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# Hot-pressing of bimodally distributed magnesium fluoride powder

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ABSTRACT

In the present study, nearly dense polycrystalline magnesium fluoride ceramics were fabricated by hot-press sintering, using bimodal size distribution $MgF_2$ powder. Densification was carried out at 710 °C under 300 MPa pressure for 60 min. The effect of weight percent of ultrafine powder on densification and also transparency, hardness and density of the samples produced are discussed. The results shown that sample with 15 wt.% ultrafine component, has the highest transparency, density, hardness, and a well developed microstructure, containing the lowest amount of porosities with small size.

## 1. Introduction

Polycrystalline high-density magnesium fluoride, preparing by the hot-pressing technique, has been extensively used as transmission windows due to the high transmittance in the infrared wavelength region, high mechanical strength and thermal stability [1,2]. This ceramic also displays other interesting properties such as chemical inertness, light weight and low refractive index [3]. $MgF_2$ optical ceramic is of particular importance for transmittance in the range of about 1–7 μm, and it does not show transparency in the ultraviolet and visible regions [4].

$MgF_2$ ceramics can be fabricated by different techniques such as melt casting, sintering, hot-isostatic pressing and hot-pressing. From the preliminary studies, hot-pressing was found to be so encouraging that an extensive program was carried out in preference to the other methods, each of which was believed to harbor limitations considered more difficult to overcome [5]. Irtran 1 is the commercial name of the first optical polycrystalline magnesium fluoride made in the early 1960s [6].

Recently, a few studies have shown the dependency of $MgF_2$ transparency on the microstructural parameters [7]. Much of the transmittance reduction in the best HP $MgF_2$ samples is likely to be caused by light scattering by residual pores. Other causes of transmission loss are scattering by grain boundary and surface roughness; absorption impurities and vibration between bound impurity-matrix state [1].

However, producing fully dense optical body, essentially free of absorption bands, of pure magnesium fluoride powder by hot-pressing is difficult. Absorption bands correspond to hydroxyl at about 2.8 μm, carbon dioxide at about 4.3 μm, bifluorides at 5 μm, and carbonate at 7 μm wavelengths, as well as the absorption bands at 3.0 and 6.1 μm for moisture. Hot-pressed $MgF_2$ has some residual water and hydroxide radicals, which are caused by the presence of atmospheric moisture during its fabrication [4,6,8].

However, pore boundaries have the greatest scattering power and therefore the biggest effect on reducing transparency as compared to other parameters. Previous work also showed that, achieving the best results requires the minimum amount of pore and also homogeneous grain distribution [1,11]. The purpose of the present work is to prepare dense $MgF_2$ bodies by using bimodal size $MgF_2$ powders. In this study the effect of using bimodal size powders on densification behavior, transmittance, microstructure and hardness of the final product is investigated.

## 2. Experimental procedure

Ultrafine $MgF_2$ powder was synthesized by precipitation method using NaF and $MgCl_2$ (Merck Co., Germany) as the raw materials. The process for precipitation was the same as what reported by other researchers [9,10]. The commercial micro-sized $MgF_2$ powder also were produced by attrition milling of 1–3 mm granulated $MgF_2$ powder (Umicore Co., France). Fig. 1 shows the micrograph of the synthesized and commercial $MgF_2$ powders obtained by these two methods and used in this study. It was found that the average particle size were ∼1 μm and ∼200 nm, for micro-sized and ultrafine $MgF_2$ powders, respectively.

These two powders were used to prepare a bimodal size $MgF_2$ powder mixture. The weight percent of the ultrafine powder in the mixture was 0, 5, 10, 15, 20 and 25 wt.%. The two different sizes powders were dispersed in acetone and mixed using an attrition mill in the presence of high-purity zirconia balls for 4 h in a plastic containers. After drying, the mixed powders were screened through a 120-mesh sieve, to produce well granulated powder. The granulated mixed powders were die-pressed using a BN-

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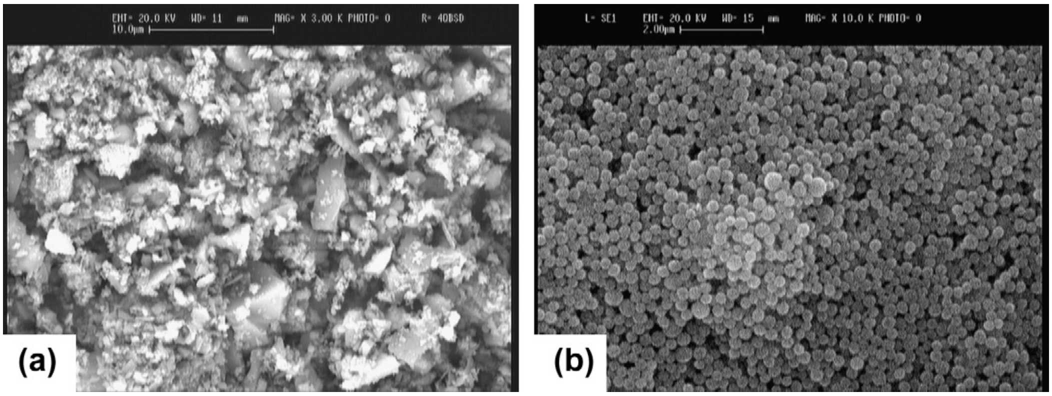

**Fig. 1.** Scanning electron micrograph, images of (a) micro-sized and (b) ultrafine powders.

coated steel die into cylindrical shape of 12 mm diameter at room temperature under 50 MPa pressure. The green samples were then hot-pressed in air at 710 °C under 300 MPa pressure for 60 min. To hot-press $MgF_2$ powder to high densities, it is not necessary to add any sintering aid. Using boron nitride (BN) powder suspension coated on the Inconel 718 die surface, eliminated any sticking between the product and the die surface during the hot-pressing process.

For transmission measurement, samples were given a final polish on both sides with 0.3 μm alumina polishing suspension to obtain a good surface finish. Transmittance was measured by Spectrum GX FTIR spectrophotometer. The density of the samples was measured by the Archimedes method. Mechanical properties of the samples was determined by micro-hardness test. The micro-hardness test was performed with the load of 100 g and dwell time of 15 s. It was done on five points for each sample. For micrograph studies, the fracture surface of the specimens was observed using a scanning electron microscope (Leica Cambridge S360).

## 3. Results and discussion

Fig. 2 shows the relative density of the consolidated samples as a function of weight percent of the ultrafine powder used. As it can be seen, the samples including partially ultrafine powder were densified to near theoretical density, 3.17–3.18 g/cm$^3$. By increasing the weight percent of the ultrafine powder in the initial mixtures in the range of 5–25%, the density first increases slightly and then decreases.

Fig. 3 shows the scanning electron micrograph of the fracture surface of the samples includes (a) 0, (b) 10, (c) 15 and (d) 20 wt.% of the ultrafine powder. It is obvious that, as it mentioned before, significant reduction in the amount of pores and average pore size occurred by the addition of ultrafine powder (a–c). With an excessive increase in the ultrafine powder, the amount and the size of the porosities increase again.

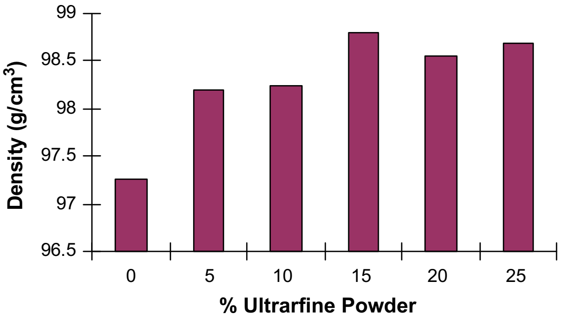

**Fig. 2.** Relative density vs. weight percent of ultrafine powder used in the initial $MgF_2$ powder mixtures.

Figs. 2 and 3 demonstrate the enhanced densification of $MgF_2$ ceramics, due to the use of bimodal size powder mixtures. This can be attributed to the improved green density, due to the void filling ability of fine particles, which cause a higher number of contact point among the particles occurs, and therefore, it enhanced the densification of low-reactivity coarse powders [12].

Also, increasing the fraction of fine particles, decreases the diffusion paths in the resultant compact, and so improves mass transport during hot-pressing [13]. The same studies carried out for investigating the effect of particle size distribution on densification behavior of alumina powder, also showed that, the initial densification rate is enhanced appreciably with broader particle size distribution [14,15]. However, these approaches are effective to a special amount of fine particles. Exceeding the optimum value (15 wt.%), causes agglomeration and structural inhomogenity, and deteriorates compaction and sintering characteristics of the compact, which leads to the decrease in the density, and presence of more porosities. It is worth mentioning that ultrafine particles are prone to agglomerate and the pores retained in the agglomerated zones decrease the density [13].

Fig. 4 shows the transparency in the IR region for hot-pressed magnesium fluoride ceramic includes (a) 0, (b) 5, (c) 10, (d) 15, (e) 20 and (f) 25 wt.% ultrafine $MgF_2$ in their raw materials. It can be seen that the addition of ultrafine synthesized magnesium fluoride up to 15 wt.%, enhanced the transparency significantly, and above this weight percent, it has an adverse effect. This result is in good agreement with density and microstructure trends shown in Figs. 2 and 3.

In a HP $MgF_2$ samples, the transmission loss of incident radiation may inevitably be due to scattering by the presence of a large number of micro-pores within the material and by the optical anisotropy [1]. High transparency at infrared wavelengths could be obtained by increasing the density and eliminating the porosity which is the main cause of scattering.

Fig. 5 shows relation between the hardness and percentage of the ultrafine component used in the initial $MgF_2$ powder mixtures. It is clear that a maximum appears at 20 wt.%.

As a fact, hardness has a direct relationship with respect to density. It is obvious that, the first traces of porosity have a disproportionately damaging effect on mechanical properties. Thus, elimination of porosity due to density increment, improves hardness of ceramics. Previous studies on different ceramics also confirmed this relationship [16,17].