Thermal Equilibrium and Test Protocol for 8mm and 4mm DSR Measurements

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FHWA Asphalt Binder ETG
Fall River, MA
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Expanded Working Group

- WRI - Mike Farrar
- MTE - Andrew Hanz
- NCSU - Cassie Castorena
- PTSI - Sonia Serna
- BSA - Kriz Pavel
- Rheometer Manufacturers – Malvern, Anton-Paar, TA
- Future
  - ✓ CDOT – Ed Trujillo
  - ✓ FHWA –
- Others?

Task force scope

- To provide guidance for the development of 4 mm PP geometry as a tool for specification testing
  - ✓ *Test method development and refinement*
  - ✓ Ruggedness testing
  - ✓ Identifying path for and facilitating technology transfer
  - ✓ Recommendations for a round robin program
  - ✓ Extending findings to 8 mm PP
- Scope does not include protocols for using test data
  - ✓ Acceptance and material specification requirements based on 4 mm PP beyond our scope
  - ✓ Executing RR beyond our scope and resources

Task Force - Specific Work Elements

- Step 1: Develop recommended testing protocol based on limited laboratory testing
  - ✓ Instrument standardization
  - ✓ Specimen preparation
  - ✓ Specimen conditioning – thermal equilibrium and physical hardening
  - ✓ Verification of data integrity
  - ✓ Provide rationale for protocol based on test results

Task Force work plan

- *Step 1: Develop testing protocol that is appropriate for routine use and that provides data of acceptable accuracy and precision(repeatability*)
  - ✓ Prepare for ruggedness testing
  - ✓ 8 and 4 mm PP geometry
- Step 2: Conduct ruggedness testing
  - ✓ More robust than typical ruggedness rest
  - ✓ Include more than one laboratory
- Step 3: Conduct round robin
  - ✓ Only when have sufficient number of laboratories on-line
  - ✓ “Technology transfer” part of task force mission

Potential Uses of 4-mm Test

- Use by producer for QC?
  - ✓ Current protocols are acceptable
  - ✓ But - Qualify results
  - ✓ Comparative use only
- Calculated parameter for specification use?
  - ✓ Primary focus
- Mastercurve or model manipulation?
- Point values for specification use?

1. Issues – Verification/Standardization

- Torque Transducer
  - ✓ Verify with reference fluid at ambient temperature
  - ✓ Current practice using 25 mm plate at ambient temperature covers needed torque range
- Angular displacement transducer
  - ✓ Not performed in user laboratory
- Temperature transducer
  - ✓ 25 mm diameter wafer (thermistor/platinum film)
  - ✓ Questionable for 8 mm PP, Unacceptable for 4 mm PP
- Machine compliance
  - ✓ Instrument and fixture specific – Assign to DSR mfg.

2. Issues - Specimen preparation

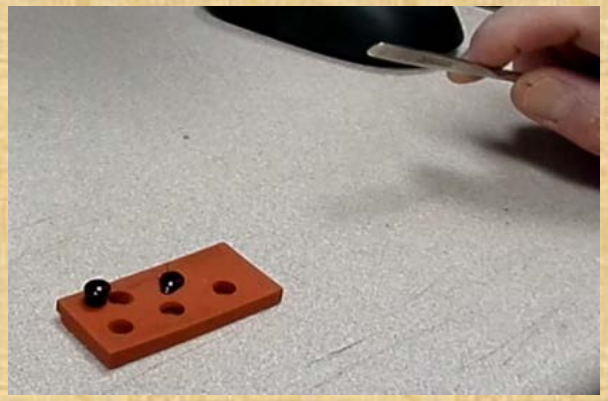
- Two protocols: WRI and MTE
- Primary differences
  - ✓ Placement of test sample
    - WRI - Hot place and heat gun
    - MTE – Preform and torch
  - ✓ Bulge formation
    - WRI at “soft” temperature
    - MTE at “hard” temperature
- Are they equivalent?
  - ✓ Both give acceptable adhesion
  - ✓ Measured values are not the same

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

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

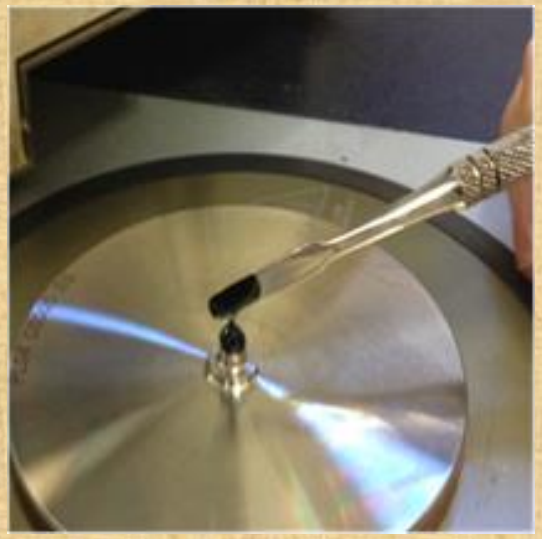
MTE - Photographs

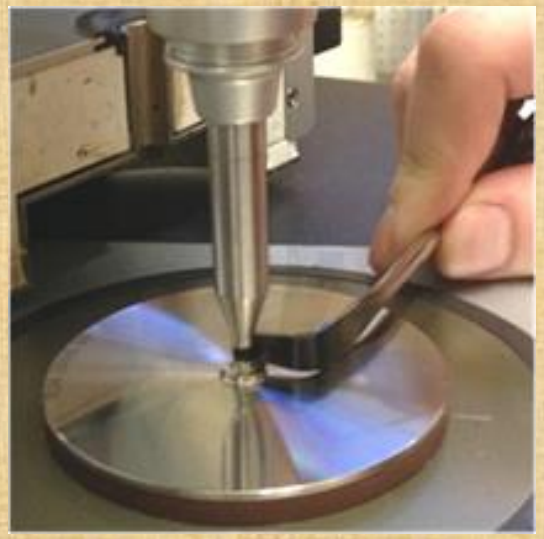


WRI Protocol

- Using direct transfer of warm binder with spatula
  - ✓ Scoop annealed sample with spatula, no 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



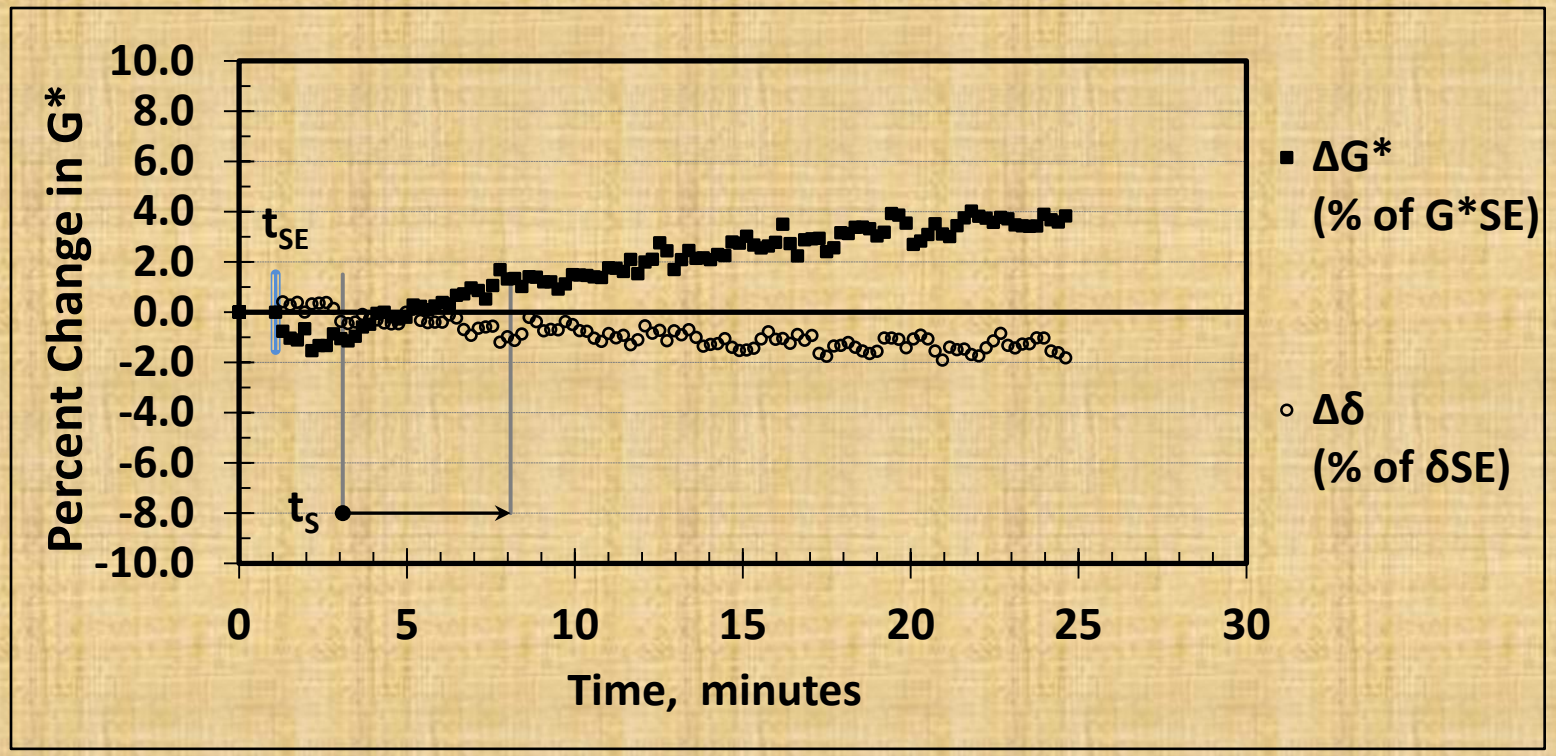


Sample Preparation - Status

- Two procedures give acceptable adhesion
  - ✓ Tentative
  - ✓ Subject to additional evaluation
- Both methods are ready to release as provisional procedures
  - ✓ Need feedback from users
  - ✓ Recommend distribution
- Caveat
  - ✓ Measured values may not be same with two methods
  - ✓ Use with caution
- Available in Specification format

Protocol for establishing wait time

New procedure added to AASHTO 315
Monitor G* vs. time
Constant G* → Specimen thermal equilibrium



WRI Protocol – G* vs. time

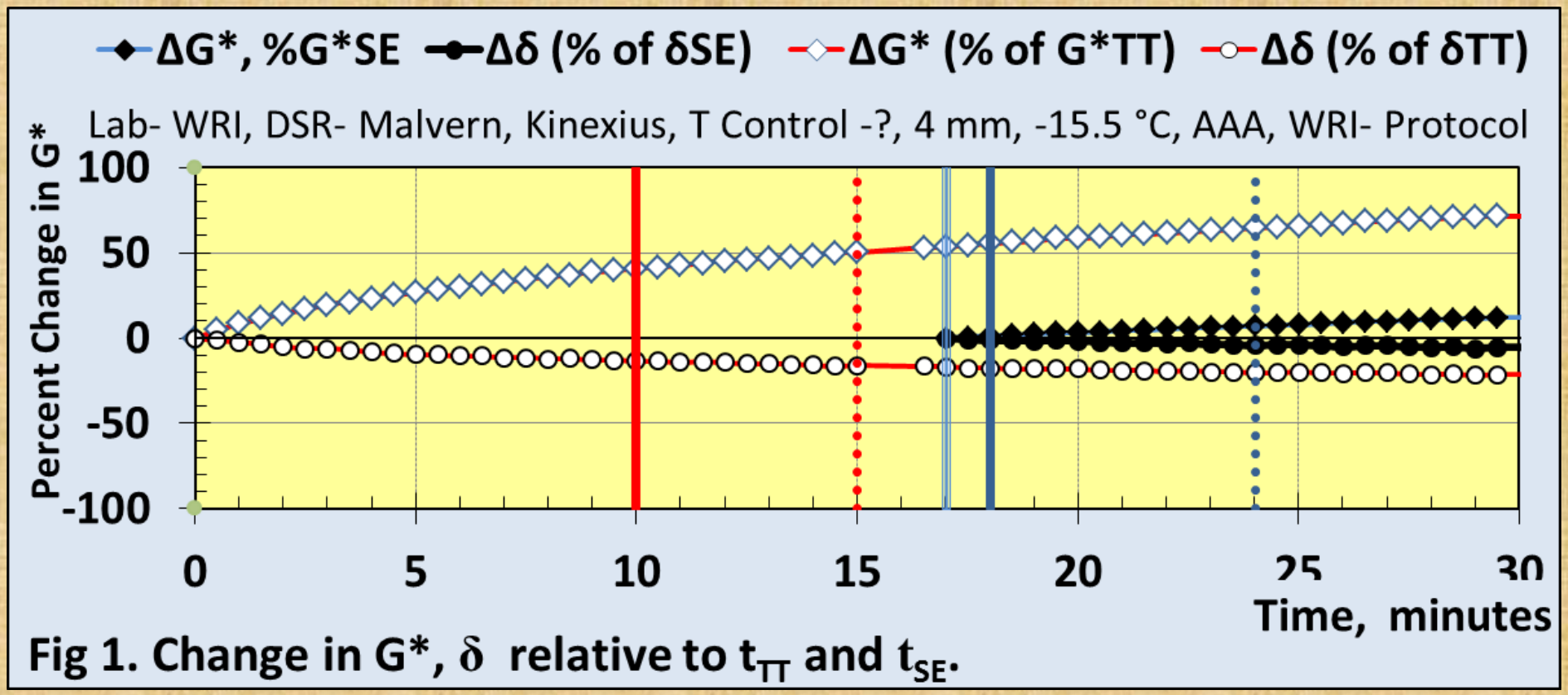


Fig 1. Change in G*, δ relative to t_{TT} and t_{SE}.

WRI Protocol – G* vs. log time

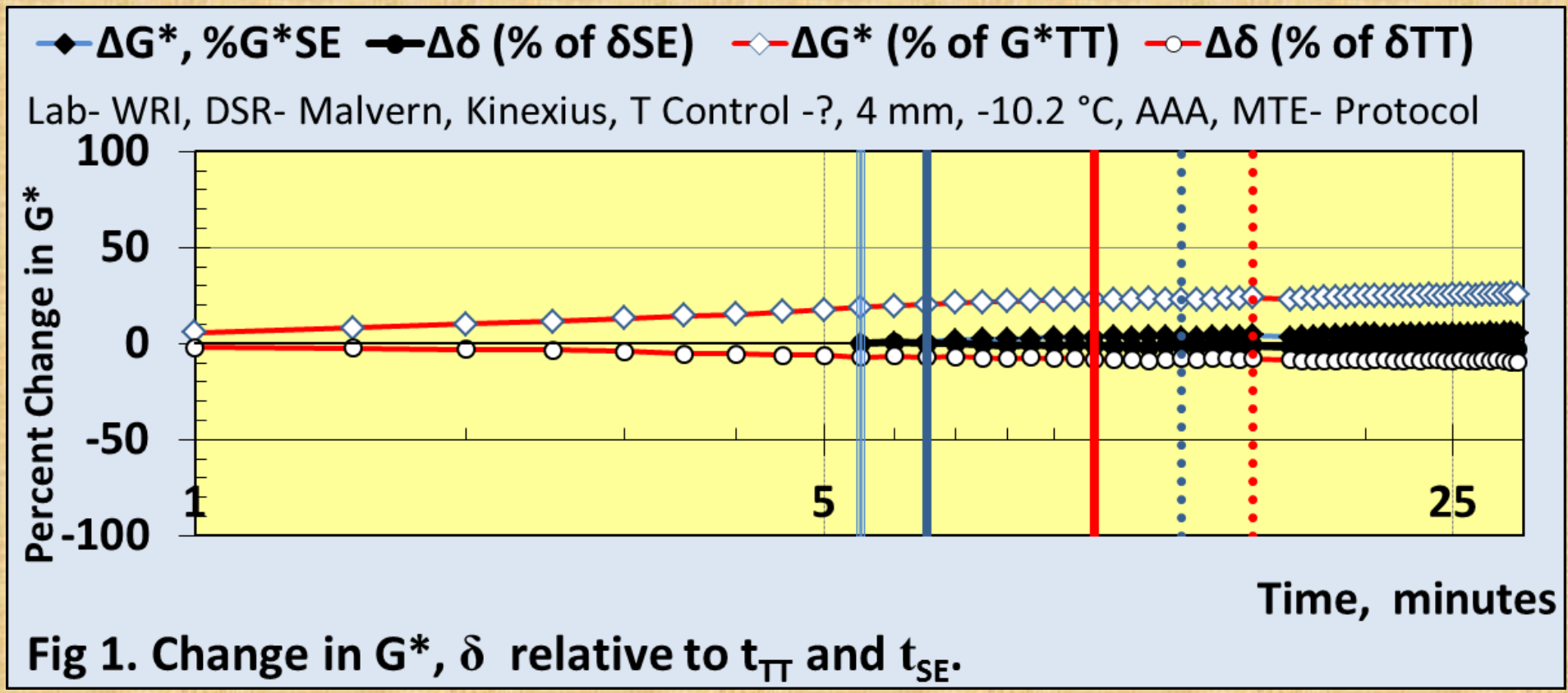


Fig 1. Change in G*, δ relative to t_{TT} and t_{SE}.

WRI Protocol – R vs. time

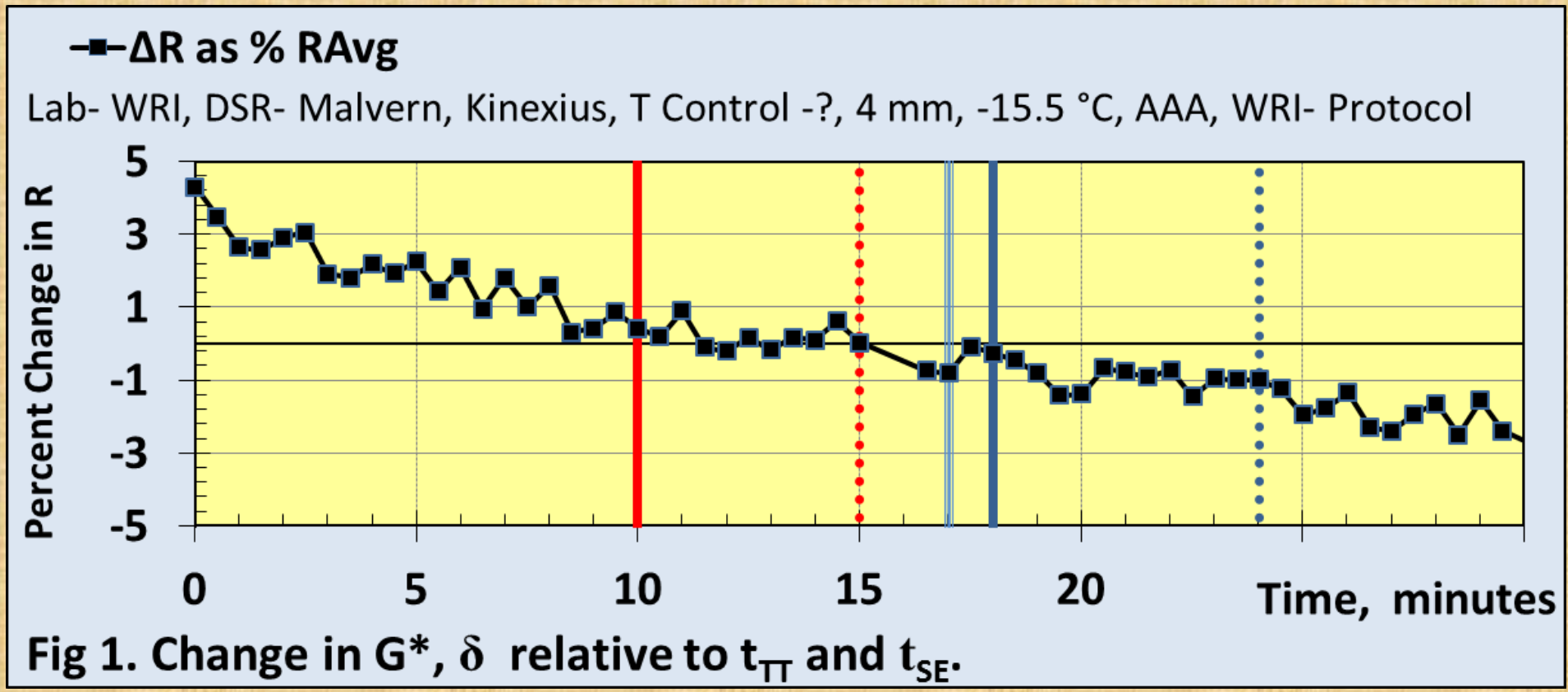


Fig 1. Change in G*, δ relative to t_{TT} and t_{SE}.

MTE Protocol – G* vs. time

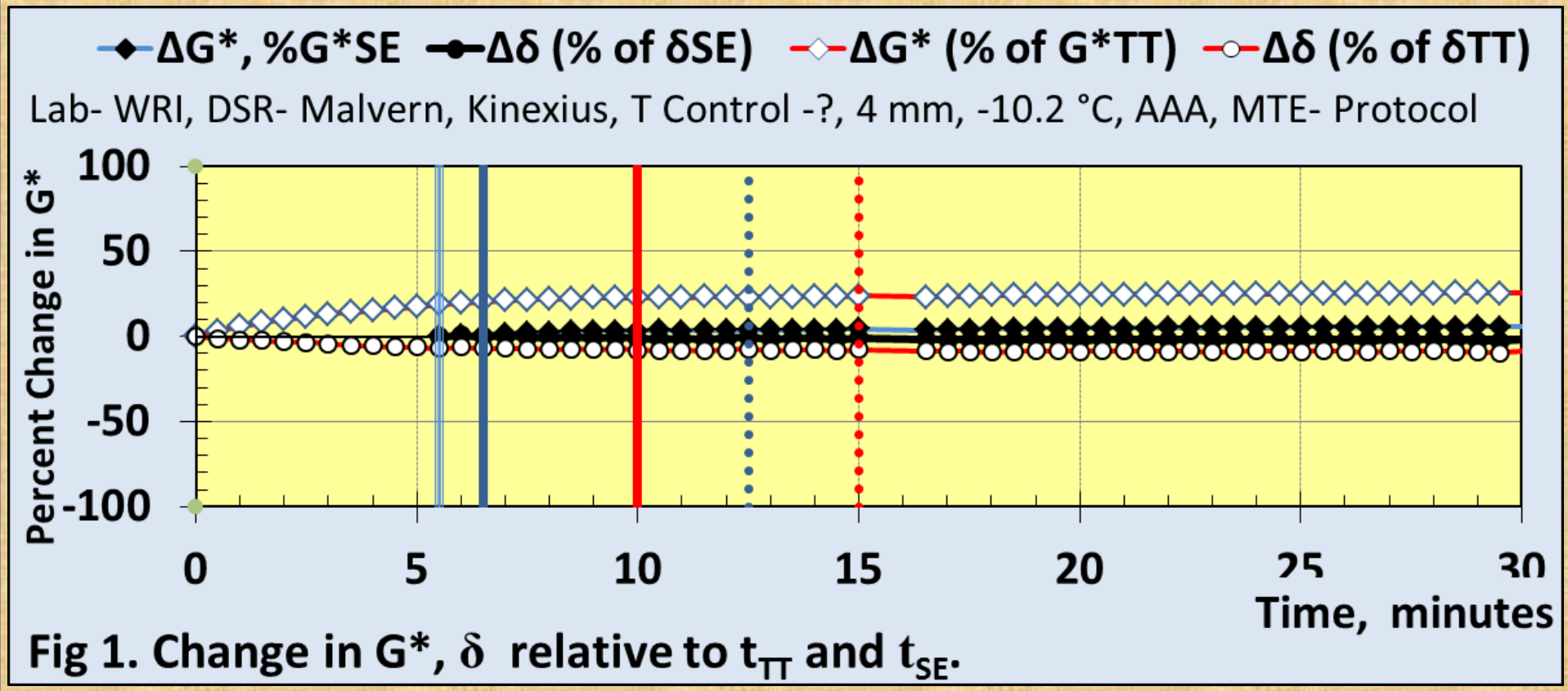
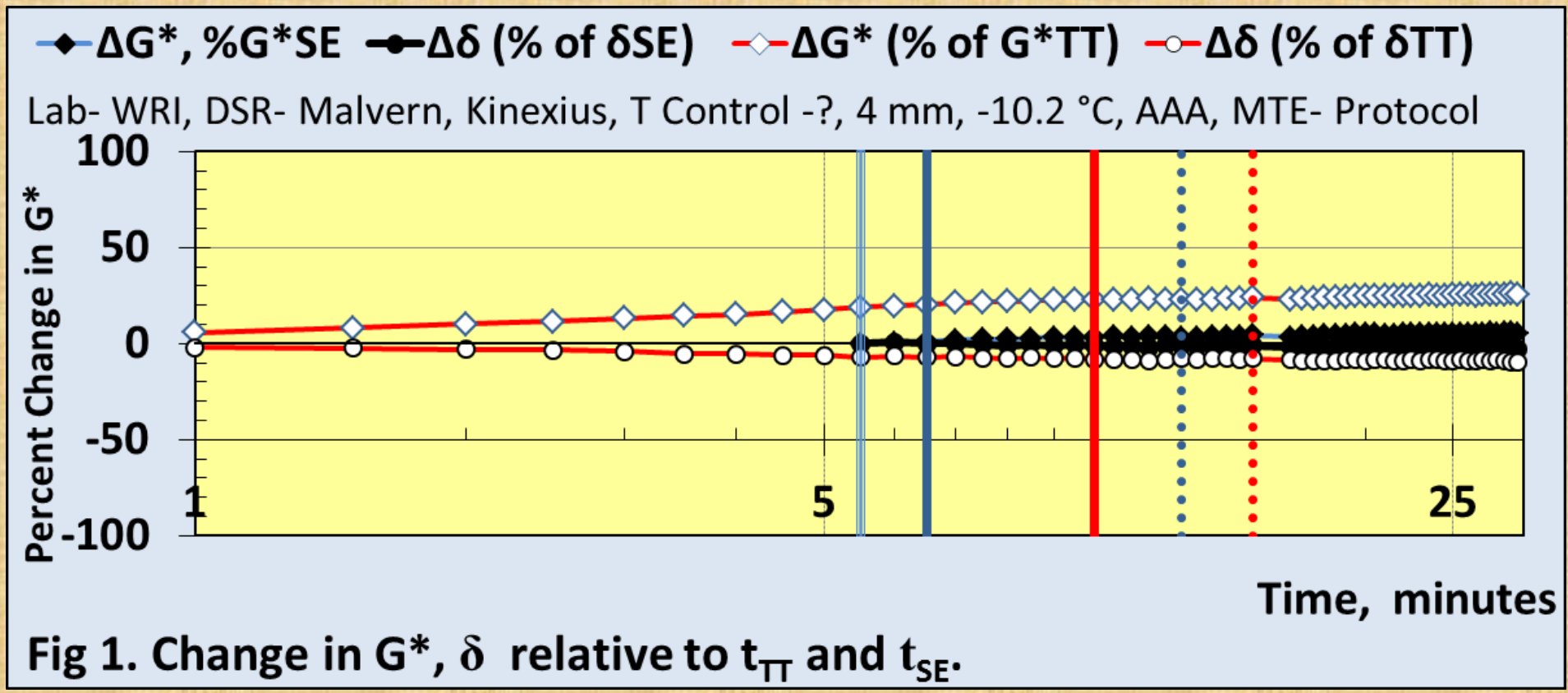


Fig 1. Change in G*, δ relative to t_{TT} and t_{SE}.

MTE Protocol – G* vs. log time



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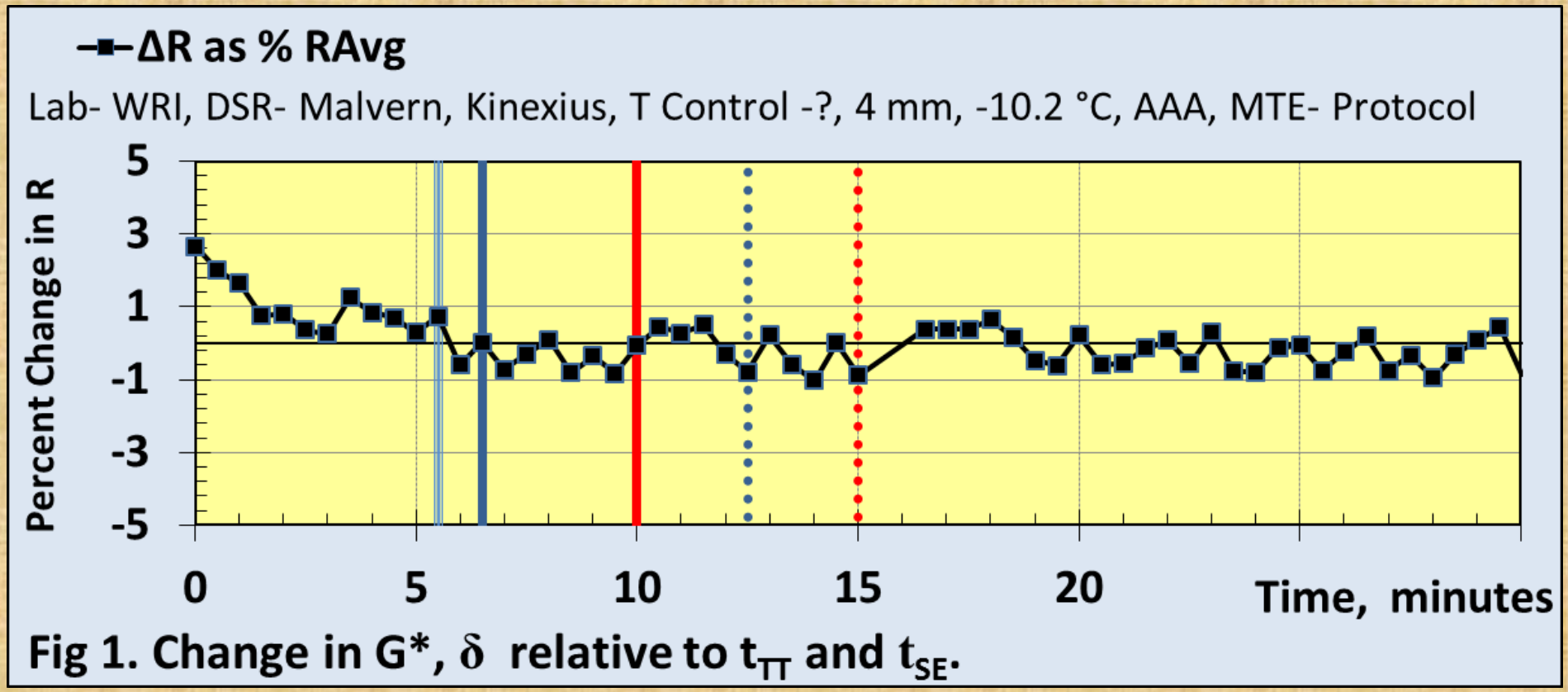


Fig 1. Change in G*, δ relative to t_{TT} and t_{SE}.

Percent Change with time (WRI Data)

Measurement	MTE	WRI	% Difference
G^*_{TT}	1.72E+08	1.56E+08	10.3
G^*_{SE}	2.04E+08	2.39E+08	-14.6
G^*_{S}	2.07E+08	2.42E+08	-14.7
G^*_{E}	2.11E+08	2.56E+08	-17.5
G^*_{10}	2.11E+08	2.19E+08	-3.8
G^*_{15}	2.12E+08	2.34E+08	-9.5

PTSI Results (Average 2 Tests)

Measured values versus time

Binder	AAA	AAA	AMM	AMM
Protocol	WRI	MTE	WRI	MTE
G*SE	5.2E+07	1.7E+08	4.0E+07	1.4E+08
G*S	2.9E+08	3.4E+08	2.2E+08	2.5E+08
G*E	2.9E+08	3.4E+08	2.2E+08	2.5E+08
G*10	3.0E+08	3.5E+08	2.3E+08	2.7E+08
G*15	2.7E+08	3.5E+08	2.1E+08	2.6E+08

PTSI Results (Average 2 Tests)

Percent change relative to T_{TT}

Binder	AAA	AAA	AAM	AMM
Protocol	WRI	MTE	MTE	WRI
G*SE	465	572	395	326
G*S	2	21	-10	-23
G*E	4	23	-7	-20
G*10	-9	16	-14	-29
G*15	-9	16	-14	-29

How are test results different?

- Vary with binder
  - ✓ Expected, Physical hardening known to be greater for AAM
- Vary with protocol, WRI vs MTE
  - ✓ Unexpected
  - ✓ MTE gives less physical hardening
- Varies with DSR
  - ✓ Unexpected

Why are they different?

- We live in a three dimensional world and asphalt binders take time to respond to our commands!!!!
- Some thoughts
  - ✓ Poisson's ratio not 0.50?
    - Literature suggests Poisson's ratio ≠ 0.50
    - Affects normal stresses
  - ✓ Stresses not fully relaxed
    - Probably true for both methods
  - ✓ Normal stresses not sufficient to suppress physical hardening
- Need some "out of the box" thinking, more analysis

3. Issues – Thermal Equilibrium

- Wait time before starting test and test window
  - ✓ Need to establish time increment to reach specimen thermal equilibrium once DSR reaches thermal equilibrium
  - ✓ Above increment plus “cushion” = wait time
- Protocol established for 8 and 25 mm does not work
  - Physical hardening swamps G* thermal stability
  - Considered with BBR and needs to be resolved for PP
  - Appears to be rheometer –specific
  - Being evaluated as part of wait time considerations
- Again – need to think “out of the box”

Conclusions to date

- Sample preparation protocol established
  - ✓ Ready for distribution as draft with caveats
- Two protocols result in significantly different test values
  - ✓ Physical hardening is different between two protocols
- Physical hardening effects differ with two protocols
- Methodology/script for generating real time data established
  - ✓ Need to acquire and analyze

4. Issues - Testing Sequence (TBD)

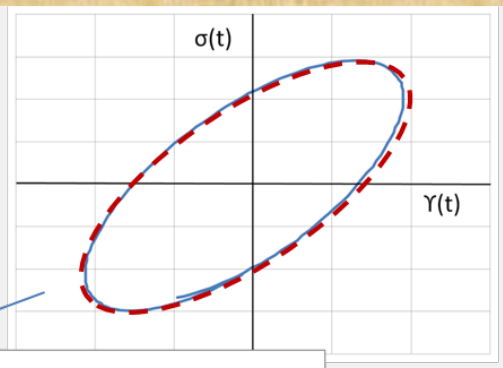
- Temperature sequencing
  - ✓ Cool to highest test temperature in test sequence, decrease temperature to lower temperatures
  - ✓ Cool to lowest test temperature in test sequence, increase temperature to higher temperatures
  - ✓ Two sequences yield different test data, data quality
- Issue needs to be resolved in order to release test method
  - ✓ Recognized at higher temperatures

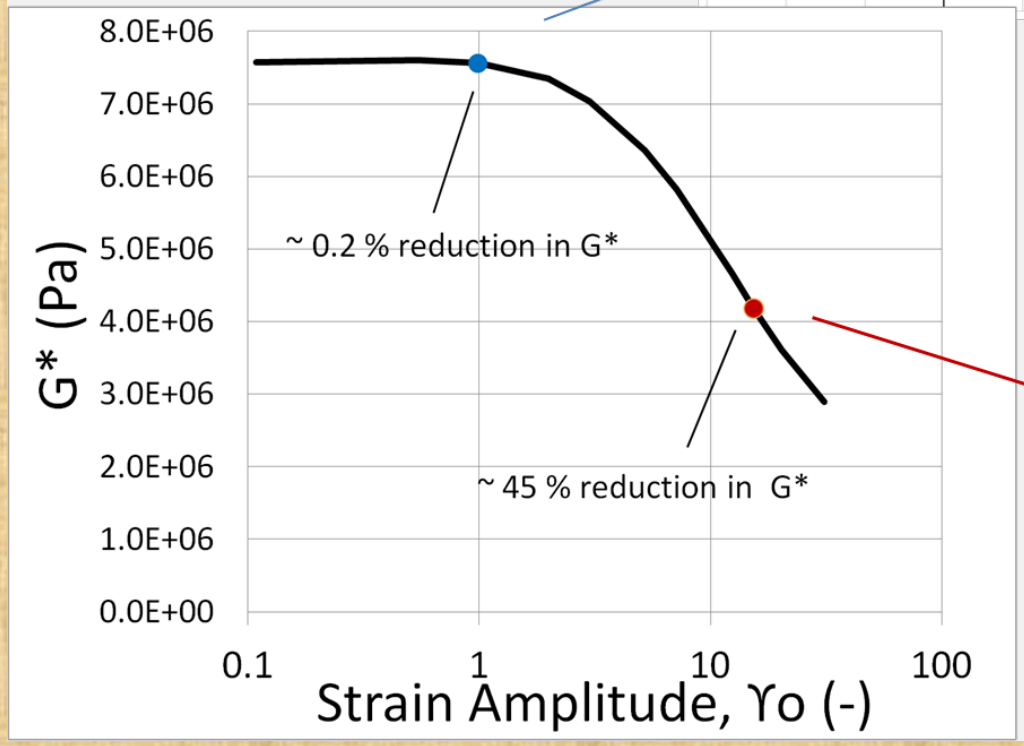
5. Issues: Verifying data integrity (TBD)

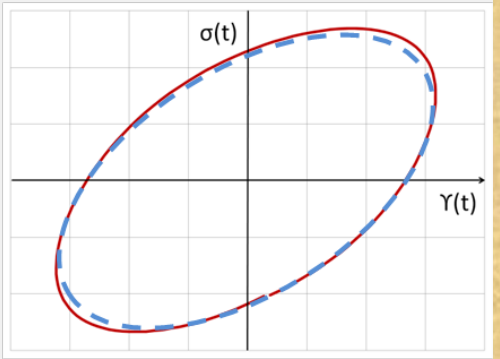
- Fall-off in G* with strain in strain sweep
- Lissajous Figures in isothermal test with varying frequency
- Odd harmonics in isothermal test with varying frequency
- Not looking at Black Space or mastercurve construction at this point
  - ✓ Subject for later follow-on studies

Lissajous Figures

Frequency = 1 Hz
T = 28°C
PG 76 - 16

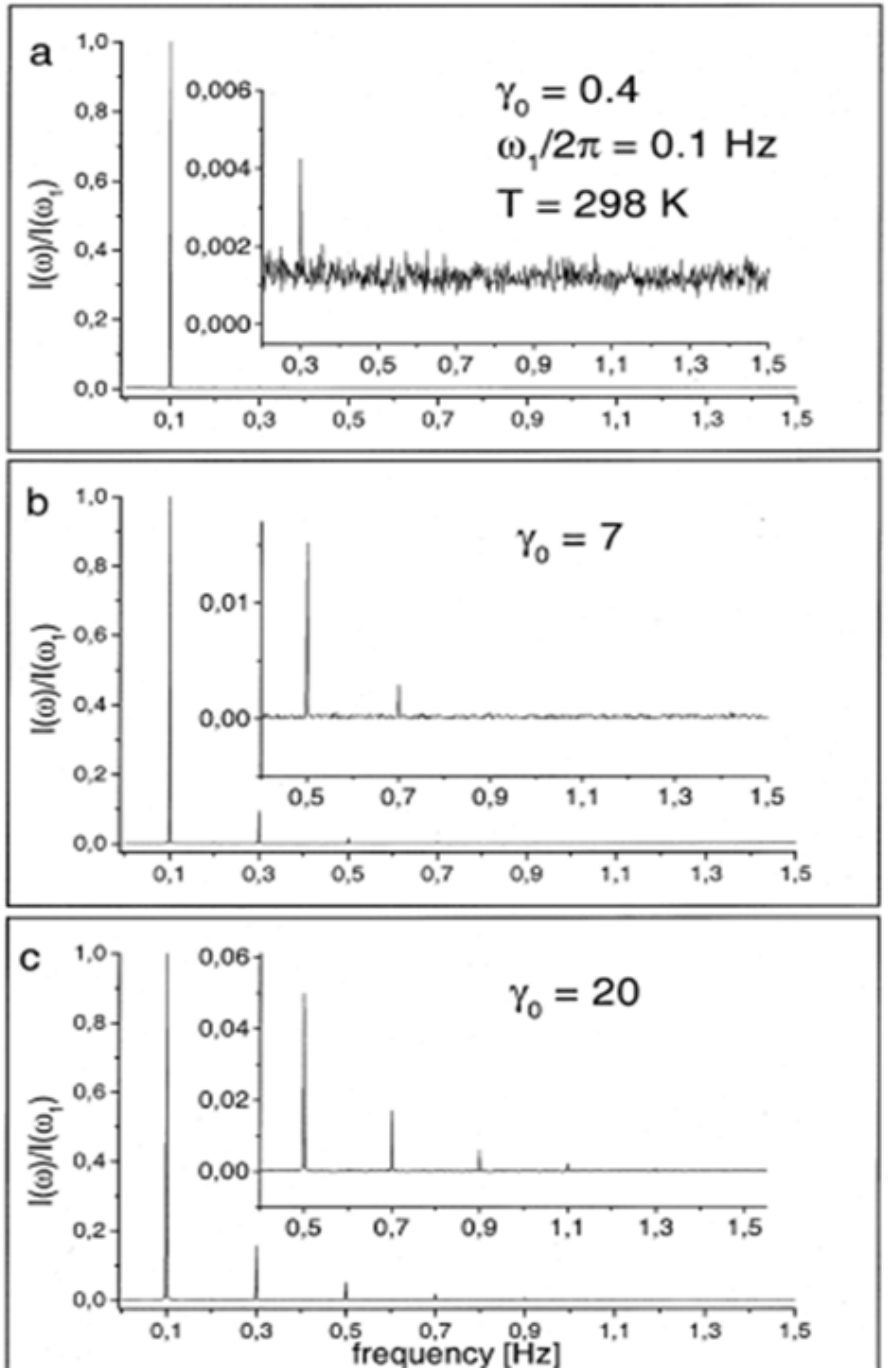






Harmonic Analysis

- 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

Summary – Needed Results

- *Recommended sample preparation protocol developed*
  - ✓ *Testing protocols in specification format*
  - ✓ *Equipment requirements established*
  - ✓ *Surprising results!!!! (PLEASE TAKE AS TENTATIVE!!!)*
- Ruggedness testing program (TBD)
  - ✓ Expect to include rheometers from 3 manufacturers
  - ✓ Somewhat more robust than typical ruggedness program
- Recommendations for training (TBD)
  - ✓ Needed before round robin to develop sufficient number of laboratories for robust round robin
- Round robin recommendations (TBD)