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Nanocrystalline yttria-doped zirconia sintered by fast firing

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ABSTRACT

Sintering of powders commonly leads to simultaneous densification and grain growth, particularly for nanocrystalline materials. Currently, methods such as spark plasma sintering (SPS), hot pressing (HP), two-step sintering (TSS) and fast firing (FF) are employed to hinder grain growth while maintaining a high densification. In this work, FF consisting in thermal treatments with high heating rates ($>500^{\circ}$ C/min) and shorter holding times (10 min or less) and conventional sintering (CS) approaches were experimentally compared in the sintering of commercial yttria doped zirconia (3YSZ and 8YSZ) compacts. CS-samples presented larger grain sizes by a factor of ~2 and ~4 in comparison to the initial 3YSZ and 8YSZ powders. Conversely, FF method significantly suppressed grain growth with a growth factor of ~1. Those results and comparison with previous work indicated that high heat inputs could indeed minimize grain growth.

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1. Introduction

Fine-grained materials constitute a paramount research focus considering their enhanced electrical [1–3], mechanical [4,5], optical [6] and thermal [7] properties. However, microstructural control during sintering is quite challenging due to overlapped thermally activated phenomena grain growth and densification. The smaller the powder, even more critical becomes grain growth due to the reactivity of the particles with high surface areas, and undesirable grain growth can arise even at low temperatures [8–12].

Spark plasma sintering (SPS) [6,13] and hot pressing (HP) [13,14] are currently appropriate adopted techniques for production of nanostructured ceramics. Nevertheless, the equipment involved is expensive and complex, limiting its widespread application, in particular for fabrication of large parts. Alternative routes as two-step sintering (TSS) [15] and fast firing (FF) [16] have been proposed, where the morphology control criterion relies on modifying the temperature–time profile to exploit the difference in kinetics between grain boundary diffusion (grain growth) and lattice diffusion (densification), making the latter predominant, with the advantage of feasible implementation in conventional furnaces [8,15–18]. In this regard, energy effective sintering cycles are arguably the best option to achieve a desired dense nanograined microstructure in a technologically and economically affordable way [19].

In this work, grain growth and densification were compared for FF and CS samples of partially and cubic stabilized zirconia (3YSZ and 8YSZ). The results and previous work, briefly discussed here, suggest a synergy effect between TSS and FF as favorable alternative method for sintering bulk nanocrystalline ceramics.

2. Material and methods

Commercial, high purity yttria stabilized zirconia powders, ZrO_2–3 mol% Y_2O_3 (TZ-3Y), with powder density of 6.05 g/cm^3, from Tosoh (Tokyo, Japan) and ZrO_2–8 mol% Y_2O_3 (TZ-8Y), with powder density of 5.95 g/cm^3, from Tosoh (Tokyo, Japan) were used for the experiments. Particle size measurements of the dispersion of the YSZ powders (0.5 wt%) in deionized water with dispersant were performed in a Nano/Zeta-sizer (ZEN 3600, Malvern).

Powders were compacted in a cylindrical die by uniaxial pressing to produce green compacts with 19.05 mm diameter, and ~0.5 mm height. No additives such as organic binders or dispersants were used to pellet forming to avoid carbon contaminations. The green density was 45% of the pore free (theoretical) density.

The sintering was performed in a tube furnace with a heating rate of ~500 °C/min (named fast firing, FF) and 10 °C/min (named conventional sintering, CS). In both cases, the samples were quenched in air to room temperature. The maximum temperature (T_{max}) used was setup at 1400 °C and the holding times at T_{max}

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were 1, 5, 10 and 100 min (counted after reaching the isotherm in both FF and CS). The continuous density change of the YSZ compacts was measured by the Archimedes method from 3 samples of each experimental condition. SEM micrographs were used to determine the grain sizes by image analysis on polished surfaces chemically etched for 2 min with hydrofluoric acid (2 wt%). The micrographs were taken in a Hitachi TM-3030 SEM and in a JEM-1011 TEM. XRD patterns were obtained using a Panalytical X-Pert. The monoclinic and tetragonal phase percentages were estimated from the correlation by Toraya et al. [20].

3. Results and discussion

XRD analysis of yttria-doped zirconia before and after thermal treatments is shown in Fig. 1. All 8YSZ patterns present single cubic phase without noticeable differences. On the other side, for 3YSZ the presence of monoclinic and tetragonal phases is evident. The amounts of monoclinic phase were estimated as $24 \pm 1\%$, $9 \pm 1\%$ and $8 \pm 1\%$ for green, CS and FF samples, respectively. Hence, the thermal treatments indicate no significant phases difference between FF and CS procedures.

Fig. 2 shows the theoretical density achieved by 3YSZ and 8YSZ compacts as function of holding time at maximum temperature, for conventional sintering (CS) and fast firing (FF). An initial difference of ~30% in densification is observed at holding time 1 min. Such variance between methods is attributed to the densification occurred during the heating procedure before reaching maximum temperature. The difference in density gets smaller with time, due to higher densification rate of FF samples at the holding temperature. Furthermore, the differences in relative density are negligible after holding time equal to 10 min.

The data show no difference in final densities when comparing both procedures, but FF evidently takes a much shorter time to achieve the densities, which could result in reduced grain growth. No cracks were observed in the samples after sintering. However, the thermal gradient may cause cracking due to internal stress. Thus, materials with low thermal expansion and higher heat conductivity are more suitable for FF. Furthermore, larger samples may present a gradient microstructure when comparing outer (denser) and inner (more porous) regions [18,19]. An examination of the grain sizes from micrographs in Fig. 3 for nearly dense samples (holding time 100 min), reveals larger grains for samples produced by conventional sintering for 3YSZ as compared to FF.

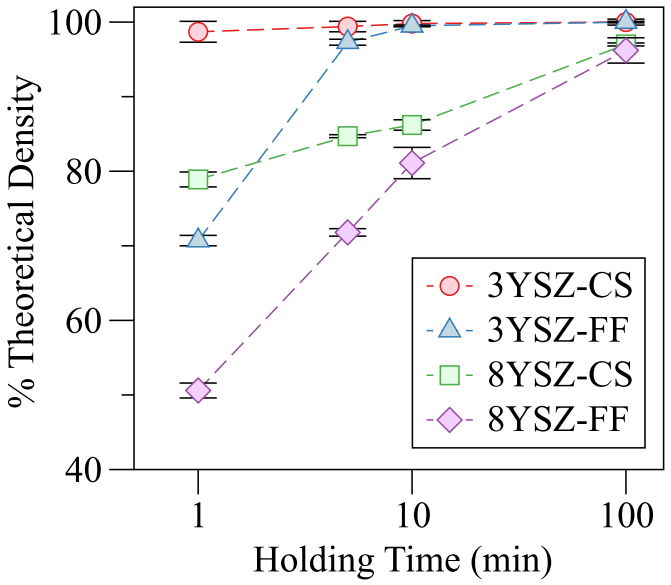

Fig. 2. 3YSZ and 8YSZ theoretical density versus holding time at maximum temperature, for compacts sintered by conventional sintering (CS) and fast firing (FF).

While this is also the case for 8YSZ, FF of this sample showed still some porosity and highly dense domains (with small grains) distributed along the microstructure.

The reduction in grain growth for FF becomes evident when observing the grain size distributions shown in Fig. 4. The grain size distribution of the unsintered 3YSZ powder is right-skewed with an average of 188 nm in agreement with TEM measurements, for a growth factor of 1.11 and 1.82 for FF and CS, respectively. For 3YSZ-samples, the differences in average grain size correspond to a decrease of 63% for FF-3YSZ compacts as compared to CS-3YSZ.

The grain size distribution of FF-8YSZ compacts is impressively close to the unsintered 8YSZ powder, revealing a limited grain growth during the process. As compared to CS-8YSZ, FF-8YSZ is narrower and with an average less than one order of magnitude: 243 nm for FF and 1103 nm for CS.

It is interesting to observe that though starting with a submicron powder (243 nm), the grain size of FF-8YSZ compacts are on the order of typical sizes achieved from powders with grain sizes below 50 nm for this same composition but using different densification techniques; i.e. 295 nm for TSS, 210 nm for SPS, and

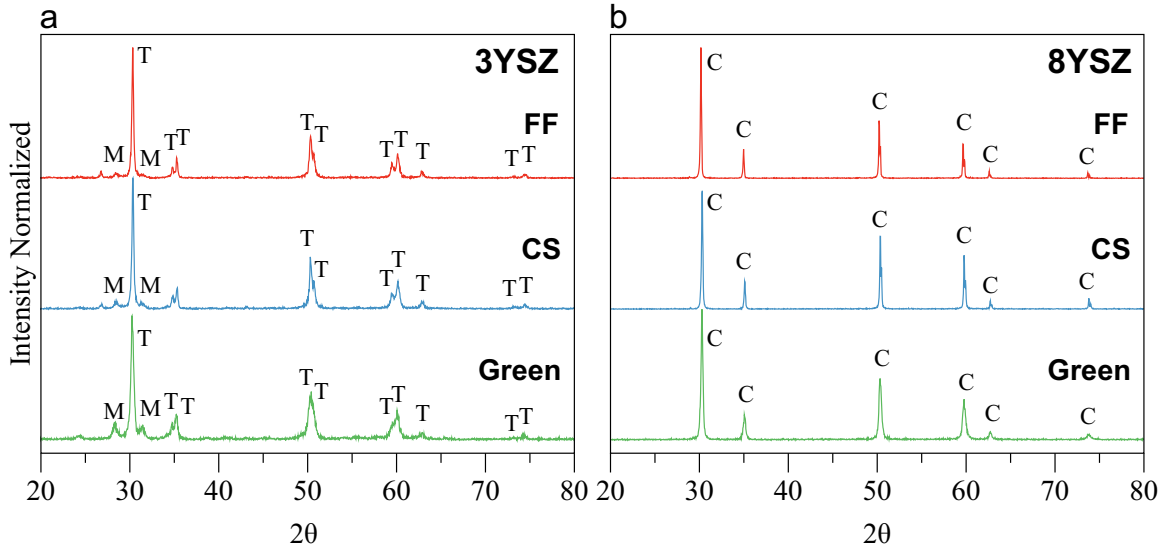

Fig. 1. Diffractograms for raw powder and for near full dense specimens sintered at 1400 °C by conventional sintering (CS), and fast firing (FF): a) 3YSZ; b) 8YSZ.

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