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Delayed Curvatures in Porcelain Tiles

Analysis and Measurement of Influencing Factors.

Abstract

Glazed and unglazed porcelain tiles exhibit a phenomenon known as 'delayed curvatures', which consist of the change in tile curvature after the tiles leave the kiln. This phenomenon becomes more problematic as tile size increases.

In this study, the variation of curvature in time has been quantified in industrial glazed porcelain tiles. It was observed that the curvature usually displays an evolution in one direction (customarily in a concave direction or vessel shape) which, after a certain time has elapsed, reverses this trend. The kinetics of

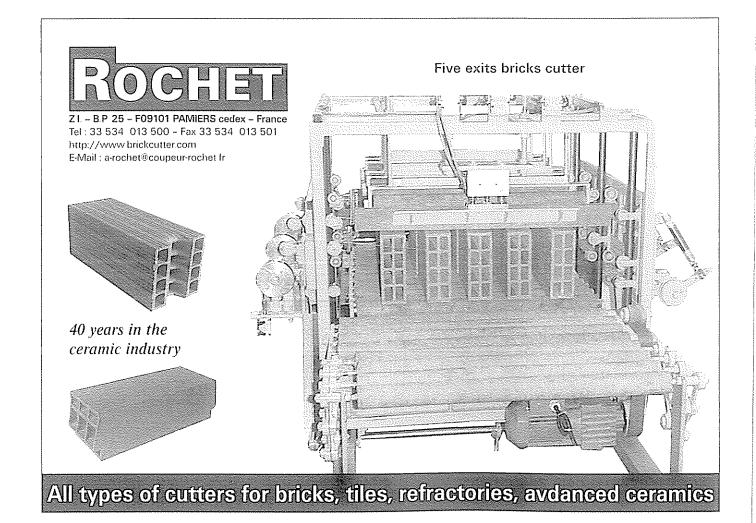
this process has been parameterised, assuming there are two simultaneous, opposing mechanisms, with different kinetics

Theoretical analyses indicate that only two factors can produce delayed curvatures: residual stresses and expansion of the tile body. In both cases, additional circumstances need to occur for delayed curvatures to appear; thus, for example, the presence of residual stresses is not synonymous with delayed curvatures, but an additional mechanism is needed that allows progressive release of these stresses, a mechanism known as creep. In addition, the condition that the stress profile is

not symmetrical with respect to the centre plane of the tile also needs to be obeyed.

In regard to the expansion of the bodies, special conditions must also occur for these to cause delayed curvatures. In particular, it is necessary for the expansion at the tile fair face and at the rib face to be different. Uniform expansion would cause a slight dimensional change, but not a delayed curvature, not even in the presence of glaze

The measurement of the factors that influence delayed curvatures is complex because different techniques from those typically used in characterising ceramic tiles are required.



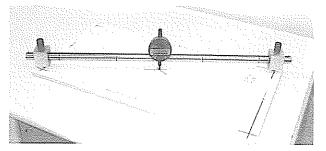


Fig. 1 Arrangement used to measure the deflection of industrial tiles

Procedures have been fine-tuned to measure the different factors that give rise to delayed curvatures, and the influence of certain variables on these factors has been studied

Introduction

A significant percentage of ceramics tiles, independently of their porosity, exhibit the phenomenon known as 'delayed curvatures' These consist of the change in tile curvature after the tile exits the kiln over a period of time that may take several weeks or even months

Although delayed curvatures are not unknown in the ceramic sector, they pose a serious manufacturing problem owing to the present larger tile sizes. Thus, the presence of delayed curvatures leads to problems in the sorting stage and in final product quality, thus it is necessary to know the final curvature that the tile will develop in order to establish with what curvature the tile needs to leave the kiln, as well as the criteria for tile classification

The delayed curvatures in porous wall tiles or earthenware tiles display a trend towards concavity with time, possibly as a result of the expansion of the bodies due to their high porosity (which allows water to penetrate), and to the presence of phases that expand when the body hydrates [1,2]. In both glazed and unglazed porcelain tile, such behaviour is particularly surprising, because the curvature displays an evolution in one direction (customarily in a concave direction) which, after a certain time, usually reverses direction The change in the evolution of the curvature, together with the very low porosity of porcelain tiles (water absorption below 0,5 %) and the low quantity of hydratable phases present in the fired tile, indicates that the cause of this behaviour is much more complex

In this study the factors that can influence delayed curvature in porcelain tiles, either directly or indirectly, have been examined. Although much work still remains to be done in the field of delayed curvatures, the results obtained lay the groundwork for a systematic study of delayed curvatures

Measurement of the **Evolution of Curvature with Time**

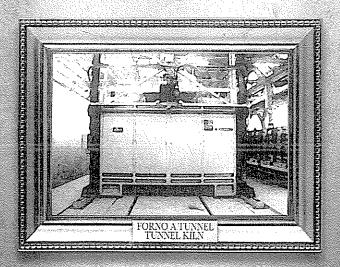
The first step in the study of delayed curvatures in porcelain tile was the quantification of the magnitude and kinetics of the delayed curvatures

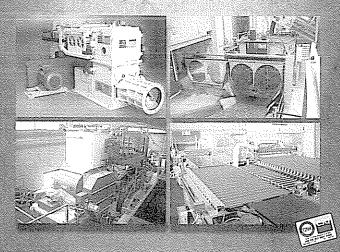
The tests consisted of measuring the deflection in the centre of the piece (δ_c) at different moments, using a dial gauge set on a stand with three supports (Fig. 1) This deflection was determined for each diagonal and the results were then averaged. It is important to begin making these measurements immediately after firing in the industrial kiln, since that is when the curvature usually evolves fastest.

Fig. 2 shows the evolution of curvature with time over a period of 13 days for the glazed porcelain tile of 450x450 mm² of the same model (Model-1) using two different body com-



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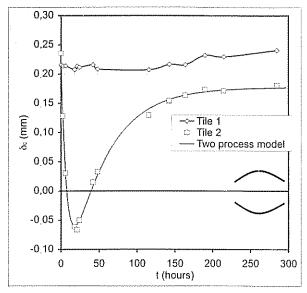


Fig. 2 Evolution of deflection in industrial tiles (Model 1, Tile 2)

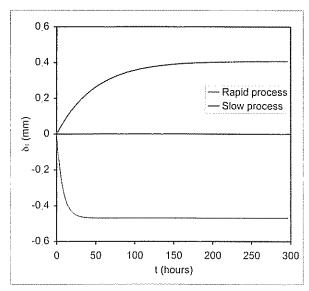
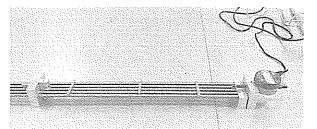


Fig. 3 Contribution of both processes (Model 1, Tile 2)

whereas the pieces formed from spray-dried powder 1 (Tile 1) leave the kiln with a convex curvature (0,22 deflection), which practically remains invariable with time, the curvature of the piece obtained from spray-dried powder 2 (Tile 2) displays an important variation with time. Thus, the pieces exhibit a convex curvature of 0,24 mm at the kiln exit, which decreases quickly and

positions. It may be observed that





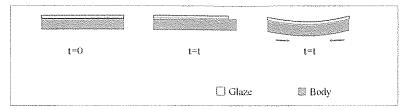


Fig. 4 Schematic illustration of the delayed curvature produced by expansion of the body

becomes a concave curvature after 20 hours (δ_{c} =-0,06 mm). After this time, the curvature of the piece evolves more slowly in the opposite direction until it practically stabilises after 8 10 days (δ_{c} =0,17 mm)

The behaviour described is that typically observed in glazed porcelain tiles, in which the magnitude of the deflection, and the speed at which this evolves, change as a function of a multitude of factors such as type of model, spray-dried powder, firing cycle, peak temperature, environmental conditions, etc.

The foregoing evolution of curvature with time suggests that the tile is subject to two opposite phenomena: a rapid one that tends to decrease the deflection and another slower one than tends to increase it. Thus, the overall evolution of the curvature may be considered due to these two phenomena. Mathematically this decomposition can be written in the following form:

$$\delta_{i} = \Lambda_{i}(1 - e^{-i \cdot t}) + \Lambda_{i}(1 - e^{-i \cdot t})$$
 (1)

Where the term $A_1(1-\exp[-t/\tau_1])$ corresponds to the first process and $A_2(1-\exp[-t/\tau_2])$ to the second. Parameter τ is a time constant related to process kinetics, whereas A indicates the strain deflection that will be reached in each process. This equation is represented for Tile 2 (red line) in Fig. 2. It may be observed that the proposed equation suitably describes the change in deflection with time. Fig. 3 illustrates the contribution of each phenomenon in the overall process.

The decomposition of the curvature evolution in two processes does not provide an indication of the originating causes; it is simply a quantification and parameterisation of the process, which may simplify the analysis of the influence that the different variables involved may have

Factors that Influence Delayed Curvatures

The factors that influence delayed curvatures in tiles may be divided

into direct and indirect factors. The former are characterised as being the ultimate causes of the delayed curvatures; the latter can affect the delayed curvatures either because they act on some direct factor, or because they modify the evolution of the delayed curvature (though they do not generate it)

Direct Factors

To modify in a fired tile its curvature with time it is necessary to make some modifications, as it can occur by the expansion of the body of a glazed tile, or if a force is applied to the tile. This last case can take place when relaxation occurs of the residual stresses present in the tile.

Thus there are basically two factors that can cause delayed curvatures: expansion of the bodies and relaxation of the residual stresses. The remaining factors are indirect

Expansion of the Bodies

In a glazed tile, the expansion of the body may lead to the appearance of delayed curvatures, as illustrated in Fig 4. The mechanism is very similar to the change in curvature that occurs during cooling in the kiln, owing to the fit between the glaze and body layers as a result of the difference in shrinkage between both layers. In this case, the expansion of the body will lead to a concave curvature, assuming that the piece is initially flat and that the glaze practically does not expand

Once one of the factors that can cause the delayed curvature had been identified, it was necessary to verify whether porcelain tile bodies expanded Although it is well known that earthenware tile bodies display appreciable moisture expansion, which may lead to problems of glaze crazing under limit conditions, this property is usually not measured in porcelain tile bodies, owing to the inexistence of crazing defects in these products and to their low porosity

Measurement Procedure

Since delayed curvature needs to be observed starting immediately after the pieces leave the kiln, it was nec-

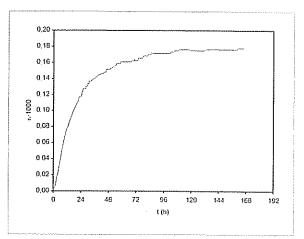


Fig. 6 Evolution of the expansion of a porcelain tile body as a function of time

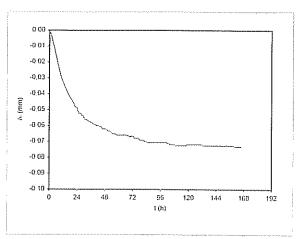


Fig 7 Delayed curvature caused by expansion of the body

essary to design and fine-tune a test procedure that would enable the determination of body expansion during this period of time. The main difficulty posed in the performance of this measurement was the temperature variation during the test, which might considerably affect measurement accuracy. In order to address these difficulties a device was built, shown in Fig. 5, which is able to offset automatically the temperature changes that the hot tile induces

Results

The evolution of the expansion of a porcelain tile body, starting when the tile exits the industrial kiln, is shown in Fig. 6. It may be observed that in the initial moments the expansion is very fast; it then stabilises after 96 hours at a value of about 0,18 %.. Though the result depends on the composition used and on the peak firing temperature, it may be considered representative for these types of bodies. In comparison, the expansion of white-firing earthenware tile bodies usually lies at about 0,35 ‰, which only differs a factor of 2 with respect to that of porcelain tiles

The expansion of the bodies, of itself, is insufficient to explain the delayed curvatures. As indicated previously, the presence of another layer that does not deform is required.

This role could be played by the glaze layer in glazed products; however, there are also unglazed porcelain tiles that exhibit the problem of delayed curvatures. On the other hand, the glaze coating on a porcelain tile is usually thin (about 100 µm), which is why the resulting curvature is also small. Fig. 7 shows the curvature deflection that a 410x410 mm² tile will display, calculated from the typical expansion of a porcelain tile body, and considering the fit with the glaze. It may be observed that the expansion of the body is not sufficient, in general, to explain the delayed curvatures, though it can play a certain role in some cases.

In the foregoing, it has been assumed that the expansion of the body is uniform throughout the thickness of the body. If there was a difference between the expansion of the body at the fair face and at the rear face, part of the delayed curvature could be explained in both glazed and unglazed products Thus, for example, a difference of 0,1 % in the expansion of a 410x410 mm² size tile could cause a delayed curvature, expressed as a deflection, of about 0,3 mm Further, even though the total expansion of both faces was the same, a difference in the expansion kinetics could cause changes in the direction

of the curvature similar to those observed in industrial tiles. Although there are indications that suggest this difference exists, there are no conclusive results yet on this point Relaxation of Residual Stresses

One of the hypotheses used to explain delayed curvatures is the presence of residual stresses in the tiles. If these stresses are relaxed by some process, such as creep, they could lead to variations in tile curvature

However, the existence of a residual stress profile is not synonymous with delayed curvatures. This profile also needs to be asymmetrical and there needs to be creep. In regard to profile asymmetry, it may be noted that

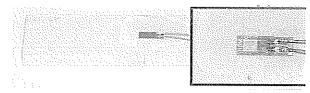


Fig. 8 Residual stress measurement test and detail of the gauge

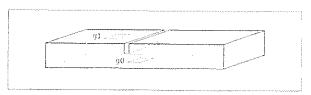


Fig. 10 Location of the gauges on the bottom (g_0) and top surface (g_1)

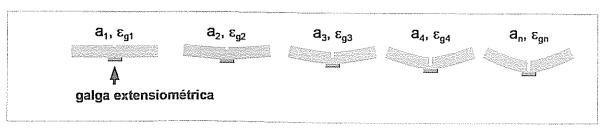


Fig 9 Schematic illustration of the principle used to measure the residual stresses

-- Model - 1 --- Model - 2 -10 -08 -06 -0,4 -02 00 02 0,4 05 08

Fig 11 Experimental strain and fit for a porcelain tile

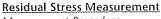
Fig. 12 Residual stress profiles of Model-1 and Model-2

a symmetrical profile means that the stress is the same on the fair face as on the rear face, so that the piece will not tend to curve towards either of the two sides.

The existence of residual stresses in ceramic tiles may be due to two causes: stresses caused by rapid cooling of the tile in the industrial kiln, associated with thermal gradi-

ents in the piece, and stresses produced by the glaze/body fit The former have been measured in this study, since they are customarily of a greater magnitude than those stemming from the glaze fit. That is because the glaze is usually much thinner than the body and because the body's modulus of elasticity is relatively high compared with that of the glaze.

Positival Stress Measurement



Measurement Procedure

The residual stresses in the body stemming from the thermal gradients were measured using the strain relaxation slotting method (SRSM) This method consists of fixing a gauge to the bottom of the unglazed piece (Fig. 8), and then making increasingly deep cuts (a_i) from the top surface, measuring the strain recorded by the gauge (ε_g) (Fig. 9).

In order to be able to calculate the residual stress profile it is necessary to know the relation between $\epsilon_g(a_i)$ and the stress at each point inside the piece. This relation is determined by certain calibration factors which need to be theoretically calculated for each geometry by a numerical method (in this case the finite element method was used) Finally, once the calibration factors have been determined, it is possible to calculate the stress profile

With a view to improving the accuracy of the method, two strain gauges are sometimes used, one on the bottom and the other on the top surface, located close to the notch area (Fig. 10)

Results

Fig 11 shows the strain measurement values of both gauges (rhombuses and triangles) and the fitting curve obtained from the finite element calculation. The analysed piece was a tile fired under industrial conditions. It may be observed that as notch depth (a) increases, the strain measured by the two gauges increases. The top gauge is very sensitive at the beginning of the cut (low values of a), but as the notch depth increases, this gauge quickly loses its sensitivity and the bottom gauge becomes more relevant.

The data in Fig. 11 allowed the residual stress profiles of two models of glazed porcelain tile to be determined (Model 1 and Model 2), displayed in Fig 12. The value ζ =-1 corresponds to the bottom surface and ζ =+1 to the top surface (fair face). In both cases the profile exhibits negative stresses (compression) at the surface and positive stresses (tension) in the centre. This is the typical profile of a tempered material

Model 1 had stresses similar to those of Model 2 in the bottom half of the piece, but slightly larger ones in the top half; generally speaking, it may be said that Model 1 was slightly more stressed than Model 2, and its profile was more symmetrical. Experimentally it was verified that Model 1 did not tend to display any problems of delayed curvatures; whereas Model 2 did. This result appears to agree with what has been indicated previously regarding the relation between stress profile symmetry and the delayed curvatures. In any event, in order to establish definitive conclusions it would be necessary to proceed further with the measurements, analysing a greater number of tiles and models.

Creep Measurement

Residual stresses, by themselves, do not cause delayed curvatures, even when they are highly asymmetric. It is necessary for there to be a mechanism that causes the tile to release

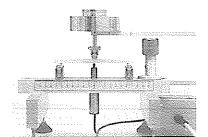


Fig. 13 Device used to measure creep

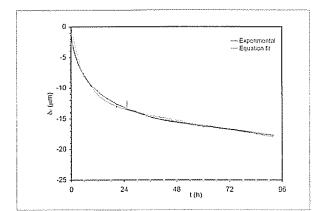


Fig. 14 Strain of a porcelain tile test piece under loading

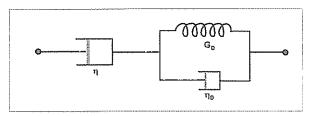


Fig.15 Linear viscoelastic model used to model creep

these stresses This mechanism is known as creep and, just as in the case of the residual stresses, it was necessary to verify whether porcelain tiles displayed this behaviour Measurement Procedure

The device shown in Fig 13 was used to measure creep. The device consists of a bending bar on which the test piece is placed. A constant load is then applied, by means of masses, and test piece strain is recorded using a displacement measurement instrument (linear variable differential transformer or LVDT) Results

Fig. 14 displays the results obtained It shows that the strain is not zero, which indicates there is a certain creep In addition, two sections may be distinguished: an initial one, in which the strain rate decreases (primary creep), and a second one in which the slope is constant (secondary creep) This behaviour can be modelled using the viscoelastic model shown in Fig. 15. This model allows the following relation between deflection and time to be established:

$$\delta_{\rm P} = \frac{S^2 \sigma_{\rm m}}{18 \text{Im}} \left(\tau_{\rm D} \left(1 - e^{-\tau/\tau} \right) + 1 \right) \tag{2}$$

where:

σ_m: Maximum applied stress (Pa)

h: Test piece thickness

η: Viscosity (Fig 15) (Pas)

η_D: Viscosity (sFig. 15) (Pa-s)

G_D: Shear modulus (Pa)

 τ_D : Quotient D/GD (s)

τ: Time constant indicating the process kinetics

The result of the application of Eq. 2 is presented in Fig. 14, which shows that the equation is able to reproduce the experimental results correctly

Indirect Factors

The characteristics of the materials used in the manufacture of porcelain tiles (spray-dried powders, glazes etc), together with the process variables used, define ceramic tile characteristics, as illustrated in the scheme of Fig 16 Although these factors are not directly responsible for the delayed curvatures, they can in some cases have an enormous influence on them This section analyses these factors first, and then presents some examples of the influence they have on both delayed curvature and the factors that originate delayed curvature.

Factors that Affect Expansion of the Body

The expansion of ceramic bodies caused by humidity is a well-known characteristic, and methods are provided in the applicable standards to measure this The values of expansion by humidity in an autoclave, with water vapour pressure cycles of 5 at 10 bar, usually range from 0,6 to 1,0 % for earthenware tile bodies, and drop to values below 0,3 % for tiles with low water absorption

The moisture expansion of porous ceramic products (earthenware and porcelain) has been widely studied owing to its relation to glaze crazing defects. In these products, moisture expansion of the ceramic bodies stems from the physical and chemical adsorption of water molecules on the free valencies existing in the hydratable phases (glassy phases and, in particular, amorphous phases from clay mineral dehydroxylation) present in the fired pieces [3]. For this reason, expansion mainly depends on the porous structure of the piece (which defines its greater or lesser accessibility by water) [1]

and on the nature and content of the phases present in the fired piece [2]. These characteristics are highly influenced by the mineralogical composition of the composition used, its particle size and the firing schedule [4]. Thus, as the fusibility of the body composition and the firing temperature or residence time increase, the moisture expansion of these bodies decreases owing to their reduced porosity and hydratable phase content [5]

However, no studies have been found on moisture expansion in bodies with very low porosity, such as porcelain tiles, probably because of the smaller expansion and absence of crazing in these products Although the objective of this study was not to determine the reason for the expansion of porcelain tile bodies after they left the kiln (moisture adsorption or other reasons), the factors that influence moisture expansion in porous bodies are also likely to do so in the case of porcelain tile bodies.

<u>Factors that Affect Residual</u> <u>Stresses</u>

When peak firing temperature is reached, the porcelain tile is made up of a great quantity of liquid phase, quartz and residual albite, and sometimes mullite [6] In this state, the piece is able to relax any stress that is applied on it, since it is highly deformable. It is during the cooling phase that residual stresses develop in the tiles, either because of a mismatch in the fit between the body and glaze layers, or because of differential shrinkage as a result of the greater cooling rate in the outer areas compared with the centre of the piece. Thus, the factors that determine residual stresses in porcelain tiles are basically thermal expansion and modulus of elasticity of the

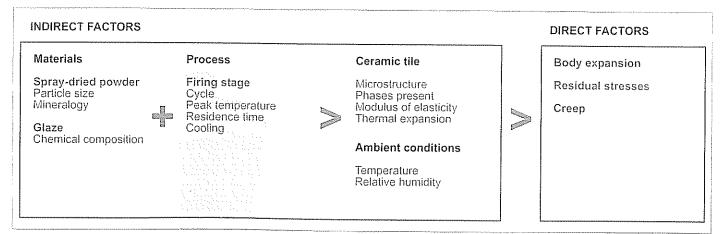
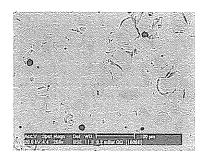


Fig 16 Factors that influence delayed curvatures

Fig. 17 Cross-section of a porcelain tile



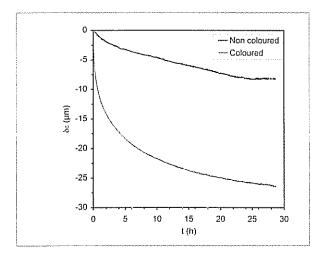


Fig. 18 Influence of dry colouring on creep

body and the glaze, their relative thicknesses, and the tile cooling rate. It has been observed that a greater mismatch of the thermal expansion of both layers, as well as a greater glaze thickness, raises residual stresses Furthermore, when cooling is faster, the temperature gradient increases inside the tile [7], leading to different shrinkage rates throughout the tile cross-section. This produces a stress profile inside the piece that becomes more pronounced as the cooling rate increases [8]. In addition, when the cooling rate is not the same at both tile faces (the usual situation in roller kilns), the resulting stress profile is not symmetrical, as set out further below.

Factors that influence creep

The factors that influence creep in porcelain tiles must be associated with the tile microstructure Fig 17 presents a cross-section of a porcelain tile observed by scanning electron microscopy (SEM) It consists of a glassy matrix containing numerous pores and microcracks. These pores are usually round and stem from the decomposition of certain impurities as well as from large pores present in the green piece [9]. The origin of the microcracks varies, though they occur in particular as a result of the mismatches between the residual quartz particles and the glassy matrix [8], and the junctures of spray-dried powder granules that were not fully deformed during pressing [10]

Creep in porcelain tile pieces is highly likely to be caused by growth of the existing microcracks in the tile when the tile is subjected to a tensile stress, so that the factors originating these microcracks will determine creep in these types of tiles. It has been verified that an increase in quartz content as well as in particle size produces a microstructure with a larger quantity of microcracks, which will probably increase tile creep. This can occur e.g. when the composition has been insufficiently milled.

Influence of Dry Colouring on Creep

Creep tests were conducted on test pieces cut from two types of industrial fired glazed porcelain tiles: these were fabricated using either a coloured spray-dried powder, or a non-coloured spray-dried powder. The test pieces measured 220x20 mm² and were machined to eliminate the rear rib. The average thickness of these test pieces was 6,4 mm and the applied load was 29 MPa

Fig 18 shows that both test pieces undergo strain (creep) with time, which is very rapid at the beginning of the test and progressively stabilises with time. It may be observed that the piece with pigment displays a considerably larger strain than the non-pigmented piece, indicating that the capacity to deform under a given stress is much greater for the pigmented pieces

Although this study has not examined the origin of this difference, the reason that the coloured piece exhibits greater creep can be related to the microstructure of this type of tile, in which the pigment particles are concentrated in certain areas of the piece (the contact areas between the spray-dried powder granules) [11] These regions can act as microcrack-initiating flaws when larger percentages of pigment are used, whose growth under the application of a particular stress could originate the greater creep observed

These results do not allow it to be directly concluded that the dry-coloured pieces will display greater problems in regard to delayed curvatures, since that depends on the other factors involved. Indeed, there are coloured models that exhibit no delayed curvatures. However, with the existence of asymmetric residual stresses in the tiles, and the other factors being equal, the tiles that present the greatest creep are more likely to change their curvature with time.

Influence of the Presence of Glaze on Delayed Curvature

Fig. 19 presents the evolution of the deflection with time for glazed porcelain tiles, size 300x600 mm², with the dry-coloured body, with and without the presence of glaze. It shows that while the tile with the glaze displays a qualitatively similar

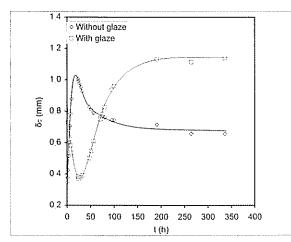


Fig 19 Evolution of tile deflection with and without glaze

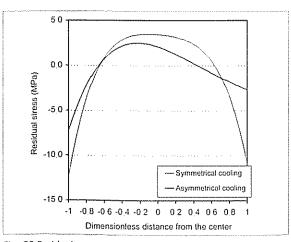


Fig. 20 Residual stress profile as a function of type of cooling

curvature evolution to that shown in section 2, the body without the glaze evolves in the opposite direction. It should be noted that the time at which the minimum curvature in the glazed piece is observed (24 h, 0,37 mm²) practically coincides with time at which the maximum curvature is detected in the tile without the glaze (23 h, 1,02 mm²).

This behaviour may be due to the existence of different rates of expansion between the fair face and the rear surface, since the tiles have been fired with the same cycle and, therefore, the residual stresses caused by cooling should be similar

This same behaviour was also observed in pieces of unglazed porcelain tiles, which seems to indicate the enormous influence of the glaze on the evolution of curvature with time

Influence of the Cooling Stage

The cooling stage in industrial roller kilns typically has a first rapid-cooling section by injection of air at ambient temperature into the kiln. This type of cooling produces high temperature gradients between the surface and the inner part of the tile, which leads to residual stresses that can originate delayed curvatures.

This part of the study examined the influence of cooling on residual stresses in the body. It was conducted using test pieces of 150x30 mm² cut from green industrial glazed porcelain tiles, which were fired in an electric laboratory kiln at 1200°C for 6 min and cooled outside the kiln (air quenching) in two different ways:

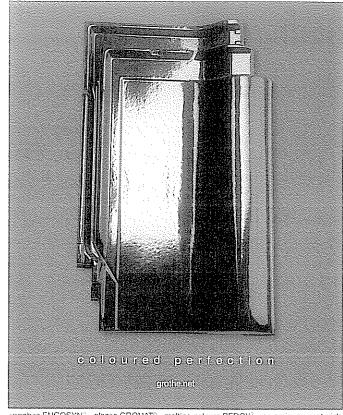
- Symmetrical cooling: with the piece in a vertical position. Both sides of the piece were identically cooled
- Asymmetrical cooling: with the piece placed horizontally on a refractory slab. In this case, cooling basically took place through the top

The stress profiles of the two test pieces cooled in different ways are shown in Fig. 20. It may be observed that in both cases the two surfaces of the pieces are subjected to a compressive stress, whereas the inside of the piece is under tension, which is the usual situation in ceramic materials. Cooling is also observed to have a considerable influence on the stress profile. Thus, when the test piece is identically cooled at both faces (piece cooled in a vertical position), the stress profile is symmetrical, whereas the piece cooled in a horizontal position on refractory slab (asymmetric cooling) displays a non-symmetrical profile. These results confirm that the differences in the cooling rate through the top and bottom of the piece can lead to asymmetrical stress profiles.

Conclusions

This study has examined the factors that may influence delayed curvatures in porcelain tiles and allows the following conclusions to be drawn:

- The variation of curvature with time in industrial porcelain tiles has been quantified, it being observed that there is a change in curvature trend (concaveconvex) This means that there are two simultaneous, opposing mechanisms, with different kinetics, which influence the process
- The theoretical analyses indicate that there are just two factors that can cause the delayed curvatures: residual expansion of the bodies, and stresses. In both cases, it is necessary for additional circumstances to be present for delayed curvatures to appear; thus, expansion needs to be different at the fair face and at the rear face. Uniform expansion would cause a slight dimensional change, but not significant delayed cur-



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- vature, not even in the presence of glaze. In regard to the residual stresses, the stress profile needs to be asymmetrical and there needs to a mechanism that allows their release (creep).
- The measurement of the factors that influence delayed curvatures is complex because it usually requires use of other techniques than those typically employed in characterising ceramic tiles Procedures have been fine-tuned in the study to measure the different factors that produce delayed curvatures A device has been designed that automatically offsets temperature for measuring the expansion of the bodies when they leave the kiln; the strain relaxation slotting method (SRSM) has been applied to measure the residual stresses in porcelain tiles, and 3-point bending tests with constant loading for
- several days have been used to verify the presence of creep in porcelain tile bodies
- It has been verified that porcelain tile bodies undergo expansion when they exit the kiln. These bodies also display residual stresses and creep, which could explain the existence of delayed curvatures.
- Experiments have been conducted modifying the type of spray-dried powder, eliminating the glaze, and modifying the cooling. These experiments have provided initial information on the influence of the characteristics of the material and of the manufacturing process on creep, residual stresses, and delayed curvature
- Though much remains to be done in the field of delayed curvatures, the results obtained lay the groundwork for a systematic study of delayed curvatures.

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