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Porcelain production by direct sintering

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ABSTRACT

Most of the energy consumed during the processing of porcelains is accounted by the heating rate, dwell time and cooling rate employed. Efforts are made in this study to lower the total energy consumption, and thereby total emissions, by optimizing the porcelain processing parameters. Porcelain samples processed at 5, 15 and 30 °C/min heating rates were compared with samples processed under direct sintering. X-ray diffraction and microstructural characterisation confirmed that the phase evolution, such as mullite formation in the porcelain, is heating rate independent while glass formation is heating rate dependent, and directly sintered porcelain had physical and mechanical properties comparable to conventionally heat-treated samples. Direct sintering reduced total processing time by ~50% and also lowered the sintering temperature from 1200 °C to 1175 °C, suggesting that the direct sintering method provides benefits meaning it may in future replace conventional procelain processing.

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1. Introduction

Porcelains are traditional ceramics, used extensively in many household, scientific and engineering applications [1–4]. High mechanical strength, low water absorption, translucence and durability are key technological advantages of porcelains [1-4]. A porcelain body is typically made of clay, feldspar and quartz along with other constituents to promote desired characteristics [1,2]. Clay provides plasticity to the mixture in the forming process while feldspar is a fluxing agent, which promotes glass formation by reacting with amorphous silica from clay and reduces sintering temperature. A fired porcelain consists predominantly of mullite, glass and undissolved quartz [1,3,5]. Mullite is formed in porcelains which are fired above 1100 °C and its amount is found to be constant over the temperature range 1200–1400 °C [6–8]. Viscous glass phase starts to form at different temperatures depending on the chemical composition of the porcelain (for potash feldspar, the eutectic melt forms at \sim 990 °C whereas the eutectic melt of soda feldspar forms at \sim 1050 °C), and the chemistry of the glassy phase in fired porcelain is located on the glass formation boundary from 1200 to 1400 °C [8–10]. Quartz dissolution is significant above 1200 °C [7,11-13] and has been demonstrated to increase as a function of temperature, and dwell time regardless of heating rate [8].

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http://dx.doi.org/10.1016/j.jeurceramsoc.2016.07.013 0955-2219/© 2016 Elsevier Ltd. All rights reserved. The major energy consumption during manufacturer of porcelains and other ceramics occurs during the firing process [14]. Hence, efforts have been made to lower the total energy consumption by modifying the heating ramps and chemical compositions [5,15–18].

The term "fast firing" was introduced to ceramic production in 1965 [19]. Studies on fast firing of traditional ceramics such as clay bricks [20], tile [21], and red clay [22] indicate that the properties of fast-fired products are comparable to those of the conventionally-fired products [21,22]. In modern porcelain manufacture, the overall firing times (cold to cold) are 35 min for mosaic tile, 40 min for wall tile, 60 min for porcelaneous-stoneware floor tile, 2 h for dinnerware, and 7 h for sanitaryware [19].

Likewise, Field Assisted Sintering Techniques (FAST) have become potential procedures for sintering ceramics in the past few years since they can produce ceramics in a few minutes while exhibiting the same fired characteristics of conventionally-fired ceramics. However, sophisticated equipment and process control are crucial and need further development for the next generation of ceramic processing technologies. Direct sintering (DS) has been demonstrated to produce glass-ceramic composites from waste materials [23]. In the DS process, samples are transferred into the furnace which is preheated to sintering temperature followed by dwelling and cooling the samples to room temperature. This sintering method employs very fast heating rate (from room temperature to peak temperature in seconds) compared with conventional sintering (typically 10 °C/min); so minimizing the total energy consumption and total processing time. The aim of this



Fig. 1. Temperature-time profiles and change in total porcelain processing time with respect to change in heating rate. Bars at the bottom of figure represent reduction in energy consumption with the change in processing time.

work is to demonstrate the feasibility of DS of porcelain by comparing microstructural evolution and end product properties of DS porcelain to those of conventionally sintered porcelain.

2. Materials and methods

Industrially-processed spray dried porcelain powder obtained from a porcelain tile manufacturer was used in as-received state without additional processing. The powder exhibited average granule size between 300 and 400 μ m (~60%). Mineralogically, the porcelain powder consists mainly kaolinite, albite, and quartz.

 2 ± 0.05 g of spray dried powder was uniaxially cold-pressed under 35 MPa into 13 mm diameter and 7 mm thick pellets in a steel die. The pellets were dried overnight at 110 °C and stored in an oven until further experiments.

Thermal behaviour of the spray dried powder, with mean particle size (D_{50}) of 10 μ m, was characterised by using a simultaneous thermogravimetry-differential thermal analyzer (TG-DTA, Netzsch STA 449C, Jupiter, Selb, Germany) under continuous air flow at a rate of 50 ml/h. from 25 to 1200 °C at a heating rate of 10 °C/min and instrument cooling rate.

The pellets were heated at rates of 5, 15, 30 °C/min to the sintering temperatures of 1150, 1175 and 1200° C. Direct sintering was performed by transferring samples directly into a front loading box furnace at the given sintering temperatures. All samples were held for 15 min at the respective sintering temperatures and cooled to 700 °C at 30 °C/min followed by cooling in furnace to room temperature. All firing schedules are presented in Fig. 1. Bars located below the firing schedule represent total energy consumption used which reduces with increasing heating rate. The percentage of total energy usage of each firing schedule was calculated using the area under the curves. 5 °C/min was used as the baseline representing 100% energy consumed and energy usage values for other heating rates were normalized to this value.

Phase contents of the sintered pellets were characterized using X-ray diffraction (XRD, Bruker D2 Phaser, Madison, WI, USA). Pellets were ground in a borosilicate glass mortar with a pestle to obtain fine powder ($<10 \,\mu$ m) prior to XRD. Diffraction patterns were



Fig. 2. TGA/DTA plot for the green porcelain powder.

analysed using commercial software (X'pert high score plus software, Pan Analytical, Cambridge, UK). Three non-overlapping peaks of mullite and albite were selected to calculate the peak area. To quantify the amount of mullite $-(1\ 1\ 0), (0\ 0\ 1), (2\ 2\ 0)$ and $(0\ 0\ 2), (0-3\ 1), (-2\ 0\ 1)$ of albite peaks were used while background was estimated via the Pearson-VII distribution function (with residual error less than 5%).

Apparent bulk density (ABD) and water absorption (WA) were obtained using an immersion method using ASTM standard (C20-00). Microstructures were examined by secondary electron imaging using a scanning electron microscope (JEOL JSM-5610LV, Tokyo, Japan). Diametral compressive strength (DCS) measurements of the samples sintered under different heating conditions was made using a Zwick/Roell Z010 unit at a crosshead speed of 1 mm/min with load applied until sample failure [24]. Vickers hardness (HV) was determined using a microhardness tester (Zwick/Roell Indentec ZHV) integrated with computer software at 1000g load, and loading duration of 10 s.

3. Results and discussion

The firing schedules used (Fig. 1) mean that the energy and processing times used in this study were only different on heating. Fig. 1 shows energy consumption for different heating rates of samples sintered at 1200 °C. By considering the slow heating rate at 5 °C/min as the baseline which consumed 100% energy then using a rate of 30 °C/min reduces the energy consumption by 40%, while the direct sintering method can reduce energy consumption by ~50%.

Fig. 2 shows TGA/DTA of the green porcelain revealing two large endothermic peaks from evaporation of physically-bound water at 100–200 °C and dehydroxylation of chemically-bound water of the clay substances between 450 and 600 °C. However, the endothermic peak of quartz inversion at 573 °C is masked by overlap with the dehydroxylation peak of the clays. The small exothermic peak around 940 °C suggests initial formation of the spinel-alumina phase from the clay species. The total weight loss of this porcelain body is ~5%, 1% from physically-bound evaporation, and 4% from chemically-bound water decomposition from the clays.

Physical properties of the samples sintered under different sintering conditions are shown in Fig. 3(a–b). Fig. 3a indicates that the changes in heating rate have minimal influence on ABD values after sintering at 1150 °C and 1175 °C which range between 2.35 g/cm^3 and 2.40 g/cm^3 . However, after sintering at 1200 °C, there is a significant change in ABD values which are 2.25 g/cm^3 (5 °C/min), 2.26 g/cm^3 (15 °C/min) and 2.31 g/cm^3 (30 °C/min) and 2.33 g/cm^3 for direct sintered samples. This change is due to bloating—it is not a heating rate effect. In other words, the bloating effect has much

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