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Solid State Studies in Ceramics

# Coupled Experimentation and Computer Modeling To Determine the Friction at a Ceramic-Ceramic Interface During Sintering

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## Background

- Unique devices such as fuel cells and micro-electronic packages require co-firing and/or a combination of different materials
- The character of the interfaces is critical to both processing and performance
- Quantifying interface properties experimentally is very difficult or impossible
- A combination of experiments and finite element simulations allows studying interfaces during sintering

## Experimental Procedure

### Materials

- 0.2 μm ZnO (Aldrich, # 255750), PVA/PGA binder
- Sintered 99% pure Al2O3 rods, cut in disks

### Preparation

- Uniaxial pressing of ZnO green bodies
  - Cylinders ($\rho_0$ = 0.57): $\sigma_z$ = 138 MPa
  - Toroids ($\rho_0$ = 0.47): $\sigma_z$ = 31 MPa

### Sintering

- Samples fired in a box furnace in air
- Oven curve: 1000°C @ 5 K/min with 30 min hold @ 550°C

## Skorohod-Olevsky Viscous Sintering (SOVS) Constitutive Model *

### Key Equations

Linear viscous case of the SOVS constitutive relationship

$$\dot{\varepsilon}_{ij}^{in.} = \frac{\sigma'_{ij}}{2\eta_0\varphi} + \frac{\sigma_{kk}/3 - P_L}{3(2\eta_0\psi)}\delta_{ij}$$

Shear Viscosity

$$\tilde{G} = \eta_0\,\varphi$$

Bulk Viscosity

$$\tilde{K} = 2\eta_0\,\psi$$

Normalized viscosities, φ and ψ and the Laplace pressure, PL (sintering stress) are functions of the instantaneous relative density, ρ.

Norm. Shear Viscosity

$$\varphi = 1.12\,\rho^{1.26}$$

Norm. Bulk Viscosity

$$\psi = \frac{2}{3}\frac{\rho^{2.26}}{(1-\rho)^{1.12}}$$

Laplace Pressure

$$P_L(\rho) = 1.7\frac{3\alpha}{r_0}\rho^{0.26}$$

* E.A. Olevsky, "Theory of Sintering: From Discrete to Continuum," Mater. Sci & Eng., R23, 41-100 (1998).
** Reiterer, M.W., Eswuk, K,G., Arguello, J.G., "An Arrhenius-Type Viscosity Function to Model Sintering Using the Skorohod-Olevsky Viscous Sintering Model within a Finite Element Code", *J. Am. Ceram. Soc.* **89** 1930-1935 (2006).

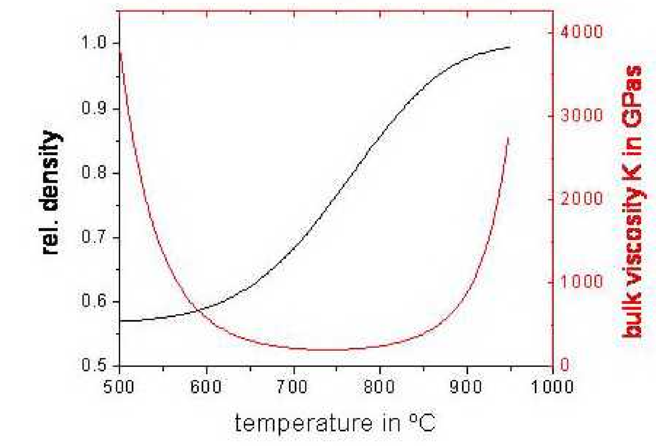
## Newly Proposed Function $\eta_0$ (T) **

**Coble Creep Law**

$$\dot{\varepsilon} = \sigma/\eta_0 = \frac{14\Omega}{kTd^2} D_{GB,0}\, e^{\frac{Q_{GB}}{RT}}\sigma$$

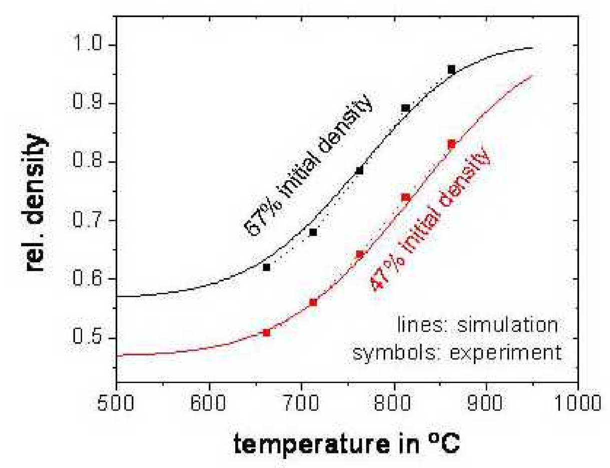
**Viscosity of the dense material's skeleton**

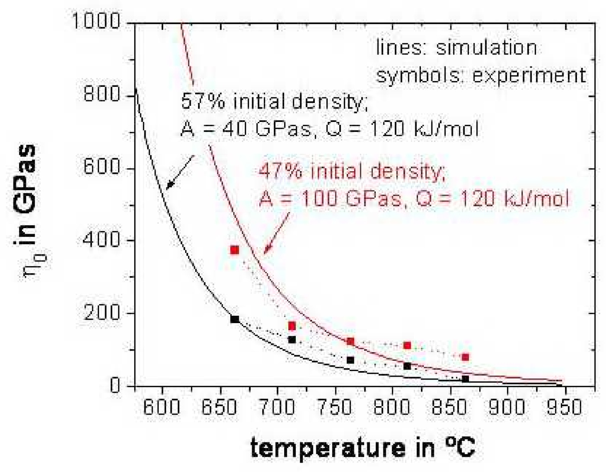
$$\eta_0(T) = A\,T\,e^{\frac{Q}{RT}}$$

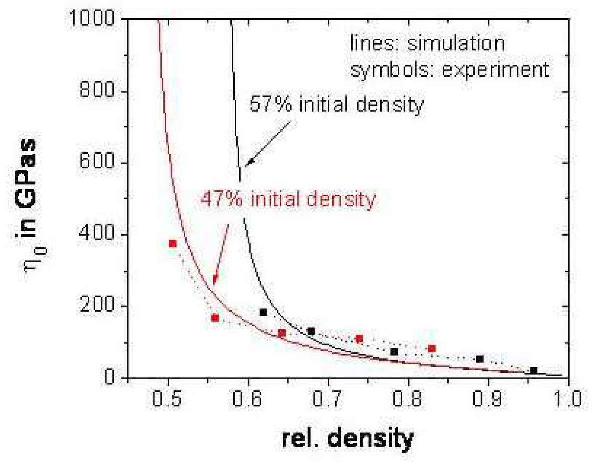



The parameters A and Q have been fitted manually to the sintering process of green bodies with 2 initial densities under consideration of the evolution of ρ (T) and $\eta_0$ (T)

The values of the A and Q are discussed in Ref.**

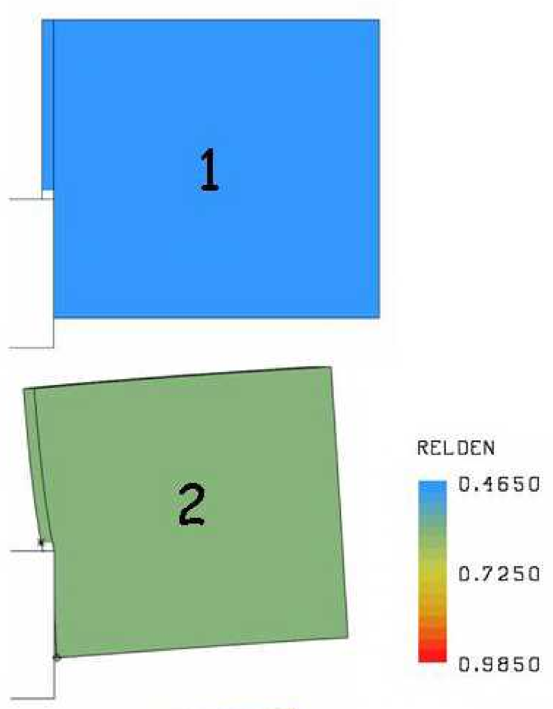


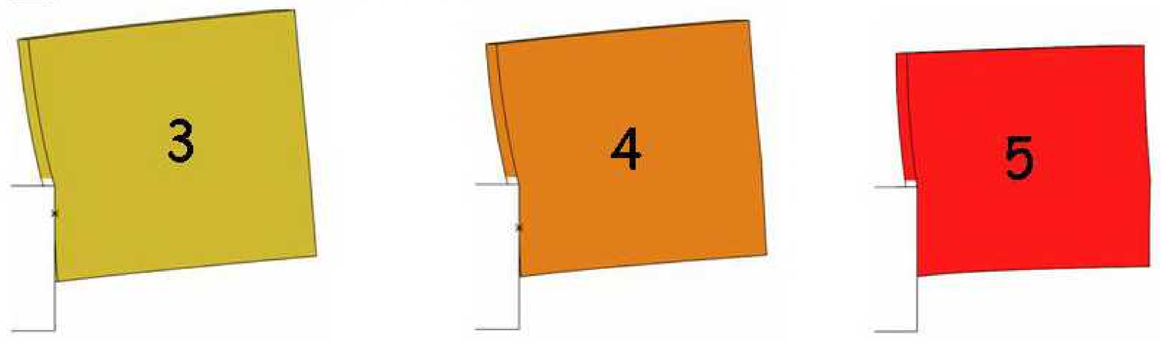



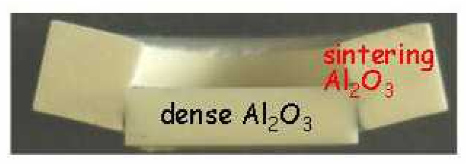



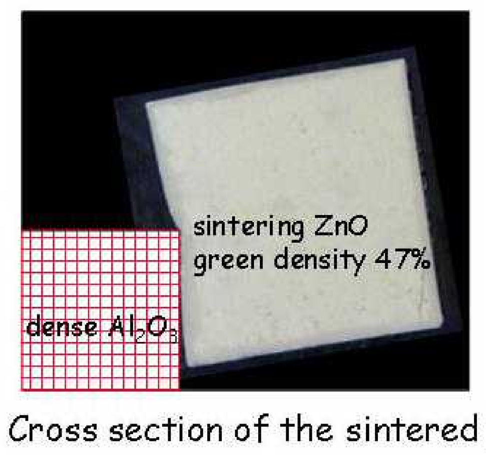

## Sintering of a Toroid with a Semi-Filled Dense Core

### Deformation and Contact Opening Due to Partial Constraining



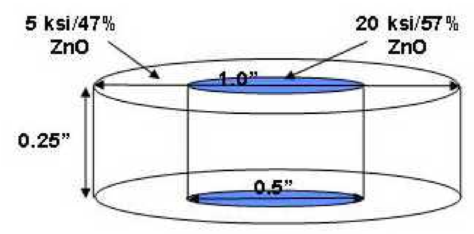



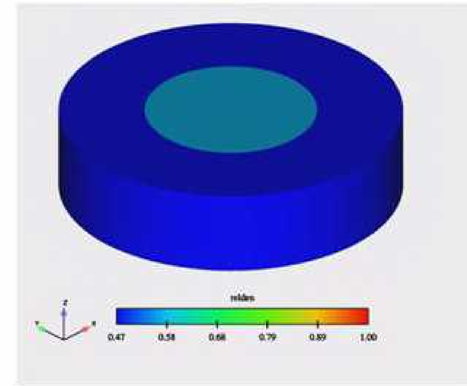

Cross section of the sintered part

*Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lookheed Martin company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000*

## Sintering of a Bidensity Concentric Cylinder

### Comparison of Experimental and Numerical Results



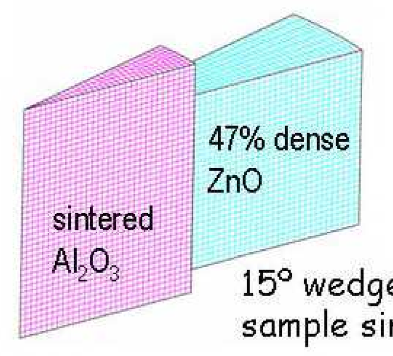



**Garino's Experiment vs. Simulation**
$\rho_0$(toroid) = 0.47 & $\rho_0$(cylinder) = 0.57

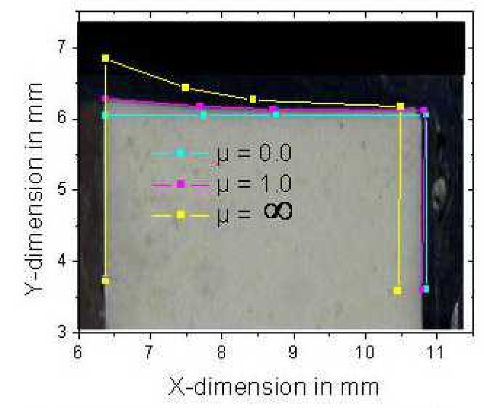
| At 925 °C | Experiment (% Shrinkage) | Simulation (% Shrinkage) | Difference |
|---|---|---|---|
| Di (12.7 mm) | 16.7 | 17.0 | 1.6% |
| Do (25.4 mm) | 19.8 | 19.3 | 2.7% |
| Ti (6.35 mm) | 15.7 | 14.1 | 10.2% |
| To (6.35 mm) | 21.0 | 20.5 | 2.4% |

## Sintering of a Toroid with a Dense (Rigid) Core

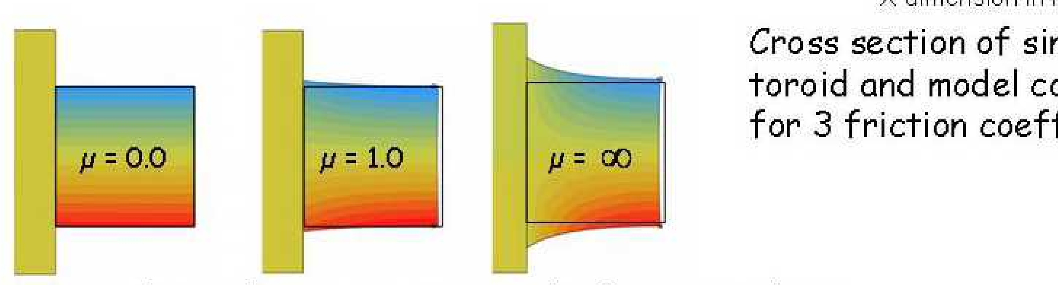
### Influence of Friction Coefficient Final Shape




15° wedge of the sample simulated




Cross section of sintered toroid and model contour line for 3 friction coefficients μ




FE result: Final cross section; z-displacement shown

## Summary

*Co-firing and a combination of different materials are required to fabricate unique devices such as fuel cells and micro-electronic packages. The character of the interfaces in a multi-material system is critical to both processing and performance; however, quantifying interface character can be difficult. In this study, a combination of experimentation and modeling was employed to determine the coefficient of static friction at a ceramic-ceramic interface during sintering. Experimentally a dense, 1.27 cm diameter alumina cylinder was mechanically inserted into the 1.27 cm inner diameter of a toroid-shaped green powder compact, and the sintering behavior of the 47% relative density, 0.2 μm average particle size ZnO toroid was characterized and simulated. Sintering simulations were made using an improved Arrhenius-type viscosity function and the Skrorohod-Olevsky Viscous Sintering (SOVS) model, as implemented within the Sandia National Laboratories JAS3D finite element (FE) code. Comparisons between the shrinkage, density, and deformation measured experiment and predicted by modeling were then used to approximate the coefficient of static friction between the sintering toroid and the rigid alumina cylinder. Excellent agreement between the experimental and numerical results was observed, and a friction coefficient of ~1 was determined for the ZnO-alumina interface. The results demonstrate the feasibility and value of a coupled experimental and numerical modeling approach to define the characteristics of interfaces during sintering in complex multi-material systems.*

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*The first author gratefully acknowledges Medtronic's support to attend the 2006 Gordon Research Conference in Solid State Studies in Ceramics. Medtronic is the global leader in medical technology - alleviating pain, restoring health and extending life for millions of people around the world.*