



## **ARC Project Emulsion Task Force Update**

**Improvement of Emulsions' Characterization and Mixture Design for Cold Bitumen Applications**

**Hussain Bahia , Andrew Hanz, Timothy Miller, & Petrina Johannes**

**December 14, 2009  
Madison, WI**

# Outline

---

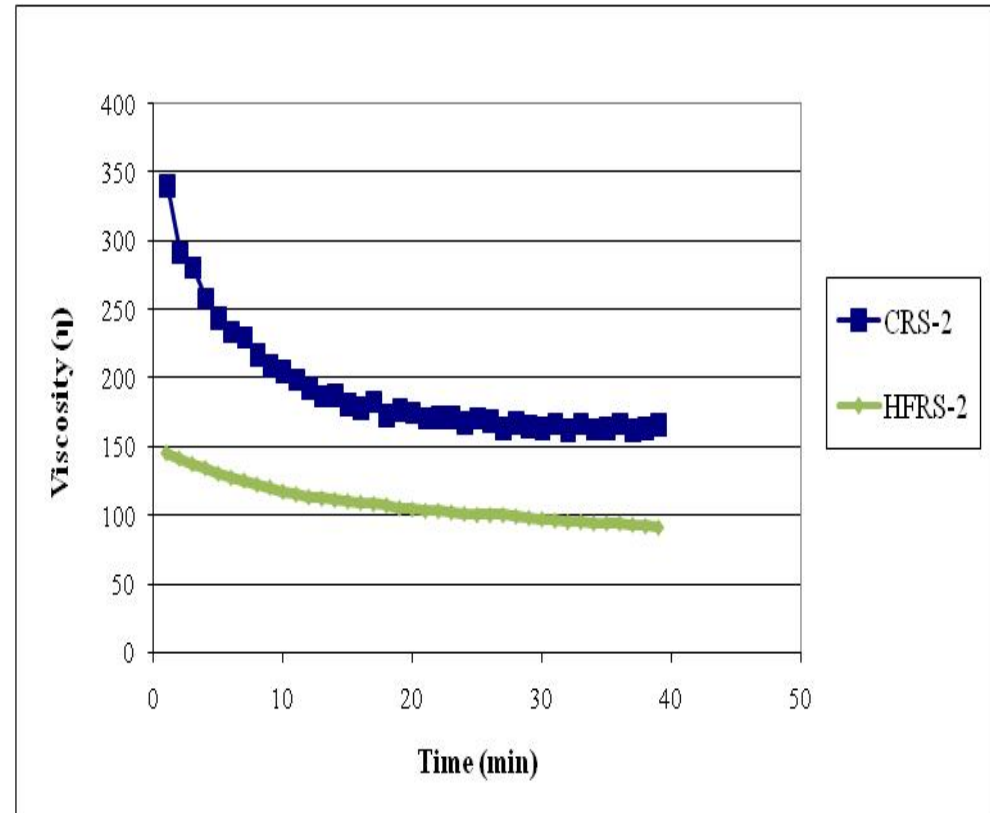
- **Construction Properties - BBS and RV**
  - **Emulsion Viscosity in the RV**
  - **Bitumen Bond Strength Testing**
- **Residue Evaluation - DSR**
  - **Aging Considerations**
  - **Performance Testing**
- **Year 4 Work Plan Focus Areas**

# Pertinent Construction (Emulsion) Properties - Chip Seals

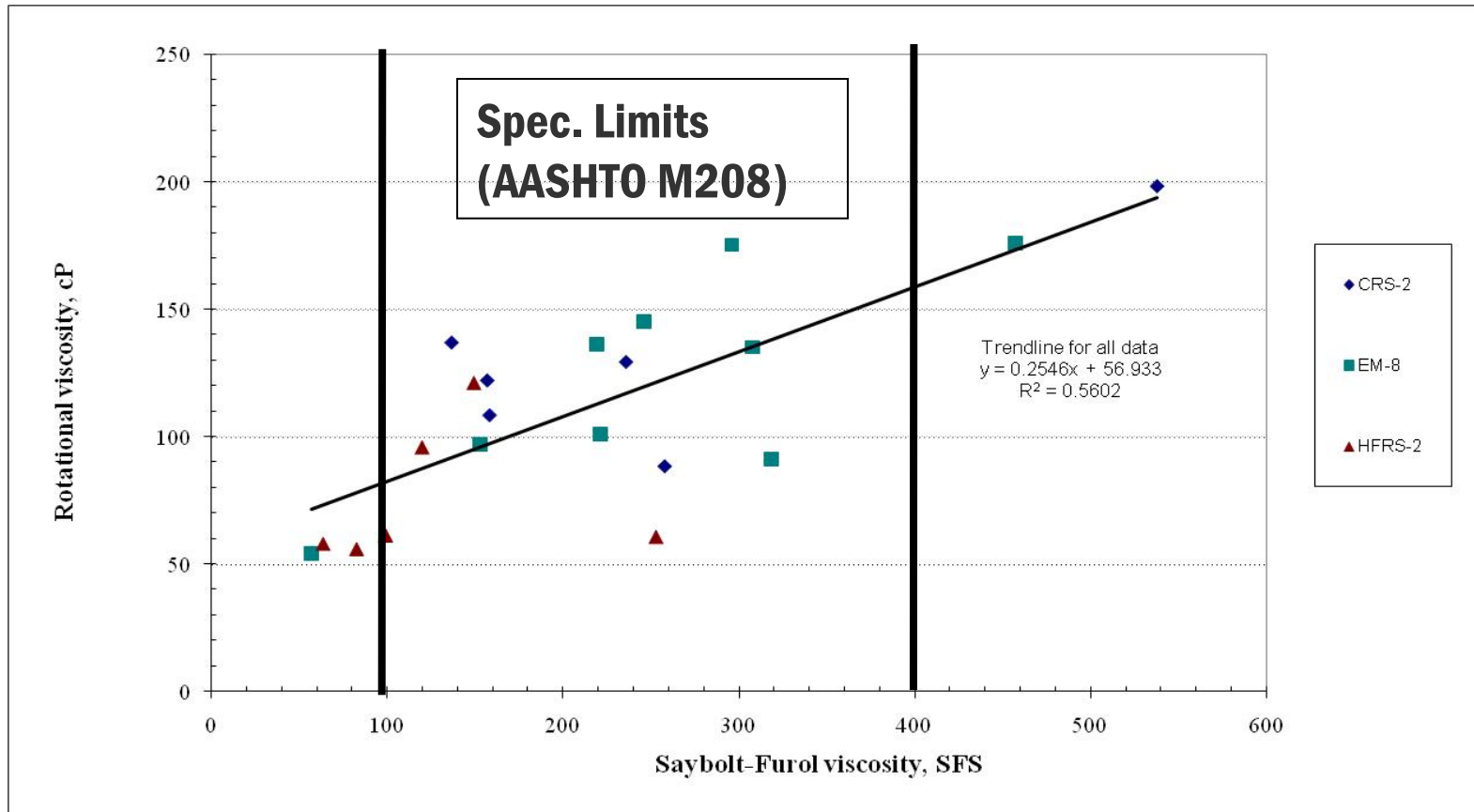
Engineering Property	Parameter(s) Measured
<b>1. Construction Properties</b>	
<b>1.1 Storage Stability</b>	Difference in residue - top and bottom of storage vessel - 24 hrs.
<b>1.2 Spray-ability and Drain Out</b>	Viscosity @ application temp. Shear rate to simulate pumping and placement
<b>1.3 Breaking / Setting Rate</b>	Change in bond strength with time.
<b>1.4 Early Raveling</b>	Bond Strength at a given curing time.

# Use of RV to Measure Emulsion Viscosity

- Evaluate steady state viscosity using RV
- Testing conditions
  - 50 °C, 50 RPM, # 21 spindle
- Relationship to current methods
  - Compare to Saybolt-Furol viscosity



# Preliminary Results – RV (50 RPM) vs. SFS



# Concepts for Evaluation of Viscosity

- **Vary Shear Rate to simulate field conditions**
  - **Spray-ability** –  $\eta$  at ***high*** shear rate
  - **Drain Out** -  $\eta$  at ***low*** shear rate
  - **Can RV producing a shear rate that simulates spraying?**
- **Relationship to Saybolt-Furol Viscosity**
  - **How relevant is it?**

# Challenges – Based on Discussion with ARC Advisory Group

- **Steady Shear Viscosity**
  - Is large drop in  $\eta$  recoverable? Or does it reflect damage in the material during initial testing?
  - Initial results show this is not recoverable.
- **Thermal History – Disconnect between lab and field.**
- **Effect of Shear Rate**
  - Define effect of shear rate on steady state viscosity.
  - Test must simulate field conditions.

# Next Steps for Viscosity

- **Address Comments provided by Advisory group.**
- **Based on results develop testing procedure**
  - **Steady state viscosity at low shear rate, then high shear rate on the same sample.**
- **Testing Conditions for initial evaluation**
  - **Temperature: 50°C, 65°C, 80°C**
  - **Shear Rates (RPM): 1, 5, 20, 50, 150**



# Adhesion Testing

- Chip seal performance highly dependent on development of adhesion between emulsion and aggregate chips.
  - Current test is qualitative – ASTM D244 Coating Ability
- Concept is to develop a simple test to measure:
  - Bond strength, development of adhesion
  - Aggregate / emulsion compatibility
- Validation – Test Entire System
  - Sweep Test (ASTM D7000) – Aggregate Loss

# Early failures due to lack of adhesion and climate effects



Constructed 27/04 and  
trafficked for 1 warm day

Constructed 29/04  
Temperature drop 30/04

Source: Gerrie Van Zyl – RSA

# Bitumen Bond Strength Test (BBS)

- **Test Method Development**
  - Procedure
  - Equipment
  - Factor Screening Experiment
- **Relationship to Chip Seal Performance**
  - Correlation with Sweep Test
- **Draft AASHTO Procedure – For ETF Review**

# BBS Procedure

- **Aggregate Plate Preparation**
  - Sawing parallel faces, lapping
- **Emulsion Application and Curing**
  - Sample weight: 0.4 – 0.5g. Curing controlled in environmental chamber.
- **Apply Stub and Acclimate to Laboratory Conditions (1 hr).**
- **Testing**

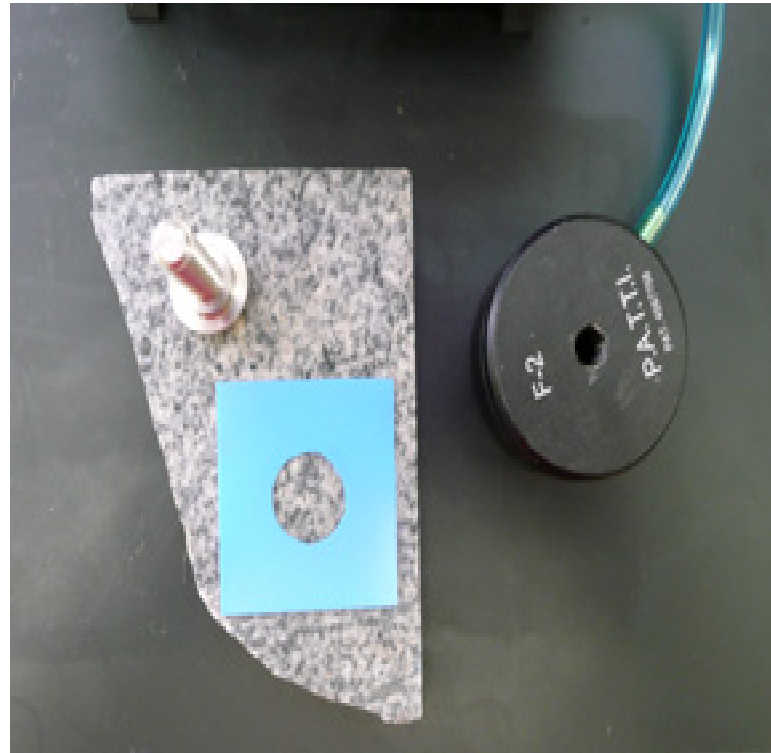
# BBS Procedure (cont)

- **Data Analysis and Interpretation**
  - **Pull Off Tensile Strength**
  - **Ensure consistent loading rate**
  - **Examine/Image Failure Surface**
    - **Adhesive Failure**
    - **Cohesive Failure**
- **Detailed steps provided in draft AASHTO standard.**

# Adhesion Testing – PATTI Quantum Gold Testing Set up



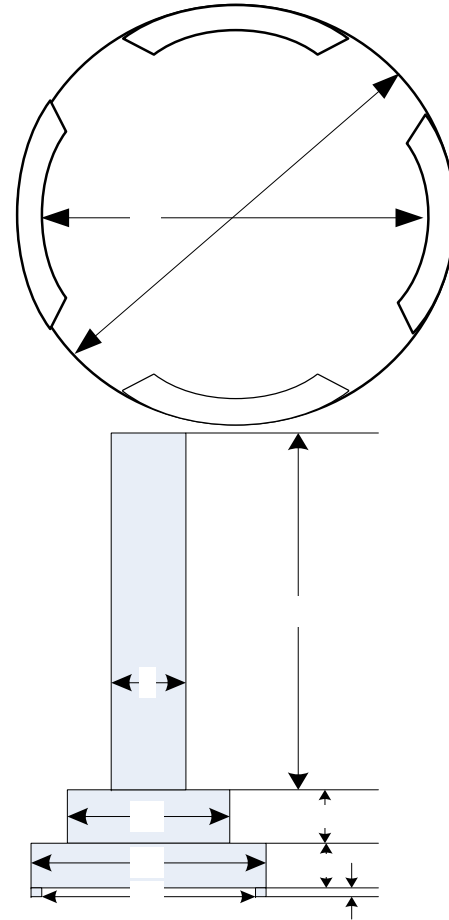
Aggregate Plate and stub.  
Digital display gives POTS.



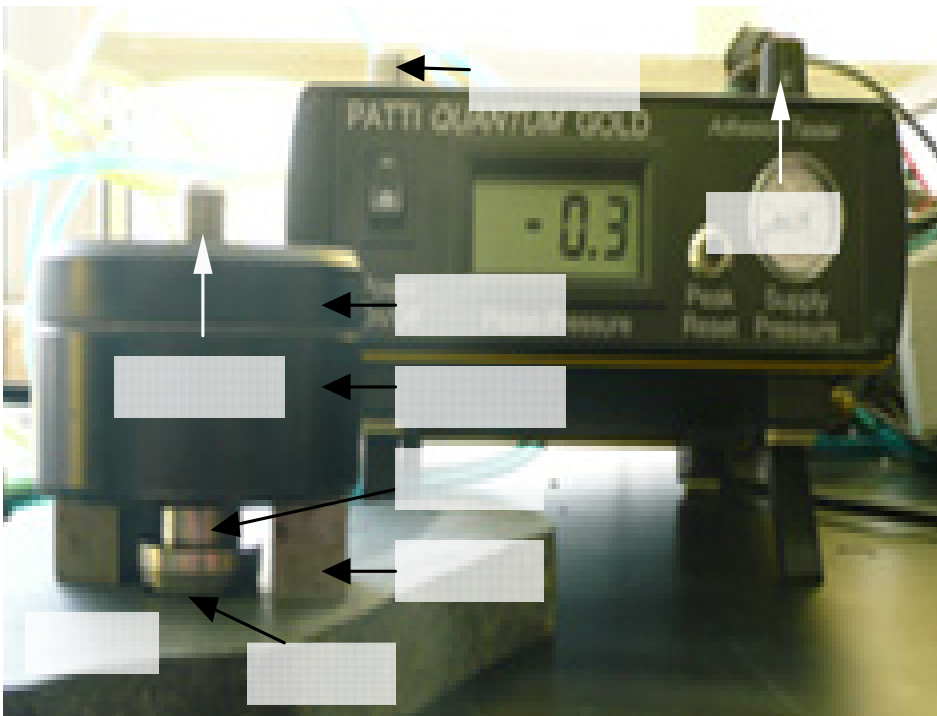
Loading plate and materials  
used in test preparation.



# BBS Stub Geometry



# Testing Apparatus

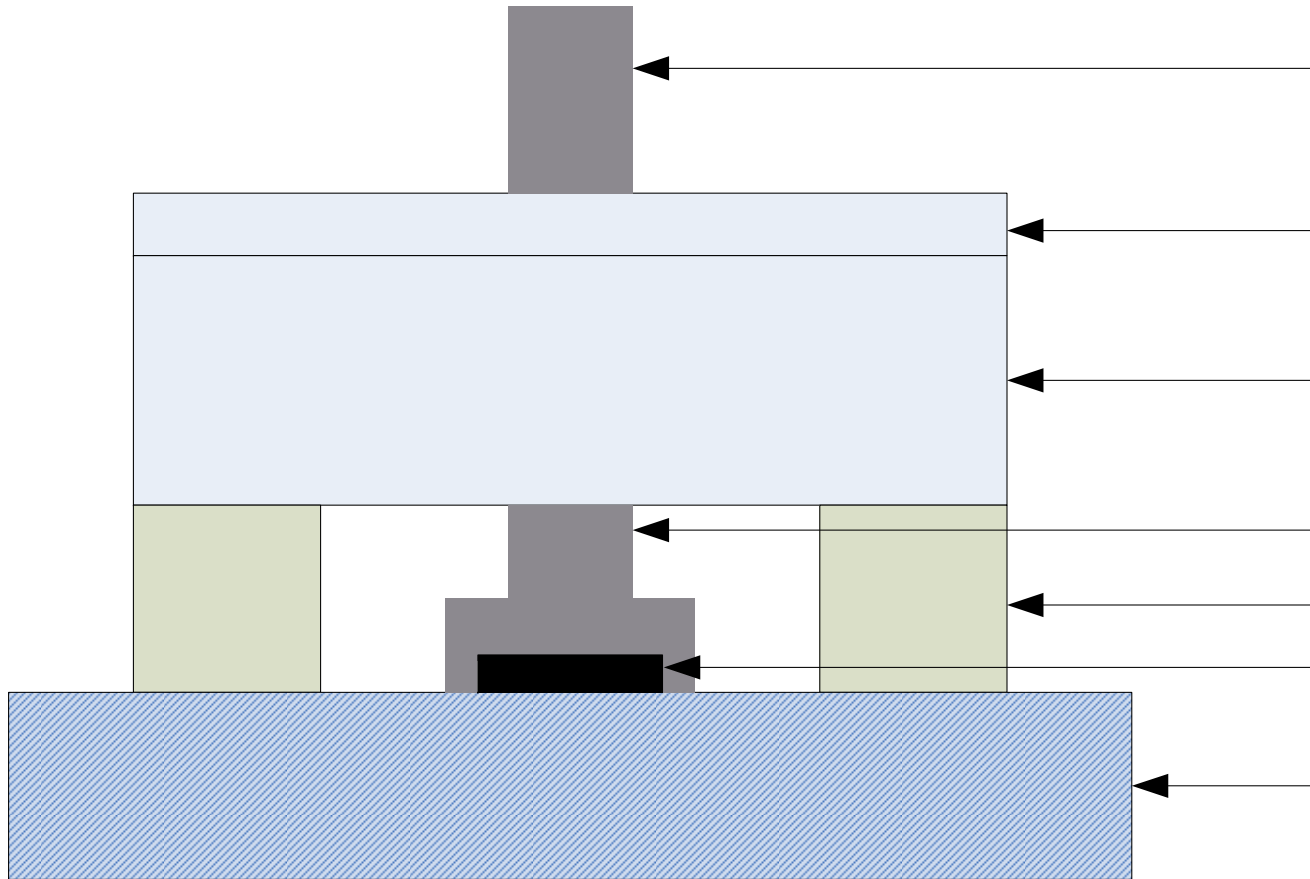


**Graded Scale for Air Flow  
Control Test Button**

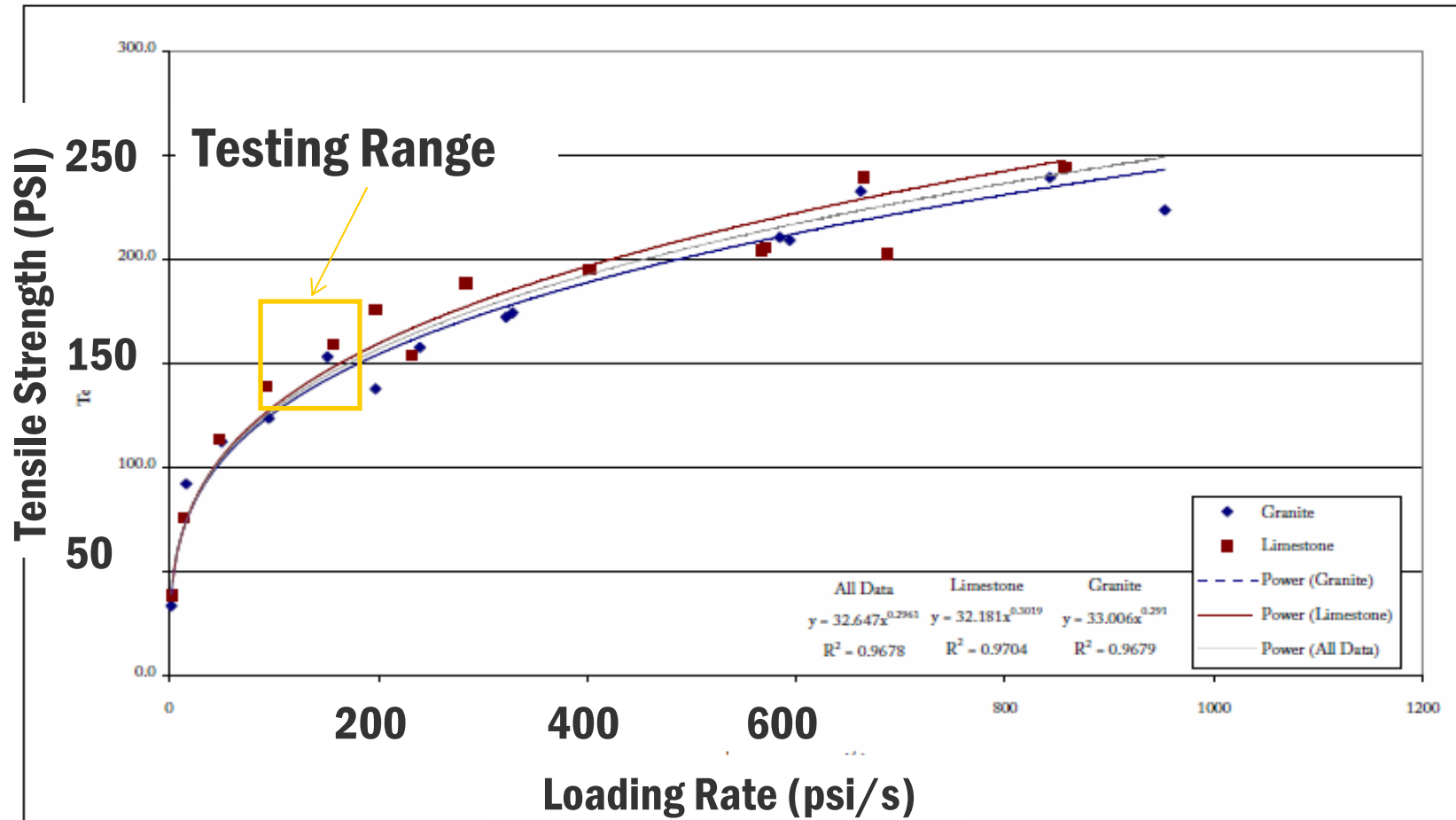
Pressure Plate



# Schematic of Complete Testing Assembly



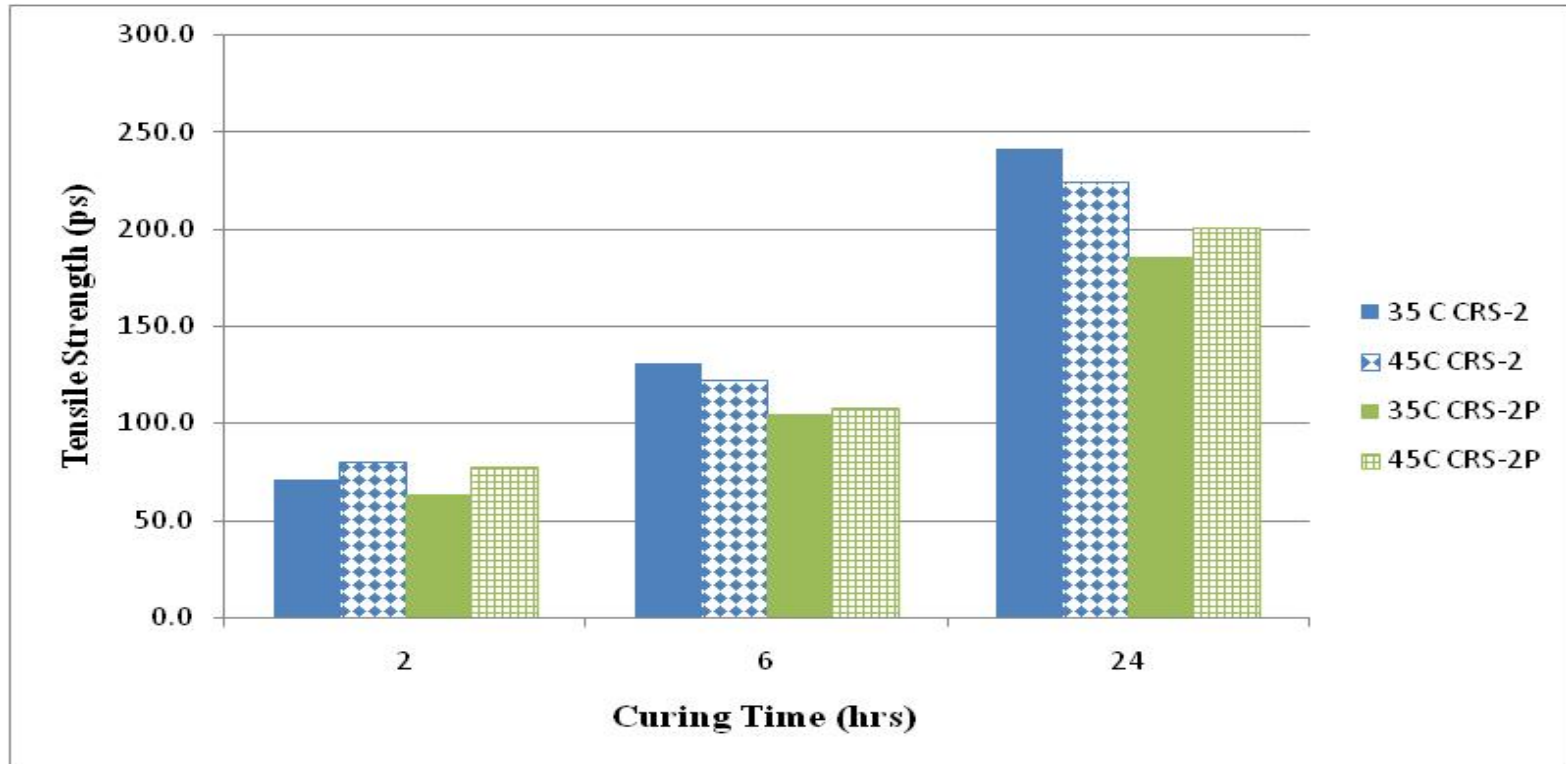
# Is Control of Flow Rate Important? Yes



# BBS Identification of Significant Factors

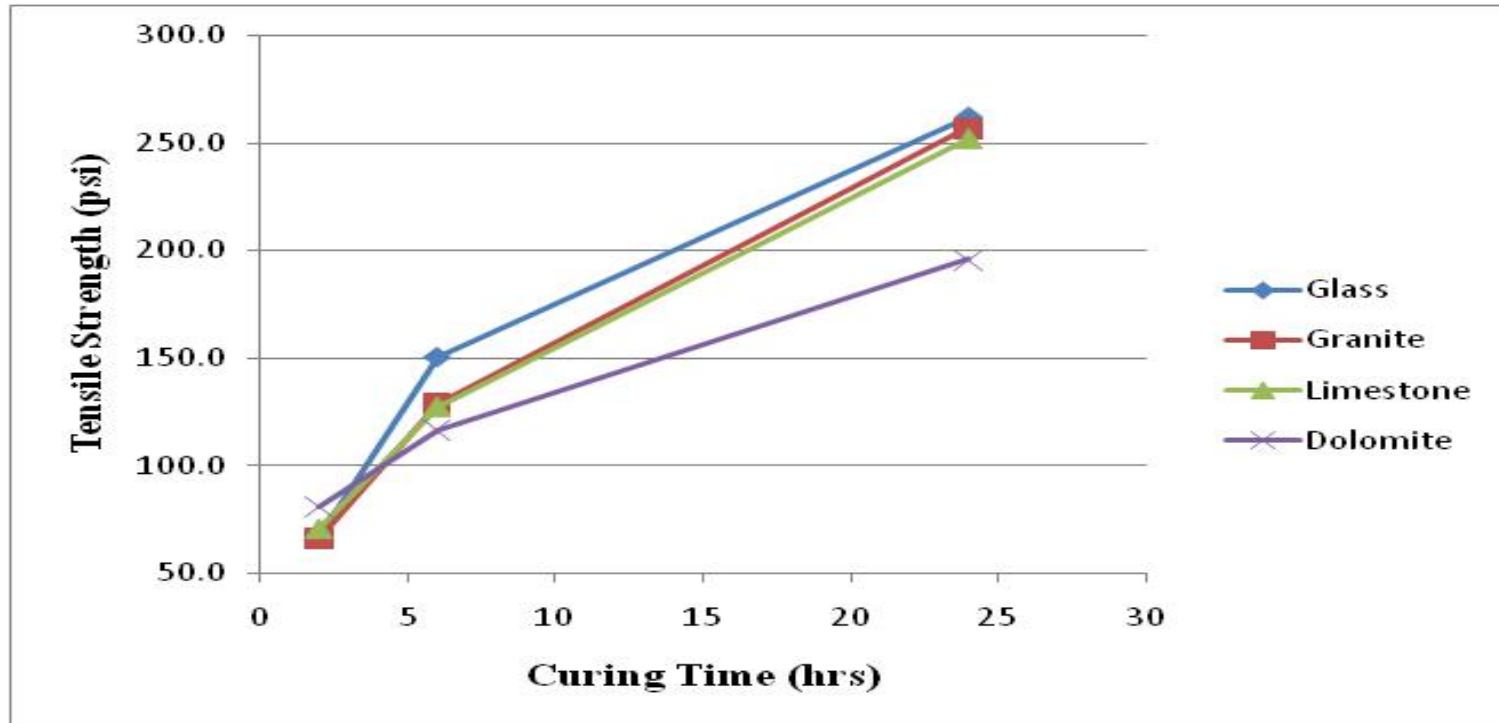
- **Environmental Conditions**
  - **Control Humidity – 30% RH**
  - **Temperature (°C) – 35, 45**
- **Aggregate Type**
  - **Glass (reference), Granite, Limestone, Dolomite**
- **Emulsion Type**
  - **CRS-2 vs. CRS-2P**
  - **Same Base Binder**

# Typical Results – Effect of Curing Temperature



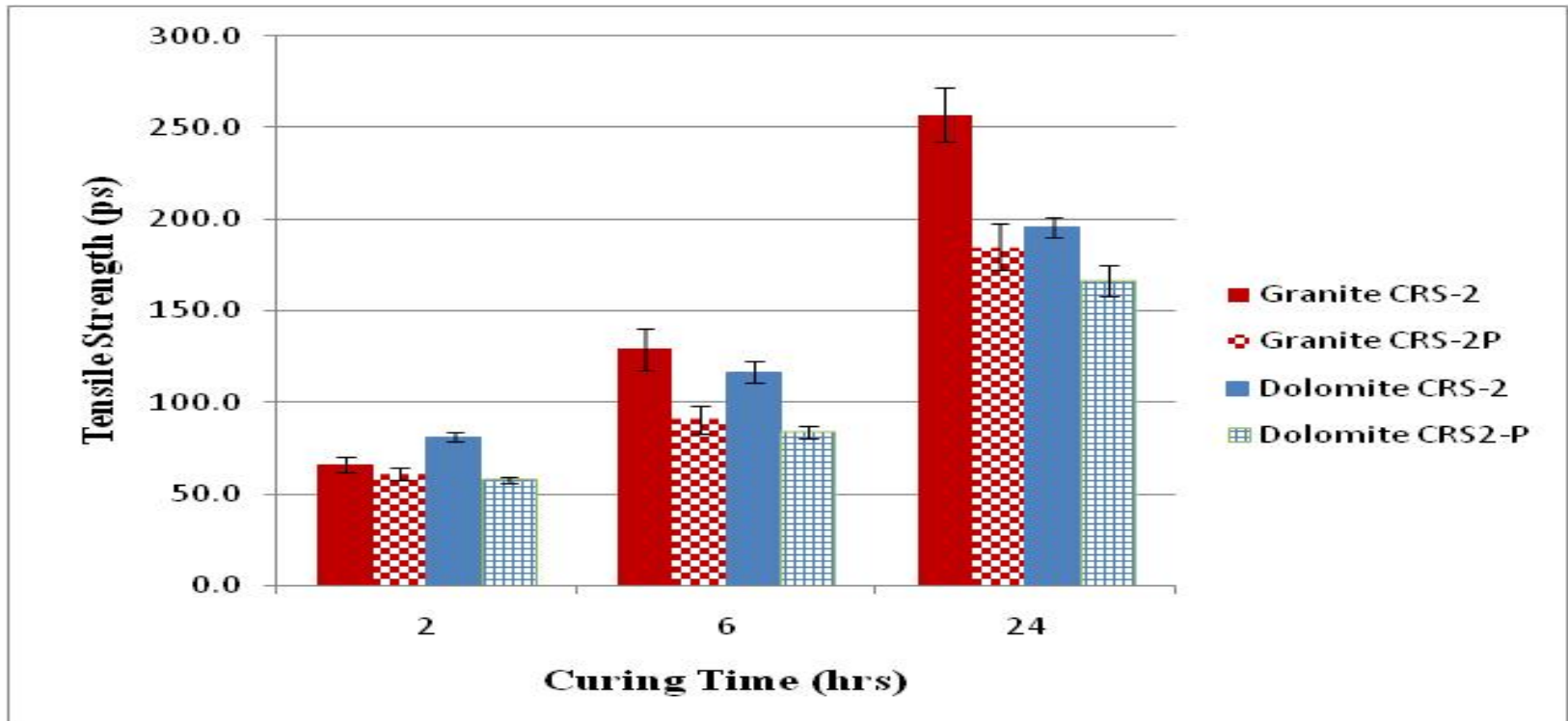
- No effect of curing temperature for either emulsion.
- Skinning at 45C? Select 35°C for further testing

# Typical Results – Effect of Substrate – CRS-2



- **Further investigation needed for performance of dolomite. Freshest surface – effect of surface charge?**

# Typical Results - Effect of Modification - CRS-2 vs CRS-2P

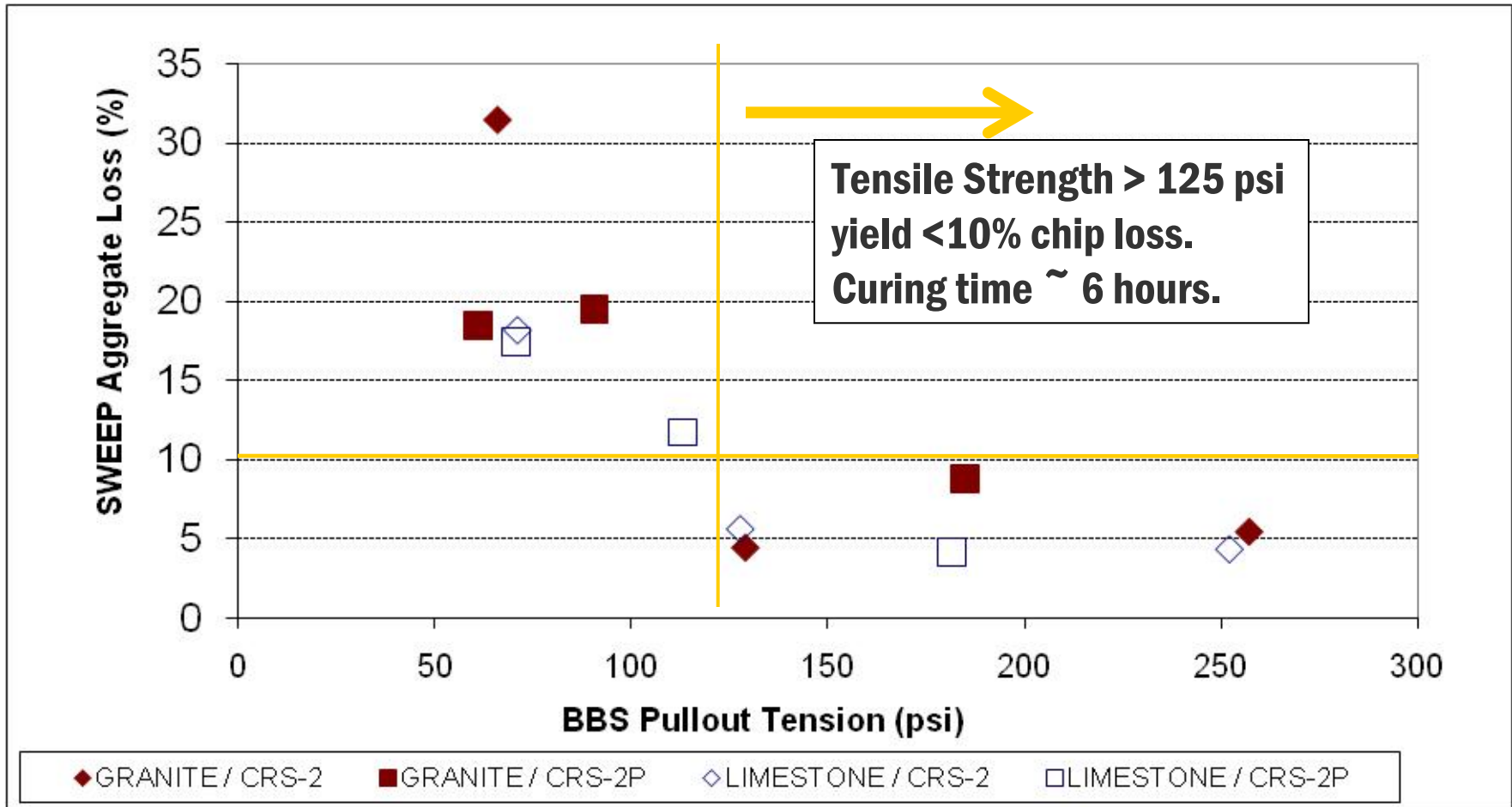


- **CRS-2 > CRS-2P in all cases. Why? Demuls/Viscosity, extra curing time needed?**

# Adhesion Testing – Completed & Next Steps

- **Screening Experiment (ANOVA) – 90% Confidence Level**
  - **Curing Conditions Significant**
  - **Aggregate Type must be considered**
  - **Aggregate Moisture (dry vs. SSD) and Surface Roughness (insignificant)**
- **Define “Optimum” Loading Rate – Replicated ANOVA @ 6 hrs cure.**
- **Evaluate effect of curing time at selected loading rate**
  - **2 hrs**
  - **24 hrs**
- **Relate to Sweep Test**

# BBS Relationship to Performance





# BBS Test – Next Steps

- **Relationship to Performance**
  - Continue comparison to Sweep Test
  - Comparisons to Field Performance
- **Test Method Evaluation**
  - Continue to conduct test on various emulsions/substrates
  - Collaboration with Stellenbosch
- **Finalize Draft Standard and establish precision/bias.**

# Discussion Points

---

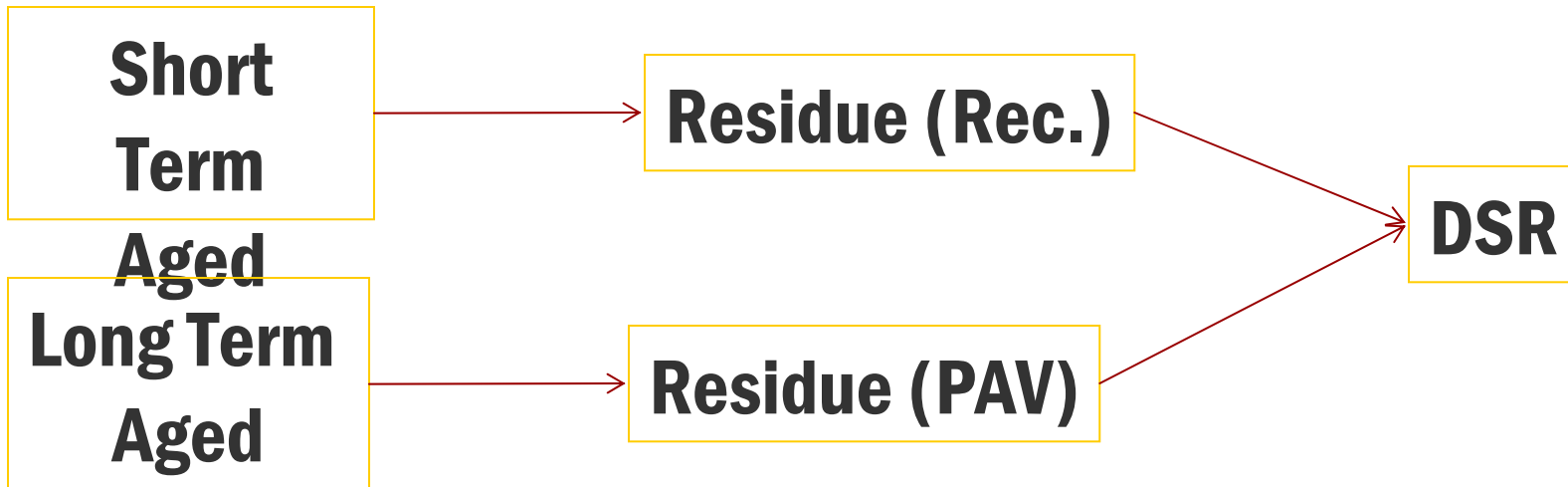
- **More ideas for new tests of emulsions using existing PG equipment. Is there a need for an adhesion test?**
- **Surface Treatment Performance**
  - **How do we define it? What are important factors (Traffic, Climate, Materials)?**
- **Are modified emulsions worth it? Develop database to quantify effects of modification on performance.**

# Emulsion Characterization - Residue Aging

## Construction Properties



## Performance Properties



# Proposed Residue Evaluation Framework

Property	Aging Level	Testing Temperature	Proposed Procedure	Potential Evaluation Criteria
<b>6.1. Resistance to Bleeding</b>	Recovered Residue	High Surface Temperature	MSCR (100 kPa/3200kPa)	<b>Jnr Stress Sensitivity</b>
<b>6.2. Resistance to Early and Late Raveling</b>	Recovered Residue PAV Residue	TBD	Strain Sweep	<b>Strain at 50% Reduction in G*</b>
<b>6.3 Fatigue Cracking</b>	PAV Residue	TBD	Frequency Sweep	<b>TBD</b>
<b>6.4 Thermal Cracking Resistance</b>	PAV Residue	10°C	Frequency Sweep	<b>Estimates of BBR Properties S(60) and m(60)</b>
<b>6.5. Polymer Identifier</b>	Recovered Residue	25°C	Elastic Recovery DSR Procedure MSCR	<b>%ER - DSR % Recovery</b>

# Emulsion Residue Aging – Short Term

- **ASTM Evaporative Residue Recovery Method**
  - Residue Rheology ~ Properties of RTFO aged base materials
  - Preserves Effects of Modification
- **Research Challenges**
  - Establish relationship to field
  - Reduce 48 hour recovery time



Kadrmaz – TRB 2009 Session  
791

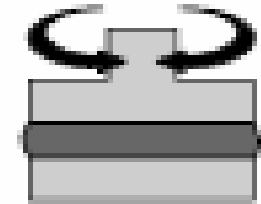
# Emulsion Residue Aging - PAV

- **Available Procedure: PAV for Hot Binders**
  - Aged at 90 – 110°C at 300 psi for 20 hours
- **Challenges in applying PAV procedure to emulsion**
  - PAV Temperature > Softening point of emulsion residue (40 - 60 °C)
  - Effect: Latex structure in emulsion residue could be compromised
- **Previous work: PAV at 85°C for 65 hours (Guet, et. al)**
  - Microscope images showed presence of polymer (SBS), however
  - Cohesion and ER greatly reduced – inconsistent with field performance.
- **Propose PAV at 60°C for 120 hours**
  - Very long aging time – but insight into rheology is needed.

# Residue Performance Characterization – Concepts and Examples

- **Previously Presented Tests**

- High Temp. – MSCR: Jnr
- Polymer Identifier – MSCR: % Recovery
- Intermediate – Strain Sweep: Failure Strain

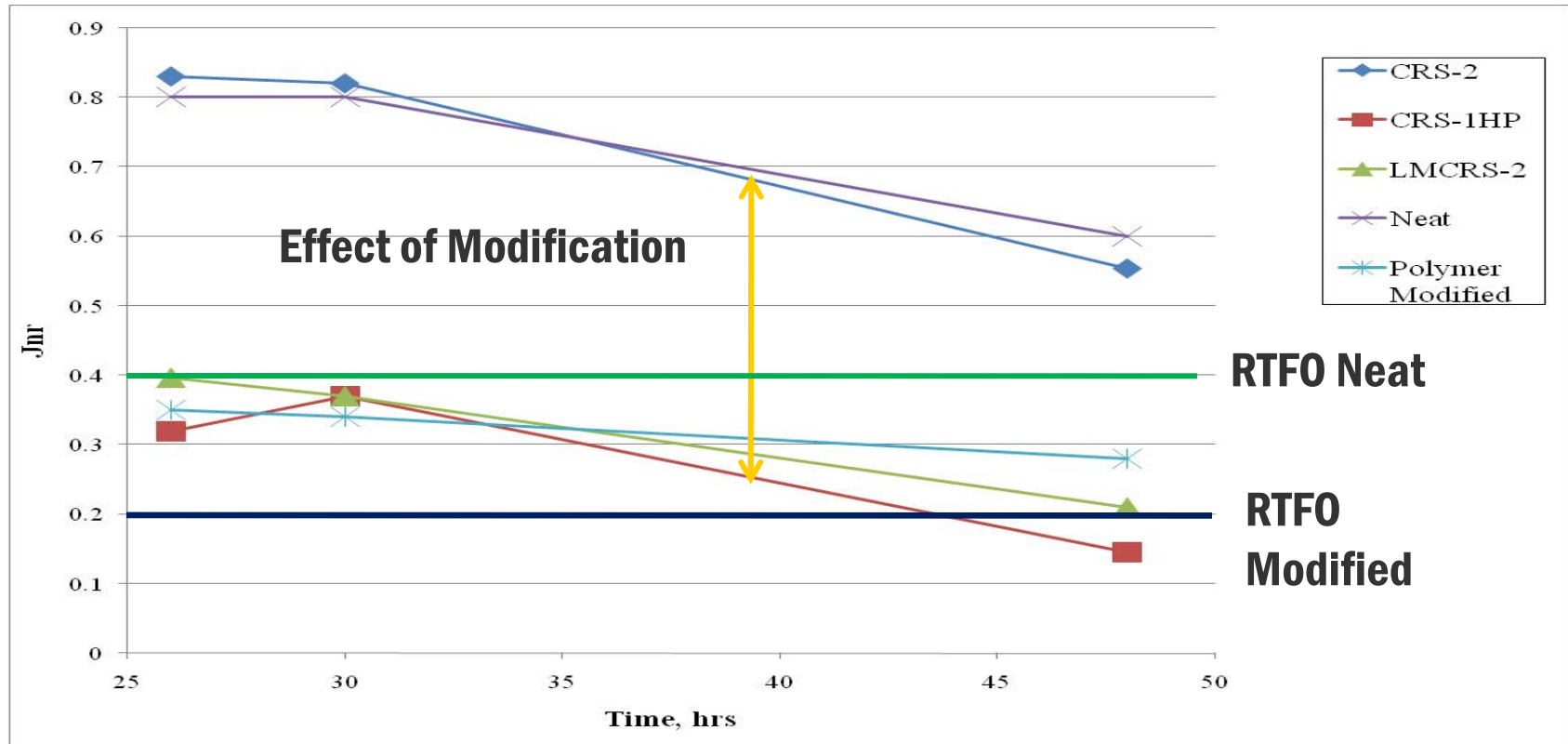


- **Newly Developed Tests**

- Elastic Recovery in DSR
- Low Temperature – DSR to estimate BBR Performance



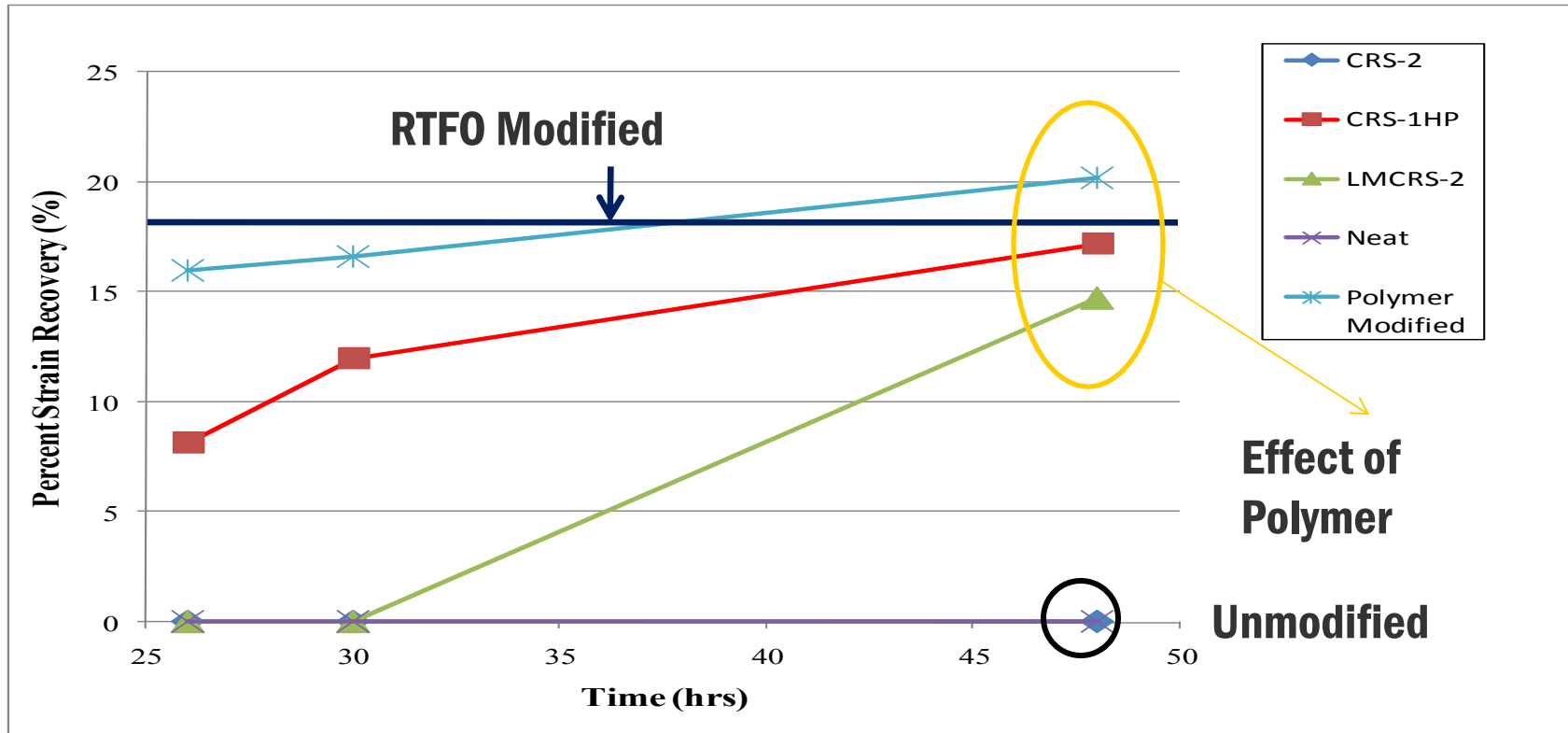
# High Temperature Evaluation - MSCR



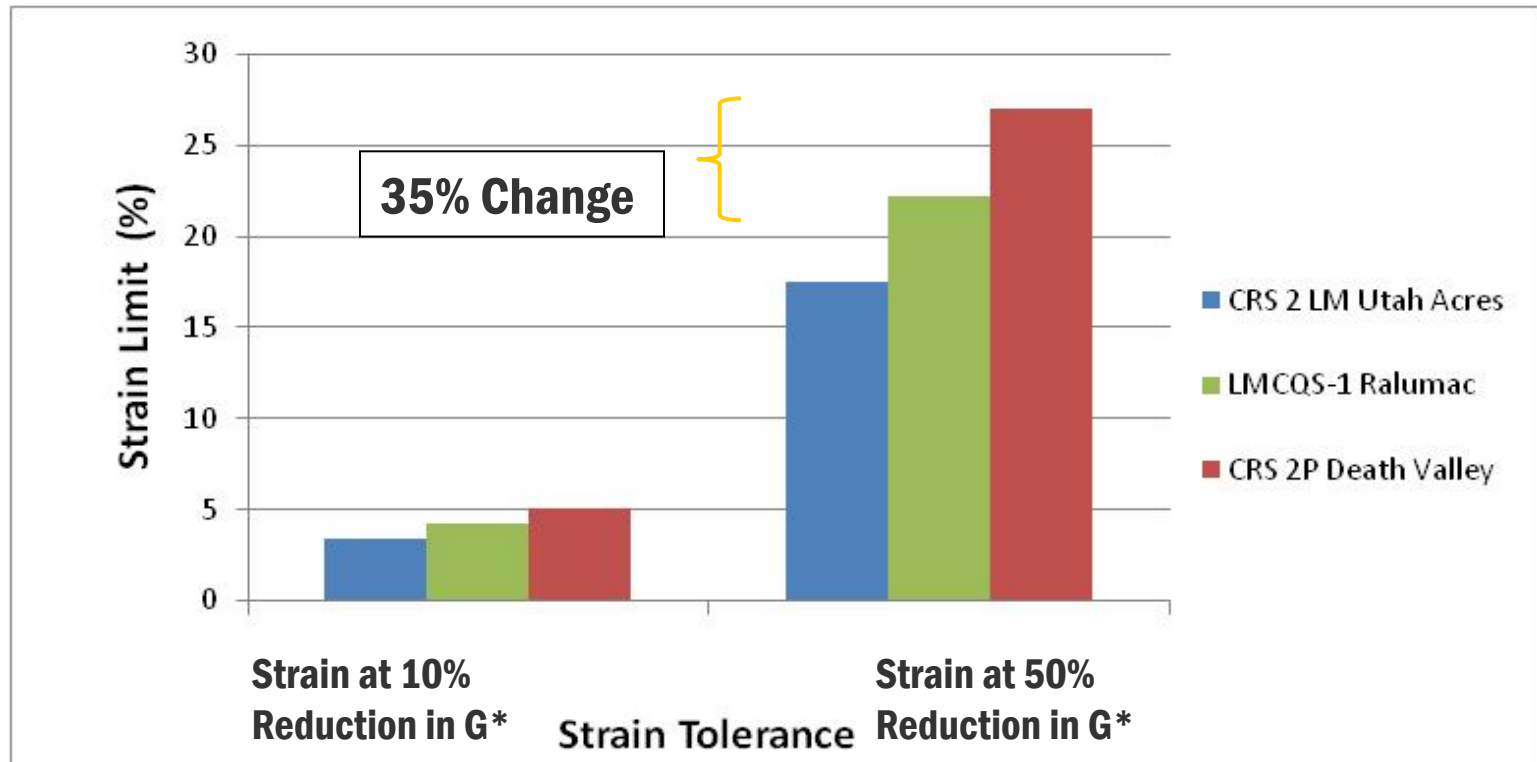
Displays effect of modification and curing time on Jnr.



# MSCR % Recovery – Effect of Modification



# Strain Sweep of residue @ 25 C (PRI data) - Effect of Modification

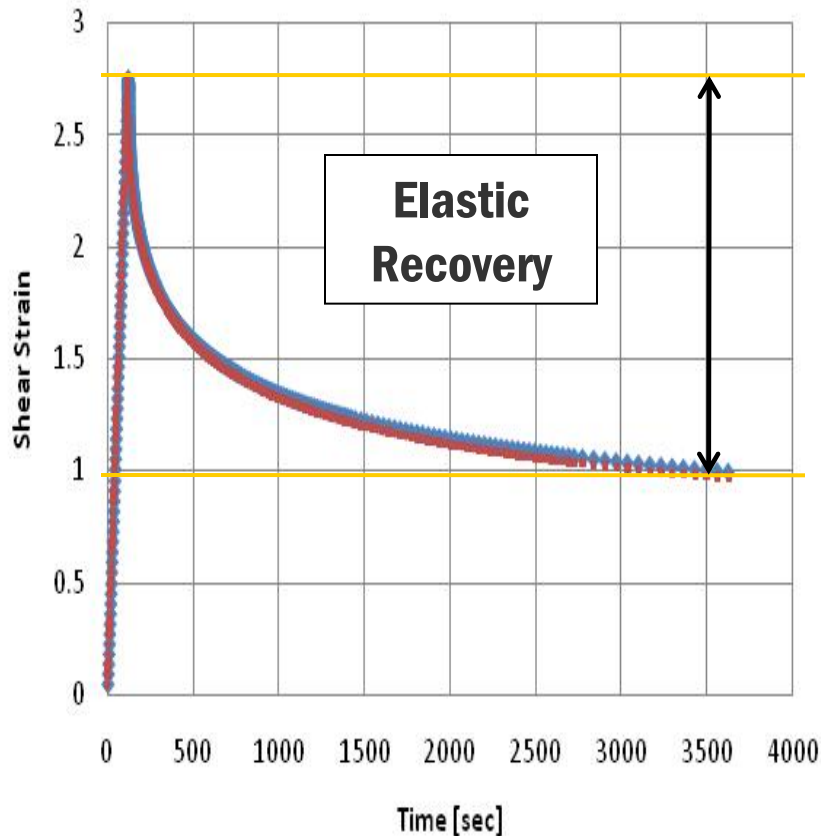


\*Data obtained from FLH Project

# Elastic Recovery in the DSR

- **Procedure**
  - **8 mm Parallel Plate Geometry**
  - **Testing Temperature: 25°C**
  - **Step 1: Strain controlled. Imposes a strain rate of 2.32%/sec for 120 s.**
  - **Step 2: Control stress to 0 Pa for 1 hour.**
- **Strain rate and loading time defined to match conditions of current Elastic Recovery test.**

# Elastic Recovery in the DSR - Schematic



- **Materials Tested**

- **Base Binders PG 58-28 to PG 64-22.**

- **Modifiers:**

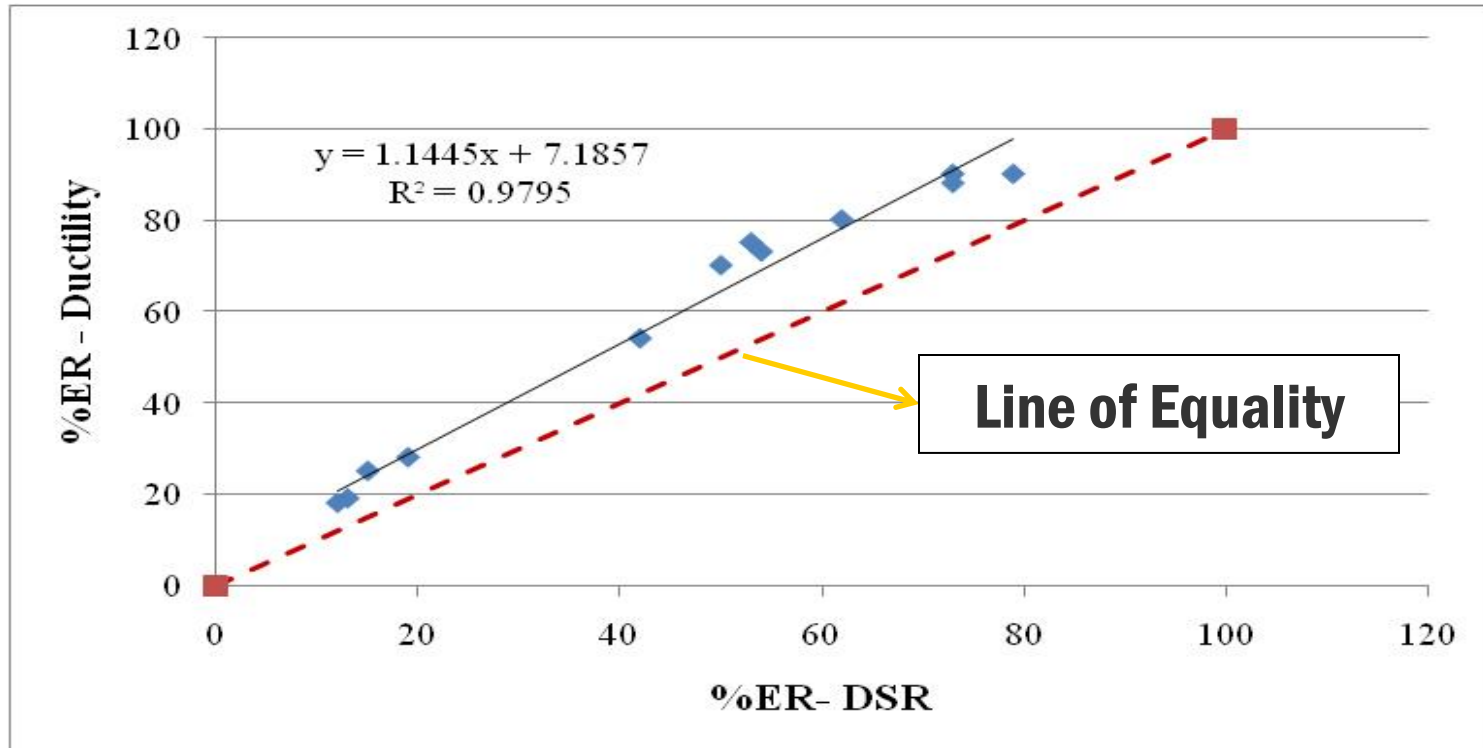
- **SBS**

- **Elvaloy**

- **PPA**

- **Base binders modified 2 levels.**

# Elastic Recovery in the DSR - Results



- **Strong relationship, but DSR test under predicts result from standard method.**

# Estimating BBR from DSR Data – Concepts

## Comparing Dynamic and Creep Measurements – solve for $\omega$

Equation (1): 
$$T_d \approx \left[ \frac{\frac{1}{273 - T_s} - 2.303R[\log(t_s\omega)]}{250,000} \right]^{-1} - 273$$

Where:

$T_d$  = test temperature for dynamic testing at frequency  $\omega$ , °C

$T_s$  = specified temperature for creep testing, °C

$R$  = ideal gas constant, 8.31 J/°K-mol

$t_s$  = specified creep loading time, s

$\omega$  = dynamic testing frequency, rad/s

- **Equivalent DSR  $\omega$  to measure  $S(60)/m(60)$  at 10°C = 20 Hz**
- **Data shows both 10Hz and 20 Hz used can be used.**

# Estimating BBR from DSR Data – Concepts (cont).

- Approximation of  $S(t)$

$$S(t) \approx \frac{3G^*(\omega)}{[1+0.2\sin(2\delta)]}$$
$$t \rightarrow \frac{1}{\omega}$$

.....  
 $S(t)$  = creep stiffness at time,  $t$ , Pa  
 $G^*(\omega)$  = complex modulus at frequency  $\omega$ , Pa  
 $\delta$  = phase angle at frequency  $\omega$ , Pa

- Approximation of  $m(t)$

$$m = \frac{d(\log G^*)}{d(\log \omega)}$$

Where:

$m$  = slope of  $G^*$  vs. Frequency plot at a given frequency  
 $\delta$  = phase angle  
 $G^*$  = complex modulus  
 $\omega$  = frequency (rad/s)

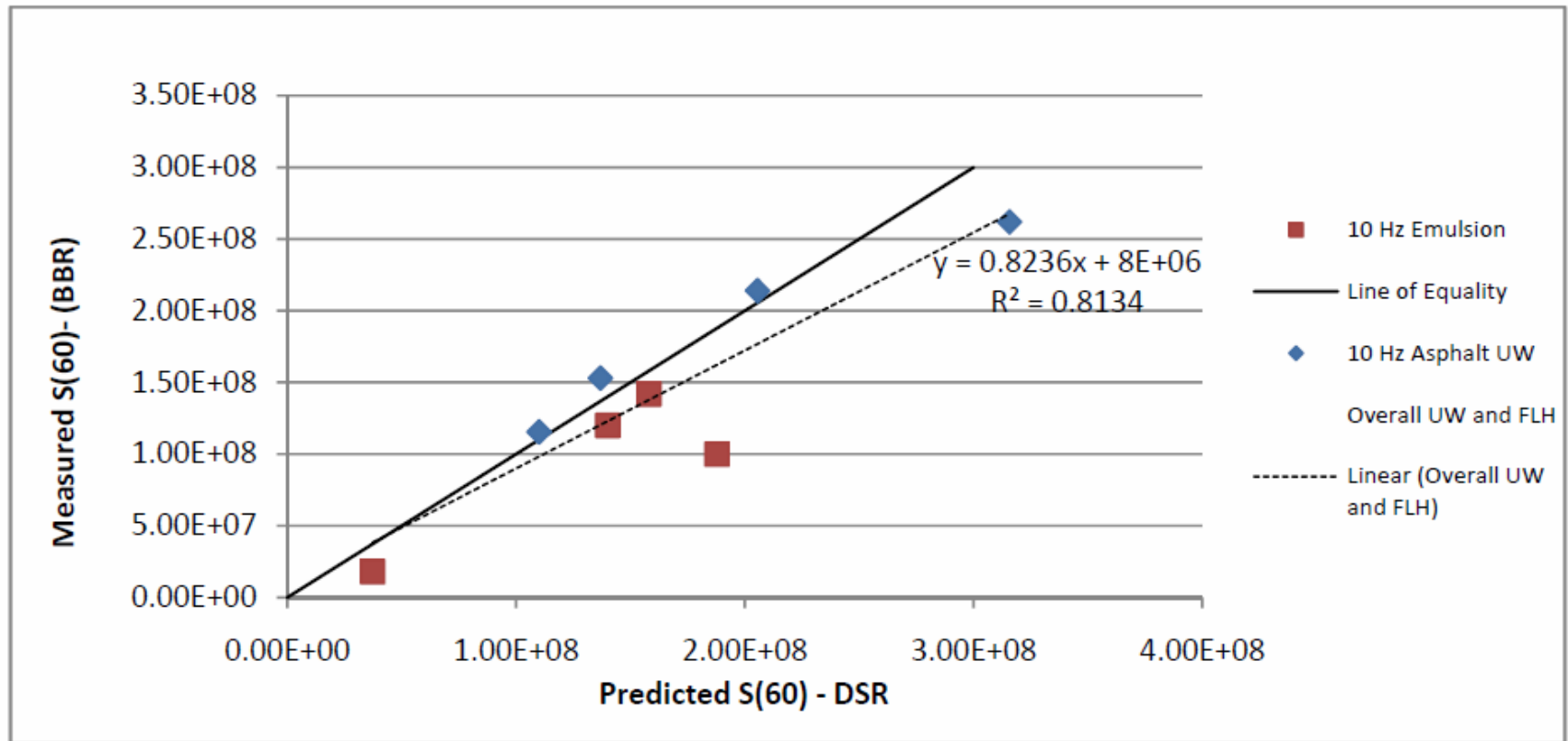
Ferry, J.D. Viscoelastic Properties of Polymers. Madison, WI: University of Wisconsin, 1980.

# Estimating BBR from DSR Data

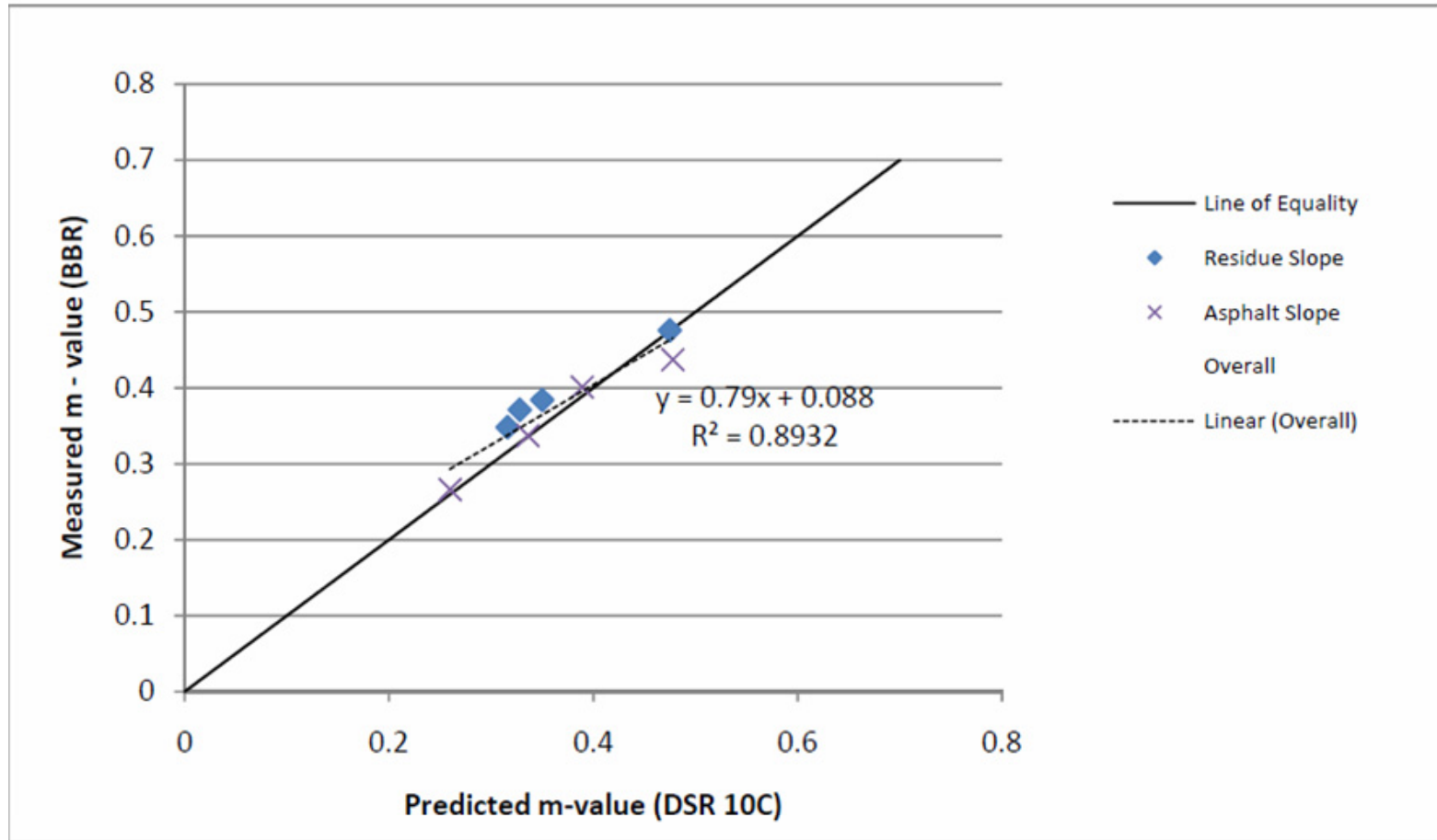
- **Procedure**
  - 8 mm Parallel Plate Geometry
  - Testing Temperature: 10°C
  - Frequency Sweep: 0.1 – 100 rad/s
- **Materials Used:**
  - Base Asphalt – 4 Levels of Aging (OB, RTFO, PAV, 2PAV)
  - Four emulsion residues from FLH project.
- **Use  $G^*$ ,  $\delta$  at 10Hz to conduct comparison (20 Hz) not available for all materials**



# Estimate of S(60)



# Estimate of m(60)



# Year 4 Work Plan Focus Areas

- **Performance Properties of Emulsions**
  - Establish range of performance for various emulsion residues.
- **Improvements to the Sweep Test**
  - Use as a design tool
  - Examine modifications to procedure
  - Apply to other distress modes: bleeding.



# Year 4 Work Plan Focus Areas

- **Field Validation**
  - Construction and performance thresholds based on field performance.
  - Identify field tests to evaluate construction properties.
  - Field vs. Laboratory aging of emulsion/residue.
  - Link performance tests (DSR) to chip seal distress modes.
- **Dense Cold Mixes**
  - Define emulsion selection framework.
  - Develop mix design procedure and evaluation parameters.

# Thank you for your time!

**Hussain U. Bahia**

**[bahia@engr.wisc.edu](mailto:bahia@engr.wisc.edu)**

**Andrew Hanz**

**[ajhanz@wisc.edu](mailto:ajhanz@wisc.edu)**

