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Research Article

Effect of Sintering and Synthesis of Transparent Alumina

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Abstract

Transparent alumina samples have been prepared by microwave sintering processing. Transparent alumina can be achieved at lower sintering temperature and shorter sintering time by microwave sintering. Transmittance is optical property which is highly dependent on the grain size and residual porosity which lead to real in line transmittance. A systematic experimental design is used to study transmittance of alumina. The following work was carried out using MgO as sintering aid for all Al_2O_3 samples. By using optimized parameters, the transmittance of single crystal alumina was obtained about 35% - 20% in visible wavelength for 2 mm thick samples. The results are significantly better.

Keywords: Transparent alumina; Single crystal; Microwave sintering; Ceramics.

Introduction

Transparent ceramics are the ceramics materials that show high total and inline transmission. Transparent ceramics are produced from various ceramics by using various sintering methods. These transparent ceramics are widely used in many applications in the form of films, coatings, and fibers [1]. Transparent ceramics has many applications in lasers, night vision, missiles, armor and many more electro-optical applications. Most ceramics such as alumina and zirconia are formed by fine grained polycrystalline micro structure, so they are opaque. Recent developments in sintering and other processes came out with polycrystalline transparent ceramics such as Y_2O_3, Y_2O_3.La_2O_3 and Al_2O_3.The length of the grains or crystallites in a polycrystalline alumina is grain size of that polycrystalline material. This grain size is expected in (5μm-0.5 μm) to get transparent polycrystalline alumina. The change in grain size must be (0.6 μm-0.4 μm).Residual pores are the isolated pores or voids trapped by air. These residual pores cause multiple reflections which are known as birefringence.

The solid state process is bulk conversion of a polycrystalline ceramic body to a single crystal body by heating the polycrystalline body to temperatures above one-half the melting temperature of the material but below the melting temperature of the material. More particularly, this invention relates to a solid state process for the bulk conversion of polycrystalline alumina (PCA) to a single crystal alumina (sapphire) [2]. The solid state conversion of PCA to sapphire is accomplished by heating a PCA body containing less than 100 ppm of magnesia to a temperature above ll00° C. but below 2050°C, the melting point of alumina. Transmission of about 80% is attained by this method. Hot pressure (HP) sintering is a high-pressure low-strain-rate powder metallurgy process for forming of a powder or powder compact at a temperature high enough to induce sintering. This is achieved by the simultaneous application of heat and pressure. Densification at high pressure works through particle rearrangement and plastic flow at the particle contacts [3].

The light transmission of polycrystalline alumina ceramics is relatively small, in contrast to single cylindrical alumina, which shows a complete transparency. This holds for the total transmission as well as for the in line transmission. The reason for the small light transmission of polycrystalline alumina is grain boundaries and residual pores acting as scattering center of the light [4]. Thus, the translucency of polycrystalline ceramic material can be increased by completely eliminating residual porosity. Residual pores are single and isolated voids caused by trapped air during sintering which is normally carried out in an air atmosphere. The sintering atmosphere plays an important role in obtaining a fully dense material

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and hence a high transmission co efficient. To eliminate the pores it is necessary to remove the gas from the pores before they become isolated. According to a basic study on the solubility of gas in alumina conducted by coble, 12 the alumina lattice shows only the some limited solubility for pure hydrogen and pure oxygen whereas nitrogen and all inert gases are not soluble. From this it came be concluded that a sintering atmosphere containing nitrogen like air, for example or inert gases will never lead to a transparent alumina body [5]. But when sintering in hydrogen a sintering process in oxygen is not practicable. A remarkable total transmission of up to 96% can be contained if alumina is of high purity and sintering time is long enough to allow the gas to a diffuse through or into the alumina lattice [6].

The disadvantage of this process however is an exaggerated grain growth because of the long sintering time, lead to the significant loss in strength compared to a fine grained alumina body. Therefore, a completely different production process of translucent alumina ceramics have been developed which prevents large grain size. The basic idea is to remove the sintering atmosphere from the pores not by diffusion during sintering but rather by evacuation before the beginning of final stage sintering. This idea can be realized by carrying out the final stage sintering. Under high vacuum, very pure alumina is purified in air in order to remove all the organic benders at atmosphere low enough before the pores become isolated subsequently the body is placed into a vacuum furnace and evacuated during the heating up period the pore, which are now free from any gas, an close completely at once, this permits the application of short sintering times which leads to a sintering body with full density and grained micro structure [7]. The total transmission turns out to be the same as that of alumina body sintered in hydrogen.

Materials and methods

Selection of ceramic material

Alumina (Al_2O_3) exhibits many interesting properties, such as high strength, high hardness and excellent corrosive resistance. This makes transparent Al_2O_3 ceramics a promising candidate for applications as electromagnetic windows, transparent armor and envelopes of high-pressure metal halide lamps [8]. Sintered Al_2O_3 ceramics with sub micrometer grain size are the hardest of all transparent armor (including sapphire). Transparent polycrystalline alumina is believed to be a promising replacement for sapphire. Optical and mechanical properties of transparent Al_2O_3 ceramics are highly dependent on their grain size and residual porosity. Various strategies have been employed to control the grain sizes and minimize the residual porosity. For this purpose, fine-grained transparent Al_2O_3 ceramics have recently attracted much attention. The fine-grained ceramics demonstrated a significant improvement in mechanical strength and optical transparency. It has been reported that typical fine grained transparent Al_2O_3 ceramics had strength of up to 600e 800 MPa and high in-line transmission of up to 60%.

Selection of sintering method

Transparent alumina samples have been successfully prepared by microwave sintering processing. In comparison to the conventional sintering processing, microwave sintering to transparent alumina can be achieved at lower sintering temperature and shorter sintering time. It was also found that the microwave heating could substantially increase the conversion rate of polycrystalline alumina to single crystalline sapphire, to improve the transparency and other properties of the transparent alumina samples.

Pretreatment of alumina powder

High purity (99.99%) commercial alumina powders with primary particle size of 0.15 Am were used as starting materials in this study. The starting powder was blended in acetone with 0.025, 0.05, 0.1, 0.25 weight % of MgO (in form of Mg $(NO_3)_2.5H_2O$) and organic binder (PCA) using alumina mortar. The samples were mixed in mixer for 4 hours for better mixing and heated in a furnace at 60ºC for 1 hr.

Processing of green body

Green samples were prepared by dry pressing uniaxially into pellets followed by lab press at a pressure of 280 MPa. The green densities of the compacts were around 52–54%. The compacted pellets were preheated at 1100ºC for 2 h in a resistance furnace to burn out the binder. The microwave sintering was carried out using a single mode microwave applicator coupled with a 1.5 kW microwave generator operating at 2.45 GHz .Pressure was used as sintering atmosphere for all samples. The transmittances were measured using UV-Visible spectrophotometer.

Optimization of parameters

Preparation of samples

The alumina powder was separated into 4 different samples and mixed with different compositions of MgO to form 4 batches. The batches (Table 1) are mixed with 50 ml of acetone and mixed in a mixer for 4 hours, After that samples were heated at 60ºC for an hour then These batches were then mixed with few drops of PCA (organic binder) and pressed in lab press to 2.5 cm × 2.5 cm square shaped dye. These samples were then sintered in microwave sintering process at 1600ºC for half an hour at a rate of 100ºC/min. The sintered samples are then cut into halve to fit the UV-Visible spectrometer. The samples were about 2.5 cm × 1.25 cm. The sintered and cut samples are then tested in UV-VISIBLE Spectrometer to obtain the transmittance % of that sample. The transmittance of the sample at different wavelength are also obtained and represented as a graphical value.

Table 1. Concentration of sintering aid in batches

Batches	Alumina (g)	Magnesia (g)
0	50	0.0
1	50	0.025
2	50	0.05
3	50	0.1
4	50	0.25

Results and discussions

The results for the transmittance of the alumina samples with different concentration of sintering aid (MgO) are summarized in the following table 2. The densification behaviors of the TA samples during the microwave sintering are shown in Fig. 1. All samples were sintered in a single mode microwave cavity at different temperatures with the same dwelling time of 30 minutes. It was observed that there was little. We believe that highly translucent with high thermal conductivity TA samples can be prepared by microwave sintering if reduction of the oxygen content and addition of suitable sintering aids are employed.

Table 2. Transmittance % of all the batches at wavelength 520 μm

Batches	Alumina (g)	Magnesia (g)	Transmittance (%)	Wavelength (μm)
0	50	0.0	30	520
1	50	0.025	25	520
2	50	0.05	23	520
3	50	0.1	20	520
4	50	0.25	18	520

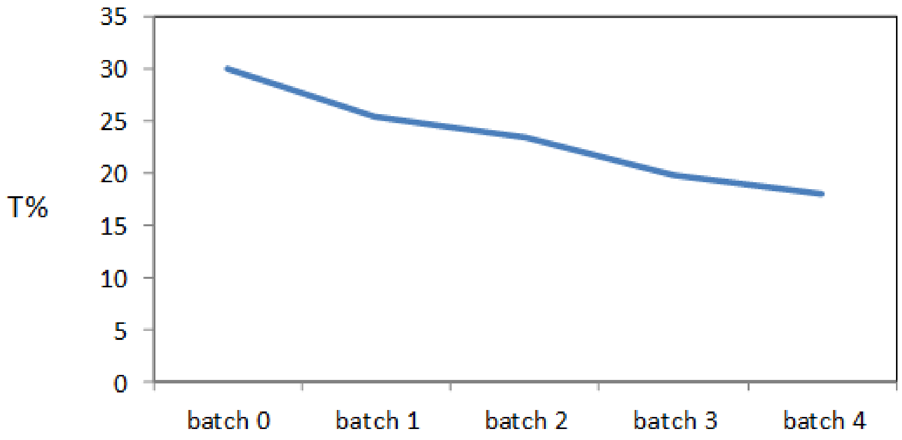

Fig. 1. The transmittance measurements of the microwave Sintered Al_2O_3 samples at wavelength of 520 μm

Conclusions

Transparent alumina samples have been successfully prepared by microwave sintering processing. In comparison to the conventional sintering processing, microwave sintering to transparent alumina can be achieved at lower sintering temperature and shorter sintering time. It was also found that the microwave heating could substantially increase the conversion rate of polycrystalline alumina to single crystalline sapphire, to improve the transparency and other properties of the transparent alumina samples. Size and thickness of transparent ceramics are important parameters for assessing their resistance to withstand thermal shock and stresses. The wide range of new transparent ceramic envelope materials represents opportunities for advanced light-sources such as beamers and automotive headlights of new designs.

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Conflict of interest

Authors declare there are no conflicts of interest.

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