

Correction to the Beam Bending Method for Measuring Glass Viscosity

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Background and *Problem*

The ASTM Standard, C1350M, is used to calculate the viscosity using a bending beam, but there are problems.

The calibration is used to “...correct experimental viscosity data...”

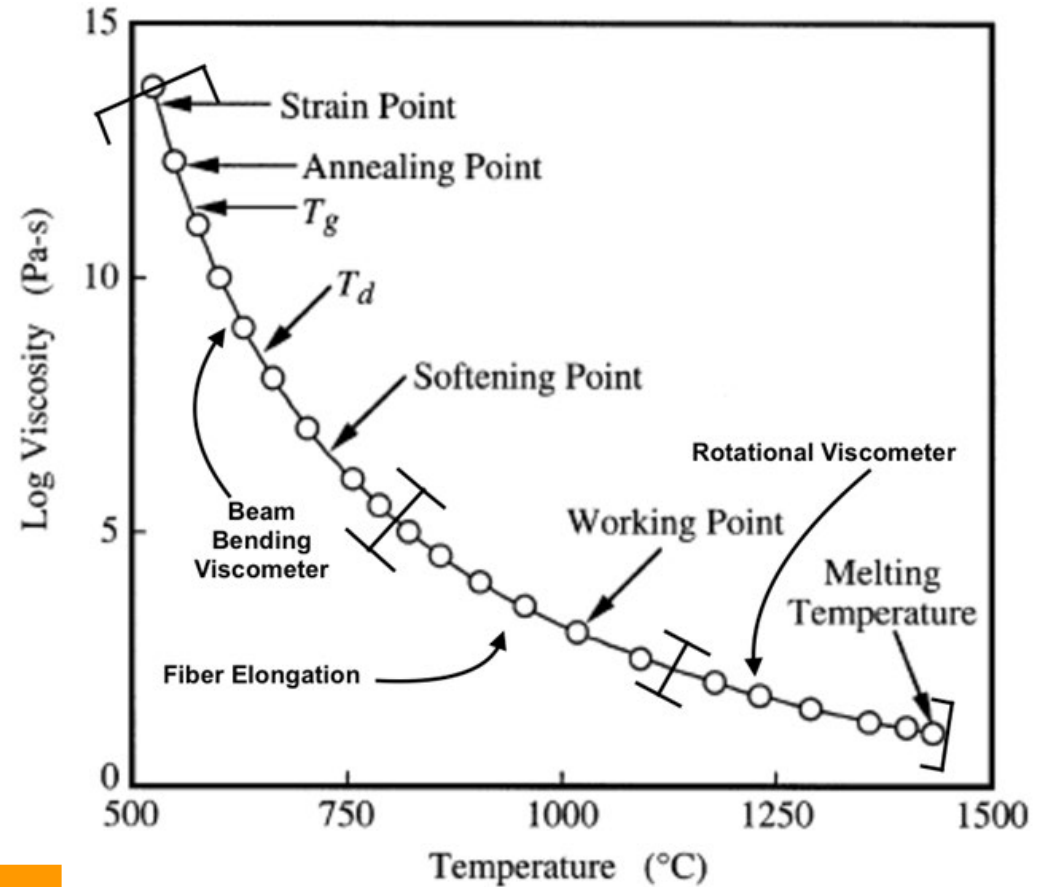
C1350 uses the mid-point deflection of a 3-point loaded beam to determine the deflection rate – *this is incorrect.*

It appears that bending beam, fiber elongation, and shear viscosity measurements do not line up. (Has this been published?)

Beam bending is a “shear-free” flow, as is fiber elongation.
Rotational viscosity is “shear flow.”

Glass Viscosity Measurement

The extremely broad range of viscosity exhibited by glass requires several measurement methods.

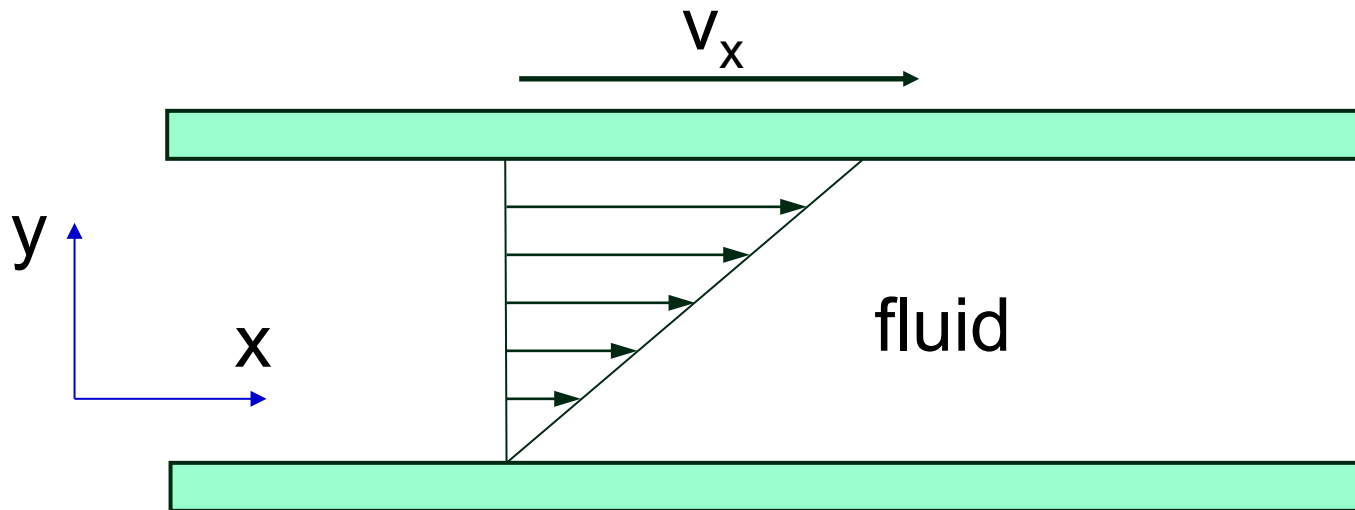


Viscosity range	Temperature	Method
$> 10^5$ Pa·s	Low	Beam Bending
$10^2 - 10^5$ Pa·s	Intermediate	Fiber Elongation
$< 10^2$ Pa·s	High	Rotational (Shear)

Definition of viscosity: *ratio of stress to strain rate*

$$\tau = \mu \left(\frac{dv_x}{dy} \right) = \eta \dot{\gamma}$$

Proportionality constant
between shear rate and
shear stress (η)



For Extensional Viscosity

$$\eta_e = \frac{\sigma}{\dot{\epsilon}}$$

$$\eta_e = \frac{F/A}{dv/dx}$$

Fiber
elongation

Shear versus Shear-free viscosity

Viscosity range	Method	ASTM	Flow Condition
$< 10^{6.5} \text{ Pa}\cdot\text{s}$	Rotational (Shear)	C965	Shear flow
$10^4 - 10^8 \text{ Pa}\cdot\text{s}$	Parallel Plate (Squeezing Flow)	C1351	Mixed (shear/shear-free)
$10^6 - 10^{11} \text{ Pa}\cdot\text{s}$	Penetration Viscometer	[not for glass]	Shear/shear-free (?)
$10^5 - 10^{15.5} \text{ Pa}\cdot\text{s}$	Fiber Elongation	C336	Shear-free
$10^7 - 10^{14} \text{ Pa}\cdot\text{s}$	Bending Beam	C1350	Shear-free

Trouton (1906)

$$3\mu = \eta_e$$

“Elongational Viscosity”
(shear-free viscosity)

“Shear Viscosity”

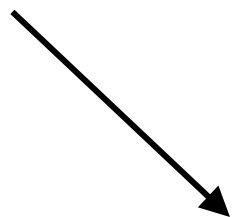
F. Trouton, “On the Coefficient of Viscous Traction and its Relation to that of Viscosity”

Proceedings of the Royal Society of London. Series A, Containing Papers of a Mathematical and Physical Character
77 [519], pp 426-440 (1906)

ASTM C1350M – 25

Standard Test Method for Measurement of Viscosity of Glass
Between Softening Point and Annealing Range (Approximately 10^8
 $\text{Pa}\cdot\text{s}$ to Approximately $10^{13} \text{ Pa}\cdot\text{s}$) by Beam Bending

Viscosity
($\text{Pa}\cdot\text{s}$)


$$\eta = \frac{g L^3}{1440 I_c (dh/dt)} \left[M + \frac{\rho AL}{1.6} \right] \left[\frac{(1 + \alpha_s T)^3}{(1 + \alpha_g T)^4} \right]$$

Foundation: H. Hagy, “Experimental Evaluation of Beam-Bending Method of Determining Glass Viscosities in the Range 10^8 to 10^{15} Poises,” *J. American Ceramic Soc.*, **46** [2], 93-97 (1963)

ASTM C1350M – 25

(Uses mid-point deflection as strain)

Standard Test Method for Measurement of Viscosity of Glass
Between Softening Point and Annealing Range (Approximately 10^8
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Viscosity
($\text{Pa}\cdot\text{s}$)

Gravity
(980 cm/s^2)

Load
(applied + load train)
(g)

Sample mass
(distributed load)
(g)

L = span (cm)

Moment of inertia
(cross section)
C598 (cm^4)

Mid-point deflection rate
(cm/s)

Thermal Expansion Correction
(dimensionless)

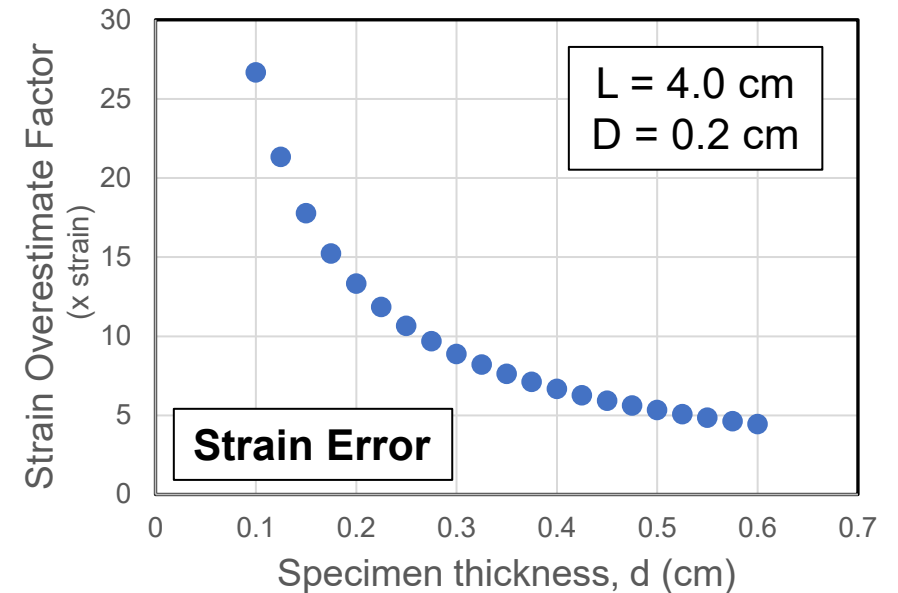
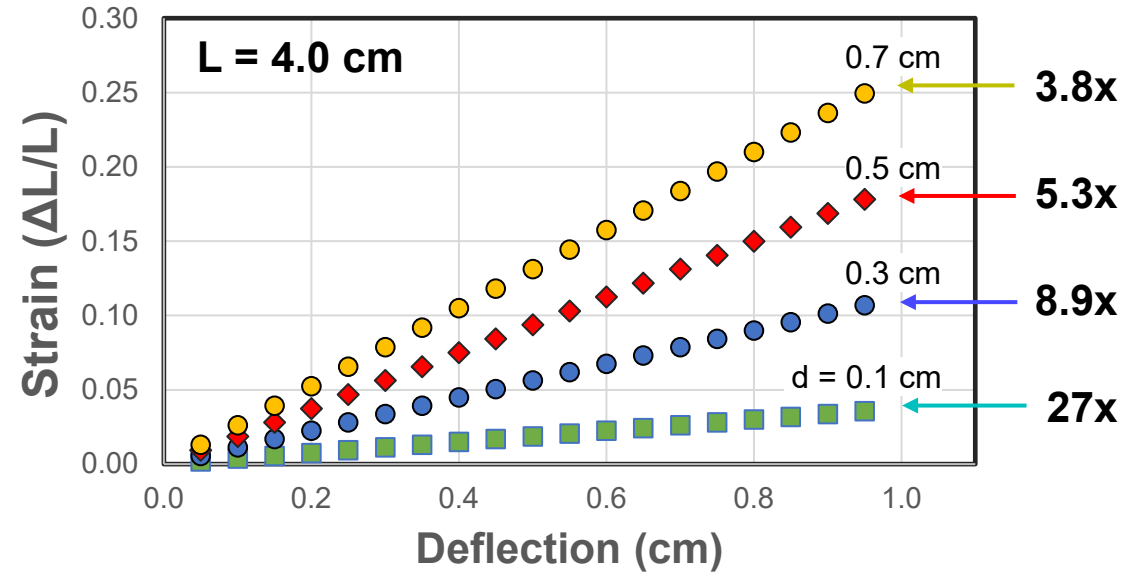
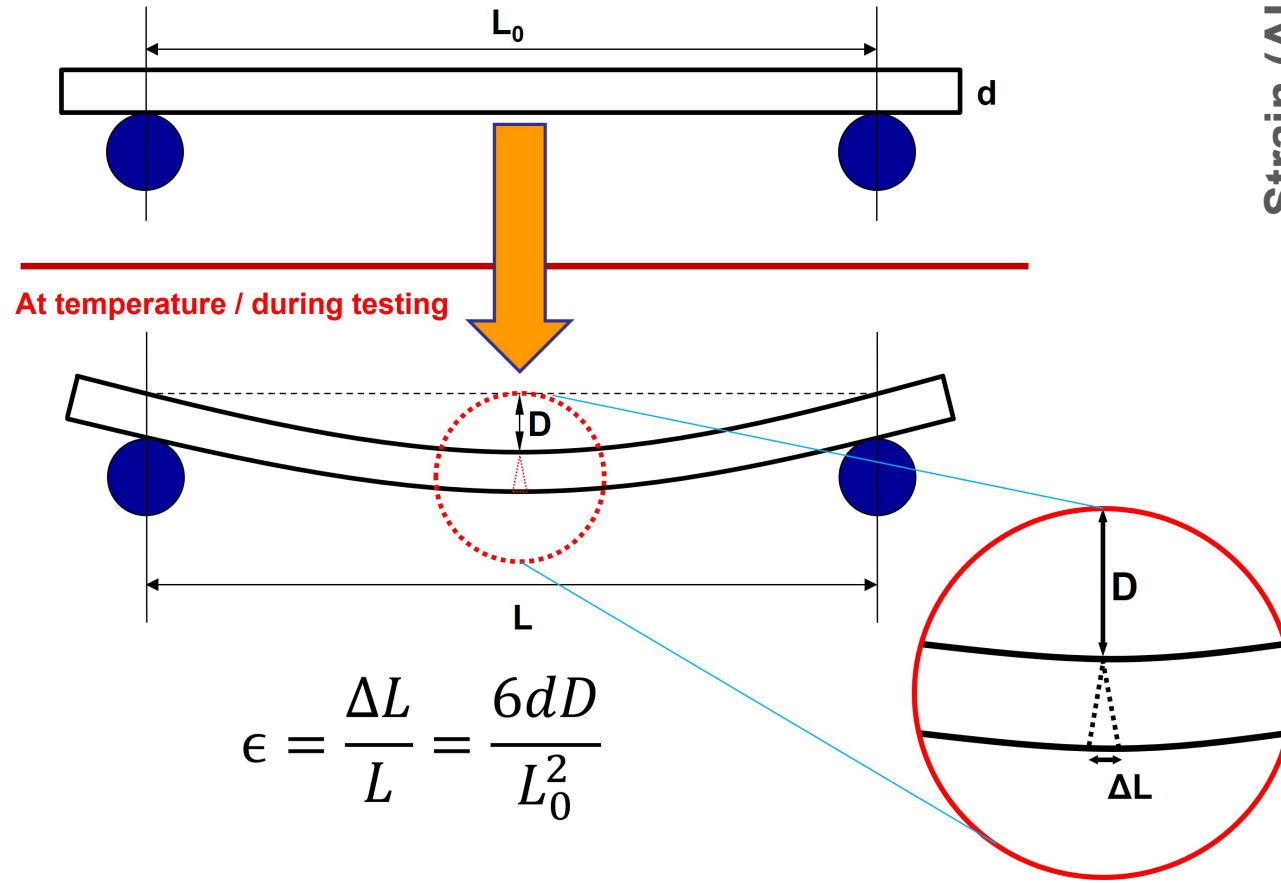
$$\eta = \frac{g L^3}{1440 I_c (dh/dt)} \left[M + \frac{\rho A L}{1.6} \right] \left[\frac{(1 + \alpha_s T)^3}{(1 + \alpha_g T)^4} \right]$$

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ASTM C1350

(Uses mid-point deflection as strain)

Deflection is not Strain. Deflection greatly overestimates the strain.
Therefore: Lower 'measured' viscosity.



Elongational viscosity via bending

Stress / strain rate

Stress from 3-point flexure relationship

$$\sigma_{rod} = \frac{8PL}{\pi d^3}$$

Circular Cross-section Rod
ASTM C1684

Rectangular Cross-section Rod
ASTM C1161

$$\sigma_{bar} = \frac{3PL}{2bd^2}$$

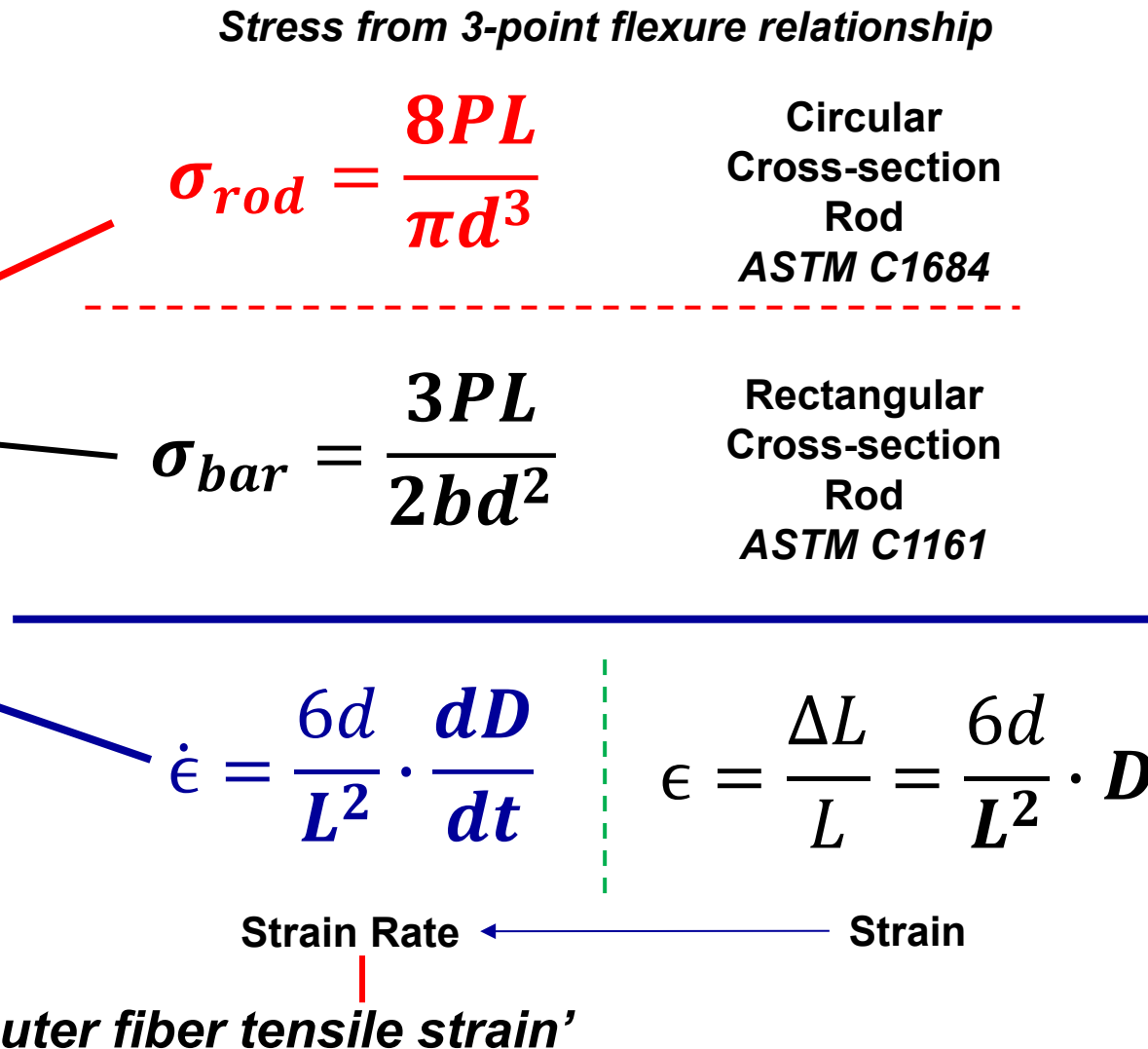
$L \geq 8d$
ASTM

$\eta_e = \frac{\sigma}{\dot{\epsilon}}$

$$\dot{\epsilon} = \frac{6d}{L^2} \cdot \frac{dD}{dt}$$
$$\epsilon = \frac{\Delta L}{L} = \frac{6d}{L^2} \cdot D$$

Strain Rate ← Strain

Strain rate from 'outer fiber tensile strain'



Elongational viscosity via bending

Stress / strain rate

(mm)	b	d	L
A	2.0	1.5	25
B	4.0	3.0	45
C	8.0	6.0	90

Stress from 3-point flexure relationship

$$\sigma_{rod} = \frac{8PL}{\pi d^3}$$

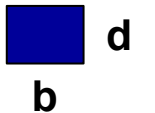
Circular
Cross-section
Rod
ASTM C1684



$L \geq 8d$
ASTM

$$\sigma_{bar} = \frac{3PL}{2bd^2}$$

Rectangular
Cross-section
Rod
ASTM C1161



$$\eta_e = \frac{\sigma}{\dot{\epsilon}}$$

$$\dot{\epsilon} = \frac{6d}{L^2} \cdot \frac{dD}{dt}$$

$$\epsilon = \frac{\Delta L}{L} = \frac{6d}{L^2} \cdot D$$

Strain Rate

Strain

Strain rate from 'outer fiber tensile strain'

Elongational viscosity via bending

Rod

$$\eta_e = \frac{\frac{8PL}{\pi d^3}}{\frac{6d}{L^2} \cdot \frac{dD}{dt}} = \frac{8PL^3}{6\pi d^4 \left(\frac{dD}{dt} \right)}$$

Cylindrical Cross Section (Rod)

Bar

$$\eta_e = \frac{\frac{3PL}{2bd^2}}{\frac{6d}{L^2} \cdot \frac{dD}{dt}} = \frac{PL^3}{4bd^3 \left(\frac{dD}{dt} \right)}$$

Rectangular Cross Section (Bar)

$$P = (\text{Sample Mass} + \text{Added Load}) \times (\text{gravity})$$

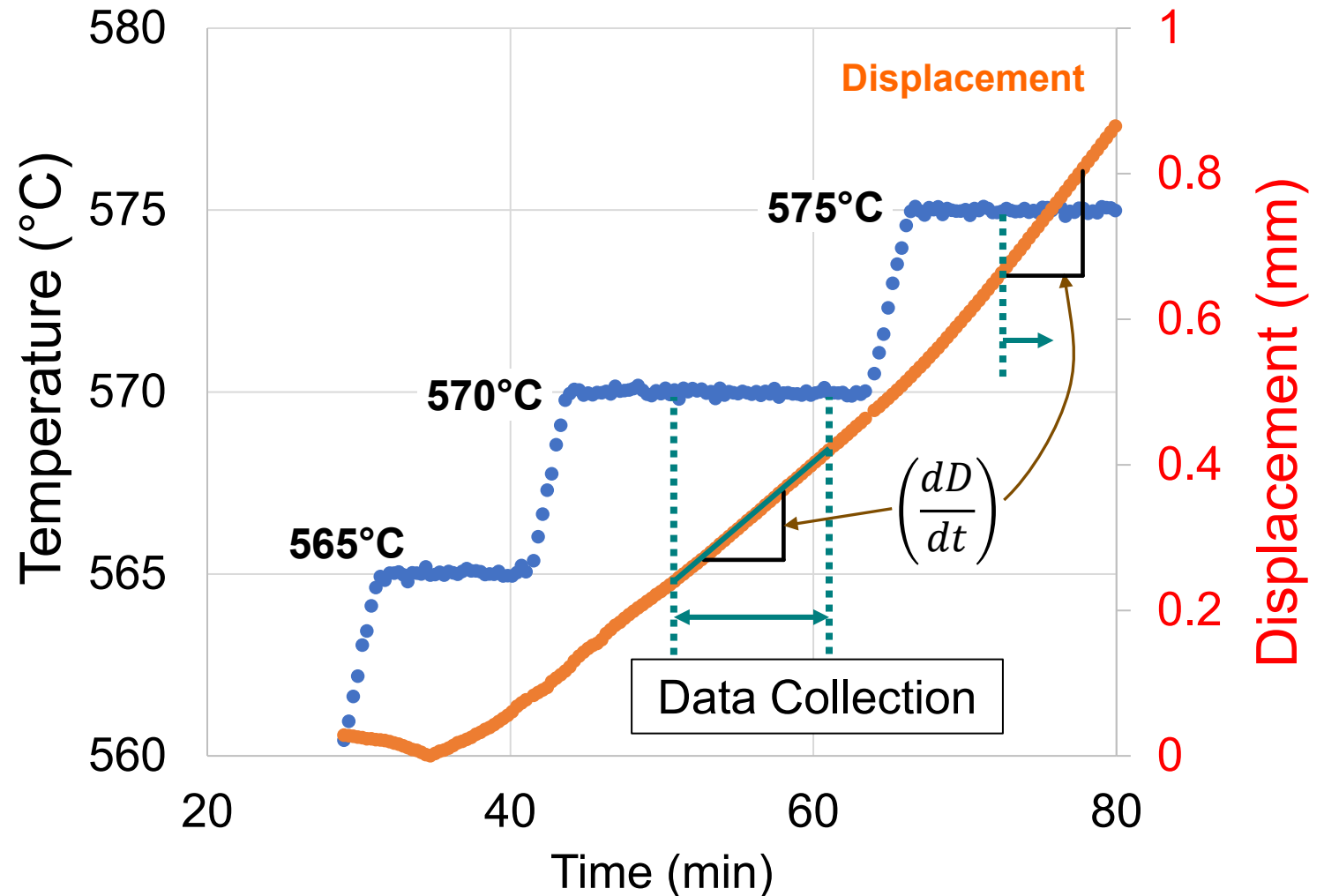
Heat, hold, collect rate data.

Heat to set-point

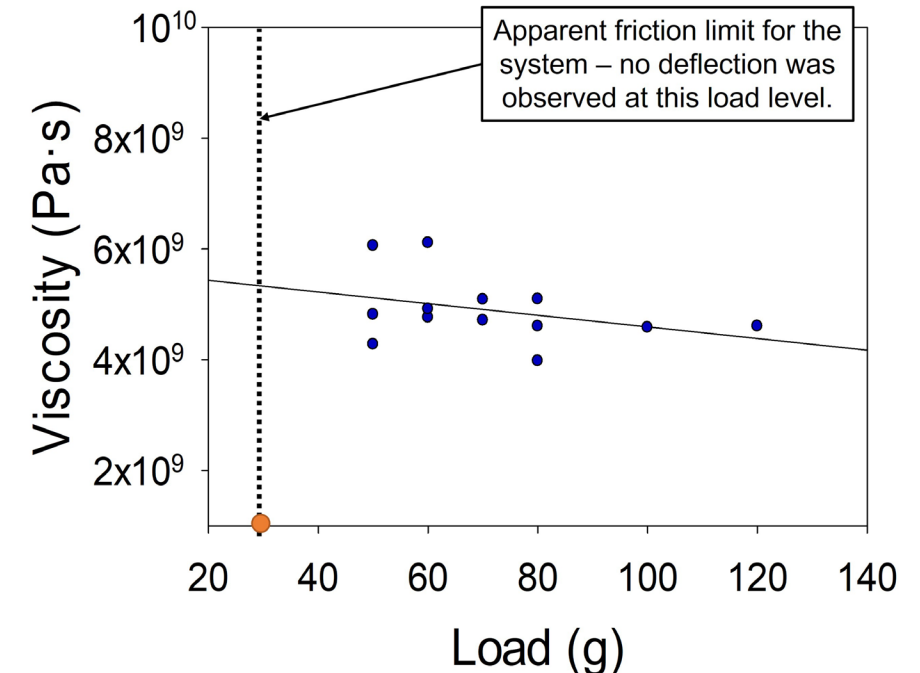
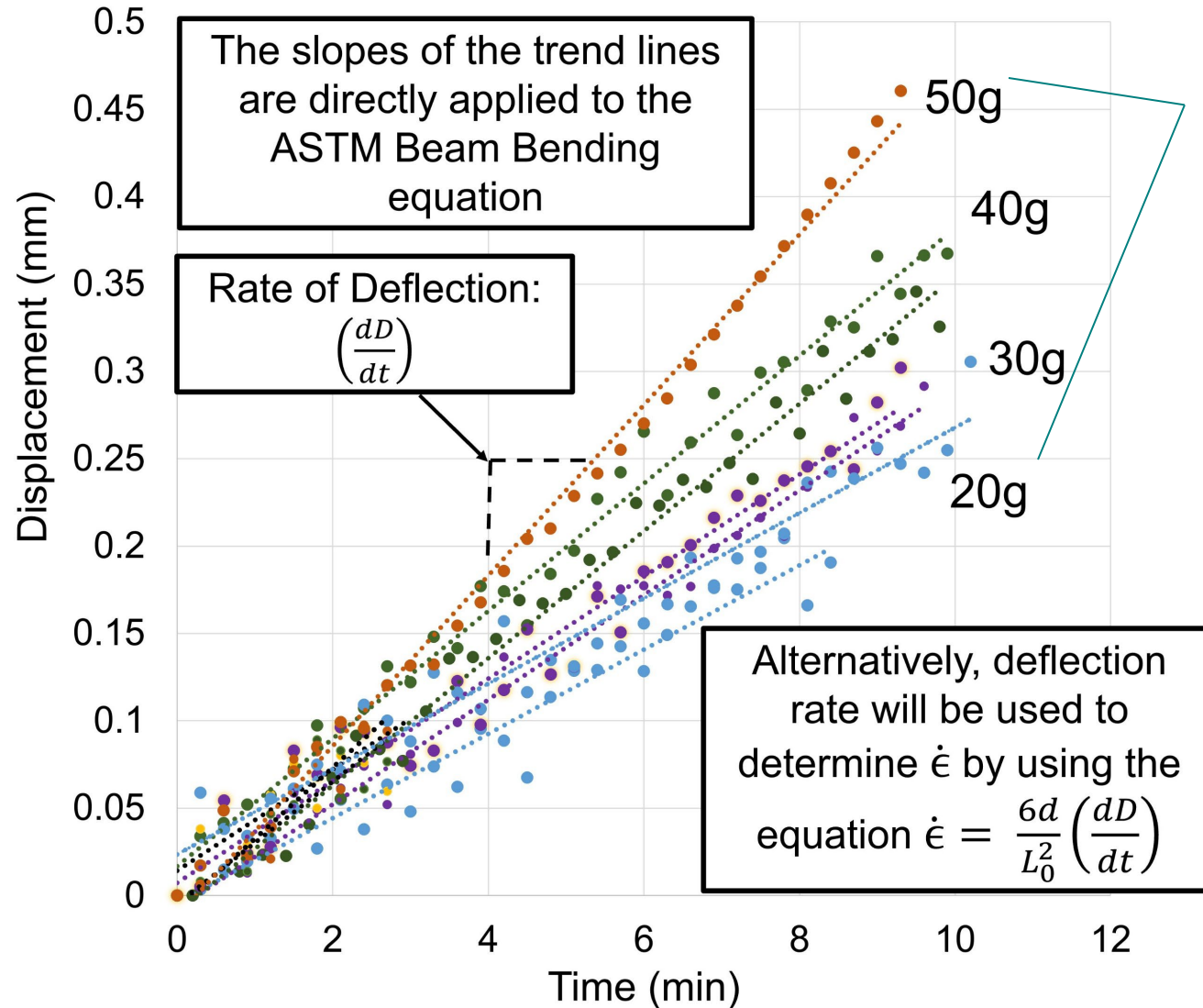
Allow to reach
steady-state
(constant load)

Collect displacement
data over time.

Calculate deflection
rate



Determining the Tensile Strain Rate at 575°C

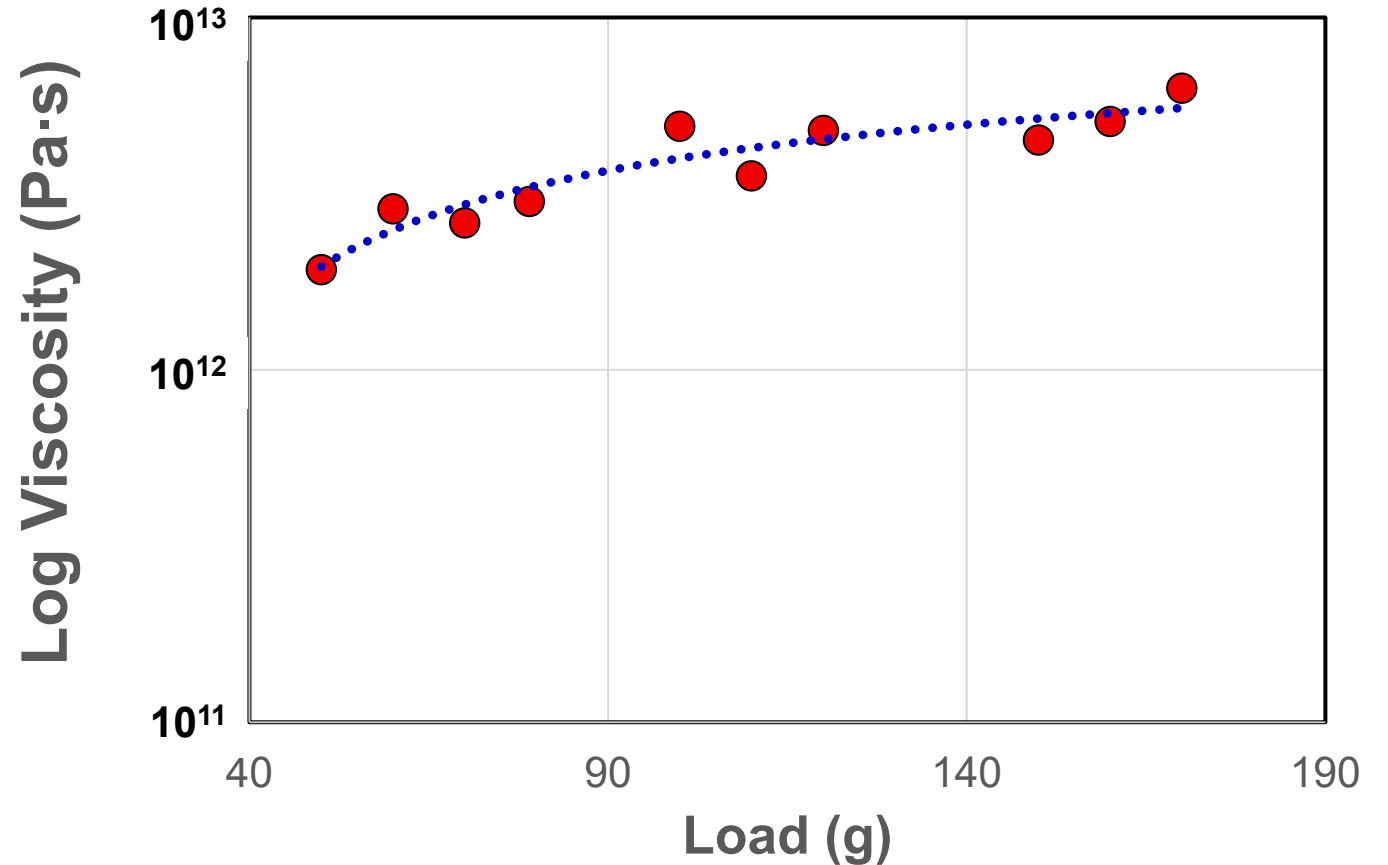


Following this “set point” the rod overcame frictional forces therefore deformation occurs with effective loads

Viscosity versus applied load

The dependence of viscosity on load is proposed to be due to *friction in the load train*.

This is an area that requires attention in instrument design.

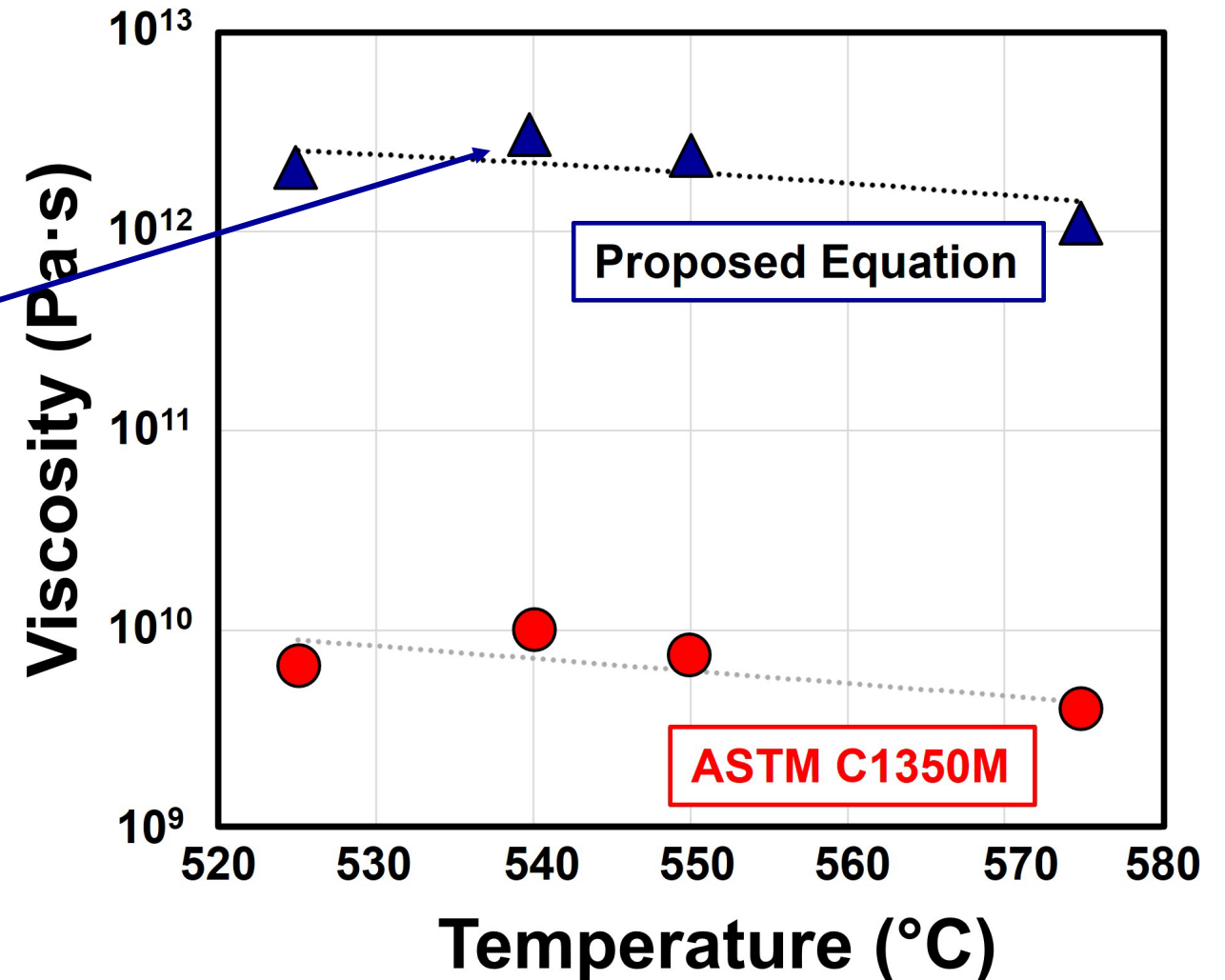


Comparison of the viscosity calculations

Measured T_G to be 540°C via
Differential Scanning Calorimetry

Viscosity at $T_G = 10^{12.4} \text{ Pa}\cdot\text{s}$

Measured log viscosity: 12.44 ± 0.2



Problems with Bending Beam Measurement Instruments

Problem	Potential Solution
Vertical loading system (mass)	Lever-based loading system
Load train friction	Reduce friction
Vertical tube furnace (challenging for specimen alignment)	Front-loading system
MoSi ₂ resistance heated system	Move to Kanthal (FeCrAl)
LVDT deflection measurement	Acceptable (if other problems fixed)
Tension-compression in <i>flexure</i> specimen?	May need to be addressed in a more sophisticated analysis

ASTM 'Corrections'

Sample mass

M = mass of load train + added mass

$$\left[\mathbf{M} + \frac{\rho AL}{1.6} \right]$$

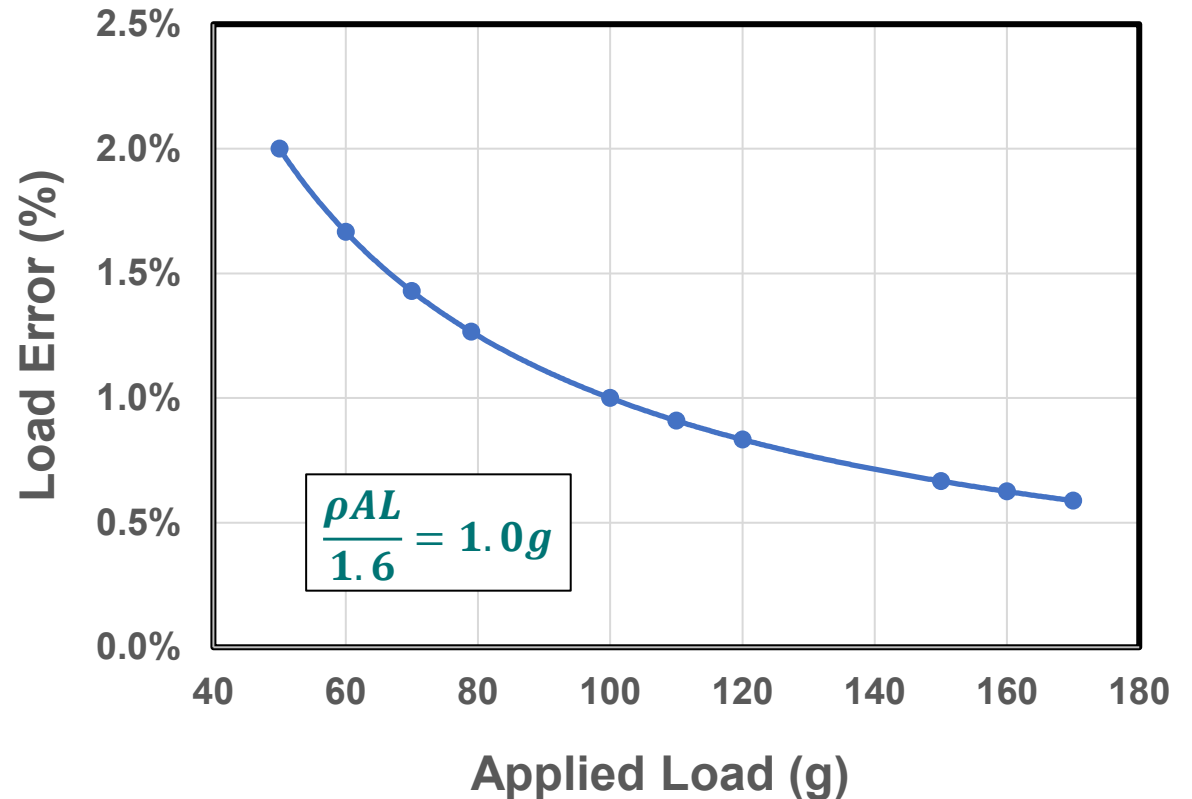
3 mm diameter rod
2.5 cm long
Density = 2.44 g/cm³

Not suggesting this is ignored, but it appears the error would be small.

If the span is larger, the mass of the sample is sufficient to create deformation.

Measured viscosity of porcelains during firing. (A. Buchtel, M.S. Thesis, 2004)

$$\eta = \frac{g L^3}{1440 I_c (dh/dt)} \left[\mathbf{M} + \frac{\rho AL}{1.6} \right] \left[\frac{(1 + \alpha_s T)^3}{(1 + \alpha_g T)^4} \right]$$



Compensation for specimen dimensions and span

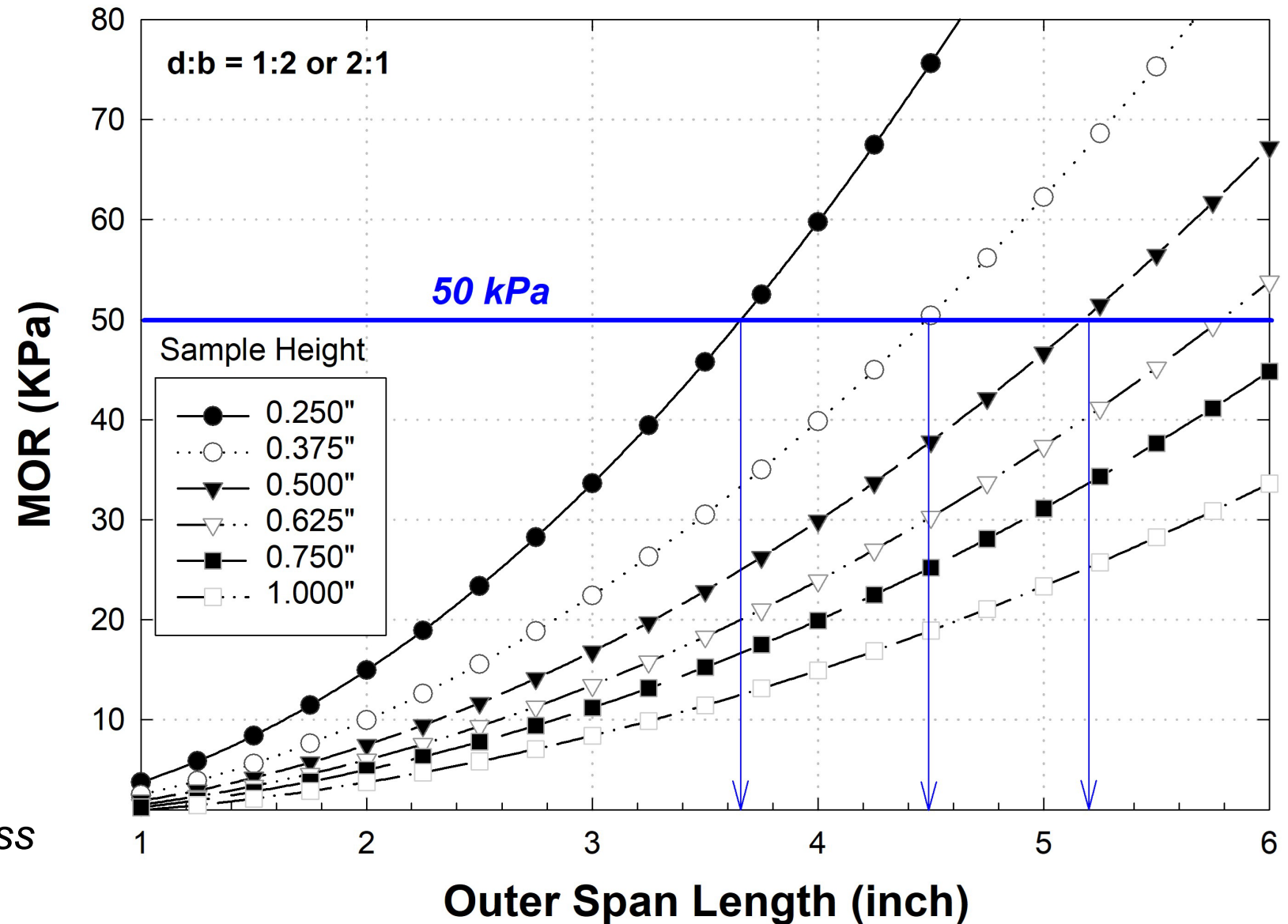
Specimen mass
contribution only.
(No additional
applied load)

OFTS:

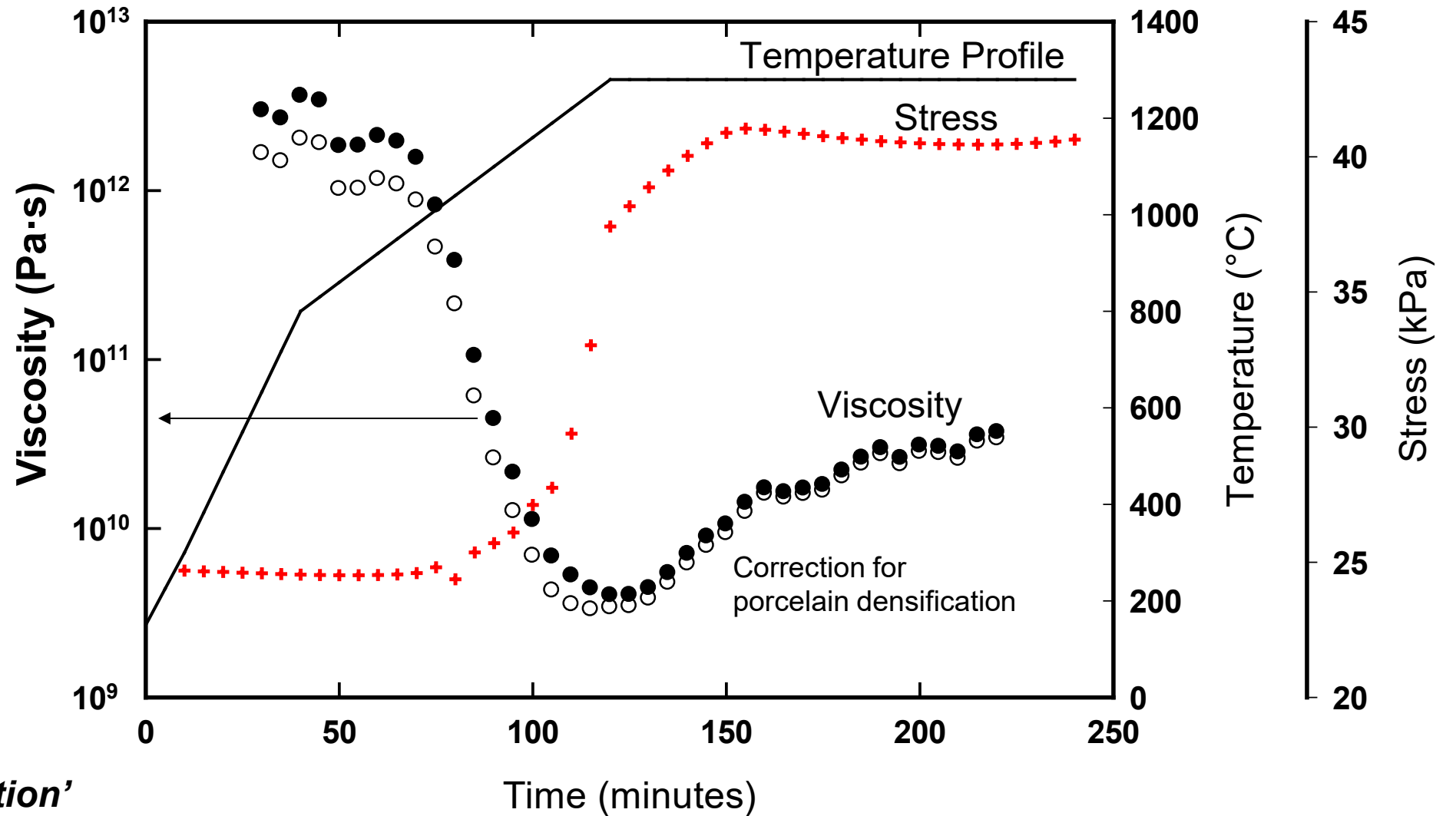
↑ with **span**

↓ with **thickness**

OFTS: *Outer Fiber Tensile Stress*
MOR: *Modulus of Rupture*



Bending Viscosity of Porcelain During Firing



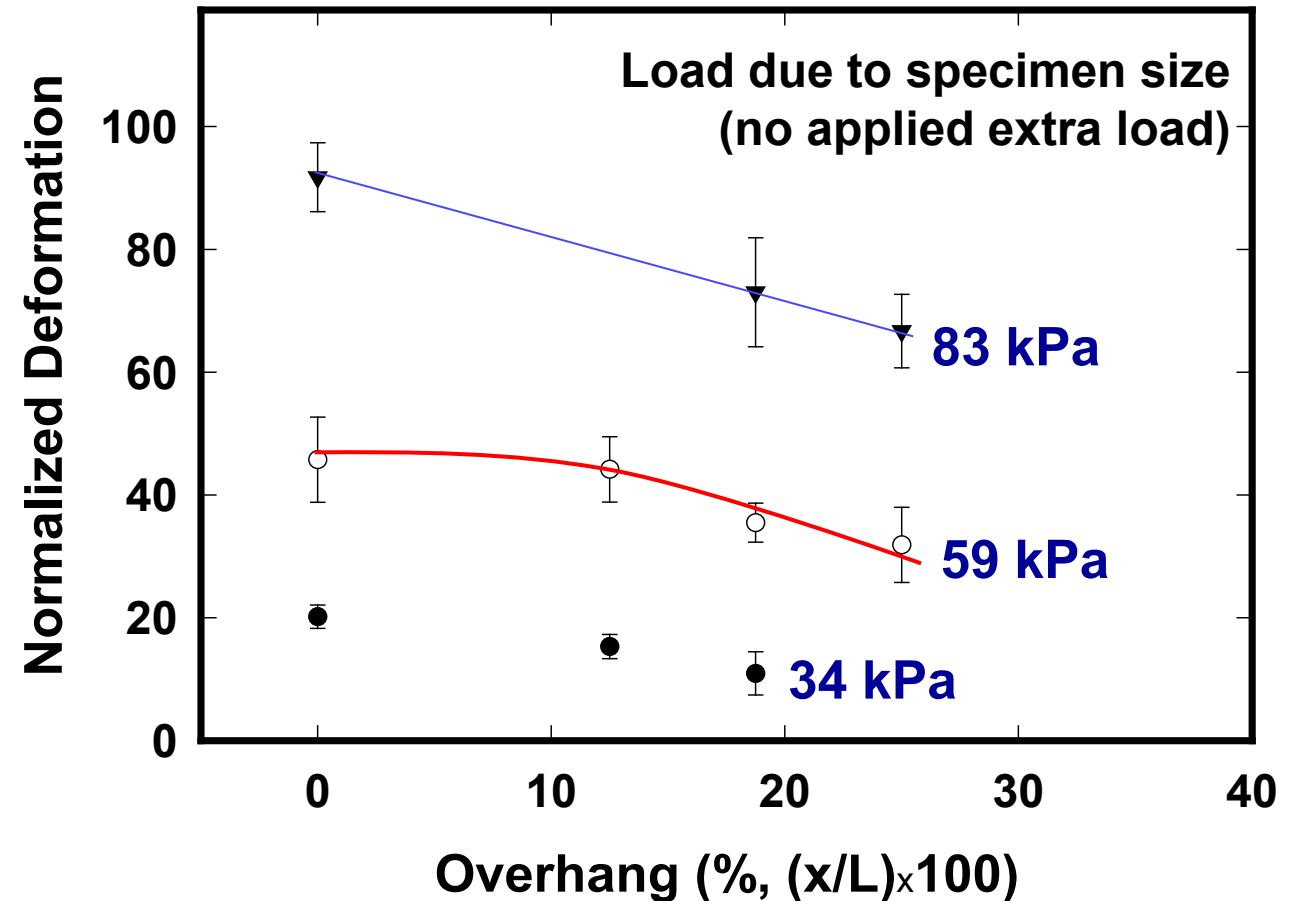
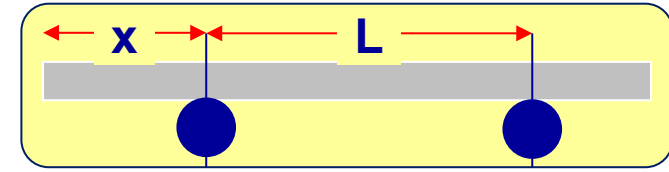
Effect of Overhang

Overhang reduces the center-point deformation.

Therefore increases the measured viscosity

ASTM should establish a specific specimen size or overhang* for this test.

In general, bending beam specimens should be standardized to improve the reliability and reproducibility of the measurement.



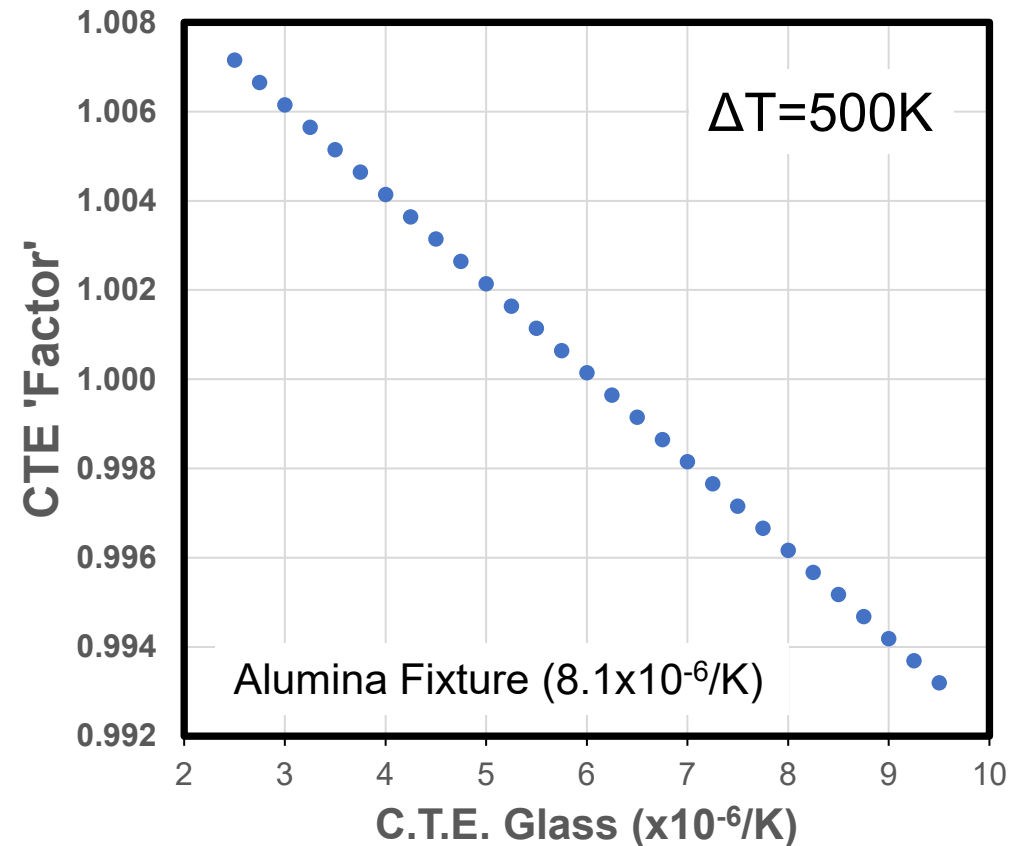
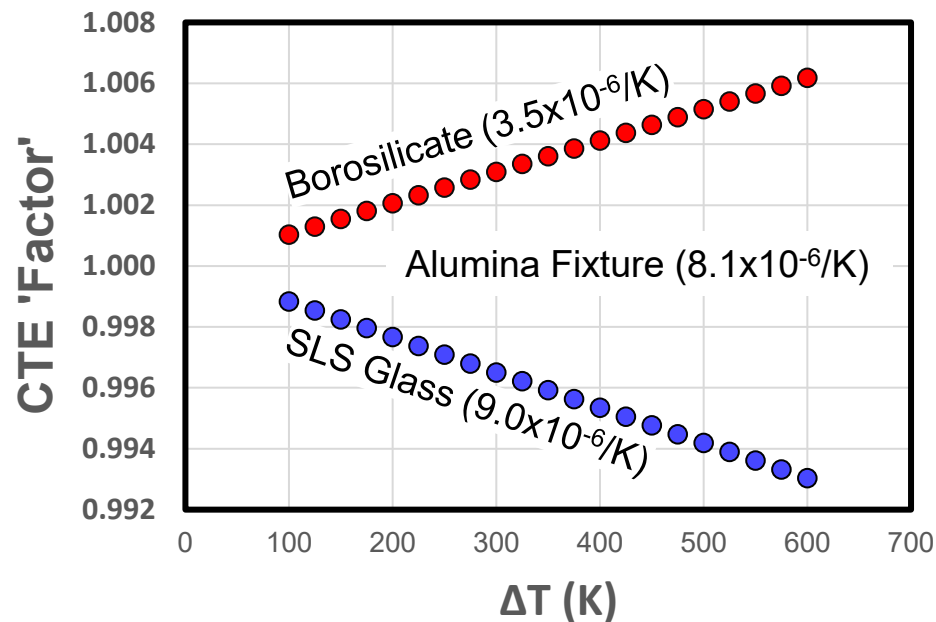
* ASTM C1499 recommends $\leq 20\%$ overhang.

Thermal Expansion Correction

$$\eta = \frac{g L^3}{1440 I_c (dh/dt)} \left[M + \frac{\rho AL}{1.6} \left[\frac{(1 + \alpha_s T)^3}{(1 + \alpha_g T)^4} \right] \right]$$

$$\frac{(1 + \alpha_s T)^3}{(1 + \alpha_g T)^4} \quad T \rightarrow \Delta T \quad \text{Correction is less than 1\%}$$

ΔT Contribution



Thermal Expansion Correction

Span (L) is the main issue.

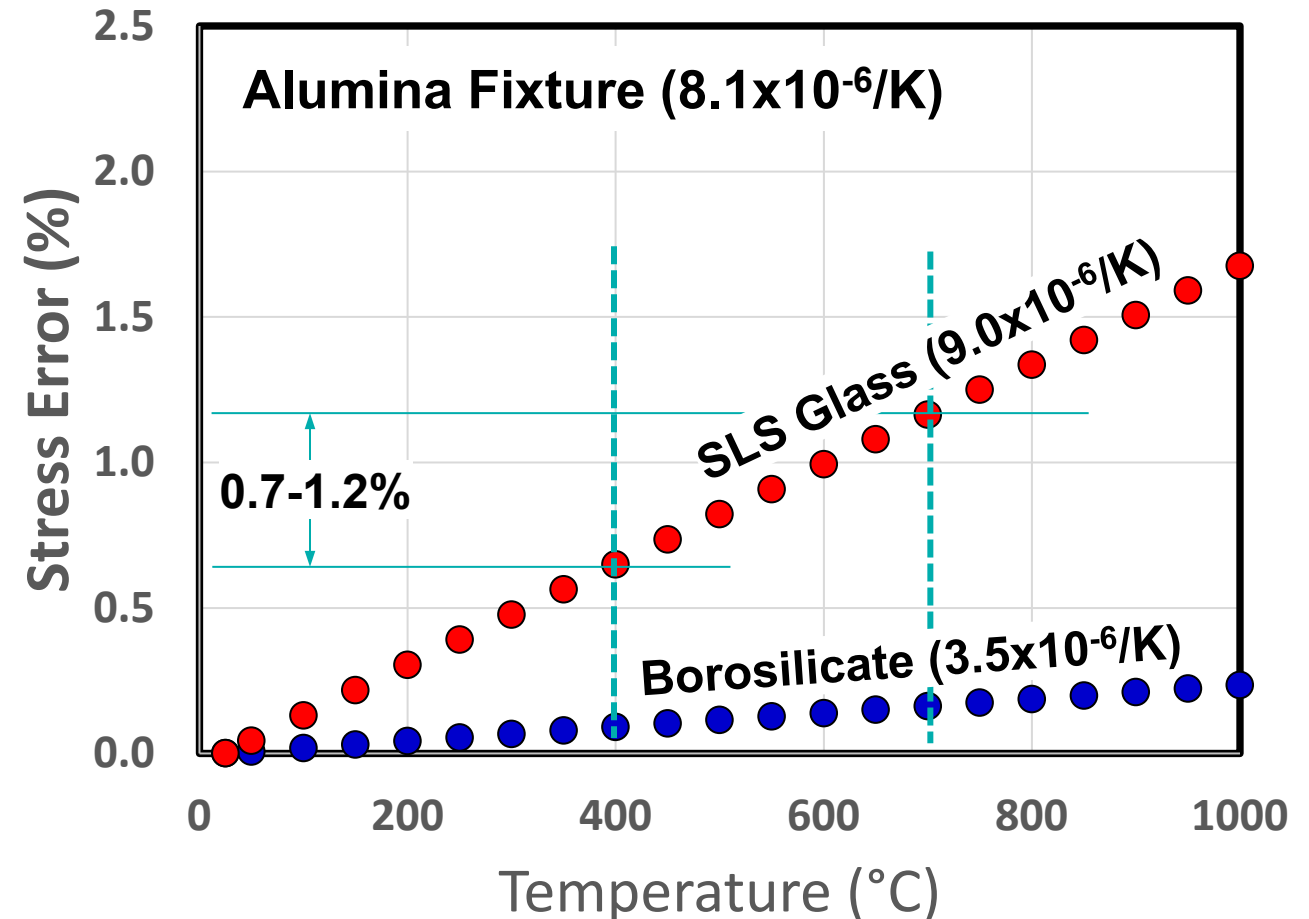
$$\frac{(1 + \alpha_s T)^3}{(1 + \alpha_g T)^4}$$

CTE of the fixture means
that L increases with
temperature.

CTE of the specimen
only alters (increases)
b and d.

Error is independent of
specimen shape.

ΔT Contribution



Summary and Conclusions

The viscosity obtained by the measurement of the deflection of a beam in bending should use the strain rate calculated from the deflection rate.

Recommend that ASTM C-14 Committee revisit C1350M for viscosity via beam bending.

Make recommendations for specimen size, perhaps following the ASTM guidelines established for flexure testing.

There are likely relatively simple changes that would make the method easier to execute experimentally.

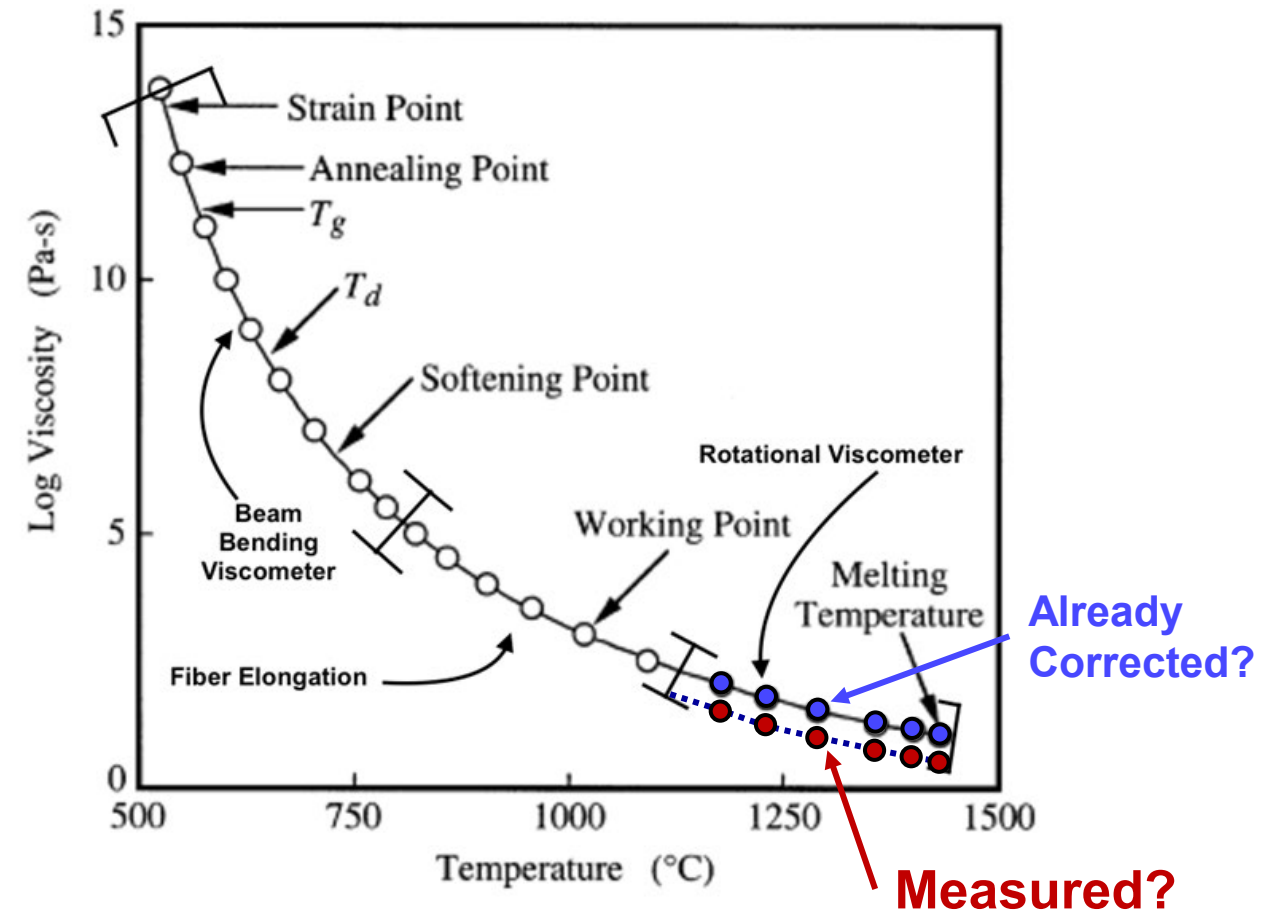
$$\text{Rod } \eta_e = \frac{8PL^3}{6\pi d^4 \left(\frac{dD}{dt} \right)}$$

$$\text{Bar } \eta_e = \frac{PL^3}{4bd^3 \left(\frac{dD}{dt} \right)}$$

Recommendation for Glass Viscosity Reporting

Most forming operations → shear-free
Shear viscosity, at high temperatures is, based on Trouton, offset by 3x (½ order of magnitude).

Recommend that shear viscosities be adjusted (3μ) to reflect this difference and therefore align with shear free measurements.



Trouton (1906) $3\mu = \eta_e$ ← “Elongational Viscosity”
(shear-free viscosity)
↑ “Shear Viscosity”

Thank You

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