Recycled Engine Oil Bottoms as Asphalt Binder Additive

Terry Arnold & Nelson Gibson

North Central Asphalt User Producer Group Feb 3 2015

Acknowledgments

- State DOTs and FHWA Federal Lands
- Crystal Clean / Heritage Research Group
- Safety-Kleen
- SES Group / Turner Fairbank Highway Research Center
  - Anant Shastry
  - Susan Needham
  - Scott Parobeck
  - Frank Davis
  - Adrian Andriescu
  - Xinjun Li
  - Lakesha Perry

AASHTO Task Force

Christopher Abadie	Louisiana DOT
Bill Ahearn	Vermont DOT (leader)
Terry Arnold	FHWA
Richard Bradbury	Maine DOT
Matthew Corrigan	FHWA
Nelson Gibson	FHWA
John Grieco	Massachusetts DOT
Matt Mueller	Illinois DOT
Christopher Peoples	North Carolina DOT
Michael San Angelo	Alaska DOT
Eileen Sheehy	New Jersey DOT

AASHTO Task Force Goals and Objectives

1. Develop a Common Understanding of the published information about REOB – mid January

2. Finalize a State by State summary of REOB specification/use status IBNLT known or unknown – end January

3. Define data gaps in knowledge and timeframes for resolution – mid February

4. Develop consensus on risk and recommended action – end February

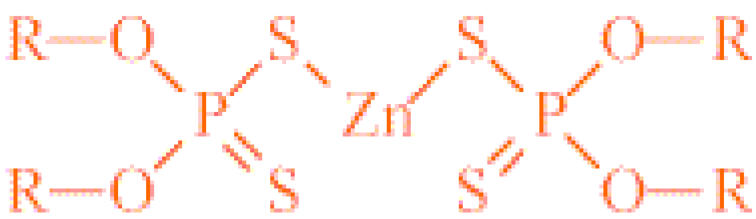
5. Finalize response to SCOH – early March

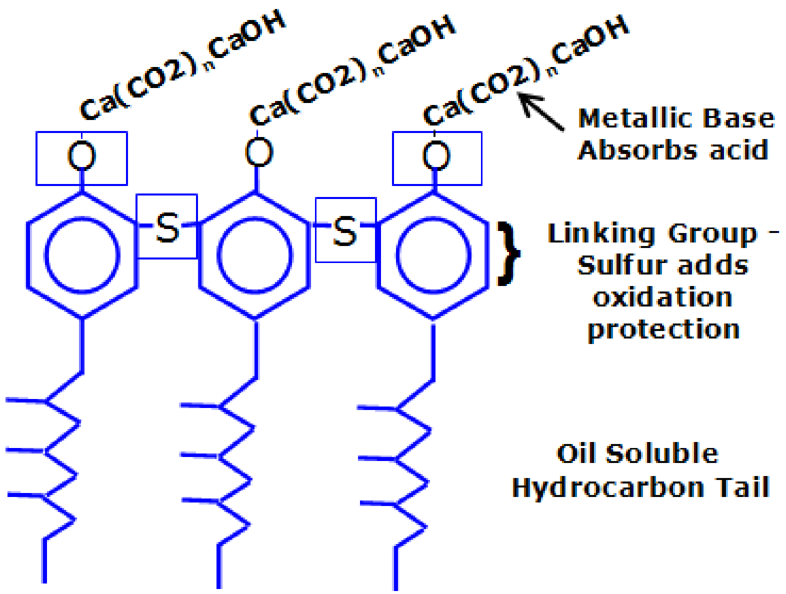
Recycled Engine Oil Bottoms are Liquids at Room Temperature

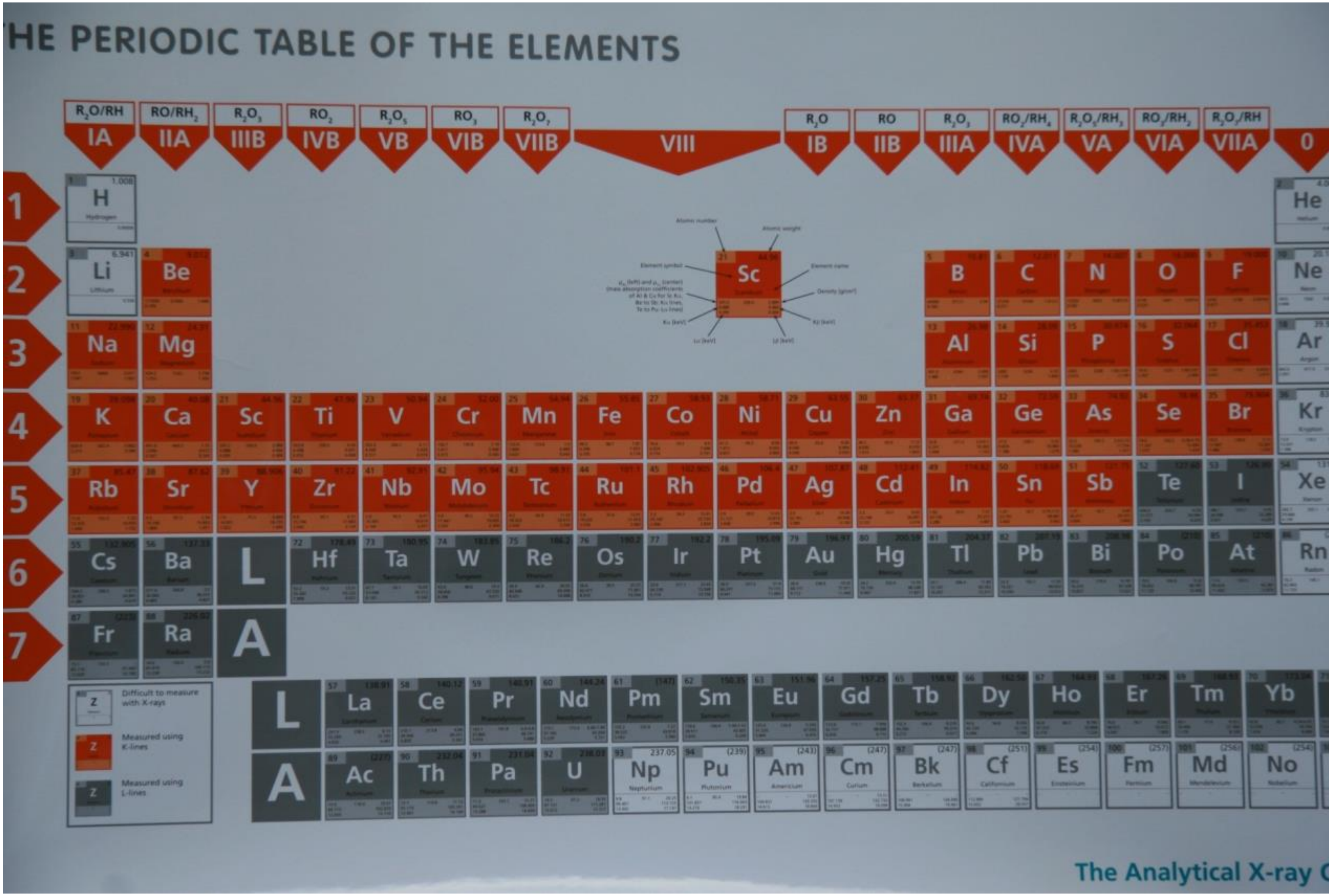
Their Brookfield Viscosities Differ Widely Between Producers

- **Producer A: 257.3 cps @ 135^{0}C**
- **Producer B: 28.2 cps @ 135^{0}C**

Lubricating Oil additives






HE PERIODIC TABLE OF THE ELEMENTS
IA IIA IIIB IVB VB VIB VIIB VIII IB IIB IIIA IVA VA VIA VIIA 0
1 H He
2 Li Be B C N O F Ne
3 Na Mg Al Si P S Cl Ar
4 K Ca Sc Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Br Kr
5 Rb Sr Y Zr Nb Mo Tc Ru Rh Pd Ag Cd In Sn Sb Te I Xe
6 Cs Ba L Hf Ta W Re Os Ir Pt Au Hg Tl Pb Bi Po At Rn
7 Fr Ra A
L La Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb
A Ac Th Pa U Np Pu Am Cm Bk Cf Es Fm Md No
Difficult to measure with X-rays
Measured using K-lines
Measured using L-lines
The Analytical X-ray C

XRF-Spectrometer


AS1-144-7

Variation and Complications
between and within REOB Suppliers

• Phosphorous	1.5 - 1.9%
• Sulfur	1.5 - 1.9%
• Calcium	7,204 - 10,901ppm
• Iron	372 - 1,838 ppm
• Copper	704 - 1,563 ppm
• Zinc	4,554 - 7,213 ppm
• Molybdenum	288 - 669 ppm

Variation and Complications – Asphalt

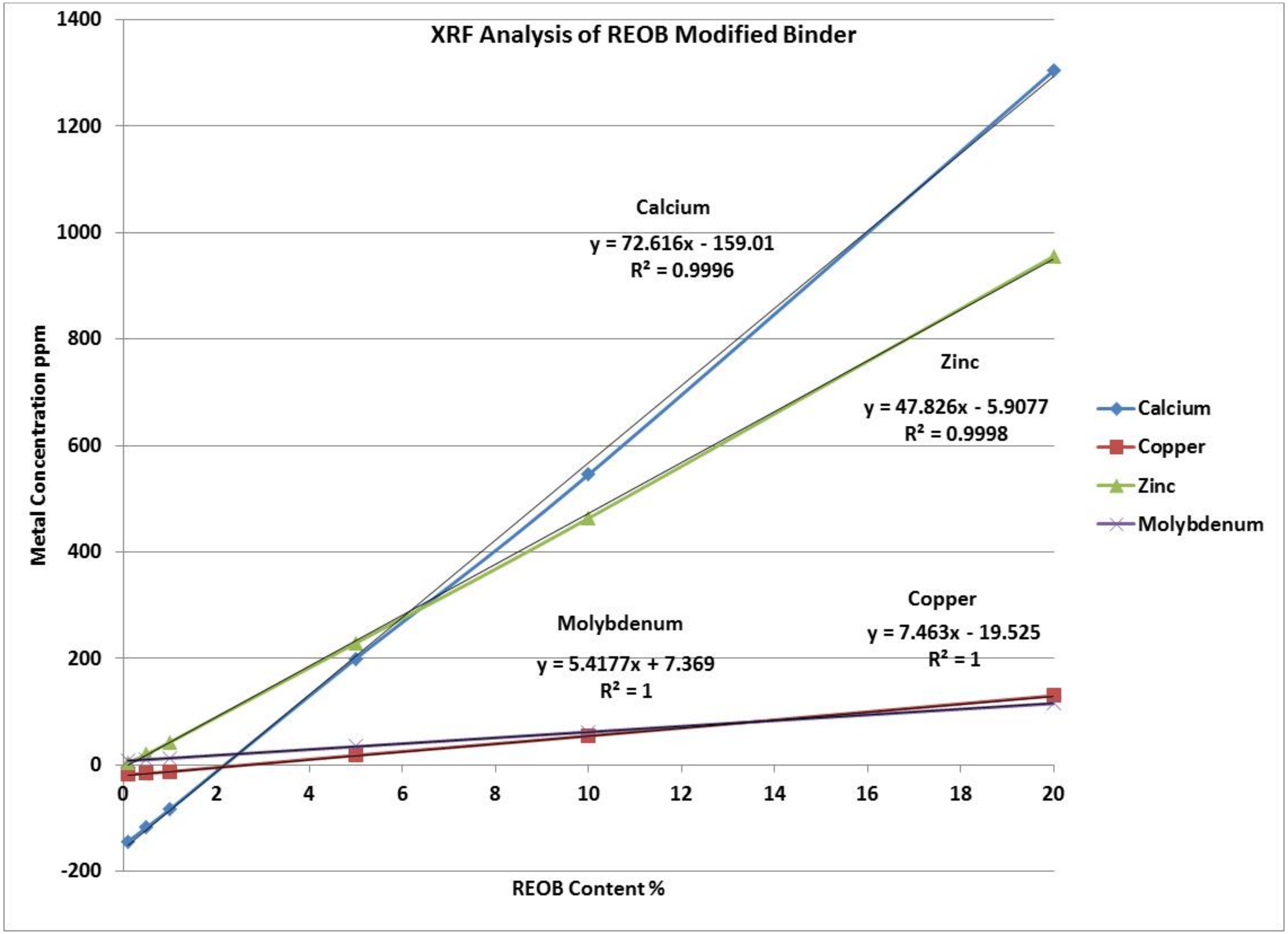
- XRF Phosphorous and Sulfur Peaks Overlap

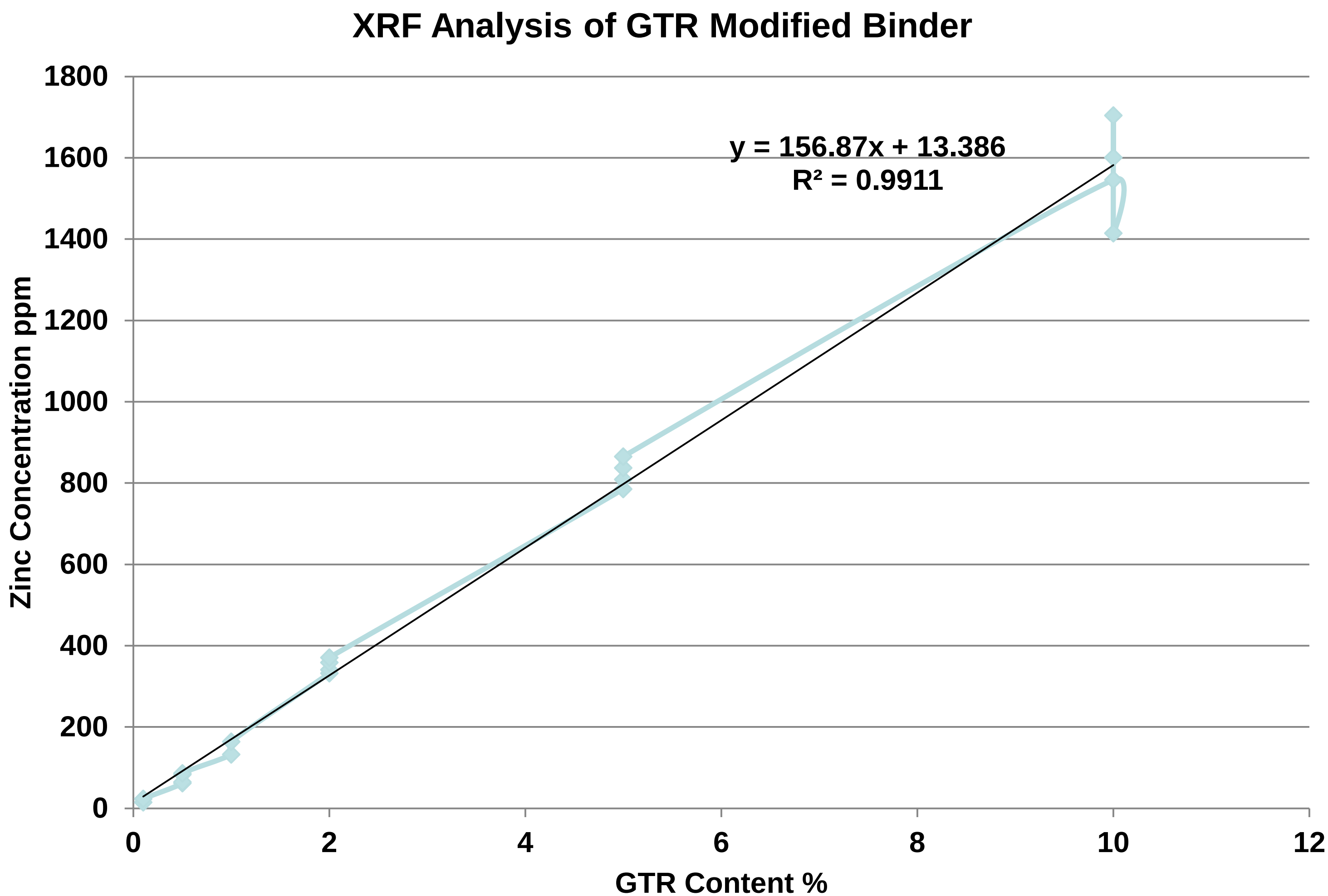
- Sulfur 3.05 - 11.49%
- Iron 8 - 115 ppm
- Molybdenum 0 - 15.7 ppm

- May contain Zinc H_2S Scavengers

XRF Analysis of REOB and GTR (ppm)

Element	REOB	GTR	Asphalt
Phosphorous	12,000	0	0
Sulfur	16,000	33,000	30-300,00
Calcium	9,000	1,600	0
Iron	1,200	2,800	8-115
Copper	900	1,000	0
Zinc	5,500	16,000	0
Molybdenum	600	0	0
Silica	-	21,000	0


XRF Analysis of REOB Modified Binder
Metal Concentration ppm
1400
1200
1000
800
600
400
200
0
-200
0
2
4
6
8
10
12
14
16
18
20
REOB Content %
Calcium
y = 72.616x - 159.01
R² = 0.9996
Zinc
y = 47.826x - 5.9077
R² = 0.9998
Molybdenum
y = 5.4177x + 7.369
R² = 1
Copper
y = 7.463x - 19.525
R² = 1
Calcium
Copper
Zinc
Molybdenum


XRF Analysis of GTR Modified Binder
y = 156.87x + 13.386
R² = 0.9911
Zinc Concentration ppm
GTR Content %
0
200
400
600
800
1000
1200
1400
1600
1800
0
2
4
6
8
10
12

REOB Content of Binders

1,208 binder samples received from 38 Agencies

		XRF Analysis ppm				REOB% (08-1001)				
State	Performance Grade	Calcium	Copper	Zinc	Molybdenum	Calcium ppm	Copper ppm	Zinc ppm	Molybdenum ppm	GTR %
IN	64-28	424.3	36.6	417.9	43.4	3	4	7	10	3
WA	64-22	479	48	424.5	44.4	4	5	7	10	3
AL	-	643.4	56.5	469.1	64.2	5	6	8	14	3
WA	64-28	576.6	51.9	480.2	51.8	5	6	8	11	3
IN	58-28	550.6	52.7	501.4	48.5	5	6	8	11	3
OK	70-28	478	50.6	548.1	44.4	4	5	9	10	3
OK	64-22 OK	874.1	124	576.6	32.8	8	12	9	8	4
TX	AC 15P	611.4	79.4	591.3	48.5	5	8	10	11	4
TX	AC 5	781.7	84.1	775.3	59.9	7	8	13	13	5
TX	AC20-5TR	-101.1	-8.1	794.7	-2	-1	0	13	1	5
TX	76-22T	-62.6	-8.7	837.9	0.6	-1	0	14	1	5
FL	76-22 AR	26.2	33.3	913.4	10.7	0	4	15	3	6
CFL	64-10	1255	200	933.3	42.1	11	19	15	9	6
AZ	76-22TR	0	18.5	1128.9	0.1	0	3	19	1	7
NE	58-28	-131.5	83.7	1203.8	5.9	-2	8	20	2	8
NE	64-30	-128.6	-14	1523.1	3.7	-2	0	25	2	10
CA	76-22TR	189.7	37.2	1761.7	10.6	1	4	29	3	11
AZ	58-22	1737.3	141	2452.4	64.1	15	14	41	14	16
TX	64-22	34.7	42.9	2558.4	-6.1	0	5	43	0	16
CA	64-28TR	782.5	145	2653.7	45.8	7	14	44	10	17

Conclusions on XRF Analysis

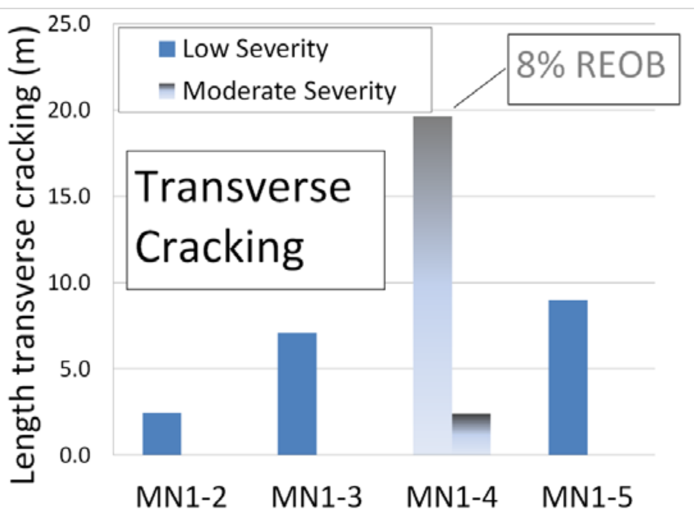
- You can readily detect REOB presence
- You cannot tell exactly how much is there

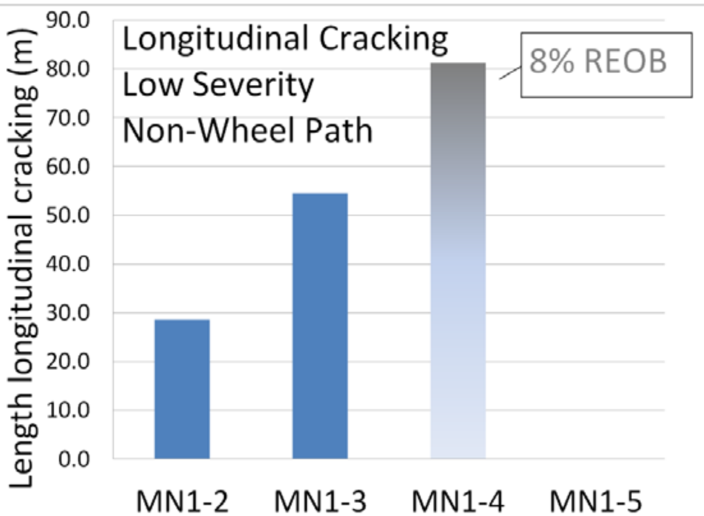
Field study

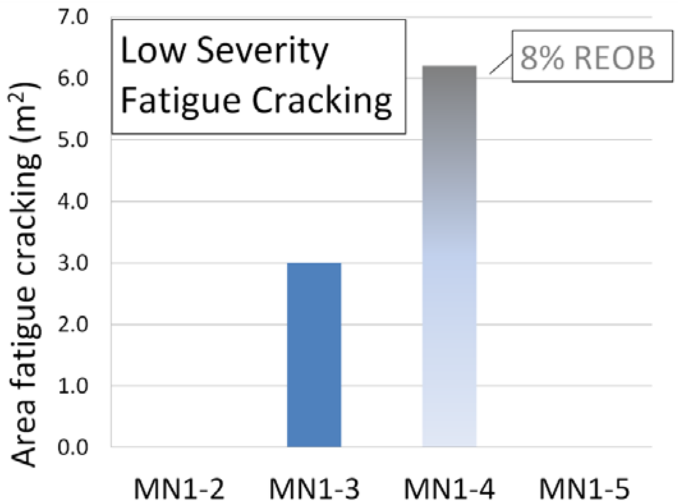
- FHWA/ARC/WRI and FHWA/ARC/NCAT Validation sites in the US and Canada -> MN site
- Only difference between the sites is the binder used??

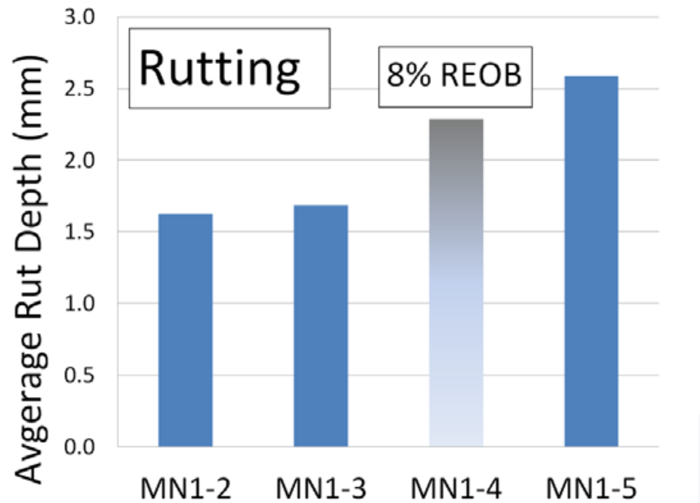
Field Study - Rochester, MN Comparative Test sites

- 2012 Distress Data (500 feet test sites)









Low Severity Transverse Cracking

__NOT ACTUAL CRACK MAPS__ - GRAPHICAL REPRESENTATION OF DATA **TO SCALE**

Low Severity Non-Wheelpath Longitudinal Cracking

MN1-2

MN1-2

MN1-4
8% REOB

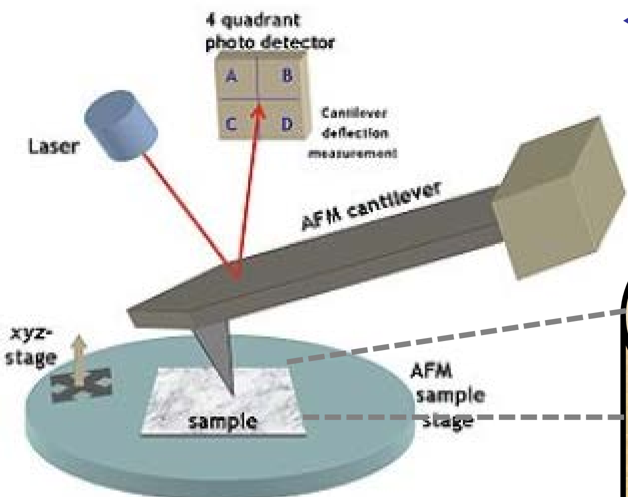
MN1-5

NOT ACTUAL CRACK MAPS - GRAPHICAL REPRESENTATION OF DATA **TO SCALE**

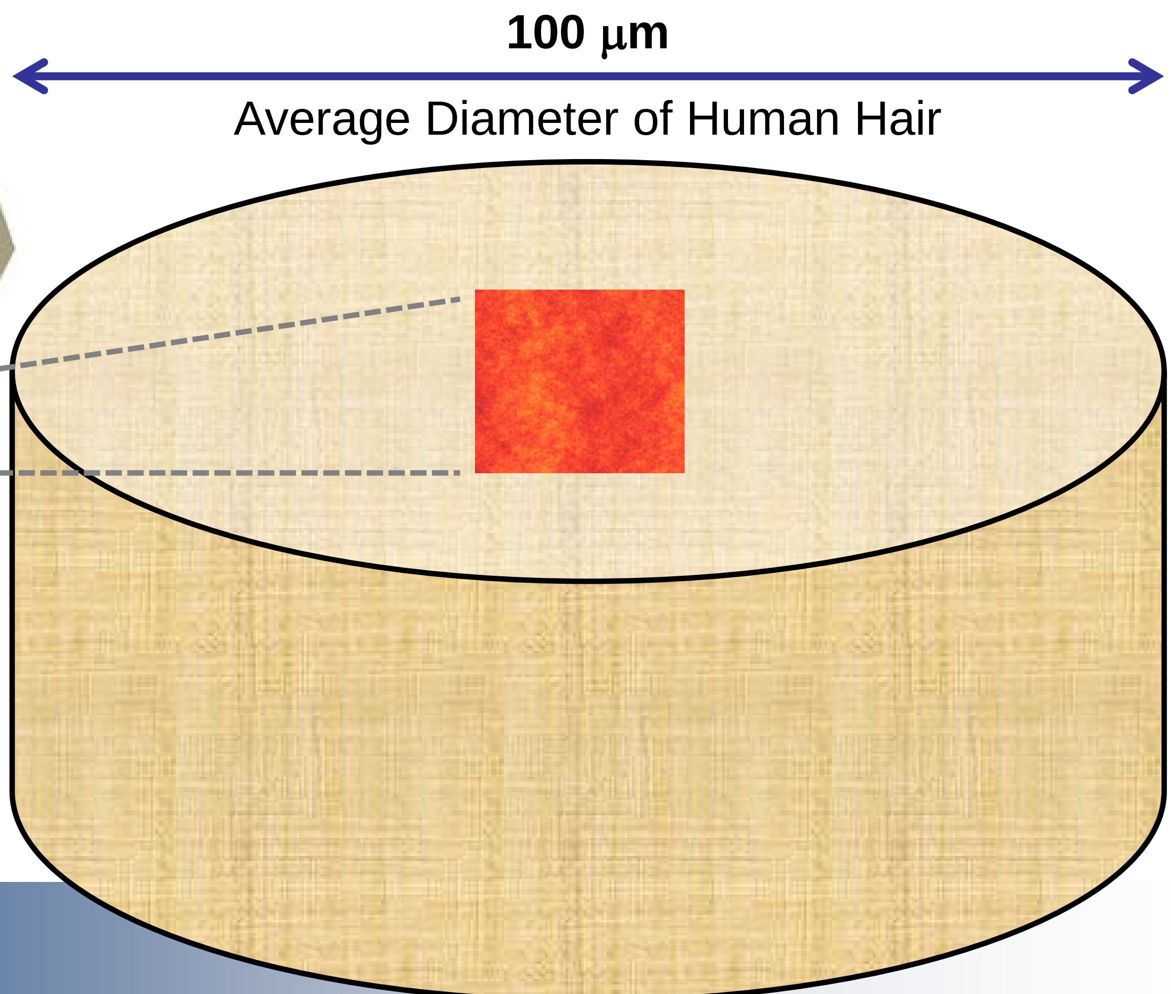
Low Severity Fatigue Cracking

<u>NOT ACTUAL CRACK MAPS</u> - GRAPHICAL REPRESENTATION OF DATA **TO SCALE**

Western Research Institute WRI Atomic Force Microscopy AFM

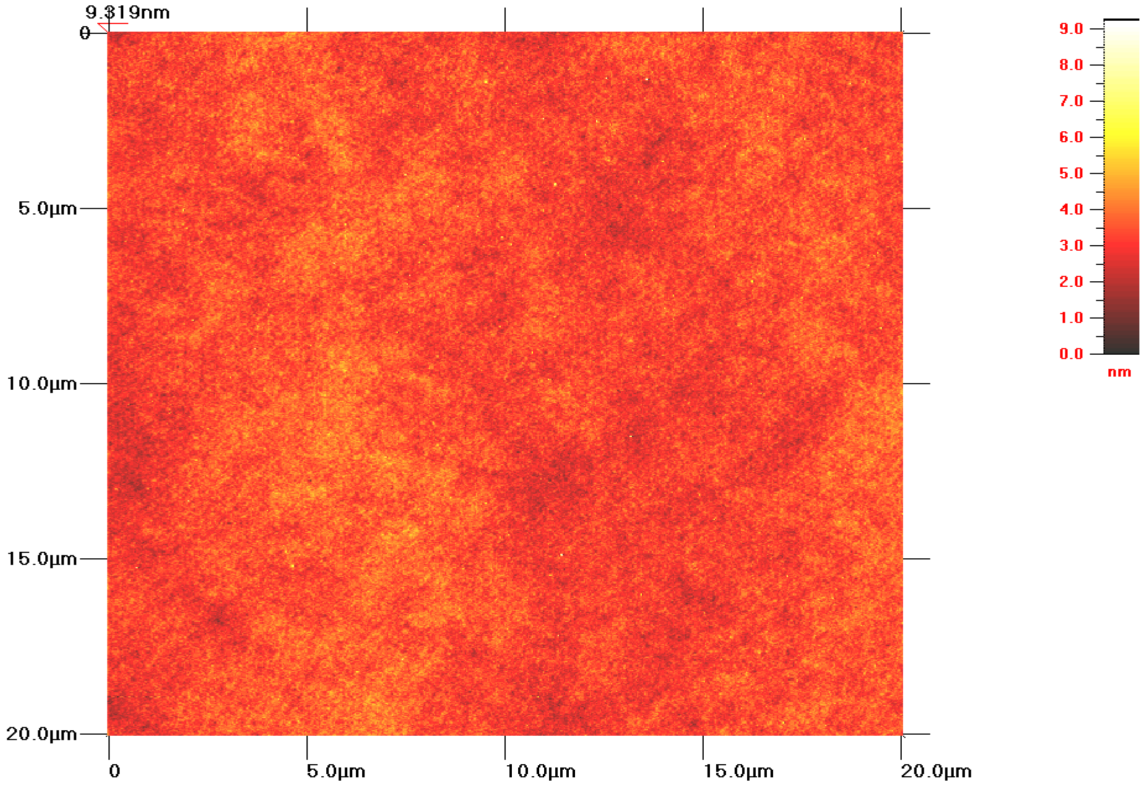


Source: Wikipedia



Binder Microstructure study: AFM

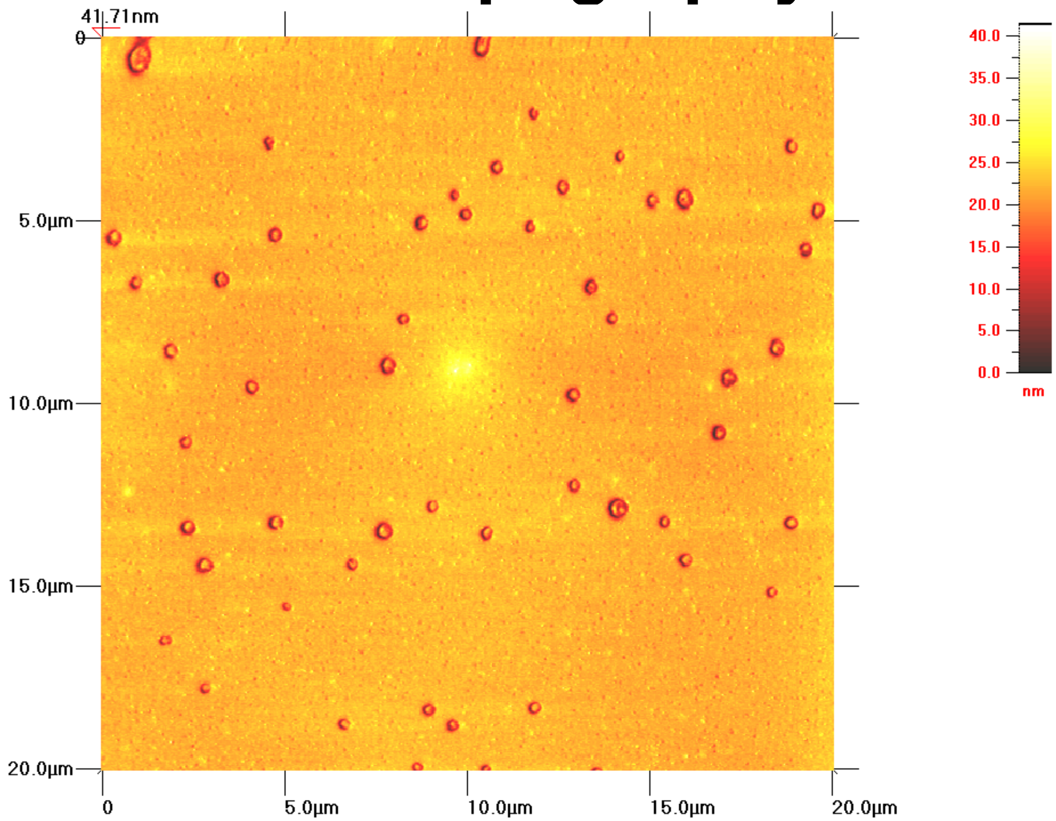
- Neat AAG-1 topography



Typical neat AAG surface Nearly flat and featureless

Binder Microstructure study: AFM

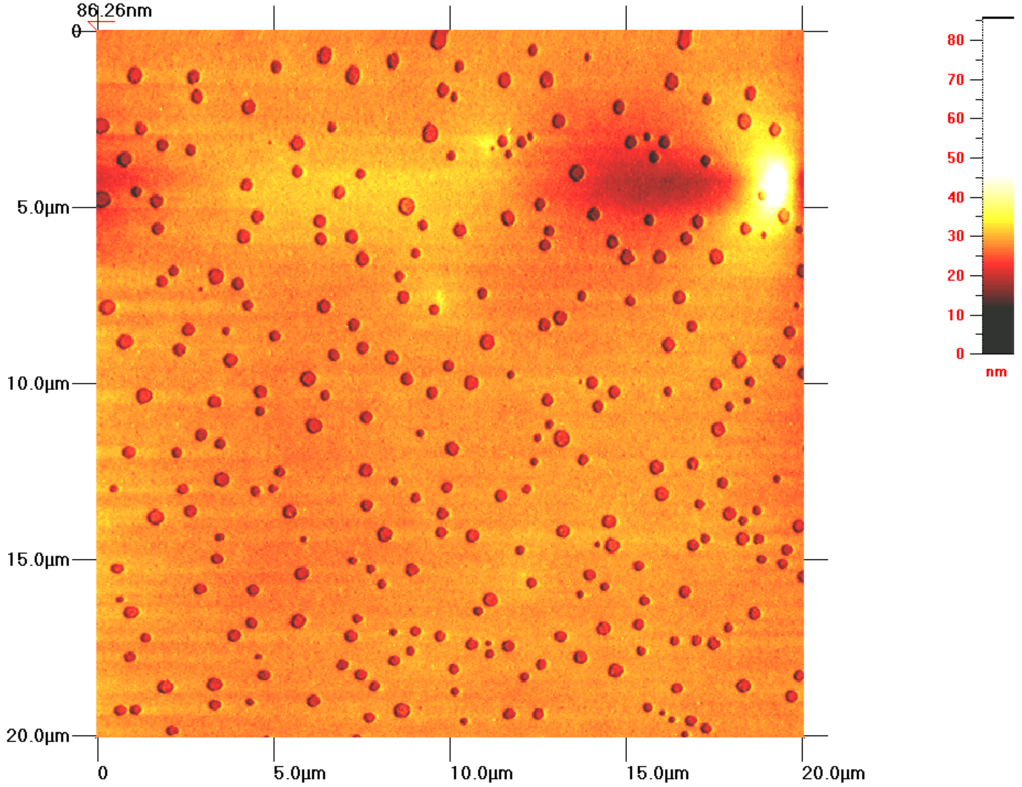
- AAG-1 + REOB topography



Topographic image indicates a relatively smooth flat surface with a number of small "holes"

Binder Microstructure study: AFM

- AAG-1 + REOB topography + Aging



Topographic image indicates a relatively smooth flat surface with more small "holes"

Summary from WRI

- A preliminary study on a very limited sampling, still on going at WRI
- However some interesting findings:
  - REOB not inert – affects microstructure, properties and aging
  - Microstructure: 2-phase structures (at least) – "holes" occurring and expanding over aging

Binders' and Mixtures' Engineering Properties

Two Modification Approaches

- **Softening an unmodified PG to another PG**

PG64-22

↓ *6% REOB**

PG58-28

**with a single REOB sample*

Two Modification Approaches

- Softening an unmodified PG to another PG

PG64-22

*6% REOB**

PG58-28

- Diluting a unmodified PG

PG69-24

+20% PG100-0

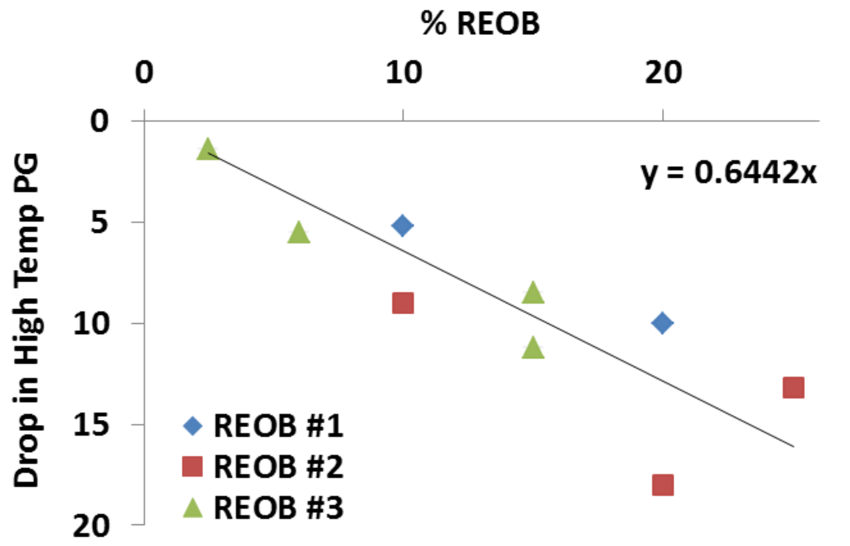
*+15% REOB**

PG58-28 PG58-28

PG58-28 PG58-28

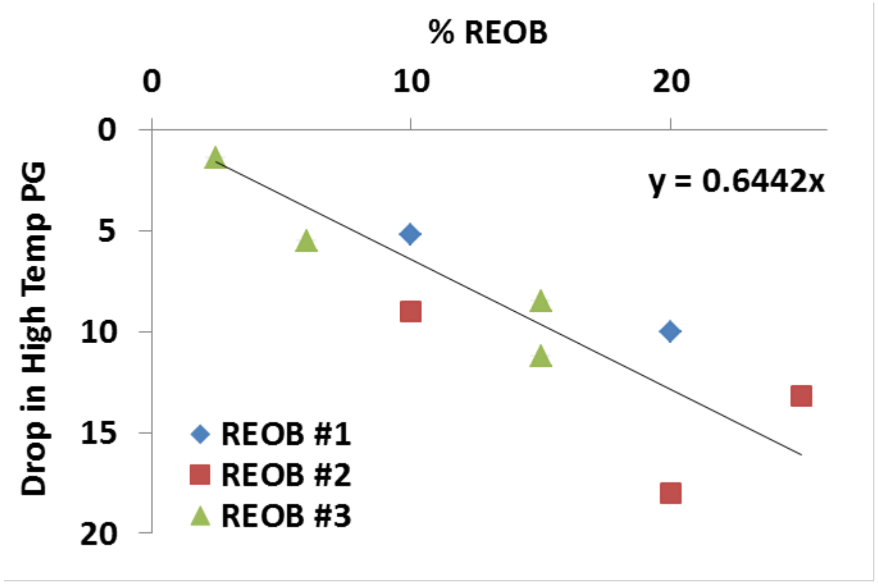
*+2.5% REOB**

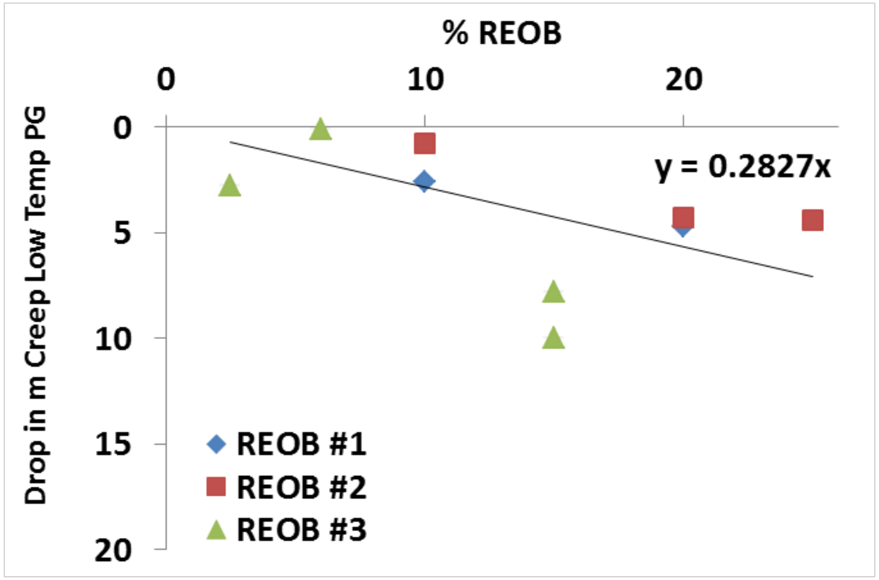
**with a single REOB sample*



- **DSR High Temp**

  ~9% REOB per PG Grade Drop



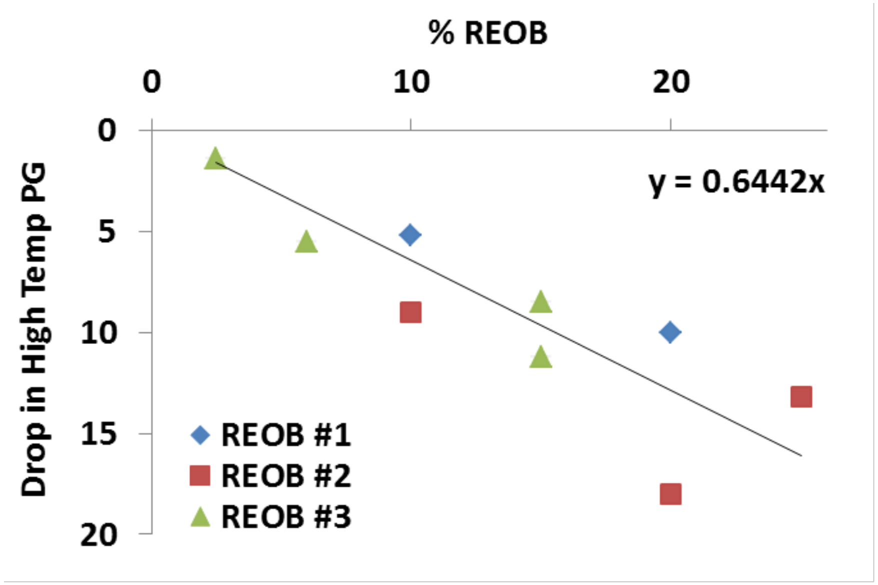


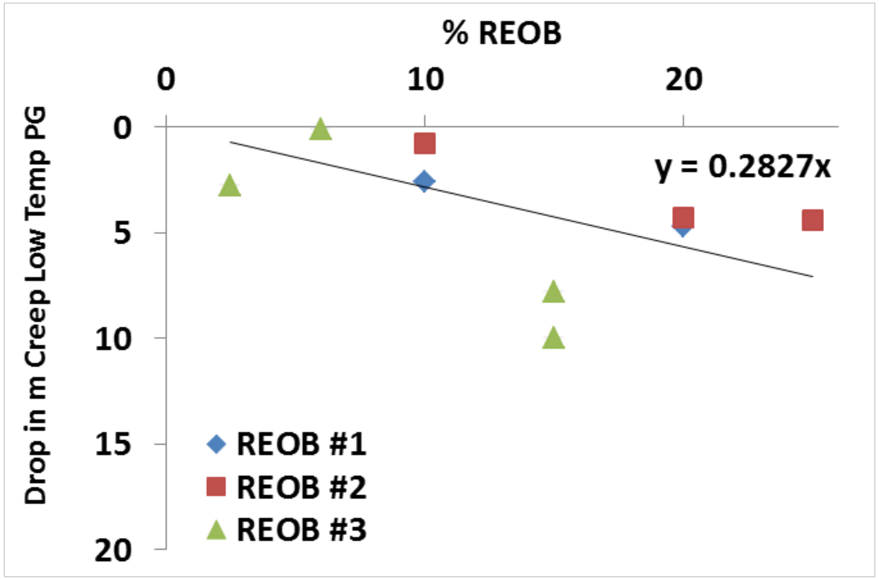
- **DSR High Temp**

  ~9% REOB per PG Grade Drop

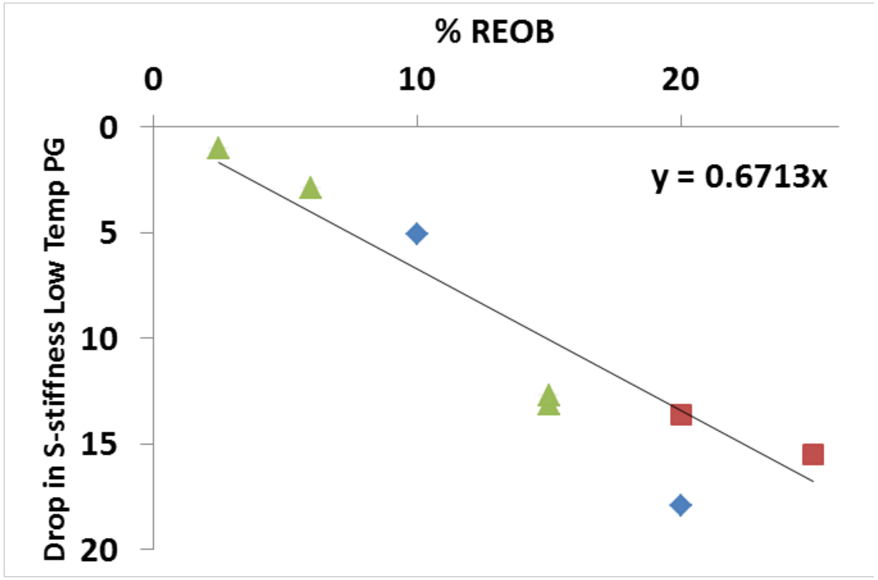
- **BBR m-Value**

  ~21% REOB per PG Grade Drop





- **DSR High Temp**
  ~9% REOB per PG Grade Drop
- **BBR m-Value**
  ~21% REOB per PG Grade Drop
- **BBR Stiffness**
  ~9% REOB per PG Grade Drop



BBR $\Delta T_{critical}$ Spread: $PG_{(S)tiffness} – PG_{(m)-creep}$

	Base	*Exploratory Blends*			**Final Blends**
		+PG100-0	*+REOB Source 1*	*+REOB Source 2*	**+REOB Source 3**
	PAV				**PAV**
Holly 58-28	**-2.0°C** **60-30**				

BBR $\Delta T_{critical}$ Spread: $PG_{(S)tiffness} - PG_{(m)-creep}$

	Base	*Exploratory Blends*			Final Blends
		+PG100-0	*+REOB Source 1*	*+REOB Source 2*	+REOB Source 3
	PAV				PAV
Holly 58-28	**-2.0°C** 60-30	*-0.8°C* *0% / 20%* *69-24*			
		-1.6°C *0% / 30%* *72-20*			

BBR $\Delta T_{critical}$ Spread: $PG_{(S)tiffness} - PG_{(m)\text{-}creep}$

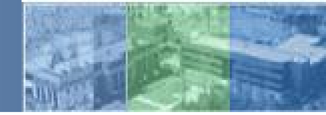
	Base	*Exploratory Blends*			**Final Blends**
		+PG100-0	*+REOB Source 1*	*+REOB Source 2*	**+REOB Source 3**
	PAV				**PAV**
Holly 58-28	**-2.0°C** **60-30**	*-0.8°C* *0% / 20%* *69-24*	*-10°C* *20% / 20%* *59-28*	*-14°C* *20% / 20%* *51-28*	
		-1.6°C *0% / 30%* *72-20*	*-13°C* *25% / 30%* *59-25*		

BBR $\Delta T_{critical}$ Spread: $PG_{(S)tiffness} - PG_{(m)-creep}$

	Base	*Exploratory Blends*			**Final Blends**
		+PG100-0	*+REOB Source 1*	*+REOB Source 2*	**+REOB Source 3**
	PAV				PAV
Holly 58-28	**-2.0°C** **60-30**	*-0.8°C* *0% / 20%* *69-24*	*-10°C* *20% / 20%* *59-28*	*-14°C* *20% / 20%* *51-28*	
		-1.6°C *0% / 30%* *72-20*	*-13°C* *25% / 30%* *59-25*		*-5.1°C* *15% / 0%* *51-40*

BBR $\Delta T_{critical}$ Spread: $PG_{(S)tiffness} - PG_{(m)\text{-}creep}$

	Base	Exploratory Blends			Final Blends
		+PG100-0	+REOB Source 1	+REOB Source 2	+REOB Source 3
	PAV				PAV
Holly 58-28	-2.0°C 60-30	-0.8°C 0% / 20% 69-24	-10°C 20% / 20% 59-28	-14°C 20% / 20% 51-28	-5.7°C 15% / 20% 58-33
		-1.6°C 0% / 30% 72-20	-13°C 25% / 30% 59-25		-5.1°C 15% / 0% 51-40
					-0.2°C 2.5% 59-33



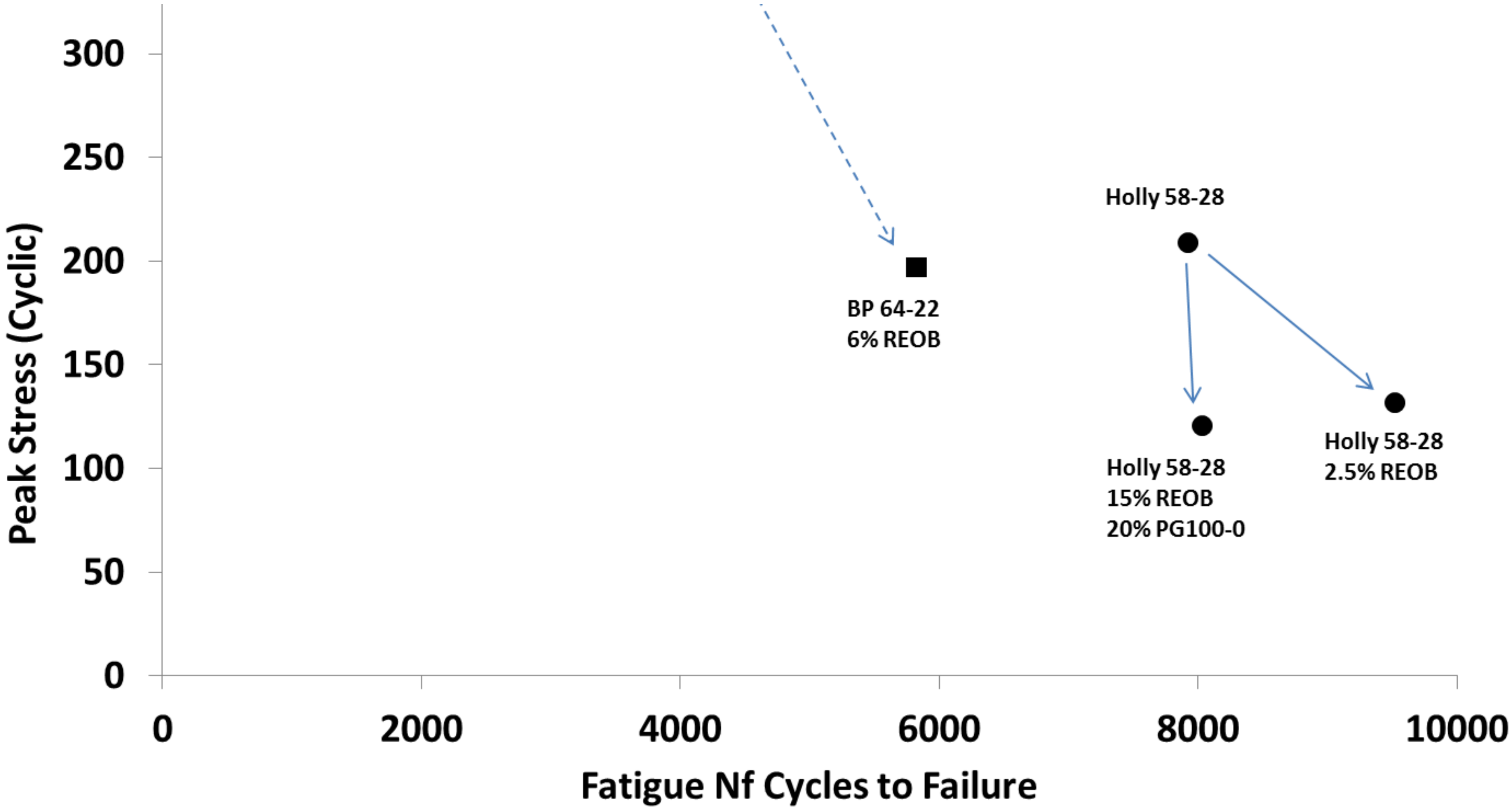
BBR $\Delta T_{critical}$ Spread: $PG_{(S)tiffness} - PG_{(m)\text{-}creep}$

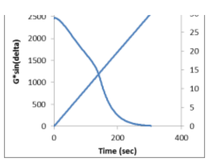
	Base	*Exploratory Blends*			**Final Blends**
	Base (PAV)	*+PG100-0*	*+REOB Source 1*	*+REOB Source 2*	**+REOB Source 3** (PAV)
Holly 58-28	**-2.0°C** **60-30**	*-0.8°C* *0% / 20%* *69-24*	*-10°C* *20% / 20%* *59-28*	*-14°C* *20% / 20%* *51-28*	**-5.7°C** **15% / 20%** **58-33**
		-1.6°C *0% / 30%* *72-20*	*-13°C* *25% / 30%* *59-25*		*-5.1°C* *15% / 0%* *51-40*
					-0.2°C **2.5%** **59-33**
BP 64-22	+0.8°C 67-27		*-1.7°C* *10%* *61-31*	*-4.0°C* *10%* *58-29*	**-2.2°C** **6%** **61-28**

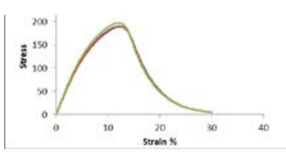
BBR $\Delta T_{critical}$ Spread: $PG_{(S)tiffness} - PG_{(m)-creep}$

	Base		Exploratory Blends			Final Blends	
	PAV	2 X PAV	+PG100-0	+REOB Source 1	+REOB Source 2	+REOB Source 3 PAV	+REOB Source 3 2 X PAV
Holly 58-28	-2.0°C 60-30	-1.1°C □-29	-0.8°C 0% / 20% 69-24	-10°C 20% / 20% 59-28	-14°C 20% / 20% 51-28	-5.7°C 15% / 20% 58-33	-10°C 15% / 20% □-26
			-1.6°C 0% / 30% 72-20	-13°C 25% / 30% 59-25		-5.1°C 15% / 0% 51-40	-10°C 15% / 0% □34
						-0.2°C 2.5% 59-33	-2.8°C 2.5% □-29
BP 64-22	+0.8°C 67-27	-1.9°C □-23		-1.7°C 10% 61-31	-4.0°C 10% 58-29	-2.2°C 6% 61-28	-2.9°C 6% □-23

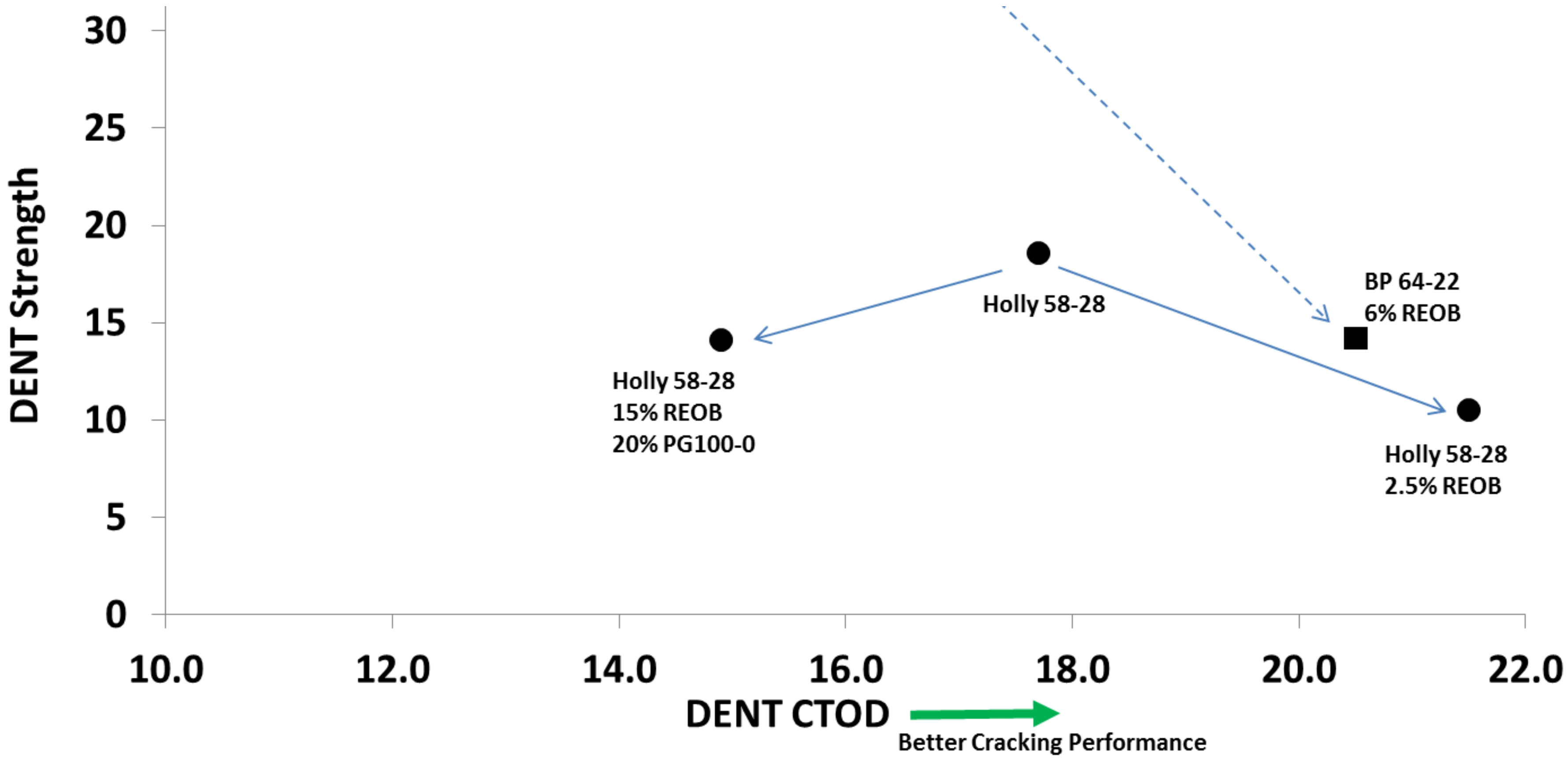
DSR Fatigue: Linear Amplitude Sweep (LAST)

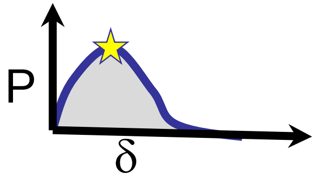






Notched Tension: Cracking Strain Tolerance

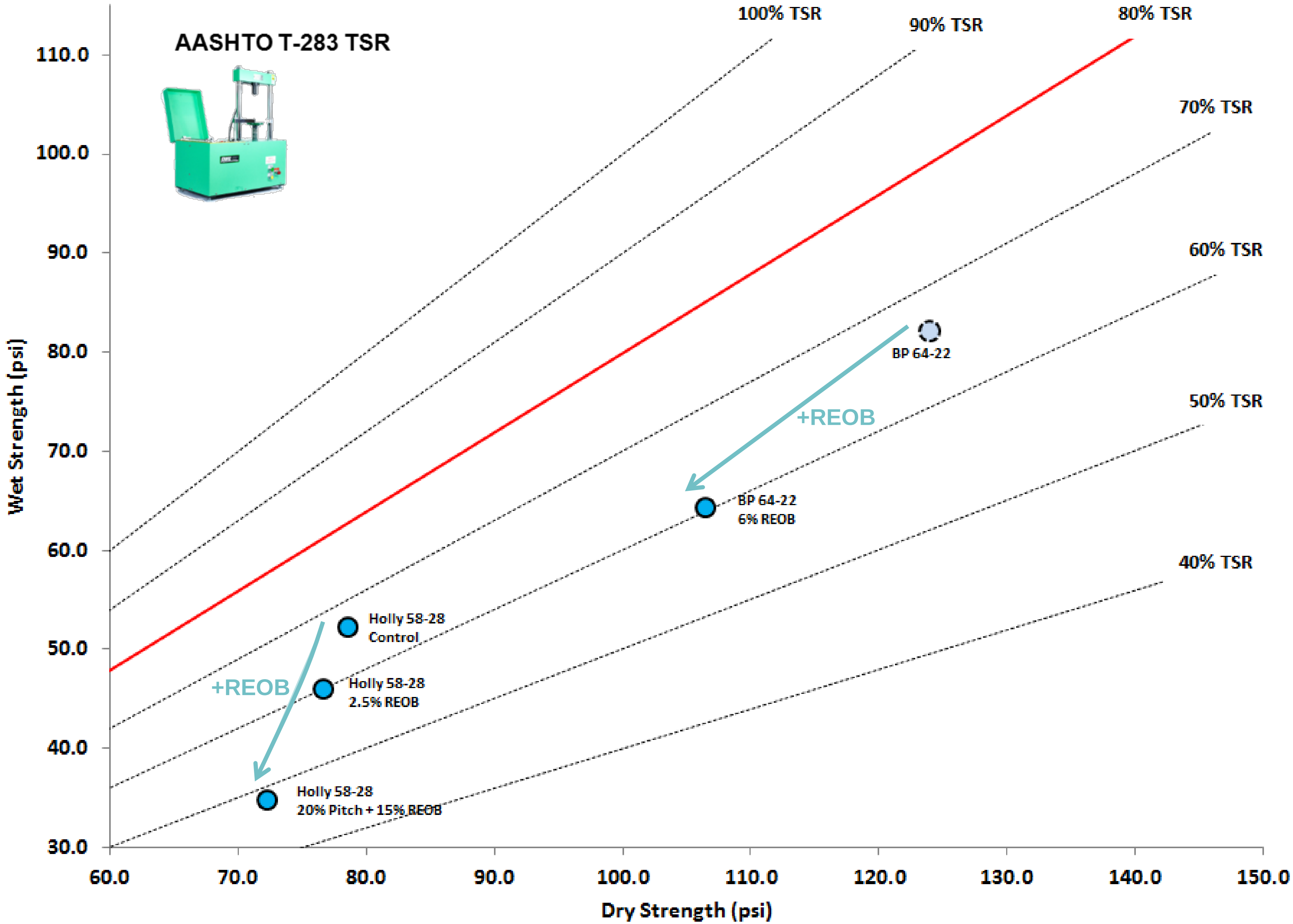


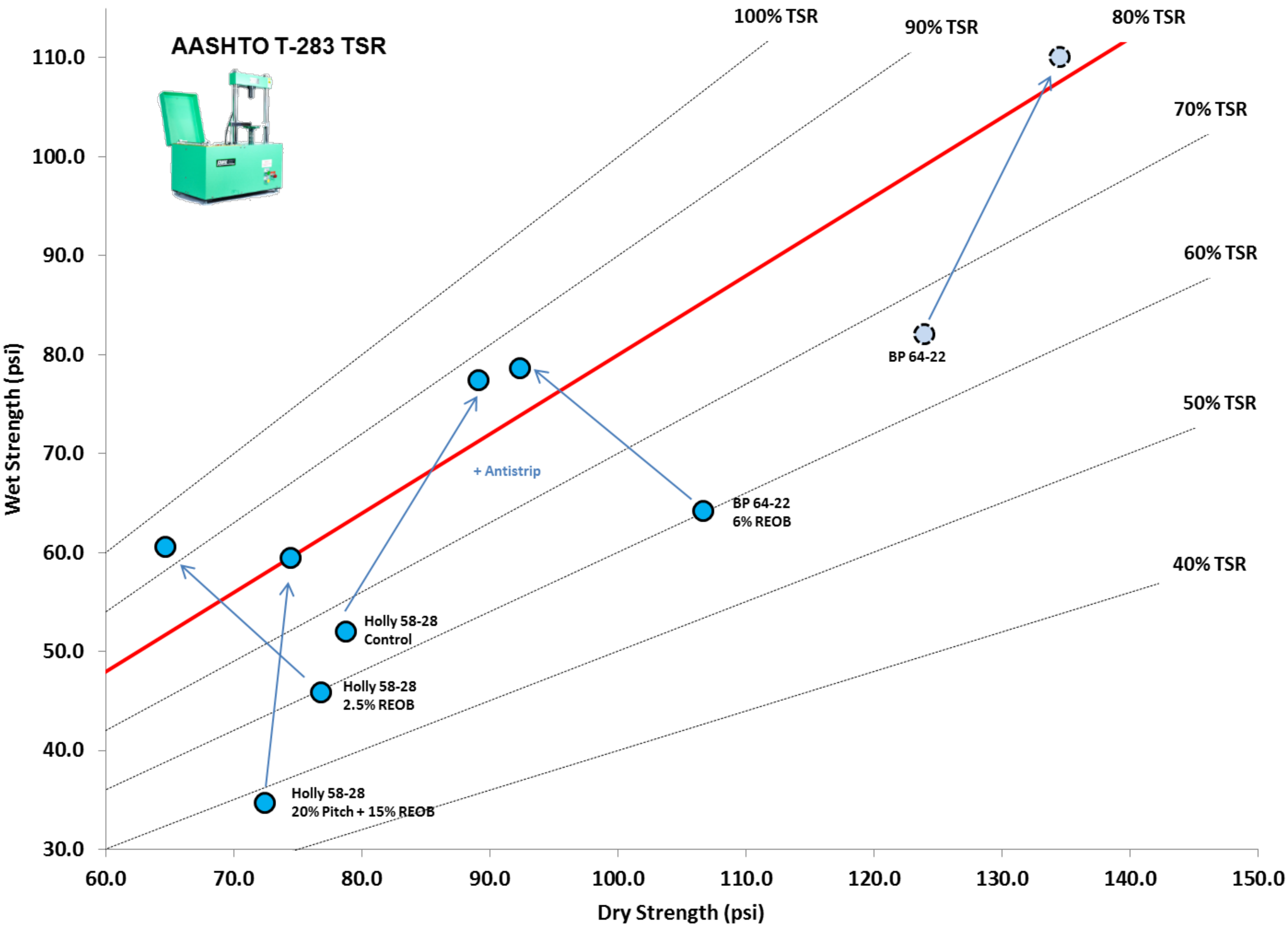


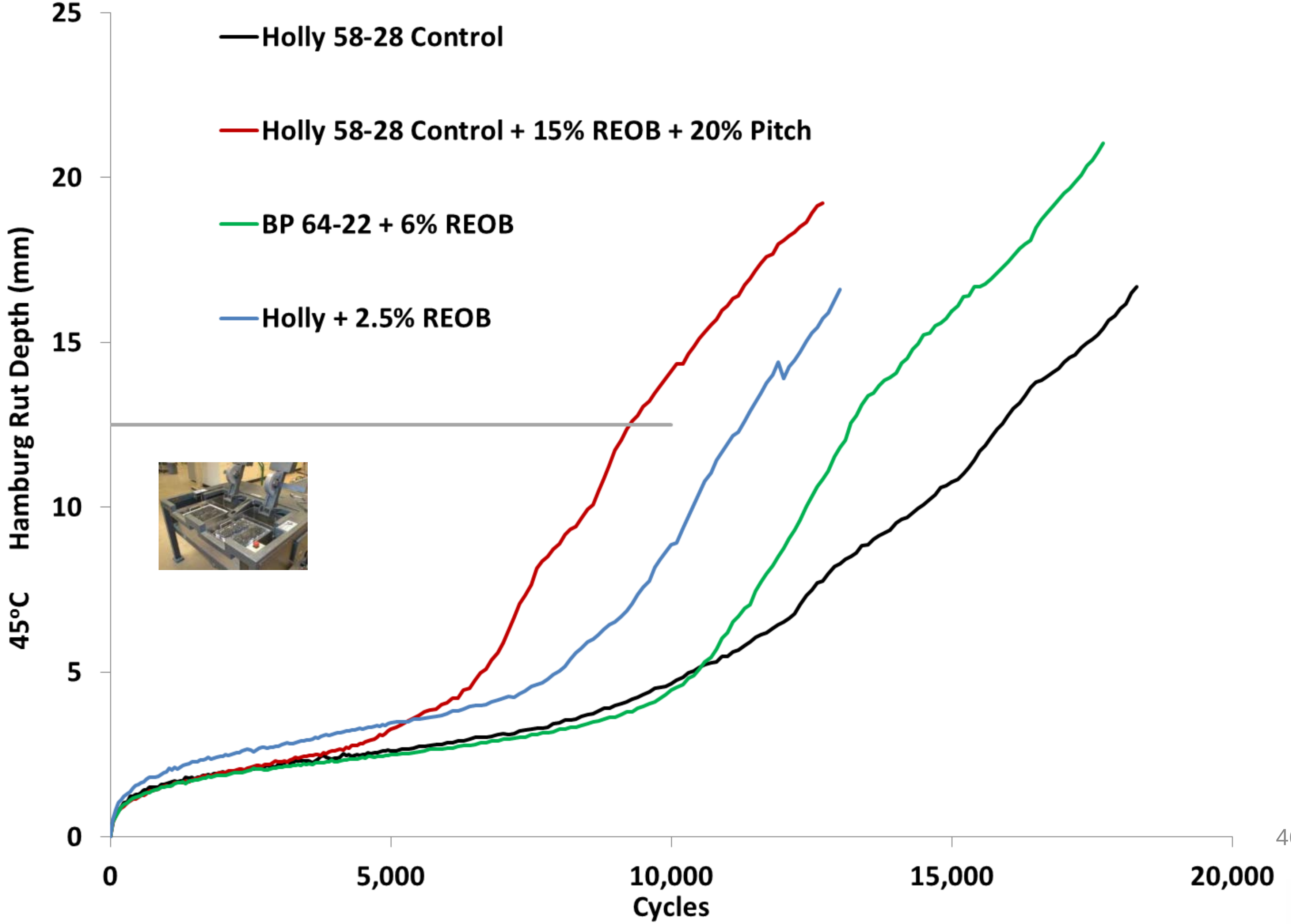
Ongoing Mixtures' Experimental Design

- **"Moisture Damage"**
  - Granite- Occoquan, VA
  - Tensile Strength Retained TSR
  - Hamburg Wheel Tracking
  - Repeated With & Without Liquid Amine Anti-strip ~~or Hydrated Lime~~

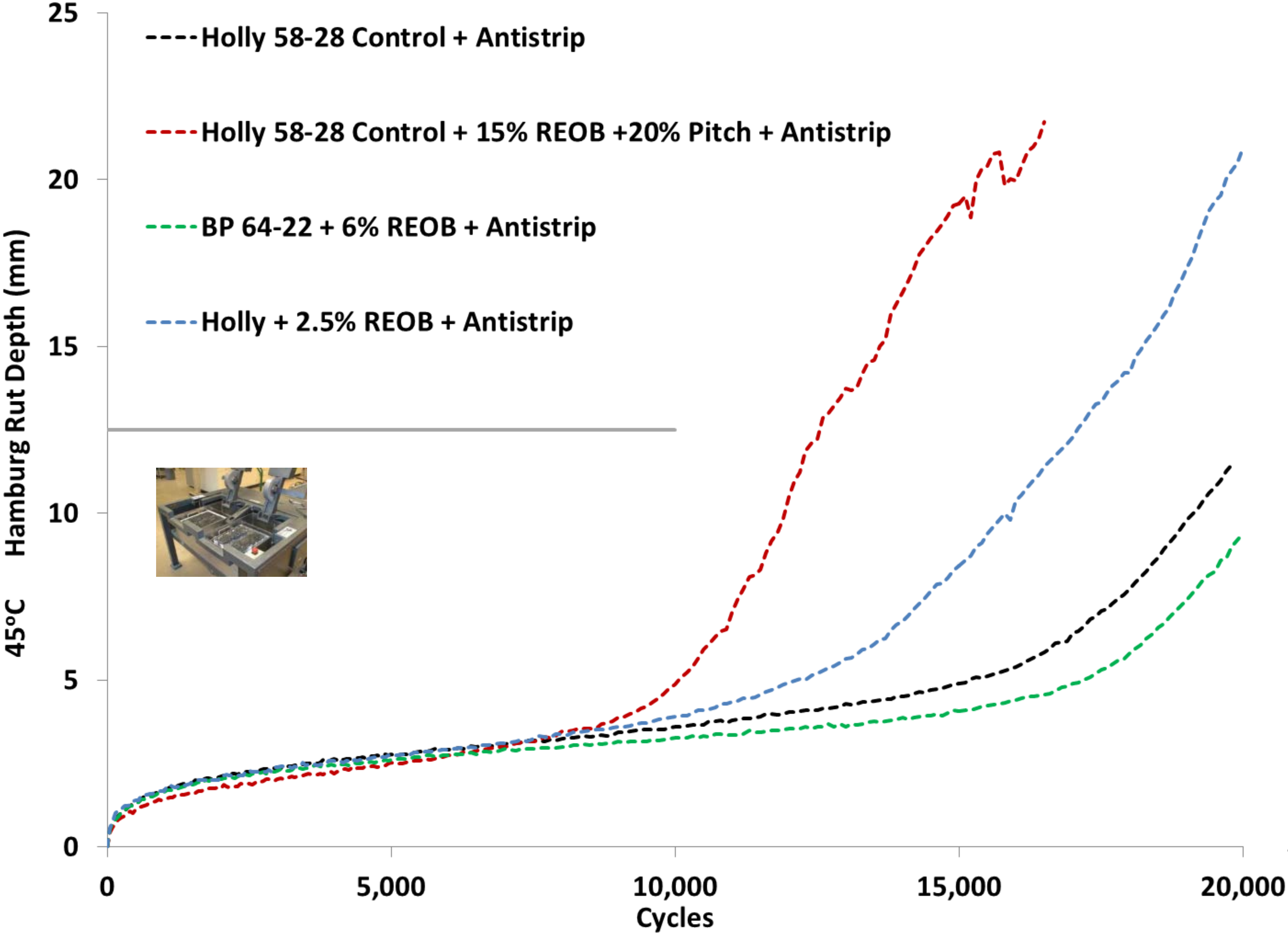
- **"Structural Performance"**
  - ALF 22% RAP Mix
  - ~~Flow Number; confined NCHRP 9-30A~~
  - Dynamic Modulus, |E*|
  - Uniaxial Fatigue - **Short and Long-Term Aged** *(loose mix 5 days @ 85°C)*
  - Thermal Stress Restrained Specimen TSRST - **Short and Long-Term Aged** *(loose mix 5 days @ 85°C)*


AASHTO T-283 TSR
100% TSR
90% TSR
80% TSR
70% TSR
60% TSR
50% TSR
40% TSR
BP 64-22
+REOB
BP 64-22
6% REOB
Holly 58-28
Control
+REOB
Holly 58-28
2.5% REOB
Holly 58-28
20% Pitch + 15% REOB
Wet Strength (psi)
Dry Strength (psi)
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0

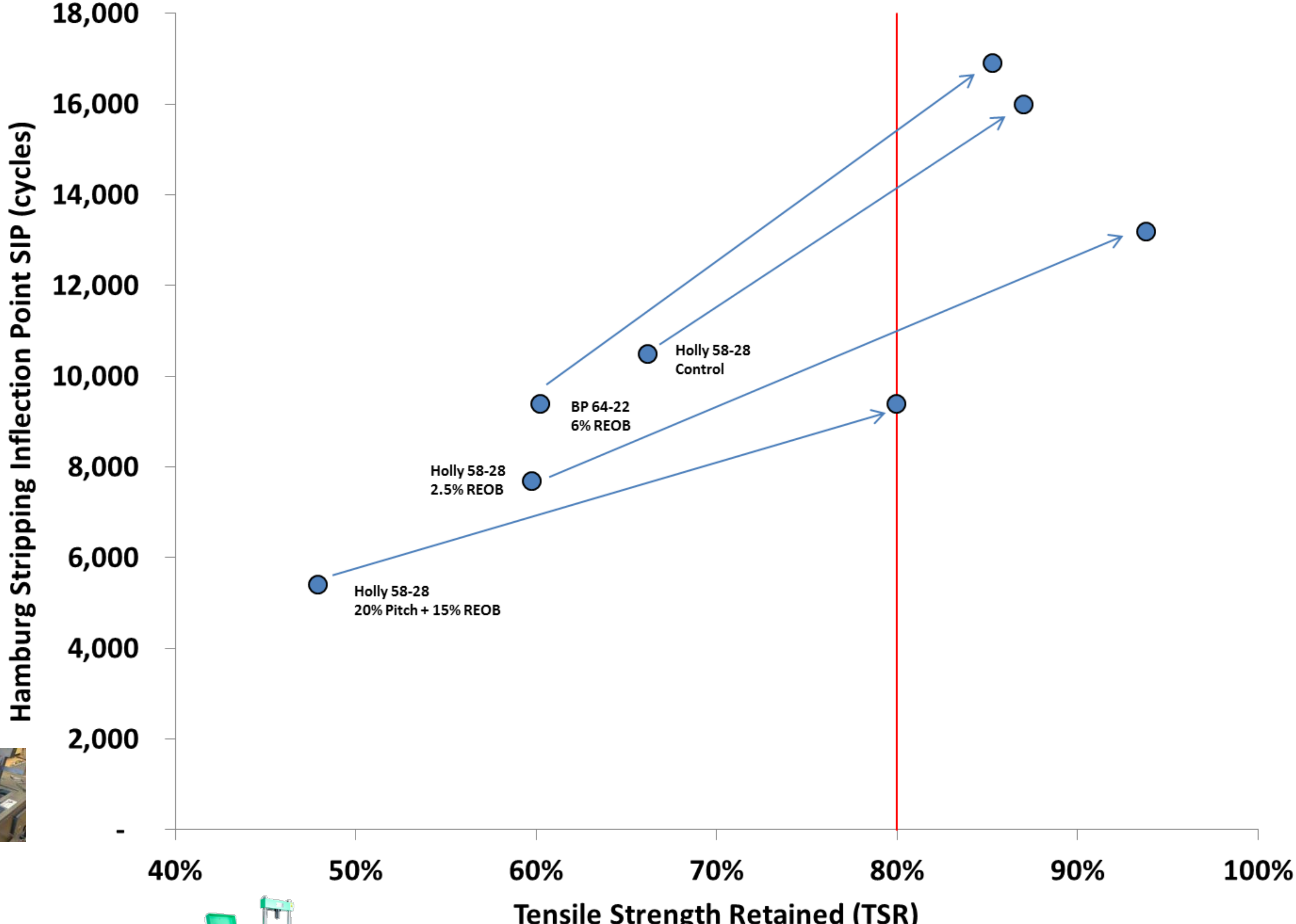

AASHTO T-283 TSR
100% TSR
90% TSR
80% TSR
70% TSR
60% TSR
50% TSR
40% TSR
BP 64-22
+ Antistrip
BP 64-22
6% REOB
Holly 58-28
Control
Holly 58-28
2.5% REOB
Holly 58-28
20% Pitch + 15% REOB
Wet Strength (psi)
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
Dry Strength (psi)



Holly 58-28 Control
Holly 58-28 Control + 15% REOB + 20% Pitch
BP 64-22 + 6% REOB
Holly + 2.5% REOB
45°C Hamburg Rut Depth (mm)
Cycles
0
5
10
15
20
25
0
5,000
10,000
15,000
20,000



Holly 58-28 Control + Antistrip
Holly 58-28 Control + 15% REOB +20% Pitch + Antistrip
BP 64-22 + 6% REOB + Antistrip
Holly + 2.5% REOB + Antistrip
45°C Hamburg Rut Depth (mm)
25
20
15
10
5
0
0
5,000
10,000
15,000
20,000
Cycles



18,000
16,000
14,000
12,000
10,000
8,000
6,000
4,000
2,000
-
Hamburg Stripping Inflection Point SIP (cycles)
40%
50%
60%
70%
80%
90%
100%
Tensile Strength Retained (TSR)
Holly 58-28
Control
BP 64-22
6% REOB
Holly 58-28
2.5% REOB
Holly 58-28
20% Pitch + 15% REOB

Conclusions (1 of 4)

1. You can readily detect REOB presence
2. You cannot tell exactly how much is there
3. Effect of REOB depends on base binder (like PPA)
4. Variation between REOB suppliers & their samples
   – Same concentration can produce different PG grades

Conclusions (2 of 4)

6. **2 X PAV is a reasonable approximation of 5 years - *where anecdotal concerns lie***
7. **REOB softens and reduces tensile strength**
   - **Binder notched tension (DENT)**
   - **Decreases mix wet and dry IDT strength**
8. **In 2 of 3 cases, REOB improved binder intermediate temperature parameters for fatigue / strain tolerance**
   - **6% and 2.5% REOB blends**
   - **CTOD and LAST**

Conclusions (3 of 4)

9. **Rheological "disruption" occurred w/ highest %REOB**
   - Differences in Low Temperature m&S
   - Did Not occur in blend with PG100-0
   - Did occur in blends with high-REOB

   - Made worse by continued aging
   - Alludes to performance deterioration
   - Corroborated by DENT CTOD & LAST & Stripping
   - Forces the issue of compatibility (extenders, rejuvenators, RAP / RAS, WMA…)

10. **Consider specification change to BBR m & S**

Conclusions (4 of 4)

10. **REOB effects on Moisture Sensitivity**
    - TSR ratio, strength and Hamburg performance decreases with increasing REOB when no anti-strip is added
    - REOB did not interfere with liquid anti-strip which improved TSR and Hamburg performance
    - Consistent results from T283 and Hamburg
      - different conditions: hot/no-freeze and cold-freeze
    - Notably, liquid ant-strip (0.4%) alters IDT strength and Hamburg deterioration more than REOB (2.5%-15%)

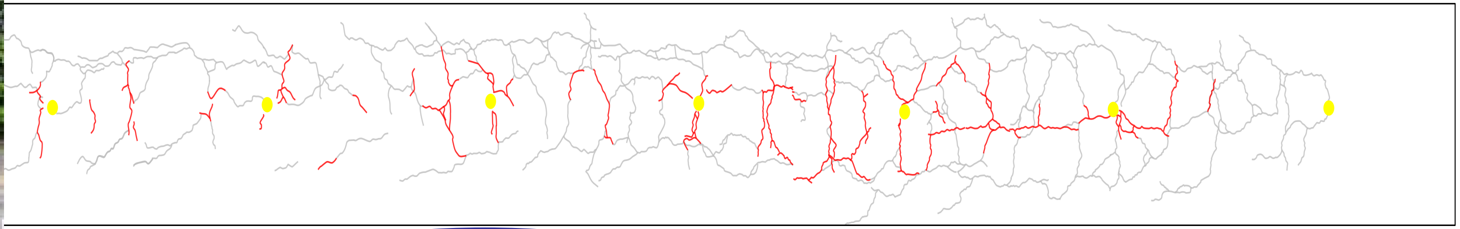
Ongoing experiments on mixture performance will be finishing February-March 2015

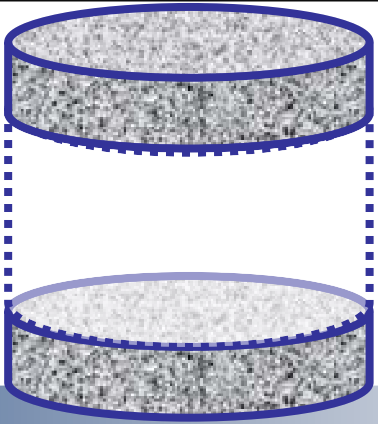
Thank You.

Questions?

Awareness of long-term performance

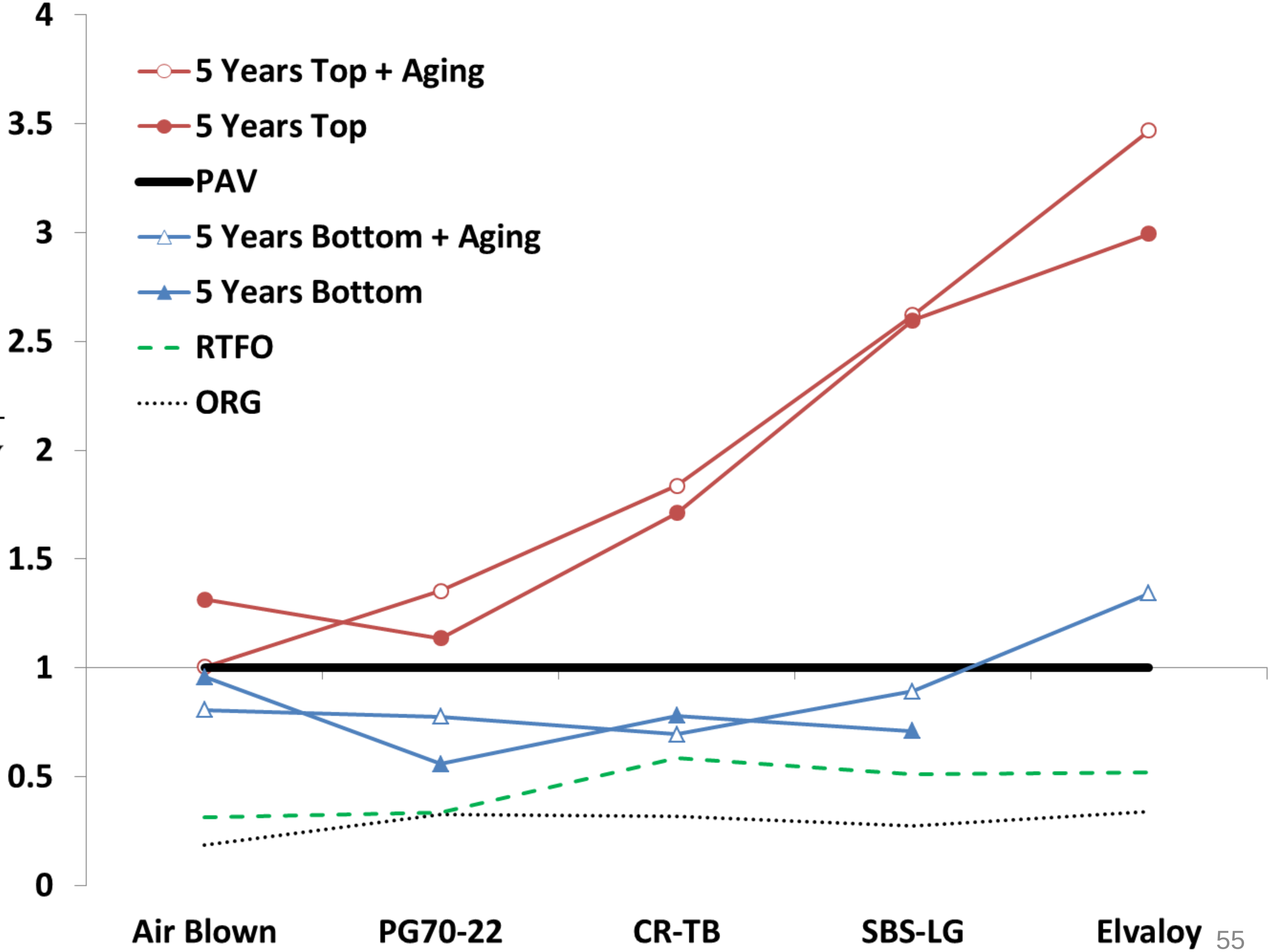
- **Utility of PAV to approximate 5-years age**
- **Poor performance after 5-years *anecdotally* attributed to REOB**
- **Data from FHWA ALF test sections**
  - **Top and bottom 1-inch of core extracted & recovered binder**

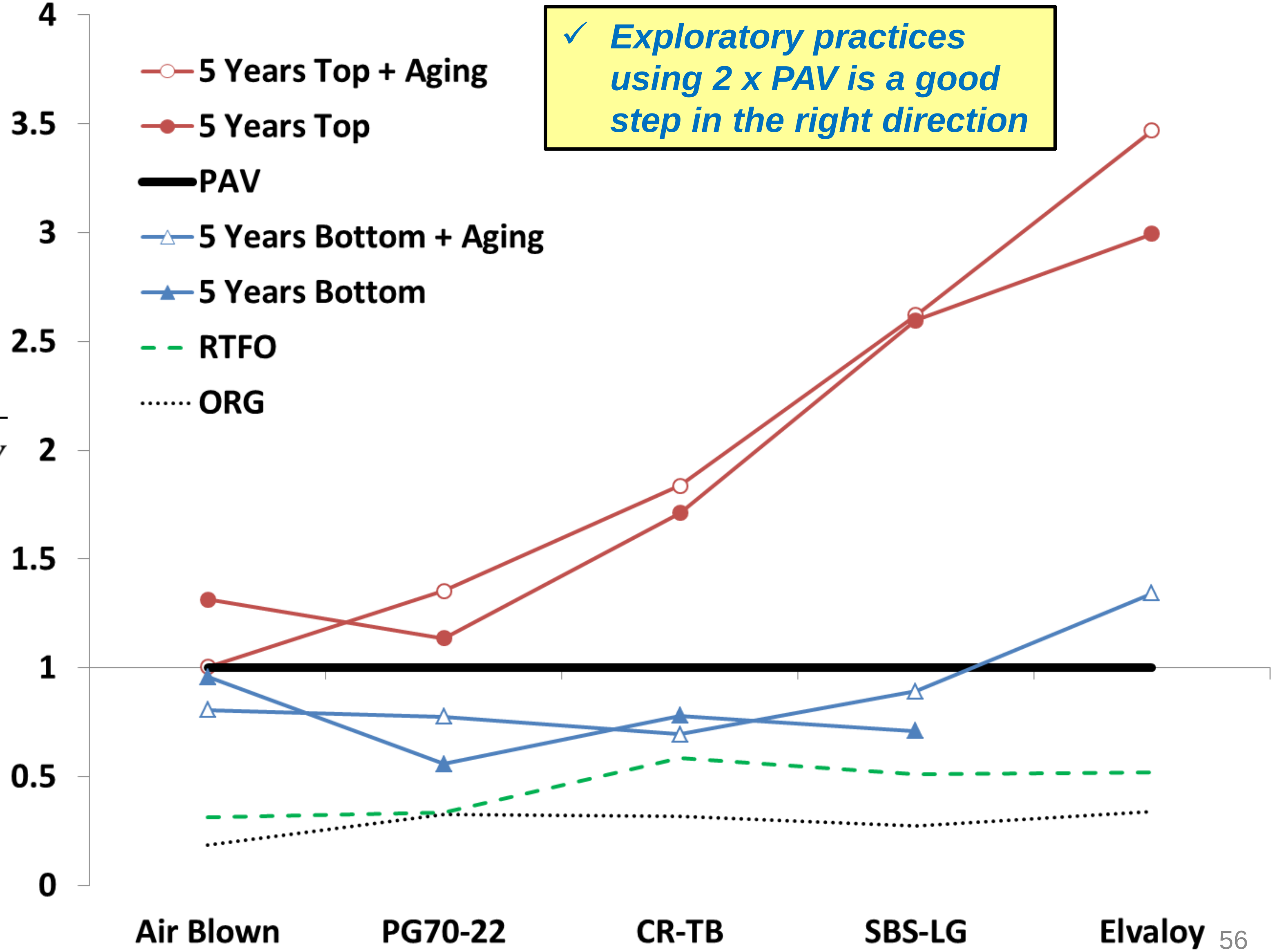




$$\frac{|G*|\sin(\delta)_x}{|G*|\sin(\delta)_{PAV}}$$

10Hz 19C



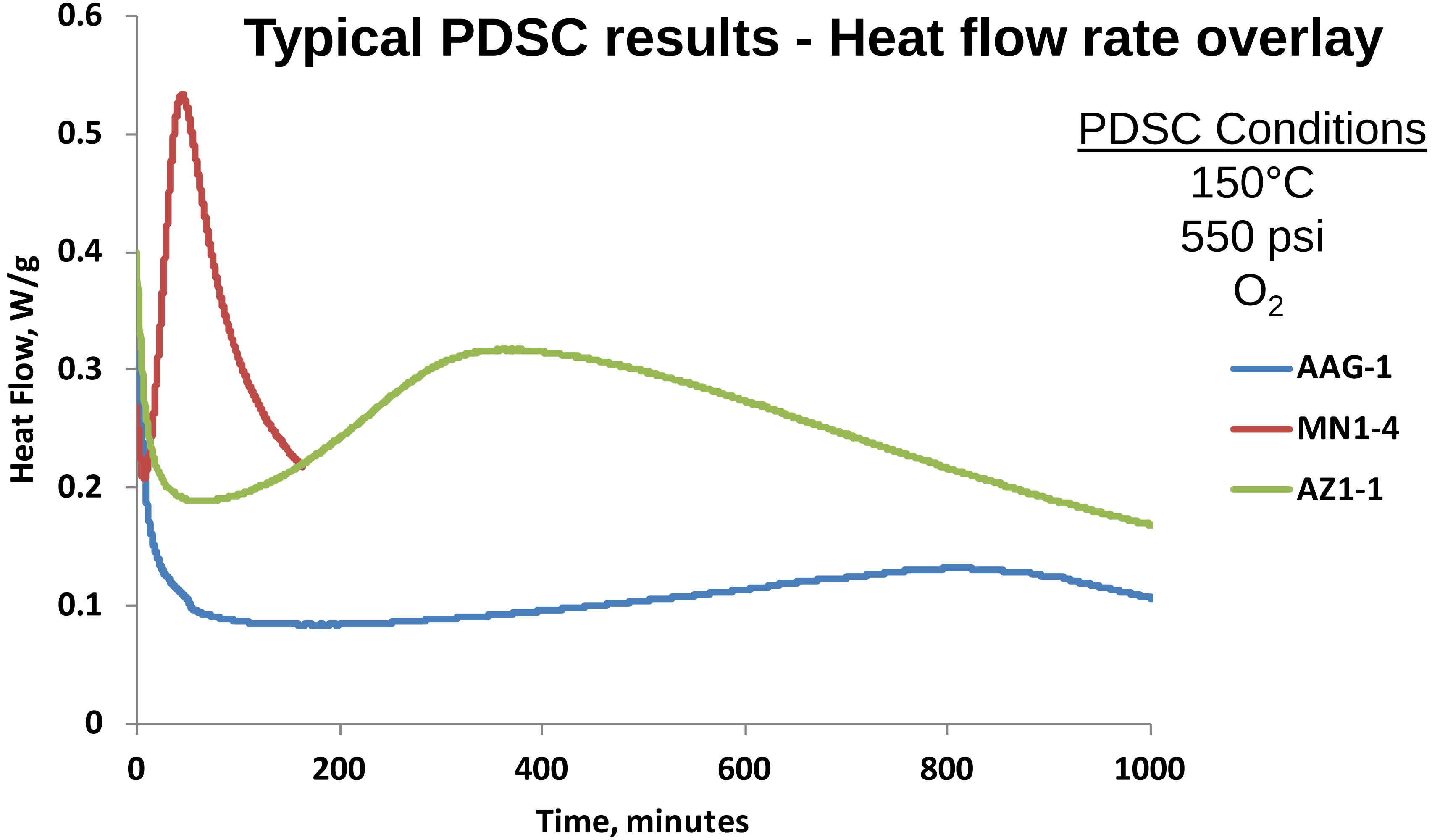


$$\frac{|G*|\sin(\delta)_x}{|G*|\sin(\delta)_{PAV}}$$

10Hz 19C

✓ *Exploratory practices using 2 x PAV is a good step in the right direction*

Aging study: High Pressure DSC



Overlap of Phosphorous and Sulfur Peaks