SEM/EDX TO EVALUATE THERMAL TREATMENTS IN THE DETERMINATION OF MOISTURE EXPANSION IN CERAMIC TILES

Mansur, H. S.; Mansur, A. A. P.

Department of Metallurgy and Materials Engineering – Federal University of Minas Gerais
Rua Espírito Santo, 35/206 – Centro, 30.160-030, Belo Horizonte/MG, Brazil
e-mail: hmansur@demet.ufmg.br

Moisture Expansion (ME) is commonly defined as an increase in physical dimensions of ceramic products. These pieces undergo irreversible expansion (under natural climatic conditions) once they cool in the kiln and then are exposed to the action of water or water vapour during storage and use. ME has been pointed out as one of the many causes of pathologies of tiles and ceramic tile systems. Because of this aspect, the determination of tile moisture expansion is very important. This evaluation could be made before its use, allowing us verify whether it meets its technical specifications, or after a pathology when installed, in order to address its contribution to the problem. The determination of ME is based on two steps: reheating and accelerated ageing with water. The reheating procedure, called reheating because the firing in production is the first heating, consists of a thermal cycle, with a predefined heating rate and maximum temperature, usually 900°C (high) for 1 hour or 550°C (low) for 2 hours. This step allows an estimation of past expansion. Past expansion is the increase in physical dimensions of ceramic tile after its cooling in the kiln until the moment of the test. It also provides a relatively stable datum for the determination of potential expansion (expansion that the tile will undergo in its lifetime), which is evaluated by the accelerated ageing method (water boiling or autoclave). Despite reheating being proposed in almost all standards related to the determination of moisture expansion, there are many doubts about how this thermal treatment affects the original material microstructure. Possible changes in the material, among many others, are the occurrence of neomineralizations and sintering. In the present work, we have used Scanning Electron Microscopy (SEM) coupled to an energy dispersive spectroscopy (EDX) microprobe for ceramic tile morphological characterization, and to help in the identification of the phases present. Micrographs were also obtained to evaluate the occurrence of sintering after the thermal treatments.

The samples were prepared from a commercial ceramic tile (BIIa). The material was first embedded in a block of resin and the exposed cross section was then ground and polished. Before examination by SEM, the polished surface was coated with a thin layer of gold. The micrographs were obtained in three conditions: reference, which is the ceramic tile as supplied, without any further thermal treatment (IC), reheated for 120 minutes at 600°C (R1) and reheated for 60 minutes at 900°C (R2).

SEM images, associated with EDX spectra, show the presence of a glassy phase and its composition (Si, Al and K are the major constituents). These characterization methods also allow the identification of other phases, which are usually found in ceramic systems. In Figure 1, for example, we can see the image and the EDX spectrum of a phase immersed in the glassy matrix. Based on its spectra, it should be a potassium feldspar ($KAlSi_3O_8$), which remains after the glassy phase formation.

Regarding the occurrence of changes in microstructure due to reheating, it was not possible verify neomineralizations with these techniques, but ceramic particle sintering was observed using SEM images. Figure 2 shows the micrographs we have obtained for sample BIIa, before and after the thermal treatments. We can identify a reduction in the network of interconnected pores with irregular shape (Figure 2a; indicated as C) and an increase in the number of small pores, some of them quite rounded (Figure 2b; indicating arrows) after reheating. In R3 situation (Figure 2c), 900°C / 60 minutes, we can observe that the small pores almost completely disappear and the ones that remain have a spherical shape.

SEM coupled to EDX is shown to be an important tool for identification of the distribution and composition of glassy and other phases. The proposed use of these techniques to verify occurrence of neomineralizations during the thermal treatments to determine past expansion was not possible. However, the micrographs in the three study situations have showed evidence of the occurrence of sintering due to reheating. Based on this information, further microstructural study is necessary to re-evaluate the standards on reheating in the determination of moisture expansion.

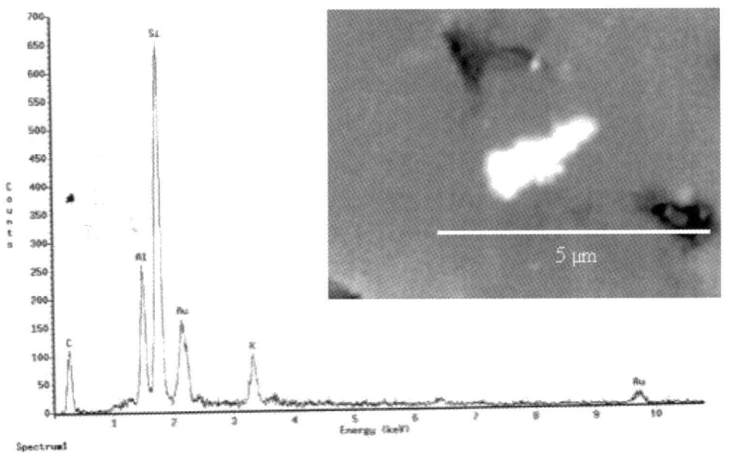

Figure 1. EDX spectrum of the immersed phase shown in detail (Backscattered electrons, BSE).

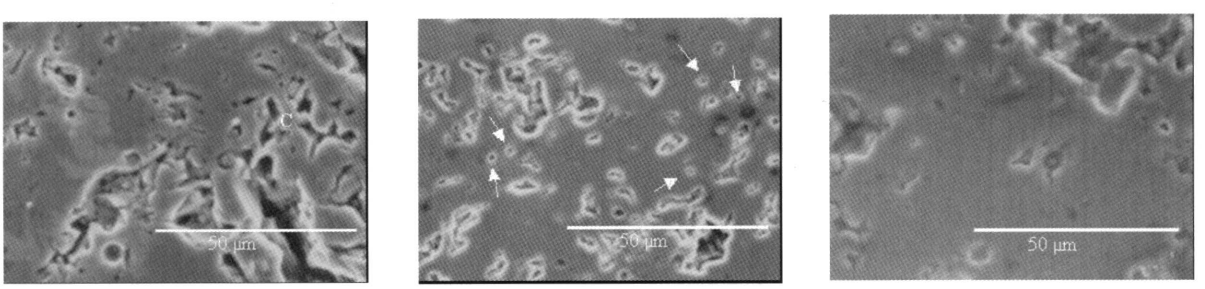

Figure 2. Effects of reheating thermal treatments for sample BIIa. (a) Reference. (b) Reheating 600°C/120 minutes. (c) Reheating 900°C/60 minutes.