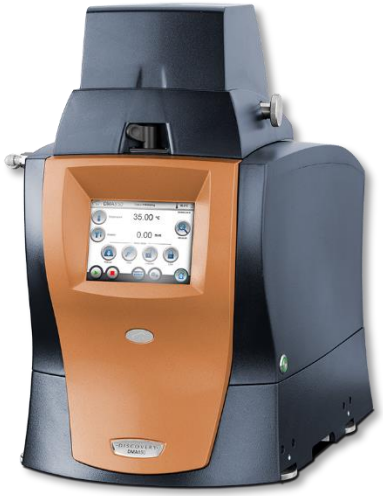


Dynamic Mechanical Analysis
Basic Theory & Applications Training
Day 2

Course outline

Part 1

- Basic Theories of Dynamic Mechanical Analysis
- DMA Instrumentation and Clamps
- Introduction to DMA Experiments
  - Dynamic tests
  - Transient tests
- Appendix: Screenshots from the instrument control software

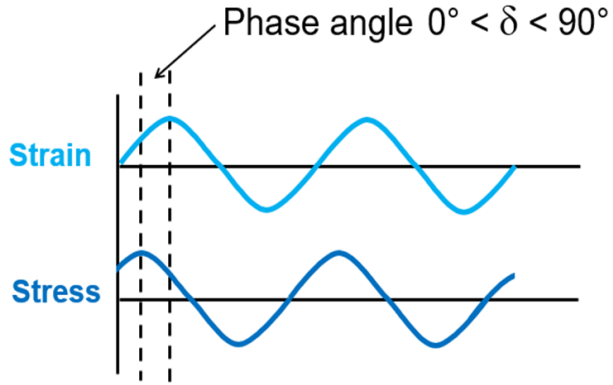
Part 2

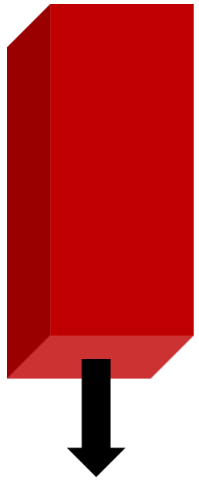
- Recap of Part 1
- DMA Applications and data interpretation
- Troubleshooting experimental Issues
- Time-temperature superposition (TTS)

Recap of Day 1

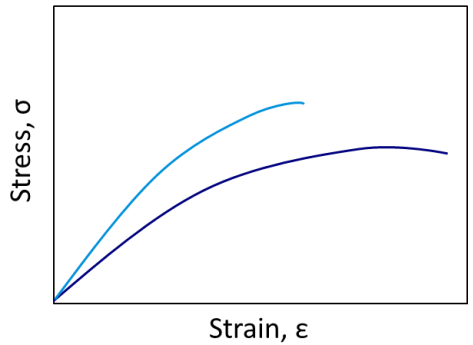
Recap: Viscoelasticity and mechanical tests

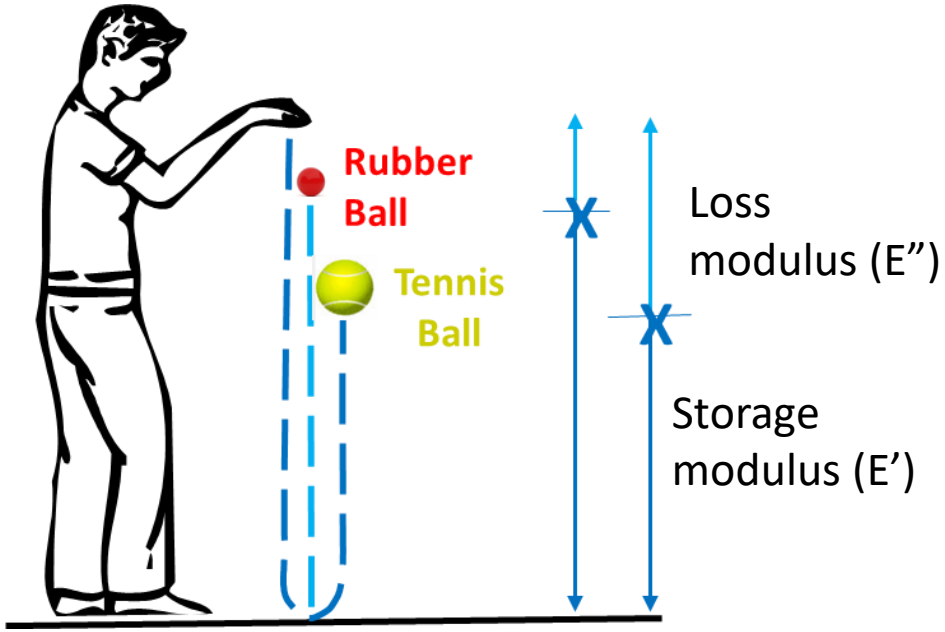
Dynamic mechanical tests





Conventional stress-strain tests





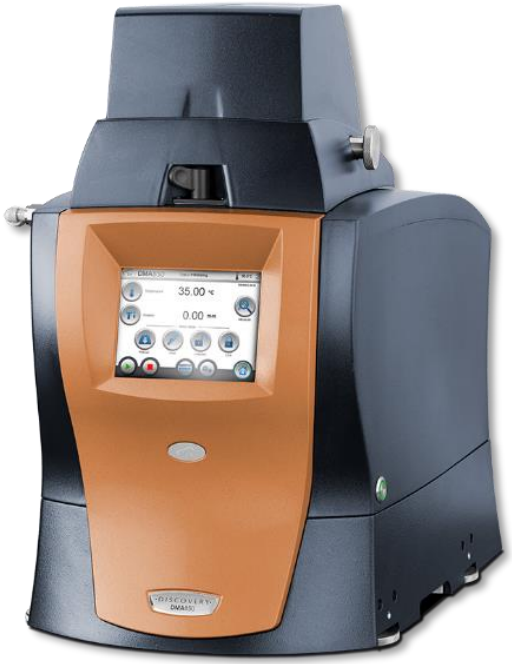
$$\tan\delta = \left(\frac{E''}{E'}\right)$$

Measure of material damping. Increasing tan δ implies a greater viscous property while having the appropriate level of stiffness.

Recap: DMA instrumentation

RSA G2

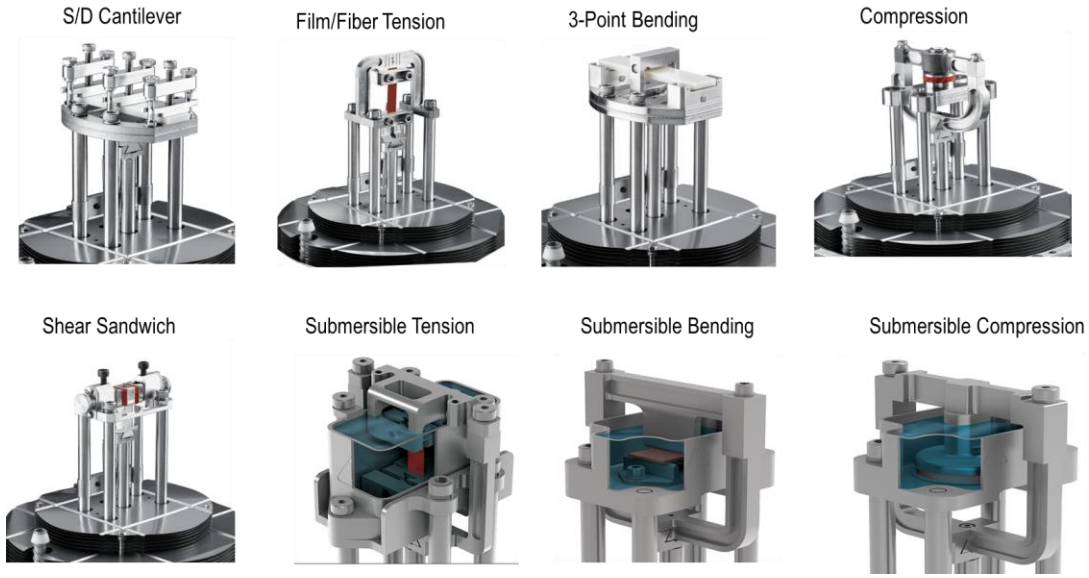
Discovery DMA850



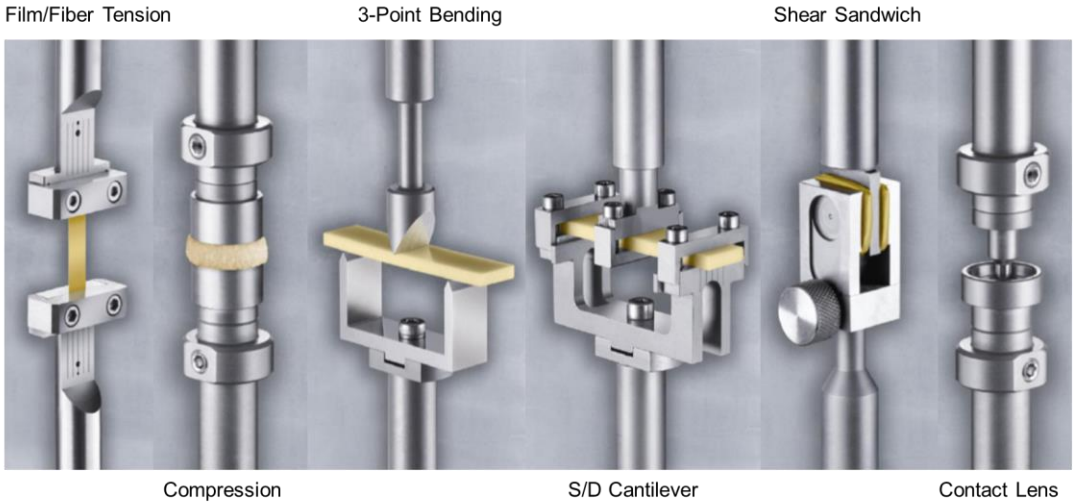
Electroforce series (high load frame, fatigue)

Recap: DMA clamps

850/800 clamps

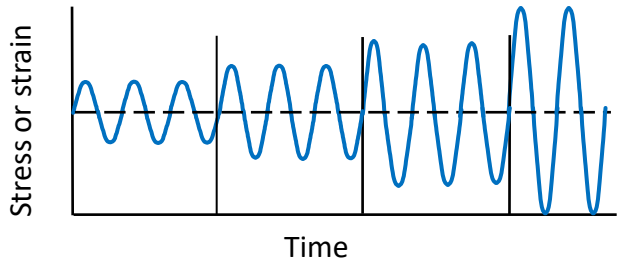


RSA-G2 clamps

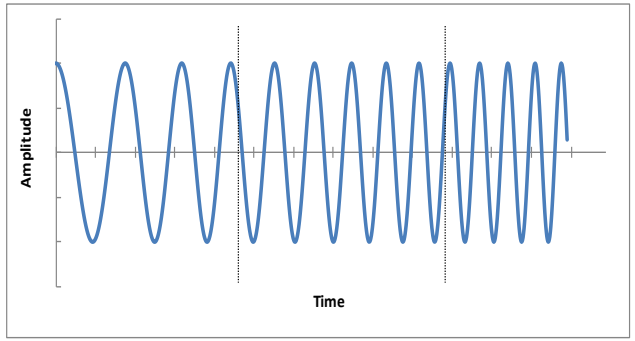


Recap: DMA dynamic tests

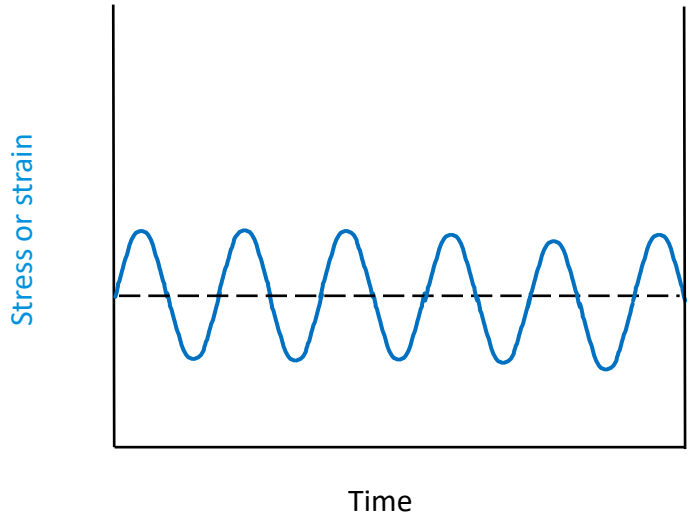
Strain Sweep



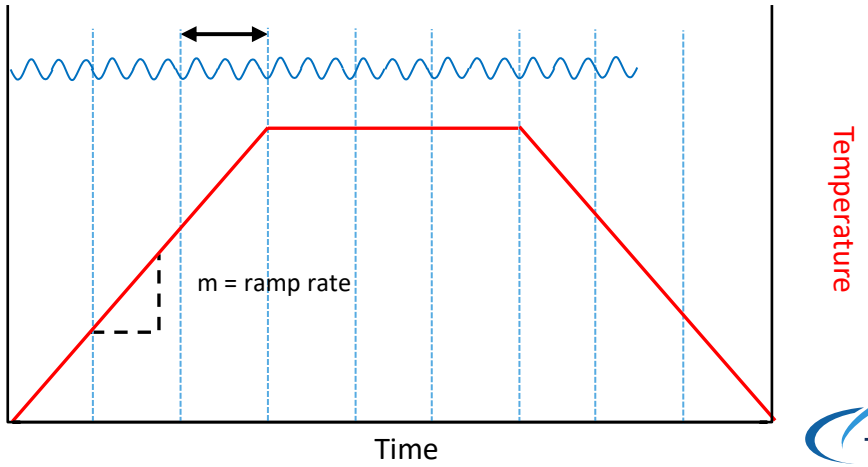
Frequency Sweep



Time sweep

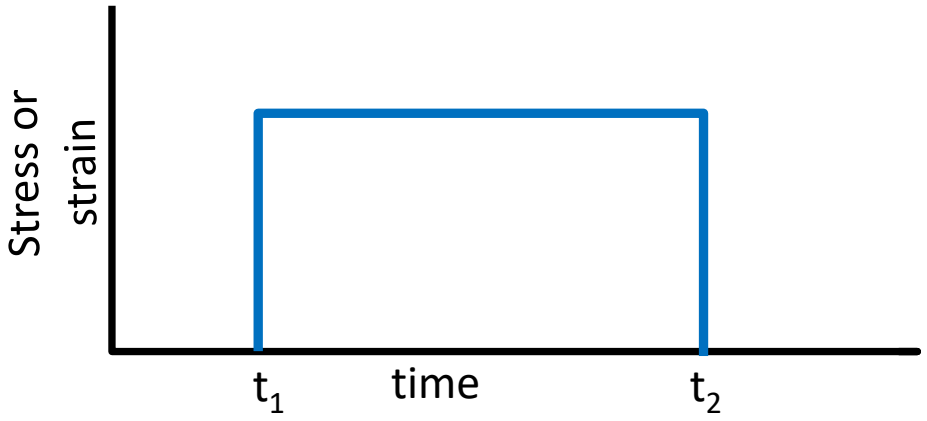


Temperature ramp/sweep

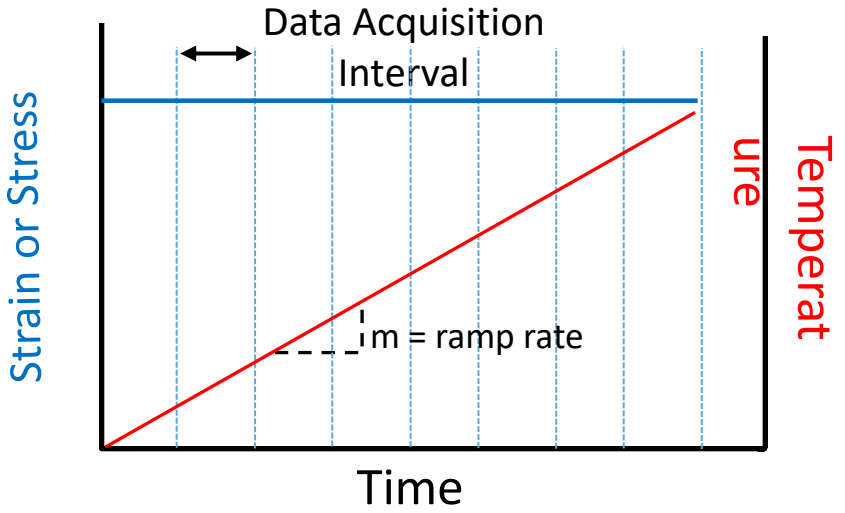


Recap: DMA transient tests

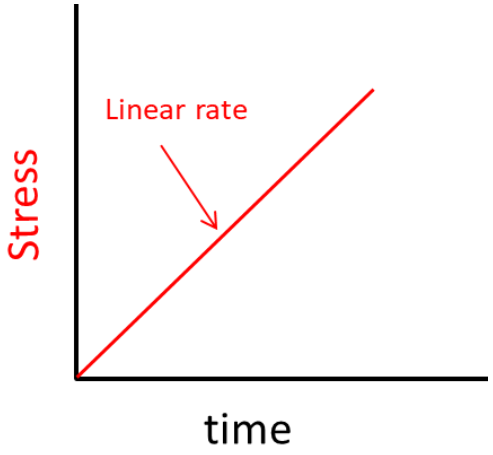
Creep/stress relaxation

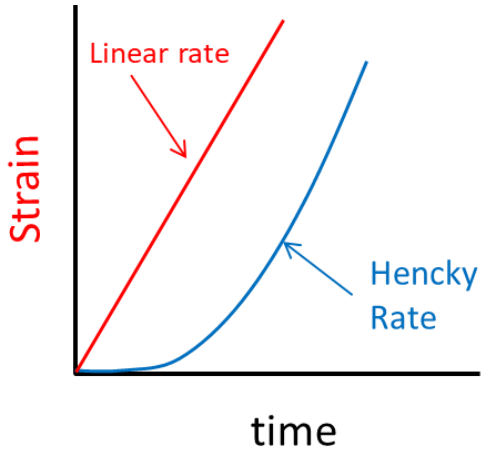


Iso-strain/Iso-stress Temperature Ramp



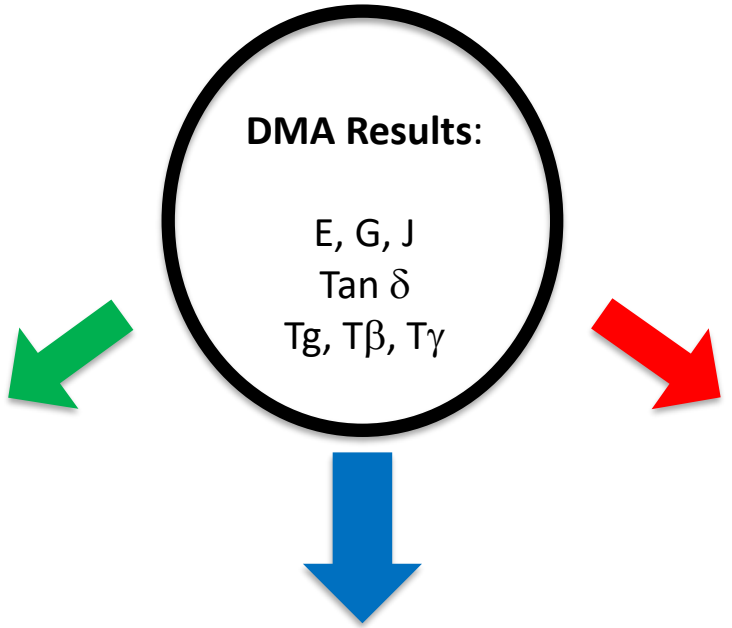
Stress-strain tests





DMA Applications and Data Interpretation

The DMA results can correlate to…



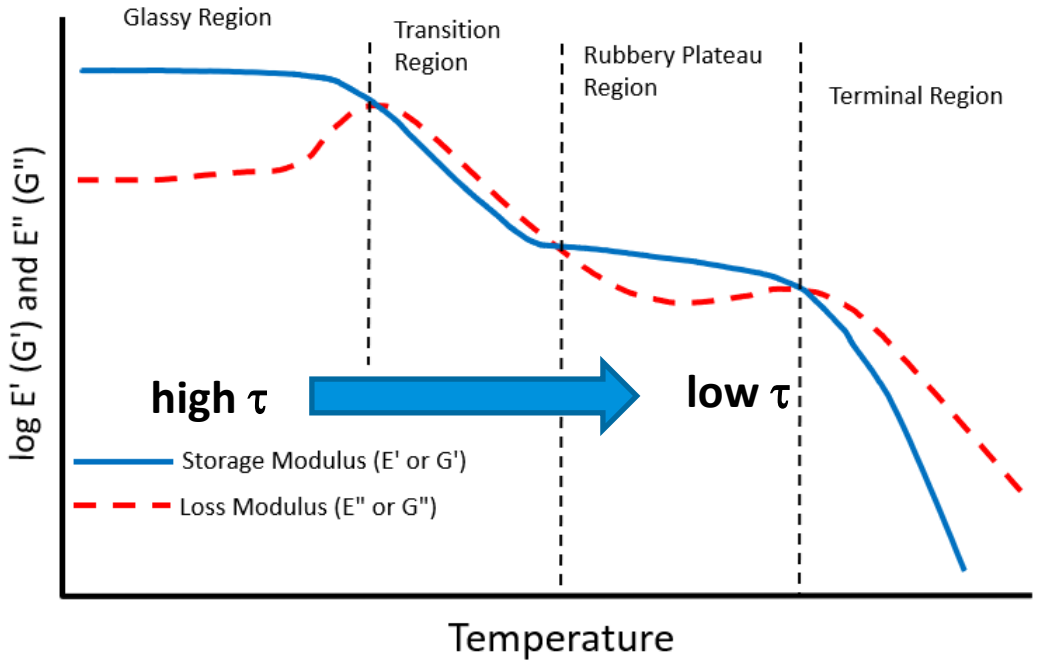
Molecular structures
- MW and MWD
- Branching
- Crystallinity
- Crosslinking
- Phase
- Relaxation

Processing:
- Heat history
- Residual stress
- Shear or orientation
- Temperature
- Fillers

Product properties
- Transition temperatures (Tg, Tβ, Tγ)
- Environmental resistance
- Impact strength
- Long term behavior, aging
- Adhesion

Most common DMA test – temperature ramp

- The most Common DMA measurement is a dynamic temperature ramp
- The test results report modulus (E*, E', E"), damping factor (Tan δ) and transition temperatures (T_g)
- Provide information to polymer's structure-property relationship



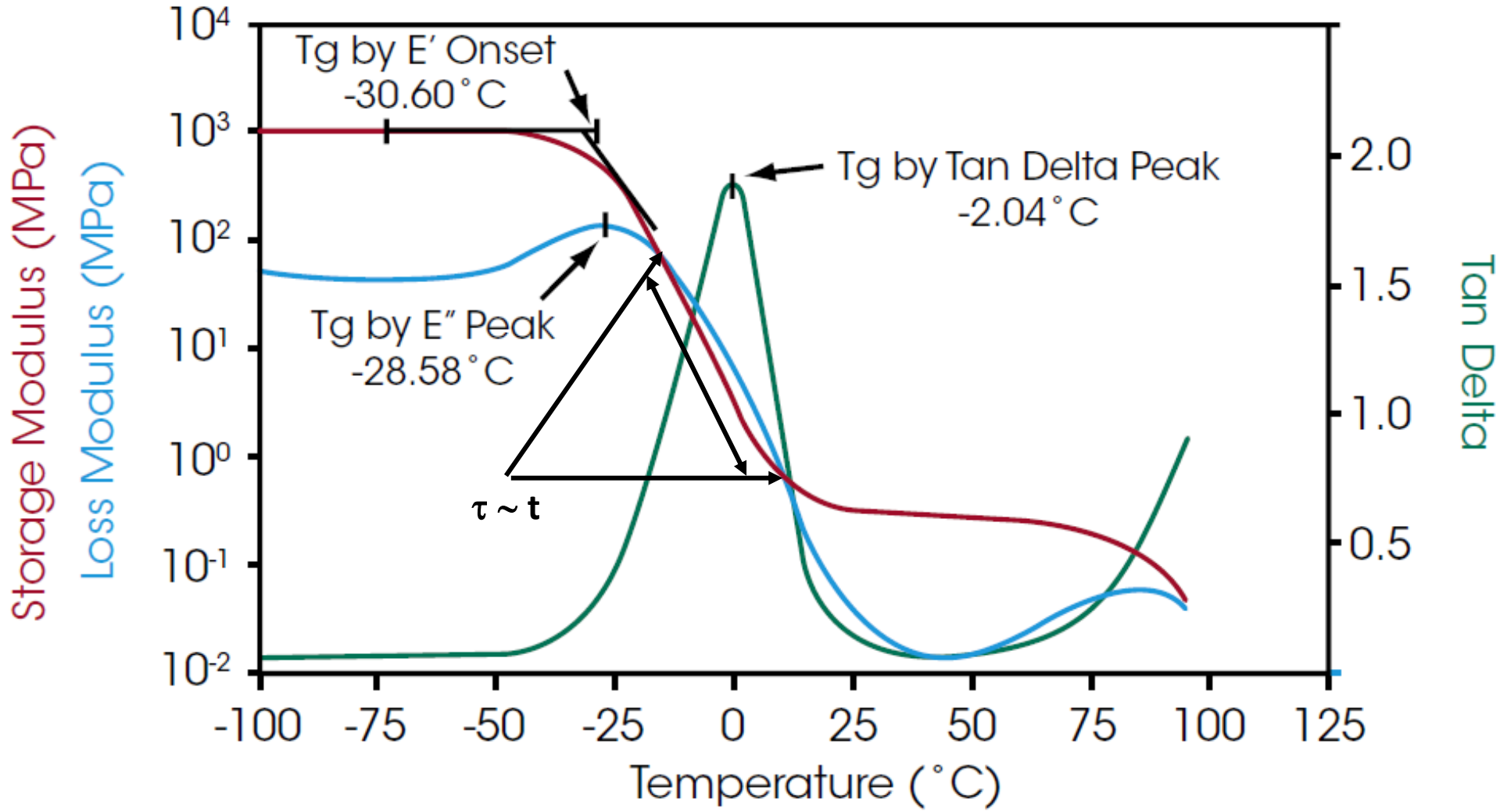
Frequency (or 1/t is fixed)

What is Glass Transition (T_g)?

- A transition over a ***range of temperature*** from a glassy state to a rubber state in an amorphous material
- Mechanical:
  - Below the Glass Transition, the material is in a brittle, glassy state, with a modulus of 10^9 Pa
  - Above the Glass Transition, the material becomes soft and flexible, and the modulus decreases two to three decades
- Molecular:
  - Below the Glass Transition, polymer chains are locked in place, without sufficient energy to overcome the barrier for rotational or translational motion.
  - At temperatures above the Glass Transition, there is molecular mobility, and chains can slide past each other

How to Define T_g in DMA?

Onset of E' **Peak of E"** **Peak of Tan δ**



Secondary Transitions

Glass Transition (T_g)

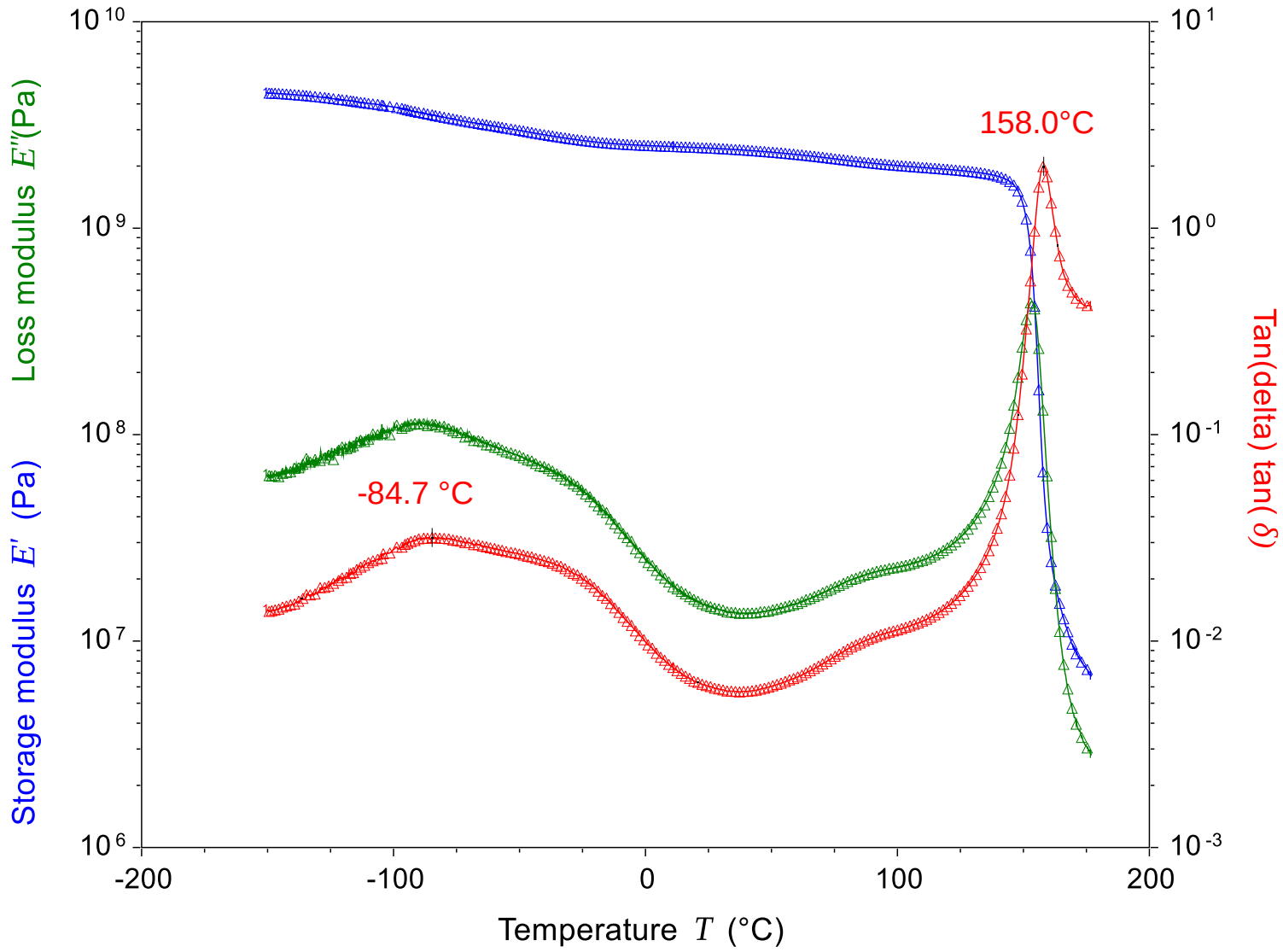
- Cooperative motion among a **large number** of chain segments, including those from neighboring polymer chains

Secondary Transitions (T_β, T_γ)

- Local Main-Chain Motion – intra-molecular rotational motion of main chain segments four to six atoms in length
- Side group motion with some cooperative motion from the main chain
- Internal motion within a side group without interference from side group.
- Motion of, or within, a small molecule or diluent dissolved in the polymer (eg. plasticizer.)

Turi,Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 487.

Primary and Secondary Transitions in PC



Instrument: DMA850
Temperature:
-150°C to 180°C
Heating rate: 3°C/min
Frequency: 1Hz
Amplitude: 15 μm

What Will Affect T_g and Modulus?

- Heating rate
  - Thermal lag
- Test frequency
- Polymer structures
  - Rigid polymer chain shows higher T_g (e.g. PS)
  - Flexible polymer chain shows lower T_g (e.g. PE)
  - Crystallization
  - Degree of crosslinking

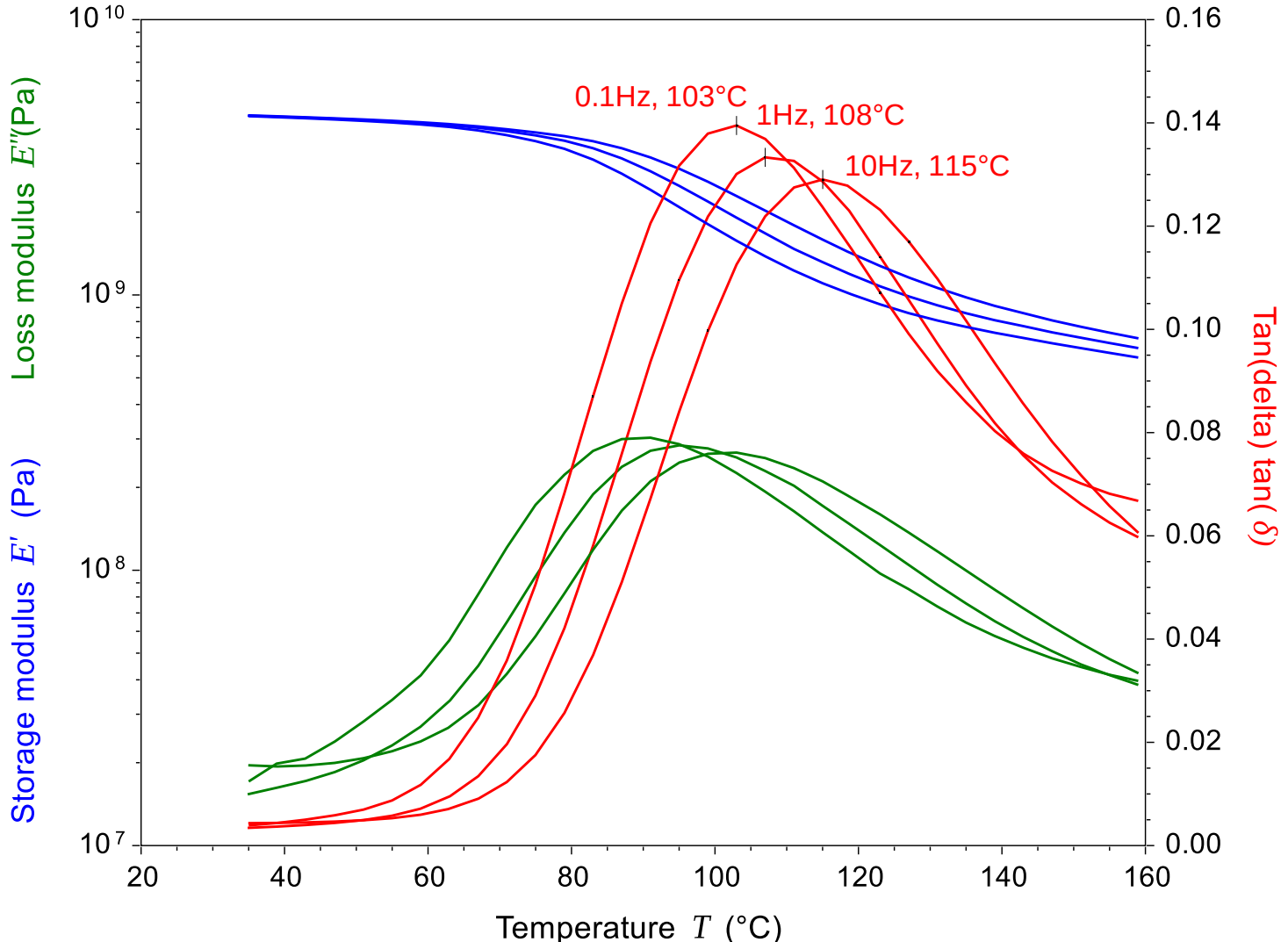
The Effect of Test Frequency on T_g

- The glass transition is strongly influenced by the frequency of the test. The T_g is a molecular relaxation that involves cooperative segmental motion.
- Because the *RATE* of segmental motion depends on temperature, as the frequency increases, the relaxations associated with the T_g (where $\tau \sim t$) can only happen at higher temperatures (higher temperatures corresponds to higher kinetic energy that is imparted to the system).
- In general, increasing the frequency will
  - Increase the T_g
  - Broaden the peak
  - Decrease the intensity of tan δ or loss modulus
  - Decrease the slope of the storage modulus curve in the region of the transition.

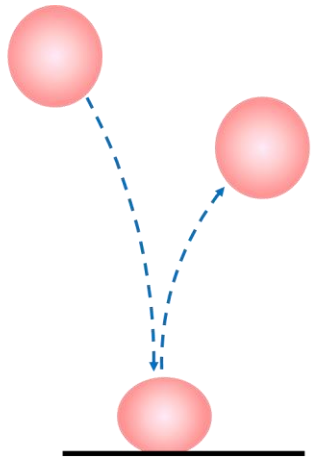
Turi, Edith, A, Thermal Characterization of Polymeric Materials, Second Edition, Volume I., Academic Press, Brooklyn, New York, P. 529.

PET Film: Effect of Frequency on T_g

- PET film tested at 0.1 Hz, 1Hz and 10 Hz

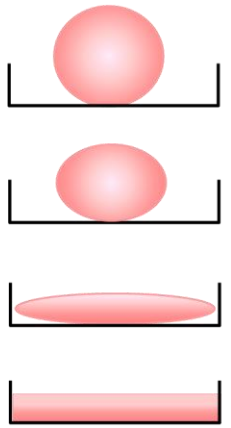


$$\tan\delta = \left(\frac{E''}{E'}\right)$$



Time-period of deformation is short (high frequency)

Increased elastic response
Higher E', lower E"
Lower tan δ

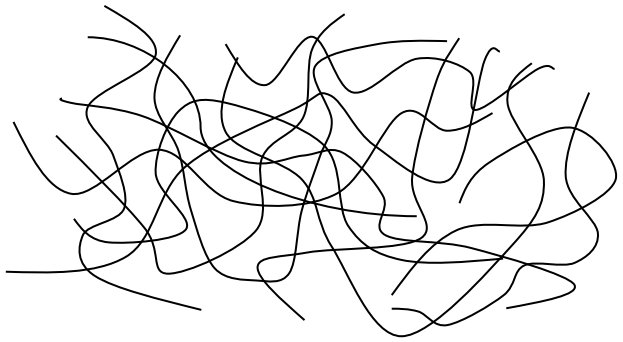


Time-period of deformation is long (low frequency)

Increased viscous response
Higher E", lower E'
Higher tan δ

Effect of Crystallinity in Polymer

"The major effect of the crystallite in a sample is to act as a crosslink in the polymer matrix. This makes the polymer behave as though it was a crosslinked network, but as the crystallite anchoring points are thermally labile, they disintegrate as the temperature approaches the melting temperature, and the material undergoes a progressive change in structure until beyond T_m, when it is molten"

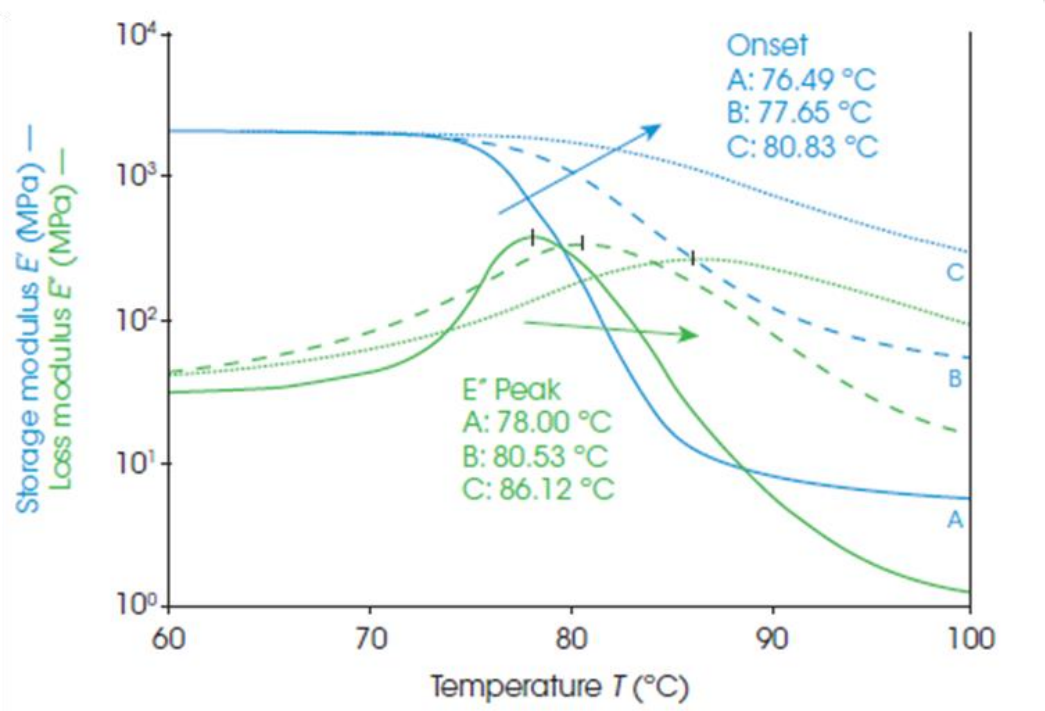


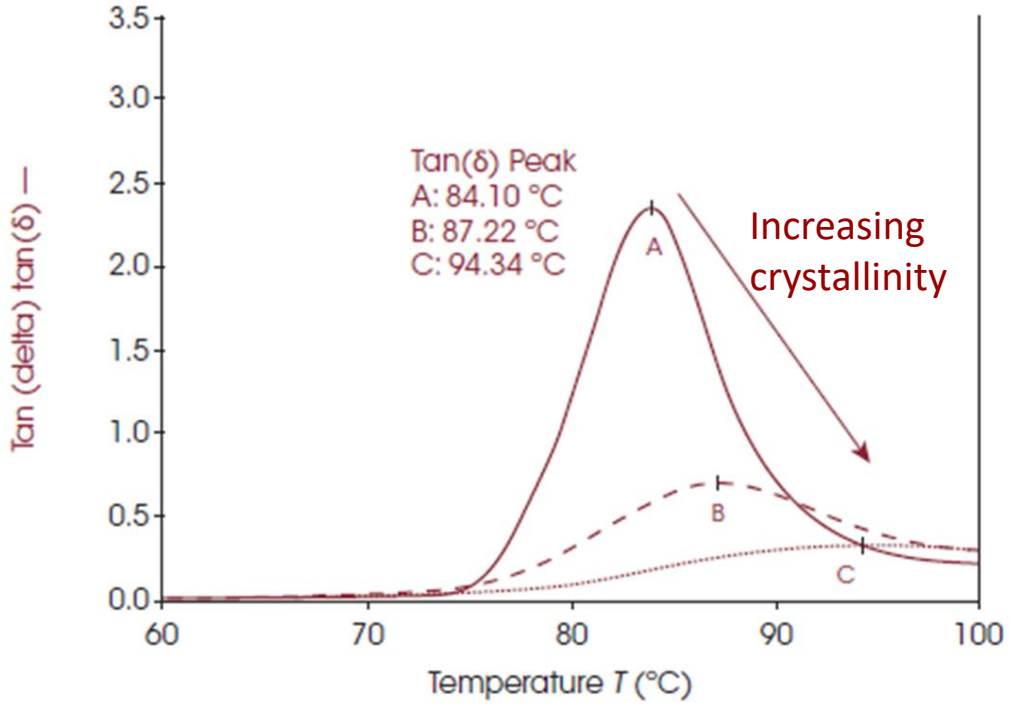
Random Chain
100% Amorphous

Fringed Micell
Crystalline

Cowie, J.M.G., Polymers: Chemistry & Physics of Modern Materials, 2nd Edition, 1991p. 330-332. ISBN 0 7514 0134 X

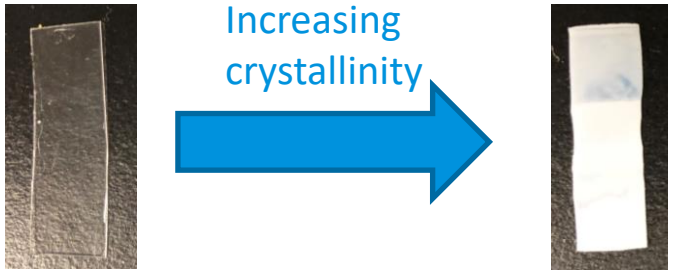
Effect of crystallinity on modulus and tan δ: PET





As polymer becomes more crystalline:

- **Tg shifted to higher temperature**
- **Transition becomes much broader and weaker (tan δ decreases)**
- **Rubbery plateau modulus increases**

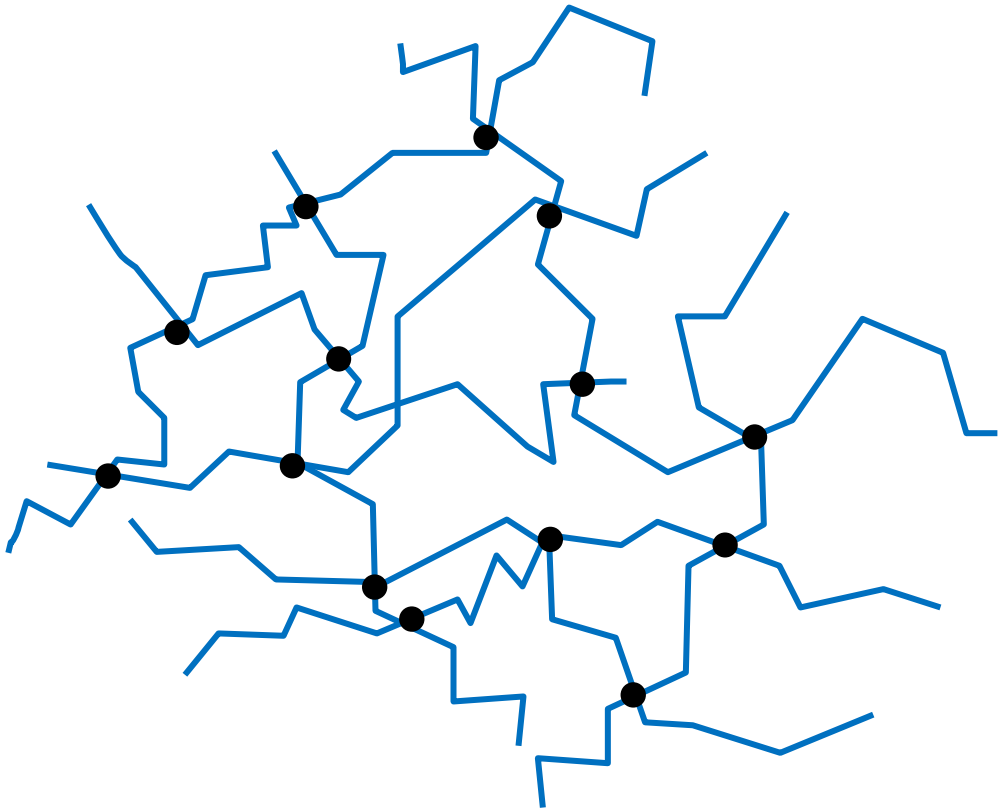


A **C**

TA Applications note: RH100 Measurement of Glass Transition Temperatures by Dynamic Mechanical Analysis and Rheology
https://www.tainstruments.com/pdf/literature/RH100.pdf

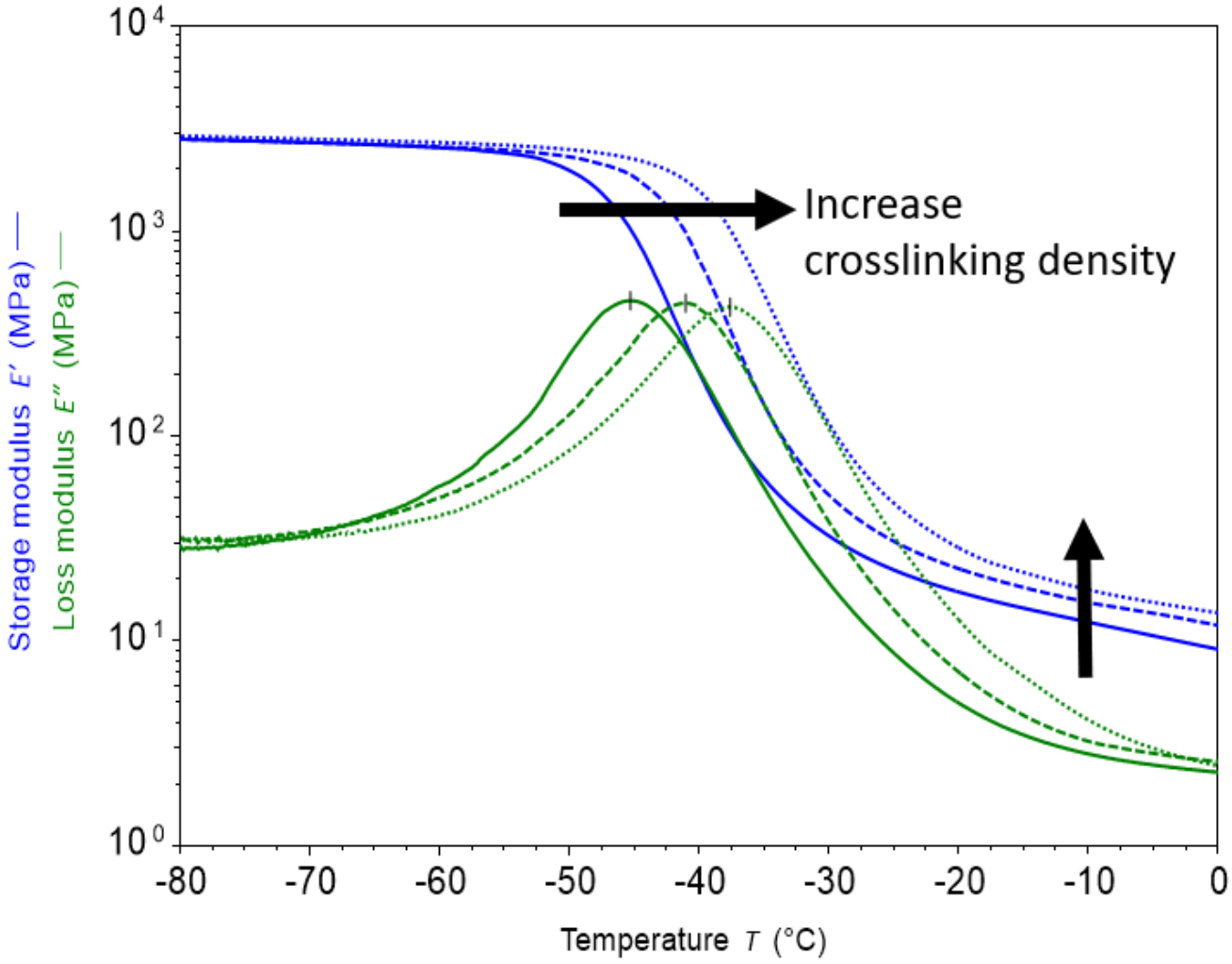
Polymer crosslinking

- Linear polymers can be chemically or physically joined at points to other chains along their length to create a crosslinked structure
- Chemically crosslinked systems are typically known as **thermosetting polymers** because the crosslinking agent is heat activated.
- Thermosetting polymers typically decompose before they can be molten.



Ward, I.M., Hadley, D.W., An Introduction to the Mechanical Properties of Solid Polymers, John Wiley & Sons Ltd., New York, 1993

Effect of crosslinking density on Tg

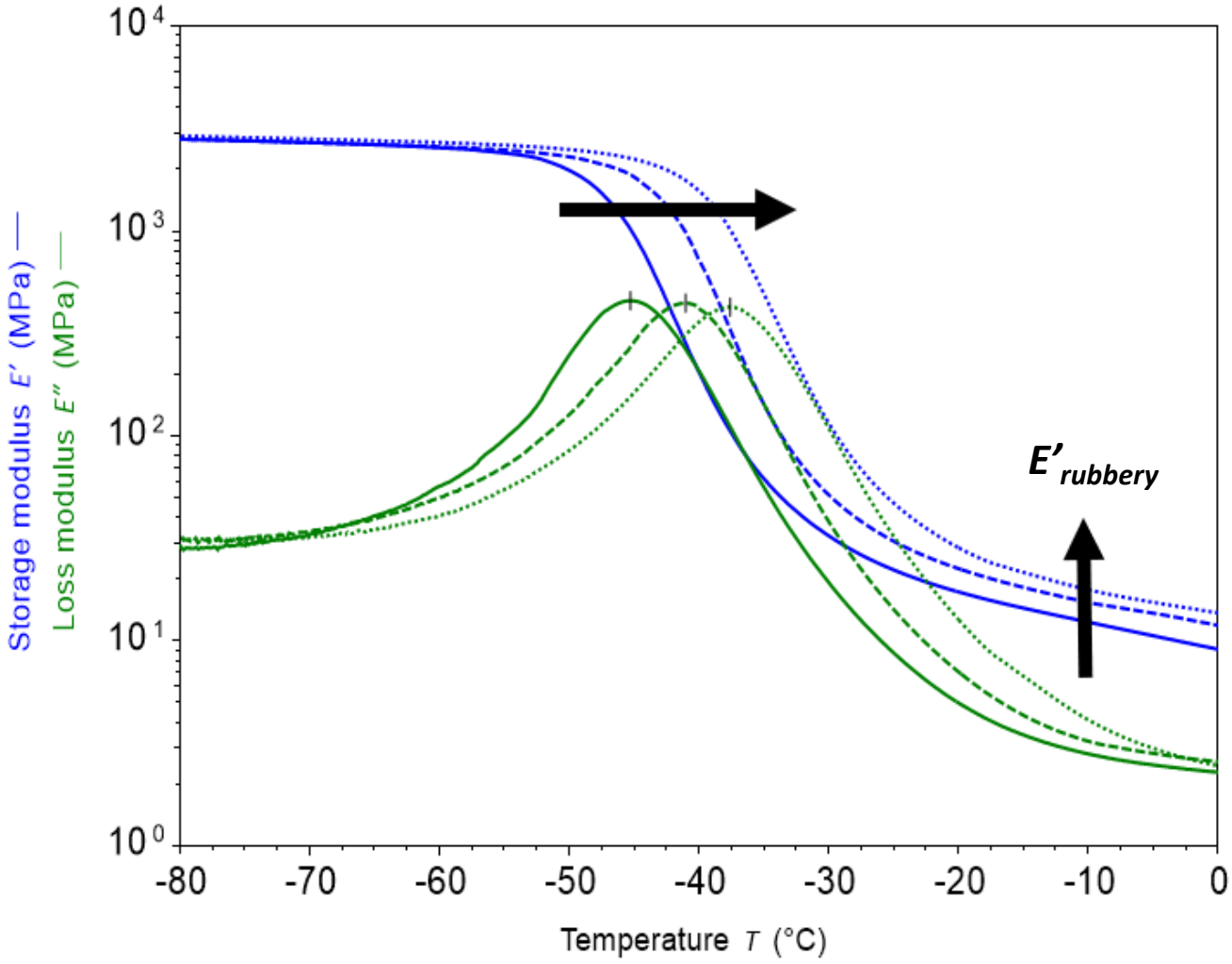


Increasing crosslinking density:

- Tg shifted to higher temperature
- Transition becomes broader and weaker (tan δ decreases)
- Rubbery plateau modulus increases

TA Applications note: RH100 Measurement of Glass Transition Temperatures by Dynamic Mechanical Analysis and Rheology
https://www.tainstruments.com/pdf/literature/RH100.pdf

Quantification of crosslinking density



- Crosslinking density:

$$M_c = \frac{3RTd}{E'_{rubbery}}$$

M_c is the molecular weight between crosslinks
R is the universal gas constant
T is the absolute temperature (in K)
d is the density of the polymer.

- A lower Mc implies higher extent of crosslinking

TA Applications note RH102: Quantifying Polymer Crosslinking Density Using Rheology and DMA, https://www.tainstruments.com/pdf/literature/RH102.pdf

Applications of DMA in Polymer blends

Polymer Blends

Miscible (single Tg)

Homogeneous

Same chemistry, different MW or different polymer structure (linear, branched).

Combinations to achieve needed properties.

Heterogeneous

PS/PVME

PSAN/PMMA

PEO/PMMA

PB/ PIP

Cost saving without sacrificing properties

Immiscible (separate Tg's)

Compatiblized

Crystalline and Amorphous blend (PC/PET, Nylon/ABS)

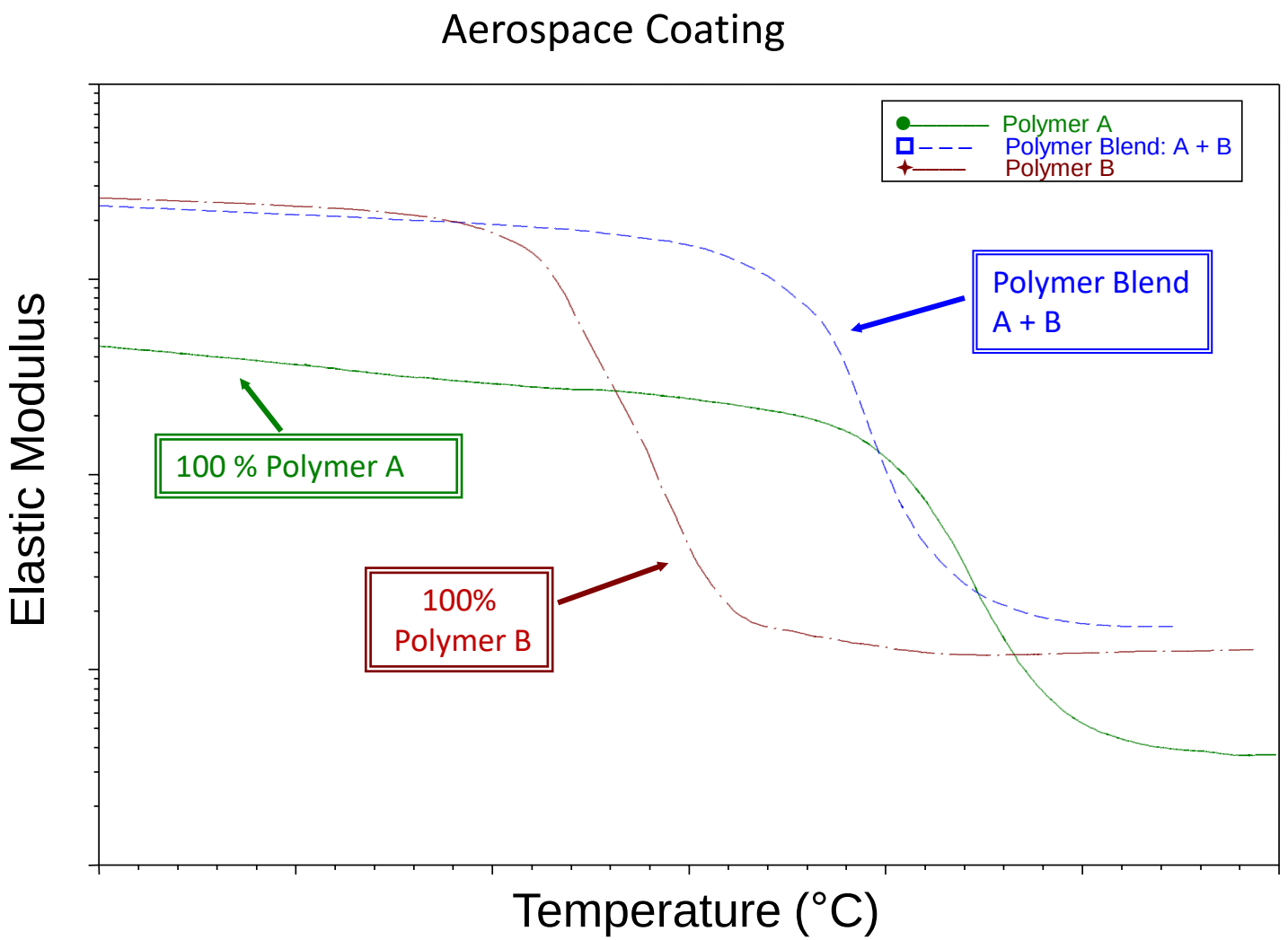
Thermoplastic and Elastomer blends (PP/EPDM). Achieve properties of both polymers.

Not compatible

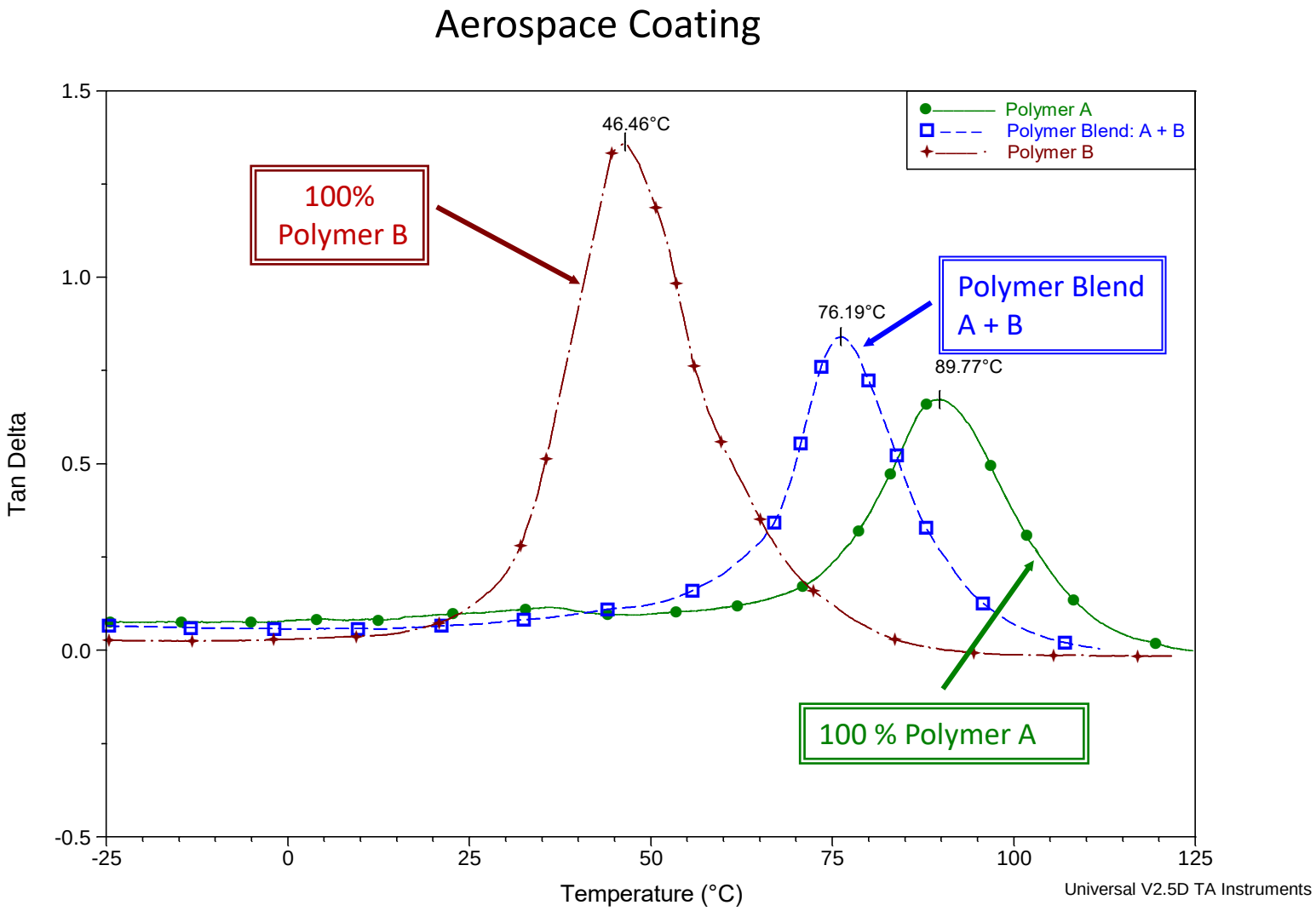
Phase Separation

Structural Failure

Polymer Blend – Miscible Case

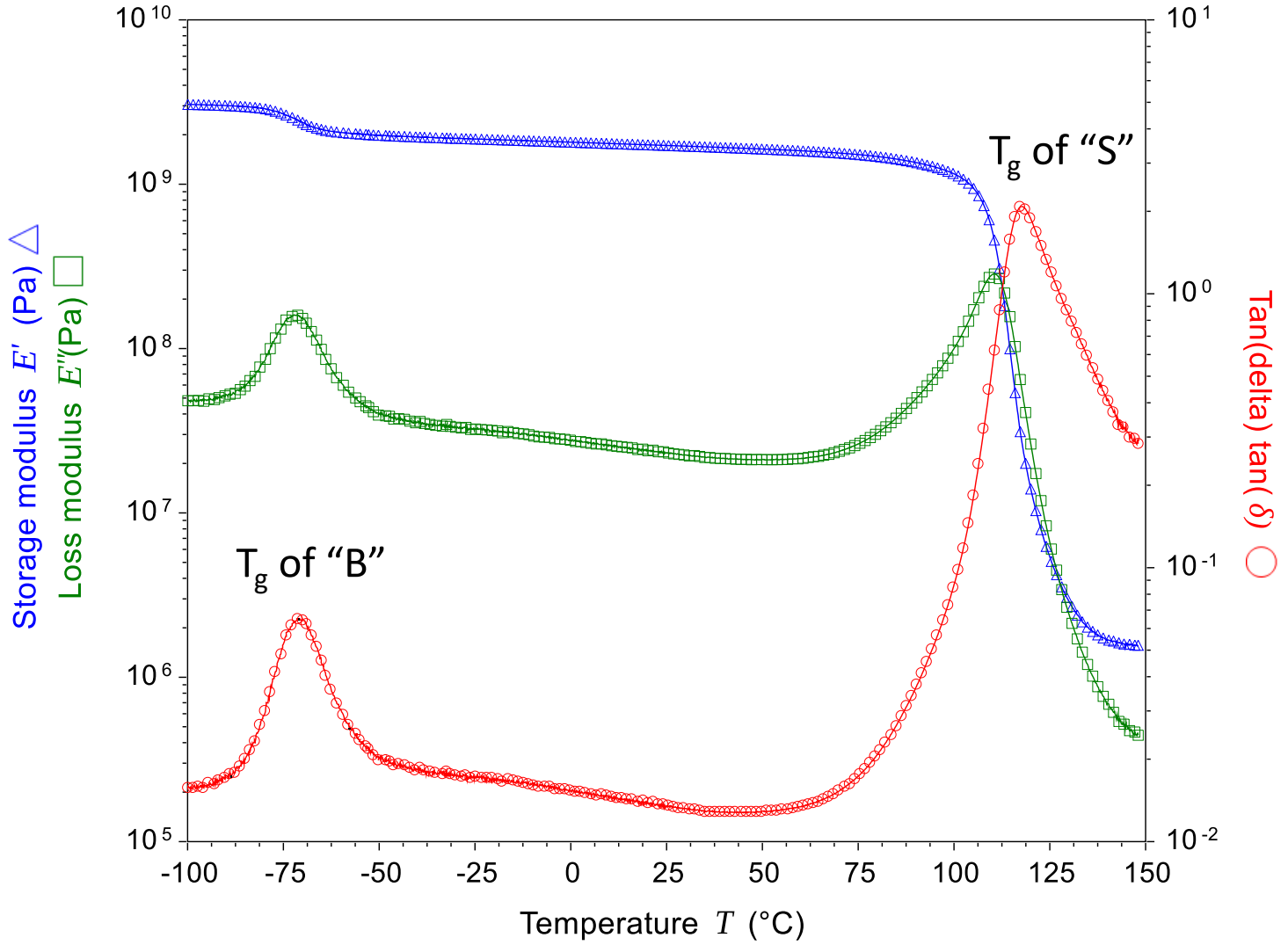


Polymer Blend – Miscible Case



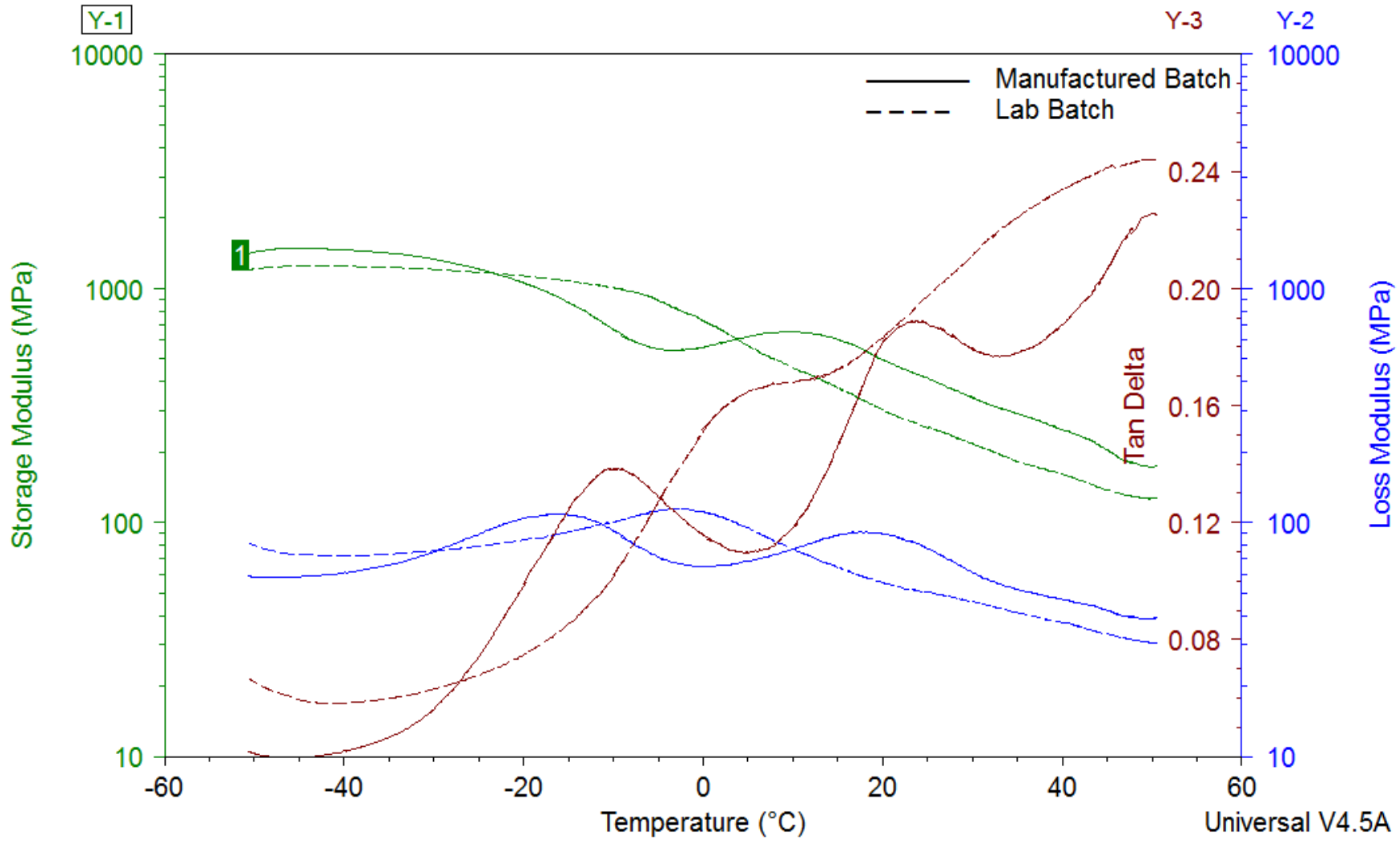
Polymer Blend - Immiscible Case

Acrylonitrile-Butadiene-Styrene (ABS) is a polymer blend



Using Glass Transition to Evaluate Blending

Insufficient blending on the manufactured batch results in two transitions



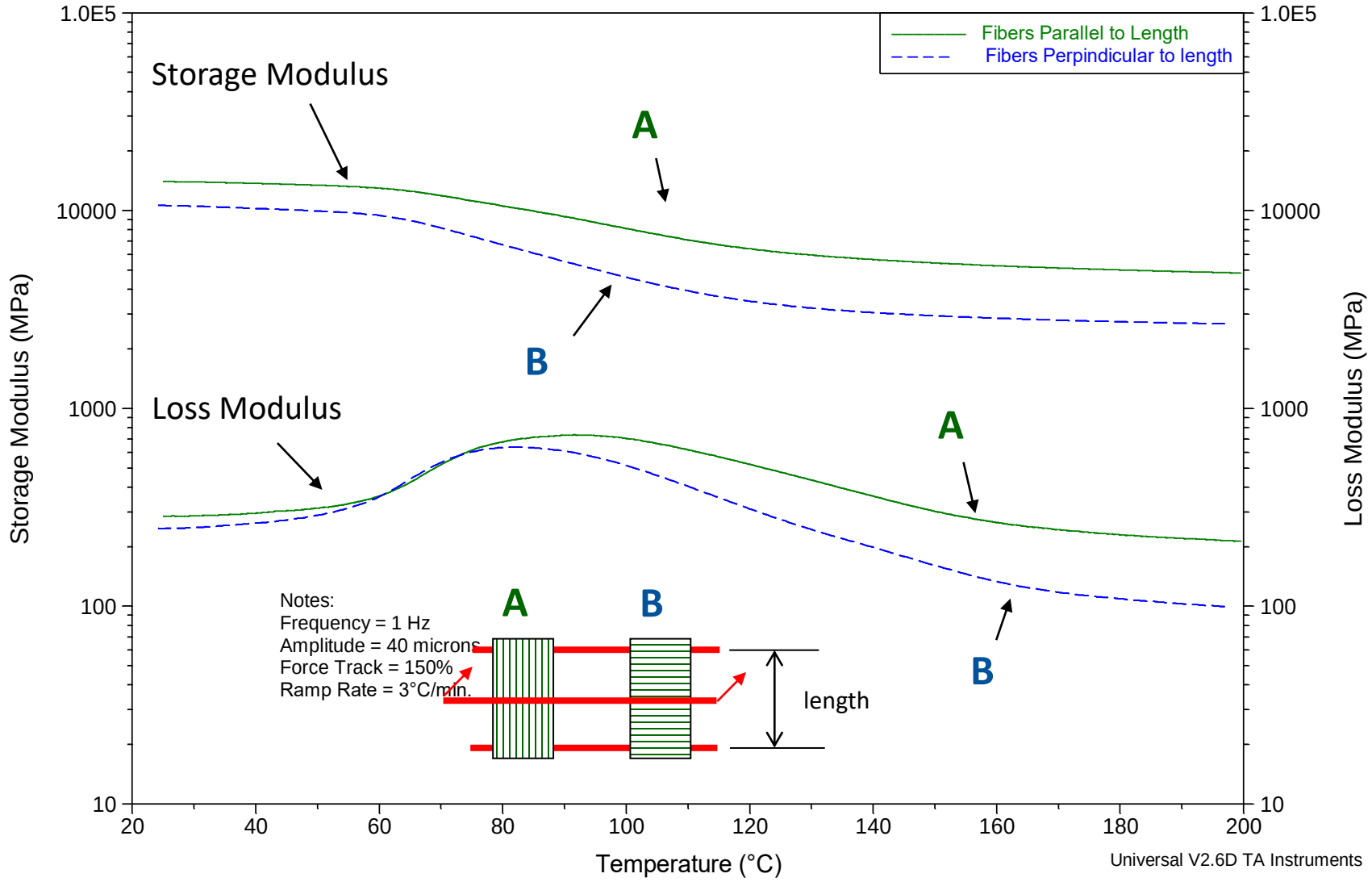
Anisotropic Materials

- Anisotropic materials have different properties in different directions.
  - fibers, wood, fiber-filled composites
  - oriented amorphous polymers, injection molded specimens
  - crystalline polymers in which the crystalline phase is not randomly oriented

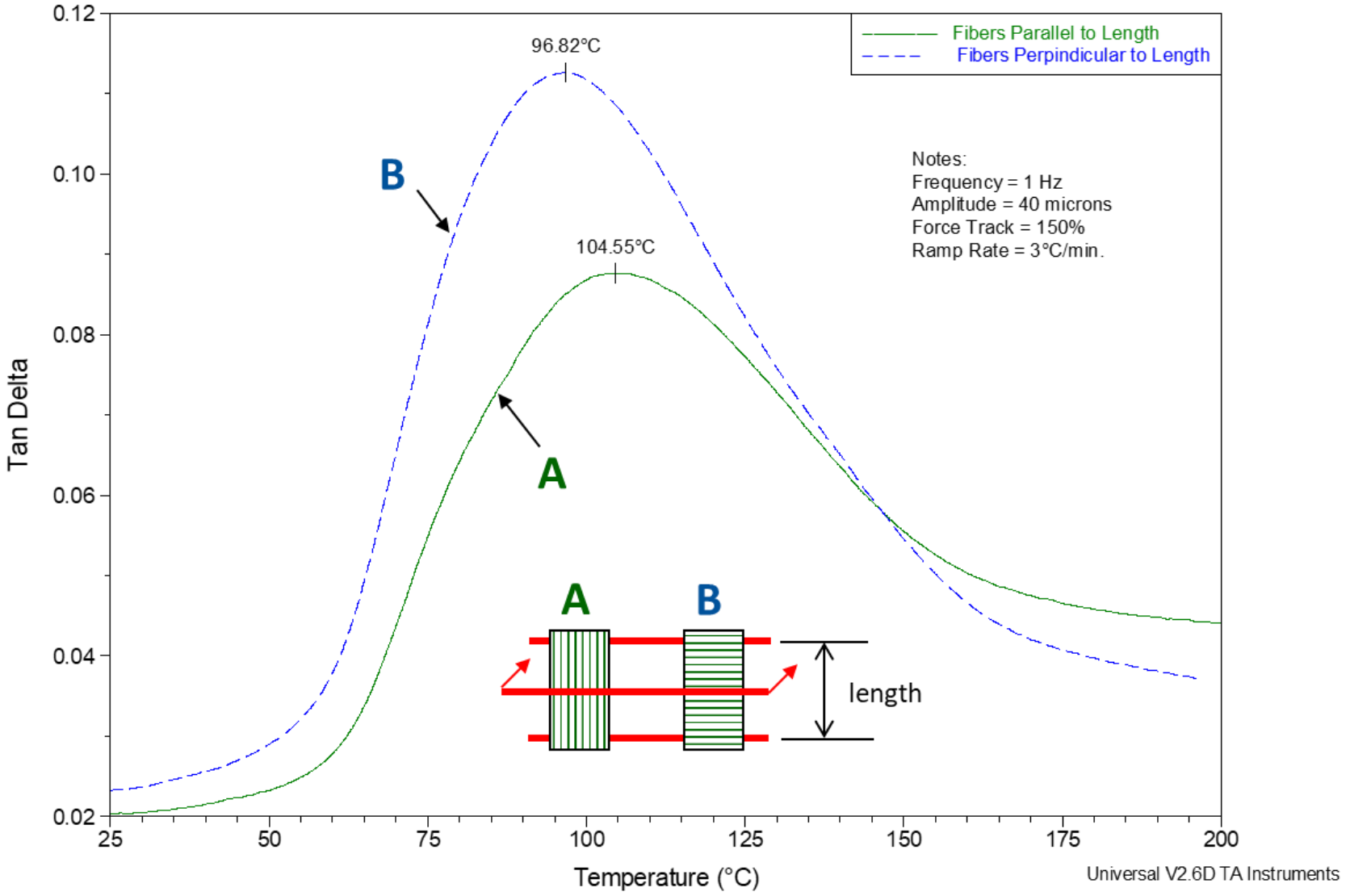
- Show different moduli at different measurement direction

Nielsen, Lawrence E., Mechanical Properties of Polymers and Composites, Marcel Dekker, Inc., New York, 1974, pp. 39-40.

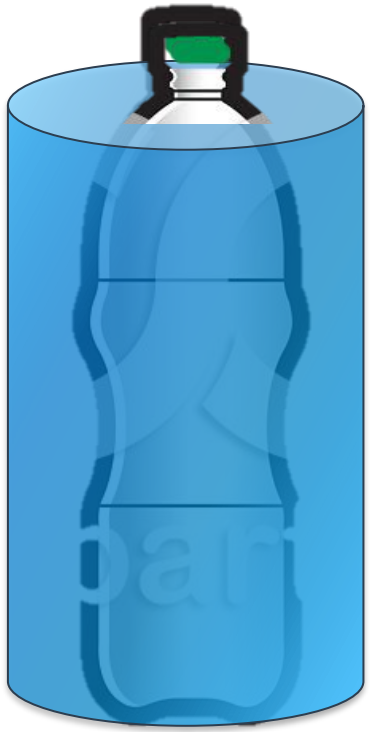
DMA of Polyester/Glass Fiber Reinforced Composite

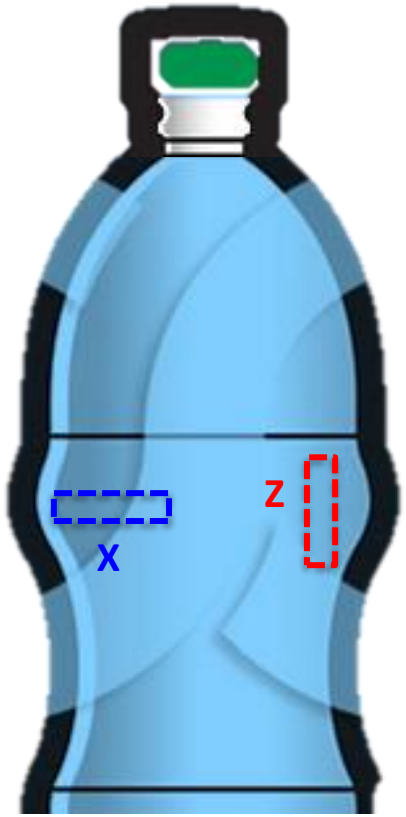


DMA of Polyester/Glass Fiber Reinforced Composite



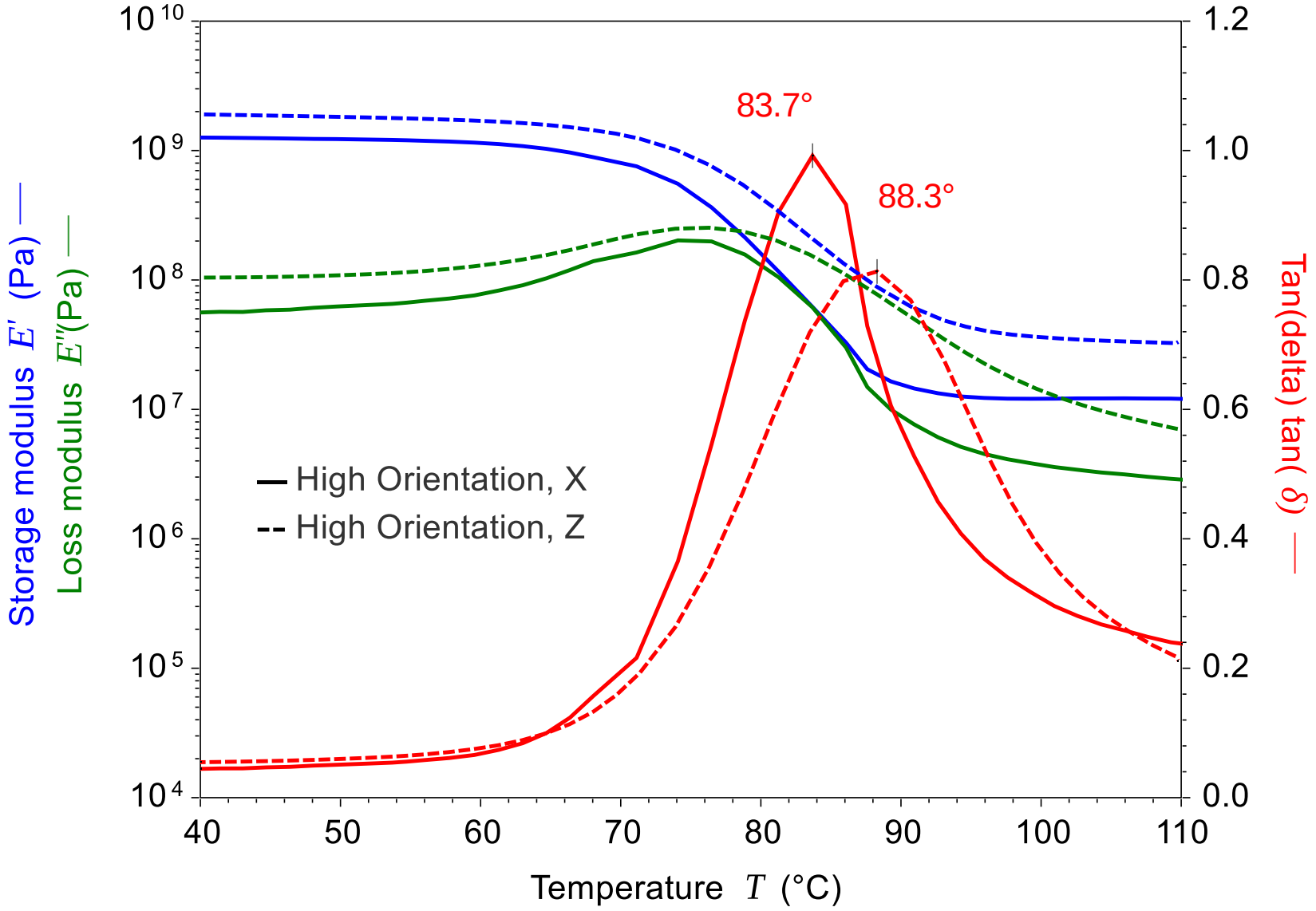
Oriented Polymer: Shrink Wrap

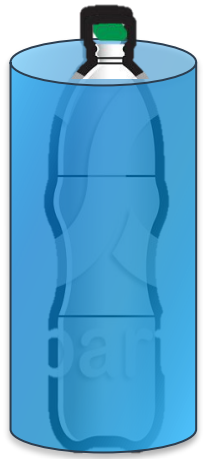




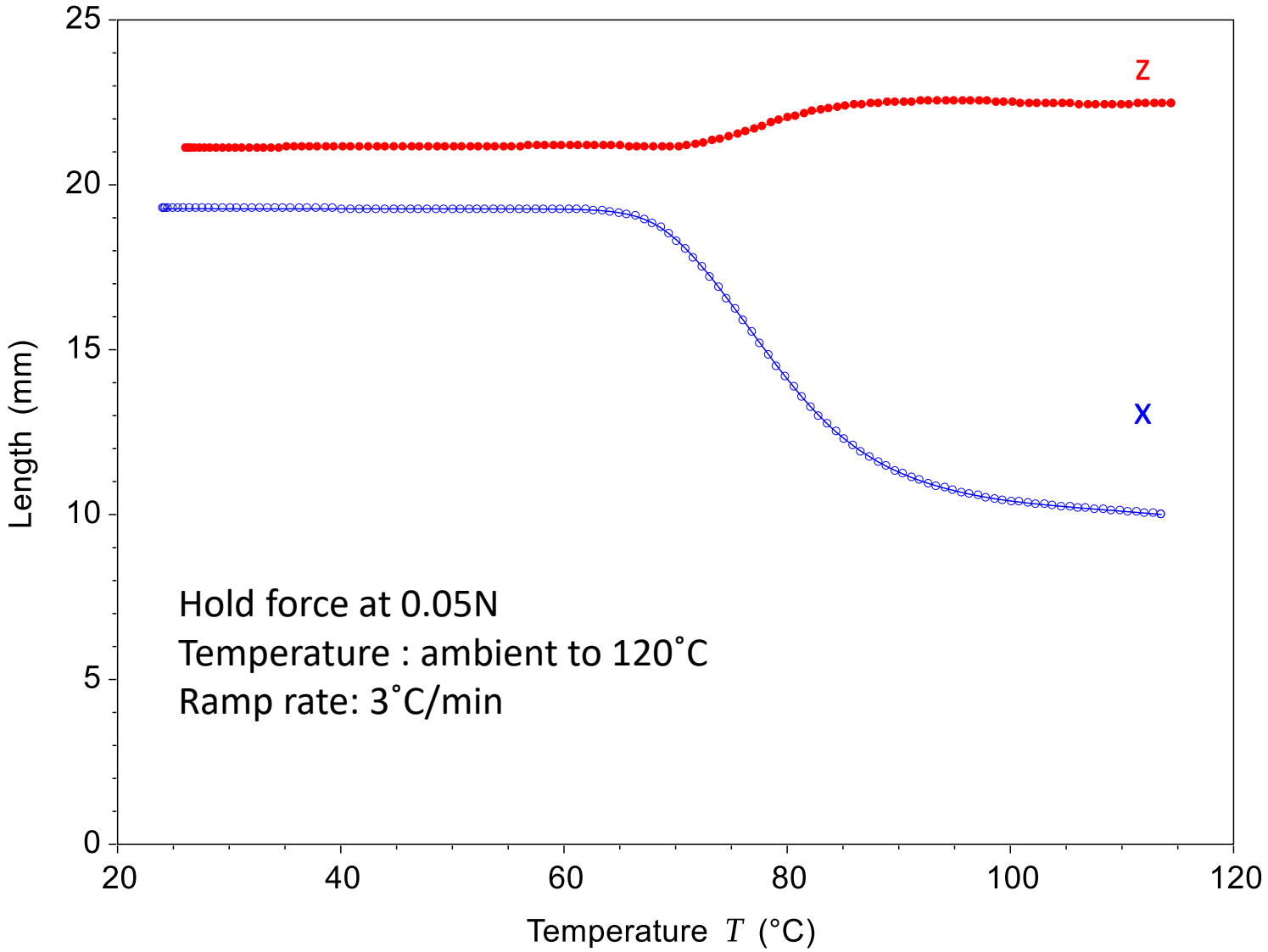
- "Shrink Wrap" is widely used in packaging to conveniently label irregularly shaped containers
- When this kind of biaxially oriented film is heated, it turns to shrink and returns to lower energy random-coil state.
- PET-based Label
  - Z-direction
  - X-direction
- RSA G2: 5˚ C per minute, 1 Hz

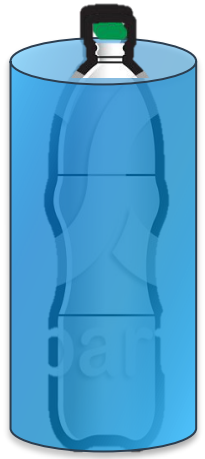
PET Film Measured at X and Z Direction



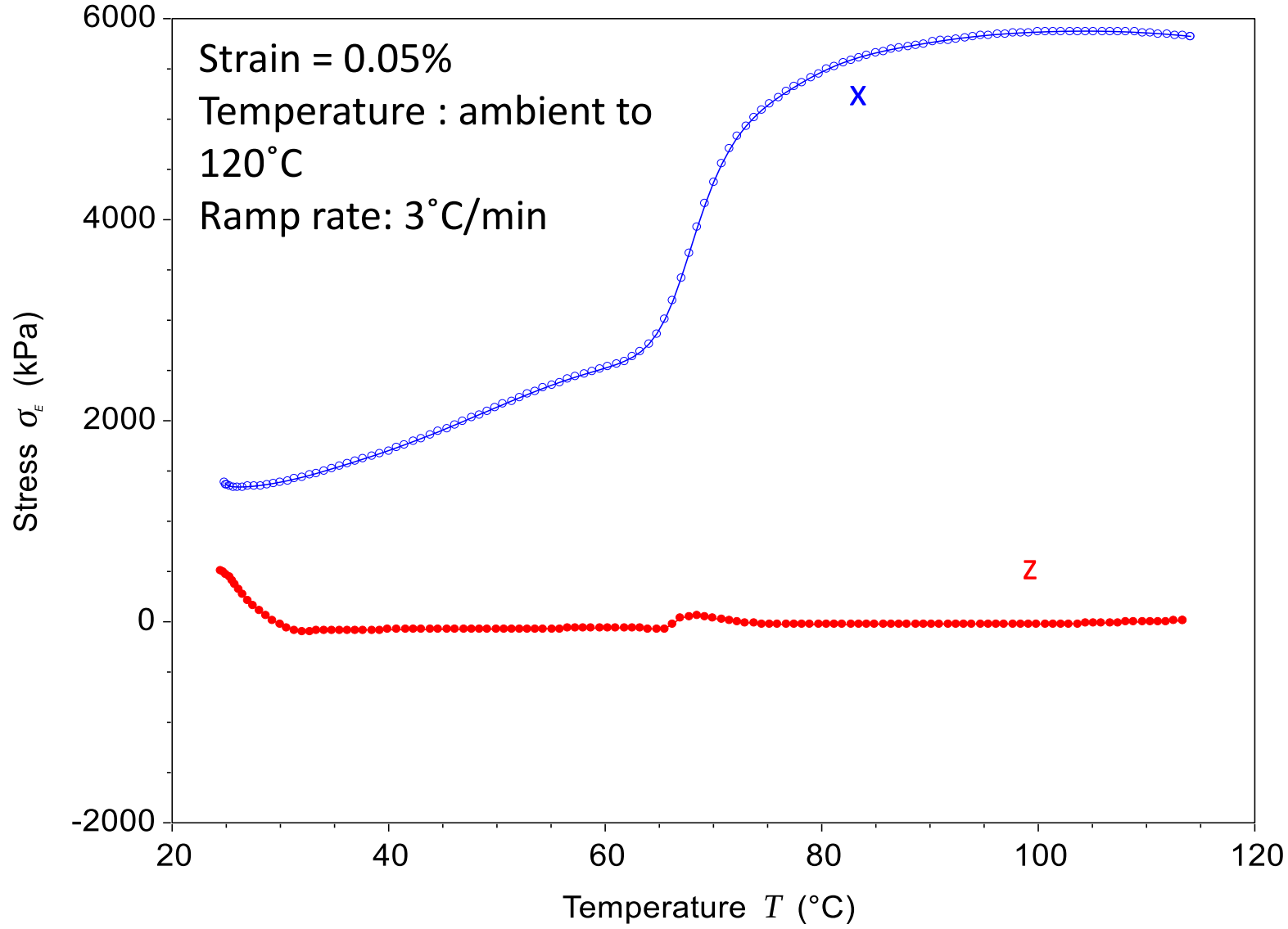


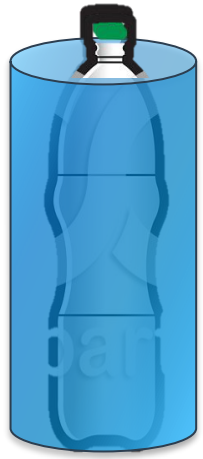
Iso-force Temp Ramp: measure shrinkage





Iso-strain Temp Ramp: measure shrinking force





Effect of filler on modulus and glass transition

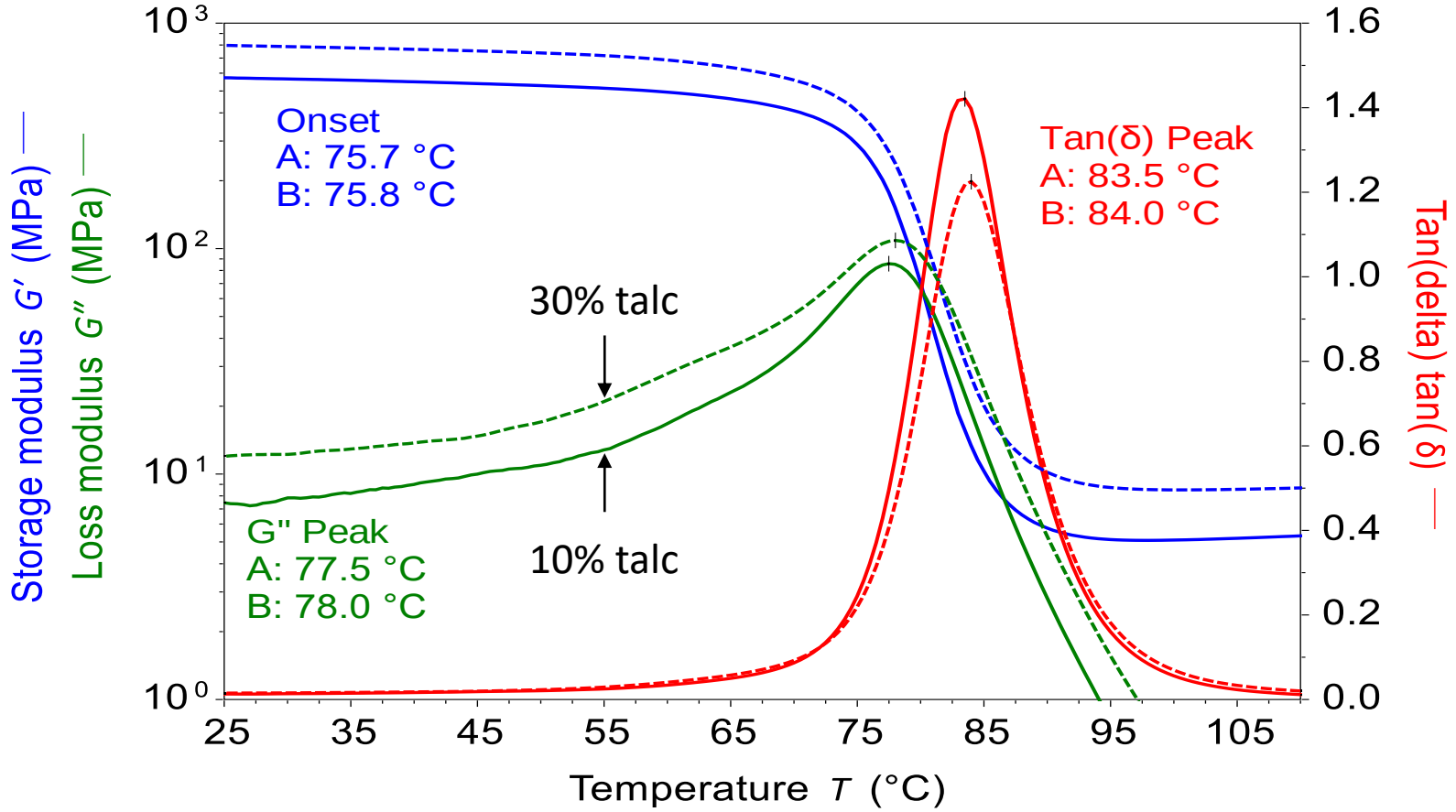
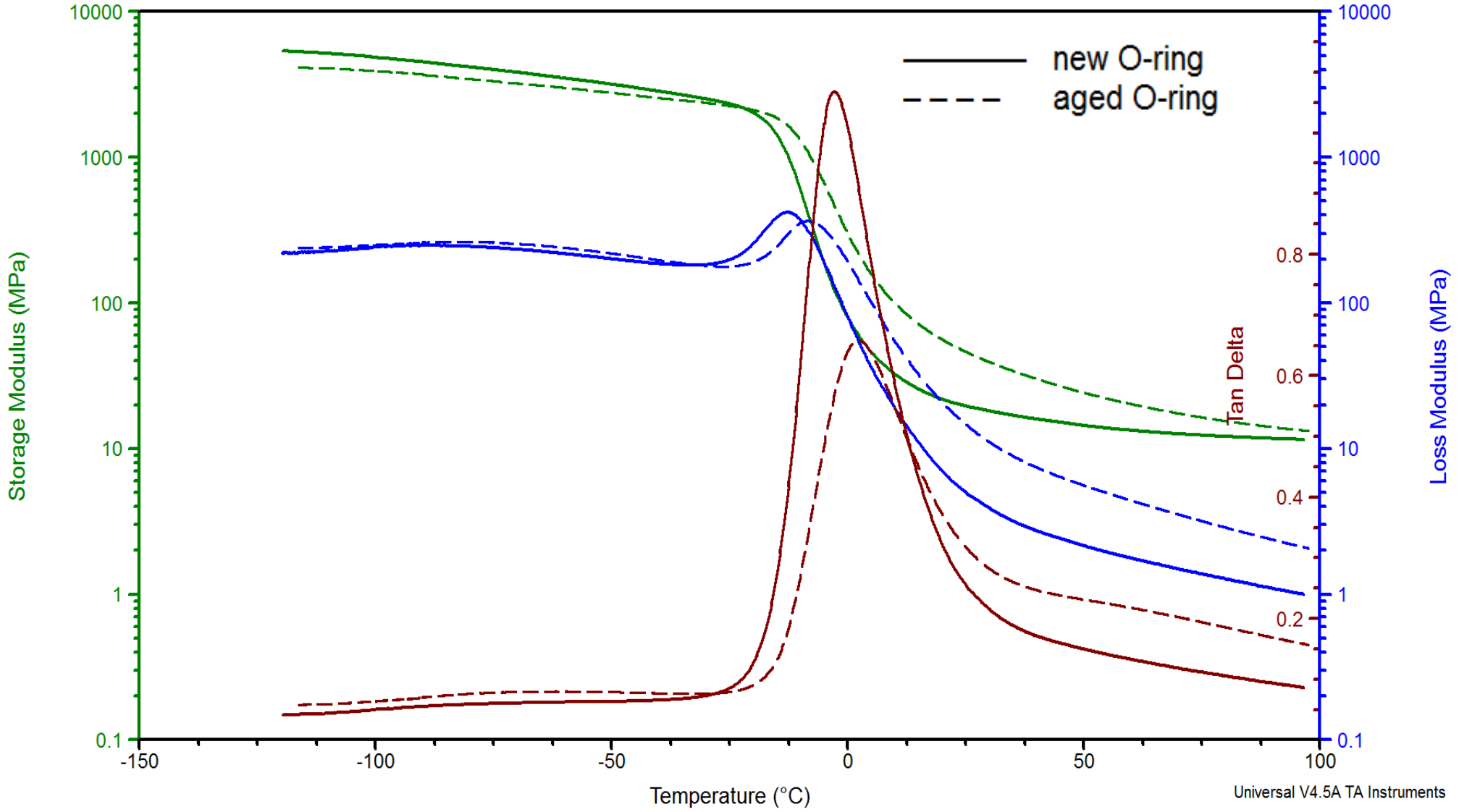


Figure 6: An epoxy sample with 10% (A) and 30 % (B) talc filler. The glass transitions are nearly identical on all signals.

TA Applications note: RH100 Measurement of Glass Transition Temperatures by Dynamic Mechanical Analysis and Rheology
https://www.tainstruments.com/pdf/literature/RH100.pdf

Effect of Aging on Elastomer O Rings



Effect of Plasticizer

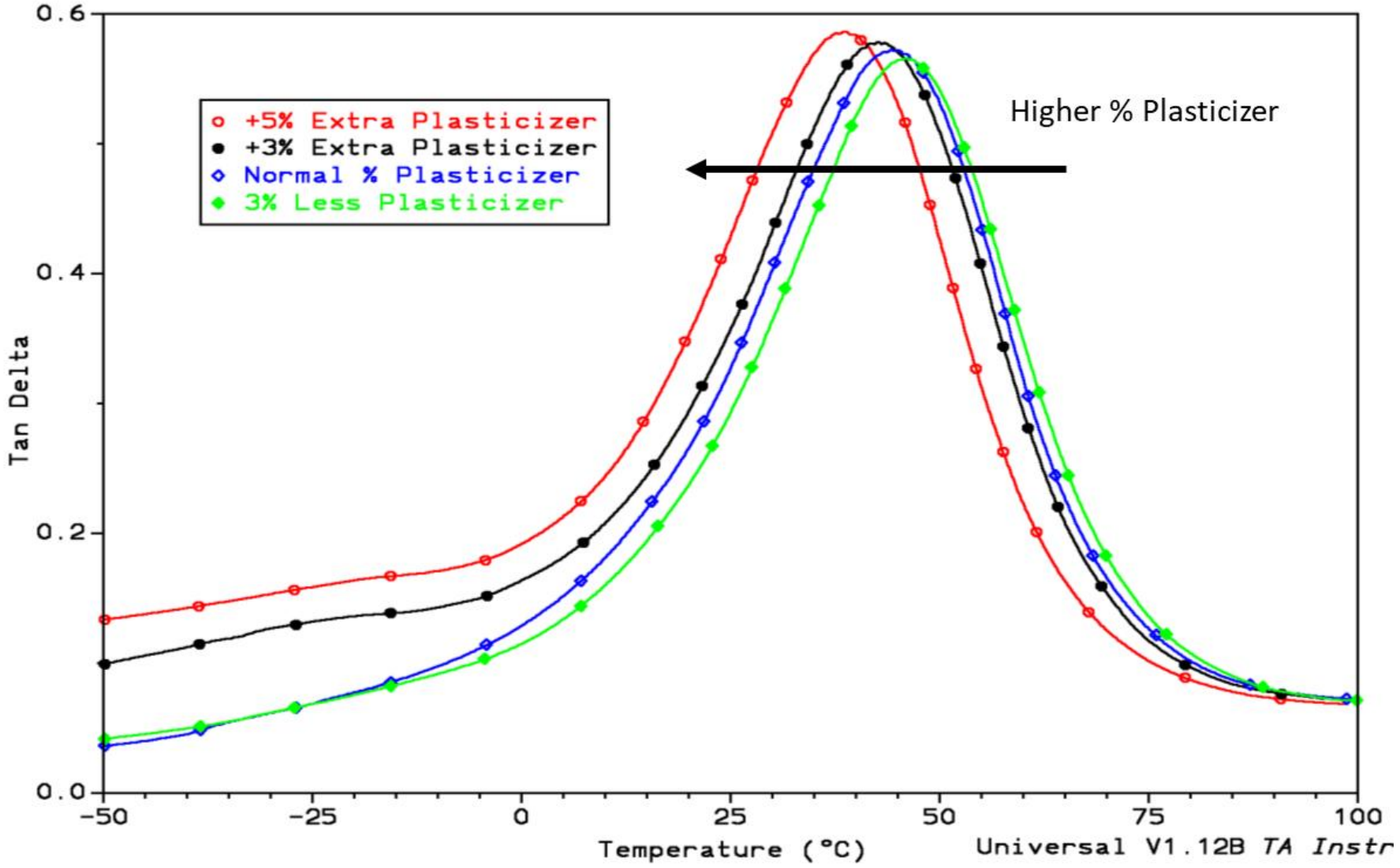
- *Plasticizers* are generally low molecular weight organic additives which are used to soften rigid polymers

- Typically added to a polymer for two reasons:
    1. To lower the T_g to make a rigid polymer become soft
    2. To make the polymer easier to process

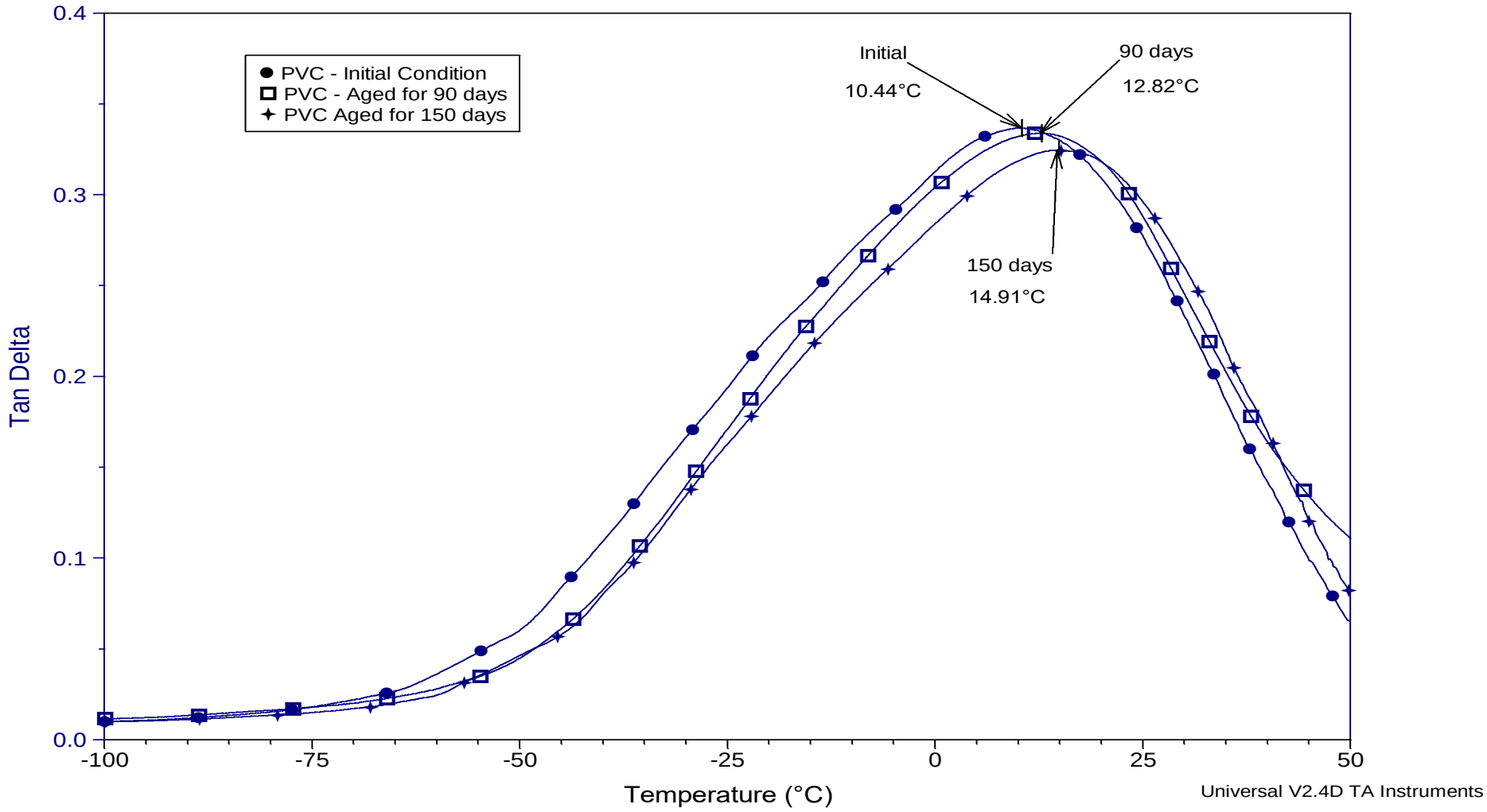
- *Plasticizers* act as lubricant in polymer chains

- Therefore plasticizers will have the effect of:
    1. Lowering the T_g and broadening tan δ peak
    2. Lowering the moduli (E' and E")

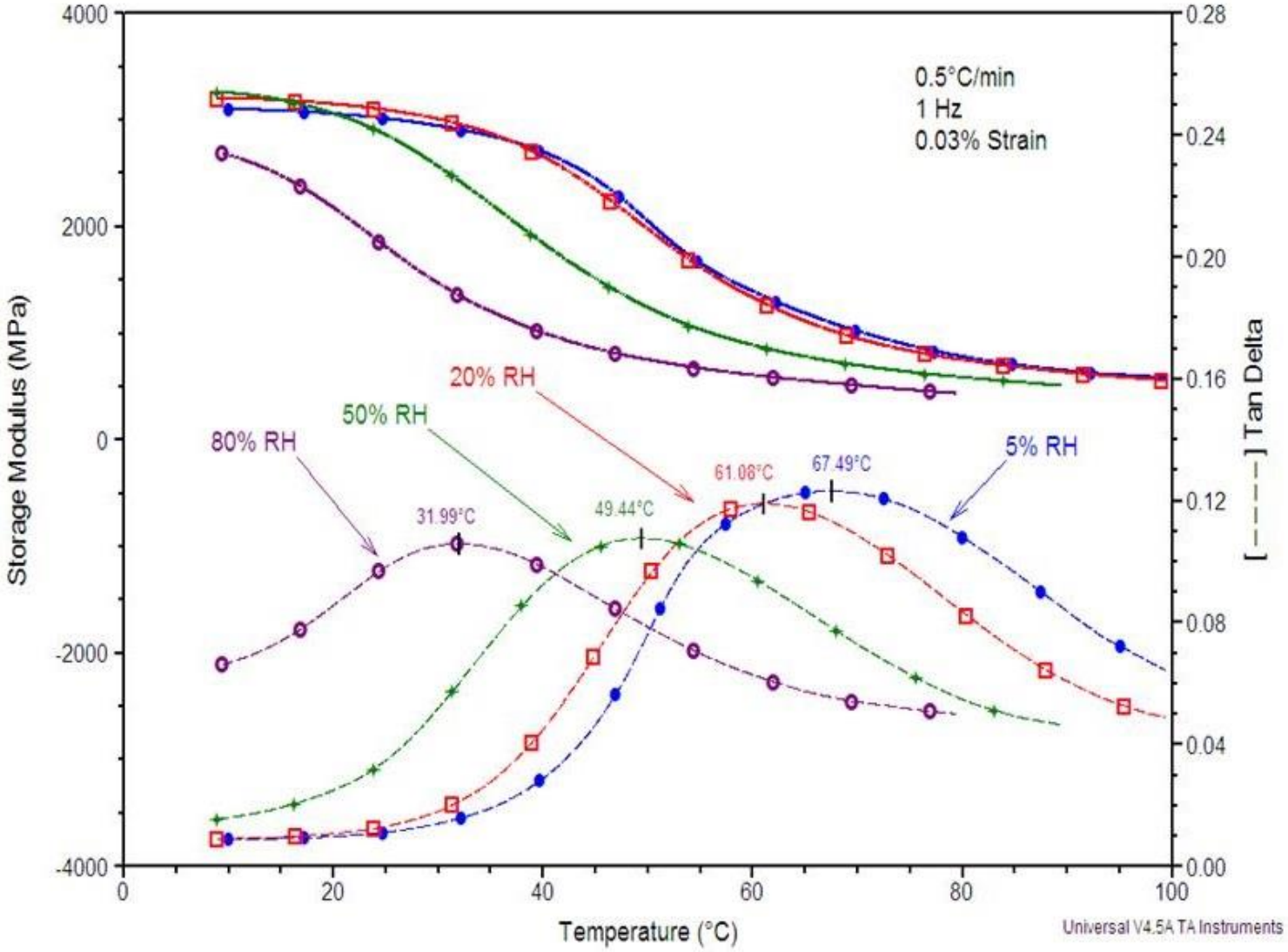
Effect of Plasticizer on Vinyl Flooring



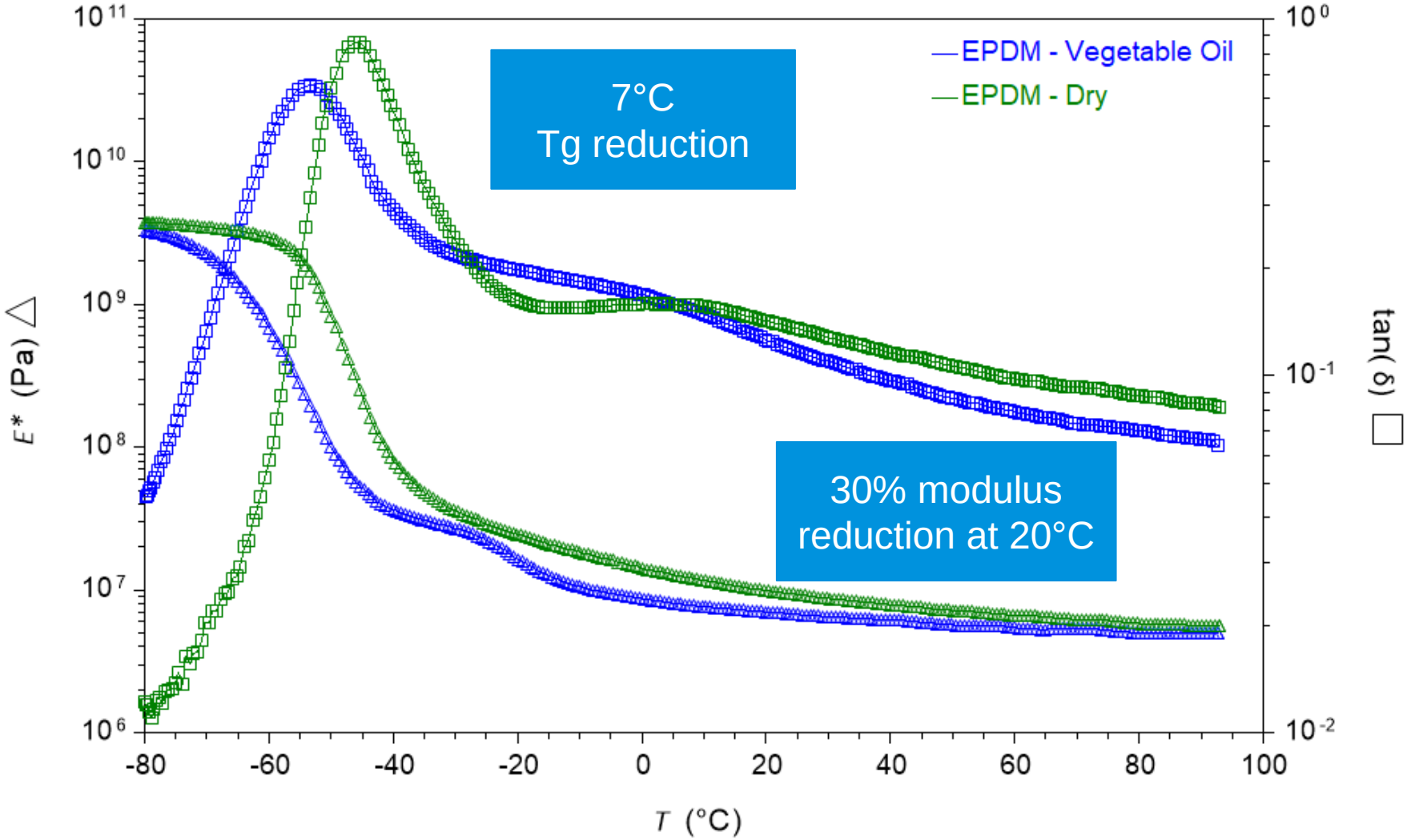
Effect of Aging on T_g of PVC Roofing Membrane



Humidity Influence: Nylon Film

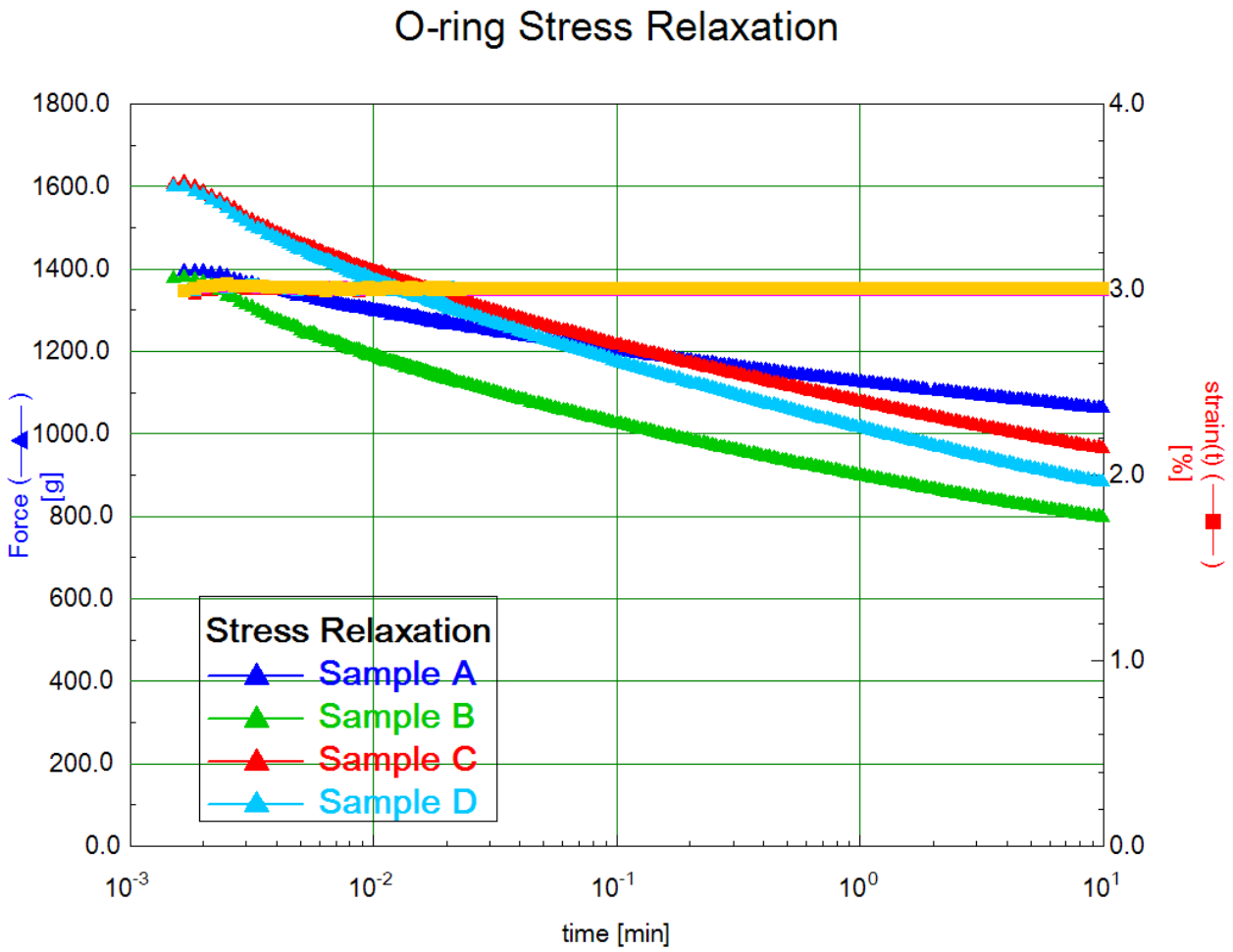


EPDM immersed in oil is subject to significant plasticizing



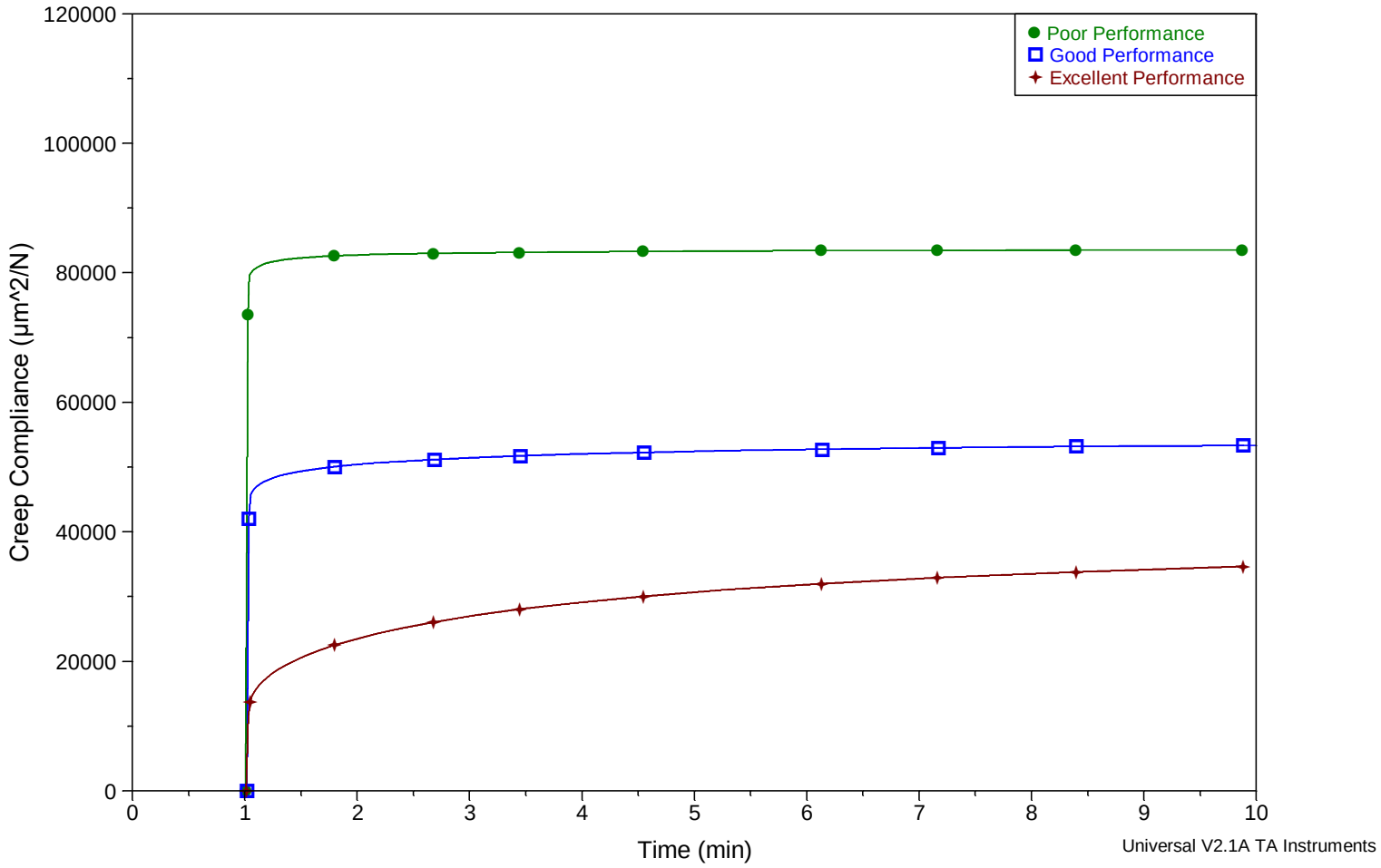
O-rings: Stress Relaxation

- Squeeze the O-ring to a certain strain. Hold it constant, then measure how long it takes for the force to relax

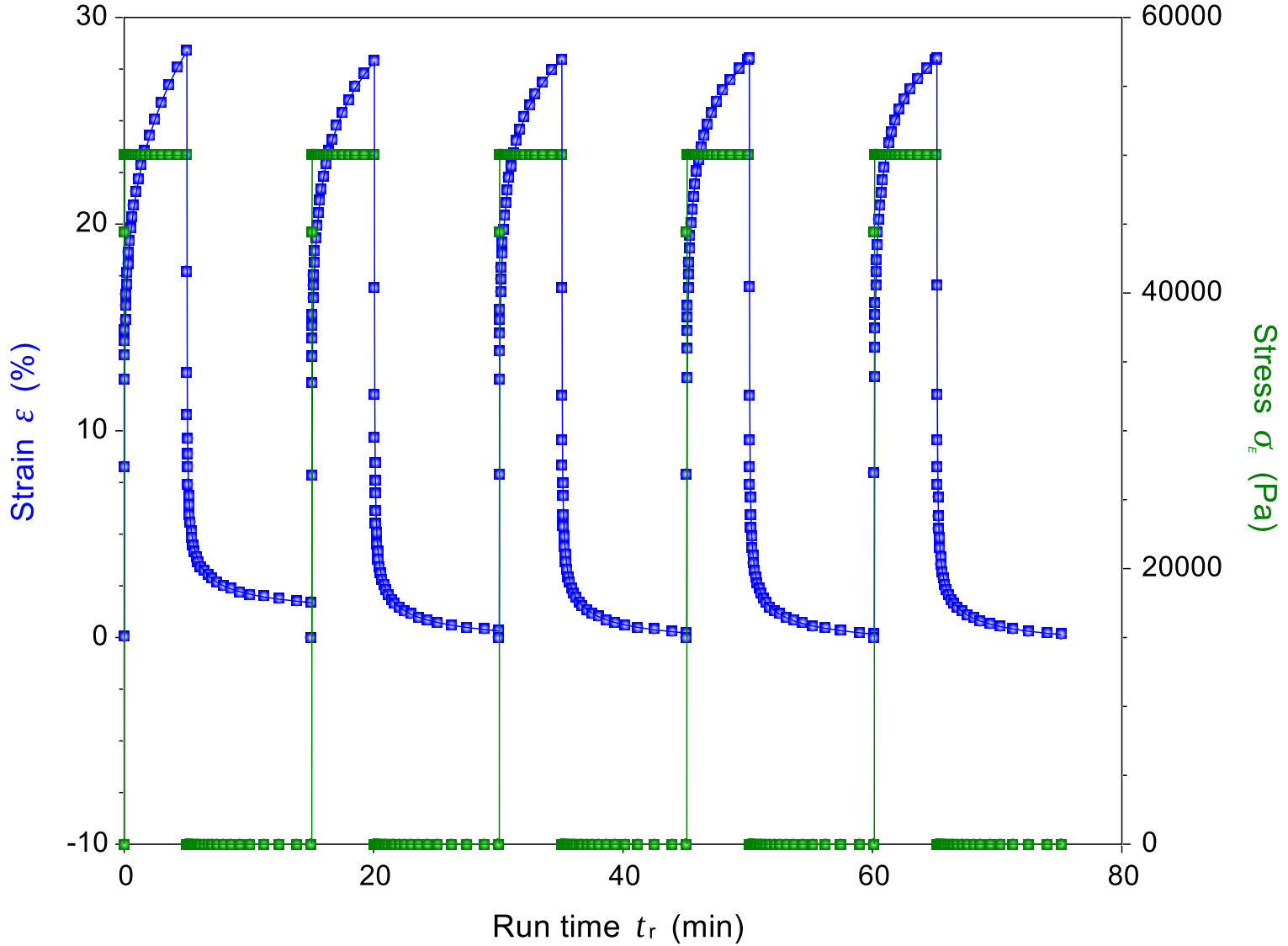


Creep Tests on Packaging Bags

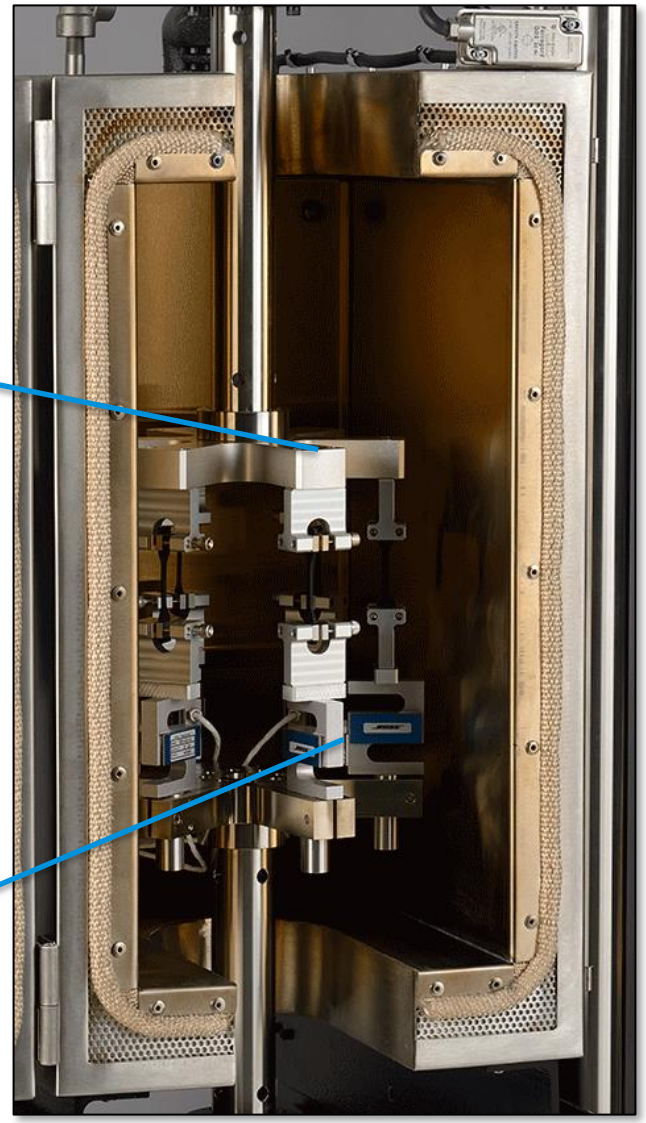
- Apply a load force, then measure how much the bag is stretched



Memory Foam: Multi-Step Creep Recovery



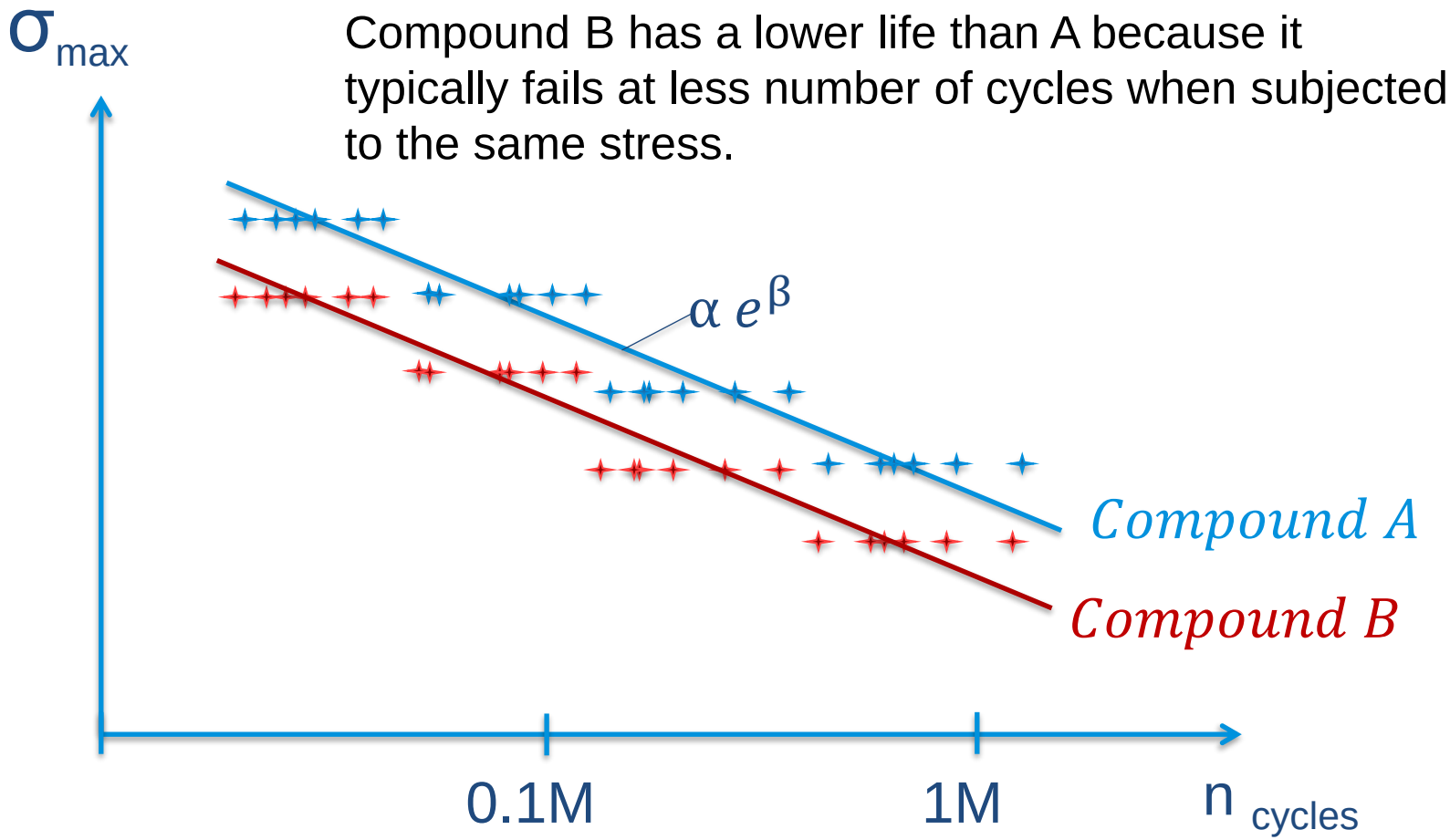
Multi-Specimen Tension Fatigue
BR Rubber (Tire Rubber) Example



6-Specimen Fixture with 6 Force Sensors

Multi-Specimen Tension Fatigue
BR Rubber (Tire Rubber) Example

- Many SN points obtained in relatively short time
  - 4 test series (24 failures) in ~1 month
- Data is fit to exponential equation (line on plot)
- Data and exponential fit is used to compare materials



Troubleshooting Experimental Issues

Factors that influence measurement results

Primary measurements
- Force
- Displacement
- Temperature

Check list
- Instrument calibrations
- Thermocouple position

Geometry
- Clamp
- Sample size
- Sample loading
- Sample integrity

Check list
- Clamp calibrations
- Proper loading torque/force
- Sample uniformity and thermal stability
- Bad contact or sample bending and twisting

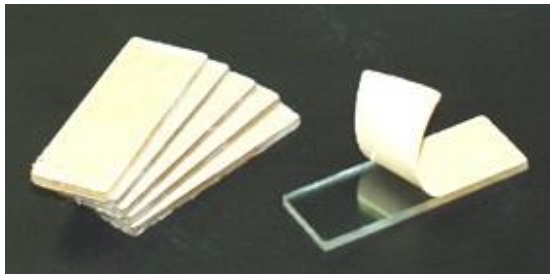
Test Parameters

Check list
- Loading force
- Proper force track
- Equilibration time
- Heating rates
- Amplitude
- Frequency

Instrument and clamp

- Perform clamp calibration every time when attaching a new clamp on the instrument
  - http://www.youtube.com/user/TATechTips

- Perform confidence verification check using polycarbonate provided by TA Instruments (see next slide)



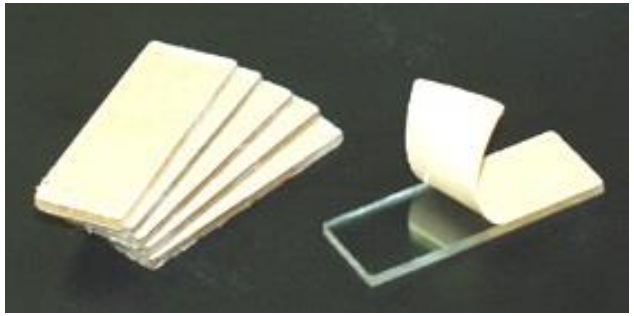
p/n: 982165.903

DMA confidence check - polycarbonate

- Load Polycarbonate (L ≈ 17.5, w ≈ 12.85, t ≈ 1.6mm)
- Use Single Cantilever Clamp
  - 20-30 micrometer amplitude
  - 1 Hz frequency
- Storage Modulus at Room Temperature
  E' = 2.35 GPa (2350 MPa) +/- 5%
- Tan Delta at Room Temperature
  Tan δ < 0.01

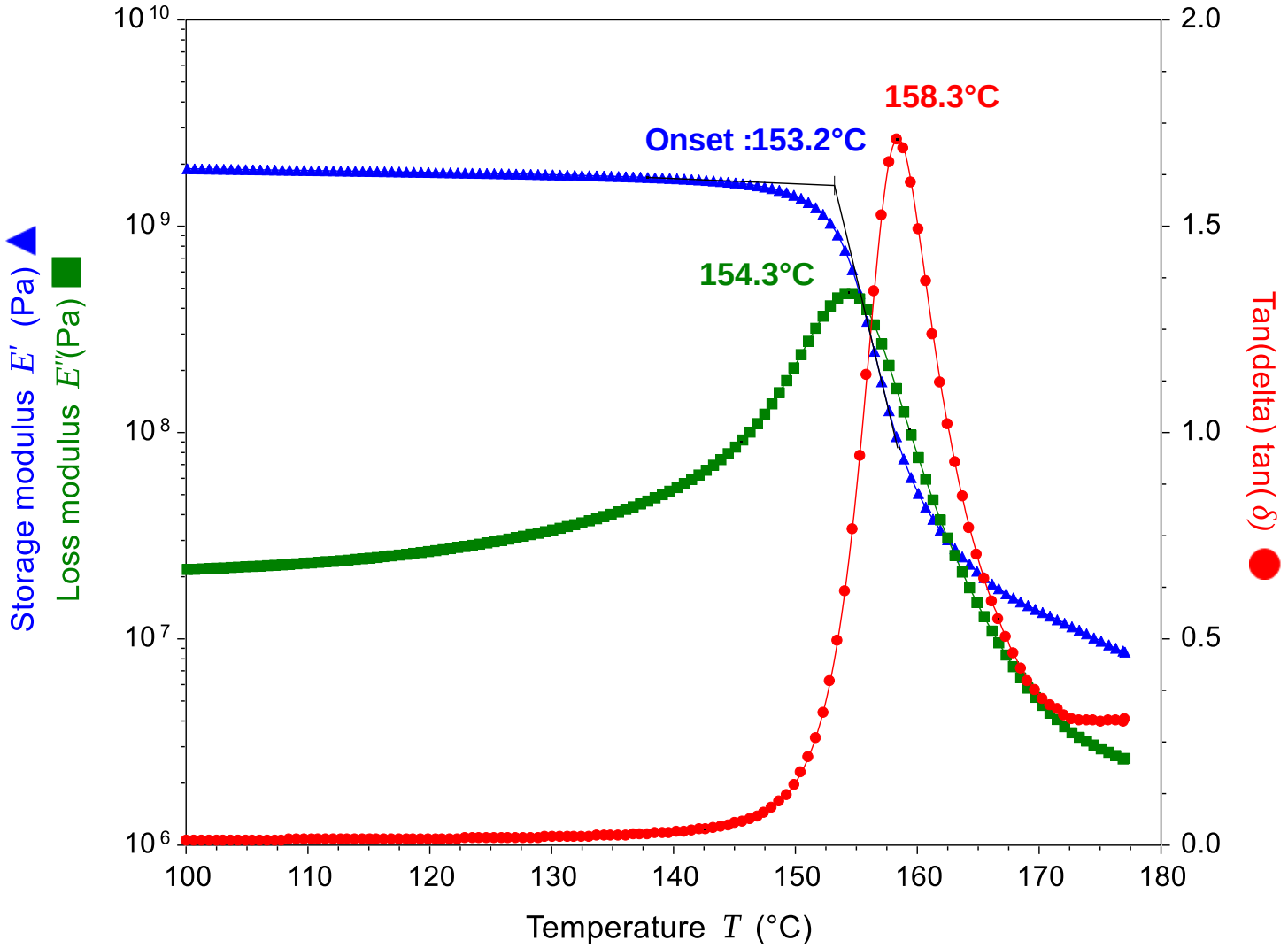
- Transition Temperature
  Tan δ peak between 155-160°C @ 1Hz, 3-5°C/min
  E" peak will be about 5°C lower

p/n: 982165.903

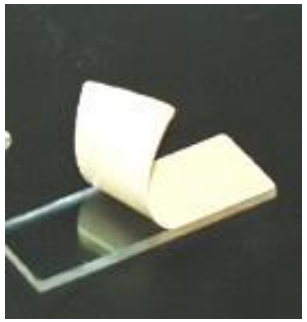


Temp ramp on polycarbonate

- Available from TA for Instrument verification



PC sample



p/n: 982165.903

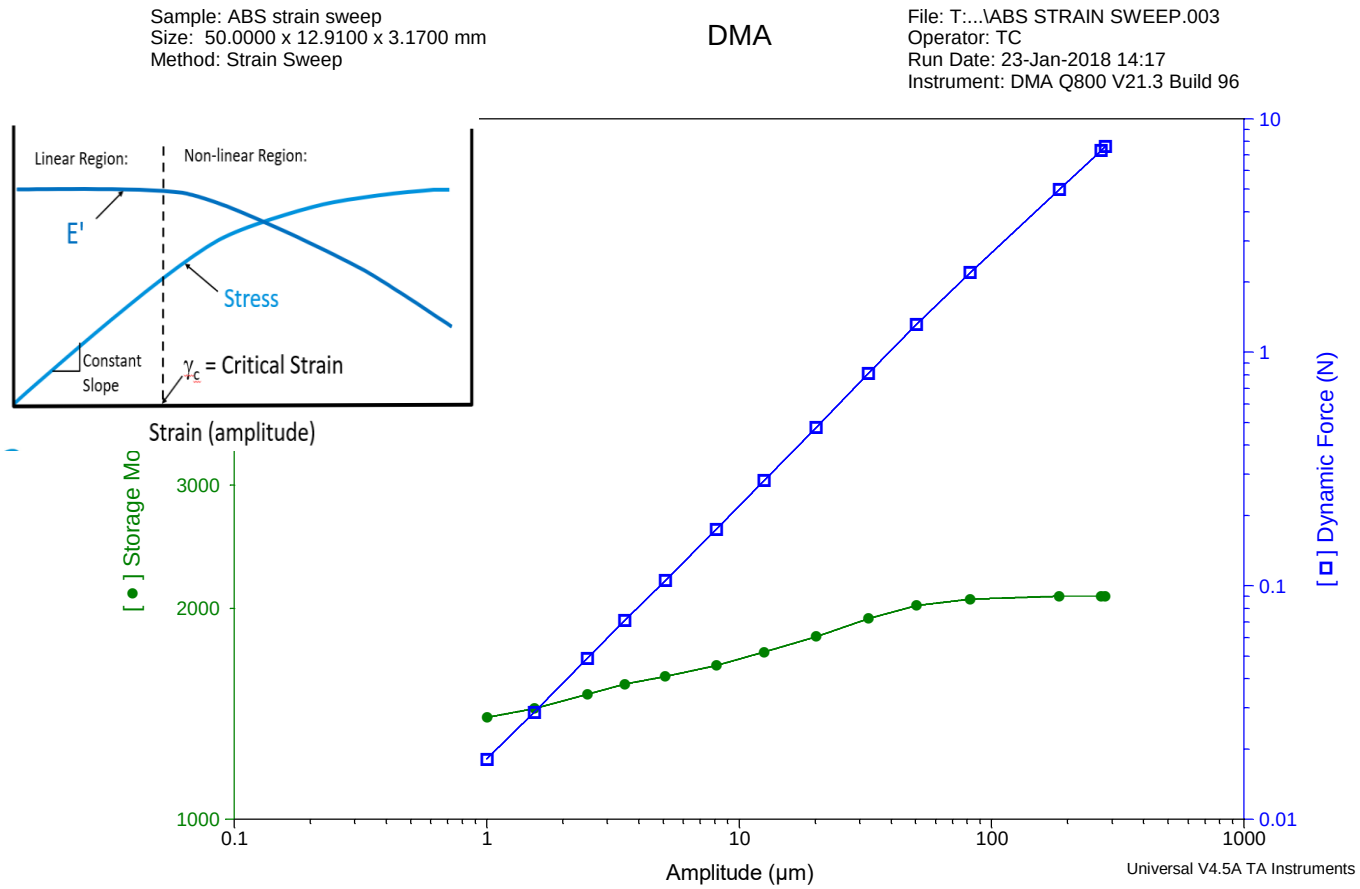
Clamp:
single cantilever
Temperature:
ambient to 180°C
Heating rate: 3°C/min
Frequency: 1 Hz
Amplitude: 20 µm

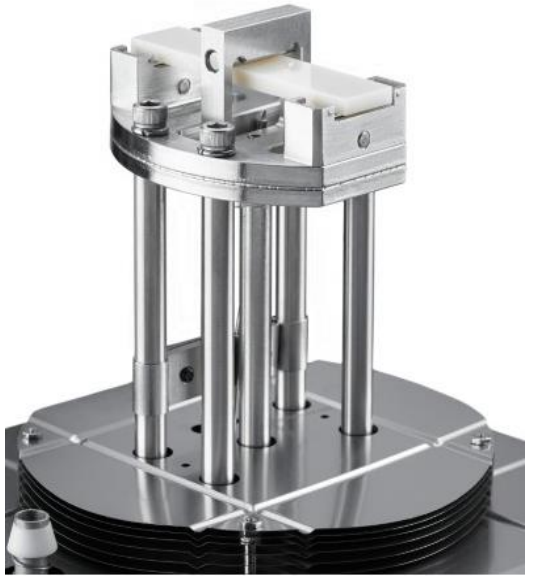
Sample preparation

- DMA measurement results are highly depending on the quality of sample preparation
- Sample flatness and uniformity have big influence to the accuracy of the test results

E’ Increase in a strain sweep

Why does E’ increase with increasing amplitude?
How to verify linear viscoelastic region?





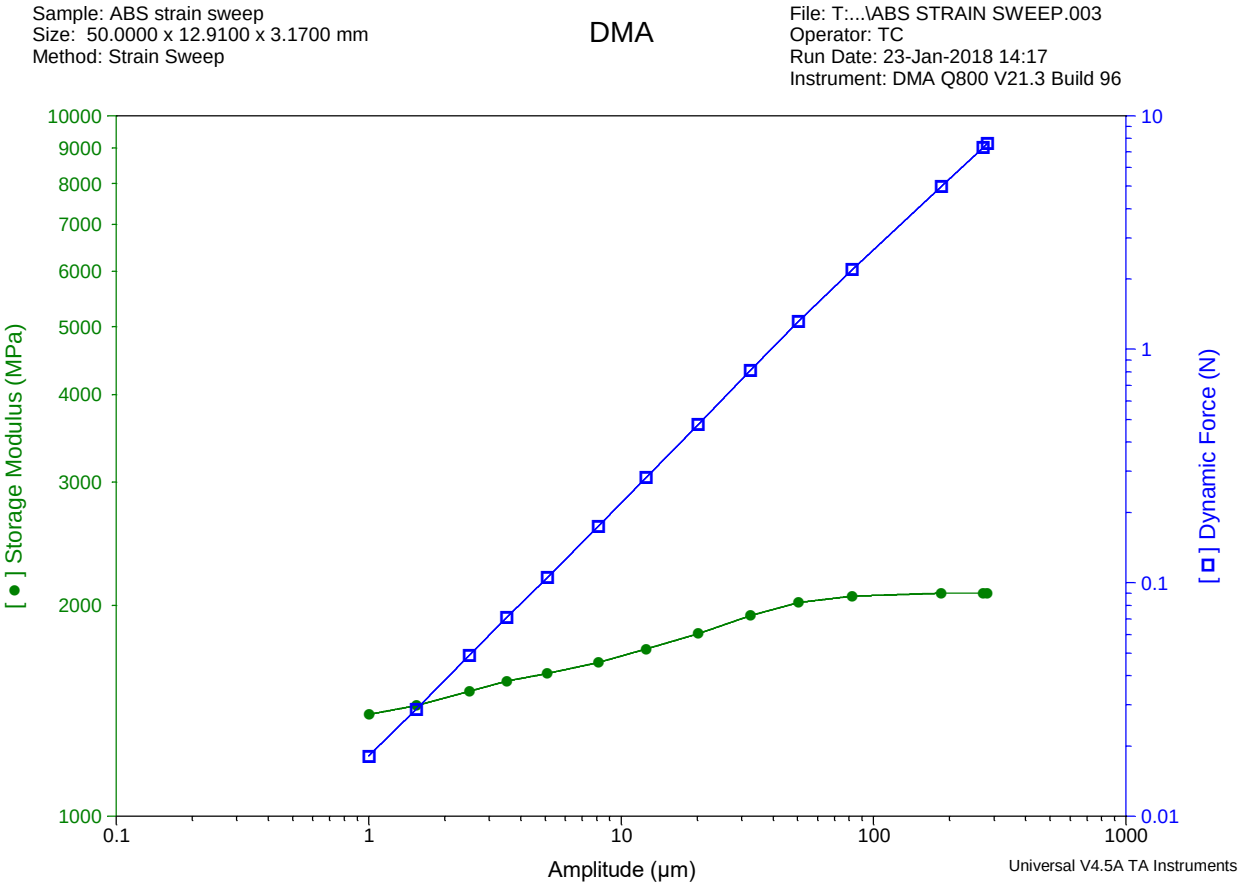
Instrument: Q800
Clamp: 3-p bending
Temperature: ambient
Amplitude: 1-500 µm
Frequency: 1Hz

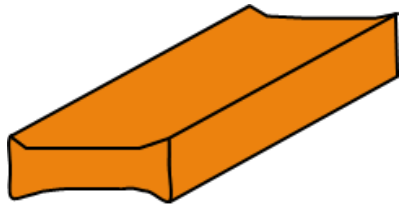
E’ Increase in a strain sweep

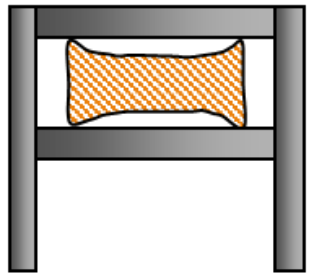
The sample is not flat and not in full contact with the clamp face.

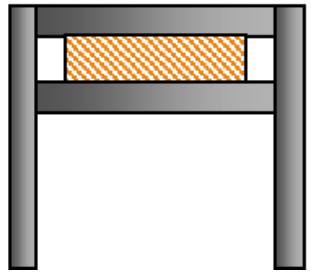
Solutions: (1) Prepare a flat sample

(2) Increase force track or increase static force



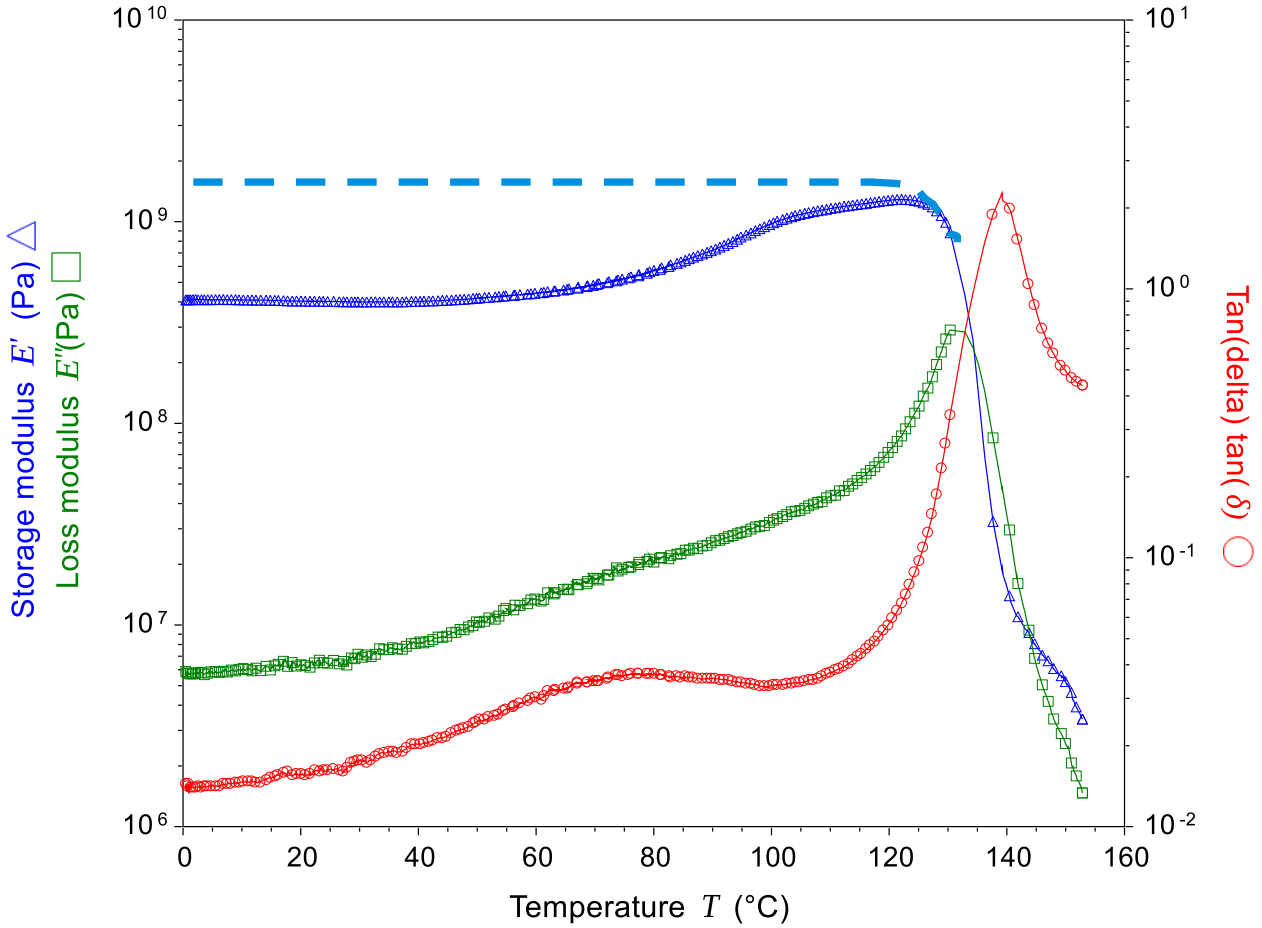






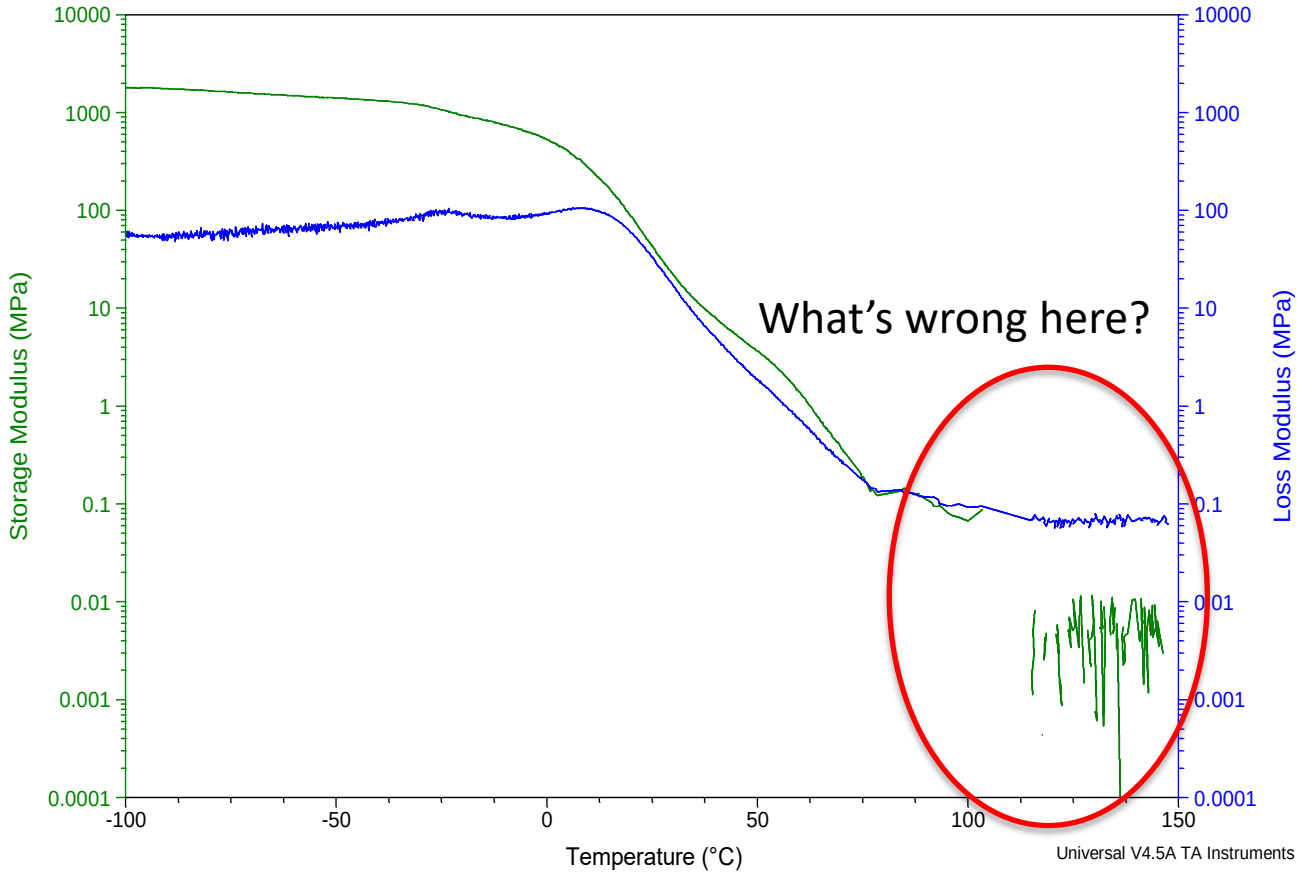
E’ increase in a temp ramp

- The E’ of a material should not increase with temperature unless it is crystalized or crosslinked



Noisy modulus after Tg

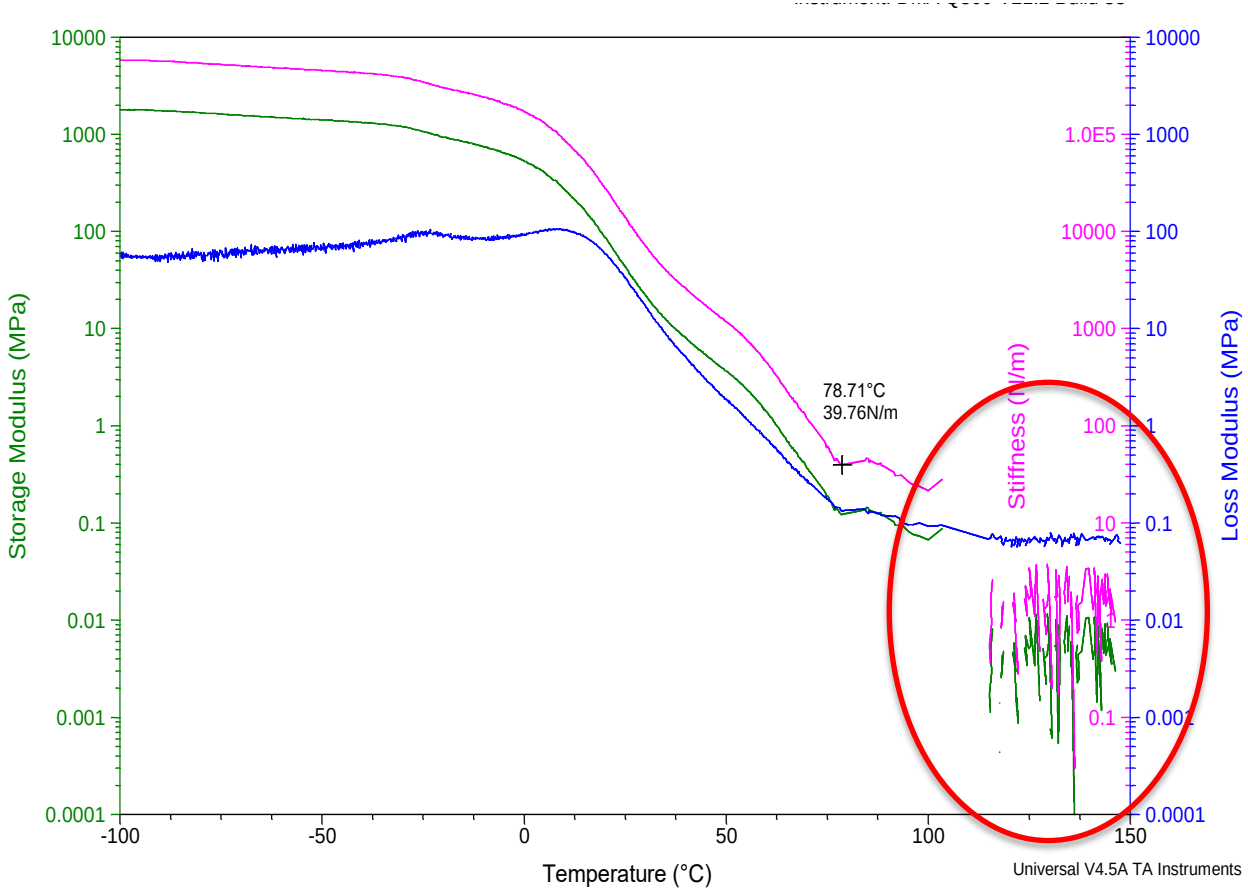
What is the problem with this data collected after T_g?



Instrument: Q800
Clamp: tension
Temperature:
-100°C to 150°C
Heating rate: 3°C/min
Frequency: 1Hz
Amplitude: 10 μm

Noisy modulus after Tg

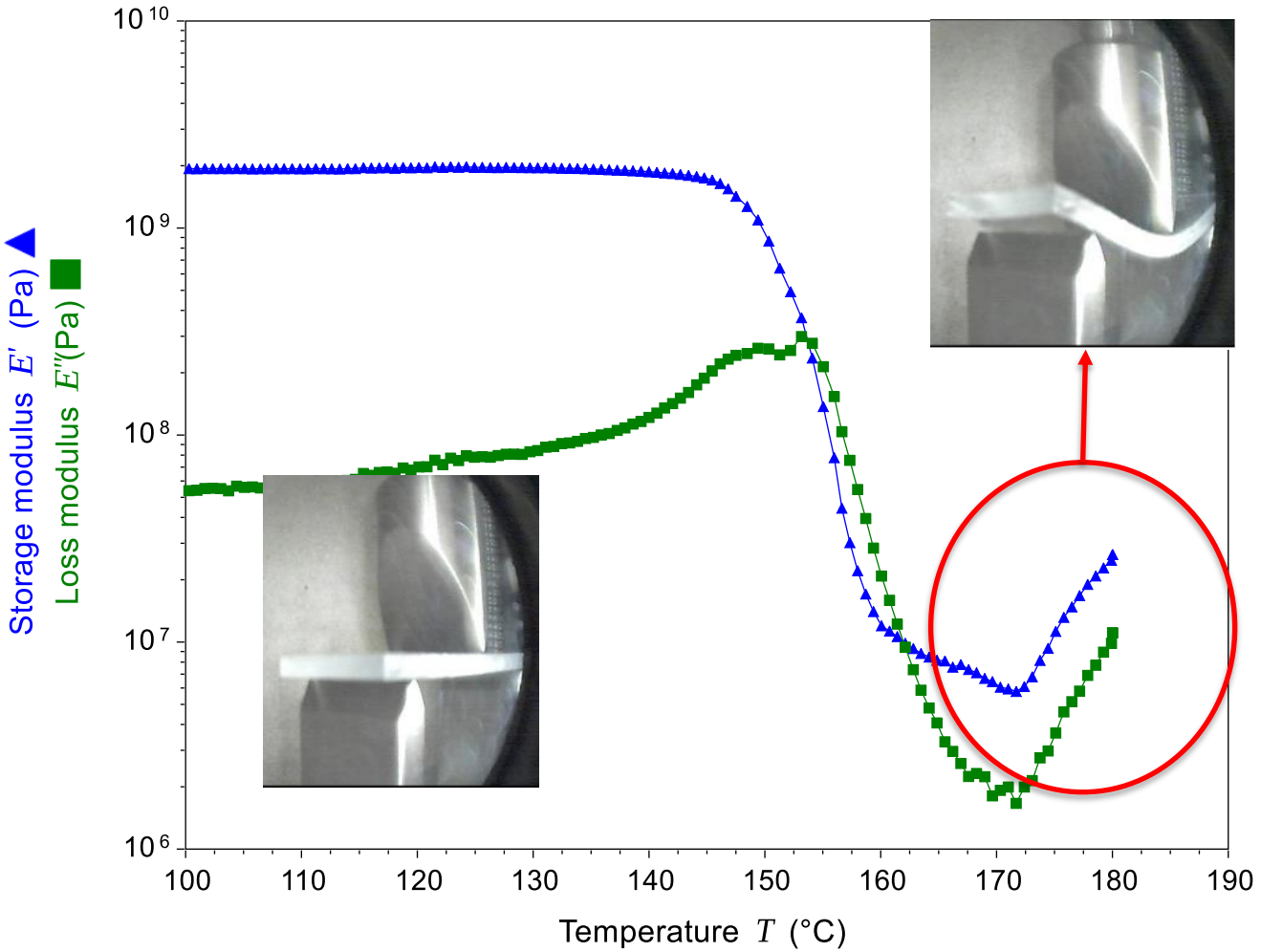
What is the problem with this data collected after T_g?



Instrument: Q800
Clamp: tension
Temperature:
-100°C to 150°C
Heating rate: 3°C/min
Frequency: 1Hz
Amplitude: 10 µm

Sample sagging

- Sample sagging after T_g
- Solution: use cantilever clamp instead of 3-p bending



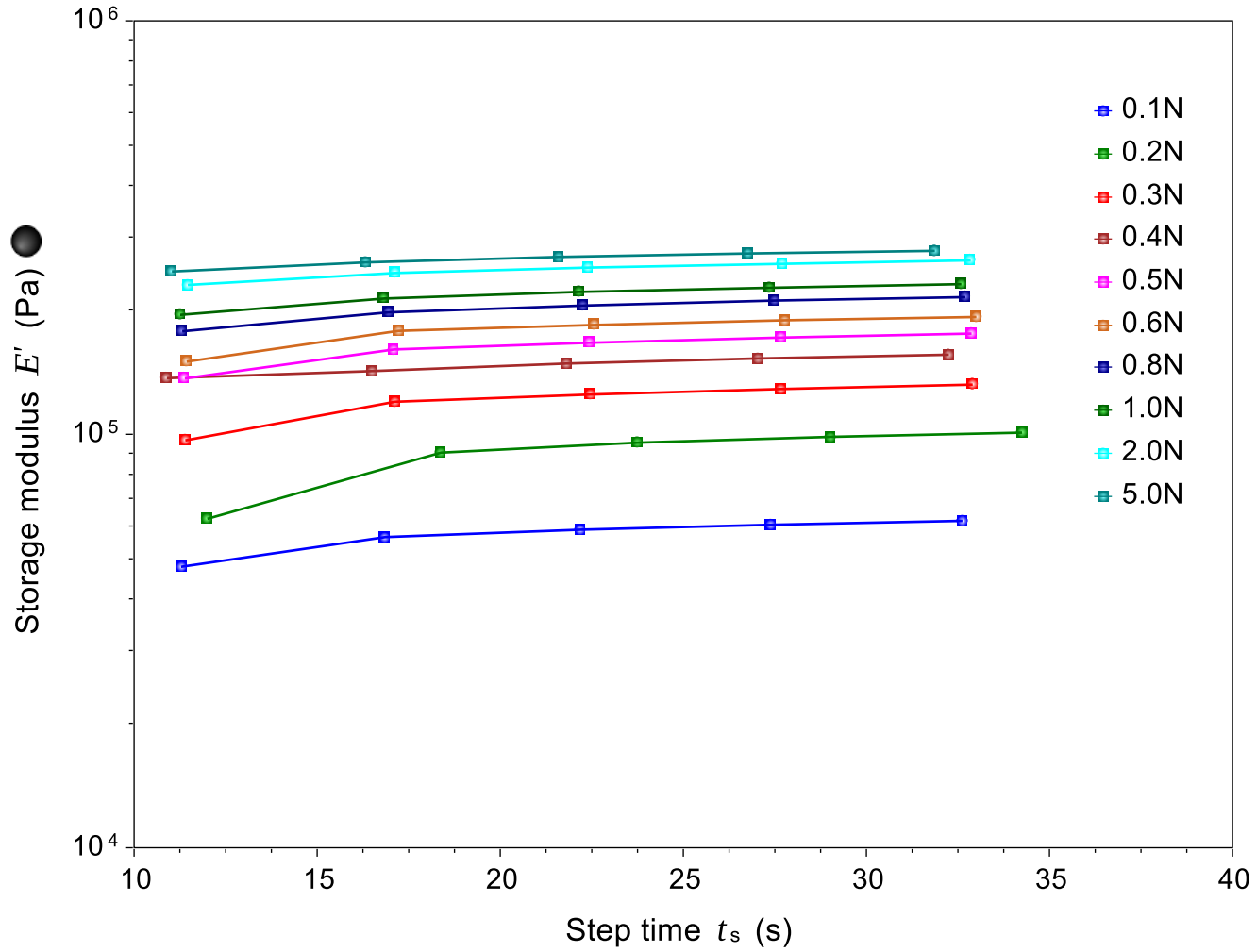
Instrument: RSA G2
Clamp: 3-p bending
Temperature:
50°C to 180°C
Heating rate: 3°C/min
Frequency: 1Hz
Amplitude: 10 μm

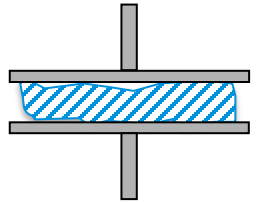
Compression testing on foam/rubber

Question: How to measure correct modulus?

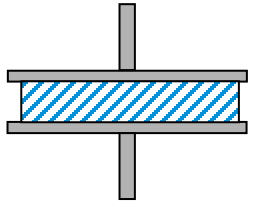
Answer: (1) Prepare regular sample

(2) Apply appropriate static force

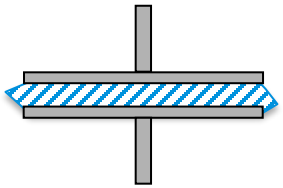




(1) Poor contact



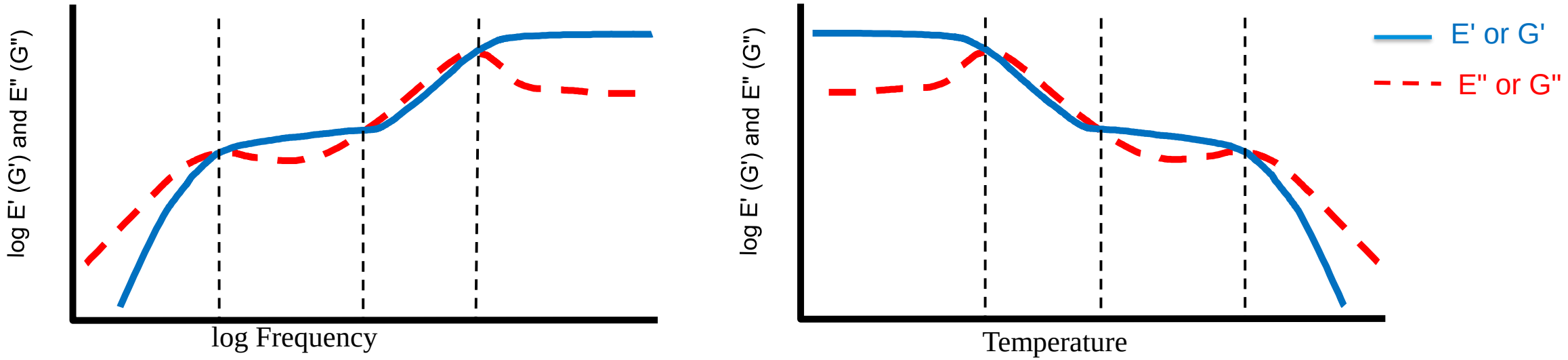
(2) Appropriate



(3) Over squeezed

Time-Temperature Superposition (TTS)

Time and temperature relationship



- Some materials show a time dependence that is proportional to the temperature dependence
  - Decreasing temperature has the same effect on viscoelastic properties as increasing the frequency, and vice versa

- For such materials, changes in temperature can be used to “re-scale” time, and predict behavior over time scales not easily measured

Dealy, J., Plazek, D., *Time-Temperature Superposition – A Users Guide*, Rheology Bulletin, **78**(20) 16 (2009)

Merits of TTS

- TTS can be used to extend the frequency beyond the instrument's range.

- Low frequency data predicts material behavior over longer timescales that cannot be practically measured on a laboratory instrument.
  - Creep or Stress Relaxation TTS can predict behavior over longer times under static load/deformation.

- High frequency data predicts material behavior at short timescales (high-speed impact, mechanical vibrations, acoustics) that are challenging to measure accurately using an analytical instrument.
  - High frequency mechanical testers almost always are influenced by inertial contributions from the instrument itself.

- Dealy J, Plazek D. *Time-Temperature Superposition – A Users Guide.* Rheology Bulletin 2009; 78**:** 16
- van Gurp M, Palmen J. *Time-temperature superposition for polymeric blends.* Rheology Bulletin 1998; 67: 5.
- Cox WP and Merz EH. *Correlation of dynamic and steady flow viscosities.* Journal of Polymer Science 1958; 28: 619

Limitations of TTS

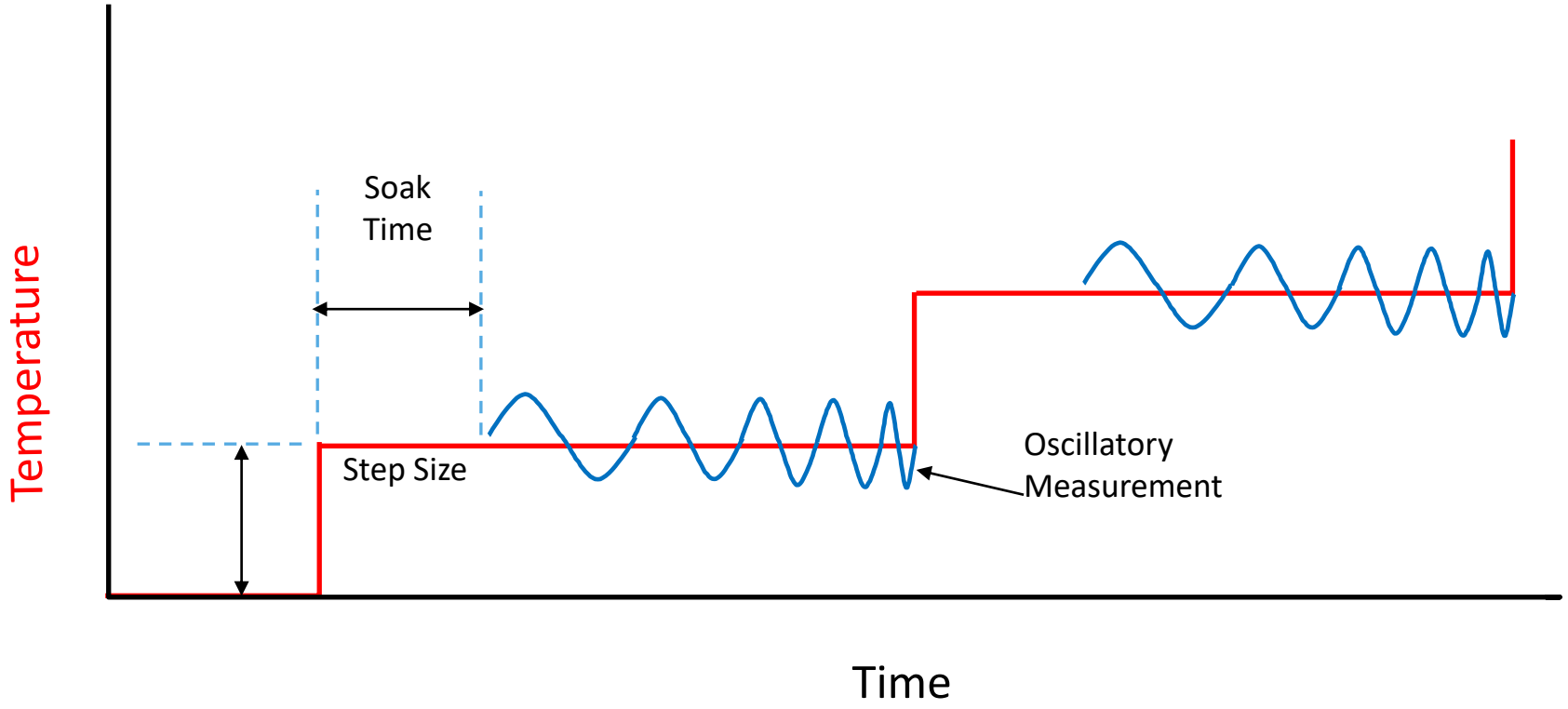
1. TTS typically works with polymers that are thermo-rheological simple

2. TTS may or may not work if:
    a. Polymer contains multiple phases: composite or blends
    b. Filled polymers

3. TTS does not work if:
    a. Sample properties change (e.g. crystalize, melt, cure, decompose) within temperature of interest

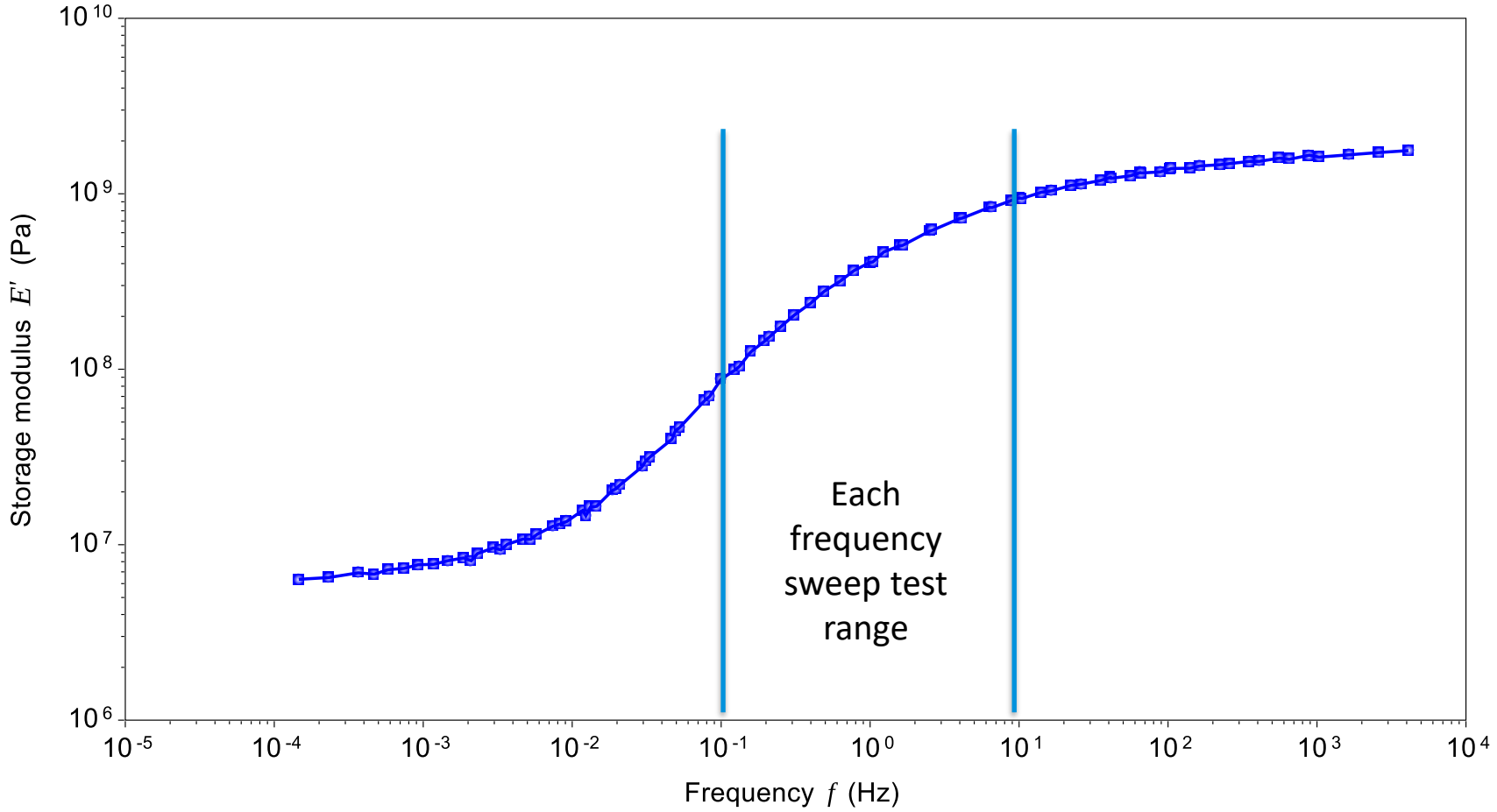
Temperature Step & Hold- Single /Multi-Frequency

- A step and hold temperature profile is applied. The material response is monitored over a range of frequencies, at constant amplitude of deformation

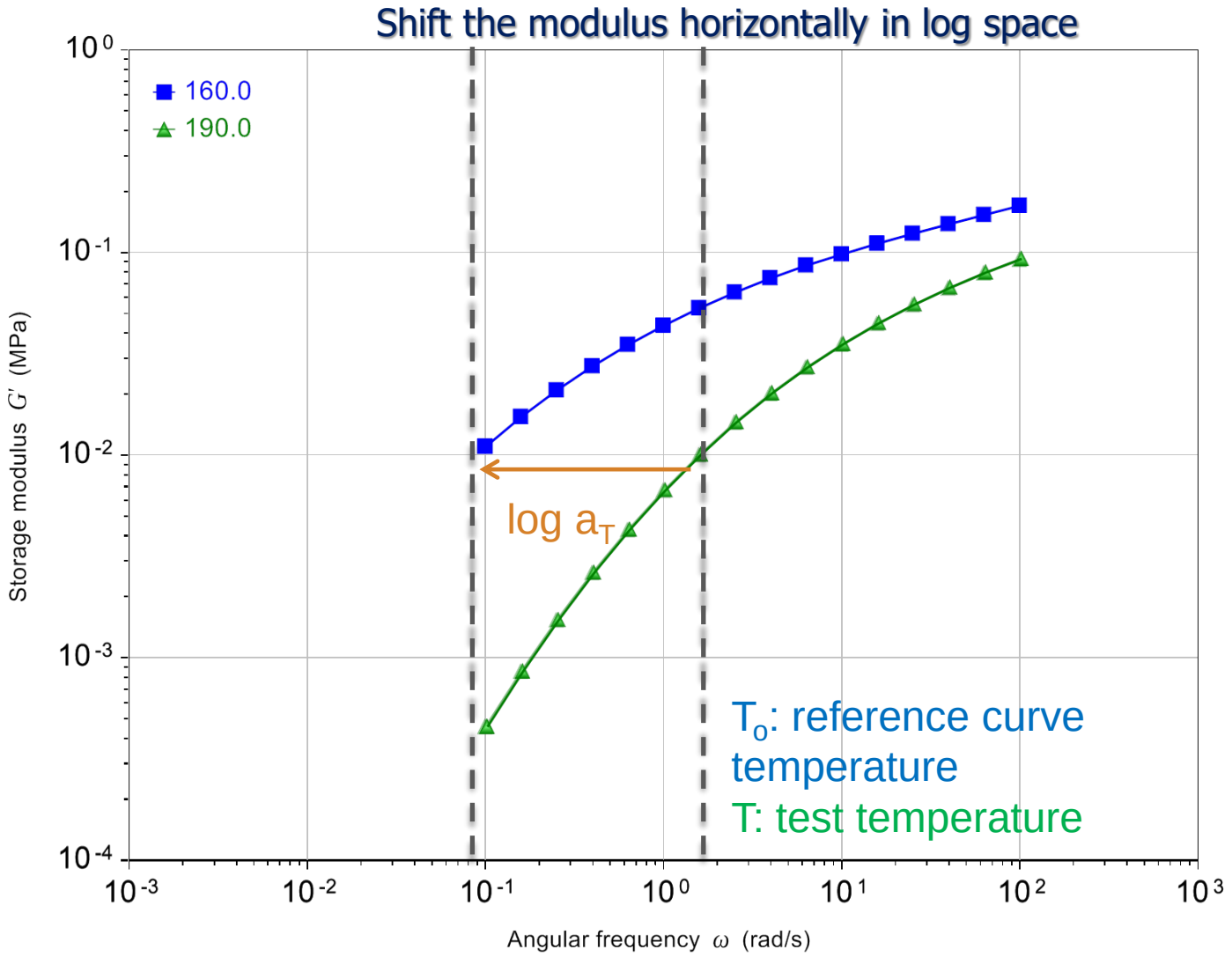


Master curve from TTS Shifting, polystyrene

- TTS Master Curve using 155°C as reference



Principle of time-temperature superposition (TTS)



Dealy J, Plazek D. *Time-Temperature Superposition – A Users Guide.* Rheology Bulletin 2009; 78**:** 16

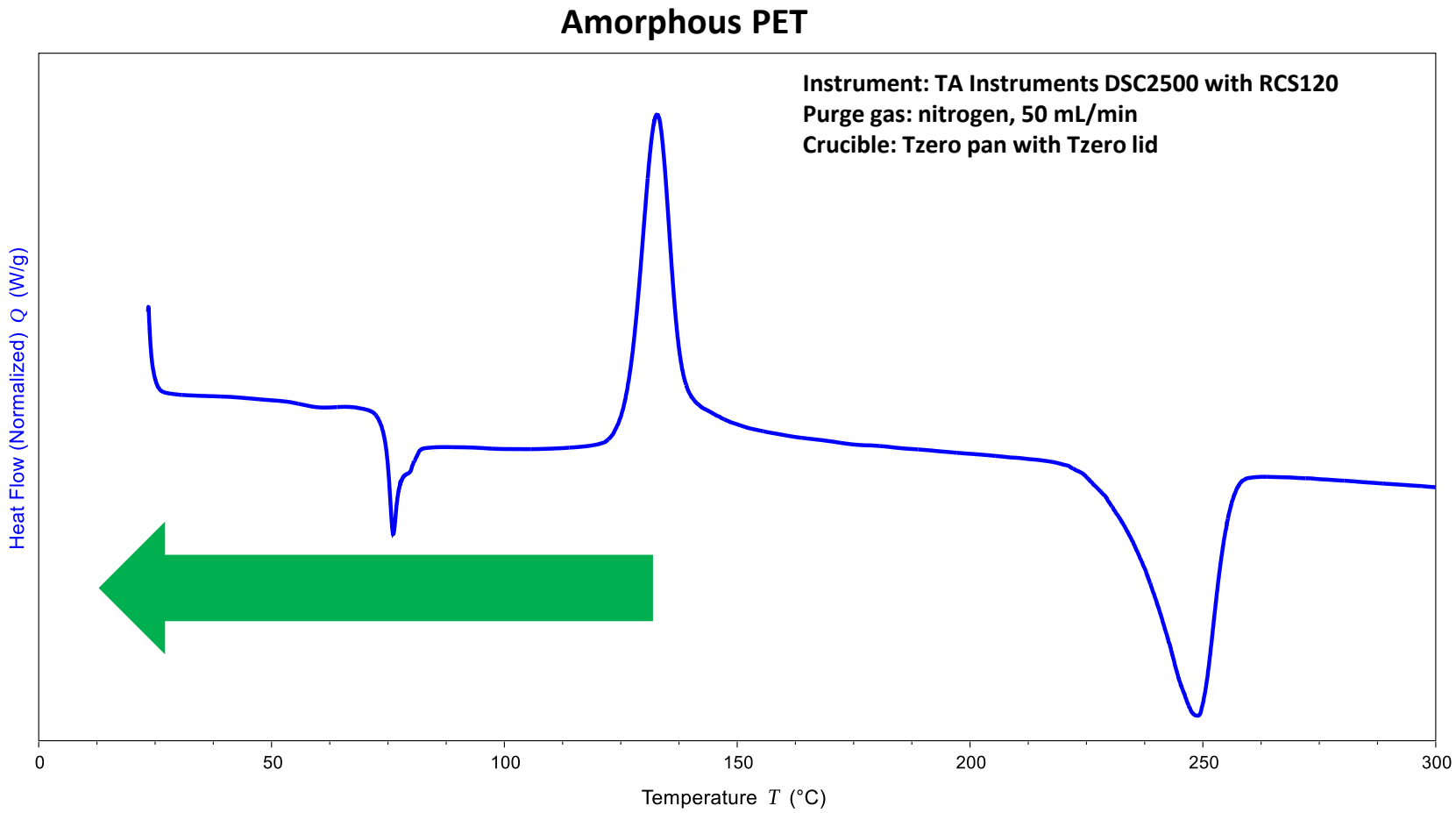
Method development for TTS studies

Selecting a reference temperature

- Decide on the Reference Temperature: T_r.
- Higher frequencies ≡ shorter times. Test temperatures < T_r.
- Lower frequencies ≡ longer times. Test temperatures > T_r.

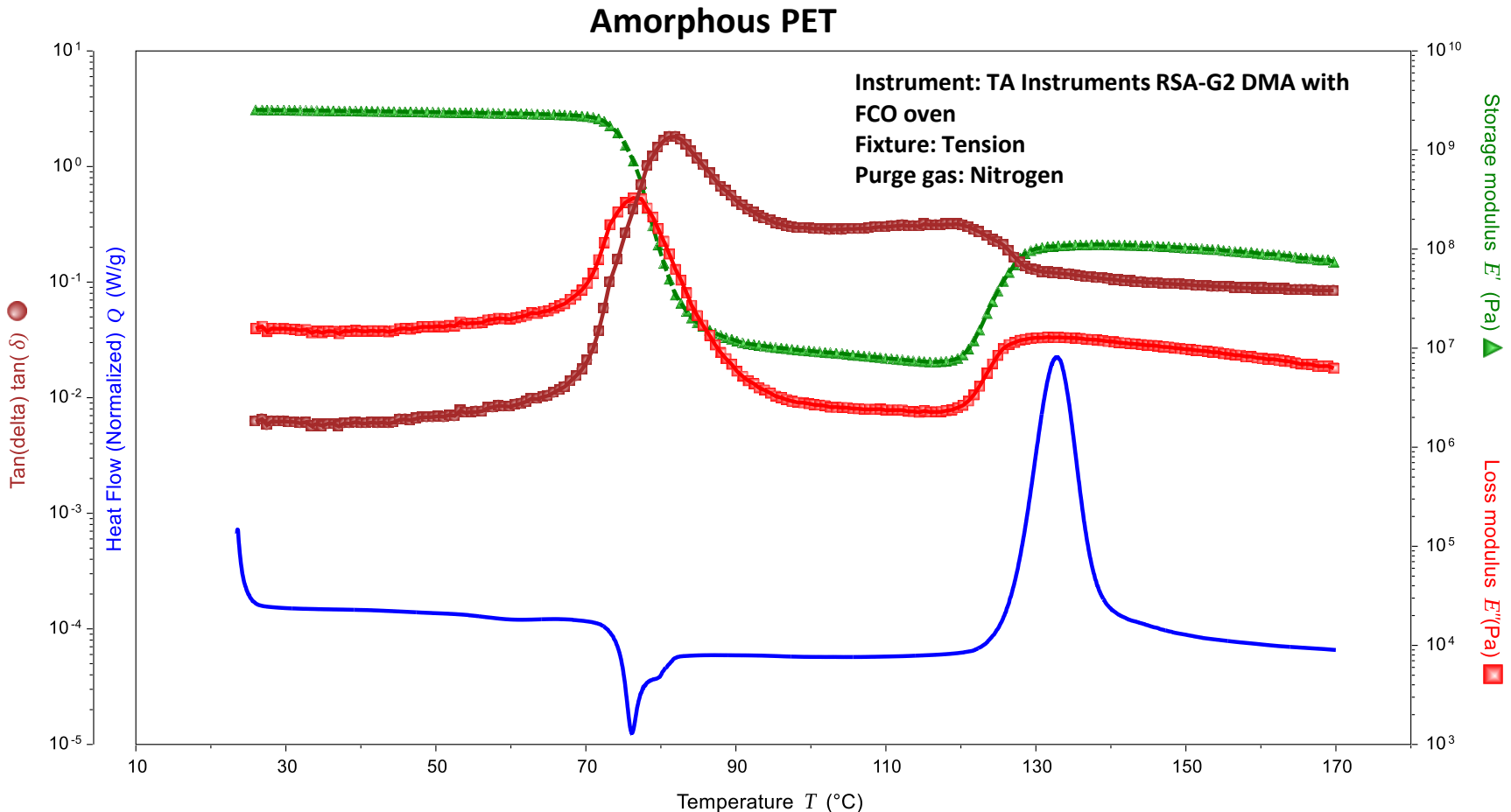
Importance of thermal analysis in TTS studies

- Sample properties change (e.g. crystalize, melt, cure, decompose) within temperature of interest
- Scan material over temperature range to get an idea of transition behavior and modulus-temperature.



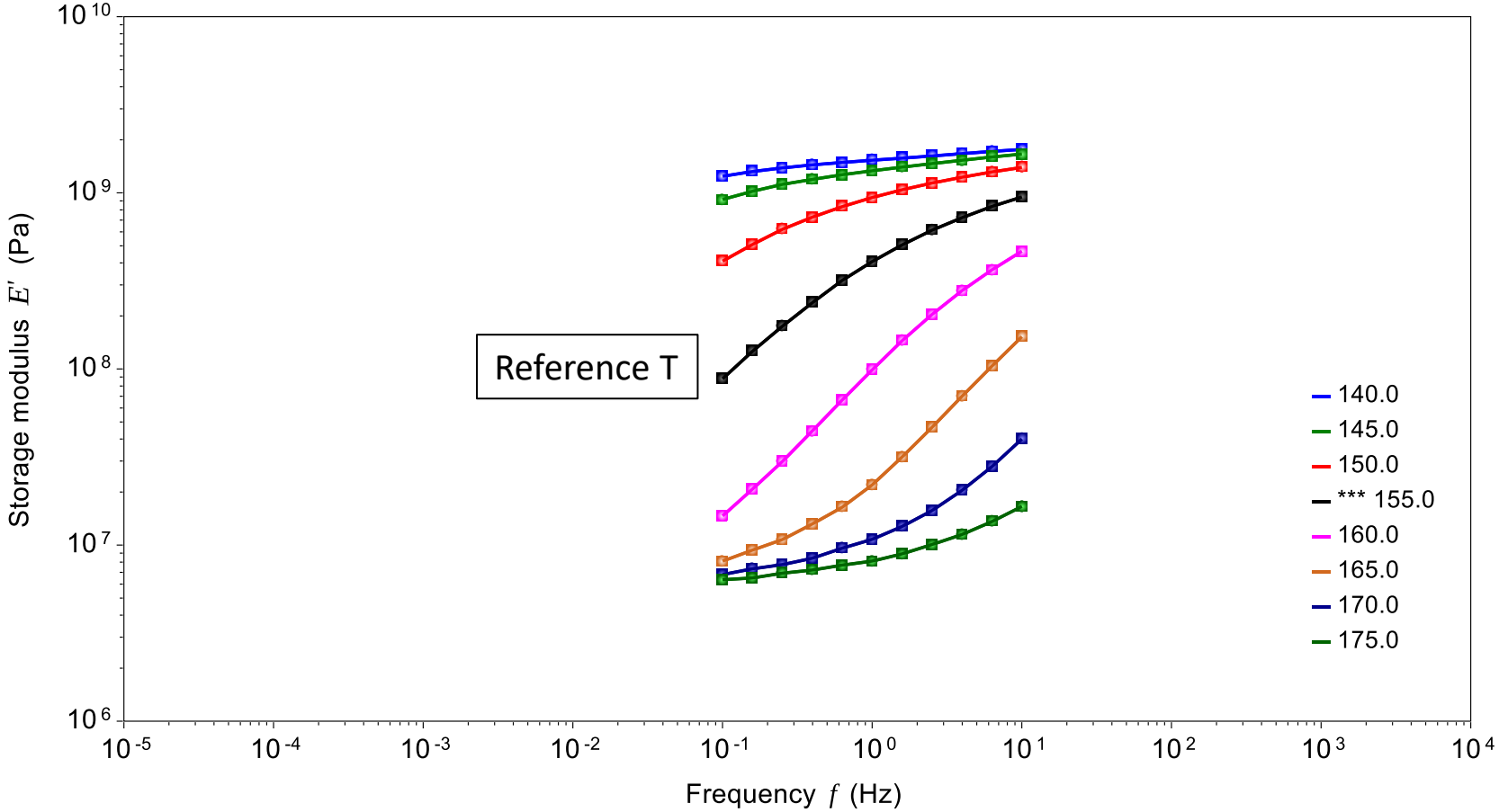
Importance of thermal analysis in TTS studies

- Scan material over temperature range to get an idea of transition behavior and modulus-temperature.
- Allows for optimizing experimental method (axial force, force track, % strain, etc.) prior to longer TTS experiments.



TTS example: shifting of individual curves, polystyrene

- Generating frequency sweep data at different temperature steps
- Select your reference temperature (e.g. 155°C)

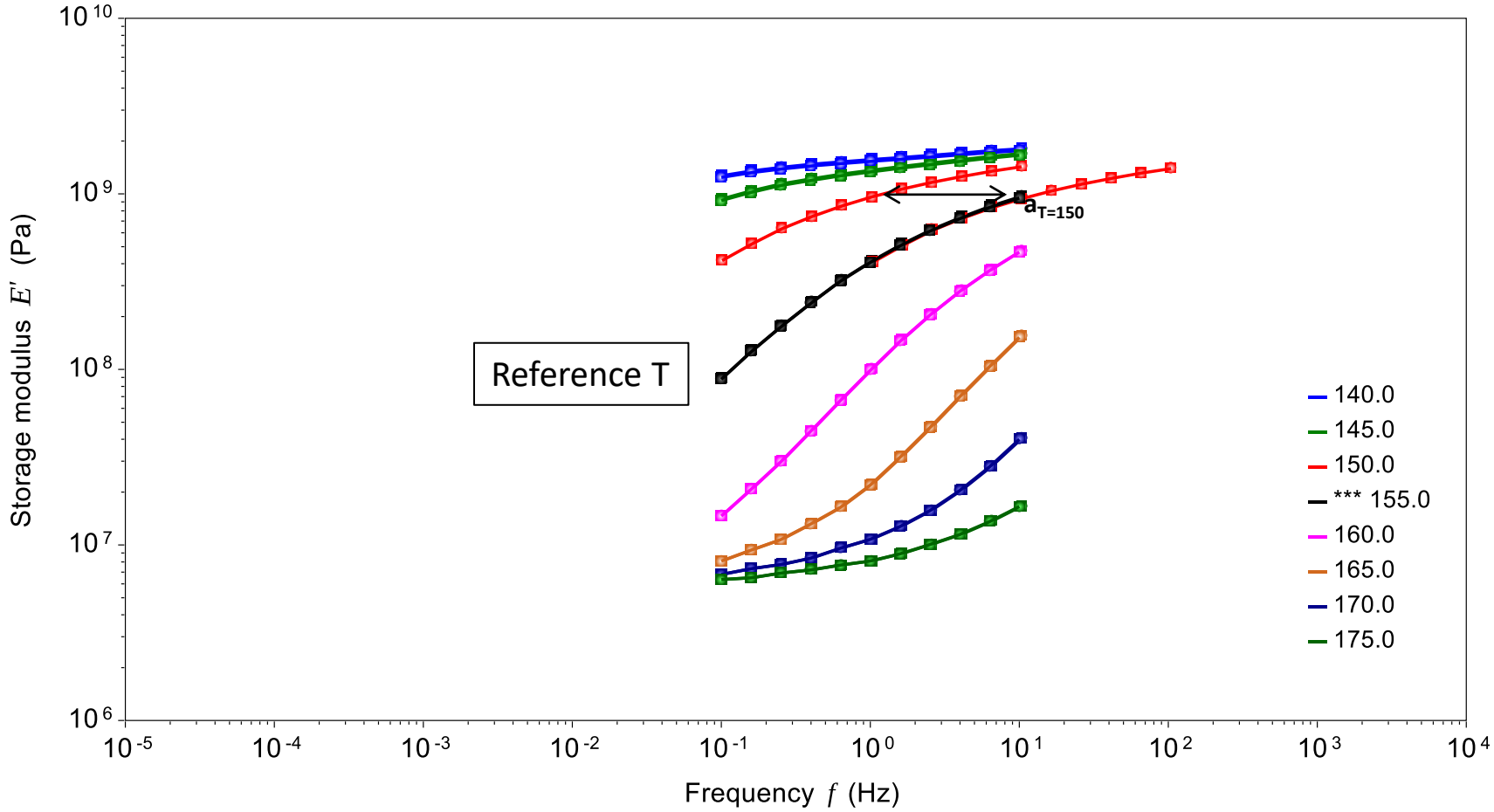


- Shifts can be performed automatically in TRIOS.

Refer to http://www.tainstruments.com/tts-and-trios-software/

TTS example: shifting of individual curves, polystyrene

- Low temperature data shift to higher frequencies



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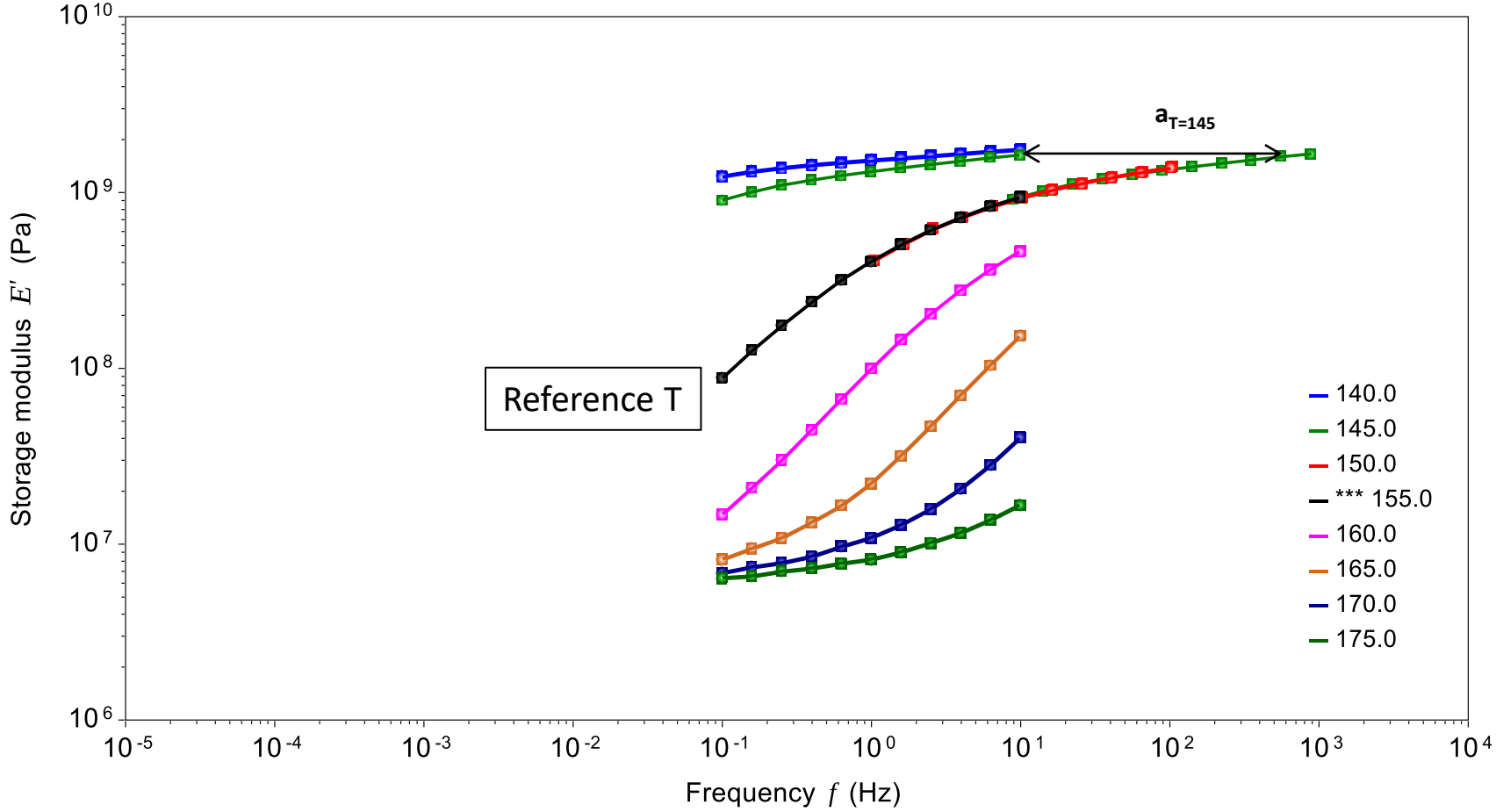
TTS example: shifting of individual curves, polystyrene

- Low temperature data shift to higher frequencies



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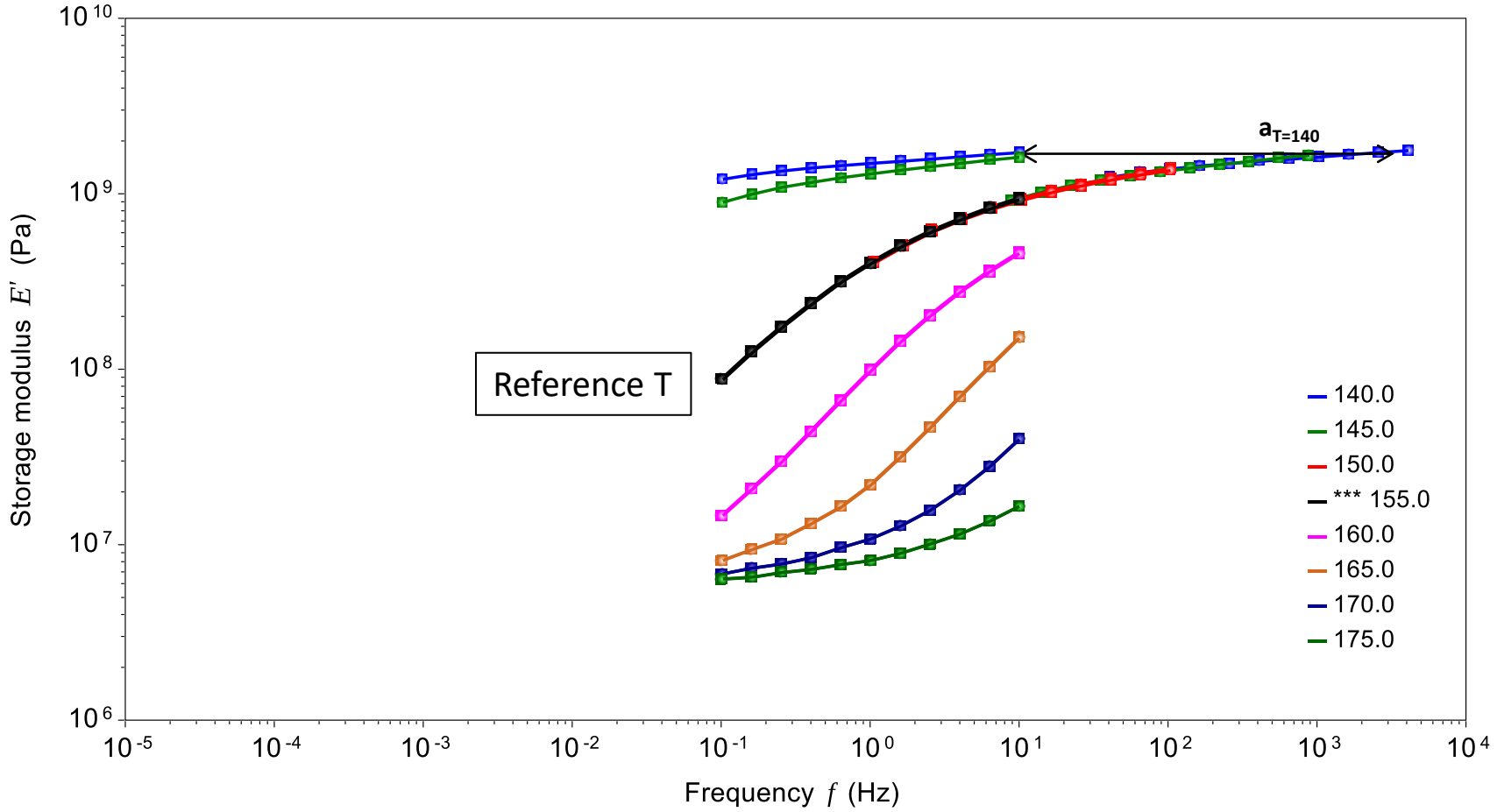
TTS example: shifting of individual curves, polystyrene

- Low temperature data shift to higher frequencies

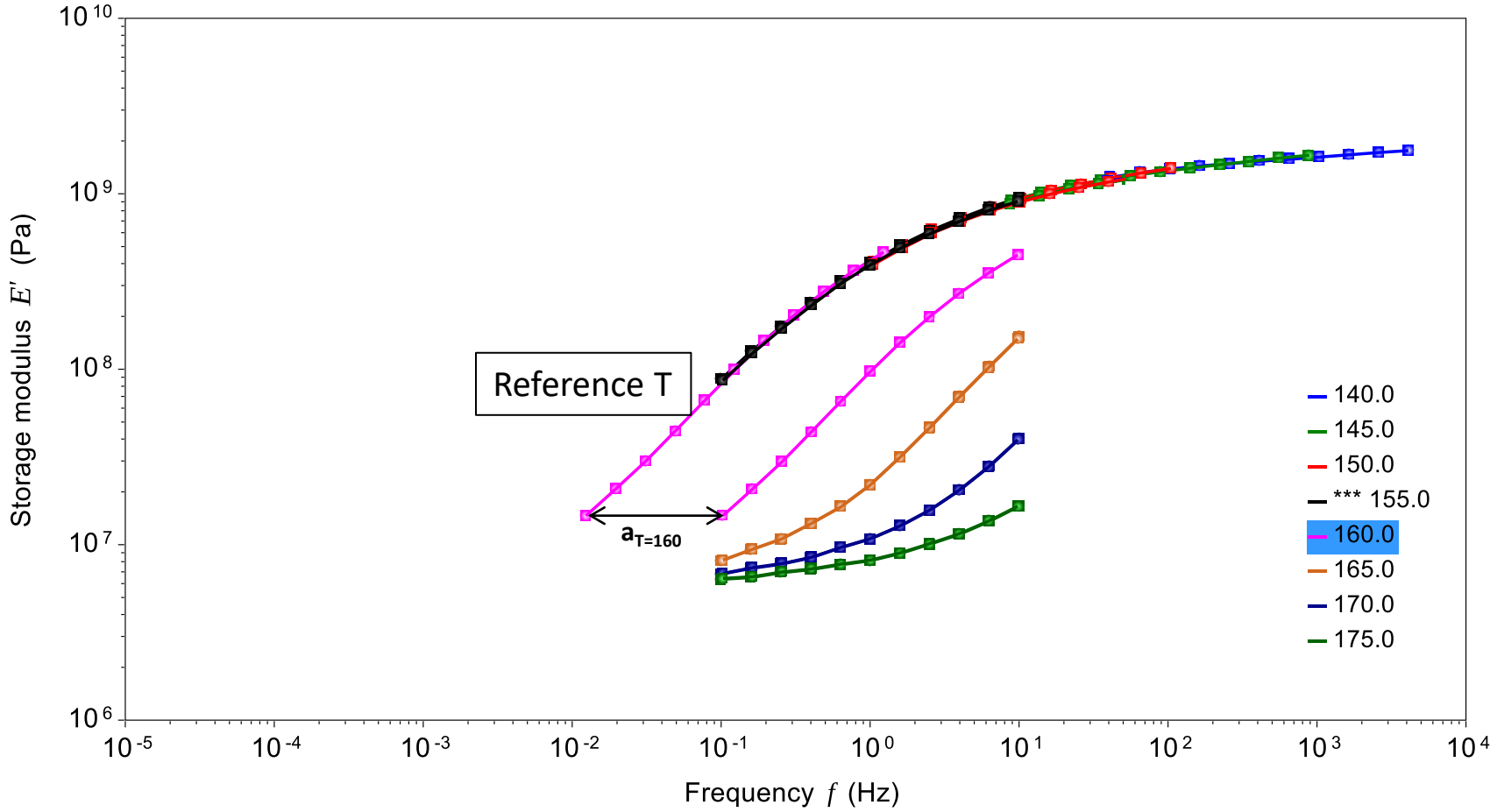


- Shifts can be performed automatically in TRIOS.

Refer to http://www.tainstruments.com/tts-and-trios-software/

TTS example: shifting of individual curves, polystyrene

- High temperature data shift to lower frequencies

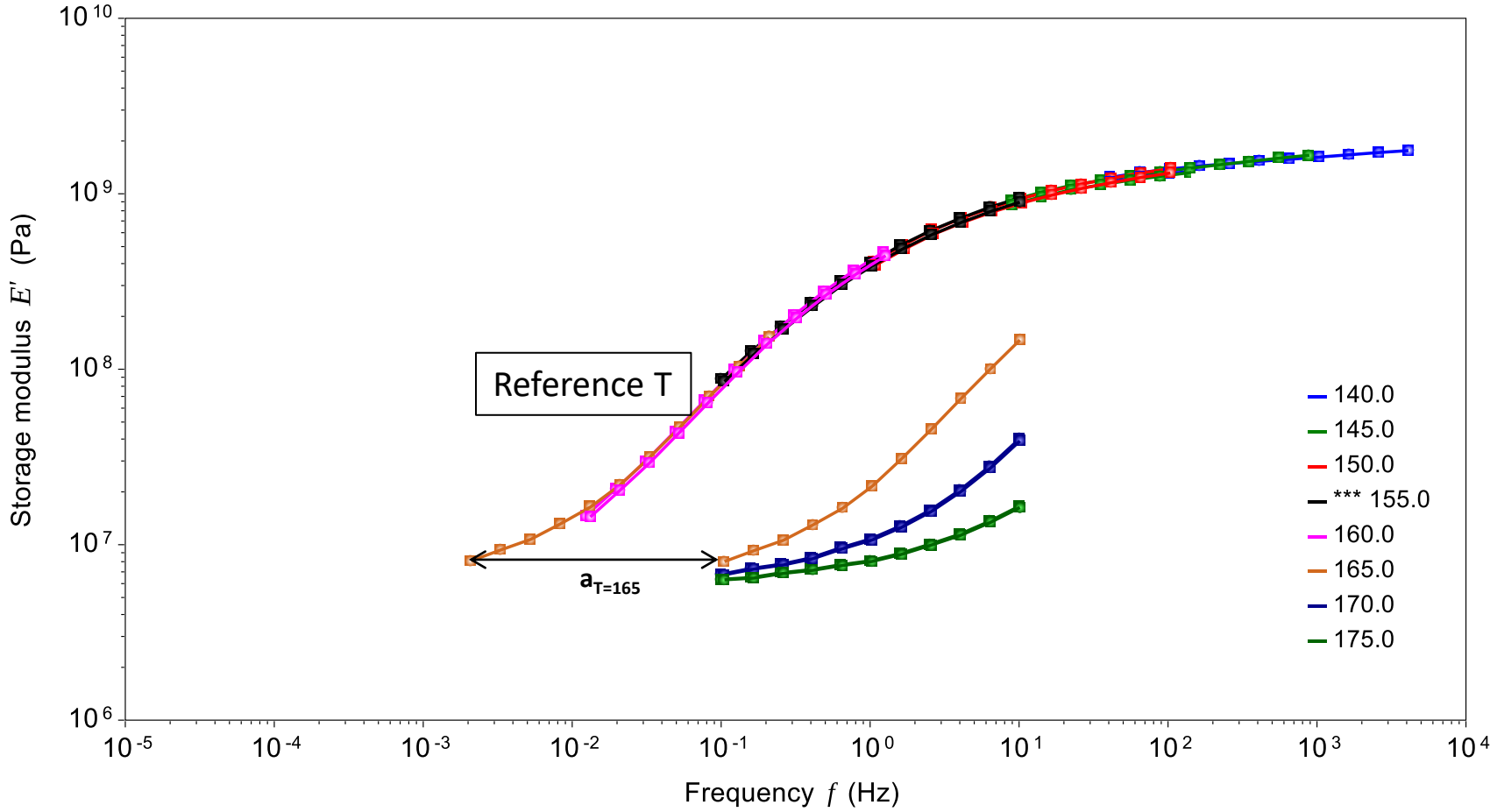


- Shifts can be performed automatically in TRIOS.

Refer to http://www.tainstruments.com/tts-and-trios-software/

TTS example: shifting of individual curves, polystyrene

- High temperature data shift to lower frequencies



- Shifts can be performed automatically in TRIOS.

Refer to http://www.tainstruments.com/tts-and-trios-software/

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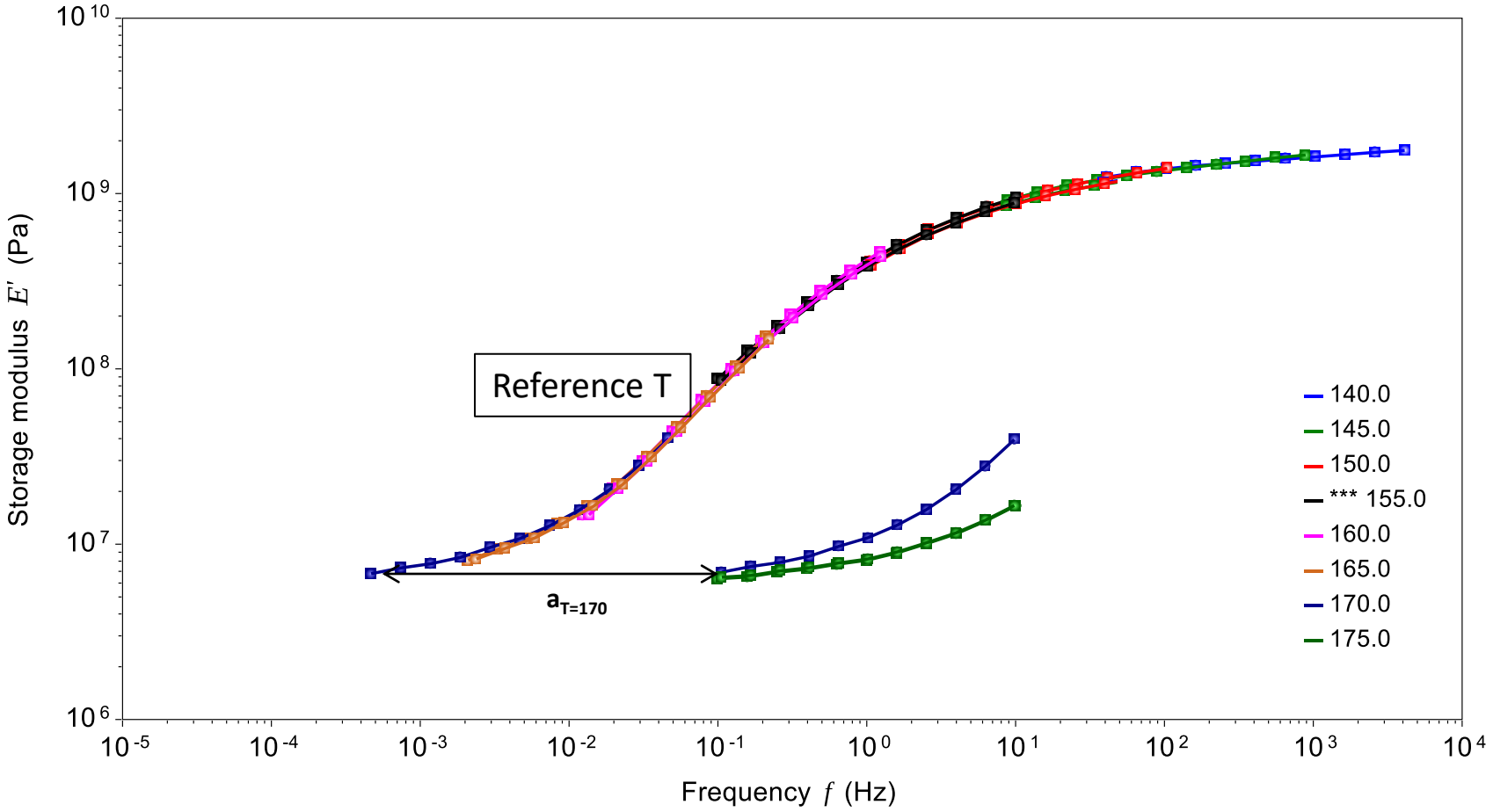

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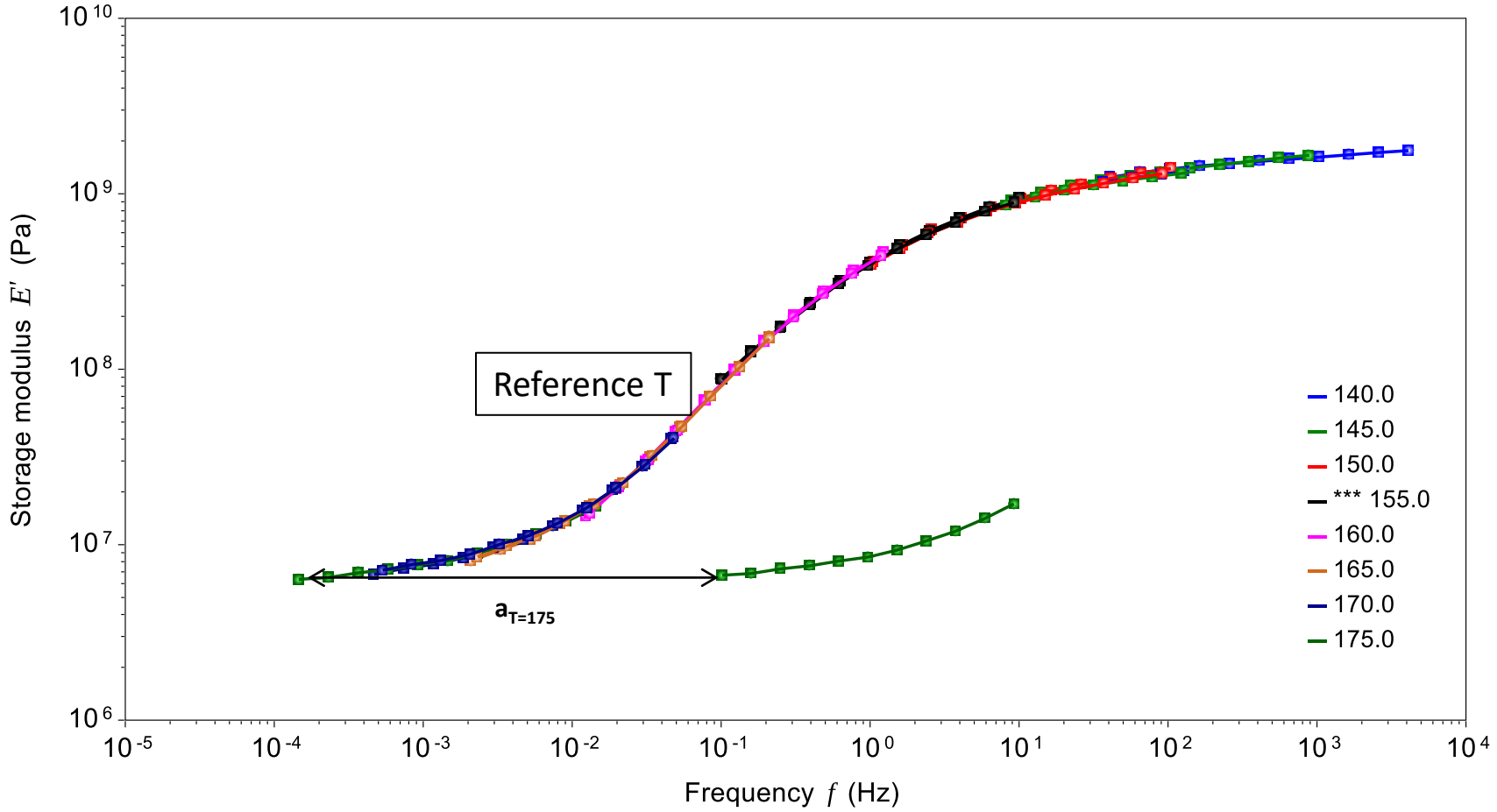

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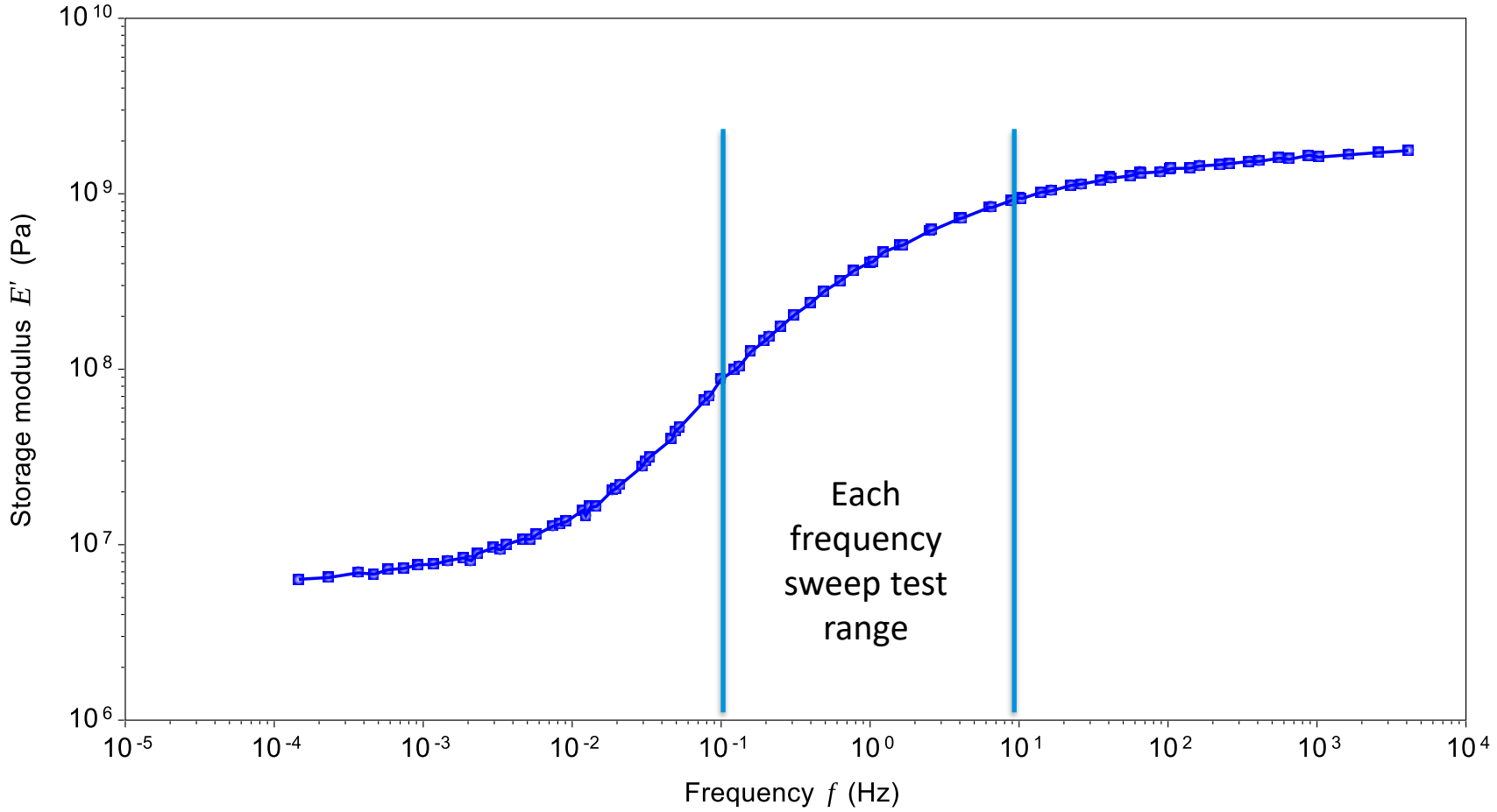
Refer to http://www.tainstruments.com/tts-and-trios-software/

Master curve from TTS Shifting, polystyrene

- TTS Master Curve using 155°C as reference



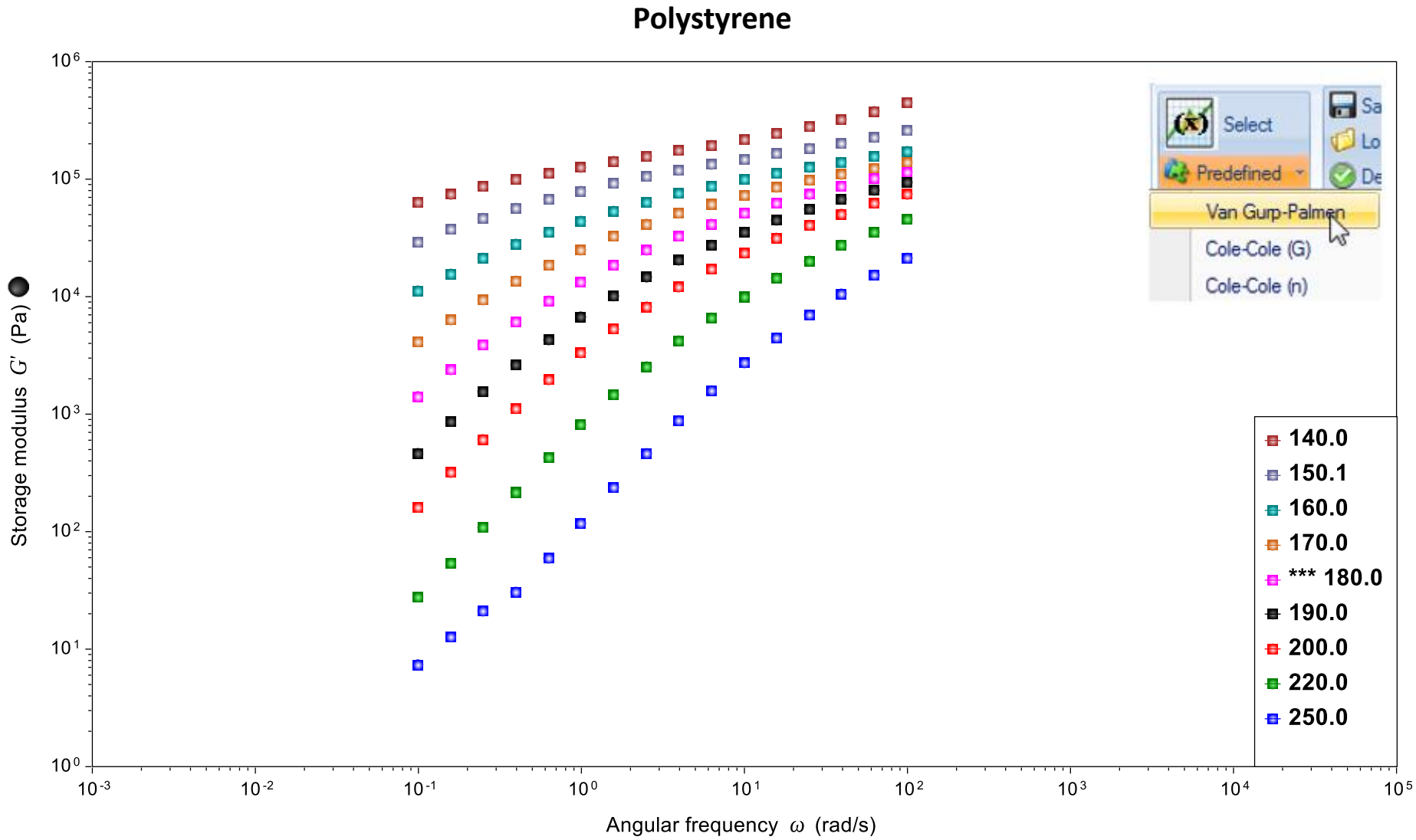
How accurate is the master curve?

Check-points to check validity of TTS

1. Van-Gurp Palmen plot should be a smooth curve
2. Cole-Cole plot should be a smooth curve
3. Shift factor (a_T) should fit the WLF or Arrhenius equation
4. Same a_T should be applicable to all viscoelastic parameters (E', E", tan δ)

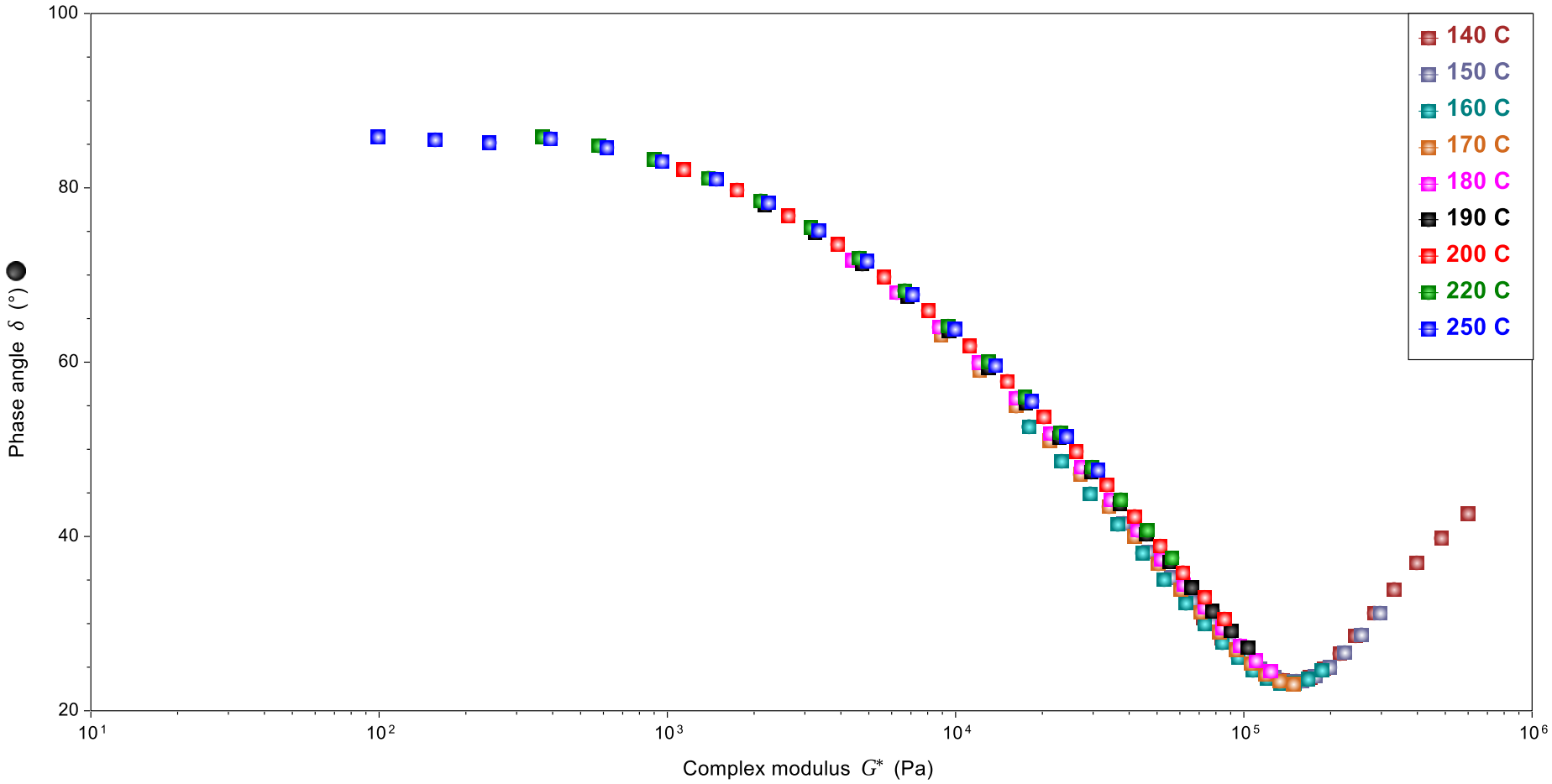
If any one of the above criteria are not met, TTS may not be applicable for your material.

1. van Gurp-Palmen plot to Validate TTS

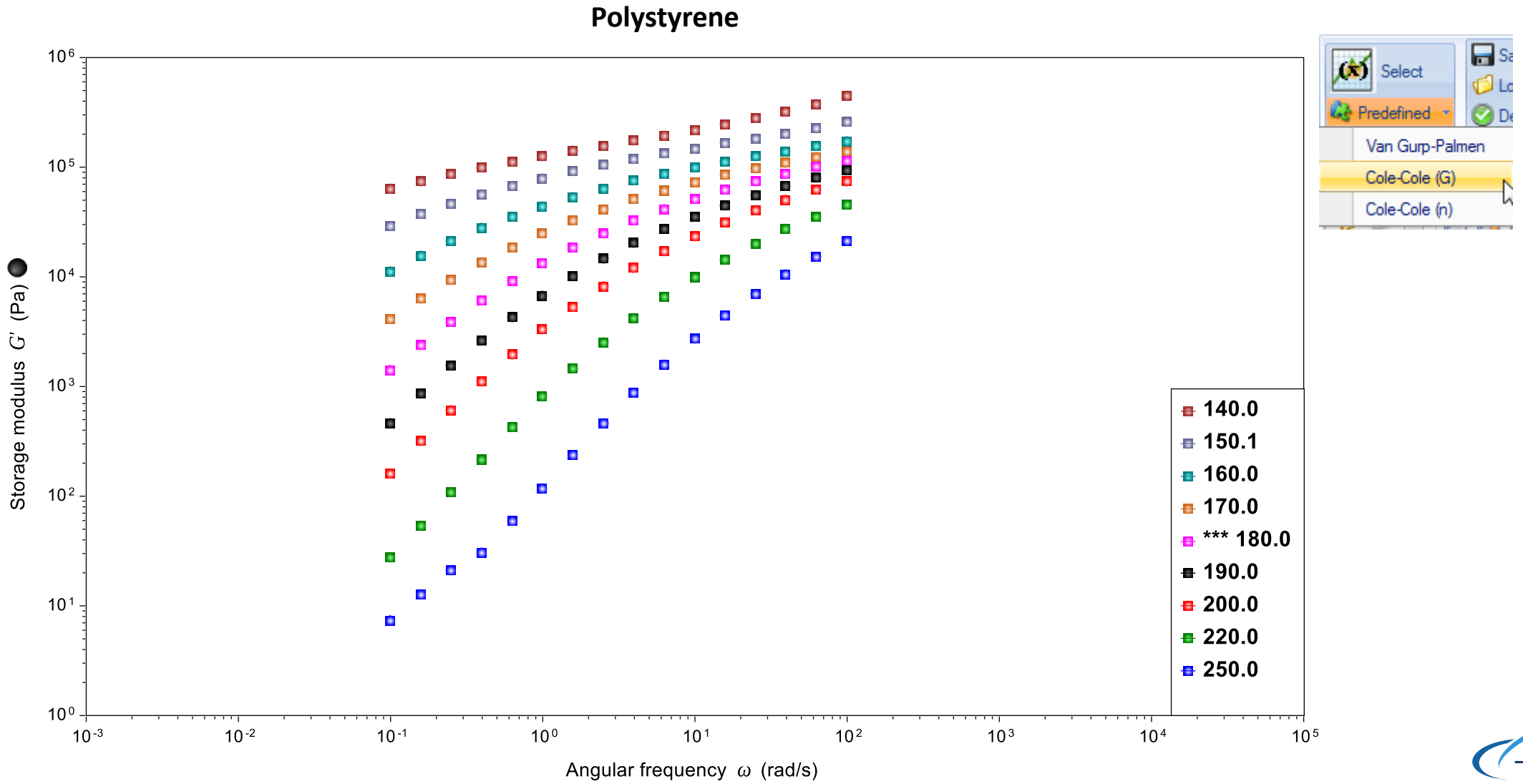


1. van Gurp-Palmen plot to Validate TTS

- The van Gurp-Palmen plot can be directly plotted in TRIOS to validate the application of (TTS).



2. Cole-Cole plot to Validate TTS



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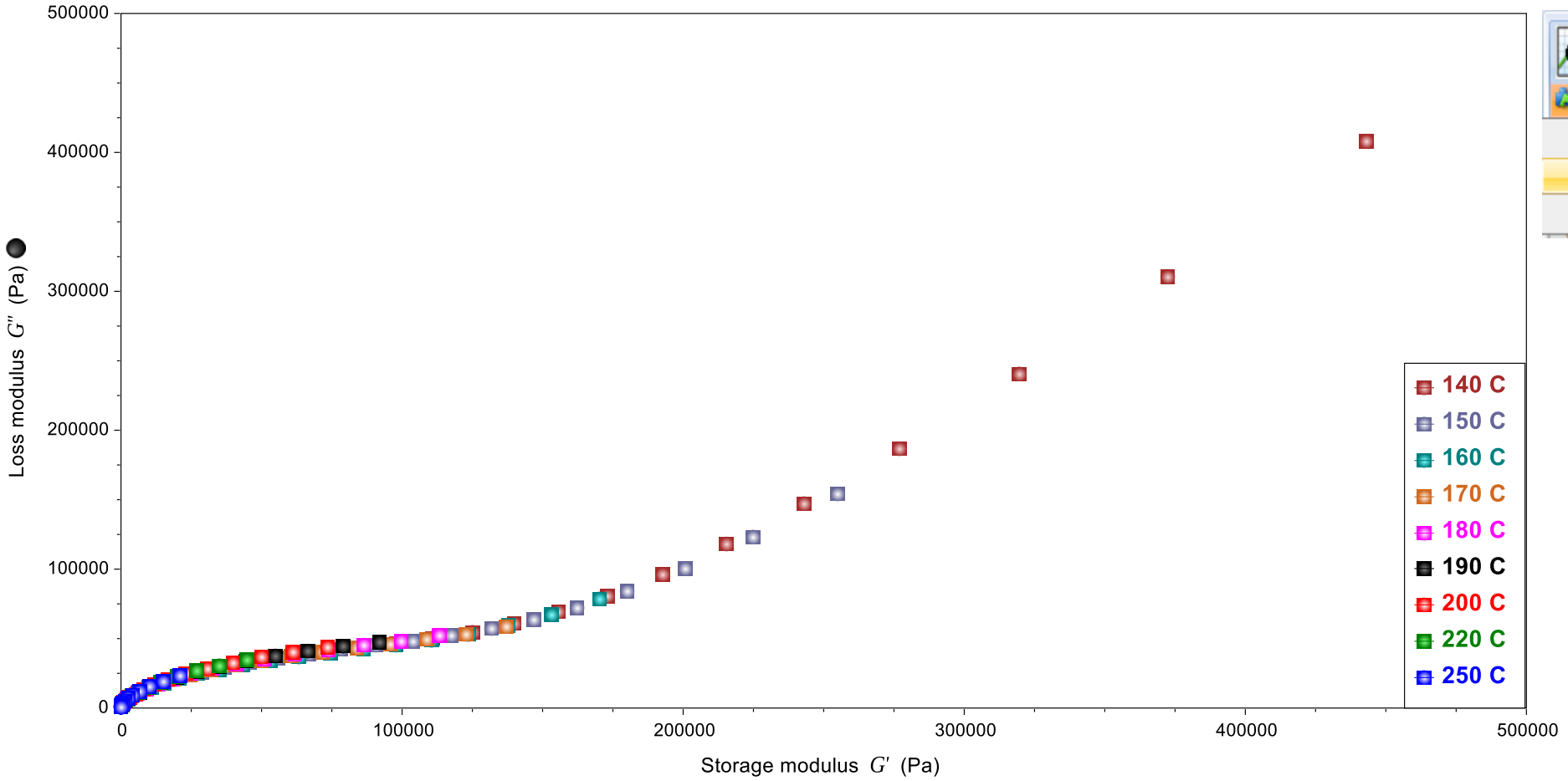


3. Shift factors should fit WLF or Arrhenius Equations

- Master Curves can be generated using shift factors derived from the Williams, Landel, Ferry (WLF) model

$$\log a_T = -c_1(T-T_0)/(c_2+(T-T_0))$$

a_T = temperature shift factor T_0 = reference temperature

c_1 & c_2 = constants from curve fitting

Generally, c_1=17.44 & c_2=51.6 when $T_0 = T_g$

- The Arrhenius model works better if
  - $T > T_g$+100°C; or $T < T_g$ and polymer is not elastomeric
  - temperature range is small, then c_1 & c_2 cannot be calculated precisely

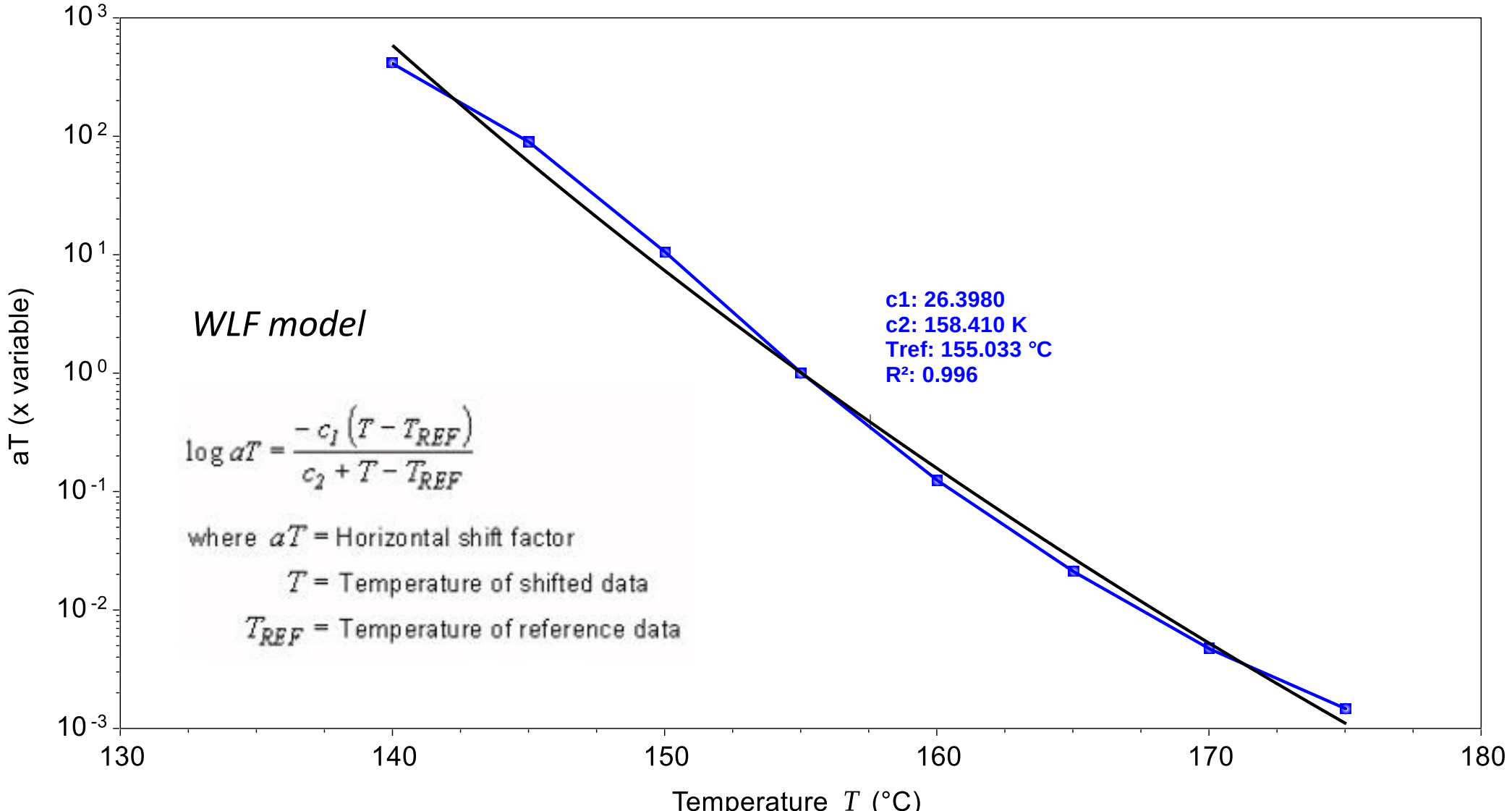
$$\ln a_T = (E_a/R)(1/T-1/T_0)$$

a_T = temperature shift factor E_a = Apparent activation energy

T_0 = reference temperature R = gas constant

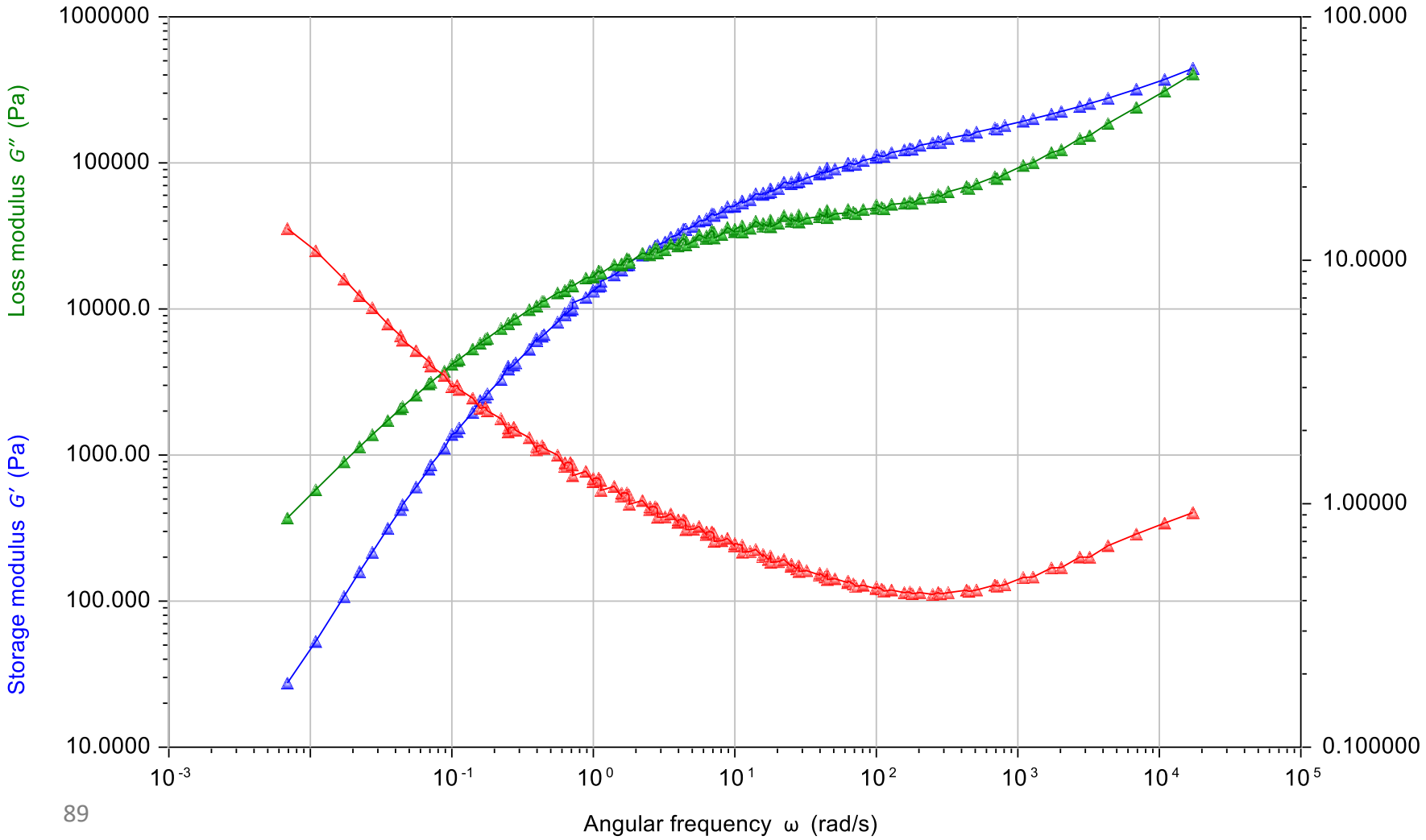
3. Shift Factors a_T vs. Temperature

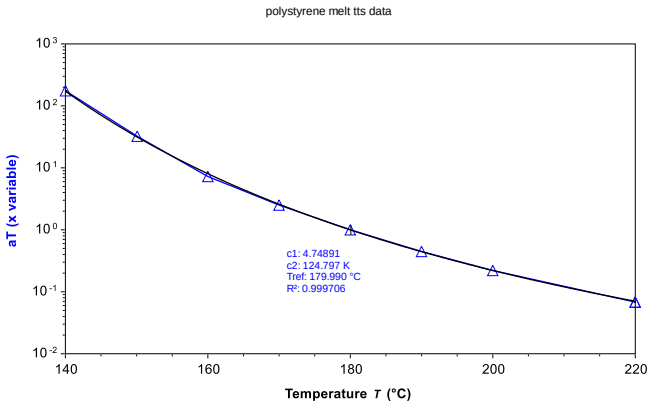
- Shift factors can be checked against the WLF and Arrhenius model directly in TRIOS



4. Same a_T should be applicable to all viscoelastic parameters (E', E", tan δ)

Curve shifting can be performed with all the parameters plotted simultaneously in TRIOS





Same a_T applied to all functions

Limitations of TTS

- TTS typically works with polymers that are thermo-rheological simple

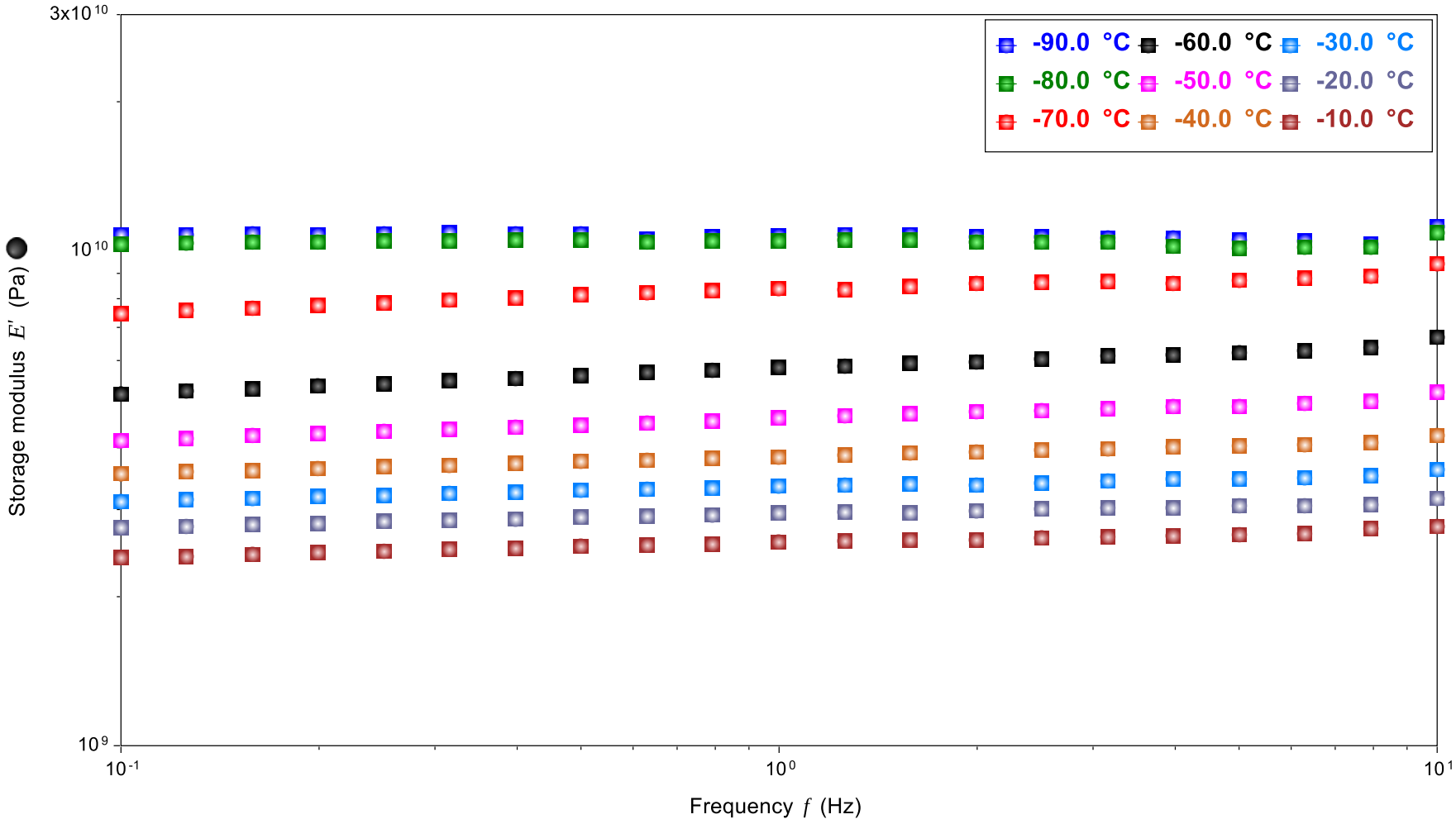
- TTS may or may not work if:
  - Polymer contains multiple phases: composite or blends
  - Filled polymers

- TTS does not work if:
  - Sample is partially crystalized, crosslinked, or highly filled
  - Sample properties change (e.g. crystalize, melt, cure, decompose) within temperature of interest

Limitations of TTS

- TTS typically works with polymers that are thermo-rheological simple
- TTS may or may not work if:
  - Polymer contains multiple phases: composite or blends
  - Filled polymers - trans polyisoprene (PIP) with 80% inorganic filler
- TTS does not work if:
  - Sample is partially crystalized, crosslinked, or highly filled
  - Sample properties change (e.g. crystalize, melt, cure, decompose) within temperature of interest

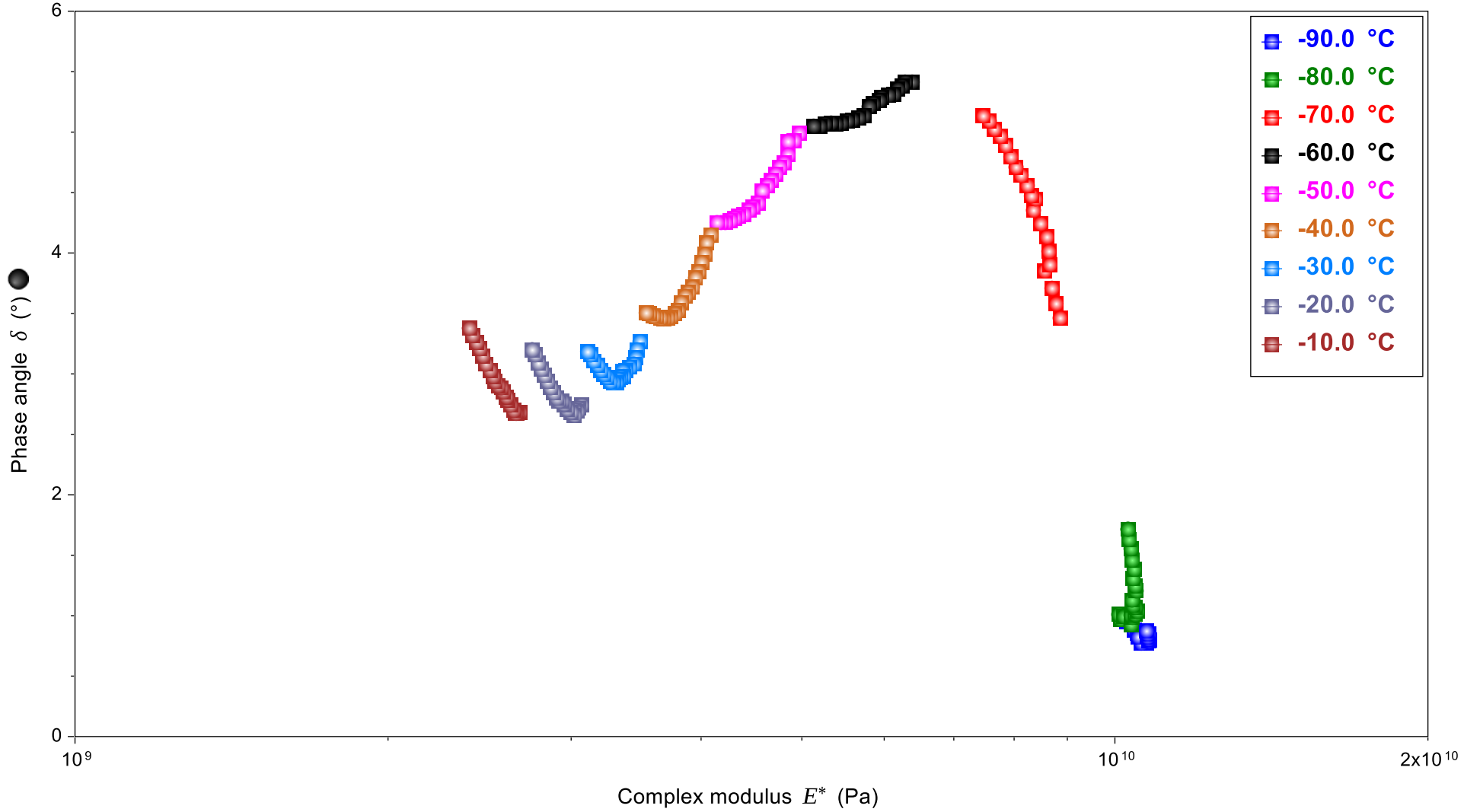
Temperature sweep Trans PIP



Instrument: RSA-G2 DMA
Fixture: 3-point bending
Purge gas: Nitrogen

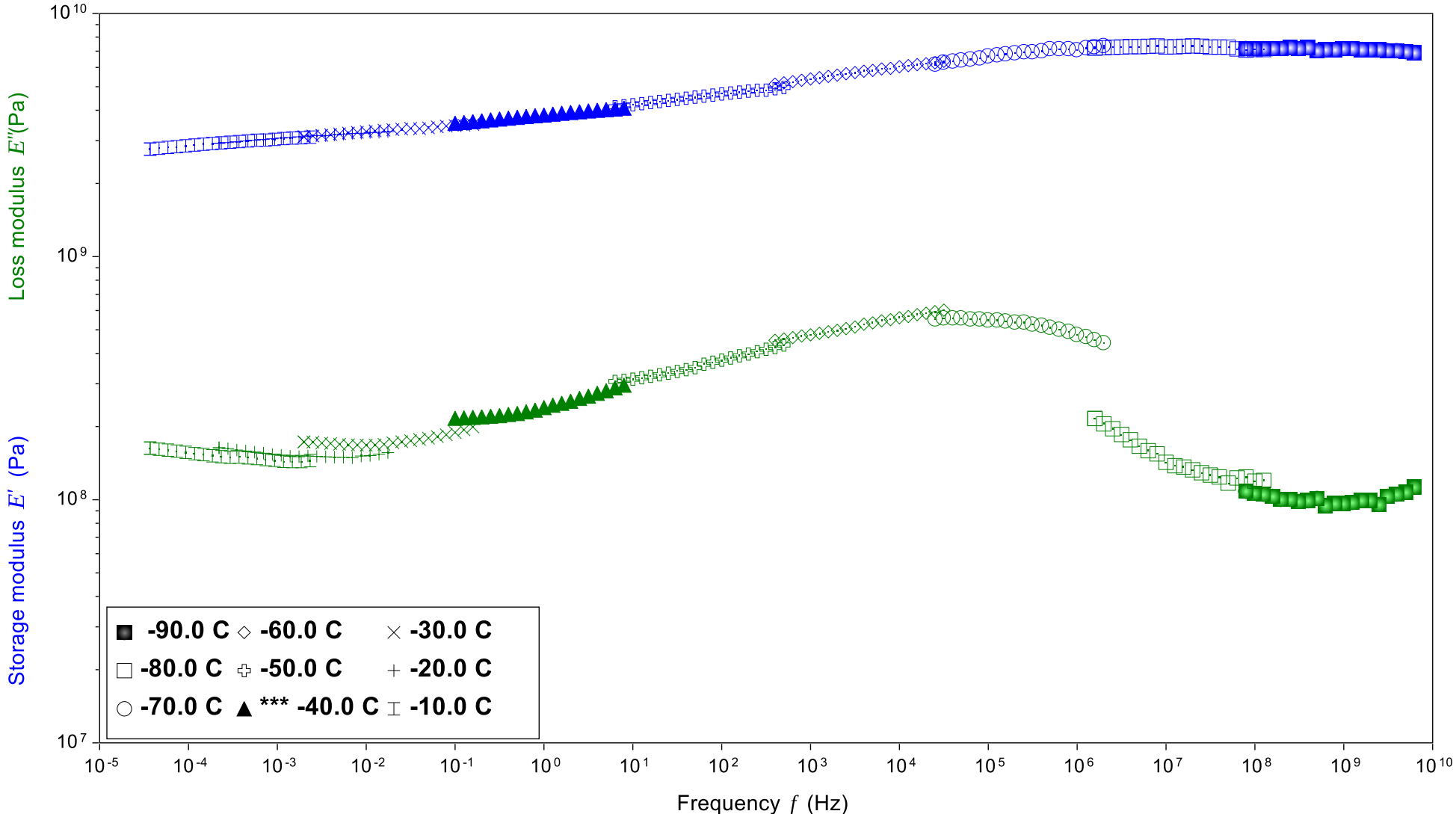
Van Gurp plot PIP

Van-Gurp plot shows discontinuity indicating TTS might not be applicable to this sample.

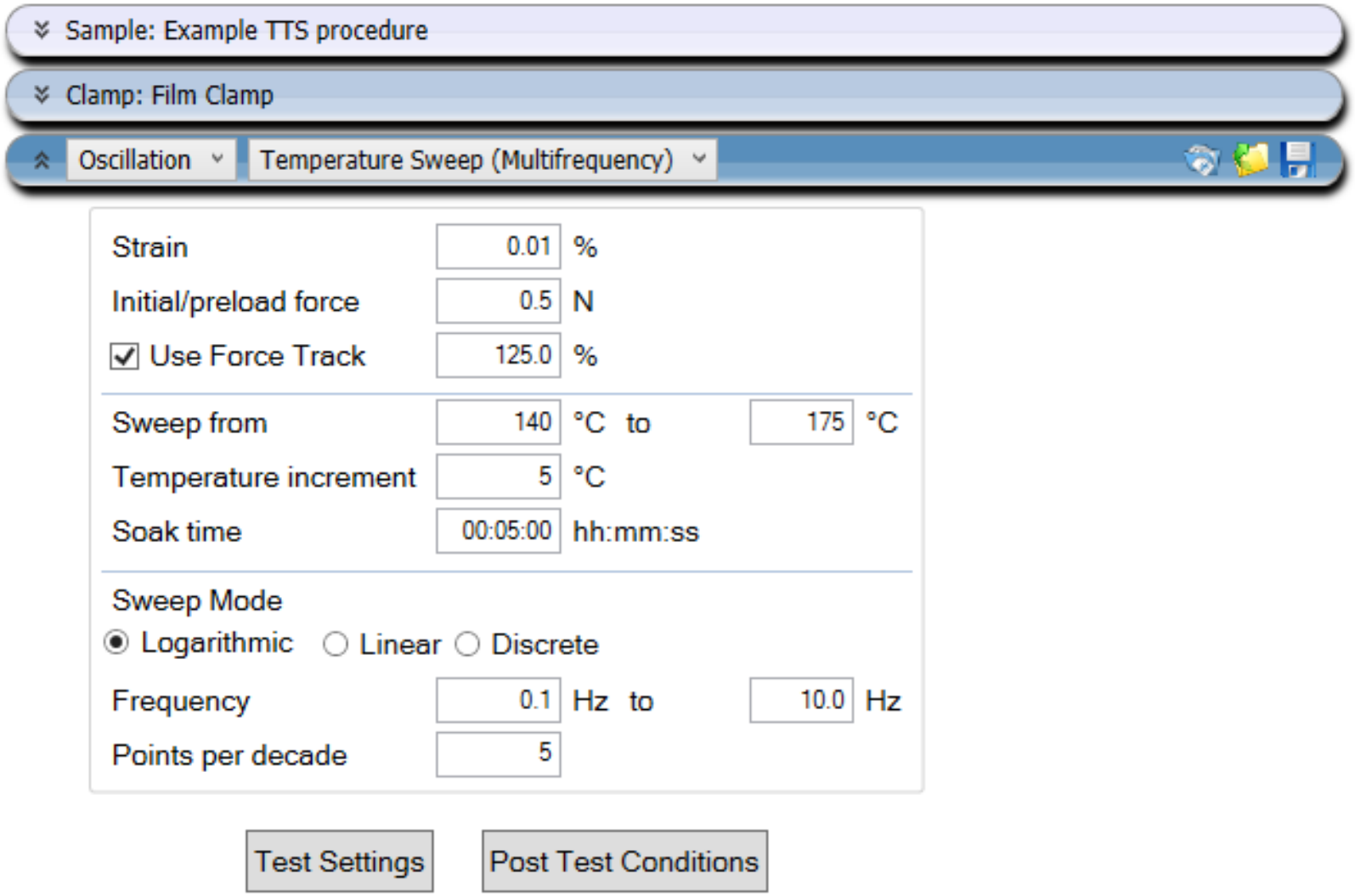


Master curve PIP

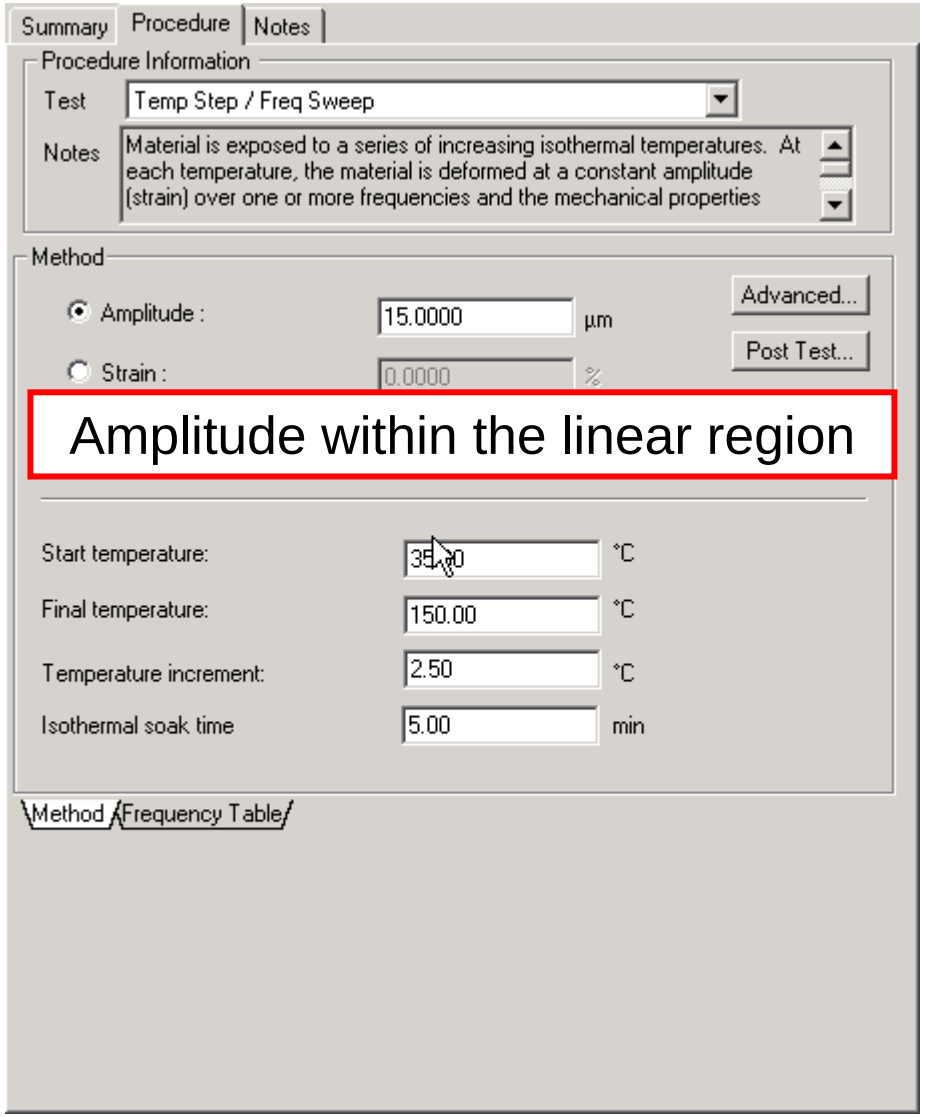
Same a_T cannot be applied to all viscoelastic parameters to obtain a continuous master curve.

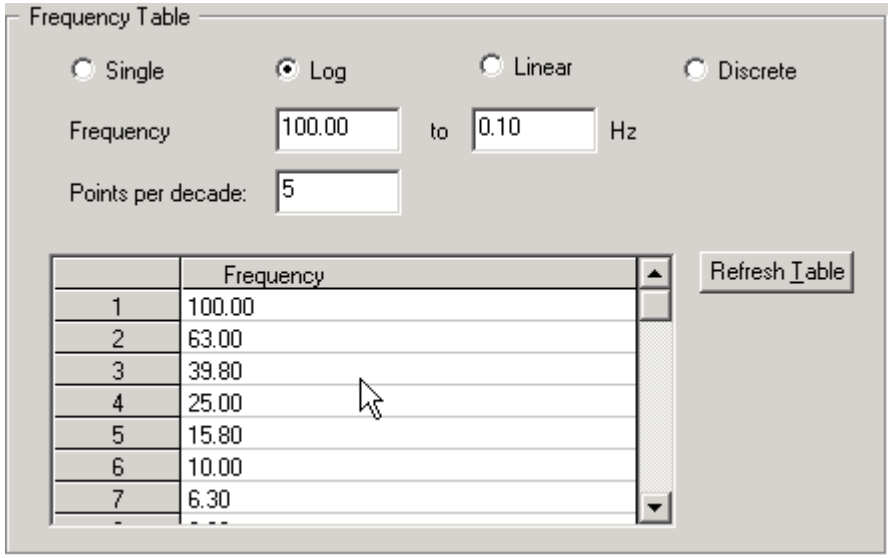


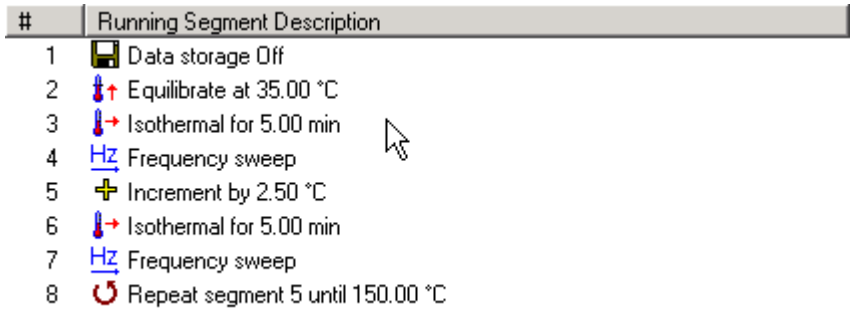
Setting up TTS procedure on DMA850



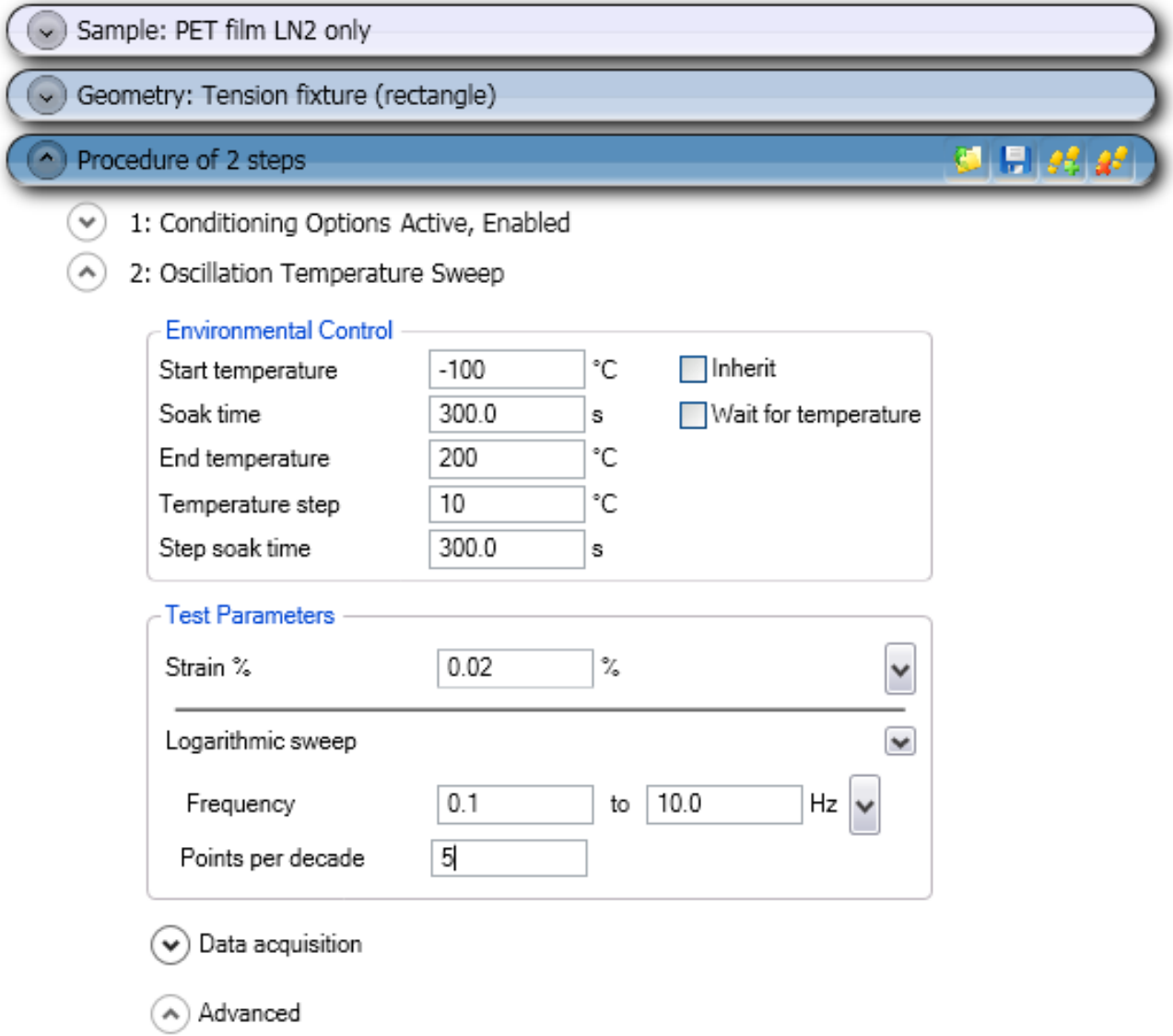
Setting up TTS procedure on Q800







Setting up TTS procedure on RSA G2



TTS concluding remarks

- Time-temperature superposition (TTS) is a valuable tool for describing the viscoelastic properties of polymeric materials over a wide range of times/frequencies.
- Thermal analysis techniques such as DSC and DMA can be valuable tools for developing the method for TTS experiments.
- The modulus vs frequency curves at various temperatures can be shifted horizontally and/or vertically depending on whether the material is thermo-rheologically simple.
- Experimental verification of part of a master curve provides additional confidence in the applicability of the TTS principles to a material.

References for TTS

1. Ferry JD. *Viscoelastic Properties of Polymers.* Wiley 1970, Chapter 11.

2. Dealy J, Plazek D. *Time-Temperature Superposition – A Users Guide.* Rheology Bulletin 2009; 78**:** 16

3. van Gurp M, Palmen J. *Time-temperature superposition for polymeric blends.* Rheology Bulletin 1998; 67: 5.

4. Cox WP and Merz EH. *Correlation of dynamic and steady flow viscosities.* Journal of Polymer Science 1958; 28: 619

5. Tajvidi M, Falk RH, Hermanson JC. *Time-temperature superposition principe applied to a kenaf-fiber/high density polyethylene composite*. J. Appl. Polym. Sci. 2005; 97: 1995

6. Lovering EG, Wooden DC*. Transitions in Trans-1,4-Polyisoprene.* J. Polym. Sci. 1969; 7: 1639

7. Mavridis H, Shroff RV. *Temperature dependence of Polyolefin Melt Rheology.* Poly. Eng & Sci 1992; 32: 1778

Summary

Summary: DMA applications

- DMA Applications and data interpretation
  - Glass transition temperature
    - Definition
    - Factors influencing Tg
    - Determination of cross-linking density
  - Evaluation of polymer blends and compatibility
  - Effect of anisotropy on mechanical properties
  - Influence of additives
    - Fillers
    - Plasticizers
  - Application of creep-recovery and stress relaxation tests

Summary: Troubleshooting DMA experiments

- Troubleshooting common experimental issues
  - Confidence check with polycarbonate
  - Sample preparation
  - Plotting the stiffness signal
  - Tips for compression testing

Summary: Time-temperature superposition (TTS)

- Time-Temperature Superposition (TTS)
  - Introduction to time-temperature superposition (TTS)
  - Performing TTS in TRIOS
  - Thermal analysis techniques to aid method development in TTS
  - Does TTS apply to my sample?
    - Van-Gurp Palmen and Cole-Cole plot
    - WLF and Arrhenius equation
    - Fitting multiple viscoelastic parameters
    - Experimental verification
  - Limitations of TTS

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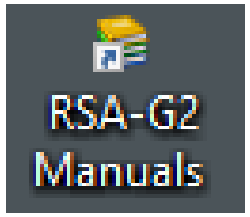

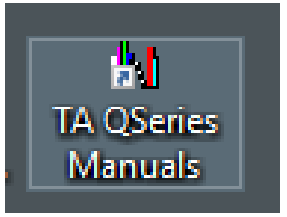

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New Features in Advantage Integrity™

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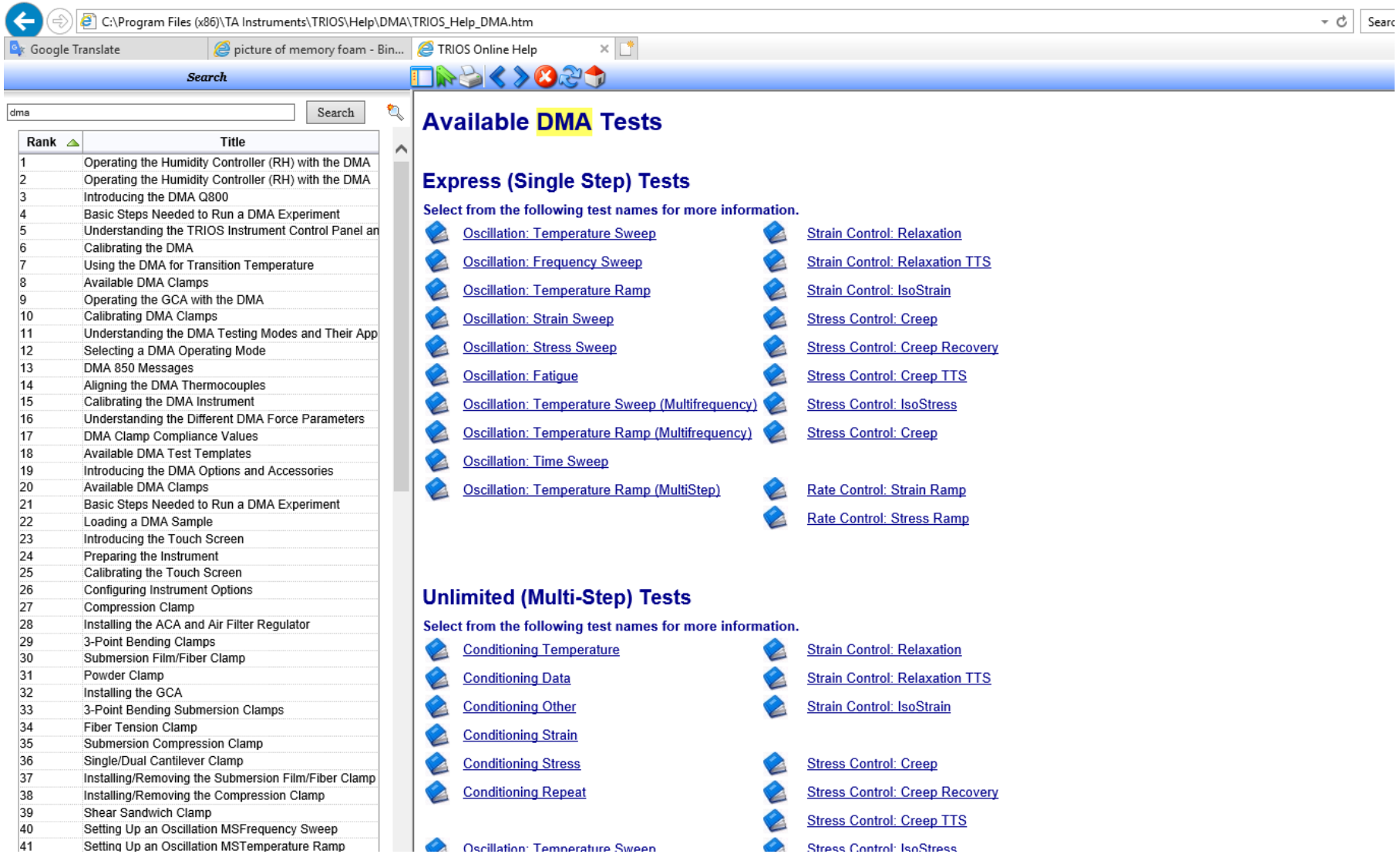

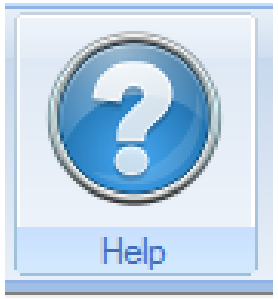

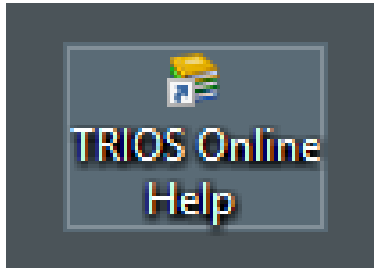

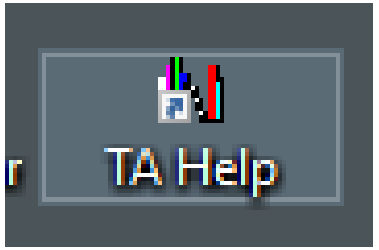

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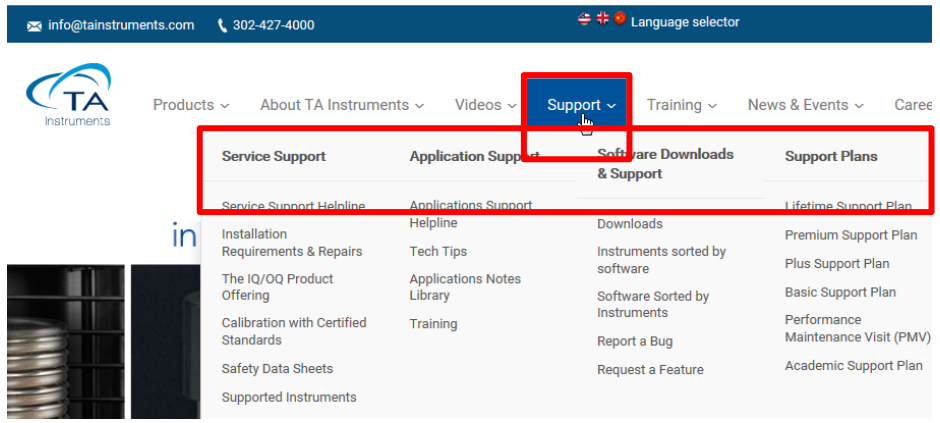

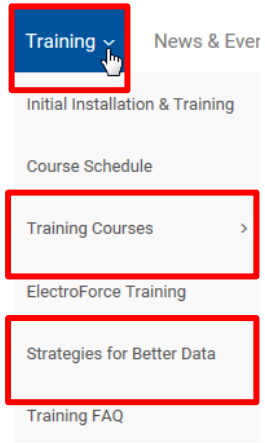

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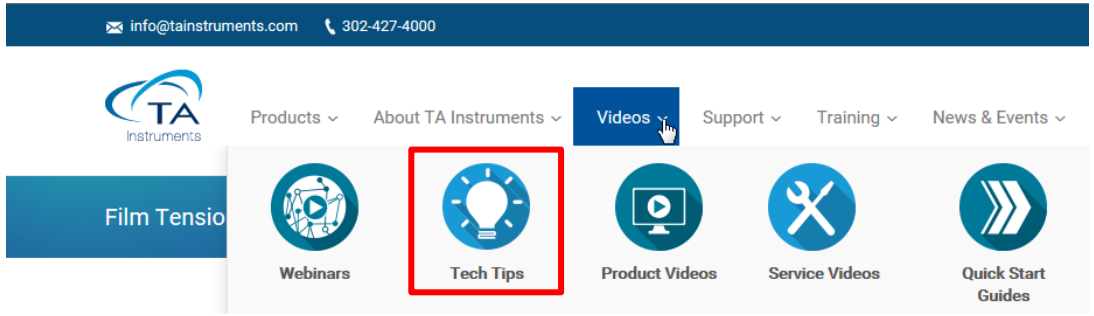

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