Polycrystalline Transparent Magnesium Aluminate Spinel Processed by a Combination of Spark Plasma Sintering (SPS) and Hot Isostatic Pressing (HIP)

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Highlights

- Extremely transparent $MgAl_2O_4$ spinel was fabricated for the first time using a combination two-stage process of SPS followed by HIP.

1. Introduction

SPS is a rapid and cost-effective advanced sintering technique, highly promising for the fabrication of transparent polycrystalline ceramics. However, using this approach for large and/or thicker parts is difficult due to limited tooling dimensions, applied force and non-homogeneous temperature distribution. The transparency of polycrystalline ceramics is highly dependent on the size and amount of residual porosity that typically remains after convectional sintering processes. The HIP process, as the final stage in fabrication, serves to reduce or eliminate this residual porosity. In the present study, we suggest combining the SPS process with a subsequent HIP treatment to obtain relatively large (30 mm in diameter and 4 mm thick) highly transparent ceramic parts.

2. Methods

Magnesium aluminate spinel ($MgAl_2O_4$) powder (S30CR, Baikowski Chemicals, La Blame de Silingy, France) containing 0.6% LiF (99.99%, Alfa-Aesar, Heysham, Lancashire, UK) was used as starting material. The powders were mixed in distilled water by stirring for 1 hour in a silica glass container, the water was removed using a freeze dry apparatus. The final mixed powder was conducted in a SPS apparatus (FCT Systems, Rauenstein, Germany) with the following parameters: 30 mm diameter die, under a 50 MPa at 1450ºC for 2 hours, with varying heating rates of 25, 50, 100 ºC/min. The measured relative density of the SPS-processed samples was >99% of theoretical density and transparent to some extent. The samples were treated by HIP (AIP6-30H, USA) with the following parameters: 1500ºC under 200 MPa of Ar gas pressure for varying durations of 0.5, 2, 5 and 10 hours. Microstructural examination was carried out using a scanning electron microscope (FEI, Quanta 200 SEM) and the real in-line transmission (RIT) was measured using spectrophotometer (JASCO, V-530 UV-VIS).

3. Results and discussion

Highly transparent and relatively large (30 mm in diameter and 4 mm thick) SPS-processed $MgAl_2O_4$ spinel specimens were obtained (Figure. 1). The samples are not highly transparent or homogenous after the SPS process (Figure 1a), however, after the HIP treatment both those issues are solved (Figure 1b) and they become extremely transparent (Figure 1c).

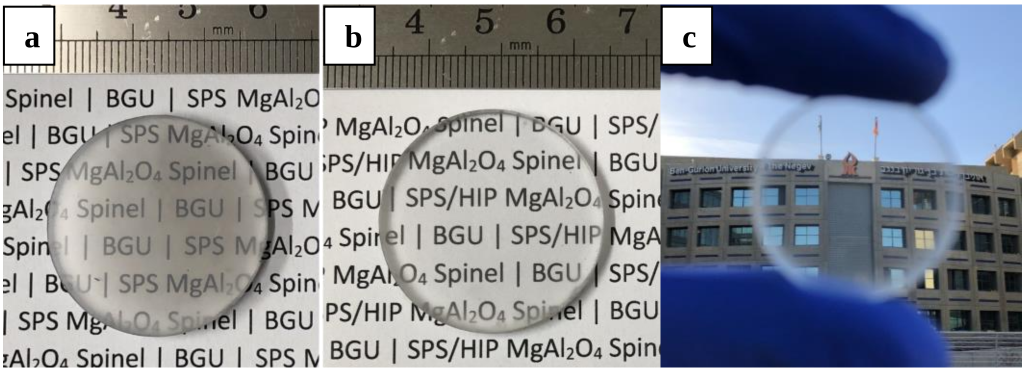

Figure 1. Polycrystalline magnesium aluminate spinel (30 mm diameter, 4 mm thick) after (a) SPS (b) SPS/HIP and (c) a portrait photo.

A remarkable improvement in the RIT of the SPS-processed samples after HIP was observed (Fig. 2). The samples after prolonged HIP treatment exhibit extremely high transmission at lower wavelengths that reflects the decreased level of a fine porosity responsible for Rayleigh scattering. However, some pore coalescence takes place resulting in light scattering at higher wavelengths. The transmittance results are, in fact, the best reported so far in literature (see the comparison with Krell et al. [1] for a two-stage pressureless + HIP process of the same Baikowski powder).

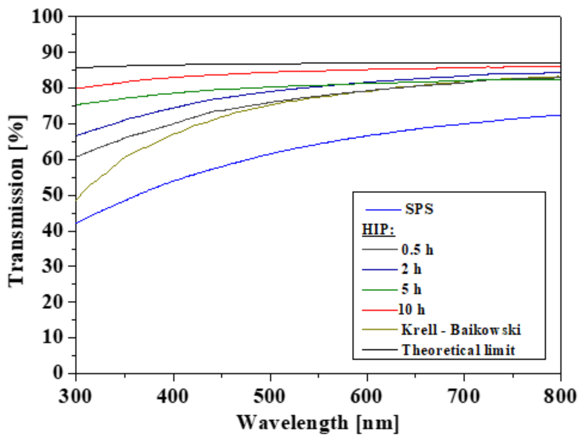

Figure 2. Transmission spectra normalized to 4 mm for SPS-processed and HIP treated for various durations.

4. Conclusions

Extremely transparent polycrystalline magnesium aluminate spinel specimens with 30 mm diameter and 4 mm thickness were fabricated by combination of the SPS process and the subsequent HIP treatment. Perhaps an important milestone for SPS upscaling viability to fabricate large transparent ceramic parts has been reached.

References

[1] Krell et al., Optical Materials, Vol. 38, 2014, P. 61-74.

Keywords
Magnesium aluminate spinel; Spark plasma sintering; Hot isostatic pressing; In-line transmittance