

EVOLUTION OF CERAMIC ROLLER MICROSTRUCTURE DURING USE

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1. INTRODUCTION

Ceramic rollers are refractory materials that are subjected during use to high temperatures and an atmosphere containing volatile compounds^[1] released during ceramic tile firing. Because the rollers get dirty, it is common practice to withdraw them for cleaning while the kiln is running, and then put them back. This fact makes roller resistance to thermal shock one of its most important characteristics^[2]. And this depends on roller composition and porosity. Inside the kiln the roller is exposed to a series of aggressions by the gases and pieces being fired, which, with time and temperature, modify roller microstructure and properties. As consequence of these transformations, the roller coefficient of thermal expansion changes^[3].

The present work studied the evolution of the composition and microstructure of ceramic rollers installed in the heating and firing zones of industrial floor tile kilns. It shows that one of the main agents responsible for these changes is the calcium contained in some engobes^[4] used on the back of ceramic tiles.

2. EXPERIMENTAL

The kiln modules from which the rollers were withdrawn, and their average operating temperatures were: heating I (800°C), heating II (950°C), pre-firing (1120°C) and firing (1200°C). Four withdrawals were effected after 2, 3.5, 6 and 12 months' time inside the kiln.

To examine the microstructure, the central areas of the rollers were selected (located in the middle of the kiln). The test specimens, cross-sectionally sliced, were embedded in an epoxy resin and polished to 3 μm . In some cases the polished test specimens were etched with aqueous solution of HF to make the crystalline phases present stand out.

All the test specimens were observed with an optical microscope and a scanning electron microscope with the secondary and back-scattered electron signals and analysed with an energy-dispersive X-ray spectrometer system (EDXA). For observation with the secondary electron detector, the sample surface was coated with conductive gold film.

3. RESULTS

Figure 1 corresponds to the cross section of a roller installed for 6 months in the heating zone II. A porous layer is observed (a), which consists of engobe stuck to the roller outer surface; and an area (b) in which the calcium and magnesium have diffused inward into the roller, with a clearer colour, without any pores. The corundum grains of the roller (c) remain unchanged, as are the zirconium silicate (d) and mullite of the rest of the roller (e).

The roller in Figure 2 spent 6 months in the pre-firing zone. An outer, more sintered engobe layer can be observed (a), which contains magnesium and calcium aluminosilicates; there is also a clearer region in the interface between the roller and the layer (b), marked with a dashed white line, produced by diffusion of calcium and magnesium inward into the roller. The corundum particles (c) at the surface of the roller, just in contact with the region (b), have converted partly to spinels by inward diffusion of magnesium; while in the innermost areas (d) the corundum remains unaltered, without transforming. In area (e) calcium is detected, which indicates that this element diffuses to a greater depth than magnesium.

Figure 3 shows the cross section of a roller installed for 3.5 months in the firing zone. The outer part of the roller has sintered, eliminating the pores. In the surface region of the roller, which corresponds to the engobe layers, calcium has crystallised as anorthite (a), and magnesium as magnesium silicate (b). Under the engobe layer, magnesium has reacted with the corundum in the roller, producing a spinel (c); also in this area, in which the proportion of calcium is very high, zircon has converted to zirconium oxide and the initial mullite has reacted with calcium to produce anorthite.

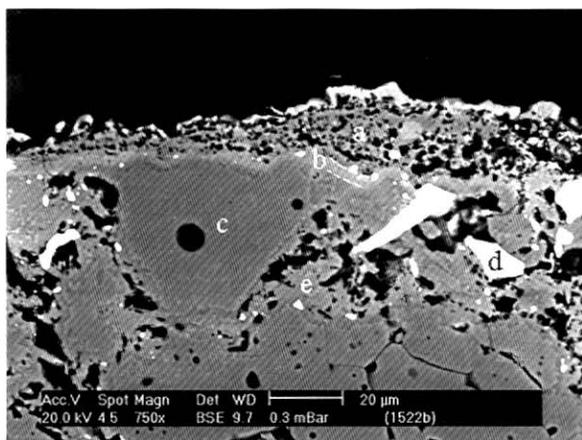


Figure 1. Roller from heating zone II.

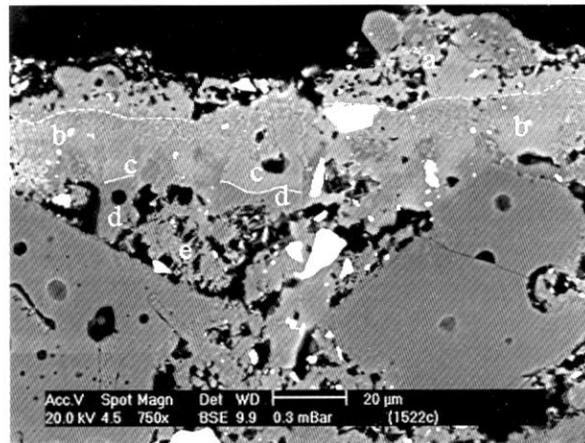


Figure 2. Roller from the pre-firing zone.

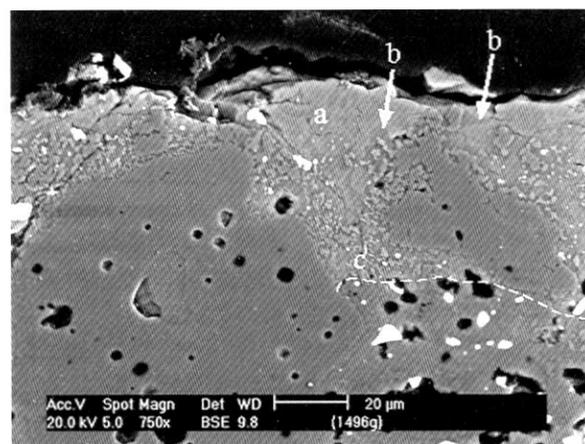


Figure 3. Roller from the firing zone.

4. CONCLUSIONS

The study shows that inside the kiln, rollers undergo attack by two different mechanisms:

- In the gas phase, by condensation of volatile elements which accumulate in the kiln atmosphere
- In the liquid phase, by elements consisting of materials stuck to the rollers, such as: tile base engobe, tile support or dirt from the glaze.

Attack by volatile elements fundamentally affects the first heating zone. The detected elements were sulphur and potassium. Sulphur is deposited as calcium sulphate, forming a porous crystal layer on the roller, which comes off easily. The calcium that forms the sulphates basically comes from the engobe applied to the back of the tiles, which has adhered to the rollers.

Attack by diffusion in the liquid phase and the reaction are essentially due to the components of the engobe applied to the back of the tiles, namely calcium and

magnesium, with calcium being the main agent responsible for the degradation of the original microstructure of the roller. This attack leads to the formation of dense layers on the roller surface, which are of importance in the pre-firing and firing zones.

5. REFERENCES

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