4 mm ETG Task Force Report

(Thermal Equilibrium - Sample Prep – Compliance)

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All three major equipment manufacturers

Many others – Mike Farrar – Alec Cookman - Gaylon Baumgardner – Sonia Serna

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Salt Lake City, UT

Historical Observation – DSR Test Method

- ✓ Early on it was recognized that thermal gradients and thermal equilibrium can affect accuracy (lab bias)
  - Thermal gradients are currently accounted for with a dummy specimen and a temperature offset
- ✓ Thermal equilibrium is considered in the current AASHTO and ASTM test methods by a finite wait time (10 min)
  - No time limit is given for completion of data acquisition
- ✓ Test procedure is built around specification measurements at 10 rad/s based on early generation DSR's
  - Measurements at temperatures where G* ranges from 100 Pa to 10 MPa
  - Non-specific fixture compliance is fine

But What are the 4-mm issues?

1. Verification of DSR as part of laboratory QC Program
   Overall operation DSR ➔ reference fluid ➔ present OK
2. Fixture-specific device compliance
   Addressed with draft protocol
3. Thermal equilibrium determination
   Addressed with draft protocol
4. Specimen preparation protocol
   Addressed with draft protocol
5. Unresolved
   Measurement of specimen temperature
   Ruggedness, round-robin testing, data reduction

Success –
Three documents released to ETG !!!!

1. Verification Issues

- Verification of torque transducer with reference fluid
  - ✓ Verifies overall operation, not the torque transducer alone
  - ✓ Verification temperature independent
  - ✓ Replacement not needed
- Verification of temperature transducer
  - ✓ Current 25 mm diameter wafer unacceptable
  - ✓ Need replacement - questionable for 8 mm
  - ✓ Most critical issue
  - ✓ Issue not resolved but some promising leads

2. Fixture-specific machine compliance

- Several procedures available (solid rod, WRI, MTE, etc.)
  - ✓ Procedures documented in literature by others
- Two methods recommended by task force
  - ✓ Method A uses ice to bond top and bottom plates
  - ✓ Method B uses “crazy glue” to plates
- Objective is to accurately determine the strain in the rheometer so that when load is applied strain in the rheometer can be subtracted from total strain
  - ✓ Generic machine compliance work fine for normal strains
  - ✓ Not so when move to 4-mm plate at low temperature

Comments – Fixture-specific machine compliance

- Not routine procedure
  - ✓ Beyond capability of user-producer laboratories
- Not rocket science!
  - ✓ Needs operator skill - potential for machine damage
- When required, fixture-specific machine compliance should be supplied by manufacturer with fixtures
  - ✓ Best left to DSR manufacturer
- When is it necessary?
  - ✓ When machine strain is significant with respect to specimen strain
  - ✓ Guidelines forthcoming

3. Test Specimen Equilibrium, t_{SE}

- Monitor G* during 30 minute isothermal time sweep
  - ✓ Determine G* at 30 second intervals – 61 data points
  - ✓ For 57 data points calculate C_{SE} at t_i calculated as average absolute deviation for 5 data points from t_{i-2} to t_{i+2}
  - ✓ Express C_{SE} as percent of mean from t_{i-2} to t_{i+2}
- Thermal equilibrium time t_{SE} obtained when $C_{SE} \leq 1\%$
  - ✓ 1% must be maintained for remainder of 30 minutes
- Start testing at t_{SE} + 2 minutes
  - ✓ Five minute test window
  - ✓ Works at all temperatures

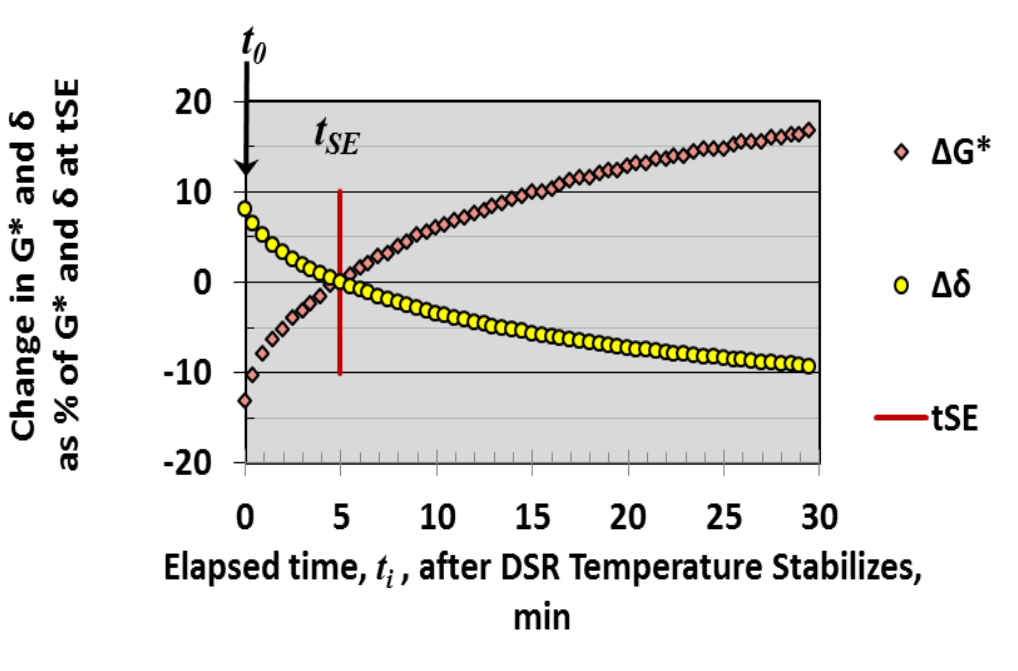
Specifying specimen equilibrium

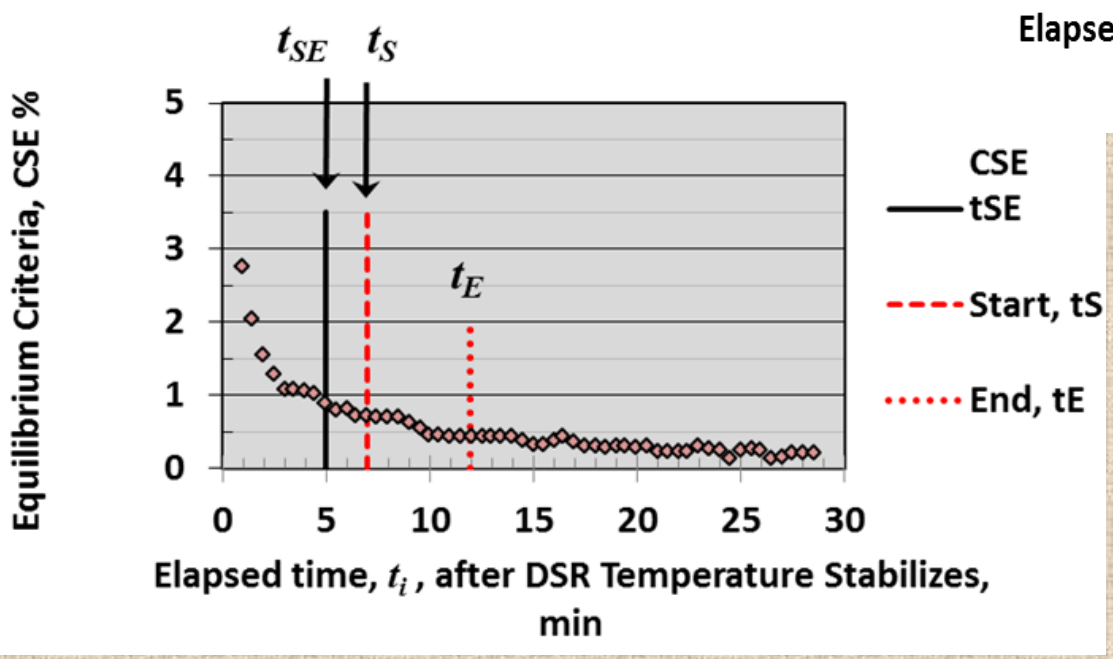
- Can we assume specimen properties and DSR mechanical properties are unchanging?
  - ✓ DSR is at equilibrium?
    - Transducer and motor properties unchanging?
    - DSR components are stable?
  - ✓ Binder properties are changing with time?
    - Measure in linear range
    - Steric and physical hardening is minimal
- G* is likely candidate to establish specimen equilibrium
  - ✓ Proposal: Monitor changes in G* with 30 min time sweep
  - ✓ Thermal equilibrium not only cause for changing G*!!!!

Thermal Equilibrium

t_{SE} is not well defined

Change may be large and continue for extended time period





Recommend test window

Extended isothermal measurements should be used with caution

Physical hardening????

4. Test Specimen Preparation

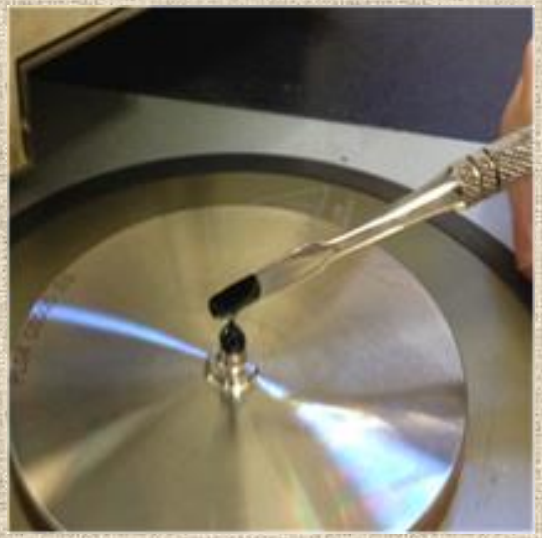
- Two protocols have been developed: WRI and MTE
- Primary differences
  - ✓ Placement of test sample
    - WRI - Hot place and heat gun
    - MTE – Preform oversize specimen in silicone mold using torch
  - ✓ Bulge formation
    - WRI at “soft” temperature
    - MTE at “hard” temperature

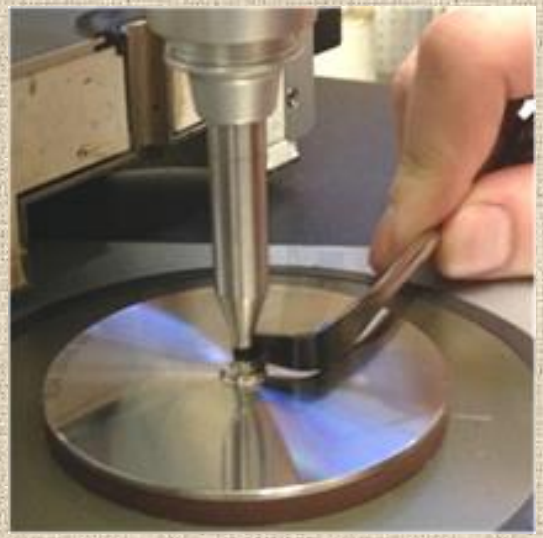
  } Ongoing controversy
- Are they equivalent?
  1. Do they both give acceptable adhesion? Answer - Yes
  2. Do they both accommodate physical hardening? Answer - Yes
  3. Are specimen thermal equilibrium times similar? Answer - Yes

WRI Protocol

- Using direct transfer of warm binder with spatula
  - ✓ Annealed sample with spatula, no preform in silicone mold
- Heat sample on spatula with heat gun to transfer to lower plate
  - ✓ Smear residue remaining on spatula on upper plate
- Loading and trim at 50°C - 60°C with 2 mm gap
- Closing Bulge at 30°C to 1.75 mm
- Cool to test temperature
  - ✓ Automatic adjust gap to control normal forces
  - ✓ Final gap will vary – calculate on actual gap

WRI Photographs



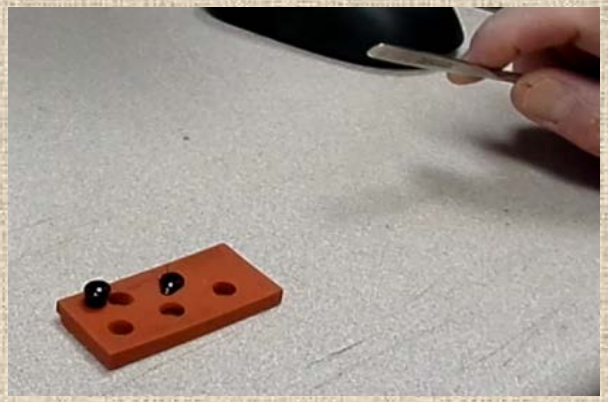


MTE Protocol

- Place sample on the end of warm spatula.
- Heat upper and lower plate with a small torch.
- Press specimen on the bottom plate so that it adheres to the bottom plate.
- Lower the upper plate so that it is embedded in the test specimen so gap is ≈ 3,000 µm, initial trim at ≈ 10°C.
- Reduce gap to ≈ 3,000 µm at ≈1°C for final trimming
- Close to final gap at ≈1°C
- Bring to test temperature

Note: Normal force is controlled during process of trimming and gap closure

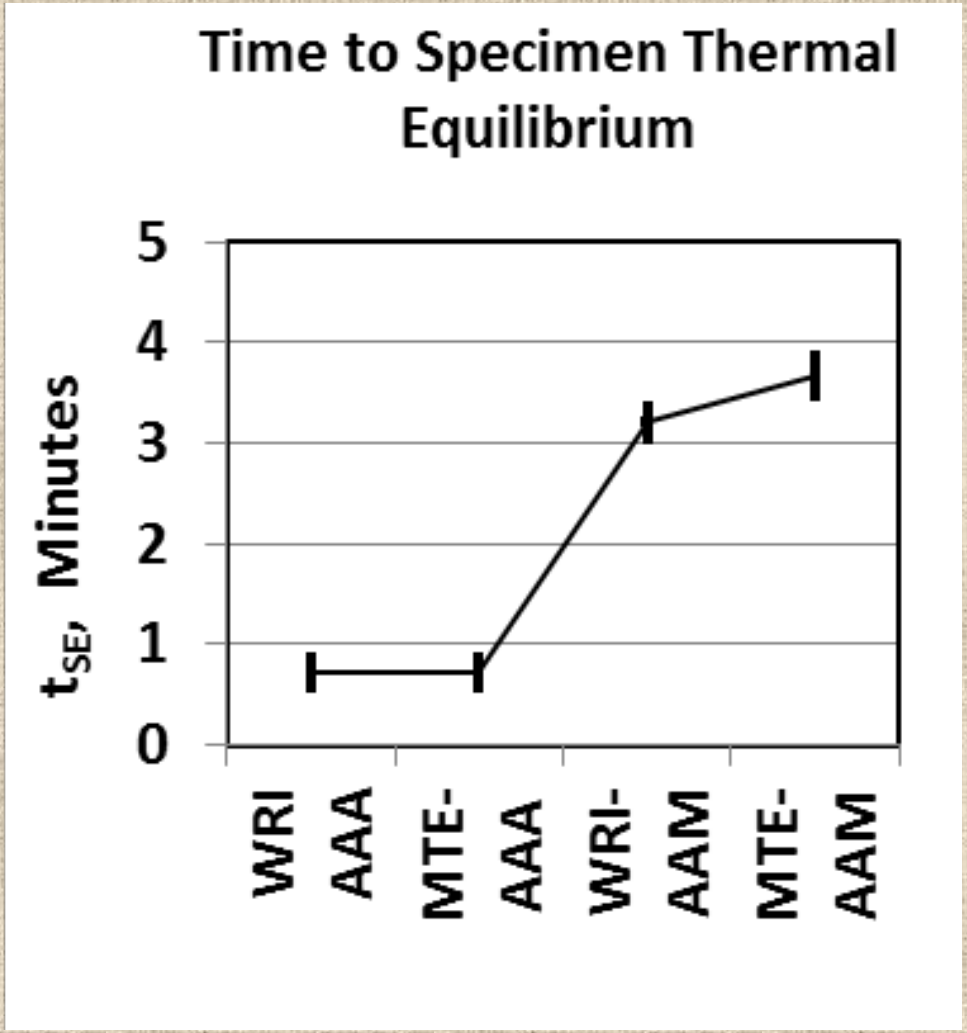
MTE - Photographs



Test Specimen Preparation Experiment

- Addressed *two* issues: Thermal equilibrium and specimen preparation
  - ✓ Five laboratories representing three DSR manufacturers
  - ✓ Two asphalt binders representing low and high degrees of physical hardening (AMRL AAA-1 and AAM-1)
  - ✓ Two sample preparation protocols (MTE and WRI)
  - ✓ Testing using thermal equilibrium protocol
- Binders PAV conditioned by TAI and sent in small tins to participants
- Returned data included complex modulus, phase angle, and normal force

Time for Specimen Thermal Equilibrium

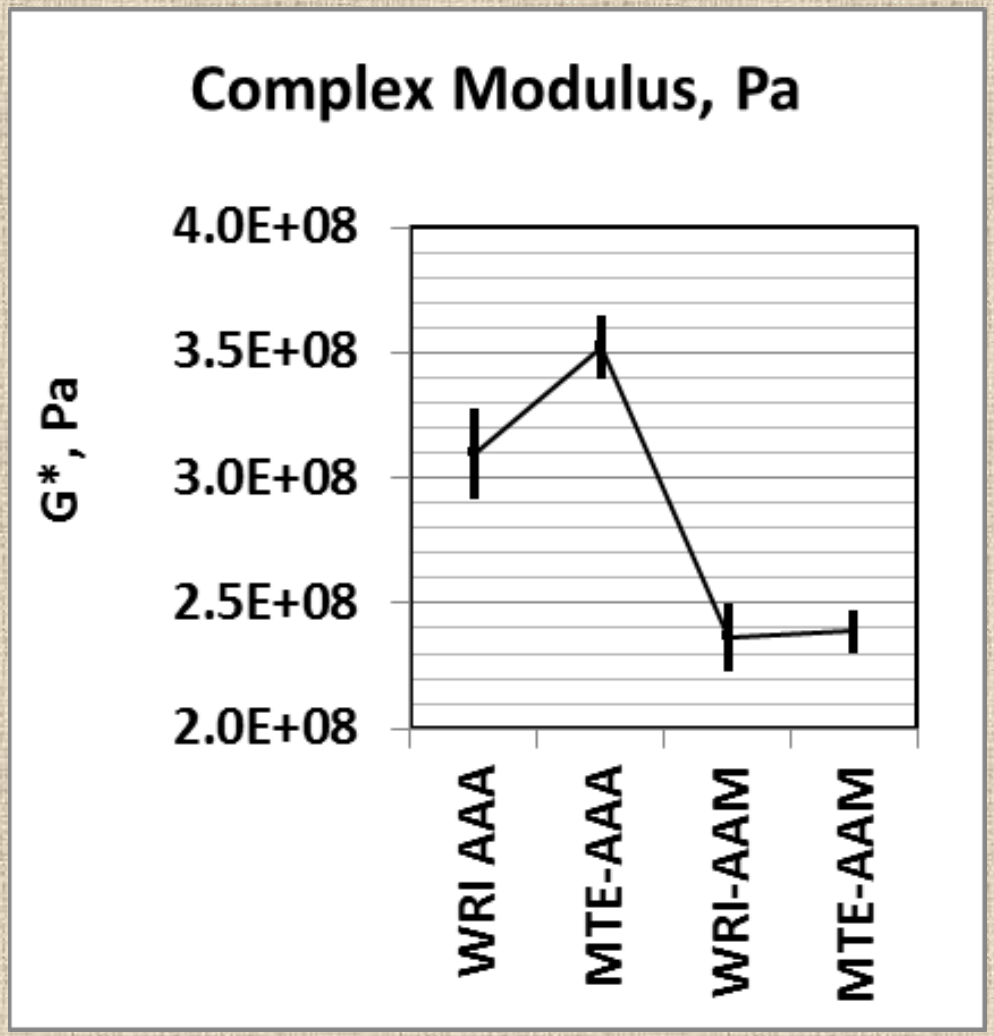


Procedure “works” at low temperature

Will have to account for physical hardening

Physical hardening affects test specimen thermal equilibrium time

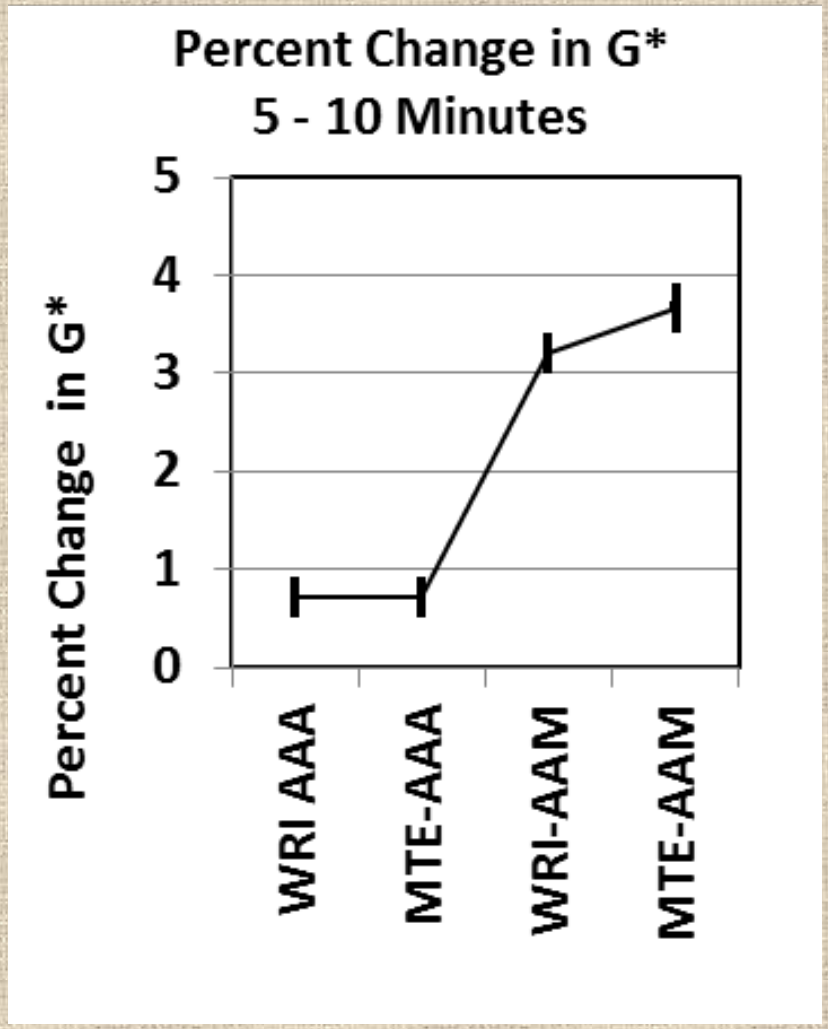
Complex Modulus, Laboratory A



- Procedure are close but not same
- Not enough data to recommend preferred procedure
- Decision depends on ultimate use of 4 mm

Change in G* - Between 5 and 10 Minutes after DSR Temperature = T_{TT}

- Reflection of physical hardening
- Time at isothermal temperature is important consideration



Summary

- Two protocols appear to give similar results
  - ✓ Draft protocol has been forwarded to ETG
- Equilibrium occurs rapidly – within few minutes
  - ✓ Time to equilibrium is affected by physical hardening
- Physical hardening is binder dependent as expected
  - ✓ Can be significant and is binder dependent
  - ✓ Test data interpretation must to account for physical hardening otherwise test variability may be unacceptable
  - ✓ Depending on purpose of testing, physical hardening may be an issue.

Issues remaining with test specimen preparation

- Specifying linear region
  - ✓ Broader than first expected
- Testing sequence
  - ✓ Increasing or decreasing temperature steps
  - ✓ Increasing or decreasing frequency
- Consideration of physical hardening
  - ✓ Test sequence?
  - ✓ Data correction by extrapolation to zero time?
- Ruggedness testing
- Round robin testing
  - ✓ Need supplier and user labs with proper training first!

What is next for Task Group?

- Recommendation continuation for short term
  - ✓ White papers with detailed data analysis
  - ✓ No additional laboratory work
    - Additional work needed is beyond a volunteer effort !!!!!!!
  - ✓ Been fun, BUT need financial resources to continue
- Ruggedness testing that includes rheometer design as variable
  - ✓ Needed before round robin and to refine procedures
- Training to establish corps of trained laboratories
  - ✓ 4-mm testing is a step up in testing capabilities
- Identify and recommend potential uses for 4-mm test results
  - ✓ Develop algorithms for incorporating procedure into specification testing
- Extend many of findings to 8-mm plate
  - ✓ Variability of 8-mm considered excessive by many