FIRING A GLAZED FLOOR TILE MASS BY MICROWAVE ENERGY

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INTRODUCTION

Several investigations have been devoted to the sintering of technical ceramics by means of microwave energy^[1,2,3]. The so-called "non-thermic effects" of this kind of energy source generally lead to a more homogeneous microstructure and absence of residual stresses in the fired bodies. Moreover, the same densification degree as of the conventional radiation/convection firing is promoted, however, in a range of 200°C to 300°C lower temperatures, in 50% shorter firing cycles.

Microwave energy heating is due to the dielectric relaxation of ceramic materials in the region of high megahertz to medium gigahertz frequency. It may be produced from capacitive or conductive phenomena. Most of the reported work on the use of microwave energy in ceramic sintering is related to high performance ceramics for which dielectric losses as a function of temperature are well known. For clayey materials such as vitrified stoneware floor tile masses apparently no pertinent data have been presented, nor with respect to characteristic, processing behaviour and final properties of typical masses. Based on results obtained for technical ceramics, this work investigated the use of microwave sintering of a commercial glazed vitrified stoneware floor tile mass (3 to 6% water absorption), compared with conventional single fast firing of the same body.

It was found that bodies fired under microwave energy displayed much higher fired tensile strength, lower water absorption and shrinkage, compared with the conventional single fast firing process. Additionally it was found that the glaze microstructure is less porous and the pores are smaller, while the body/glaze interface is better defined and stronger. All these good characteristics were attained at approximately 100 °C lower temperature in at least 25% less firing time.

MATERIALS AND METHODS

The Mass – The investigated mass was a proprietary composition of vitrified stoneware floor tile type of commercial grade produced by Ceramica Eliane from the State of Santa Catarina Brazil.

The Specimens – Cylindrical specimens of 2.5cm diameter and 0.5cm thickness were prepared under 45MPa pressing pressure of the spray-dried mass. The resulting as-pressed density was around 1.92g/cm³. The Glaze was applied by spraying.

The Firing Schedule - The firing schedule used for the microwave sintering consisted of a heating rate of 50°C/min. from room temperature to 800°C; from 800°C to 1010°C a heating rate of 20°C/min was used, followed by natural cooling. In this case the total firing cycle took 32 minutes.

On the other hand, the firing schedule used in conventional single fast firing was 50°C/min from room temperature to 1150°C in an electric furnace, with a residence time of 3 minutes, and cooling rate of 50°C/min by forced convection.

The Microwave Furnace – The microwave oven used was of a domestic type powered by a multimode 950 Watt magnetron, working under 2.45 GHz frequency. The power was delivered by a wave phase controller. The temperature was evaluated by a shielded and grounded K-type thermocouple. Silicon Carbide susceptor bars were used to help heat up the pile of specimens (stack of three), in order for them to start coupling with the microwave field.

Properties Evaluated – Vitrification Behaviour (Water Absorption and Firing Shrinkage), Fracture Strength (Brazilian Test), Microstructure Features (Optical Microscopy).

RESULTS AND DISCUSSION

Figure 1 shows the vitrification diagrams for the microwave sintered and the conventional fired specimens. It is clear that the microwave sintered specimens matured at lower temperature presenting lower values for firing shrinkage at the optimum firing temperature, the same occurring for water absorption.

Figures 2 and 3 show that the body, glaze, and interface regions exhibit better characteristics for the microwave processed specimens, than for the conventionally sintered ones. The body seems to be denser, the interface smoother and the glaze more homogeneous and dense. The respective glaze bulk appearances can be better appreciated in Figures 4 and 5. It is clear that the microwave processed specimens have a smaller volume of pores and which are of smaller dimensions.

To conclude Table I presents the respective fracture strengths for specimens fired at 1050°C (optimum firing temperature for the microwave processed specimens), and 1150°C (optimum firing temperature under conventional sintering). It can be seen that the microwave processed specimens present less deviation, and are stronger when fired at 100°C lower temperature comparatively to the conventionally sintered specimens. A drastic decrease in strength can also be noticed at 1150°C for the microwave sintered specimens, which confirms the overfiring displayed by the vitrification diagrams.

| | Strength (1050°C) MPa | Strength (1150°C) MPa |
|-------------------------------|-----------------------|-----------------------|
| Microwave Processed Bodies | 16.3 ± 4.1 | 4.1 ± 1.2 |
| Conventional Processed Bodies | 9.7 ± 1.9 | 20.1 ± 7.6 |

Table I. Fracture Strengths Versus Temperature of firing for the two different firing procedures.

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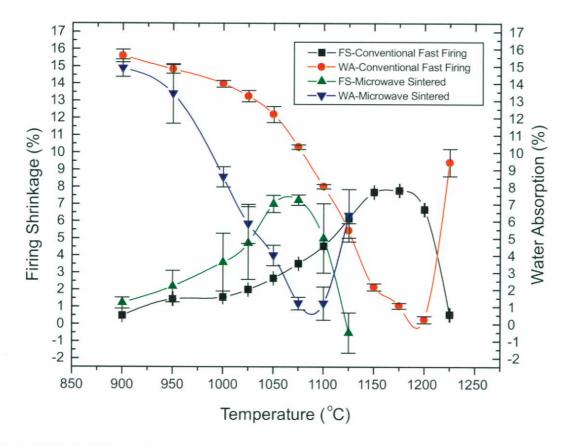


Fig. 1. Vitrification Diagrams for the Conventionally and Microwave fired specimens of vitrified floor tile composition.

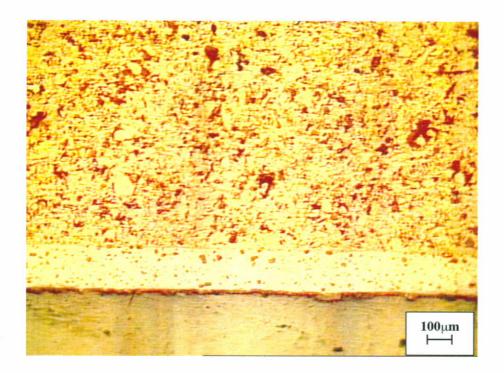


Fig. 2. Optical Micrograph displaying the general aspects of glaze/body interface in a vitrified stoneware floor tile mass single fired under microwave energy at 1050°C during 3 minutes. Heating rate 50°C/minute.

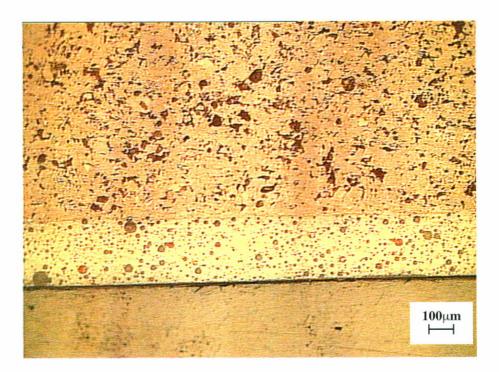


Fig. 3. Optical Micrograph displaying the general aspects of glaze/body interface in a vitrified stoneware floor tile mass fast fired at 1150°C during 3 minutes in a conventional eletric furnace. Heating/cooling rate used was 50°C/minute.

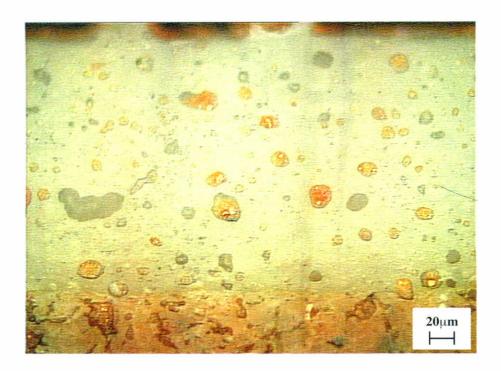


Fig. 4. Optical Micrograph showing the general aspects of glaze/body interface and glaze bulk, of a single fired glazed vitrified stoneware floor tile mass under microwave energy at 1050°C for 3 minutes under 50°C/minute heating rate.

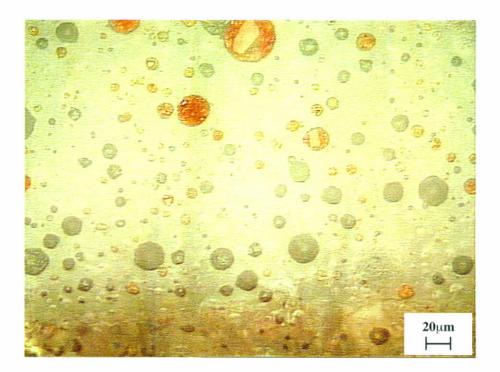


Fig. 5. Optical Micrograph showing the general aspects of glaze/body interface and glaze bulk, of a single fired in a conventional vitrified glazed stoneware floor tile mass in conventional eletric furnace during 3 minutes under heating/cooling rate of 50°C/minute.