Densification of alumina by FAST and conventional hot pressing: Comparative study

M. Demuynck

INISMa-U.C. Louvain, Avenue Gouverneur Cornez 4, 7000, MONS (BELGIQUE)
Outline of the presentation

- Introduction: What is FAST?
- Objectives of the thesis
- FAST of insulating materials – Case study: Al$_2$O$_3$
  - Effect of the sintering parameters on grain size and density
  - Hypotheses concerning the mechanisms
- Conclusion
- Perspectives
Field Assisted Sintering Technique (FAST) vs conventional Hot Pressing (HP) (1/2)

- Also known under the acronym
  - SPS (Spark Plasma Sintering)
  - PECS (Pulsed Electric Current Sintering)
  - PAS (Pulsed Activated Sintering)
  - ...
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- Similarities
Field Assisted Sintering Technique (FAST) vs conventional Hot Pressing (HP) (2/2)

Main differences: heating modes and heating rates

- FAST: Joule effect → 1000°C/min (250°C/min)
- HP: inductive heating → 150°C/min (100°C/min)
- HP: resistive heating → 50°C/min (10°C/min)

With the equipments used in this study
Field Assisted Sintering Technique (FAST) vs conventional Hot Pressing (HP) (2/2)

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  - Applicable to a broad range of materials → conductive or **insulating**
  - Short thermal cycles and lower T → more economical  
    → microstructure control,…
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- **FAST limitations**
  - Mechanisms ??
  - Simple shapes and small dimensions

*With the equipments used in this study*
Objectives of the PhD

- **Evaluation of the real advantages and limitations of FAST**
  - Comparing with conventional uniaxial hot pressing (HP)
  - Effect of the sintering parameters on density and grain size

- **Try to identify the mechanisms**
  - More particularly in the FAST sintering of alumina (FAST = « very rapid hot pressing » or real effect of the technique on the sintering of insulating materials?)

- **Influence of thermal and electrical properties**
  - Sintering of composite materials (without additives)
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Al₂O₃: Sintering conditions and densities

- **Temperature:** 1300 → 1600°C
- **Dwell time:** 2 or 6 min
- **Load:**
  - 16 – 32 – 48 MPa
  - 16/48 MPa
- **Heating rate:**
  - FAST: 10 → 250°C/min
  - HP inductive: 20 → 100 °C/min
  - HP resistive: 2 → 10 °C/min
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- **All samples are fully dense**
  - except those sintered at 1300°C for 6min under a load of 16MPa

<table>
<thead>
<tr>
<th>1300°C 6min 16MPa</th>
<th>Heating rate °C/min</th>
<th>Relative density %</th>
</tr>
</thead>
<tbody>
<tr>
<td>FAST</td>
<td>10</td>
<td>92-98</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>83-98</td>
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<td>50</td>
<td>82-97</td>
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<td>150</td>
<td>91</td>
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<tr>
<td></td>
<td>200</td>
<td>94</td>
</tr>
<tr>
<td></td>
<td>250</td>
<td>88</td>
</tr>
<tr>
<td>HP resistive</td>
<td>2</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>69</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>68</td>
</tr>
<tr>
<td>HP inductive</td>
<td>20</td>
<td>68</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>69</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>72</td>
</tr>
</tbody>
</table>

*FAST: pulses 10ms “on” – 5ms “off”*
Effect of temperature and dwell time on grain size ($\rho > 99\%$)
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Al$_2$O$_3$ (P172SB) - Grain size

Effect of temperature and dwell time

Higher $T$ → Higher density at the beginning of the dwell
Effect of heating rate and heating mode on grain size ($\rho > 99\%$)

- $t_{\text{min}}$ for $\rho_{\text{rel}} > 99\%$
- Design
Effect of heating rate and heating mode on grain size ($\rho > 99\%$)

$t_{\text{min}}$ for $\rho_{\text{rel}} > 99\%$ increases with lower heating rate
Effect of heating rate and heating mode on grain size ($\rho > 99\%$)

**Al$_2$O$_3$ (P172SB)**

- 2°C/min
- 5°C/min
- 20°C/min
- 50°C/min
- 150°C/min
- 200°C/min

Constant load of 16MPa

Grain size ($\mu$m)

1500°C

Relative density (%)

$\text{t}_{\text{min}}$ for $\rho_{\text{rel}} > 99\%$ increases with higher heating rate
Effect of heating rate and heating mode on grain size ($\rho > 99\%$)

Al$_2$O$_3$ (P172SB)

Constant load of 16MPa

1500°C

Grain size ($\mu$m)

$t_{\text{min}}$ for $\rho_{\text{rel}} > 99\%$: no significative influence of heating rate
Effect of applied load on grain size

Grain size increases if higher applied load

![Graph showing the effect of applied load on grain size. The graph compares grain size at different heating rates and loads. The y-axis represents grain size in micrometers, and the x-axis represents heating rate in °C/min.]
Effect of applied load on grain size

Grain size increases if higher applied load

$\tau_{\text{min}}$ for $\rho_{\text{rel}} > 99%$
Sintering trajectories (Grain size vs $\rho_{\text{rel}}$)
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Sintering of alumina

Grain size ($\mu$m)

Relative density (%)
Sintering trajectories (Grain size vs $\rho_{rel}$)

FAST/HP: Same sintering trajectory

→ Importance of a good parameters optimisation

According to Langer et al., Acta Materialia 57, 2009
Effect of the sintering technique on density

- 1300°C – 16MPa – 6min $\Rightarrow \rho_{\text{rel}} < \rho_{\text{max}}$
- FAST $\Rightarrow$ Dispersion $\Rightarrow$ effect of heating rate??

Sintering of $\text{Al}_2\text{O}_3$ (P172SB) - 6min - 16MPa:
Effect of heating rate and heating mode

Final relative density (%) vs. Heating rate (°C/min)
Effect of the sintering technique on density

- $1300^\circ C - 16\text{MPa} - 6\text{min} \rightarrow \rho_{rel} < \rho_{max}$
- FAST $\rightarrow$ Dispersion $\rightarrow$ effect of heating rate??
- $\rho_{\text{FAST}} > \rho_{\text{HP}}$

Effects of the electric field?

![Graph showing the effect of heating rate and heating mode on final relative density. The graph compares different heating rates and heating modes (FAST, HP inductive, HP resistive) with varying final relative densities.]
Potential effect of the electric field

Enhancement of surface phenomena like surface diffusion

How?

→ Modification of energies and interface kinetics

Ghosh et al., J. Amer. Ceram. Soc. 92 [8], 2009, pp. 1856-1859
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Surface diffusion

→ non-densifying mechanism (does not induce shrinkage)

→ BUT can influence densification (formation of sintering necks)
Potential effect of the electric field

Enhancement of surface phenomena like surface diffusion

- Surface diffusion at lower T:
  - sintering necks formation
  - positive effect on densification

- Surface diffusion at higher T
  - Grain growth
  - Negative effect on densification

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Effect of electric field at lower T

- Interrupted sintering cycles
- Measurement of $\alpha$ at $T_{\text{amb}}$

L. Stanciu et al., J. Amer. Ceram. Soc. 90 [9], 2007
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\[ \alpha \rho = A \frac{K_0}{C_p} \exp \left( -\frac{Q}{nRT} \right) \]

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- Influence the formation of sintering necks

Effect of electric field

- Electromigration observed on conductive materials
  
  Bradbury and Olevsky, Scripta Materialia 63 (2010)
  Olevsky and Froyen, Scripta Materialia 55, 2006
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Yang and Conrad, Scripta Materialia 41 (4), 1999
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Effect of electric field
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Role of impurities
- Apparition of charges at the particles surfaces
  - $\sigma_{\text{surf}} > \sigma_{\text{bulk}}$
  - Small currents but no Joule effect

- Enhancement of diffusion mechanisms and sintering necks formation
Conclusion

- **FAST of insulating materials:**
  - Grain size increases if higher sintering temperature, higher load or longer dwell time (like in conventional sintering)
  - No direct effect of the current for insulating materials
  - Effect of the electric field (enhancement of the mobility at interfaces and particles surfaces, grains rearrangement,...)

- **Mechanisms: enhancement of surface phenomena**
  - Advantage: the surface diffusion at lower temperature enhance the formation of sintering necks
Perspectives

FAST has an positive influence in the sintering of alumina through the presence of an electric field BUT:

- Do we have the same effect whatever the insulating material?
- What are the relative influences of:
  - the powder properties (grain size, purity,…)
  - the intensity of the electric field
  - ...

in the enhancement of the sintering of insulating materials by FAST?
Thank you for your attention

Questions?

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\textsuperscript{1} INISMa-CRIBC, Avenue Gouverneur Cornez 4, 7000, MONS
\textsuperscript{2} K.U.Leuven, Kasteelpark Arenberg 44, 3001, HEVERLEE
\textsuperscript{3} U.C.Louvain, Place Sainte Barbe 2, 1348, LOUVAIN-LA-NEUVE