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# Sintering of Heterogeneous Bodies

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# Motivation

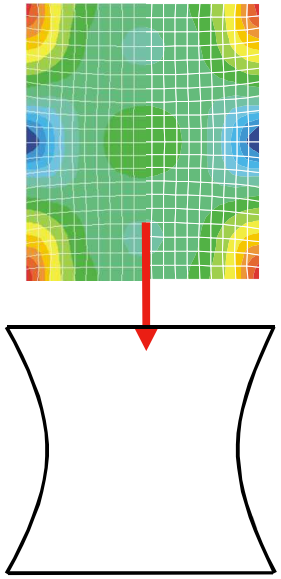
## The Problem:

## Predictive Densification Modeling

- Compaction model is highly advanced
- Current sintering model is highly generalized
  - Grain & pore size, & pore separation
  - Microstructure heterogeneities

  } current models insufficient
- Models are not linked

density distribution in green compact



shape after sintering

## The Solution *(partial):*

- Determine weakness of current model
- Develop a detailed constitutive numerical model

Numerical Model → Finite Element Model

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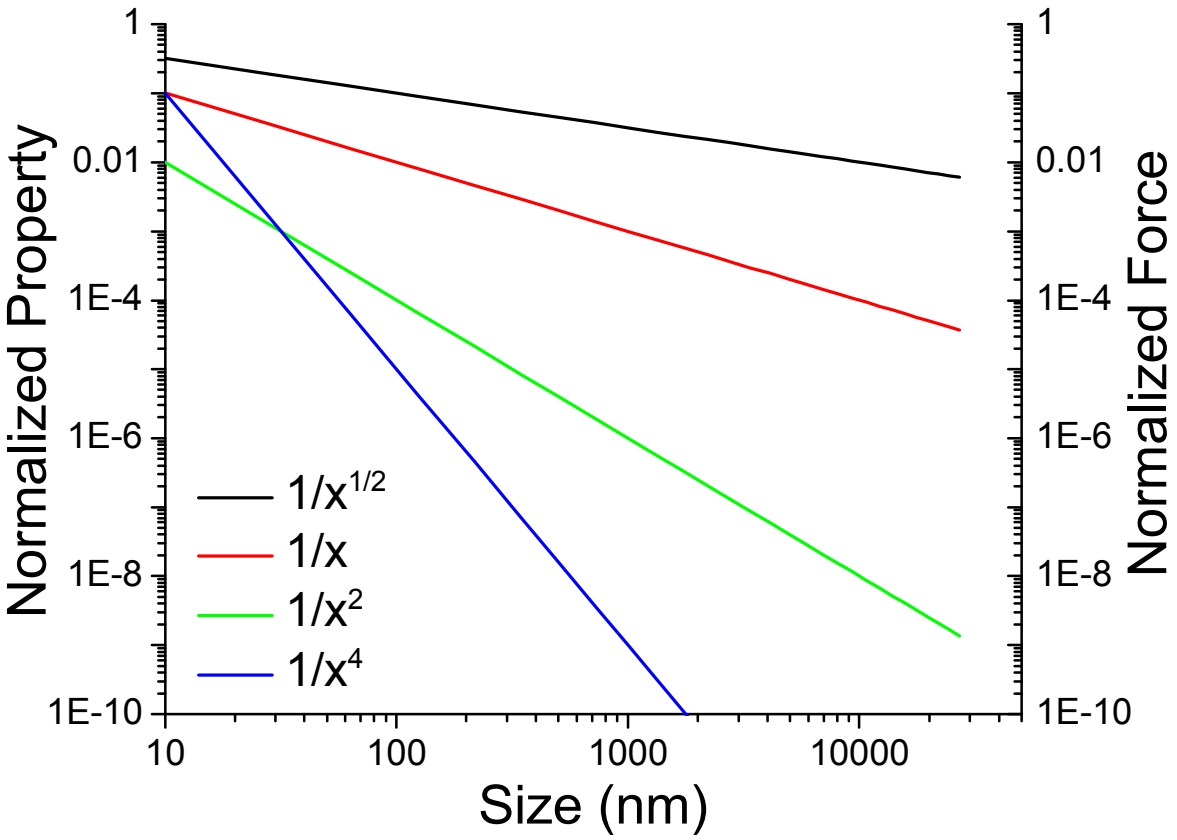
# The factors contributing to heterogeneity development increase as the particle size is reduced

| Property | Force |
|---|---|
| Strength | Sintering Stress |
| Fracture Toughness | Sintering Rate |
| Resistance | Van der Waals |
| Creep | Compaction Friction |

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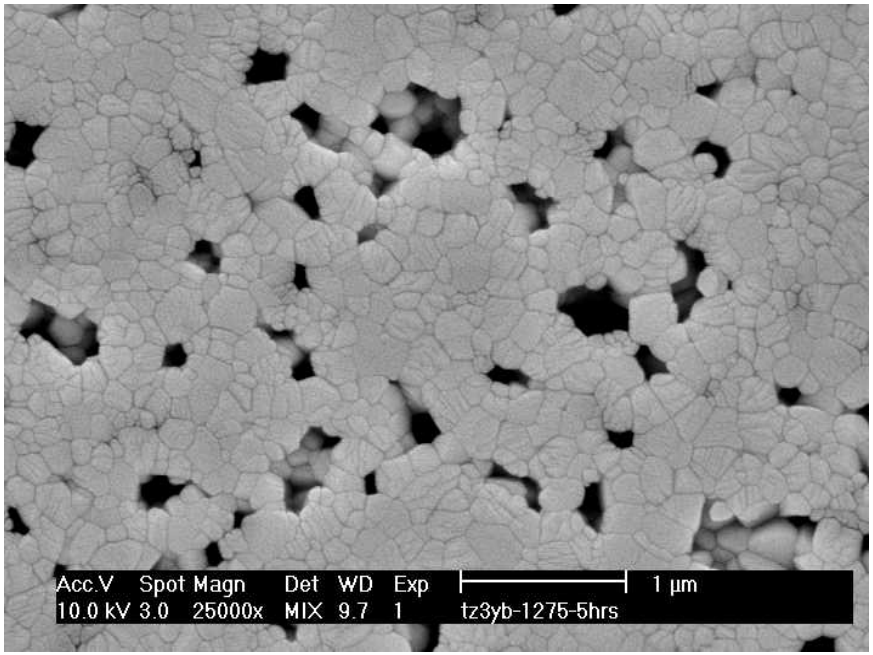
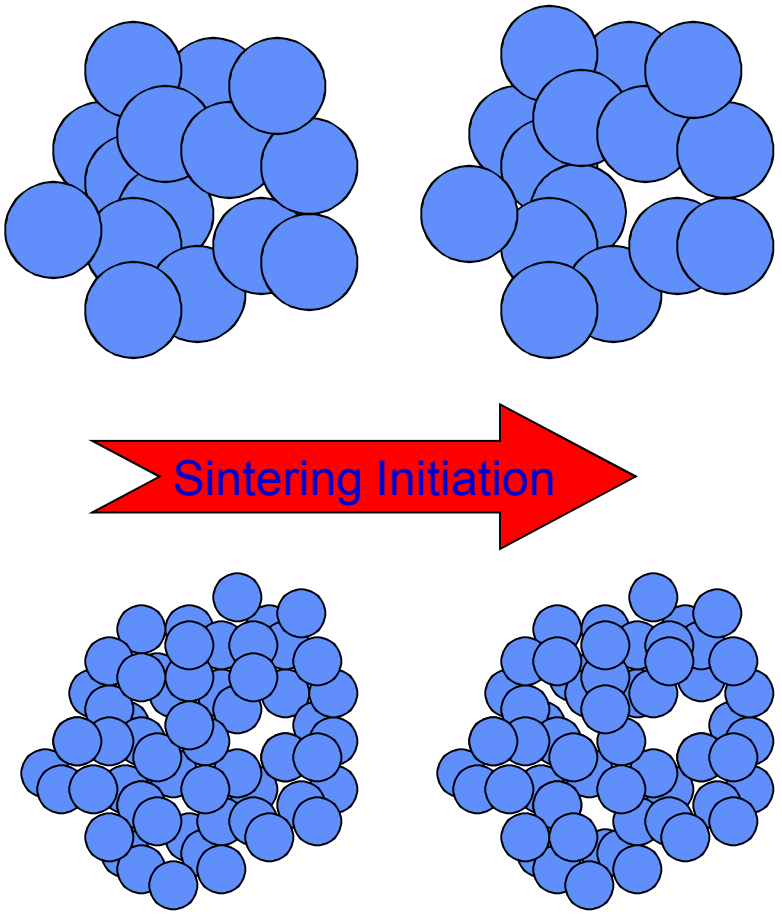
# Packing and forming defects result in a heterogeneous microstructure

## Heterogeneity Origins

- Density gradients
- Particle size distribution
- Agglomerates

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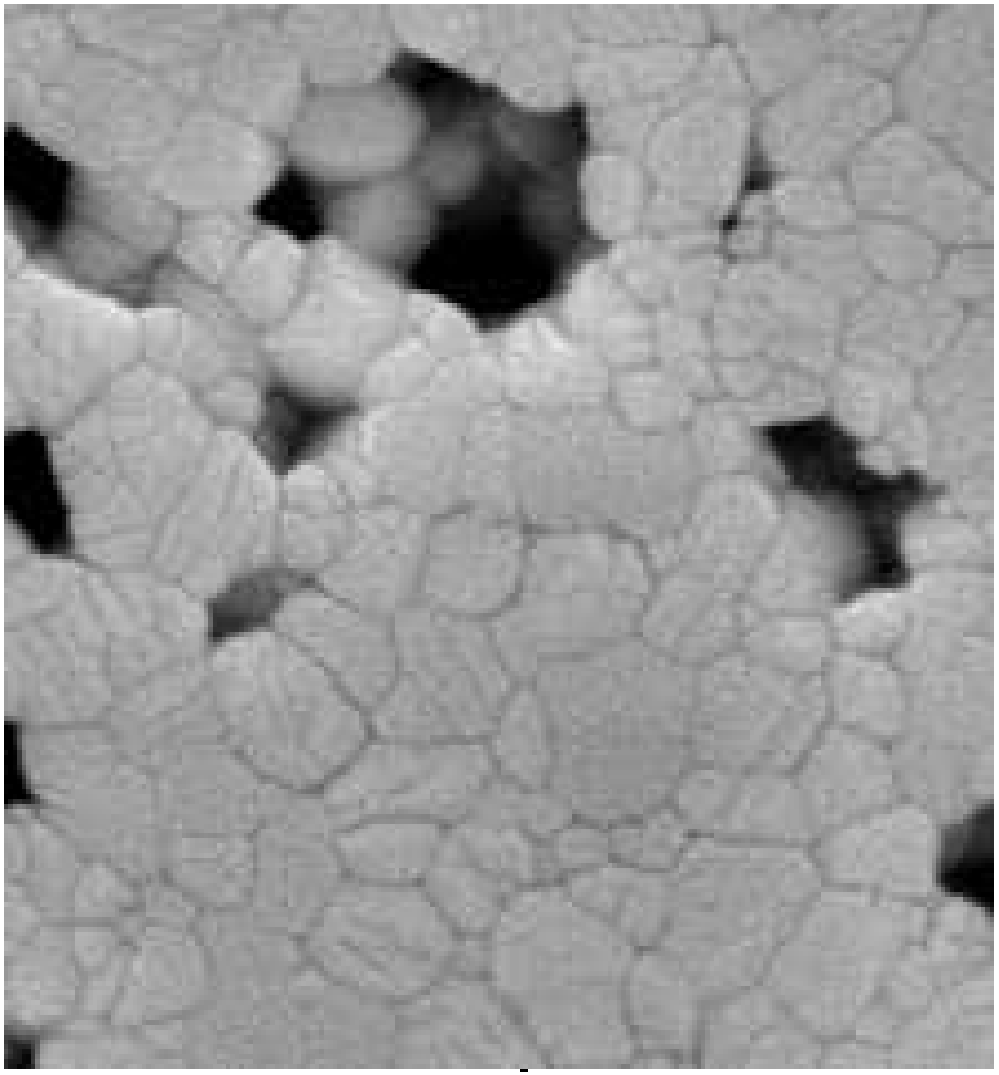
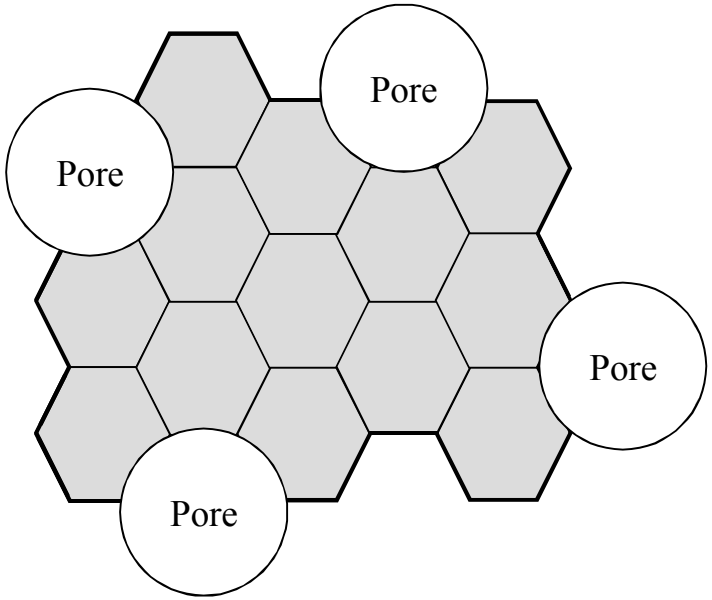
# Sintering models do not address the heterogeneity in real microstructures



Real




$$\frac{\dot{\rho}}{\rho} = ?$$



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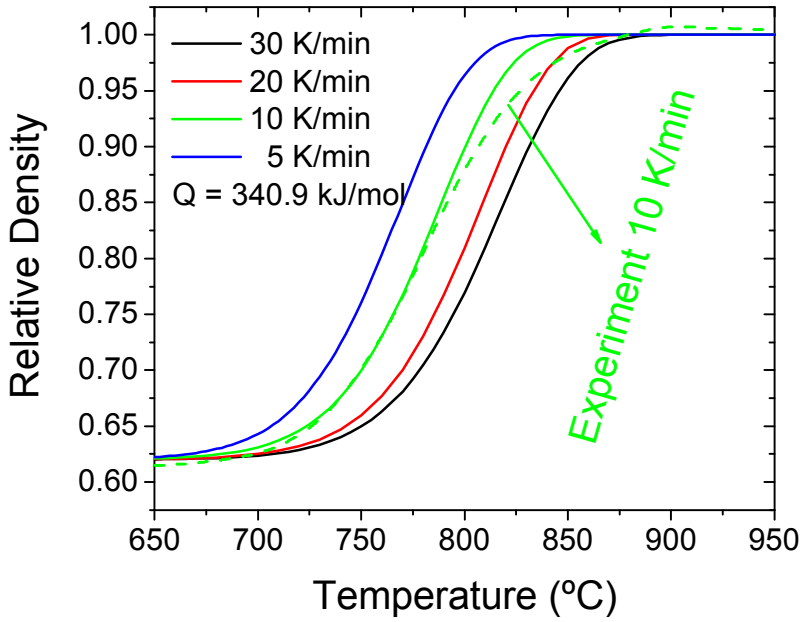
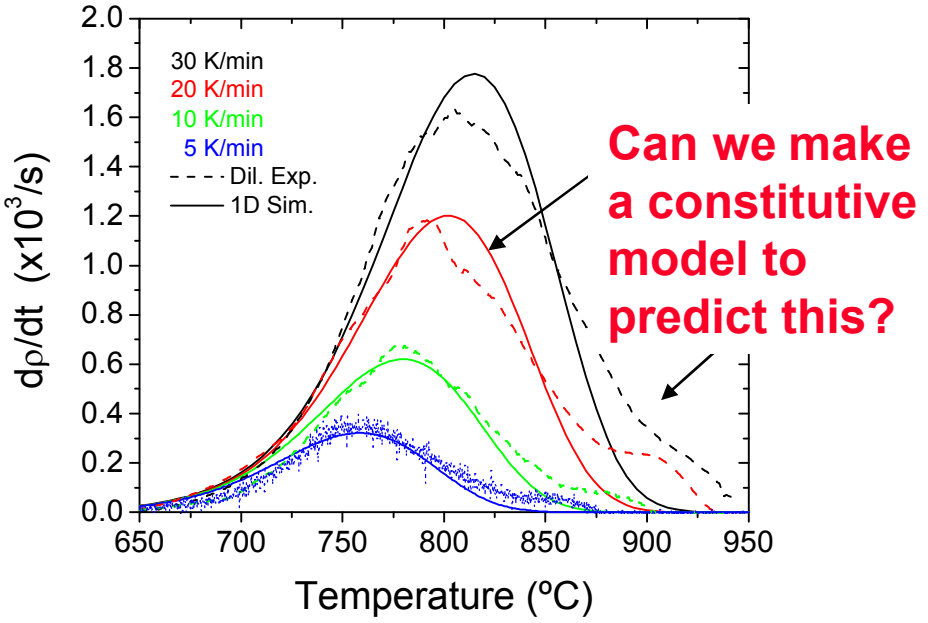
# "Real" microstructure determines the actual densification behavior

**Current finite element sintering models:**

R-S[1] model is based on the repeat unit of classical sintering models

SOVS[2] uses a viscous analogy




**Can we make a constitutive model to predict this?**

[1]Svoboda J, Riedel H, Zipse H *Acta Metall. et Mater.* 42 (2): 435-443 FEB 1994
[2]Reiterer MW, Ewsuk KG, Arguello JG, *J. Am. Ceram. Soc.* 89 (6): 1930-1935 JUN 2006

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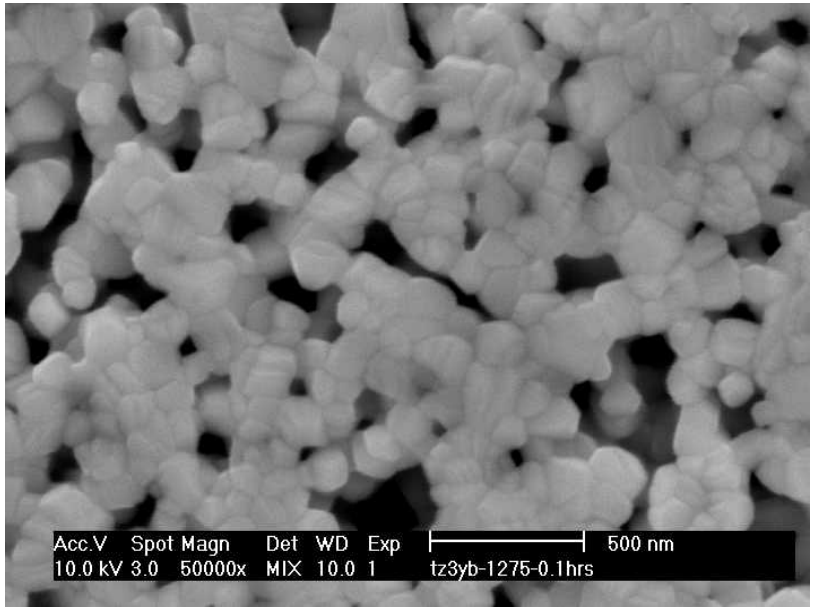
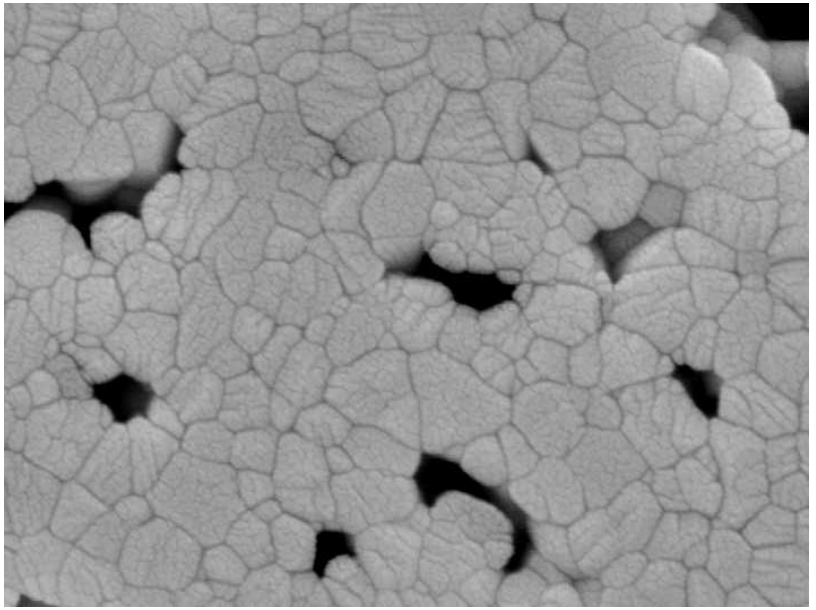
# A fine grain size powder with a hierarchical structure was chosen to develop analysis tools

TOSOH 3YB: 0.1 h




500 nm

TOSOH 3YB: 5 h



500 nm

Non-ideal structure
Multiple grains between pores
Pore elimination occurs during sintering

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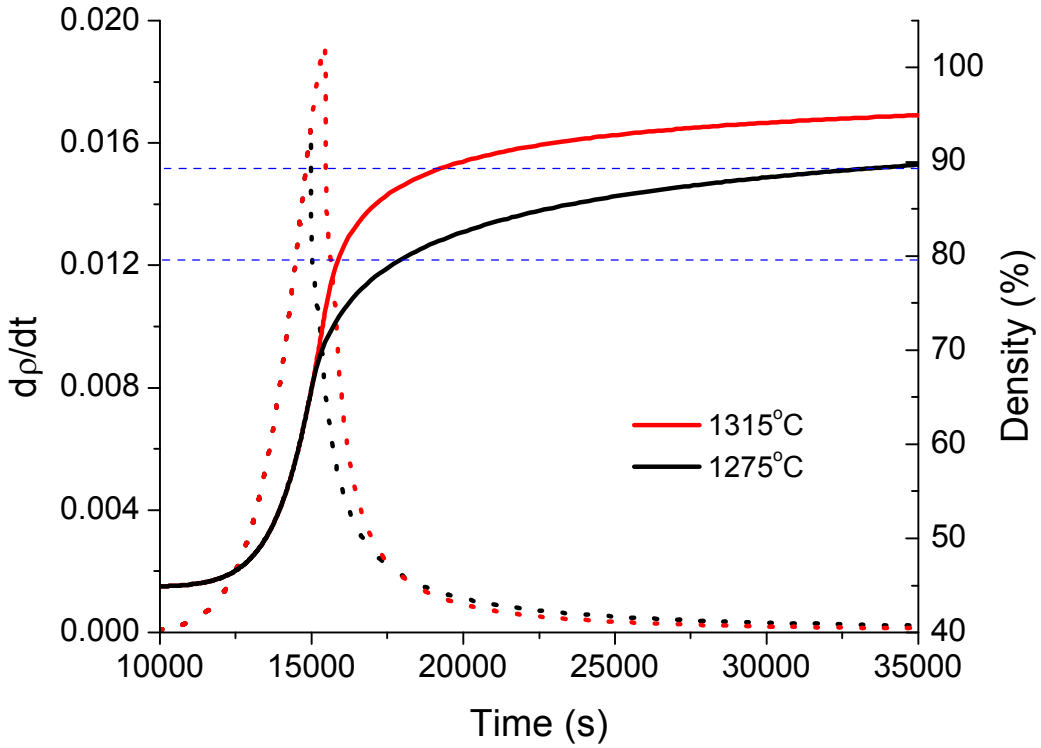
# Isothermal sintering experiments were preformed to evaluate microstructure evolution



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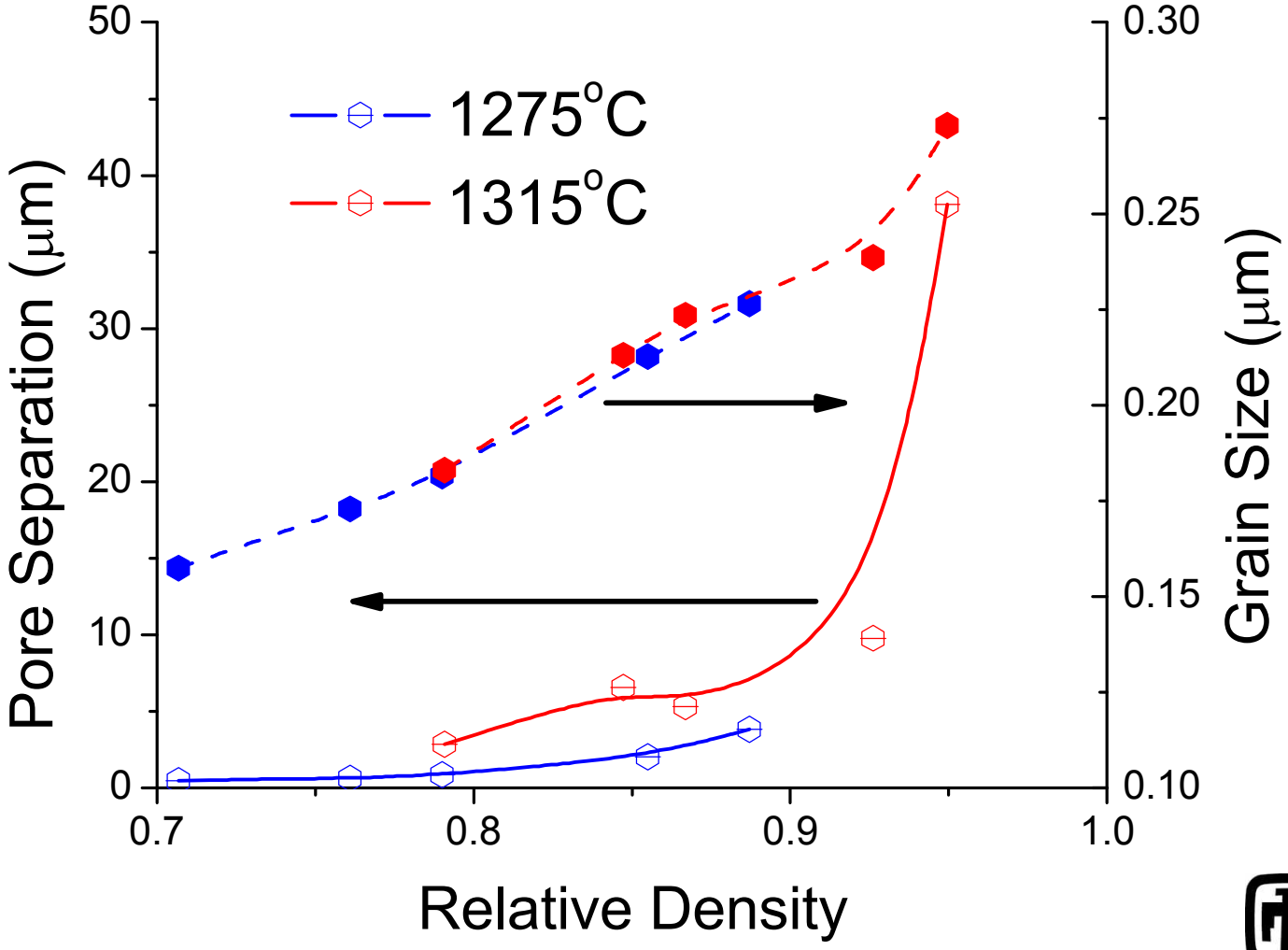
# Image analysis reveals pore separation increases with isothermal sintering time *prior to grain growth*

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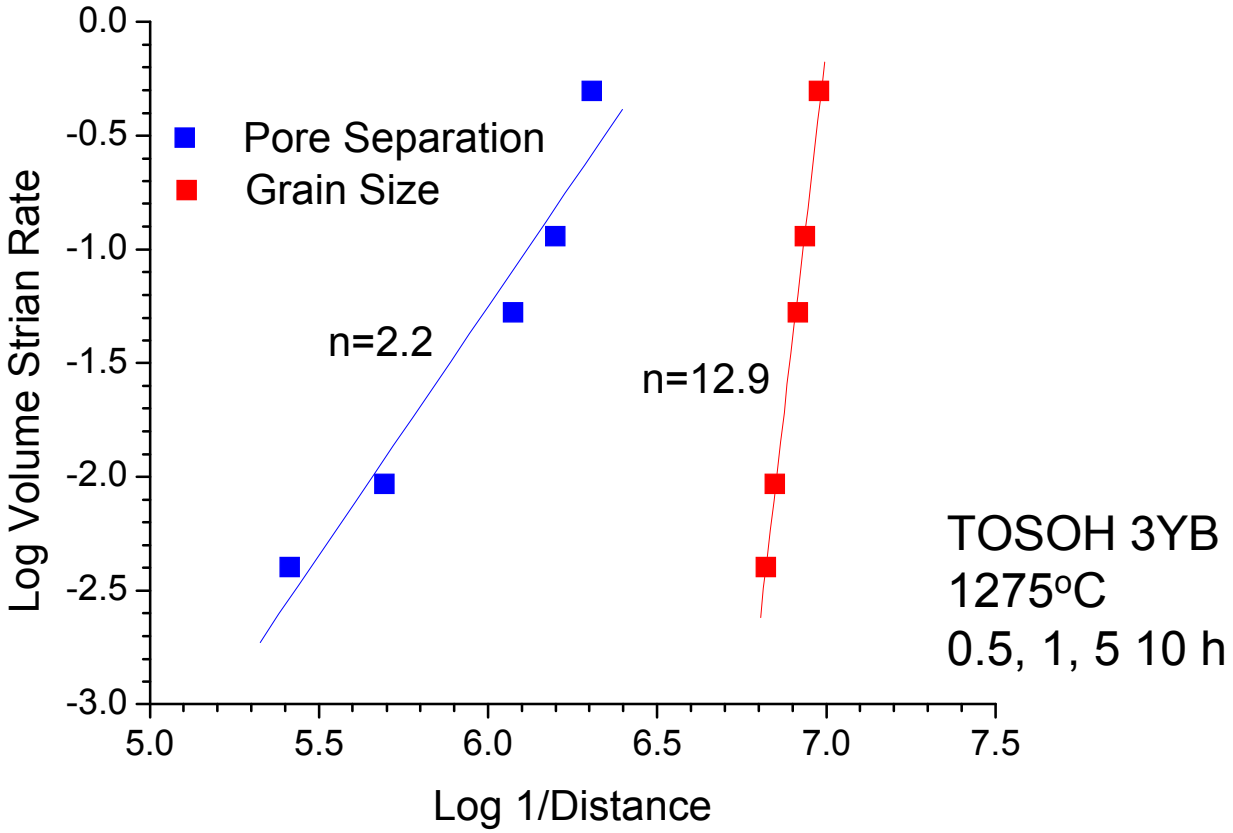
# Inter-pore distance is the actual scale of the diffusion length not the grain size

$$\frac{\dot{\rho}}{\rho} \propto \frac{1}{D^{n?}}$$




Power law relationship does not fit the data



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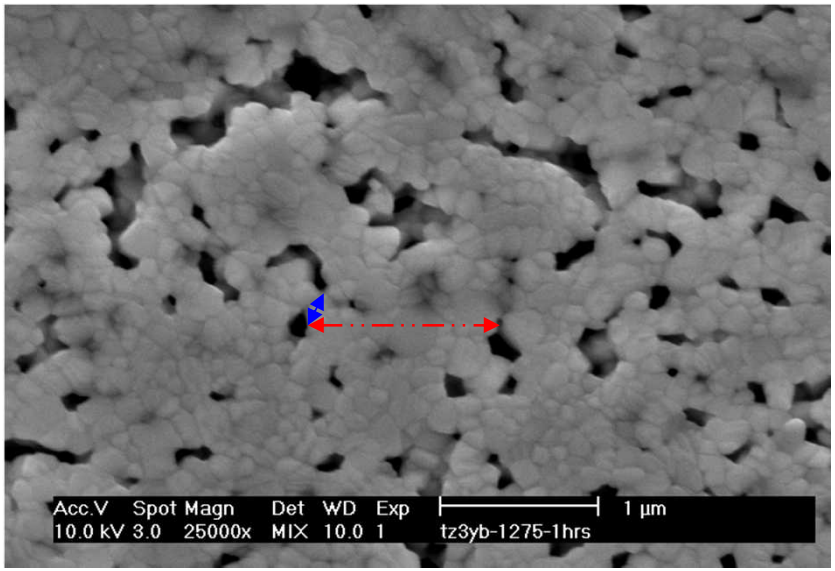
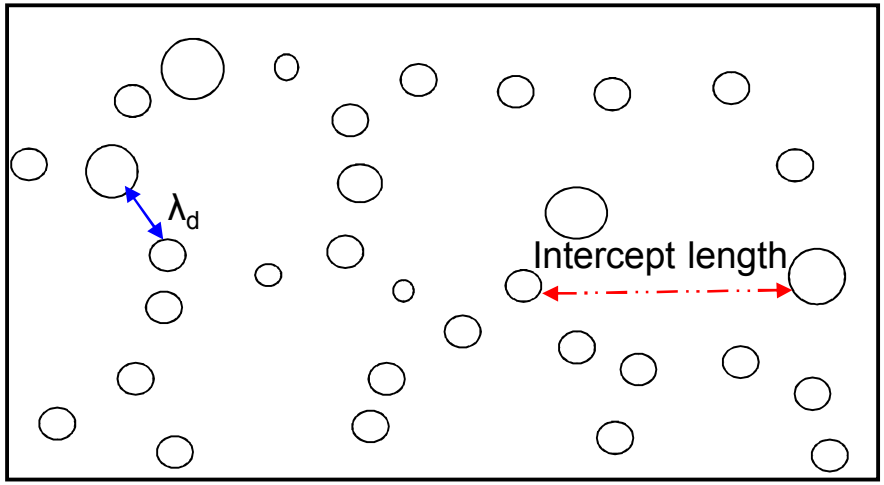
# The intercept length overestimates the diffusion distance in aggregated structures




: Intercept length  :Diffusion shortcuts

The flux equation: $J=k(\Delta c)/\lambda$.

Longer distance, less mass transferred in limited time

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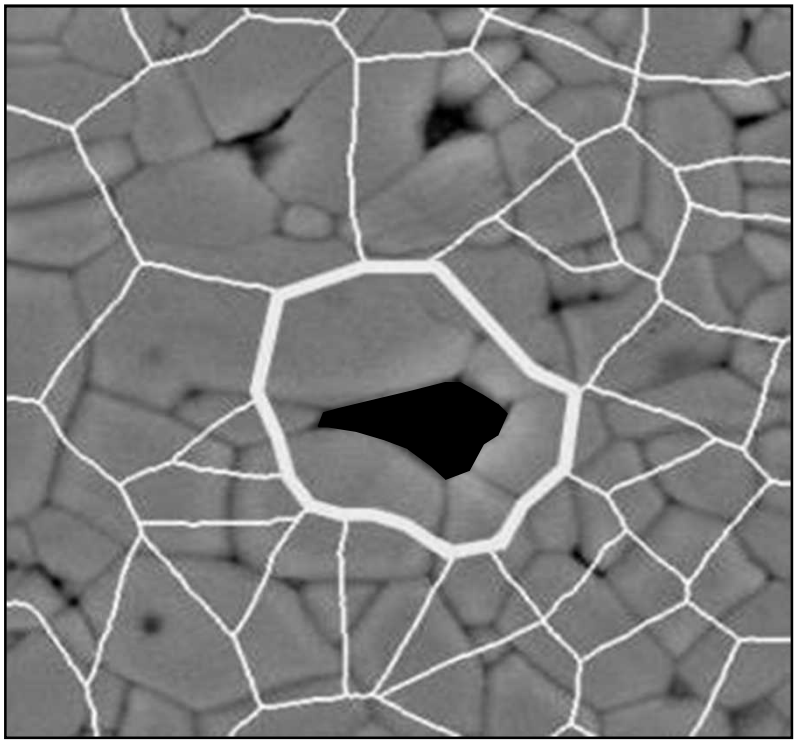
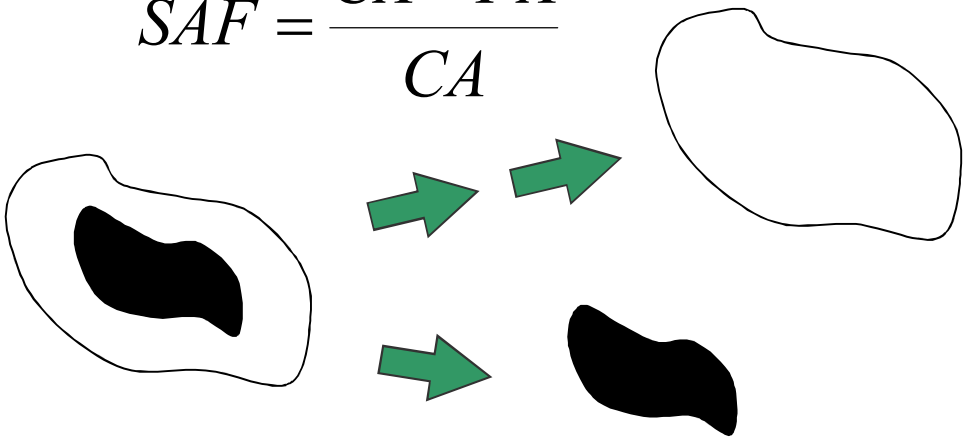
# Microstructure can be uniquely characterized using pore boundary tessellation



$$SAF = \frac{CA - PA}{CA}$$



Similar to construction of a Voronoi polyhedron

A tessellation cell contain a pore and fractions of the surrounding grains

Sensitive to local arrangements of grains around the pore

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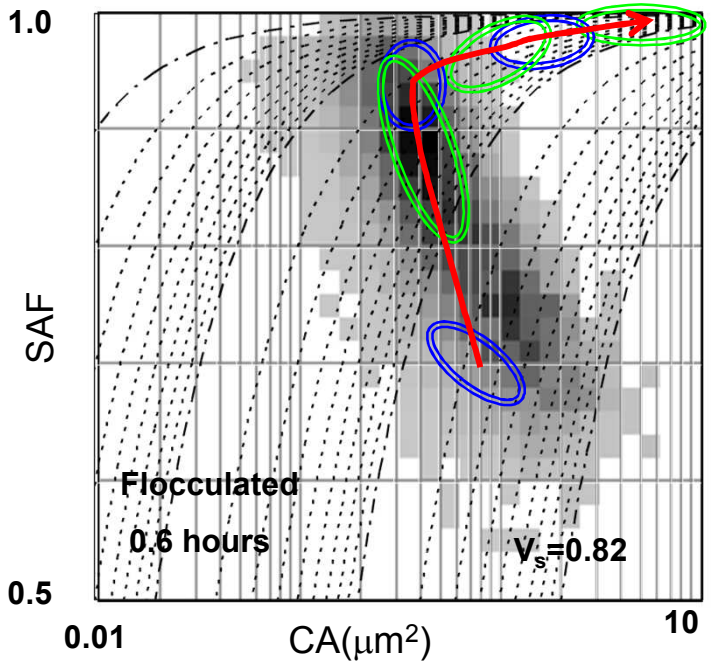
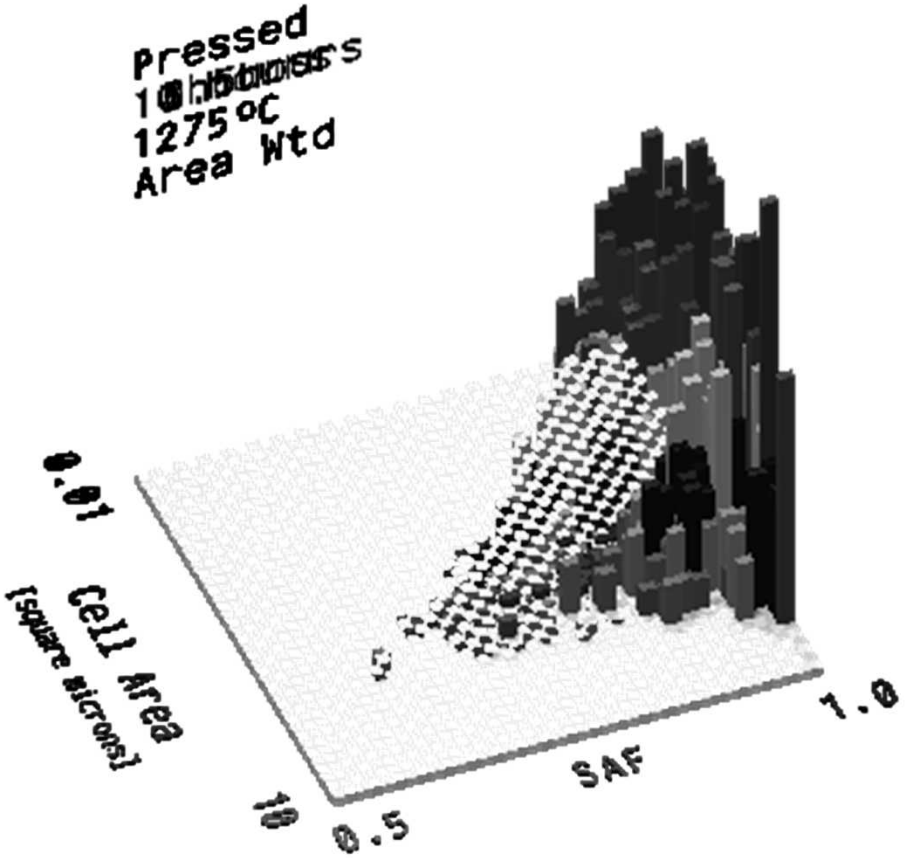
# Pore boundary tessellation is powerful tool for following microstructure evolution





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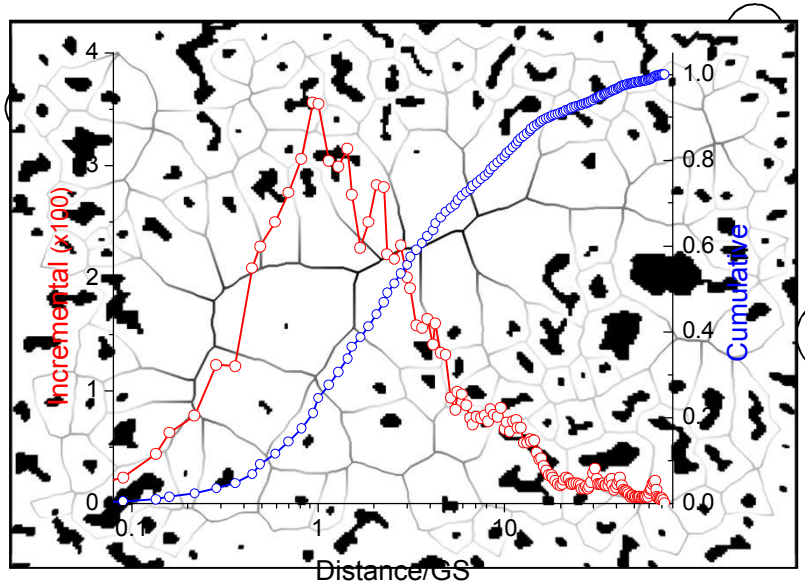
# Dilation of tessellated images can be used to determine the effective diffusion lengths

Process SEM images into binary pore images.

Distance map with the tessellation cell boundaries obtained from pore images.

The tessellation cell boundaries fall at the maximum distance from all pores.

The grey scale of cell boundaries represent the distance between the pores. The dark, the further.

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$$\lambda_d = n/(\sum_i \frac{1}{\lambda_i})$$



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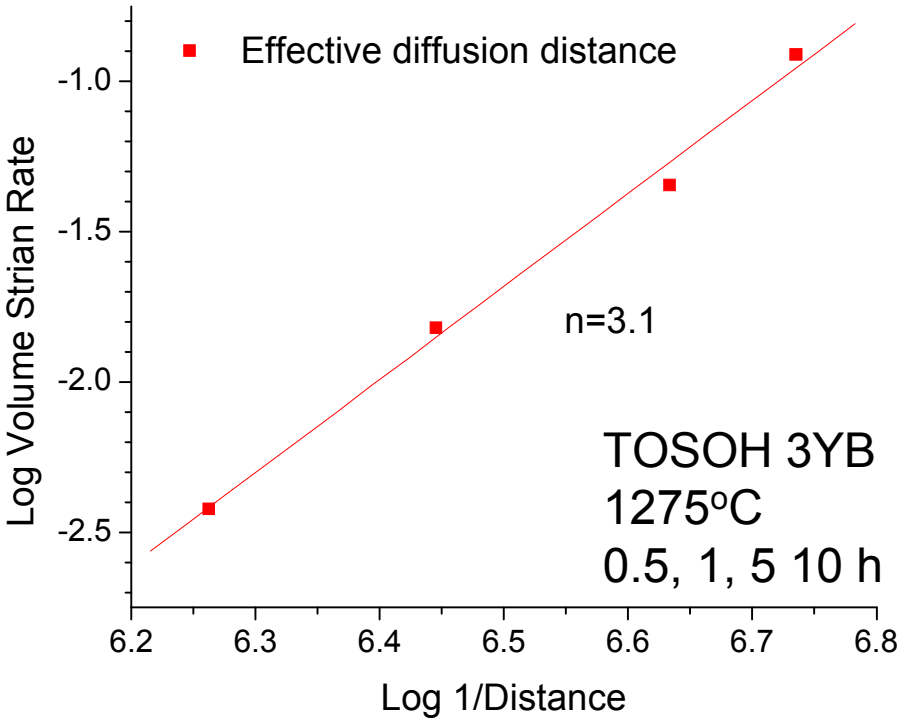
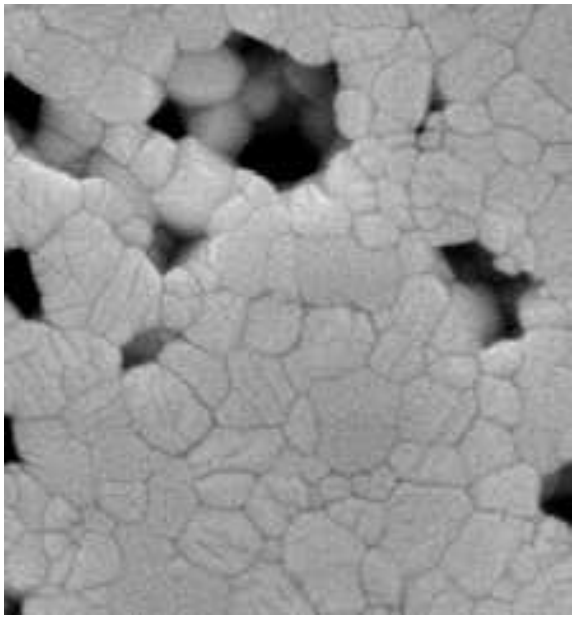
# The effective diffusion distance calculated from pore boundary tessellation is a good measure of the controlling flux distance

A $\lambda_{di}$ is calculated for each tessellation cell, then the average value of $\lambda_d$ is calculated for all cells




Many possible diffusion paths across multiple grains between two pores



The exponent may not represent a classical diffusion mechanism value

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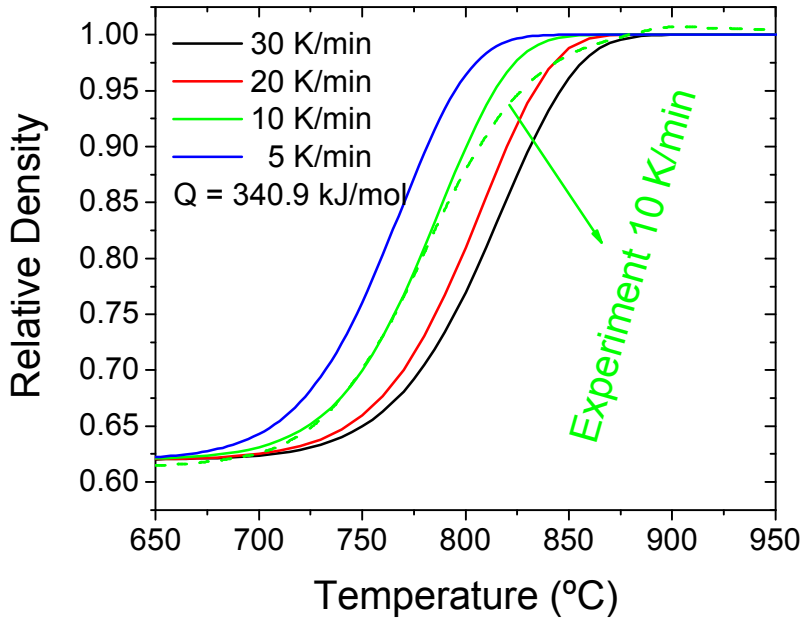
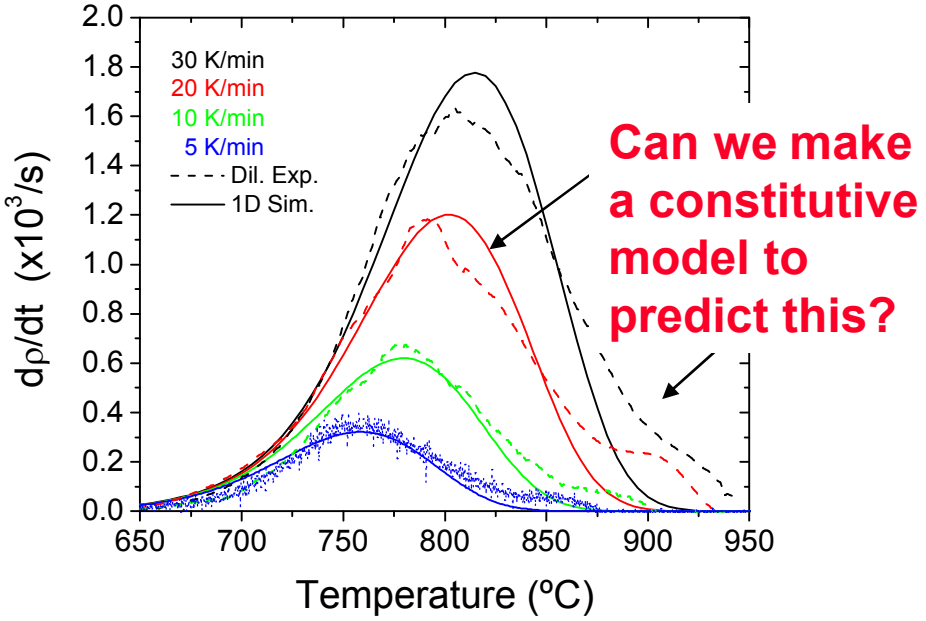
# There are several possible approaches to achieve more effective and realistic finite element models

**Incorporation of the effective diffusion distance into existing models**

**Use pore boundary tessellation to validate microstructure evolution models to determine parameters for global finite element simulations**

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# Summary

Processing of ultrafine ceramic powders leads to heterogeneous microstructure evolution

Current numerical and FE models do not sufficiently account for heterogeneous microstructure evolution

Effective diffusion distance is a better measure of scale than traditional grain size and pore size measurements

*Can the effective diffusion distance be incorporated into a constitutive model for heterogeneous sintering?*

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# Can the effective diffusion distance and porosimetry be combined into an effective model?

Two possible approaches

Combined stage sintering / Master sintering curve

Microstructure & Properties:

$$\Phi(\rho) = \frac{k}{\gamma_s \Omega D_0} \int_{\rho_0}^{\rho} \frac{G((\rho))^n}{3\rho\,\Gamma(\rho)} d\rho$$

Temperature-time profile:

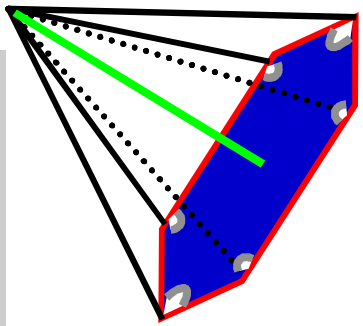
$$\equiv \theta(t, T(t)) = \int_0^t \frac{1}{T} e^{-\frac{Q_{app}}{RT}} dt$$

$$\Gamma_b = \frac{\alpha C_k C_b}{C_\lambda C_a C_h}$$

Stress intensification factor

$$\dot{\varepsilon} = -K\sigma\phi^{(m+1)/2} F^{m/2} / G^m$$

$$\dot{\rho} = n(\rho) g(\rho)(1 - \sigma_m / \Sigma) l^{-a}$$



α: Chemical potential

$C_k$: Pore curvature

$C_b$: Grain boundary diffusion area

$C_\lambda$: Diffusion distance

$C_a$: Cell area

$C_h$: Cell height

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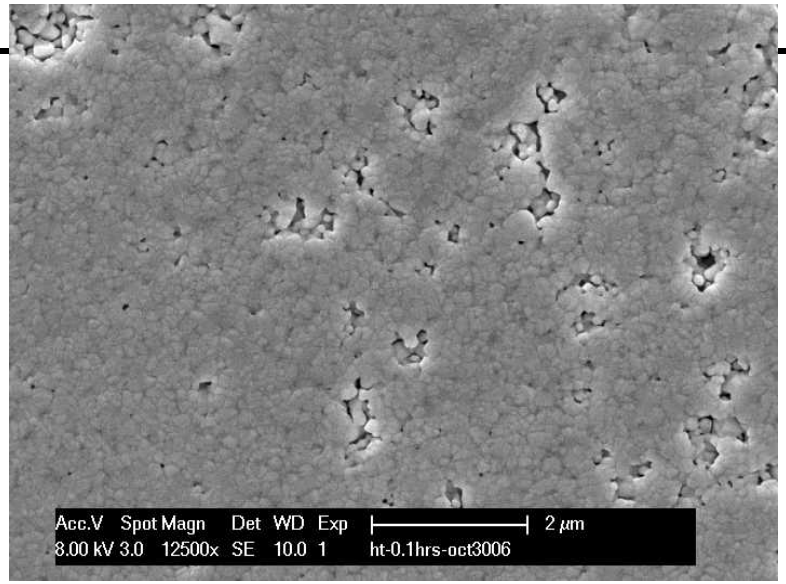
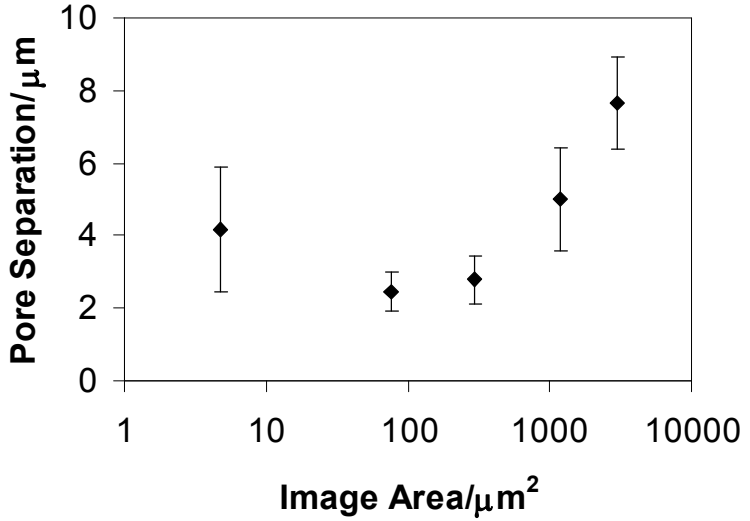
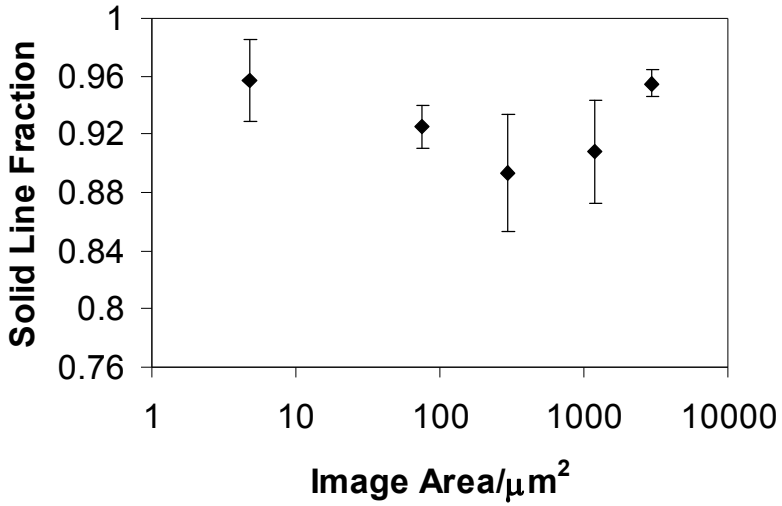
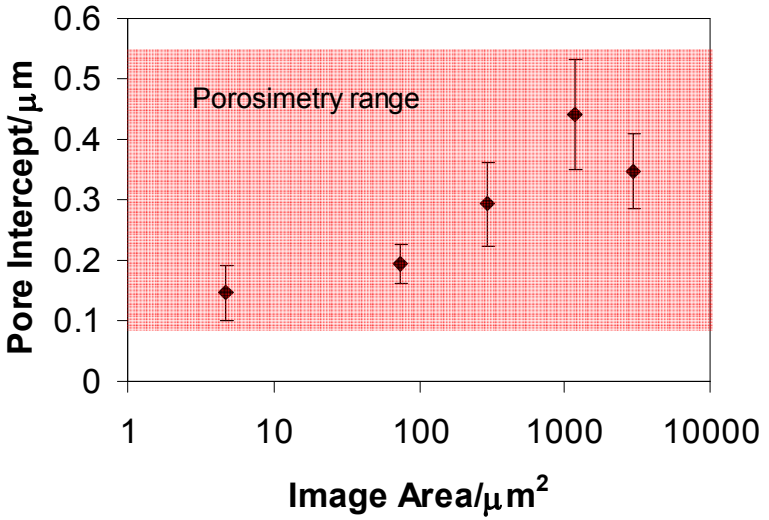
# Heterogeneous Microstructures Are More Difficult To Characterize



# Porosimetry complements image analysis