

# Glass Density Effects in Glass-To-Metal Sealing

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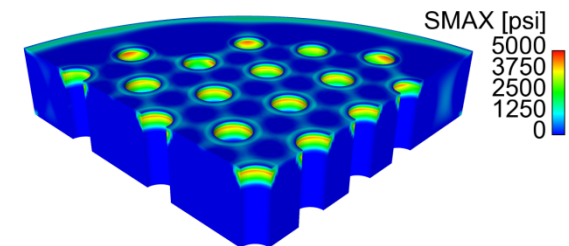
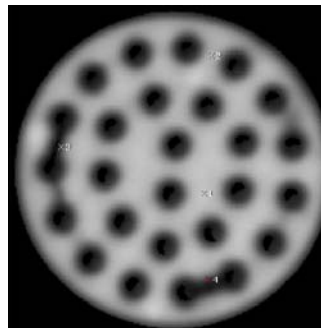
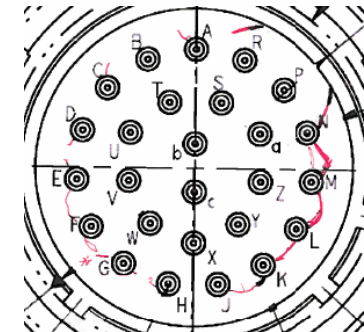
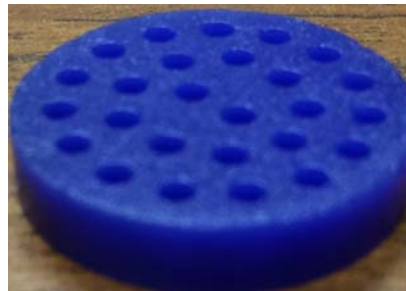
Rio Grande Regional  
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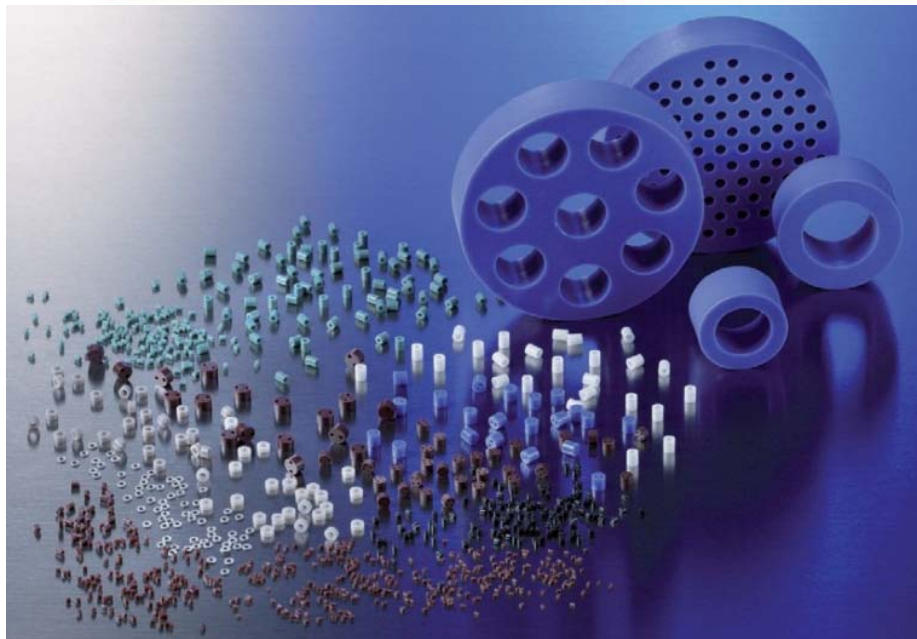
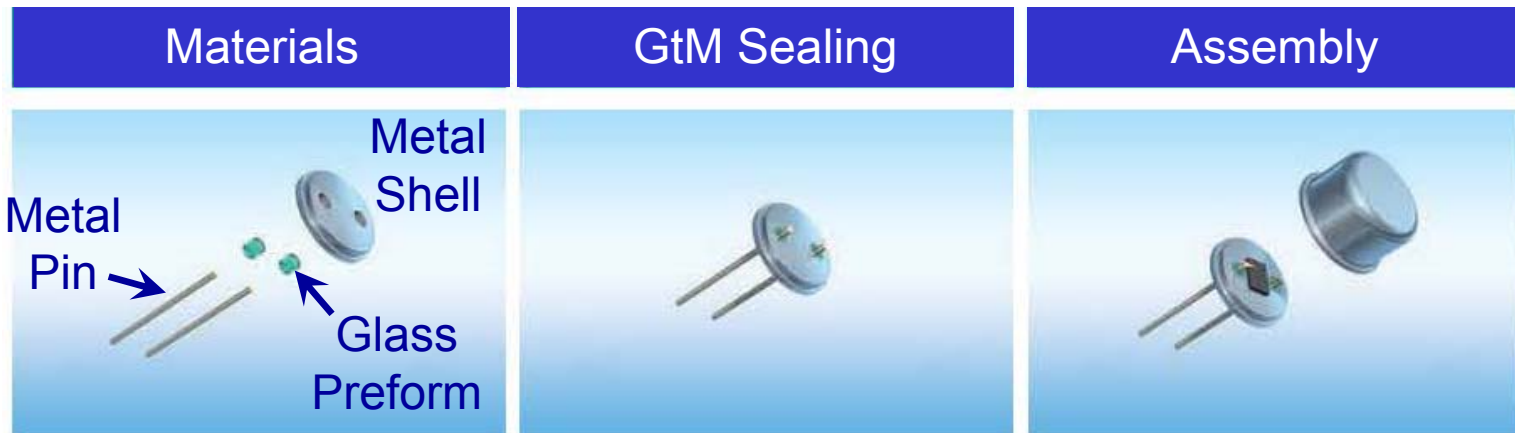
v141001e

# Glass to Metal (GtM) Seals Are Used To Make Hermetic Electrical Feed-Throughs

- GtM Seal Applications
  - Automotive
  - Aviation
  - Telecommunications
  - Medical
  - Energy (Fuel Cells)



# Glass Preforms Are Fabricated, Assembled, And Reflowed To Produce A GtM Seal



## ■ Glass Preforms

### ○ Types

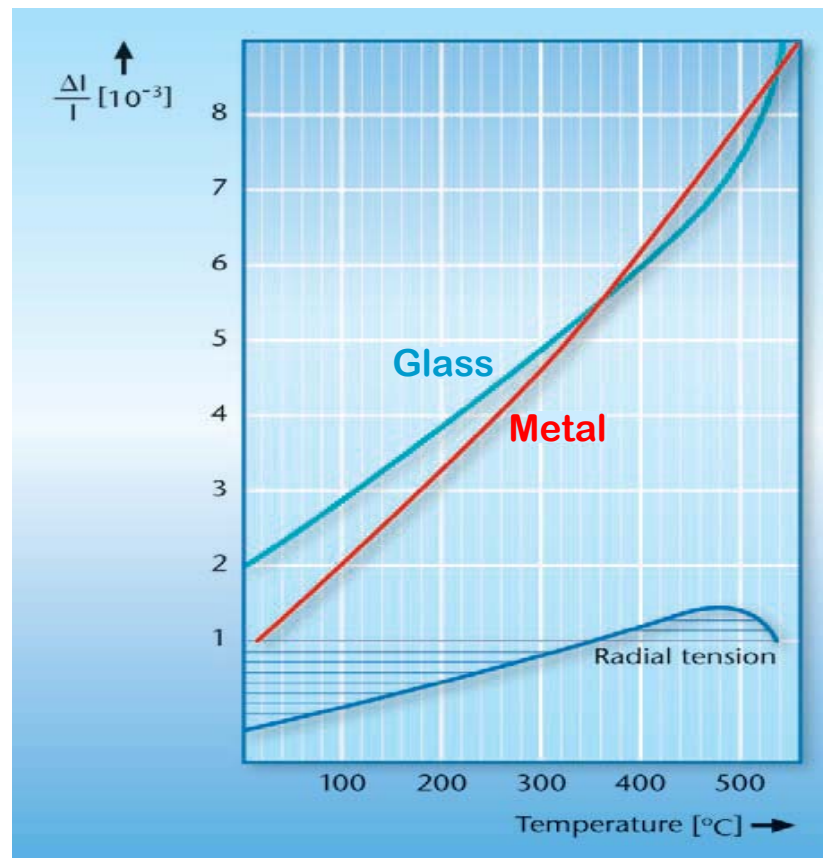
- (solid) glass tube
- (porous) sintered powder preforms

### ○ Desirable Characteristics

- Precise dimensions & mass
- Smooth Surfaces

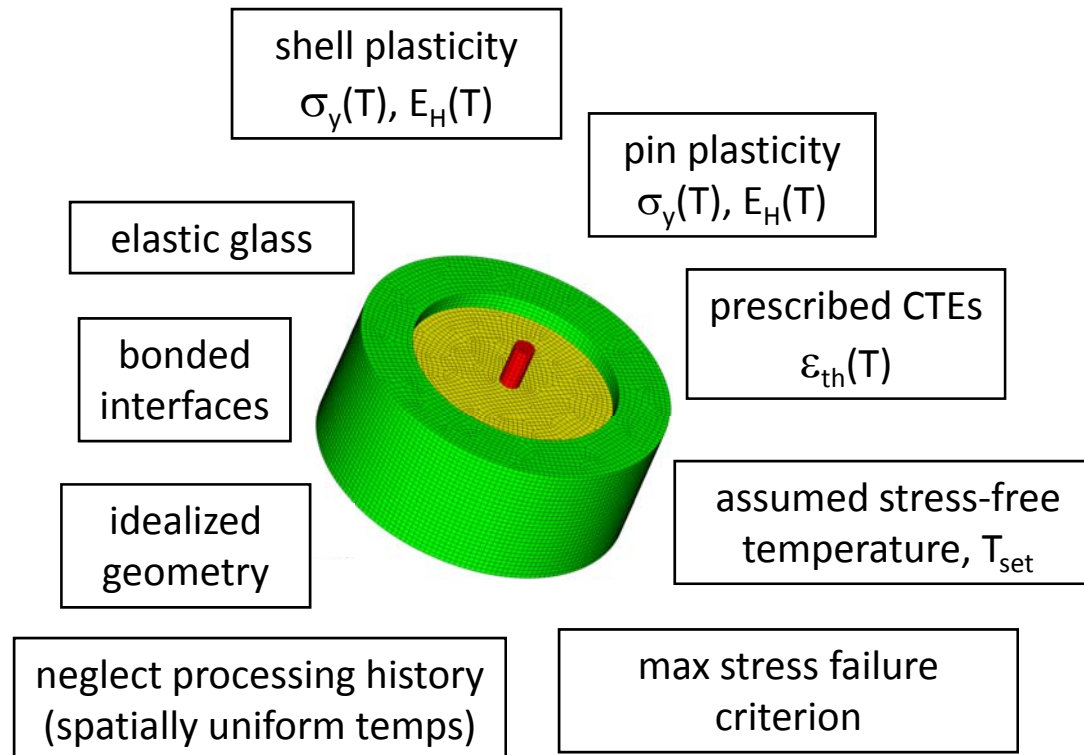
# A Compression Seal Is Produced When The $CTE_{\text{metal}} > CTE_{\text{glass}}$

- Compression GtM Seal
  - Corrosion & Pressure Tolerant



Heating/Cooling  
Changes The  
Stress In The  
Glass Seal

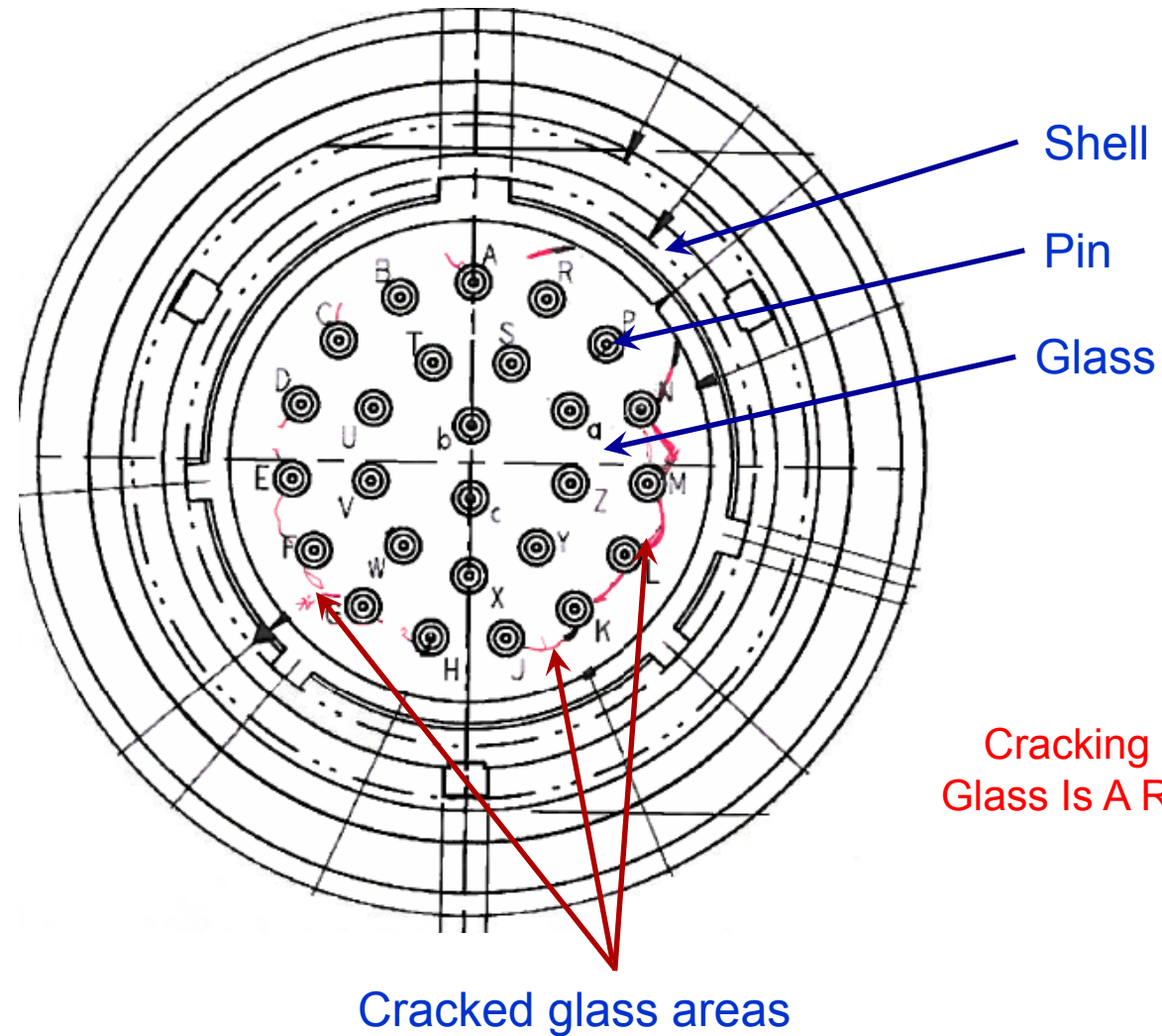
# Finite Element (FE) Stress Predictions Are Used To Assess GtM Seal Performance & Reliability



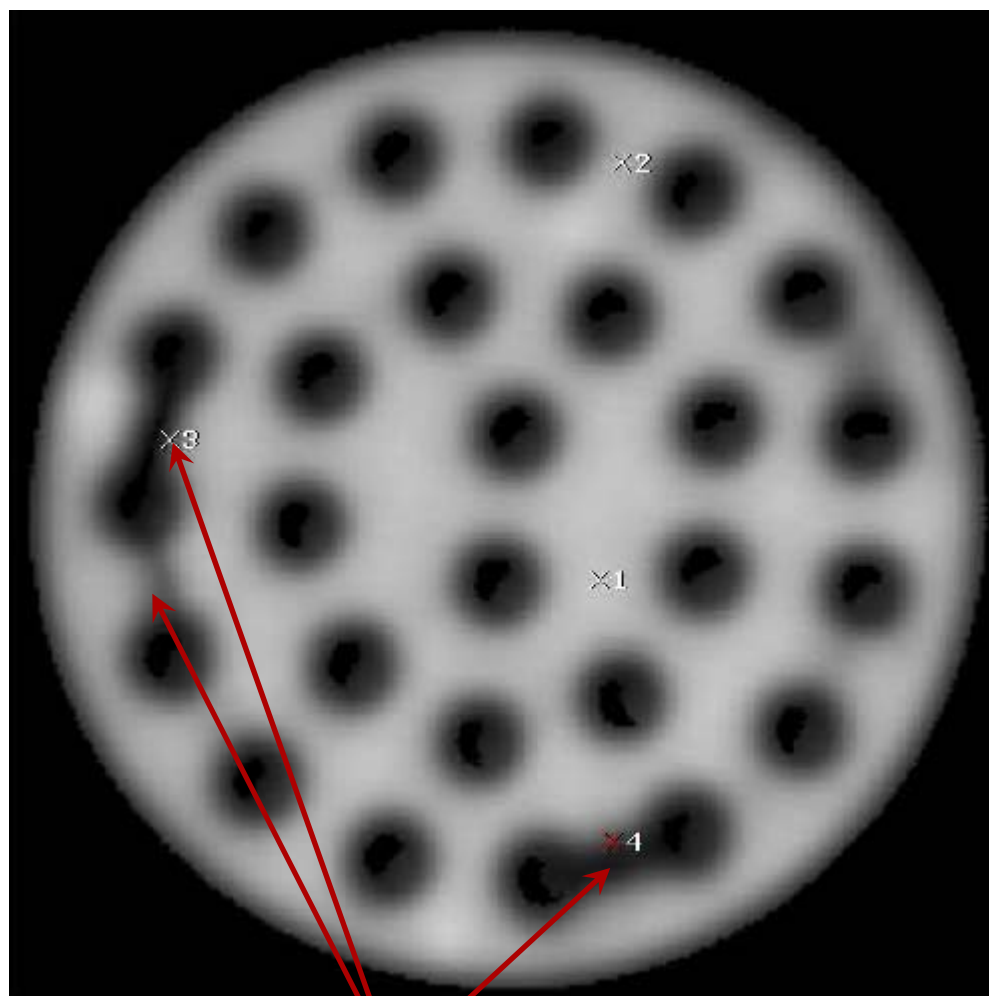
Materials/Modeling Assumptions Determine  
The Fidelity Of FE Stress Predictions



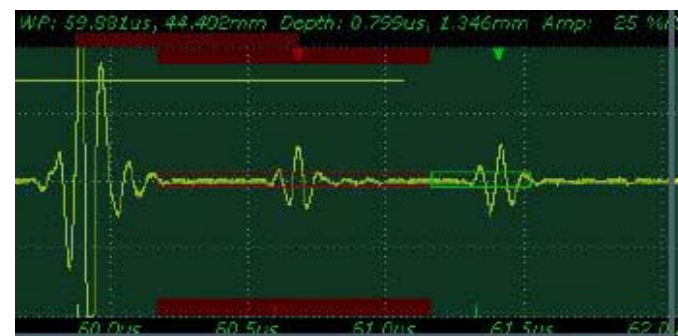
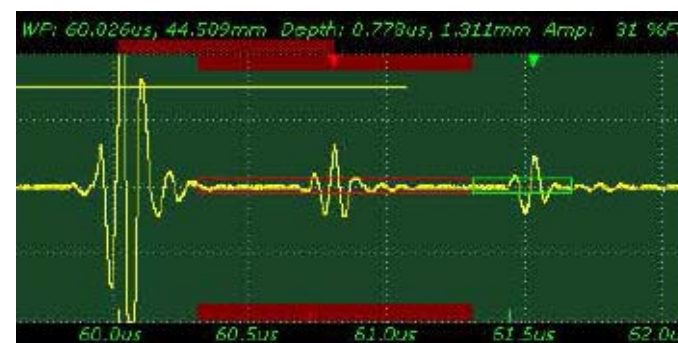
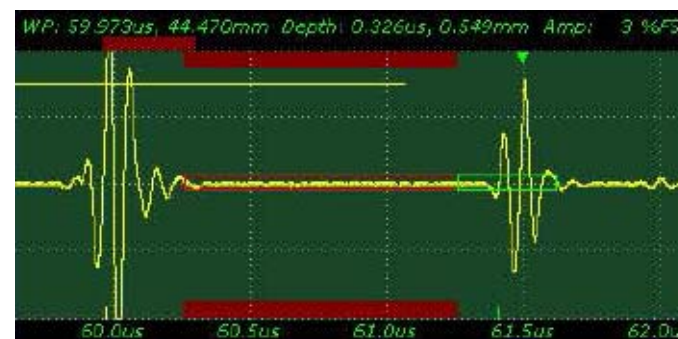
# Post Stress Test Visual Inspection Identified Cracks Between The Outer Pins



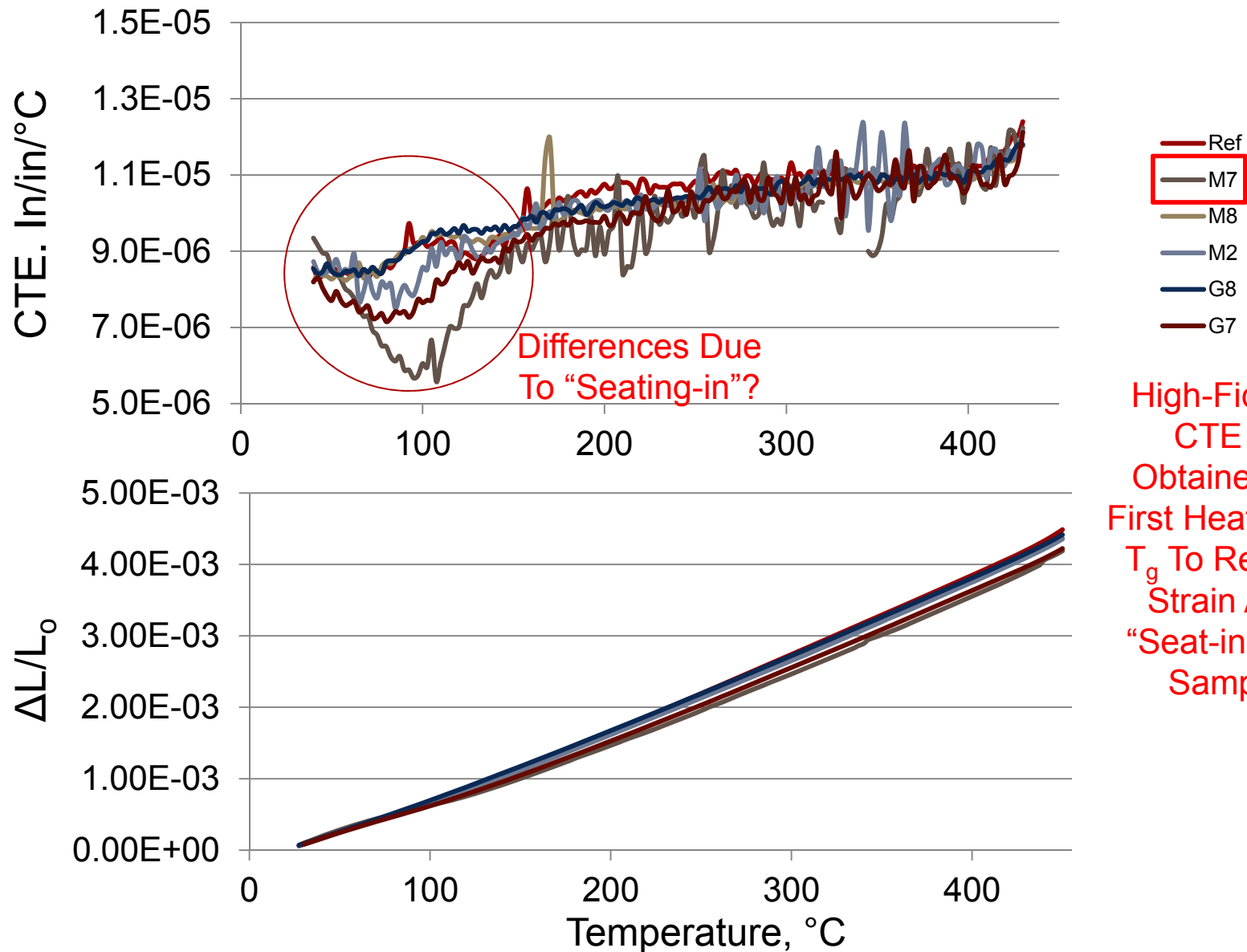
# Post Stress Test Ultrasound Imaging Revealed Low Density Areas Between The Outer Pins



Low-density regions



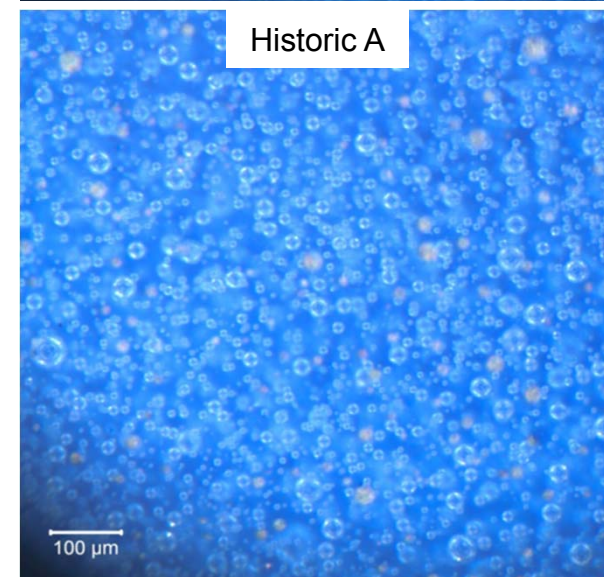
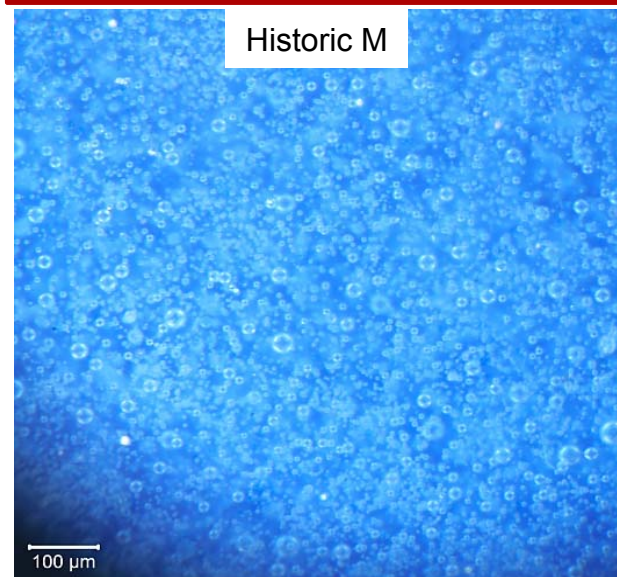
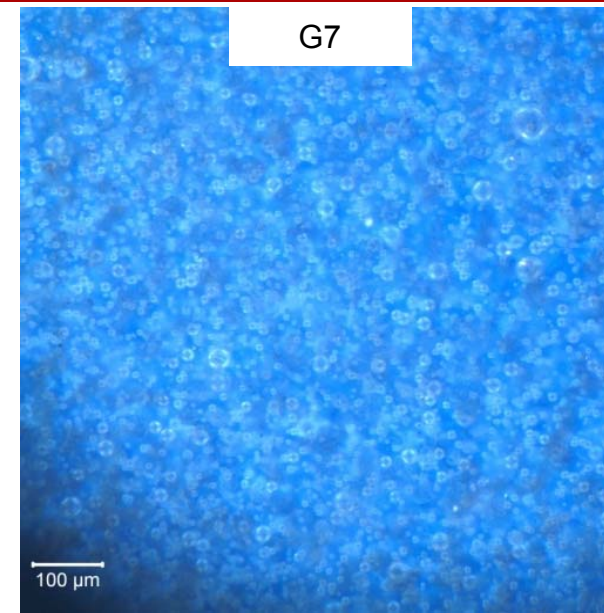
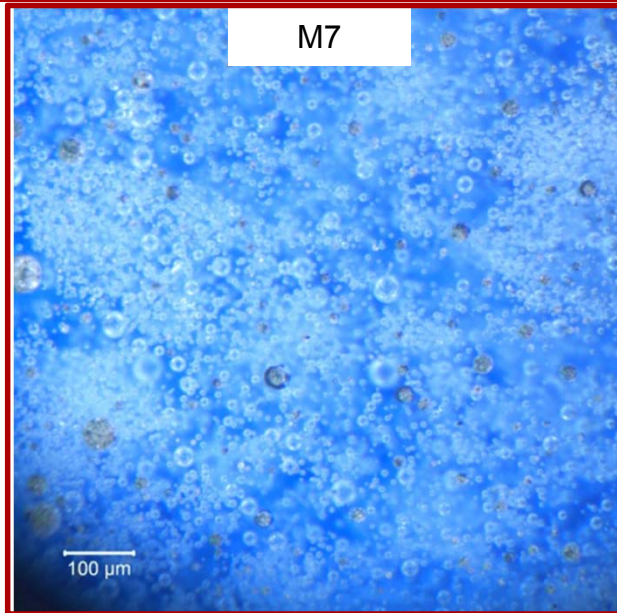
# Heating Coefficient Of Thermal Expansion Curves Show No Significant Differences



High-Fidelity  
CTE Is  
Obtained By  
First Heating To  
 $T_g$  To Relieve  
Strain And  
"Seat-in" The  
Sample

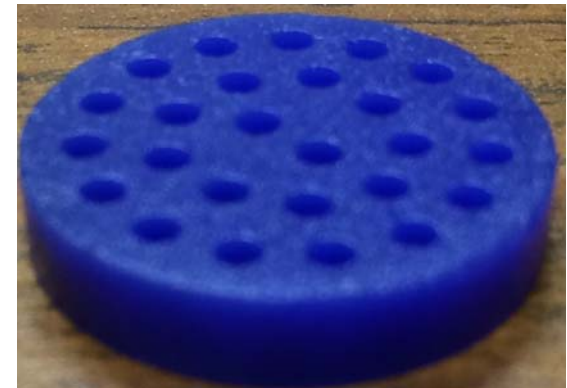


# The M4047 Glass Looks Heterogeneous & “Cloudy” After Sealing

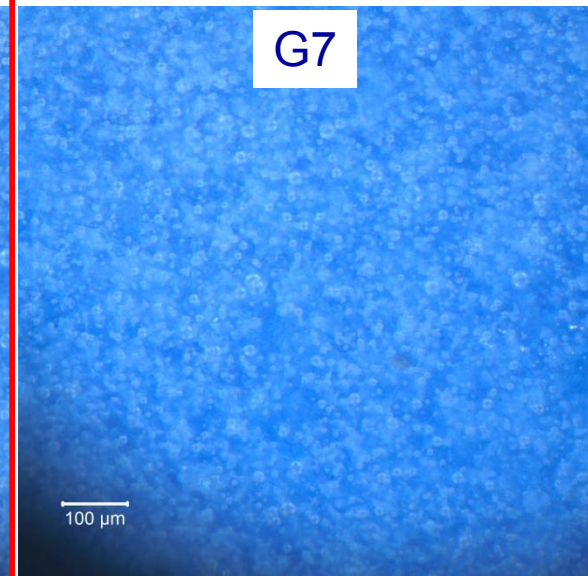
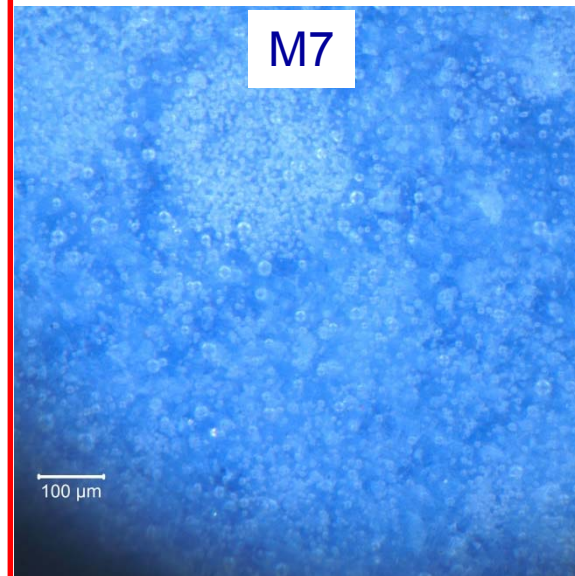


# Preform Processing Affects The Look Of The Glass Before & After Sealing

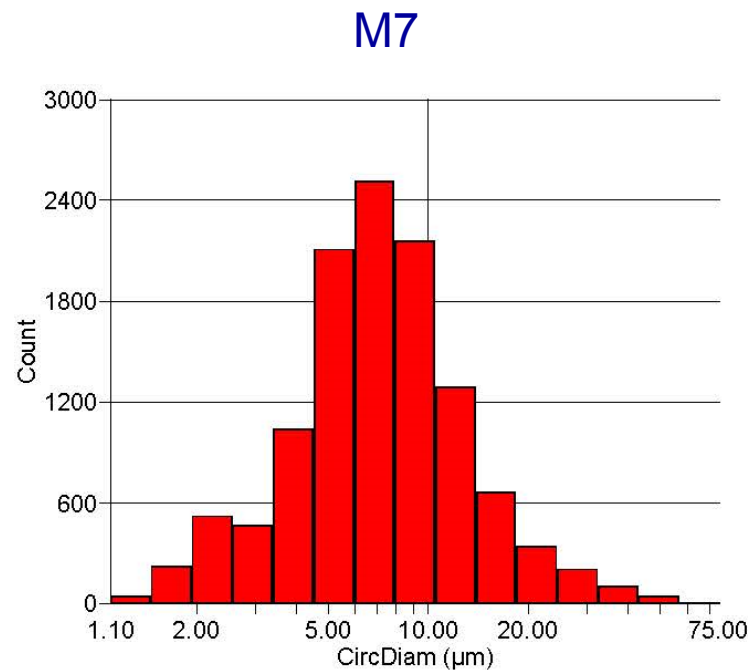
Preform →



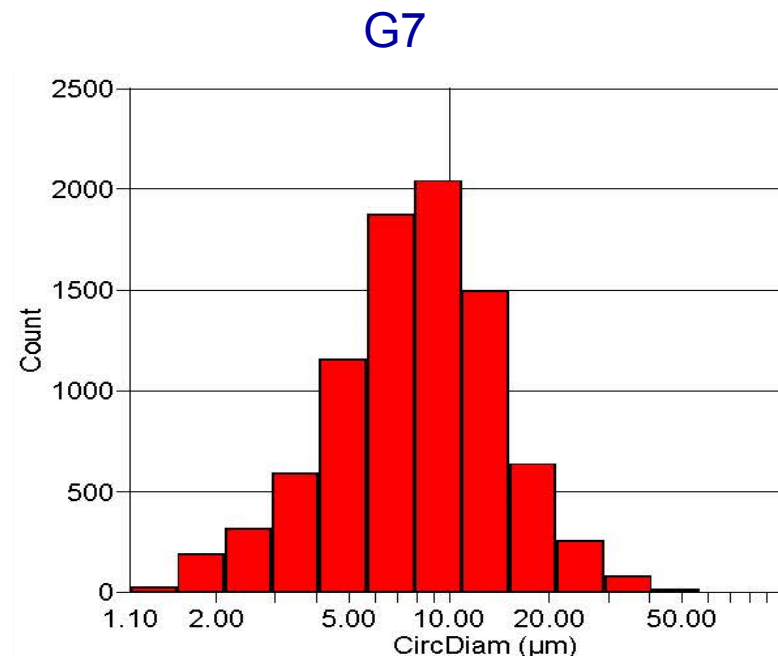
After Sealing →



# Quantitative Stereology Shows Only Minor Differences in Pore Size/Distribution



Minimum:	1.14	$\mu\text{m}$
Maximum:	71.35	$\mu\text{m}$
Mean:	8.53	$\mu\text{m}$
Std Dev.:	6.26	$\mu\text{m}$
Sum:	99943.62	$\mu\text{m}$
Count:	11717	
Under:	0	
Over:	0	
Accepted:	100.00	%
Field Count:	10	
Field Area:	1464348.54	$\mu\text{m}^2$
Total Area:	14643485.36	$\mu\text{m}^2$
D10:	3.13	$\mu\text{m}$
D50:	7.00	$\mu\text{m}$
D90:	14.80	$\mu\text{m}$



Minimum:	1.14	$\mu\text{m}$
Maximum:	108.43	$\mu\text{m}$
Mean:	9.29	$\mu\text{m}$
Std Dev.:	5.84	$\mu\text{m}$
Sum:	80512.02	$\mu\text{m}$
Count:	8666	
Under:	0	
Over:	0	
Accepted:	100.00	%
Field Count:	10	
Field Area:	1464348.54	$\mu\text{m}^2$
Total Area:	14643485.36	$\mu\text{m}^2$
D10:	3.73	$\mu\text{m}$
D50:	8.14	$\mu\text{m}$
D90:	15.71	$\mu\text{m}$

QS Does  
Not  
Discern  
The  
Differences  
Seen By  
Eye



# Cracked Parts Have Lower Preform And Glass Seal Density



A High Density Preform  
Is Required To Produce  
A High Density Seal

# Glass Microstructure/Pore Shape Change During Sealing

Preform

Seal

M7

Irregular shape  
angular pores

Spherical  
pores

G7

Irregular shape  
pores

Spherical  
pores

50  $\mu$ m

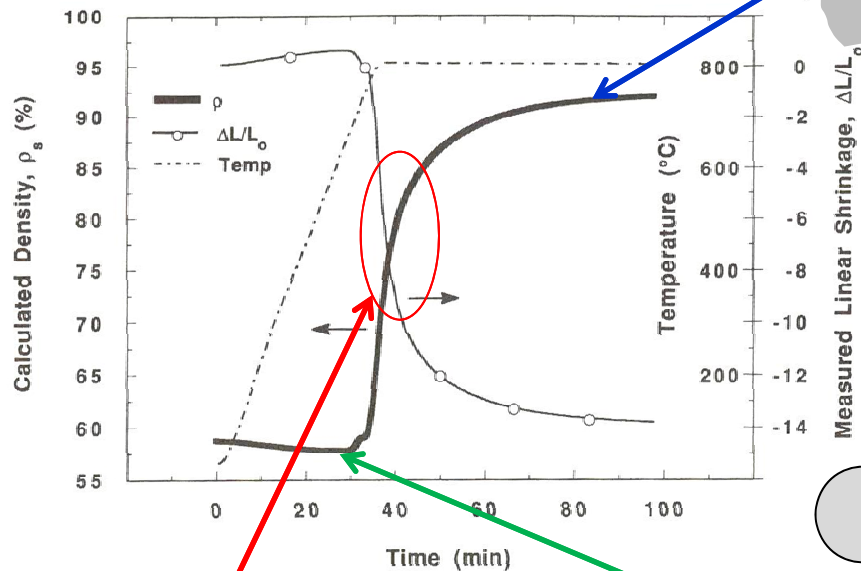
50  $\mu$ m



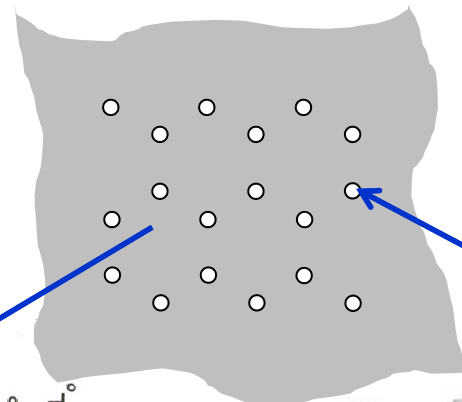
# Pore Size & Shape Change During Sintering, And Indicate Microstructure Maturity

## ■ Typical Sintering

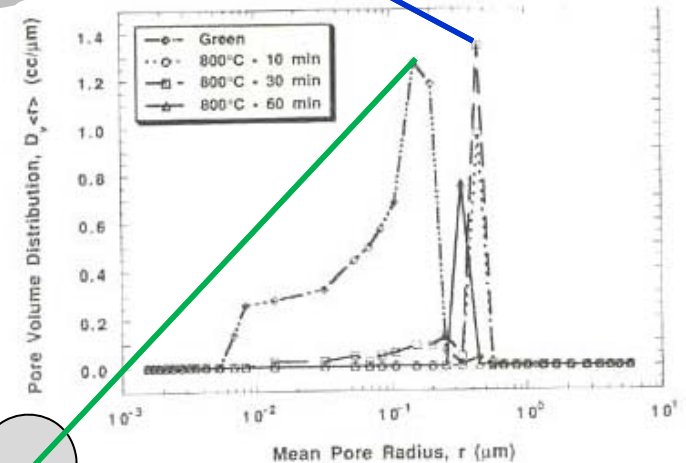
- Angular pores spheroidize
- Shrinkage of part & pores
  - Fine pores eliminate preferentially
  - Closed pores swell
- Densification



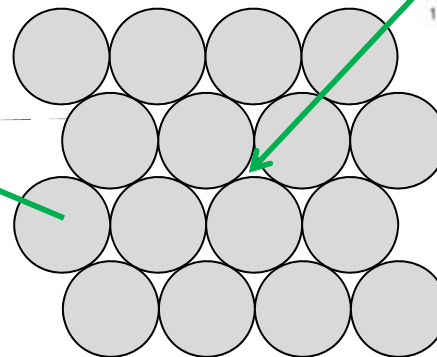
Angular Porosity Indicates An Immature Microstructure And High Manufacturing Variability



Seal

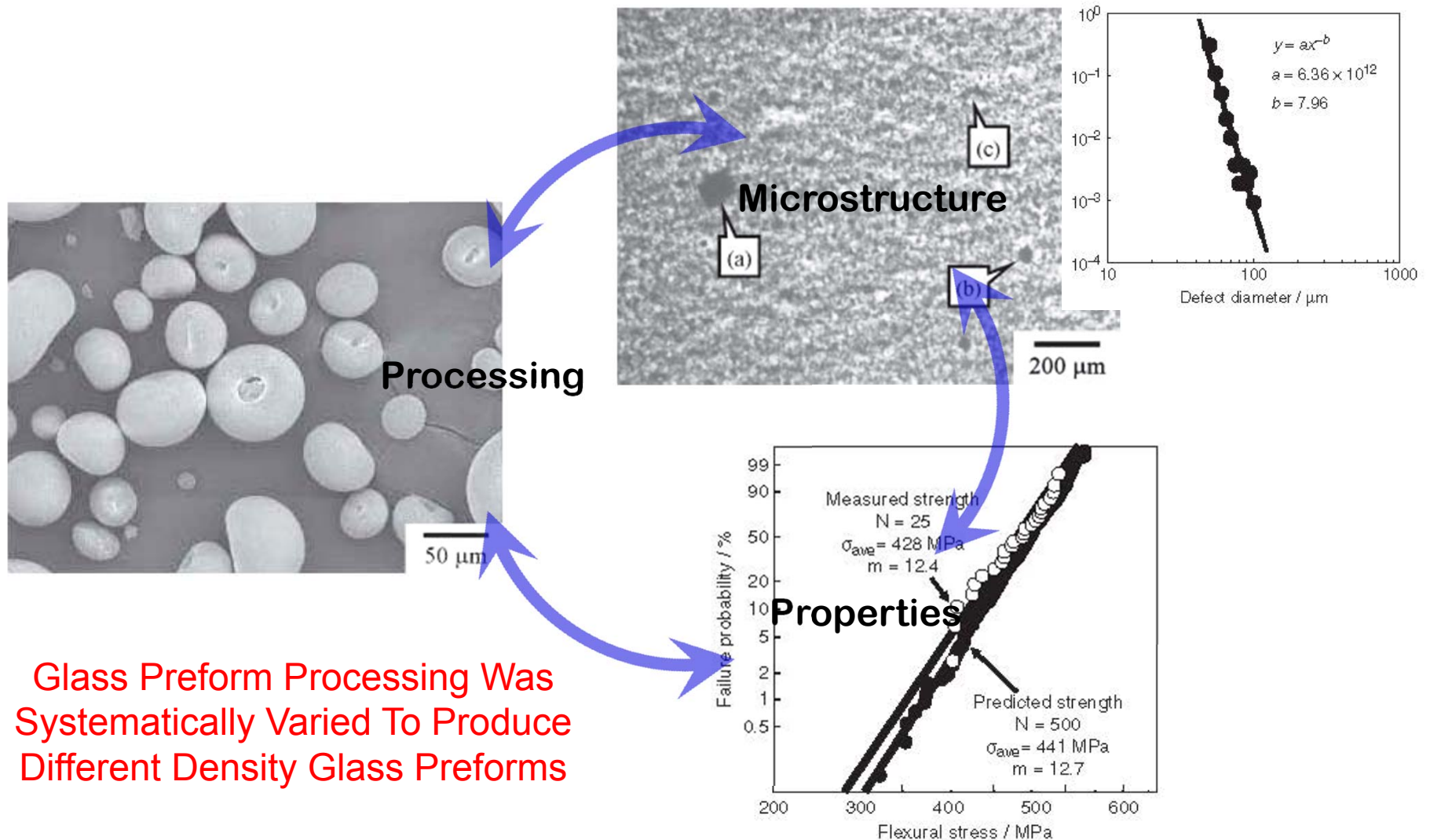


Preform



Ewsuk, "Consolidation of Bulk Ceramics," in Characterization of Ceramics, ed. Loehman Butterworth-Heinemann, Greenwich, CT 1993

# Process-Structure-Property Understanding & Control Are Critical To Performance & Reliability



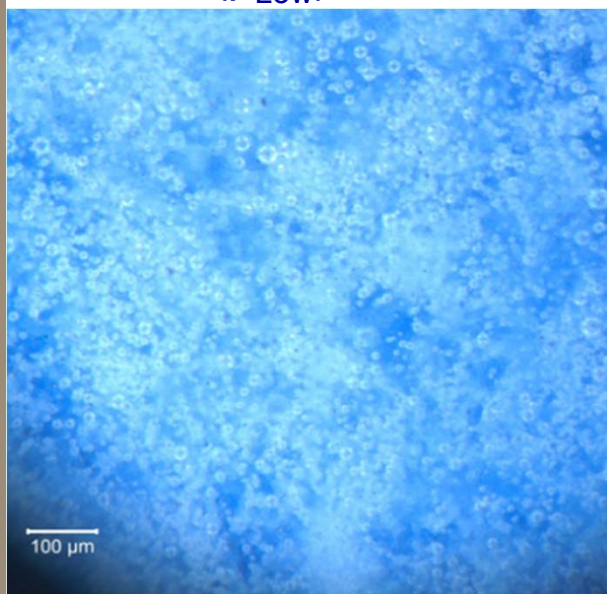
Glass Preform Processing Was Systematically Varied To Produce Different Density Glass Preforms

S. Nakamura, S. Tanaka, Z. Kato, & K. Uematsu, "Strength-Processing Defects Relationship Based on Micrographic Analysis & Fracture Mechanics in  $\text{Al}_2\text{O}_3$  Ceramics," J. Am. Ceram. Soc., 92 [3] 688–693 (2009).

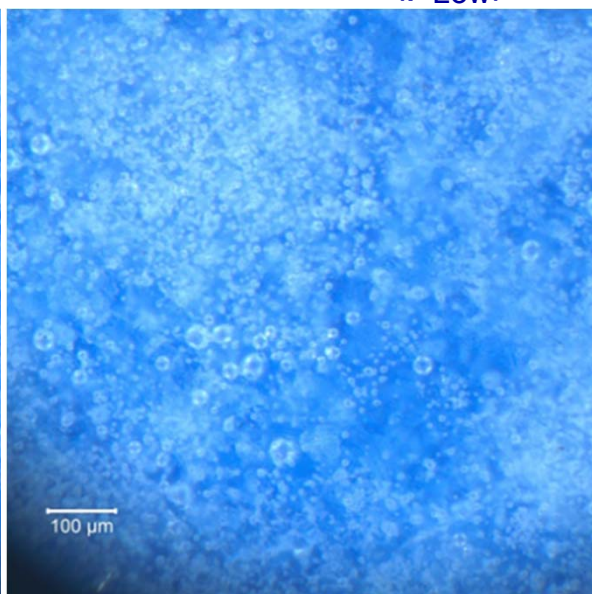
# Systematic Process Changes Did Not Significantly Affect Bubble Size/Distribution

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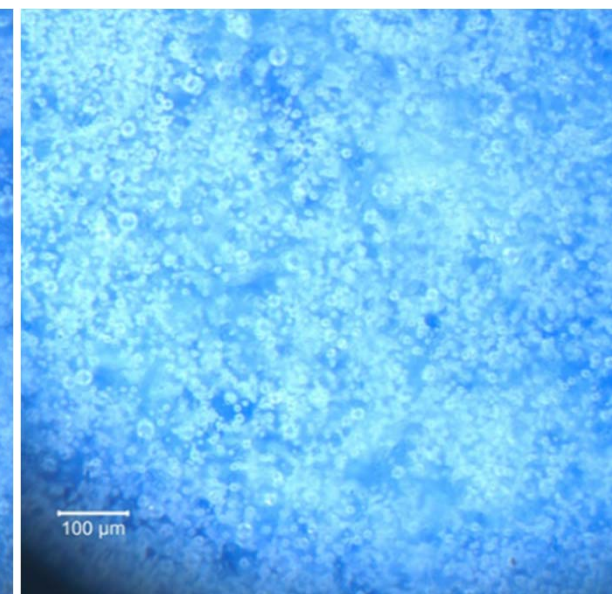
Md1 ( $\rho_{\text{Low}}$ ) Preform



Reflowed Md2 ( $\rho_{\text{Low}}$ )



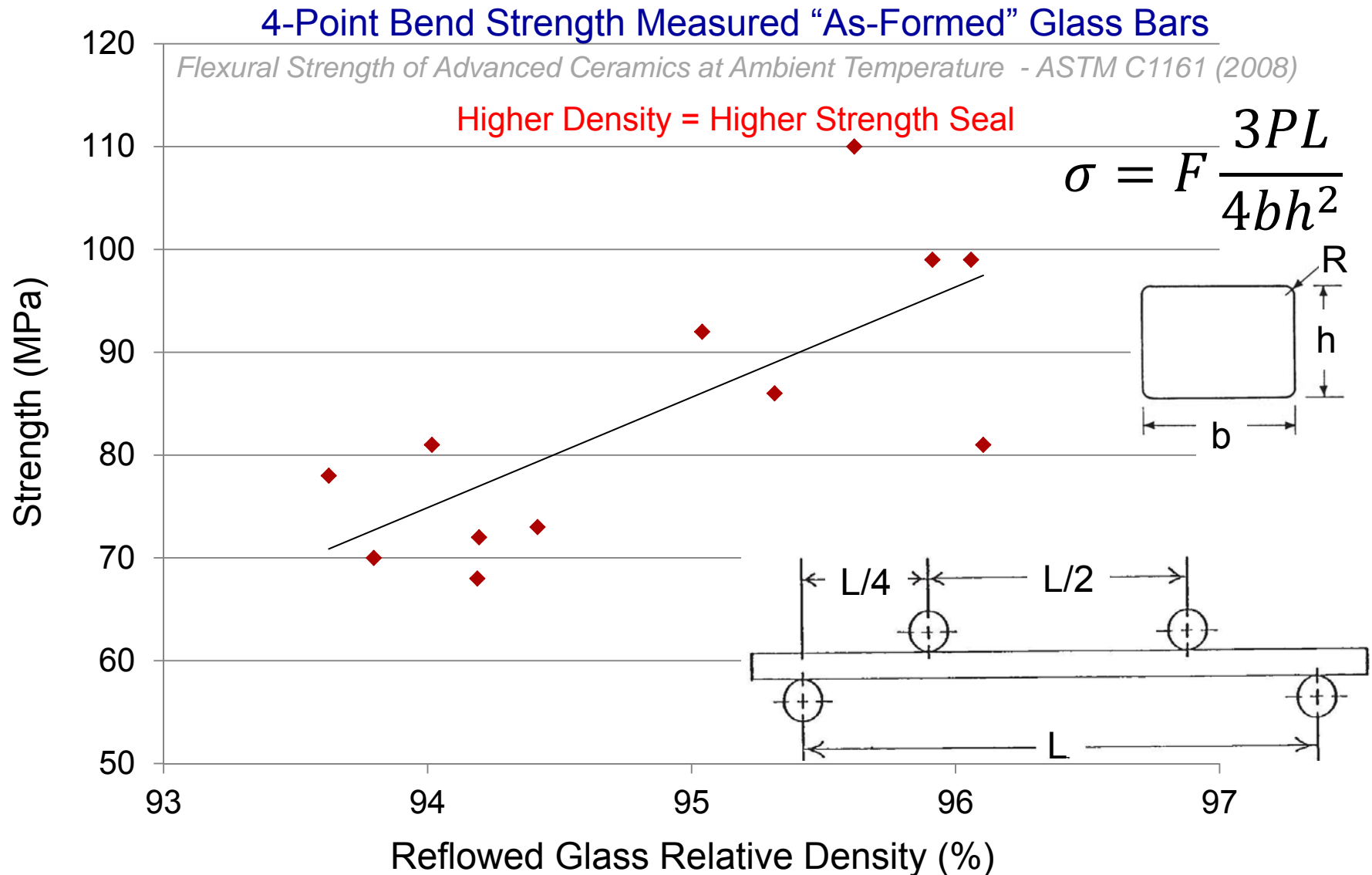
Reflowed Md2



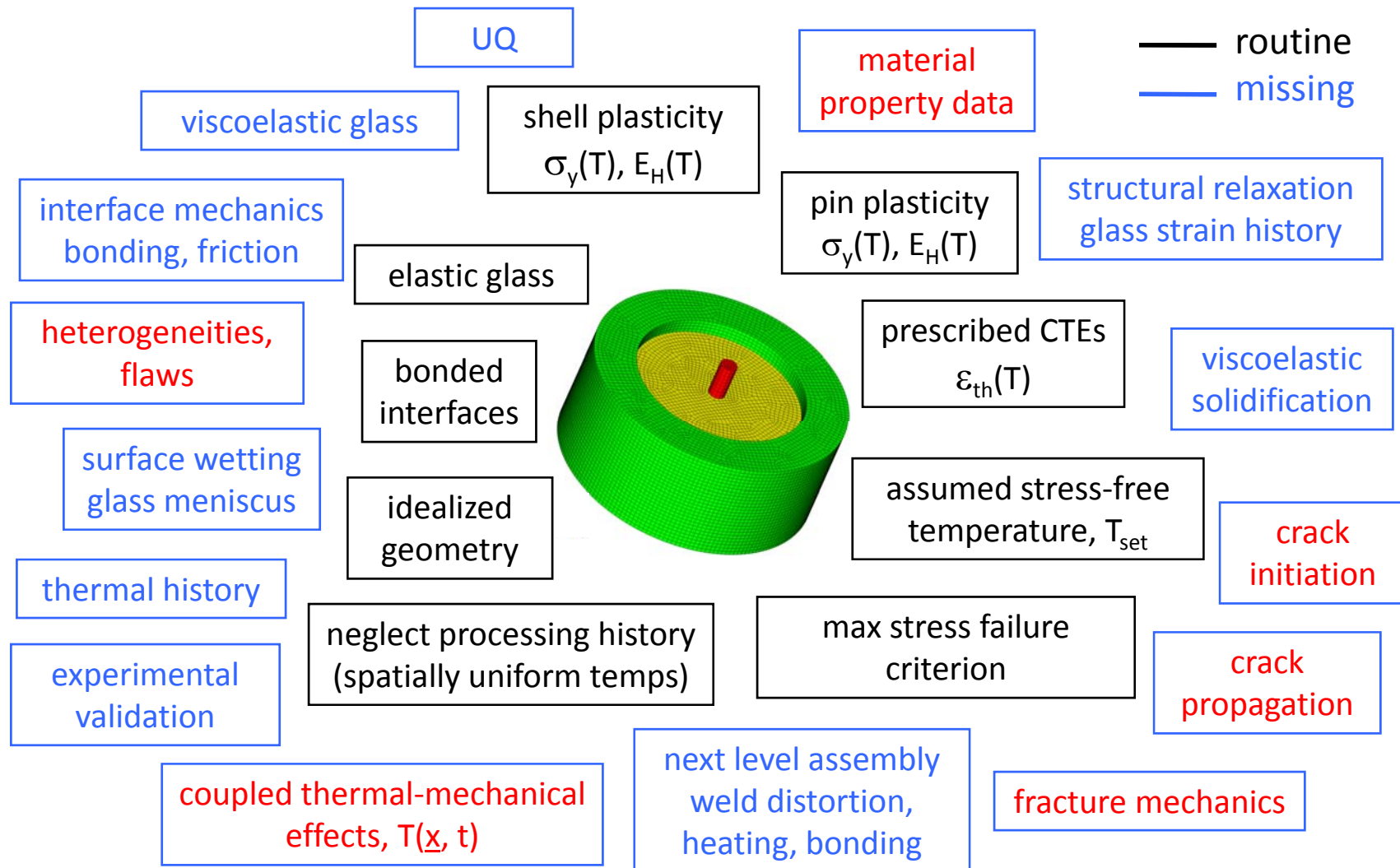
## Potential Supply Chain Issues

- Lot-to-Lot Process Variability
- Limited Process Understanding & Control  
(it was not possible to replicate the low preform density)

# Reflowed/Sealed Glass Strength Trends Up With Density



# Residual Stress & Cracking Will Be Affected By Thermo-Mechanical, & Structure-Properties



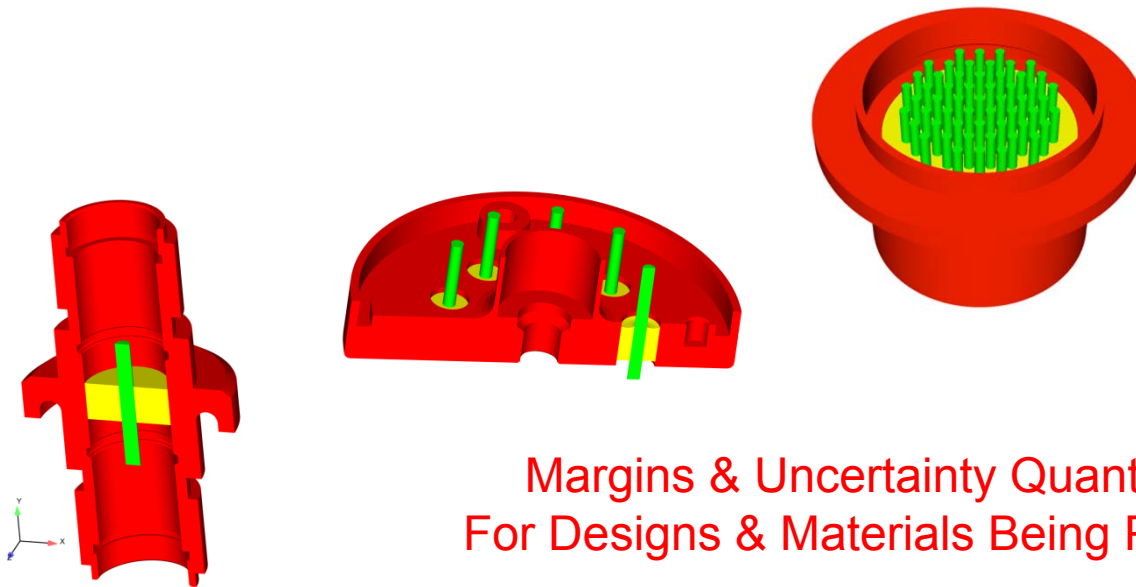
The Seal Geometry, Materials, Processing, & Model Details Are Critical For High-Fidelity Stress, Performance, & Reliability Predictions



# GtM Sealing/Manufacturability Improves With Glass Preform Consistency/Quality

## ■ What's Different With GtM Seals Today?

- More complex designs/geometries
  - more pins & tighter spacing
- More demanding requirements
  - Higher stress, longer lifetime
- Approaching materials & processing limits



Margins & Uncertainty Quantification Are Critical  
For Designs & Materials Being Pushed To Their Limits

# Summary & Conclusions

## ■ Summary

- Stress-Testing resulted in glass seal cracking – a Red Flag
- No materials differences indicated by CTE measurements
- Process differences indicated by density & microstructure measurements
  - Preform variability due to insufficient process understanding/control
- Seal density < preform density
  - Bloat density is a “quality” metric
  - Self-consistent results (Archimedes and QS)
- Strength increases with glass seal density

## ■ Conclusions

- Pre-heating to  $T_g$  is recommended for high-fidelity CTE measurements
  - Quick measurements are subject to errors.
- QS cannot discern the differences (heterogeneity) seen by eye.
  - Meso-structure characterization techniques may be needed (e.g., tessellation)
- Consistent, high density preforms = high quality
  - Preform quality = GtM seal manufacturing yield & quality