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Feature article

Influence of post-HIP temperature on microstructural and optical properties of pure $MgAl_2O_4$ spinel: From opaque to transparent ceramics

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ABSTRACT

Transparent $MgAl_2O_4$ 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 $MgAl_2O_4$ 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).

1. Introduction

Transparent magnesium aluminate spinel ($MgAl_2O_4$) 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_2O_3 [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 $MgAl_2O_4$ spinel with LiF [29]. Indeed, the authors presented the reaction of LiF with Al_2O_3 on the particle surfaces, which leaves MgO-rich areas and the formation of $LiAlO_2$ 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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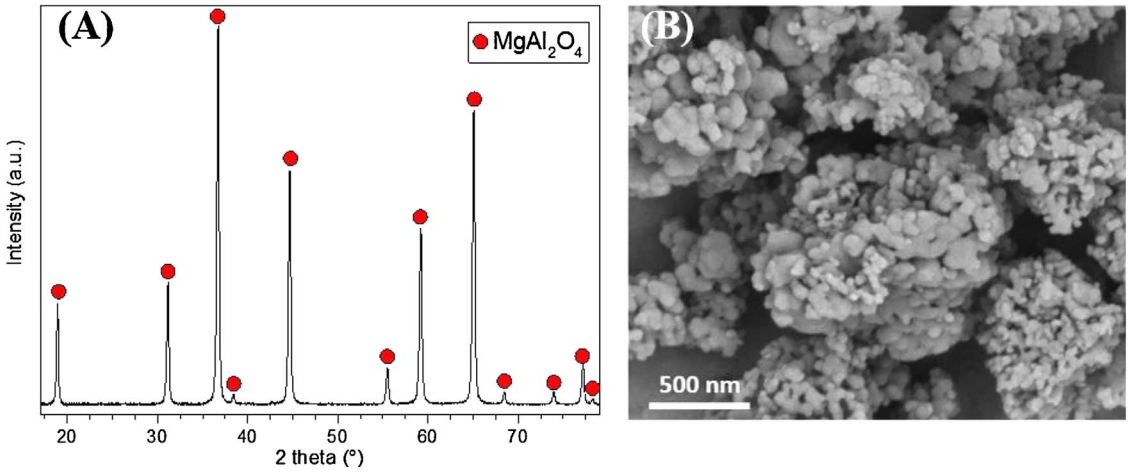

Fig. 1. XRD pattern (A) and SEM observation (B) of the commercial spinel $MgAl_2O_4$ powder.

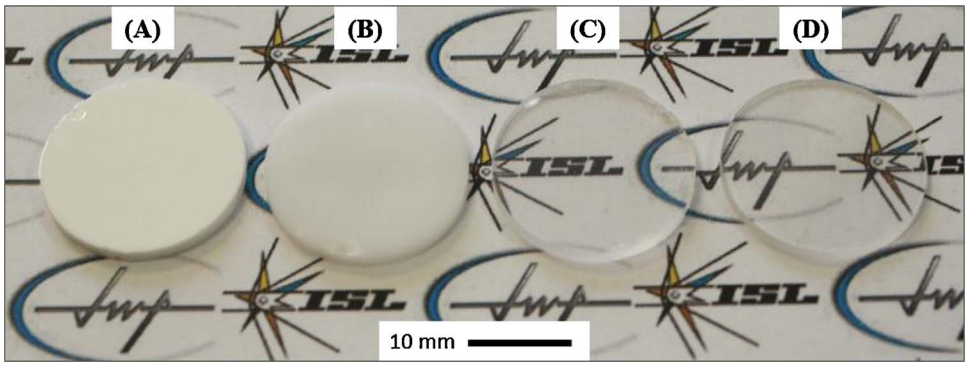

Fig. 2. Visual aspect of $MgAl_2O_4$ spinel after pressureless sintering (A) and after post-HIP at 1500 °C for 10 h (B) and 1800 °C for 1 h (C) and 10 h (D).

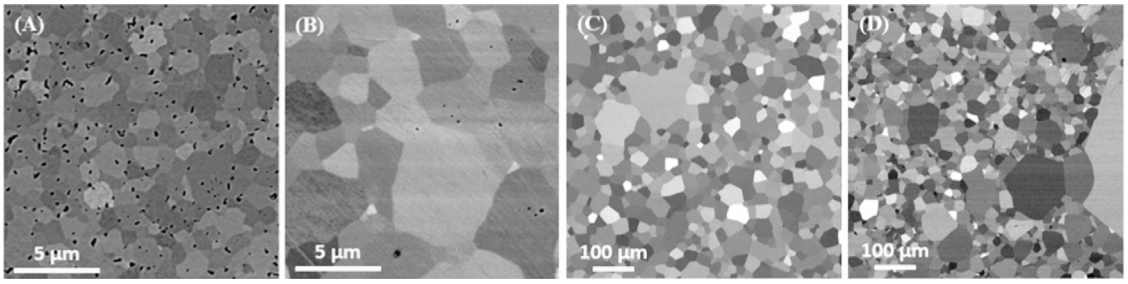

Fig. 3. SEM microstructures of $MgAl_2O_4$ spinel after sintering (A) and after post-HIP at 1500 °C (B) and 1800 °C for 1 h (C) and 10 h (D).

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 $MgAl_2O_4$ spinel has been studied and is presented in this paper.

2. Experimental procedure

Commercial $MgAl_2O_4$ spinel powder (grade S25CR, Baikowski, France) with a grain size of 0.43 µm (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 Cu Kα radiation ($\lambda = 0.15406$ nm) on the commercial $MgAl_2O_4$ 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:

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