INFLUENCE OF PARTICLE SIZE REDUCTION ON FRIT REACTIVITY WITH THE DISPERSANT MEDIUM

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1. ABSTRACT

This study examines how the particle size reduction of some industrial glazes affected their reactivity with the dispersant medium and its consequences in the finished product. Six industrial frits were selected for the study: a calcium and zinc transparent frit, a zircon opaque frit, a calcium matt frit, a barium matt frit, a strontium matt frit, and a barium and zinc matt frit. The particle size distributions studied exhibited a mean diameter of about 10μm, 5 μm, and 0.7μm. The microstructural and visual characteristics of the fired glazes were then determined. It was verified that, in the case of the barium frit, important alterations developed in its reactivity with the organic dispersant, which significantly altered glaze behaviour in the firing stage and fired glaze visual characteristics.

2. EXPERIMENTAL PROCEDURE

Six glazes were prepared by adding 8 wt% kaolin to each chosen test frit. Three suspensions of different particle sizes were prepared for each glaze. The distributions with a mean diameter of about 10µm (T1) and 5µm (T2) were obtained by conventional milling. The finest distribution, with a mean diameter of 0.7µm (T3), was obtained by milling T2 in a high-speed mill, using an organic suspending agent (Figure 1). The sintering/melting curves were obtained for each type of glaze and particle size in a hot stage microscope, determining test piece silhouette area, A, continuously at 25K/min, and expressing the results as $\varepsilon_A = \ln(A/A_0)$, where A_0 is test piece silhouette area at sintering onset. In addition, the suspensions were applied onto conventional unfired ceramic bodies and fired in a laboratory kiln according to a standard cycle. After firing, the microstructural characteristics were determined by SEM–EDX.

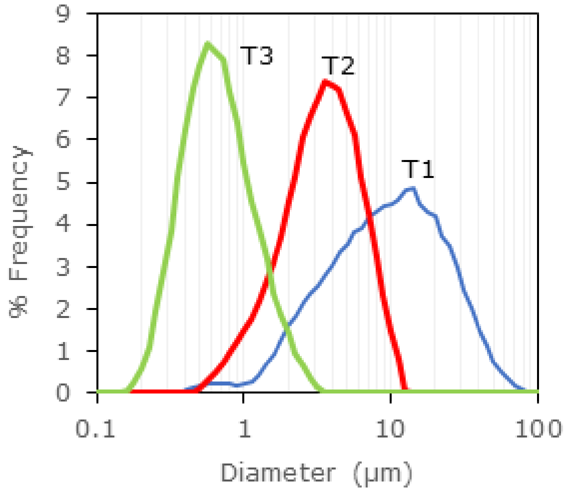

Figure 1. *Particle size distributions corresponding to the glaze prepared with the frit of high barium content.*

3. RESULTS AND DISCUSSION

It was verified that only distribution T3 of the barium frit exhibited anomalous behaviour both in sintering/melting and in the visual appearance and microstructure of the fired glaze. It was observed (Figure 2a) that, as the mean diameter of the distribution decreased, sintering began at lower temperatures. However, in the case of T3, process arrest (shrinkage stabilisation), due to celsian crystallisation, occurred when densification had already practically ended. In contrast, for particle sizes T1 and T2, this phenomenon occurred when the glaze was still very porous, shrinkage starting again at higher temperatures when celsian began to melt. Such behaviour is typical for frits that devitrify abundant crystalline phase. The anomaly lay in the fact that, for distribution T3, when partial melting of celsian began, the piece expanded greatly, evidenced in the fired glaze by excessive closed pore growth (Figure 3). Preheating the glaze at temperatures above 500ºC (Figure 2b) only encouraged crystallisation (shrinkage arrest occurred at lower shrinkages), owing to the nucleating effect of thermal treatment. However, expansion still took place. This behaviour was due to interaction between the barium in the glaze and the organic medium used.

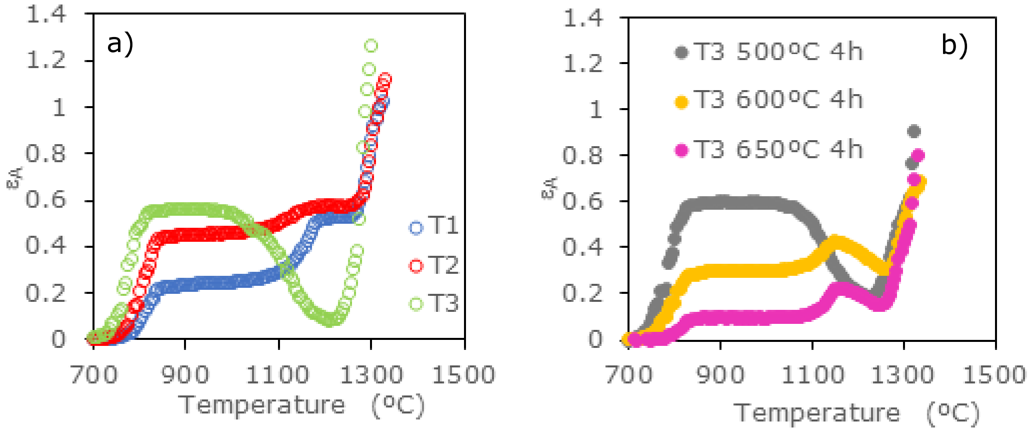

Figure 2. *Evolution of test piece silhouette surface shrinkage,* $\varepsilon_A=\ln(A/A_0)$*: a) corresponding to the three particle size distributions of the barium glaze and b) T3 with different preliminary thermal treatments.*

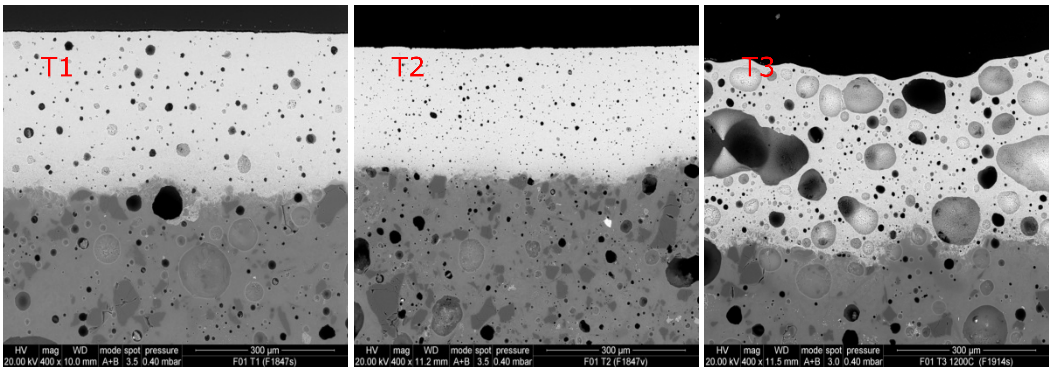

Figure 3. *Microstructure of the glazes obtained using the frit with high barium content at different particle sizes: T1, T2, and T3.*