“Validating Thermal Processing For More Than 125 Years”

Gradient Furnace: The Most Powerful Tool in the Glass/Ceramic Lab

Artem A. Trofimov

Orton Ceramic Foundation, Westerville, Ohio, USA

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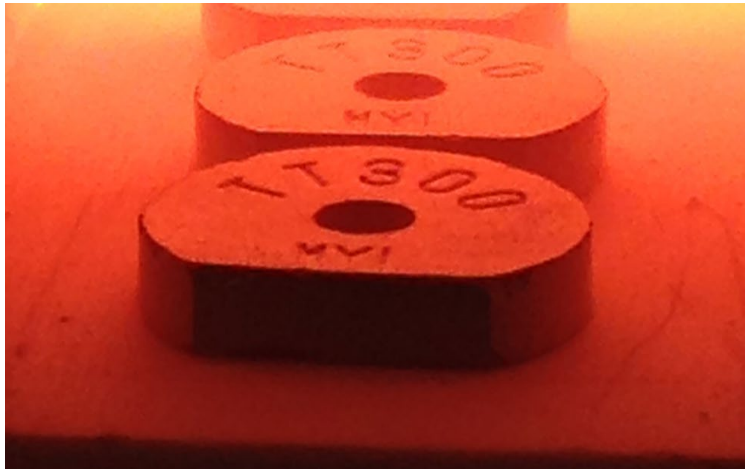
GLASS MANUFACTURING INDUSTRY COUNCIL

"Validating Thermal Processing For More Than 125 Years"

Temperature Monitoring & Kiln Products



Thermal Analysis & Glass Testing Instruments

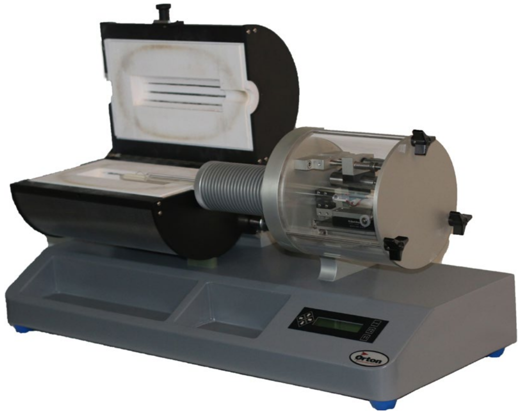
Materials Testing & Research Center

- Thermal Properties
- Physical Properties
- Failure Analysis

<u>100+ ASTM Test Procedures</u>

Instruments

Dilatometers

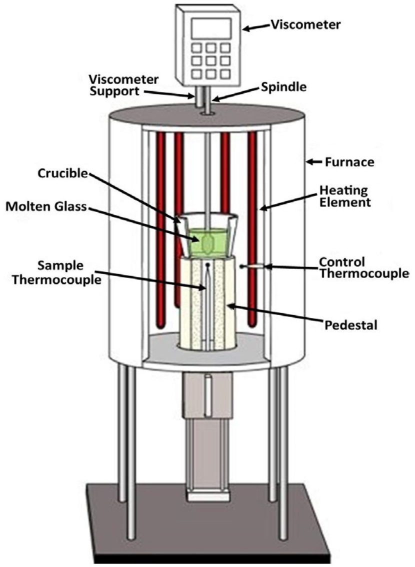


- -190 °C to 1600 °C
- Air or inert atmosphere

Determines:

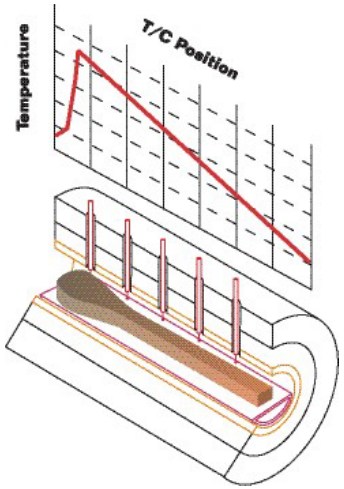
- Thermal Expansion (CTE)
- Shrinkage + Sintering rate
- Glass transition
- Phase transition

High Temperature Viscometers



- Up to 1700 °C
- Log_{10}(Poises): 1.2 to 14.0
- Rotating Spindle Viscometer
- Parallel Plate Viscometer
- Beam Bending Viscometer

Gradient Furnaces



- Up to 1600 °C
- 10-12 °C per inch linear gradient
- 12- or 16-inch monitored zones

Thermal Conductivity

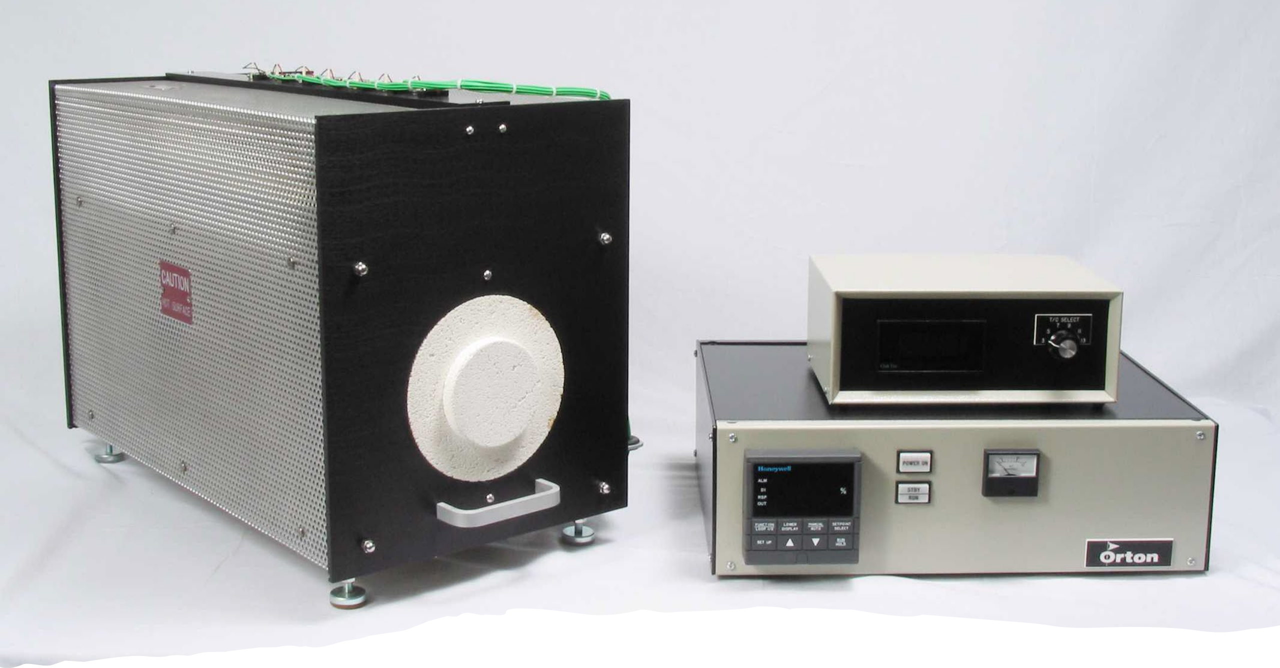
- Up to 1000 °C
- Flexible sample size

Tests:

- Solids
- Powders
- Liquids
- Thin films

Glass Properties

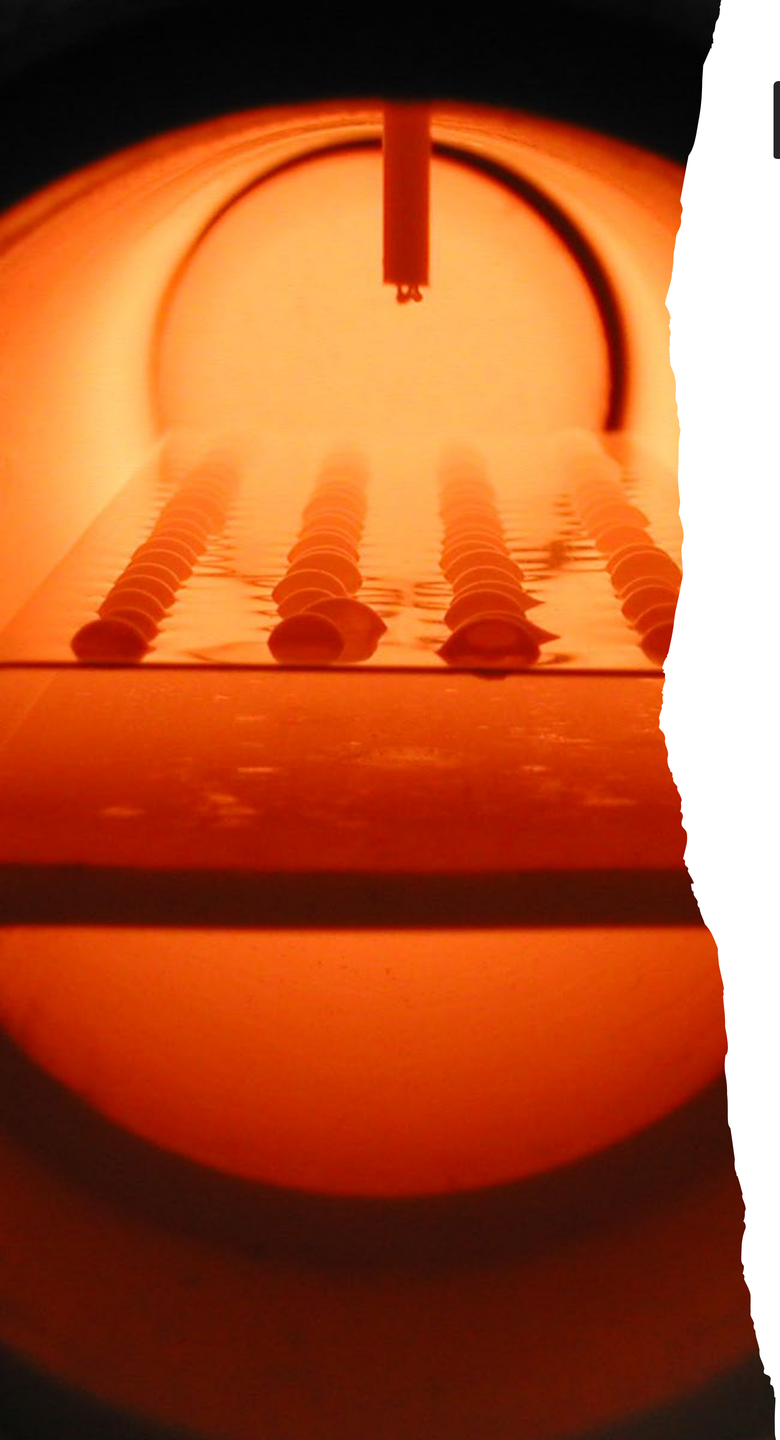
- Annealing point
- Strain point
- Softening point



What is Gradient Tube Furnace (GTF)?

- Horizontal tube furnace used to generate a controlled linear temperature gradient across a sample or a group of samples
- Enables evaluation of **material response** to temperature variations in a single experiment
- Provides a wide temperature range (*ca*. 120-160°C) across a sample or a group of samples
- Offers a cost-effective alternative to multiple-point testing.

Study the effects of a temperature range in ONE firing



Most Powerful Tool in Glass/Ceramic Lab

- **Fire multiple samples simultaneously**

Study how different materials respond to a range of temperatures in a single firing cycle.

- **“Shotgun” approach to thermal analysis**

Rapidly identify critical temperature ranges where material behavior changes — crystallization, softening, warping, melting, etc.

- **Side-by-side comparison of materials**

Fire a known material alongside a test sample to benchmark behavior and performance.

- **Pre-screen new raw materials or batches**

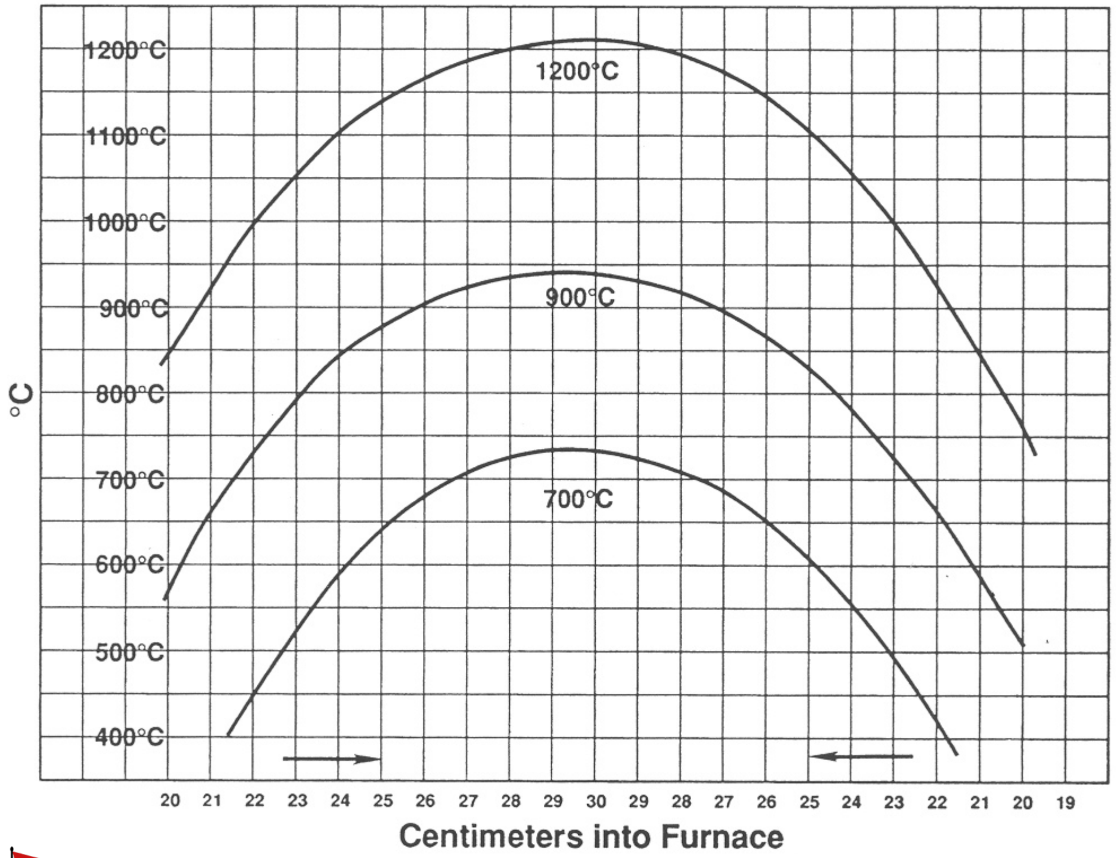
Evaluate new formulations before committing to expensive and time-consuming production trials.

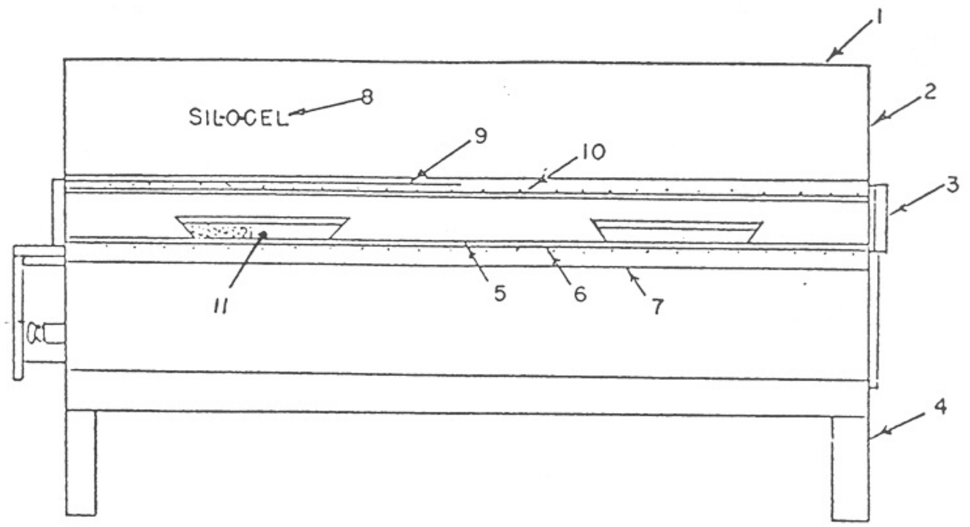
- **Ideal for both QC and R&D**

A versatile tool used across the development cycle—from material selection to process optimization.

Traditional Gradient Furnace

- **Hottest zone fixed in the center**
- **Short monitored zone** – <u>about 3.9”/10 cm long</u>
  - ➢ Restricts the number of usable data points per firing
- **Large thermal gradient** – <u>about 60-80°C per inch</u>
  - ➢ Reduced resolution – temperature changes too rapidly to precisely identify critical transitions





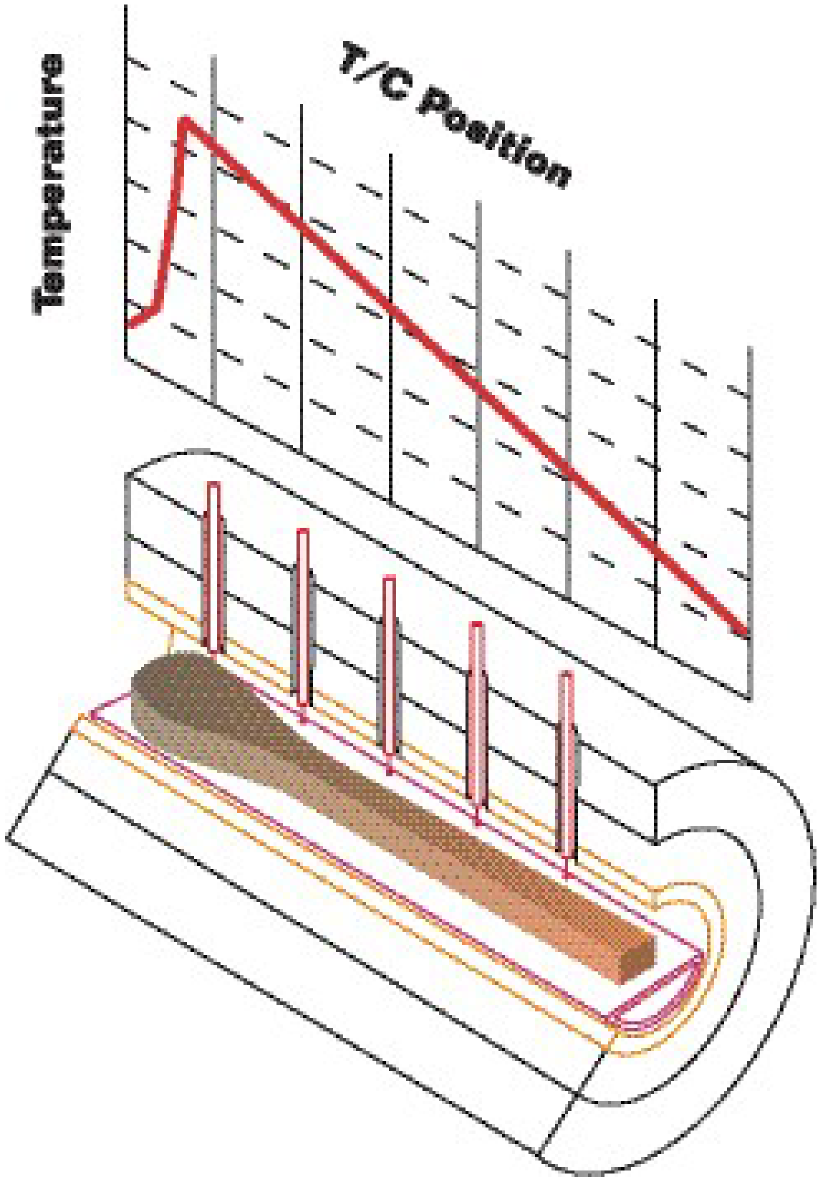
NOTE 1—See A1.1 for further description.

1. Outer shell (stainless steel)
2. End plate (Transite)[4]
3. End plate (quartz)
4. Stand
7. Outer protection tube
8. Sil-O-Cel[5] insulation
9. Control thermocouple (platinum/rhodium)
10. Heating element wire

*ASTM C829 “Standard Practices for Measurement of Liquidus Temperature of Glass by the Gradient Furnace Method”

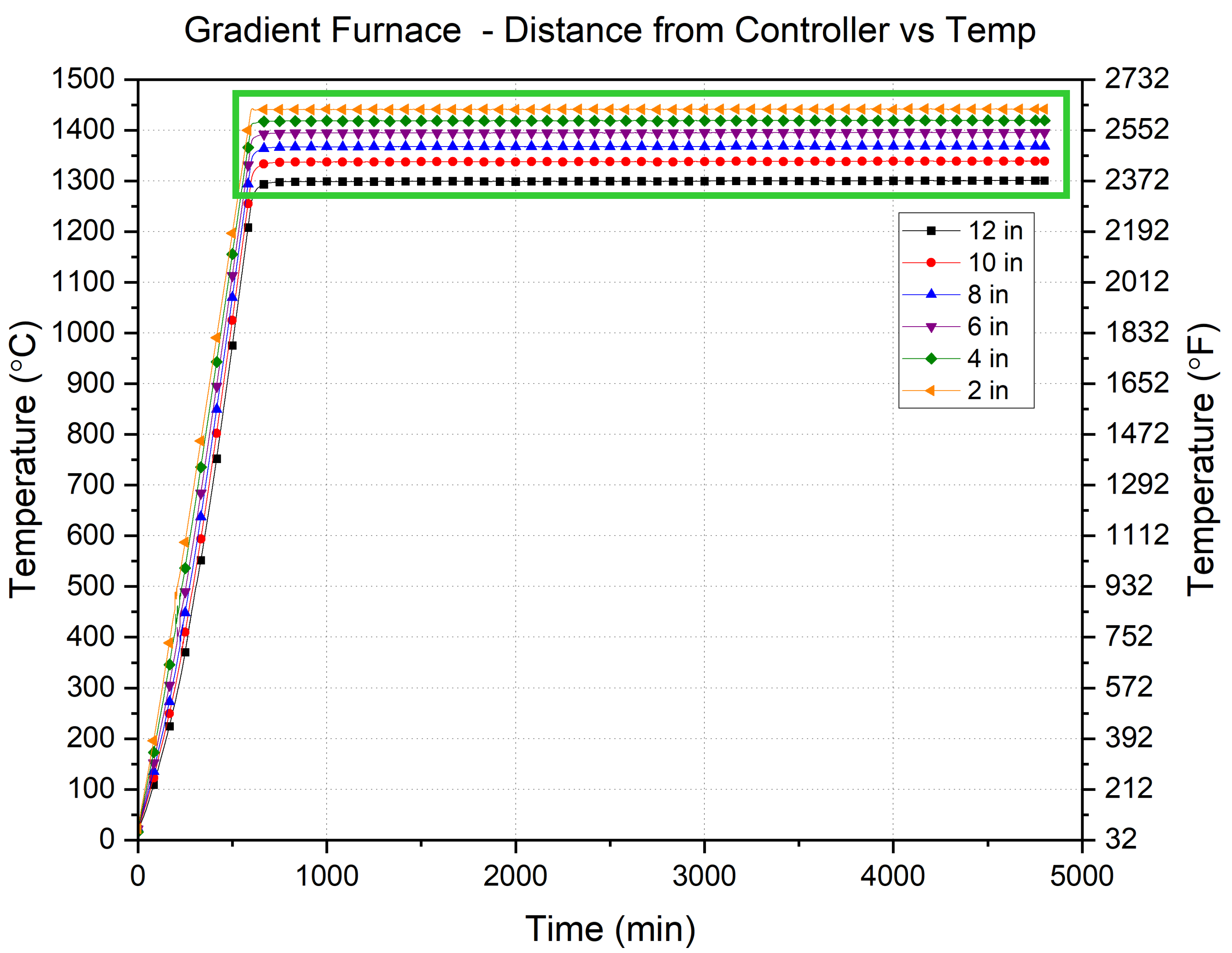
Orton Gradient Furnace

- **Hottest zone placed at the end**
- **Longer monitored zone** – 12 inches (30.5 cm) or longer
  - More samples or larger sample lengths in a single firing
- **Shallower thermal gradient** – About 10-12°C per inch
  - Finer temperature gradient resolution, ideal for detecting subtle changes in phase, structure, or behavior
- **Software** – for remote programming/datalogging



- Monitoring thermocouples hang from ceiling
- D-Tube hearth holds samples

Simulate Production Firing Schedule



Simple heat-and-soak or multi-segment thermal cycles

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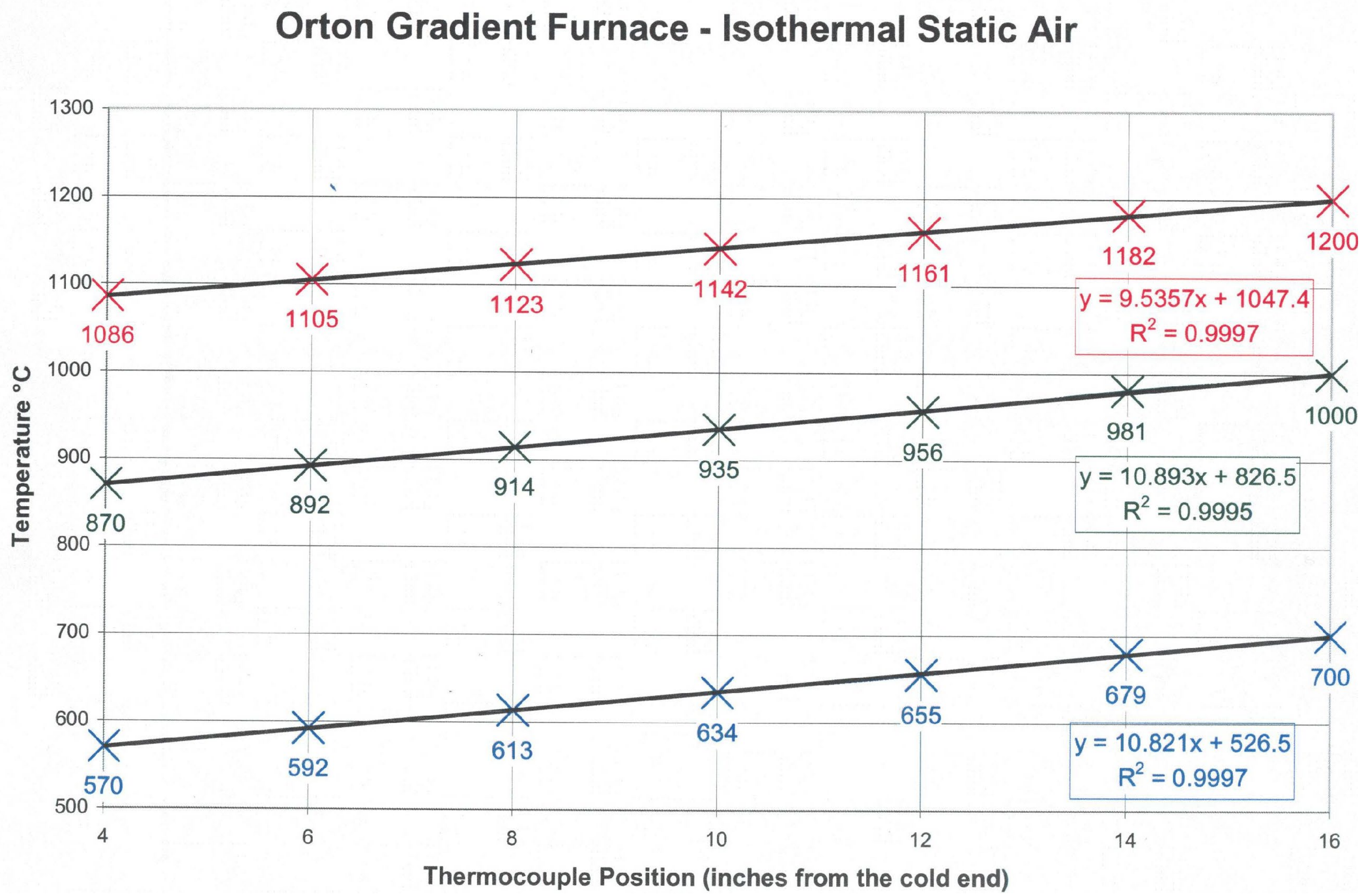
Isothermal Gradient vs Set Point



Excellent linear gradient

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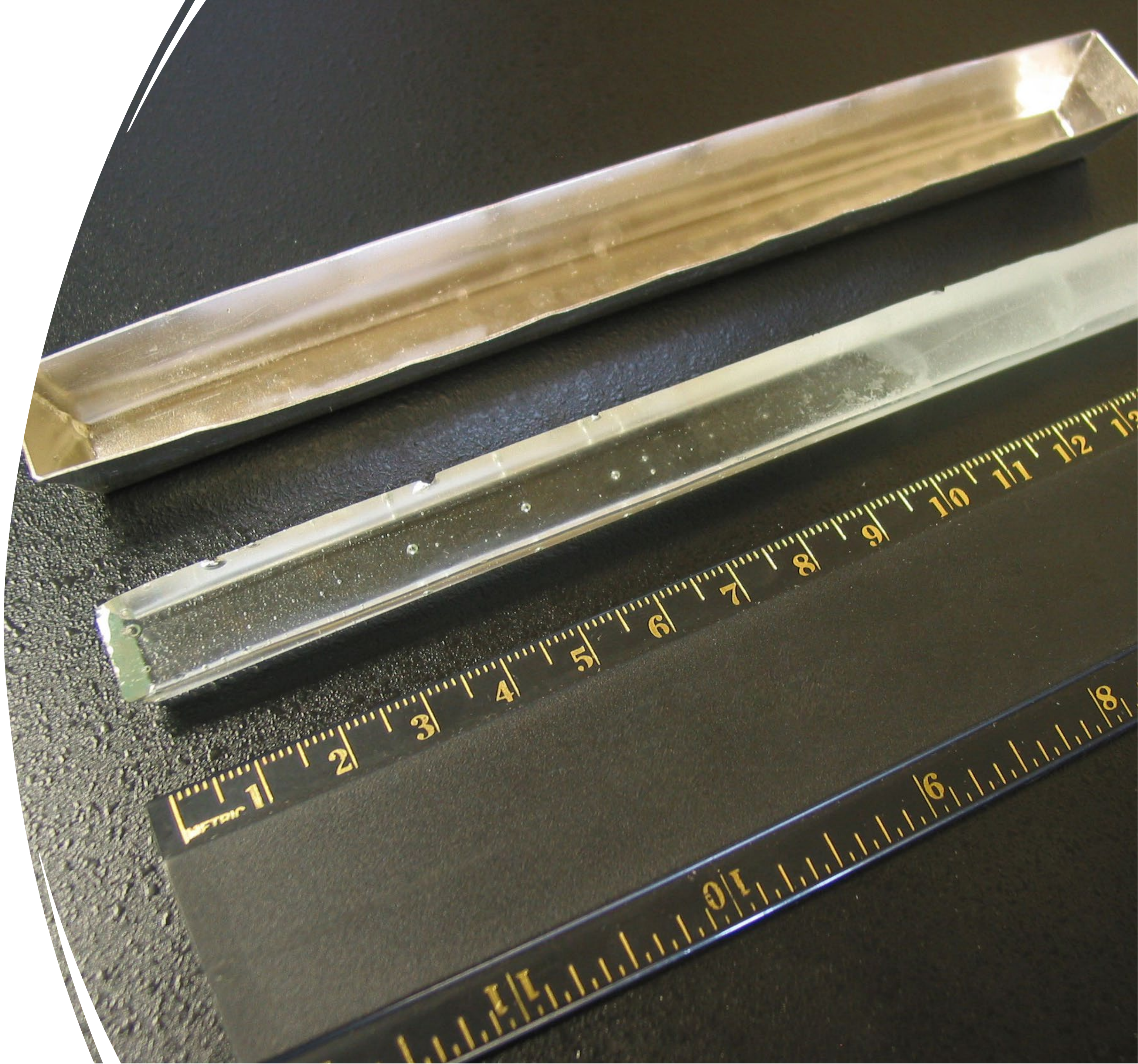
Liquidus Point Determination - Glass

1. Fill platinum boat with glass
2. Hold boat at an isothermal soak for several hours until the equilibrium crystal plane develops
3. Analyze bar under a microscope for plane of crystal development
4. Correlate position of plane in bar with position in furnace to determine temperature at that plane



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Liquidus Point Determination - Glass

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Materials Testing and Research Center
ASTM C829 - Liquidus Temperature of Glass by the Gradient Furnace Method

Engineer:	BR	**Customer:**	Orton
Technician:	BR	**Customer PO:**	NA
Method:	A, Platinum Boat	**Report Date:**	

Ramp Rate 1 (C/min):	5
Temperature 1 (C):	905
Ramp Rate 2 (C/min):	2
Temperature 2 (C):	1025
Hold Time (hr):	25

Position (in)	**Temp. (C)**
16	1027
14	1006
12	974
10	946
8	913

Sample ID:	NIST 773
Prep. Method:	
Notes:	

Position of Last Crystal (in): 13.06

Liquidus Temperature (C): 988

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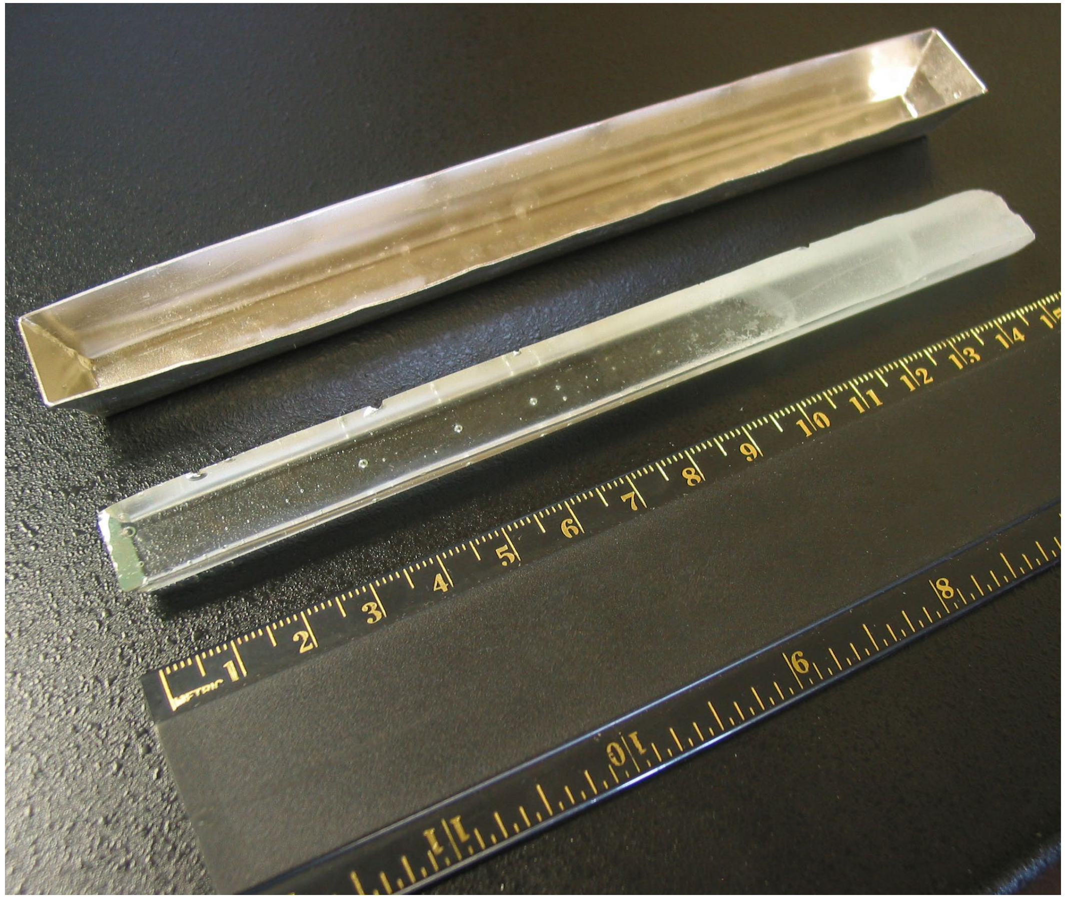


Standard Reference Material 773

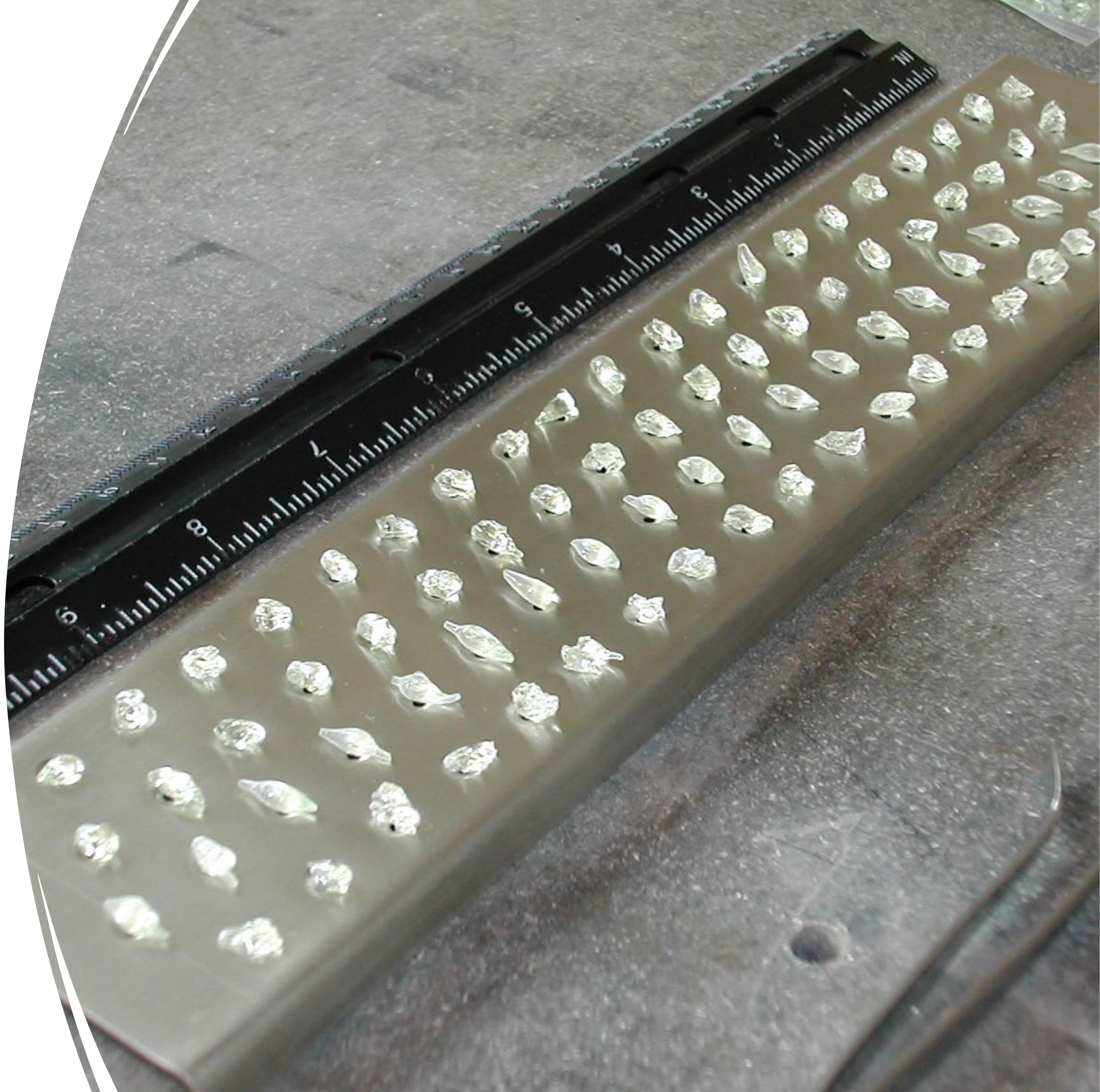
Soda-Lime-Silica Glass for

Gradient-Furnace Liquidus Temperature

Method	Liquidus Temperature
A (boat)	988 ± 3 °C
B (perforated plate)	991 ± 5 °C

Liquidus Point Determination - Glass

1. Platinum tray with holes – place chunks of glass on holes
2. Hold tray at an isothermal soak for several hours until the equilibrium crystals develop
3. Analyze each chunk/hole for crystal development
4. Correlate position of tray with position in furnace to determine temperature at that hole



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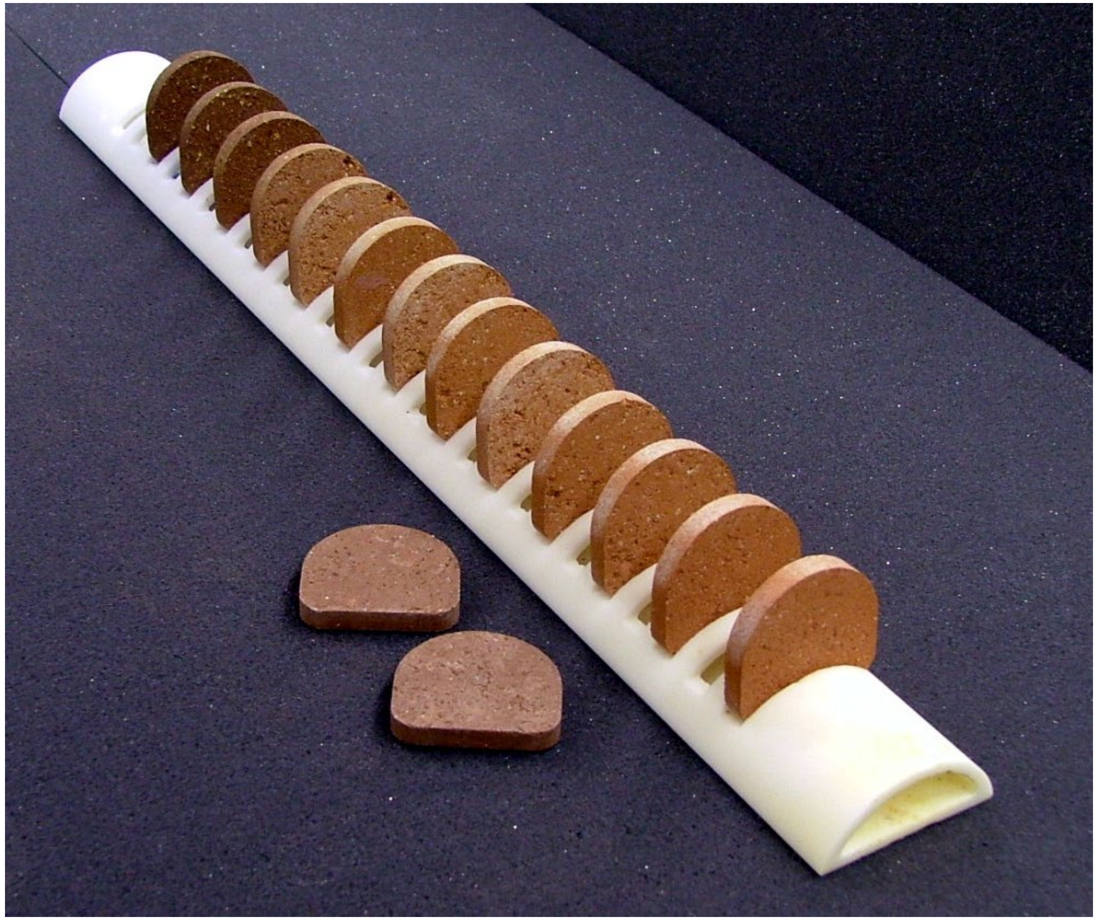
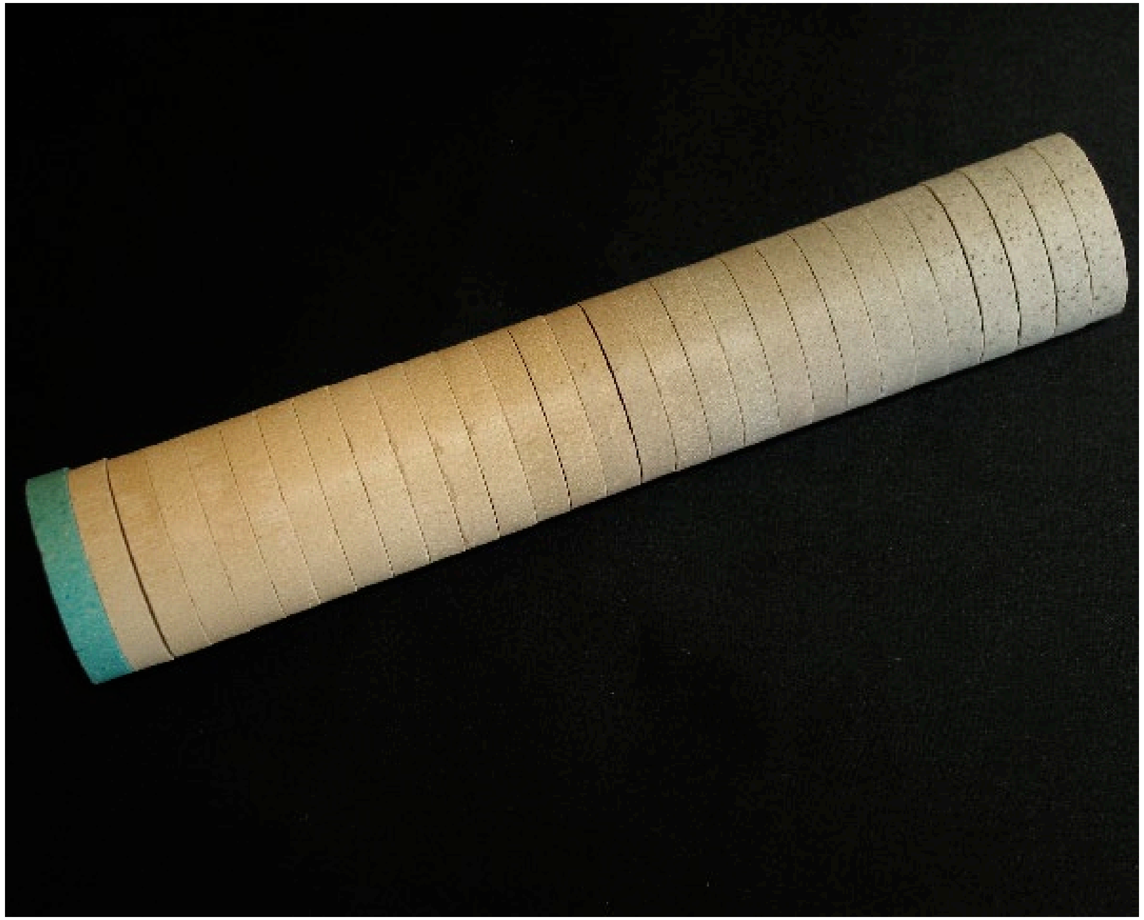
Temperature Effect on Ceramic Materials – Dry Pressed Body





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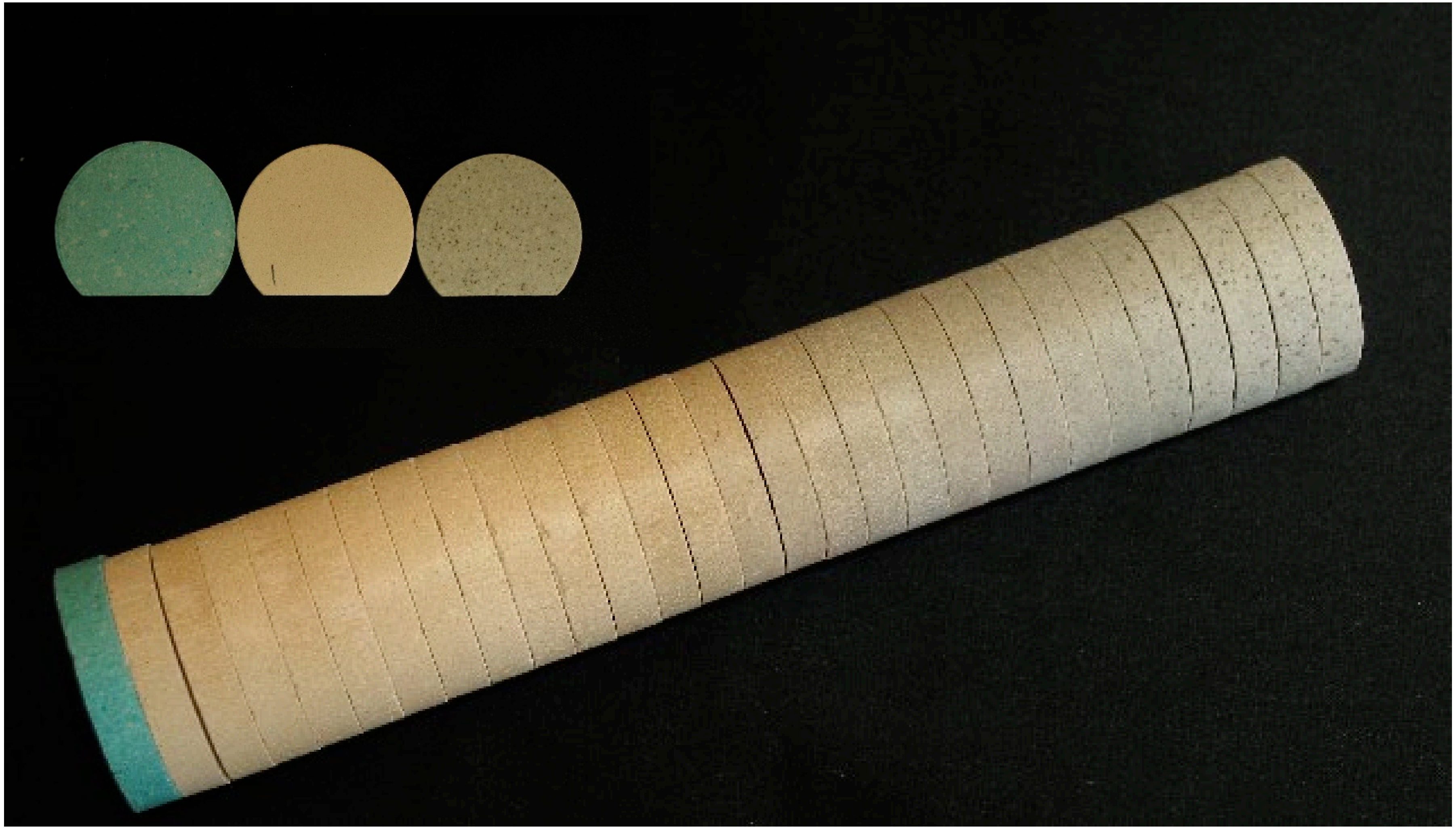
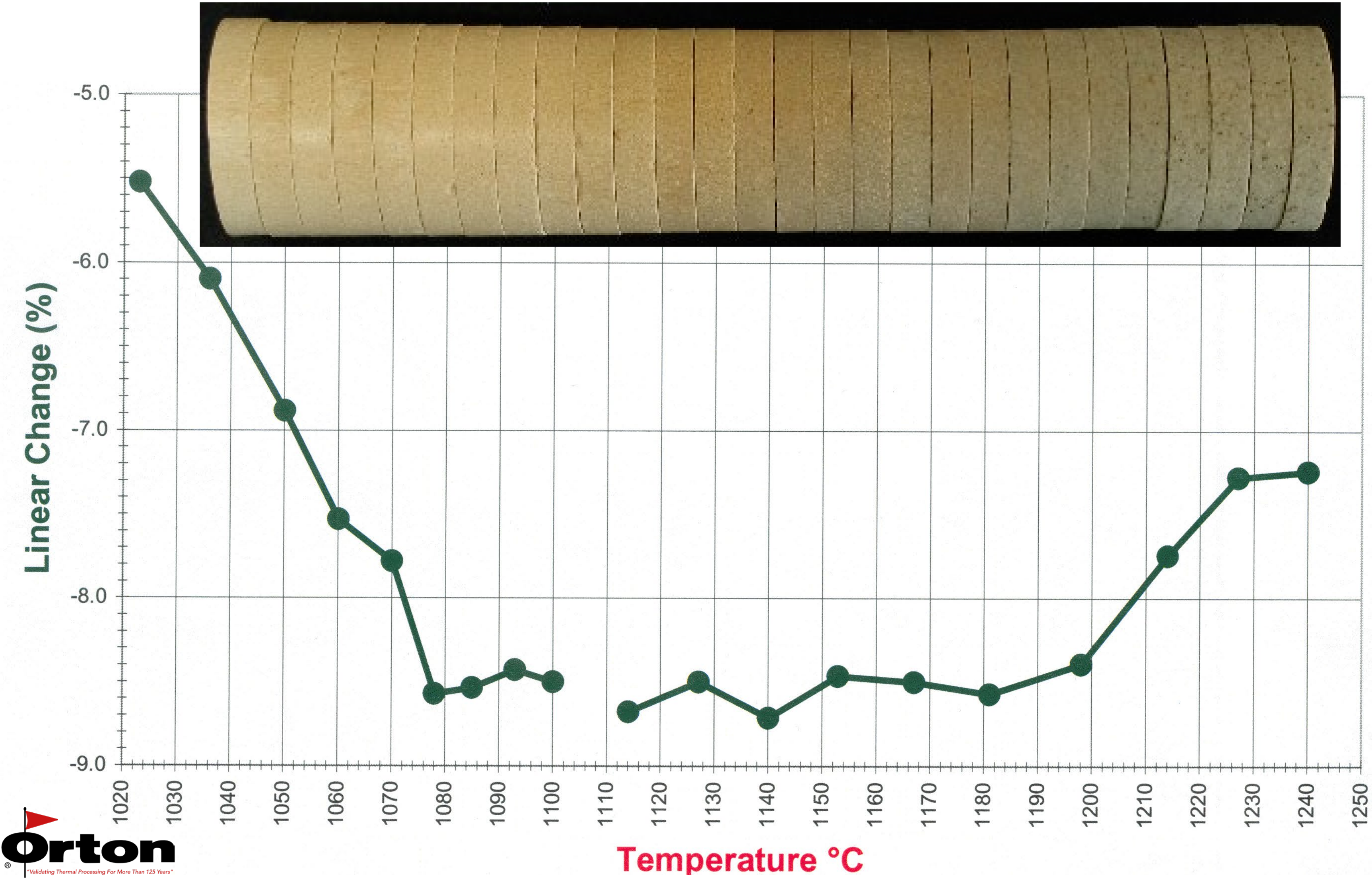
Color Change vs Temperature



Shrinkage/Expansion vs Temperature



Identify the ideal firing window

1. **Initial Decrease in Linear Dimension**

   Drying and initial sintering shrinkage

2. **Plateau**

   Minimal Dimensional Change – "stable window"

3. **Increase in Linear Dimension**

   Expansion due to bloating and overfiring effects

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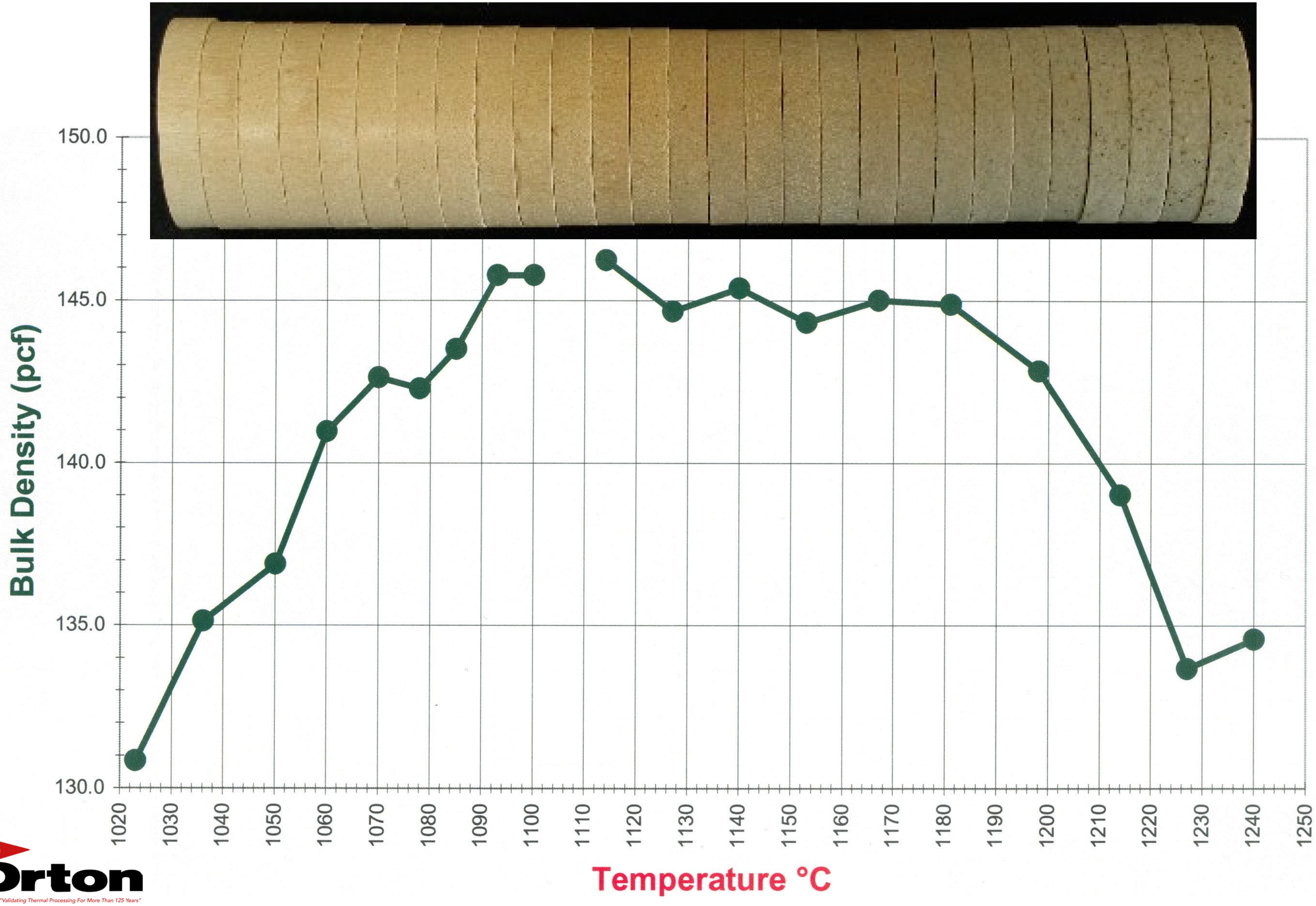
Bulk Density vs Temperature



<u>Identify the ideal firing window</u>

1. **Sintering-densification**

   Reduction of open porosity

2. **Plateau**

   Pore closure is mostly complete, no major changes to mass or volume

3. **Overfiring**

   Structure swells, volume increases while mass stays constant

Bulk density follows both porosity and total volume change

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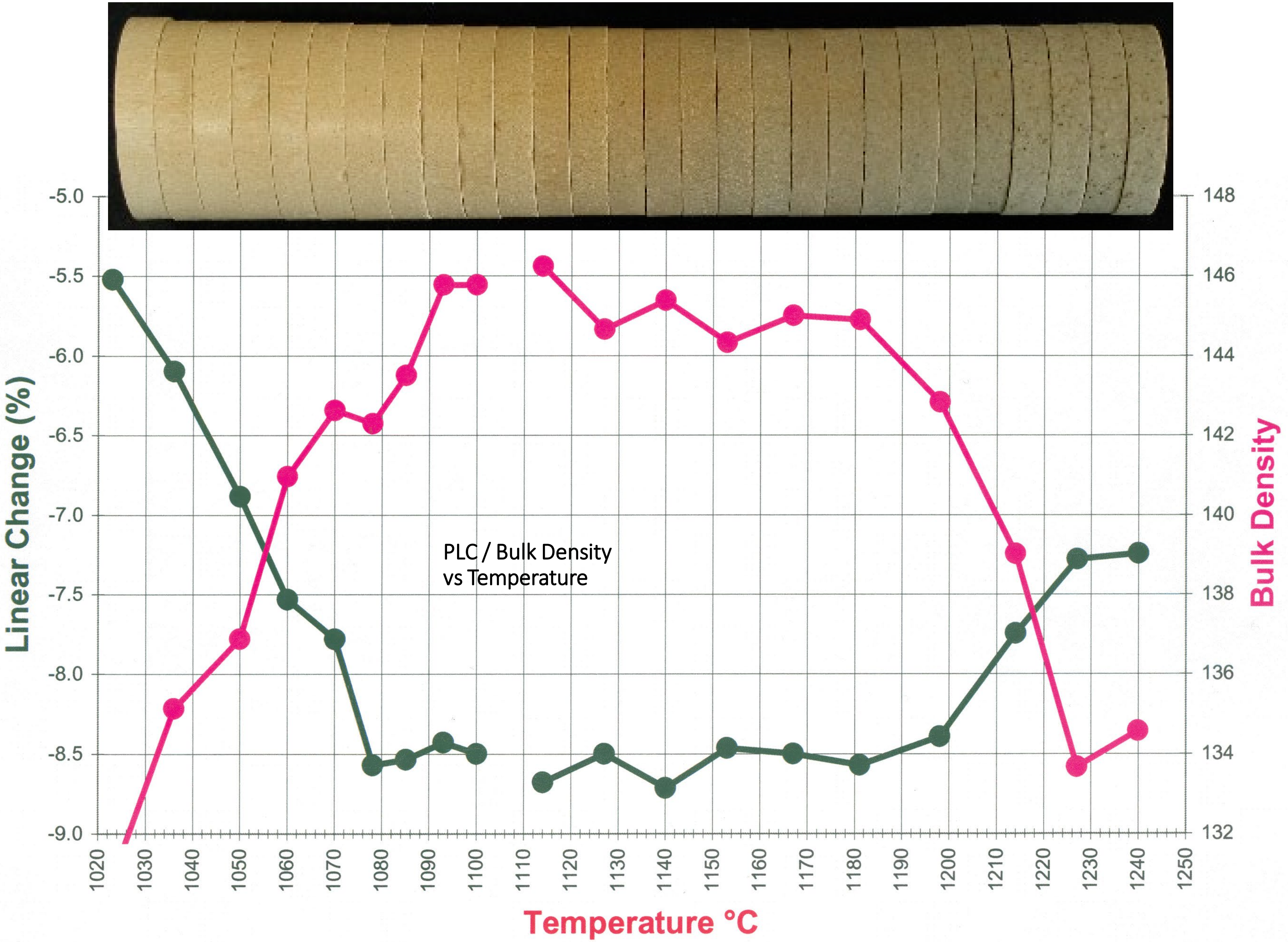
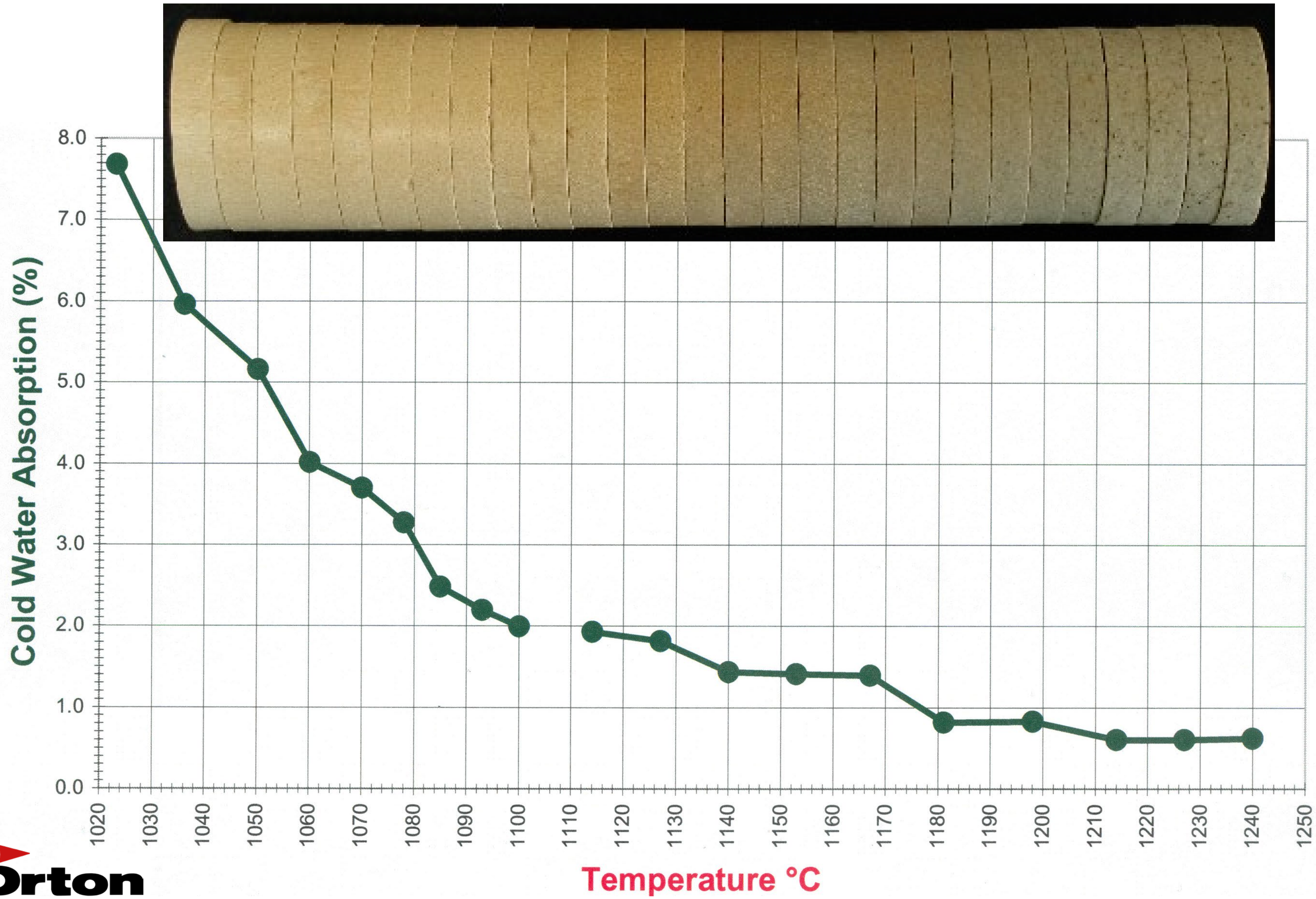

PLC / Bulk Density
vs Temperature
Linear Change (%)
-5.0
-5.5
-6.0
-6.5
-7.0
-7.5
-8.0
-8.5
-9.0
Bulk Density
148
146
144
142
140
138
136
134
132
1020
1030
1040
1050
1060
1070
1080
1090
1100
1110
1120
1130
1140
1150
1160
1170
1180
1190
1200
1210
1220
1230
1240
1250
Temperature °C

Percent Absorption vs Temperature



Identify the ideal firing window

1. **Pores close due to sintering**

   Less water can penetrate

2. **Pore closure is mostly complete**

   Further temperature increase doesn't significantly affect CWA

CWA mainly follows the loss of open porosity

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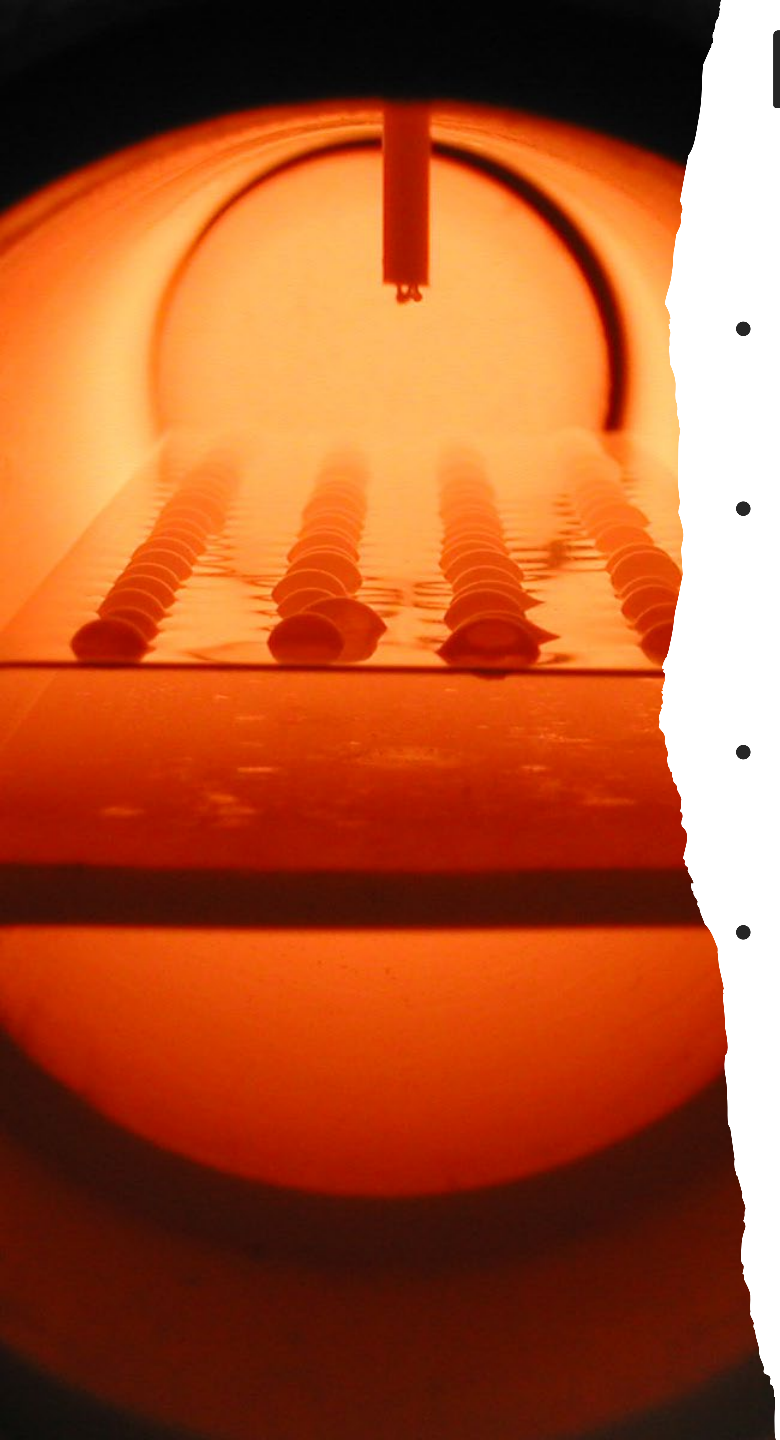

Most Powerful Tool in Glass/Ceramic Lab

- Powerful tool for both R&D and QC
- Critical for liquidus temperature determination and devitrification studies
- Efficient temperature profiling
- Orton GTF: Enhanced design for modern testing needs

Make the mistakes in the Gradient Furnace,
NOT in the Production Furnace!

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Thank You for Your Attention

Visit Orton's Booth #57

Contact: Dr. Artem Trofimov, Senior Application Scientist, trofimov@ortonceramic.com

6991 Old 3C Highway, Westerville, Ohio 43082 USA www.ortonceramic.com

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