DILATOMETRIC AGREEMENT IN DOUBLE COMPACTION PORCELAIN TILE

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ABSTRACT

During the heating of double compaction porcelain tile, the substrate and the micronised layer can undergo incompatible expansions, because in this stage the micronised layer displays a greater quantity of liquid phase owing to the greater reactivity between its particles, in accordance with the dimensional variations that the substrate displays. This state remains up to a certain cooling stage in the kiln, where the micronised layer again behaves as a solid and displays its own shrinkage. From that moment on, if the substrate and the micronised layer exhibit incompatible shrinkages, stresses will be generated in the layer interface and curvature develops, with a view to alleviating the arising stresses. In this sense, the control of the thermal expansion of the substrate and of the micronised layer becomes particularly important for the control of ceramic tile curvature. In order to keep these characteristics within certain limits, it is necessary to know all the possible causes of stresses in the substrate-micronised layer interface. In this study a double compaction type of porcelain tile was examined with a view to identifying the variables that cause curvature during firing. For this, the thickness of the micronised layer was altered in proportions of 15/85, 30/70 and 45/65 in relation to the thickness of the substrate, the tiles being compacted at 430kgf/cm^2 *and fired at 1205°C in a 50min cycle in a roller kiln. For each formulation, the three-point bending strength, modulus of elasticity, thermal expansion, softening point, and curvature were determined. The results suggest that the thickness of the micronised layer, its modulus of elasticity, and thermal expansion are the factors that most influence the resulting curvatures in double compacted porcelain tiles.*

1. INTRODUCTION

The expansion that most of the materials suffer is due to atomic vibrations caused by the action of heat, which leads to a greater spacing between the atoms, and hence to an increase in their internal energy. This dimensional increase is characteristic of each material and is expressed by a factor dependent on temperature, termed the coefficient of expansion. In addition, in order to determine the effective fit between a layer and its substrate, it is necessary to know the effective fit temperature, in addition to the softening point and the transformations points[1-3].

The factors that influence the thermal expansion of sintered ceramics depend on the nature and quantities of the glassy and crystalline phases formed during firing. Thus, the thermal expansion of the fired ceramic will be a function of the composition of the body ceramic and of the sintering temperature, and to a lesser extent of porosity[1-6]. Porcelain tile is a denser product owing to its greater quantity of glassy phase, which increases the intrinsic value of the expansion coefficient, with values close to 70.10-7°C^{-1}.

During the cooling of a tile in the kiln, at the effective fit temperature the surface layer (as occurs with the micronised layers in double compaction porcelain tile) ceases to behave as a viscous material and becomes a solid, so quickly that it is unable to absorb any stress to which it is subjected. Beyond this temperature, if the surface layer and the substrate undergo different shrinkages, stresses will originate between both materials, possibly leading to curvature of the sintered pieces[3-6].

The fit temperature (Ta) of the surface layer must be in the range of temperatures between the softening temperature (Tm) and the glass transition temperature (Tg) of the material, but its exact value is difficult to estimate. The optical methods and the Steger method used to determine the evolution of the stresses between the surface layer and the substrate as a function of temperature resulted in an estimate for the Ta values[3-7].

During firing, ceramic tiles are constantly subjected to variations in size due to their thermal expansion. If a ceramic body undergoes shrinkages of different magnitudes throughout its length, the end product can display deviations, problems of rectangularity, planarity and parallelism of its sides. Two main causes exist for differential shrinkages between the layers in a slab: temperature gradients in the dryers or kilns; and density variations in the tile, due to differences in homogeneity[5-8].

Even with a perfectly adjusted kiln (or dryer), dimensional deviations can occur owing to compaction heterogeneities during pressing, or variations in density in the same tile. The intensity of the strains caused by the variations in bulk density stem from problems in the pressing stage or the degree of tile vitrification during firing[1-4].

The elastic modulus is another parameter to be considered in the dilatometric agreement between the substrate and its covering. The elastic modulus changes as a function of the porosity in the material, and the formed porosity is a function of the sintering temperature: the higher the firing temperature, the greater is the formation of glassy phase and the smaller is the resulting porosity. Thus, the reduction in porosity leads to an increase in the elastic modulus. Other factors, such as the dissolution of the quartz present and the development of new crystalline phases considerably affect the elastic modulus[1-3].

Finally, in relation to the dilatometric agreement between the substrate and its covering, during the heating of the tile in the kiln, the substrate and the surface covering layer can undergo incompatible expansions, because in this stage the surface layer exhibits a greater quantity of liquid phase owing to greater reactivity between its particles, accompanying the dimensional variations of the substrate. This state will last until a cooling stage of the kiln when the surface layer again behaves as a solid and displays its own shrinkage. At that moment, if the substrate and the surface layer present incompatible shrinkages, stresses will be generated in the interface between the layers. The curvatures that arise will seek to alleviate the developed stresses[7,8].

In addition to the agreement or compatibility between the covering and bases being evaluated by their coefficients of expansion, this effect is not so direct, because the interfaces formed between the surface layer and the substrate considerably affect the stresses between the layers due to the compositional variations during firing. The lack of compatibility between the layers leads to the arising curvatures (concave or convex) in the end product[1-4].

From the knowledge of the relations between the thermo-mechanical behaviour of the substrate and of its surface layer, it is possible to identify the most likely causes of the dimensional deviations that ceramic products like double compaction porcelain tile display.

2. MATERIALS AND METHODS

In this study a basic formulation of spray-dried porcelain tile powder was used, forming the body of the substrate, and the same micronised formulation, forming the surface covering layer. The chemical composition of the base body was determined by X-ray fluorescence (XRF). The spray-dried powder and the micronised one were industrially obtained. In the double compaction the thickness of the micronised layer was altered in proportions of 15/85, 30/70 and 45/65 in relation to the thickness of the substrate, the tiles being compacted at 430kgf/cm^2 and fired at 1205°C in a 50min cycle in an industrial roller kiln. The isothermal heating used was 5min and the temperature was quickly reduced to 560°C.

For each formulation (double compaction) the three-point bending strength, modulus of elasticity, thermal expansion, and softening point were determined, the results being compared with the propensity to curvature of the trade product. The bending strength and the modulus of elasticity were determined in a universal testing machine with a rate of advance of 10mm/min using three support points. The coefficient of thermal expansion and the characteristic temperatures (Tm and Tg) were determined by dilatometry (10°C/min). The expansion was determined in the range of temperatures between 25°C and 325°C, and the characteristic temperatures to softening of the sample were determined.

3. RESULTS AND DISCUSSION

Table 1 gives the chemical analysis of the base body, formed basically by feldspars (albite and microcline), clays (muscovite, kaolinite, and illite) and other materials such as quartz and zircon, contaminated by anatase and goethite. It is a commercially used body by a porcelain tile company.

SiO_2	**Al_2O_3**	**TiO_2**	**Fe_2O_3**	**MgO**	**K_2O**	**Na_2O**	**ZrO_2**	**LOI.**
65,3	20,2	0,3	0,6	0,3	1,7	3,6	3,2	4,8

Table 1. Chemical analysis of the porcelain tile body (% by mass)

With regard to thermal behaviour, the body presents a glass transition temperature of 888°C and softening temperature of 1062°C, with a calculated fit temperature of 972°C. Its coefficient of thermal expansion is 70,5.10-7°C^{-1}. The micronised layer presents a Tg of 830°C, Tm of 1037°C and calculated Ta of 813°C, with an expansion of 72,0. 10-7°C^{-1}. The obtained results show there is a small difference between the coefficients of expansion of the body and of the micronised layer, which would not cause dilatometric agreement problems. In addition the differences between the softening and glass transition temperatures of the substrate and the micronised layer are large, which causes a large difference in the calculated fit temperature. Since the body is the same for the substrate and the micronised layer, the difference is only due to the processing of each layer.

Figure 1 shows the linear shrinkage with the variation in thickness of the micronised layer. There is a clear reduction of the linear shrinkage with the increase in the thickness of the surface layer due to greater density of this layer. Granulated powders present a greater green density when they are pressed and, therefore, a smaller shrinkage during firing.

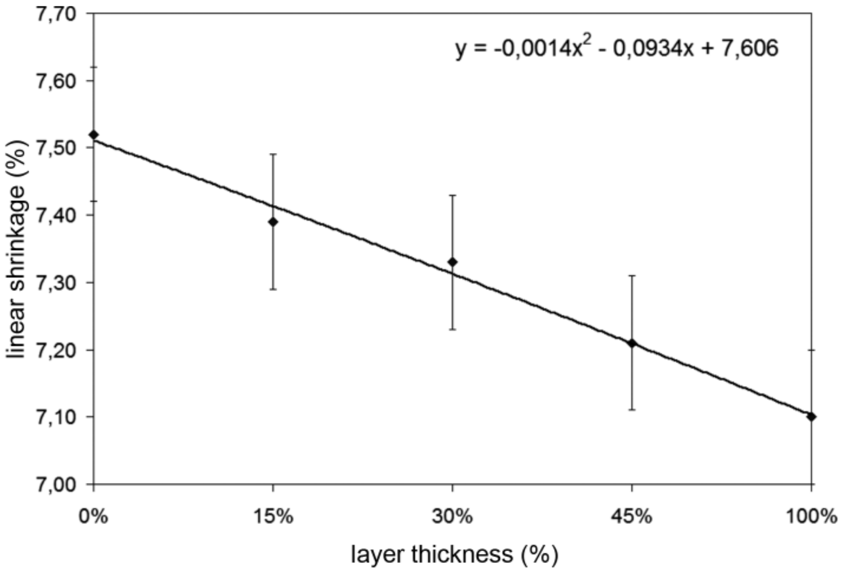

Figure 1. Linear shrinkage as a function of the variation in thickness of the surface layer

The modulus of rupture (Figure 2) is optimum with the relation 30/70 for the thickness of micronised layer. Both the modulus of the base and that of the micronised layer have lower values, which indicates the efficiency of the double compaction for the porcelain tile. Thus, with a 30% thickness of the surface layer, the greatest interaction probably occurs between the layers. The modulus of elasticity displays the same behaviour as the modulus of rupture, which was to be expected.

The double compaction allows the obtainment of a base layer consisting in about 75% of the thickness of the substrate, where the surface layer would not jeopardise the structural characteristics of the end product. The results of the modulus of rupture and elastic modulus were obtained taking the strain measurements of the machine, and not of the trials, requiring some corrections for a more precise calculation of these properties.

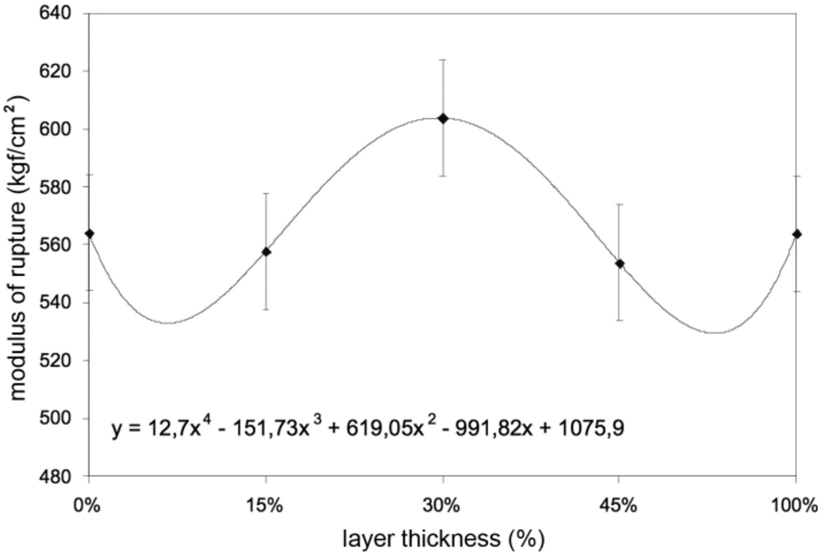

Figure 2. Modulus of rupture as a function of the variation in surface layer thickness

4. CONCLUSION

Double compaction is an industrial practice used for the obtainment of special technical or aesthetic effects for porcelain tile. However, the lack of agreement control between the substrate and its surface layer leads to dimensional problems such as curvatures and loss of planarity, which become very critical in polishing. In this research some aspects of the process have been verified that need to be better controlled in order to avoid fit problems.

With regard to the thermal expansion, the effect was verified of the compaction density of each constituent (substrate and micronised layer) on its coefficients of thermal expansion and characteristic temperatures (glass transition and softening). With the same chemical composition and phases as the substrate, the denser micronised layer had a slightly greater coefficient of thermal expansion. Its glass transition temperature, on the contrary, was lower.

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