Application of Master Sintering Curve Theory To Predict And Control Nano-Crystalline Ceramic Sintering

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A Science-Based Approach Can Be Used To Improve Process Control

The Problem: Processing defects impact manufacturing cost, and component performance and reliability

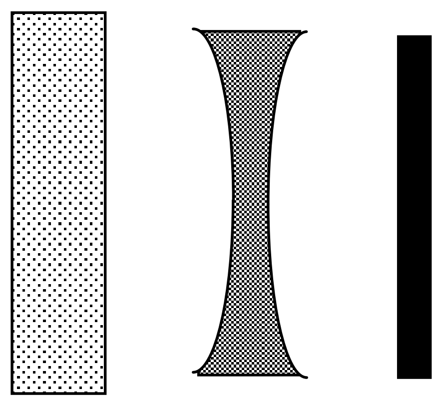
Technical Solution:

Apply fundamental scientific understanding to design reproducible processes to manufacture high performance and reliability ceramics

- characterization
- predictive modeling

Science-Based Processing

Form -> Sinter



Form -> Sinter -> Grind ($$'s)

Traditional Processing

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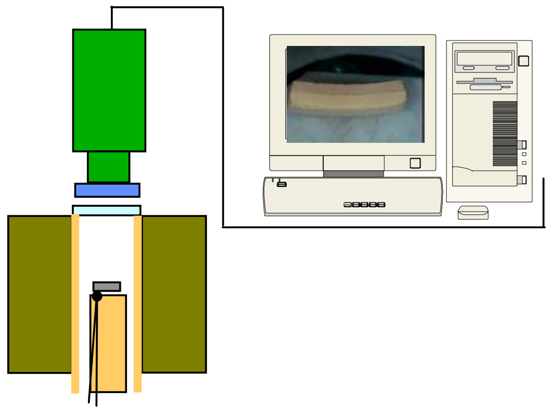
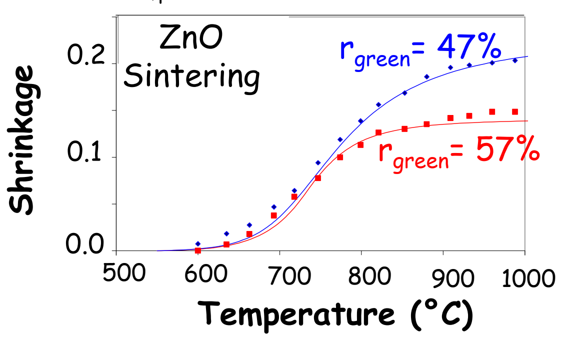
Characterization & Modeling Are Advancing Sintering Science

Bilayer Sintering Experiment

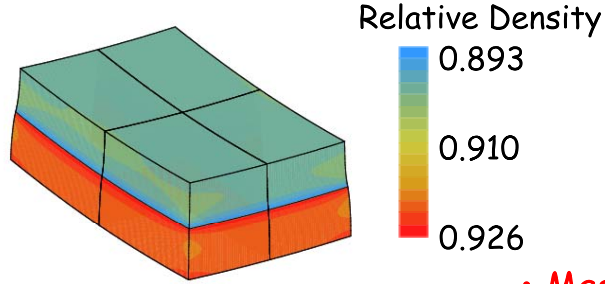




Characterization

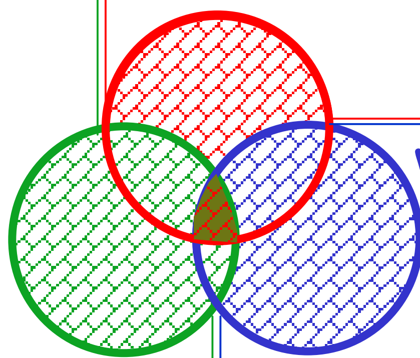
- Shrinkage (non-contact video)
- Shape Deformation (in-situ)
- Constitutive behavior
- Pore Characteristics (NMRI)

Bilayer Sintering Simulation



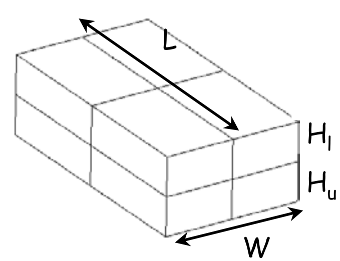
Modeling

- Master Sintering Curve Theory
- 3D FE Modeling
- Microstructure Modeling



Validation

Sintering Model Validation



	Experiment	Model	Δ
L 8.049 mm	6.7 mm	6.431	4.0%
H_u 1.323 mm	2.15 mm	1.001	1.2%
H_l 1.308 mm		1.123	
W 4.790 mm	3.89	3.888	0.1%

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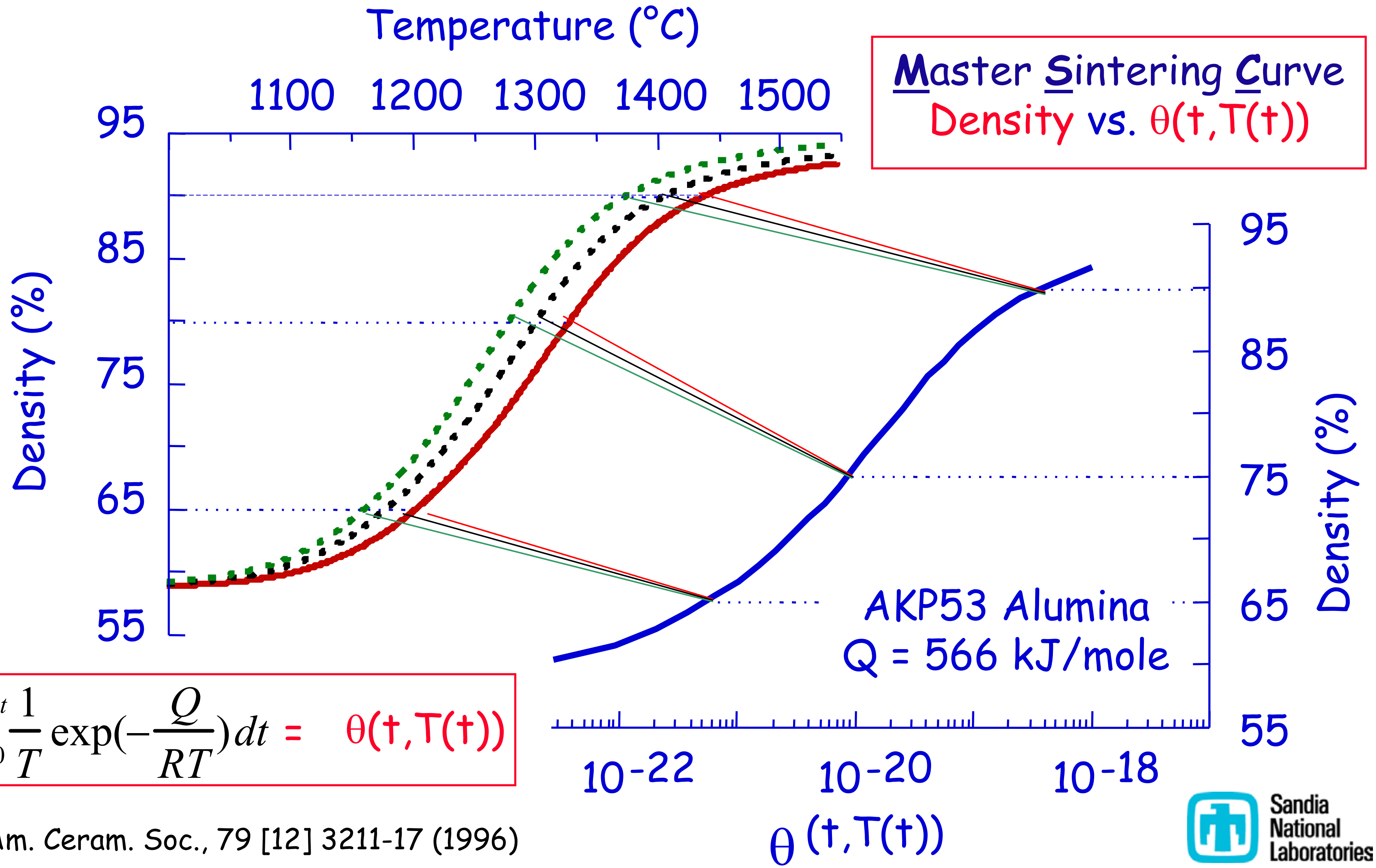
Densification Can Be Predicted And Controlled With The MSC



$$\int_0^t \frac{1}{T}\exp\left(-\frac{Q}{RT}\right)dt = \theta(t,T(t))$$

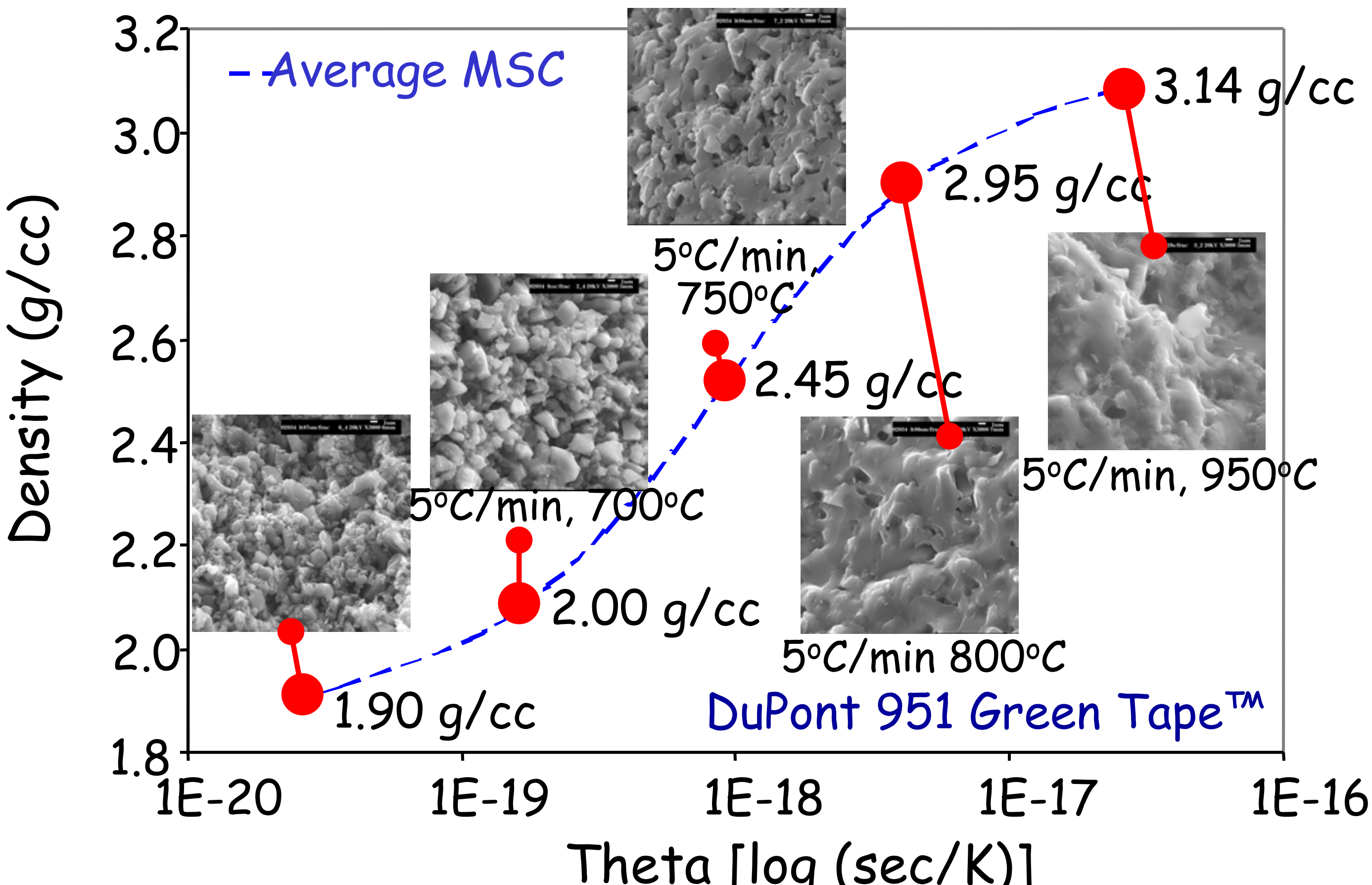
J. Am. Ceram. Soc., 79 [12] 3211-17 (1996)

The MSC Offers Processing, Microstructure, & Property Control

Sintering Time And Temperature

$$\int_o^t \frac{1}{T}\exp(-\frac{Q}{RT})dt \equiv \rho \equiv \frac{k}{\gamma\Omega D_o}\int_{\rho_o}^{\rho}\frac{(G(\rho))^n}{3\rho\Gamma(\rho)}d\rho$$

Microstructure & Materials Properties



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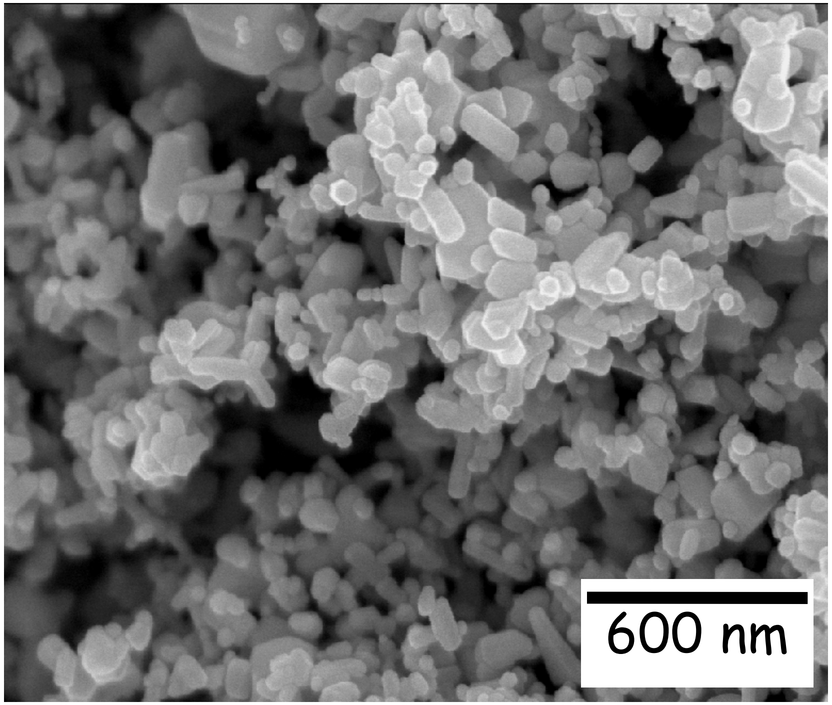
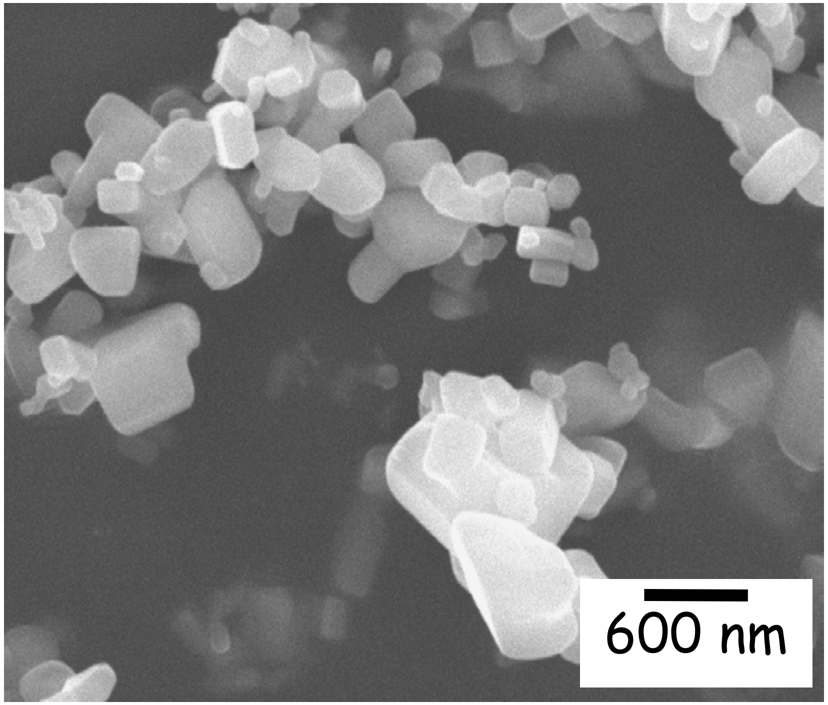
Two Different ZnO Ceramic Powders Were Examined

Nano-Crystalline ZnO



48 nm average particle size
30 m^2/g specific surface area

Micro-Crystalline ZnO



790 nm average particle size
3.2 m^2/g specific surface area

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The ZnO Powders Were Granulated, Pressed, And Sintered

Nano-Crystalline ZnO

Pan Granulated
3 Wt% 50:50 PVA:PEG

Ground and Sieved
<150 μm

Dry Pressed (6 x 6mm dia pellets)
140 MPa uniaxial
210 MPa Isostatic

Binder Burned out
RT - 600°C in Air

Sintered
5, 10, 20, or 30°C/min to 1550°C
Netzsch Dilatometer 402ED

Micro-Crystalline ZnO

Spray Granulated (CBM)
3 Wt% 50:50 PVA:PEG

Sieved
>45μm to <150 μm

Dry Pressed (5 x 6 x 50 mm bars)
18 MPa uniaxial
210 MPa Isostatic

Binder Burned out
RT - 600°C in Air

Sintered (4 x 5 x 10 mm bars)
5, 10, 20, or 30°C/min to 1550°C
Netzsch Dilatometer 402

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62% TD ZnO Powder Compacts Sintered To Greater Than 91% TD

Nano-Crystalline ZnO

Compact Density (w/o binder)
62.6 ± 0.0% TD

Sintered Density
99.2 ± 0.2% TD

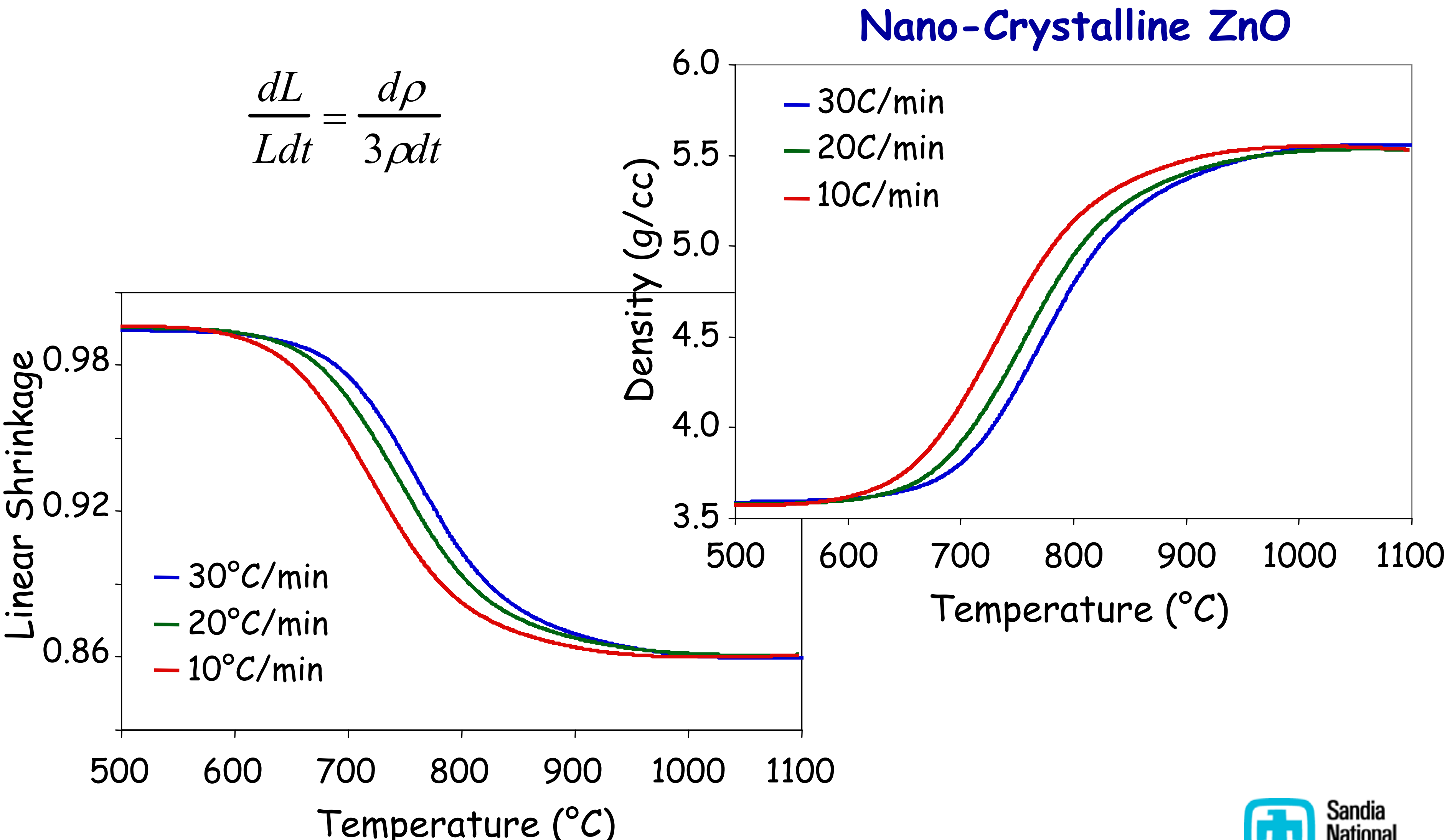
Micro-Crystalline ZnO

Compact Density (w/o binder)
62.2 ± 0.0% TD

Sintered Density
91.3 ± 0.3% TD

Sintered Density Was Determined From Measured Linear Shrinkage

$$\frac{dL}{Ldt} = \frac{d\rho}{3\rho dt}$$



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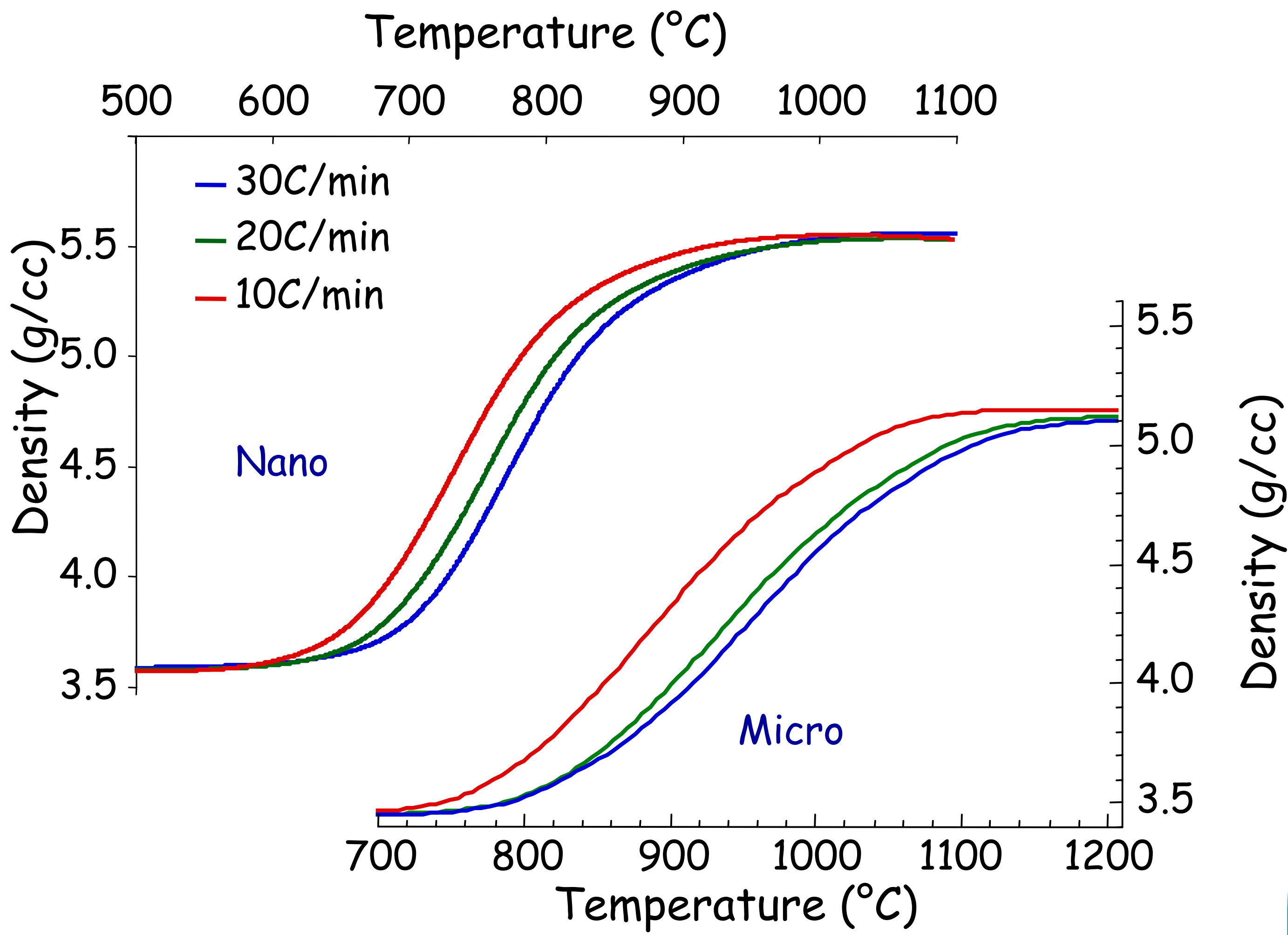
Nano-Crystalline ZnO Sinters Faster Than Micro-Crystalline ZnO

Crystalline ZnO sintering



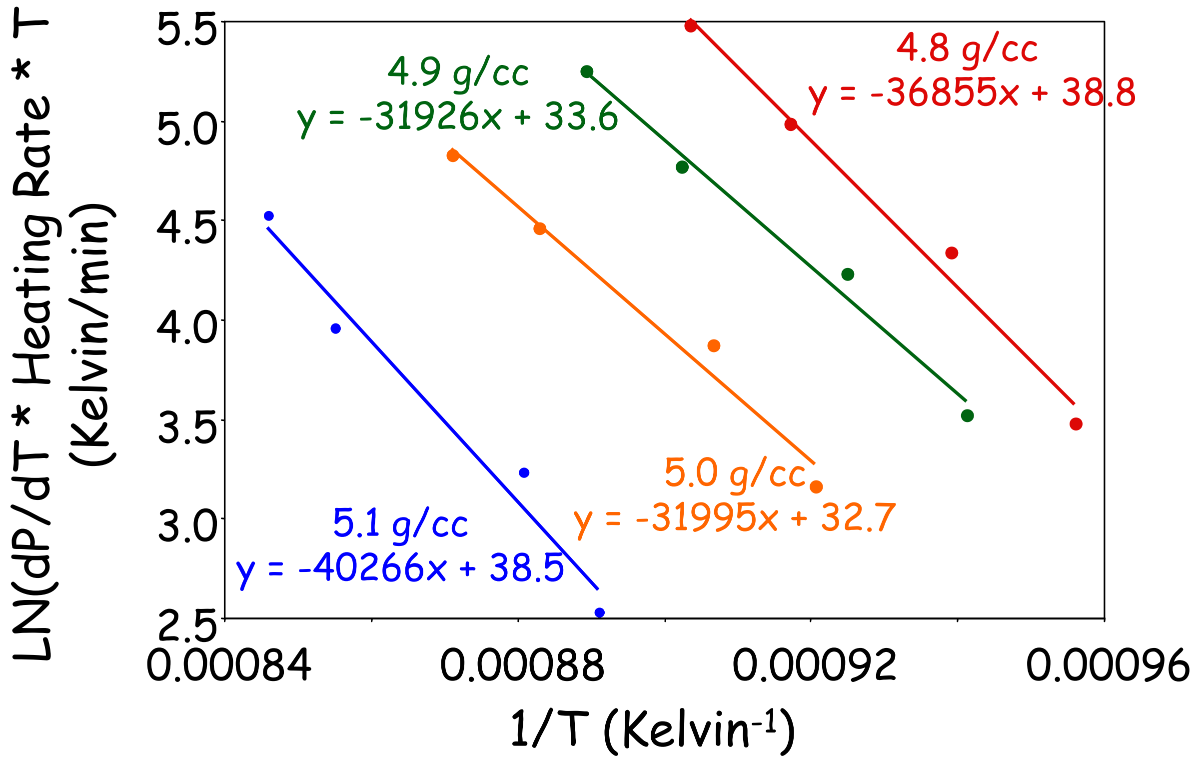
A Consistent Activation Energy (Q) Was Determined For ZnO Sintering

*Nano-Crystalline ZnO

Q for Densification (65-90%TD)
268 ± 25 kJ/mole

Micro-Crystalline ZnO

Q for Densification (65-90%TD)
296 ± 21 kJ/mole



Q = slope * R

Q = 276 ± 13 kJ/mole Gupta & Coble - J Am Ceram Soc 1968

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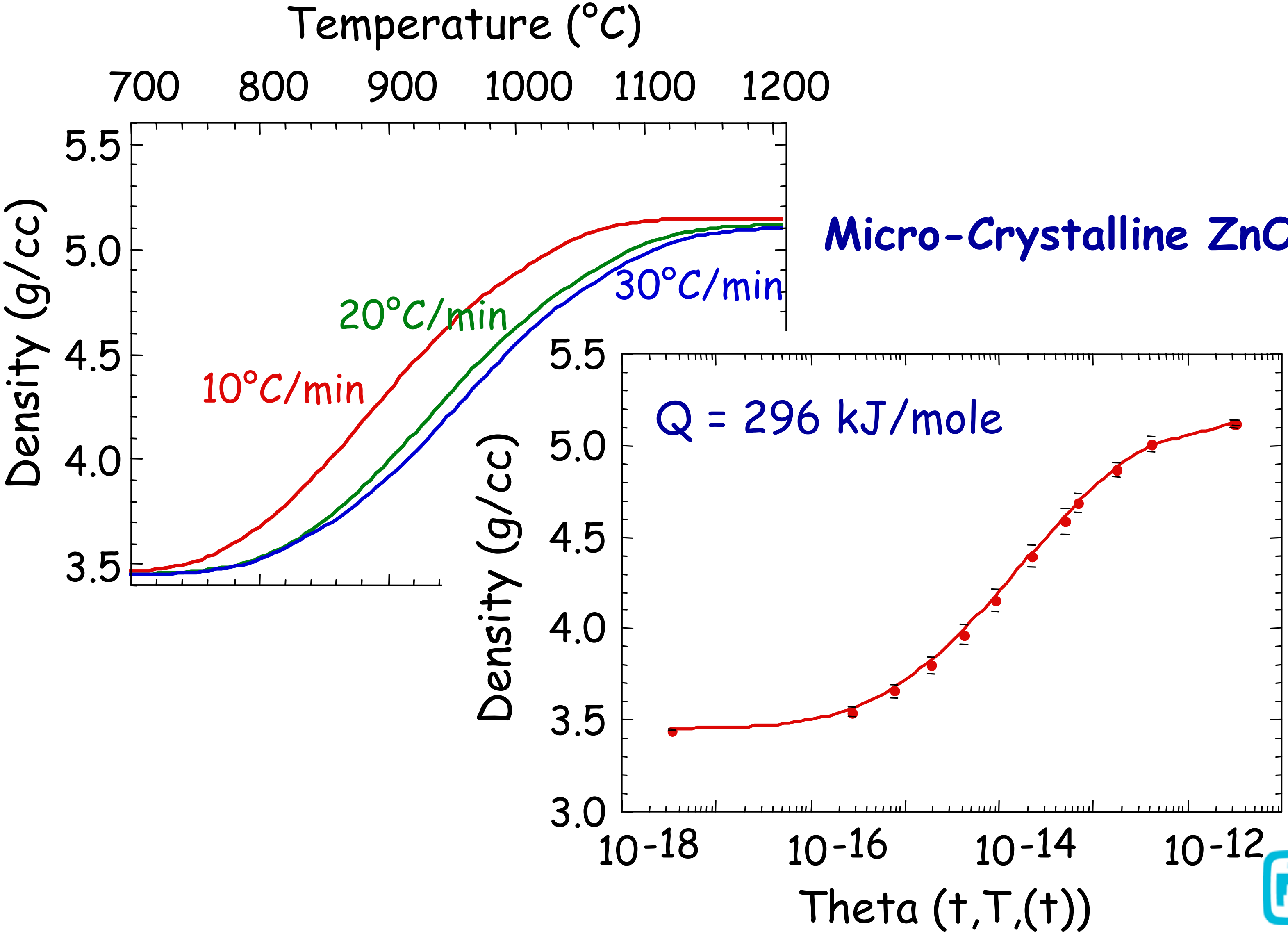
A Master Sintering Curve Was Constructed For Micro-Crystalline ZnO



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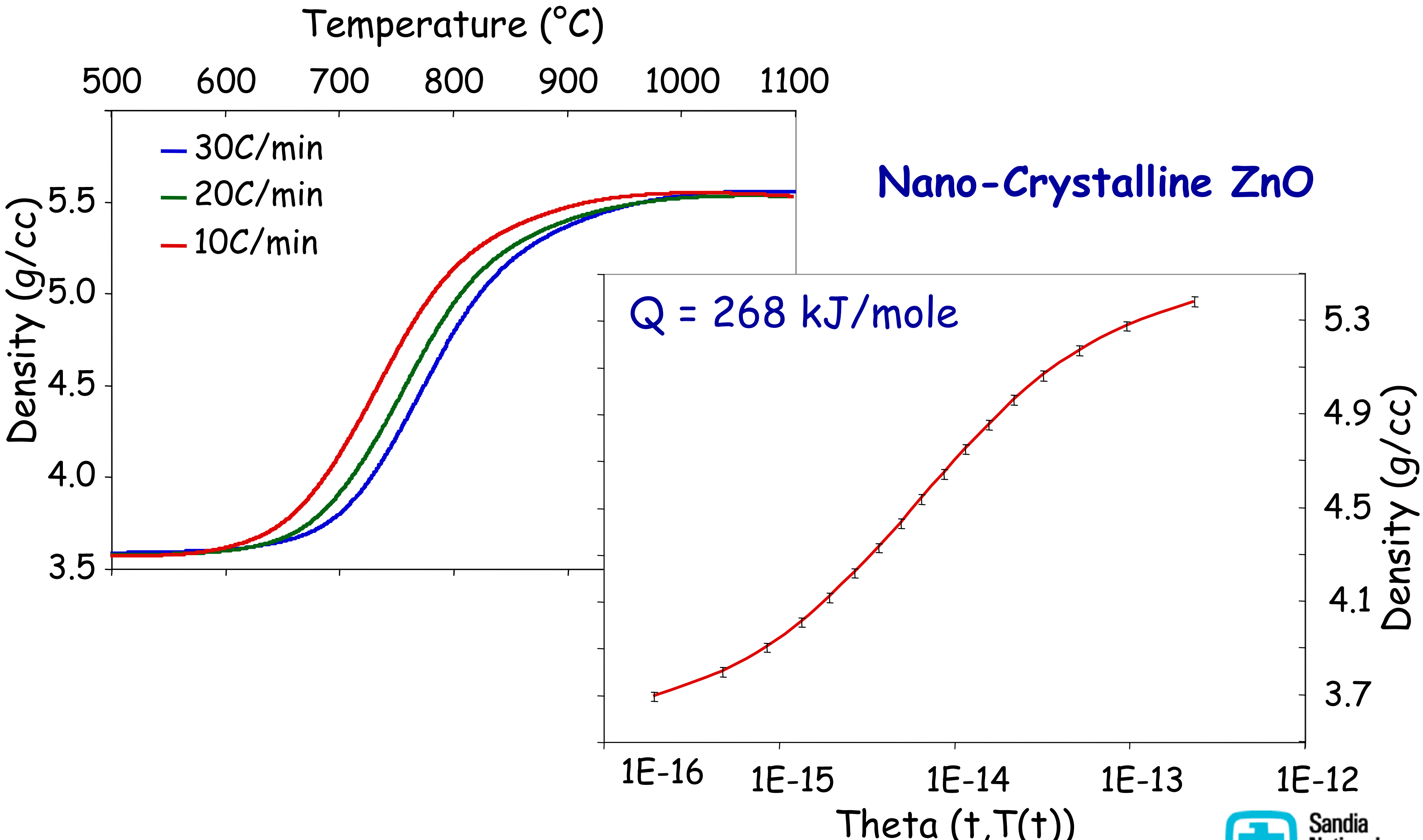
A Master Sintering Curve Was Constructed For Nano-Crystalline ZnO



Nano-Crystalline ZnO

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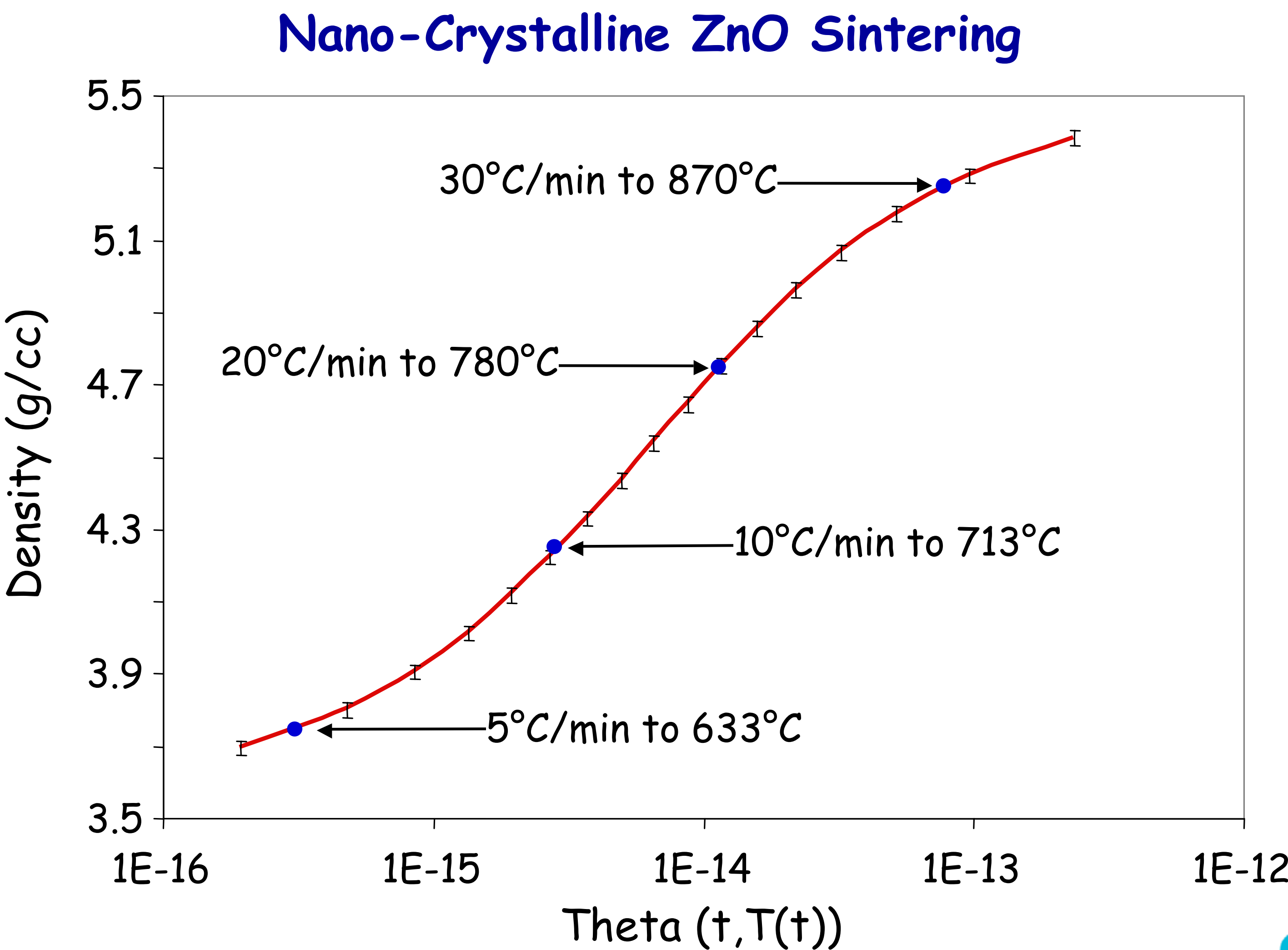
Master Sintering Curve Predictions Were Tested & Validated



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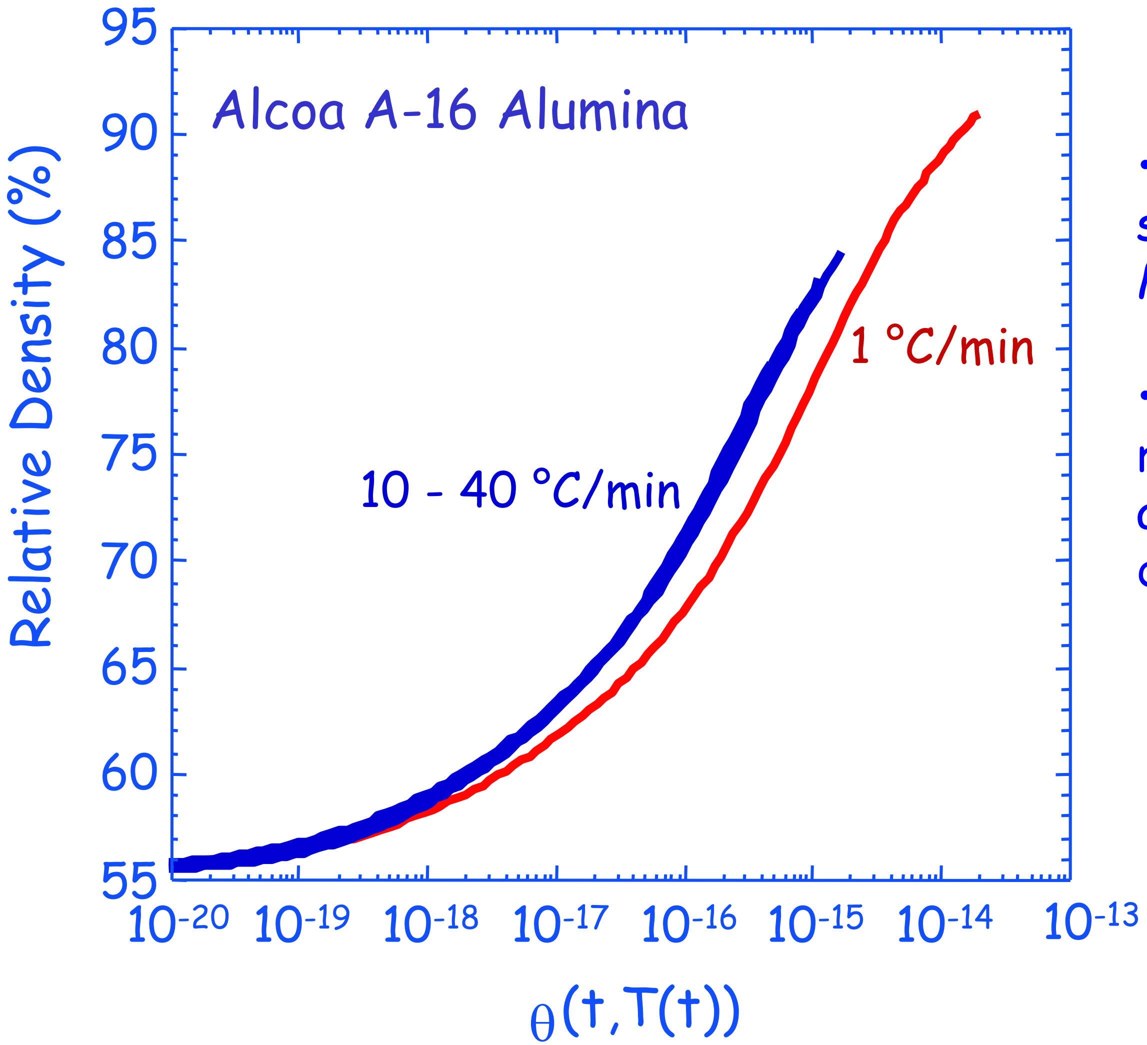
A Slower Heating Rate May Alter Sintering And The MSC



- A-16 Al_2O_3 Sshows seviation from the MSC at 1°C/min.
- Surface diffusion results in coarsening at the expense of densification.

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MSC Theory Was Applied To Nano-Crystalline ZnO Sintering

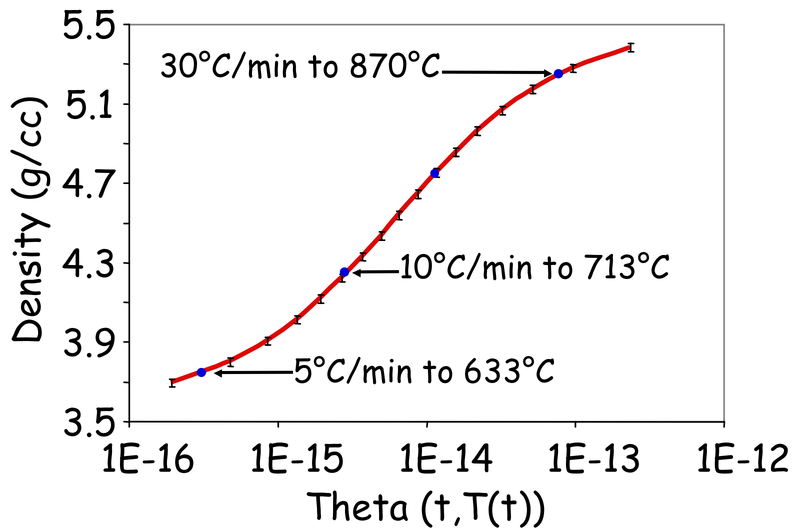
Nano-Crystalline ZnO Sintering

48 nm average particle size
30 m^2/g specific surface area

62.6% TD compacts sintered to 99.2% TD

Q = 268 ± 25 kJ/mole

A master sintering curve (MSC) was constructed



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