

# Bulk, Dense, Nanocrystalline Yttrium Aluminum Garnet by **Consolidation of Amorphous Powders at Low Temperatures and High Pressures**

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Amorphous Al<sub>2</sub>O<sub>3</sub>-37.5% Y<sub>2</sub>O<sub>3</sub> powders, prepared using spray pyrolysis followed by partial or complete thermal decomposition, were hot-pressed at 315°-640°C and 500 or 750 MPa uniaxial pressure. Hot pressing of fully decomposed amorphous powder at 450°-640°C at pressures up to 750 MPa led to densification (up to 96%) as well as nanocrystallization of yttrium aluminum garnet (YAG). When the pressure was applied during heating, instead of after reaching the final temperature, higher relative densities resulted. Fully crystalline starting powder did not densify. The low true density of the amorphous phase  $(3.1 \text{ g} \cdot \text{cm}^{-3})$  was believed to be responsible for the densification through enhanced ionic mobilities.

## I. Introduction

I T RECENTLY has been shown<sup>1-3</sup> that amorphous  $ZrO_2$ -Al<sub>2</sub>O<sub>3</sub> powders, prepared using spray pyrolysis of aqueous solutions of nitrates, can be hot-pressed to high relative densities (95%-99%) while retaining the amorphous state. Bulk nanocrystalline metastable tetragonal- $ZrO_2(Al_2O_3)$  solid solutions with 8 nm grain size have been produced by crystallization of the dense amorphous samples. Under the conditions used for consolidation (uniaxial pressures of 500-750 MPa and temperatures of 450°-650°C), the amorphous powders exhibit densification behavior that is largely independent of time. The relative density increases monotonically and instantaneously with applied pressure. The true density of the amorphous phase with composition ZrO<sub>2</sub>-40 mol% Al<sub>2</sub>O<sub>3</sub>, at 3.4 g·cm<sup>-3</sup>, is considerably less than that of the equilibrium phases (5.0 g·cm<sup>-3</sup> for a mixture of monoclinic-ZrO<sub>2</sub> and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>). It is suspected that the open structure of the metastable amorphous phase can, in part, be responsible for the ease of plastic deformation at the relatively low temperatures of consolidation. The low yield stress of the amorphous phase can be inferred from the low room-temperature hardness of 4 GPa (compared with  $\sim 10$  and 18 GPa for ZrO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>, respectively), and, subsequently, the ability to plastically deform under uniaxial compression at stresses of  $\sim$ 360 MPa at 600°–700°C has been established.<sup>4</sup>

It also was found that as-sprayed, partially decomposed Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> powders ex hibit marked sinterability when heated to their sintering temperature through the temperature range of complete decomposition (up to 750°C). Sintering does not occur once the thermal decomposition is complete. While this feature cannot be exploited during hot pressing because of the need to allow volatile species to escape, there remains the possibility that these partially

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decomposed phases can be densified under pressure, while remaining below the critical temperature at which further gas evolution occurs. The above considerations also extend to higher temperatures and pressures (700°–900°C and 1 GPa),<sup>5,6</sup> under which conditions the amorphous phase crystallizes during densification. At 700°C, the relative density achieved is higher in the presence of the metastable phases of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and tetragonal-ZrO<sub>2</sub> than at  $800^{\circ}-900^{\circ}C$ , where the equilibrium phases of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and monoclinic-ZrO<sub>2</sub> appear.

To investigate the generality of the above phenomena, an amorphous material with composition corresponding to yttrium aluminum garnet (YAG, Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>) has been chosen for consolidation using a route similar to that described above for Al2O3-ZrO<sub>2</sub>. Precursor-derived amorphous phases in Al<sub>2</sub>O<sub>3</sub>-Y<sub>2</sub>O<sub>3</sub> crystallize at temperatures as high as  $\sim 900^{\circ}$ C,<sup>7</sup> which allows a wide range of temperatures for hot pressing. Moreover, the radius of the  $Y^{3+}$  ion is slightly larger than that of the  $Zr^{4+}$  ion<sup>8</sup> and can be expected to lead to open, low-density structures similar to that encountered in Al<sub>2</sub>O<sub>3</sub>–ZrO<sub>2</sub>. The ability to produce the amorphous counterpart of YAG allows the synthesis of a material with new electronic and optical properties.

Chemical precursor synthesis of phase-pure and nanocrystalline YAG has been widely studied. Metastable microstructures with phases such as orthorhombic- or hexagonal-YAlO<sub>3</sub> and  $\alpha$ - or  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> are encountered even in nominal compositions corresponding to YAG (37.5 mol% Y2O3).9,10 YAG synthesis experiments frequently have produced single-phase YAG as the first (and only) product of crystallization,<sup>11–15</sup> although, occasionally, an intermediate hexagonal-YAlO<sub>3</sub> has served as the crystalline precursor to YAG.<sup>16-19</sup> Similar intermediate phases form during mechanochemical synthesis.<sup>20</sup> These results indicate the need to preserve molecular homogeneity during precursor synthesis, including sol-gel and spray pyrolysis routes. In situations where segregated phases appear (e.g., Y2O3, Y4Al2O9, and YAlO3), subsequent reaction is needed at higher temperatures to produce garnet.<sup>16</sup> However, it has been shown in recent reports that phase-pure YAG powders can be synthesized through lowtemperature chemical precursor synthesis routes, such as spray pyrolysis,<sup>7</sup> coprecipitation,<sup>21,22</sup> glycothermal method,<sup>23</sup> and mixed-metal citrate precursor method.<sup>24</sup> Most of these routes yield nanocrystalline YAG powders at temperatures <1000°C.

Processing of bulk, dense nanocrystalline YAG has proved to be as difficult as processing other nanocrystalline oxides. The usual tradeoff between residual porosity and grain growth<sup>25–30</sup> has been made in sintering of nanometer-sized powder particles.<sup>21,31–33</sup> Sintering temperatures >>1000°C are usually required for obtaining high relative density. In this context, the present research studies the processing of bulk, nanocrystalline YAG, as well as the densification of spraypyrolyzed amorphous powders with the YAG composition of Al<sub>2</sub>O<sub>3</sub>-37.5%  $Y_2O_3$  at temperatures of  $\leq 640^{\circ}C$ .

### **II. Experimental Procedure**

An aqueous solution (200 g·L<sup>-1</sup>) of Al(NO<sub>3</sub>)<sub>3</sub> and Y(NO<sub>3</sub>)<sub>3</sub> corresponding to the composition of YAG (37.5 mol% Y<sub>2</sub>O<sub>3</sub>) was

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sprayed with compressed air onto a Teflon-coated aluminum pan maintained at 285°-305°C. The resulting partially decomposed powder was studied using thermogravimetric analysis (TGA; Model TG-171, Cahn Instruments, Cerritos, CA) at a heating rate of 10°C/min to identify the temperature range of weight loss accompanying complete thermal decomposition. Differential thermal analysis (DTA; Polymer Laboratories, Epsom, U.K.) performed at 10°C/min on the as-sprayed powder and X-ray diffractometry (XRD; Model JDX-8030, JEOL, Tokyo, Japan) performed on heat-treated powders were used to identify the crystallization temperature and the product of crystallization. Amorphous powders with an increasing degree of thermal decomposition were obtained by heat treatments at 365°, 450°, and 600°C, whereas fully crystalline YAG powder was obtained by heat treatment at 1000°C. The heating rate was 10°C/min and the holding time was 1 min, after which the sample was air cooled. Sedimentation was conducted in a 15 cm tall column of xylene to obtain particles  $<20 \ \mu m$ .

The true densities of the heat-treated powders were measured using pycnometry with specific gravity bottles of 10 mL volume and xylene as the fluid medium. The powders were ground using a mortar and a pestle to break agglomerates. A period of 10 d was allowed to achieve full infiltration of the finest interparticle voids.

Uniaxial hot pressing was conducted at temperatures ranging from  $315^{\circ}$  to  $640^{\circ}$ C, in a 5 mm diameter cylindrical die made of nickel-based superalloy (Alloy 718, Midhani, Hyderabad, India). In the cases of partially decomposed powders, hot pressing was performed at a temperature lower than or equal to the heat treatment to minimize the evolution of volatile matter and buildup of gas pressure inside the compact. Most of the experiments were performed under load control using a servo-hydraulic universal testing frame (Model 9500 Console, DARTEC, Stourbridge, U.K.). The pressure was applied at 10.2 MPa·s<sup>-1</sup>, either at room temperature or after the final temperature was reached. A few experiments were performed under a constant displacement rate of 10  $\mu$ m·s<sup>-1</sup> in a screw-driven universal testing frame (Instron, Canton, MA) fitted with a temperature-compensated extensometer that recorded the change in sample thickness during hot pressing.<sup>3</sup>

Hot-pressed samples were checked for crystallinity using XRD and for porosity using optical image analysis and, where possible, the Archimedes method. Image analysis was done on optical micrographs captured with a computer-controlled display (CCD) camera at a magnification of 600. The porosity of a sample was measured by specifying the gray-scale range of the pores (dark regions) and measuring the area fraction of this range. The error range of  $\pm 1.5\%$  in relative density was estimated by measurements made by first including and then excluding fine pores (<5 µm), which had lighter contrast. Including these pores led to the expansion of areas of larger pores and an overestimate of porosity.



Fig. 1. TGA of as-sprayed  $Al_2O_3$ –37.5%  $Y_2O_3$  powders prepared by spray pyrolysis of aqueous nitrate solutions.



Fig. 2. DTA of as-sprayed  $Al_2O_3$ -37.5%  $Y_2O_3$  powders show exothermic peak with maximum at ~921°C, which corresponds to the crystallization of YAG.

The relative density was quoted as 100%-% porosity. Transmission electron microscopy (TEM; Model 000FX-II, JEOL) was performed on hot-pressed samples after they were crushed to fine powders, mounted on a copper grid, and coated with carbon.

#### III. Results and Discussion

Figure 1 shows the TGA plot of the as-sprayed  $Al_2O_3$ -37.5%  $Y_2O_3$  powder. The major weight loss, which follows the loss of adsorbed moisture up to ~100°C, occurs between 325° and 480°C, which is attributed to the completion of thermal decomposition. DTA reveals an exothermic peak at 921°C (Fig. 2). The endotherms at low temperatures correspond to the weight loss due to thermal decomposition. As discussed below, this peak is due to the crystallization of YAG. Based on the above information, the four temperatures of heat treatment (365°, 450°, 600°, and 1000°C) of the as-sprayed powder were selected for hot-pressing experiments.

XRD patterns of the as-sprayed powder in Fig. 3 show that it is amorphous. After heat treatment at 600°C, broad and weak crystalline peaks that correspond to  $Y_2O_3$  appear, which indicates



Fig. 3. (a) XRD patterns of  $Al_2O_3$ -37.5%  $Y_2O_3$  powder heat-treated at 365°C shows that it is amorphous. (b) Heat treatment at 600°C yields predominantly amorphous phase, with some  $Y_2O_3$  because of minor segregation during spray pyrolysis.





**Fig. 4.** XRD pattern of  $Al_2O_3$ -37.5%  $Y_2O_3$  powder heat treated at 875°C shows the beginning of YAG formation from the amorphous phase and residual  $Y_2O_3$  (Y). Heat treatment at 1000°C produces single-phase YAG.

some segregation during spray pyrolysis. However, a sample heated to 875°C (Fig. 4) shows the beginning of YAG formation and some residual  $Y_2O_3$ , whereas, by 1000°C, only single-phase YAG is formed, and no residual  $Y_2O_3$  is detected using XRD. The fact that the heat treatment of the as-sprayed powder at these temperatures produces YAG, and no other phase, indicates that the segregation is minor.<sup>7</sup> The possibility of forming YAG when the major volume fraction has composition slightly leaner in  $Y_2O_3$  than 37.5% cannot be ruled out.<sup>15</sup> The influence of the small amount of yttrium-rich regions on the densification behavior has been neglected.

The true densities obtained using pycnometry measurements of the powders heat treated to 365°, 450°, and 600°C are 2.7, 3.0, and 3.1 g·cm<sup>-3</sup>, respectively. Although the densities of the amorphous powders increase with degree of decomposition, the values are considerably lower than that of the equilibrium garnet, which has a density of 4.55 g·cm<sup>-3</sup>. Table I summarizes the results of the hot-pressing experiments. Representative optical micrographs of polished samples on which porosity measurements have been made after hot pressing are shown in Fig. 5. Table I shows that the partially decomposed powders do not achieve significant densities, irrespective of whether pressure is applied during or after heating. On the other hand, the powder that is fully decomposed at 600°C can be pressed to relative densities as high as 96%, provided the load is maintained during heating. Other conditions remaining the same, the relative density increases from 83% to 96% (samples 4 and 9,



**Fig. 5.** Photomicrographs of samples hot-pressed at 750 MPa at (a) 640°C (sample 9 in Table I), and (b) 450°C (sample 4). Subsurface porosity, corresponding to diffuse intermediate contrast, has been omitted during porosity measurement.

respectively) as the hot-pressing temperature increases from 450° to 640°C. A significant amount of densification (up to 95%) occurs when predominantly amorphous powder is hotpressed (sample 6), as compared with fully crystalline powder hot-pressed under the same conditions (sample 10). The final relative density of sample 10 is very close to the initial density expected after cold compaction. This implies that the crystalline powder is not sinterable under the conditions used here. Indeed,

Sample	Starting powder condition	Heat treatment (°C)	Hot-pressing temperature (°C)	Applied pressure (MPa)			Relative
				Cold press	Hot press	Time (min)	density (%)
1	Partially	365	315	50	500	30	70
2	decomposed	450	400	50	500	30	72
3			450	$750^{\dagger}$		0	78
4	Fully	600	450	$750^{+}$		0	83
5	decomposed		550	50	500	30	78
6			600	$750^{+}$		0	95, 91 <sup>‡</sup>
7			600	50	750	0	84
8			600	100	750	15	84
9			640	$750^{\dagger}$		0	96
10	Crystallized	1000	600	$750^{\dagger}$		0	68 <sup>§</sup>

Table I. Results of Hot-Pressing Experiments on Amorphous and Crystalline Powders of  $Al_2O_3$ -37.5%  $Y_2O_3$  Prepared Using Spray Pyrolysis<sup>†</sup>

<sup>†</sup>Heating under pressure. <sup>‡</sup>Repeated experiment. <sup>§</sup>Density measured using Archimedes principle is 3.09 g·cm<sup>-3</sup>.

Intensity

 $\mathbf{20}$ 

the extensive interconnected porosity precludes a ceramographic polish in sample 10.

Figure 6(a) shows a plot of the punch displacement against the increasing pressure, and Fig. 6(b) shows displacement as a function of time with the pressure held constant at 500 MPa during hot pressing of powders with varying degrees of thermal decomposition. The greater part of densification is achieved during pressurizing with only a small increase with time when the pressure is held constant at the hot-pressing temperature. XRD traces of selected hot-pressed pellets are shown in Fig. 7. The samples that reach near theoretical density have crystallized, whereas the onset of crystallization is detected in samples hotpressed under less-severe conditions, e.g., at 450°C and 750 MPa (sample 4) or 550°C and 500 MPa (sample 5). The grain size has been estimated from X-ray peak broadening scanned in the range of 20°-40° 20. The hot-pressed pellet, when heated to 1000°C (sample 9), has a grain size ( $\sim 27$  nm) comparable with that obtained when the as-sprayed powder is heated to the same temperature. This implies that crystallization during hot pressing does not affect the subsequent grain growth.

In experiments in which pressure is applied during heating of the amorphous powder compacts, densification up to  $\sim$ 80% occurs before the initiation of crystallization. Densification and increase in the volume fraction of YAG occur after the crystallization stage. Figure 8 shows a bright-field TEM micrograph of the sample pressed at 450°C and 750 MPa. Nanocrystals of YAG appear in the matrix of the amorphous phase. Although the volume fraction of the amorphous phase continues to decrease, it is expected to continue to participate in the densification process. It is also likely that the transformation to YAG assists densification. However, a compact pressurized to 750 MPa after reaching 600°C (sample 7) is crystalline after pressing, and its final relative density is 84%,



**Fig. 6.** (a) Change in the compact thickness as a function of applied pressure during hot-pressing of  $Al_2O_3$ -37.5%  $Y_2O_3$  powders with varying degrees of decomposition as indicated by the heat-treatment temperatures. (b) Change in the thickness as a function of time, at 500 MPa.



Fig. 7. XRD patterns of hot-pressed pellets of  $Al_2O_3$ -37.5%  $Y_2O_3$  (sample details are presented in Table I). Crystalline peaks correspond to YAG.

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**2**0

compared with 91%–95% obtained by heating compacts of sample 6 to 600°C at 750 MPa. Therefore, it is important to heat a compact under pressure to obtain high relative densities. When the XRD patterns of samples 6 and 7 are compared (Fig. 7), it is evident that heating under load also leads to higher degree of crystallization.

G A A To m

**Fig. 8.** Bright-field TEM micrograph of  $Al_2O_3$ –37.5%  $Y_2O_3$  sample hot-pressed at 450°C and 750 MPa shows nanocrystals of YAG (G) in the matrix of amorphous phase (A). Inset shows electron diffraction pattern with crystalline ring pattern corresponding to YAG and an amorphous halo. Amorphous phase can continue to participate in densification, even as the volume fraction transformed to YAG increases during hot pressing.

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When the amorphous phase is exposed to pressure at lower temperatures before significant crystallization, higher densification occurs than if the pressure is applied only at temperatures of rapid crystallization. The implication is that the contribution of the amorphous phase to densification is dominant over any possible densification induced by crystallization. The higher sinterability of the amorphous phase is probably due to the open structure, as indicated by its high molar volume (low density), as compared with the crystalline phase (YAG), leading to higher ionic mobilities.

The applied pressure accelerates the crystallization of YAG, as expected from the molar volume difference between the amorphous phase and YAG. Similar acceleration of crystallization to tetragonal- $ZrO_2(Al_2O_3)$  solid solution, as well as its subsequent evolution into the equilibrium phases, occurs in hot-pressing experiments on  $ZrO_2$ –40%  $Al_2O_3$ , although at slightly higher temperatures and hydrostatic pressures (700°C and 1 GPa).<sup>5</sup> The present results are similar to the densification of amorphous  $ZrO_2$ – $Al_2O_3$ , in that the high-pressure, low-temperature consolidation of initially amorphous powders leads to dense compacts. However, the overlap of crystallization and densification in the present work appears to preclude the retention of the amorphous phase.

#### **IV.** Conclusions

Amorphous powders of Al<sub>2</sub>O<sub>3</sub>–37.5% Y<sub>2</sub>O<sub>3</sub> prepared by spray pyrolysis have been hot-pressed at low temperatures (315°– 640°C), under moderately high pressures (500–750 MPa). High relative densities (96%) are obtained when fully thermally decomposed amorphous powders are used. The results establish the generality of the phenomenon of densification of the amorphous phase found in the ZrO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub> system. Partially decomposed powders do not densify to high relative densities. Crystallization of YAG with grain size of ~20 nm accompanies densification. This feature allows the processing of almost fully dense nanocrystalline YAG at temperatures as low as 600°C, under moderately high pressures. Heating the compacts under pressure is important for obtaining high relative densities. Crystallization precludes the retention of amorphous phase in the dense compacts, in contrast with the hot-pressing behavior of amorphous ZrO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub>.

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