

High-alumina porcelain with the addition of a Li_2O -bearing fluxing agent

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Abstract

We have studied the influence of increasing the amount of β -spodumene ($\text{LiAlSi}_2\text{O}_6$), as a Li_2O -containing flux, on the phase composition, the microstructure evolution and the physical properties of high-alumina porcelain. Quartz reacts with β -spodumene in the temperature range 1150–1250 °C, forming lithium aluminium silicates with a larger amount of SiO_2 . The presence of lithium minerals contributes to a lower CTE for the fired bodies. At 1300 °C an improved flexural strength is achieved with compositions containing 1.0 or 1.2 wt.% of Li_2O , as a result of a more uniform microstructure. With increasing amounts of Li_2O the overfiring effect is greatly enhanced. The most favourable characteristics from an industrial perspective with regard to flexural strength and deformation during firing were attained by using a high-alumina porcelain composition containing 1.0 wt.% Li_2O .

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1. Introduction

Technical porcelains, which are classified in the alkaline alumina silicate porcelain group, find diverse application in electrotechnics, most often as the insulating parts in electricity transmission, distribution and protection. The developments in electrical engineering dictate the need for technical porcelains with high reliability, and it has been shown that the reliability of porcelain strongly depends on the microstructure and phase composition.^{1,2} The typical requirements for an electrical porcelain are a high mechanical strength and a good thermal shock resistance. To achieve a better mechanical strength for the porcelain body, alumina with its higher Young's modulus is used instead of quartz as a filler.^{3,4} This increasing amount of alumina greatly contributes to the strength enhancement, but at the same time causes the thermal expansion to increase, thus reducing the thermal shock resistance. Therefore, to reduce the expansion of high-alumina porcelain it is necessary to reduce the expansion of the glassy phase or introduce low-expansion phases.

The viscous liquid that is formed during the firing of the porcelain body enables densification and the formation of the final microstructure.⁵ The temperatures at which this liquid is formed are lowered by the introduction of flux, with sodium- and potassium-based feldspars being the most commonly used fluxes in porcelain. It has also been reported that a combination of feldspar and nepheline syenite contributes to the increased mechanical strength of porcelain bodies.⁶ The level of vitrification, the pyroplastic deformation and the microstructure are affected by the mineralogical composition of the raw materials and by the level of equilibrium achieved.⁷

The use of lithium-bearing minerals as a liquid-phase sintering aid has been investigated in various types of ceramics. It was reported that spodumene as a partial replacement for feldspar can lower the firing temperature of sanitary chinaware bodies with a high content of flux when the feldspar-to-spodumene ratio is 70:30.⁸ Bodies of electrical porcelain with a fairly high thermal shock resistance and mechanical strength were developed using 18.5–20.5 wt.% of spodumene and firing at 1380 °C.⁹ Good properties for tableware porcelains were attained when the Li_2O content did not exceed 1.5 wt.%, and these compositions matured at temperatures 100–120 °C lower than standard, triaxial porcelain formulations.¹⁰ The presence of spodumene, when 10 wt.% was added as a partial replacement for sodium feldspar,

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reduced the firing temperature and the shrinkage of the body mixes for stoneware tiles.¹¹ The use of lithium-bearing minerals as a liquid-phase sintering aid has also been investigated in other types of ceramics, for example, in mullite ceramics,^{12,13} to lower the firing temperature and in alumina ceramics¹⁴ to improve the thermal shock resistance. The influence of a Li₂O-bearing fluxing agent in a high-alumina porcelain body has not yet been investigated.

The aim of our work was to investigate the influence of Li₂O in combination with K₂O as a flux on the sintering behaviour and the physical properties of high-alumina porcelain bodies. The increasing amounts of Li₂O were introduced in compositions via a commercial spodumene concentrate. The investigations were assisted by a study of the phase composition and the microstructural development at different firing temperatures. All the characteristics were compared with an alumina porcelain body containing K₂O and Na₂O as the flux. New body formulations, suitable for extrusion and industrial applications, were developed.

2. Materials and experimental procedure

We compared the reference formulation (EN) with three model formulations (A, B, and C) of high-alumina porcelain. The model formulations were prepared using calcined alumina (HVA, Almatix GmbH, D), kaolin (Zettlitz Ia, Sedlecký kaolin a.s., CZ), illite clay (M1M, Stephan Smidt, D), bentonite (Portaclay A, Ankerpoort NV, NL), calcined spodumene (SC 7.5, Gwalia Consolidated Ltd, Australia) and potassium feldspar (Dorkasil 90, Dorfner, D). Additionally, 0.3 wt.% of binder based on polyvinyl alcohol was admixed to improve the plasticity for extrusion.

Conventional ceramic processing routes were used to prepare the model alumina porcelain bodies on a laboratory scale. First, the materials were wet milled with alumina balls for around 11 h. The particle size and the particle size distribution after milling were measured by laser sizer (Matroc Microtrac S3500). The distribution for all the model compositions was bimodal, with the first maximum around 0.6 μm and the second one between 3 and 4 μm . The EN reference composition was milled in an industrial mill with silica balls, and its particle size distribution was comparable to that of the model compositions.

Suspensions of the milled model compositions A, B and C were dried on plaster moulds at room temperature. The EN suspension was spray dried and mixed with water in a Z-mixer. Test samples in the form of extruded circular bars were prepared on a laboratory vacuum-extrusion machine (Netzsch, V5). The samples were dried for one day at room temperature followed by 4 h drying in an oven at 105 °C.

The samples were fired in a laboratory electric furnace (Naber N20/14) in the temperature range 950–1350 °C or in a gas kiln. Before firing the test samples were set in refractory saggars. In the laboratory furnace the firing process involved a 4 °C/min heating rate and a 15-min soaking time, and was the same for each firing, except for the maximum temperature. The seven different maximum temperatures were 950, 1050, 1150, 1200, 1250, 1300 and 1350 °C. After soaking at the maximum tem-

perature the samples were cooled to room temperature at the natural rate for the laboratory furnace. In the gas kiln the firing was carried out at a maximum temperature of 1315 °C and the duration of the complete firing cycle was 11 h. The atmosphere was changed during the firing cycle, with oxidation up to 1100 °C, followed by reduction until the end of the firing at the maximum temperature. The cooling process was in an oxidising atmosphere.

Characterization of the investigated high-alumina porcelain bodies after firing involved both physical and structural characterizations. The physical characterization included assessments of the open porosity, the bulk density, and the thermal and mechanical properties. The structural characterization included measurements of the phase composition and the microstructure.

The bulk density and the open porosity were measured using the standard method, in accordance with the IEC 60672-2 standard¹⁵ with deionized water as the immersion medium. Two samples of half-cut test bars, $\varnothing 10 \times 120$ mm, were used for the open-porosity and bulk-density measurements.

The behaviour of the samples during heating was assessed from the DTA and TG curves, which were recorded on powdered mixtures in the temperature range 25–1200 °C (Netzsch STA 429, 5 °C/min), and from sintering curves recorded on test samples in the form of bars $\varnothing 7 \times 25$ mm in the temperature range 20–1340 °C using a dilatometer (Baehr, heating rate 5 °C/min). The linear thermal expansion and the corresponding curves of the coefficient of thermal expansion (CTE) were measured on fired test bars, $\varnothing 6 \times 50$ mm, using a dilatometer (Netzsch DIL 402EP, heating rate 5 °C/min) in the temperature range 30–980 °C. The thermal shock evaluations were determined in accordance with the IEC 60672-2 standard.¹⁵ The deformation of the samples during firing in a gas kiln was measured as the height reduction of the test bars relative to a horizontal surface. The test bars, $\varnothing 10 \times 180$ mm, were positioned on a refractory support with a height of 100 mm and a span of 150 mm. Five test bars were measured to evaluate the deformation during firing.

The flexural strength was determined on circular test bars, $\varnothing 10 \times 120$ mm, using a three-point bend tester (Netzsch 401/3) with a 100-mm span. Seven test bars were used for each set of measurements. The maximum and minimum values were eliminated from the results, thus the flexural strength was calculated as the average of five measured values.

The phase composition was studied from X-ray powder diffraction (XRD) patterns recorded on powdered samples (<63 μm) at room temperature (PANalytical, X'Pert PRO MPD, The Netherlands) using Cu-K α radiation. The data were collected in the 2θ range from 10 to 70°, in steps of 0.034°, with an integration time of 100 s. The crystal phases were identified using a complete ICDD powder pattern file and the X'Pert PC software. The microstructure was observed using optical and scanning electron microscopy (SEM, JEOL 5800 equipped with an energy-dispersive X-ray analyzer (EDX)). The samples for the microstructure observation were cut from $\varnothing 10$ -mm test bars with a cross-sectional orientation and prepared by grinding and polishing using standard metallographic techniques. Prior to any analysis in the SEM, the samples were coated with carbon to provide the electrical conductivity.

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