that of sides B and D.

When top chamber temperature was higher than that of the bottom chamber, wall tile curvature was concave. Deformation maximised at around 900°C.

Figs. 12 and 13 present the profile of side B of the wall tiles that underwent the various preheating treatments. These profiles clearly show how the tiles kept the history of their thermal treatment in the curved line of their sides. This is possible owing to the elastoplastic behaviour of the ceramic material. Thus, the sides parallel to the direction of tile movement (sides B and D) exhibited convex curvature in the regions at the ends and a concave curvature in the centre in the first preheating stages, while this trend subsequently inverted or the tile deformed under its own weight (Fig. 12 curves PP4 and PP5).

In contrast, the lateral profiles (Fig. 12 curves PP9 and PP10) exhibited concave curvature at the ends and convex curvature in the middle when the tile presented a concave curvature with a subsequent inversion of this tendency in the first preheating stage. As remarked, these variations could be caused by the difference in temperature between the top and bottom chambers, or as a result of the evolution of material properties with temperature.

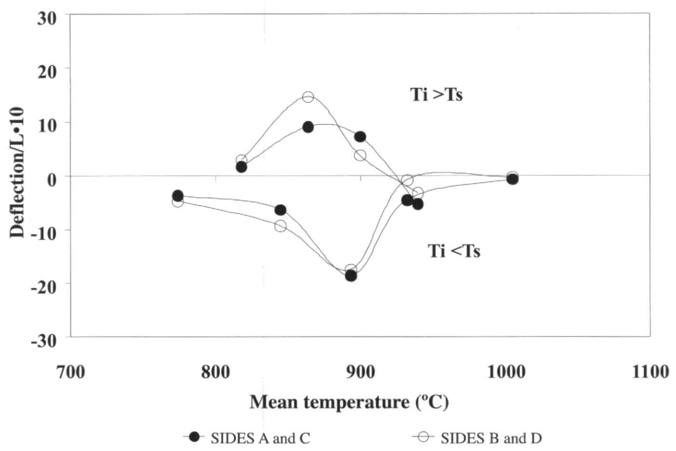

Figure 11. *Wall tile curvature. Preheating.*

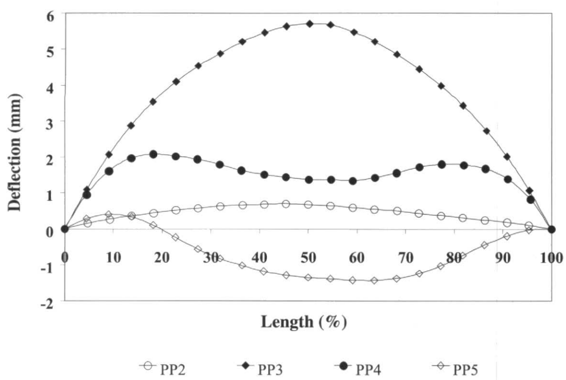

Figure 12. *Wall tile profiles. Preheating.*

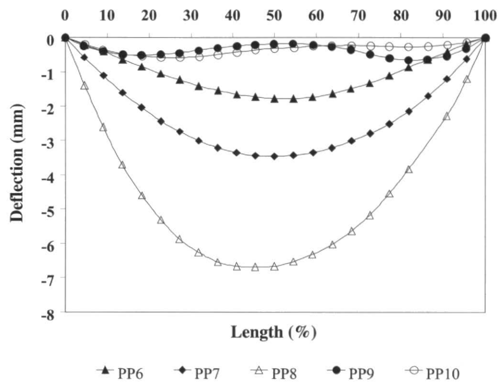

Figure 13. *Wall tile profiles. Preheating.*

4.2. RESULTS OF THE FIRING ZONE EXPERIMENTS

Figs. 14 and 15 present certain lateral floor tile profiles (side B) obtained after the experiments in the firing zone. They are all very similar. After these experiments a certain residual curvature was only to be remarked at the ends of sides B and D owing to the supporting effect.

In the case of the wall tiles, which were more rigid than the floor tiles [4], the temperature gradient was found to have a considerable influence (Fig. 16). However, the magnitude of the curvature was less than in the curvatures found in preheating, as at firing temperature, tile rigidity was lower.

The lateral profiles presented in Figs. 16 and 17 show that tile curvature matched that found in the preheating stage especially with the tiles that underwent the highest temperatures in the preheating stage.

In the programmed cycle, the tiles exhibited a convex curvature at the end of preheating, so that during the firing stage, this curvature can be reinforced or attenuated according to the sign of the thermal gradient. Thus, when bottom chamber temperature was higher than top chamber temperature (PC9 and PC8) the convex curvature was attenuated. However, this curve is determined to a great extent by tile deformability and the supporting effect, since, as Fig. 16 shows, the tile centre area is approximately flat and the ends have dropped.

On the other hand, when bottom chamber temperature was lower than that of the top chamber (PC5 and PC6), the side ends exhibited convex curvature whereas the tile centre area presented concave curvature, which increased with the rise in the thermal gradient.

The modification in maximum temperature, keeping a steady thermal gradient, did not appreciably affect wall tile curvature (Fig. 17).

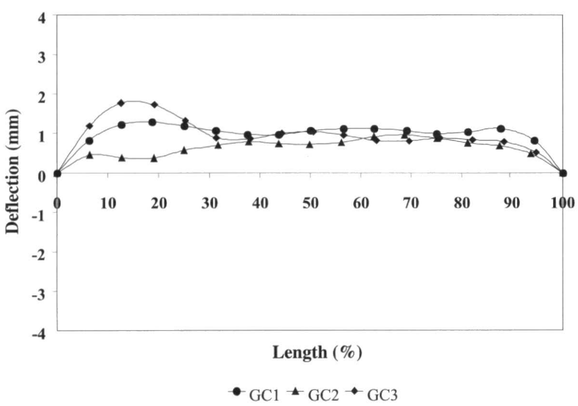

Figure 14. Floor tile profiles. Firing. Modifications in maximum temperature.

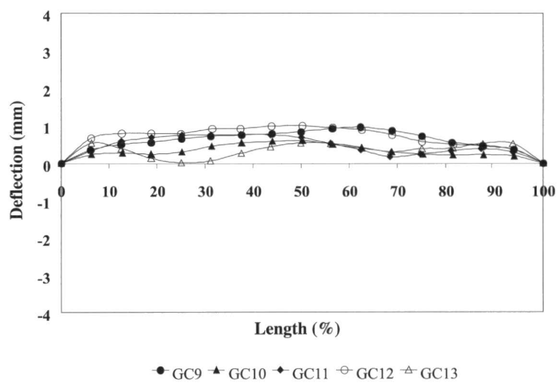

Figure 15. Floor tile profiles. Firing. Modifications in thermal gradient.

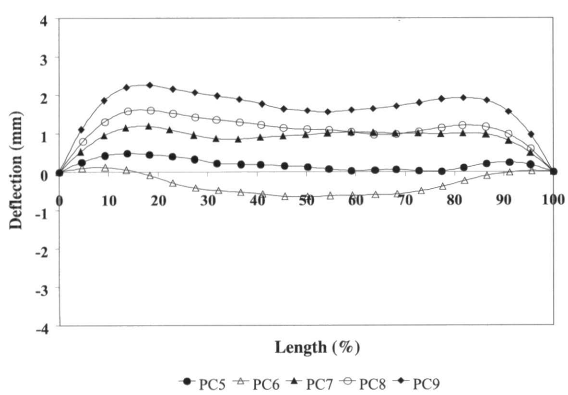

Figure 16. Wall tile profiles. Firing. Modifications in thermal gradient.

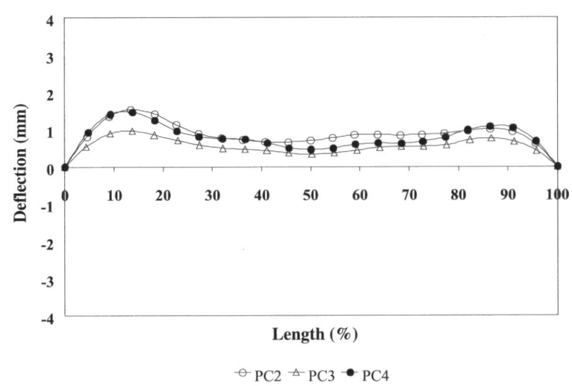

Figure 17. Wall tile profiles. Firing. Modifications in maximum temperature.

4.3. RESULTS OF THE COOLING ZONE EXPERIMENTS.

Figs. 18 and 19 respectively show the different curvature of the floor and wall tiles, when the glaze coating was transparent (with high expansion) or opaque (with low expansion) for each thermal cycle used.

The influence was found to be notable, and capable of producing a variation from convex curvature in the case of the opaque glaze, to concave curvature in the case of the transparent glaze (cf. curves EGB2 and EGC2 of Fig. 19). The magnitude of the arising variation was similar to that which could be achieved by modifying the temperature gradient. The consequence of this fact is obvious, and matches industrial experience, according to which it is convenient to use glazes with similar thermal expansion curves to that of the fired body. Otherwise, the curvature produced by the glaze can hardly be corrected by modifying the thermal cycle.

The pilot kiln used in these experiments enabled acting on the cooling stage. The thermal gradients described in the experimental part of this study were produced to try and modify body temperature when glaze fitting was to occur [11 and 12] and thus vary tile curvature.

Figs. 18 and 19 exhibit the evolution of curvature as a function of temperature difference between the top (glazed) and bottom tile surfaces. However, it was found on using unglazed tile bodies subjected to the same thermal cycles, that the arising curvature was similar to that shown in Fig. 18 and 19. These findings indicate that before modification of the curvature can be effected by a variation of the glaze-body fitting temperature, this occurs as a result of the body's own deformation, as was the case in the preheating stage.

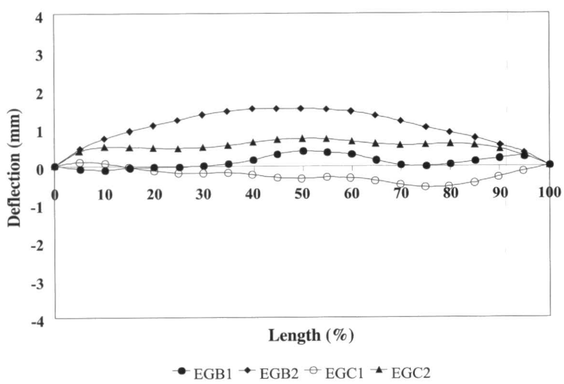

Figure 18. Modification of the cooling stage. Floor tile.

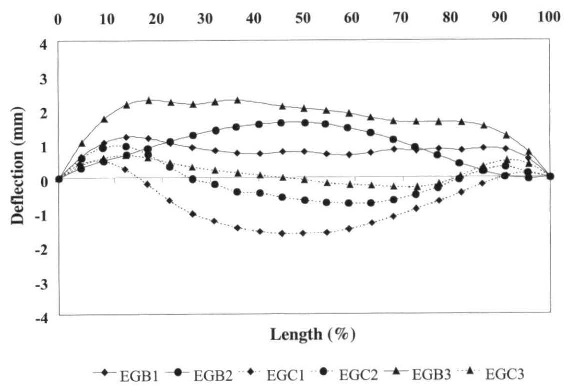

Figure 19. Modification of the cooling stage. Wall tile..

5. CONCLUSIONS

The findings of the present study have shown that it is possible to reproduce the curvatures observed in industrial tile manufacture by using a batch pilot kiln.

Performing a series of programmed experiments enabled evaluating the influence of the various thermal cycle stages on tile deformation as well as confirming the marked effect of the supporting system on this deformation.

The results obtained, together with the literature consulted, show that during firing, ceramic tiles basically undergo a succession of deformations that are recorded in the shape they adopt, owing to tile elastoplastic properties. The resulting end curvature of a given product is a consequence of the dynamics and characteristics of the thermal cycle and conveying system.

Using a pilot kiln, such as the one employed in this study, allows overcoming the enormous difficulties involved in attempting to estimate the shape that a tile will eventually adopt by theoretical considerations, or more specifically, the magnitude of tile curvature after firing in a given industrial cycle. This is especially so if the method is furthermore required to predict the modifications that are to be made to the thermal cycle to achieve minimum final curvature.

Thus, after running the experiments, the tiles were observed to exhibit an approximate symmetry with regard to the centre axis of the sides parallel to tile advance inside the kiln. There were therefore in general two characteristic deformations: the deformation found at about the centre of the side (centre deflection), and the bending observed close to the ends (which can be assessed as the slope exhibited by the tile ends).

When the value that these two magnitudes adopt (expressed as dimensionless modules) are plotted versus the temperature difference that produced them, a plot is found similar to the one presented in Fig. 20.

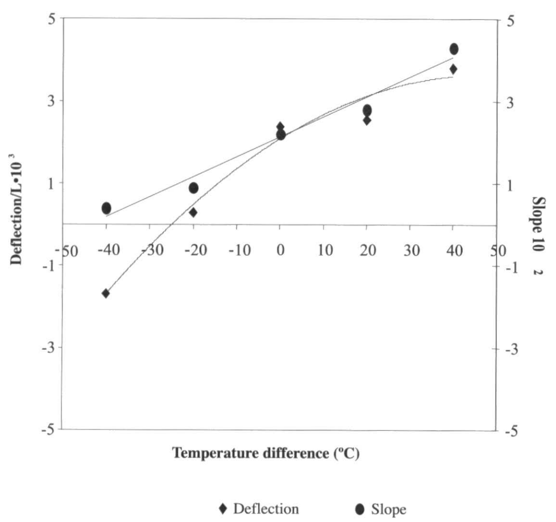

Figure 20. Evolution of side curvature and end bending with temperature.

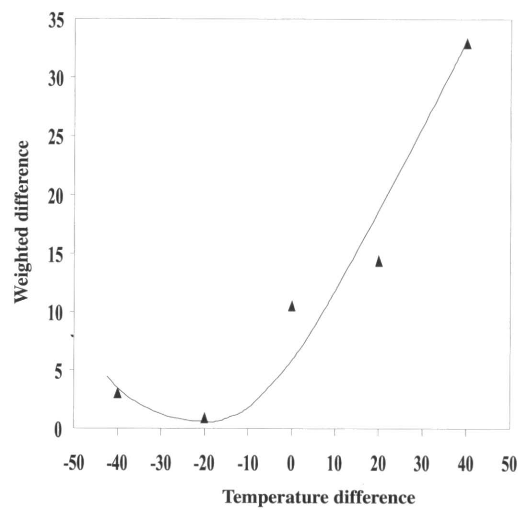

Figure 21. Joint deformation. Estimation of optimum temperature difference during firing.

On the other hand, the condition that end bending and centre deflection need to be minimum can be adopted as a comparative criterion for the tiles to be flat. This criterion can be accurately expressed by stating that the weighted sum of both factors, in absolute value, must be minimum. When overall tile deformation is thus calculated, a plot is obtained like the one shown in Fig. 21, from which the modification to be effected on a given firing cycle to minimise tile final curvature can be determined.

6. REFERENCES

[1] CLAUSELL, A; GALINDO, R. *Apuntes de operaciones básicas en la fabricación de pavimentos y revestimientos cerámicos.* Valencia: Consellería de Cultura, Educación y Ciencia. 1991.

[2] AMORÓS, J.L.; ENRIQUE, J.E.; BLASCO, A., ET AL. *Hornos para la fabricación de pavimentos y revestimientos cerámicos. Características y criterios de elección.* Técnica Cerámica, 104, 750-768, 1982.

[3] JARQUE, J.C.; MORENO, A.; ENRIQUE, J.E., ET AL. *Optimización de las condiciones de funcionamiento en hornos monoestrato [IV]. Mecanismos de transmisión de la energía calorífica.* Técnica Cerámica, 247, 566-572, 1996.

[4] CANTAVELLA, V. *Simulación de la deformación de baldosas cerámicas durante la cocción.* Tesis doctoral. Departamento de Ingeniería Química, Universitat Jaume I de Castelló. Castellón. 1998

[5] MALLOL, G.; LLORENS, D.; BELTRÁN, G., ET AL. *Reproducción de las condiciones industriales de cocción de las baldosas cerámicas utilizando un horno piloto de rodillos.* Técnica Cerámica, 276, 855-864, 1999.

[6] LORICI, L. *El horno de rodillos y su influencia sobre la planaridad del producto final.* Cerámica y Cristal, 114, 19-28, 1995.

[7] *Tecnología de la fabricación de azulejos.* Castellón: Asociación de Técnicos Cerámicos, 1990.

[8] MONZÓ, M. *Estudio de las causas de los defectos de fabricación en la industria de pavimentos y revestimientos cerámicos.* Castelló: Universitat Jaume I, Departamento de Ingeniería Química, 1994. Tesis doctoral.

[9] AMORÓS, J.L.; BELTRÁN, V.; BLASCO, A. ET AL. *Defectos de fabricación de pavimentos y revestimientos cerámicos.* Castellón. Asociación de Investigación de las Industrias Cerámicas, 1991, p. 159-160.

[10] ESCARDINO, A.; AMORÓS, J.L.; NEGRE, F., ET AL. *Defectos de planaridad en las piezas de pavimento gresificado motivados por la deformación piroplástica. Influencia de las variables de proceso.* Taulells, 3, 3-9, 1985.

[11] AMORÓS, J.L.; NEGRE, F.; BELDA, A., ET AL. *Acuerdo esmalte-soporte [I]. Causas y factores de los que depende.* Técnica Cerámica, 178, 582-592, 1989.

[12] AMORÓS, J.L.; BLASCO, A.; CARCELLER, J.V., ET AL. *Acuerdo esmalte-soporte [II]. Expansión térmica de soportes y esmaltes cerámicos.* Técnica Cerámica, 179, 644-657, 1989.