

Further experimental investigation on fast densification mechanism of bimodal powder during pressureless sintering of transparent ALON ceramics

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ABSTRACT

In our recent work we demonstrated that a bimodal particle size distribution (PSD) ALON powder could be fast densified by pressureless sintering (Shan et al., *Ceram. Int.* 41 3992–3998 (2015); Shan et al., *J. Eur. Ceram. Soc.* 36 67–78 (2016)). To obtain a further insight of its fast densification mechanism, this powder was held for 30 min and 60 min at 1500–1700 °C during heating, respectively. However, the added holdings resulted in a decrease in both relative density and transmittance, compared to that of the sample without holding during heating. Further investigation into phase transformation and microstructure evolution of these samples indicates that high heating rate enables the sintering mixtures to keep a near sphere particle shape in bimodal PSD until phase transformation from Al₂O₃ to ALON is fully completed. Then mass transport between ALON grains of different size can simultaneously happen at the final fast densification stage, which benefits less formation of pores and fast ALON grains growth. Therefore, high heating rate plays a key role to fast and better consolidation of the bimodal ALON powder.

1. Introduction

Transparent aluminum oxynitride (γ -ALON) of cubic structure has attracted a growing interest due to its excellent mechanical properties, superior resistance to chemical attack, and high transparency ranging from ultraviolet to mid-infrared, for that has many advanced applications [1–5]. ALON transparent ceramics can be fabricated by conventional sintering technique either with or without pressure in high temperature [6]. Pressureless sintering technique is favored for its low cost and easy shaping compared to hot pressing or hot isostatic pressing techniques [7–11]. Nevertheless, the pressureless sintering temperature of transparent ALON by using traditional powder is up to 1850–2000 °C and ≥ 6 h holding time is also needed. Consequently, energy consumption of pressureless sintering of transparent ALON by using traditional powder is still very high.

Recently, our work has demonstrated that the cost of pressureless sintering transparent ALON can be effectively cut by controlling the particle size distribution (PSD) of starting ALON powder [12,13]. It was confirmed that ALON powder having a bimodal PSD can be more efficiently sintered for its fast heating (40 °C/min) and a lower sintering temperature (1820–1880 °C). More importantly, holding

time of pressureless sintering transparent ALON using this bimodal powder can be remarkably shortened to 1.5–2.5 h.

In Wang's study, a surface treated ALON powder characterized by small and narrow pore size distribution of green body was obtained through a modified aqueous gelcasting forming technique. The fabricated ALON ceramics was highly transparent because small and narrow pore size distribution of green body favors elimination of pores during sintering [14]. Similarly, for a bimodal powder, its excellent gap filling ability of small particle benefits to reduce pore size and narrow pore size distribution, which should contribute to fast densification of the bimodal ALON powder [15,16].

Krell et al. have reported that pore within the transparent ceramics can result in its optical inhomogeneity and severely reduce its in-line transmission [17]. Apetz's study revealed that decreasing grain size (grain size > 30 μ m) can lead to a broader scattering profile because the light has to pass an increased number of grain boundaries [18]. Apparently, it is necessary to manipulate the densification and grain growth process to obtain the lowest porosity and wanted grain size for the best possible transparency. In these two key processes, phase assemblage and microstructure of sintered mixture should be carefully monitored to reveal its phase transformation and microstructure evolution mechanism.

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Willems et al. have revealed that AlON is thermodynamically unstable below a temperature between 1600 °C and 1640 °C [19], and our work demonstrated that Al_2O_3 , AlN and AlON may coexist in the samples during heating (1400–1700 °C) [13]. Phase assemblage analysis suggests that, with the bimodal powder heated to 1400 °C, all the AlON particles are transformed into Al_2O_3 , where fine AlON particles are transformed into hexagonal $\alpha\text{-Al}_2\text{O}_3$ and AlN, while coarse AlON particles are transformed into cubic $\eta\text{-Al}_2\text{O}_3$ and AlN. Further heating to > 1600 °C, Al_2O_3 and AlN are retransformed back to AlON. After the phase transformation from Al_2O_3 to AlON is fully completed, the sintering mixture still has a bimodal grain size distribution. As a result, a much faster (less holding) and better (less and smaller defects) densification at the final stage of sintering can be obtained, and the holding time is shortened to 1.5–2.5 h [12,13].

In this study, the phase transformation and microstructure evolution of bimodal AlON powder, being held for 0, 30 and 60 min at 1500, 1600 and 1700 °C respectively during heating, were monitored to reveal the effects of holding time or heating rate on phase transformation and microstructure evolution of the sintering mixtures. Then three samples, i.e. no holding during heating, holding 30 min at 1500 °C and holding 30 min at 1600 °C, were further heated to 1860 °C and held for 90 min before cooling to detect their phase assemblages, observe their microstructures and measure their transmittances. The results revealed that more and larger pores were formed in the samples with the added holdings (30 min) during heating, which leads to a decrease in both relative density and transmittance of the AlON samples, compared to the samples without holding (0 min) during heating. Therefore, it is important to keep a high heating rate during heating to maintain the near sphere bimodal PSD of the sintered mixtures before the final stage of sintering starts, which benefits to grain growth and better densification.

2. Experiment procedure

2.1. Preparation of bimodal PSD AlON powder

AlON powder was firstly synthesized by carbothermal reduction and nitridation (CRN) method, then 0.5 wt% Y_2O_3 (Grade C, Starck, Germany) was added into the obtained AlON powder. Using Si_3N_4 balls (having 5, 8 and 10 mm diameter, weight ratio at 1.7:1:1.3) as milling media, keeping the ball-to-powder weight ratio at 7:1, 30g of mixture powders of AlON and Y_2O_3 were ground in absolute ethyl alcohol (weight ratio: absolute ethyl alcohol/powder = 3.93) for 24 h at 170 rpm, then the bimodal AlON powder (~1.1 μm and ~2.2 μm) was obtained [13]. The morphology of the milled powder was observed by scanning electron microscope (SEM; supra 55, Zeiss, Germany) as shown in Fig. 1. Detailed fabrication process of the bimodal AlON powders was given in Ref. [12] and Ref. [13].

2.2. Sintering of AlON powder

1.6 g of the obtained bimodal AlON powder was packed into a pellet

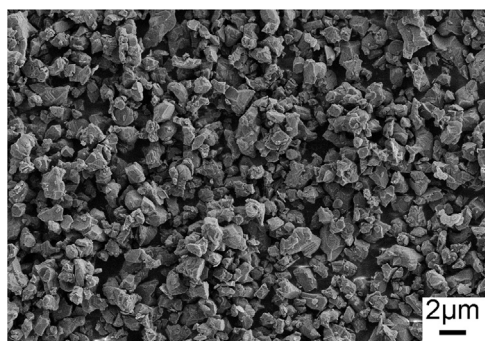


Fig. 1. SEM image of the bimodal AlON powder.

of 13 mm in diameter under 50 MPa. The pellet was then pressureless sintered within a graphite furnace in an atmosphere of 0.1 MPa N_2 . Two identical samples were put into graphite furnace each time to be heated at a heating rate of 40 °C/min to 1500 °C, 1600 °C and 1700 °C, and held at each temperature for 0 min, 30 min and 60 min, respectively. At the same time, samples directly heated to 1800 °C also were prepared. Then the furnace was cooled down to room temperature to take out the sintered samples for further testing and characteristic. In addition, the following three typical samples, i.e. no holding during heating, holding 30 min at 1500 °C and holding 30 min at 1600 °C, were directly heated to 1860 °C and held for 90 min before cooling.

2.3. Characterization of sintered samples

The phase assemblages of the sintered samples were characterized by X-ray diffractometry (XRD; D/Max-ULTIMA+, Rigaku, Japan) using Si as an internal standard. Scanning electron microscope was used to observe the microstructure of sintered samples. The bulk densities of all the samples sintered at different conditions were measured by Archimedes principle. The sample sintered at 1860 °C with holding 90 min was ground and polished on both sides to a thickness of 3 mm for transmittance measurement. Transmittance within the wave length of 2500–6000 nm was recorded by a Fourier transform infrared spectroscopy (FTIR; Nexus 670, Thermo Nicolet, USA).

3. Results and discussion

3.1. Phase transformation

Fig. 2 shows the XRD patterns of the samples pressurelessly heated to 1500 °C, 1600 °C and 1700 °C and held for 0 min, 30 min and 60 min, respectively. From Fig. 2a, it can be seen that no AlON can be detected right after temperature was raised to 1500 °C (0 min). Instead, $\eta\text{-Al}_2\text{O}_3$ and $\alpha\text{-Al}_2\text{O}_3$ are the major phases of the sample, which is dominated by $\eta\text{-Al}_2\text{O}_3$. Meanwhile, a little AlN can also be detected. It confirms that all the AlON particles at this stage were transformed into Al_2O_3 and AlN, where fine AlON particles were transformed into hexagonal $\alpha\text{-Al}_2\text{O}_3$ and AlN, while coarse AlON particles were transformed into cubic $\eta\text{-Al}_2\text{O}_3$ and AlN [13]. However, further holding at 1500 °C (both 30 min and 60 min), a large amount of AlON appears and no $\eta\text{-Al}_2\text{O}_3$ can be found in the samples. Correspondingly, the content of $\alpha\text{-Al}_2\text{O}_3$ and AlN increases slightly. It indicates that all $\eta\text{-Al}_2\text{O}_3$ can be retransformed into AlON during holding at 1500 °C. At the beginning of holding at 1500 °C (0–30 min), the increase in the content of $\alpha\text{-Al}_2\text{O}_3$ and AlN could be either attributed to transformation from $\eta\text{-Al}_2\text{O}_3$ to $\alpha\text{-Al}_2\text{O}_3$ (observed in Ref [13]) or retransformation from the newly formed small AlON particles. Further holding at this temperature (from 30 min to 60 min), it is evident that part of newly formed small AlON particles were transformed into $\alpha\text{-Al}_2\text{O}_3$, which resulted in a marginal increase in the content of $\alpha\text{-Al}_2\text{O}_3$ and AlON.

For the sample directly heated to 1600 °C (Fig. 2b), $\eta\text{-Al}_2\text{O}_3$, $\alpha\text{-Al}_2\text{O}_3$ and AlN coexisted in the sintered mixture (Fig. 2b, 0 min). After holding 30–60 min at 1600 °C (Fig. 2b, 30 min and 60 min), all $\eta\text{-Al}_2\text{O}_3$ were again transformed back to AlON. It should be noticed that the content of $\alpha\text{-Al}_2\text{O}_3$ decreases evidently during holding, which indicates that $\alpha\text{-Al}_2\text{O}_3$ starts to retransform back to AlON at 1600 °C after holding.

From Fig. 2c, it can be seen that all $\eta\text{-Al}_2\text{O}_3$ have been retransformed back to AlON once the temperature was directly heated up to 1700 °C at 40 °C/min, i.e., it only took 5 min for $\eta\text{-Al}_2\text{O}_3$ to be fully retransformed back to AlON from 1500 °C to 1700 °C. As a result, AlON, $\alpha\text{-Al}_2\text{O}_3$ and AlN coexisted in the mixture at this stage. Although it is not clear how long it may take to fully retransform from $\eta\text{-Al}_2\text{O}_3$ back to AlON at 1600 °C (< 30 min according to Fig. 2b), it is reasonable to suggest that a rise in temperature leads to a faster

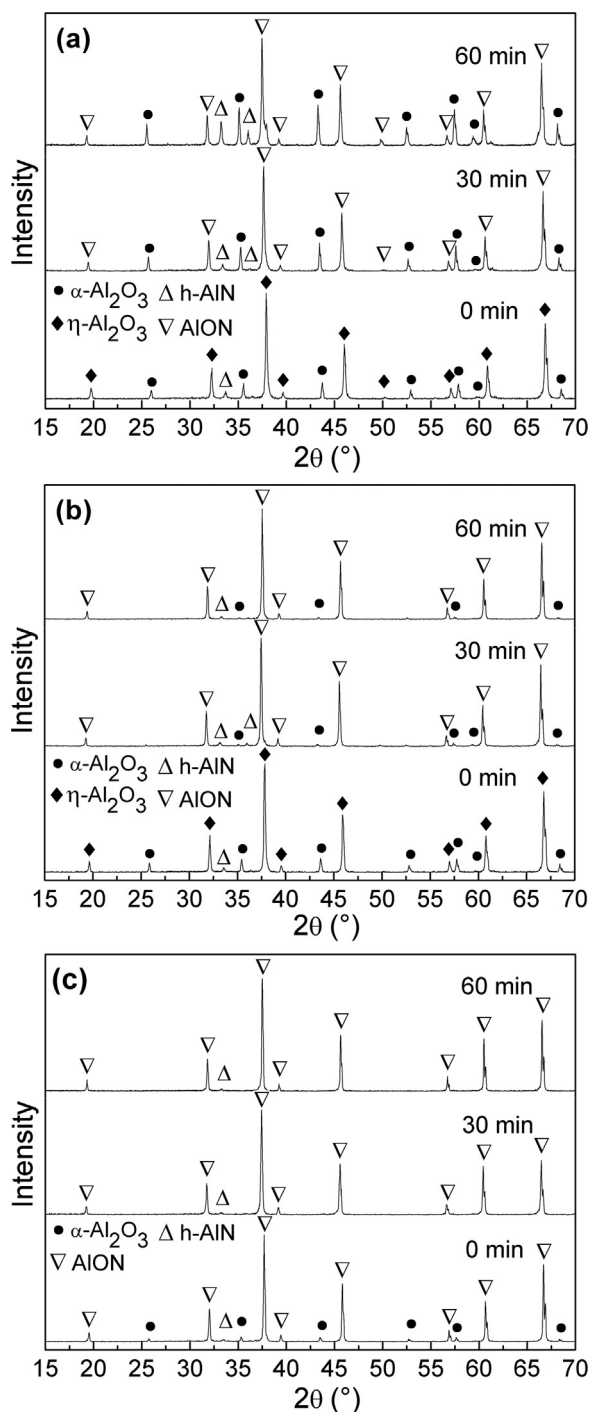


Fig. 2. XRD patterns of the samples sintered at 1500 °C (a), 1600 °C (b) and 1700 °C (c), respectively.

retransformation from η - Al_2O_3 back to AlON. After holding 30 min and 60 min at 1700 °C, all α - Al_2O_3 were retransformed back to AlON. It should be noted that α - Al_2O_3 were not fully retransformed back to AlON at 1600 °C even after 60 min holding, which was completed in 30 min at 1700 °C. Therefore, full phase transformation from η - Al_2O_3 back to AlON and from α - Al_2O_3 back to AlON can be rapidly accomplished at a temperature higher than their own phase transformation temperatures.

All the analysis above indicates that both sintering temperature and holding time contribute to the phase transformations among AlON, η - Al_2O_3 , α - Al_2O_3 and AlN during heating at 1500–1700 °C. Holding at 1500–1600 °C within 30 min or directly heating to 1700 °C all benefit

to the phase transformation from η - Al_2O_3 to AlON, and holding at 1500 °C also results in the transformation from small AlON to α - Al_2O_3 , but α - Al_2O_3 can be retransformed back to AlON at 1600–1700 °C by holding. It implies that the phase assemblages of the AlON samples can be deliberately controlled at 1500–1700 °C via heating rate and holding time.

3.2. Microstructure evolution

To better understand microstructure evolution of the samples held for different duration at different phase transformation temperatures, the SEM images and the relative densities of the sintered samples after pressureless sintering at 1500 °C, 1600 °C and 1700 °C held for 0 min, 30 min and 60 min are presented in Fig. 3 and Fig. 4, respectively. It is observed that the grain morphology, grain size, grain size distribution, pore size and relative density of sintered samples are strongly related to holding time and sintering temperature at 1500–1700 °C.

At 1500 °C, grain morphology of samples held for 0 min and 30 min develops towards sphere compared to the starting AlON powder (Fig. 1 vs. Fig. 3a and b), and the microstructure of the two samples demonstrates a bimodal PSD, where large particles are either η - Al_2O_3 (retransformed into AlON after 30 min holding) or AlON and small particles mainly are α - Al_2O_3 . The inherited bimodal PSD and very limited grain growth indicate that surface diffusion should be the main mass transport mechanism during holding within 30 min at 1500 °C [20,21]. It should be pointed out that small particles in this duration mainly are α - Al_2O_3 , which confines mass transport between large and small particles in the mixture due to their dissimilarity in grain structure. As a result, most mass transport takes place individually, i.e., large to large and small to small particles. Consequently, the excellent gap filling and grain rearrangement abilities of bimodal PSD are weakened. Instead, the separated surface diffusion paths lead to many enlarged voids and large pores within the sintered mixture shown in Fig. 3b. On the other hand, a little grain growth of large particles and limited grain growth of small particles can also be observed in the sample held 30 min compared to the one held 0 min (Fig. 3a vs. b). The grain growth of large particles should be mainly attributed to retransformation from η - Al_2O_3 into AlON and faster grain growth ability of AlON (Fig. 2a) [13]. Fig. 4 shows that only limited increase in relative density of the mixtures (from 52.42% to 57.70%) is measured during first 30 min holding at 1500 °C, which implies that the voids in the bimodal PSD mixtures didn't shrink effectively via surface diffusion. Further holding the sample up to 60 min at 1500 °C, most grains present a sharp edge and the grains are getting closer (Fig. 3b vs. c). It indicates that both surface diffusion and grain boundary diffusion should have happened during holding from 30 min to 60 min at 1500 °C [20–22]. It is evident that grain boundary diffusion only happens in part of grains in Fig. 3c. Nevertheless, many large pores can still be observed. The measured relative density for the sample held 60 min at 1500 °C indicates a faster densification progress occurred during holding from 30 min to 60 min (Fig. 4), which can be mainly attributed to surface diffusion and limited grain boundary diffusion. Additionally, the increase in α - Al_2O_3 content in samples should also contribute to the improvement of relative density during holding at 1500 °C.

Heating green body up to 1600 °C, then held for 0 min, 30 min and 60 min, respectively. The sample held 0 min at 1600 °C still keeps near sphere morphology, limited grain growth and bimodal grain size distribution compared to the sample held 0 min at 1500 °C (Fig. 3d vs. a). It means that surface diffusion still be the main mass transport mechanism during heating from 1500 °C to 1600 °C. However, grains characterized with sharp edge are observed in sample held for 30 min and 60 min, which should be caused by both surface diffusion and a little grain boundary diffusion (Fig. 3e and f). As shown in Fig. 2b, α - Al_2O_3 can be detected in the mixtures held both 30 min and 60 min at 1600 °C, which is less than that in the sample held 60 min at 1500 °C.

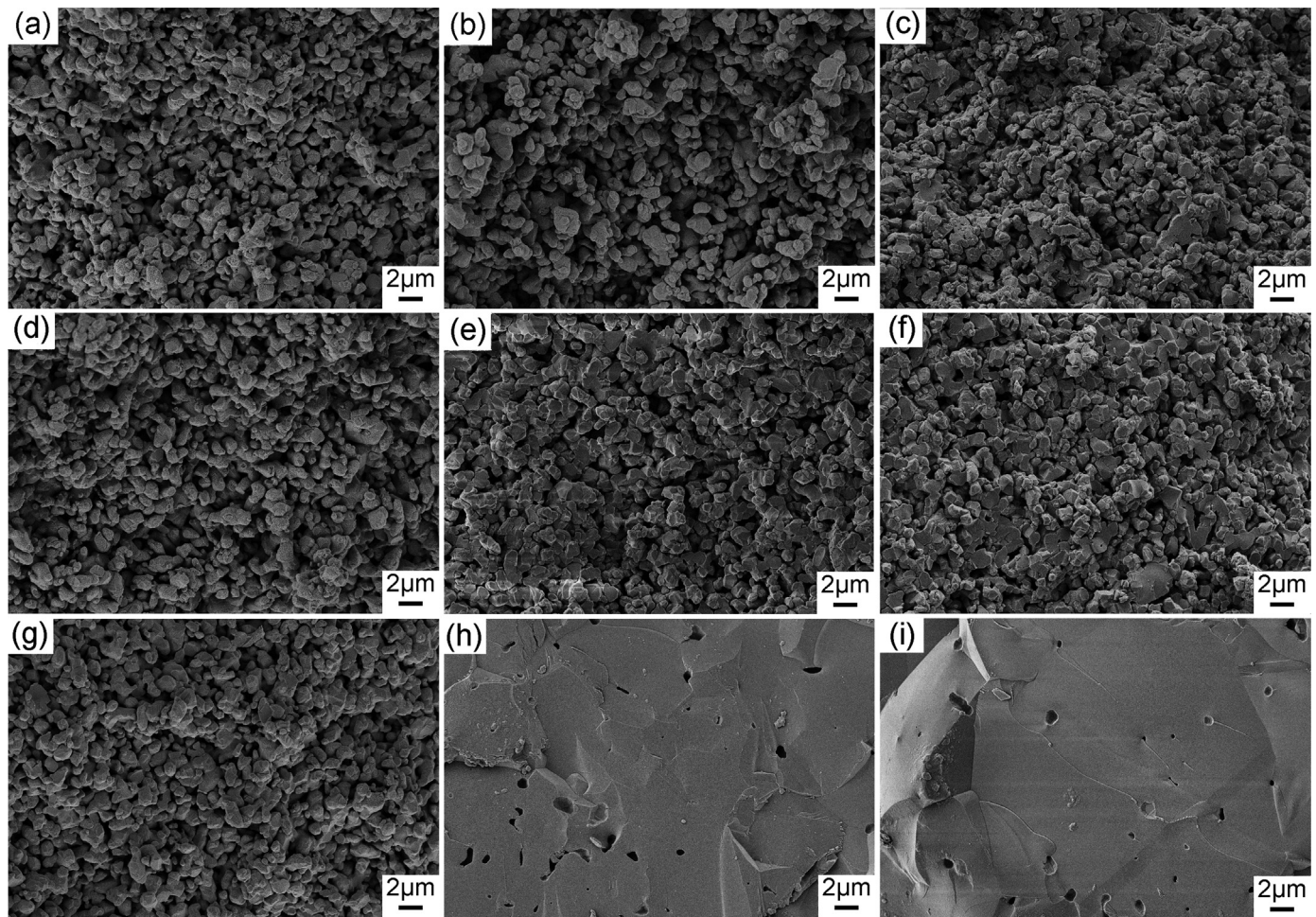


Fig. 3. SEM images of the fracture surfaces of the samples heated to 1500 °C holding for 0 min (a), 30 min (b) and 60 min (c), 1600 °C holding for 0 min (d), 30 min (e) and 60 min (f), and 1700 °C holding for 0 min (g), 30 min (h) and 60 min (i), respectively.

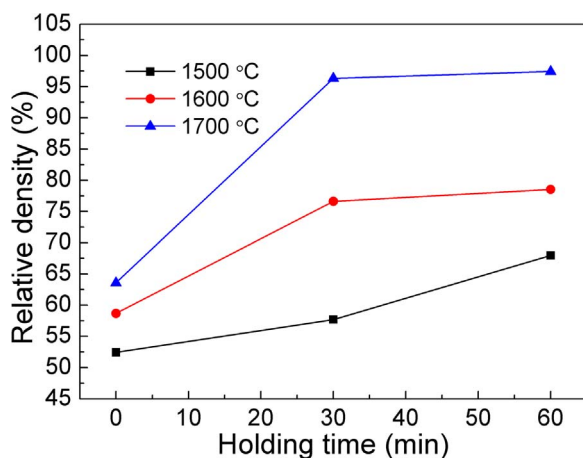


Fig. 4. Effects of holding time on relative density of the samples sintered at 1500, 1600 and 1700 °C, respectively.

This indicates that there are more ALON grains in the mixtures sintered at 1600 °C. Apparently, mass transport between ALON grains is easier compared to other adjacent particle pairs. Therefore, mass transport at 1600 °C is faster than that at 1500 °C. As a result, grains with sharp edge can be observed in the sample held 30 min at 1600 °C and 18% improvement in relative density (58.68% (0 min) vs. 76.62% (30 min)) within the first 30 min holding at 1600 °C. However, the residual α - Al_2O_3 intercepted mass transport in the sample, which results in large

amount of pores in the mixtures (Fig. 3e and f). Consequently, only a marginal increase in relative density (~2%) is measured when holding time extended to 60 min from 30 min at 1600 °C (Fig. 4). As a matter of fact, the particle size distribution shown in Fig. 3e and f is more similar to a unimodal pattern. It indicates that the microstructure characterized by sharp edge, uniform grain and a large amount of pores (shown in Fig. 3e) is difficult to be densified even though nearly all α - Al_2O_3 has been retransformed back to ALON due to the lower rearrangement and grain growth ability.

Further heating the sintering mixture to 1700 °C, the microstructure of the sample held for 0 min (Fig. 3g) is still similar to that of samples held 0 min at 1500 °C and 1600 °C. However, as shown in Fig. 3h and Fig. 4, significant grain growth and fast densification happens during the first 30 min holding at 1700 °C. The curve of relative density vs. holding time shown in Fig. 4 confirms that relative density of 96.33% is achieved for the sample held for 30 min, which is much higher than that of 63.57% of sample held 0 min. The fast grain growth and quick densification should be attributed to great rearrangement ability of the near sphere particles in bimodal PSD (Fig. 3g) and fast grain boundary migration of ALON because no α - Al_2O_3 can be detected in sample held 30 min (Fig. 2c). However, there are a lot of pores can be observed in Fig. 3h and Fig. 5a (SEM image of sample held 30 min at 1700 °C). On contrast, much less pores can be identified from Fig. 5b (SEM image of sample directly heated to 1800 °C at 40 °C/min). Further holding to 60 min, only limited relative density increasing (~1%) is measured with a lot of pores remained in the sample (Fig. 3i). It indicates the pores left in the sample are difficult to be eliminated during holding, which remarkably affects the transparency

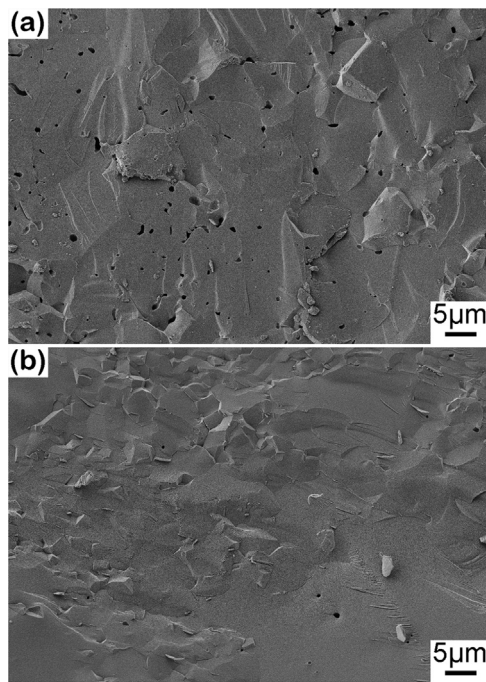


Fig. 5. SEM images of the samples held 30 min at 1700 °C (a) and held 0 min at 1800 °C (b).

and mechanical properties of the sintered AlON ceramics.

Based on the above phase assemblage and microstructure analysis of the bimodal AlON samples, three typical samples of the first ramping up period were selected to fabricate transparent AlON ceramics, which are:

- 1) Holding for 30 min at 1500 °C, closed to sphere grain and bimodal grain size distribution with large voids and pores, identified as A15;
- 2) Holding for 30 min at 1600 °C, sharp edge grain and more unimodal grain size distribution with large pores, identified as B16;
- 3) No holding during heating, closed to sphere grain and bimodal grain size distribution in the whole heating process (1500–1700 °C), identified as C.

Before cooling, these three samples were further heated to 1860 °C and held for 90 min to fabricate transparent AlON ceramics.

3.3. Properties of transparent AlON ceramics

The phase assemblage analysis of three fabricated transparent AlON ceramics samples is shown in Fig. 6, which reveals that single phase AlON ceramics have been obtained after sintering at 1860 °C for 90 min due to only AlON crystalline was detected in all the fabricated samples. It also indicates that all the α -Al₂O₃ formed during heating have been retransformed back to AlON after holding 90 min at 1860 °C.

Fig. 7 depicts the SEM images of all samples sintered at 1860 °C. Pores can be obviously observed in both samples A15 and B16 with more pores being identified in sample A15 than sample B16 and no apparent pores can be detected in sample C. Further relative density measurement results shown in Fig. 8 demonstrates that sample A15 has the lowest relative density of 98.75%, and a slightly higher relative density of 99.00% is measured for sample B16, sample C has the highest relative density. At the end of the first ramping up period of the three samples, sample C has the lowest relative density, compared to that of sample A15 and B16 (Fig. 4). However, only much less and smaller pores were formed in sample C when all the particles were retransformed back to AlON and AlON grains inherited a bimodal PSD

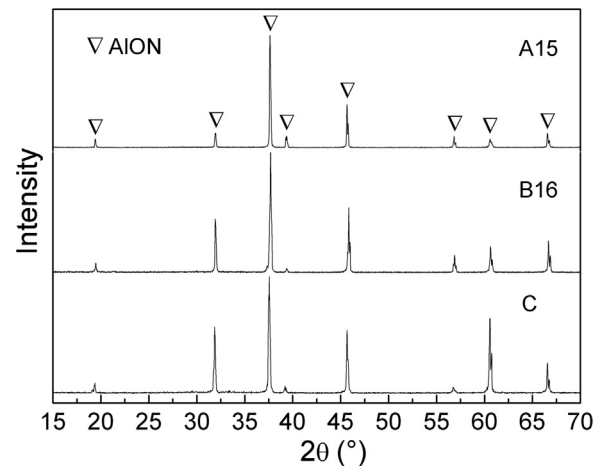


Fig. 6. XRD patterns of the samples held 90 min at 1860 °C: A15 vs. 1500 °C (30 min)+1860 °C (90 min); B16 vs. 1600 °C (30 min)+1860 °C (90 min); C vs. 1860 °C (90 min).

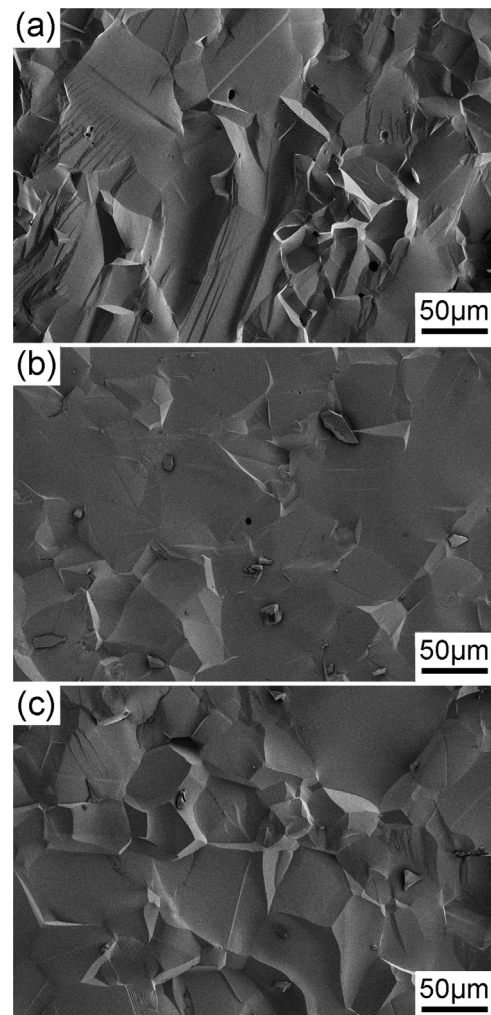


Fig. 7. SEM images of the fracture surfaces of the samples held 90 min at 1860 °C: (a) A15 vs. 1500 °C (30 min)+1860 °C (90 min); B16 vs. 1600 °C (30 min)+1860 °C (90 min); (c) C vs. 1860 °C (90 min).

(Fig. 5b and Ref. [13]), which further benefits its fast and better densification. For other two samples, a lot of large pores are formed at the end of the first ramping up period because of the interception of residual α -Al₂O₃. Apparently, these large pores are difficult to be eliminated in the final stage of densification. It indicates that both

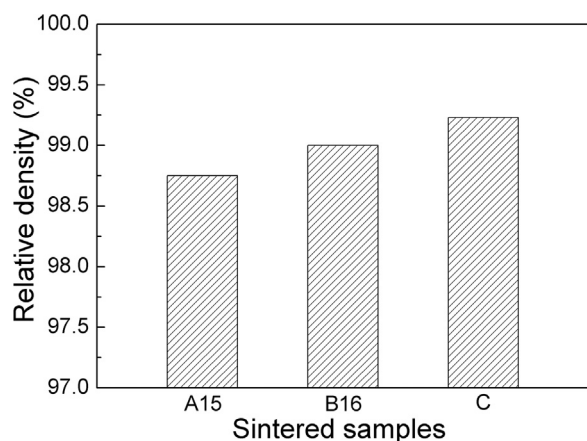


Fig. 8. The relative density of samples held 90 min at 1860 °C.

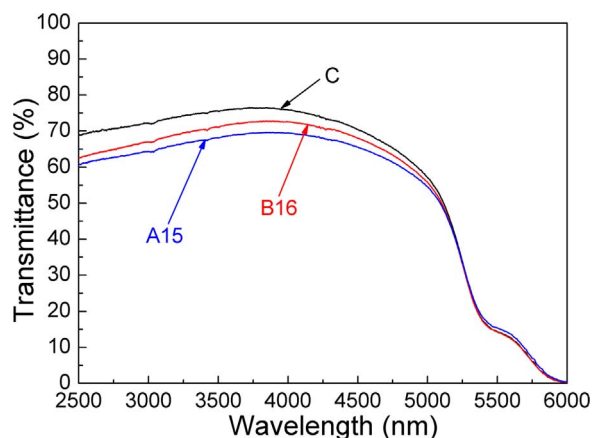


Fig. 9. Transmittance of the AlON samples sintered at 1860 °C for 90 min.

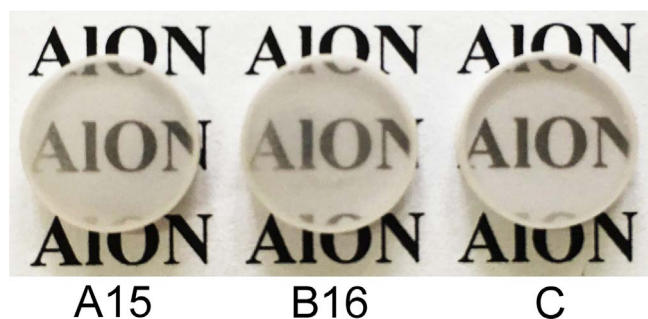


Fig. 10. Optical images of the transparent AlON samples sintered at 1860 °C for 90 min.

phase assemblage and microstructure after the first step ramping significantly affect the followed densification process and microstructure evolution of AlON ceramics.

Further transmittance measure shown in Fig. 9 reveals that sample C has the highest transmittance of 76.49%, which is higher ~4% to 7% than that of samples A15 and B16. The pictures of three samples are presented in Fig. 10.

Above measurements confirms that fast heating is favorable for a better densification of the bimodal AlON sample by keeping the particles in near sphere shape and bimodal PSD during Al_2O_3 is retransformed back to AlON. Once the phase transformation from Al_2O_3 to AlON is fully completed, the fast grain growth of AlON will happen. Without the interception of small residual $\alpha\text{-Al}_2\text{O}_3$ particles, mass transport between large to large, large to small and small to small AlON grains can simultaneously happen, which means the advantage of bimodal AlON grain size distribution can be fully taken. Consequently,

much less and smaller pores would be formed during this fast and massive mass diffusion process, which are much easier to be eliminated during holding, i.e., higher relative density and less defects can be achieved. Therefore, based on the bimodal grain size distribution benefited from fast heating, bimodal grain size distribution is kept in the whole sintering process of the bimodal samples [12,13], which leads to fast grain growth and fast densification of the sample.

4. Conclusions

- 1) Two opposite phase transformations between AlON and Al_2O_3 can be identified during heating, and a higher temperature can accelerate the phase transformation process from Al_2O_3 to AlON.
- 2) During holding at 1500 °C and 1600 °C, mass transport mainly happens between large AlON grains, the residual small Al_2O_3 particles retard mass transport between large and small particles in the sintering mixtures. As a result, a great number of large pores are formed within the mixtures, which are difficult to be eliminated at the final stage of sintering.
- 3) To achieve a faster and higher densification of the bimodal AlON powder, fast heating is favorable because it is helpful to keep near sphere bimodal grain size distribution during heating. Then mass transport between large to large, large to small and small to small AlON grains can simultaneously happen during fast densification (after all Al_2O_3 is transformed back to AlON) without the interception of small residual $\alpha\text{-Al}_2\text{O}_3$ particles. As a result, much less and smaller pores would be formed, which are much easier to be eliminated during holding at the final stage of sintering.

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