RESIDUAL STRESSES IN PORCELAIN TILES FORMED BY TWO-CHARGE PRESSING

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ABSTRACT

The two-charge pressing technique used in making certain types of porcelain tile adds an additional series of demands to the process, especially if the compositions making up the two layers exhibit very different dilatometric curves. In these cases, problems of curvature or residual stresses can appear, which can hinder cutting for tile installation.

It was found in this study, that the difference in thermal expansion between the top layer or second charge, and the bottom or base layer, was the cause of residual stresses. Analysis of the stress profile of a two-layer tile showed that on subjecting the tile to point impact or stress, failure tended to occur at a certain distance from the surface, coinciding with the interface between the two layers.

A simple method is proposed for assessing residual stresses in two-charge porcelain tile based on the determination of mechanical strength by three-point bending. This is done by calculating the difference in mechanical strength between a tile subjected to the test at its proper face (normal position) and another tile subjected to the test at the rib face (inverted position). A mathematical expression derived from the Timoshenko equation then enables the residual stress to be evaluated in the top layer-base layer interface (where failure occurs) from the difference in mechanical strength found by the proposed procedure.

The derived model was used to verify the influence of the cooling and thermal expansion rate of the two layers on residual stresses, and to obtain a composition that minimises these stresses. The laboratory study was compared with the data obtained with industrial tiles on a semi-industrial scale.

1. INTRODUCTION

From a strictly technical point of view, porcelain tile is a whiteware floor tile of very low porosity (less than 0.5% water absorption), traditionally decorated by colouring the body by adding pigments, which is generally polished to enhance tile gloss.

The strong growth of porcelain tile, not just in Spain but also worldwide, has led to an important rise in the range of products and finishes that can be included in this type of tile. Thus, the limited range of models marketed in Spain about a decade ago, such as the well-known plain-coloured and granite or "salt and pepper", polished and unpolished porcelain tiles, has been broadened by decorations with soluble stains and more recently, with finishes produced in the press itself by combining different feeding systems with simultaneous or successive powder charges [1].

One of these decorating systems, popularly known as the "two-charge" system, consists of a first press charge with a base spray-dried powder, which is then covered by a second charge comprising a mixture of the base powder with one or more spray-dried, micronised, granulated, pigmented or unpigmented powders. This technique, which varies in terms of the mechanical feeding facilities and characteristics of the pressing powders used, yields a great variety of aesthetic effects such as veining, shading, stratification, etc.

This combination of pressing powders with such different characteristics can sometimes cause certain faults to appear in the fired tile stemming from the different characteristics or properties of the layers of powder used. It has been repeatedly found in industry, that porcelain tile made by the two-charge system is more prone to curving during firing, breaks more easily during polishing and, when cut for tile installation, gives rise to a greater number of defective tiles as a result of greater fragility in cutting.

The residual stresses arising in a product made by combining layers of different thickness and nature are, just as in glazed tiles, the main source of some of these faults [2]. The stresses have different origins: on the one hand there are the layer characteristics (thickness, thermal expansion, Young's modulus, thermal diffusivity, etc.), and on the other the tile cooling cycle. Finally, as some authors have indicated [3][4], the polishing process can also contribute to raising residual stresses, especially at the tile surface.

Various methods are found in the literature for assessing residual stresses in materials and structures [5-9], however, their application to ceramic tiles is very restricted, as excessively complicated techniques are involved for use in quality control.

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[1] FERRER, C. *Posibilidades decorativas del gres porcelánico.* II Jornadas en Técnicas decorativas y tratamientos superficiales. Castellón, 23 y 24 de Junio de 1999 (Conferencia presentada en las Jornadas).

[2] BELDA, A.; BLASCO, A.; MONTIEL, E; ORTS, M.J. *Glaze support adhesion and its industrial control.* Tile Brick Int., 7(1), 15-22, 1991.

[3] SAMUEL, R.; CHANDRASEKAR, S.; FARRIS, T.N.; LICHT, R.H. *Effect of residual stresses on the fracture of ground ceramics.* J. Am. Ceram. Soc., 72(10), 1960-66, 1989.

[4] TUAN, W.H.; KUO, J.C. *Contribution of residual stress to the strength of abrasive ground alumina.* J. Eur. Ceram. Soc., 19, 1593-97, 1999.

Some of these methods are as follows: incorporating strain gauges in holes bored in the piece, X-ray diffraction, photoelasticity, etc. The indentation technique can also be used for determining residual stresses. This last technique is simple, but its point character limits its application to homogeneous materials such as metals, plastics, glasses, etc., and not to polyphase materials such as ceramic tiles. Finally, there is the Steger method, which is widely used to determine stresses in tile [2], though the characteristics of the equipment do not allow it to be used with laboratory test specimens. Moreover, in the case of porcelain tile, it entails the added disadvantage of the complexity of the machining required to be able to fit the item into the measuring equipment, given the high mechanical resistance and hardness of the product.

2. STRESS DISTRIBUTION IN TWO-LAYER PORCELAIN TILE

During thermal treatment, both in heating and cooling, stresses develop between the top and bottom layer of the body. If the thermal expansion of the base layer exceeds that of the top layer, which is the usual case in two-charge porcelain tile manufacture, these stresses qualitatively adopt the shape depicted in Figure 1. It shows that compressive stresses are involved in the top layer and bottom region of the base layer, whereas tensile stresses are found in the centre of the tile.

To explain why compressive stress arises in the bottom region of the base layer, it is necessary to consider what occurs to the body during cooling. When the body cools below the temperature at which the solid is considered sufficiently rigid not to undergo permanent deformation (in the glaze-fit this arbitrary temperature is called the effective fitting temperature and is referenced T_a), the greater shrinkage of the body puts it under tensile stress. The tile bends to minimise these stresses, producing a *"curvature-induced stress"* with maximum compression in the bottom region (σ<0) and maximum tension in the top region (σ>0), varying linearly inside the body. This *"curvature-induced stress"* is added to the stress associated with the different shrinkage of both layers, yielding the profile shown in the figure.

In practice, the foregoing situation is usually more complicated, owing to the role played by pyroplastic deformation, which is significant in the case of porcelain tile, and the cooling rate of the industrial firing cycle, which can both give rise to tiles exhibiting similar curvature, but with different residual stresses.

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[5] SCHAJER, G.S. *Measurement of non-uniform residual stresses using the hole drilling method.* J. Eng. Mat. Tech., 110, 338-349, 1988.

[6] ASTM E837. *Determining residual stresses by the hole-drilling strain-gage method.*

[7] LANGE, F.; JAMES, M.R.; GREEN, D.J. *Determination of residual surface stresses caused by grinding in polycrystalline Al_2O_3.* J. Am. Ceram. Soc., 66(2), C16-C17, 1983.

[8] BERNAL, E.; KOEPKE, B.G. *Residual stresses in machined MgO crystals.* J. Am. Ceram. Soc., 56(12), 634-39, 1973.

[9] COOK, R.F.; LAWN, B.R.; DABBS, T.P.; CHANTIKUL, B. *Effect of machining damage on the strength of a glass ceramic.* J. Am. Ceram. Soc., 64(9), C121-C122, 1981.

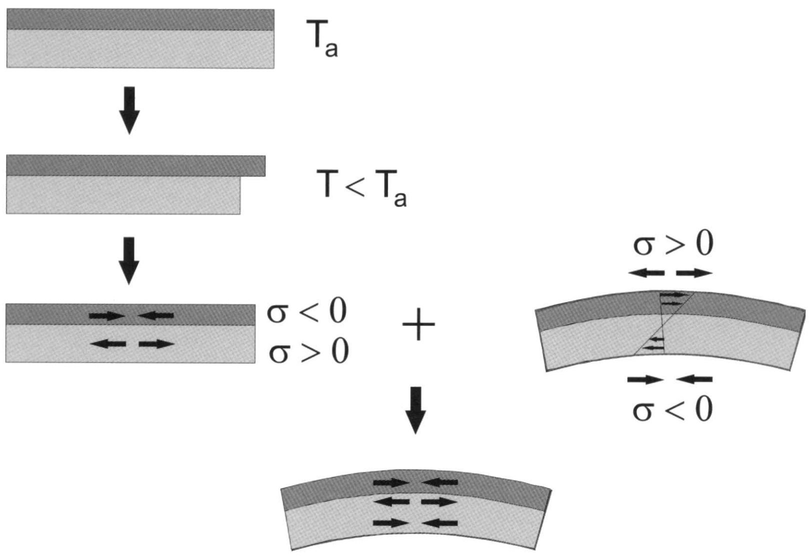

Figure 1. *Origin of stress distribution in the top and bottom layer of two-charge porcelain tile.*

The Timoshenko equation [10] establishes to which stress the base layer is subject (σ_{CB}) and to which the top layer is subject (σ_{CT}), i.e., it allows determining the stress profile. The profile adopts the form defined by Eqs. (1) and (2):

(1)
$$\sigma_{CB} = \left[\frac{K_R}{h}(y - y_0) + \frac{mn}{1+mn}\right] E_{CB}\Delta\varepsilon_f$$

(2)
$$\sigma_{CT} = \left[\frac{K_R}{h}(y - y_0) - \frac{1}{1+mn}\right] nE_{CB}\Delta\varepsilon_f$$

where:

- h: tile thickness (m)
- y_0: position of the neutral axis (m) (position corresponding to the plane dividing the tile in two equal parts, parallel to the proper face, when Young's modulus is the same for the two layers)
- E_{CB}: Young's modulus of the base (kg/cm^2)
- E_{CT}: Young's modulus of the top layer (kg/cm^2)
- n: Young's modulus ratio: E_{CT}/E_{CB}
- h_{CB}: botton layer thickness (m)
- h_{CT}: top layer thickness (m)
- m: thickness ratio: h_{CT}/h_{CB}
- $\Delta\varepsilon_f$: difference in thermal expansion between the top and bottom layer

K_R is a constant that only depends on n and m, and is given by:

(3)
$$K_R = \frac{6(m+1)^2 mn}{m^4n^2 + 4m^3n + 6m^2n + 4mn + 1}$$

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[10] SCHERER, G.H. *Relaxation in glass and composites.* New York: Wiley, 1986. Chap.16: Split Ring Composite (Bimaterial Strip).

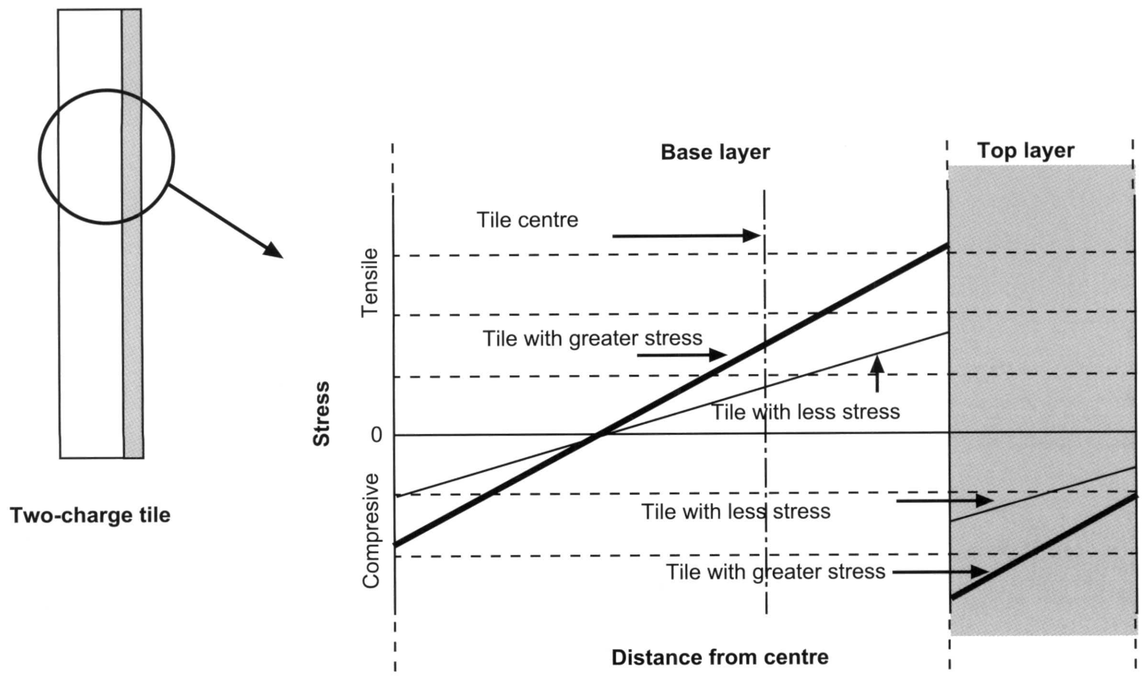

Figure 2. Residual stress distribution in a two-charge porcelain tile.

Figure 2 presents the stress distribution when the base layer shrinks more than the top layer, for two levels of stress: a slightly stressed and a highly stressed tile. The profile is found on applying Eqs. 1 and 2 to the two layers of a two-charge porcelain tile. It can be observed that in general, compressive stress arises in the top layer, which maximises next to the top-base layer interface. The base layer exhibits tensile stress next to the interface and compression on the opposite side.

3. OBJECTIVE

As a result of the foregoing, and in view of the absence of studies in the literature on the determination of stress distribution in ceramic tiles, the present study was undertaken with the following aims. Firstly, fine-tuning a method for determining residual stresses in two-charge porcelain tile. Secondly, relating these stresses to the failures they produce during tile cutting for fixing and finally, designing new compositions to minimise these stresses.

4. EXPERIMENTAL

4.1 MATERIALS

An industrial porcelain tile spray-dried powder consisting of a mixture of illitic-kaolinitic clays and sodium-potassium feldspars was used for the base layer composition. A spray-dried powder was used for the top layer in the two-charge tile, which is employed for this purpose in industry, formulated with illitic-kaolinitic clays, kaolin, sodium-potassium feldspars and white pigment. The samples were respectively referenced B (base or bottom layer) and T (top or second charge layer)

These two industrial compositions were chosen because they had previously been found to yield two-charge porcelain tiles, which gave rise to a considerably higher number of faulty tiles on cutting them for tile installation than conventional porcelain tiles.

4.2 EXPERIMENTAL PROCEDURE

4.2.1 Making the test specimens by two-charge pressing

On conditioning the above two spray-dried powder samples at a moisture content of 5.5% (by weight on dry solid), prism-shaped test specimens measuring 2x8x0.7 cm were formed on a laboratory hydraulic press. The base powder was fed in and scraped flush, then dropping the lower punch. The second powder (T) was subsequently fed in, scraped flush, and the set pressing pressure applied (400 kg/cm^2). The height of both powder samples had been suitably adjusted beforehand to achieve a top/bottom layer thickness ratio in the green specimen of 0.35.

The compacts were oven-dried at 110°C, determining bulk density by the mercury immersion method. The specimens whose bulk density exceeded the mean value by $0.01 g/cm^3$ were discarded.

4.2.2 Characterisation of the resulting test specimens

The dry test specimens were fired in an electric laboratory kiln. The firing cycle used consisted of fast heating up to peak firing temperature with a 6-min hold at peak temperature. Given the influence of cooling on residual stresses, three cooling cycles were tested: fast cooling (F), in which the specimen was withdrawn after ending the relevant peak firing segment, normal firing (N), by forced convection across the whole temperature range except for the stretch between 500-600°C, which was by natural convection (without a fan), and slow cooling (S) just by natural convection. Peak temperatures were tested in the range 1150-1225°C. Figure 3 illustrates the firing cycles used.

The most extensively tested firing cycle was the schedule that included fast cooling, given its greater similarity to industrial conditions. The other cooling rates were tested when it was attempted to determine the effect of cooling rate on residual stresses.

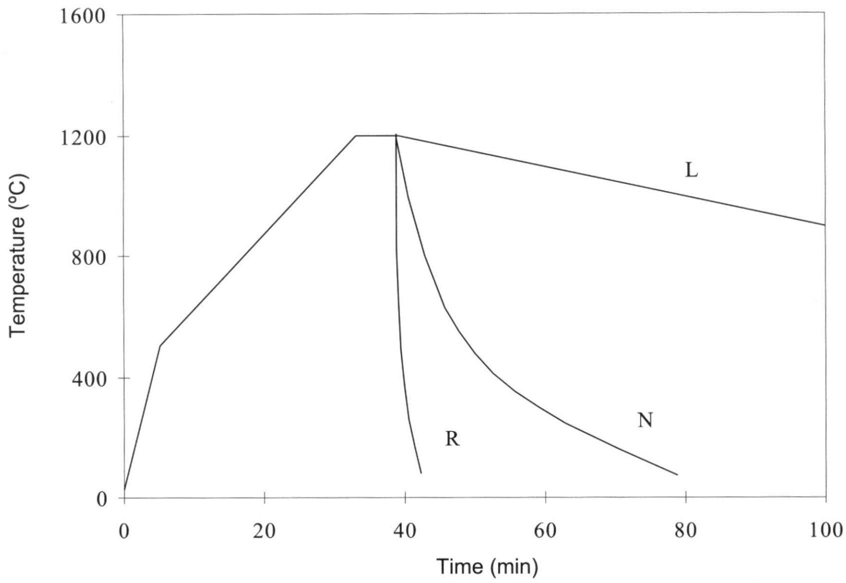

Figure 3. *Firing cycles used for preparing the two-charge test specimens.*

The following tests were run on the fired test specimens: bulk density by mercury immersion, thermal expansion on a dilatometer, mechanical strength and Young's modulus. These last mechanical properties were assessed by 3-point bending, using a universal mechanical testing machine [11]. At least ten specimens were tested to evaluate mechanical strength and Young's modulus, averaging the results.

To separately characterise the resulting two layers, these properties were determined on specimens made with samples B and T, using the same methods as those employed for the two-charge specimens. In this case, the firing cycle with normal cooling (N) was used.

5. RESULTS AND DISCUSSION

5.1 CHARACTERISTICS OF THE TWO LAYERS IN THE TWO-CHARGE TILE

Table 1 details the characteristics of the test specimens made with powder samples B and T fired at 1200ºC, the temperature at which they attained maximum compaction or relative density. The maximum density found for both compositions was 0.91.

Spray-dried powder	σ_R (kg/cm^2)	E (kg/cm^2)$\cdot 10^{-3}$	$\alpha_{(300\text{-}500ºC)} \cdot 10^7$(ºC^{-1})
B	710±20	350±10	94±1
T	740±20	360±10	87±1

Table 1. *Mechanical strength (σ_R), Young's modulus (E), and coefficient of expansion between 300-500ºC ($\alpha_{300\text{-}500ºC}$) of the specimens made with compositions B and T fired at 1200ºC.*

[11] AMORÓS, J.L.; FELIU, C.; GINÉS, F.; AGRAMUNT, J.V. *Resistencia mecánica y microestructura de soportes cerámicos en crudo.* En: IV Congreso Mundial de la Calidad del Azulejo y Pavimento Cerámico, Castellón: QUALICER. 1996, vol.I, pag.153-171.

The table shows that the most significant differences between both samples occurred in the coefficient of expansion values (the specimens made with the base composition exhibited greater thermal expansion), as the mechanical strength and Young's modulus data lie within the measurement scatter itself, so that these differences cannot be considered significant. Hence, as with glazed tiles, the mismatch between layer thermal expansion is the cause of the residual stresses arising in two-charge porcelain tile manufacture.

5.2. DETERMINATION OF RESIDUAL STRESSES IN LABORATORY-SCALE TESTS

5.2.1 Estimation of failure point in a two-charge porcelain tile subject to a point force

On applying a force to a tile, the arising stress will be the sum of the stress from the applied force plus the residual stress. Failure will occur, if at some point, total stress exceeds the material's mechanical strength.

The question is: at which point will the tile fail, when subject to an arbitrary force? The response will depend on the type of stress. Force is often applied at a single point (impact, levering to break a tile, etc.). In this case, a complex stress field is generated, containing several components [12], as shown in Figure 4. σ_z corresponds to the stress according to an axis perpendicular to the surface, σ_r represents the stress according to the direction defined by the straight line that connects the load application point to the point of stress assessment. Finally, σ_θ describes the stress in a direction parallel to the surface and perpendicular to the direction of σ_r application. To simplify stress analysis, the contribution of shear stresses was not considered.

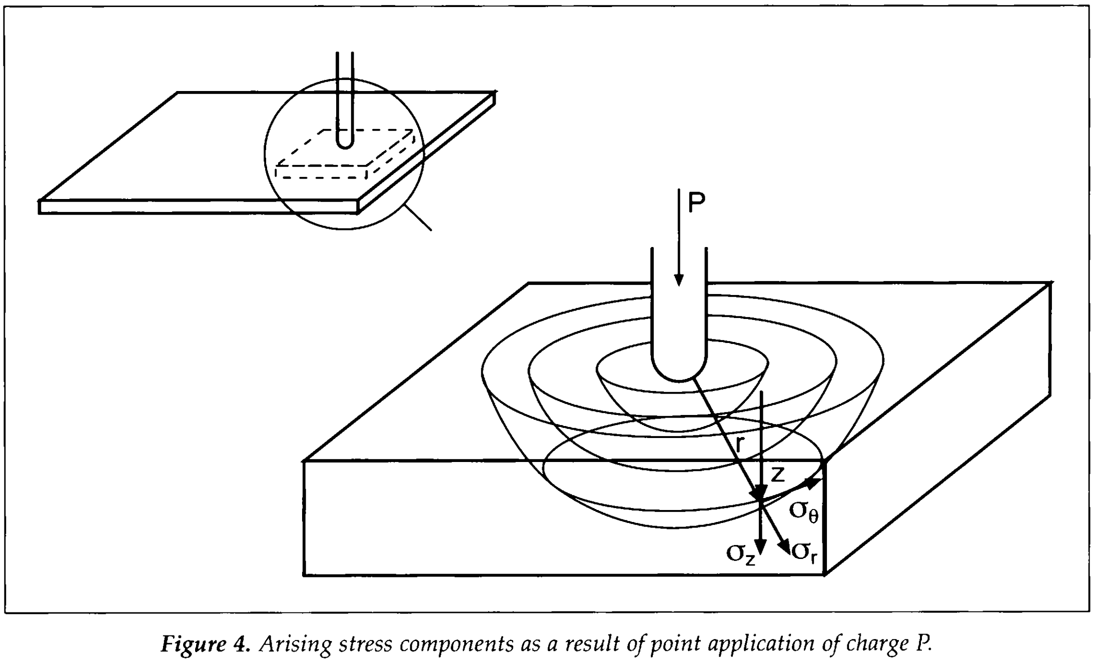

Figure 4. *Arising stress components as a result of point application of charge P.*

[12] TIMOSHENKO, S.; GOODIER, J.N. *Teoría de la elasticidad.* 2ª ed. Bilbao: Urmo, 1975. pag. 394-396.

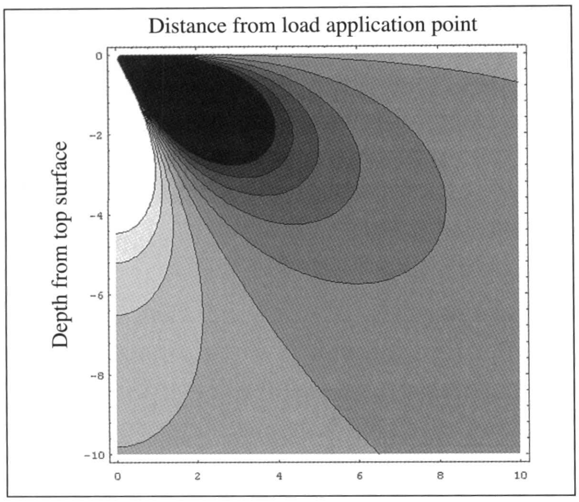

Figure 5.. σ_r as a function of distance from load application point and depth. The darkest points correspond to compression stresses and the lightest ones to tensile stresses.

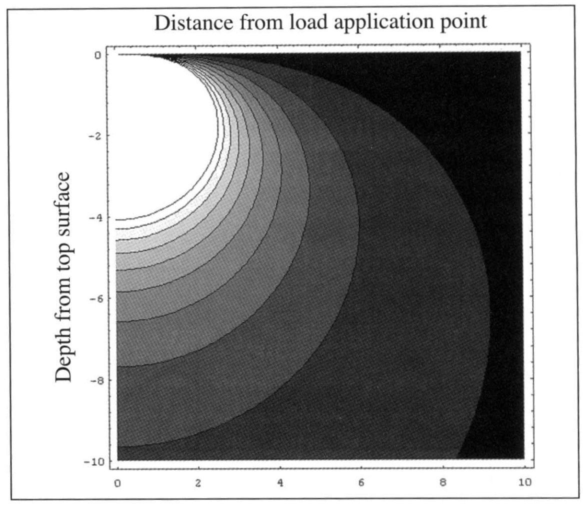

Figure 6. σ_θ as a function of distance from load application point and depth. The lightest points correspond to the highest tensile stresses.

The stress in the z direction (σ_z) is always negative (compressive stress). Taking into account the greater resistance of ceramic materials to compression, it is unlikely that σ_z will cause tile failure. σ_r can be positive as well as negative, depending on the point considered, while the stress in the θ direction (σ_θ) is always positive (tensile stress). Thus, as depicted in Figures 5 and 6, failure can be caused by stress in the r direction or in the θ direction. In either case, the profiles show that stress is zero when z=0. Maximum tensile stress does therefore not occur at the tile surface, but at distance z from the surface.

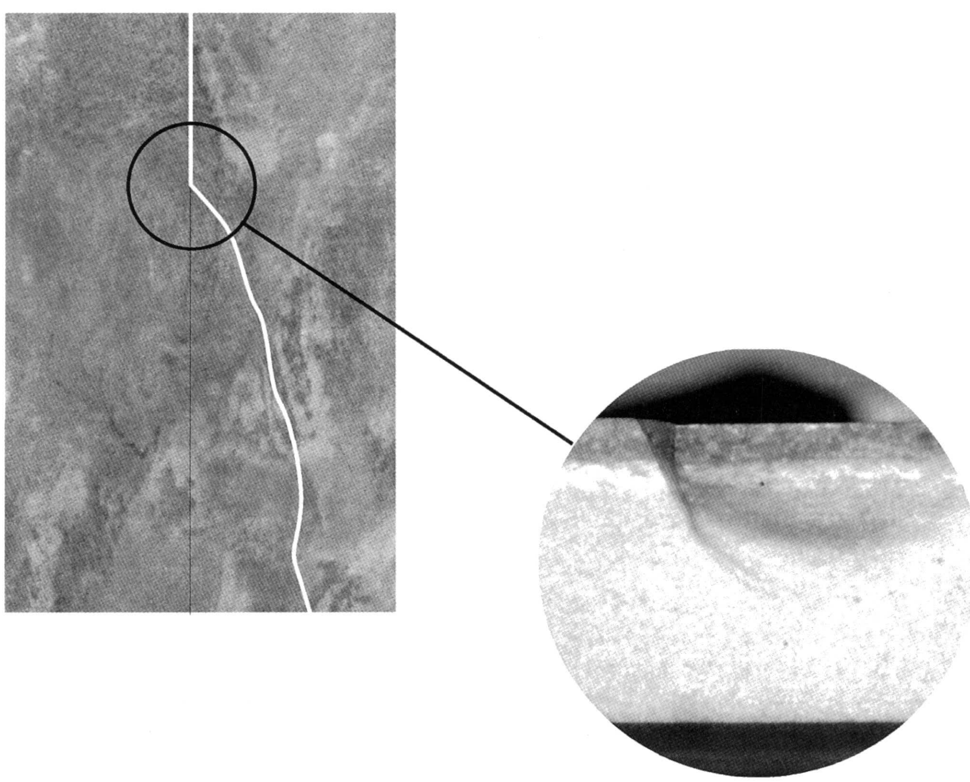

***Figure 7.** Fracture surface of a stressed, two-charge porcelain tile.*

According to the above, when the top layer undergoes impact or tensile stress, as is the case when a levering force is applied to the tile after cutting to separate the pieces, the break can be expected to take place in the interface between the base and top layer, which is where residual tensile stress tends to be greatest (Figure 2) and the tensile stress produced by impact will also be the largest.

To verify this hypothesis, the fracture surface of a two-charge porcelain tile was photographed on a stereoscopic microscope. As Figure 7 shows, the examined area corresponded to the region where the fracture surface deviated from the cutting line made with a diamond-tipped tool. The photograph shows that the most stressed area appears to coincide with the top-bottom layer interface, exhibiting an enormous chipped area in this interface.

5.2.2 Estimation of residual stress

The stress to which the base is subjected next to the interface was estimated by 3-point bending tests. These tests were performed for a single type of tile, with the top layer face up (normal position) and face down (inverted position).

To qualitatively illustrate how it is possible to estimate residual stresses from these tests, it is useful to imagine two pieces (A and B). Piece A exhibits less compressive stress in the top layer than B (Figure 8). If the test is conducted on the inverted piece, raising the load lowers the compressive stress in the top layer (dark layer), as shown in the figure, until the load reaches value p_1. Under these conditions, piece A exhibits zero compressive stress, while this continues to be larger than zero in B. If the applied force is increased, there comes a moment (p2) at which compressive stress in the two-charge layer of B is zero, while this layer is already under tensile stress in A. Finally, at a certain loading value (p3), the stress in A reaches a point at which failure occurs. Thus, "apparent mechanical strength " of piece B is greater than that of A.

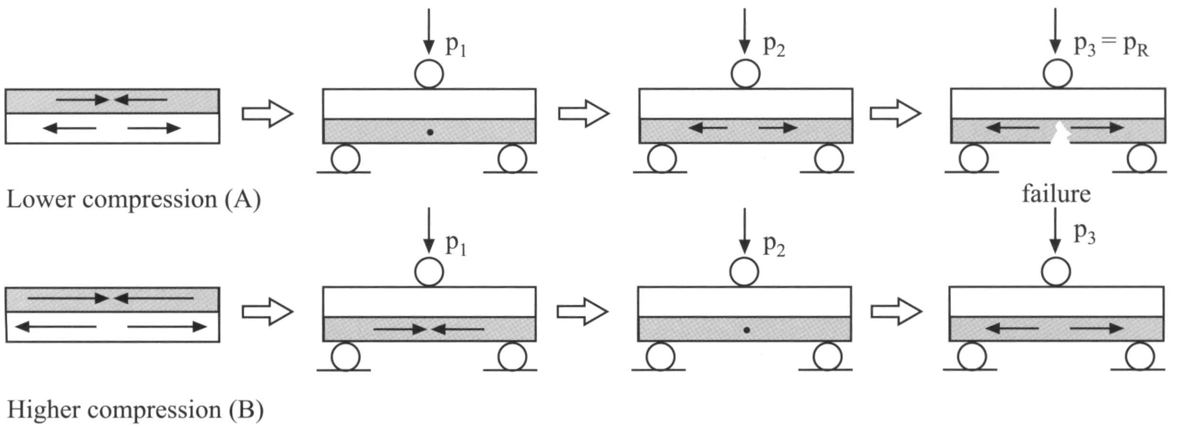

Figure 8. *Illustration of the effect of residual stresses on "apparent mechanical strength" in two-charge porcelain tile with the top layer subject to two levels of residual compressive stress: low (A) and high (B)*

The physical meaning of this apparent mechanical strength is that if there were no residual stresses, apparent mechanical strength and mechanical strength, which is a material property, would coincide. If the residual stress on the top layer is compressive stress, apparent mechanical strength exceeds the material's mechanical strength. When this residual stress is tensile stress, the opposite is true. The figure also highlights an interesting feature: a more highly stressed tile will not always yield lower mechanical strength.

In view of the foregoing, it is of interest to determine the stress in the base, next to the interface with the top layer. This stress, designated σ_{CBi}, which corresponds to the interface, can be calculated from Eq. (1) and a value for y= h_{CB}-h/2, according to the reference system adopted (Figure 9):

(4) $$\sigma_{CBi} = \frac{mn(4+3m+m^3 n)}{m^4 n^2 + 4m^3 n + 6m^2 n + 4mn + 1} E_{CB}\Delta\varepsilon_f$$

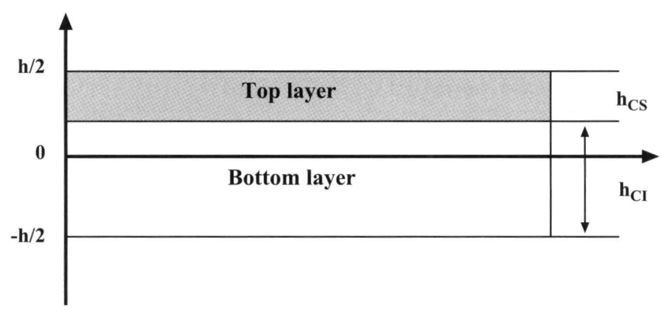

***Figure 9.** Reference system adopted for calculating residual stresses in two-charge porcelain tile.*

When the top layer lies in the normal position (face up), fracture initiates in the base. The maximum stress that the piece can withstand (σ_R: mechanical strength) will be the sum of the "apparent mechanical strength (σ_{RCT})" and residual stress expressed by Eq. (1):

(5) $$\sigma_R = \sigma_{RCT} + \left[\frac{K_R}{h}\left(\frac{-h-y_0}{2}\right) + \frac{mn}{1+mn}\right] E_{CB}\Delta\varepsilon_f$$

Analogously, when the mechanical strength test is performed with the top layer face down (inverted position), fracture initiates in this layer. The maximum stress that the piece can withstand will be σ_R as before, and is the sum of "apparent mechanical strength (σ_{RCB})" and residual stress derived from Eq (2):

(6) $$\sigma_R = \sigma_{RCT} + \left[\frac{K_R}{h}\left(\frac{-h-y_0}{2}\right) + \frac{mn}{1+mn}\right] E_{CB}\Delta\varepsilon_f$$

Eqs. 5 and 6 yield $E_{CB}\Delta\varepsilon_f$:

(7) $$E_{CB}\Delta\varepsilon_f = \frac{m^4 n^2 + 4m^3 n + 6m^2 n + 4mn + 1}{(1+m)n(-m^3 n + 3m^2 n + 3m - 1)} \Delta\sigma_{R(CT-CB)}$$

and replacing this term in Eq. 4 enables establishing the relation between σ_{CBi} and $\Delta\sigma_{R(CT-CB)}$:

(8)
$$\sigma_{CBi} = \frac{m(4+3m+m^3 n)}{(1+m)(-m^3 n+3m^2 n+3m-1)} \Delta\sigma_{R(CT-CB)}$$

The stress to which the outside of the top layer (σ_{CTe}) is subject can be determined similarly to the derivation of σ_{CBi}, giving:

(9)
$$\sigma_{CTe} = -\frac{-2m^3 n-3m^2 n+1}{(1+m)(-m^3 n+3m^2 n+3m-1)} \Delta\sigma_{R(CT-CB)}$$

Table 2 presents the mechanical strength data found in the normal position (top side up), σ_{RCT}, and in the inverted position with the base facing upwards (σ_{RCB}), as well as with the estimation of the stress in the outside of the top layer (σ_{CTe}) and in the base, next to the interface with the top layer (σ_{CBi}).

Type of specimen	σ_{RCT} (kg/cm^2)	σ_{RCB} (kg/cm^2)	$\sigma_{R(CT-CB)}$ (kg/cm^2)	σ_{CTe} (kg/cm^2)	σ_{CBi} (kg/cm^2)
T on B	930	745	185	-198	645

Tabla 2. *Apparent mechanical strength with the two specimen positions (σ_{RCT}) and (σ_{RCB}), and residual stresses in the interface (σ_{CBi}) and in the outside of the top layer (σ_{CTe}).*

The table shows, first of all, the difference in mechanical strength with the top layer face up or face down. Considering that Young's modulus and mechanical strength of the individual layers are similar (Table 1), this means that there are residual stresses inside the piece. As indicated above, these residual stresses are caused by the difference in thermal expansion of the two layers. Furthermore, the differences in temperature in the specimens caused by cooling could also make these stresses more pronounced.

The outside of the top layer is subject to compressive stress (negative value of σ_{CTe}), whereas high tensile stress is found in the base next to the interface (σ_{CBi}). To avoid failure, the tensile stress at the interface needs to be minimised.

5.2.3 Influence of cooling rate on residual stress in two-charge porcelain tile

With a view to verifying the effect of cooling rate on residual stresses, two-charge test specimens were prepared with compositions B and T, fired according to the other two cooling cycles set out in point 4.2.2: normal (N) and slow (S). In both cases, the cooling rate was decreased compared to starting conditions (fast cycle).

The 3-point bending tests were performed on both sides of the pieces, and the corresponding residual stress values in the interface (σ_{CBi}) were calculated from Eq. (8). Table 3 details the data for the tested cooling cycles (N and S). For comparative proposes, the corresponding data for the fast-cooling schedule discussed above have also been included.

Cooling	σ_{RCT} (kg/cm^2)	σ_{RCB} (kg/cm^2)	$\sigma_{R(CT-CB)}$ (kg/cm^2)	σ_{CBi} (kg/cm^2)
Fast (F)	930	745	185	645
Normal (N)	800	700	100	362
Slow (S)	780	760	20	48

Table 3. *Apparent mechanical strength and residual stress in the two-charge specimens obtained on a laboratory scale, using three different cooling schedules (F, N and S).*

The data show that as the cooling rate fell (R→N→L), the difference in mechanical strength found in both experiments decreased as was to be expected. The residual tensile stress that acts at the top-bottom layer interface (σ_{CBi}) therefore also dropped. Thus, lowering the cooling rate raised temperature uniformity in the whole specimen cross section at every temperature, so that the effect of the residual stresses caused by temperature gradients also dropped. These stresses are particularly relevant in the first cooling stretch, i.e., from peak firing temperature to the temperature range between 800-700°C. In this first cooling stretch, the piece exhibits markedly viscoelastic behaviour [13][14], so that the deformations that arise as a result of temperature gradients acquire, at least in part, a permanent character, subsequently found as residual stresses.

Unfortunately, industrial cooling resembles the fast cooling schedule more than the other tested cycles in the laboratory, so that appropriate design of the two-charge compositions is required to minimise the influence of temperature gradients in the first cooling stretch on residual stresses.

5.2.4 Influence of thermal expansion of both layers on residual stress

The difference in thermal expansion between the two layers making up the porcelain tile was shown to be the cause of residual stresses. To quantify this effect by the proposed method, two-charge specimens were prepared on a laboratory scale in which compositions were combined with different coefficients of thermal expansion. This was done by adding incremental quantities of quartz with a mean particle size of 20 μm to industrial powder T. The base layer composition used in each case was the result of mixing the T powder with 8 wt% quartz. Table 4 presents the three types of two-charge specimens, with their references. The table also includes the coefficient of thermal expansion ($\alpha_{300\text{-}500^{\circ}C}$) of the layers.

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[13] Cantavella, V. *Simulación de la deformación de baldosas cerámicas durante la cocción.* Castellón: Universidad Jaume I, 1998. Tesis doctoral.

[14] Enrique, J.E; Cantavella, V.; Negre, F.; Sánchez, E. *Model predicts tile deformation in firing.* Am. Ceram. Soc. Bull., 78(10), 65-69, 1999.

Specimen reference	Base layer (BL)	Top layer (TL)	$\alpha_{(300\text{-}500°C)} \cdot 10^7 °C^{-1}$ (BL)	$\alpha_{(300\text{-}500°C)} \cdot 10^7 °C^{-1}$ (TL)
80	T powder + 8% quartz	T powder	92	87
88	T powder + 8% quartz	T powder + 8% quartz	92	92
816	T powder + 8% quartz	T powder + 16% quartz	92	97

Table 4. *Two-charge specimens prepared with spray-dried powder T + quartz.*

In every case, industrial spray-dried powder T was mixed with quartz in a laboratory ball mill, using acetone as a suspending liquid. The dry powder was wetted to a moisture content of 5.5% by weight. The two-charge specimens were prepared in the same way as in point 4.2.1.

Three-point bending tests were run on both specimen sides to assess residual stresses. Table 5 presents the apparent mechanical strength data, together with the residual stress values at the interface (σ_{CBi}), obtained from Eq. (8).

Type of specimen	σ_{RCT} (kg/cm^2)	σ_{RCB} (kg/cm^2)	$\sigma_{R(CT\text{-}CB)}$ (kg/cm^2)	σ_{CBi} (kg/cm^2)
80	700	640	60	485
88	620	600	20	130
816	510	580	-10	-570

Table 5. *Apparent mechanical strength and residual stress in the two-charge specimens obtained on a laboratory scale, using compositions with different proportions of quartz.*

The data indicate that raising the coefficient of thermal expansion of the top layer (as the quartz proportion was increased) lowered residual tensile stress at the interface, until this even inverted to compressive stress in the case of specimen 816 (16% quartz). According to this line of reasoning, it appears reasonable to maximise the difference in thermal expansion between the top and bottom layer, in favour of the top layer. This observation will be true provided failure takes place at the interface itself, and not in another part of the piece, such as on the surface, as raising the thermal expansion of the top layer increases the residual tensile stress in the proper surface (Figure 1), so that the region in which fracture originates could shift towards the surface. A more conservative approach would be to attempt to obtain matching coefficients of thermal expansion in both layers, or at least, to keep a slightly greater thermal expansion in the top layer compared to the base.

It can furthermore be observed that specimen 88, which exhibited the same thermal expansion in both layers (in fact the same composition is involved but applied in two charges), did not exhibit zero residual stress as might be expected, but slight tensile stress. This apparent anomaly is due to the residual stresses produced by fast cooling.

5.3. REFORMULATION OF THE T COMPOSITION WITH A VIEW TO REDUCING RESIDUAL STRESSES. SEMI-INDUSTRIAL-SCALE TESTS.

In view of the findings with regard to the influence of thermal expansion in both layers, a new composition was formulated for application onto base powder B to reduce residual stresses. The formulation criterion used was to raise the coefficient of thermal expansion of the top layer (powder T) to match that of base composition B. The following modifications were made, not only to minimise residual stresses but also to keep the other product characteristics steady (whiteness, processability, etc.): raising the proportion of kaolinitic clayey mineral and incorporating feldspathic sand. The new composition was referenced T2 and its coefficient of expansion, determined under the same conditions as the foregoing samples ($\alpha_{300\text{-}500°C}$), was 92×10^{-7} $°C^{-1}$.

Two-charge specimens measuring 15x20 cm were prepared on a semi-industrial press with compositions B, T and T2, using sample B as base spray-dried powder. The top/bottom layer thickness ratio of the green body was 0.3 for both types of specimens, a slightly lower value than the one tested in the laboratory. Firing was performed in an industrial roller kiln, with a standard porcelain tile firing schedule.

Three-point bending tests were run on both sides of the specimens to assess residual stresses. Table 6 lists the mechanical strength data and residual stress values at the interface (σ_{CIi}) obtained from Eq. (8).

Specimen	σ_{RCT} (kg/cm^2)	σ_{RCB} (kg/cm^2)	$\sigma_{R(CT\text{-}CB)}$ (kg/cm^2)	σ_{CBi} (kg/cm^2)
T on B	650	540	110	870
T2 on B	570	660	-90	-740

Table 6. *Mechanical strength and residual stress in two-charge specimens made on a semi-industrial scale with compositions B, T and T2.*

It can be observed that the interface between the two layers of the specimens made from composition T2, with a greater thermal expansion than T, and therefore with characteristics closer to the base composition, was subject to compressive stress, whereas in the specimens made from powder T, tensile stress was found in this region. These results qualitatively match the laboratory-scale outcomes (points 5.2.2 and 5.2.4), though they differ in the magnitude of the calculated stresses as a result of the differences in the compositions and working conditions involved on both scales.

As a result of the above, the point stresses to which a porcelain tile may be subjected, which can occur at certain moments during polishing and especially during cutting with a diamond tool, will be more readily assimilated by two-charge porcelain tiles made by combining compositions T2 and B than with the powder with a lower thermal expansion (T).

To verify this, 50 two-charge tiles made with compositions T and T2 were cut with a diamond-tipped cutting tool. The proportion of tile failure as a result of fractures that separated from the cutting line was 10% and 2% respectively for compositions T and T2.

Finally, the results obtained confirm the validity of the method proposed in this study for estimating the residual stresses in two-charge porcelain tiles, which owing to its simplicity and speed can be used for industrial quality control.

6 CONCLUSIONS

The following conclusions were drawn from the present study:

- The different thermal expansion between the base composition and the top layer is the origin of the residual stresses that arise in two-charge porcelain tiles. The effect is similar to the curvature that occurs in glazed tiles, although in this case, the low deformability of the porcelain tile body hardly allows such severe curvature happen as in the case of glazed tiles.

- The high cooling rate in industrial kilns enormously encourages the residual stresses, as it raises the temperature gradient inside the tile.

- Residual stress has been related to the failures that occur on cutting two-charge porcelain tile. A lower residual tensile stress at the interface between the two layers reduces the fragility of the tile and avoids uncontrolled crack propagation, yielding a cleaner cut.

- A method has been developed for estimating residual stresses. The procedure involves determining the difference in mechanical strength of a tile on performing the test (3-point bending test) on the tile proper face and rear side. Using the Timoshenko equation, the difference in mechanical strength can be related to the residual stress present at the interface between the two layers that make up the tile.

- It was confirmed that raising thermal expansion in the top layer decreases the residual tensile stress acting at the interface, which favours tile cutting. Raising this thermal expansion is advisable provided it does not excessively increase tensile stress on the tile proper face.

- Taking this criterion into account, a composition was prepared on a semi-industrial scale for two-charge application, which yielded greater compressive stress in the tile fracture region (interface between the two layers). The two-charge tiles made with the new composition performed better during cutting with a diamond-tipped tool.

- The validity of the proposed method for determining residual stresses was confirmed both on a laboratory and on a semi-industrial scale, which permits using it for the design of new compositions or products (laboratory) and for industrial quality control.