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# Experimental Study of Typical Defects in Traditional Ceramic Products Caused by Deformations and State of Tension Occurring During Industrial Thermal Cycle

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**Abstract**

Deformations of traditional ceramic materials during and after firing may depend on several factors and are in general too complex to be studied theoretically. If the products are made up of a single material, such deformations are mainly due to pyroplastic phenomena. In the case of glazed materials, two further factors must be considered: the state of tension established between glaze and body, and their differences in behaviour during sintering. A novel optical technique is described to measure the state of tension of ceramic glazed bodies and applied to the study of delayed crazing and pyroplastic deformation of the body. In addition, this article outlines how can ceramists avail themselves of new optical equipment in order to design high quality products and avoid ordinary defects. Combining dilatometric tests and bending analysis, a full study of residual stresses on glazed ceramic can be performed, therefore this is how planarity problems can be solved. Furthermore the Optical Fleximeter is able to get information about viscosity changing during firing process in traditional sintered ceramic bodies, like stone-ware or porcelain-ware: the viscous flow occurring during the sintering process, where the driving force is mainly given by the surface tension of the liquid glassy phase, and the speed of the process is controlled by the viscosity of the glassy phase. This kind of analysis is crucial for the complete comprehension of pyroplastic behaviour. As a fundamental result, the bending and expansion curves obtained experimentally with optical techniques proved to be a valid help for the study of the deformations and state of tension in glazed ceramic materials.



## Deformations And Stresses In Glazed Tiles

In glazed or double layer tiles, deformations may be generated from the different behaviour of the two overlapped layers, during both heating and cooling phases. The coupling of materials with different thermal behaviours inevitably gives rise to a system of stresses due to the thermal incompatibility between the layers. The ceramic support shows the characteristics of an elastic solid, while glasses and glazes exhibit a strongly temperature-dependent mechanical behaviour. At room temperature, they behave as elastic solids, obeying Hooke's law; at temperatures higher than their glass transition temperature (Tg) they behave as plastic fluids and their viscosity decreases as temperature rises, in accordance to Arrhenius' law.

The study of the deformations and state of tension in a glazed ceramic material may be tackled from different points of view, both theoretical or experimental.

### 2.1 Theoretical deformations and stress

Coupling between glaze and ceramic body was studied by Amoros, Negre, Belda and Sanchez, which used a formula derived from Timoshenko equation to calculate theoretically the curvature induced in a glazed ceramic tile by the state of tension (compression/traction) of the glaze. They introduced some simplifying hypotheses: isotropic, homogeneous, perfectly elastic materials, no interface development between support and glaze, same temperature among the layers, elasticity moduli ratio constant during cooling: temperature among the layers, elasticity moduli ratio constant during cooling:

$$D = \frac{1}{8}\frac{L^2}{h}K_R\Delta C \qquad (1)$$

$$\sigma = E_g K'_R \Delta C \qquad (2)$$

Where:

D = deformation intended as "deflection" [mm]

$\sigma$ = glaze stress [MPa]

L = length [mm]

h = thickness [mm]

$\Delta C$ = percentage difference between the single dilatometric curves of the ceramic body and the glaze at room temperature (after translating the glaze dilatometric curve so that it coincides with the body dilatometric curve in correspondence of the coupling temperature)

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

$$K'_R = \frac{nm^3 + nm^2 + 1}{m^4n^2 + 4m^3n + 6m^2n + 4mn + 1}$$

Where:

$m = S_g / S_s$

$n = E_g / E_s$

$S_s$ = support thickness

$S_g$ = glaze thickness

$E_s$ = support elasticity modulus

$E_g$ = glaze elasticity modulus

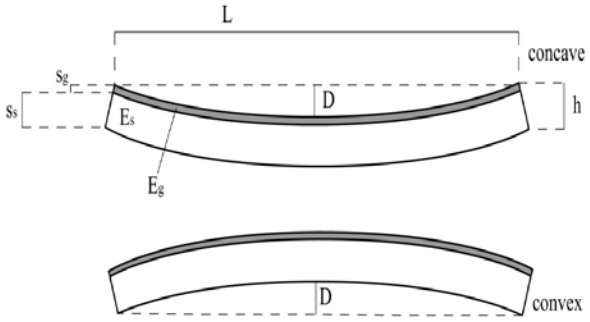


**Figure 1:** Quantities used in equations 1 and 2

In a further publication, Amoros, Moreno, Negre and Orts point out that, during firing, diffusion and dissolution phenomena between glaze and support layers occur and a glaze-body interphase develops. Thus, glaze and body do not behave as independent layers because of the presence of such interphase, which affects the properties of the final product, including planarity. For this reason, they state that coupling between glaze and support should be investigated experimentally, using the experimental conditions which better reproduce the industrial ones. Considering the limits to determine theoretically the flexion behaviour of a glazed ceramic material, optical techniques allows to characterize the material behaviour during firing and cooling without entering in contact with the specimen and thus with no interference caused by the measuring system, obtaining a good comprehension of the material real behaviour in an actual industrial firing cycle.

The instrument used in this paper to study the deformations and the state of tension in ceramic materials is the optical Fleximeter combined to the Horizontal Dilatometer (MISURA® ODLT® FLEX®)

The curves obtained with these instruments allow to identify the coupling temperature between glaze and ceramic body, to obtain information about the sample planarity and to study qualitatively and quantitatively the state of tension established between glaze and body [1].

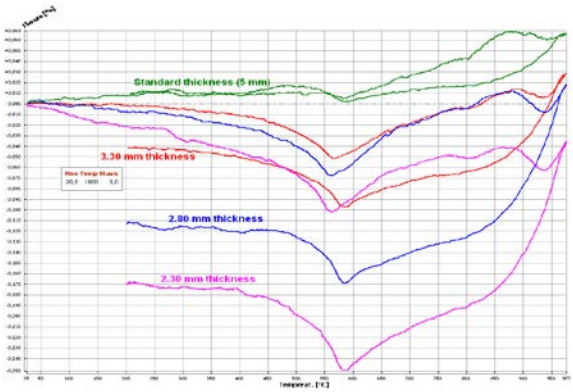


**Figure 2:** bending curves obtained reducing the body thickness

### 2.2 Planarity defect

The increased tile dimensions, the reduction of their thickness and the use of fast firing cycles, are all factors which required an accurate planarity control. By considering all these important reasons,a real case-study will be presented: two different porcelain stone-wares were analyzed by mean of the optical fleximeter varying the body thickness and keeping unchanged the one of the glaze. In addition, the

samples have the same body composition for both the cases but different type of glaze. Proceeding in this way we will appreciate how the body thickness reduction can affect both the bending deformation and the state of tension established in the ware. As we can easily see from the bending curves in Figure 2, reducing the body thickness from 5 mm up to 2.30 mm leads to an increase of the final bending deformation from ~10 microns (upward) up to ~120 mm (downward). This result represents the direct practical consequence of the theoretical deformation study expressed by equation 1 and 2: in fact, if we keep in mind that deformation intended as "deflection" is inversely proportional to the square of the ware thickness, we can easily understand why reducing the body thickness allows us to appreciate greater bending deformations.

On the contrary the level of stress established is noticeably increased. This behavior, rightly anticipated by the equation 2, is probably due to the dilatometric features both of glaze and body: by consequence, for having the right comprehension of the phenomena occurring in the glazed ware, it's very important to know also the expansion curves of both glaze and body, not only the results coming from the bending tests.

For these reasons the results obtained are plotted. Thanks to the curves in Figure 3, we can point out that there is another variable affecting the state of tension established between glaze and body: depending on the cooling rate, in fact, a volume decrease occurs both in the glaze and in the body.

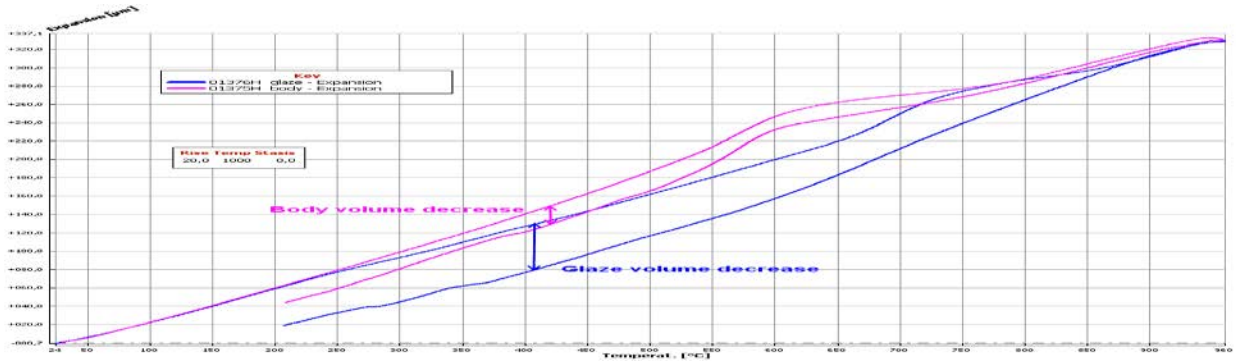


**Figure 3:** expansion curves of glaze (in blue) and body (in light-red)

By consequence a further study lies in focusing on detecting volume changes occurring after the heating and the cooling phase. This is the reason why a great bending deformation difference occurs between the heating and cooling phase of the bending curve. It must be pointed out that, regardless of the cooling rate applied during the analysis (natural or controlled), the greatest volume change is already occurred due to the first firing cycle applied rather than to the different cooling conditions. Both setting a natural cooling phase or a controlled cooling phase, in fact, leads to very different conditions from the ones verifying during an industrial process, when wares moving through the ending part of the kiln undergo cold airflow treatments freezing the inner volume of the glaze.

Proceeding this way, the Horizontal Dilatometer MISURA® ODLT® allows to study the thermal expansion dependence on the prior thermal history. Expansion data of a ceramic glaze melted and fast cooled during an industrial process, subjected to a two-way dilatation test (both for heating and cooling) are expression of important results. In order to study the change in volume which occurs in the cooling it is important to use the optical dilatometer with the thermostatic control of the optical bench mounted on a the granite base to minimize the mechanical drift of the instrument.

### 2.3 Pyroplasticity

The pyroplasticity is the attitude of the body to deform within a given temperature range. It is parameter of great technological importance is pyroplasticity. This parameter affects the quality of a production plant because it determines the relationship between the thickness and the rate of deformation of the material as a function of firing temperature and firing interval.

It is now possible to study changes in pyroplasticity during the thermal treatment by measuring the bending of a body sample suspended between two supports 70 mm apart. The bending occurring inside the kiln can be calculated using the formula that defines the deflection of a beam supported at the ends and subjected to its own weight (formula valid when deformation is purely elastic):

$$y_{max} = \frac{5\rho g l^4}{32Eh^2} \qquad (3)$$

Where:

$y_{max}$ = deflection [mm]

ρ= density

g = gravitational acceleration

l = distance between supports [mm]

E = elastic modulus [MPa]

h = thickness

When the deflection of the beam is caused by viscous flow, it increases as a function of time.

The rate of deformation is therefore a function of viscosity and, for small deflection, the values the elastic modulus E can be replaced by the viscosity [4]:

E→η

And the deflection y can be replaced with the deflection rate $\dot{y}$

y→ý

Considering solely the maximum deflexion at the center of the beam we obtain:

$$\dot{y}_{max} = \frac{5\rho g l^4}{32\eta h^2} \qquad (4)$$

The exact application of this formula is quite complex, because many of the parameters are subject to changes during the firing process. Take for example a typical porcelain body:

- the density raises from 2 g/cm$^3$ up to 2,4 g/cm$^3$,
- the thickness reduces according to the shrinkage (6-8 %),
- the viscosity is supposed to decrease with temperature according to the Arrhenius law.

It may be more productive to use a simplified theoretical approach to analyze how the pyroplastic deformation is affected by industrial parameters, assuming that the properties of the material:

- density ρ,
- viscosity η,
- thickness *h*,

do not change during the thermal cycle.

This approach may be considered too much simplified, but, looking at the formula, it may still yield useful information since the parameter which is affecting most the speed of deformation is the distance of the holders supporting the beam. In an industrial roller kiln this is represented by the roller gap.

During the industrial firing cycle inside a roller kiln, the material moves over the rollers and the supporting points shift continuously across the entire width of the piece. First of all we have to think to make a picture of the process, so that, instantaneously, we can assume that the product upon the kiln rollers is a beam supported at the ends, which is subject to its own weight, as previously mentioned. In other words, if we assume that the material properties do not change, the maximum speed of pyroplastic deformation will vary according to the fourth power of the gap between the rollers.

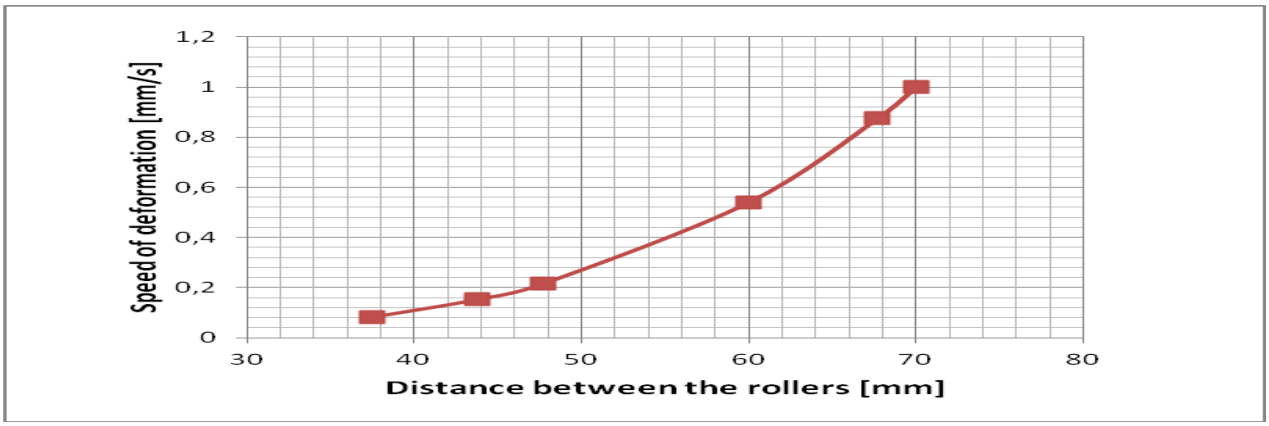


**Figure 4:** Speed of deformation change as a function of the distance between the rollers

$$\frac{\dot{y}_{max}}{l^4} = \frac{5\rho g}{32\eta h^2} = constant \qquad (5)$$

Proceeding this way allows us to easily study the effects on the maximum deflection caused by a change of the rollers gap. As a first result, we can now clearly see that the factor affecting most the speed of deformation is the gap between the rollers. If we suppose to change the gap between the rollers and plot the change of speed of deformation we get the behavior illustrated by the plot in Figure 4. Firing the same body composition in a kiln with a roller gap of 60 mm reduces the speed of deformation by the half!


**Figure 5:** pyroplastic deformation and speed of deformation of two different body compositions

The figure 5 shows the different pyroplastic behavior of two different body composition submitted to the same heat treatment as measure with the optical fleximeter MISURA FLEX, which has the sample holding rods with a gap of 70 mm.

- The blue body deformation curve reach a maximum deformation of 6534 micron at 1230 °C and it goes out of scale, reaching a speed of deformation equal to: $dy/dT$= 28,71 $10^{-3}$.
- The green body, at 1230 °C, reaches a maximum deformation of 2821 micron at and a speed of deformation equal to: $dy/dT$= *4,7* $10^{-3}$.

Surprisingly, the green body reduces the speed of deformation while the temperature increases: clear sign that the glassy phases are changing their compositions, becoming more viscous. This example shows how pyroplasticity and deformation rate may adversely affect the quality of a production plant. Since it is not yet possible to determine “ab initio” the deformation behaviour of a material, the analysis performed with the optical Fleximeter is of fundamental importance to characterize the material behaviour during heat treatment [3].

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