



Electrophoretic deposition of yttria-stabilised zirconia powder from aqueous suspensions for fabricating ceramics with channel-like pores

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Abstract

Electrophoretic deposition with simultaneous gas bubble formation by electrolysis can be used for producing ceramic green bodies, typically few millimetres in thickness, with unidirectionally aligned channel-like pores. The method is successfully applied to yttria-stabilised zirconia. Two types of aqueous suspension compositions are investigated. Suspensions with acetic acid additions are particularly suitable for forming green bodies with fine pore channels. Only small amounts of acetic acid, promoting the gas evolution, are needed for this purpose. Dissolution of yttria in the acidic range has to be considered, but the required low acid concentrations do not measurably affect the yttrium content of the deposits. Yttria dissolution can be minimised by a suspension composition containing an anionic polyelectrolyte and ammonia instead of acetic acid. The ammonia concentration influences the size of the tubular pores of the deposits formed under constant-voltage conditions. Using structured deposition electrodes, the regularity of the pore arrangement can be enhanced.

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1. Introduction

Electrophoretic deposition (EPD) is known as shaping, infiltration, or coating technique. Main advantages which are used in ceramic processing are the applicability of this method to powders with particle sizes in the submicron or nanometre range and the dense, homogeneous particle packing which can be obtained by EPD from well-dispersed, stable suspensions. When aqueous suspensions are used in EPD, gas bubbles are formed by electrolysis above the decomposition voltage of water. Pores resulting from this gas generation have to be prevented in most applications by suitable approaches such as the EPD at a membrane positioned in front of the deposition electrode¹ or the EPD in a pulsed DC² or asymmetric AC field³ instead of a continuous DC field. On the other side, gas bubble formation during electrophoretic deposition can be used for fabricating porous materials, but only few publications about this method are known.^{4,5} Potential applications of ceramics

with the resulting tubular or conical pores are filters,⁶ catalyst carriers, substrates for gas sensors, or porous bioceramics. Previous own publications^{7–9} describe the successful use of this electrophoretic method with simultaneous gas generation for producing zirconia ceramics with aligned pore channels from aqueous suspensions of the yttria-stabilised ZrO₂ powder TZ-3Y (Tosoh, Japan).

The commercially available powder TZ-3Y, containing approx. 3 mol% Y₂O₃, is widely used for fabricating Y-TZP ceramics (yttria-stabilised tetragonal zirconia polycrystals). The stabilising oxide Y₂O₃ prevents the tetragonal to monoclinic phase transformation, associated with volume expansion, on cooling down from sintering temperature. In this way, it enables toughening by transformation of metastable tetragonal grains to the monoclinic phase under mechanical load. The effectiveness of the stress-induced transformation toughening is mainly controlled by the yttria content and the grain size of the sintered ceramic.¹⁰ Additionally, it depends on how the yttria is distributed in the zirconia – homogeneously on an atomic scale as in the case of the commonly used co-precipitated powders or less homogeneously as, for example, in the case of ZrO₂ powders coated with Y₂O₃.¹¹ Commercial Y-TZP ceramics are prepared with Y₂O₃ contents in the range between 1.75 and 3.5 mol%,¹²

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usually between 2 and 3 mol%.¹³ The 3 mol% Y_2O_3 composition provides insurance against spontaneous transformation to the monoclinic form and allows in comparison with lower yttria contents a larger critical grain size to remain metastable on exposure to moisture at temperatures around 200 °C.¹⁰ As reported by Basu et al.,¹¹ the toughness of ceramics based on Tosoh powders TZ-3Y (3 mol% Y_2O_3), TZ-2Y (2 mol% Y_2O_3), or powder mixtures of TZ-0 (monoclinic ZrO_2 without Y_2O_3) and TZ-3Y, however, increases with decreasing total yttria content down to 2 mol%. It is shown in the same publication that the yttria content must be above 1.75 wt% to inhibit spontaneous transformation of tetragonal ZrO_2 grains during cooling from sintering to room temperature. The tetragonal-to-cubic phase ratio of commercial Y-TZP ceramics, which depends on composition, firing temperature, and holding time, lies typically in the range between 100%/0% and 60%/40%.¹²

When aqueous suspensions of yttria-stabilised zirconia powders are used in ceramic processing, it is necessary to be aware of the preferential dissolution of yttria in the acidic range.^{14–17} In the aforementioned investigations concerning the fabrication of porous ZrO_2 ceramics by electrophoretic deposition with simultaneous gas bubble formation from suspensions of the powder TZ-3Y,^{7–9} small amounts of acetic acid were added to the suspensions in order to increase and adjust the intensity of electrolytic gas evolution.

It is important that the extent of dissolution of yttria at the particle surfaces in the suspension has to remain moderate at the used acid concentrations to prevent a decline in mechanical properties of the Y-TZP ceramic.

The present paper describes the electrokinetic and rheological behaviour of the TZ-3Y suspensions as a function of the acetic acid concentration and shows examples for pore structures obtained by the EPD/electrolysis method. Particular emphasis is laid on yttria dissolution under conditions relevant for the EPD experiments in order to prove the applicability of these suspensions with respect to the required yttria content of the ceramic. The yttria content must not significantly decrease. For minimising the dissolution of yttria, suspensions containing an anionic polyelectrolyte and ammonia solution were examined as alternative composition. The suitability of this suspension type for fabricating similar porous structures as obtained using the suspensions with acetic acid was investigated.

2. Materials and methods

The used Y_2O_3 -stabilised ZrO_2 powder TZ-3Y from Tosoh/Japan had a specific surface area of 15.3 m²/g and a crystallite size of 260 Å (manufacturer's certificate of analysis). The Y_2O_3 content was 5.2% by weight.

For preparing the suspensions, the powder was dispersed in pure deionised water or deionised water containing acetic acid (first suspension type) or the commercially available polyelectrolyte solution Dolapix PC 21 and ammonia (second suspension type). The suspensions were stirred for 10 min and then subjected to ultrasonic agitation for 3 min (ultrasonic homogeniser "Sonopuls HD 2200", Bandelin, Berlin, Germany) in order to

destroy agglomerates of the fine particles. Heating during ultrasonication was avoided by means of a cooling bath. After this sonication, the temperature of the suspensions was adjusted to 21 °C.

The influence of glacial acetic acid (100% CH_3COOH , Merck, Germany) on the electrokinetic behaviour of a 40 wt% solids content suspension was investigated by measurements of the electrokinetic sonic amplitude (ESA) using the ESA 8000 System with SP-80 probe (Matec Instruments/Hopkinton, MA, USA). ESA signal (which is a measure for the zeta potential and the electrophoretic mobility) and specific electrical conductivity were measured as a function of the acetic acid concentration. The glacial acetic acid was diluted with deionised water in a ratio of 1:1 for this purpose. This solution was added in steps of 0.001 ml to the suspension (initial suspension volume: 70 ml) by an automatic titrator. (In the diagram, see Section 3.1.1, the ESA signal was plotted against the corresponding amount of undiluted glacial acetic acid per g of powder.)

The same solids content as in the above-mentioned previous own work on electrophoretic deposition from suspensions containing acetic acid,^{7–9} 40% by weight, was used for rheological measurements, investigation of yttria dissolution, and EPD experiments. In addition, a 65 wt% suspension was investigated because increased solids contents provide higher electrophoretic deposition rates.

Shear stress and dynamic viscosity of suspensions with different concentrations of acetic acid were measured as a function of shear rate by means of a rotational rheometer RheoStress RS 150 (Haake, Karlsruhe, Germany) with double-slit measuring cell DG 41 Ti. The shear rate was increased from 0 s⁻¹ to 800 s⁻¹ in 300 s, kept at 800 s⁻¹ for 90 s and then decreased to 0 s⁻¹ in 300 s.

The amount of yttria dissolved from the powder surface after 1.5 h, a typical time of contact between powder and liquid in the EPD experiments (including suspension preparation), was investigated in centrifugation experiments. Using the example of 40 wt% suspensions, the influence of an increased contact time was also shown. In this case, the suspension was divided in half: one half was stirred for 1.5 h and the other one for 24 h by means of a magnetic stirrer. After the contact time of 1.5 or 24 h, the solid was separated from the liquid by centrifugation at 5000 rpm for 1 h. The centrifugates (supernatants) were removed by a pipette and centrifuged again (at 5500 rpm for 1.5 h) to remove remaining particles. The pH values of the suspensions and of the centrifugates at 21 °C were measured using a pH meter "pH 95" (wtw, Weilheim, Germany). The yttrium content in the centrifugates was analysed by ICP-OES (inductively coupled plasma optical emission spectrometry) using an INTEGRA XM Sequential ICP-OES Spectrometer (GBC Scientific Equipment).

Rectangular planar green bodies were produced by electrophoretic deposition at constant DC voltage (5, 10, or 15 V). The deposition time, chosen depending on the suspension and the applied voltage, was 30, 35, or 60 min. Platinum foil or platinum gauze (225 meshes per cm², wire diameter: 0.12 mm) positioned on platinum foil was used as deposition electrode,

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