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# Influence of post-HIP temperature on microstructural and optical properties of pure MgAl<sub>2</sub>O<sub>4</sub> spinel: From opaque to transparent ceramics

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## A B S T R A C T

Transparent MgAl<sub>2</sub>O<sub>4</sub> spinel ceramics were processed from sub-micrometric commercial powder by applying a two-step procedure: pressureless sintering under vacuum followed by hot isostatic pressing. To limit grain growth and to avoid secondary reactions or impurities, no additives or sintering aids were added to the powder. First, pressureless sintering at 1500 °C during 2 h under vacuum led to opaque samples due to a high level of porosity. To improve the optical quality of the MgAl<sub>2</sub>O<sub>4</sub> ceramics and the in-line transmission in the visible range, a post-treatment by hot isostatic pressing was applied. Highly transparent ceramics were obtained after a post-treatment at 1800 °C for 10 h with an in-line transmission of 81% at 400 nm and 86% from 950 to 3000 nm for a thickness of 2 mm (98.8% of the theoretical transmission).

### Keywords:

Spinel  
MgAl<sub>2</sub>O<sub>4</sub>  
Transparent ceramic  
Pressureless sintering  
Hot isostatic pressing

## 1. Introduction

Transparent magnesium aluminate spinel (MgAl<sub>2</sub>O<sub>4</sub>) ceramic exhibits a high potential for applications in which a combination of optical and mechanical properties is needed: an isotropic propagation of the light due to a cubic structure, a wide range of transmission, a low density and high toughness [1]. Thus, spinel ceramics can be found as laser ignitors, transparent domes for IR-seeking missiles and ballistic protection [1,2].

Transparent spinel has generally been fabricated either directly from pure spinel powder [3–12] or by reactive sintering [10,13–15], by combining pressureless sintering (PS) and hot isostatic pressing (HIP) [3,6,8,9,11–14,16–18] and by hot press or Spark Plasma Sintering [15,19–24]. High transparency is conditioned to the achievement of a microstructure exempt of scattering centres, such as pores and impurities. In the visible range, pores of 0.1–1 μm are the most detrimental, as the diameters of the defects match the wavelengths. Moreover, transparent material needs to exhibit a residual porosity less than 0.01% with pore size below 100 nm [1,6,19]. A small difference of refractive index, because of secondary phases or impurities, can lead to light reflection and refraction, both phenomena promoting a loss of transparency [24–28].

To meet the strict requirements for transparent ceramics [23,25–27], sintering aids might be used, as they enhance the densification kinetics and may remove some impurities. Sintering aids, such as

LiF, have been used with sintering under pressure [4,15,20,29,30], while B<sub>2</sub>O<sub>3</sub> [11] or CaO [18] have been tested in the pressureless sintering approach. Despite their positive effect on the grain size or optical properties, they may cause the formation of secondary phases or defects in the microstructure, yielding to translucency, as Villalobos et al. pointed out in their study on hot-pressed MgAl<sub>2</sub>O<sub>4</sub> spinel with LiF [29]. Indeed, the authors presented the reaction of LiF with Al<sub>2</sub>O<sub>3</sub> on the particle surfaces, which leaves MgO-rich areas and the formation of LiAlO<sub>2</sub> phase. The presence of these phases caused white regions due to the incomplete elimination of LiF located at grain boundaries. Nevertheless, highly transparent spinel ceramics have been successfully fabricated with a two-step approach on treated spinel powder (ultrasounds on a suspension with dispersing agents) without sintering aids. For example, Krell et al. applied a pressureless sintering at 1250 °C and a post-HIP at 1260 °C for 15 h and measured over 80% in-line transmission (ILT) at 640 nm for spinel ceramics with thicknesses between 3 and 4 mm [6]. Likewise, good results have been obtained by Goldstein et al. (~80% ILT at 640 nm) after a double step sintering (PS at 1480 °C/3 h and HIP at 1550 °C/4 h) [16,17] and slightly less by Witek (> 70% ILT at 640 nm) after sintering in air at 1700 °C for 1 h and post-HIP at 1800 °C for 110 min [12].

Various works in which pressureless sintering has been used focus on the powder optimisation to subsequently obtain homogeneous green bodies, which seems to be essential for highly transparent ceramics [6,14,16]. Several powder processing steps have been employed such as

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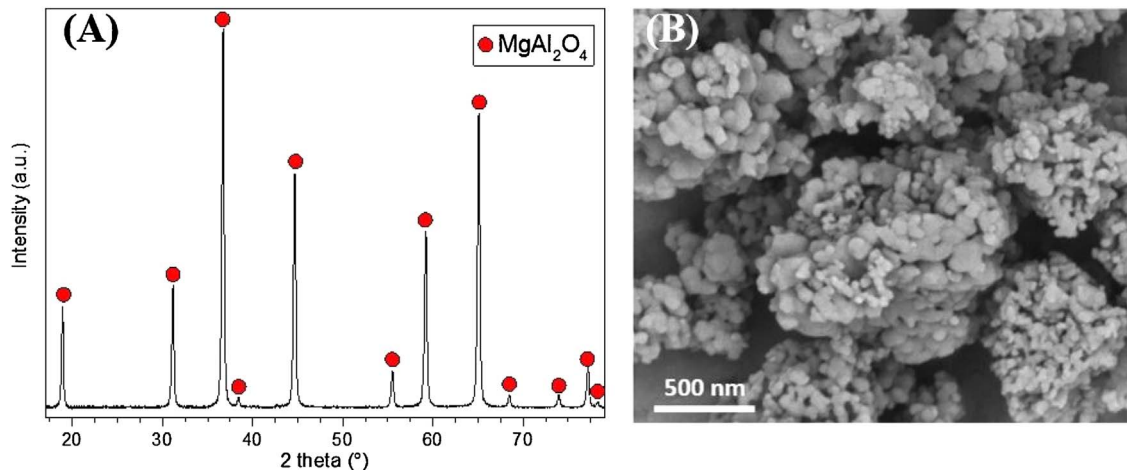


Fig. 1. XRD pattern (A) and SEM observation (B) of the commercial spinel  $\text{MgAl}_2\text{O}_4$  powder.

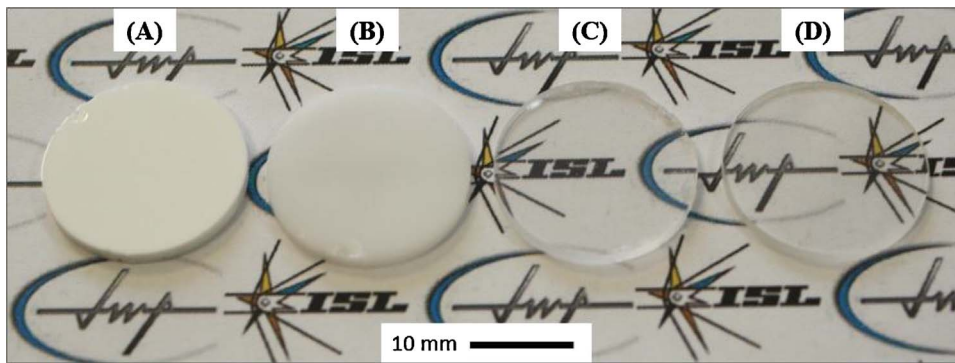


Fig. 2. Visual aspect of  $\text{MgAl}_2\text{O}_4$  spinel after pressureless sintering (A) and after post-HIP at 1500 °C for 10 h (B) and 1800 °C for 1 h (C) and 10 h (D).

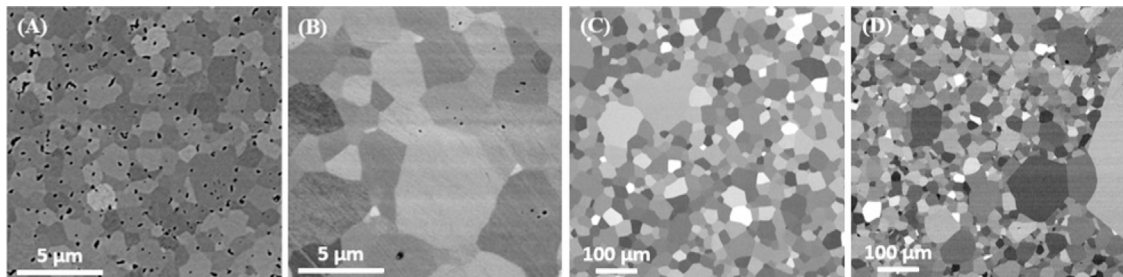


Fig. 3. SEM microstructures of  $\text{MgAl}_2\text{O}_4$  spinel after sintering (A) and after post-HIP at 1500 °C (B) and 1800 °C for 1 h (C) and 10 h (D).

deagglomeration (ultrasonication [16] or ball milling [6,11,18]) or even re-agglomeration to improve the final powder flowability [8,9,31]. Homogeneous green bodies can be obtained by dry compaction [6,11,12,16,17], gel casting [6,14] or slip casting [9,14] via the addition of different additives. However, the optimisation of the green body via powder treatments might also introduce some impurities deleterious to the optical transmission of the transparent ceramics. Besides, the introduction of organics requires in general a burn-out step that can weaken the green body or lead to cracks [8].

A similar approach to the procedure followed by Maca et al. [3] is presented in this paper: without pre-treatment, commercial powder is dry shaped by uniaxial and cold isostatic pressing; then, pressureless sintering at 1550 °C is applied, followed by HIP at 1500 °C/1 h/200 MPa. Without using sintering aids, Maca et al. [3] obtained 60.2% ILT (at 632 nm) for 1.1 mm of thickness. To further enhance the transparency of the spinel ceramics, the influence of HIP temperature on the microstructural and optical properties of  $\text{MgAl}_2\text{O}_4$  spinel has been studied and is presented in this paper.

## 2. Experimental procedure

Commercial  $\text{MgAl}_2\text{O}_4$  spinel powder (grade S25CR, Baikowski, France) with a grain size of 0.43  $\mu\text{m}$  ( $d_{50}$ ) and containing a low amount of impurities (total of 50 ppm with Ca, Fe, K, Na and Si) was used in this study. Additional chemical analysis revealed a sulfur level of 37 ppm. The specific surface area (SSA) was measured by nitrogen adsorption according to the Brunauer-Emmett-Teller (BET) method (Micromeritics, ASAP 2020). X-ray diffraction (XRD) analysis was performed with a D8 Advanced Bruker AXS using  $\text{Cu K}\alpha$  radiation ( $\lambda = 0.15406 \text{ nm}$ ) on the commercial  $\text{MgAl}_2\text{O}_4$  powder to identify crystalline phases. Complementary characterization was performed by inductively coupled plasma (ICP) to determine the molar ratio of Mg/Al of the spinel powder.

Green bodies were prepared by uniaxial pre-compaction followed by cold isostatic pressing at 300 MPa. The obtained samples were subsequently sintered in a vacuum furnace (Lilliput, ECM Technology) at 1500 °C for 2 h, and post-treated by HIP (EPSI Inc.) under argon pressure of 190 MPa. Two temperatures of post-HIP were considered:

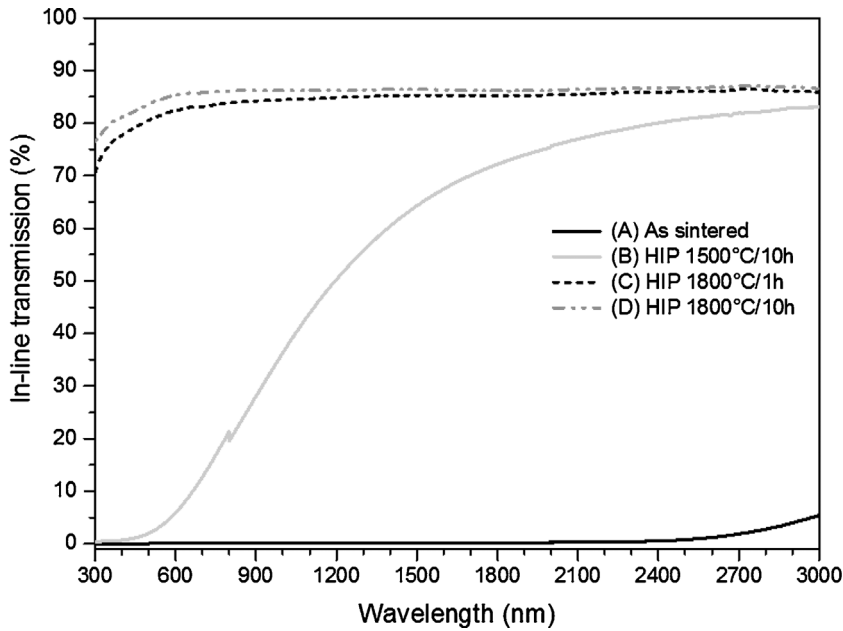


Fig. 4. In-line transmission of MgAl<sub>2</sub>O<sub>4</sub> spinel after pressureless sintering under vacuum, post-HIP at 1500 °C and 1800 °C.

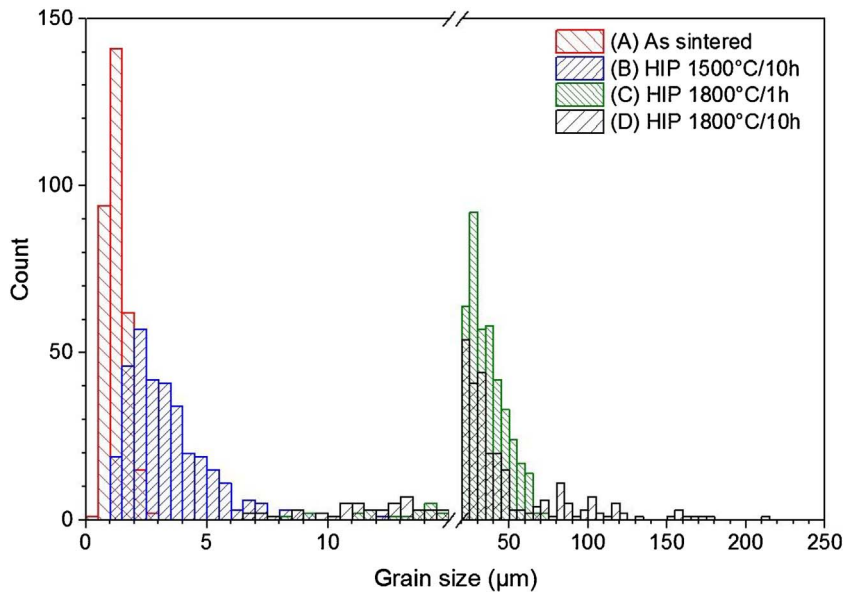


Fig. 5. Grain size distribution of MgAl<sub>2</sub>O<sub>4</sub> spinel after pressureless sintering (A) and after post-HIP at 1500 °C (B) and 1800 °C for 1 h (C) and 10 h (D).

1500 °C and 1800 °C with a holding time of 1 h or 10 h. Finally, sintered ceramics were mirror-polished on both sides.

Surface microstructures were investigated by scanning electron microscopy (SEM) with a Nova NanoSEM 450 (FEI Company). Average grain size was determined from the measurements of at least three hundred grains. Relative densities of the MgAl<sub>2</sub>O<sub>4</sub> ceramics were determined according to the Archimedes principle by hydrostatic weighing in distilled water. The in-line transmission was measured from 300 nm to 3000 nm with a UV-vis-IR spectrophotometer (Cary 7000 UMS, Agilent Technologies). Knoop hardness was determined with the aid of a micro indenter (Buehler).

### 3. Results and discussion

Results of XRD analysis and SEM observations are shown Fig. 1. The obtained XRD pattern was indexed with pure cubic MgAl<sub>2</sub>O<sub>4</sub> spinel structure (JCPDS 01-72-72-32). Purity was also confirmed by ICP: the Mg/Al molar ratio is equal to 0.50194, which is close to the theoretical value of a stoichiometric spinel powder. The commercial powder

presented agglomerates of  $1.1 \pm 0.4 \mu\text{m}$  composed of rounded shaped particles of  $70 \pm 20 \text{ nm}$ . This information correlates the particle size deduced from the specific surface area obtained on the powder ( $100 \text{ nm}$  for  $\text{SSA} = 16.6 \pm 0.1 \text{ m}^2/\text{g}$ ). However, this is not in accordance with the given commercial value of  $d_{50}$ .

Fig. 2 presents the visual aspect of the polished MgAl<sub>2</sub>O<sub>4</sub> ceramics after pressureless sintering and post-HIP (diameter: 21 mm – thickness: 2 mm). After pressureless sintering under vacuum, the ceramic was white and opaque (Fig. 2A). The application of a post-treatment by HIP at 1500 °C slightly improved the optical quality of the spinel sample, which became slightly translucent (Fig. 2B).

At higher temperature (1800 °C) for 1 and 10 h, the MgAl<sub>2</sub>O<sub>4</sub> ceramic is transparent, as shown by the fact that the text below is clearly visible (Fig. 2C and D).

Microstructures of the MgAl<sub>2</sub>O<sub>4</sub> ceramics after sintering and after post-HIP at 1500 and 1800 °C were analysed by SEM (Fig. 3). The surface observation of the pressureless sintered spinel revealed numerous inter and intragranular pores, which explains the relative density value of  $97.4 \pm 0.1\%$ . These defects caused light scattering,

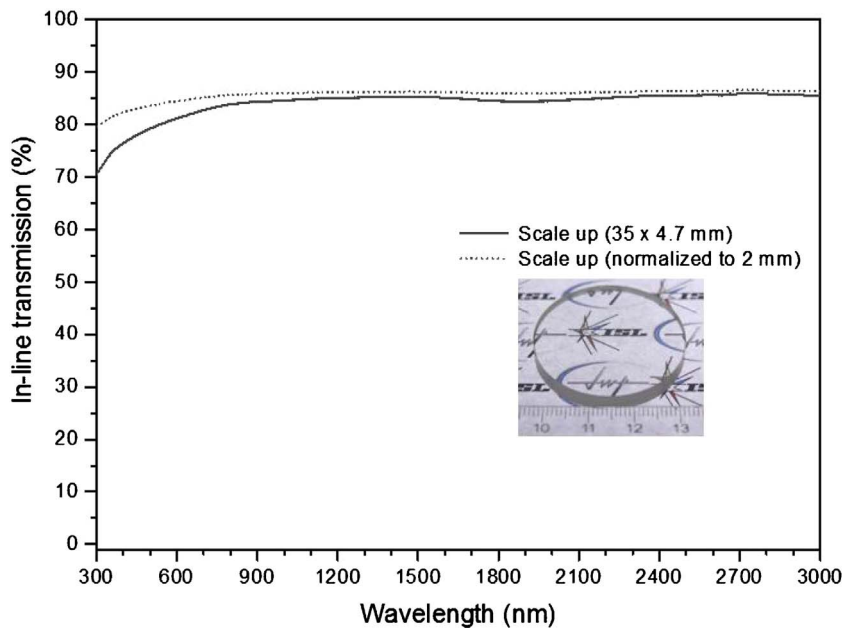


Fig. 6. In-line transmission of a scale up of  $\text{MgAl}_2\text{O}_4$  spinel ceramic and normalized to 2 mm thick.

leading to no transmission in the visible range and a very low maximum value of 5% ILT at 3000 nm (Fig. 4). A partial elimination of porosity was observed after the application of a post-treatment by HIP at 1500 °C (Fig. 3B), implying an increase of density up to  $99.8 \pm 0.1\%$  and less light scattering. The transmission slightly improved in the visible range (0–15% between 400 and 800 nm), whereas in the IR range, a transmission of 82% was reached at 3000 nm. A post-treatment at 1800 °C led to a fully dense ( $> 99.9\%$ ) and highly transparent spinel by elimination of residual pores (Fig. 3C and D). The higher temperature of HIP resulted in a great improvement of the optical quality in the visible range with ILT values between 78% and 84% for a holding time of 1 h, and 81% and 86% for a holding time of 10 h (Fig. 4). Furthermore, the transmission remained high (close to theoretical value) in the near-IR without any optical loss.

Spinel ceramic after pressureless sintering at 1500 °C presented a homogeneous microstructure with an average grain size of  $1.2 \pm 0.3 \mu\text{m}$ , as shown in the particle size distribution where the majority of grain sizes was between 1 and  $1.5 \mu\text{m}$  (Fig. 5). The application of a subsequent HIP step at the same temperature (1500 °C) for 10 h caused a slight increase of grain diameter up to  $3.4 \pm 1.3 \mu\text{m}$ . However, the range of grain sizes was wider from 1 to  $13 \mu\text{m}$ . Finally, the higher temperature of HIP (1800 °C) induced a remarkable grain growth. With a post-HIP of 1 h, the grain size reached  $35 \pm 11 \mu\text{m}$  with smallest grain size of  $10 \mu\text{m}$  and largest of  $70 \mu\text{m}$ . A longer holding time of HIP caused heterogeneous microstructure, where some very large grains were detected up to  $210 \mu\text{m}$ . The two populations of grains presented an average size of  $28 \pm 10 \mu\text{m}$  and  $125 \pm 50 \mu\text{m}$ . As it can be noticed, a heterogeneous microstructure did not affect transparency and in-line transmission in the visible and infrared ranges (Fig. 4).

To study the influence of microstructure on the mechanical properties of the spinel ceramics, Knoop hardness was determined for each sample with a loading of 4.9 N. The as-sintered spinel ceramics presented a hardness of  $11.6 \pm 0.1 \text{ GPa}$ . With the application of a post-HIP at 1500 °C, hardness increased to  $12.6 \pm 0.2 \text{ GPa}$ . This increase was clearly due to the elimination of porosity (from 97.4% to 99.8% of relative density). A higher HIP temperature (1800 °C) did not modify the hardness, as the samples showed values of  $12.3 \pm 0.3 \text{ GPa}$  and  $12.5 \pm 0.2 \text{ GPa}$  for a holding time of 1 h and 10 h, respectively. These results are close to the one reported by Maca et al. with 13 GPa [3].

Surprisingly, the large grain growth did not result in lower hardness. When comparing samples obtained by post-HIP at 1500 °C for

10 h and at 1800 °C for 1 h, the expected deleterious effect of grain growth may have been compensated by the increase of relative density from 99.8% to  $\sim 100\%$ . With a longer holding time at 1800 °C, as the relative density remained close to 100% and as the grain size increased, lower hardness values could have been expected. Sokol et al. [32] reported similar results on post-HIPed spinel samples at 1700 °C for different holding times (4–10 h). The grain size increased with the holding time:  $15 \mu\text{m}$ ,  $25 \mu\text{m}$  and  $170 \mu\text{m}$  respectively. Vickers hardness was determined for each sample and it revealed that the grain size did not influence the values, as they presented a hardness of 13.2, 12.9 and 13.2 GPa respectively.

This two steps sintering process allowed to increase the size of the samples (both thickness and diameter) while maintaining a very high optical quality. An example of ILT curve of a scale up from 21 to 35 mm in diameter and from 2 to 4.7 mm in thickness is shown on Fig. 6, along with the visual aspect of this larger spinel sample. After normalisation of the scale up data at 2 mm in thickness, the ILT curve exhibited minimum values of 80% between 300 and 3000 nm, close to the results of the smaller ceramic samples (21 mm in diameter) obtained after post-HIP at 1800 °C for 10 h.

#### 4. Conclusion

This work showed the possibility to obtain highly transparent  $\text{MgAl}_2\text{O}_4$  spinel ceramics via a two-step procedure without any additives or sintering aids. Furthermore, by avoiding supplementary treatment such as deagglomeration or granulation, the possible introduction of impurities was decreased, hence preventing the formation of secondary phases that can cause light scattering. By combining dry powder compaction of commercial spinel, pressureless sintering under vacuum and hot isostatic pressing, values of in-line transmission close to the theory were reached with 82% at 400 nm (94.2% of the theoretical value) and up to 86% in the 950–3000 nm wavelength domain (98.8% of the theoretical value). The application of the post-treatment by hot isostatic pressing allowed to enhance the optical quality, as well as the in-line transmission, by eliminating the inter-granular porosity. Knoop hardness values were not influenced by the increase of the grain sizes. This process also has the advantage of allowing the production of numerous samples and the preparation of larger and thicker samples without loss of optical properties.

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