Roofing Asphalt Characterization:

John Casola
Malvern Panalytical

Association of Modified Asphalt Producers Meeting
8 February 2018

Roofing asphalt characterization: simplified testing using intelligent rheology

- **This presentation will review some alternate testing techniques which relate to several commonly performed roofing asphalt tests. These methods will be discussed along with actual data presented from automated DSR determination of softening point, penetration, capillary and rotational viscosity. When implemented for quality control, intelligent software will be used for logical decision making which can be used for determination of pass/fail criteria, report generation and escalation decisions for out of spec materials to company procedures.**

- **Other techniques to investigate adhesive pull off, prediction of sag, as well as, high shear to simulate processing conditions will also be presented.**

Data Presented

- **10 Samples of various roofing products**
  - Flux
  - Highly Polymer Modified
  - Oxidized
  - Highly Mineral Filled
- **Broad range of Applications**
  - Mopping
  - Shingles
  - Adhesives
  - Etc…
- **Broad range of Customer Requirements**
  - Most products used internally. Internal QC/QA
  - Some products are provide to other Manufacturers. External QA & Specific Testing
- **Broad range of Material Properties**
  - We will focus on basic, common tests

The Goal

- **Managing a broad range of products with a broad range of testing conditions**
  - Temperature
  - Pass/Fail Limits
  - Viscosity
  - Etc…

- **Making the testing easy, reliable and highly reproducible**
  - Standard Operation Procedures (SOP) driven to ensure exact procedure is performed
  - Test set up automatically by product
  - Improving statistical accuracy

- **Providing immediate feedback with next step solutions**
  - Logical decision making based on results to provide operator ‘what next’ to policy

Tests Performed For This Presentation

- Viscosity
- Penetration
- Softening Point
- Pull-Off

- Notes on Units:
- While many manufacturers use Imperial (CGS) units, such as, Fahrenheit, Poise and Inches, I’ve chosen to present most thing in SI units.

- Temperature in Celsius.
- Viscosity will be in Pas
- Frequency in Hertz

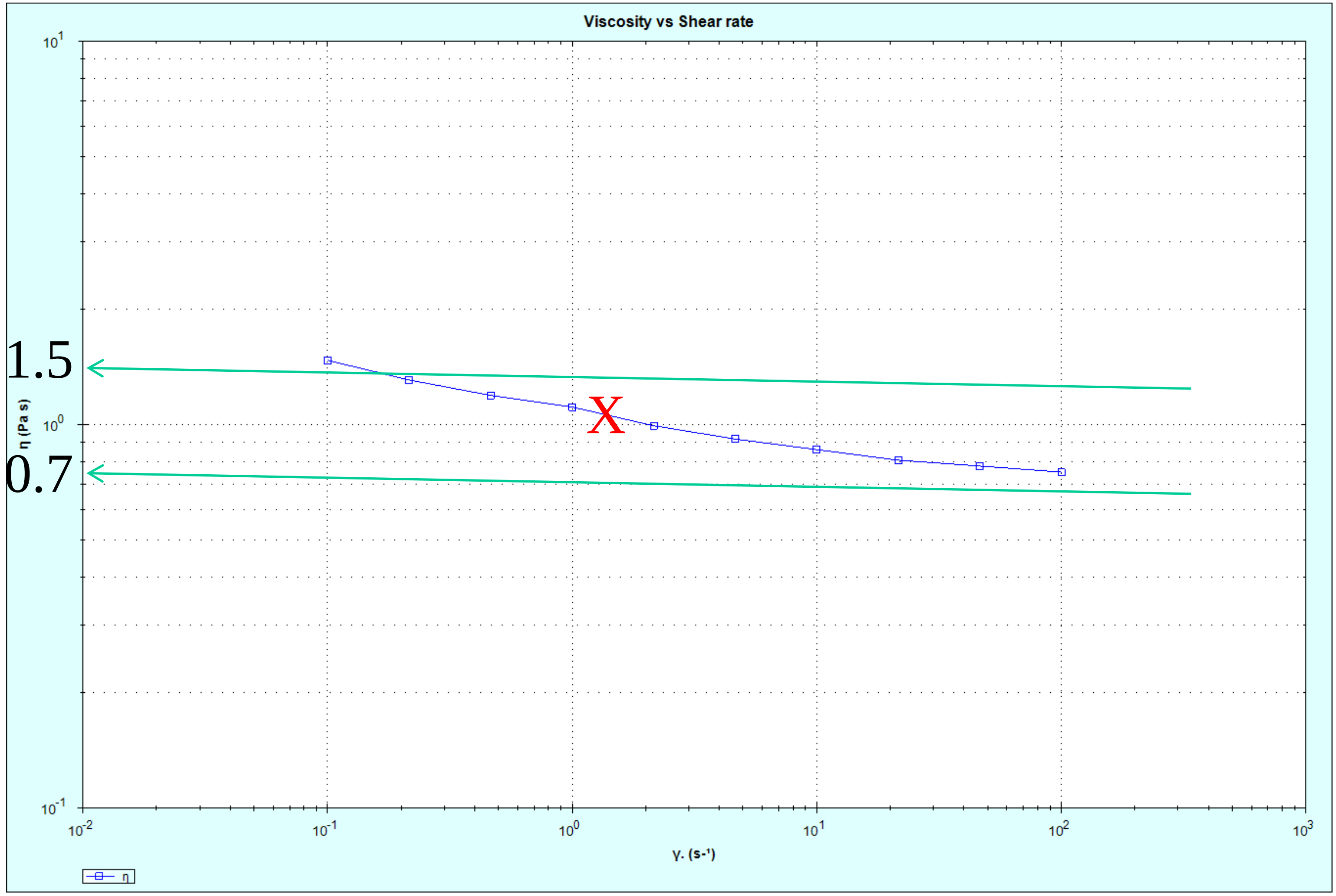
Tests Performed

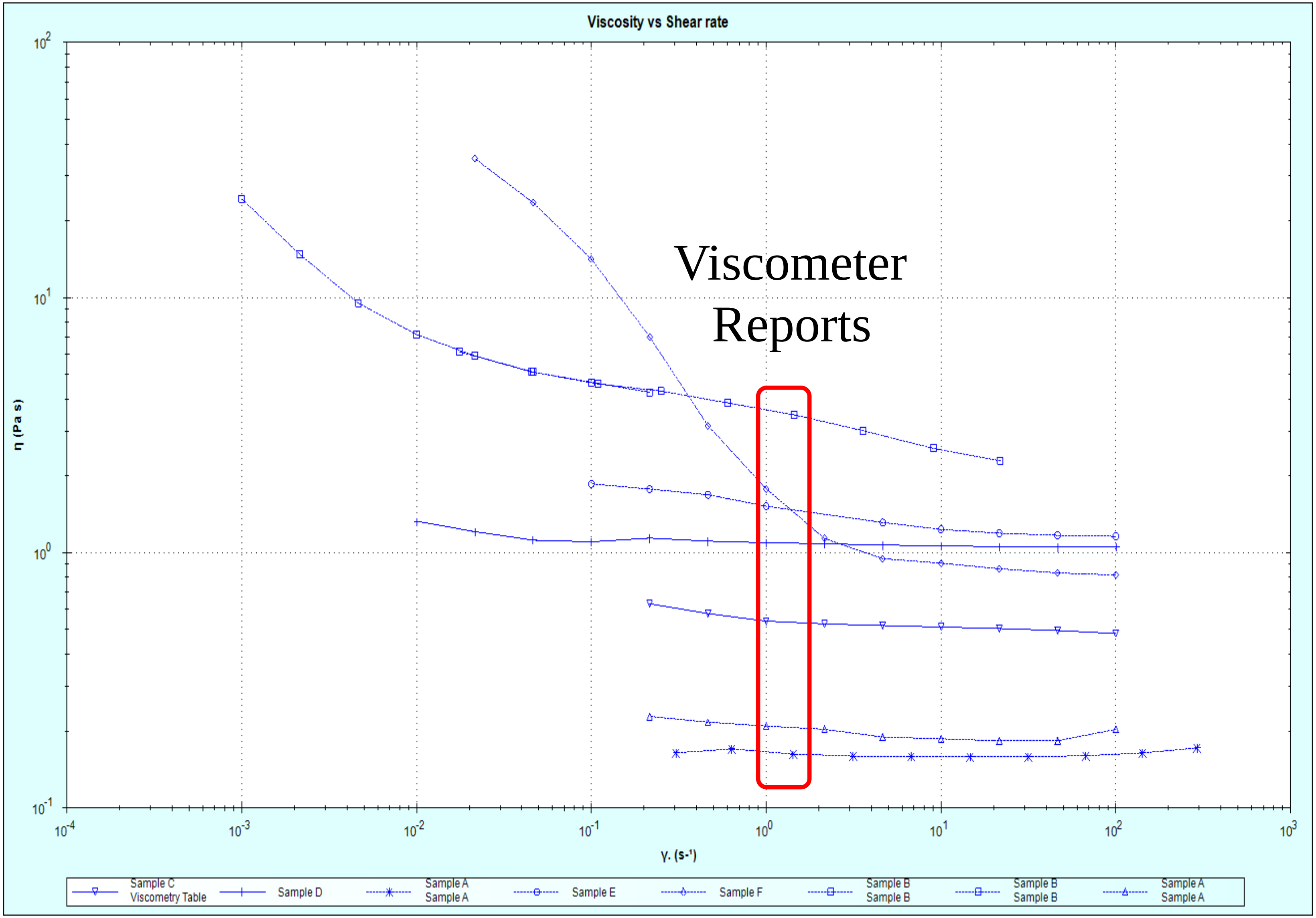
- **Viscosity, at various specified temperatures & acceptance limits**
  - 204ºC, 190ºC, 177ºC, 163 ºC, 149 ºC, 125 ºC & 0 ºC
- **Penetration at 25ºC & 0ºC**
- **Softening Point**
  - Measured with static force loading
  - Measured with dynamic force loading
- **Pull-Off**
  - 0.1 inch/min
  - 1.0 inch/min
  - 12 inch/min
- **High Shear Viscosity for discussion**

Rotational Viscosity

- **Very common test measurement**
- **Performed at a single rotation rate**
- **Performed at a single temperature**
- **Performed of a specific period of time**
- **Question: What does it really tell us about the product?**

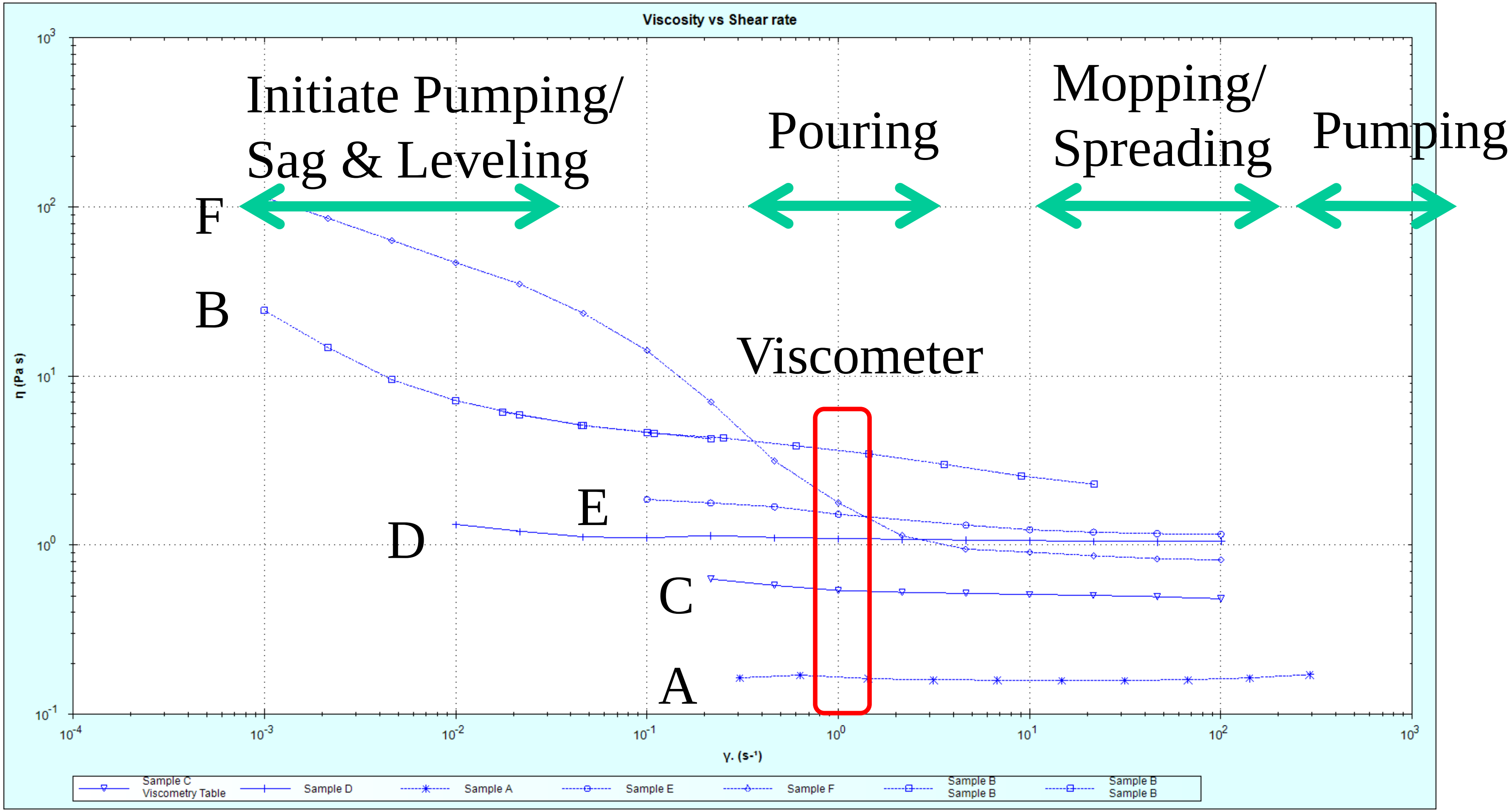
Sample H; 1012mPas @ 163C



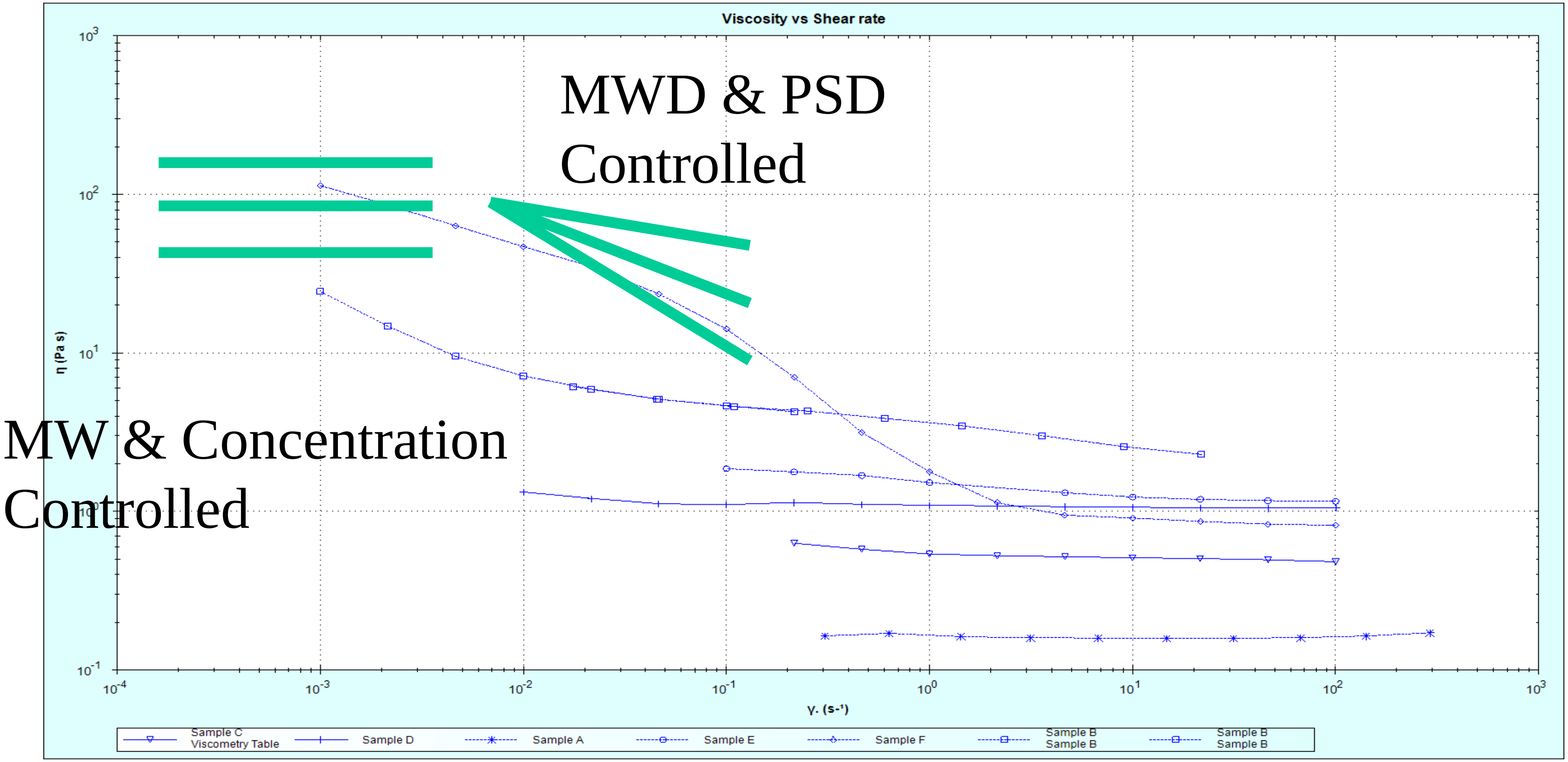


Viscosity vs Shear rate
Viscometer Reports
η (Pa s)
γ. (s-¹)
Sample C Viscometry Table
Sample D
Sample A Sample A
Sample E
Sample F
Sample B Sample B
Sample B Sample B
Sample A Sample A

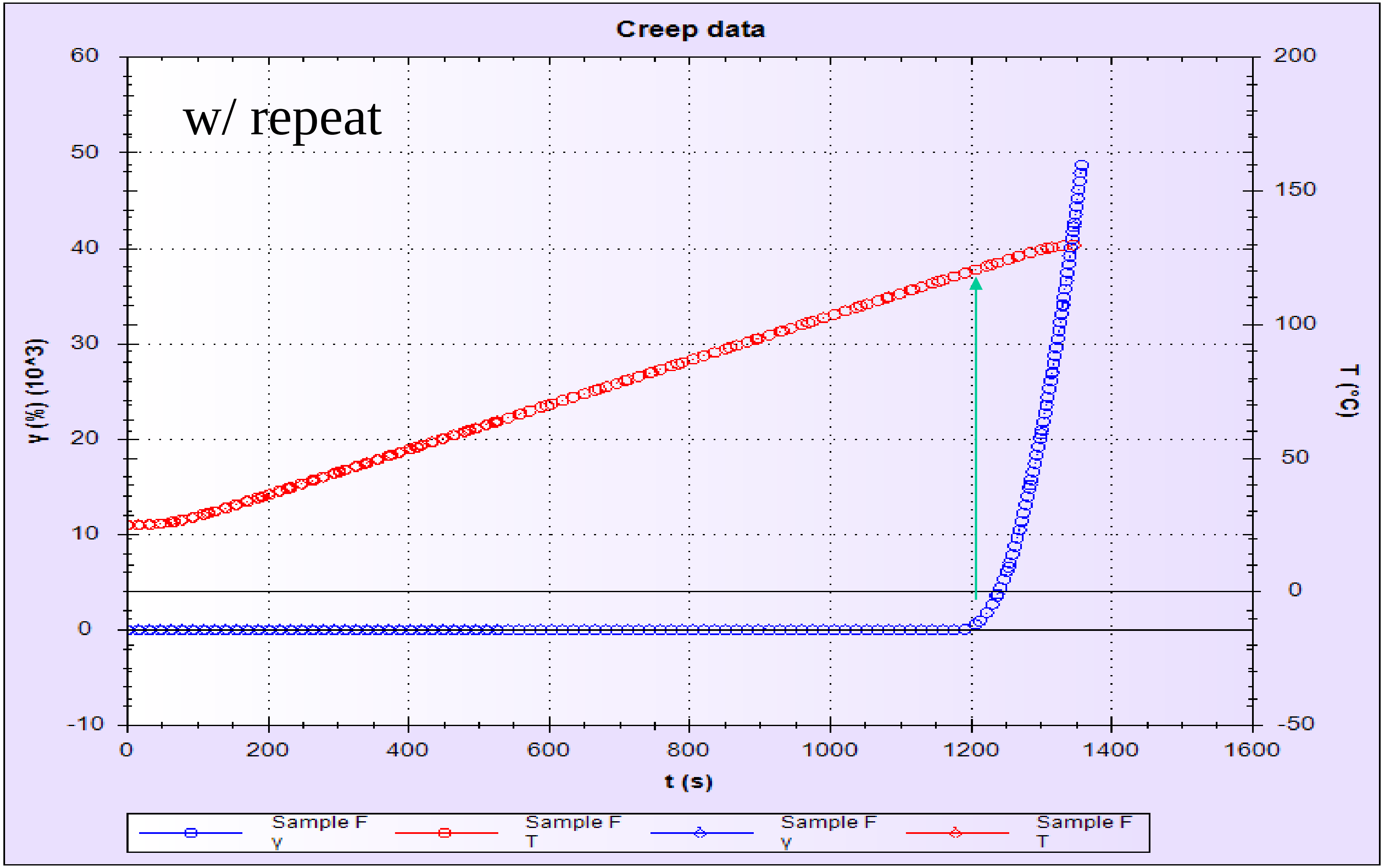
DSR Shear Viscosity



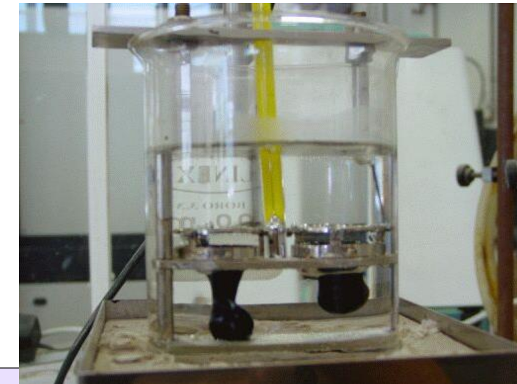
DSR Shear Viscosity
Tuminello & Shaw for polymer MW & MWD
Krieger Dougherty & Einstein Equation PS & PSD

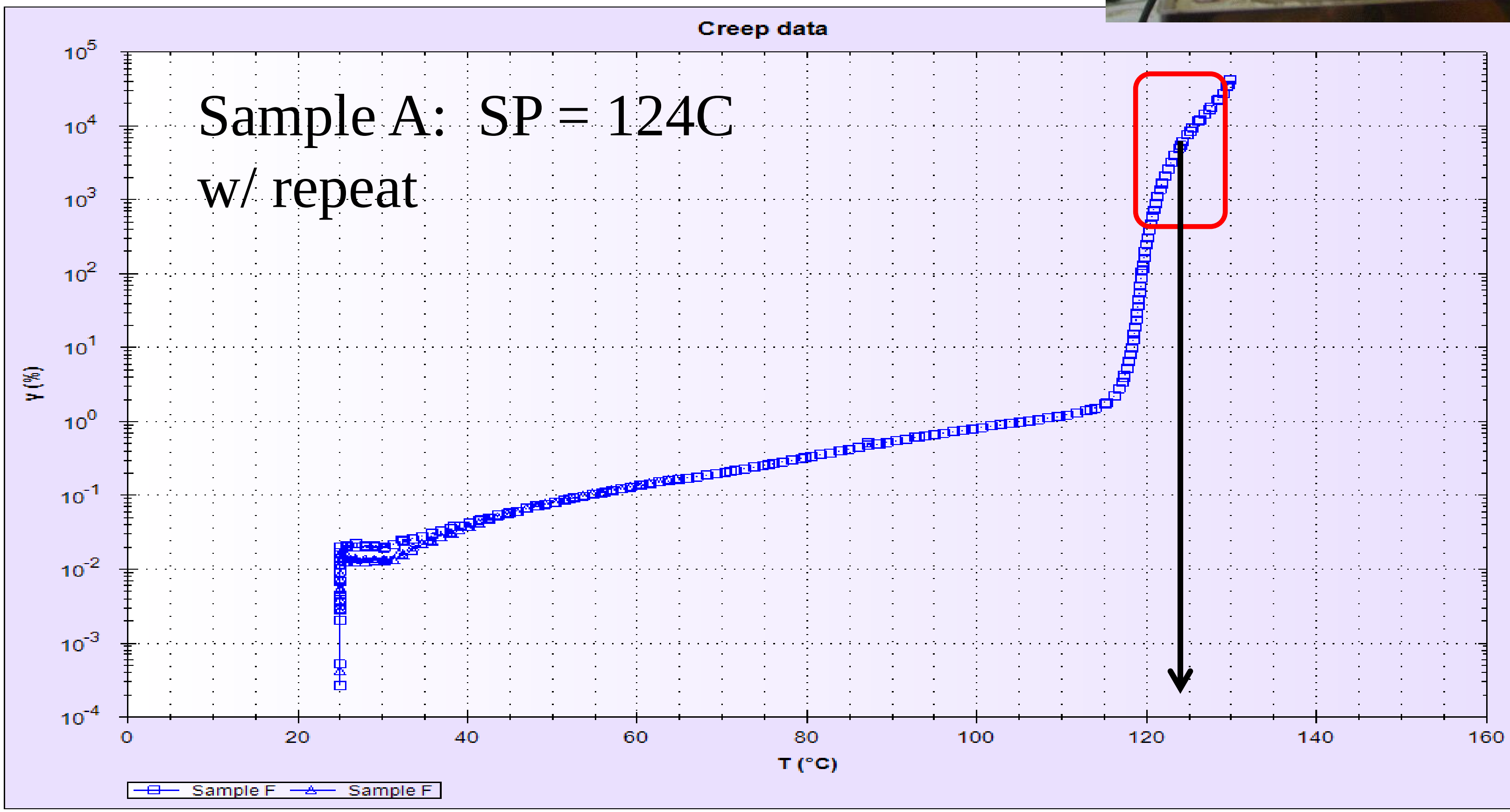


Static Load to Monitor Softening

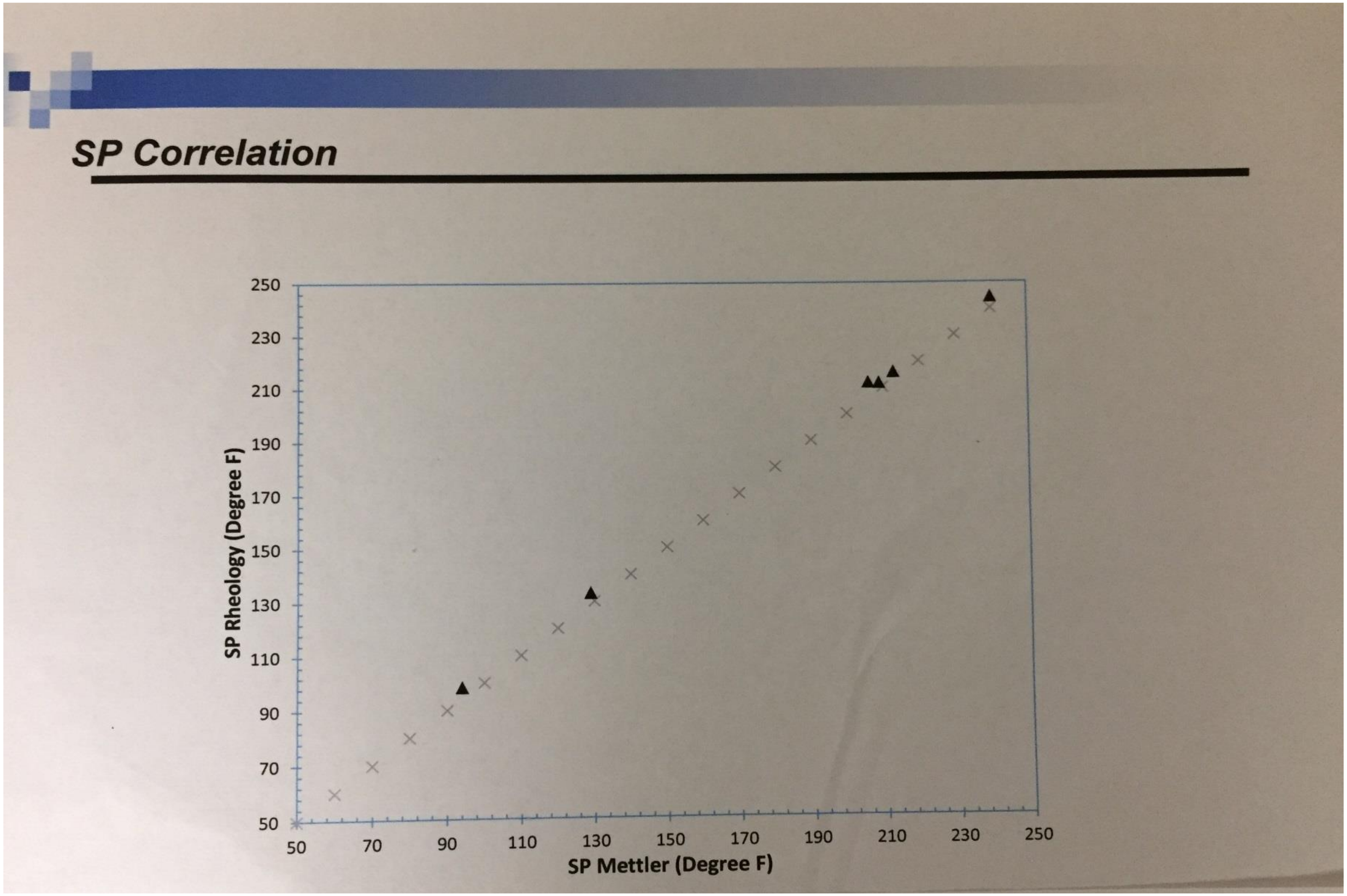


Static Determination of Softening Point

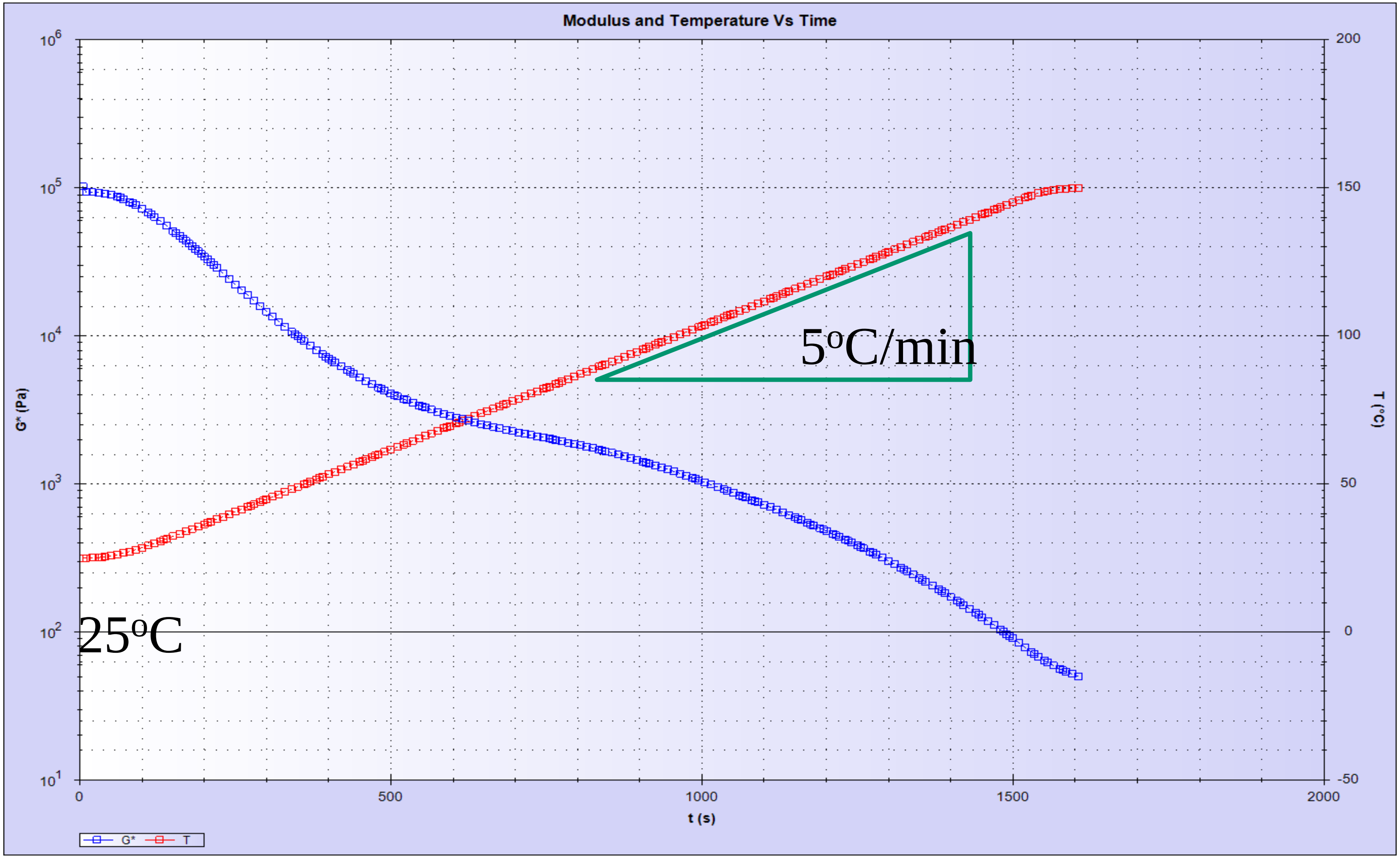




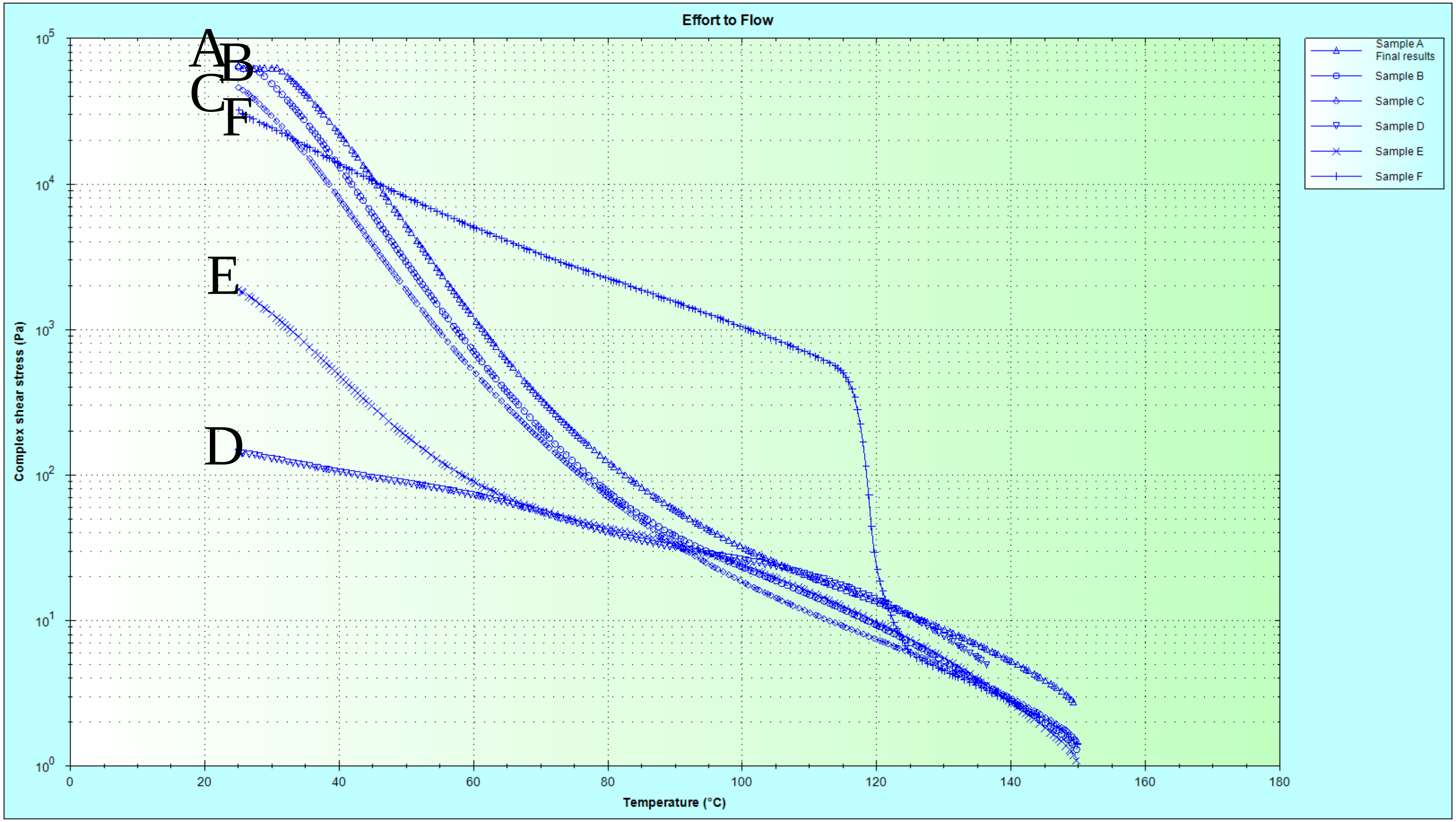
Oxidized Roofing Asphalt using Static Load ~2009



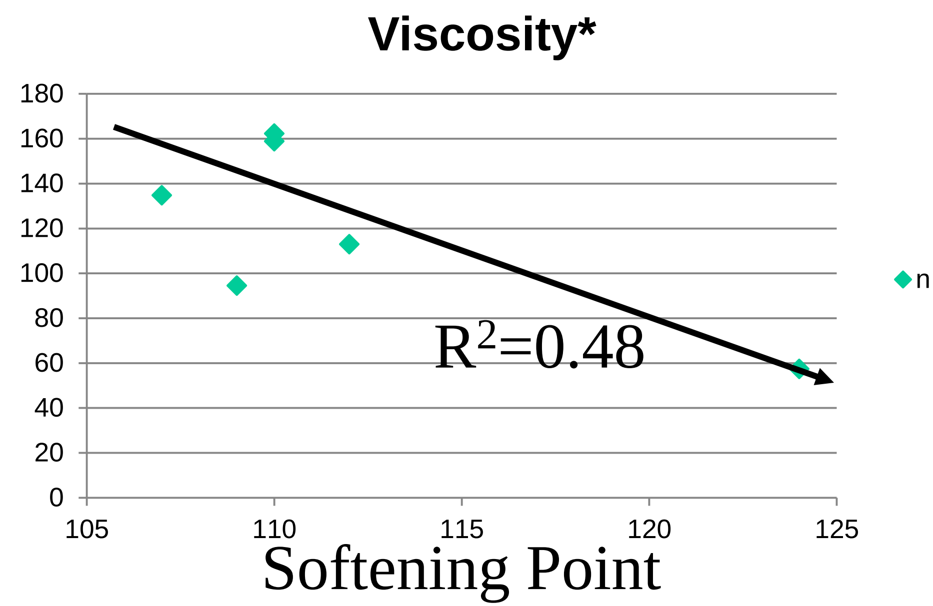
Dynamic Determination of Softening Point

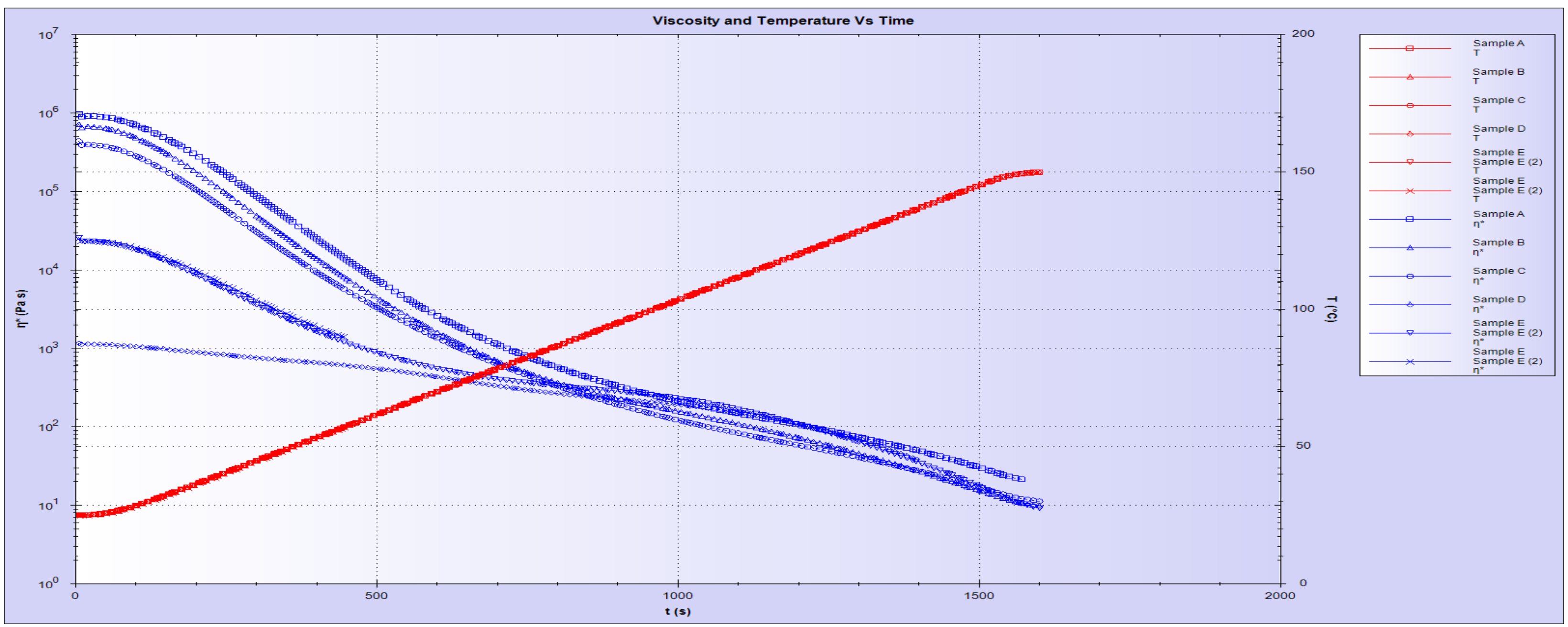


Comparison of Effort to Flow; Interesting Behavior?

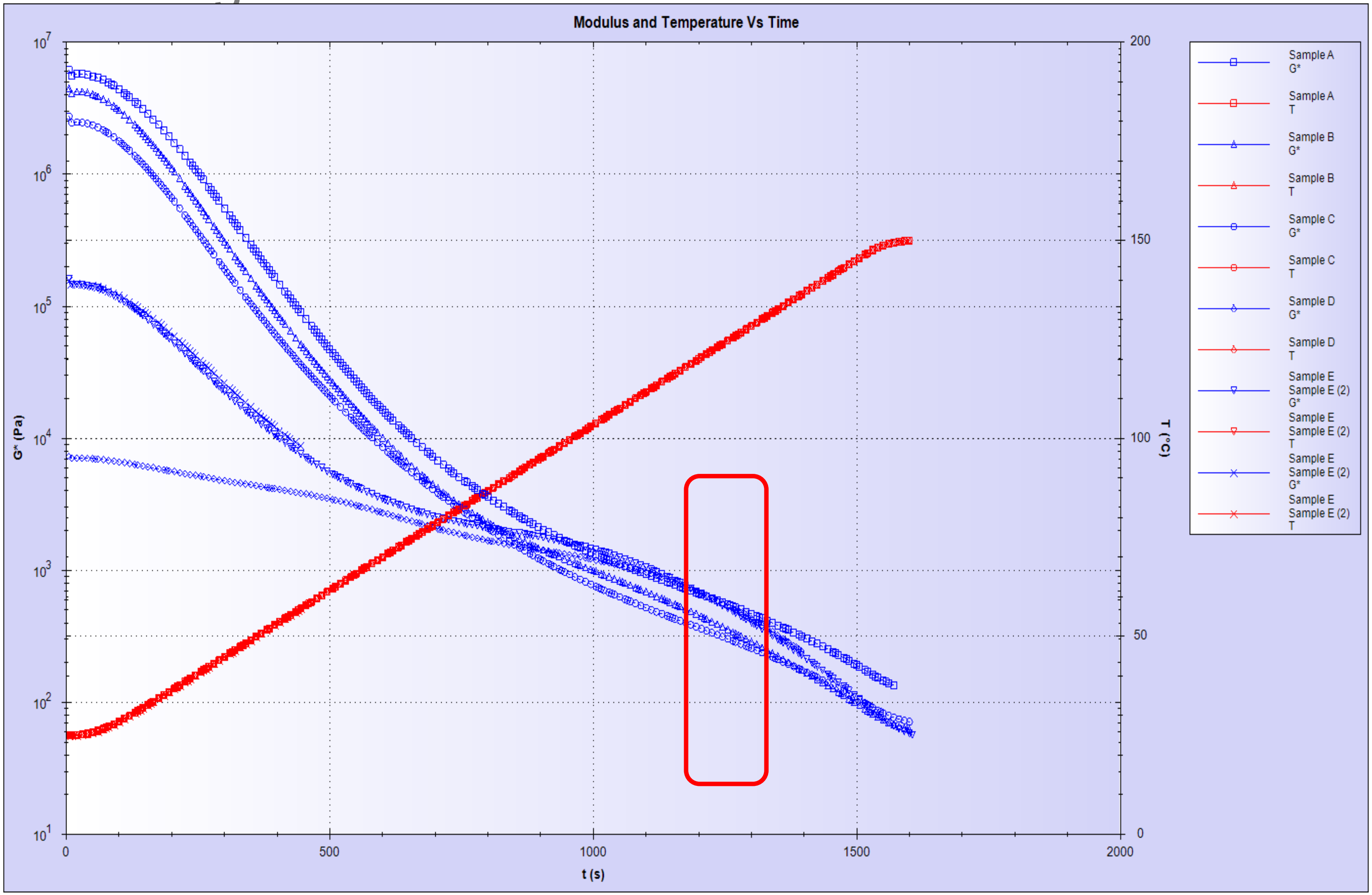


Viscosity* to Reported Softening Point (poor fit)



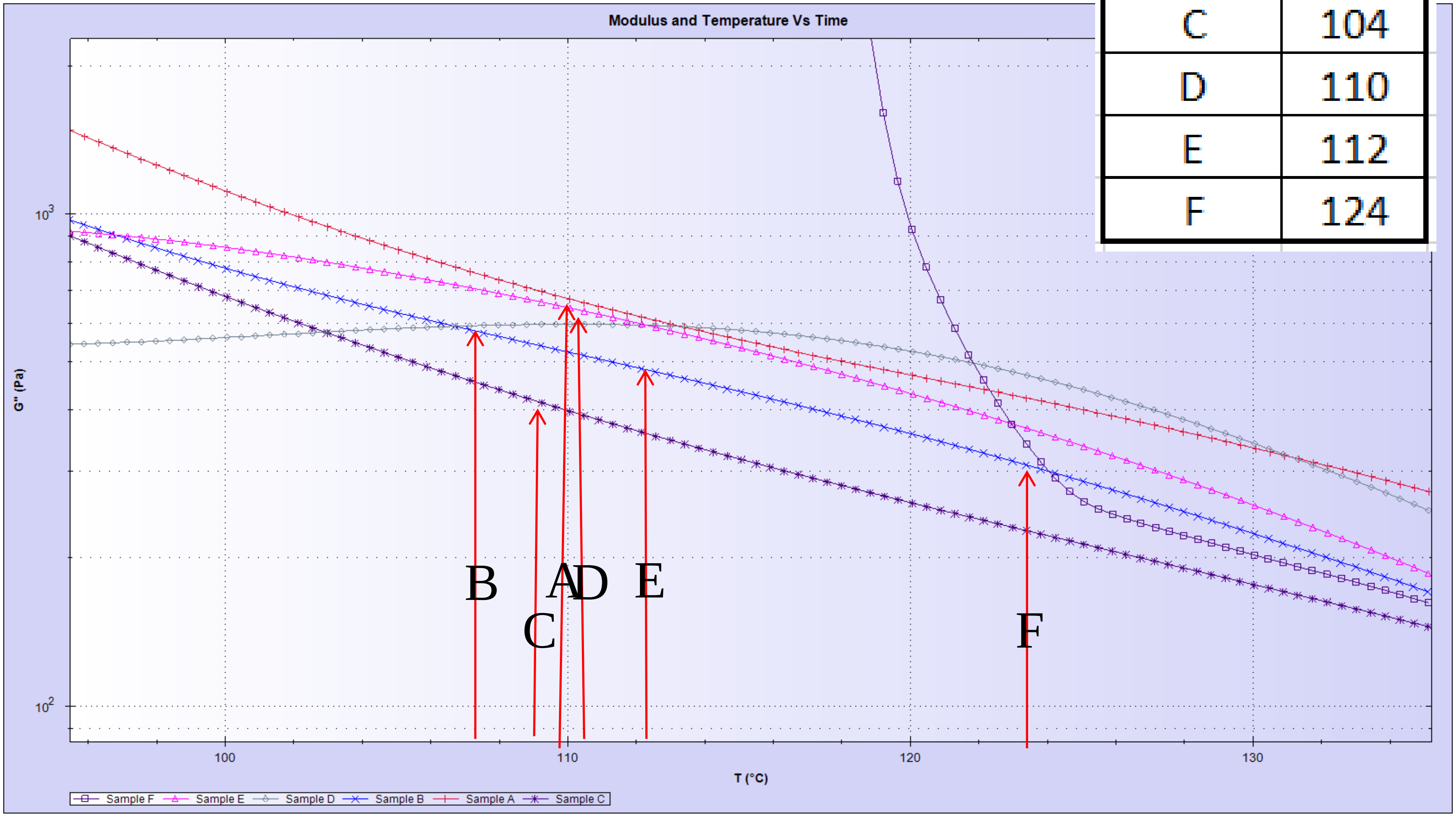


Modulus to Reported Softening Point



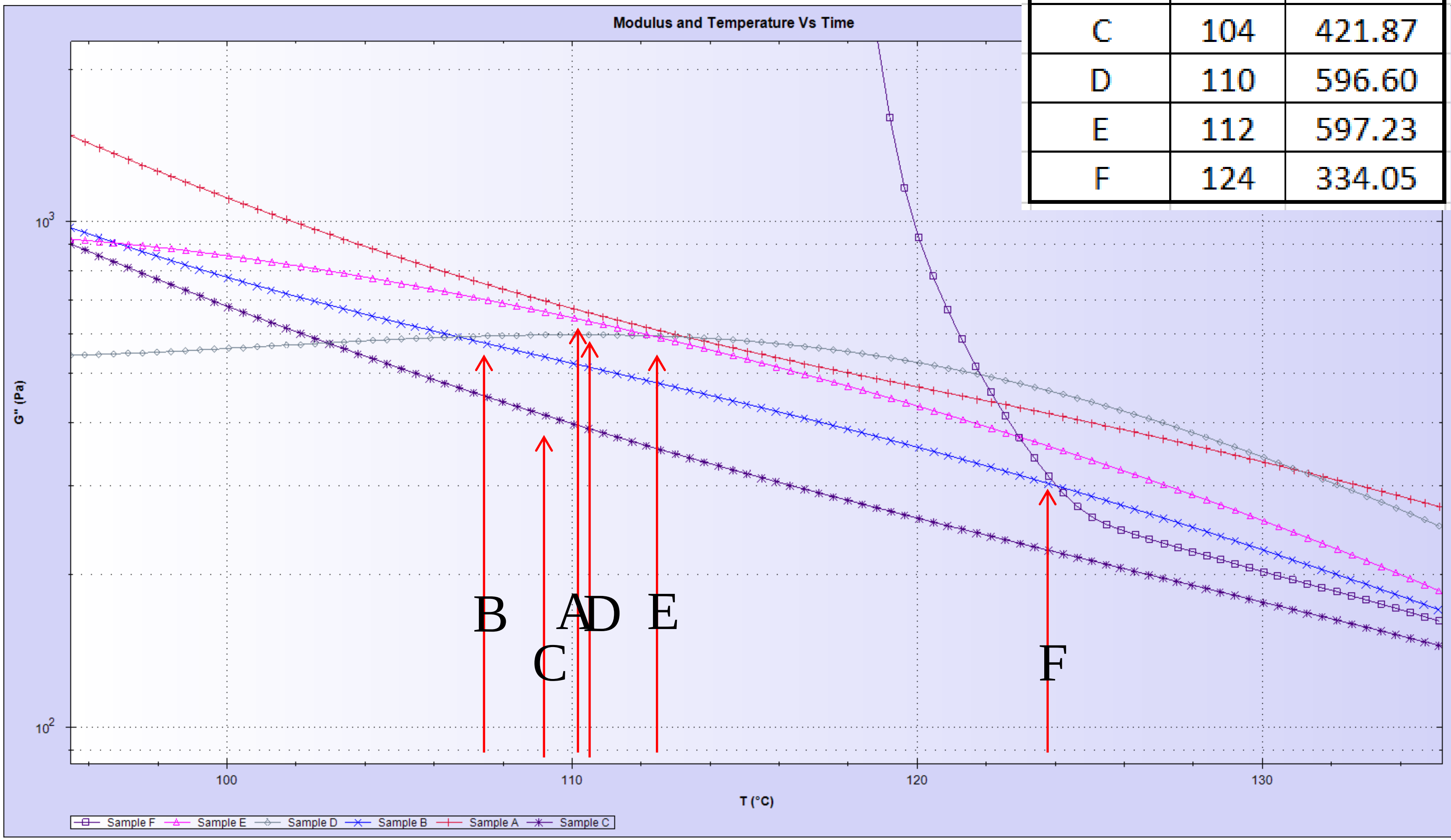
Loss Modulus (G”) to Reported Softening Point

Sample	SP
A	110
B	107
C	104
D	110
E	112
F	124

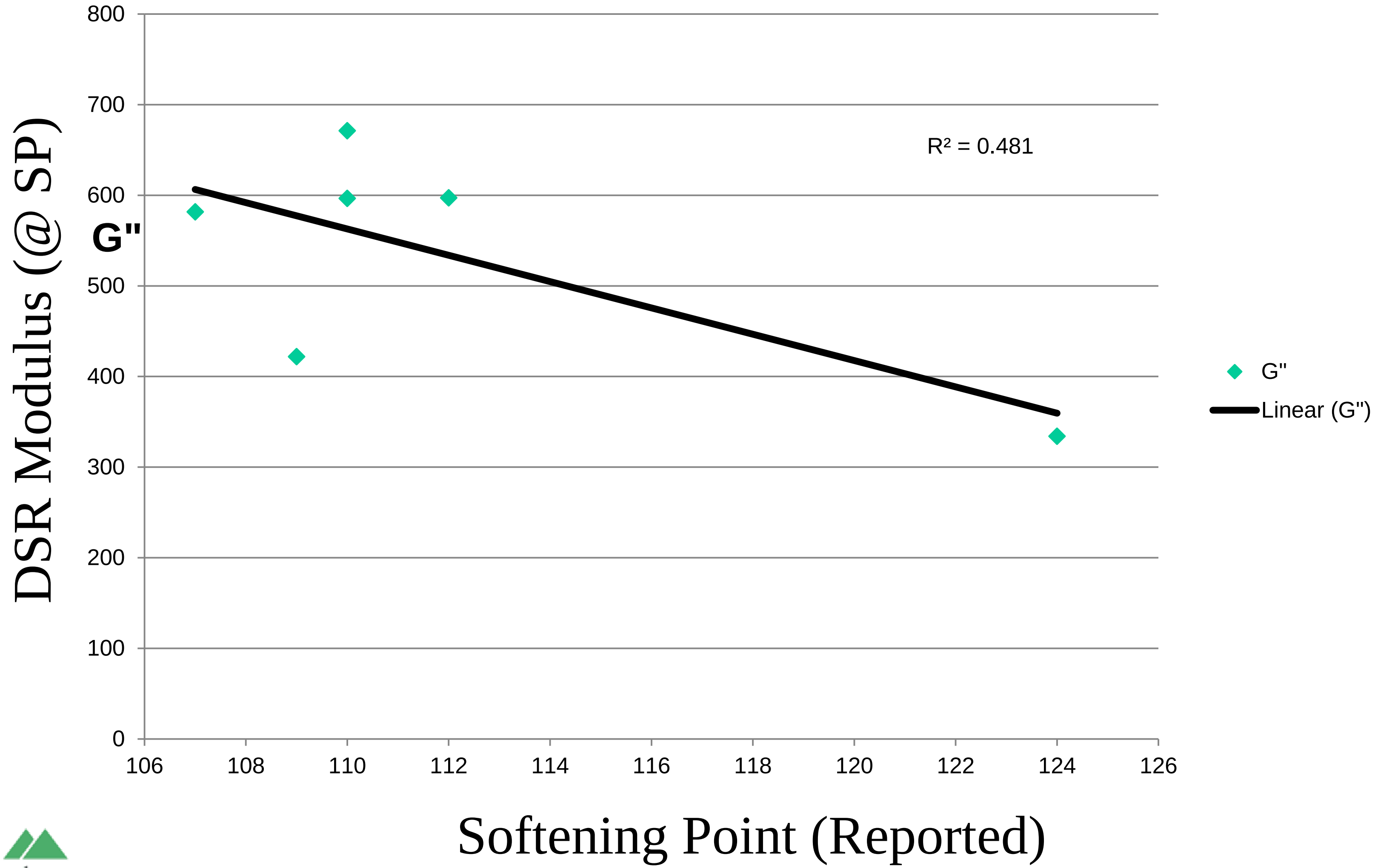


Loss Modulus (G”) to Reported SP

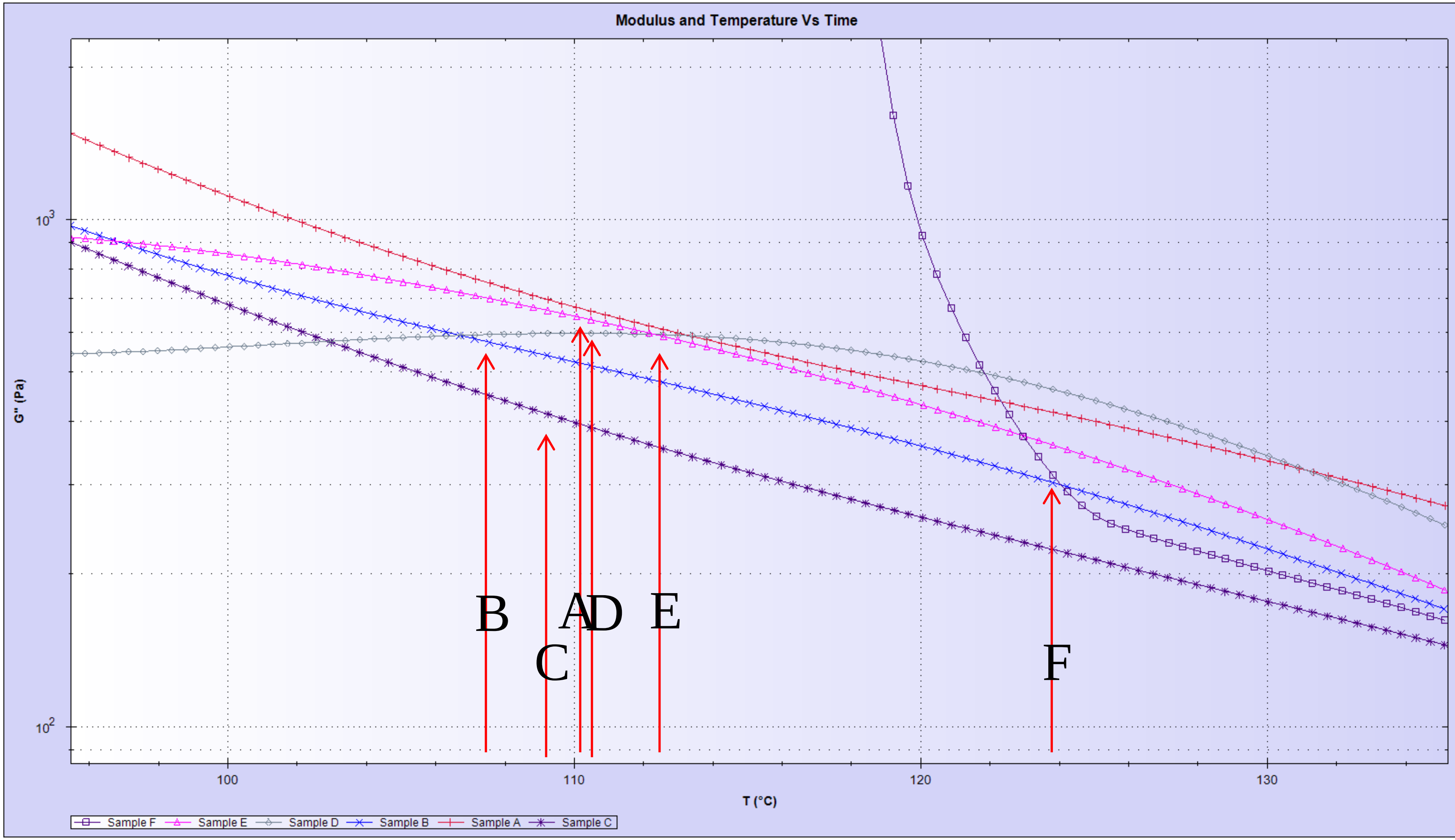
Sample	SP	G"
A	110	671.15
B	107	581.78
C	104	421.87
D	110	596.60
E	112	597.23
F	124	334.05



Modulus to Softening Point Direct Correlation (poor fit)

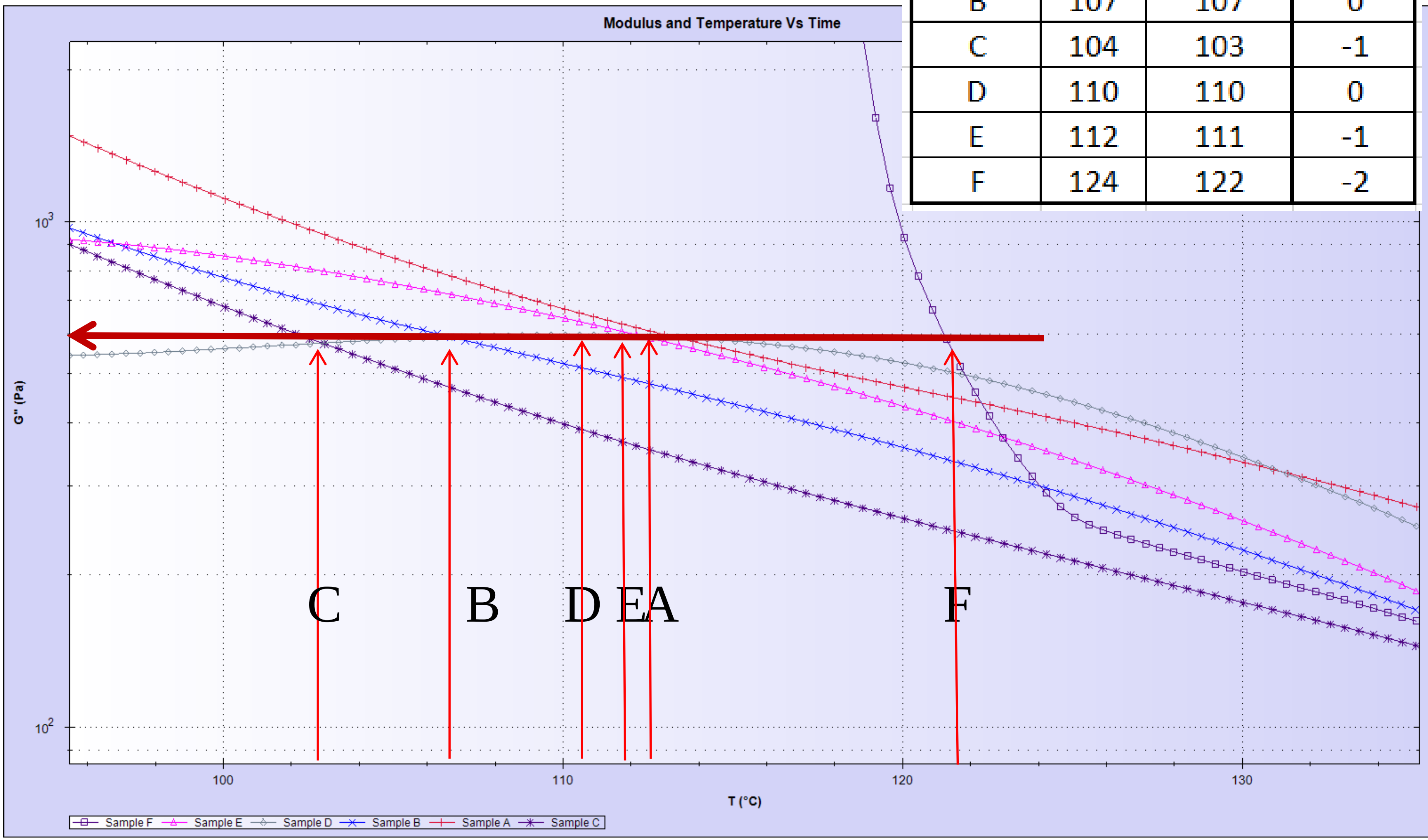


Loss Modulus (G”) to Reported Softening Point

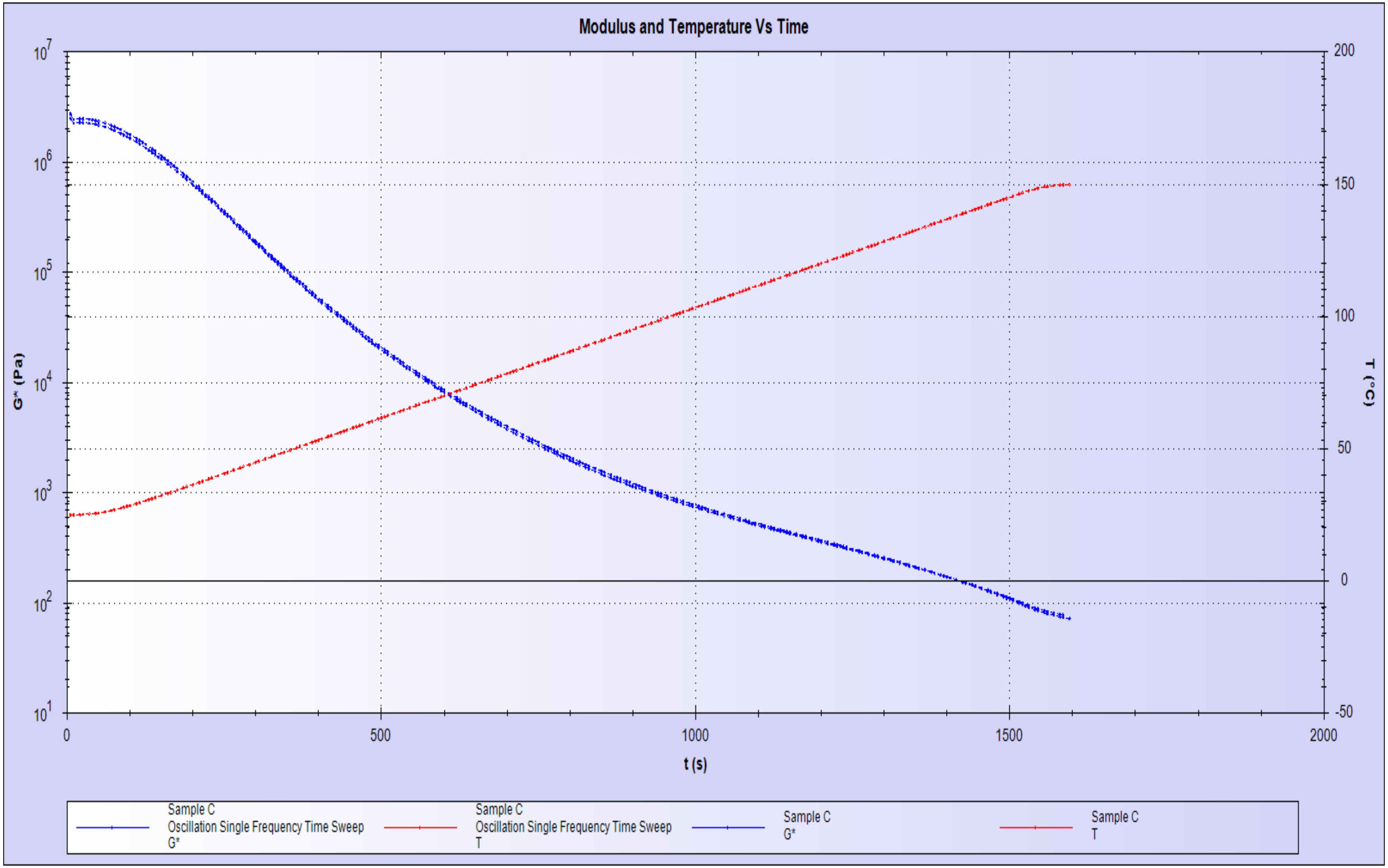


Adjust SP to Equal Modulus G" is an EXCELLENT FIT

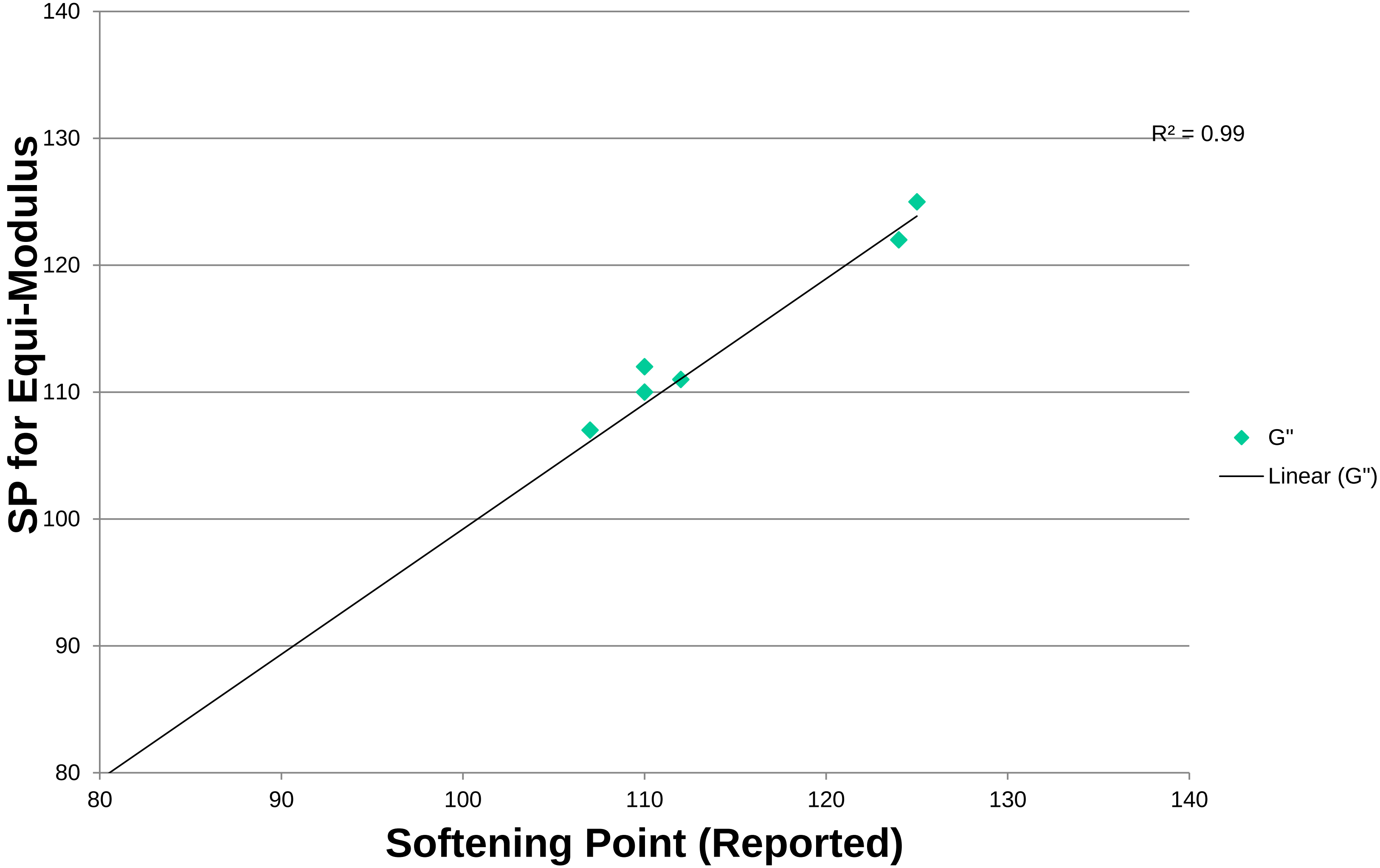
Sample	SP	G"	Diff
A	110	112	2
B	107	107	0
C	104	103	-1
D	110	110	0
E	112	111	-1
F	124	122	-2



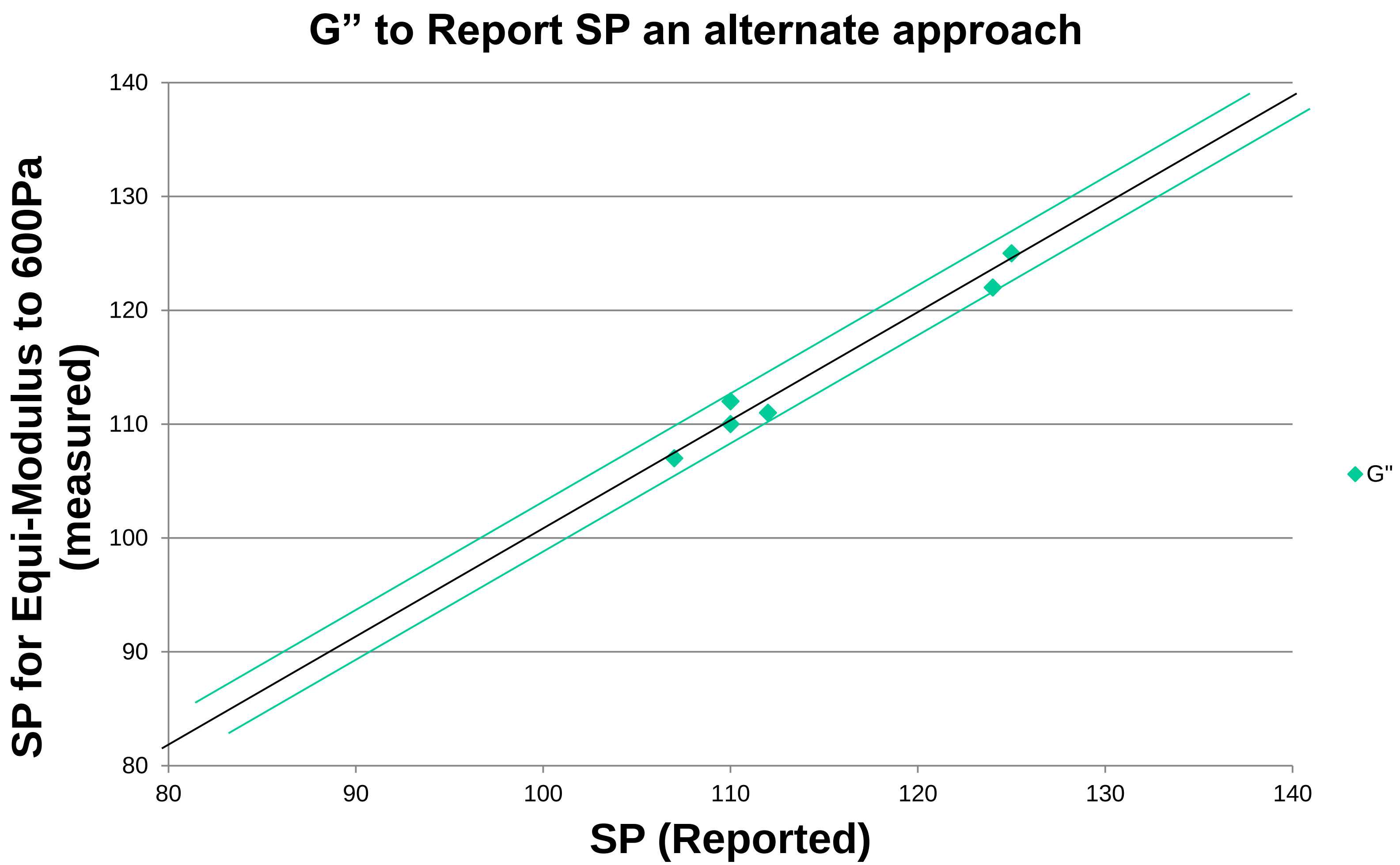
Sample C w/ Repeat; Results within 1%



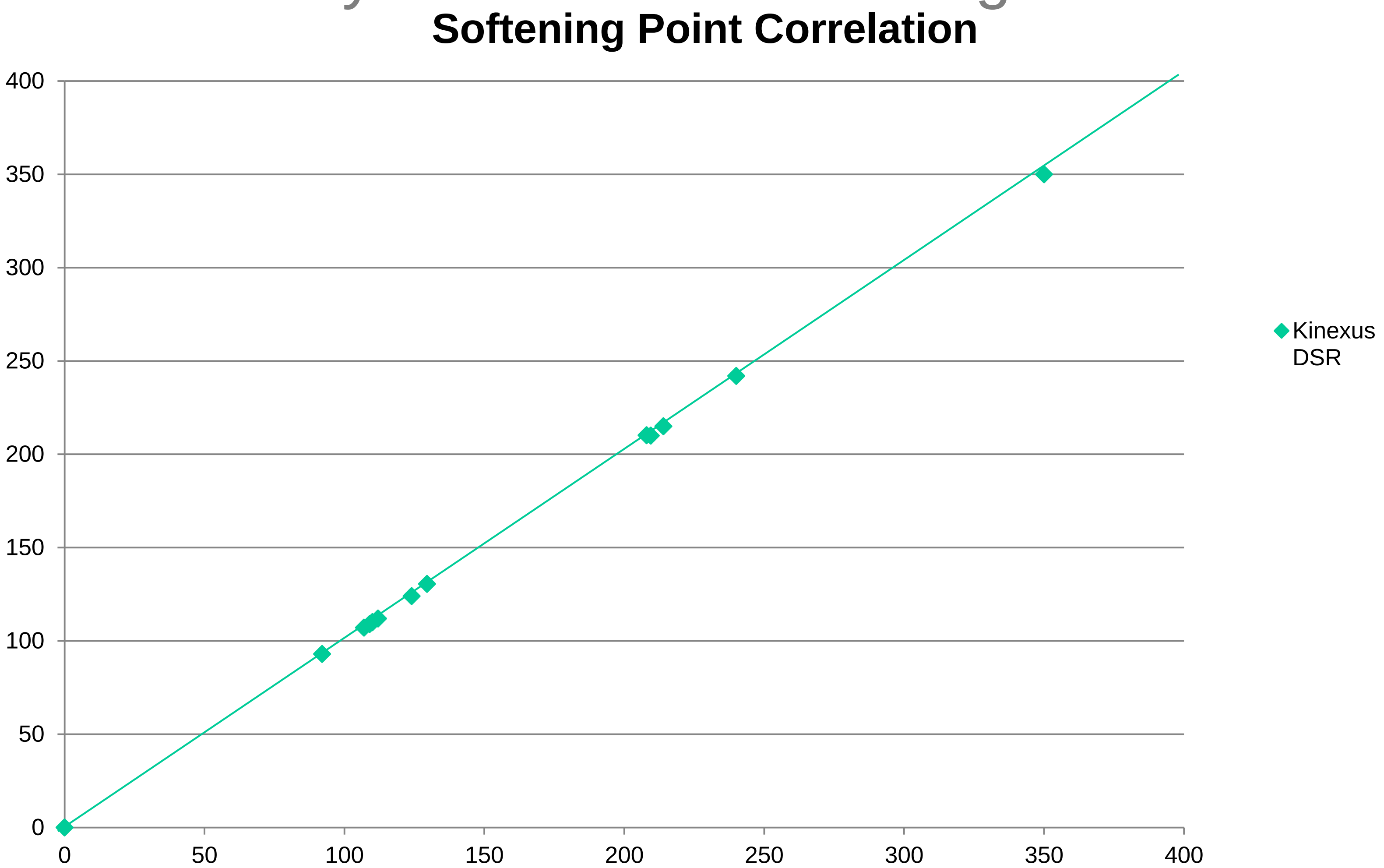
DSR Correlation to Softening Point



Line of Equality +/-2^{o}C

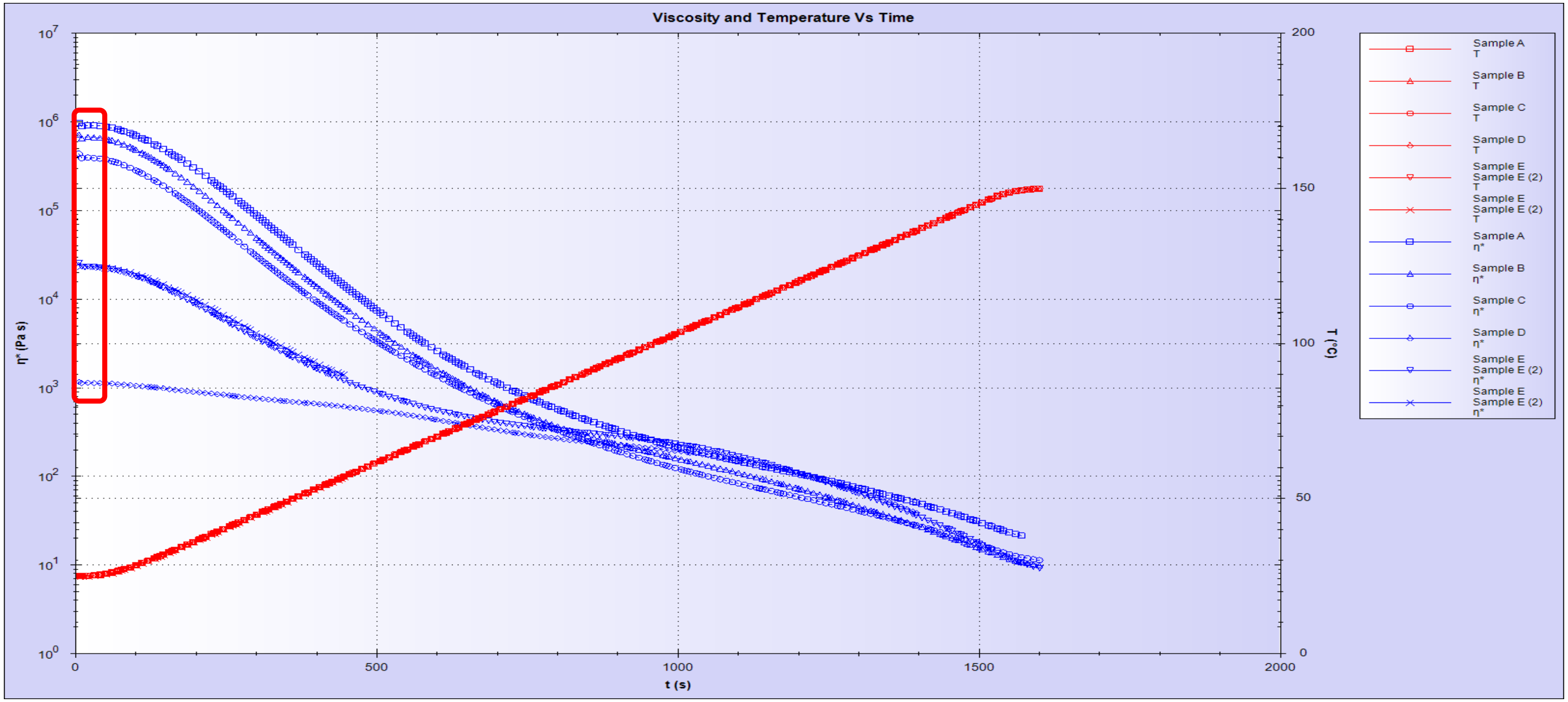


DSR vs. SP
combined dynamic & static loading

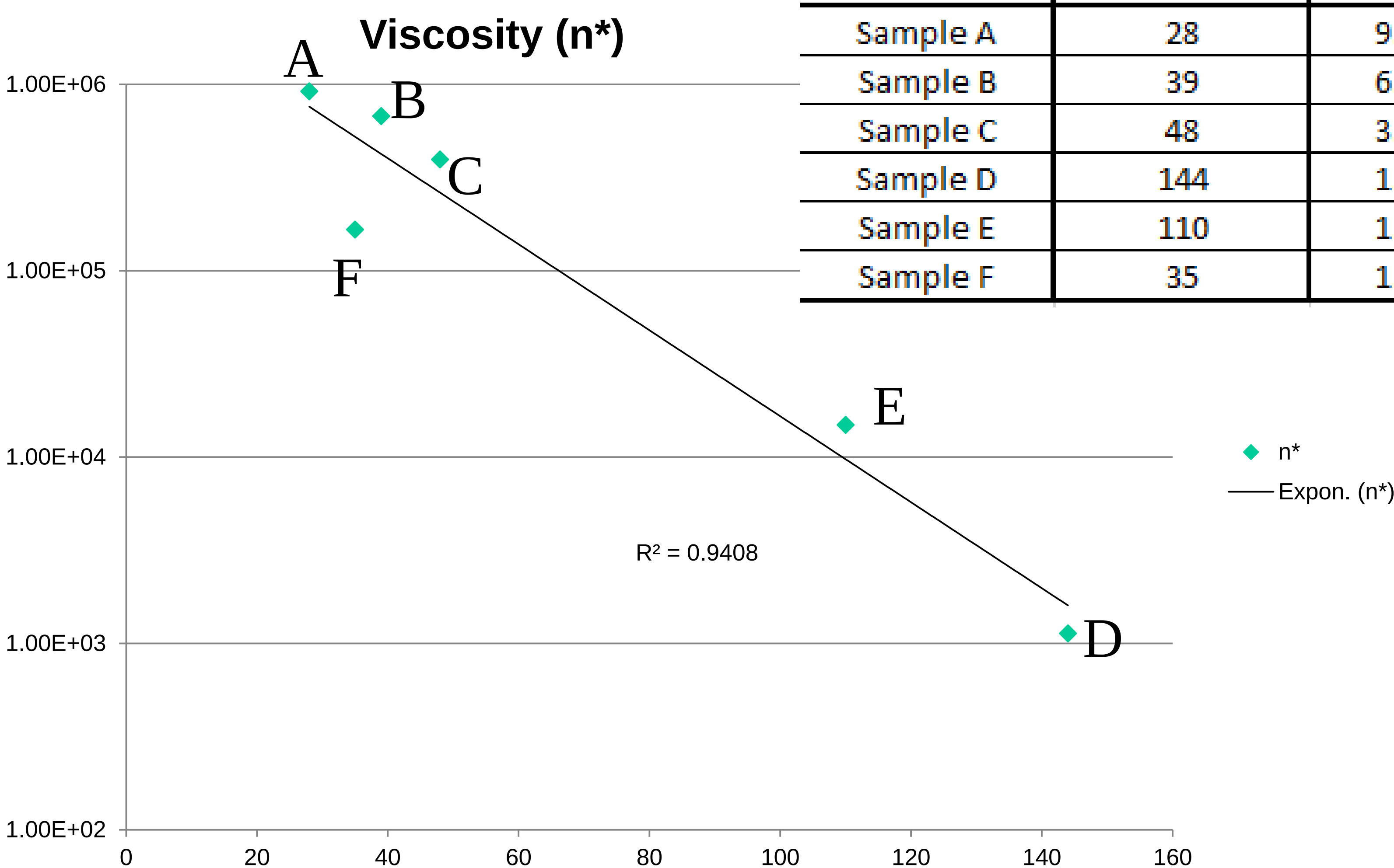


DSR Viscosity* to Reported Penetration

	Pen	n*
Sample A	28	9.18E+05
Sample B	39	6.76E+05
Sample C	48	3.96E+05
Sample D	144	1.13E+03
Sample E	110	1.49E+04
Sample F	35	1.66E+05

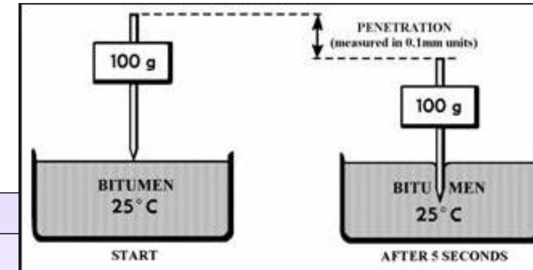


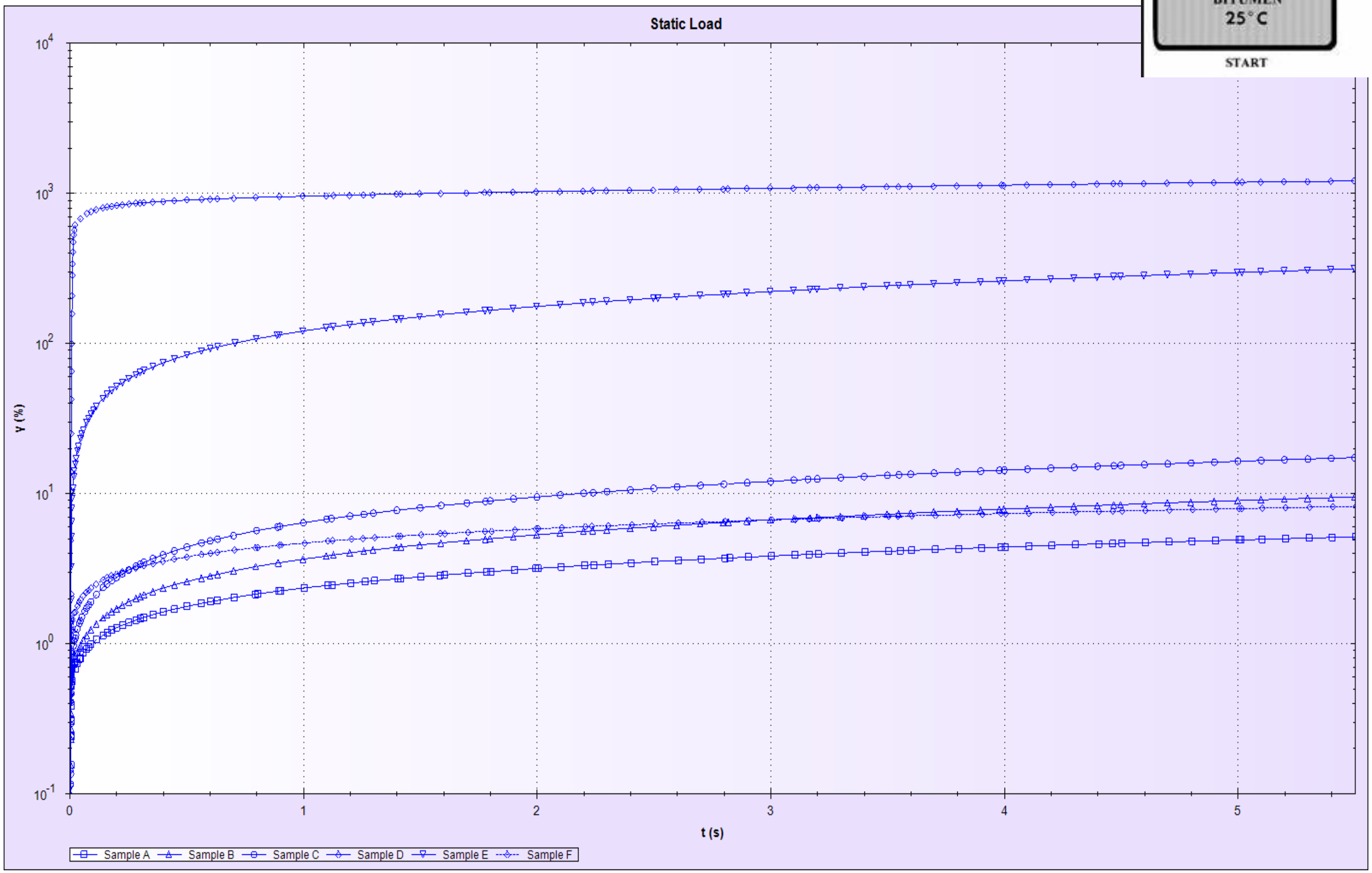
Relationship of Viscosity to Pen



	Pen	n*
Sample A	28	9.18E+05
Sample B	39	6.76E+05
Sample C	48	3.96E+05
Sample D	144	1.13E+03
Sample E	110	1.49E+04
Sample F	35	1.66E+05

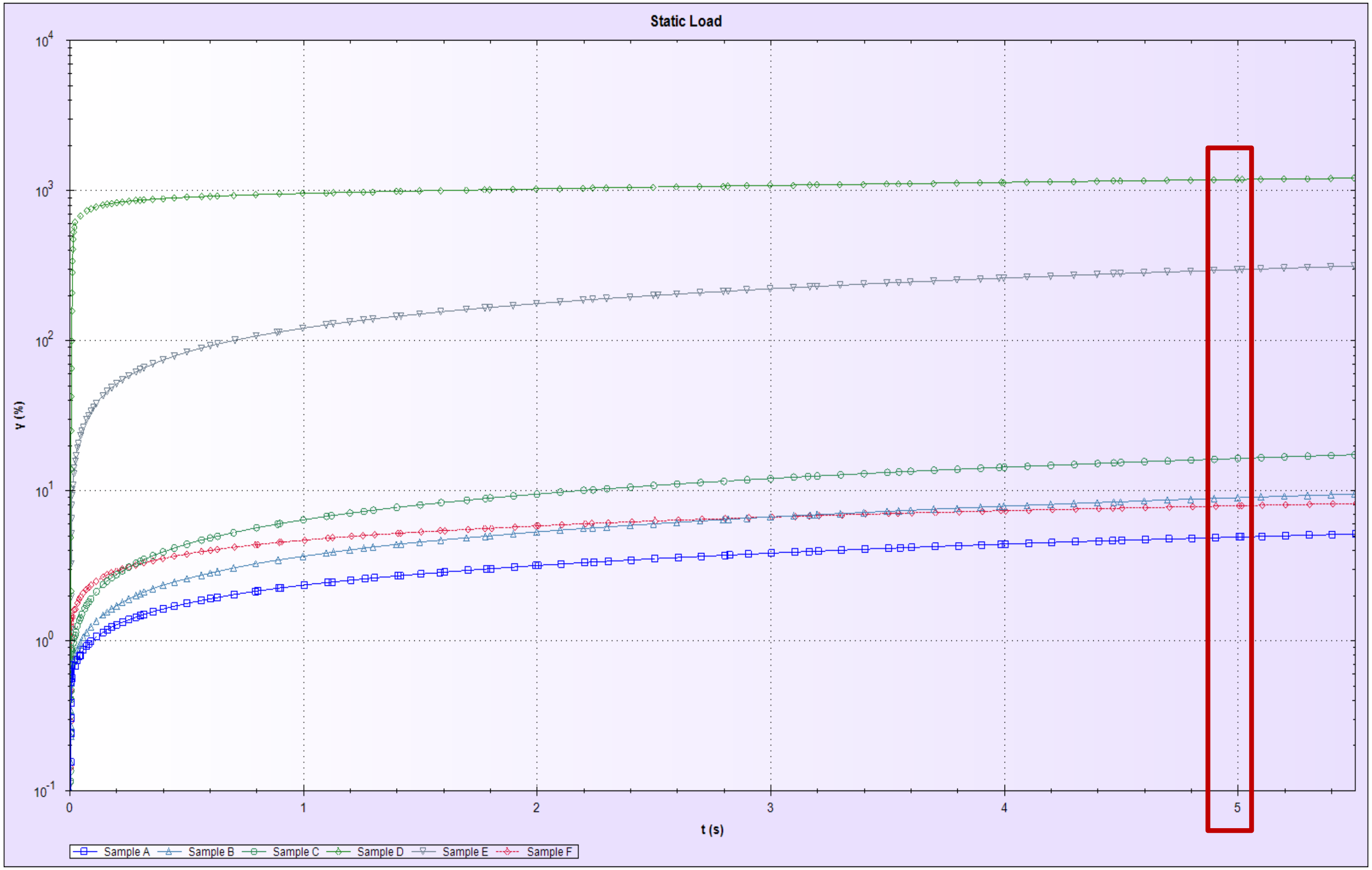
Let's Look at Static Load to Penetration





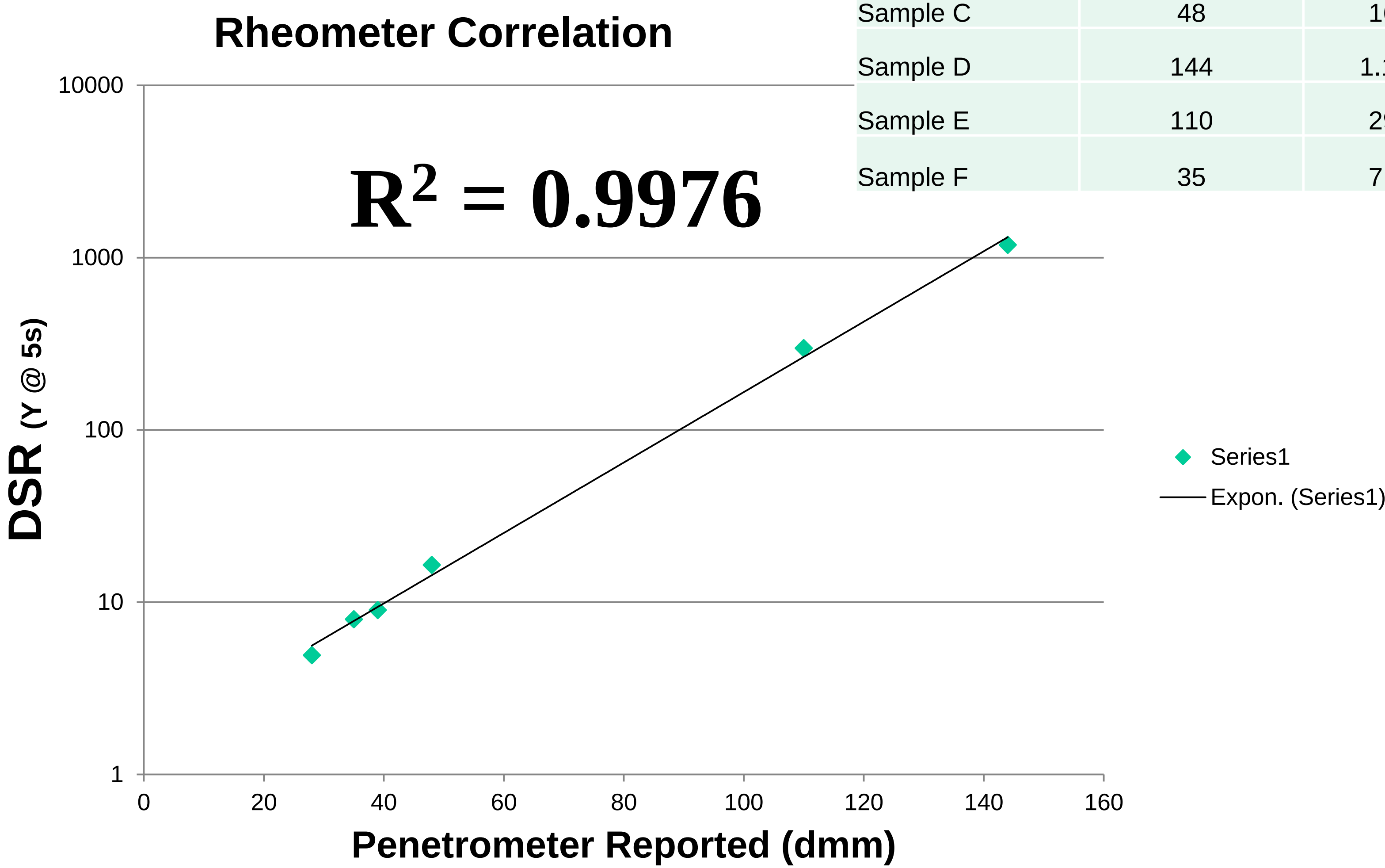
D
E
C
B
F
A

Relationship of Creep Strain at 5s



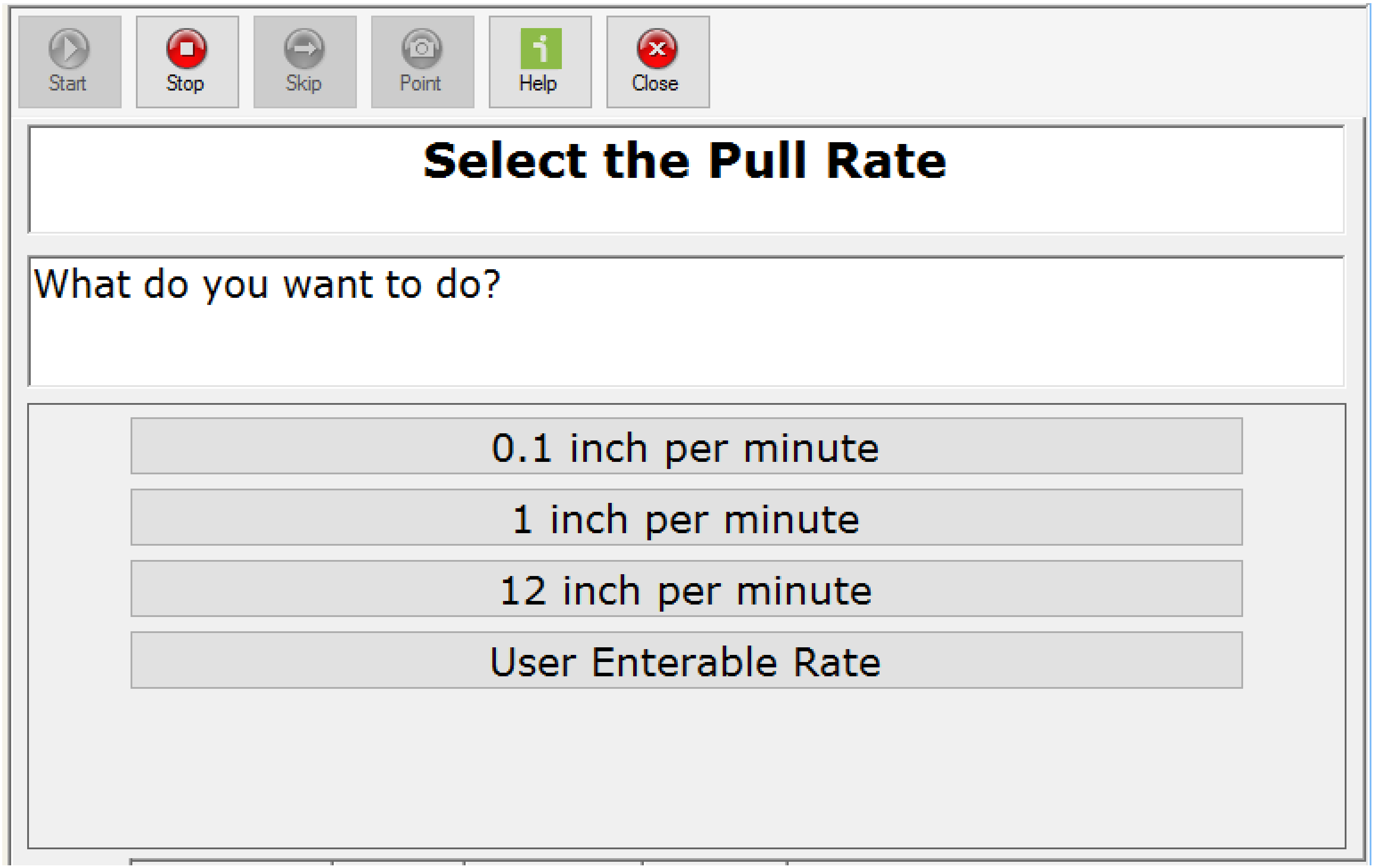
	Pen
D	144
E	110
C	48
B	39
F	35
A	28

Static Loading to Penetration

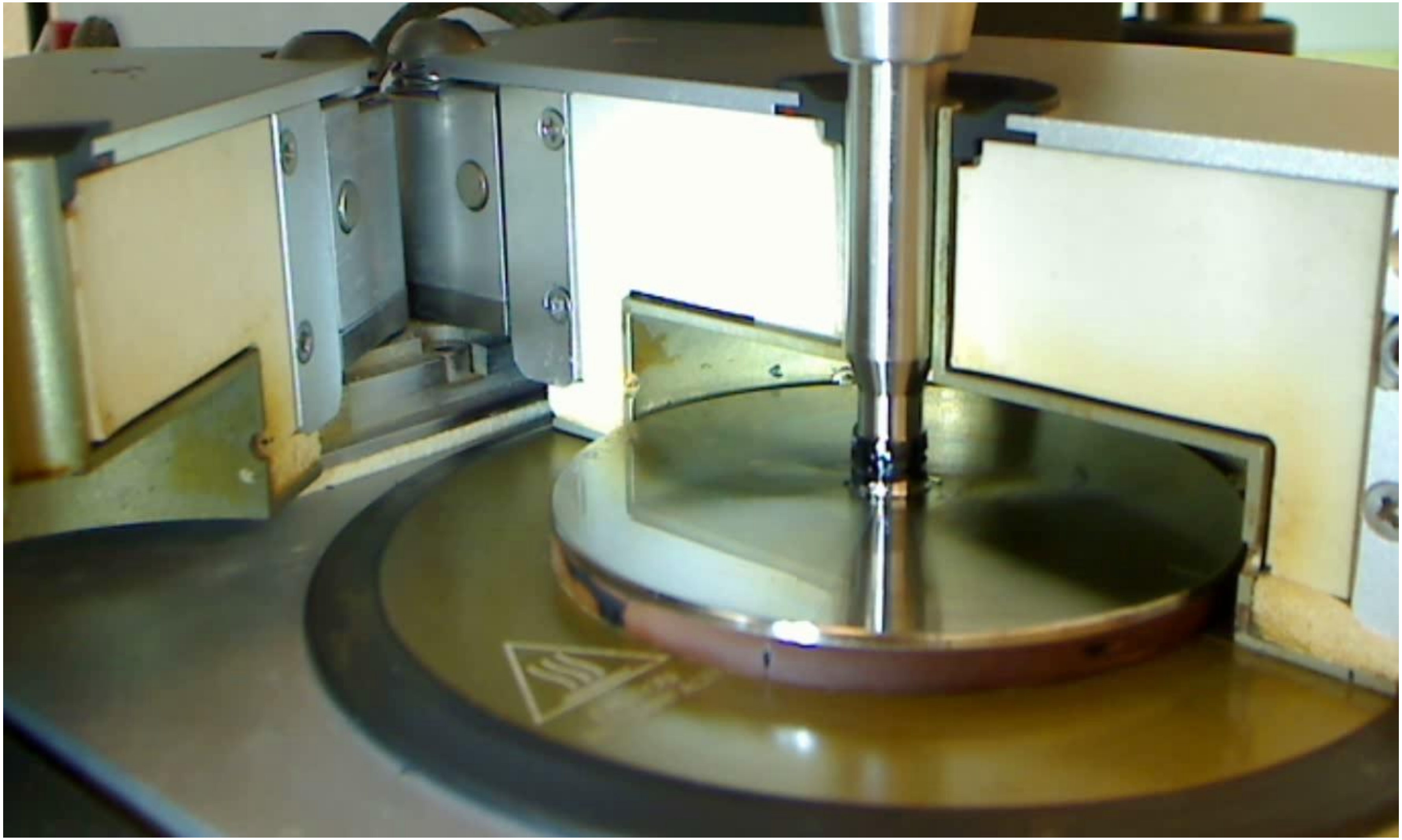


	Pen @ 25C	Strain @ 25C
Sample A	28	4.91572
Sample B	39	8.99743
Sample C	48	16.4315
Sample D	144	1.19E+03
Sample E	110	297.965
Sample F	35	7.95222

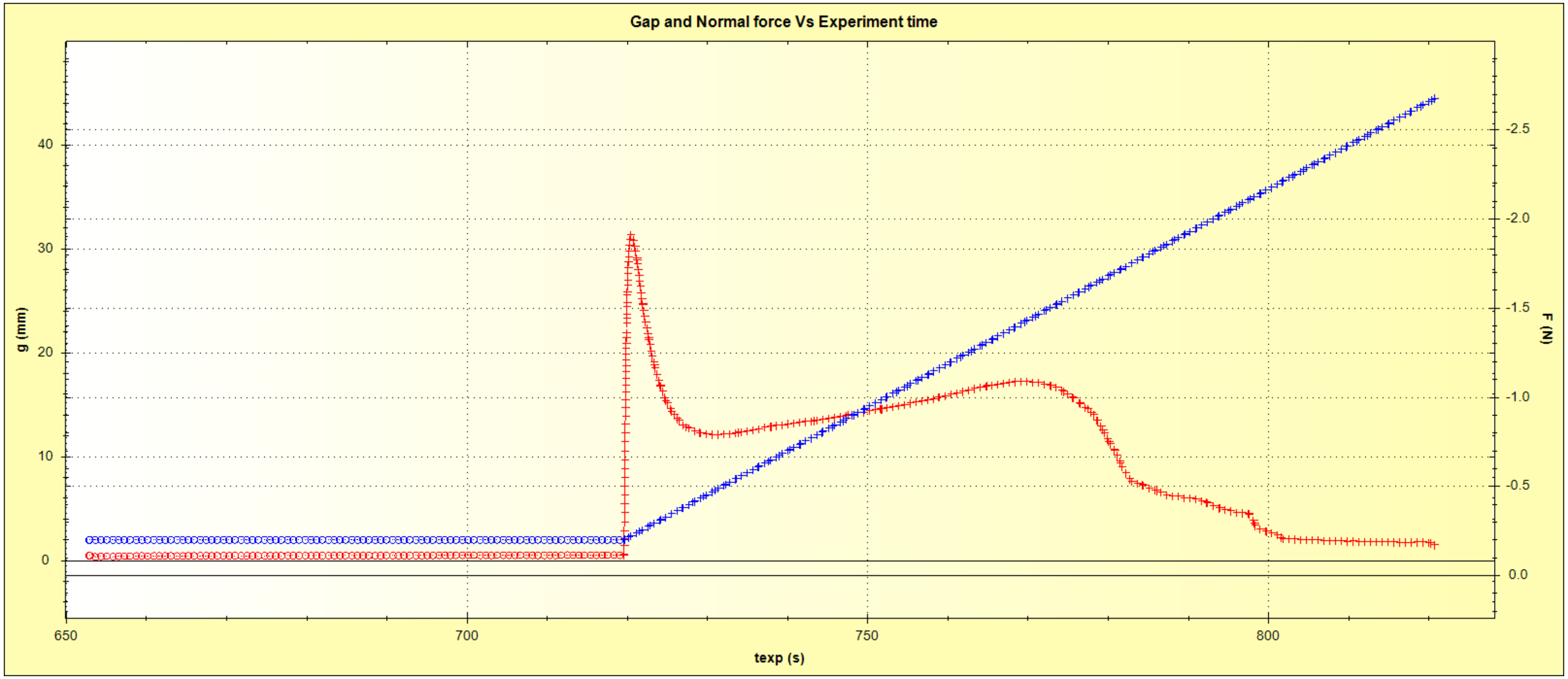
Adhesion & Cohesion



Adhesion & Cohesion: Pull rate 1 inch/min

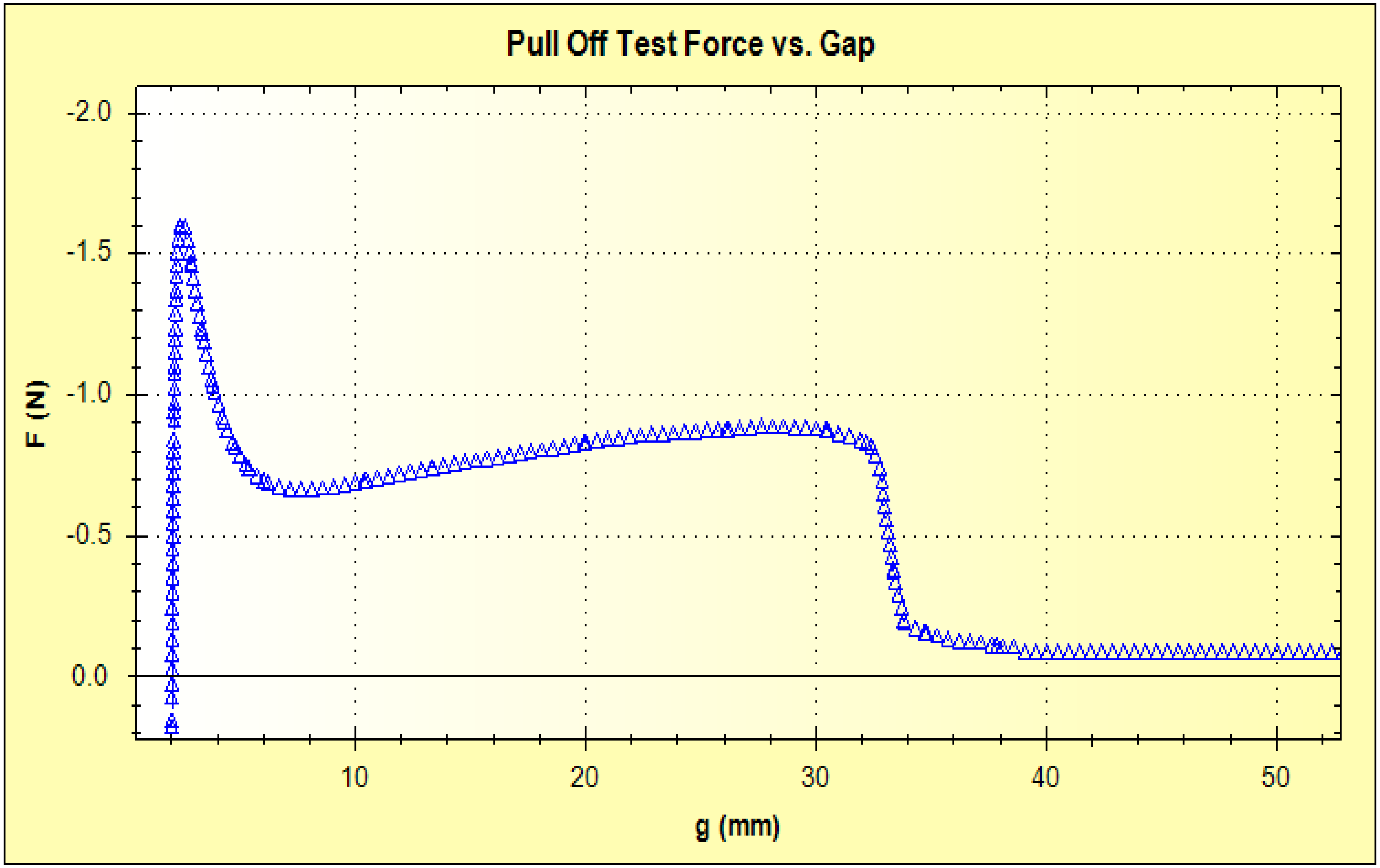


1.0 inch/min



Gap and Normal force Vs Experiment time
g (mm)
F (N)
texp (s)
0
10
20
30
40
-2.5
-2.0
-1.5
-1.0
-0.5
0.0
650
700
750
800

1.0 inch/min: Force vs. Gap



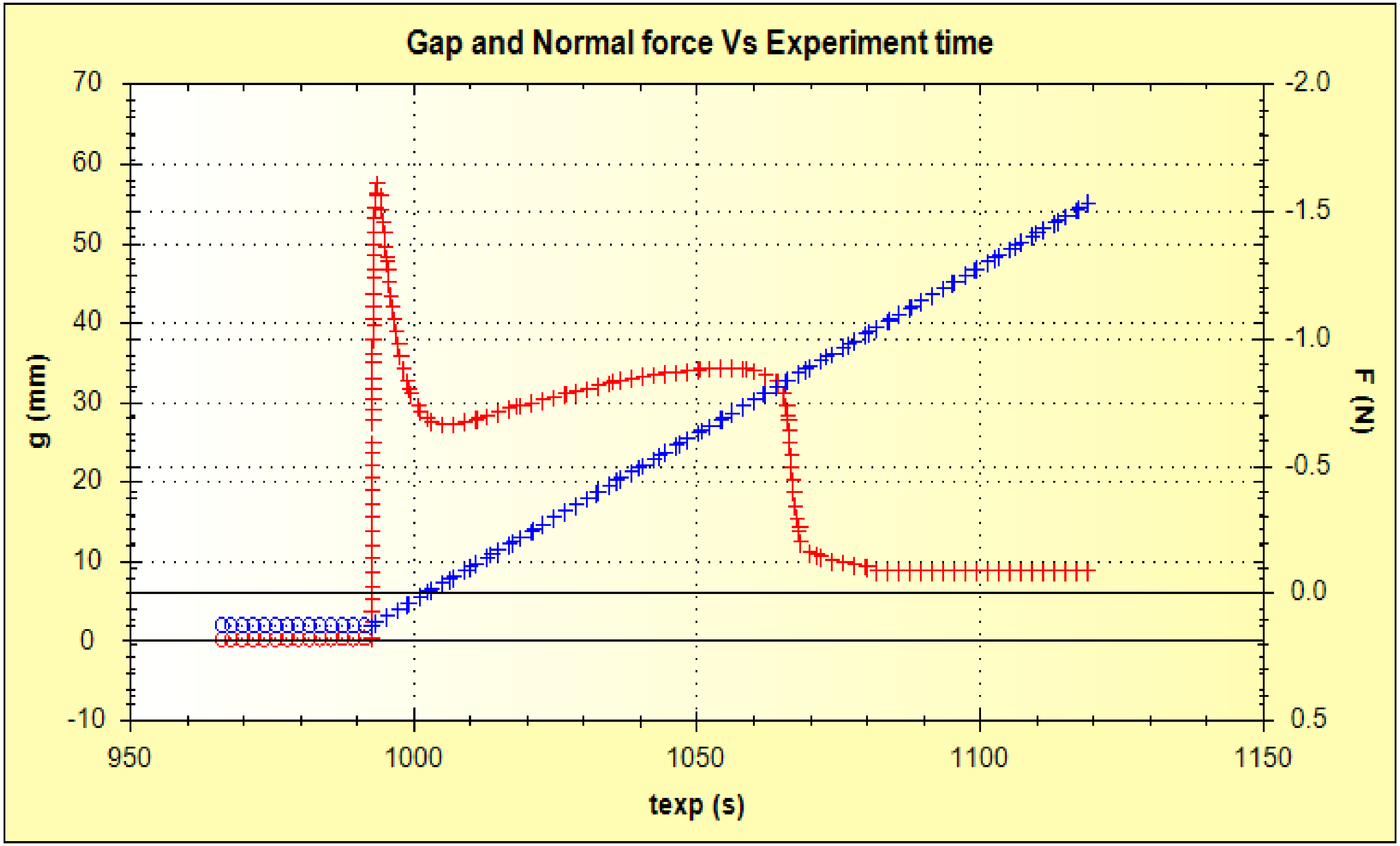
	Indx.	Samp.	Actn.	T(°C)	t(s)	F(N)	g(mm)
	493	Sample A Pull off 0.1inch	1.0 inch/min	59.95	4.930	-0.9610	4.0614
	494	Sample A Pull off 0.1inch	1.0 inch/min	59.95	4.940	-0.9599	4.0656
	495	Sample A Pull off 0.1inch	1.0 inch/min	59.95	4.950	-0.9587	4.0698
	496	Sample A Pull off 0.1inch	1.0 inch/min	59.95	4.960	-0.9580	4.0740
	497	Sample A Pull off 0.1inch	1.0 inch/min	59.95	4.970	-0.9569	4.0782
	498	Sample A Pull off 0.1inch	1.0 inch/min	59.95	4.980	-0.9551	4.0824
	499	Sample A Pull off 0.1inch	1.0 inch/min	59.95	4.990	-0.9549	4.0866
	500	Sample A Pull off 0.1inch	1.0 inch/min	59.95	5.000	-0.9541	4.0908
	501	Sample A Pull off 0.1inch	1.0 inch/min	59.95	5.010	-0.9532	4.0950
	502	Sample A Pull off 0.1inch	1.0 inch/min	59.95	5.020	-0.9522	4.0992

Point Index	1
Sample Description	Sample A Pull off 1.0 inch
Action Name	Area calculation
Table Name	Result data (F(N) vs t(s))
Area result	67.08
Point Notes	

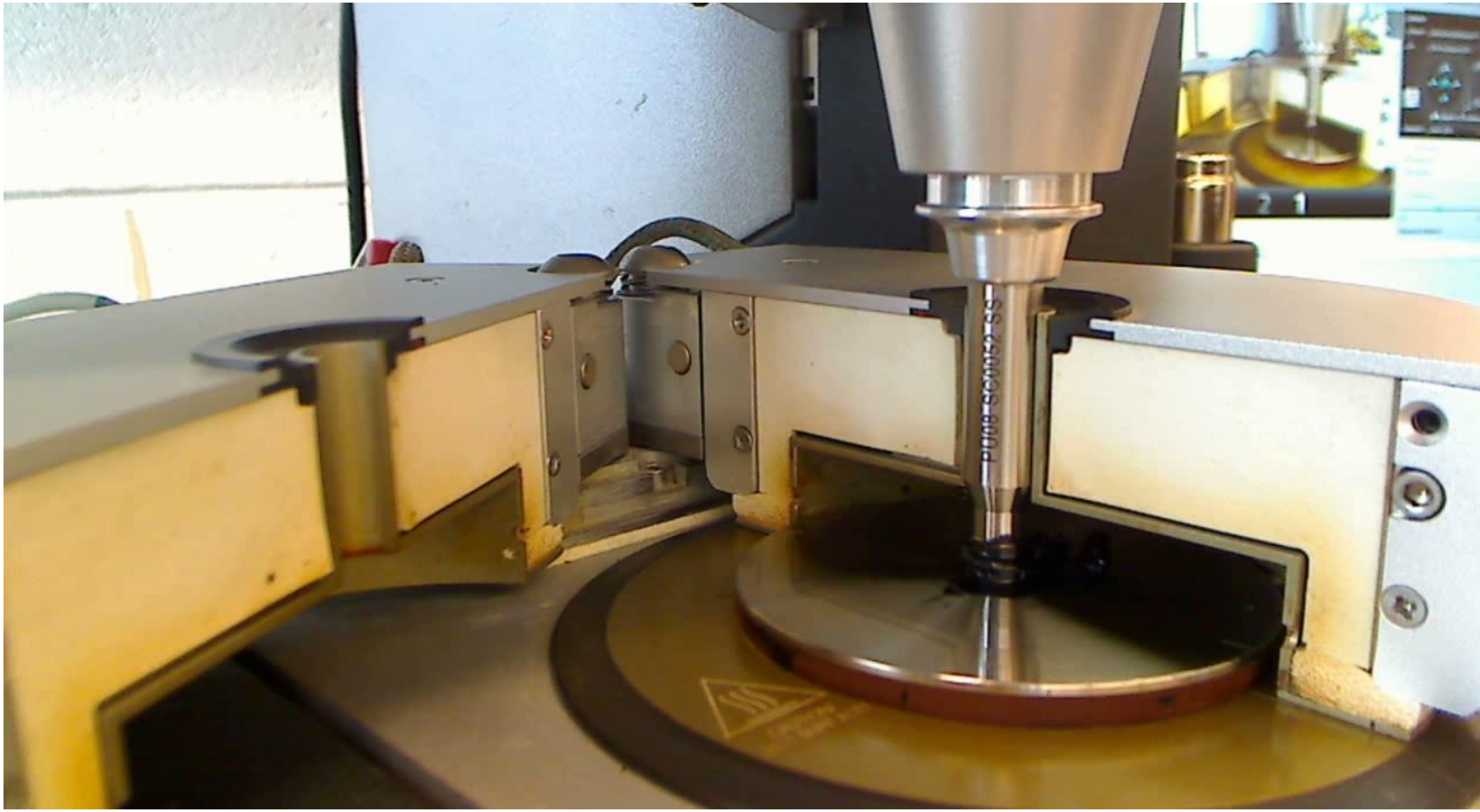
1.0 inch/min w/ repeat

Point Index	1	1
Sample Description	Sample A Pull off 1.0 inch	Sample A Pull off 1.0 inch
Action Name	Area calculation	Area calculation
Table Name	Result data (F(N) vs t(s))	Result data (F(N) vs t(s))
Area result	66.23	67.08
Point Notes		

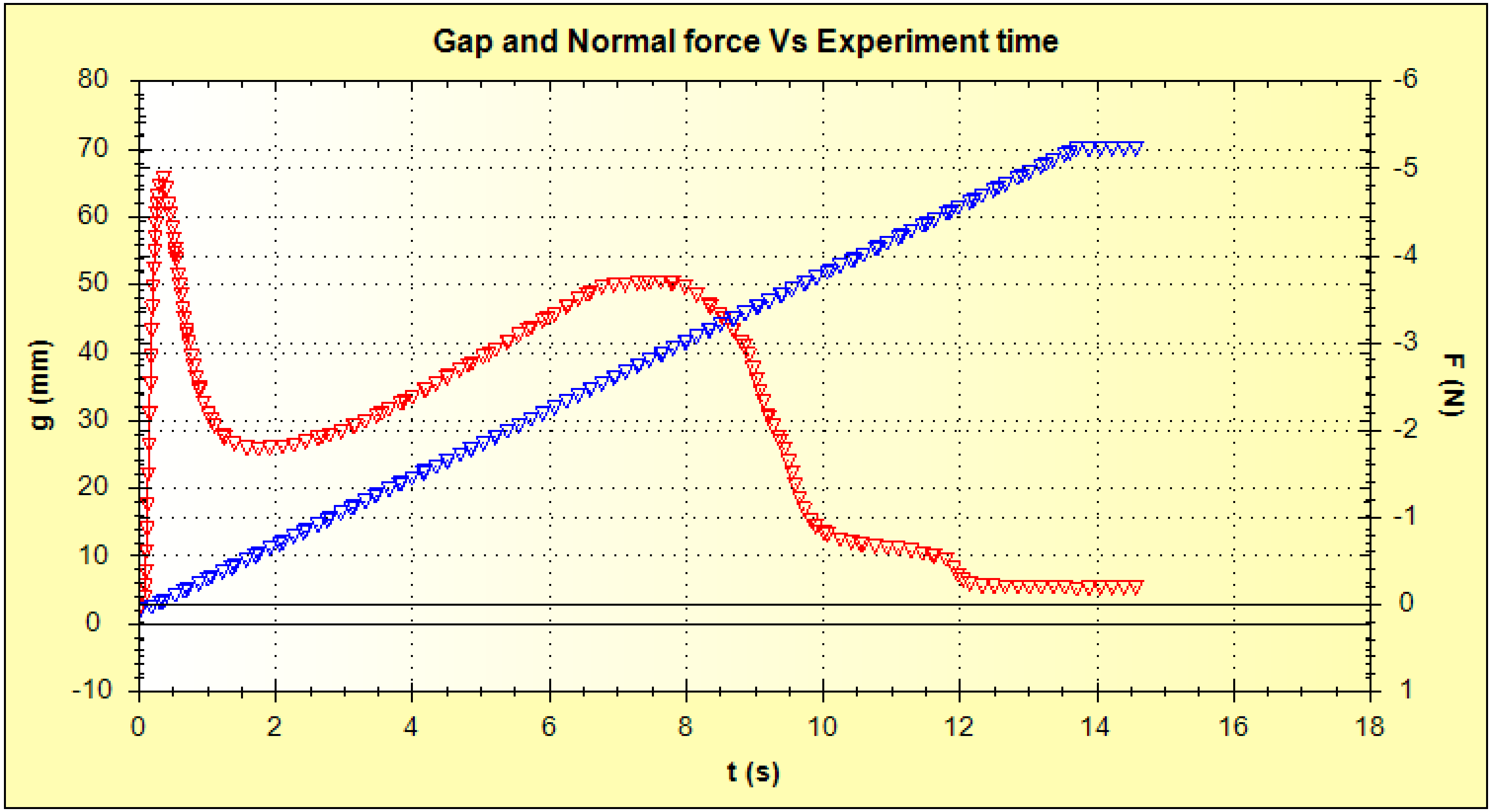
1.0 inch/min: Force & Gap vs.Time



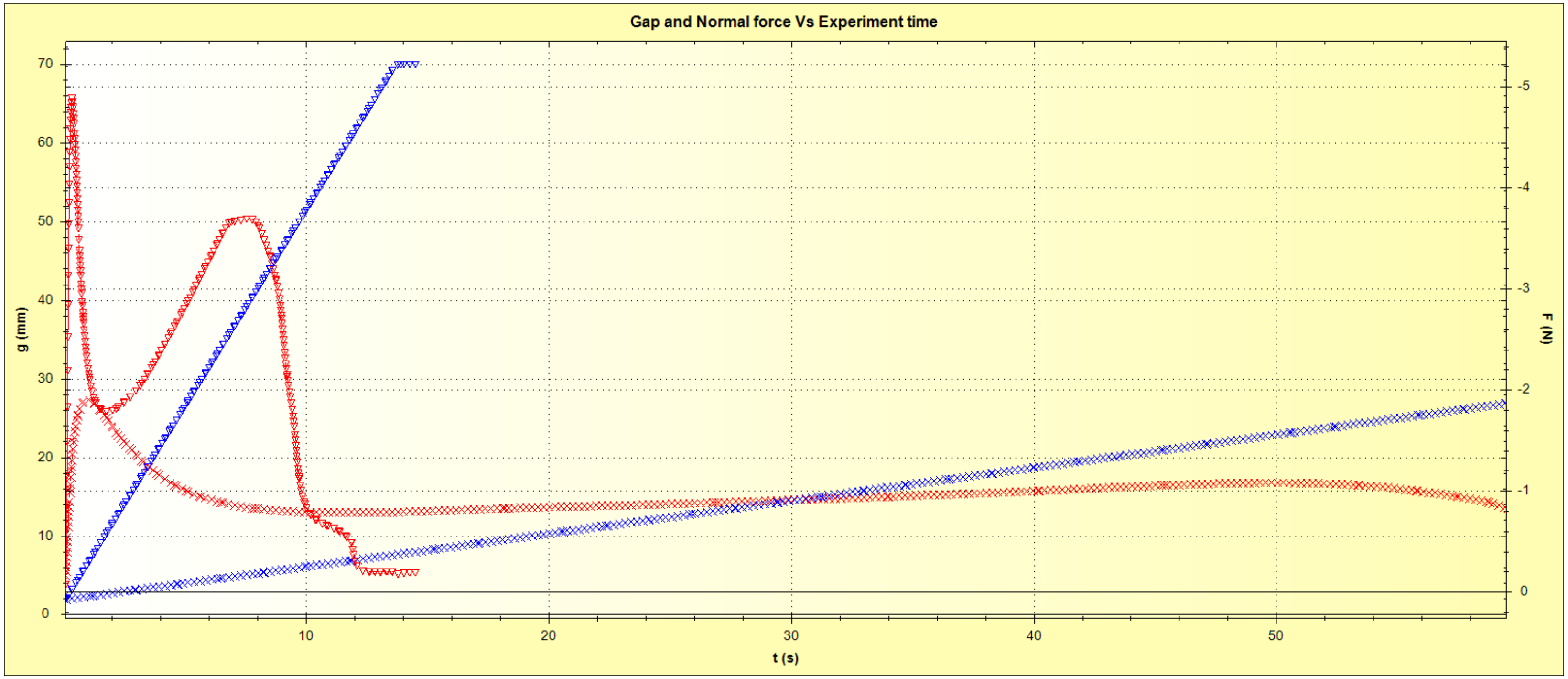
Adhesion & Cohesion: Pull rate 12 inch/min



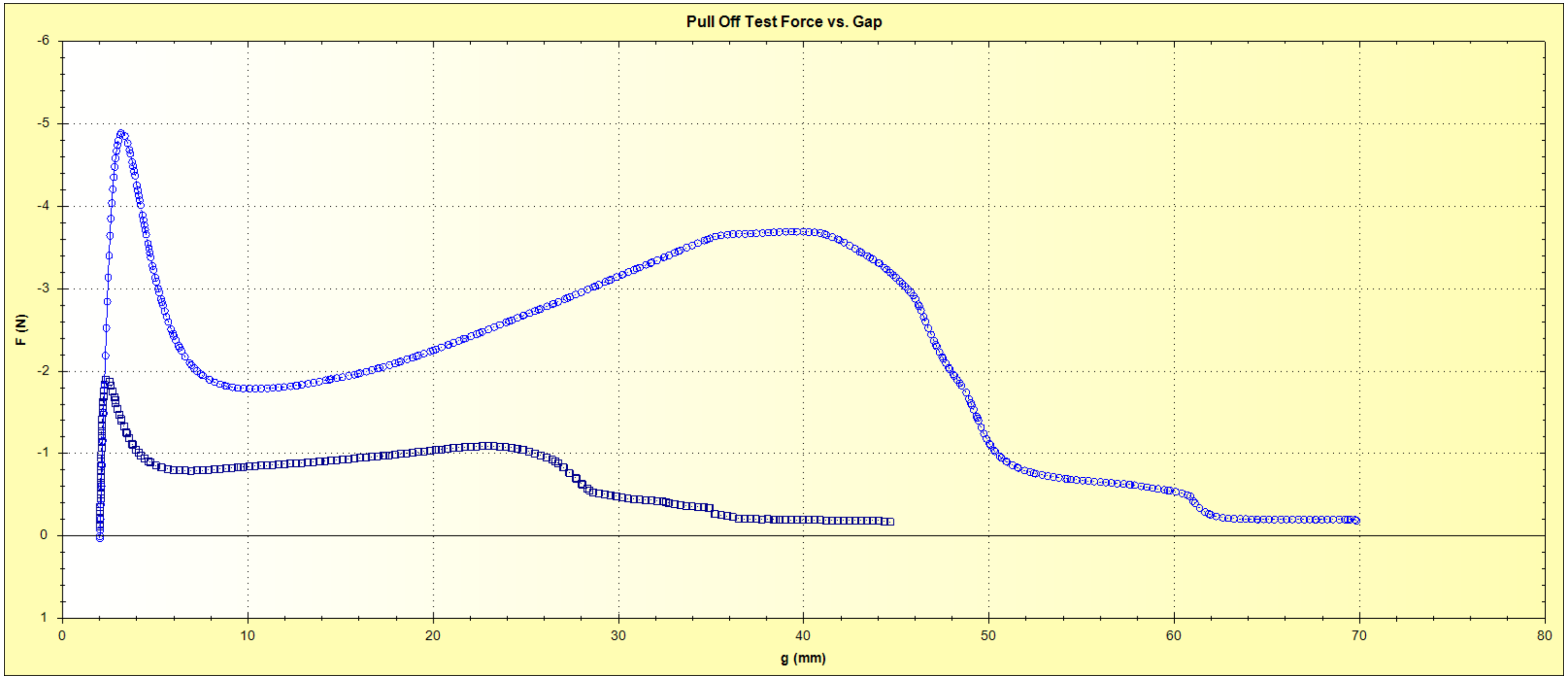
12 inch/min   Sample A



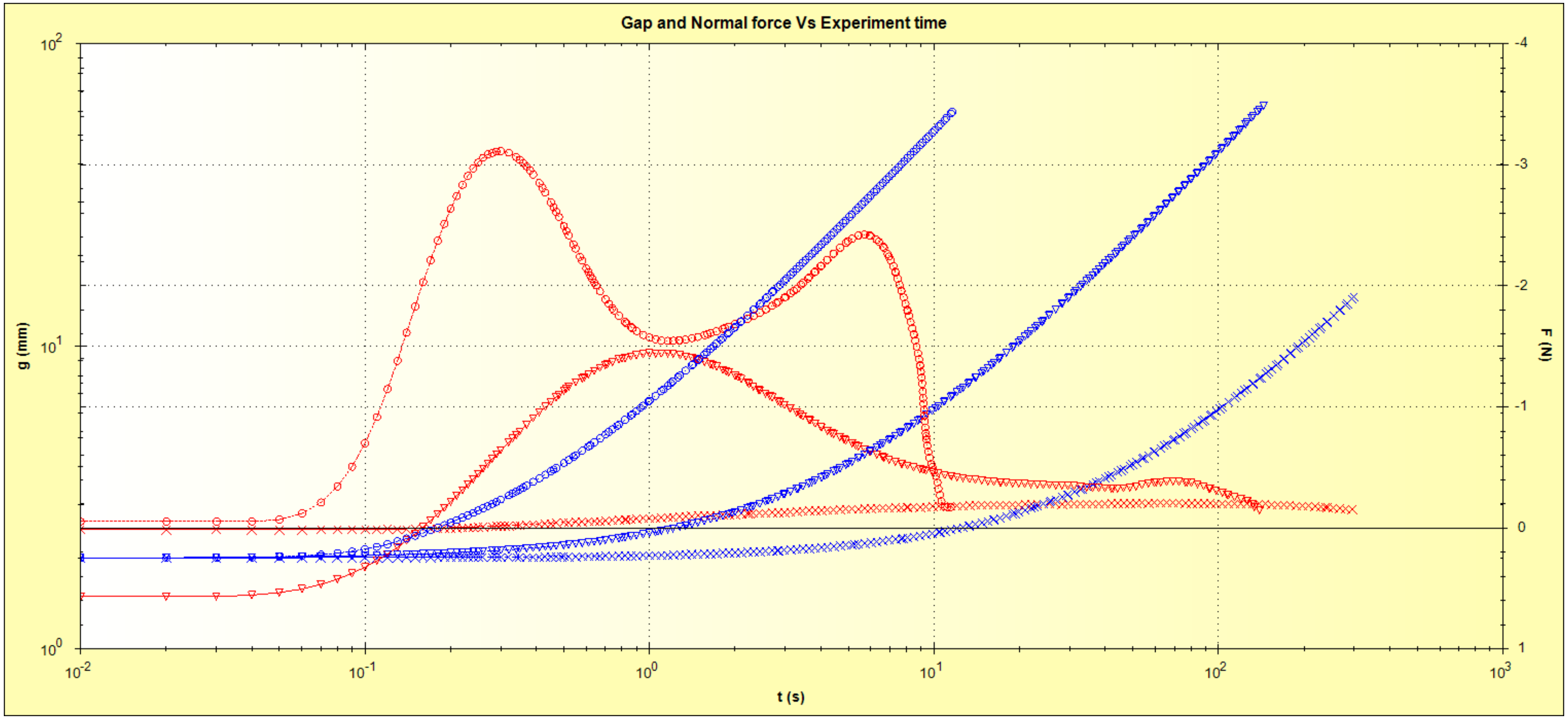
12 vs 1.0 inch/min Comparison: Sample A



12 vs. 1.0 inch/min Comparison: Sample F

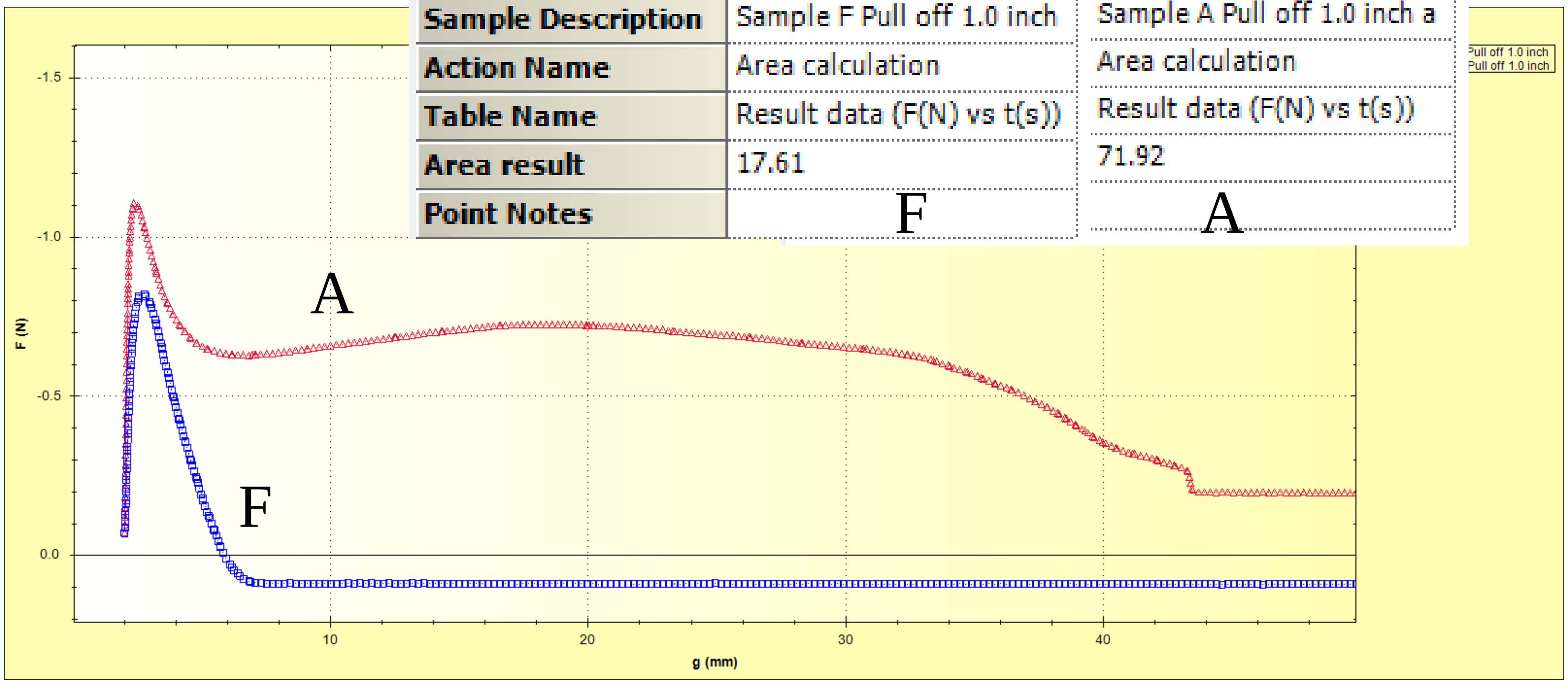


12.0, 1.0, 0.1 inch/min comparison Sample A

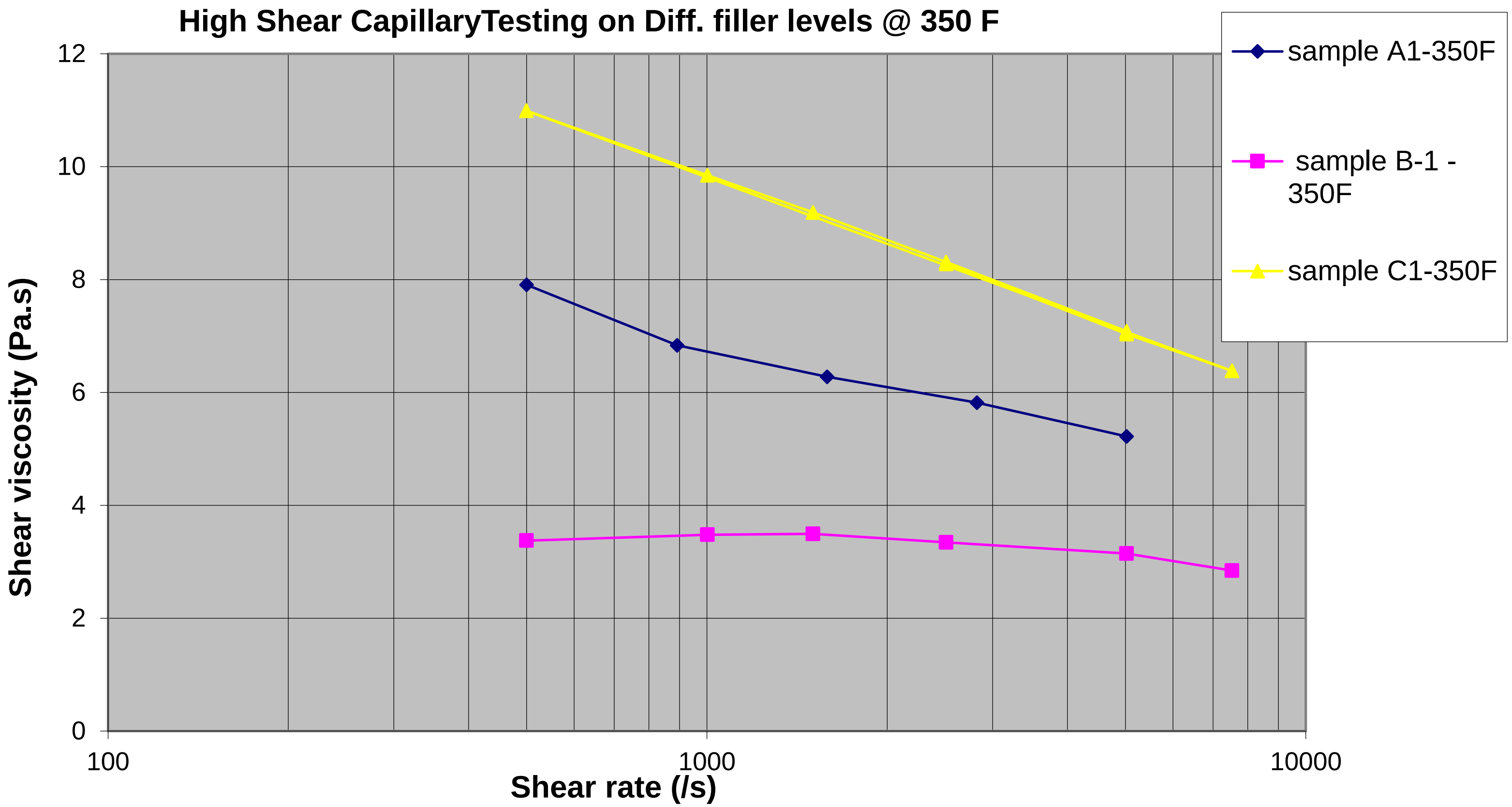


Sample A vs. F 1 inch/min Comparison

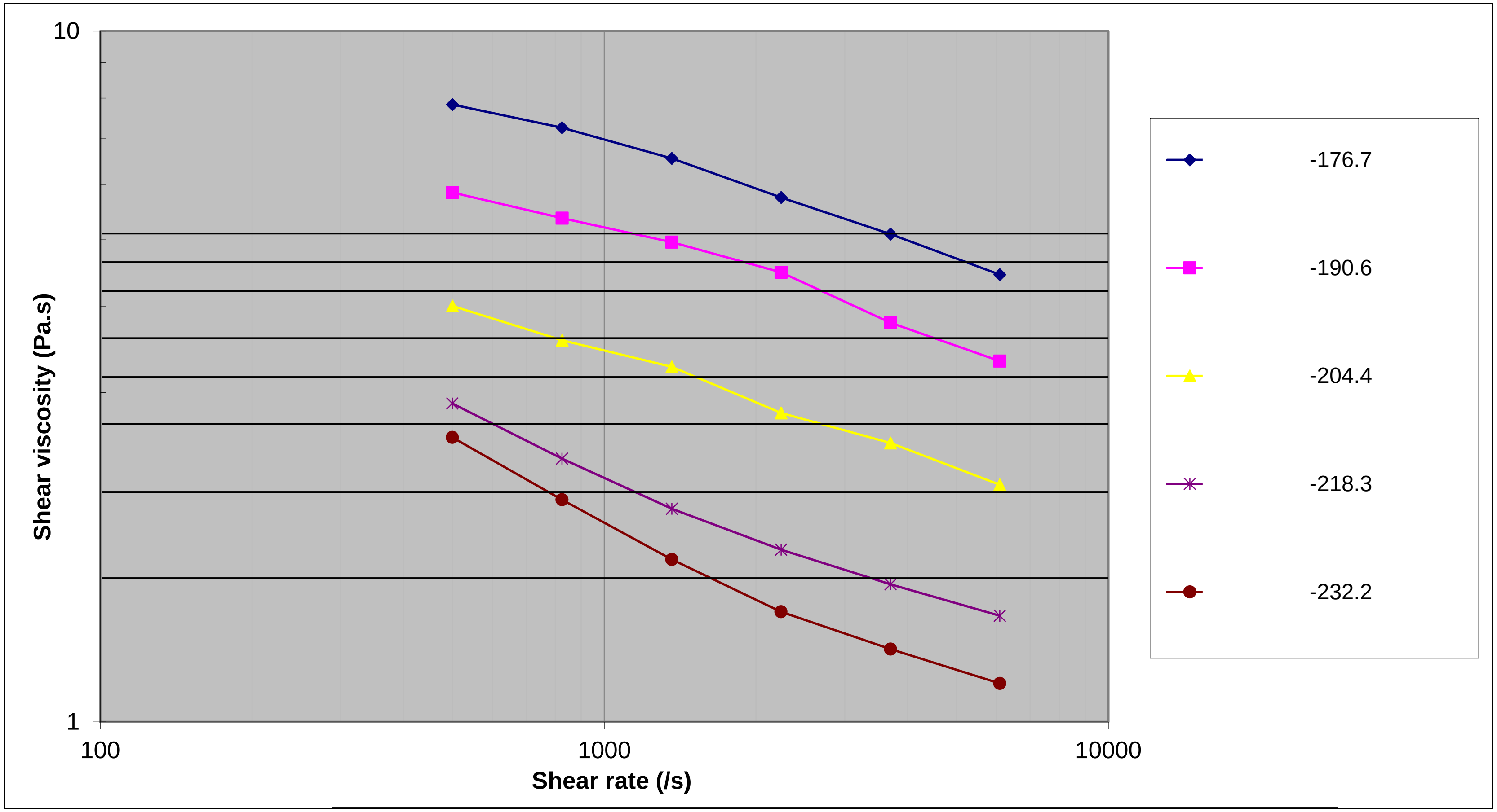
Point Index	1	1
Sample Description	Sample F Pull off 1.0 inch	Sample A Pull off 1.0 inch a
Action Name	Area calculation	Area calculation
Table Name	Result data (F(N) vs t(s))	Result data (F(N) vs t(s))
Area result	17.61	71.92
Point Notes	F	A



Differing Filler Volume Fractions Shingle Asphalt



Filled Shingle Asphalt; Differing Processing Temperatures



Abstract :

SqueezeFlow_0001 Alternative Flow Curve

Abstract

This sequence runs a logarithmic table of shear rates and measures the viscosity. It prompts the user for appropriate information before starting.

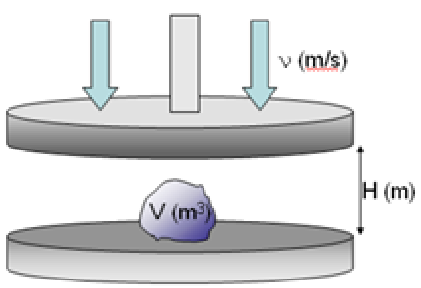
This sequence requires a sample to be loaded in a specific way when prompted. Simply follow the on screen instructions.

Category

Squeeze flow

When might I use this sequence?

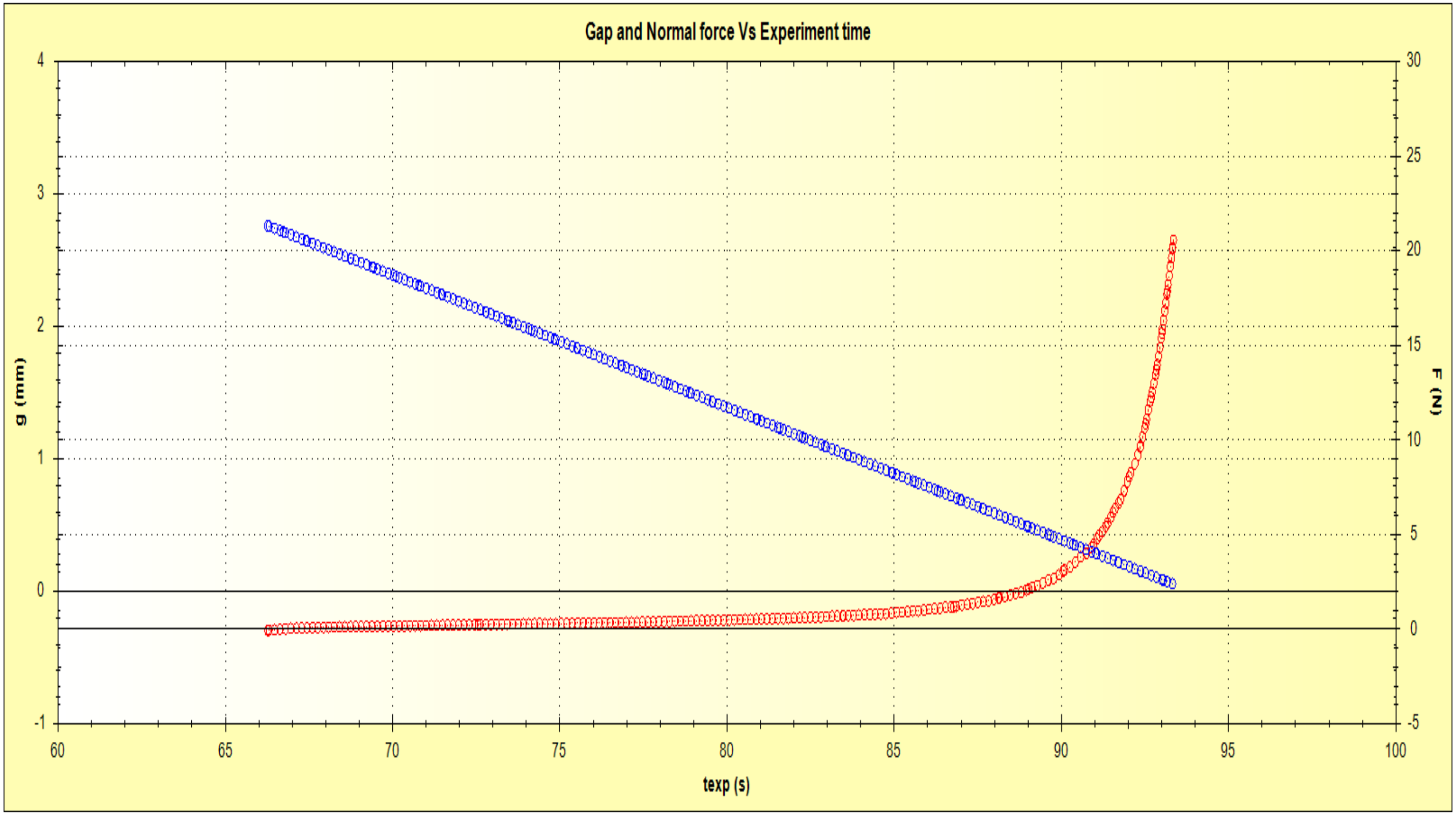
This an alternative method for measuring the shear viscosity of a material over a range of shear rates It is particularly useful for highly filled yield stress type samples which show power law type flow behavior as these samples tend to be challenging to measuring under standard rotational shear, these sample types tend to slip and fracture (even with roughened plates) which makes measurements problematic. However, these same properties are perfect for squeeze flow where the sample needs to slip across the surface of the plates.



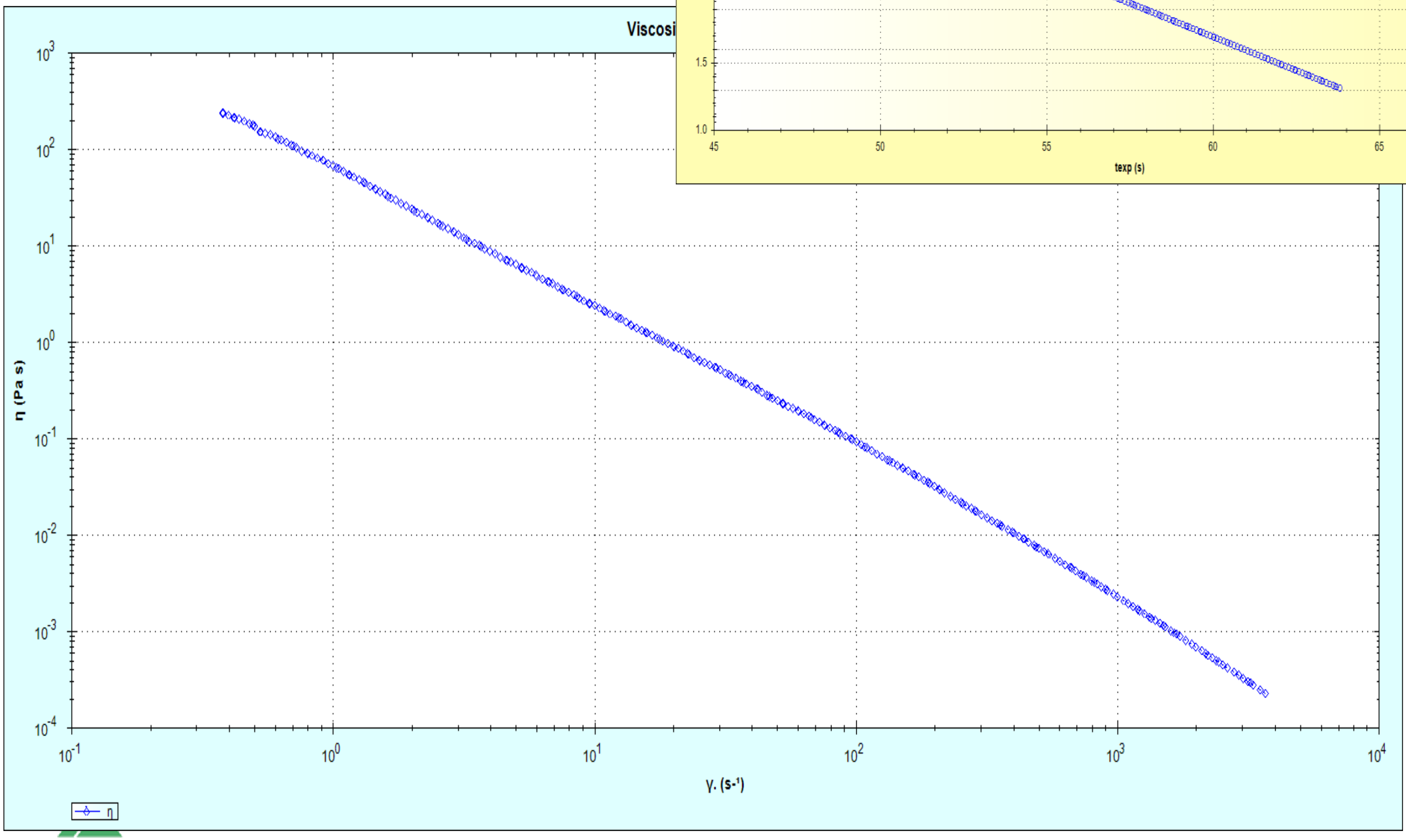
The algorithm is based on a technique described in J. Non-Newtonian Fluid Mech., 81 (1999) 1-15
"Analytical solutions for squeeze flow with partial wall slip" by H.M. Laun, M. Rady, O. Hassager

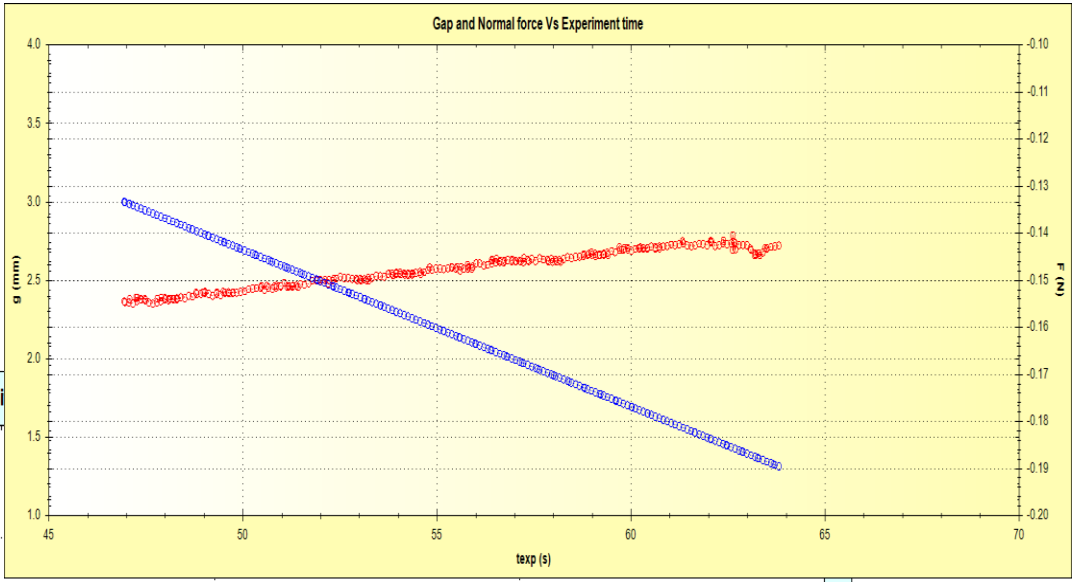
As with all methods which approximate the shear flow behavior of a non-Newtonian sample, the deformation may not follow the prescribed profile. Therefore, this method should be validated with the samples type of interest before considering the viscosity values to be the same as those recorded under standard conditions.

Squeeze Flow



Sample G at 150C
Squeeze Flow Method





Keys to Automated QC Testing

Manage Sample History and Quality of the Measurement

› How do we manage sample history?

- Understanding the sample!
- Loading to minimize stress
- Precondition to establish sample time zero
- Time to permit sample to relax before testing or between testing
- Standard Operating Procedures

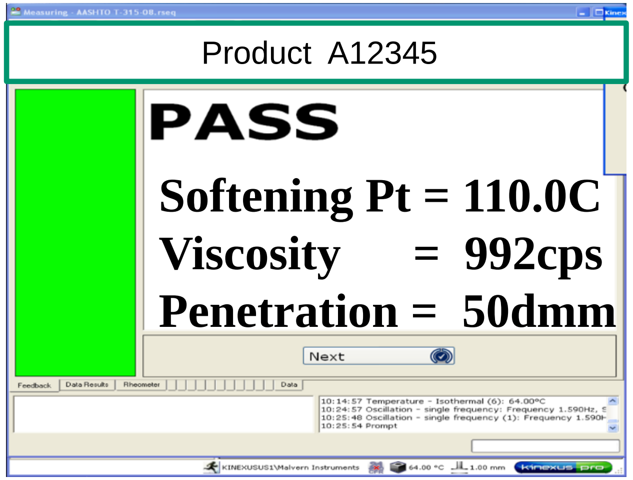
› Measurement Quality

- Monitor normal force; indicator of internal sample stress
- Monitor approach to steady state (m-Value); tells us when the sample’s at equilibrium
- Monitor % harmonic distortion; provides a measure of the quality of oscillation data
- Automatically determine LVER; optimizes the measurement

› Using the Instrument to improve the quality and reproducibility of the measurements

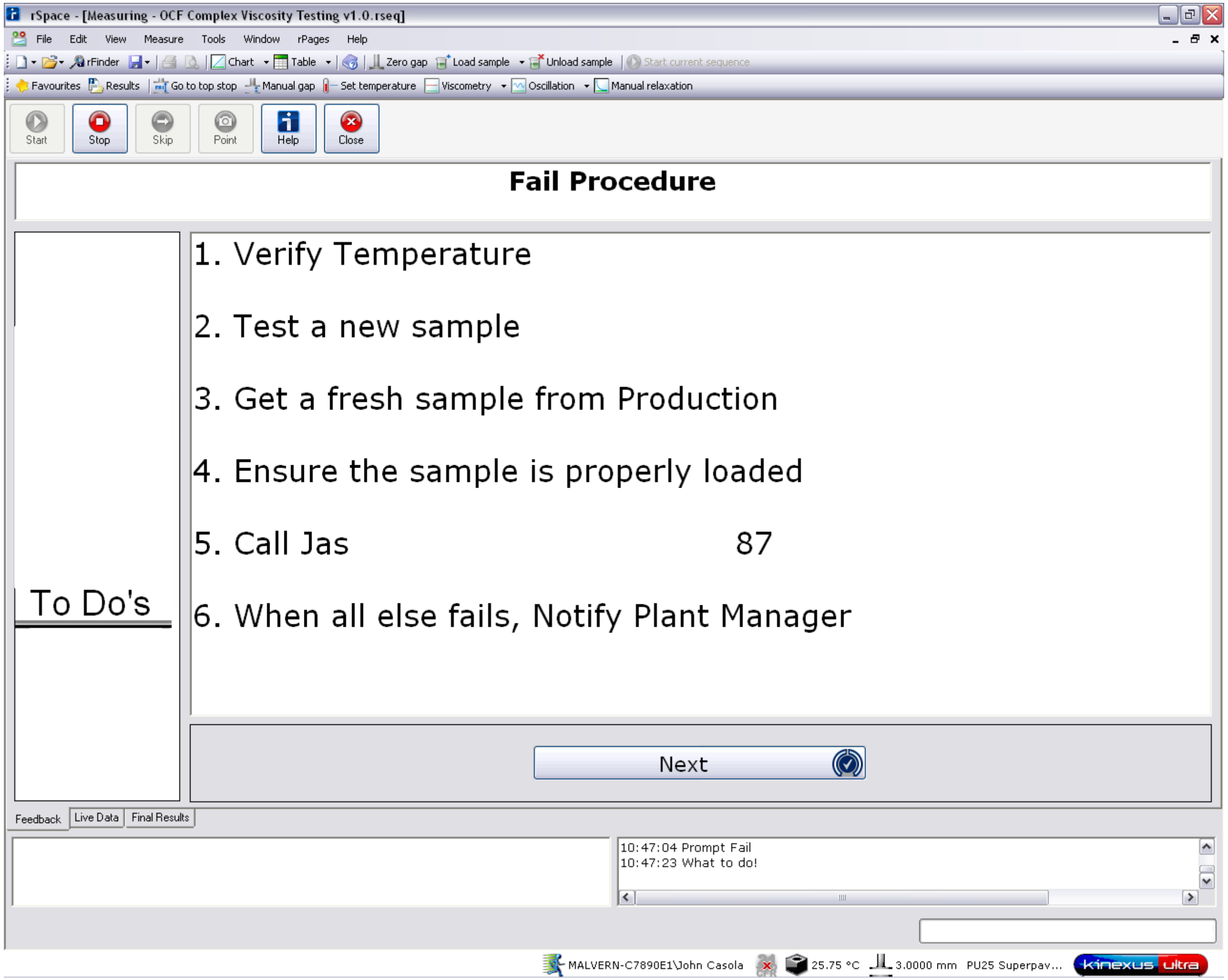
Logical Decision Making

- Know your product & know your plan of attack
- Establish acceptance criteria
  - Life’s great when everything passes!



- What to do when it fails?
  - Can the data lead to the next appropriate step?
    - **Add more of something?**
    - **Let down?**
    - **Process differently?**
    - **Specify differently?**
- Let’s use the instrument to provide positive feedback.

Customizable Escalation Procedures



Malvern

www.malvern.com

How many tests were used to collect all the data?

2 for DSR

- 2 sample loadings
- 1 - test to perform Pen, Softening Point and Viscosity on same sample. 25mm plates
  - **Could just as easily add additional non-destructive tests, for example: Frequency sweeps for Master curve generation**
- 1 - test to perform sample Pull-Off. 8mm plates

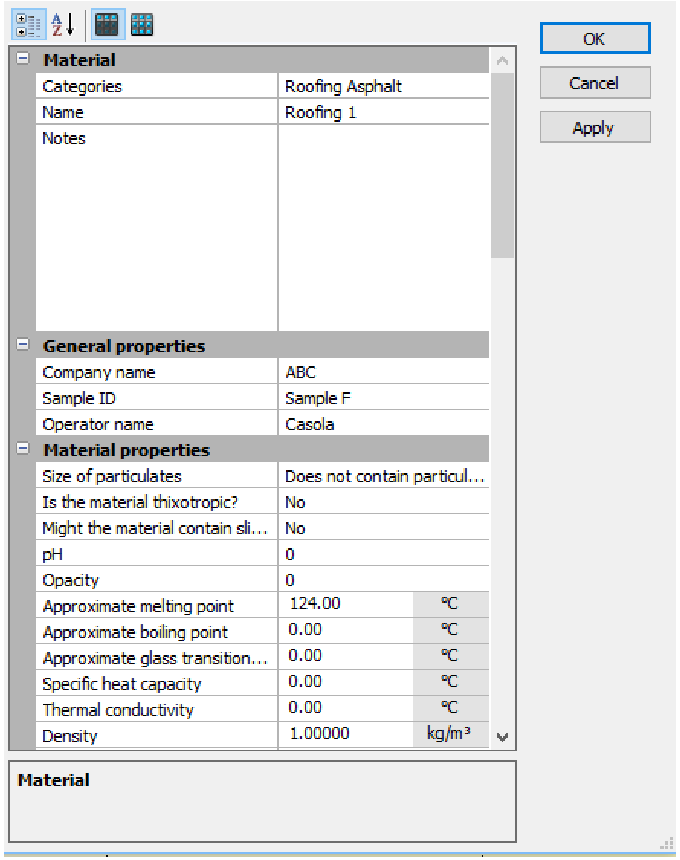
1 for Capillary

- High shear viscosity

Materials Data Base (bi-directional)

› Set up by Product, Project, Customer, Application...

- Min – Max Softening Point Temperature Limits
- Min-Max Viscosity Limits
- Min – Max Pen Limits
- Softening Point Temperature
- Temperature for Viscosity
- Temperature for Penetration
- Modulus at Softening Point
- Penetration Value
- Pull Off Energy
- Strain & Stress for LVER Testing
- Particle size, shape & distribution
- Sample Loading Temperature
- Clean Up Temperature
- And, any other property or parameter…



Thank you for your interest!

Questions ?