FROM WET SPONGES TO OPTOCERAMICS

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T02: High Temperature Processes and Advanced Sintering

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Topics of discussion

- Context and objective
- Growing and modifying the “sponges”
- Consolidation – spark plasma sintering
  - Characterization of ceramics
  - Conclusions and perspectives
Motivation

To control the grain size of ceramics on our way to self-healing nano-grained transparent ceramics

Applications as optical windows in nuclear (fusion) power plants, transparent armors, nose cones for heat seeking missiles (YAG), space engineering, medicine ...
Growing alumina monoliths*

99% porosity of the monolith makes it easy to dope by gas or liquid

1 cm/h = 0.5 g (43 m% water)

80% humidity, 25°C, cooling plate 23°C

Al plate + Ag-Hg amalgam -> Al(OH)₃ monolith


Spark plasma sintering

- Vacuum or argon
- Conductive or not
- 3.5 – 50 kN
- 20 – 2000°C
- Sample displacement

- Joule effect
  => fast process, grain size control
- High temp + pressure
  => dense materials

![Graph showing displacement and pressure over time](image)

**Dr. Sinter LAB Series SPS-515S**

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Alumina ceramics

From previous work in LSPM*

Powder crystallite size: 10 nm

Powder crystallite size: 50 nm

Although our γ-alumina powder crystallite size is smaller than the commercial powder’s, the sintered ceramic has bigger grain size

Effect of green body preparation

SPS cycle: 3’ at 1450°C, 300 MPa

our alumina, no pre-pressing

$\rho = 96.6\%$

Pre-pressing with 4t for better densification leads to a tenfold increase in grain size

our alumina, pre-pressing with 4t

$\rho = 98.6\%$
XRD texture study

5° × 5° grid with 0° ≤ χ ≤ 55° and 0° ≤ φ ≤ 355°

As received from SPS

Post-annealing at 1000°C

- Mild fibrous texture aligned with SPS pressure-axis
- Crystallite shape “spherifizes” during annealing
Mullite, spinel – reactive sintering

SPS cycle: 20’ at 1450°C + 10’ at 1150°C, 100 MPa
Our alumina doped with TMES in gas phase OR in liquid phase with Mg(NO$_3$)$_2$·6H$_2$O

- Stoichiometric Al$_2$O$_3$:MgO ratio gives monophasic spinel ceramic
- Average grain size of spinel produced by reactive sintering is about 5 times larger than that of spinel produced from commercial powder via the same cycle.
Cathodoluminescence study

MgAl$_2$O$_4$ ceramics compared to a single crystal*

- 1.8 eV -> the R-lines of Cr$^{3+}$ impurity**
- 2.4 eV -> tetrahedral Mn$^{2+}$ impurity**
- 2.4 – 4 eV -> F, F$^+$ centres + complex intrinsic defects***
- 4.5-6.5 eV -> “anti-site” defects**

Conclusions

• Dense ceramics with various grain sizes and transparencies, starting from UPA
• Starting from a phase with smaller crystallite size does not insure a smaller-grained ceramic
• Pre-pressing leads to a tenfold increase in grain size
• The spinel samples need annealing in air to get rid of coloration
• Slight fiber-like texture aligned with the pressure axis of SPS, crystallites more spherical after annealing
• High inversion level for spinels and common impurities
Thank you for your attention!

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