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Effects of Microstructural Hierarchy in the Consolidation and Sintering of Ultrafine Powders

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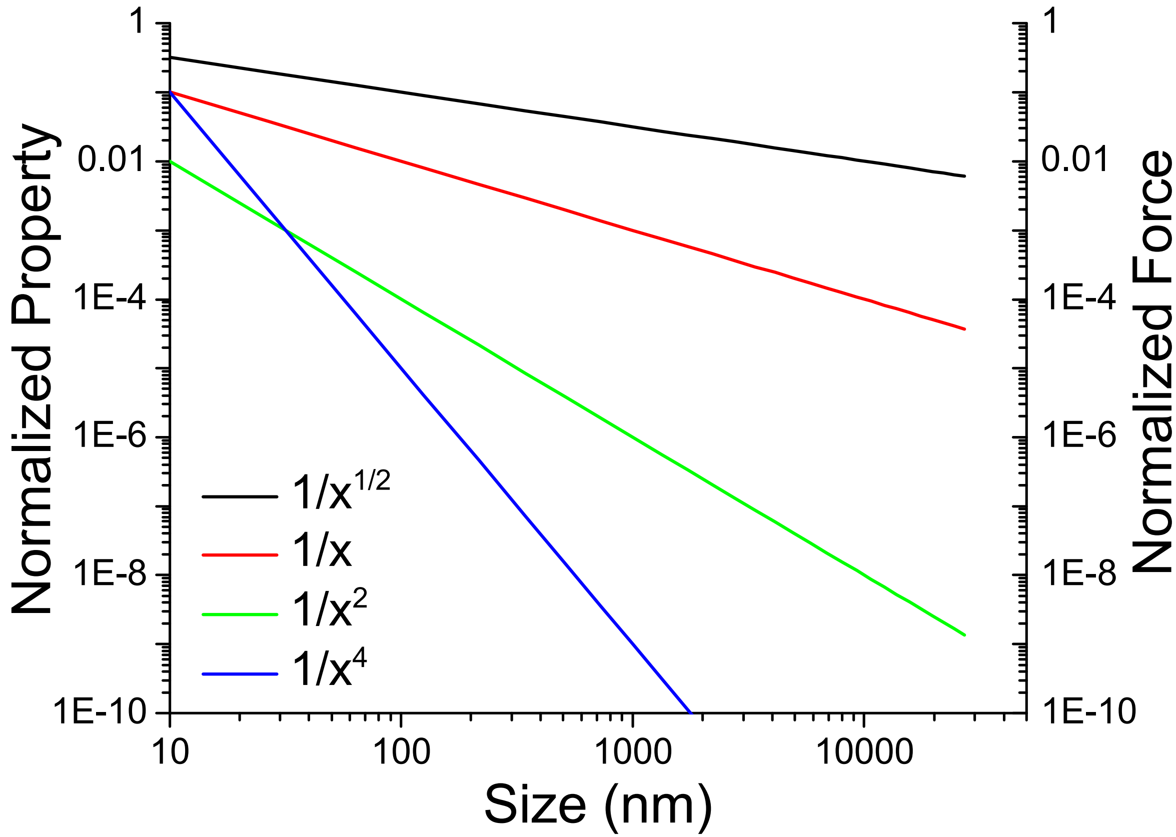
There Are Trade-offs Between Properties And Processing

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



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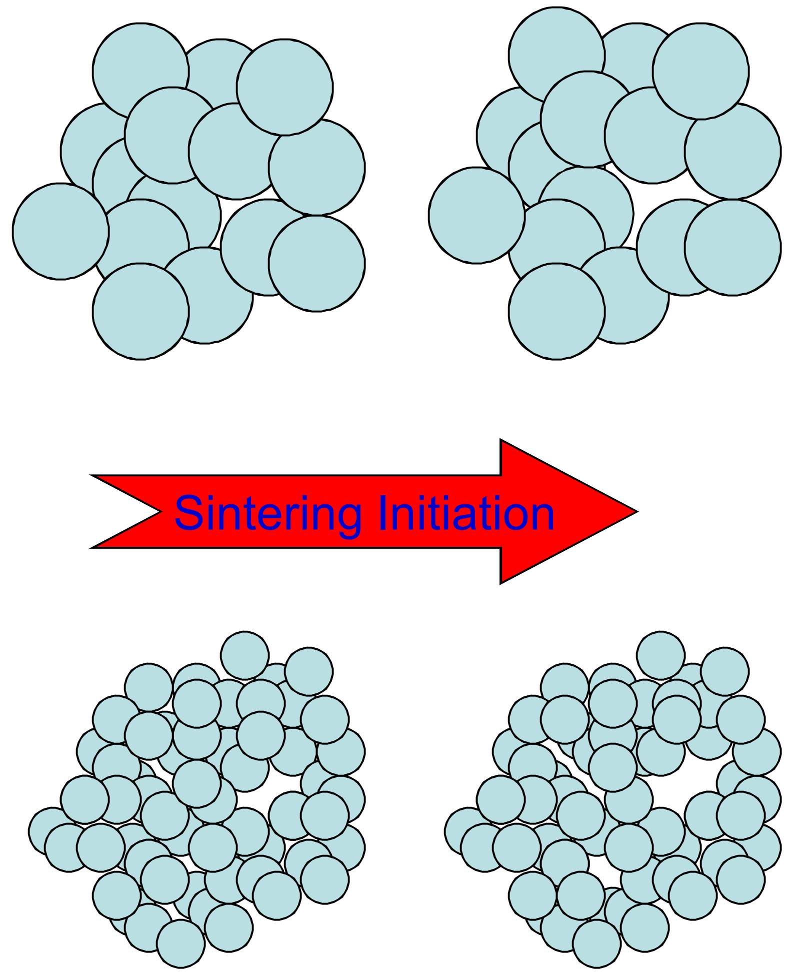
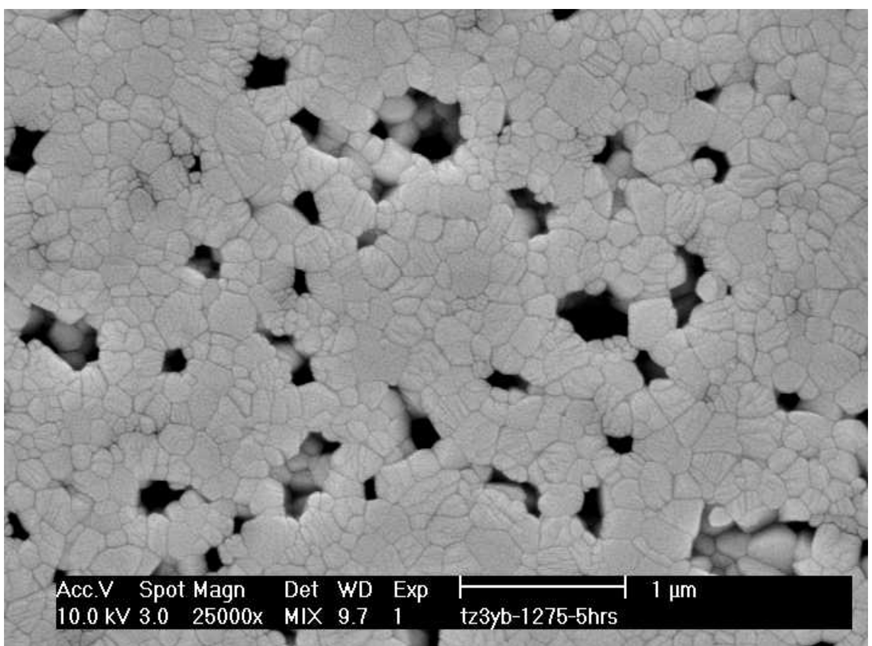
Packing And Forming Defects Result In A Heterogeneous Microstructure

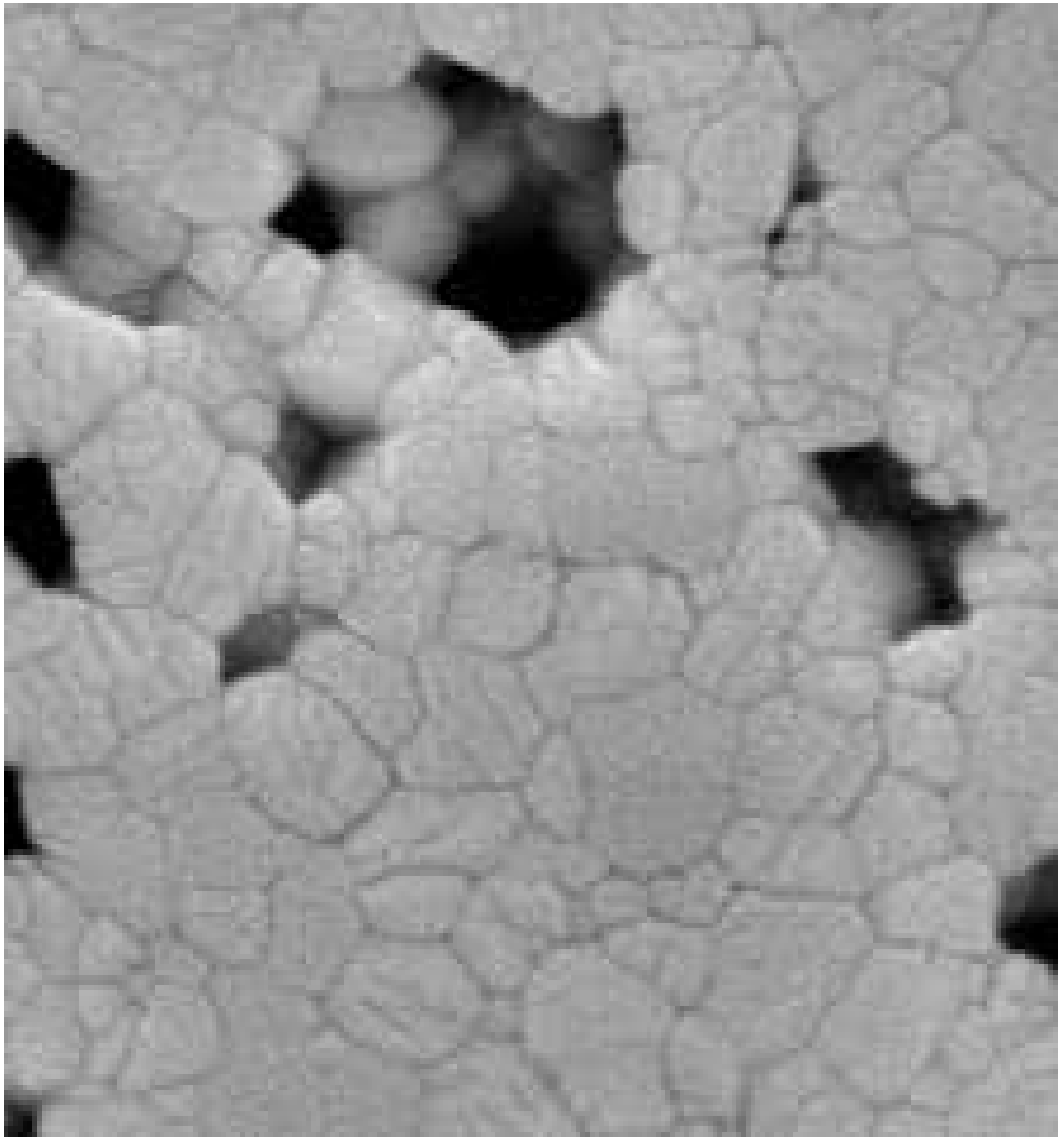
Heterogeneity Origins

- Density gradients
- Particle size distribution
- Agglomerates

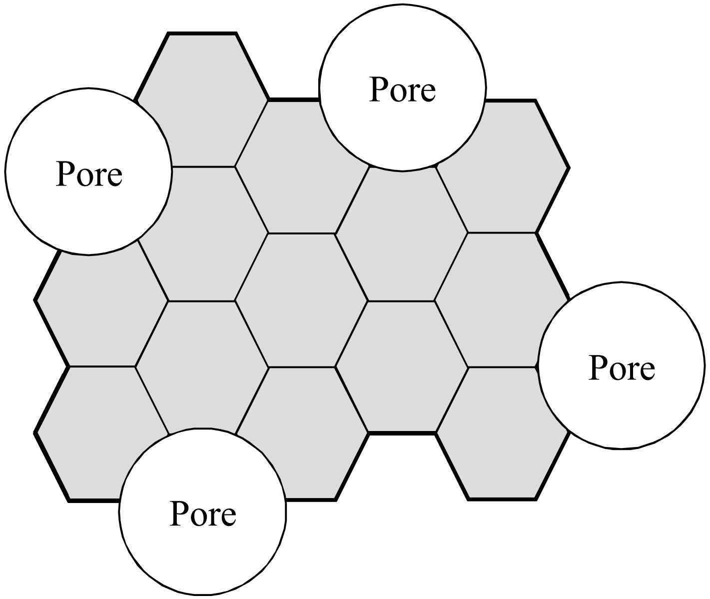




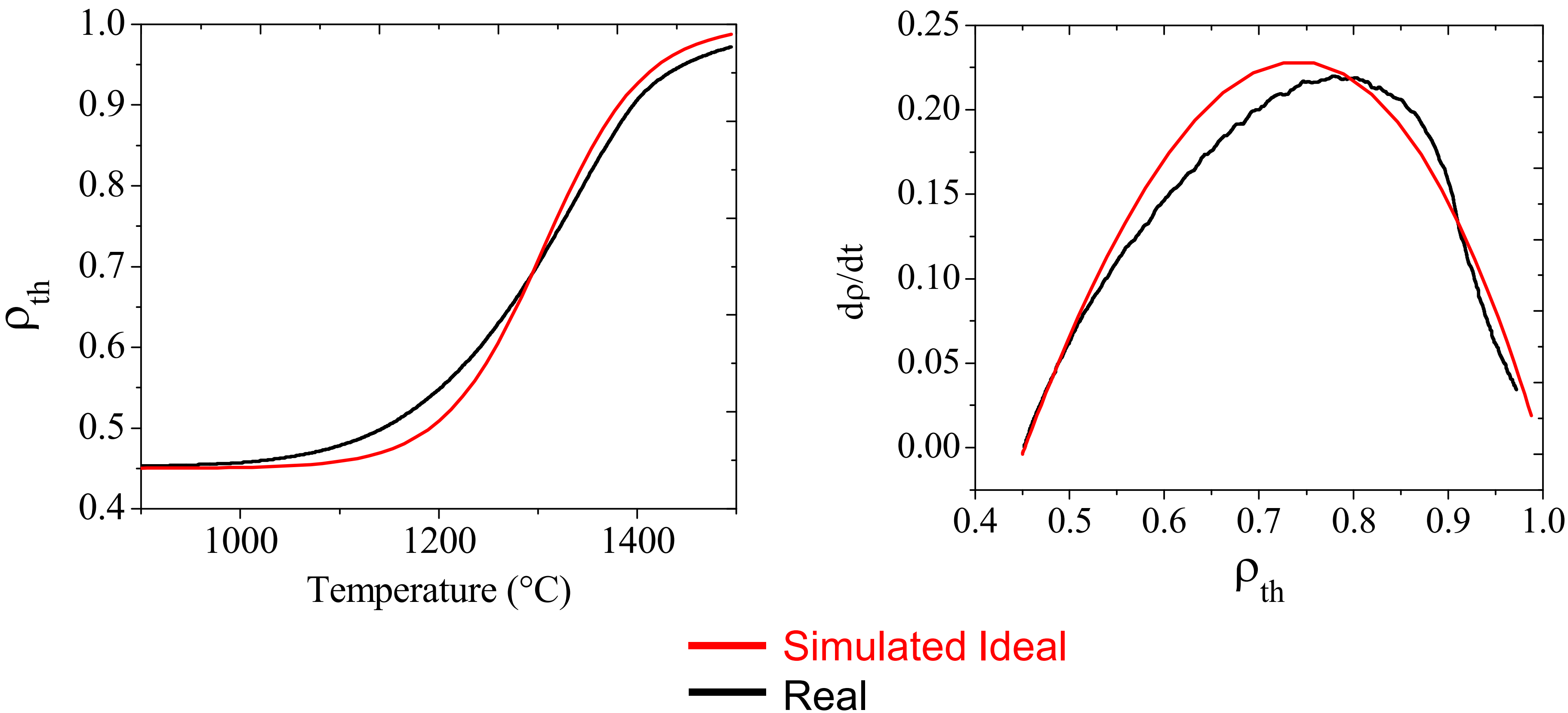
Sintering Models Do Not Address The Heterogeneity In Real Microstructures



Real



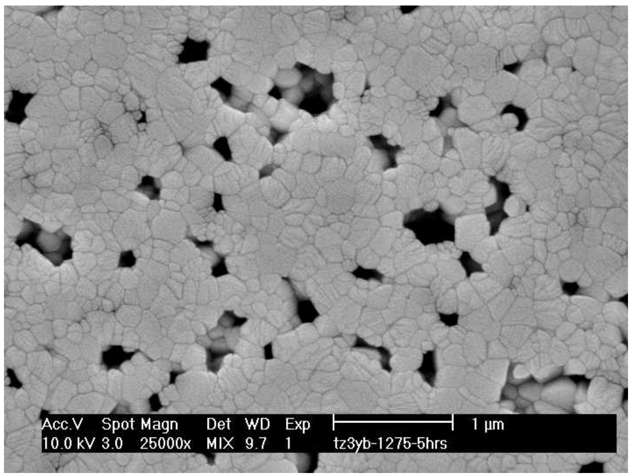
"Real" Microstructure Determines The Actual Densification Behavior



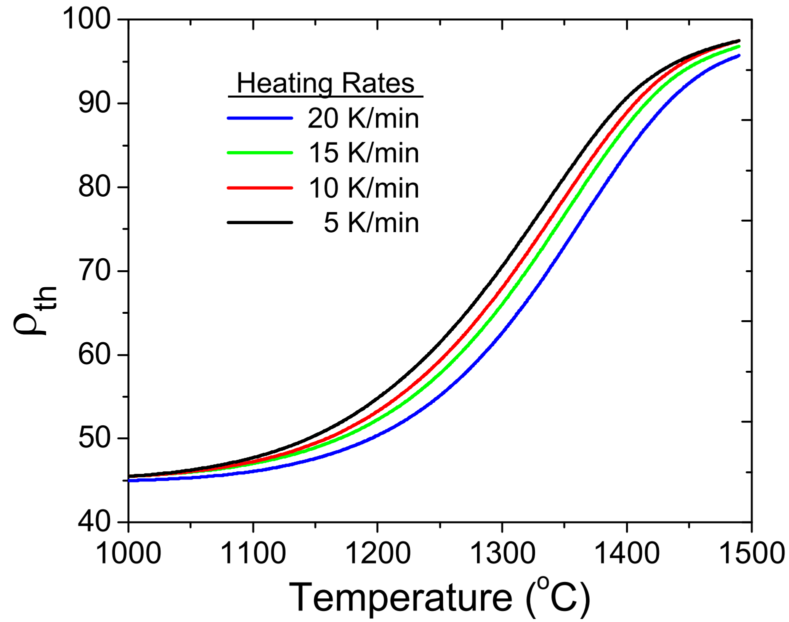
Master Sintering Curve (MSC) Links Processing & Microstructure

Instantaneous linear shrinkage

$$-\frac{dL}{L\,dt} = \frac{\gamma_s \Omega}{kT}\left(\frac{\Gamma_v D_v}{G^3} + \frac{\Gamma_b D_b}{G^4}\right)$$



TOSOH 3YB Densification Curves



Microstructure & Properties:

Temperature-time profile:

$$\Phi(\rho) = \frac{k}{\gamma_s \Omega D_0} \int_{\rho_0}^{\rho} \frac{\boxed{G((\rho))^n}}{3\,\rho\,\boxed{\Gamma(\rho)}}\, d\rho \equiv \boxed{\theta(t, T(t))} = \int_0^t \frac{1}{T} e^{-\frac{Q_{app}}{RT}}\, dt$$

H. Su, D.L Johnson, “Master Sintering Curve: A Practical Approach to Sintering”
J. Am. Ceram. Soc. **79** 3211-17 (1996)

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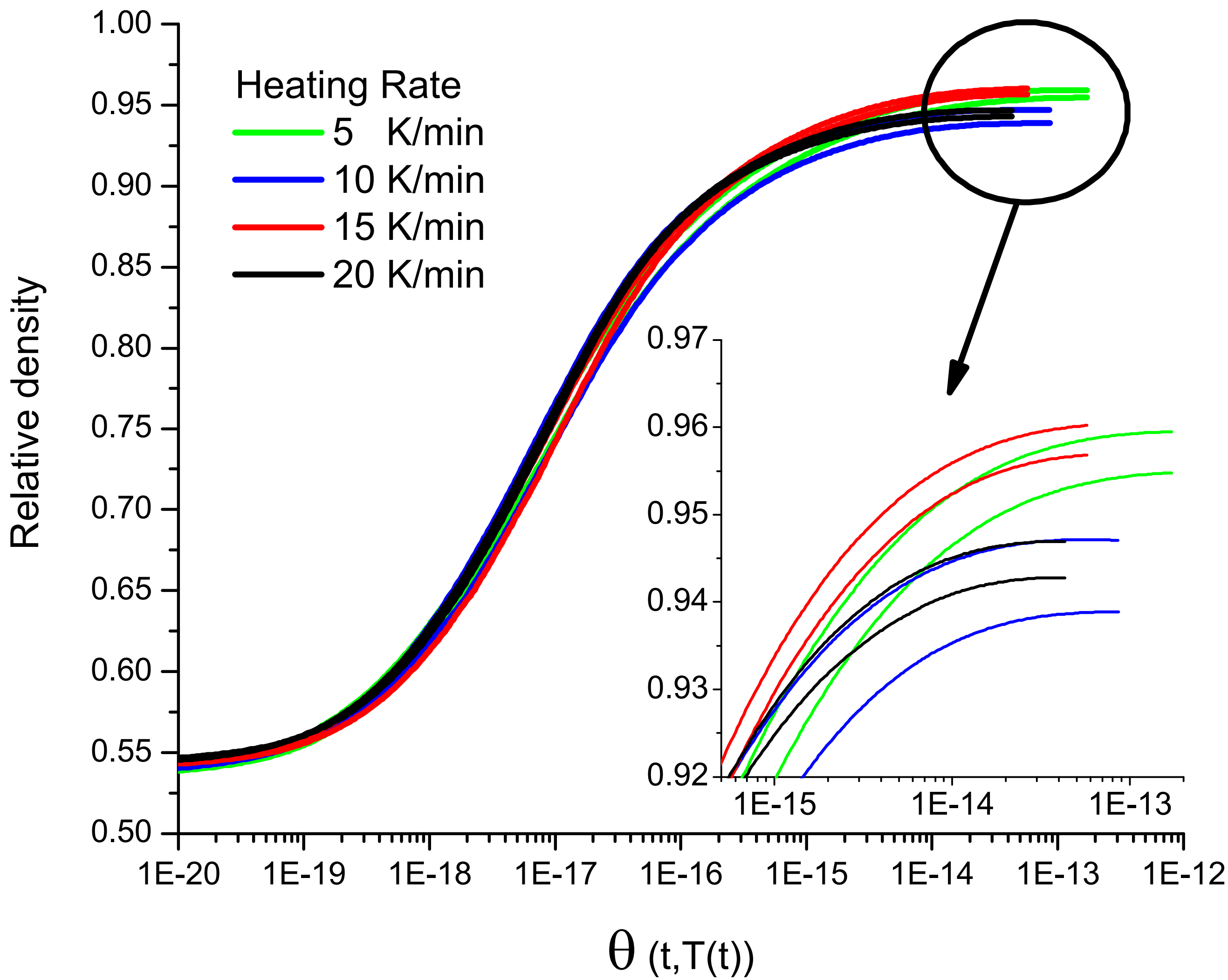
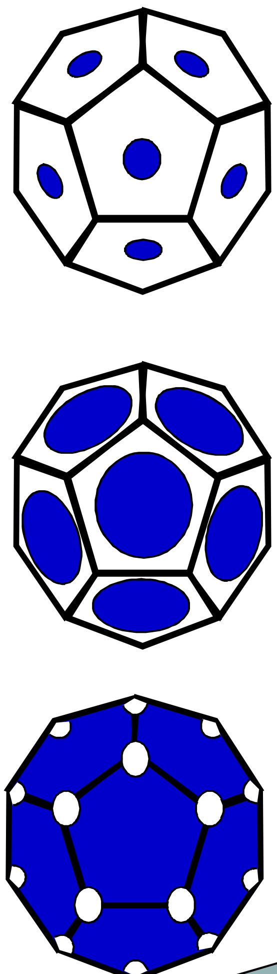
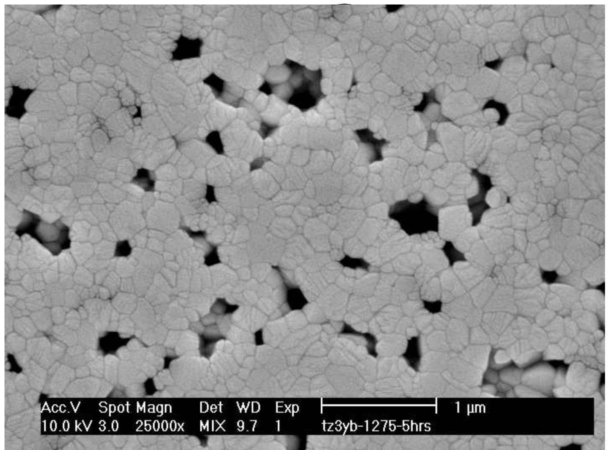
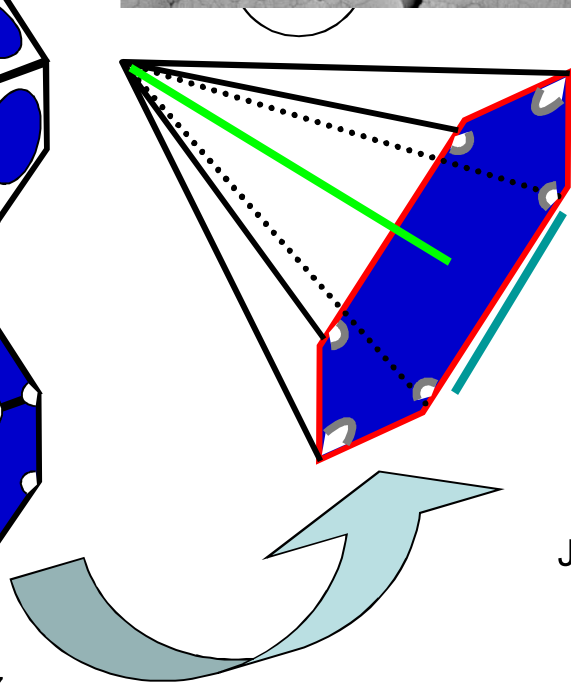
MSC Deviations Reflect Microstructure-Density Trajectory Changes



Diffusion Distance Increases With Grain Growth And Pore Separation







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

α: Chemical potential

C_k: Pore curvature

C_b: Grain boundary diffusion area

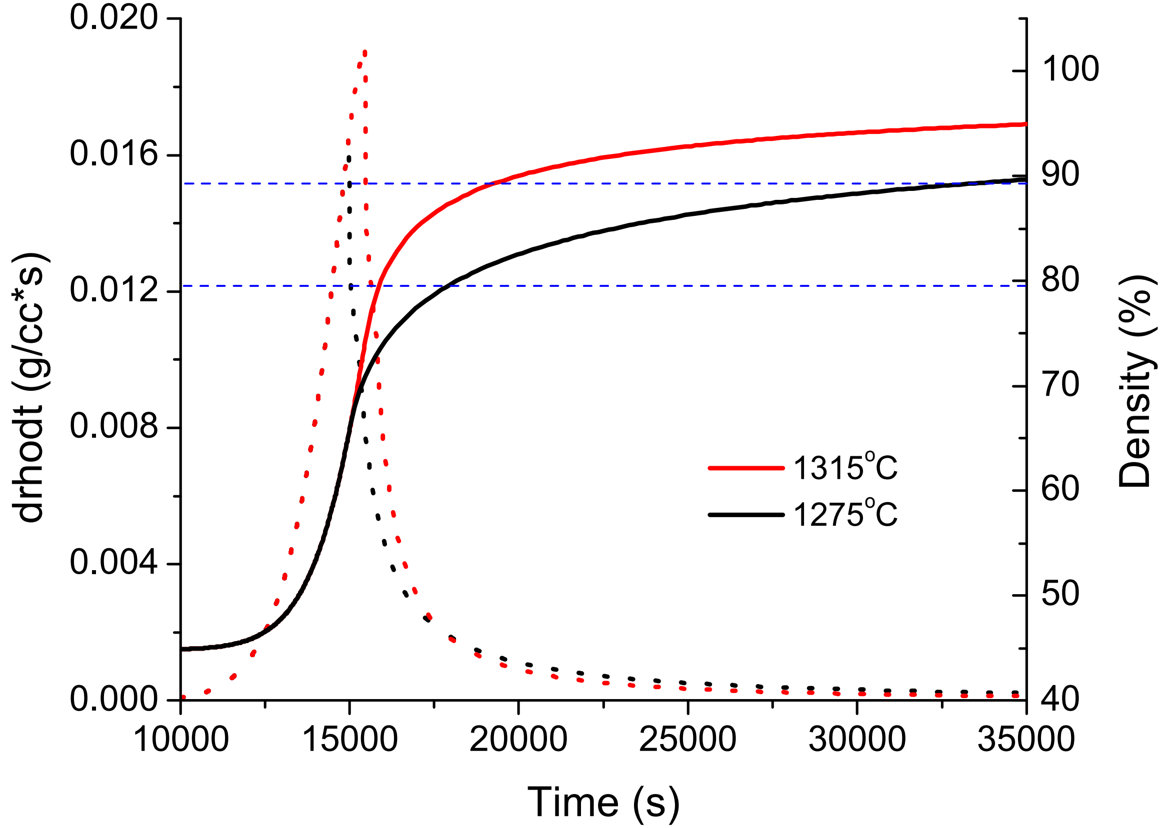
C_λ: Diffusion distance

C_a: Cell area

C_h: Cell height

J.D. Hansen, R.P. Rusin, M.H. Teng, D.L Johnson, "Combined-Stage Sintering Model" *J. Am. Ceram. Soc.* **75** 1129-35 (1992)

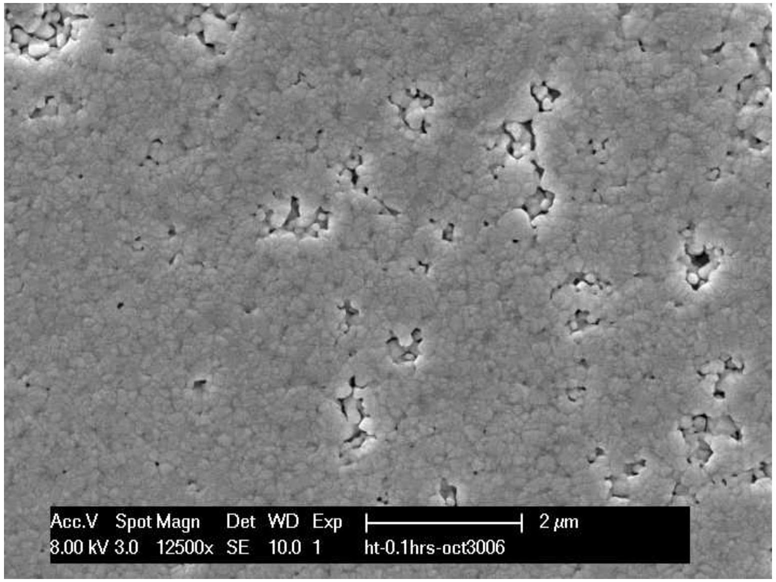
Real Systems Undergo Heterogeneous Microstructure Evolution

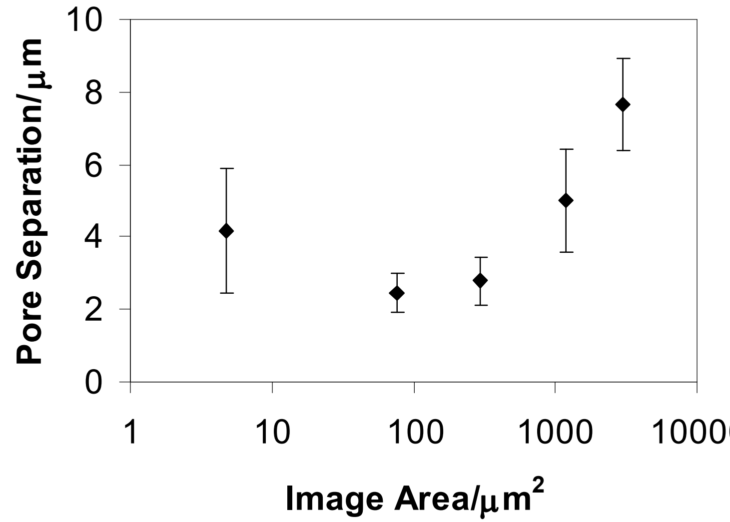


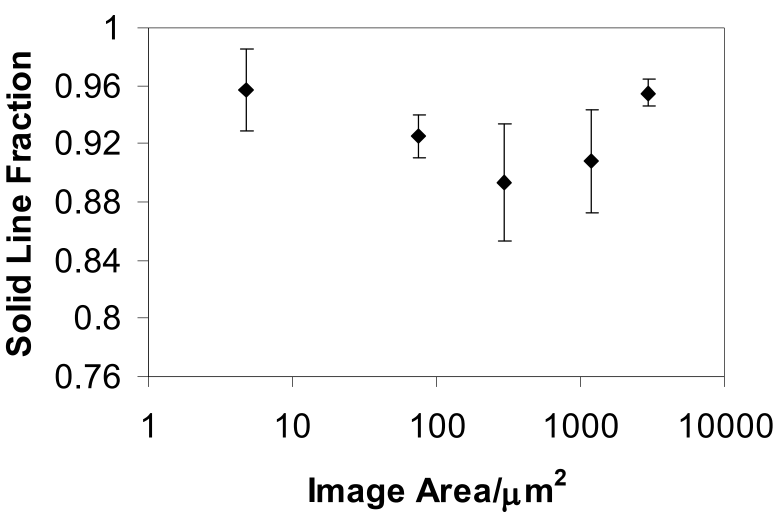
ρ=0.79	Measured Density	Pore Size (μm)	Grain Size (μm)	**Pore Separation (μm)**
1275°C 1h	0.84 ± 0.0[illegible]	0.27 ± 0.04	0.06 ± 0.01	**4.04 ± 0.76**
1315°C 1h	0.96 ± 0.0[illegible]	0.[illegible]1 ± 0.06	0.05 ± 0.01	**5.32 ± 1.80**

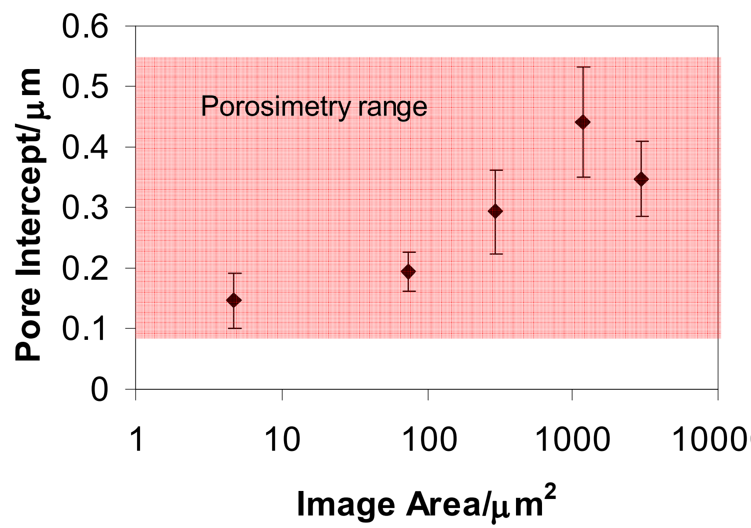
ρ=0.88	Measured Density	Pore Size (μm)	**Pore Separation (μm)**
1275°C 1h	0.93 ± 0.02	1.30 ± 0.13	**1.90 ± 0.09**
1315°C 1h	0.95 ± 0.0[illegible]	[illegible].25 ± 0.12	**4.[illegible] ± 1.72**

Heterogeneous Microstructures Are More Difficult To Characterize









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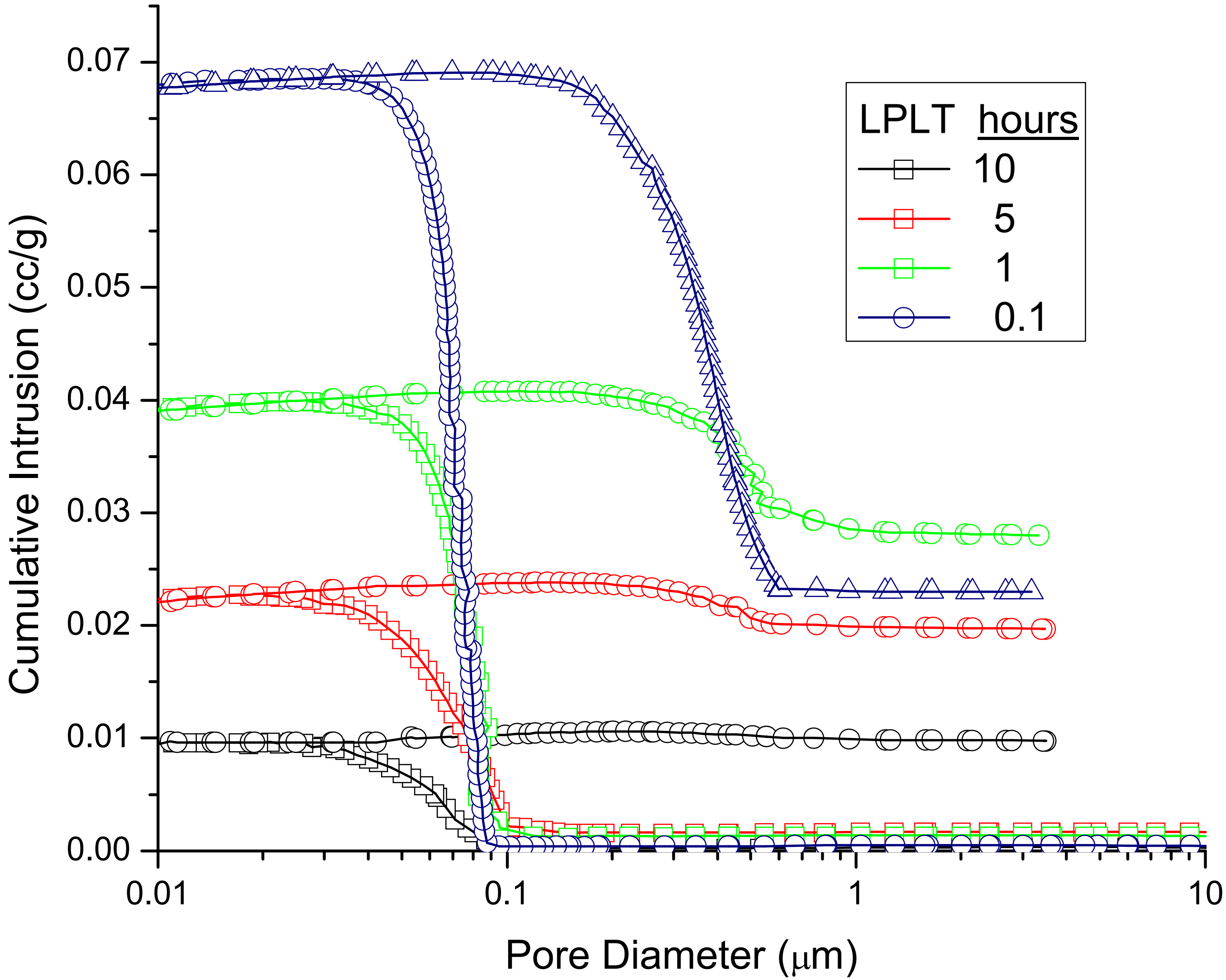
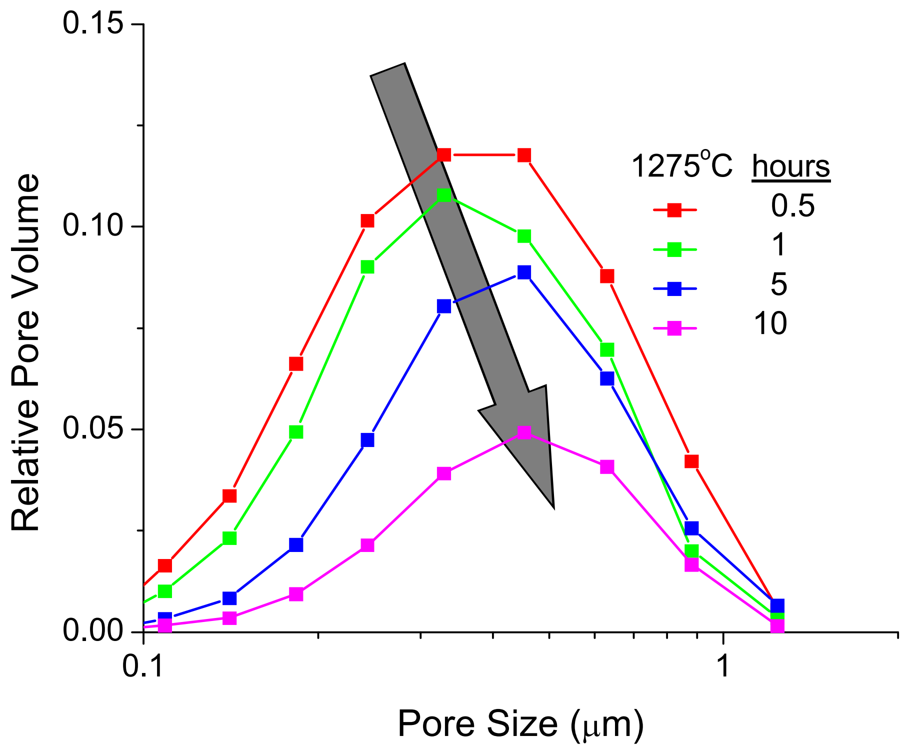
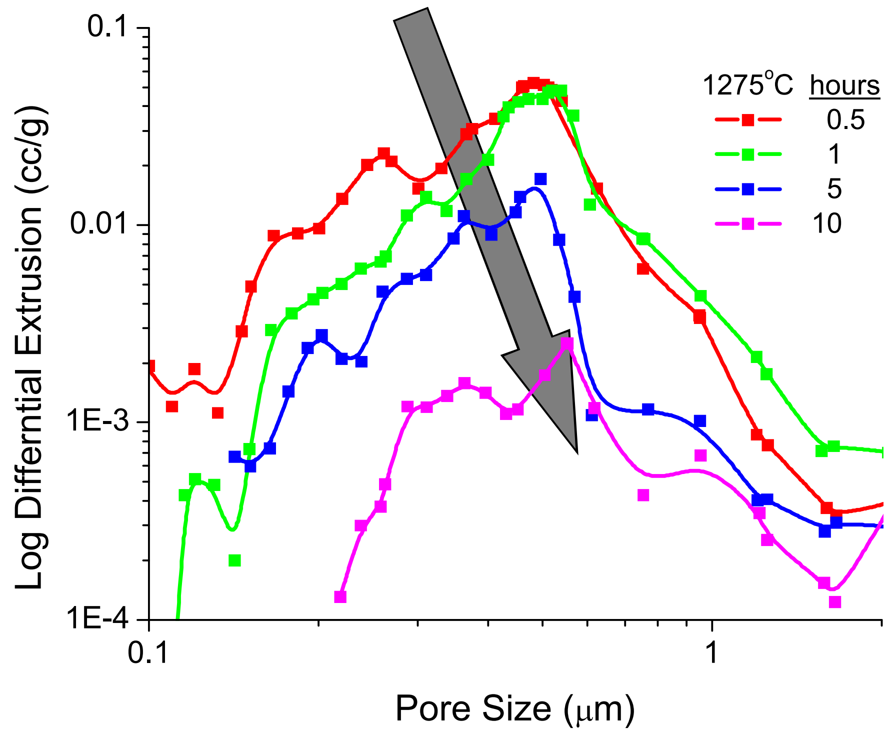
All Of The Porosity Is Characterized With Porosimetry

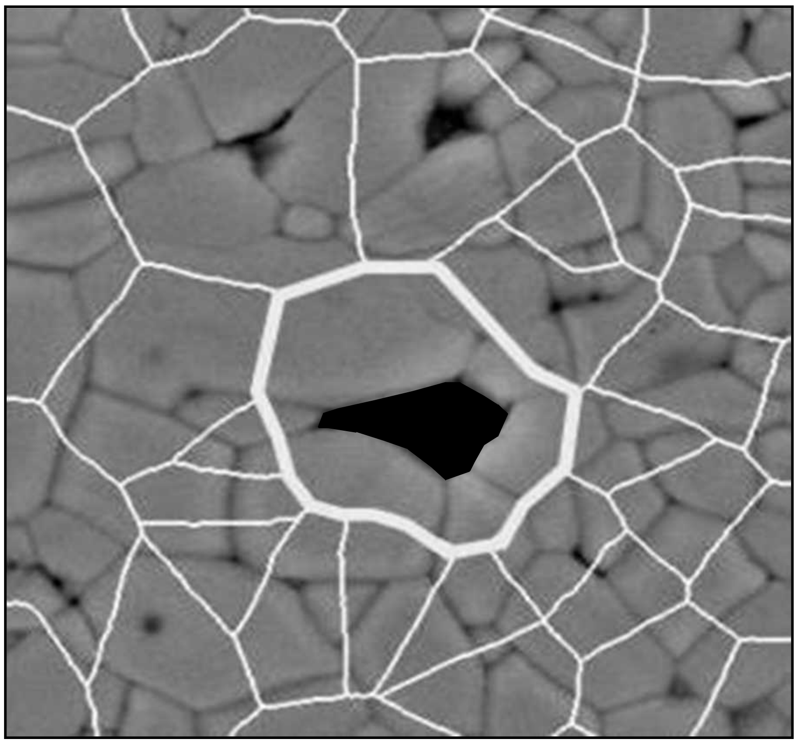


Porosimetry Complements Image Analysis

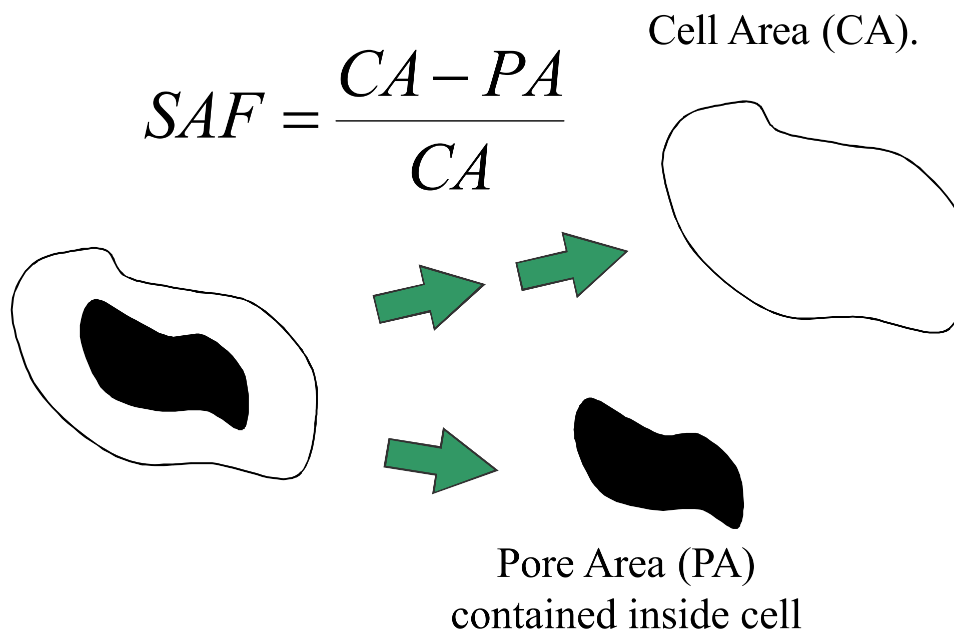




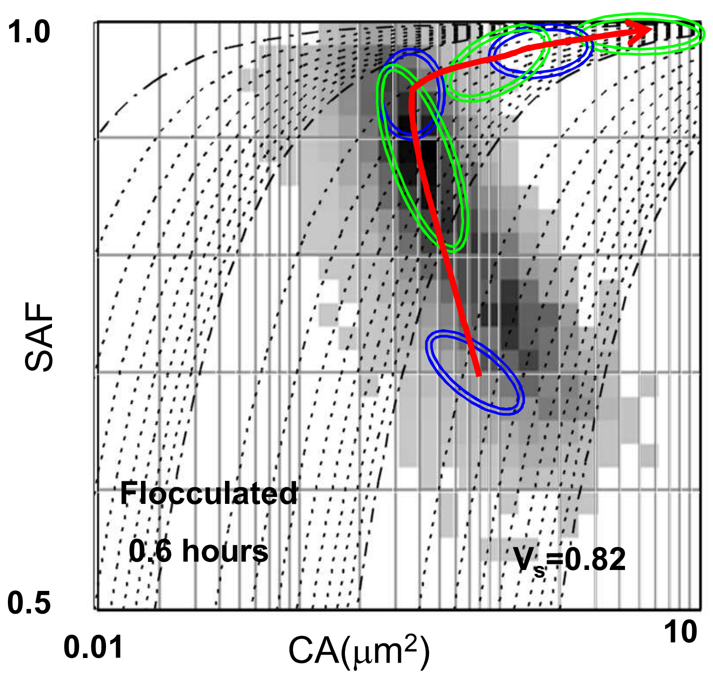
Porosity Can Be Characterized Using Pore Boundary Tessellation

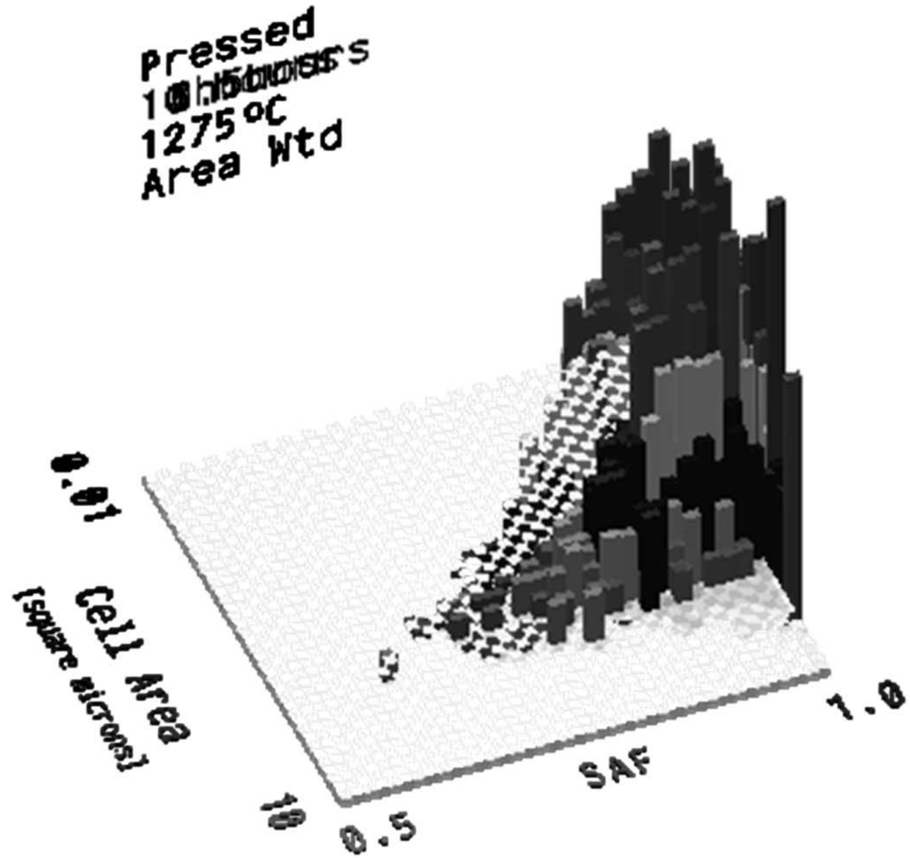


$$SAF = \frac{CA - PA}{CA}$$



PBT Results Are Consistent With Image Analysis And Porosimetry





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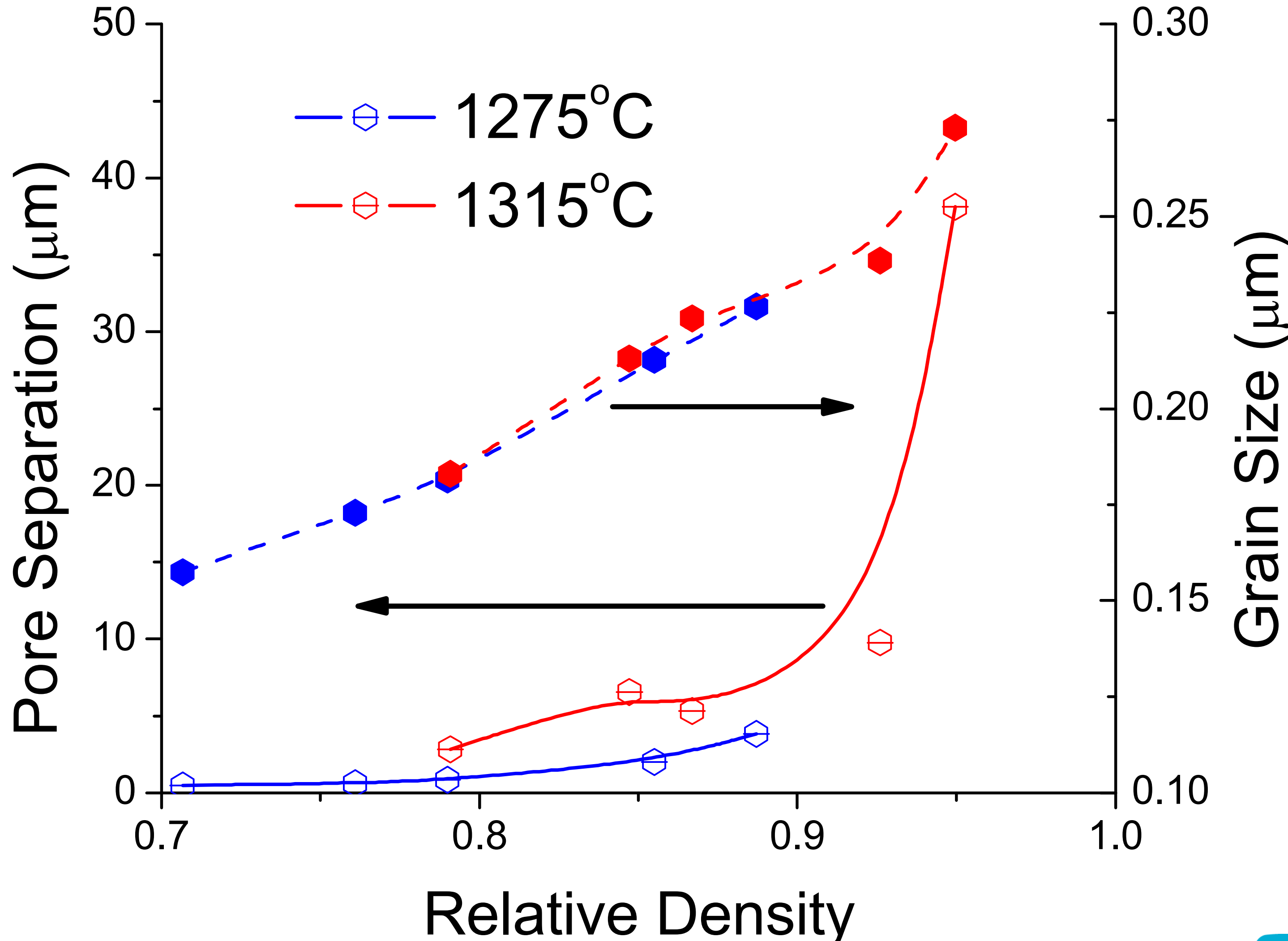
PBT Shows Pore Separation Increases With Isothermal Sintering Time



Summary

Pore Based Microstructure Evolution

Pore separation may contribute more to the coarsening of ultrafine microstructures than *grain growth*