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Evolution of the mechanical strength of industrially dried ceramic tiles during storage

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Abstract

Industrially fast-dried ceramic tiles were used to determine the variation of dried tile mechanical strength with storage time in a moisture-free container. Dried tile mechanical strength rose with storage time under these conditions. Under the most favourable conditions, dry mechanical strength increased by up to 60% of the initial value. The reason for the rise in mechanical strength is attributed to the relaxation of stresses that develop during rapid industrial drying. A model was derived making the assumption that on exiting the dryer, the tile acts as a Maxwell linear viscoelastic system. The relation was determined between tile moisture content and mechanical strength, which was shown to be independent of whether tile moisture content was the result of drying wet tiles, or dried tile adsorption of ambient humidity. This relation and the stress relaxation model were used to derive a model that describes the evolution of dried tile mechanical strength during subsequent storage in ambient air very well.

Keywords: Drying; Mechanical properties; Strength; Tiles; Traditional ceramics; Residual stresses

1. Introduction

Green mechanical properties are of singular importance in the ceramic tile manufacturing process. Inadequate mechanical properties lead to rejects (both in green and fired products), with the ensuing cost involved in scrap recycling in the production process, or disposal and resulting environmental impact.[1,2]

The annual economic losses associated with cracking and failure in green ceramic tiles can be assessed at around 200 million euros in the EU, the world's major tile producer, so that even a minor reduction in flawed green tile would entail significant economic savings.

There are several reasons for focusing on the drying operation when studying tile mechanical strength. Firstly, reducing residual moisture content generally raises mechanical strength. However, completely dry tiles sometimes exhibit a higher rate of failure than tiles with a certain residual moisture content.[3] Secondly, if the drying operation takes place too rapidly, mechanical strength may be impaired by fatigue, and in extreme situations failure may occur. Finally, the drying operation has essentially been studied in terms of energetics or economics,[4,9] but hardly from a mechanical point of view.

The appearance of cracks in tiles is the phenomenon limiting the maximum rate at which the drying operation can take place.[5,6] Two simultaneous transport phenomena occur inside the tile during drying: heat is conducted inward into the tile from the tile surface, while water vapour diffuses from inside the tile to the surface. From a mechanical standpoint, both phenomena produce stresses if they do not develop uniformly throughout the piece: non-uniform heating will cause differential expansion, while non-uniform drying will produce differential shrinkage. Such stresses will obviously increase as heating and drying rates rise.

The temperature differences between the tile surface and tile interior are quite insignificant, as heat transfer does not generally constrain industrial drying.[4] Moisture gradients, i.e. tile inner moisture profile, can therefore be inferred to play a major role. This is particularly true in the fast drying schedules (20 min or less) currently used in industrial tile dryers.

Stress distribution in industrial tile drying is assumed to develop as shown in Fig. 1. During fast drying a

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Nomenclature

E	Young's modulus (Pa)
X	moisture content (kg water/kg dry solid)
α	parameter which relates moisture content to relaxation time [kg dry solid/(kg water s)]
β	parameter which relates moisture content to mechanical strength (kg dry solid/kg water)
ε	total strain
ε_e	elastic strain
ε_v	viscous strain
γ	relative relaxed stresses
η	viscous modulus (Pa s)
σ	residual stress (Pa)
σ_0	initial residual stress (Pa)
σ_{RD}	mechanical strength of a possibly stressed dried tile (Pa)
$\sigma_{RD\infty}$	mechanical strength of a dried tile after stress relaxation (Pa)
σ_{RR}	mechanical strength of a possibly wet stress-free tile (Pa)
σ_{RR0}	mechanical strength of a stress-free tile after drying (Pa)
σ_R	mechanical strength of a tile after drying and stress relaxation (Pa)
τ	relaxation time (s)
τ_0	relaxation time of a dry tile (s)

moisture gradient is rapidly established, leading to greater free shrinkage (shrinkage that would occur if there were no mechanical stresses). This free shrinkage gradient causes a stress profile to appear in the tile, bringing out a compression layer in the inner part of the tile and tension layers at both surfaces. Tile stresses are unlikely to be immediately eliminated when the tile exits the dryer, owing to the nature of the material. Tile mechanical strength will therefore vary as these residual stresses relax.

In this work, a study was undertaken of the evolution of tile mechanical strength after industrial drying, with a view to advancing a kinetic model that suitably describes stress relaxation after drying.

2. Experimental

The study was conducted on 31×31 cm tiles made by industrial pressing of a standard spray-dried powder used in stoneware floor tile manufacture. Drying was performed in a horizontal industrial dryer according to the standard thermal cycle (20 min).

Two types of unfired tiles were tested: tiles off the press and tiles exiting the dryer. Both types of unfired tiles underwent two series of tests, in which the tiles were placed in bags to keep them from losing or gaining moisture, or stored in cars to analyse the effect of ambient conditions.

This yielded the following four series of experiments: as-pressed tiles placed in bags (PB series), as-pressed tiles stored in cars (PC series), tiles exiting the dryer placed in bags (series SB), and tiles exiting the dryer stored in cars (SC series).

Moisture content and mechanical strength were determined of all the industrial tiles in the four series of experiments. Mechanical strength was determined by three-point bend testing (3PB). Ten tiles were used for each test condition, averaging the data.

3. Results

Figs. 2 and 3 plot tile mechanical strength, σ_R, and moisture content, X, versus storage time of the four series of tests.

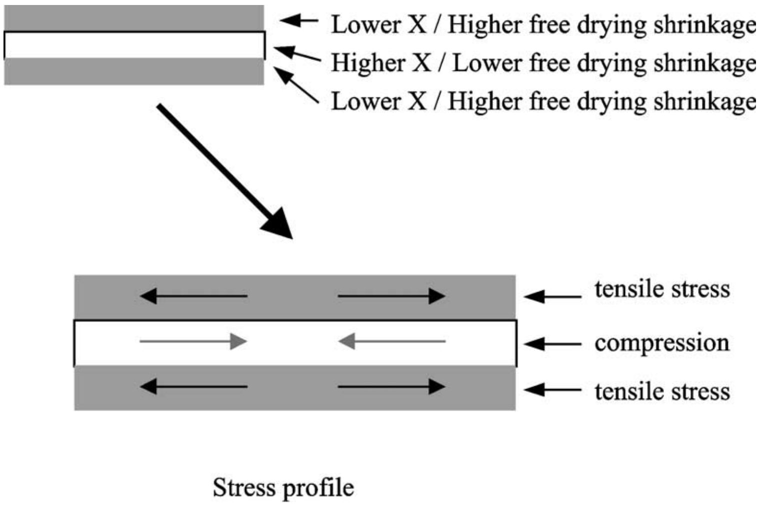

Fig. 1. Stress profile model arising during ceramic tile drying.

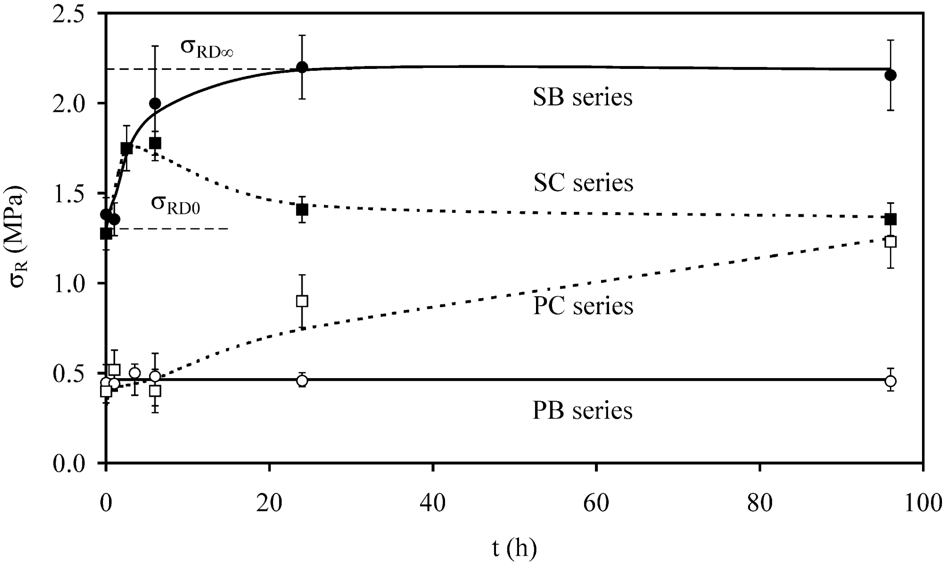

Fig. 2. Evolution of tile mechanical strength (σ_R) with storage time.

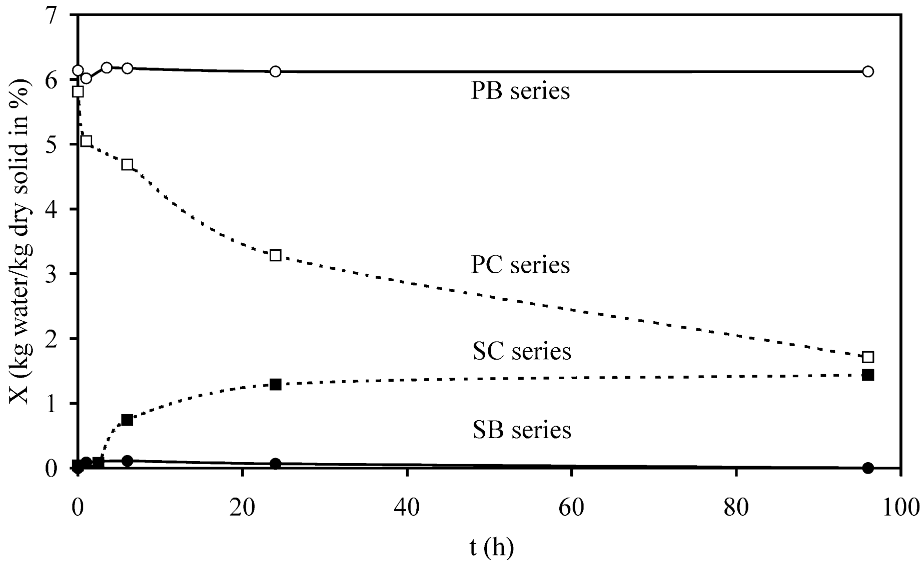

Fig. 3. Evolution of tile moisture content (X) with storage time.

The figures show:

1. The mechanical strength of as-pressed tiles stored in bags to retain their moisture (Fig. 3, PB series) remains practically constant (Fig. 2). This indicates an absence of residual stresses in the freshly pressed tiles, in all likelihood due to the almost instant relaxation of any stresses that may have been generated in the industrial pressing operation.
2. The as-pressed tiles stored in ambient air at 24 °C and around 60% relative humidity (PC series), which dry progressively (Fig. 3), also exhibit a progressive increase in mechanical strength (Fig. 2). Rising mechanical strength correlates with a parallel decrease in tile moisture content in this type of body, as shown elsewhere.[3,7]
3. The tiles placed in bags on exiting the dryer (SB series) to keep them dry (Fig. 3) exhibit a noticeable rise in mechanical strength (Fig. 2), which increases most at the beginning of this process. The considerable rise in mechanical strength must be caused by relaxation of stresses produced in the tile during drying, since the relaxation of stresses generated in pressing, the other possible cause, is practically immediate.[8]
4. The mechanical strength of tiles exiting the dryer and stored in the industrial ambient rises, peaking at 6 h storage (Fig. 2), after which it decreases, tending asymptotically to a constant

value. This behaviour is due to the simultaneous occurrence of two opposing processes: stress relaxation on exiting the dryer, which tends to raise tile mechanical strength, and adsorption of ambient humidity, which tends to lower tile mechanical strength (Fig. 3).

5. Comparison of the mechanical strength, σ_R, of tiles stored in ambient air for 4 days (SC and PC series) shows that these values practically coincide, as does their moisture content, (X), evidencing virtual tile moisture equilibrium with atmospheric air humidity, regardless of whether this proximity in humidity is caused by adsorption of ambient humidity (SC series) or drying (PC series). Furthermore, the coincidence or proximity of tile mechanical strength data indicates that at long storage times, when relaxation processes have ended, moisture content is the determining factor in tile mechanical strength.

3.1. Relaxation of tile stresses on exiting the dryer—proposed kinetic model

The proposed model is based on the following assumptions:

(i) Each tile is assumed to consist of a series of uniform layers of infinitesimal thickness, each of which is subject to different residual stress according to its position (Fig. 4a). Owing to tile geometry and the way in which heat and mass are transferred to the layers during drying, thermal and moisture content gradients only form across the thickness of the tile. The stresses they produce will therefore be uniform in each layer.

(ii) The viscoelastic behaviour of each infinitesimal layer can be described mathematically by a Maxwell element (Fig. 4b). Under these conditions, the following equations relate elastic (ε_e), viscous (ε_v) and total strain (ε) to the mechanical properties of the solid:[3]

$$\left.\begin{array}{l}\varepsilon = \varepsilon_e + \varepsilon_v \\ \varepsilon_e = \dfrac{\sigma}{E}; \quad \dot{\varepsilon}_v = \dfrac{\sigma}{\eta}\end{array}\right| \rightarrow \dot{\varepsilon} = \frac{\dot{\sigma}}{E} + \frac{\sigma}{\eta} \tag{1}$$

where E is Young's modulus and η is a modulus that quantifies the material's viscous behaviour.

(iii) The tile was experimentally verified to undergo no curvature after leaving the dryer, indicating that tile total strain (ε) is uniform (the total strain that each layer undergoes does not depend on its position). As each layer is subject to different residual stress, it follows from both conditions that ε and $\dot{\varepsilon}$ are independent of residual stress, so that $\dot{\varepsilon}(\sigma) = \dot{\varepsilon}(-\sigma)$. On the other hand, Eq. (1) enables inferring that $\dot{\varepsilon}(\sigma) = -\dot{\varepsilon}(-\sigma)$. On combining the last two equalities, total strain is found to be not only independent of position, as already observed, but also of time ($\dot{\varepsilon}(\sigma) = 0$).

Applying this last condition to Eq. (1) and integrating the resulting differential equation between $t=0$ and a generic time gives:

$$\sigma = \sigma_0 \exp\left(-\frac{t}{\tau}\right); \quad \tau = \frac{\eta}{E} \tag{2}$$

where σ_0 is the initial residual stress (at dryer exit). This equation describes the variation of each infinitesimal layer's residual stress with time. τ is a parameter, having units of time, which determines stress relaxation rate.

The mechanical strength measured in a bending test is the maximum tensile stress that the bottom layer can withstand (Fig. 5). In accordance with Eq. (2), at a sufficiently long time, the residual stress that each of these hypothetical layers undergoes will be cancelled, as a result of which the tile will achieve its peak mechanical strength ($\sigma_{RD\infty}$) (subscript D stands for dry). During the relaxation process, tile mechanical strength (σ_{RD}) will therefore be the difference between peak mechanical strength ($\sigma_{RD\infty}$) and the residual stress (σ) acting in this layer: $\sigma_{RD} = \sigma_{RD\infty} - \sigma$. Incorporating the variation of σ with time [Eq. (2)] in this equation yields:

$$\sigma_{RD} = \sigma_{RD\infty}(1 - \gamma e^{-t/\tau}) \tag{3}$$

where:

$$\gamma = \frac{\sigma_{RD\infty} - \sigma_{RD0}}{\sigma_{RD\infty}} \tag{4}$$

This equation describes the increase in mechanical strength of a dried tile during relaxation. The solid line of the SB series (Fig. 2) is the plot of Eq. (3) found by fitting the experimental data. It should be noted that moisture content was kept constant, as shown in Fig. 3

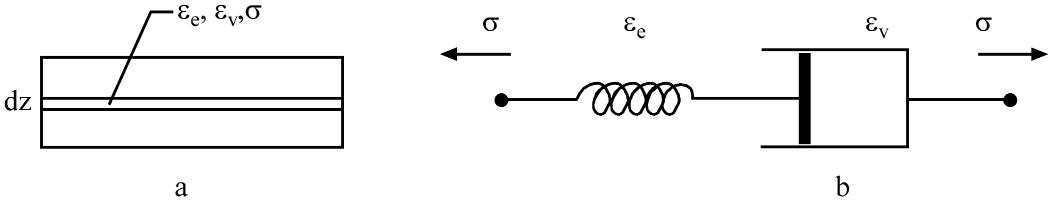

Fig. 4. Scheme of the viscoelastic model used.

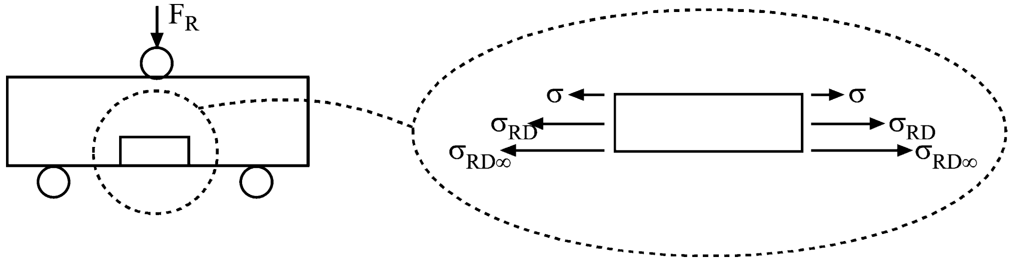

Fig. 5. Stress distribution at the centre of the bottom layer in a bending test.

Table 1
Values of the parameters of the equations used in the model

Equation	Parameter	Value
3	$\sigma_{RD\infty}$	2.20 MPa
	γ	0.4
	τ	16.9 ks (4.69 h)
5	$\sigma_{RR0}=\sigma_{RD\infty}$ (Eq. 3)	–
	β	33 (kg dry solid/kg water)/100
7	$\sigma_{R\infty}=\sigma_{RD\infty}$ (Eq. 3)	–
	$\tau_0=\tau$ (Eq. 3)	–
	α	100 (kg dry solid/(kg water·h))

(SB series). Excellent fit is found. The values of the parameters are detailed in Table 1.

3.2. Relation between tile mechanical strength and moisture content

If dried tiles are stored in a relatively humid environment, they will adsorb ambient humidity. This would lower tile mechanical strength, if the tile were free of residual stresses. Fig. 6 plots the mechanical strength of stress-free tiles (σ_{RR}) (the second subscript R stands for "relaxed") versus moisture content. These data are mainly from the PC series in Fig. 3.

According to some researchers,[3,7] the equation which relates σ_{RR} with moisture content (X) reads:

$$\frac{\sigma_{RR}}{\sigma_{RR0}} = \exp(-\beta X) \tag{5}$$

The plot of this equation is also shown in Fig. 6. Table 1 lists the values of Eq. (5) parameters. The value σ_{RR0} equals $\sigma_{RD\infty}$. This is an expected result, because the final mechanical strength of an initially stress-free, as-pressed tile, which is then completely dried, must be the same as that of an initially dry tile containing stresses, which then relaxes. The resulting tile state is the same: dry and stress-free.

3.3. Evolution of dried tile mechanical strength on storage in ambient air

If a tile freshly emerging from the dryer is stored in ambient air, it will simultaneously be subject to moisture

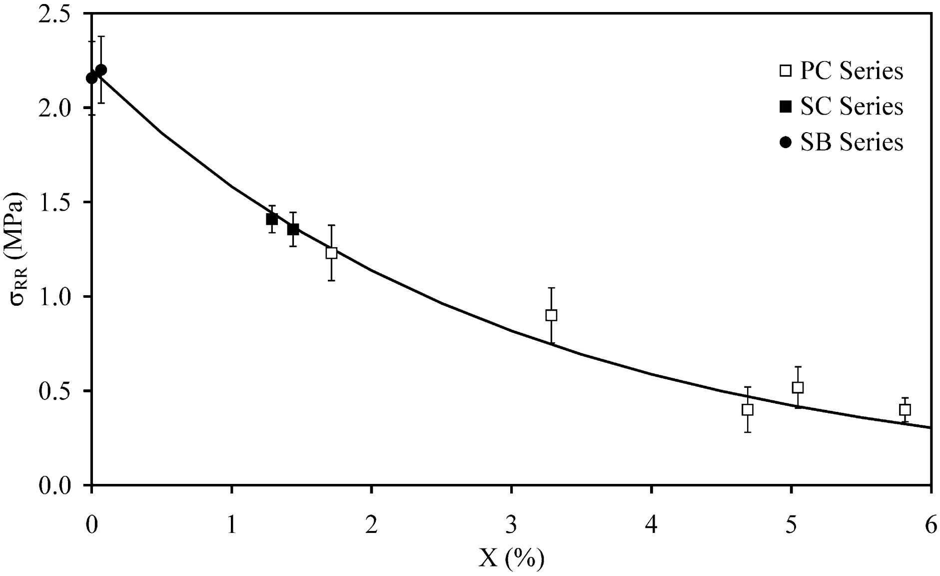

Fig. 6. Effect of moisture content on the mechanical strength of stress-free tiles.

adsorption (SC series, Fig. 3) and stress relaxation. These opposing processes cause the variation of tile mechanical strength with storage time to exhibit a peak (SC series, Fig. 2). Ambient condition mean values during the industrial tests were $T = 20$ °C and relative humidity $\varphi = 70\%$.

Deriving a model that takes moisture sorption as well as stress relaxation processes into account requires making certain assumptions. The model assumes that Eq. (3) is also valid if moisture is not constant, provided the following modifications are made:

(i) As moisture content increases, stress relaxation is quicker, so τ must depend on X, T and the nature of the clay. One of the simplest relationships to explain the effect of X on τ is to assume:

$$\frac{1}{\tau} = \frac{1}{\tau_0} + \alpha X \tag{6}$$

where τ_0 and α are parameters that only depend on T (and the nature of the clay). When $X = 0$, $\tau = \tau_0$, so that τ_0 will be the value of τ found from the fit of Eq. (3).

(ii) $\sigma_{\mathrm{RD}\infty}$, which corresponds to the mechanical strength of a relaxed tile, depends on moisture content analogously to Eq. (5).

(iii) γ, which represents the ratio of relaxable stresses to mechanical strength, does not depend on moisture content. This parameter can therefore be directly found from the previous fit [Eq. (3)].

Taking into account the aforementioned assumptions, the final equation reads:

$$\frac{\sigma_{\mathrm{R}}}{\sigma_{\mathrm{R}\infty}} = \exp(-\beta X)\left[1 - \gamma \exp\left(-\left(\frac{1}{\tau_0} + \alpha X\right)t\right)\right] \tag{7}$$

Fig. 2 (SC series) depicts the curve calculated using Eq. (7), whose parameters are detailed in Table 1. Moisture content was experimentally measured (if moisture sorption kinetics were known, it would generally be possible to express X as a function of time, relative air humidity and temperature). The agreement between Eq. (7) and the experimental data is quite good. The equation specifically enables reproducing the peak mechanical strength attained after a certain time (about 6 h after the tiles have exited the dryer).

4. Conclusions

The study shows that the mechanical strength of ceramic tiles changes significantly after the tiles exit the industrial dryer.

The increase in mechanical strength of completely dry tiles can be explained by assuming that the tiles contain stresses produced by the drying process, which subsequently relax.

A model has been derived, based on Maxwell's viscoelastic elements, which enables explaining the rise in mechanical strength.

The relation between tile mechanical strength and moisture content was found to be independent of whether the process involved in modifying tile moisture content occurred by drying tiles with a high initial moisture content, or by moisture adsorption from ambient air of previously dried tiles.

If tiles adsorb humidity after exiting the dryer, their mechanical strength rises and then decreases. This is the result of two opposing processes: stress relaxation raises mechanical strength, while the concurrent rise in moisture content lowers mechanical strength. This joint behaviour has also been successfully modelled.

Acknowledgements

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