Asphalt Binder Testing Protocol for Dynamic Shear Rheometer

Presentation by Dave Anderson

Mike Farrar (Alec Cookman) - WRI

Gerry Reinke (Andrew Hanz) - MTE

Sonia Sterna (Codrin Daranga) - PTSI

Mike Anderson - TAI

Cassie Castorena - NCSU

Pavel Kriz - Imperial Oil

Olli-Ville Laukkanen – Allto University

Asphalt Binder ETG

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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

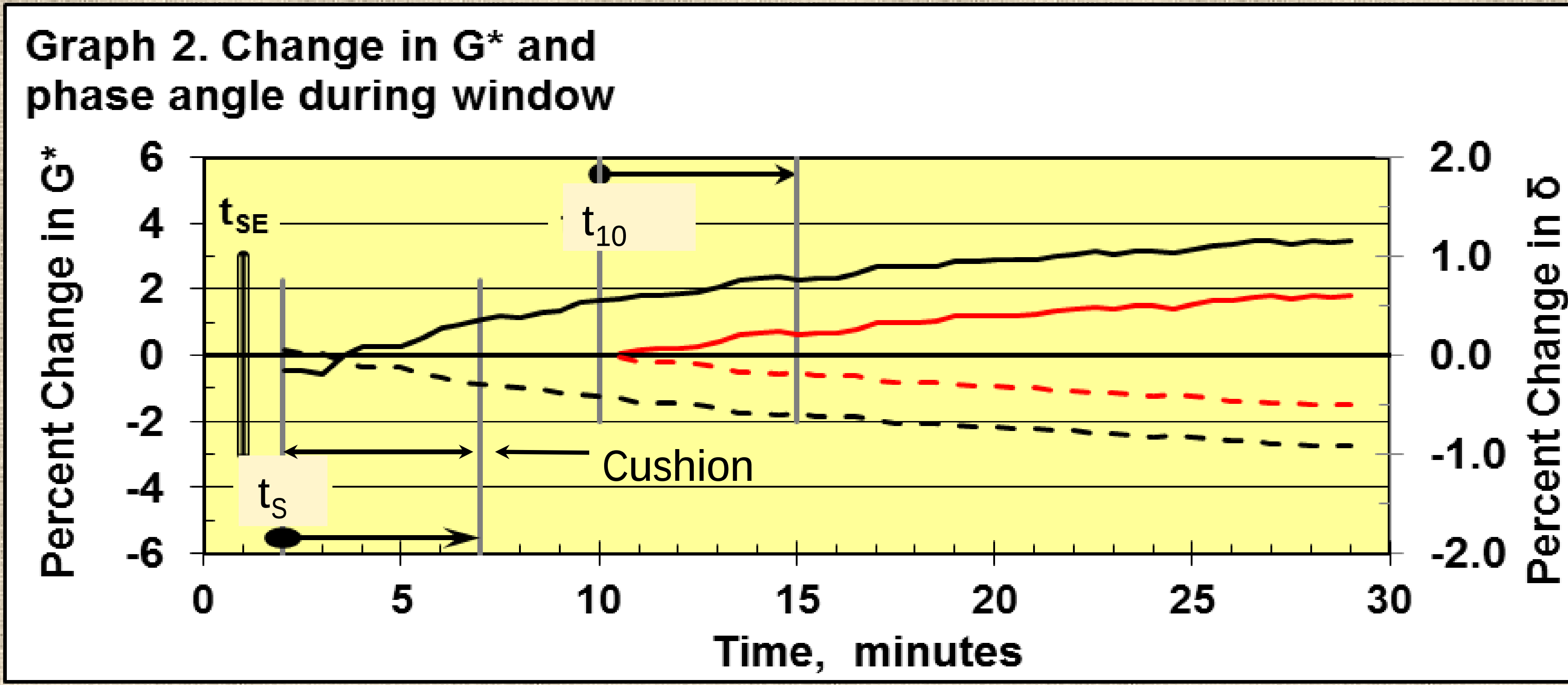
Early ETG Task Group on Thermal Equilibrium

- Initial concern was that 10 minute wait time was insufficient to obtain specimen thermal equilibrium
  - ✓ Test method did not include procedure for determining specimen thermal equilibrium
- Based on extensive series of tests recommended:
  - ✓ Change in G*with time was recommended as the criterion
  - ✓ Ten minute wait time is excessive
  - ✓ Wait time is instrument- specific
  - ✓ Test window should include both a “start” and “stop” time
- Expect adoption in ASTM and AASHTO DSR test method

Specifying specimen equilibrium

- Specimen equilibrium implies specimen mechanical properties are constant as long as can assume:
  - ✓ DSR is at thermal equilibrium – still may have gradients!
    - Transducer and motor properties unchanging
    - DSR components are stable
  - ✓ Binder properties are not 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

Definition of terms



t_{TT} – time zero, DSR indicates target temperature ±0.1°C

t_{SE} – time when specimen is at thermal equilibrium

t_C – cushion between equilibrium and start time

t_S , t_E – time at start an end of test window

t_{10} – 10 minute wait time as per AASHTO 315, ASTM 7125

Recommendation for determining 25 and 8 mm specimen equilibrium, t_{SE}

- Monitor G* during 30 minute isothermal time sweep
  - ✓ Determine G* at 30 second intervals – 61 data points
  - ✓ Calculate C_{SE} – average <u>absolute</u> deviation for 5 data points as percent of the average of the 5 data points
  - ✓ Moving average, calculate for 61 - 4 = 59 data points
  - ✓ Plot C_{SE} vs time
- Thermal equilibrium time t_{SE} obtained when $C_{SE} \leq 1\%$
  - ✓ 1% must be maintained for remainder of 30 minutes
- Start time is time required for specimen thermal equilibrium plus a cushion, t_C
  - ✓ Five minute test window starts at $t_S = t_{SE} + t_C$

4mm Plate - Historical

- Pioneer work performed by Mike Farrar, WRI and Gerry Reinke, Mathy
  - ✓ Currently being used by a number of researchers but without any standardization
- Promising protocol with many applications
  - ✓ Facilitates implementation of revised aging protocols
  - ✓ Useful for asphalt emulsion work and recovered binders
  - ✓ Potential replacement for BBR
  - ✓ Master curve generation at low temperatures
- For all of the above applications revisions to AASHTO T315, ASTM D7125 will be necessary

4mm Task Group Objectives

- 4-mm is a different “ball game”
  - ✓ FHWA Binder ETG Task Group established to provide guidance for the development of 4 mm geometry as a tool for purchase specification testing
  - ✓ *Focus of task is on test method development and standardization to facilitate implementation*
- Future work beyond scope of task group must include
  - ✓ Ruggedness testing
  - ✓ Technology transfer to ramp up the learning curve
  - ✓ Recommendations for a round robin program
  - ✓ Extending findings to 8 mm

1. 4-mm Issues - Verification

- 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

1. 4-mm Issues – Verification, cont'd

- Verification of machine compliance
  - ✓ Several procedures available (WRI, MTE, etc.)
  - ✓ Two methods recommended by task force
    - Method A uses ice to bond top and bottom plates
    - Method B uses "crazy glue" to bond top and bottom plates
  - ✓ Objective is to determine DSR response when plates are held rigid
  - ✓ ASTM task force established to refine and validate equivalency of two methods
- Temperature and compliance critical verification steps

2. 4-mm Issues - Specimen preparation

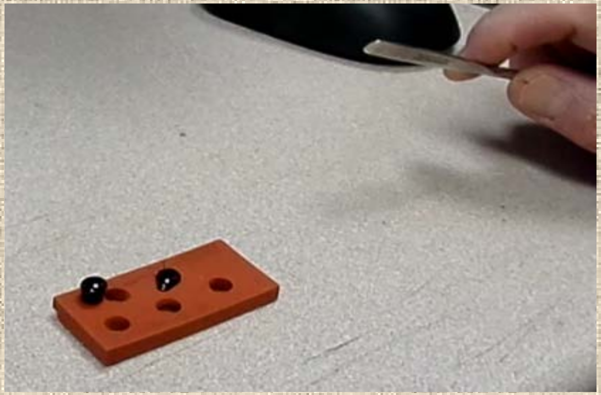
- Two protocols have been developed: WRI and MTE
- Primary differences
  - ✓ Placement of test sample
    - WRI - Hot place and heat gun
    - MTE – Preform oversize specimen in silicone mold using torch
  - ✓ Bulge formation
    - WRI at “soft” temperature
    - MTE at “hard” temperature
- Are they equivalent?
  - ✓ Do they both give acceptable adhesion?
  - ✓ Do they both accommodate physical hardening?
  - ✓ Are specimen thermal equilibrium times similar?

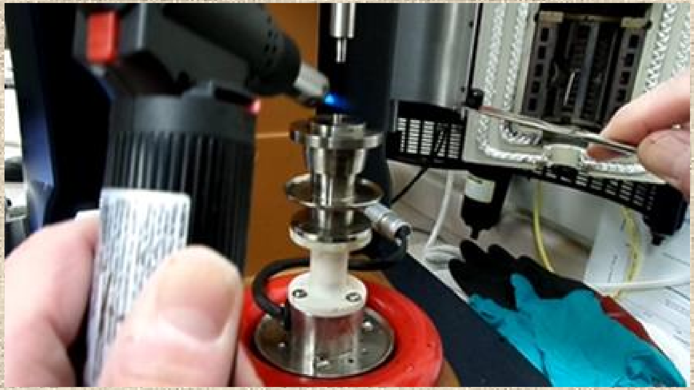
MTE Protocol

- 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

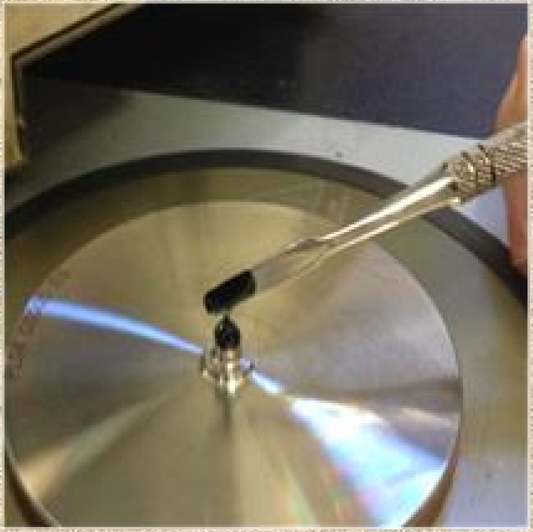


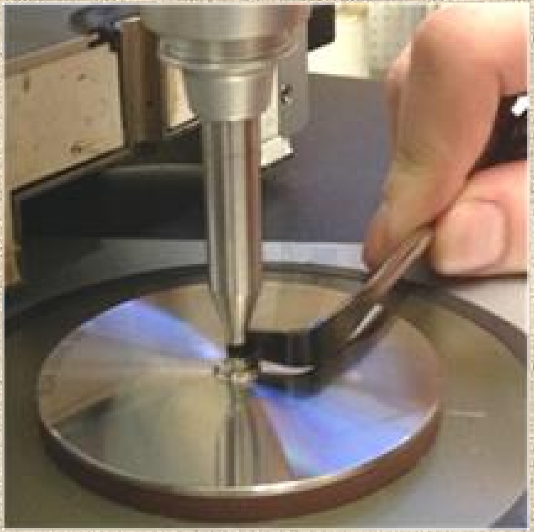


WRI Protocol

- 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





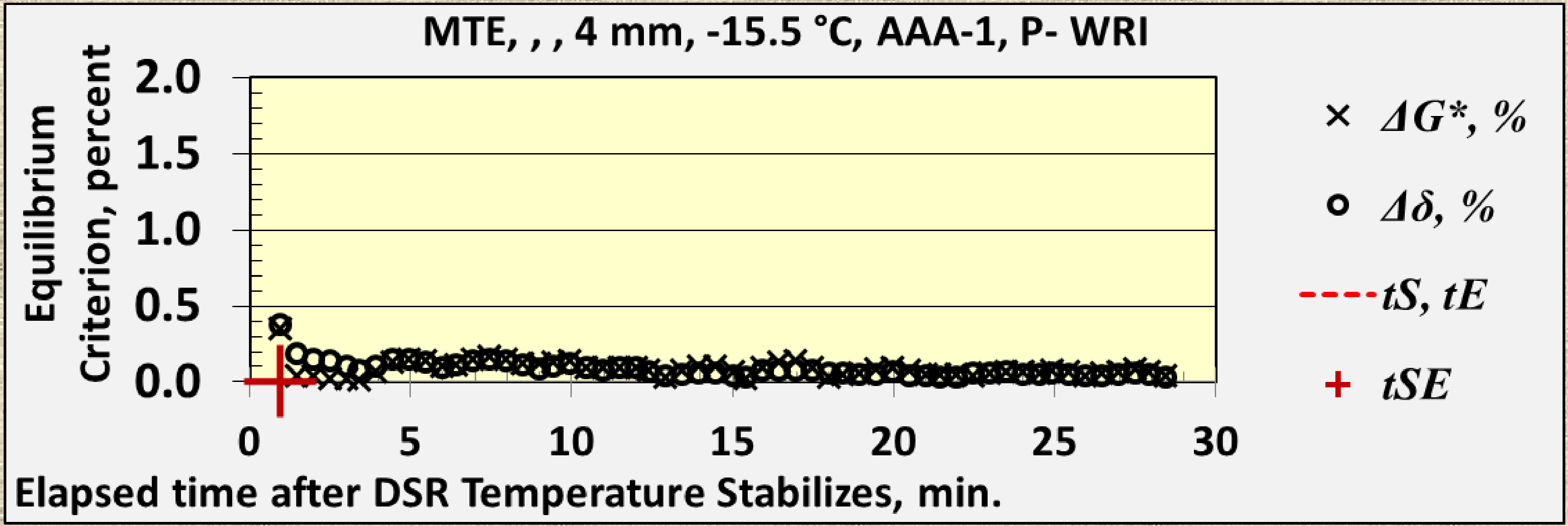
3. 4-mm Issues - Thermal Equilibrium

- Is the procedure established for the 8 and 25 mm plate valid for low temperature measurements with the 4 mm plate?
  - ✓ If the procedure is valid what are the criteria?
  - ✓ Should there be a “start-end” testing window?
  - ✓ Is physical hardening a factor in establishing thermal equilibrium?

Task Group Experiment

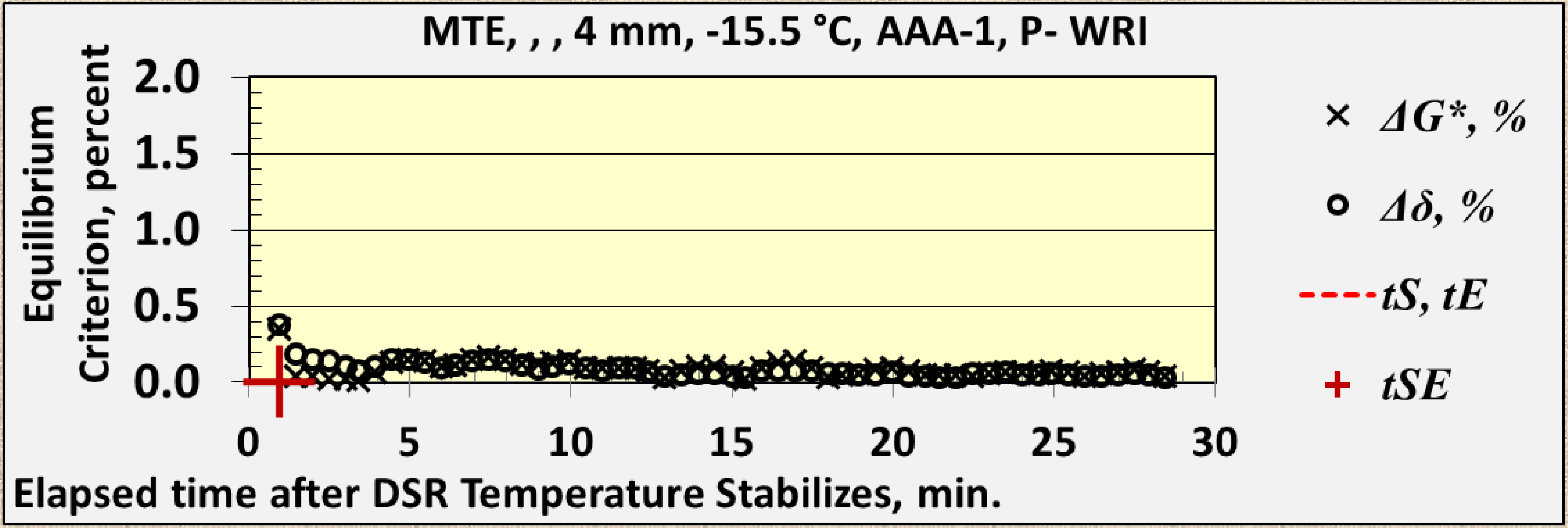
- Addresses *two* issues: Thermal equilibrium and specimen preparation
  - ✓ Five laboratories representing three DSR manufacturers
  - ✓ Two asphalt binders representing low and high degrees of physical hardening (AMRL AAA-1 and AAM-1)
  - ✓ Two sample preparation protocols (MTE and WRI)
  - ✓ Testing 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

Typical Result – Criterion vs. Time



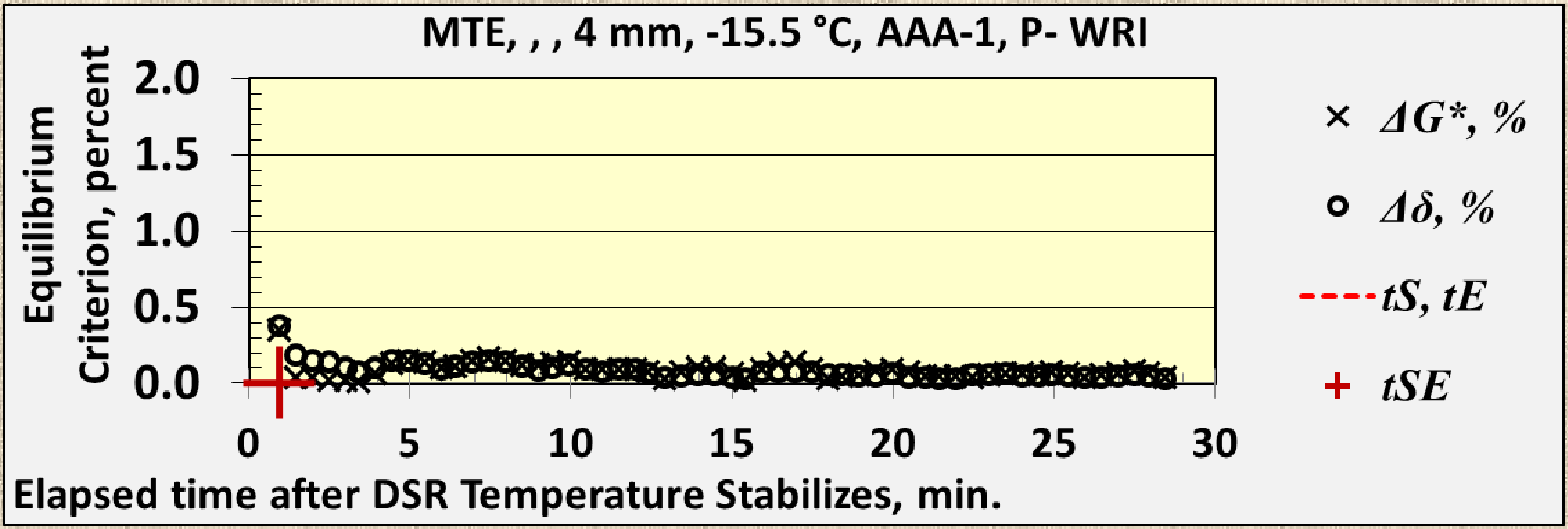
- Specimen equilibrium is reached quickly
  - ✓ More rapid than expected
  - ✓ Attributable to small specimen size?
- As with 8 and 25 mm plate 10 minute wait excessive

G* vs. δ as Criterion



- Give equivalent results
  - ✓ Phase angle tends to be less noisy
  - ✓ G* used for 8 and 28 mm
  - ✓ Recommend G* at 1% change

Typical Result - G* vs. δ as Criterion



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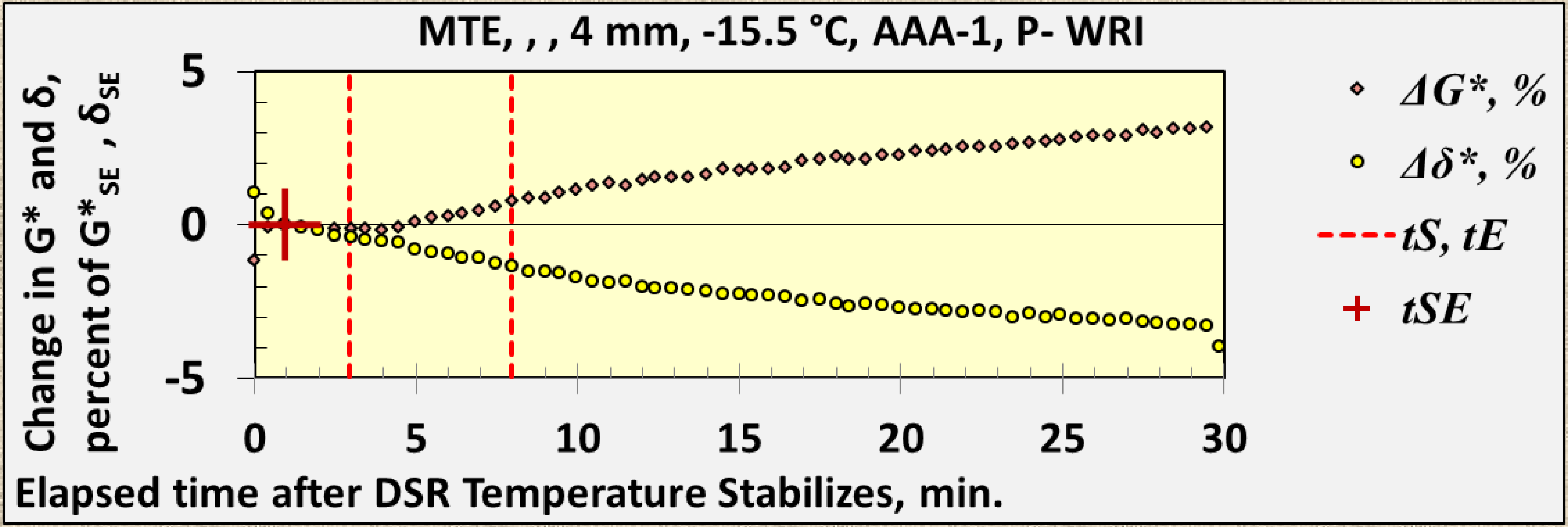
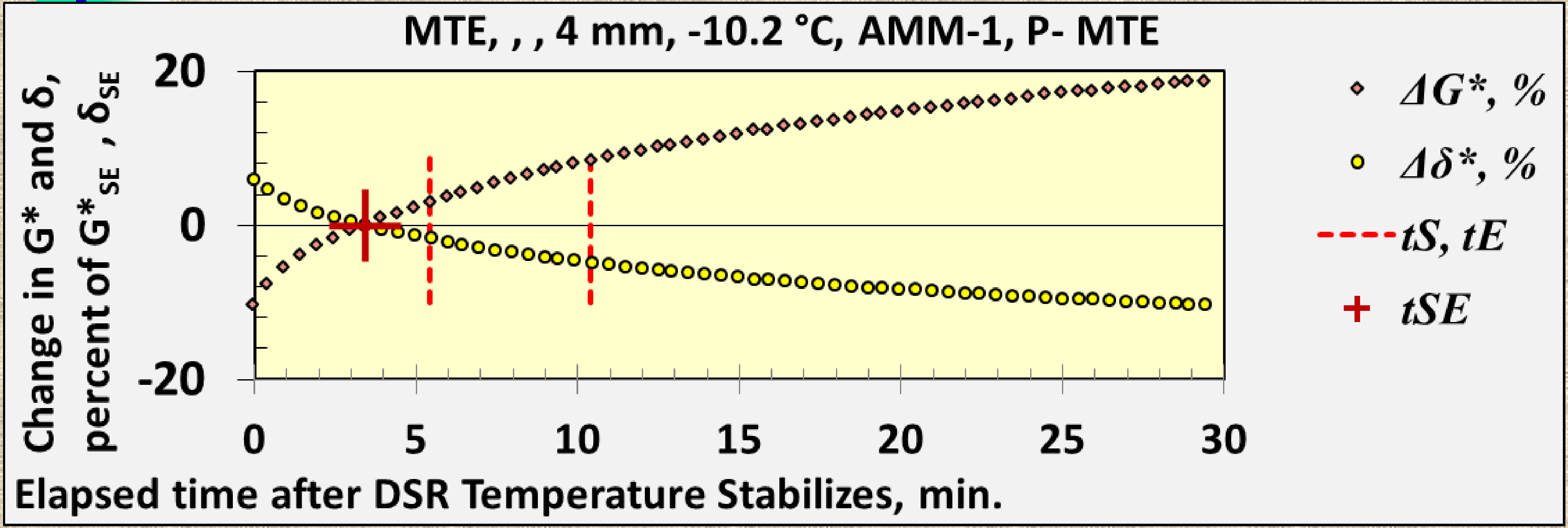
Percent Change in G* and δ with time, AAA-1



- Small change within test window
  - ✓ Protocol appears to be acceptable
  - ✓ Physical hardening minimal as expected with AAA-1

Percent Change in G* and δ with time, AAM-1



- Larger change within test window
  - ✓ Physical hardening causes 20% change in 20 minutes
  - ✓ Need to account for physical hardening in some manner

Summary

- Two protocols appear to give similar results
  - ✓ Draft protocol is available for general distribution
- Equilibrium occurs rapidly – within few minutes
  - ✓ Time to equilibrium is not an issue
- Physical hardening is binder dependent as expected
  - ✓ Can be significant/Binder dependent
  - ✓ Need to develop test protocols that account for physical hardening
  - ✓ If unaccounted for test variability may be unacceptable
- Depending on purpose of testing, physical hardening may be an issue.

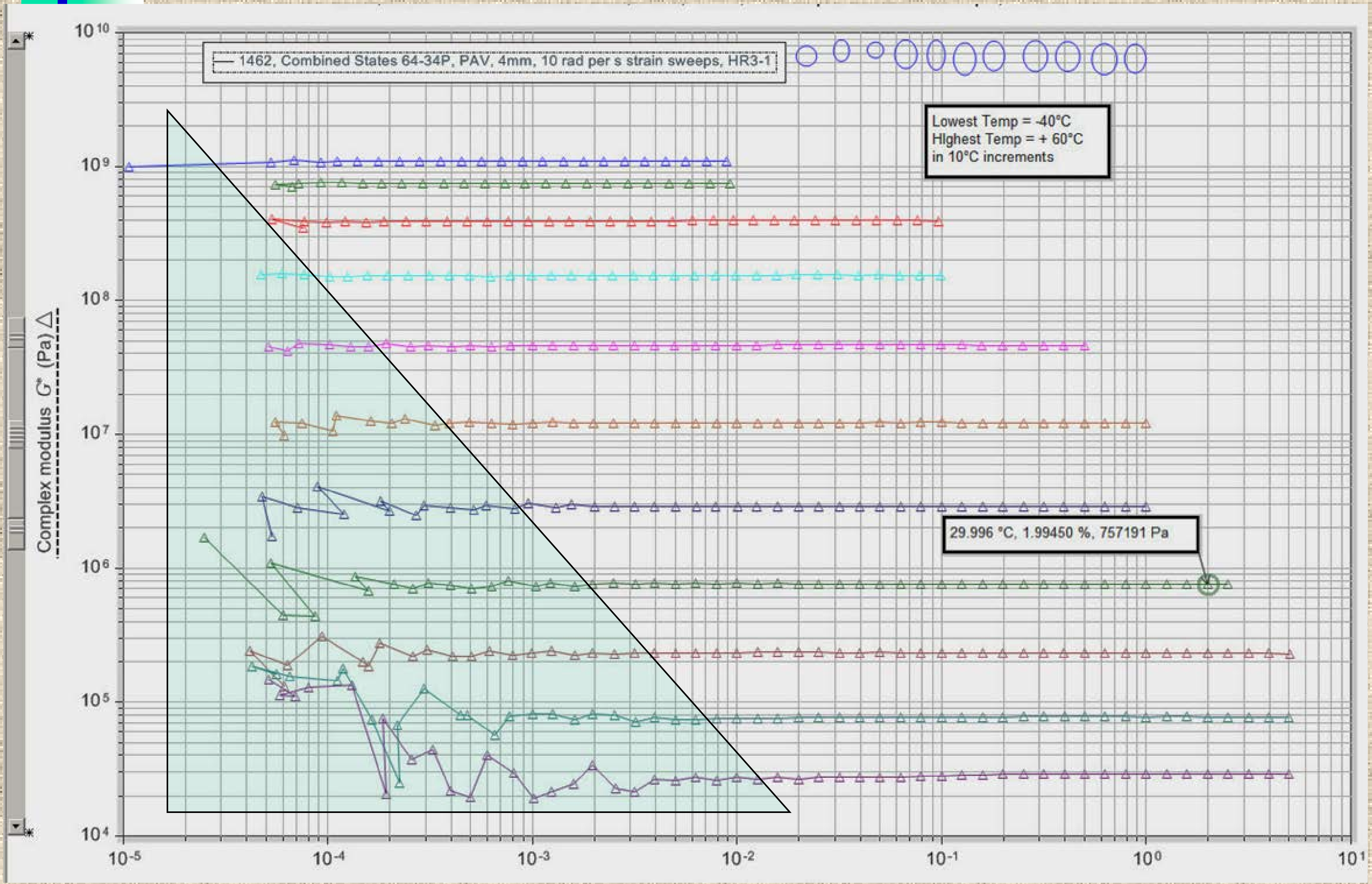
5. Issues Remaining

- 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!

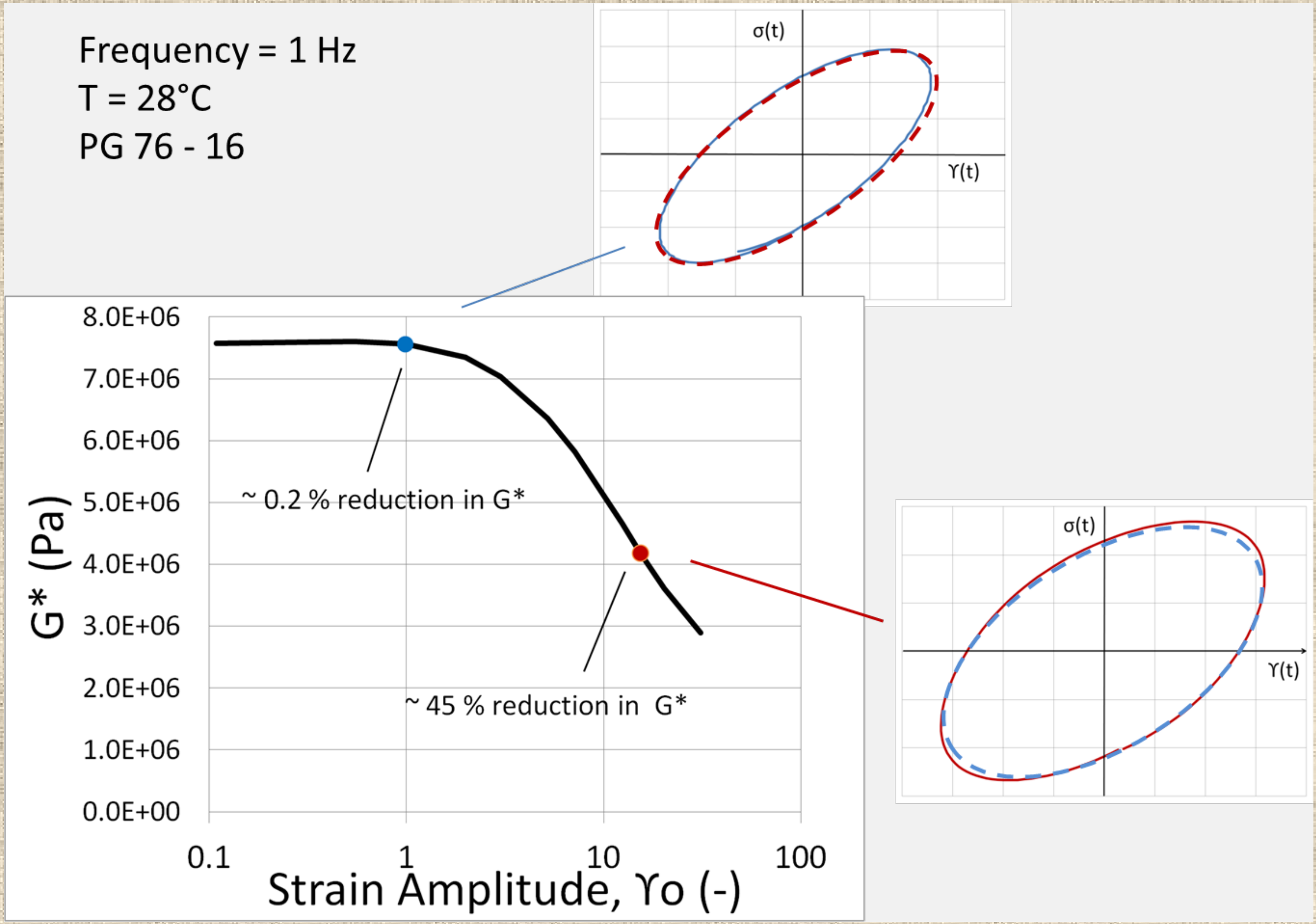
Current Status

- All test data for main experiment is complete
  - ✓ Data mining essentially complete
- Data have been organized into manageable database
- Data analysis underway

Example strain sweeps to show linearity

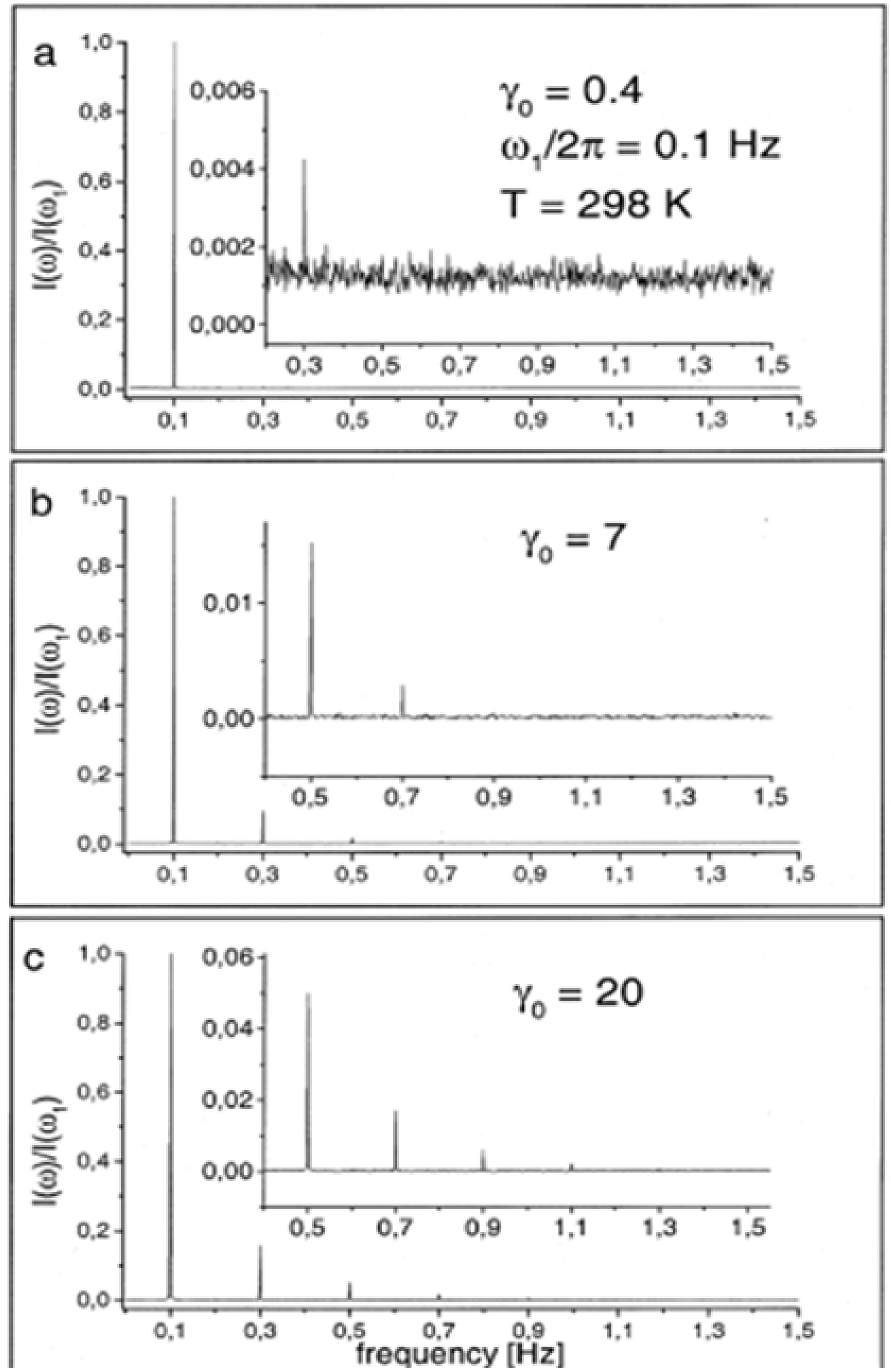


Lissajous Figures for data integrity



Harmonic Analysis

- Manfred Wilhelm
  - ✓ Analysis of harmonics
  - ✓ Used ratio of 1st and 3rd to validate data integrity
  - ✓ Patented analysis???



Wilhelm, M., Macromolecular Materials and Engineering 2002, 287, No. 2

Status to date – some findings

- Machine compliance protocols available
  - ✓ Methods considered tentative until evaluated in ruggedness testing
- Two sample preparation protocols established
  - ✓ Available on request
- 25 and 8 mm thermal equilibrium methodology is appropriate for 4 mm at low temperatures
  - ✓ Specimen thermal equilibrium occurs rapidly
  - ✓ Physical hardening present with both methods
- Both specimen preparation procedures produce acceptable test specimens

What do we need for full implementation?

- ❑ Recommended protocols for specimen preparation and determining specimen thermal equilibrium (Done)
- ❑ Protocol for determining machine compliance (TBD)
- ❑ Ruggedness testing program (TBD)
  - ✓ Expect to include rheometers from 3 manufacturers
  - ✓ Somewhat more robust than typical ruggedness program
- ❑ Training so that have sufficient labs for round robin (TBD)
  - ✓ Needed before round robin to develop sufficient number of laboratories for robust round robin
- ❑ Round robin (TBD)