Ultrahigh Temperature Microwave Processing

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Troy, New York

36th International Conference on Advanced Ceramics and Composites
Symposium 8: SPS and Microwave Assisted Technology

Daytona Beach, Florida
4:20 PM  January 24, 2012
Outline

- Goals
- UHT Microwave Processing
- Dielectric Properties & Modeling
- UHT Thermal Package Materials
- Ceralink UHT Microwave Experiments
Goals

- Quickly process UHTCs, in any shape
- Processing over 1700 C
- Dense, net shape UHT ceramics
- Eliminate complex microwave thermal packages
- Develop first UHT Microwave Assist Technology furnace
UHT Microwave Processing

- Penn State - single mode system
  - No susceptor used
  - Single mode limited to small samples

- University Maryland - multi mode
  - Buried samples in powder bed of AlN + TiC
  - Thermal insulation package approach

- Shanghai Institute of Ceramics
  - Boron nitride insulation materials
  - Multimode, thermal package based processing

- Oak Ridge National Laboratories
  - Various thermal package studies on UHT ceramics

- University of Bayreuth
  - Molybdenum Hot-Wall Microwave Assist Technology furnace
  - Rated to 1500 °C
High Temperature Microwave Processing

Will benefits of fine grain size be achieved for UHTCs?

Conventional Sintered Alumina Ceramic ~1600 °C  Microwave Sintered
UHT MW Dielectric Properties

- No one can measure them!
  - Highest T possible at Microwave Properties North and University of Nottingham, ~1450 °C
- We measured the refractory oxides to the limit of 1400 °C

$$D_{HP} = \frac{\ln 2}{2} \alpha^{-1} = \frac{e \ln 2}{2(2\pi f)\varepsilon_0} \left( \frac{2}{\sqrt{1 + (\tan \delta)^2} - 1}\varepsilon' \right)^{\frac{1}{2}}$$

Will dielectric materials become too conductive?

Can refractory metals become resistive enough to absorb?
UHT Microwave Dielectric Properties

- We can extrapolate dielectric loss and permittivity data to higher temperatures, above intrinsic conductivity.
- This case for zirconia fiber insulation.

\[ \varepsilon''_r = \frac{\sigma_{\text{intrinsic}}}{\omega \varepsilon_0} e^{-\frac{-E_g}{2k_B T}} \]

![Graph showing dielectric properties](image)
Microwave Modeling, QWED®

- Used commercial QWED QuickWave-3D software
- With Dr. Vadim Yakovlev of WPI
- Explored E-field distribution in MAT furnace
  - Isothermal slices
  - Calculated power dissipation
- Developing MAT capability
Modeling, MATModel™

- Developed with RPI
- Uses GiD® solid modeler
- Compared alumina vs yttria insulation
- Programmed electric element heating profile
- Imported dielectric & thermal properties of materials

Models followed
Identical MAT Heating profile and MW power input

Alumina-silica Insulation (Zircar Ceramics)

SiC

Yttria felt insulation (Zircar Zirconia)
Microwave Equipment
CPI Autowave – Microwave-only System

- 2-color optical pyrometer up to 3,000 C
- Vacuum/inert atmosphere
- 6 kW, 2.45 GHz power
- Oxygen and dewpoint sensors
Ceralink Thermal Package Design
Ultra High Temperature – 2,000 °C+

Needs
- To withstand temperatures
- Be microwave transparent

Boron nitride
Magnesium oxide
Yttrium oxide
Ceralink Ultrahigh Temperature Susceptors

Bonded SiC susceptor
- Reliable to 1550 °C
- SiO creates thermal package problems at UHTs

ZrO$_2$-SiC susceptor
- Heats to 1900 °C+ in air, nitrogen, and argon
- Silica from SiC vaporizes, contaminates
- Developed encasement technique
- Zirconia reduces or nitrides

MgO-ZrC susceptor
- ZrC susceptible to oxidation >~300 °C
- ZrC UHT oxidation by-product is benign, oxygen getter
- Easily heats to >1900 °C in nitrogen and argon
Thermal Package Example
Commercial AlN

Properties
- 3.31-3.33 g/cc density
- 115 W/m-K
- 1-2 µm grain size
- 0.02 tan delta (2-10 GHz)

MW vs. conventional:
- Equivalent density
- Finer grain size → Lower thermal conductivity
Thermal Package Example
AlN + CNT (Florida International University)

Microwave sintered AlN-CNT
90-95 % Dense
Finer grain size

Spark plasma sintered AlN-CNT
85 % Dense
Thermal Package Example
Reaction Bonded Silicon Carbide

- $\text{Si}_m + \text{C} \rightarrow \text{SiC} + \text{Heat}$
- Carbon preform
- Boron nitride crucible
- Alumina-mullite insulation

- Melt silicon granules, over 1410 °C
- Strong exotherm
Reaction Bonded Silicon Carbide

Exothermic reaction results in instantaneous $1530 \, ^\circ C \rightarrow 2662 \, ^\circ C$

Enthalpy -68 kJ/mol

Currently NSF Phase I SBIR Supported
Next Steps – UHT MAT

- Graphite furnace with Microwave Assist Technology
  - Beginning stages of furnace design
  - NSF Phase II SBIR
- Removes Thermal package from process
- Utilizing existing UHT furnace technology
- Provides a scale-up and manufacturing opportunity
Summary

- UHT Microwave limited by lack of systems
- Limitations to dependence on thermal package
  - Small samples
  - Significant materials challenges
- Build UHT microwave with existing UHT technology
- Benefits of microwave for UHT
  - Many questions remain!
Acknowledgments

Sponsors
National Science Foundation
   SBIR Phase I & II, UHTCs
   SBIR Phase I, Microwave RBSC

New York State Energy Research and Development Authority

Collaborators
Microwave Properties North
   Dr. Ron Hutcheon

Worcester Polytechnic Institute
   Dr. Vadim Yakovlev

Rensselaer Polytechnic Institute
   Dr. Jeff Braunstein

Florida International University
   Dr. K.H. Wu

Materials Focus Inc.
   Lori Bracamonte