CALCULATION OF THE RELAXATION MODULUS MASTER CURVE FROM DTT TEST DATA AND THE DETERMINATION OF CRITICAL CRACKING TEMPERATURE

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Objectives

- To improve procedures for the definition of master curves from DTT testing
- To review capability of DTT for low temperature $T_{\text{crit}}$ prediction
Typical DTT

stress

strain

brittle

brittle-ductile

ductile
The DTT can define rheology

\[ \sigma(t) = \gamma \int E(t) \, dt \]

E(t) is the tangent slope of the stress strain curve.
Information from DTT test

- Master curve of relaxation modulus from DTT
- Initial work presented at binder ETG – April 2001 in Phoenix
How good is the DTT at defining the low temperature rheology?
Determination of $E(t)$

- Initial scheme involves fitting tangent modulus to data from curve
- Several schemes being evaluated
  - Numerical averaging
  - Thor-Smith method
  - CAM model
- April 2001 - numerical appears to be more robust
- Start of test often results in early part of isotherm being rejected
Isotherms of $E(t)$
Master Curve $E(t)$

Sample ID: B-6232

Creep modulus CAM, Rms Err 2.35%
$T_{ref} = -20$

Acceptable master curve
Unacceptable master curve
Development of visco-elastic behavior

- Numerical analysis used to convert data
  - Hopkins and Hamming used with BBR to convert $S(t)$ to $E(t)$
  - DTT yields $E(t)$ direct
- Spectra fitted to data – used to convert between time and frequency domains
- Comparisons of visco-elastic properties
Comparison of master curves

E(t) master curve - DTT vs BBR

- 6228 DTT
- 6228 BBR
Overlaid Master Curves
Overlaid Master Curves – Expanded Cold Region, $G'$ & $G''$
Overlaid Master Curves – Expanded
Cold Region, $G^*$, Phase Angle
Overlaid Master Curves, $G(t)$ – Shear Relaxation Modulus
Effect of Loading Rate on DTT MC

LAMONT SECTION 3 @ 1mm/min @ -27 C vs. 0.388mm

\[ E(t), \text{ MPa} \]

1 mm/min

0.388 mm/min

\[ t, \text{ sec} \]
Master Curves

- Reasonable agreement on master curves is obtained.
- The data from the DTT appears to define a glass transition at a lower temperature based upon a peak in $G''$.
- More work was needed to look at numerical procedures.
Problems found

- Data sets contained fanned data
- Start up – zero condition not well defined if using DTT as rheometer
- Errors in DTT master curve greater than BBR
Low temperature cracking

- Removal of fanned data sets
- Adjustment of start conditions
- Work with binders tested at Calgary
Fanned data

- Removal of fanned data sets is required to ensure that data is compatible
- Fanned data is generally a result of poor sample preparation
- Fanned data is identified by inspection of E(t) criteria
Variable data from DTT
Start up errors – different slopes

Near-Origin Stress, MPa vs. Time, sec. [CITG067-23,DDD-1,8C], Strain rate = 3.06 %/min, -18°C
Start-up errors

- As control is passed from analogue to digital control some difficulties are always obtained at start conditions.
- Strain rates cannot be applied in an infinite time.
- Result is some error in the initial start conditions.
Start up errors – stress vs. strain

Graphic: Near-Origin Stress, MPa vs. Strain, Natural [PG67-22]; 6 Tests; Strain rate = 0.00 %/min, -12°C

Legend:
- Test 1
- Test 2
- Test 3
- Test 4
- Test 5
- Test 6
Start up errors – strain vs. time

Near-Origin Strain, Natural vs. Time, sec. [PG67-22] ; 6 Tests Strain rate = 3.0% min, -42°C
Start up errors – non-zero corrections
Definition of binder stiffness, $E(t)$

- Binder stiffness estimated from fitting model to DTT data

$$S(\xi) = S_{\text{glassy}} \left[ 1 + \left( \frac{\xi}{\lambda} \right)^{\beta} \right]^{-\kappa / \beta}$$

$s_{\text{glassy}}, \lambda, \beta$ and $\kappa$ are fitted.

- Model then used to obtain binder stiffness at time associated with failure
Adjustments to start up

The derived form of CAM equation (with usual three parameters, say A, B, C) is as follows:

\[ \sigma(t) = 3000 \times t \dot{\varepsilon} \left[ 1 + \left( \frac{t}{A} \right)^B \left( \frac{-C}{B} \right) \right] \]

Two additional unknowns D, E must now be introduced so that this equation can be applied to the actual raw data that doesn’t pass through the origin.

Used within Linear Visco-elastic limits
Examples of zero adjustments
Stress adjustment for high temperature (-28°C) test, conventional asphalt
Linear visco-elastic limit

-1
0
1
2
3
4
5
6789101112
log10 (G* Pa)

0
1
2
3
4
5

log10 (Strain %)

log10 (G* Pa)

SHRP
Method 2
Application of VE limit to isotherm construction

\[ E(t), \text{MPa } \text{vs. Time, sec. [U of Calgary]}, \text{ 6 Tests Strain rate } = 3.48 \% / \text{min, -28}^\circ \text{C} \]
Typical data set

![Graph showing all tests raw data. The x-axis represents time in seconds, ranging from 0 to 14. The y-axis represents stress in MPa, ranging from 0 to 4.5. Six different test curves are plotted: Test1 (blue), Test2 (magenta), Test3 (yellow), Test4 (cyan), Test5 (orange), and Test6 (green). Each curve shows the relationship between time and stress for the corresponding test.]
Identification of outlier using $E(t)$ plot

- Test 1: 0.94%
- Test 2: 0.93%
- Test 3: 1.73%
- Test 4: 1.36%
- Test 5: 1.33%
- Test 6: 1.54%

Outlier
Effect of data analysis scheme

- Effect of different analysis scheme
- Removal of start-up data not useful in improving reliability of isotherm
Isotherms with and without adjustment

Average isotherm from fitting CAM equation
Average isotherm from using slope information

No Slack removal
Slack removal

Average isotherm from fitting CAM equation
Analysis of Calgary Binders
Binders evaluated

- The binders used in the analysis had numerical references of 1293, 1719, 1730 and 1733
- Included highly modified and conventional binders
- Binder test data from the University of Calgary was analyzed on a “blind” basis.
**DTT test results for 4 binders**

<table>
<thead>
<tr>
<th>Binder Reference</th>
<th>Temperature (C)</th>
<th>Mean Stress at Break (MPa)</th>
<th>Mean Strain at Break (%)</th>
<th>Mean Energy at Break (mJ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1293</td>
<td>-18</td>
<td>5.328</td>
<td>1.395</td>
<td>44.36</td>
</tr>
<tr>
<td></td>
<td>-24</td>
<td>6.306</td>
<td>.7812</td>
<td>27.24</td>
</tr>
<tr>
<td>1719</td>
<td>-18</td>
<td>3.879</td>
<td>2.784</td>
<td>71.92</td>
</tr>
<tr>
<td></td>
<td>-24</td>
<td>4.635</td>
<td>1.001</td>
<td>27.47</td>
</tr>
<tr>
<td>1730</td>
<td>-24</td>
<td>6.580</td>
<td>3.454</td>
<td>164.3</td>
</tr>
<tr>
<td></td>
<td>-30</td>
<td>7.156</td>
<td>1.263</td>
<td>54.44</td>
</tr>
<tr>
<td>1733</td>
<td>-30</td>
<td>7.734</td>
<td>1.620</td>
<td>72.88</td>
</tr>
<tr>
<td></td>
<td>-34</td>
<td>8.125</td>
<td>1.076</td>
<td>48.18</td>
</tr>
</tbody>
</table>
Critical Cracking Temperatures ($T_{\text{crit}}$) of 4 Asphalt Binders using MP1 Specification

<table>
<thead>
<tr>
<th>Binder Reference</th>
<th>$T_{\text{crit}}$ (C)</th>
<th>Controlling parameter (S or m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1293</td>
<td>-28.3</td>
<td>m</td>
</tr>
<tr>
<td>1719</td>
<td>-33.7</td>
<td>both equal</td>
</tr>
<tr>
<td>1730</td>
<td>-36.3</td>
<td>m</td>
</tr>
<tr>
<td>1733</td>
<td>-38.4</td>
<td>S</td>
</tr>
</tbody>
</table>
MP1a Calculation

- Thermal stress derived from BBR
- In this work thermal stress was calculated from BBR and DDT
BBR isotherms
BBR master curve
Sample ID: 1293

Fracture Strength & Thermal Stress, MPa
Critical Temperature -29.0 °C

T, °C

Thermal Stress or Strength, MPa
$T_{\text{crit}}$ results

<table>
<thead>
<tr>
<th>Binder</th>
<th>$T_{\text{crit}, \text{MP1}}$</th>
<th>$T_{\text{crit}, \text{MP1A}}$</th>
<th>$T_{\text{crit}, \text{DTT}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1293</td>
<td>-28.4</td>
<td>-29.0</td>
<td>-31.9</td>
</tr>
<tr>
<td>1719</td>
<td>-33.8</td>
<td>-32.9</td>
<td>-31.7</td>
</tr>
<tr>
<td>1730</td>
<td>-36.3</td>
<td>-37.0</td>
<td>-37.1</td>
</tr>
<tr>
<td>1733</td>
<td>-38.4</td>
<td>-42.3</td>
<td>-43.3</td>
</tr>
</tbody>
</table>
$T_{\text{crit}}$ from different methods

![Graph showing $T_{\text{crit}}$ values for different methods at various temperatures.](image)
Summary

- Data gives good prediction of T\text{crit} from just DTT
- Study needs extension with more materials
- The results show the DTT-only method calculates the critical cracking temperature within 2.9°C of the current MP1A calculated values with the results being on average 0.7°C higher.
Thank you