THERMAL EQUILIBRIUM

A Status Report

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Historical Observation – DSR Test Method

- Early on it was recognized that thermal gradients and thermal equilibrium can affect test measurement repeatability (especially laboratory bias)
  - ✓ Thermal gradients are currently accounted for with a temperature measurement between test plates
- Thermal equilibrium is addressed in the current AASHTO and ASTM test methods by a finite wait time (10 min)
  - ✓ No time limit is given for completion of measurements
- Current test procedure is built around specification measurements at 10 rad/s based on early generation DSR's many of which used water to maintain temperature control
  - ✓ Measurements were obtained 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
  - ✓ No procedure for determining instrument-specific thermal equilibrium
- Based on extensive series of tests the following was reported to the FHWA Asphalt Binder Expert Task Group:
  - ✓ dG*/dt time was recommended as the preferred criterion for determining when the test specimen approaches thermal equilibrium
  - ✓ Ten minute wait time appeared to be excessive
  - ✓ Wait time may be instrument- and fixture-specific
  - ✓ Test window should include both a “start” and “stop” time
- Anticipated adoption in ASTM and AASHTO DSR test methods
  - ✓ Not over yet – Time to rethink the issue

Factors That Can Affect Equilibrium

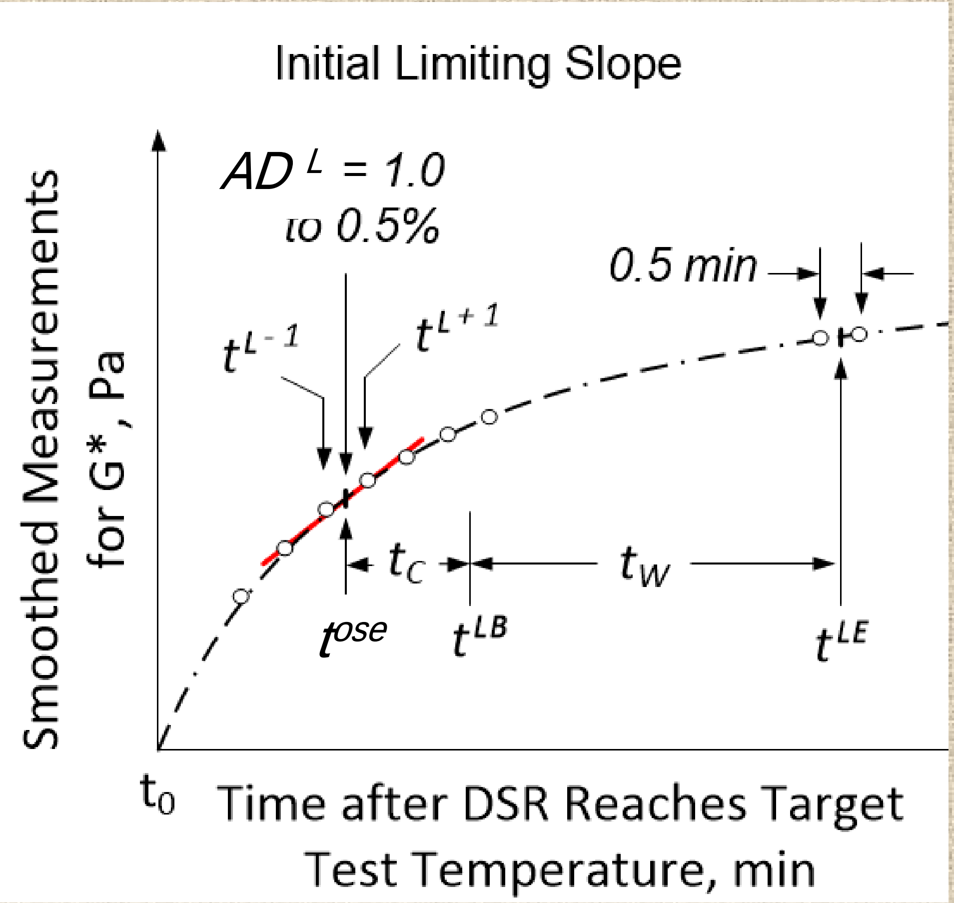
- Thermal equilibrium
  - ✓ Design of temperature control system
  - ✓ Difference between starting and target temperature – heat-cool rate
  - ✓ Fixture geometry
- Changes in binder properties – all give reversible hardening
  - ✓ Steric hardening
  - ✓ Physical hardening
  - ✓ Wax crystallization
- DSR equilibrium
  - ✓ Transducer equilibration
- DSR measurements will vary with time during isothermal test
  - ✓ Simple fact of life – need to choose some “Standard Conditions”
  - ✓ Question – how much variation is too much variation?

Specifying Onset of Specimen Equilibrium

- Specimen thermal equilibrium implies specimen mechanical properties (G^*, δ) are constant with following assumptions:
  - ✓ DSR is at thermal equilibrium – still may have gradients!
  - ✓ DSR components are stable – no drift in electronics or transducers
  - ✓ Binder properties are unchanging with time
    - Steric and physical hardening is minimal with time
- Procedure for estimating the approach of specimen thermal equilibrium – *time when additional changes can be neglected*
  - ✓ Proposal: Monitor changes in G^* with 30 min time sweep
  - ✓ Identify equilibrium as time when dG^*/dt is a “limiting value”
    - Complete equilibrium is reached exponentially with time
  - ✓ Procedure has been under development for several years
    - Still refining the “limiting value” and machine/fixture effects

5-Point Average Deviation Criteria

- ✓ AD^L – Limiting Average Deviation that defines approach of specimen thermal equilibrium
- ✓ t^{L-1} – Last time when Average Deviation of 5 measurements is greater than AD^L
- ✓ t^{L+1} – First time when Average Absolute Deviation of 5 measurements is less than AD^L
- ✓ t^{ose} – Time interpolated to AD^L
- ✓ t_C – Cushion (Factor of Safety)
- ✓ t^{BEGIN} – Beginning of test window
- ✓ t^{END} – End of test window
- ✓ t^W – Test window



Summary of Calculations

- Measure G* at 30-s intervals during 30 minute isothermal time sweep
  - ✓ Smooth measurements using 5-Pt moving average to minimize noise
- Assume equilibrium is approached at t^{ose}
  - ✓ t^{ose} is defined as the time when the moving average deviation reaches a limiting value, AD^L (AD^L) is approximately 0.5 to 1.0% change in G*/min)
  - ✓ Onset of thermal equilibrium
  - ✓ Find t^L -1, the longest time when the 5-Pt moving average deviation (AD) for all previous sets of 5 consecutive points is greater than S^L and,
  - ✓ Find t^L+1, the shortest time when the 5-Pt moving average deviation (AD) for all subsequent sets of 5 consecutive points is less than S^L
  - ✓ Interpolate to find t^{ose} as time when slope equals assumed limiting AD

Other Equilibrium Criteria

- Use fixed time as per current standard but review 10 minute value
  - ✓ Smooth measurements using 5-Pt moving average to minimize noise
  - ✓ May reduce to 8
- Define minimum allowable slope at which thermal equilibrium occurs
  - ✓ Hypothesis is that slope will will become zero
  - ✓ Use 11-Point slope
  - ✓ This will minimize thermal effects but will enhance steric hardening effects

Measurement Goal

- Select a measurement window that offers the best compromise between the effects of thermal equilibrium and time-dependent changes in binder properties
- Compromise must consider following:
  - ✓ If test too early then thermal equilibrium be the dominant effect on measurement repeatability
  - ✓ If wait too long then changes in binder will be the dominant effect
  - ✓ If test window varies between laboratories then the change in binder properties may affect variability in measured properties
- Recommend an approximate thermal equilibrium time using simple quantitative measurements that can be performed with a spreadsheet or incorporated into DSR software
  - ✓ Subjective criterion not acceptable

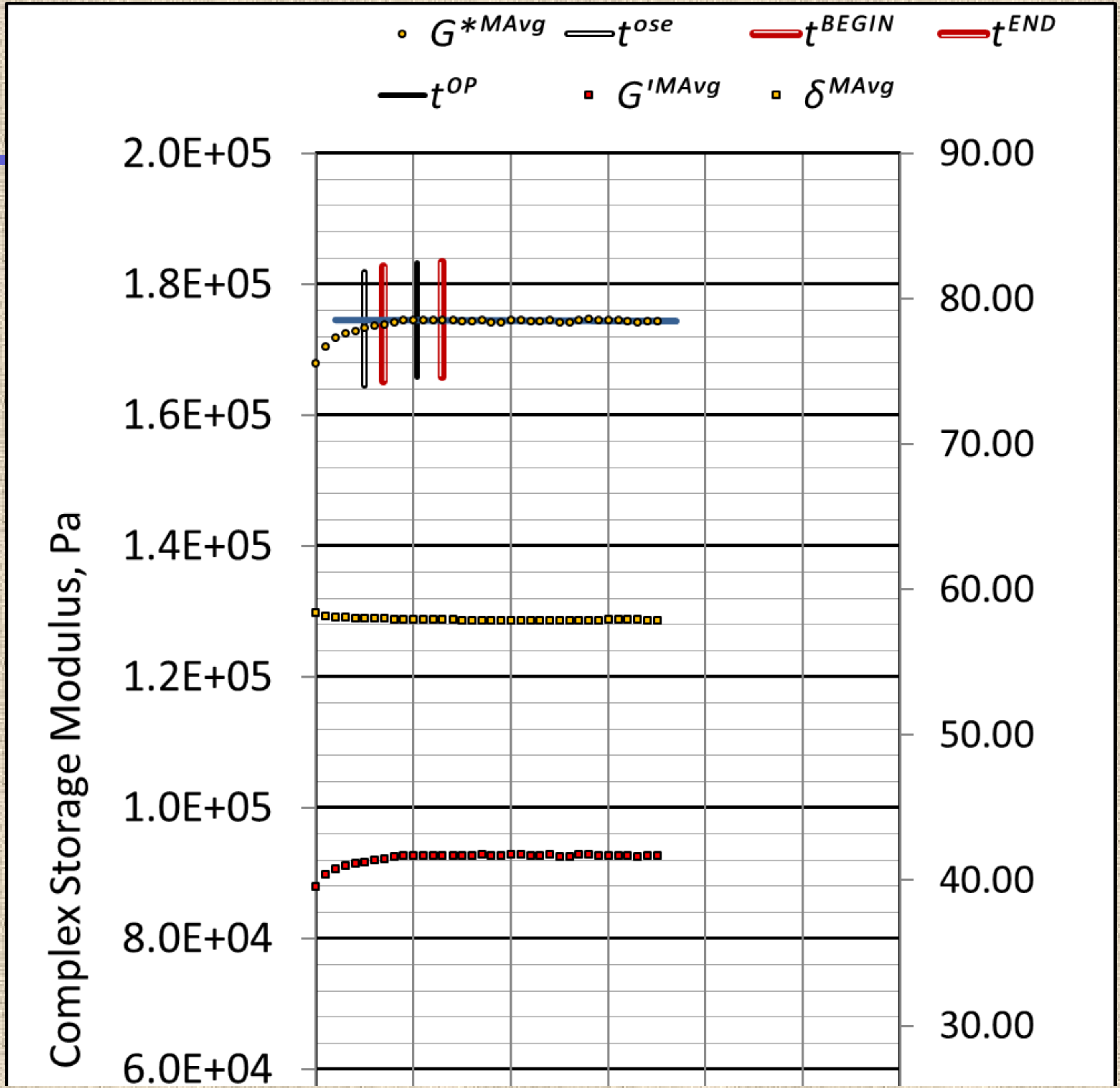
Analysis……………………

- Time to “step back and punt”
  - ✓ Previously reported times that were as small as few minutes is under review
  - ✓ Should we specify single time or device-specific, temperature time?
- What if analysis is now underway
  - ✓ Vary AD^L, window and fixed wait time
  - ✓ Extend to newer intermediate test temperature database as well as to previous data especially low temperature data
- Recommend test window that is a compromise between the effects of thermal equilibrium, DSR drift and time-dependent changes in binder properties

Some Selected Results

- Analysis is ongoing

Reference Fluid



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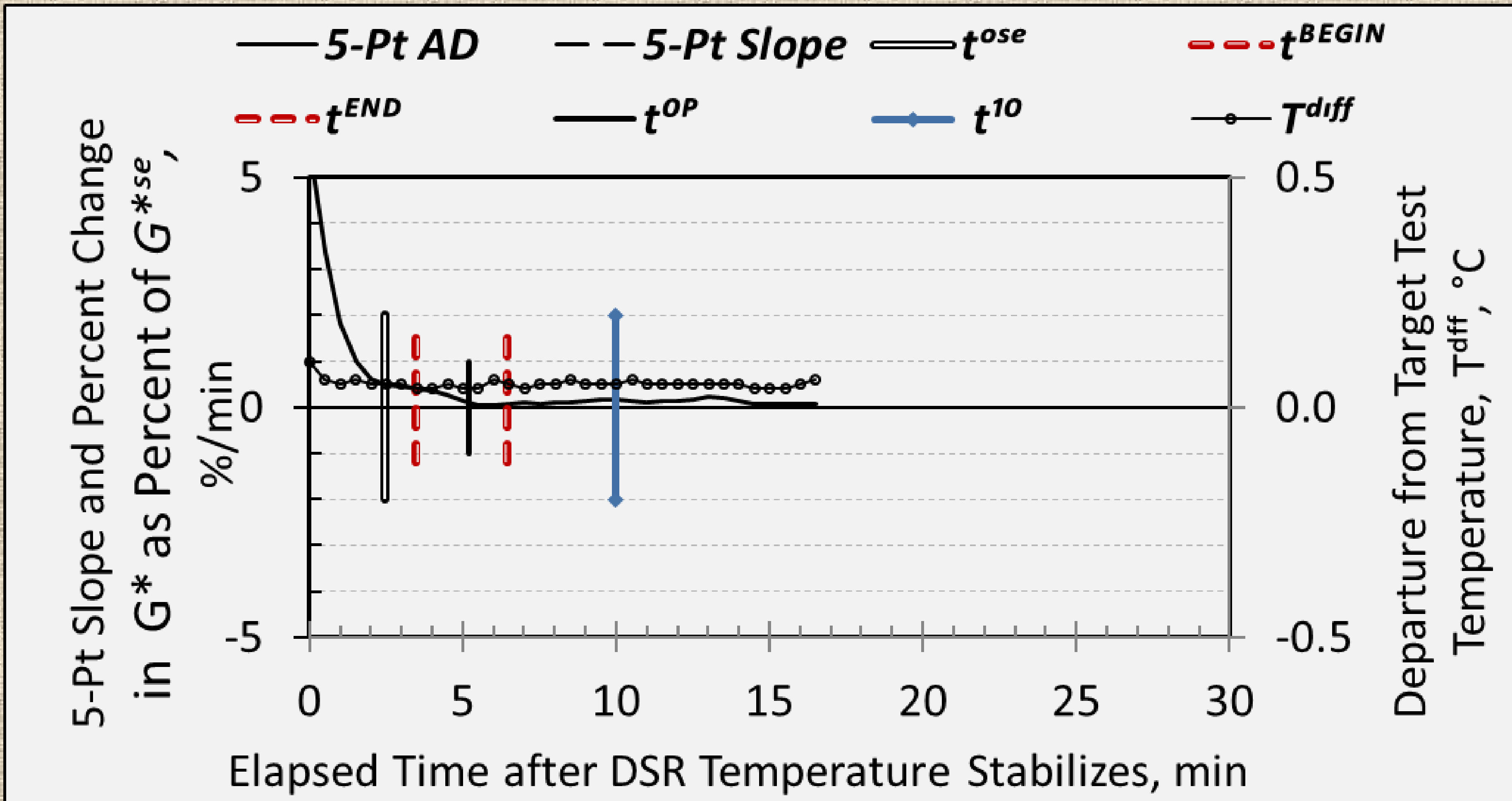

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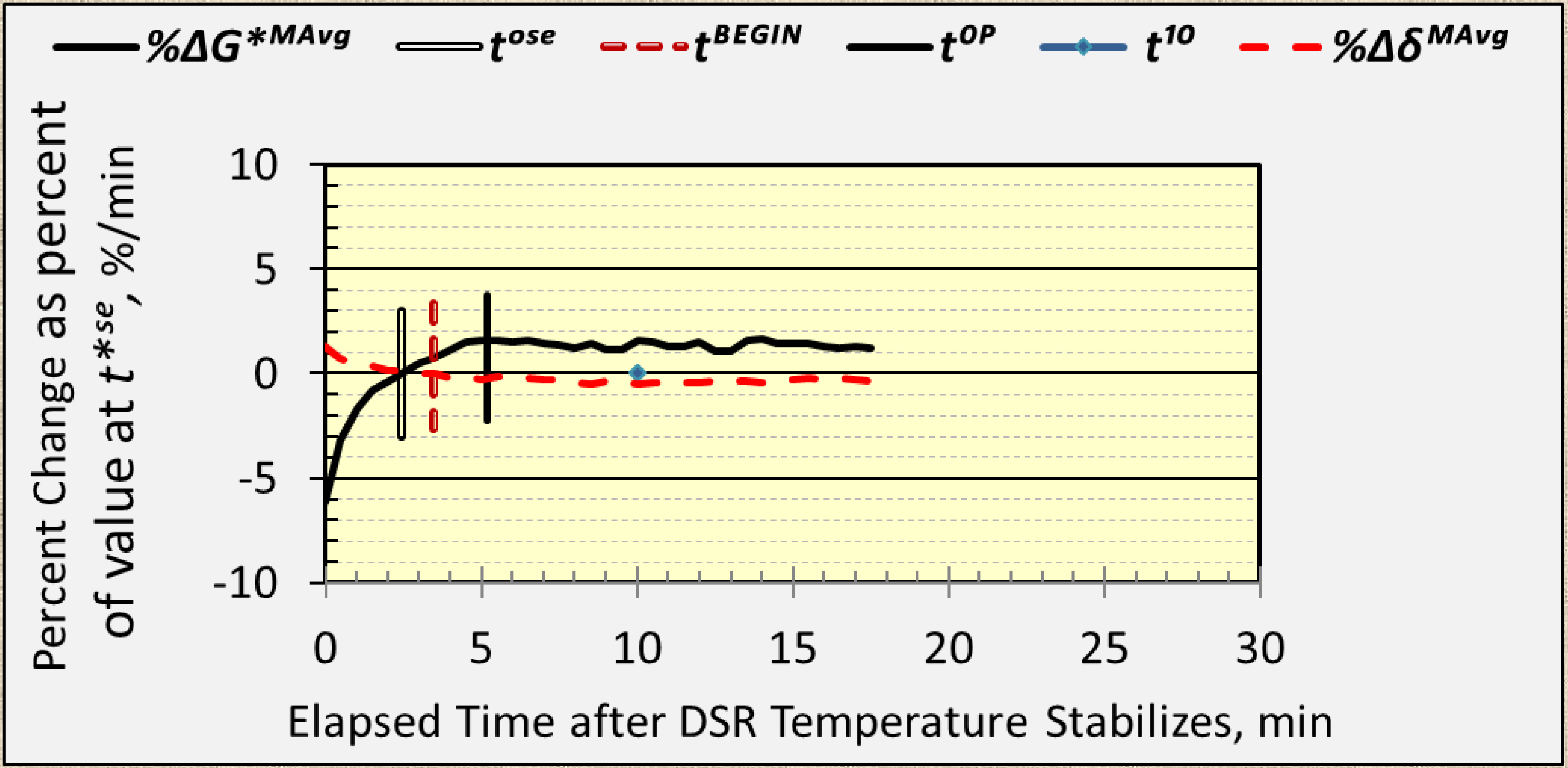
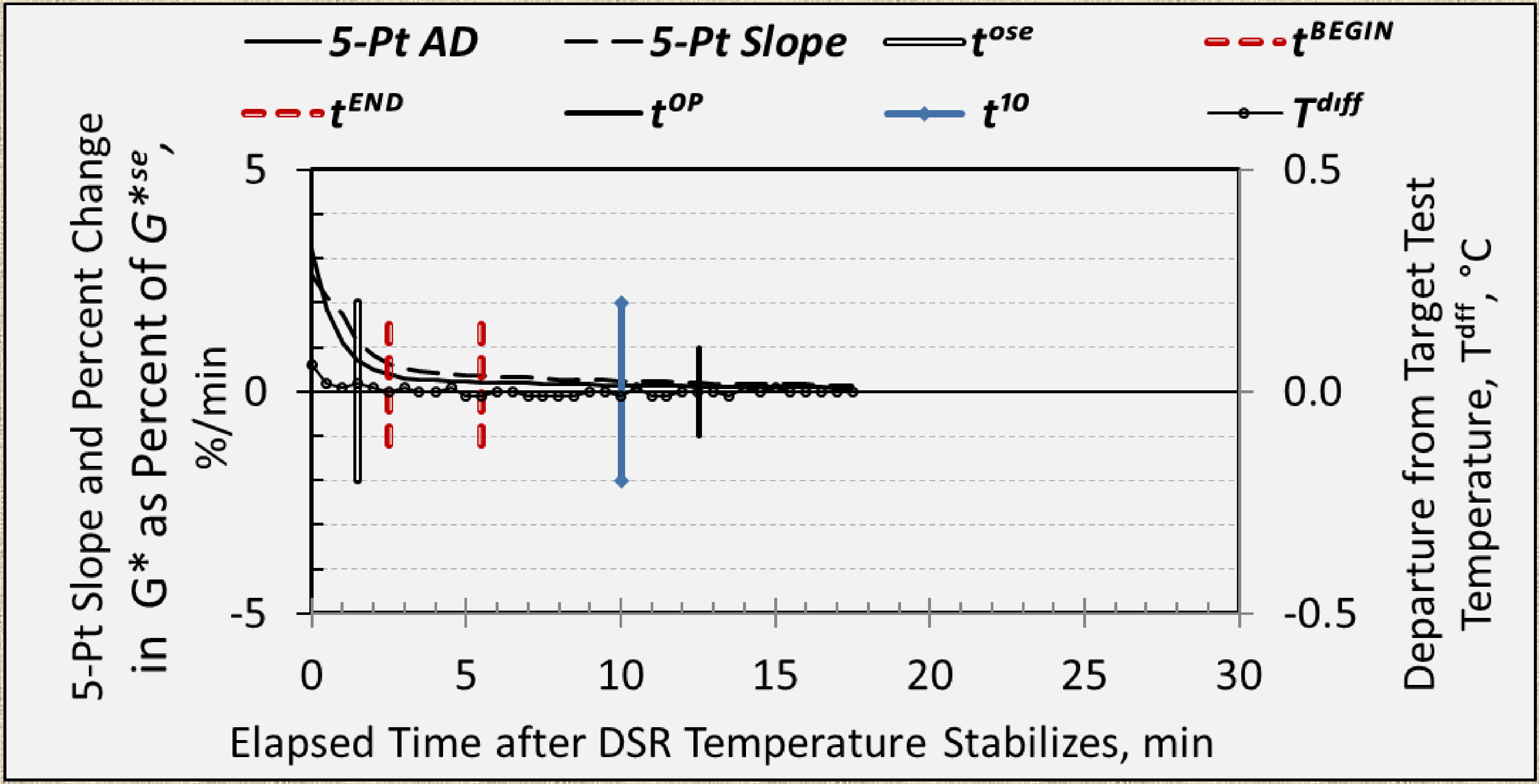
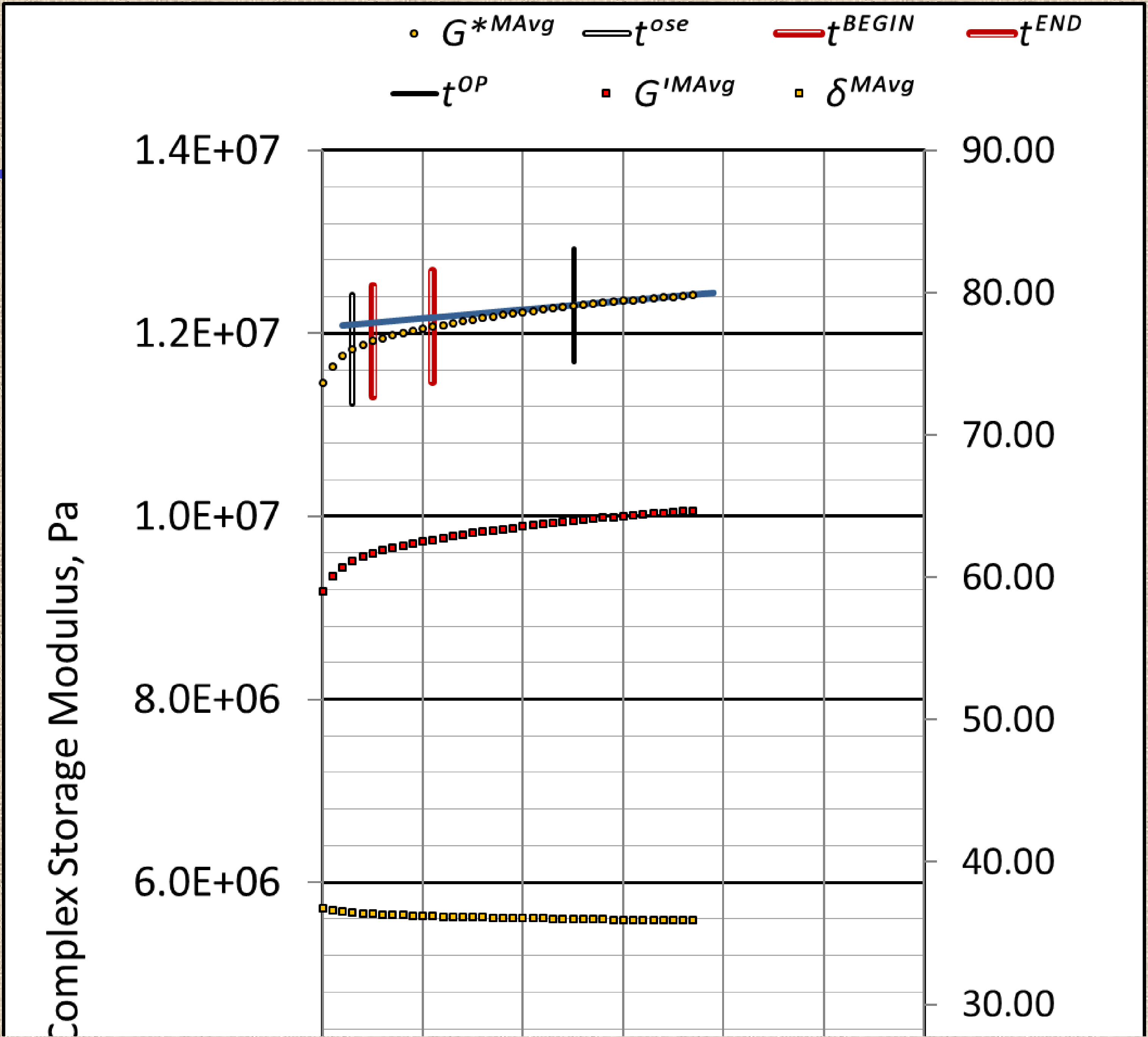


Asphalt Binder



Asphalt Binder



Asphalt Binder