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Sintering of α-Al_2O_3-seeded nanocrystalline γ-Al_2O_3 powders

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Abstract

This paper demonstrates that seeding nanocrystalline transition alumina powders is a viable option for producing high quality, alumina-based ceramics. By using α-Al_2O_3 concentrations of $\geqslant$1.25 wt.% α-Al_2O_3 seed particles (equivalent to 5×10^{14} seeds/cm^3 of γ-Al_2O_3) the sintering temperature is reduced from 1600°C for unseeded γ-Al_2O_3 to 1300–1400°C in dry pressed powders. The scale of the sintered microstructure is related to $N_v^{-1/3}$ and thus a 100-nm grain size is obtained. It is apparent that seeding is necessary for producing dense, alumina-based ceramics from nanocrystalline transition alumina powders. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Al_2O_3; Microstructure-final; Seeding; Sintering

1. Introduction

Alpha alumina (α-Al_2O_3) is utilized in many areas of modern industry because of its unique mechanical, electrical, and optical properties. Since the properties exhibited by a ceramic are governed by its microstructure, refining α-Al_2O_3 processing techniques to obtain superior microstructures is of great interest. Recently, transition alumina has attracted much attention because of its intrinsically nanocrystalline nature and because it can be synthesized by a variety of techniques. The most common transition alumina is γ-Al_2O_3 which forms from virtually all alumina precursors, except diaspore.

Upon heating, γ-Al_2O_3 undergoes a series of polymorphic transformations: [1]

$$\gamma\text{-}Al_2O_3 \Rightarrow \delta- \Rightarrow \theta- \Rightarrow \alpha\text{-}Al_2O_3$$

The α-Al_2O_3 phase transformation occurs by nucleation and growth.[2] During the reconstructive transformation from theta to alpha alumina there is a specific volume reduction (28.6–25.6 cm^3/mol Al_2O_3), due to the difference in theoretical density (3.6–3.986 g/cm^3 Al_2O_3).[1,4] A low intrinsic nucleation density results in large spacing between nucleation events and the formation of micrometer scale, single crystal α-Al_2O_3 grains with dendritic protrusions surrounded by continuous pore channels. The resultant vermicular microstructure requires sintering temperatures >1600°C to reach high densities.[2] To obtain dense, fine-grained α-Al_2O_3 at low temperatures, the scale of the vermicular microstructure must be minimized.

A number of papers report the effect of a small amount of ceramic oxide seed particles on microstructure evolution during the θ to α-Al_2O_3 phase transformation.[3–21] In unseeded boehmite, the intrinsic nucleation density is 10^8–10^{11} nuclei/cm^3 θ-Al_2O_3 and ≈1200°C is required to fully transform to alpha alumina.[2–7] The addition of seeds, which are isostructural with α-Al_2O_3, provides low energy sites for heterogeneous nucleation and thus reduces the energy barrier required for nucleation.[10,11] The seeds have a number of contacts with θ-Al_2O_3 and thus, provide multiple nucleation sites.[12] In conjunction with the lower energy barrier and the larger number of nucleation sites, the phase transformation takes place at lower temperatures. For 5 wt.% seeded γ-Al_2O_3 samples, the transformation temperature and activation energy were decreased by 75°C and 170 kJ/mol, respectively, compared to unseeded γ-Al_2O_3.[3]

Kumagai and Messing reported that a seed concentration of 5×10^{13} seeds/cm^3 γ-Al_2O_3 is optimal for controlling the microstructure evolution during the θ to α-Al_2O_3 phase transformation.[11] By using fine particles

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(<100 nm), a high seed concentration (10^{14-15} seeds/cm^3 γ-Al_2O_3) can be achieved at small weight percentage additions (<5 wt.% α-Al_2O_3).[11,13–15] The high seed concentration reduces the distance between nucleation sites causing early impingement of growing α-Al_2O_3 colonies. As a result of the earlier impingement, the size of the dendritic protrusions is limited and a homogeneous, fine-grained microstructure is obtained during sintering. A number of other researchers have attempted to improve these results by using other starting materials or by varying the seed concentration .[4–8,13,16–19]

Prasad et al. investigated the densification behavior of pressed γ-Al_2O_3 powder.[20] After sintering for 5 h at 1600°C, the initially 43% dense samples were only 73.5% dense. Wu et al. used high-pressure cold isostatic pressing to achieve a green density of 63% in an effort to improve the sinterability of γ-Al_2O_3 powder.[8] Samples sintered to 72% density at 1150°C and 82% at 1350°C. The vermicular microstructure still developed during the phase transformation and inhibited complete densification.

To circumvent the formation of dendritic shaped grains as a means for obtaining dense α-Al_2O_3 at lower temperatures, Wu et al. heated nanocrystalline γ-Al_2O_3 powders above the θ to α-Al_2O_3 phase transformation temperature prior to compaction.[8] The aim of the experiments was to investigate whether nanocrystalline α-Al_2O_3 powder obtained from γ-Al_2O_3 would sinter better than commercially available α-Al_2O_3 powder. The transformed α-Al_2O_3 powders sintered to 95% density and a grain size of ·1 μm at 1350°C. Freim et al. used rapid heating with a microwave furnace to alter the transformation, but no significant benefit was reported.[21]

Tsai et al. investigated the effects of ceramic oxide dopants on the phase transformation and densification of colloidal gels made from high purity, fumed γ-Al_2O_3.[17] Undoped γ-Al_2O_3 samples transformed to α-Al_2O_3 at 1260°C and sintered to 53% density at 1400°C and 75% at 1600°C. Densities for samples sintered at 1600°C for 4 h increased when doped with 2 wt.% TiO_2 (98%), MgO (82%), and ZrO_2 (80%) but there was no significant effect on the phase transformation. Many sesquioxide dopants (e.g. α-Fe_2O_3), that are isostructural with α-Al_2O_3, have been shown to nucleate the phase transformation.[22,23]

Unseeded bohemite (γ-AlOOH) transformed to 70% dense α-Al_2O_3 after 100 min at 1200°C, and reached 97% density after 100 min at 1600°C.[11] Using commercial boehmite Kumagai and Messing showed that seeding with 1.5 wt.% α-Al_2O_3 resulted in 99% density after sintering for 100 min at 1200°C.[10] In another investigation, they reported that they could achieve 95% density after 6 h at 1140°C by seeding a high purity, alkoxide-derived boehmite.[14]

From the previous investigations, it is evident that the number concentration of α-Al_2O_3 seeds, the homogeneity of the seed distribution in the transition alumina matrix and the purity of the starting materials have significant effects on the transformation kinetics. The use of commercial powders, which are inherently agglomerated, limits the attainable homogeneity of the seed distribution, but, avoids the costly and time intensive preparation of high purity sols from alkoxide precursors. The objectives of this investigation, therefore, were to (1) investigate the effect of α-Al_2O_3 seed addition on the microstructure evolution, and densification behavior of high purity, commercial γ-Al_2O_3, and (2) determine the effects of agglomeration and green density on the effectiveness of α-Al_2O_3 seeding. The γ-Al_2O_3 powder selected for this investigation is the same as that used by Tsai and Hsieh.[17] The phase transformation kinetics of the seeded powder described in this paper was reported earlier.[3]

2. Experimental procedure

A high purity, nanocrystalline fumed γ-Al_2O_3 powder[2] produced by flame hydrolysis of aluminum chloride, was selected for this investigation. A 2.5 wt.% dispersion was prepared by adding deionized water and adjusting the pH to 3.0 with 2 M nitric acid. To reduce the agglomerate size, the dispersion was stirred and periodically sonicated for three days. The dispersed agglomerate size, determined by dynamic light scattering[3] was ·86 nm.

Commercial α-Al_2O_3 powder was used to obtain fine seed particles.[4] A 10 wt.% dispersion was prepared by adding α-Al_2O_3 powder to deionized water and adjusting the pH to 3.0 with 2 M nitric acid. The dispersion was stirred for three days and periodically sonicated to break up agglomerates. The seeds were obtained by removing agglomerates by centrifugation for 45 min at 2000 rpm (0.2 m radius). The seed dispersion (i.e. the supernatant after centrifuging) was added to the γ-Al_2O_3 dispersion while stirring to obtain seed concentrations of 0, 1.25, 2.5 and 5 wt.% α-Al_2O_3, on a γ-Al_2O_3 weight basis. The seeded sols were rapidly dried in a rotary evaporator to prevent mass segregation of the α-Al_2O_3 seeds during drying.

After drying, the powders were ground into agglomerates <44 μm (−325 mesh) with a high purity alumina mortar and pestle. The powder was uniaxially pressed at 280 MPa in a zinc stearate lubricated hardened steel die. The pellets were then cold isostatically pressed [5] in latex bags at 280 MPa for 30 min. The pellet dimensions were

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[2] Aerosil® Aluminum Oxide C, Degussa Corp., Ridgefield Park, NJ.

[3] Microtrac UPA, Leeds & Northrup, St. Petersburg, FL.

[4] Taimei DAR, Taimei Chemical Industrial Co., Nagano, Japan.

[5] Cold Isostatic Press, Autoclave Engineers, Erie, PA.

6.2 mm diameter and ≈3 mm thick. The green densities of the pressed pellets, determined by the dimensional technique, were ·54%, based on the γ-Al_2O_3 theoretical density of 3.2 g/cm^3.[1]

For comparison, a commercial alumina powder containing 35% δ-Al_2O_3 and 65% α-Al_2O_3[6] was also sintered. A dispersion was prepared by adding 10 wt.% powder to deionized water and adjusting the pH to 3.0 with 2 M nitric acid. The dispersion was stirred for three days and periodically sonicated to break up agglomerates. The dispersed agglomerate size, determined by dynamic light scattering[3], was ·0.3 ≈μm. The powders were then rapidly dried in a rotary evaporator and lightly ground into agglomerates <45 μm with a high purity alumina mortar and pestle. Pellets of 6.2 mm diameter were prepared by die pressing at 280 MPa and then cold isostatically pressed at 280 MPa for 30 min. The green densities of the pressed pellets were estimated to be 59%, based on a calculated theoretical density of 3.67 g/cm^3.

Samples were supported on a Pt foil and sintered in air in a box furnace.[7] The furnace was heated at 5°C/min to 850°C and then at 15°C/min to the sintering temperature of 1000–1600°C and held for 30 min. The furnace was cooled at 15°C/min to 400°C and then turned off. To examine morphological development during the θ to α-Al_2O_3 phase transformation, samples were polished and thermally etched for 30 min at 50°C lower than the sintering temperature for samples sintered at ⩽1200°C and 100°C lower than the sintering temperature for samples sintered at ⩾1400°C. The samples were sputter coated with gold to prevent charging during SEM examination. Grain size was determined from micrographs using the mean linear intercept method and multiplying by 1.56.

3. Results and discussion

The particle size distribution of the seed particles, as determined by centrifugal sedimentation,[8] is shown in Table 1. There is some agglomeration of the seeds in the 100 to 200 nm size range, however, a significant portion of the seeds is <60 nm. For the sake of calculation, particles <50 nm are assumed to have an average particle size of 25 nm. The size distribution was confirmed by inspection of the seeds using transmission electron microscopy (TEM).[9] Crystallite size was determined by X-ray line broadening and calculated using the Scherrer equation:

Table 1
Particle size distribution of α-Al_2O_3 seeds

Particle diameter (nm)	Volume frequency (%)	Number frequency (%)
300–400	0.1	0.0001
200–300	4.2	0.01
100–200	29.4	0.34
90–100	0.6	0.03
80–90	3.1	0.19
70–80	3.2	0.29
60–70	5.3	0.74
50–60	15.6	3.61
<50	38.5	94.8

$$d = \frac{0.9\lambda}{B\cos\theta}$$

$$\text{where } B = \left(B_o^2 - B_c^2\right)^{1/2} \tag{1}$$

where B_o is the full width at half maximum (in °2θ), B_c is the correction factor for instrument broadening, θ is the peak maximum (in °2θ), and λ is the CuK_α weighted average wavelength. The γ-Al_2O_3 and α-Al_2O_3 crystallite sizes were 6.5 and 37 nm, respectively. The TEM and X-ray analysis results are consistent with the particle size distributions given in Table 1.

The seed frequency, f_s, was calculated from the seed particle size distribution and the seed concentration as follows:

$$f_s = \frac{N_s}{V_{tot}} = \frac{6m_\alpha}{\pi\rho_\alpha V_{tot}}\sum_{i=1}^{n}\frac{wf_i}{d_i^3} \tag{2}$$

where N_s is the number of seeds added, V_{tot} is the total volume of the seed powder, m_α is the weight of seeds added, ρ_α is the theoretical density, and wf_i is the weight fraction of seeds with diameter, d_i. The seed number densities were calculated from the data in Table 1. At a seed concentration of 1.25 wt.% the seed frequency is 5×10^{14} seeds/cm^3 γ-Al_2O_3 and the frequency increases to 2×10^{15} seeds/cm^3 γ-Al_2O_3 at a seed concentration of 5 wt.%.

The surface area of the transition alumina powder is 115 m^2/g. The transition alumina powder is a mixture of gamma and delta alumina. The major impurities are 320 ppm Fe, 170 ppm Si and 390 ppm Ti. The chlorine content was not measured but was listed by the manufacturer as <0.5%. Most studies on seeded boehmite have used gels stabilized at pH 3 and no effects of alumina solubility on the α-Al_2O_3 transformation have been observed.

All samples transformed to >95% α-Al_2O_3 when sintered at 1100°C for 100 min as seen in Fig. 1. The unseeded sample exhibits dendrite grain shapes (0.2 μm) and continuous pore channels; indicative of a vermi-

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[6] Baikalox CR-30, Baikowski International Co., Charlotte, NC.

[7] High Temperature Furnace 46100, Barnstead/Thermolyne, Dubuque, IA.

[8] Model CAPA-700, Horiba Instruments, Ltd., Irvine, CA.

[9] Model EM 420T, Philips, Netherlands.

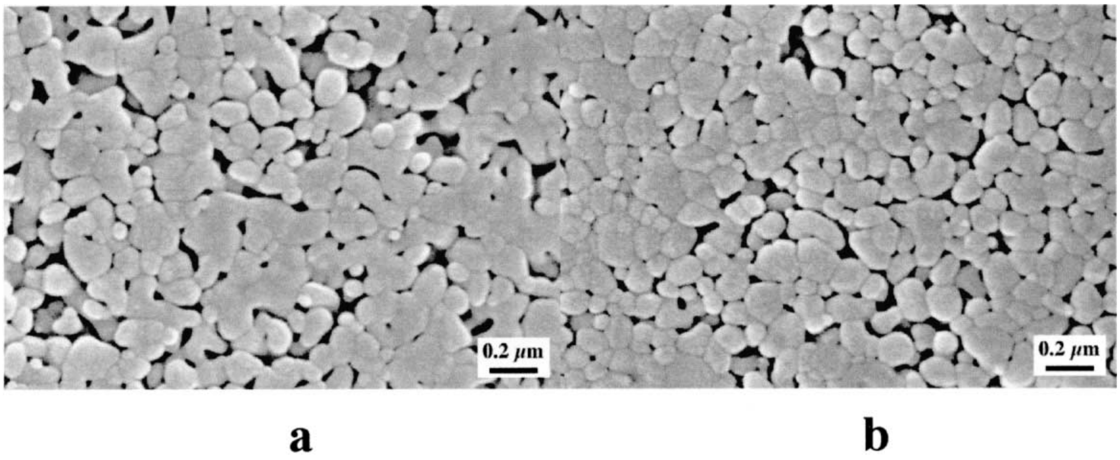

Fig. 1. Microstructures of (a) 0 wt.% and (b) 5 wt.% seeded γ-Al_2O_3 samples sintered for 100 min at 1100°C.

cular microstructure. As seed concentration increases, the size of the dendrites decreases and the porosity becomes more uniformly distributed. The vermicular grains are a result of the more heterogeneous distribution of seeds in the matrix and the large agglomerate sizes in the γ-Al_2O_3 powder. The 5 wt.% seeded sample exhibits a homogeneous microstructure of 0.1 μm α-Al_2O_3 grains.

The microstructures of γ-Al_2O_3 samples with 0–5 wt.% seeds sintered for 100 min at 1200°C are shown in Fig. 2. All of the samples have completely transformed and have undergone grain coarsening with a two-fold

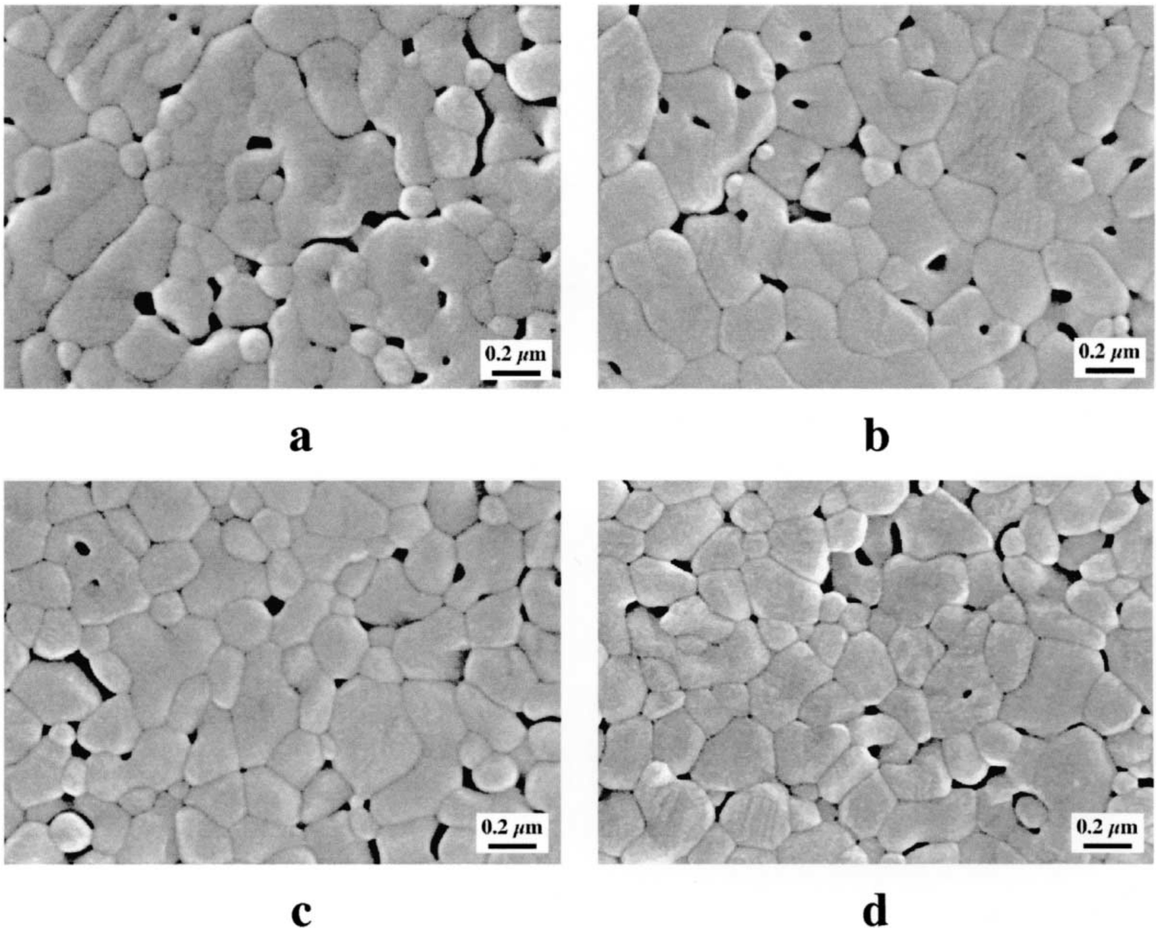

Fig. 2. Microstructures of (a) 0 wt.%, (b) 1.25 wt.%, (c) 2.5 wt.%, and (d) 5 wt.% seeded γ-Al_2O_3 samples sintered for 100 min at 1200°C.

increase in grain size. Rapid grain coarsening of the fine-grained microstructure resulted in pore entrapment within some of the grains. However, the size and number of entrapped pores decreases with increasing seed concentration. In addition, the grain morphologies appear more equiaxed at higher seed concentrations.

The microstructures of γ-Al$_2$O$_3$ samples seeded with 0–5 wt.% α-Al$_2$O$_3$ and sintered for 100 min at 1400°C are shown in Fig. 3. The unseeded sample contains continuous pore channels and large irregular grains of ·0.8 µm. The seeded samples have sintered to high densities with equiaxed grains (0.5–2 µm). The microstructures of γ-Al$_2$O$_3$ samples seeded with 0–5 wt.% α-Al$_2$O$_3$ and sintered at 1500°C for 100 min are shown in Fig. 4. Grains become more equiaxed in the unseeded sample as the matrix continues to densify. A few large pores are still present; however, a majority of the porosity is isolated at grain boundary junctions. Since seeded samples achieved high densities at 1400°C, sintering at higher temperature results in significant grain growth. The 5 wt.% seeded sample exhibits exaggerated grain growth resulting in a broad grain size distribution (1–12 µm). Without the use of grain growth inhibitors or dopants like MgO, the presence of impurities such as SiO$_2$ and TiO$_2$ in the as-received powder results in grain growth in preferential directions and the formation of faceted grains.[24,25]

The microstructures of the commercial δ/α-Al$_2$O$_3$ powder sintered at 1400, 1500, and 1600°C for 100 min are shown in Fig. 5. The sample sintered at 1400°C exhibits a broad grain size distribution of 0.2–2 µm grains with a few large pores and less porosity located on grain boundaries. The sample sintered at 1600°C appears dense with a few small pores. Although there are heterogeneities in the seeded samples due to agglomeration of the nanocrystalline powders, a more homogeneous microstructure, compared to the δ/α-Al$_2$O$_3$ mixture, of 0.5 µm grains was obtained at 1400°C from seeded transition alumina powder.

The densification of the seeded γ-Al$_2$O$_3$ and the commercial δ/α-Al$_2$O$_3$ powder are shown in Fig. 6. The unseeded γ-Al$_2$O$_3$ samples sinter to 65% relative density at 1400°C and reached 90% at 1600°C. All of the seeded γ-Al$_2$O$_3$ samples sintered to $\geqslant$98% relative density at 1400°C and reached ·100% density at 1500°C. It is reasonable to conclude that $>$95% density would be obtained by sintering seeded γ-Al$_2$O$_3$ at lower temperatures for longer periods of time. The δ/α-Al$_2$O$_3$ mixture

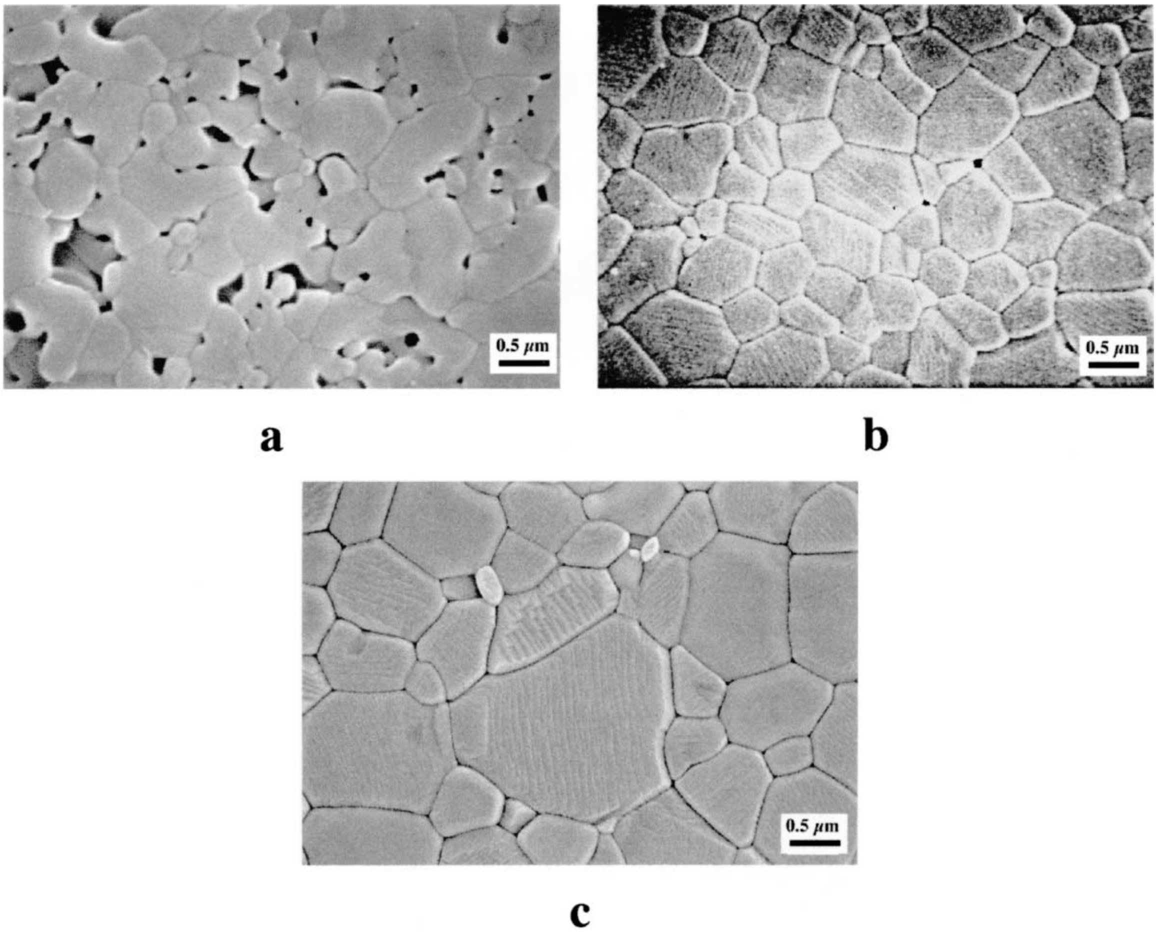

Fig. 3. Microstructures of (a) 0 wt.%, (b) 1.25 wt.%, and (c) 5 wt.% seeded γ-Al$_2$O$_3$ samples sintered for 100 min at 1400°C.

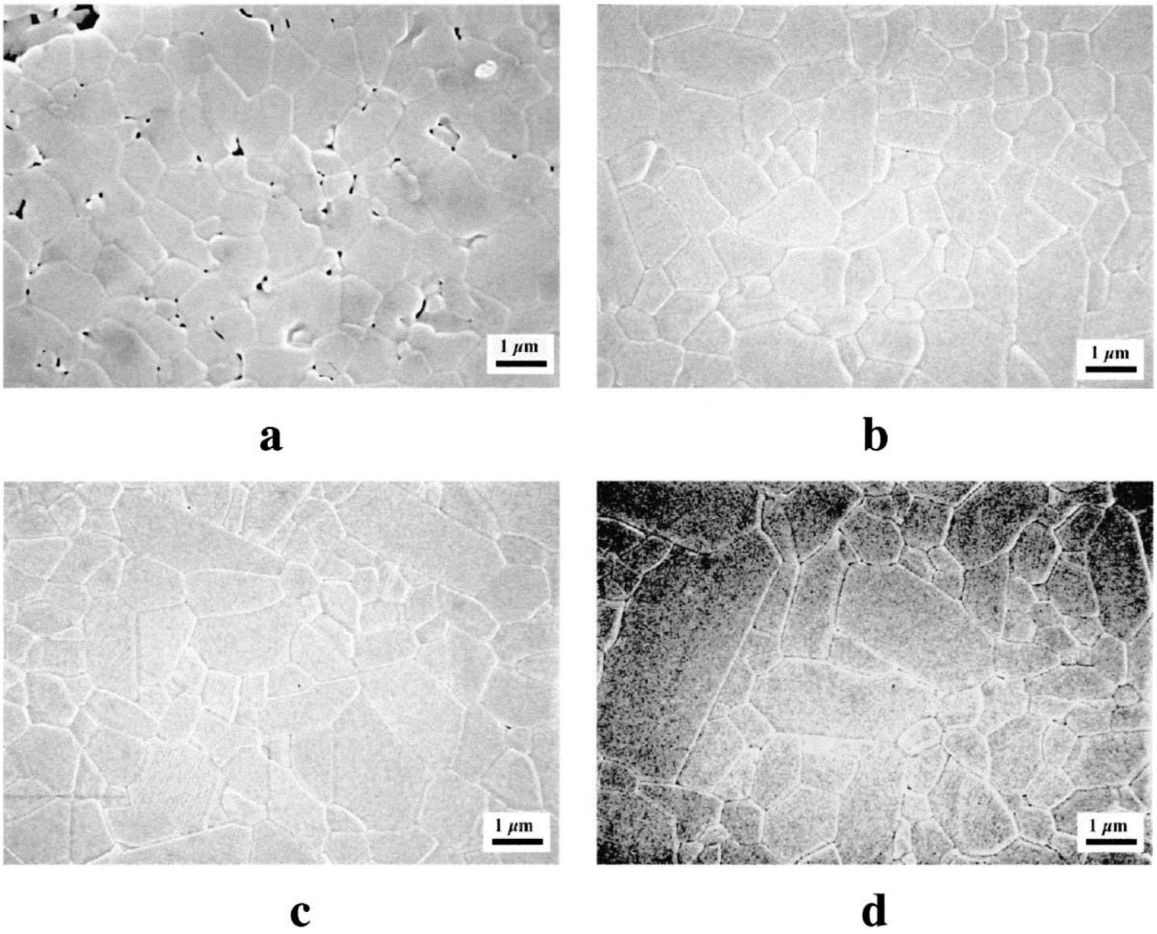

Fig. 4. Microstructures of (a) 0 wt.%, (b) 1.25 wt.%, (c) 2.5 wt.%, and (d) 5 wt.% seeded γ-Al_2O_3 samples sintered for 100 min at 1500°C.

sintered to 88% relative density at 1400°C and reached 98% relative density at 1600°C.

The δ/α-Al_2O_3 mixture sintered better than the unseeded γ-Al_2O_3 sample due to a higher green density and the presence of α-Al_2O_3 particles which act to seed the phase transformation in the transition alumina. However, the density of the δ/α-Al_2O_3 mixture did not exceed the sintered densities of the seeded γ-Al_2O_3 samples. Sintering temperatures >1600°C were required to reach full density using the δ/α-Al_2O_3 mixture powders. The seeded γ-Al_2O_3 sintered better than the commercial δ/α-Al_2O_3 powder because the nucleation density in the seeded powders was significantly higher. Close examination of the commercial powder showed that the α-Al_2O_3 was ·1 μm diameter. Since the seed number density (N_v) scales with $d^{-1/3}$, the seed number density from the α-Al_2O_3 in the commercial δ/α-Al_2O_3 powder is at least three orders of magnitude lower than in the seeded samples. As shown here and in previous papers on seeded boehmite, the number frequency is the key parameter controlling the particle size of the transformed α-Al_2O_3, which in turns affects the sintering temperature.[11,14]

Green density is known to affect the phase transformation. Therefore, seeded γ-Al_2O_3 powders were uniaxially pressed in a zinc stearate lubricated WC die to obtain pellets of 6.2 mm diameter and ·2 mm height. The green densities of the pressed pellets, determined by the dimensional technique, were 48, 53, 56, and 59% ($\rho_o = 3.2$ g/cm^3) for samples pressed at 140, 280, 420, and 560 MPa, respectively. The densification behavior of seeded γ-Al_2O_3 pellets as a function of die pressure is shown in Fig. 7. At 1000°C, the sintered densities range from 43% for the 140 MPa pressed sample to 55% for the 560 MPa pressed sample. The pellets sinter rapidly between 1200 and 1400°C. The pellets reach full density at 1400 and 1500°C when pressed at 280 and 140 MPa, respectively. By increasing the packing density of the γ-Al_2O_3 powders, spacing between nucleation events in the matrix is slightly reduced and the number of particle contacts between the matrix phase and the seed particles is increased, both of which reduce the scale of the vermicular microstructure. As a result, higher sintered densities are attainable at lower temperatures. The sintered densities for samples die pressed at >280 MPa exceed any previous reports about pressureless sintering undoped γ-Al_2O_3 powders.

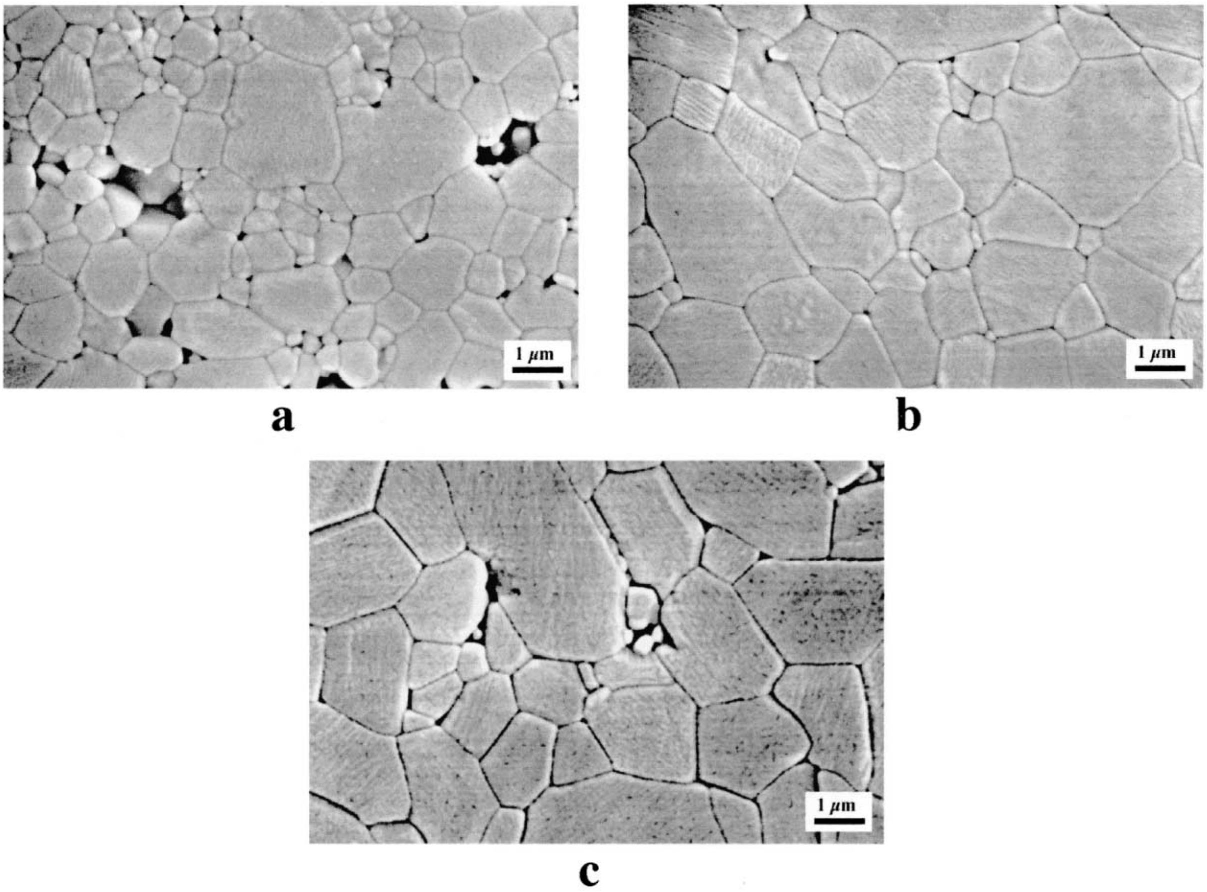

Fig. 5. Microstructures of δ/α-Al_2O_3 mixture (CR-30) powders sintered at (a) 1400, (b) 1500, and (c) 1600°C for 100 min.

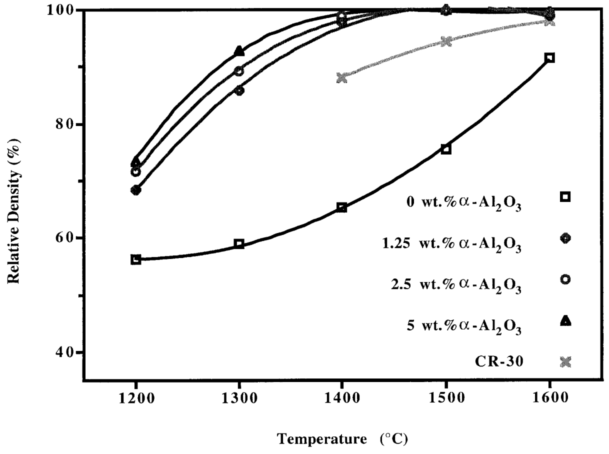

Fig. 6. Sintered densities of seeded γ-Al_2O_3 and δ/α-Al_2O_3 samples as a function of seed concentration and temperature (100 min).

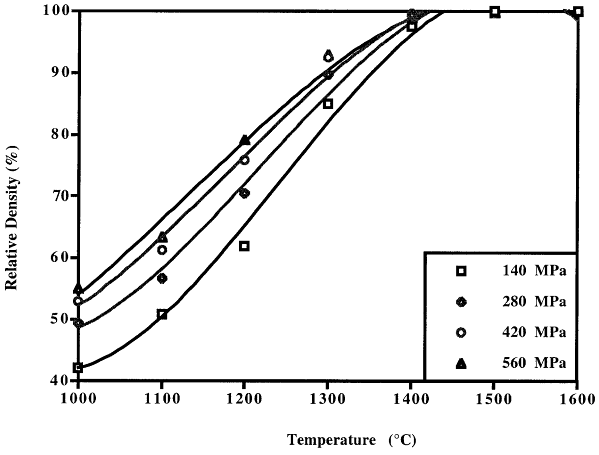

Fig. 7. Sintered densities of 5 wt.% seeded γ-Al_2O_3 samples pressed at various pressures and sintered at various temperatures for 100 min.

4. Summary

The addition of α-Al_2O_3 seeds to commercial γ-Al_2O_3 powders has a significant effect on microstructure evolution and densification. The addition of seeds provides a large number of nuclei for α-Al_2O_3 formation and thus vermicular growth during the phase transformation was limited. The seeded γ-Al_2O_3 samples sintered to full density with a 1.5 μm grain size at 1400°C after 100 min. The results of this investigation are a significant improvement relative to earlier reports for densification of either pressureless or pressure assisted sintering of undoped boehmite and γ-Al_2O_3 samples.

The use of nanocrystalline γ-Al_2O_3 powders as a starting material provides a higher bulk density than boehmite gel systems and thus less shrinkage during sintering. In addition, the time consuming calcination step required for making bulk samples from boehmite powders was avoided. In comparison to a commercial δ/α-Al_2O_3 powder, the seeded γ-Al_2O_3 samples sintered to full density at 1400°C, while the δ/α-Al_2O_3 powder samples sintered to 98% density at 1500°C after 100 min. It is proposed that reduction in the agglomerate size (<50 nm), or removal of the agglomerates, will further lower the sintering temperature to less than 1400°C. We also conclude that seeding with other sesquioxides, which have been demonstrated to be effective for boehmite gels, would be successful for seeding the phase transformation and sintering of transition aluminas.

References

1. Wefers, K. and Misra, C. Oxides and hydroxides of aluminum. *ALCOA Technical Paper* No. 19, Rev. ALCOA Labs, 1987.
2. Dynys, F. W. and Halloran, J. W., Alpha alumina formation in alum-derived gamma alumina. *J. Am. Ceram. Soc.,* 1982, **65**(9), 442–448.
3. Nordahl, C. S. and Messing, G. L., Thermal analysis of phase transformation kinetics in alpha alumina seeded boehmite and gamma alumina. *Thermochimica Acta,* 1998, **318**(2), 187–200.
4. Jagota, S. and Raj, R., Model for the crystallization and sintering of unseeded and seeded boehmite gels. *J. Mater. Sci.,* 1992, **27**(8), 2551–2557.
5. Pach, L., Roy, R. and Komarneni, S., Nucleation of alpha alumina in boehmite gel. *J. Mater. Res.,* 1990, **5**(2), 278–285.
6. Urretavizcaya, G. and Porto Lopez, J. M., Thermal transformations of sol gel alumina into α-phase. Effect of α-Al_2O_3 seeding. *Mar. Res. Bull,* 1992, **27**(4), 375–385.
7. Yarbrough, W. A. and Roy, R., Microstructural evolution in sintering of AlOOH gels. *J. Mater. Res.,* 1987, **2**(4), 494–515.
8. Wu, S. J., De Jonghe, L. C. and Rahaman, M. N., Sintering of nanophase γ-Al_2O_3 powder. *J. Am. Ceram. Soc.,* 1996, **79**(8), 2207–2211.
9. Badkar, P. A., Bailey, J. E. and Baker, H. A., Sintering behavior of boehmite gels. In *Sintering and Related Phenomenon, Material Science Research,* Vol. 6, ed. G. C. Kuczynski. Plenum, New York, 1973, pp. 311–322.
10. Kumagai, M. and Messing, G. L., Enhanced densification of boehmite sol-gels by α-alumina seeding. *J. Am. Ceram. Soc.,* 1984, **67**(11), C-230–C-231.
11. Kumagai, M. and Messing, G. L., Controlled transformation and sintering of a boehmite sol-gel by α-alumina seeding. *J. Am. Ceram. Soc.,* 1985, **68**(9), 500–505.
12. Suwa, Y., Komarneni, S. and Roy, R., Solid-state epitaxy demonstrated by thermal reactions of structurally diphasic xerogels: the system Al_2O_3. *J. Mater. Sci. Lett.,* 1986, **5**(1), 21–24.
13. Shelleman, R. A., Messing, G. L. and Kumagai, M., Alpha alumina transformation in seeded boehmite gels. *J. Non-Cryst. Sol,* 1986, **82**(1-3), 277–285.
14. Messing, G. L. and Kumagai, M., Low-temperature sintering of α-alumina-seeded boehmite gels. *Am. Ceram. Soc. Bull.,* 1994, **73**(10), 88–91.
15. Nordahl, C. S. and Messing, G. L., Transformation and densification of nanocrystalline θ-Al_2O_3 during sinter forging. *J. Am. Ceram. Soc.,* 1996, **79**(12), 3149–3154.
16. Prouzet, E., Fargeot, D. and Baumard, J. F., Sintering of boehmite-derived transition alumina seeded with corundum. *J. Mater. Sci. Lett.,* 1990, **9**(7), 779–781.
17. Tsai, D.-S. and Hsieh, C.-C., Controlled gelation and sintering of monolithic gels prepared from γ-alumina fume powder. *J. Am. Ceram. Soc.,* 1991, **74**(4), 830–836.
18. Hrabê, Z., Spaldon, O. M., Pach, L. and Kozánková, J., Thermal transformations of boehmite gel in controlled furnace atmospheres. *Mater. Res. Bull,* 1992, **27**(4), 397–404.
19. Suryanarayana, K. V., Panda, R. K., Prabhu, N. and Rao, B. T., Effect of simultaneous additions of niobia and magnesia on the sintering and microstructure of seeded boehmite. *Ceram. Inter.,* 1995, **21**(3), 173–179.
20. Prasad, J., Agrawal, G. N. and Narasimhan, M. D., Sintering of alumina (γ-Al_2O_3) in presence of ($CaO + Fe_2O_3$). *Trans. Ind. Ceram. Soc.,* 1983, **42**(2), 31–34.
21. Freim, J., McKittrick, J., Katz, J. and Sickafus, K., Microwave sintering of nanocrystalline gamma-Al_2O_3. *Nanostr. Matl.,* 1994, **4**(4), 371–385.
22. McArdle, J. L. and Messing, G. L., Transformation and microstructure control in boehmite-derived alumina by ferric oxide seeding. *Adv. Ceram. Mater.,* 1988, **3**(4), 387–392.
23. McArdle, J. L. and Messing, G. L., Transformation, microstructure development and densification in α-Fe_2O_3 seeded boehmite-derived alumina. *J. Am. Ceram. Soc.,* 1993, **76**(1), 214–222.
24. Horn, D. S. and Messing, G. L., Anisotropic grain growth in TiO_2-doped alumina. *Mat. Sci. Engr.,* 1995, **A195**, 169–178.
25. Kebbede, A., Parai, J. and Carim, A. H., Anisotropic grain growth in α-Al_2O_3 with SiO_2 and TiO_2 additions. *J. Am. Ceram. Soc.,* 2000, **83**(11), 2845–2851.