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

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Background

- High stiffness rheological measurements with 4 mm parallel plate geometry (PP) is a recent concept
  - ✓ Championed initially by WRI, picked up by others
- Attractive as result of
  - ✓ General availability of more sophisticated rheometers
  - ✓ Improvements in analytical tools that facilitate more sophisticated data analysis
  - ✓ Increased interest in basic rheology as a routine tool for specifying and characterizing asphalt
- Adoption of 4 mm PP for specification use will require an extension of the current DSR test method

Historical – Rheological measurements SHRP era

- Mid to Upper Temperature – DSR PP
  - ✓ Early DSR's in common use had were less robust and lacked normal force capability
  - ✓ ASTM D7175/AASHTO T315 limited measurements to range where 100 Pa > G* < 100 MPa
  - ✓ Parallel plate geometry limited by machine compliance and resolution
- Lower Temperature - BBR and DTT
  - ✓ BBR 30 MPa to 1 GPa
  - ✓ DSR torsion bar research tool, not practical for spec use
  - ✓ DTT to characterize brittle, brittle-ductile failure

Recommendations from SHRP 370

- G* < 1 kPa
  - 50-mm PP
- 1.0 kPa < G* < 100 kPa
  - 25-mm PP with a 1-mm gap
- 0.1 MPa < G* < 30 MPa
  - 8-mm PP with a 2-mm
- G* > 30 MPa
  - Bending beam rheometer
  - Torsion bar geometry when

Rheological measurements - Capability

1. Upper temperature - 25 mm PP: OK
2. Intermediate temperature - 8 mm PP: repeatability issue
3. Lower temperature – BBR: OK

Property	d2s, % *	
	Within	Between
DSR, 25 mm, Tank	6.4	17.0
DSR, 25 mm, Original	9.0	22.0
DSR, 8 mm, PAV	13.8	40.2
BBR, S(60), PAV	7.2	17.8
BBR, m(60), PAV	2.9	6.8

* Difference at which 2 test results are suspect

4. Capabilities of 4 mm PP geometry are unknown

Task force scope

- To provide guidance for the development of 4 mm PP geometry as a tool for specification testing
  - ✓ Test 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 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

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
- Step 1 result:
  - ✓ Prepare for formal ruggedness testing
  - ✓ Define minimum requirements for suitable rheometersl

Step 1: Specific issues to be addressed

- Issues grouped by category
  1. Instrument verification-standardization
  2. Specimen preparation
  3. Conditioning prior to testing
  4. Testing sequence
  5. Linearity region
  6. Data quality
- Resolution of above issues depends on use of data
  - ✓ Point values for specification use?
  - ✓ Calculated parameter for specification use?
  - ✓ Use by producer for QC?
  - ✓ Mastercurve or model manipulation?

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

2. Issues - Specimen preparation

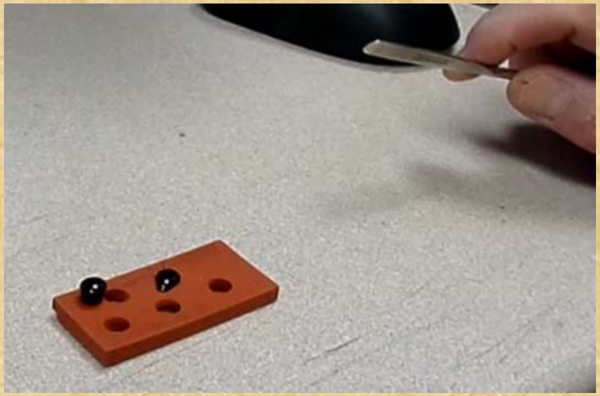
- Placement of sample on plates – requires new protocol
  - ✓ Adhesion primary concern
  - ✓ Requires heat at binder-plate interface
  - ✓ Significant for 8 mm, Critical for 4 mm
- Trimming – Current protocol with more care
  - ✓ Hot knife/scraping tool/torch or heat gun
- Bulge and specimen dimensions – requires new protocol
  - ✓ Temperature at which bulge and final gap is formed
  - ✓ Control of normal forces during final closure
- Two protocols: WRI and MTE
  - ✓ Primary difference in bulge formation
  - ✓ Two procedures need to be refined and evaluated

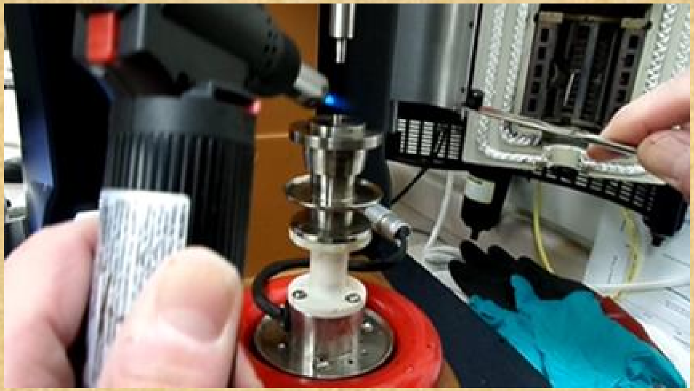
MTE Protocol

- 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

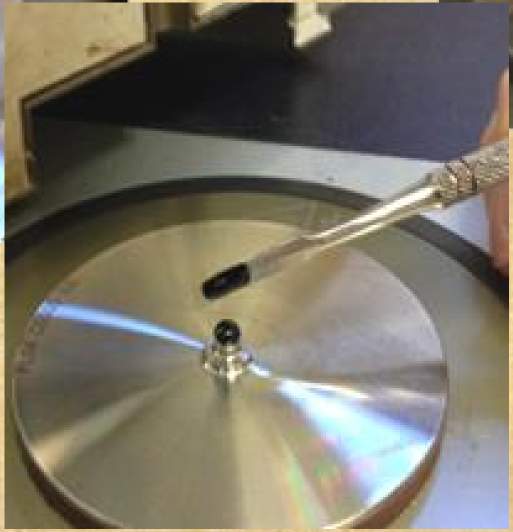




WRI Protocol

- 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

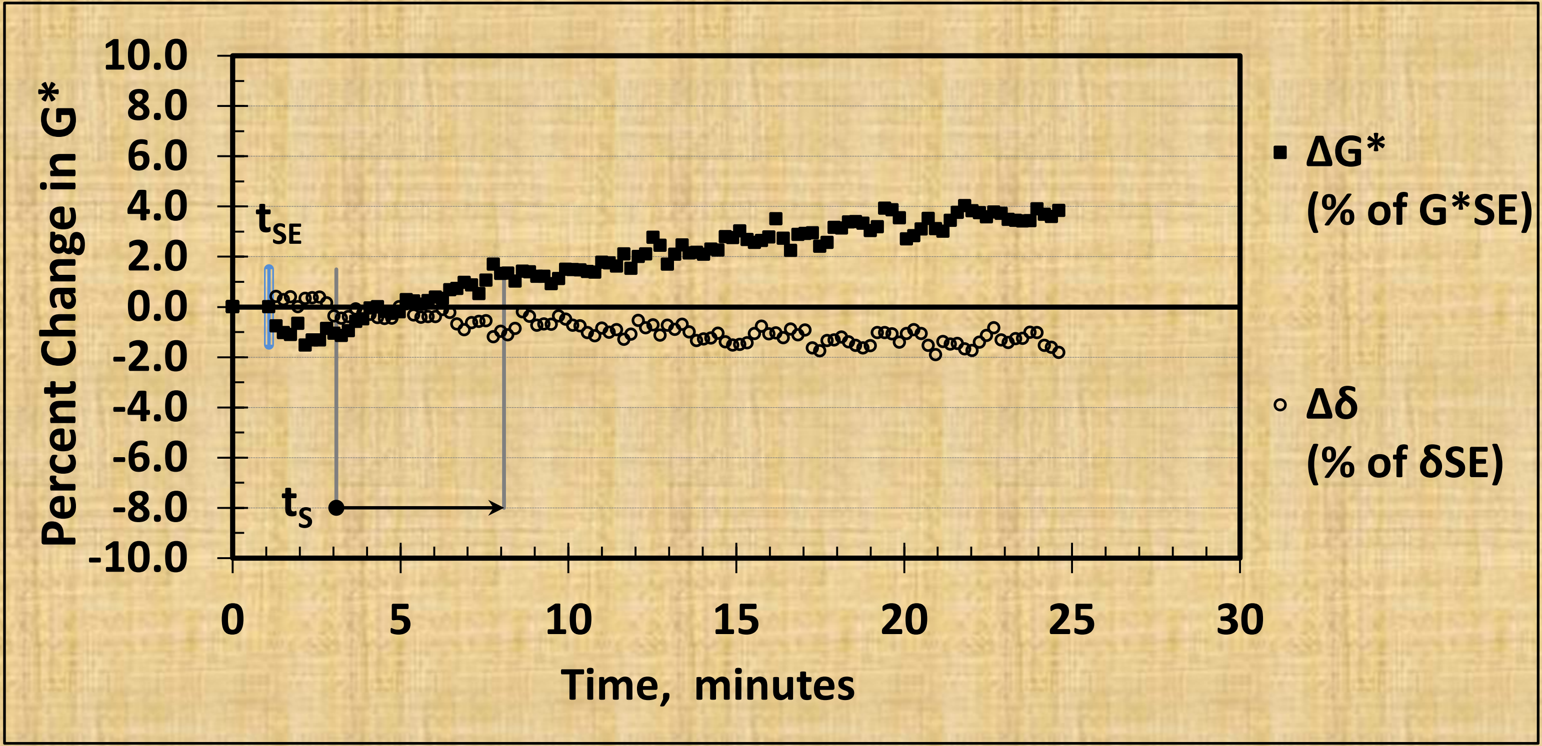


3. Issues - Conditioning prior to testing

- Wait time
  - ✓ Need to establish time increment required to reach specimen thermal equilibrium once DSR reaches thermal equilibrium
  - ✓ Above increment plus "cushion" = wait time
  - ✓ Use protocol established for 8 and 25 mm plates
  - ✓ Times for 4 mm similar to 8 and 25 mm plates, ± 2 min
- Physical hardening?
  - Considered with BBR and needs to be resolved for PP
  - Appears to be rheometer –specific
  - Being evaluated as part of wait time considerations

Protocol for establishing wait time

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



4. Issues - Testing Sequence

- 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

5. Issues – Linear region

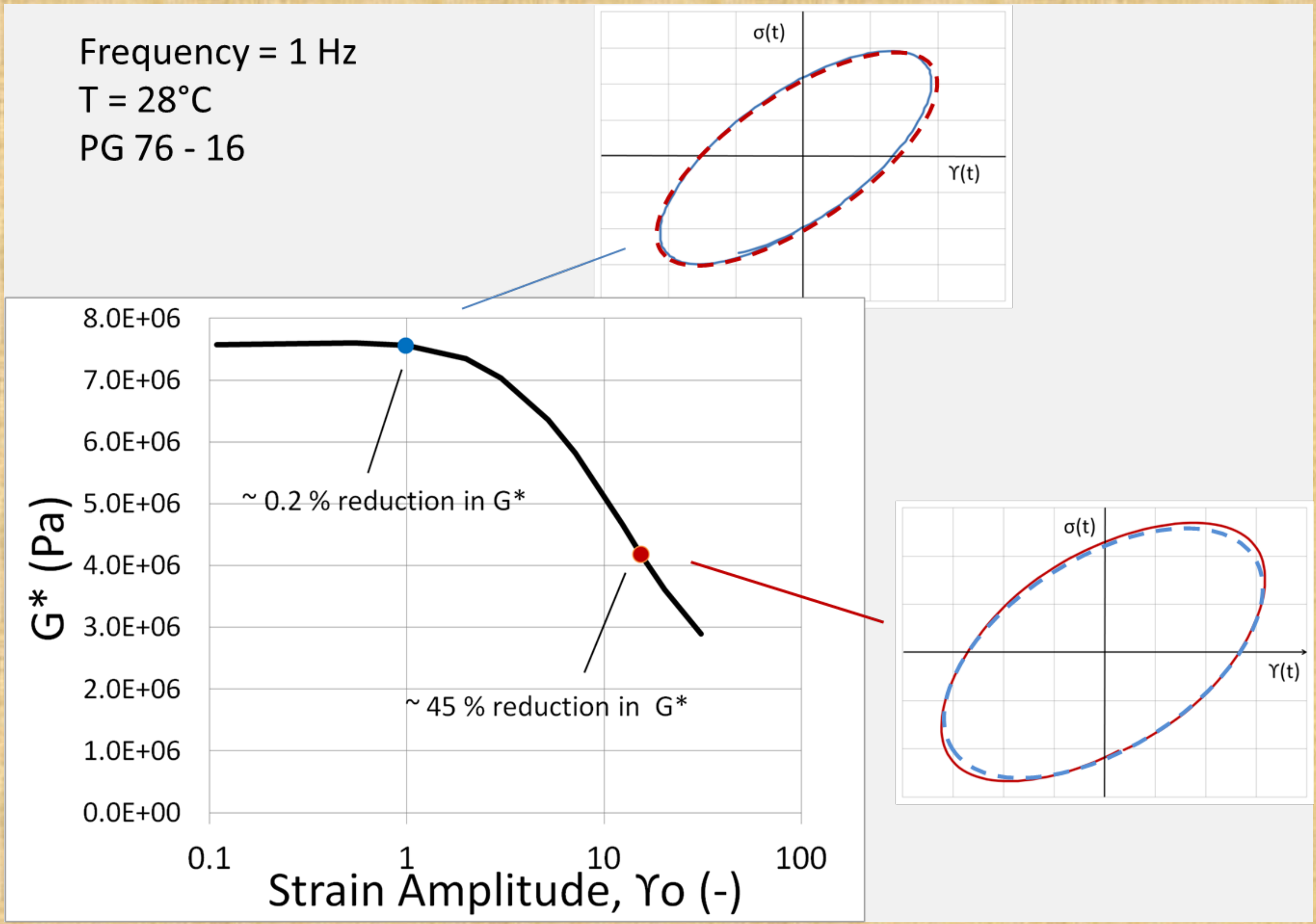
- Protocol is based on linear behavior
  - ✓ How do we ensure linear behavior?
  - ✓ Specify strain limits?
  - ✓ Perform strain sweeps?
- Specify strain as function of modulus

TEMP	MEDIAN COMPLEX MODULUS, Pa	MEDIAN TORQUE, µN·m	MTE TARGET % STRAIN	WRI TARGET % STRAIN
-40	1.22E+09	680	0.005	0.0015
-30	9.60E+08	540	0.005	0.0025
-20	5.60E+08	1738	0.02	0.005
-10	2.50E+08	1625	0.05	0.02
0	8.40E+07	1125	0.1	0.075
10	2.20E+07	590	0.2	0.25
20	5.20E+06	670	1	1
30	1.20E+06	300	2	2.5
40	3.00E+05	135	4	5
50	8.20E+04	50	5	10
60	2.00E+04	50	6	30

6. Issues: Verifying data integrity

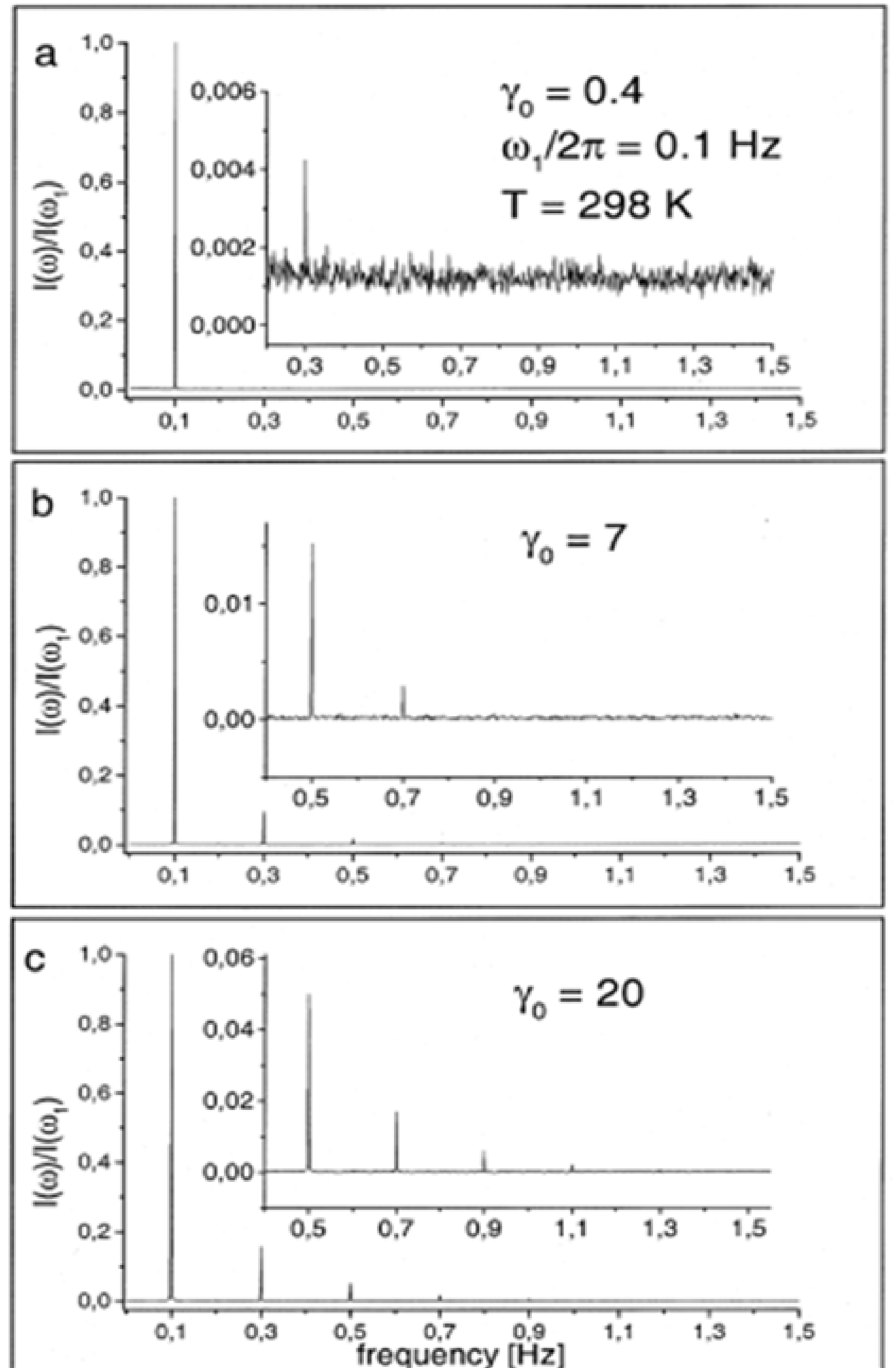
- Fall-off in G* with strain in strain sweep
- Lissajous Figures in isothermal test with varying frequency
- First and third Harmonics in isothermal test with varying frequency
- Not looking at Black Space or mastercurve construction at this point
  - ✓ Subject for later follow-on studies

Lissajous Figures



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

Results Step 1 – Testing Protocol

- Machine compliance
  - ✓ Resolved - Responsibility on manufacturer
- Sample preparation protocol
  - ✓ In progress
  - ✓ (2 Binders) x (WRI-MTE Protocols) x (Gap vs. Normal Control) x (3 Labs/Manufacturers)
- Temperature sequencing
  - ✓ Upcoming
- Protocol for evaluating data integrity
  - ✓ Upcoming

Specific Work Elements, cont'd

- 2^{nd} Step: Ruggedness Testing
  - ✓ Develop testing plan
  - ✓ Conduct plan using resources of Task Force
- 3^{rd} Step: Round-Robin Testing
  - ✓ Develop testing plan
  - ✓ Need more players to execute
- Training element
  - ✓ Many will need to "step up" proficiency to use 4 mm PP
  - ✓ Formal means of technology transfer will be required

Summary – Expected results

- Recommended protocol for using 4 mm and 8 mm PP geometry in dynamic shear
  - ✓ Testing protocols in specification format
  - ✓ Equipment requirements
- Ruggedness testing program
  - ✓ Expect to include rheometers from 3 manufacturers
  - ✓ Somewhat more robust than typical ruggedness program
- Recommendations for training
  - ✓ Needed before round robin to develop sufficient number of laboratories for robust round robin
- Round robin recommendations

Expanded Working Group

- Tom Bennert — Rutgers
- Kriz Pavel — BSA - Asphalt
- Ed Trujillo — CODOT
- Horst Winter — UMass
- Olli-Ville Laukkanen — UMass
- Three rheometer manufacturers
  - ✓ Intimately involved to date
  - ✓ Cooperation is greatly appreciated
- Group will be expanded slowly as work of task force continues