

Advancements in Testing Roofing Asphalt Binder

Mike Anderson, Asphalt Institute

AMAP Annual Meeting
Ft. Lauderdale, FL
February 5-7, 2019

Note: For narration of this presentation,
[click here to visit AMAP's technical library](#)
to download the PowerPoint show file.

ASTM D 312

Property	Type I		Type II		Type III		Type IV	
	Min	Max	Min	Max	Min	Max	Min	Max
Soft. Point, °C(°F)	57(135)	66(151)	70(158)	80(176)	85(185)	96(205)	99(210)	107(225)
Flashpoint, °C(°F)	260(500)	...	260(500)	...	260(500)	...	260(500)	...
Penetration, dmm								
at 0°C(32°F)	3	...	6	...	6	...	6	...
at 25°C(77°F)	18	60	18	40	15	35	12	25
at 46°C(115°F)	90	180	...	100	...	90	...	75
Duct at 25°C(77°F),cm	10.0	...	3.0	...	2.5	...	1.5	...
Solubility in TCE, %	99	...	99	...	99	...	99	...



Rheological Testing and Evaluation

- Rheology Task Force
 - Gaylon Baumgardner, Mike Franzen, Keith Stephens, Mike Anderson
 - Formed to evaluate rheological parameters of roofing asphalts for possible use in a specification



	RPG 58 (Type I)		RPG 70 (Type II)		RPG 82 (Type III)		RPG 94 (Type IV)	
Property	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Flash Point, °C (°F)	260(500)	---	260(500)	---	260(500)	---	260(500)	---
Equi-viscous Temperature, °C	Report		Report		Report		Report	
High Equi-stiffness Temperature, °C	57	66	70	80	85	96	99	107
Complex Viscosity @ 71.1°C, kPa-s	0.2	---	2.0	---	20.0	---	200.0	---
G*(0.4 Hz) @ 50°C, MPa	0.02	0.10	0.04	0.20	0.05	0.30	0.07	0.40
δ(0.4 Hz) @ 50°C, degrees	Report		Report		Report		Report	
G*(0.4 Hz) @ 25°C, MPa	0.3	1.8	0.6	3.2	0.8	4.9	1.1	7.1
G*(0.4 Hz) @ 5°C, MPa	3.6	16.0	17.0	28.0	23.0	42.0	32.0	62.0
δ(0.4 Hz) @ 5°C, degrees	Report		Report		Report		Report	
G*(15.9 Hz) @ 25°C, MPa	---	20	---	30	---	40	---	50
Solubility, %	99.0	---	99.0	---	99.0	---	99.0	---



	RPG 58 (Type I)		RPG 70 (Type II)		RPG 82 (Type III)		RPG 94 (Type IV)	
Property	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Flash Point C (°F)	260(500)	---	260(500)	---	260(500)	---	260(500)	---
Equi-viscous Temperature, °C	Report		Report		Report		Report	
Softening Point	57	66	70	80	85	96	99	107
Complex Viscosity @ 71.1°C, kPa-s	0.2	---	2.0	---	20.0	---	200.0	---
Penetration @ 46C	0.02	0.10	0.04	0.20	0.05	0.30	0.07	0.40
$\delta(0.4 \text{ Hz}) @ 50^\circ\text{C}$, degrees	Report		Report		Report		Report	
Penetration @ 25C	0.3	1.8	0.6	3.2	0.8	4.9	1.1	7.1
Penetration @ 0C	3.6	16.0	17.0	28.0	23.0	42.0	32.0	62.0
$\delta(0.4 \text{ Hz}) @ 5^\circ\text{C}$, degrees	Report		Report		Report		Report	
Ductility	---	20	---	30	---	40	---	50
Solubility	99.0	---	99.0	---	99.0	---	99.0	---



Rheological Testing and Evaluation

- Evaluation of Rheological Properties of Good/Bad Coatings
 - Start with 2 Good and 2 Bad Fluxes
 - Supplied by Owens Corning and GAF
 - Blow to 4 different Softening Points
 - Submit samples to AI and Paragon Technical Services for testing
 - Mastercurve determination
 - Compare data with conventional parameters



Rheological Testing and Evaluation

- Asphalt Binders
 - Owens Corning
 - Two Asphalt Binders
 - OC-A
 - OC-B
 - Four Softening Points
 - Blown to R&B Softening Points of 200, 207, 214, and 220°F



Rheological Testing and Evaluation

- Asphalt Binders
 - GAF
 - Two Asphalt Binders
 - F0965112
 - F106312
 - Blown to 3-4 Softening Points
 - F0965112
 - 3 softening points between 207 and 220°F
 - F106312
 - 4 softening points between 200 and 220°F



Rheological Testing and Evaluation

- Testing
 - Conventional
 - R&B Softening Point
 - Penetration (25°C)



Rheological Testing and Evaluation

- Testing
 - Rheological
 - Temperature-Frequency Sweep (High)
 - 0.1-10 rad/s (10 pts/decade)
 - 25-mm parallel plate geometry
 - 1-mm gap
 - 2% shear strain
 - 50-100°C (10°C increments)
 - Temperature-Frequency Sweep (Low)
 - 0.1-10 rad/s (10 pts/decade)
 - 8-mm parallel plate geometry
 - 2-mm gap
 - 1% shear strain
 - 10-40°C (5°C increments)



Rheological Testing and Evaluation

- Testing
 - Rheological
 - Mastercurve development
 - Using high and low data sets separately
 - Can be combined
 - Derived parameters
 - G^* , δ at 2.5 rad/s
 - 5°C, 25°C, 50°C
 - T_c at 2.5 rad/s
 - Determined where $G^* = 2000$ Pa
 - G^* , δ at 100 rad/s
 - 25°C
 - Complex Viscosity (η^*) at 71.1°C and 1 rad/s



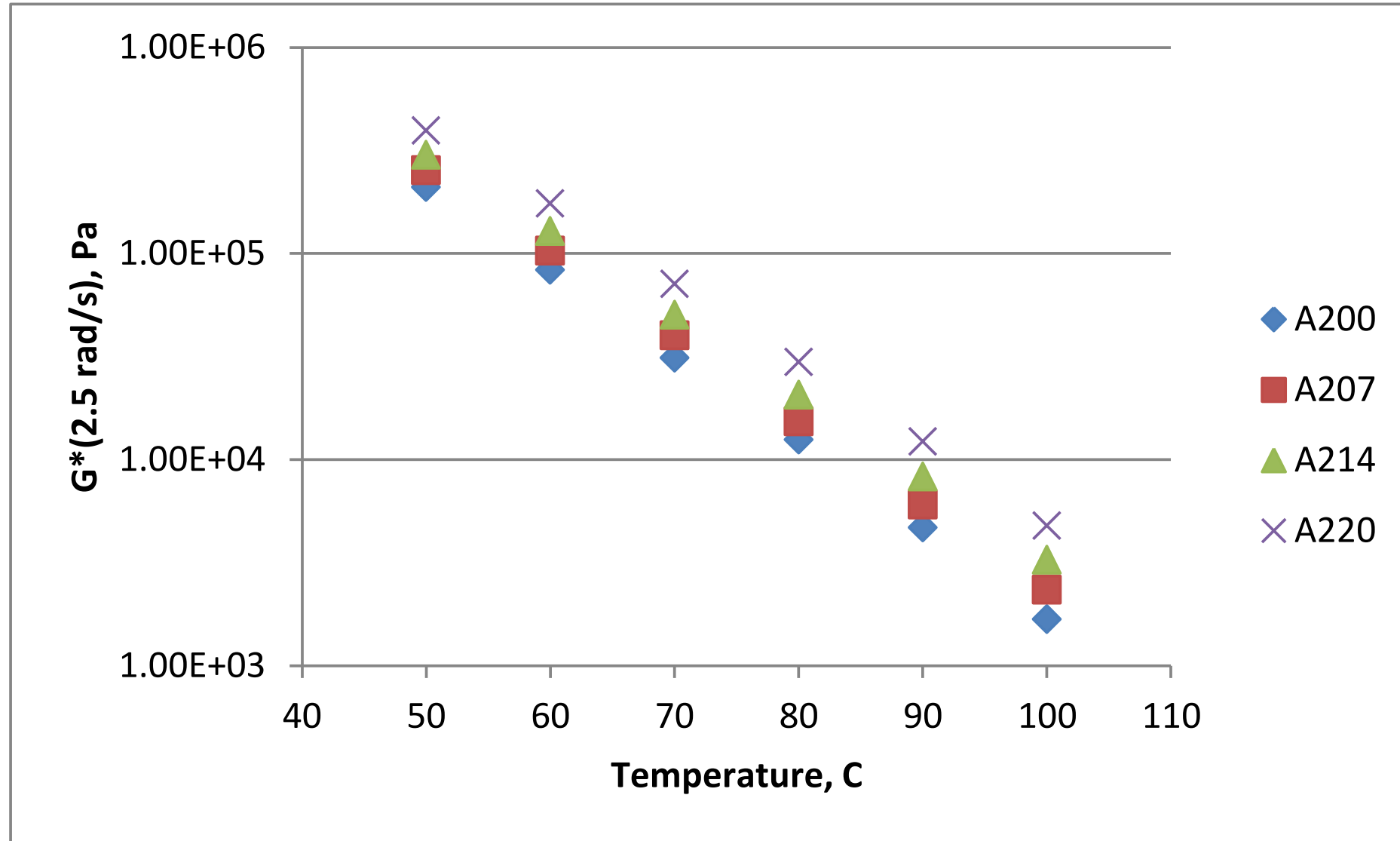
High Temperature (Paragon)

Paragon	50		71.1				50	
	2.5 rad/s		1 rad/s	2.5 rad/s	$\log G^* = Ax + B$		2.5 rad/s	R&B
	G^* , Pa	δ , deg.	η^* , Pa-s	T_c , C	A	B	$G(t)$, Pa	SP, C
A200	2.08E+05	37.8	17,604	98.6	-0.0418	7.4235		96.7
A207	2.52E+05	36.5	22,417	101.6	-0.0409	7.4565		101.1
A214	2.99E+05	35.6	29,625	105.5	-0.0394	7.4588		105.8
A220	3.95E+05	33.1	41,846	110.2	-0.0384	7.5345		110.5
B200	6.11E+05	40.4	32,184	98.9	-0.0518	8.4241		93.7
B207	8.33E+05	40.0	50,979	104.7	-0.0481	8.3392		104
B214	1.07E+06	36.0	73,220	108.2	-0.0476	8.451		104.4
B220	1.37E+06	33.8	106,931	113.4	-0.0454	8.4478		110



OC-A-xxx Binders: $G^*(2.5 \text{ rad/s}) = f(T)$

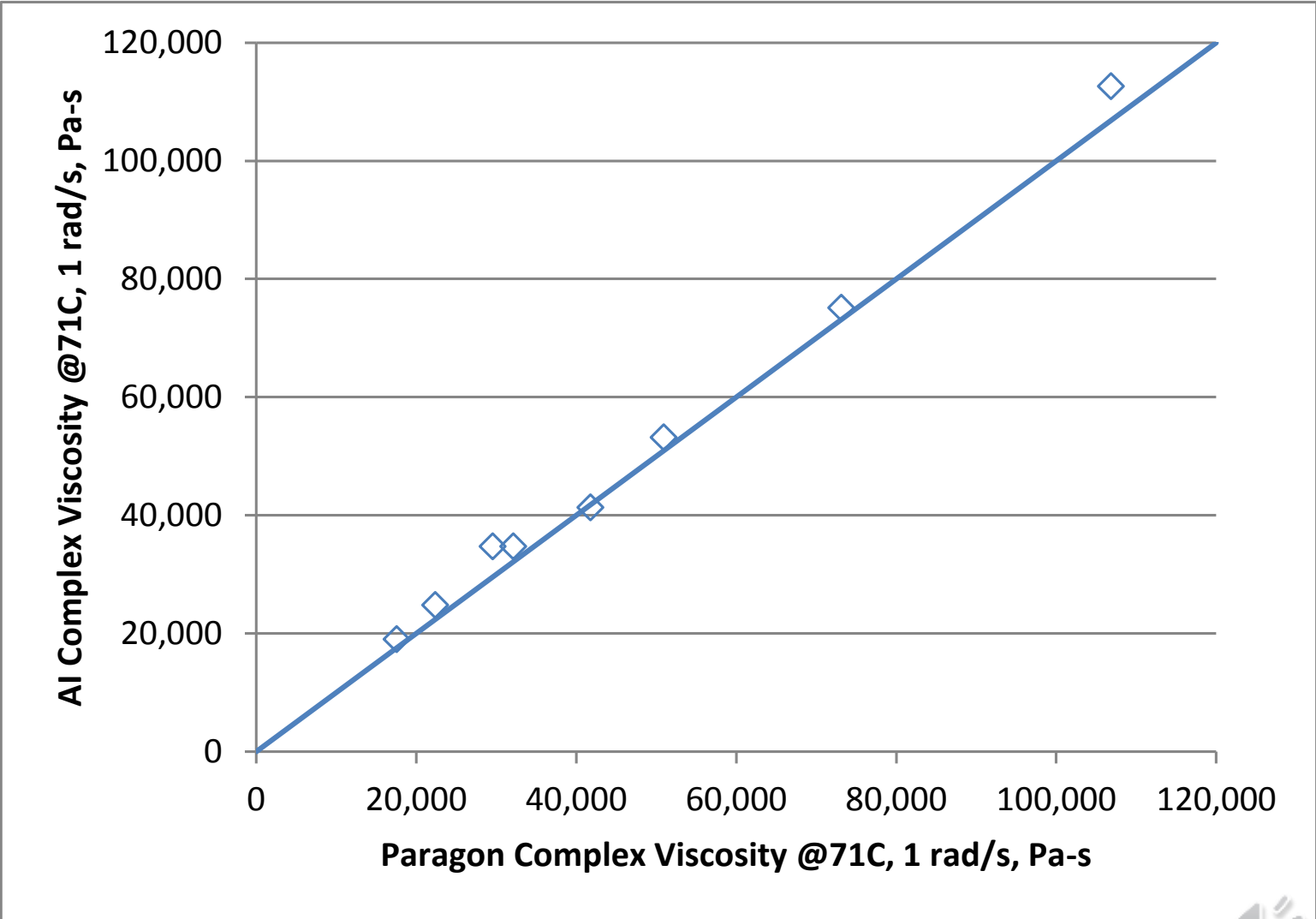
Paragon



Complex Viscosity (η^*): 71.1°C, 1 rad/s

	AI	Paragon	
	71.1	71.1	
	1 rad/s	1 rad/s	
	η^* , Pa-s	η^* , Pa-s	d2s%
A200	19,015	17,604	8%
A207	24,785	22,417	10%
A214	34,753	29,625	16%
A220	41,322	41,846	1%
B200	34,754	32,184	8%
B207	53,168	50,979	4%
B214	75,140	73,220	3%
B220	112,602	106,931	5%

Multi-laboratory d2s% for rotational viscosity (AASHTO T316) = 12.1%

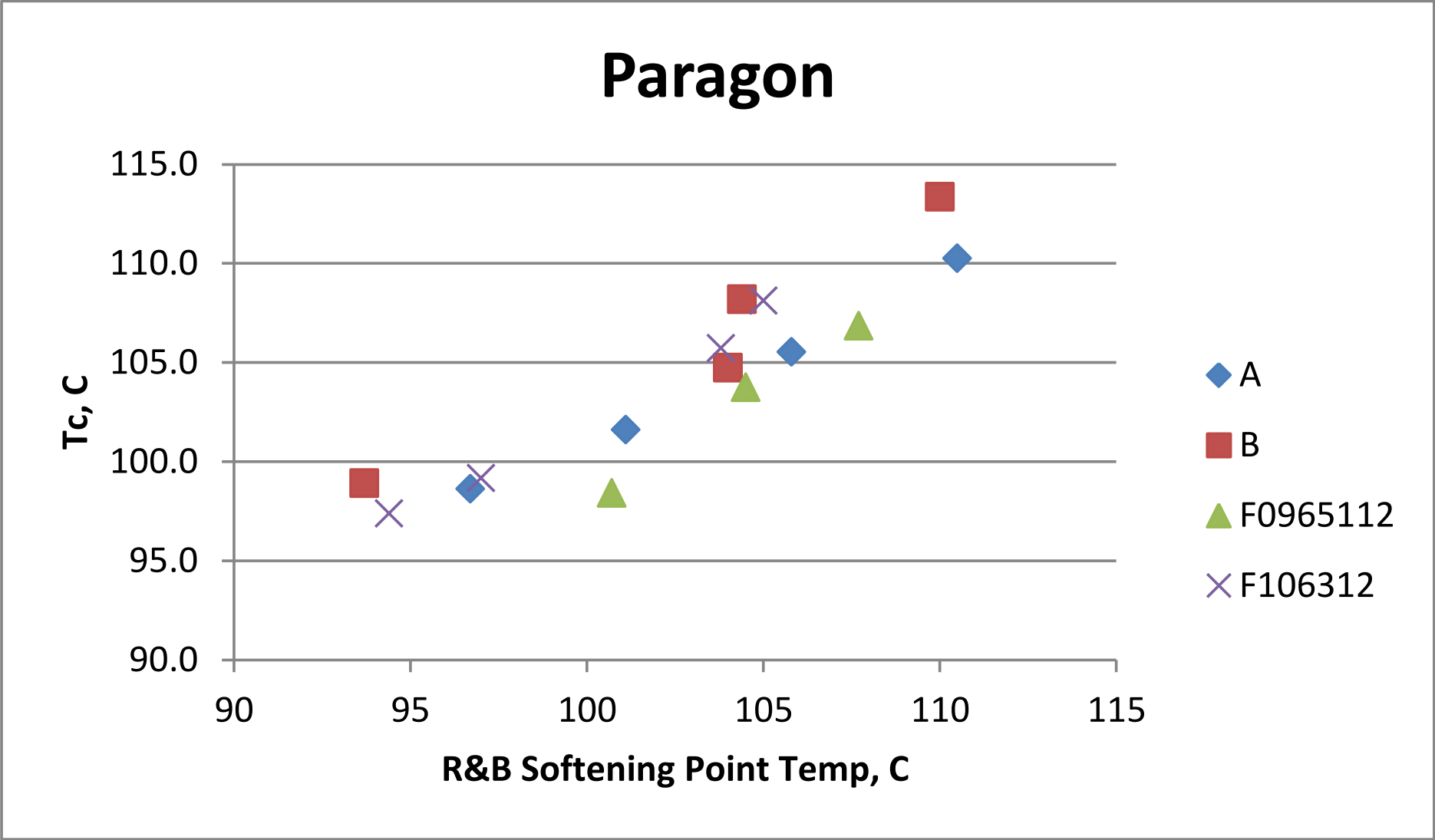


High Temperature (Paragon)

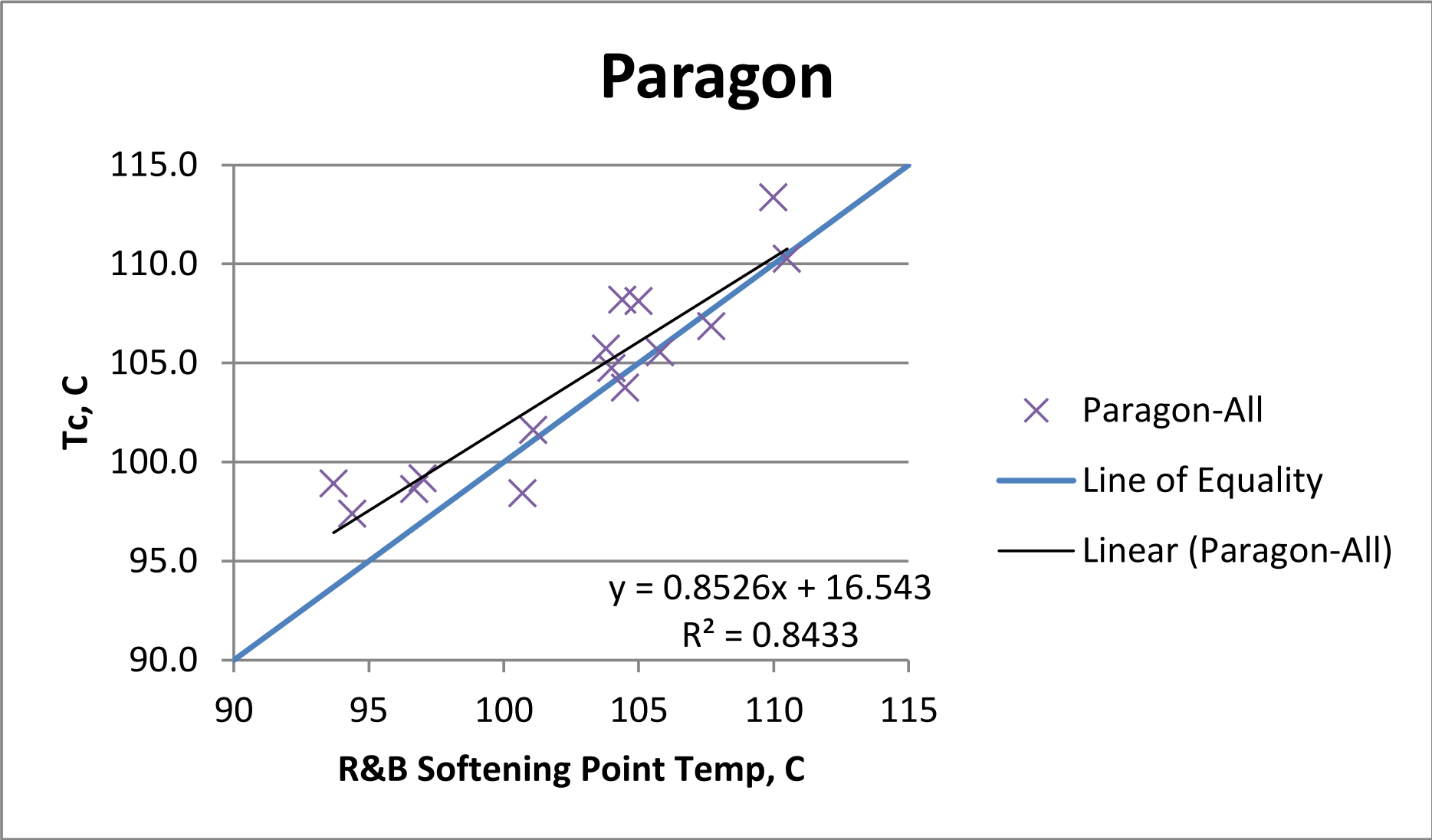
	50		71.1				50	
	2.5 rad/s		1 rad/s	2.5 rad/s	log G* = Ax + B		2.5 rad/s	R&B
	G*, Pa	δ , deg.	η^* , Pa-s	Tc, C	A	B	G(t), Pa	SP, C
F0965112-A	2.37E+05	37.7	17,742	98.4	-0.0429	7.5225		100.7
F0965112-B	3.24E+05	35.9	26,651	103.7	-0.0411	7.5649		104.5
F0965112-C	3.92E+05	34.3	34,725	106.8	-0.0403	7.6066		107.7
F106312-A	2.37E+05	39.6	17,405	97.4	-0.0443	7.6153		94.4
F106312-B	2.62E+05	39.0	19,966	99.2	-0.0435	7.6143		97
F106312-C	3.73E+05	36.2	33,240	105.7	-0.0411	7.6464		103.8
F106312-D	4.11E+05	34.8	39,113	108.1	-0.0402	7.6472		105



Calculated T_c Compared to R&B Softening Point



Calculated T_c Compared to R&B Softening Point



Data Summary – Low/Intermediate Temperature

	5		25		25		25		25
	2.5 rad/s		2.5 rad/s		100 rad/s		log G* = Ax + B		
	G*, Pa	δ , deg.	G*, Pa	δ , deg.	G*, Pa	δ , deg.	A	B	Pen, dmm
A200	2.40E+07	23.0	3.76E+06	28.5	1.12E+07	25.4	-0.0413	7.5871	18
A207	2.60E+07	21.8	4.17E+06	27.2	1.18E+07	23.9	-0.041	7.6206	16.3
A214	2.51E+07	22.0	4.31E+06	27.1	1.21E+07	24.0	-0.0394	7.596	16
A220	2.48E+07	21.4	4.53E+06	26.2	1.24E+07	23.3	-0.038	7.5853	13.3
B200	5.97E+07	19.8	1.05E+07	26.9	2.89E+07	22.9	-0.0387	7.9698	6.3
B207	7.24E+07	19.2	1.38E+07	25.8	3.61E+07	21.9	-0.037	8.0446	4.3
B214	6.58E+07	18.4	1.40E+07	24.1	3.45E+07	20.6	-0.0346	7.9911	5
B220	6.73E+07	18.3	1.48E+07	23.6	3.62E+07	20.5	-0.0338	7.9969	4.3

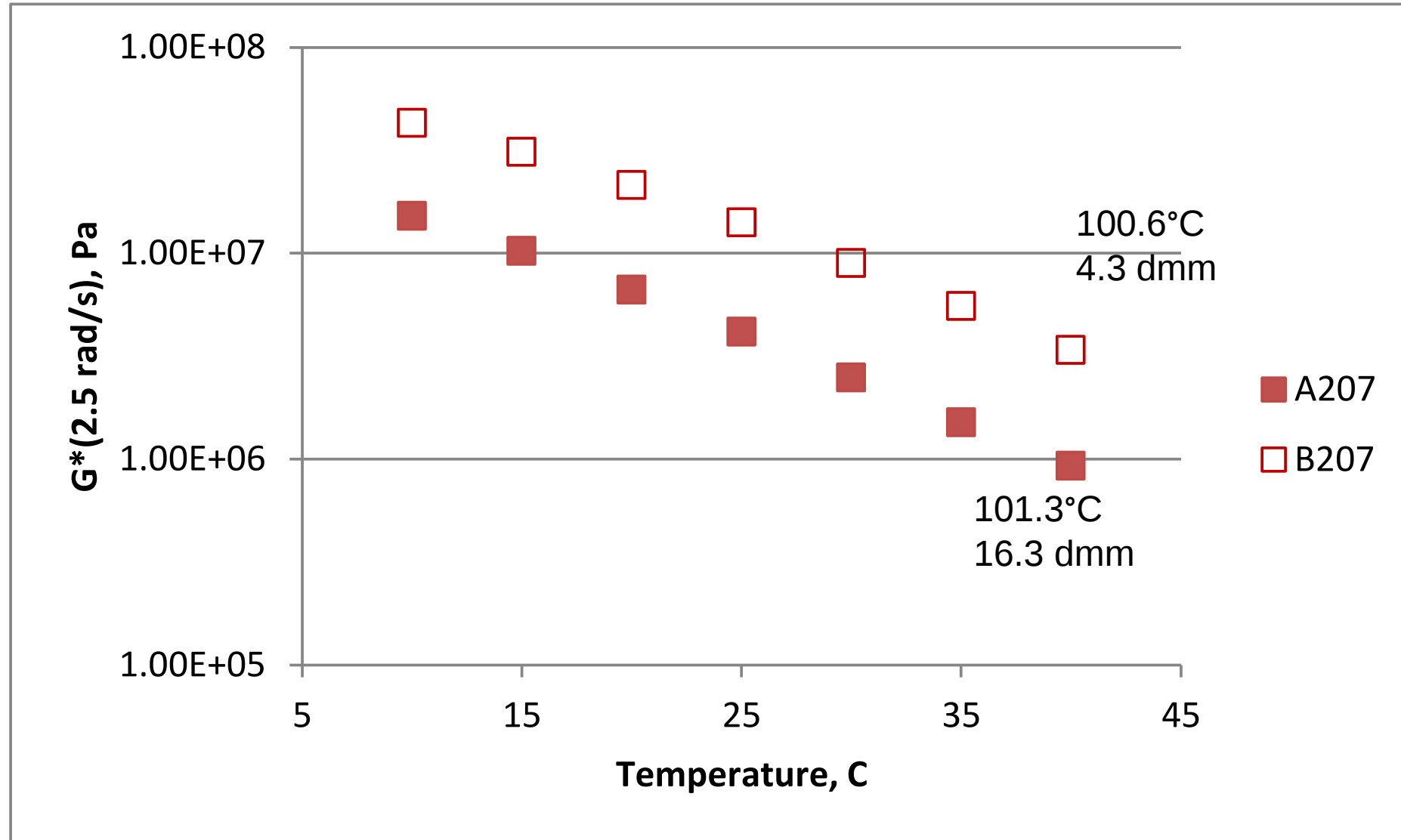


Data Summary – Low/Intermediate Temperature

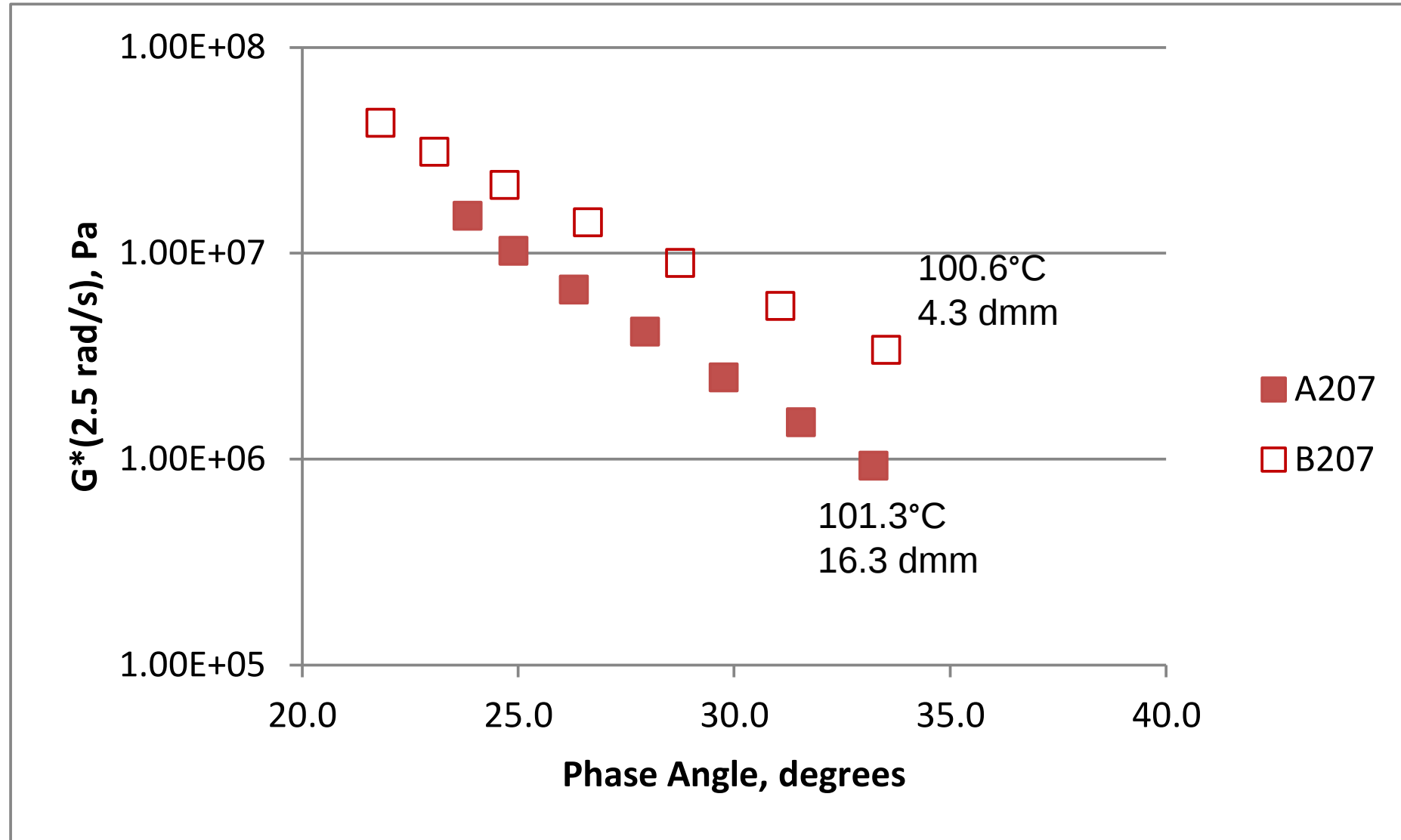
	5		25		25		25		25
	2.5 rad/s		2.5 rad/s		100 rad/s		log G* = Ax +B		
	G*, Pa	δ , deg.	G*, Pa	δ , deg.	G*, Pa	δ , deg.	A	B	Pen, dmm
F0965112-A	2.26E+07	24.0	3.55E+06	29.6	1.09E+07	25.8	-0.0407	7.5573	21
F0965112-B	2.45E+07	23.0	4.13E+06	27.8	1.21E+07	24.7	-0.0393	7.5861	20
F0965112-C	2.35E+07	22.6	4.24E+06	27.2	1.21E+07	24.1	-0.0379	7.5599	20
F106312-A	3.18E+07	23.1	4.55E+06	29.6	1.41E+07	26.0	-0.0429	7.7166	21
F106312-B	3.05E+07	22.8	4.57E+06	29.1	1.39E+07	25.7	-0.0419	7.6939	19
F106312-C	3.44E+07	22.0	5.56E+06	27.6	1.61E+07	24.4	-0.0402	7.7379	16
F106312-D	3.42E+07	21.6	5.82E+06	26.9	1.64E+07	23.9	-0.0391	7.7291	16



Similar Softening Point: $G^*(2.5 \text{ rad/s}) = f(T)$



Similar Softening Point: $G^*(2.5 \text{ rad/s}) = f(\delta)$



Gershkoff (1995)

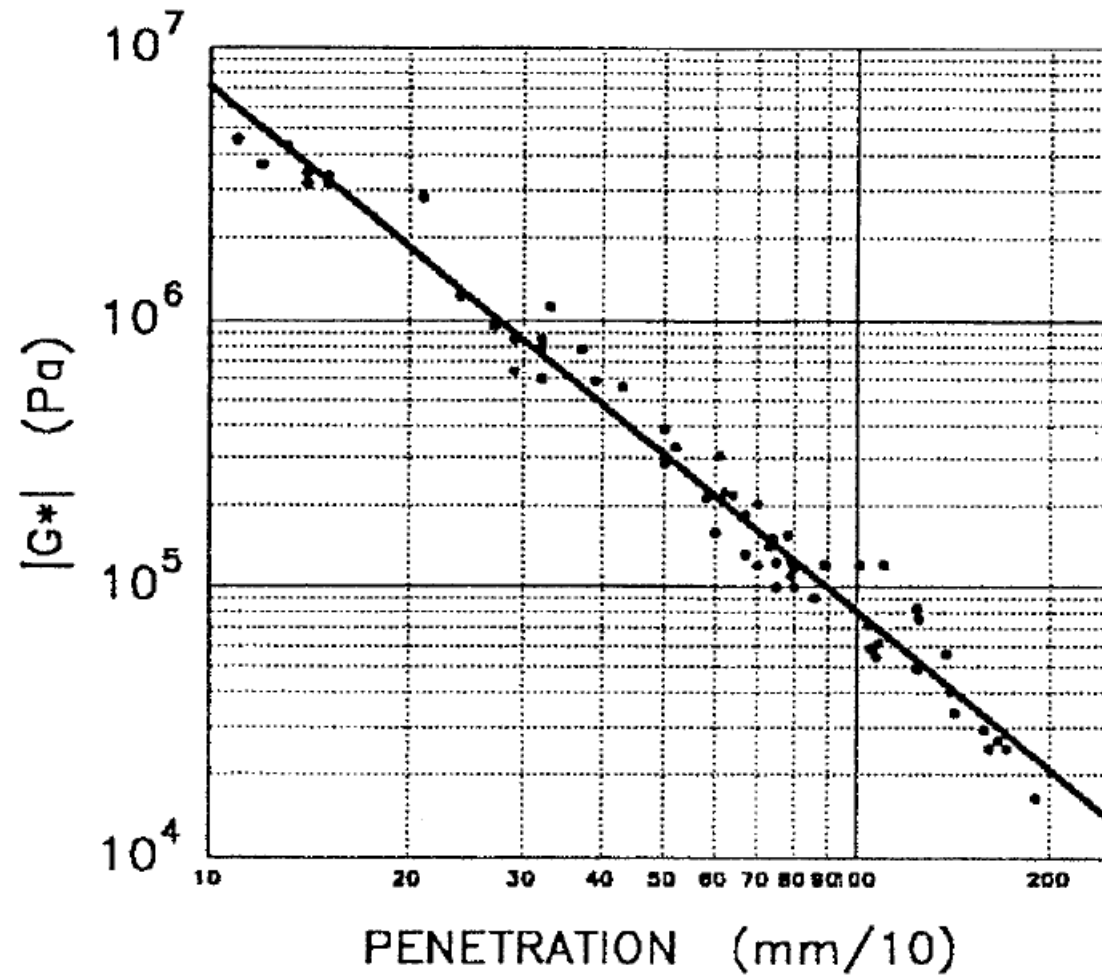
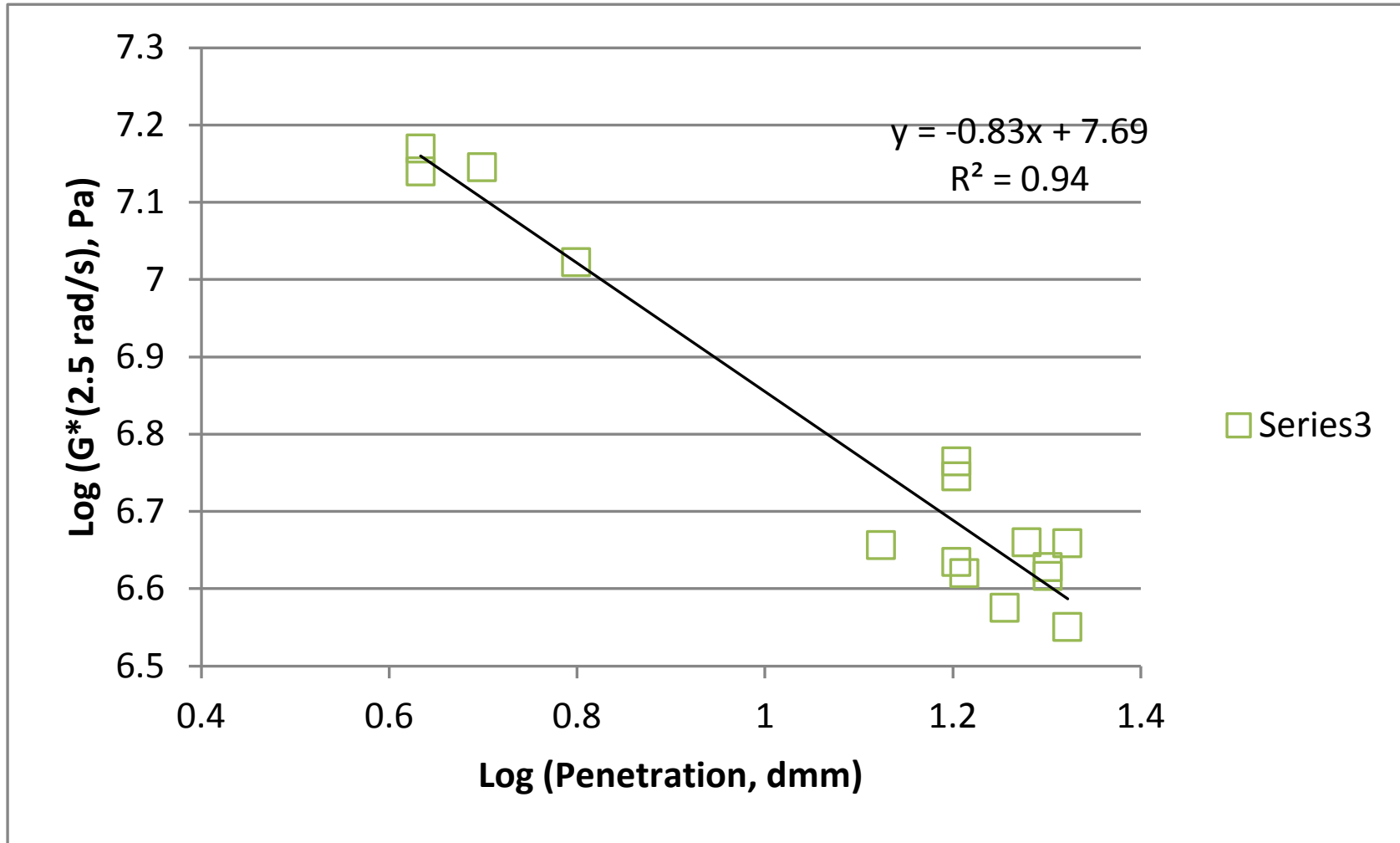


Figure 1 - Modulus at 0.4 s and Penetration Value



Relationship of $G^*(2.5 \text{ rad/s})$ to Penetration @ 25C



Gershkoff: $\text{Log}(G^*) = -1.95 \cdot \text{Log}(\text{Pen}) + 8.80$



Coating Asphalt

- Proposed Procedure to Replace Penetration at 25°C
 - Perform single-point temperature test on DSR
 - 8-mm parallel plate, 1% strain, 2.5 rad/s, 25°C
 - Determine $G^*_{2.5}$ and compare to recommended specification value



Optional Rheological Testing

- Intermediate Temperature DSR – Determination of $G^*_{2.5}$ at 25°C
 - Generally follow the equipment and specimen preparation procedures as described in ASTM D7175, *Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer*.
 - Use 8-mm parallel plate geometry with a 2-mm gap.

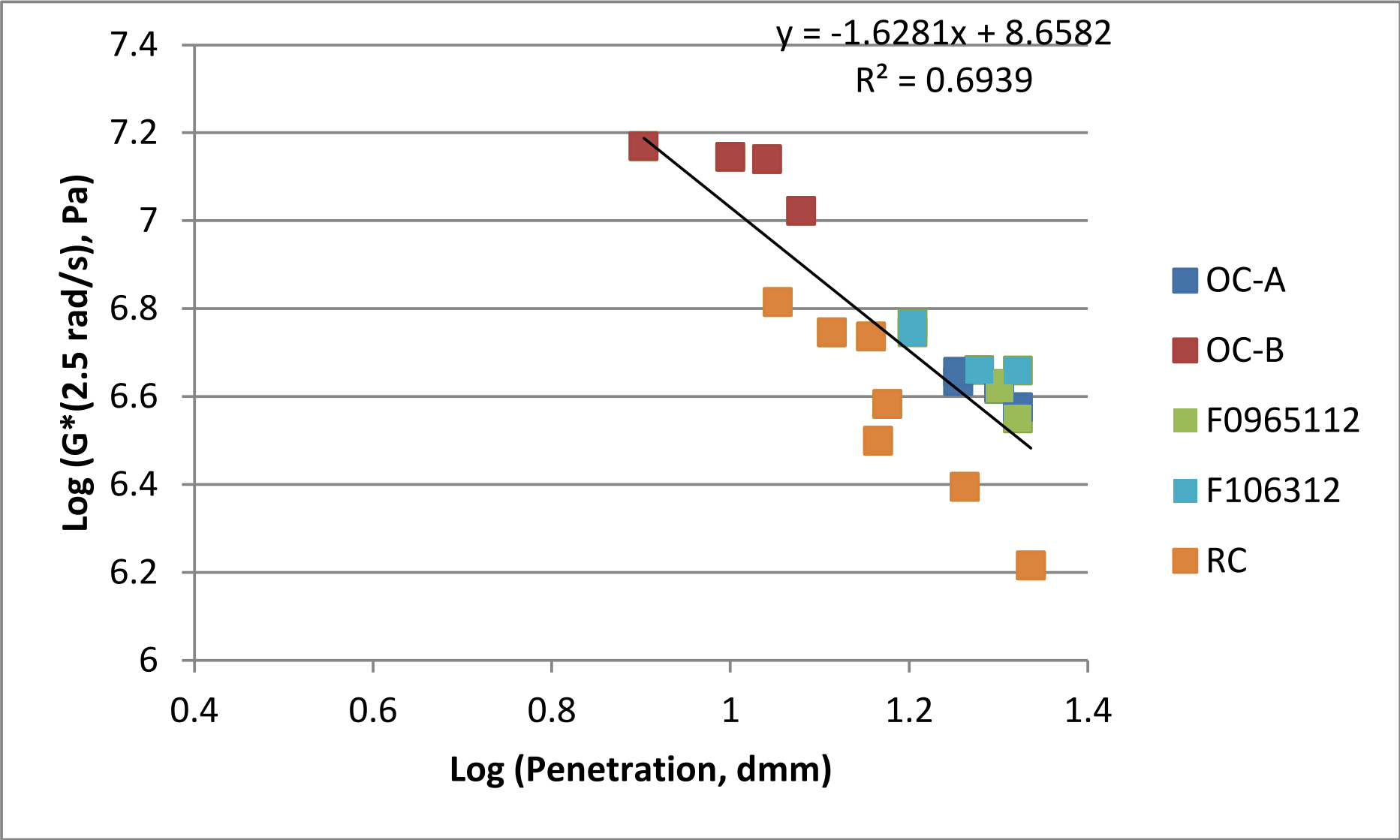


Optional Rheological Testing

- Intermediate Temperature DSR – Determination of $G^*_{2.5}$ at 25°C
 - Set the gap at 25°C. Increase the temperature to 75°C and load, trim the sample. Lower the spindle to the testing gap (2 mm). Reduce the temperature to 25°C and allow the sample to achieve temperature equilibrium before testing.
 - Start testing at 25°C in oscillatory mode using 1% shear strain and a loading frequency of 2.5 rad/s. Determine the complex modulus, G^* , in Pa.
 - Unload the sample and clean the DSR.



Relationship Between $G^*_{2.5}$ and Penetration at 25°C



Relationship Between $G^*_{2.5}$ and Penetration at 25°C

Sample	Penetration (25C), dmm			
	>20	16-20	11-15	≤10
A200	3.76E+06			
A207		4.17E+06		
A214		4.31E+06		
A220		4.53E+06		
B200			1.05E+07	
B207			1.38E+07	
B214				1.40E+07
B220				1.48E+07
F0965112-A	3.55E+06			
F0965112-B		4.13E+06		
F0965112-C		4.24E+06		
F106312-A	4.55E+06			
F106312-B		4.57E+06		
F106312-C		5.56E+06		
F106312-D		5.82E+06		
RC-01			5.56E+06	
RC-02			6.53E+06	
RC-03			3.82E+06	
RC-04			3.15E+06	
RC-05		2.48E+06		
RC-06	1.64E+06			
RC-07			5.45E+06	



Relationship Between $G^*_{2.5}$ and Penetration at 25°C

	>20	16-20	11-15	≤10
Ave $G^*_{2.5}$ (25°C)	3.37E+06	4.42E+06	6.98E+06	1.44E+07
Ave Pen (25°C)	21	18	13	9
# of Samples	4	9	7	2



Relationship Between $G^*_{2.5}$ and Penetration at 25°C

- Relationship from all data:
 - $\text{Log } G^*_{2.5} = -1.6281 * \text{Log}(\text{Pen}) + 8.6582$
- Penetration = 12 dmm
 - $G^*_{2.5} = 7.96\text{E}+06 \text{ Pa}$
- Penetration = 25 dmm
 - $G^*_{2.5} = 2.41\text{E}+06 \text{ Pa}$



Relationship Between $G^*_{2.5}$ and Penetration at 25°C

- Proposed Specification Values

$$2.50\text{E}+06 \leq G^*_{2.5}(25^\circ\text{C}) \leq 8.00\text{E}+06$$

- Testing Variability for T315 (PAV DSR)

- single-operator d2s% = 13.8%

- Acceptable range

7.45E+06 to 8.55E+06 Pa

2.33E+06 to 2.67E+06 Pa



Relationship Between $G^*_{2.5}$ and Penetration at 25°C

- Acceptable range for variability
7.45E+06 to 8.55E+06 Pa
12.5 to 11.5 dmm calculated Pen values
2.33E+06 to 2.67E+06 Pa
25.5 to 23.5 dmm calculated Pen values
- Testing variability for Penetration
 - single-operator d2s = 2.3 dmm (Pen < 60)



Coating Asphalt

- Proposed Procedure to Replace Softening Point
 - Perform temperature sweep on DSR
 - 25-mm parallel plate, 10% strain, 1 rad/s
 - Start at 90°C, increase to 110°C in 10°C increments
 - Plot $\eta^*(1 \text{ rad/s})$ versus temperature on semi-log graph
 - Calculate T_c where $\eta^*(1 \text{ rad/s}) = 1200 \text{ Pa-s}$



Optional Rheological Testing

- High Temperature DSR – Determination of T_c where $\eta^* = 1200 \text{ Pa-s}$
 - Generally follow the equipment and specimen preparation procedures as described in ASTM D7175, *Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer*.
 - Use 25-mm parallel plate geometry with a 1-mm gap.
 - Load, trim the sample at 90°C. Allow the sample to achieve temperature equilibrium at 90°C before testing.



Optional Rheological Testing

- High Temperature DSR – Determination of T_c where $\eta^* = 1200 \text{ Pa-s}$
 - Start testing at 90°C in oscillatory mode using 10% shear strain and a loading frequency of 1 rad/s. Determine the complex viscosity, η^* , in Pa-s.
 - Without removing the sample, increase the temperature to 100°C . Allow the sample to achieve temperature equilibrium at 100°C before testing.
 - Start testing at 100°C in oscillatory mode using 10% shear strain and a loading frequency of 1 rad/s. Determine the complex viscosity, η^* , in Pa-s.



Optional Rheological Testing

- High Temperature DSR – Determination of T_c where $\eta^* = 1200 \text{ Pa-s}$
 - Without removing the sample, increase the temperature to 110°C . Allow the sample to achieve temperature equilibrium at 110°C before testing.
 - Start testing at 110°C in oscillatory mode using 10% shear strain and a loading frequency of 1 rad/s. Determine the complex viscosity, η^* , in Pa-s.
 - Unload the sample and clean the DSR.

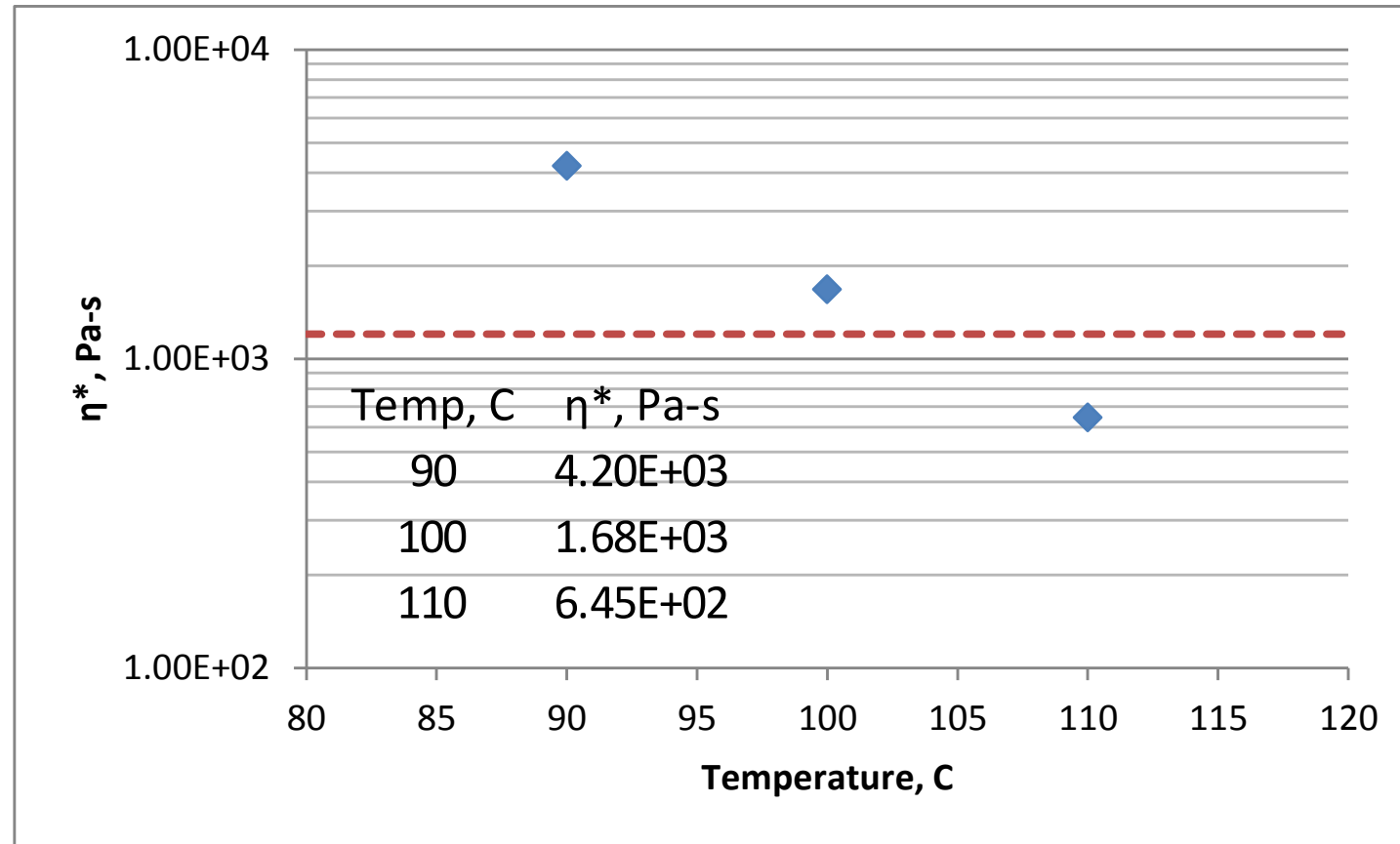


Optional Rheological Testing

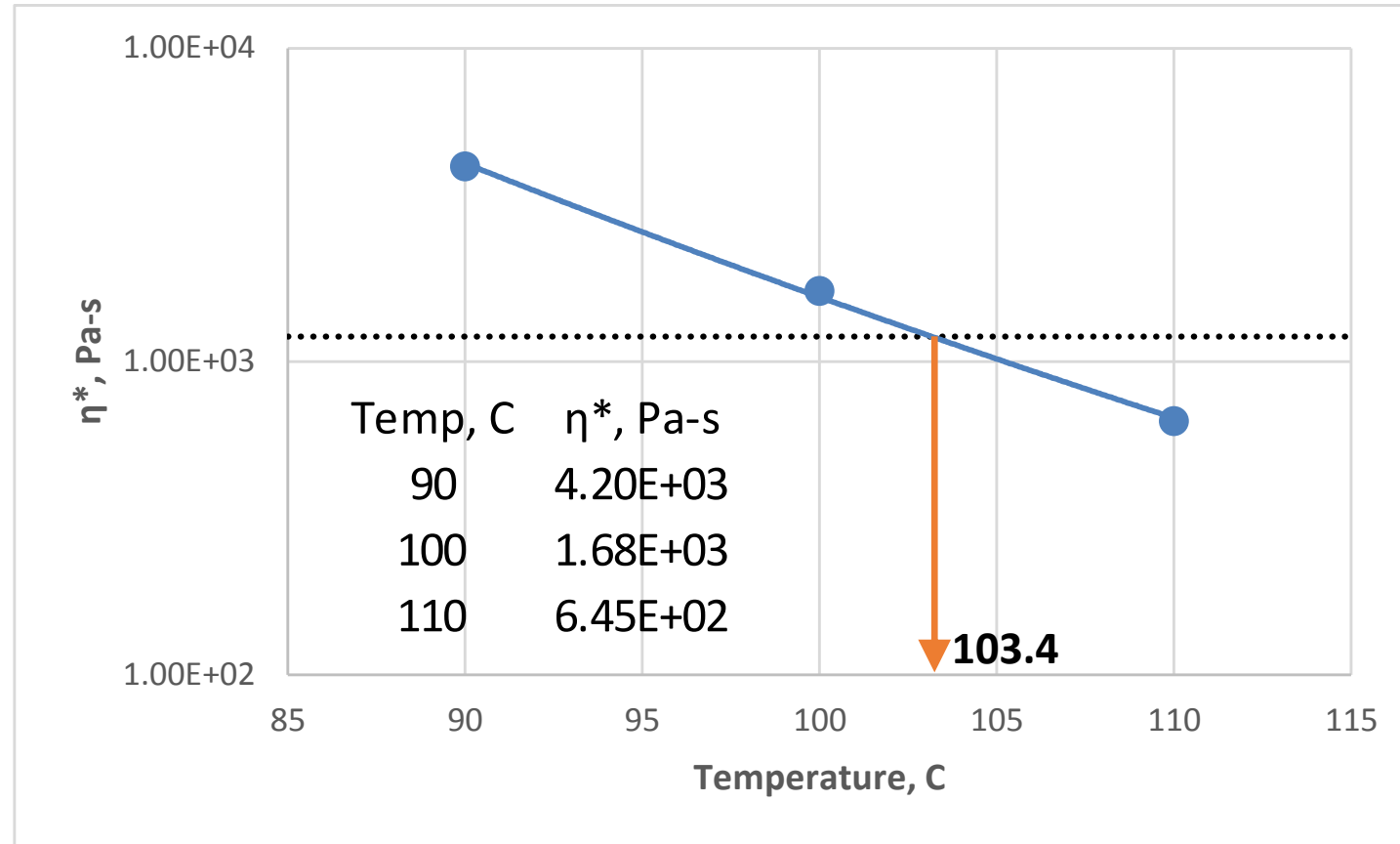
- High Temperature DSR – Determination of T_c where $\eta^* = 1200 \text{ Pa-s}$
 - Plot complex viscosity, η^* , as a function of temperature. Determine the temperature, to the nearest 0.1°C , where η^* is equal to 1200 Pa-s . This temperature may be interpolated or extrapolated.



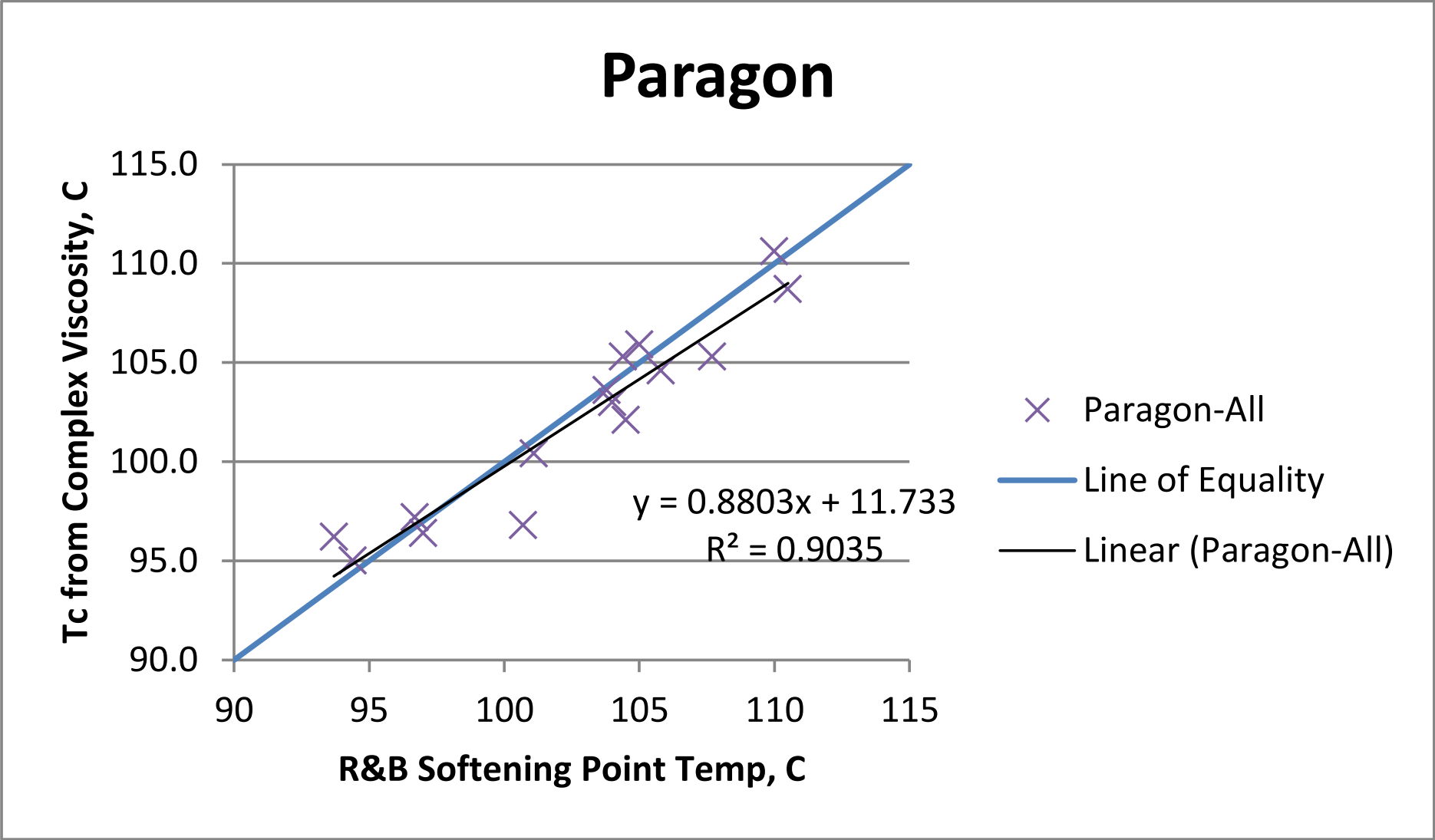
Optional Rheological Testing (2014)



Optional Rheological Testing (2014)



Relationship Between Measured R&B Softening Point and T_c (based on $\eta^*_1 = 1200 \text{ Pa-s}$)



Roofing PSP: Rheological Testing (2017)

	T _c @ $\eta^*=1200$ Pa-s, °C	R&B Softening Point, °C
Lab 2	107.7	110.0
Lab 3	110.5	106.7
Lab 4	108.4	109.4
Lab 5	111.3	108.0
Lab 6	108.8	107.8
Lab 7	107.5	107.8
Lab 9	110.0	112.2
Lab 11	109.0	110.6
Lab 13	111.4	110.0
Lab 14	112.0	110.0
Lab 15	108.7	109.9
Lab 16	109.4	112.2



Roofing PSP: Rheological Testing (2017)

	Tc @ $\eta^*=1200$ Pa-s, °C	R&B Softening Point, °C
Average	109.6	109.5
StDev	1.4	1.6



Roofing PSP: Rheological Testing (2017)

	Meas. $G^*_{2.5}$ @25°C, Pa	Penetration @ 25°C, dmm
Lab 2	4.81E+06	17
Lab 3	3.24E+06	16
Lab 4	4.72E+06	18
Lab 5	3.64E+06	15
Lab 6	4.50E+06	16.3
Lab 7	4.53E+06	18
Lab 11	4.21E+06	20
Lab 13	4.64E+06	12
Lab 14	5.12E+06	16
Lab 15	3.52E+06	16.1
Lab 16	4.60E+06	15

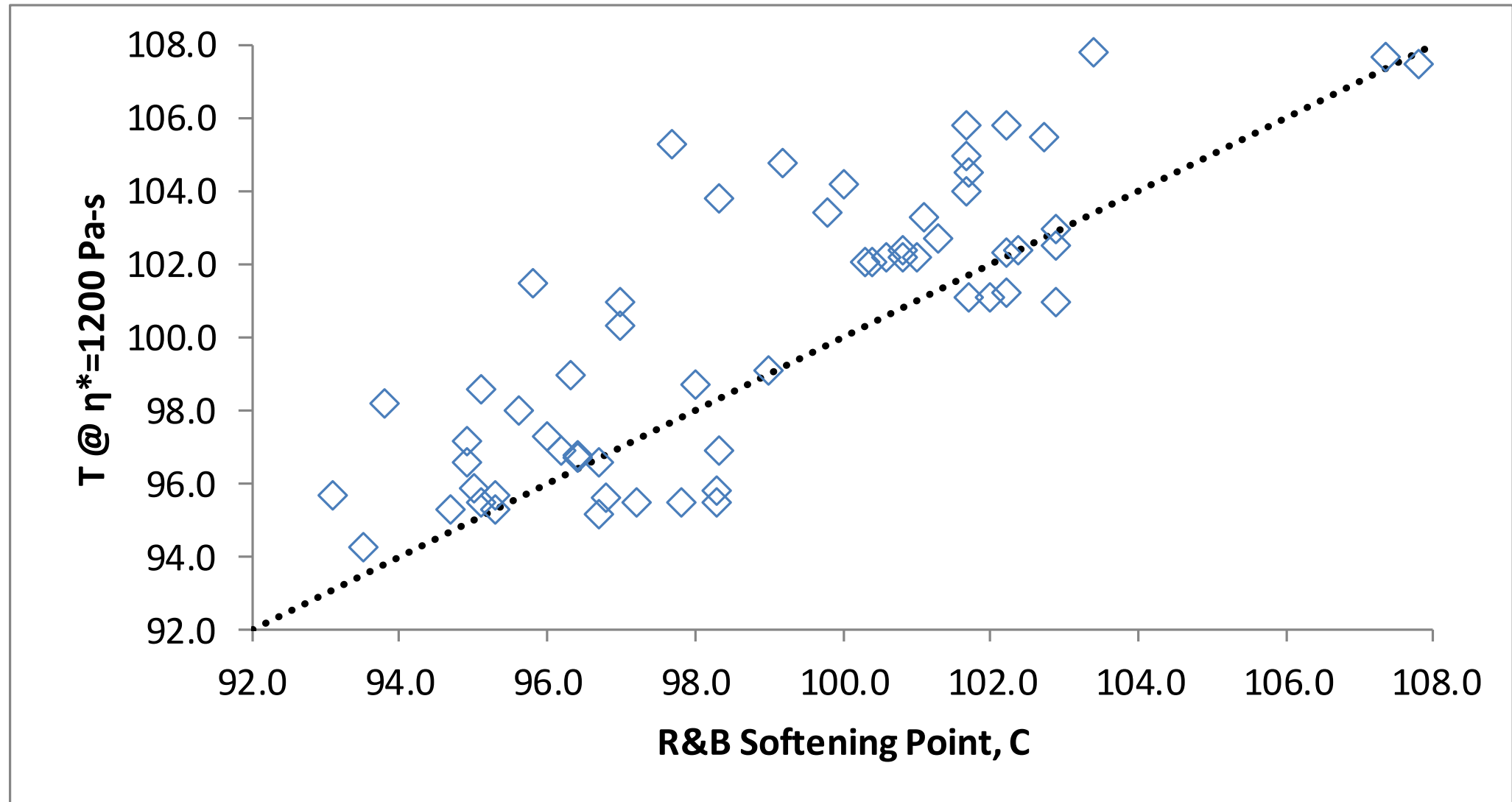


Roofing PSP: Rheological Testing (2017)

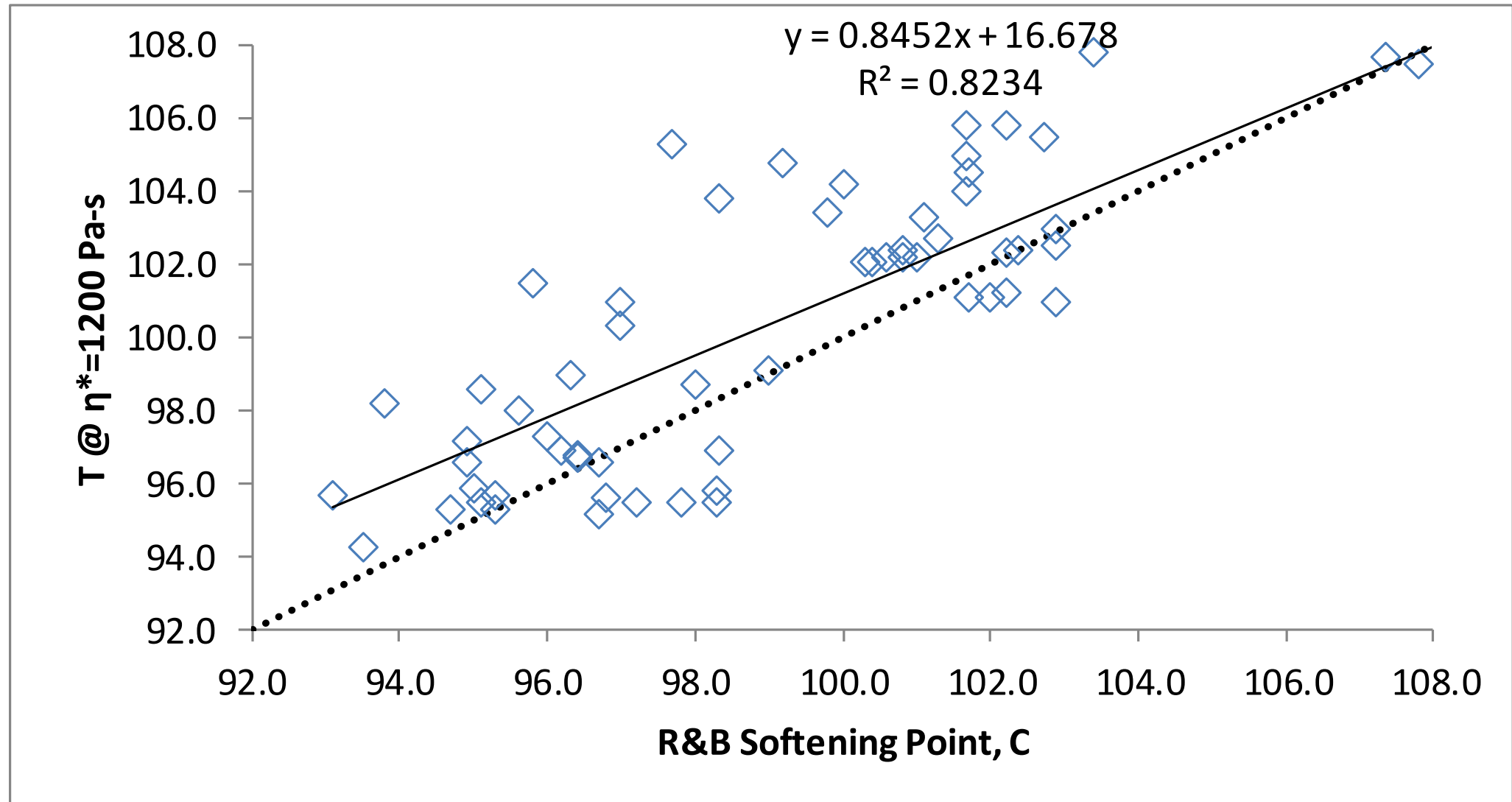
	Meas. $G^*_{2.5}$ @25°C, Pa	Penetration @ 25°C, dmm
Average	4.32E+06	16.3
StDev	0.57E+06	2.0
CV	13.2%	12.0%



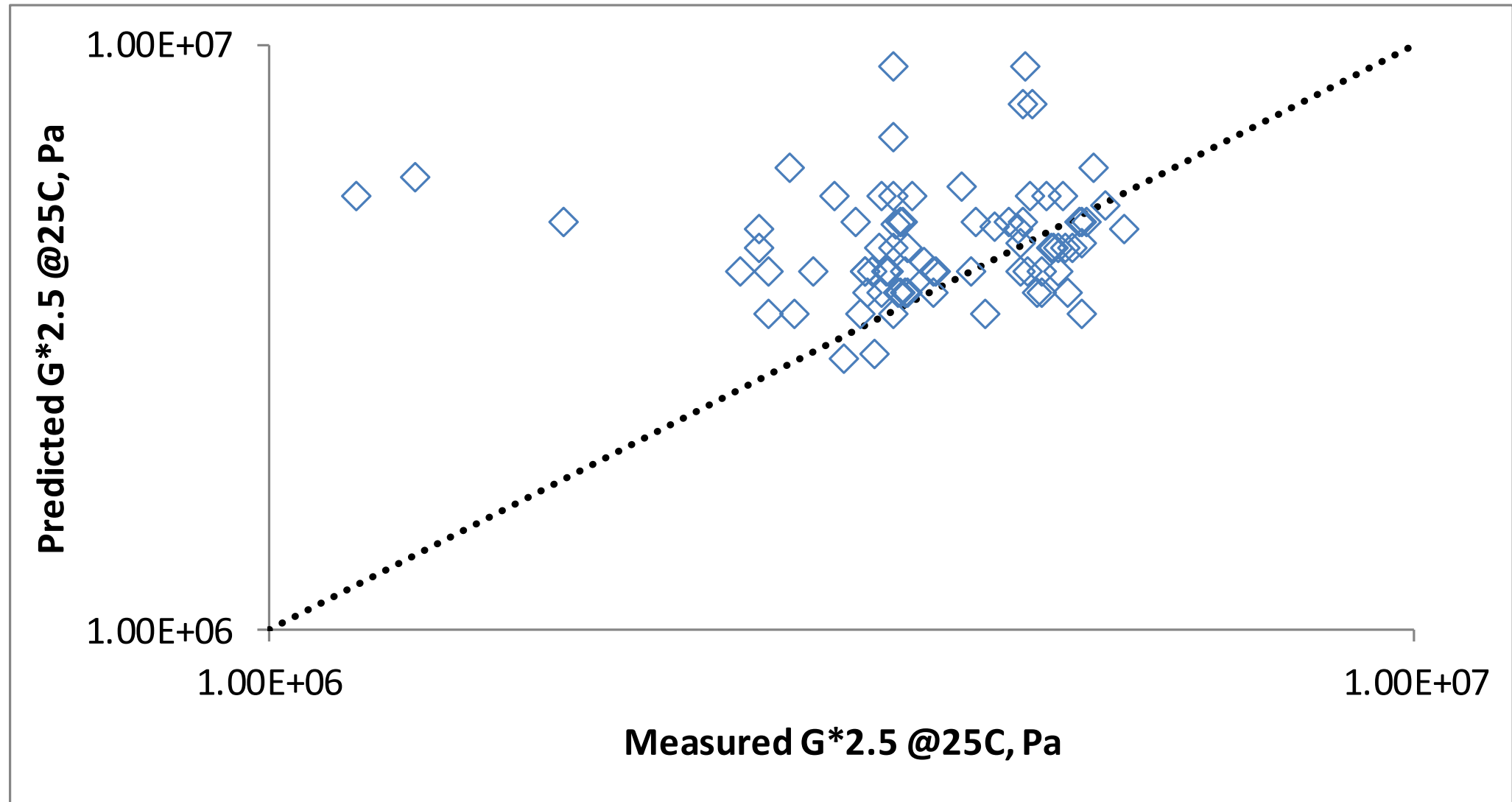
Roofing PSP: Rheological Testing (2014-17)



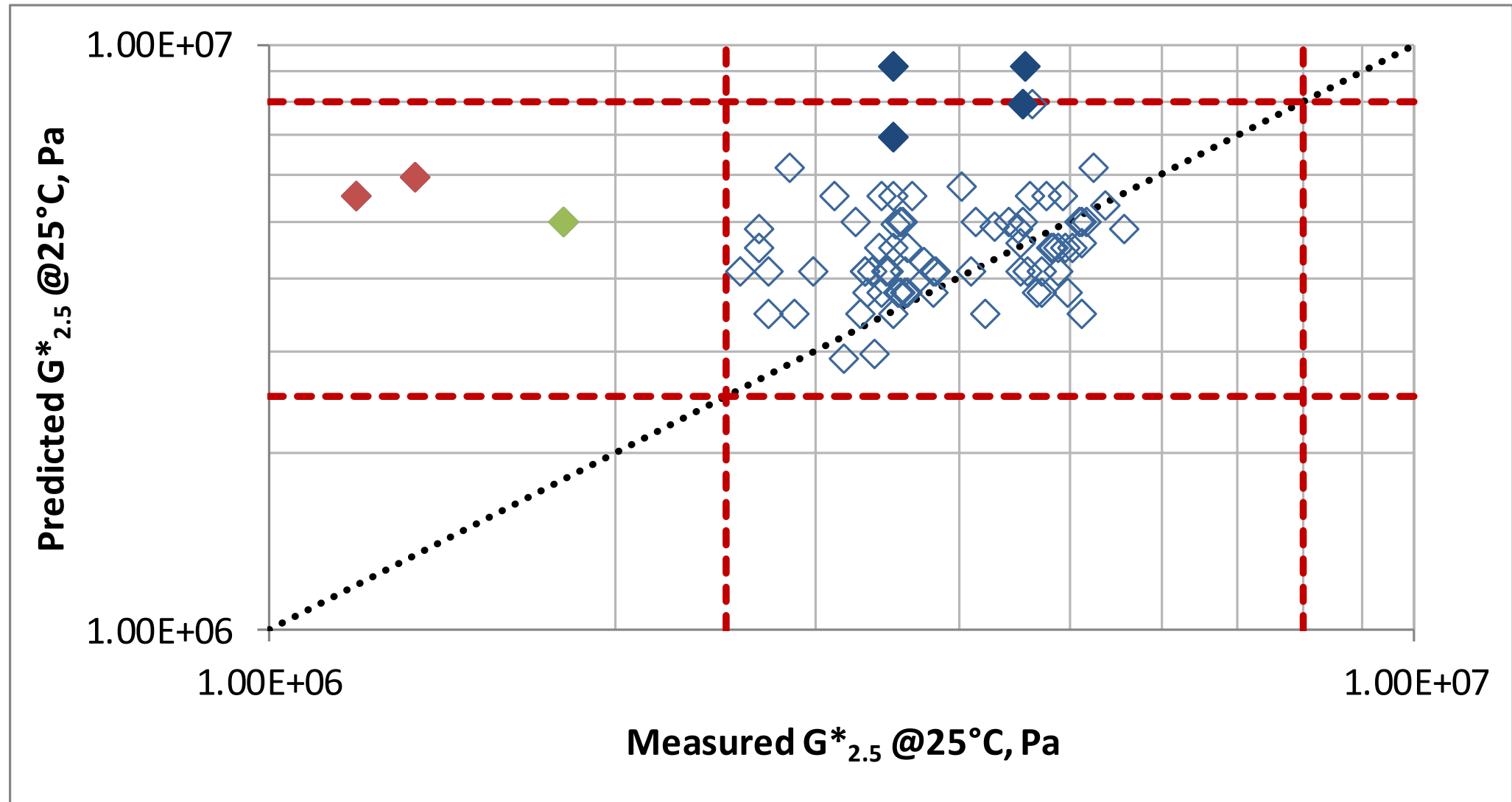
Roofing PSP: Rheological Testing (2014-17)



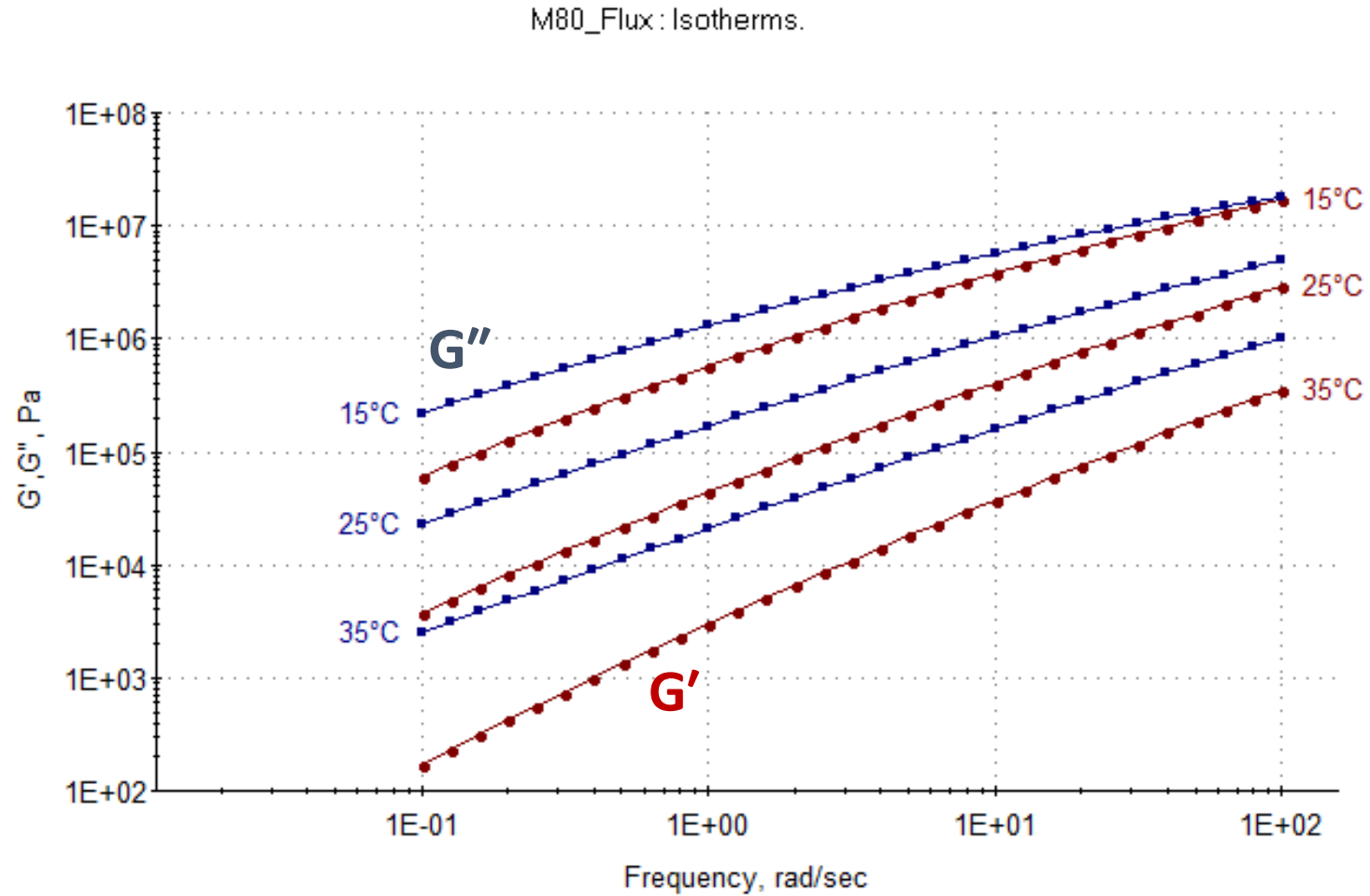
Roofing PSP: Rheological Testing (2014-17)



Roofing PSP: Rheological Testing (2014-17)



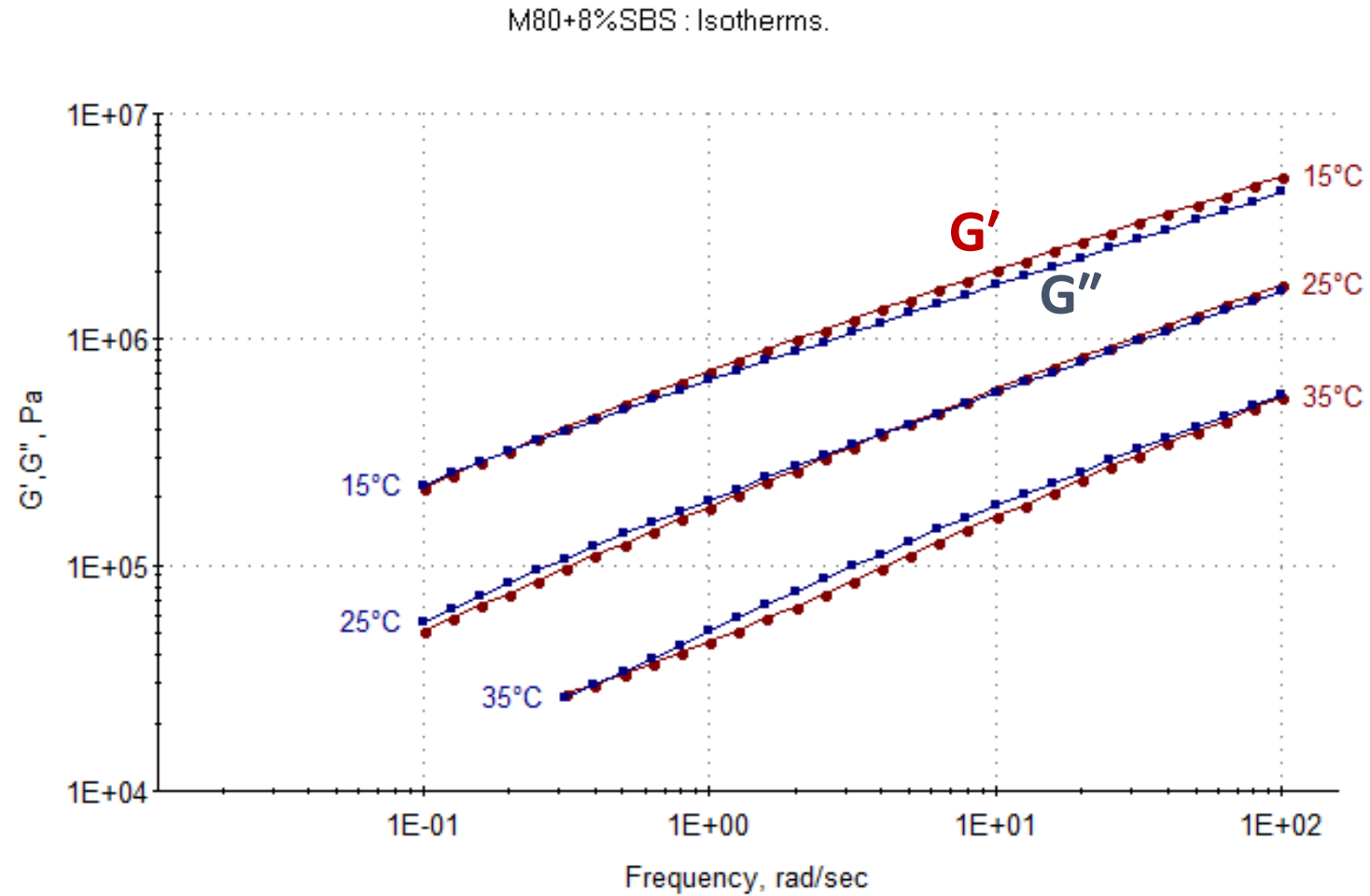
Isotherms: Flux (80 PEN)



Sample ID: M80_Flux_rhea



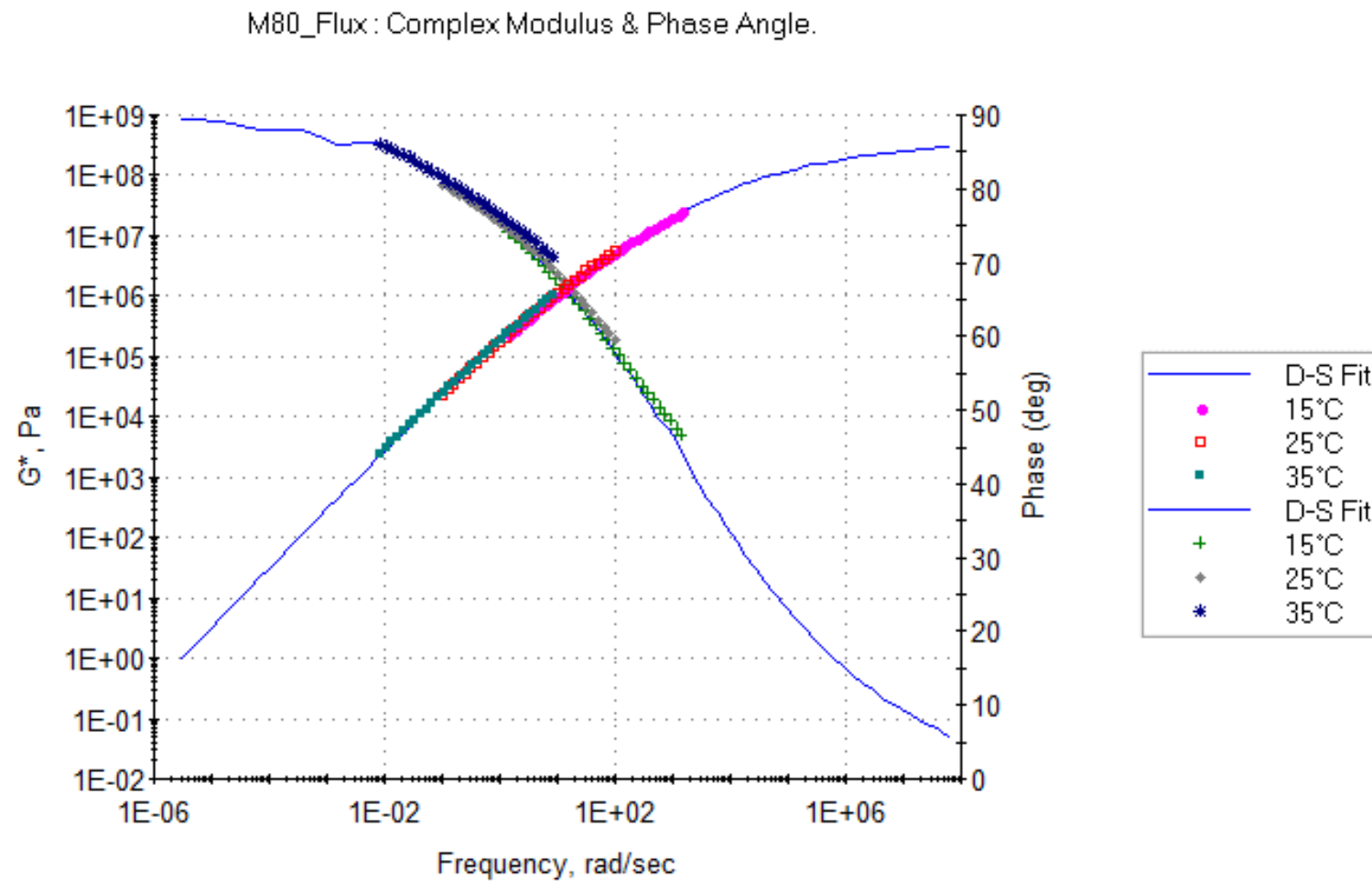
Isotherms: Flux (80 PEN) + 8% SBS



Sample ID: Mal-8SBS_rhea



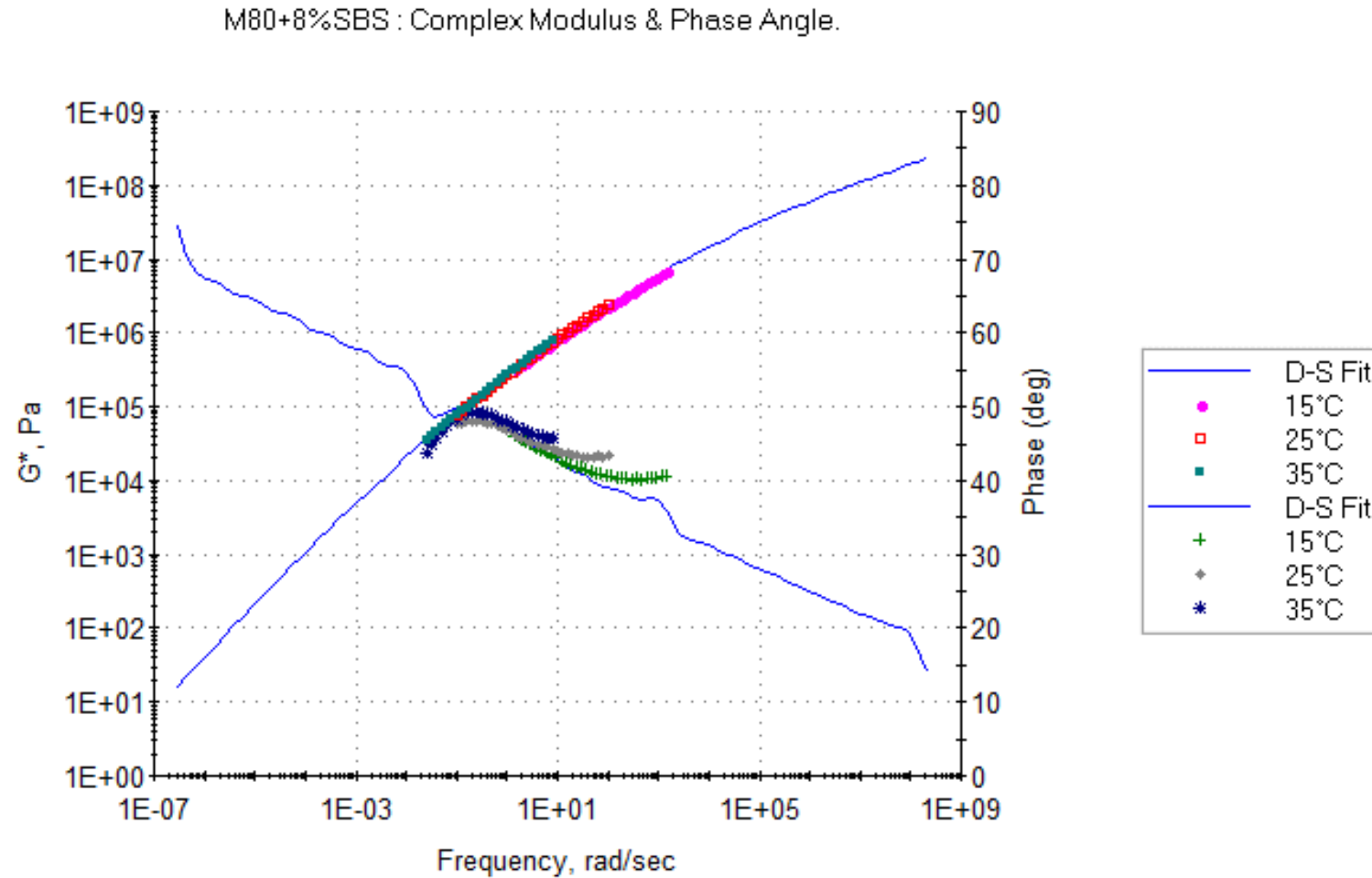
Complex Modulus (G^*) and Phase Angle (δ): Flux (80 PEN)



Sample ID: M80_Flux_rhea



Complex Modulus (G^*) and Phase Angle (δ): Flux (80 PEN) + 8% SBS

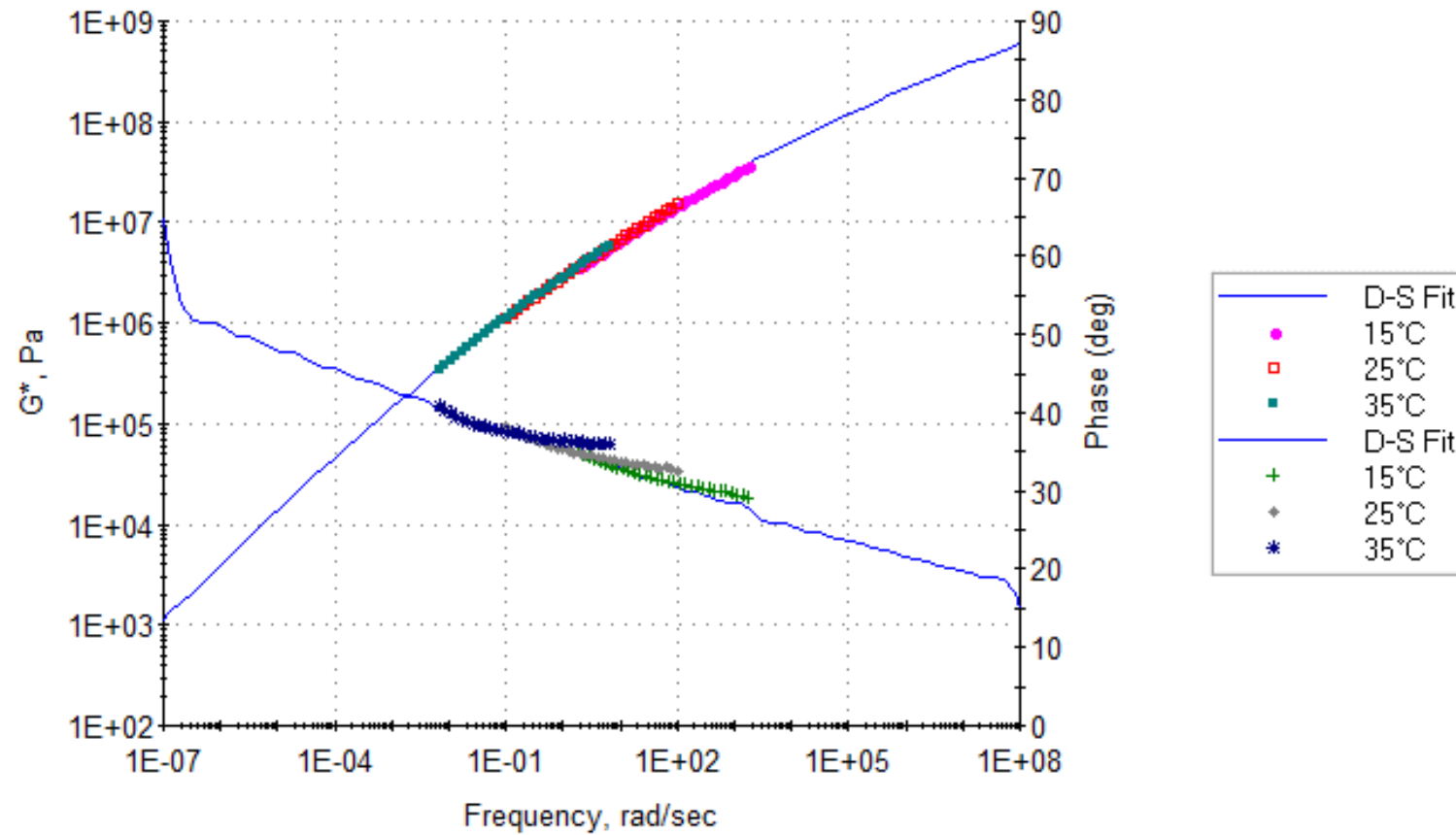


Sample ID: Mal-8SBS_rhea



Complex Modulus (G^*) and Phase Angle (δ): Coating

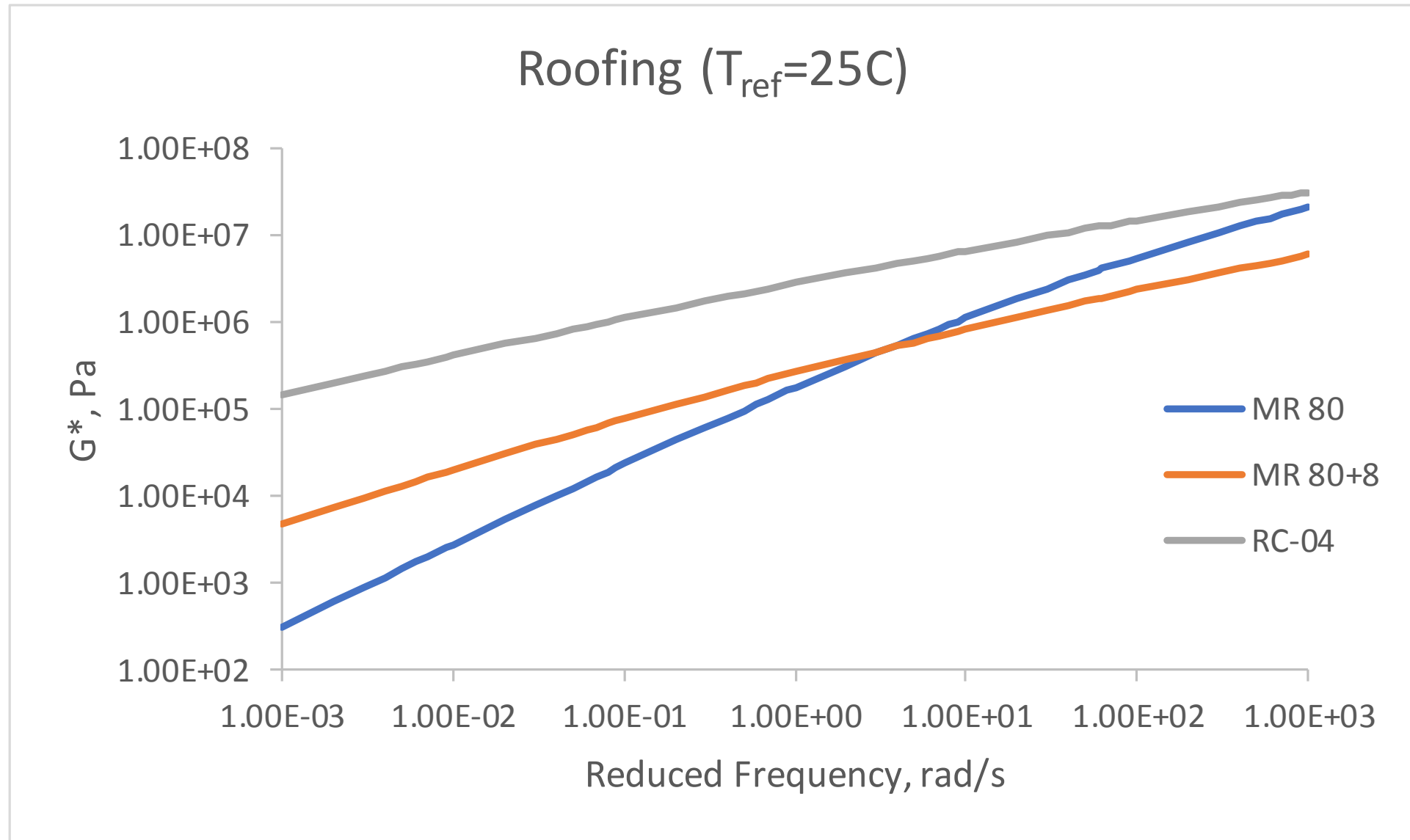
RC-04 : Complex Modulus & Phase Angle.



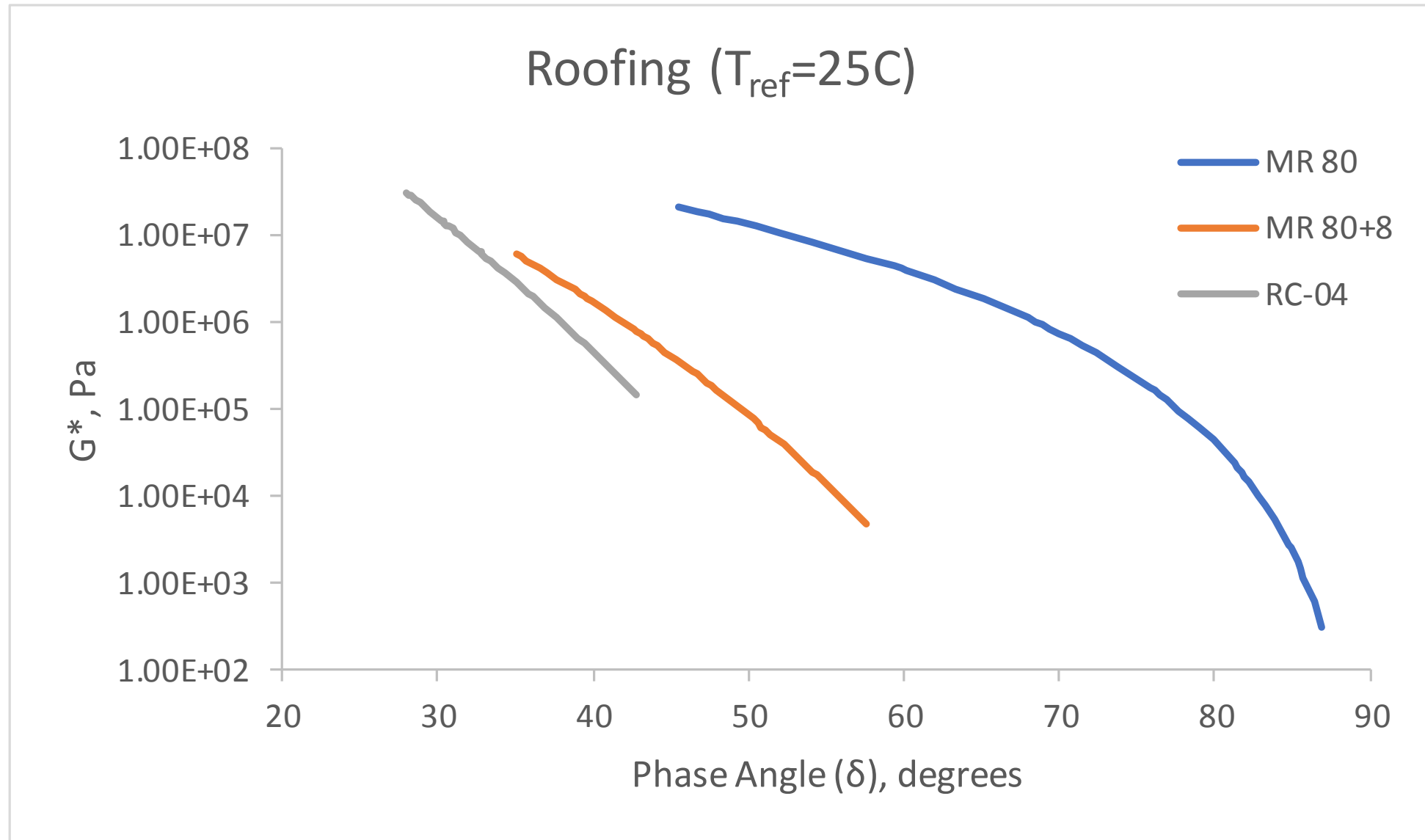
Sample ID: RC-04_rhea



Mastercurves at 25°C (Frequency Dependence)



Black Space at 25°C



Summary: Rheological Testing of Roofing Asphalt

- Rheological Parameters
 - Respond rationally to changes in softening point and penetration
 - Effects of continued oxidation are readily seen in increased G^* and δ decreased
 - Not a direct correlation
 - Shouldn't necessarily be expected since R&B Softening Point and Penetration are less fundamental (more empirical)
 - Variability appears reasonable compared to conventional tests
 - More information available from mastercurve analysis



Thanks!

Global, International, Regular, Associate and Canadian members



Affiliate and Commercial members

