# Two-Step Sintering of Mg-Doped Alumina

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## Introduction

Alumina is one of the most widely studied ceramics. The manufacturing process, from green body preparation to dopant selection and sintering conditions, greatly affect the microstructure and properties of these materials. Sintering serves the purpose of promoting densification through facilitated diffusion at high temperatures, which is also accompanied by grain growth.

Chen and Wang[1] proposed a two-stage sintering method, commonly referred to as TSS-CW, aiming to suppress grain growth while still allowing for densification to occur. They attempted this using yttria, barium titanate and Ni-Cu-Zn ferrite and managed to maintain a constant grain size while achieving near-full density[2].

The first part of the two-step sintering regime is to heat the sample up to a high temperature and then immediately lower the temperature with no isothermal dwell. This is done to reach an initial density high enough for further densification at a lower temperature. The second part involves a long isothermal dwell for continued densification by grain boundary diffusion but without grain boundary motion (no grain growth).

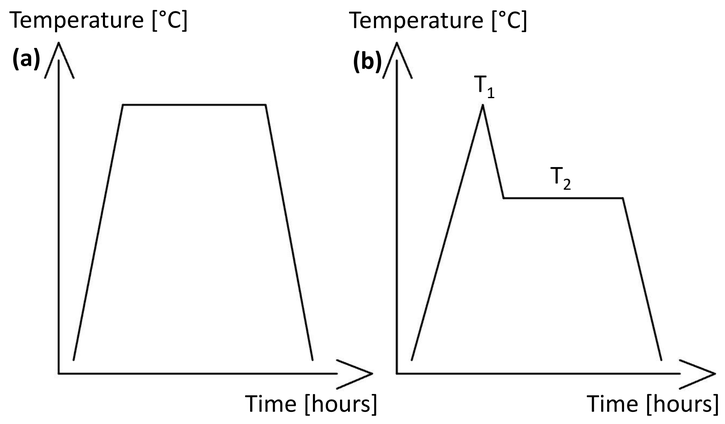



**Fig 1** – The conventional sintering (a) and two-step sintering (b) heating profiles.

## Motivation

The goal of this study was to experimentally determine the optimal temperatures for each stage of the TSS regime for alumina doped with magnesium, which is the most commonly used and efficient grain growth suppressor[3].

The study also aimed to observe the microstructure resulting from two-step sintering at different stages in comparison to normal sintering.

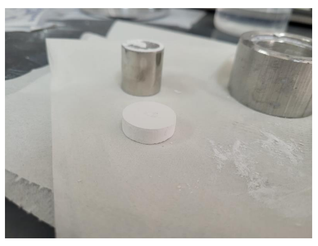
While two-step sintering may not result in densities as high as those achieved via normal sintering and is less cost effective due to longer treatment durations, it usually results in a finer microstructure.

## Experimental Procedure

**Green Body Preparation:**

Mg-doped alumina powder ~300 nm average grain size.

Uniaxial pressing @ 20MPa.



Pressed green body.

Vacuum seal.

Cold isostatic pressing @ 200 MPa.

Sample firing in air @ 650°C, 2 hr & @1000°C, 2hr.

**Sintering:**

Sintering in air.

Heating rate 15°C/min up to $T_1$ = 1400°C, 1450°C, 1500°C.

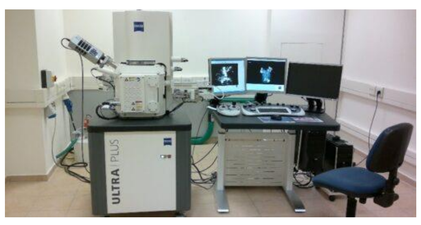
5 min isothermal dwell @ $T_1$

Cooling rate 15°C/min down to $T_2$ = 1150°C, 1200°C, 1250°C.

4 – 24 hr isothermal dwell @ $T_2$

Cooling rate 15°C/min down to room temperature.

One sample was quenched in water upon reaching 1500 °C.




Cutting the samples for density measurements (Archimedes method) and SEM observations of fracture surfaces and polished cross sections.

## Results & Discussion

**Table 1** – Summary of initial results for all samples.

| Sample | $T_1$ [°C] | $T_2$ [°C] | $T_2$ Isothermal Dwell [hr] | Relative Density [%] | Estimated Mean Grain Size [µm] |
|---|---|---|---|---|---|
| #2 | 1400 | 1150 | 4 | 85.9 | 0.4 |
| #3 | 1400 | 1200 | 4 | 87.9 | 0.5 |
| #4 | 1400 | 1250 | 4 | 86.9 | 0.5 |
| #5 | 1450 | 1150 | 4 | 92.9 | 0.7 |
| #8 | 1450 | 1150 | 16 | 94.2 | 0.7 |
| #9 | 1450 | 1250 | 4 | 94.7 | 0.7 |
| Quenched | 1500 | X | X | 96.3 | 0.8 |
| #6 | 1500 | 1150 | 4 | 95.3 | 0.9 |
| #10 | 1500 | 1150 | 16 | 97.3 | 0.8 |
| #7 | 1500 | 1250 | 4 | 97.4 | 1.0 |
| #11 | 1500 | 1250 | 24 | 98.1 | 1.1 |
| Conventional Sintering | 1500 | X | 2 @ $T_1$ | 99.6 | 3.2 |

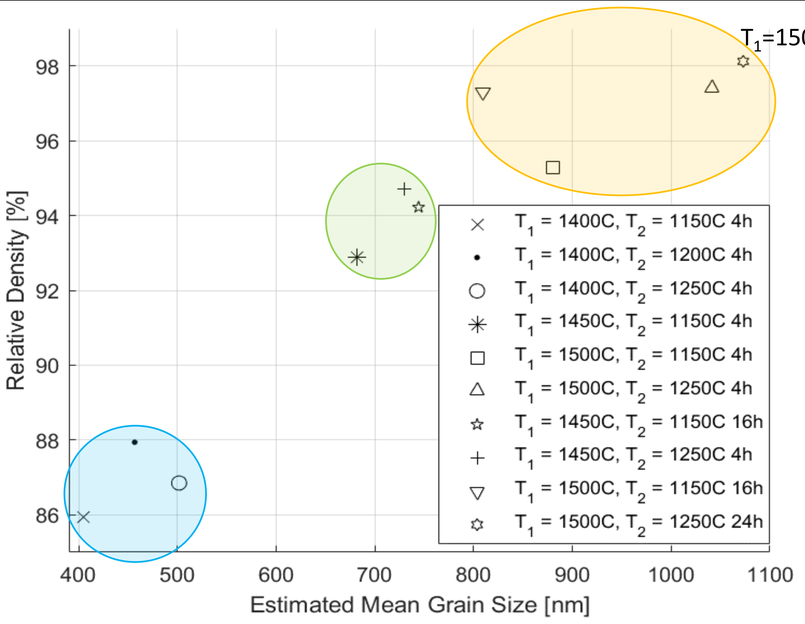



**Fig 3** – Density vs estimated mean grain size of all samples prepared by two-step sintering with a $T_1$ dwell time of 5 minutes.

Figure 3 displays the resulting density and mean grain size estimations from fracture surfaces micrographs for samples that underwent two-step sintering. Samples with a similar $T_1$ are grouped in circles. Samples with $T_1$ = 1400°C and 1450°C appear to not reach sufficient initial densification during the first part of the sintering regime, resulting in relatively low final densities.

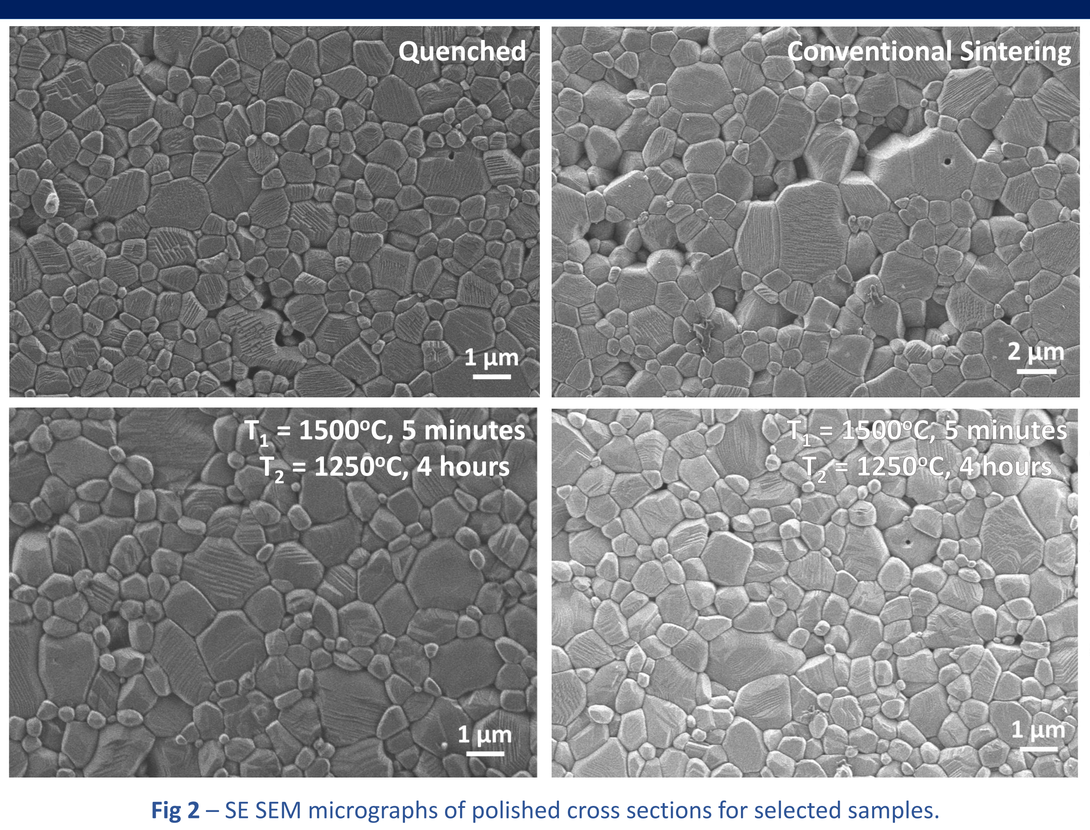



**Fig 2** – SE SEM micrographs of polished cross sections for selected samples.

Figure 2 presents SE SEM micrographs of polished cross sections of four samples. The sample that was quenched upon reaching 1500°C, as expected, is less dense and has smaller grains in comparison to the sample that was sintered at 1500°C for 2 hours and the two samples which underwent further sintering at 1250°C for 4 and 24 hours after the $T_1$ isothermal dwell of 5 minutes.

We can see that the denser, less porous material obtained via conventional sintering has much larger grains compared to the samples that were initially heated up to the same temperature but underwent two-step sintering.

Meanwhile, no significant increase in grain size is observed by extending the dwell time at $T_2$ by 20 hours, indicating that grain boundary motion is indeed suppressed.

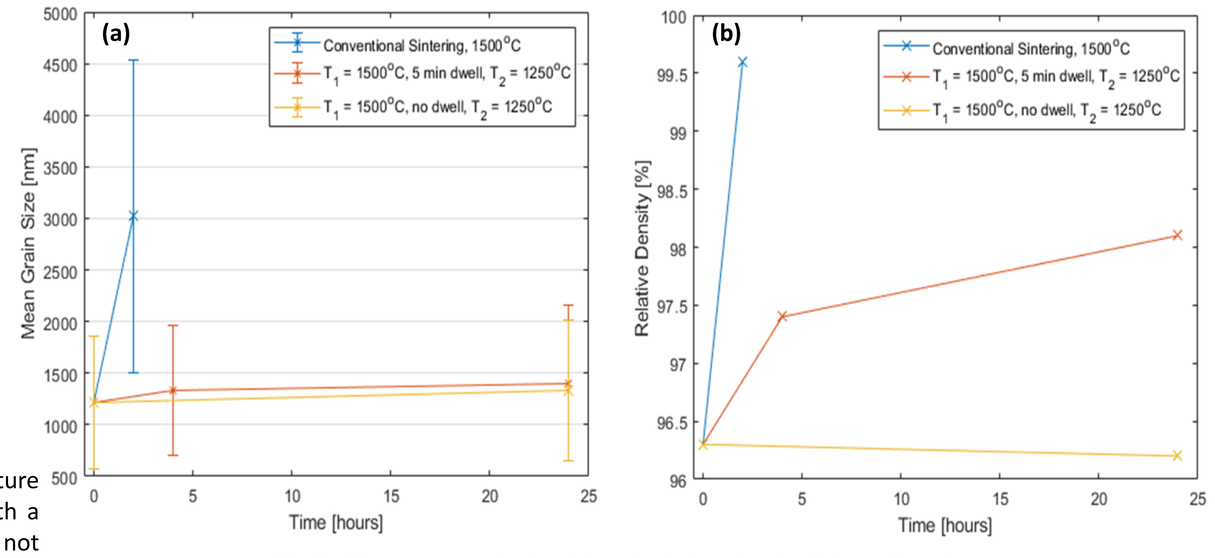



**Fig 4** – The mean grain size (a) and relative density (b) as a function of the isothermal dwell time at $T_2$ of samples with $T_1$= 1500°C.

Figure 4 highlights the resulting density and mean grain size as a function of dwell time at $T_2$ for all samples with $T_1$ = 1500°C, in comparison to the one that was sintered conventionally. The quenched sample was used as a starting point (t = 0).

While figure 4(b) demonstrates the advantage of conventional sintering; providing a higher final density after a much shorter treatment, figure 4(a) demonstrates the finer microstructure achieved by two-step sintering.

Furthermore, little to no additional grain growth takes place as a result of increasing the dwell time at $T_2$.

## Summary & Conclusions

- The optimal temperatures for two-step sintering of Mg-doped alumina using this powder as the raw material are $T_1$ = 1500°C and $T_2$ = 1250°C.
- Two-step sintering does provide a finer microstructure and a slightly lower density compared to conventional sintering by allowing grain boundary diffusion without disconnection activation (grain growth), but is significantly less cost effective.
- Continued isothermal dwell @ $T_2$ = 1250°C is insufficient when the first part of the two-step sintering regime doesn't result in enough initial densification, no matter how long.

## Further Research

- Different temperatures for both parts of the two-step sintering regime, heating/cooling rates and isothermal dwell durations at $T_1$ can be examined. Furthermore, using smaller-sized powder should be considered.
- A similarly executed project can be carried out using alumina with different dopants. Further alterations in sintering conditions can be explored such as different environments other than air.

**References**

[1] Wang, X.-H., Chen, P.-L. and Chen, I.-W. (2006), Two-Step Sintering of Ceramics with Constant Grain-Size, I. Y2O3. Journal of the American Ceramic Society, 89: 431-437.

[2] Wang, X.-H., Deng, X.-Y., Bai, H.-L., Zhou, H., Qu, W.-G., Li, L.-T. and Chen, I.-W. (2006), Two-Step Sintering of Ceramics with Constant Grain-Size, II: BaTiO3 and Ni–Cu–Zn Ferrite. Journal of the American Ceramic Society, 89: 438-443.

[3] Sathiyakumar, M. & Gnanam, F. D. (2003), Infuence of additives on density, microstructure and mechanical properties of alumina. J. Mater. Process. Technol. 133, 282–286.