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Porous cordierite-based ceramics processed by starch consolidation casting – Microstructure and high-temperature mechanical behavior

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ABSTRACT

Porous cordierite-based ceramics with different microstructural features and mechanical behavior were formed by starch consolidation casting (SCC) using native potato and corn starches and sintered at 1275, 1300 and 1330 °C. The composition and microstructure of the ceramic materials were investigated via quantitative phase analysis using X-ray diffraction (with Rietveld refinement), the Archimedes method, mercury porosimetry, scanning electron microscopy and optical microscopy with stereology-based image analysis. The mechanical behavior of samples was evaluated by diametral compression tests at room temperature, 1000 and 1100 °C. The type of starch used and the sintering temperatures were the main factors determining the characteristics of the developed porous microstructures. Materials prepared with corn starch achieved the lowest porosity and the lowest values of mean chord length, mean pore distance and pore throat size. Because of these features, these materials thus presented, in general, higher values of apparent Young's modulus, elastic limit and mechanical strength than those prepared with potato starch. Despite the presence of a silicate glassy phase, both porous materials, mainly those prepared with corn starch, still enhanced the basic mechanical properties at high temperature, in particular, the mechanical strength and the apparent Young's modulus due to the special combination of the porous microstructure features.

1. Introduction

Cordierite ceramics ($\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$) are well known for their low thermal expansion coefficient, which provides them with a high thermal shock resistance suitable for applications involving temperature cycling. Therefore, in combination with satisfactory mechanical properties, chemical stability and corrosion resistance, low thermal conductivity, a low dielectric constant and low dielectric loss factor, the range of applications for cordierite-based ceramics is very wide, ranging from kiln furniture [1] and thermally and electrically insulating ceramics [2,3] to diesel particulate filters [4] and other filters [5], as well as membrane supports [6], catalyst supports [7] and electronic substrates [8], to name only the most important. Although the properties of cordierite-based ceramics have been investigated for quite some time [9–11], a look into the current literature shows that their processing and properties are still among the hot topics of ceramic research and development [12–14].

Nowadays, porous cordierite-based ceramics and ceramic foams in which the thermal insulation behavior, specific surface and

permeability can be tailored by controlling the porosity, pore surface area and pore size (and shape), are of particular interest [15–21]. In this field, one of the early applications of starch consolidation casting (SCC) concerned cordierite ceramics [15,17,18].

Currently, the problem is that cordierite-based ceramics, especially those types that are prepared by cheap natural raw materials (mainly kaolin and talc), are complex multiphase materials that may contain, apart from cordierite as the main phase, several other crystalline phases (mullite, alumina, cristobalite, magnesia-alumina spinel [13,14,22,23], enstatite [14,24] and sapphirine [24]) and usually a siliceous glassy phase [23]. When natural raw materials are used, it is clear that even for nominally similar compositions the resulting materials can be different and exhibit different properties. This has significant implications for industrial production, because the raw materials that are available in a certain region of the world may differ from those available elsewhere.

Previous work by the authors concerned porous cordierite-based ceramics prepared by SCC from Argentinian kaolin and Chinese talc, and using potato and cassava (tapioca) starches as a pore former and

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stiffener [17,18]. While talc is a strategic raw material for which world production is largely concentrated in certain countries (mainly China, Australia and USA) [25] and distributed by a few world-wide companies, kaolin is usually from more local deposits that differ from region to region. In Central Europe, for example, Czech kaolin is one of the most widely used. On the other hand, cassava (tapioca) starch is not commonly produced in many industrialized regions of the world, such as in Europe. Therefore, the present work investigates the porous microstructures developed in cordierite-based ceramics prepared from Czech kaolin, Australian talc and potato and corn starches. Moreover, the high temperature mechanical behavior of cordierite-based ceramics, an aspect that has been scarcely studied in spite of its importance for successful applications of these materials, was also investigated. Finally, the obtained results were discussed from the viewpoint of phase evolution, microstructure and high-temperature mechanical properties.

2. Experimental procedure

2.1. Processing and characterization of porous green bodies

A cordierite precursor mixture (denoted PM) composed of kaolin 37 wt% (Sedlec Ia, Sedlecký kaolin a.s., Božičany, Czech Republic), talc 41 wt% (EC 75, Luzenac Group, Three Springs, Australia) and alumina 22 wt% (CT3000 SG, Almatix GmbH, Ludwigshafen, Germany) was used as raw ceramic material for obtaining cordierite-based bodies [26]. Commercially available potato starch (Solamyl, Natura a.s., Havlíčkův Brod, Czech Republic) and corn starch (Gustin, Dr. Oetker, Kladno, Czech Republic) were used as stiffening agents (binders) and pore formers. The complete characterization of both the ceramic and starch powders was reported in a previous work by the authors [26]. The main features of the raw materials used are shown in Table 1.

Green disks 18.6 ± 0.9 mm in diameter and 3.7 ± 0.7 mm in height were formed by SCC. Aqueous ceramic-starch (PM-potato or PM-corn) suspensions with a total solid content of 60 wt% were prepared by mixing (impeller mixer) ceramic powders in distilled water with 1 wt% Dolapix CE-64 (Zschimmer & Schwarz, Lahnstein, Germany) and 0.5 wt% sodium naphthalenesulfonate (Nutrimer Argentina S.A., Buenos Aires, Argentina), both amounts with respect to the ceramic solid content, homogenizing in a ball mill for 4 h, degassing for 20 min, and finally homogenizing the inorganic solids with starch and extra water for 1–2 min in order to obtain the desired total solid loading. The experimental conditions used for preparing these systems were previously established and reported [26]. The volume fraction of starch related to the suspension volume (15 vol%) was calculated by considering the pycnometric density value (2.40 ± 0.05 g/cm³) determined by He-pycnometry for the precursor mixture with starch. This amount was selected taking into account previously obtained results [27] and data reported in the literature in order to obtain ceramic materials with porosities higher than 35% by starch consolidation casting [28].

Table 1
Characterization of raw materials.

Raw materials	Chemical analysis	Mineralogical analysis	D_{50} (μm)
Kaolin	$Al_2O_3/SiO_2 = 0.8$ $Fe_2O_3, CaO, MgO, Na_2O < 1.9$ wt% $K_2O \sim 0.9$ wt%	Kaolinite ^a ($Al_2Si_2O_5(OH)_4$) Quartz ^b (SiO_2) Hallosite ^b ($Al_2Si_2O_5(OH)_4$)	3.2
Talc	$SiO_2/MgO = 1.9$ $Fe_2O_3 \sim 0.9$ wt% $CaO \sim 0.4$ wt%	Talc ^a ($Mg_3Si_4O_{10}(OH)_2$) Traces of hematite (Fe_2O_3)	9.0
Alumina	$Al_2O_3 = 99.85$ wt% Alkaline, alk. earth impurities < 0.2 wt% Silica, iron oxides ~ 0.05 wt%	Corundum (Al_2O_3)	0.8
Potato starch	Polysaccharide ($C_6H_{10}O_5$) _n , trace elements K, P, Ca < 0.2 wt%, in total		45
Corn starch	Polysaccharide ($C_6H_{10}O_5$) _n , trace elements K, P, Na, Cl < 0.2 wt% in total		15

^a Majority phase.

^b Secondary phase.

The prepared suspensions were poured into cylindrical stainless steel molds which were covered with Teflon, and subsequently heated in an electric stove with forced circulation of air (UFP 400, Memmert, Schwabach, Germany) at 80 °C for 4 h and dried at 40 °C for 12 h. The experimental conditions (temperature and dwell time) used for the consolidation of suspensions were established on the basis of results previously reported by the authors [17] and other published data [15]. Once the consolidation was completed, the samples were taken out of their molds.

A previous study [26] indicated that the total green porosity of the both materials was very similar ($38 \pm 6\%$ for PM-potato and $36 \pm 5\%$ for PM-corn) and only a small amount of cavities with starch-granule-specific morphology and interconnected channels (windows) in the cavity walls were developed. These particular features, which were not usually reported in previous work [29,30], could be attributed to the combined effect of certain specific characteristics of the gelatinization process and the presence of phyllosilicate raw materials with oblate particles (kaolin and talc) in the studied systems [26].

2.2. Microstructural evolution as a function of temperature

Thermal treatments for the calcining and reaction-sintering of green disks were performed in an electric furnace with SiC heating elements (RHF 16/3, Carbolite, Hope Valley, UK). Three sintering temperatures (T_s) were applied, viz., 1275, 1300 and 1330 °C, while keeping the thermal cycle constant as follows: heating rate 1 °C/min up to 650 °C; 2 h dwell at 650 °C; heating rate 3 °C/min up to T_s , 4 h dwell at T_s ; cooling rate 5 °C/min down to room temperature. The burnout temperature (650 °C) was selected based on previous results from thermogravimetric analyses (TGA-50, Shimadzu, Kyoto, Japan; at 10 °C/min up to 700 °C, in air) of the native starches. During the first stage of the thermal treatment (≤ 650 °C), complete removal of the starch and organic additives is achieved (burnout), together with the dehydroxylation of kaolinite, while dehydroxylation of talc and the reaction of the ceramic precursors occur, concomitantly with sintering, during the second stage (reaction-sintering).

The crystalline phase evolution as a function of T_s was studied by qualitative and semi-quantitative phase analysis via X-ray diffraction (X'Pert PRO, PANalytical, Almelo, The Netherlands, with Cu K α radiation at 40 kV and 40 mA, and 0.01 steps per second) of powder samples of sintered disks. In particular, semi-quantitative analysis was performed using Rietveld refinement, which was carried out employing a commercial multipurpose profile-fitting software (FullProf) [31]. The general methodology employed to obtain the phase contents from the Rietveld optimized scale factors normalizes the results to 100% of crystalline phases (i.e. the presence of an amorphous phase is not taken into account). Nevertheless, bearing in mind that a siliceous liquid phase is generated during the thermal treatments of the cordierite precursor mixture used in this work, and therefore, a silicate glassy

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