Processing effects on sintering of transparent Al_2O_3 and $MgAl_2O_4$ ceramics by SPS

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Highlights

- Obtain materials as large and transparent as possible by reducing the main transparency losses
- Fabrication process optimization of transparent alumina samples: reduce grain growth
- Sintering parameters optimization to obtain large size (diameter 40mm) transparent spinel samples

1. Introduction

The Flash sintering (SPS) is a technique widely used since several years and allows the sintering of material with specific microstructure, not accessible by conventional sintering. It is the case of transparent ceramics, particularly birefringent ceramics which grain size is a limiting parameter to transparency. One of major advantage of Spark plasma Sintering is its fast heating rate. It allows a sintering of ceramics at lower temperature and limits the grain growth. In this work, we will present a fabricated process optimization of two transparent ceramics: alumina (Al_2O_3) and spinel ($MgAl_2O_4$). The objective was to obtain materials as big and transparent as possible by reducing the main transparency losses that are secondary phases, light scattering by porosity and for birefringent ceramics, light scattering at grain boundaries.

2. Methods

To fabricate transparent alumina the global studied process is at first a step of slurry doping, then a shaping step (freeze drying or slip casting) and to finish a flash sintering step.

In the case of transparent spinel, the material is fabricated with a granulated commercial powder, shaping and sintering in the SPS. Processing parameters which allow to obtain small (20 mm diameter) transparent samples will be presented. Then, the impact of stress heterogeneity of uniaxial pressing and temperature heterogeneity between the center and the edges of the sample in case of large sample diameter (40mm) will be studied.

For both, the transparency (Real In Line Transmission, RIT) is measured with a spectrophotometer.

3. Results and discussion

In case of alumina, several parameters have been studied. Two dopants have been tested: zirconium and lanthanum. Both reduce the diffusion across grain boundary, and the grain boundary solubility limit is between 280 and 570 ppm for zirconium and between 200 and 310 ppm for lanthanum. Secondary phases have been observed at 310ppm in case of lanthanum and at 570 ppm in case of zirconium. However, with lanthanum doping, the densification is better and the alumina grain size is smaller than with zirconium doping. Therefore, lanthanum was selected for the rest of the processing. For the shaping step, the more appropriate shape process was the slip casting. It allows to obtain denser green body with of smaller size pore population. The optimized SPS sintering cycle depends of the properties of green body sample. A *dilatometric SPS* experiment has been done to determine the best sintering temperature T_f of the green body, and then a specific cycle is realized (as presented in the works of Kim and Morita) (cf. figure 1). The objective in the case of alumina is to get a fully densify sample with a limited grain growth. An alumina sample (thickness 0.88mm) with a RIT of 70% at 640nm is obtained with the optimized process [1] (cf. figure 2).

In case of spinel material, we show that to fabricate small sample (diameter 20 mm), SPS sintering is an appropriated method. Sample (cf. figure 3) with 79% RIT for 1.85 mm thickness have been obtained [2].

When the sample has a bigger size (diameter 40 mm), several problems appear. Heterogeneity in temperature between the center and the edges of the sample and heterogeneity of applied stress lead to density heterogeneity in bulk which disturb transparency homogeneity. Another phenomena that appear with large diameter, is the carbon diffusion in sample, which limit the transparency.

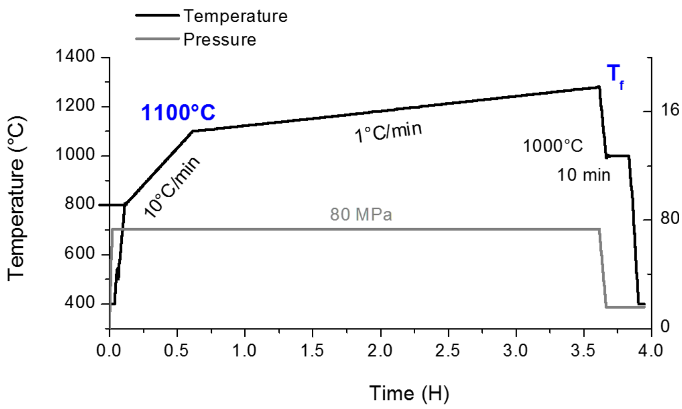

Figure 1. Classical SPS cycle for birefringent ceramic

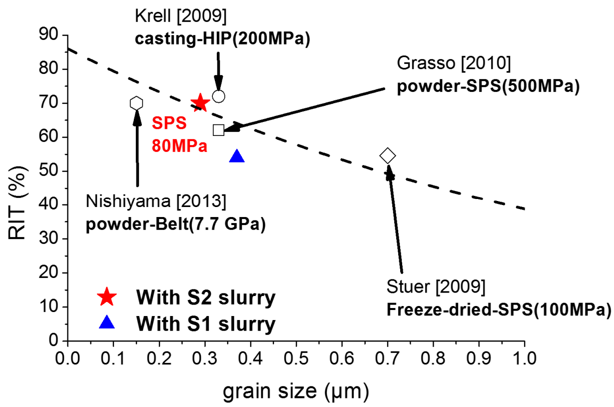

Figure 2. Optical properties of samples elaborated via slip casting - SPS

Figure 3. Spinel sample RIT = 79% at 550 nm for 1.85 mm thickness

4. Conclusions

Alumina samples (thickness 0.88mm) with a RIT of 70% are obtained with an optimized process: 120cat ppm of lanthanum doping, slip casting as shaping step and SPS sintering at 1140°C under 80MPa.

For transparent spinel, SPS is an effective sintering technique to prepare small samples (Ø 20mm). Samples with a 79% RIT for 1.85mm thickness have been fabricated. SPS is a more complicated sintering technique to prepare large samples due to sample heterogeneity, thermal gradient, stress gradient and carbon contamination.

References

[1] Roussel N., Lallemant .L et al., J. Am. Ceram. Soc., (2013), 1 - 4.
[2] Bonnefont et al., Cer. Int. 38 (2012) 131–140

Keywords
Transparency, Alumina, Spinel, Fast sintering