KEY CERAMIC PROCESSING VARIABLES THAT LEAD TO FLAKING OF CERAMIC TILE GLAZES

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The objective of this study has been to investigate the possible causes of the proneness to flaking of the glazed surface of ceramic tiles. For this, scanning electron microscopy was used to observe the body/engobe and engobe/glaze interfaces of ceramic pieces with a high tendency to flake, with a view to comparing the degree of adhesion with tiles that have little tendency to exhibit this defect. Using thermal expansion analysis of the body, glazes and engobes, the stress state was determined to which the glazes and engobes used in the study were subjected, as a function of the evolution of firing temperature.

Tests were conducted in the industrial glazing lines, attempting to correlate the influence of engobe and glaze layer thickness with proneness to flaking. The pieces were prepared so that they could pass through the bell to receive one or two layers of engobe or glaze. The specimens were subjected to firing in an industrial kiln. After firing, they were scratched with a diamond-tipped material, trying to apply the same force and speed. It was observed that the samples that underwent least flaking damage were those that had two consecutive applications of glaze layers. The proneness to flaking was even smaller when a single layer of engobe was applied.

Systematic studies of engobe, glaze and body thermal expansion, in samples of products collected at companies that displayed flaking, enabled concluding that the critical factor in determining flaking was not always the state of stress (Δc) to which the glazes were subjected at ambient temperature. Thus, in the studied cases, when analysis was performed of the glaze/engobe and glaze/body fit by means of thermal expansion curves, the glaze always displayed a small state of compression, therefore reproducing the same situation observed in the case of products from companies where there was never any appearance of flaking. However, in most products of the companies that displayed flaking of the glazed layer, the engobes had a higher initial densification temperature and/or the rate of shrinkage above this temperature tended comparatively to be smaller.

The interfaces of the glazed pieces were observed in a scanning electron microscope. It was found that the engobes of the pieces that displayed flaking of the glaze layer had a very porous structure, which was a microstructural configuration typically exhibited by engobes with a high softening point. The body/engobe interfaces were very similar in all the samples, with good adhesion. The engobe/glaze interfaces in which flaking normally occurred were quite different in the pieces that displayed flaking (Figure 1-b), which exhibited a smaller "penetration" of the glaze in the engobe compared with tiles that did not present any flaking, and which showed a greater interaction of the glaze with the engobe (Figure 1-a). These engobes, with a comparatively lower initial densification temperature, provided a greater degree of "anchoring" and larger area of reactivity for the generation of crystalline phases in the interface with the glaze, fomenting glaze bonding and avoiding flaking.

In vitrification curves made with fusion test specimens for the engobe of the product that frequently displayed flaking (Figure 2), it can be observed that at the industrial working temperature (a temperature that corresponded to a water absorption of 5% for the body), the engobe had quite a high water absorption around 16%. This shows that in the end pieces the engobe is more porous than the body of the substrate. That is to say, it is quite a refractory engobe. It can also be observed (Figure 2) that a variation in the industrial kiln temperature of ±10°C around the working temperature produces a variation of approximately ± 2.0% in engobe water absorption.

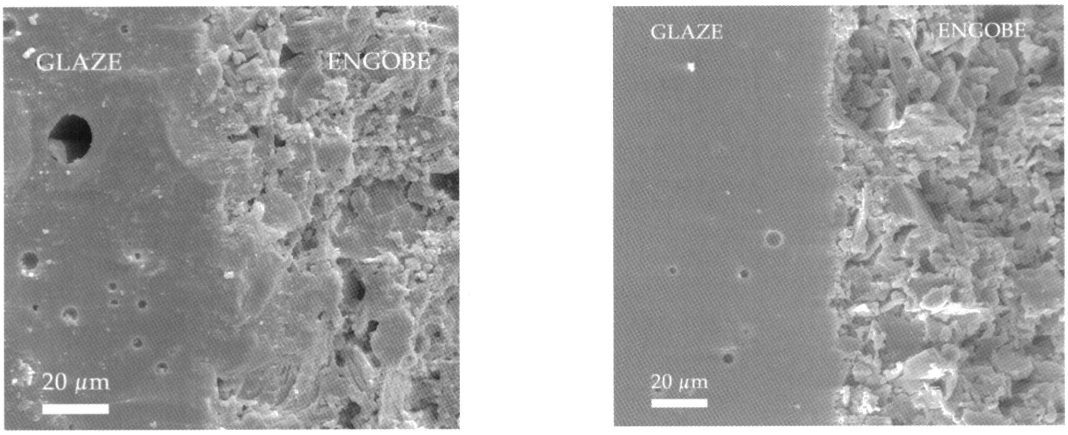

Figure 1. Interface of the sample that displayed no flaking (a) and that displayed flaking (b).

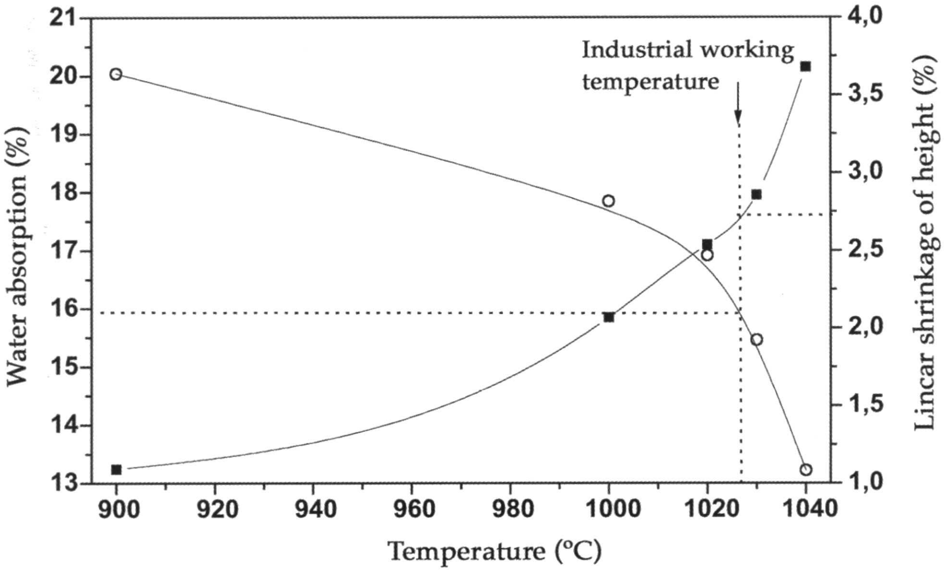

Figure 2. Vitrification behaviour of the engobe of the product exhibiting a proneness to flaking of the glaze layer.

The results obtained show that the tendency to flake can not normally be attributed to one single cause but there are a series of factors relating to the specification of materials, manufacturing process and specification for use, which in certain circumstances combine in a critical way to cause the defect. However, in the studied cases, one main cause is related to lack of adhesion of the glaze/engobe interface, mainly due to an engobe with inadequate fusibility for the fast firing cycles used in the companies where the products that tended to flake were collected.

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