Cold Sintering Process for 8 mol%Y₂O₃-Stabilized ZrO₂ Ceramics

Hanzheng Guo, Thorsten J.M. Bayer, Jing Guo, Amanda Baker, and Clive A. Randall [†]

Materials Research Institute, The Pennsylvania State University, University Park, PA 16802,

USA

[†] Author to whom correspondence should be addressed. Email: car4@psu.edu

Abstract: In this communication, the cold sintering process **was** applied to benefit the green body compaction of 8 mol%Y₂O₃-stabilized ZrO₂ ceramics (8Y-YSZ). **Compared to conventionally processed ceramics,** an enhanced densification behavior was demonstrated **in cold sintering related ones** following a second step conventional sintering process. Dense ceramics up to ~96% of theoretical density were achieved after sintering at 1200 °C. The resulted ceramics demonstrated a fine microstructure with a grain size ~200 nm. A mechanical performance with a Vickers hardness of 13.6 GPa and a fracture toughness of 2.85 MPa m^{1/2} **was** also reported.

Keywords: Sintering, Cold sintering process, Densification, Yttria-stabilized zirconia, Fine grain size

I. Introduction

We recently established a novel sintering approach, named the cold sintering process (CSP), to consolidate ceramic particulates at substantially reduced sintering temperatures.¹⁻³ Dense ceramics which cover a wide variety of chemistries and structures, have been successfully achieved. In terms of the functionality, they include microwave dielectrics, ferroelectrics, refractories, semiconductors, thermoelectrics, and battery electrolytes.¹⁻⁸ In addition to the polycrystalline ceramic form, we have also demonstrated that CSP is applicable to process multilayer ceramic structures and functional polymer/ceramic composites with higher ceramic contents at extraordinarily low temperatures.^{9,10}

Historically, there has been early studies to consolidate amorphous, polycrystalline, or polymorphous materials under hydrothermal environment with the appearance of pressure. This approach is often referred to as hydrothermal hot pressing (HHP) or hydrothermal reaction sintering, which is generally employed to prepare cements, glasses, ceramics, as well as metal oxides from metals.¹¹⁻¹⁷ Similar to HHP, the CSP is actually a liquid phase sintering process with an assistance of pressure in the range of 50-500 MPa.² The densification of ceramic particulates is assisted by a liquid phase-induced dissolution-precipitation process: the liquid phase (generally aqueous solutions) partially dissolves the surfaces of ceramic particles, and then evaporation occurs upon heating, leading to a supersaturated state for precipitation; kinetically, mass transport is enhanced by accelerating the diffusion of solute ions; and also, the liquid phase lubricates the particle surfaces, hence further benefits particle rearrangement for compaction under pressure.² We have demonstrated the feasibility of CSP to densify over 50 chemistries with densities of 80% – 99% of theoretical at extremely low temperatures of 25 °C – 300 °C.²

As is well-known, 8 mol% Y_2O_3 -doped ZrO_2 (8Y-YSZ) with a cubic structure has been extensively investigated due to its excellent chemical and thermal stability and high oxygen ionic conductivity in a wide range of oxygen partial pressures and temperature window.¹⁸ It thus has been widely manufactured as solid electrolytes for the applications in oxygen sensors and solid oxide fuel cells.¹⁸⁻²⁰ However, one of the challenges to manufacture Y_2O_3 -doped ZrO_2 ceramics is the high temperature process. To obtain dense ceramics, the conventional sintering activity is normally carried out at high temperatures.²¹ Numerous efforts have been made to effectively reduce the sintering temperature of ZrO_2 -based ceramics, and generally this has been achieved by (1) developing new sintering techniques, such as two-step sintering, spark plasma sintering, field-assisted sintering *et al.*, or (2) improving the green density of ceramic compacts using nanoparticles to enhance the densification process, or (3) a combination thereof.²²⁻²⁹ In our initial studies, we tried to perform CSP to densify 8Y-YSZ ceramics simply using water as sintering aid to consolidate the nanoparticles, since it has been noted earlier that water can effectively react with Zr-O-Zr bond in ZrO_2 , and trigger a particle growth in water vapor environment.^{30,31} This observation indicates a fact that the dissolution-precipitation process indeed takes place, and **may** be utilized to densify 8Y-YSZ ceramics under the scheme of CSP. However, unlike our previous CSP studies, 8Y-YSZ ceramics have limited densification after the cold sintering process. However, though a second step conventional sintering **was** applied for further densification, we still noticed that CSP **was** of major benefit in the green body compaction of 8Y-YSZ ceramics, and this **led** to an enhanced densification and fine grains. In this communication, we **demonstrated** such an observation, and also briefly **outlined** the initial process-structure-property relationship.

II. Experimental Procedure

The 8 mol% Y₂O₃-stabilized ZrO₂ nanoparticles (8Y-YSZ, 99.95%, 20 nm) were purchased from commercial source (SkySpring Nanomaterials, Inc., Houston, Texas). For the cold sintering process, raw powders were mixed with 30 wt.% deionized water using pestle and mortar for 1-2 min. The mixture was uniaxially pressed under 350 MPa first at room temperature (25 °C) for 10 minutes, and then the temperature was ramped up to 180 °C with a rate of 9 °C/min. The temperature was isothermally kept for 1 h. The as-prepared ceramic pellets were first baked at 200 °C overnight to remove possible water residue, and then further conventionally sintered at 500 – 1200 °C for 6 h with a temperature ramp rate of 10 °C/min in air (these ceramics are referred to as CSP-CS produced ceramics hereafter). To make a comparison, green pellets using the same raw nanoparticles were dry pressed under 350 MPa at room temperature without adding water and any binder, and then were conventionally sintered in the same way. The densities were measured by mass/geometry ratio. A theoretical density of 6.0 g/cm³ was adopted. The crystal structure was checked by X-ray diffraction (Panalytical, X'Pert PRO) with Cu-K α radiation. Vickers Hardness (H_v) was evaluated using the indentation technique on polished ceramic surfaces. H_v was calculated by $H_v = 0.0018544 (P/d^2)$, where P = applied force, and d = average diagonal length of the indentation pyramid. Fraction toughness (K_{IC}) was roughly evaluated by the indentation fracture toughness measurement at a load of 1 – 5 kg for a duration time of 15 s. The crack length C was averaged by the lengths at four corners of the indentation pyramid. Since zirconia was prone to slow crack growth,^{32,33} 10 min was waited between the indentation was done and the measurement of the crack size. Fraction toughness was then evaluated by the equation of $K_{IC} = 0.016 (E/H_v)^{1/2} (P/C^{3/2})$, where E = Young's modulus, P = applied force, and C = crack length derived from the indentation center. Transmission Electron Microscopy (TEM) specimens were prepared via standard procedures including mechanical thinning, polishing, and

ion milling. Microstructures were imaged using a Talos (FEI, Talos) microscopy at an accelerating voltage of 200 kV.

III. Results and Discussion

Compared to the green pellets (~48% of theoretical) that are dry pressed without water addition, the cold-sintered ceramics demonstrate an improved density (~56% of theoretical). Figure 1 shows TEM micrographs of the cold-sintered 8Y-YSZ ceramics. Under TEM, it has been noticed that there are a large amount of pores in cold-sintered ceramics, which minimize the densification. Up-close view at the local environment reveals that the size of nanoparticles in cold-sintered ceramics are still ~20 nm (Figure 1a), but amorphous phase and precipitates at a few nanometers scale appear in the grain-grain interstitials (Figures 1b-1c). This indicates that dissolution and some extent of precipitation process indeed occurs during CSP, but obviously, higher temperature environment is needed to trigger further densification via epitaxial grain growth and/or Ostwald ripening process.

On applying a further heat treatment with a conventional sintering process, the CSP-CS produced ceramics demonstrate an enhanced densification compared to the typically conventionally sintered ones: the density is slightly improved at the temperature ≤ 900 °C; an effective densification starts ~900 °C with a jump appears at 1000-1100 °C; and finally dense compacts ~ 96% of theoretical are achieved at 1200 °C (Figure 2a). Such a densification development produced by other published sintering techniques for 8Y-YSZ nanoceramics is contrasted in Fig. 2b.^{22,24-26,28} A density-grain size map of 8Y-YSZ nanoceramics produced by various sintering approaches is also compiled in Figure 3c.^{22,25,26,28,34} As shown in the TEM micrograph (inset in Figure 2c), the CSP-CS produced 8Y-YSZ ceramics sintered at 1200 °C demonstrate a fine microstructure with a grain size ~200 nm. Generally speaking, dense ceramics

produced by conventional sintering process are often manifested by coarse grains in their microstructures, since high temperatures in conventional sintering processes are generally required to enable such high densities.²²⁻²⁹ It can be seen from this density-grain size map that CSP-CS separates from the conventional sintering data, and is linked with other innovative sintering approaches, such as two-step sintering, microwave sintering, and spark plasma sintering, to produce dense 8Y-YSZ ceramics with limited grain growth. More details about the raw particle size, green density, processing parameters, final density, and resulting grain size can be found in Table I.

Figure 3a demonstrates the XRD profiles of 8Y-YSZ ceramics after cold sintering at 180 °C and subsequent conventional sintering at 500 - 1200 °C. Crystallographically, the raw 8Y-YSZ nanoparticles used in this study adopt a cubic structure but with a slight portion (~4.2 wt.%) of monoclinic phase.³⁵ No noticeable phase transformation is observed after cold sintering, since an almost identical XRD profile is obtained. This observation is consistent with a previous study, which has found that the cubic structured Y_2O_3 - ZrO_2 ceramics are resistive to phase transformation in aqueous environment.³⁰ Close-view around the (111) spectra range reveals that the monoclinic phase gradually transforms into the cubic phase with the increase of sintering temperature, and finally is completely removed at 1000 °C (Fig. 3b). Simultaneously, it is also noted that the cubic (111) diffraction peak is gradually intensified and becomes sharper, which indicates a gradual crystal growth process.

As shown in Figure 4a, the H_V evolution curve is found to be coincident very well with the densification development. The H_V profile keeps an almost plateau configuration (~ 1.5 GPa) at the temperatures ≤ 900 °C, and then an apparent bump appears at ~1000 °C ($H_V \sim 3$ GPa); slightly increasing the sintering temperature to 1200 °C, the H_V remarkably reaches 13.6 GPa,

which may be attributed to the significantly improved density from ~65% (at 1000 °C) to ~96% (at 1200 °C). Similar to the H_V evolution, an overall increment trend is also observed for the K_{IC} ; a highest value of 2.85 MPa m^{1/2} is found after sintering at 1100 °C. Figures 4b-4c are the literature summary of H_V (and K_{IC}) versus sintering temperature plot for 8Y-YSZ ceramics produced by various sintering techniques (see details in Table II).^{22,25,28,36-40} It is noted that the mechanical properties ($H_V=13.6$ GPa, $K_{IC}=2.85$ MPa m^{1/2}) of CSP-CS produced ceramics are still in comparable ranges, but are obtained in relatively lower temperatures, as well as with significantly reduced final grain size (see grain size details in Table II).

IV. Conclusions

In summary, we demonstrated the applicability of CSP to improve the green density of 8Y-YSZ ceramics, and thus yielded to an enhanced densification process.²² TEM studies suggested a dissolution process and some extent of precipitation process when 8Y-YSZ nanoparticles were exposed in aqueous environment during CSP. The transient liquid phase was known to kinetically accelerate the mass transport as well as mechanically benefit the particle compaction.^{2,31} Both facts were suggested to contribute to the improved green density. Following a second step conventional sintering process, dense ceramics >95% of theoretical density was achieved at ~ 1200 °C with a fine microstructure of ~200 nm grain size. The demonstrated H_V (13.6 GPa at 1200 °C) and K_{IC} (2.85 MPa m^{1/2} at 1100 °C) are comparable to those values in the literature, but they are achieved at lower processing temperatures with a substantially limited grain growth.

Acknowledgements

This material is based upon work supported by the National Science Foundation, as part of the Center for Dielectrics and Piezoelectrics under Grant Nos. IIP-1361571 and 1361503. The authors would like to thank Dr. David Shelleman (MTL and MRI-MCL at The Pennsylvania State University) for the mechanical property measurements.

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Table I. Literature summary of 8Y-YSZ ceramics as processed by nanoparticles in a size range of 20-30 nm. The data are compiled from references [22,24-26,28,34].

Particle Size (nm)	Green Density (%)	Pressure (MPa)	T (°C) or T ₁ /T ₂ for TSS	Density (%)	Grain Size (nm)	Method	Ref.
24.5	35.8-48.5	100-600	950-1500	65.4-97.5	50-2150	CS	22
			1250/1050	82.5-97.6	200-295	TSS	
21			1200 (20-160MPa)	60.7-98.5	88-148	SPS	24
21			1200 (160MPa)	86.2-99.1	82-127	SPS	
24.5	48	600	950-1400	67-97.8	50-2350	MVS	25
			950-1500	65.8-98	61-909	MVS	
20-30	52.7	400	1150	83.2	370	FAST	26
22	46-47	280	1125	97	185-220	TSS	28
25	45-47	140	1430	99.8		MVS	34
20	48.3	350	1200	81.3		CS	This work
20	56.2	350	1200	95.7	200	CSP-CS	This work

Table II. Literature summary of mechanical properties of 8Y-YSZ ceramics prepared by various sintering techniques. The data are compiled from references [22,25,28,36-40]. CS=Conventional Sintering; TSS=Two-Step Sintering; MVS=Microwave Sintering; CSP=Cold Sintering Process.

T (°C) or T ₁ /T ₂ for TSS	Density (%)	Method	GS (μm)	Hv (GPa)	K _{IC} (MPa m ^{1/2})	Ref.
1500	97.5	CS	2.15	12.87	1.61	22
1250/1050	97.6	TSS	0.295	13.51	3.16	
1450	~97	CS	~2.65	~12.5	1.2	36
1450	~99	CS	5-6.5		1.5	37
1400	98.7	CS	~5-10	9.17	3.47	28
1400	96	CS	4	11.7	1.3	38
1700	96	CS	50		1.84	39
1500	95	CS	13		2.11	
1353	94	CS	2.6		2.84	
1220	97	CS	0.7		4.12	
1600	~90	CS		8.9	3.2	40
1500	97.5	CS	2.14	12.87	1.61	25
1400	98	MVS	2.35	12.97	1.9	
1500	98	MVS	0.9	13.72	3.17	
1200	95.7	CSP-CS	~0.2	13.6	2.85 (1100 °C)	This work

Figure Captions

Fig. 1. TEM micrographs of 8Y-YSZ ceramics after cold sintering at 180 °C. (a) Overall view. (b) and (c) Up-close view.

Fig.2. (a) A comparison of the densification process of 8Y-YSZ ceramics prepared by CSP-CS and CS approaches using the same raw nanoparticles. (b) Densification evolution of 8Y-YSZ ceramics processed by nanoparticles of 20-30 nm regarding various sintering techniques. (c) Density-grain size summary of 8Y-YSZ ceramics processed by nanoparticles of 20-30 nm. CS=Conventional Sintering; SPS=Spark Plasma Sintering; MVS=Microwave Sintering; FAST=Field Assisted Sintering; TSS=Two Step Sintering; CSP=Cold Sintering Process. The data are compiled from references [22,24-26,28,34]. The inset in (c) is the TEM micrograph of 8Y-YSZ ceramics processed by CSP-CS after sintering at 1200 °C. It is ~96% relative density and has a mean grain size ~200 nm.

Fig. 3. (a) XRD spectra of 8Y-YSZ ceramics after cold sintering at 180 °C and subsequent conventional sintering at 500 - 1200 °C. (b) Close-view of (111) spectra showing the characteristic diffraction peaks of monoclinic and cubic phases.

Fig. 4. (a) Vickers hardness and fracture toughness of 8Y-YSZ ceramics after cold sintering at 180 °C and subsequent conventional sintering at 600 - 1200 °C. Literature summary of, (b) Vickers hardness and (c) fracture toughness, versus sintering temperatures of 8Y-YSZ ceramics prepared by different techniques. These plots are compiled from references [22,25,28,36-40].