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Effect of Pre-coarsening on the Microstructural Evolution of Fast-Fired and MgO-Doped Al_2O_3

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Abstract

The effect of an initial pre-coarsening step (50 to 200 h at 800 °C) on the subsequent densification and microstructural evolution of high quality compacts of undoped and MgO-doped Al_2O_3 has been investigated during fast-firing (5 min at 1750 °C) and during constant heating rate sintering (4 °C/min to 1450 °C). In constant heating rate sintering of both the undoped and MgO-doped Al_2O_3, a refinement of the microstructure was achieved for the pre-coarsened compact. A combination of the pre-coarsening step and MgO doping produced the most significant refinement of the microstructure. In fast-firing of the MgO-doped Al_2O_3, the pre-coarsening step produced a measurable increase in the density and a small refinement of the grain size, when compared with similar compacts fast-fired conventionally (i.e., without the pre-coarsening step). This result indicates that the accepted view of the deleterious role of coarsening in the sintering of real powder compacts must be re-examined. While extensive coarsening after the onset of densification must be reduced for the achievement of high density, limited coarsening *prior to* densification is beneficial for subsequent sintering.

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I. Introduction

Two-step sintering, involving an initial pre-coarsening step prior to densification, has been demonstrated by De Jonghe and coworkers [1-6] to be an effective approach for refining the microstructure of MgO, ZnO and Al_2O_3 powder compacts during constant heating rate sintering (4 - 10 °C/min) and during isothermal sintering. For these systems, the approach has been applied successfully to heterogeneous compacts of highly agglomerated commercial powders [1-4], to compacts of fine, unclassified commercial powders [3,4] and to compacts formed by high-pressure cold isostatic pressing or slip casting of a fine powder with a narrow particle size distribution [5,6].

Detailed investigations of the densification kinetics and microstructural evolution during two-step sintering indicate that the pre-coarsening step leads to a stronger compact with a more homogeneous microstructure [4,6]. Matter transport (presumably by an Ostwald ripening process) leads to a limited increase in the average particle size (thereby reducing the driving force for subsequent densification). However, while the finest particles are also eliminated, the pore channels become more uniform. The experimental data combined with semi-quantitative modeling of the evolution of the inter-pore distance [4] and of pore closure [6] during subsequent sintering have been used to identify possible mechanisms for the microstructural refinement observed in two-step sintering. The stronger compact is better able to resist sintering damage due to differential densification, while the elimination of the finest particles at the same time reduces the driving force for such differential densification. These two factors lead to a reduction in the scale of density fluctuations during subsequent sintering, i.e., to a more uniform microstructure. The improved structural uniformity of both the particle network and the pore channels acts to delay the pinch-off of the continuous pore channels.

Porosity remains open to higher densities, thereby inhibiting grain growth very effectively. One consequence is that the densification rate is reduced in the early stages of subsequent sintering. However, the benefits of the more homogeneous evolution of the microstructure are expressed in the later stages, so that a refinement of the final microstructure is achieved.

The intentional coarsening in two-step sintering may, at first glance, be considered to represent a deviation from the widely accepted approach for the production of polycrystalline ceramics by the powder processing route. For the achievement of high density and controlled (fine) grain size, a requirement is the decrease in the rate of coarsening relative to the rate of densification [7]. While this requirement is satisfied by most of the fabrication routes which are known to be effective [8], it is perhaps best exemplified by the fast-firing (or rapid sintering) technique. In fast-firing, the objective is to enhance the ratio the densification rate to the coarsening rate by a rapid approach to the sintering temperature. Since the coarsening mechanisms (e.g., surface diffusion and vapor transport) commonly dominate over the densification mechanisms (e.g., lattice and grain boundary diffusion) at lower temperatures, it has been suggested that rapid heating to higher temperatures can be beneficial for the achievement of high density coupled with fine grain size [7]. In this case the shorter time spent at lower temperatures serves to reduce the extent of coarsening, while at the same time the driving force for densification would not be decreased significantly. The merit of this proposal has been demonstrated practically [9] in the fast-firing of Al_2O_3, a system for which the activation energy for densification is higher than that for coarsening (or grain growth). For MgO, fast-firing has not been effective, but the relationship between the activation energies for densification and coarsening is uncertain, so that it is more difficult to specify an optimum temperature path for fast-firing.

In distinguishing the merits of fast-firing and two-step sintering, it should be realized that the fast-firing approach is based primarily on a consideration of the relative rates of densification and coarsening. However, it is well known that microstructure of the powder compact can also have a significant if not dominant effect on the subsequent densification and the microstructure evolution [10-19]. The two-step sintering approach exploits the microstructural homogenization produced by the pre-coarsening step.

The objective of the present work was to explore if a pre-coarsening step (50-200 h at 800 °C) prior to fast-firing and prior to the constant heating rate sintering of MgO-doped Al_2O_3 would offer any benefit at all. The powder compacts, with a relatively uniform packing, were formed by high pressure cold isostatic pressing or slip casting of a fine powder with a narrow distribution of particle sizes. This system was considered to provide a fairly homogeneous green microstructure for which conventional fast-firing by itself (i.e., without a pre-coarsening step) would be particularly effective for densification. MgO is known to be an effective dopant for the production, by conventional sintering, of Al_2O_3 with nearly theoretical density and a fairly uniform distribution of grain sizes. While the experiments are important for exploring the wider applicability of the two-step sintering approach, the results may also provide further insight into the microstructure control of ceramics.

II. Experimental Procedure

(1) Powder Processing and Consolidation

A commercial, high-purity Al_2O_3 powder (AKP-50; Sumitomo Chemical America, Inc., New York, NY) was used in the experiments. According to the manufacturer's specifications, the as-received powder had an average particle size of 0.18 μm and a surface area of 10.4 m^2/g.

As described in detail elsewhere [5,6], the powder was processed to remove hard agglomerates prior to compaction. Briefly, the powder was dispersed in water at a pH value of 3 - 4, and the suspension was ultrasonically agitated and allowed to settle gravitationally for ≈24 h. The supernatant was decanted and dried rapidly under an infrared lamp. The dried material was lightly ground in a mortar and pestle, passed through a nylon sieve (100 mesh; aperture ≈126 μm) and then ball-milled in the dry state using plastic milling media. The average particle size of the resulting powder, measured by a centrifugal sedimentation technique (Model CAPA-700, Horiba Instruments Inc., Irvine, CA) was 0.17 μm. The distribution of particle sizes was relatively narrow, with a geometrical standard deviation equal to 1.6 (defined as the ratio d_{84}/d_{50}, where 84% and 50% of the cumulative mass of the particles are finer than the sizes denoted by d_{84} and d_{50}, respectively). The specific surface area of the powder, measured by nitrogen adsorption by the BET method (Digisorb 2600, Micromeritics Instrument Corp., Norcross, GA) was 10.9 m^2/g.

In the preparation of MgO-doped Al_2O_3, magnesium nitrate [$Mg(NO_3)_2 \cdot 6H_2O$; purity = 99.999%; EM Industries Inc., Gibbstown, NJ] was dissolved in deionized water and the required amount of Al_2O_3 powder was added to obtain a dopant concentration of 250 ppm (i.e., Mg/Al atomic ratio = 250 ppm). After rapid drying under an infrared lamp while being stirred vigorously, the mixture was ground lightly in a mortar and pestle and ball milled as described above for the undoped powder. Calcination of the doped material, to decompose the $Mg(NO_3)_2$ and to incorporate the Mg into solid solution, was performed after compaction of the powder to reduce the possibility of forming hard agglomerates in the fine-grained Al_2O_3 powder.

Powder compacts for sintering were formed by uniaxial pressing in a WC-Co die

followed by cold isostatic pressing under high pressure. No binder was used in the process. Die pressing was performed under a pressure of ≈50 MPa to produce compacts (6.35 mm in diameter by 6.3 mm) with a density of ≈0.53 of the theoretical density of α-Al_2O_3 (3.986 g/cm^3). After cold isostatic pressing under a pressure of ≈1.4 GPa, the relative density of the compacts increased to 0.60 ± 0.01.

Compacts were also formed by slip casting of aqueous suspensions. The powder was dispersed in water at a pH of 3.5 to give a solids concentration of ≈40 vol%. The slurry was continuously poured into molds, made of plastic templates placed on top of a block of plaster of Paris, until a solid sample was formed. The samples were dried for ≈24 h at room temperature, removed from the molds and then heated for 1 h at 450 °C (heating rate = 0.5 °C/min) to burn off the organic additives. The density of the compacts was 0.62 ± 0.01.

(2) Sintering and Characterization

Compacts of the undoped Al_2O_3 and the MgO-doped Al_2O_3 were sintered by the two-step technique involving the use of a pre-coarsening step prior to subsequent densification. Pre-coarsening was carried out in air for 50 to 200 h at 800 °C. These conditions were found earlier to produce significant coarsening without measurable densification [4,6]. After the pre-coarsening step, the compacts were densified by constant heating rate sintering or by fast-firing. Constant heating rate sintering was performed in air at 4 °C/min to 1450 °C in a dilatometer (Model 1600D; Edward Orton Jr. Ceramic Foundation, Westerville, OH) that allowed continuous monitoring of the shrinkage kinetics. The density of the compact at any temperature was determined from the initial density and the measured shrinkage. In the fast firing schedule, the compact was heated in vacuum at 340 °C/min to 1750 °C and held at this

temperature for 5 min. For comparison, compacts were also fired without the pre-coarsening step. In this case the firing schedule is referred to as "conventional". In the conventional routine, the compact was sintered or fast-fired under the same conditions used in the two-step technique but without the pre-coarsening step.

In the constant heating rate experiments employing the two-step technique, a compact was placed in the dilatometer, heated at 1 °C/min to 600 °C and held at this temperature for 1 h to decompose the $Mg(NO_3)_2$ in the doped powder compacts. The compact was then heated at 4 °C/min to 800 °C and pre-coarsened for 50 to 200 h at this temperature, after which it was heated at 4 °C/min to 1450 °C. A different compact was used in each experiment. In the fast-firing experiments employing the two-step technique, after the decomposition process and the pre-coarsening step at 800 °C, the compact was cooled, removed from the dilatometer and then fast-fired in a vacuum furnace.

The final densities of the compacts were measured by the Archimedes method. The microstructures were observed by scanning electron microscopy (SEM) of polished and thermally etched surfaces. Thermal etching was performed in air for 1 h at 1280 °C. Average grain size was measured from SEM micrographs by the lineal intercept technique [20,21], using more than 500 grains for each sample.

III. Results and Discussion

The results for the two sintering schedules after the pre-coarsening step are discussed separately in the following sections.

(1) Fast Firing

Figure 1 shows the microstructures of the MgO-doped Al_2O_3 powder compacts (formed by slip casting) after conventional fast-firing (i.e., without the pre-coarsening step; rapidly brought to 1750 °C and held for 5 min) and by the two-step technique involving an initial pre-coarsening step (50 h and 100 h at 800 °C) followed by fast-firing. Differences in the grain characteristics resulting from the pre-coarsening step are not very obvious. However, the density of the compact fast-fired conventionally is slightly ***lower*** than those for the compacts fast-fired by the two-step technique. It is evident from Fig. 1 that pores located at the triple junctions of the grains are much more prevalent in the sample fast-fired conventionally. The final densities and average grain sizes of the compacts are summarized in Table 1.

It is clear that the refinement in the final microstructure and the increased density produced by the two-step approach is small in the present case. However, its occurrence, however small, in a system where conventional fast-firing (i.e., without the pre-coarsening step) is expected to be particularly effective indicates that the widely accepted view of the coarsening role in the sintering of real powder compacts must be re-examined. While significant coarsening *after the onset of* densification must be reduced for the achievement of high density, its limted occurrence *prior to* densification can be beneficial for subsequent sintering. A simple interpretation of this observation is that the microstructural features that drive rapid coarsening are also the ones that drive rapid densification. Local coarsening, however, serves to homogenize the microstructure with a concurrent decrease of the driving forces, while local densification produces sintering damage.

(2) Constant Heating Rate Sintering

Figure 2 shows the results for the relative density versus temperature during subsequent sintering (at a constant heating rate of 4 °C/min) for the undoped Al_2O_3 and the MgO-doped Al_2O_3 compacts (formed by cold isostatic pressing) with and without a pre-coarsening step (50 h at 800 °C). For both the undoped and MgO-doped compacts, the precoarsening step produces a delay in the onset of measurable densification and, initially, the density of the pre-coarsened compact is somewhat lower than that of the conventionally sintered compact (i.e., the compact sintered without the pre-coarsening step). However, later in the sintering process, the pre-coarsened compact densifies faster than the conventionally sintered compact (Fig. 3), and the compacts reach almost the same final density. The final densities are in the range of 0.99 to 1.00. The densification behavior shows trends similar to those found in earlier work with other powders [4,6].

The microstructures of slip cast powder compacts of undoped Al_2O_3 after sintering to 1450 °C with and without the pre-coarsening step (50 h at 800 °C) are shown in Fig. 4. Corresponding microstructures for the MgO-doped Al_2O_3 are shown in Fig. 5. The final densities and average grain sizes of the sintered materials are summarized in Table 2. For both the undoped Al_2O_3 and the MgO-doped Al_2O_3, two-step sintering leads to a reduction in the grain size. However, the relative magnitude of the reduction is larger for the MgO-doped Al_2O_3 ($\approx$25%) than for the undoped Al_2O_3 ($\approx$15%). The data also indicate that the presence of the MgO leads to a reduction in the final grain size for both conventional sintering and two-step sintering.

The data in Table 2 indicate that a combination of MgO doping and two-step sintering is most effective for the refinement of the grain size. The effects of two-step sintering and

MgO doping on the refinement of the grain size therefore appear to be additive. It would be interesting to examine the role of MgO in the sintering of Al_2O_3 and the mechanism of microstructural refinement during two-step sintering to determine the basis for this additivity of effects. Harmer and coworkers [22,23] have shown that the dominant role of MgO in the sintering of Al_2O_3 is its ability to reduce the grain boundary mobility. From a consideration of model equations for densification and grain growth, they suggested that the reduction in the boundary mobility shifts the onset of abnormal grain growth (pore-boundary separation) to larger grain size. As a result, sintering can proceed to full density without entering the separation region. From measurements of the pore surface area and the grain boundary area during sintering of undoped and MgO-doped Al_2O_3, Shaw and Brook [24] suggested that MgO acts as a microstructural homogenizer in that it restrains grain growth in the dense regions of the compact, until the less dense regions have an opportunity to densify. In this way, the MgO acts to smooth out the consequences of microstructural heterogeneity.

As described earlier, in two-step sintering, De Jonghe and coworkers [4,6] have shown that the most significant effect of the initial pre-coarsening step on subsequent densification is the production of a stronger, more homogeneous compact. A common feature of the MgO role (as outlined by Shaw and Brook) and the pre-coarsening step (in two-step sintering) is seen as the ability to produce a homogenization of the microstructure. The pre-coarsening step acts to homogenize the microstructure prior to densification, while, according to Shaw and Brook, the MgO acts to homogenize the microstructure during densification. The effectiveness of the combination of MgO doping and two-step sintering in refining the microstructure of Al_2O_3 can therefore been seen in their complementary roles.

IV. Conclusion

Two-step sintering, in which an initial pre-coarsening step is employed prior to densification, provides a small refinement in the final microstucture of fast-fired and MgO-doped Al_2O_3 when compared to similar compacts sintered conventionally (i.e., without the pre-coarsening step). While the effectiveness of fast-firing is commonly discussed primarily in terms of the relative rates of densification and coarsening, a more practical approach for real powder compacts requires a consideration of both kinetics and the homogeneity of the microstructure. The effectiveness of MgO in controlling the grain growth during sintering of Al_2O_3 is enhanced in two-step sintering. Two-step sintering appears to be a generally valid approach for the improvement of the microstructure of practical ceramic powder systems.

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Table 1. Final density and average grain size of MgO-doped Al_2O_3 powder compacts (formed by slip casting) after fast-firing by the conventional approach (i.e., without a pre-coarsening step) and by the two-step technique involving a pre-coarsening step (50 h or 100 h at 800 °C) followed by fast-firing. Fast-firing was performed in vacuum at 340 °C/min to 1750 °C with a soaking time of 5 min.

Fast-Firing Approach	Final Density	Average Grain Size
Conventional	0.993	6.15 μm
Two-Step (50 h at 800 °C)	0.998	5.94 μm
Two-Step (100 h at 800 °C)	0.998	6.03 μm

Table 2. Final density and average grain size of undoped and MgO-doped Al_2O_3 powder compacts (formed by slip casting) after constant heating rate sintering (4 °C/min to 1450 °C) by the conventional approach and by the two-step approach involving a pre-coarsening step for 50 h at 800 °C.

Al_2O_3 Sample	Sintering Approach	Final Density	Average Grain Size
Undoped	Conventional	0.998	2.16 μm
Undoped	Two-Step	0.998	1.84 μm
MgO-doped	Conventional	0.997	1.44 μm
MgO-doped	Two-Step	0.997	1.07 μm

Figure Captions

Figure 1. Scanning electron micrographs of polished and thermally etched surfaces of MgO-doped Al_2O_3 compacts formed by slip casting and fast-fired in vacuum at 340 °C/min to 1750 °C for 5 min: (a) conventional approach (i.e., no pre-coarsening), (b) two-step approach (pre-coarsening for 50 h at 800 °C prior to fast-firing), and (c) two-step approach (pre-coarsening for 100 h at 800 °C prior to fast firing). The densities and grain sizes are given in Table 1.

Figure 2. Relative density as a function of temperature for undoped and MgO-doped Al_2O_3 compacts formed by cold isostatic pressing and sintered at 4 °C/min to 1450 °C by the conventional approach (no pre-coarsening) and by the two-step approach (pre-coarsening for 50 h at 800 °C).

Figure 3. Densification rate as a function of temperature for the samples described in Fig.2.

Figure 4. Scanning electron micrographs of polished and thermally etched surfaces of the undoped Al_2O_3 compacts sintered under the conditions described in Fig. 2: (a) conventional approach (no pre-coarsening) and (b) two-step approach (pre-coarsenig for 50 h at 800 °C). The densities and grain sizes are given in Table 2.

Figure 5. Scanning electron micrographs of polished and thermally etched surfaces of the MgO-doped Al_2O_3 compacts sintered under the conditions described in Fig. 2: (a) conventional approach (no pre-coarsening) and (b) two-step approach (pre-coarsenig for 50 h at 800 °C). The densities and grain sizes are given in Table 2.

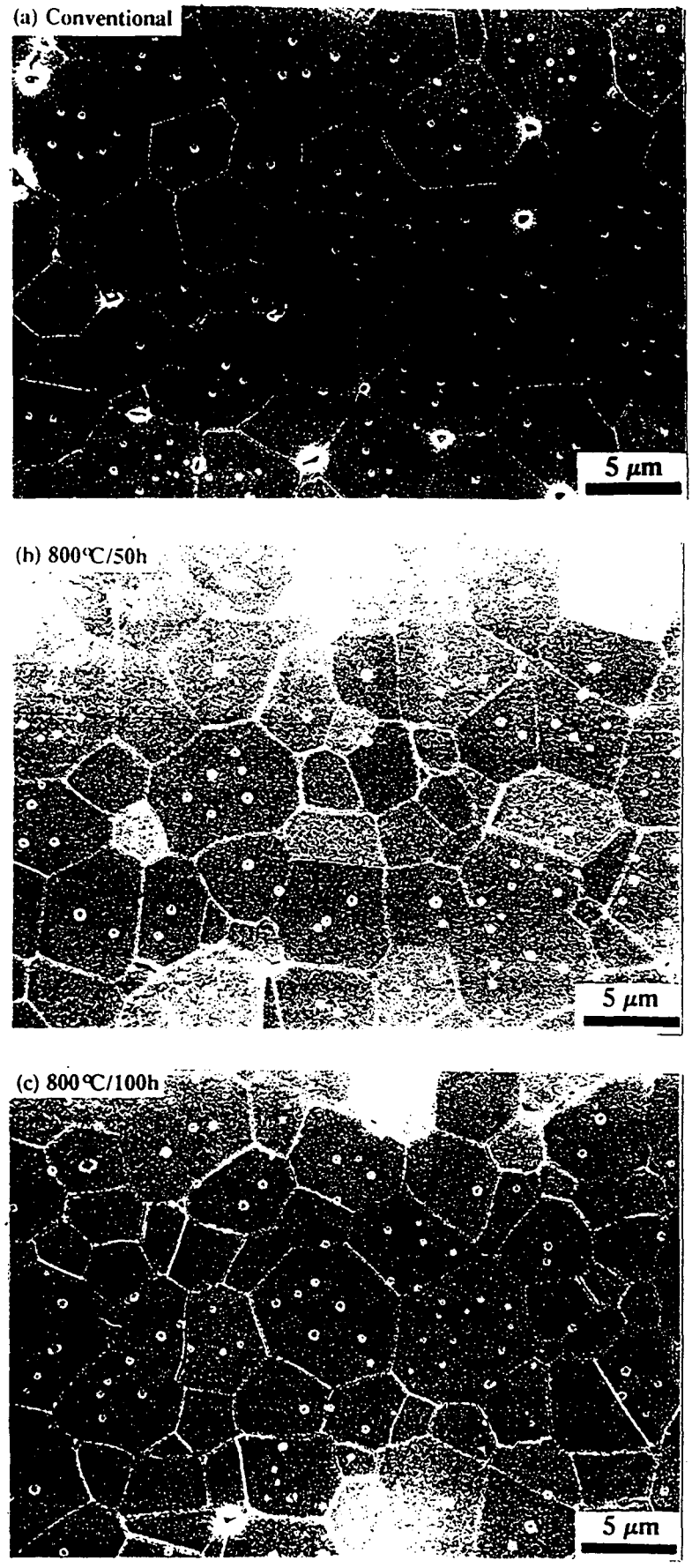

Figure 1

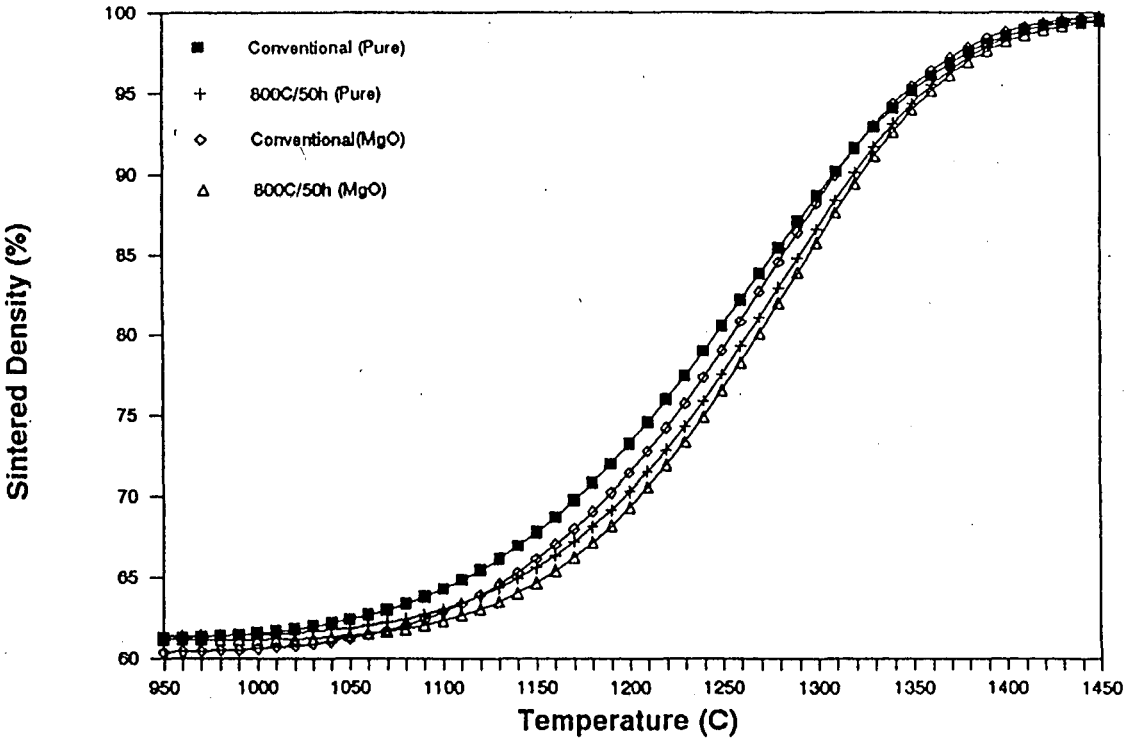
Conventional (Pure)
800C/50h (Pure)
Conventional(MgO)
800C/50h (MgO)
Sintered Density (%)
100
95
90
85
80
75
70
65
60
950
1000
1050
1100
1150
1200
1250
1300
1350
1400
1450
Temperature (C)

Figure 2

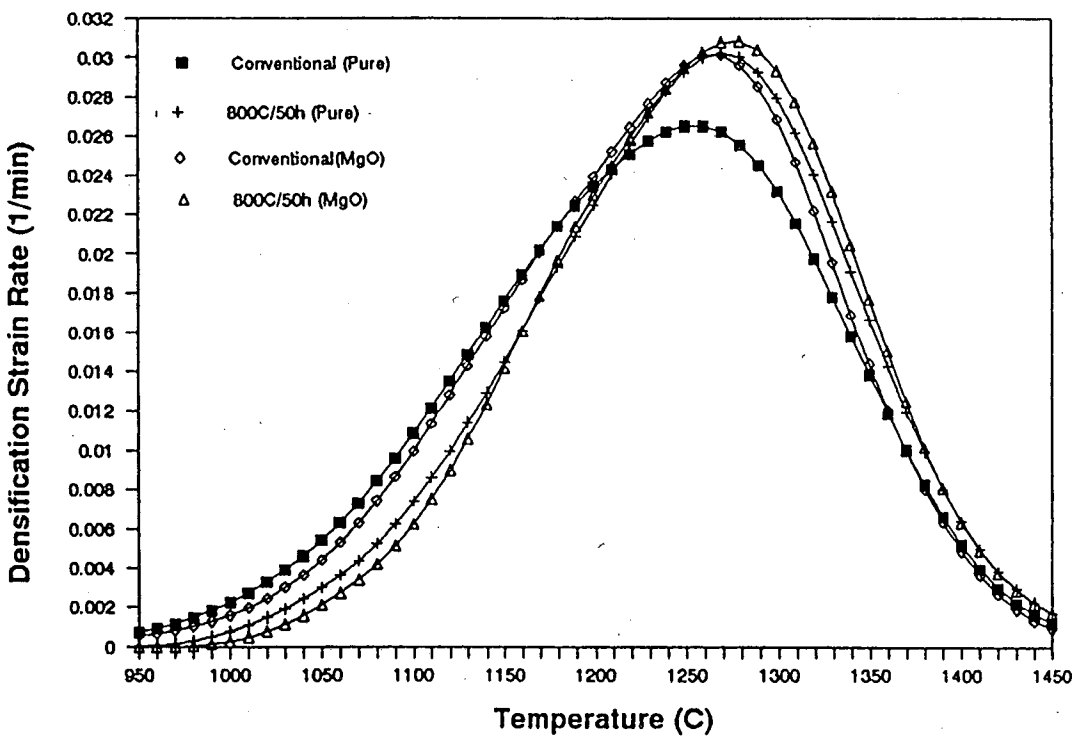
Conventional (Pure)
800C/50h (Pure)
Conventional(MgO)
800C/50h (MgO)
Densification Strain Rate (1/min)
0.032
0.03
0.028
0.026
0.024
0.022
0.02
0.018
0.016
0.014
0.012
0.01
0.008
0.006
0.004
0.002
0
950
1000
1050
1100
1150
1200
1250
1300
1350
1400
1450
Temperature (C)

Figure 3

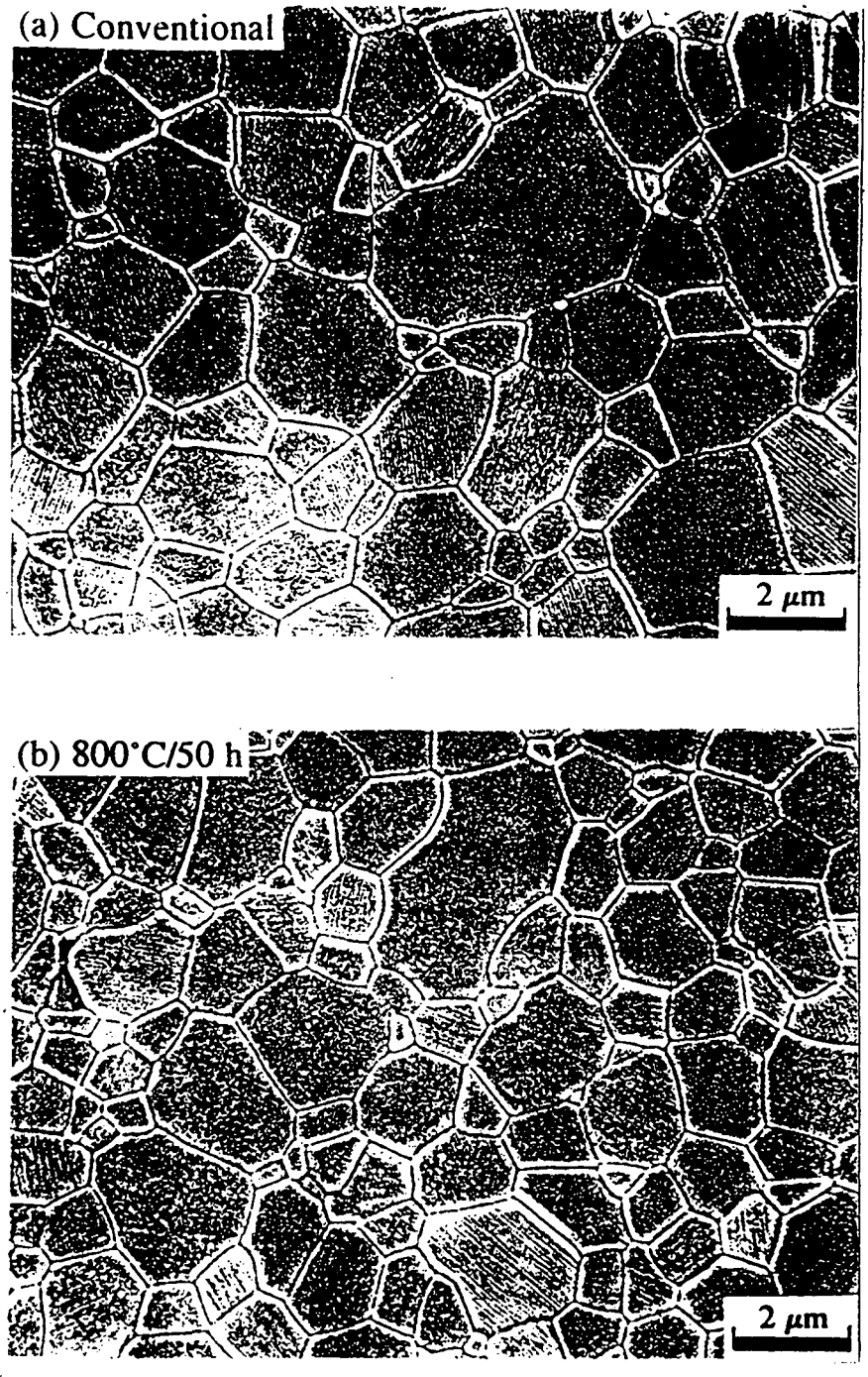

Figure 4

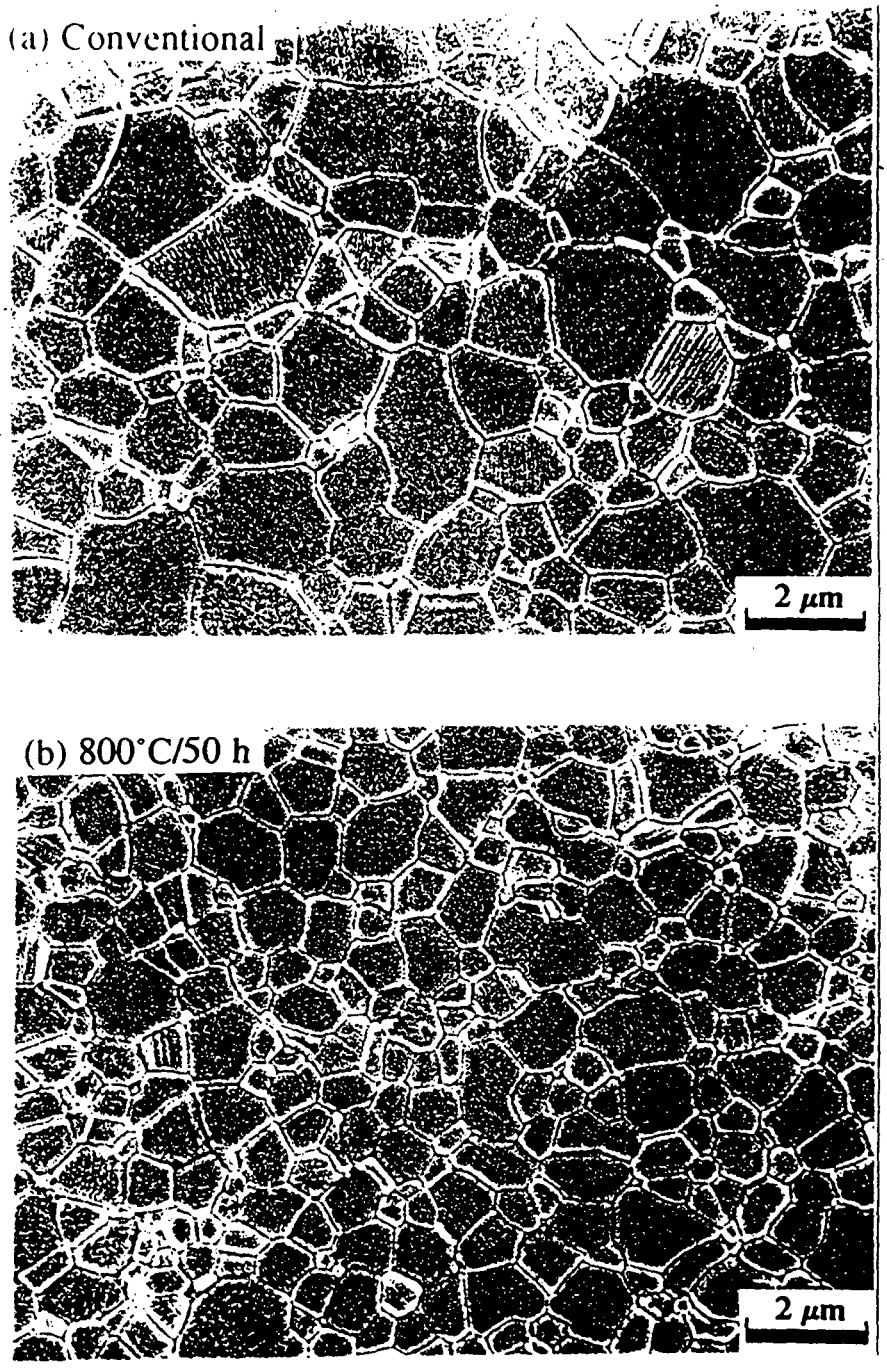

Figure 5

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