# Low-temperature Flexibility of Mastic Asphalt and Asphalt Concrete with Polymer-modified Bitumen

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ABSTRACT: The European Asphalt Specification covers a wide range of test procedures to evaluate asphalt characteristics. In particular, tests to evaluate crack resistance and the performance of asphalt at low temperature have been included in prEN 12697-46 since a few months. This standard is based on the German testing procedure that allows for the evaluation of asphalt characteristics at low temperatures. The test procedure consists of uniaxial tension stress tests (UTST) and thermal stress restrained specimen tests (TSRST) at different temperatures. TSRST yield the failure stress, failure temperature, and cryogenic tensile strength versus temperature to assess low-temperature flexibility of asphalt mixes.

The present paper will describe the principle of the testing procedure and the low-temperature flexibility of mastic asphalt and asphalt concrete, each containing a different polymer-modified bitumen of the same sort (25/55-55 A and 10/40-65 A) from various manufacturers. The effects on asphalt mix properties under oxidative and/or thermal stress in the laboratory will be presented. These special stresses reflect potential changes of the asphalt mixes during the production process and storage. The results obtained will be assessed taking the existing German evaluation background into account.

KEYWORDS: Asphalt, thermal stress restrained specimen test (TSRST), low-temperature, oxidative and/ or thermal stress

#### 1 INTRODUCTION

Bitumen manufacturers are pursuing various approaches to producing high-quality and durable polymer-modified bitumen (PmB). It is frequently found by laboratory studies that PmBs of the same sort, but from various manufacturers exhibit various properties.

Various properties are obtained by force ductility tests, for instance. Conventional binder tests, such as needle penetration or softening point ring and ball tests, reveal only small differences in the delivered state, which is in accordance with the requirements. However, it is largely unknown whether and to what an extent these differences of characteristics affect usage properties of the asphalts in practice.

Studies (Büchler et al. 2008) show relationships between bitumen characteristics measured by a dynamic shear rheometer and force ductility tests to assess the cryogenic properties of stone mastic asphalts. The cryogenic flexibility results obtained by the Karlsruhe Institute of Technology, Institute of Highway and Railroad Engineering (Roos et al. 2009) in TSRST using asphalt concretes and mastic asphalts with various PmBs produced by various manufacturers will be evaluated and interpreted below.

#### 2 TEST DESCRIPTION

#### 2.1 Principle

To examine asphalt properties at low temperatures, a test device was developed at the Road Engineering Department of the Braunschweig Institute of Technology (Arand 1993, Eulitz 1987, TP A-StB 1994). This test device is placed in a temperature chamber which can generate temperatures down to -40 °C with an accuracy of ±0.3 K. The temperature chamber contains a test frame which consists of a base plate of high bending resistance and two columns supporting a stiff crossbeam. A gearbox with stepping motor is fixed to the base plate and can generate movements with an accuracy of 0.05 µm. The equipment for measuring compressive and tensile stress is fixed to the crossbeam. To avoid radial and/or transverse forces as well as moments in the specimen, it is placed between the gearbox and the pressure measurement equipment using two gimbal suspensions. As the steel frame is exposed to the same thermal changes as the examined specimens, it reacts with thermal shrinkage and expansion. Consequently, correct measurement of the actual strain of the specimen requires a basis of constant length at various temperatures. Two thermally indifferent measurement bases made of carbon fibers are used to measure the real deformation of the specimen and to counterbalance the thermal strain of the test equipment. Figure 1 shows a principle sketch and a photo of the test device.

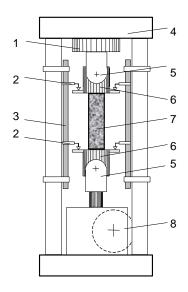
In the thermal stress restrained specimen test, the specimen length is kept constant, while its temperature is decreased with a constant cooling rate. Due to prohibited thermal shrinkage, the specimen is subjected to a (cryogenic) tensile stress. It is recommended to start the test at a temperature of +20 °C. Then, a cooling rate of -10 K/h is applied. This cooling rate induces tensile loads comparable to a deformation rate of 1 mm/min (TP A-StB). During the test, the core temperature of the specimen lags behind the air temperature in the test chamber. To record the correct specimen temperature, measurement of the temperature in an additional specimen during the test or pre-evaluation of the temperature lag between air temperature and specimen temperature is required (Karcher and Mollenhauer 2009).

As results, the temperature-dependent cryogenic stress  $\sigma_{cry}(T)$  [MPa], failure stress  $\sigma_{cry,F}$  [MPa], and failure temperature  $T_F$  [°C] are obtained. The increase of cryogenic stress is shown in Figure 4.

#### 2.2 Specimens

Prismatic specimens of 40 x 40 x 160 mm³ in dimension of asphalt mixes with a maximum grain size of 11 mm are used for the uniaxial tests. For mixtures with coarser aggregates, the cross-section is increased up to 50 x 50 mm². The specimens are sawn from laboratory-compacted asphalt plates or from field cores of about 300 mm in diameter. In this way, the longitudinal axes of the specimens are orthogonal to compaction direction. Thus, the test simulates horizontal evolution of thermal stress superposed by mechanical stress due to bending.

The specimen is fixed centrally within two adapters using a 2-component epoxy resin adhesive. After curing of the adhesive, the specimen is put between the gimbal suspensions of the test device (Karcher and Mollenhauer 2009).





- 1 Load Cell
- 2 Displacement Transducer
- 3 Thermally Indifferent Measurement Base
- 4 Crossbeam

- 5 Gimbal Suspension
- 6 Adapter
- 7 Specimen
- 8 Gearbox with Stepping Motor

Figure 1: Principle sketch and photo of the low-temperature TSRST device used by the KIT, Institute of Highway and Railroad Engineering

#### 2.3 Evaluation of the TSRST

So far, German Technical Standards do not contain any requirements on the cooling behavior of asphalts. Eulitz (2008) proposes an evaluation scheme to divide the asphalt surface layers into RStO 01 zones of cryogenic impact depending on failure temperature. Roads in Germany are dimensioned according to the Regulations for the Standardization of Road Pavements (RStO 01). The composition and thickness of the road construction depends on the road's location in one of the frost impact zones I to III. These zones are distinguished as a function of frost intensity. Frost impact zone III is subjected to the highest load. The experimental failure temperatures in the frost impact zones are listed in Table 1 for orientation purposes.

Table 1: Failure temperatures of asphalt surface layers (Eulitz 2008) for orientation purposes

| Frost impact zone         | I     | II    | III   |
|---------------------------|-------|-------|-------|
| Failure temperature in °C | ≤ -15 | ≤ -20 | ≤ -25 |

In Austria, a standard has already been drafted (ÖNORM B 3580-2:2008), in which asphalt surface and binder layers have to meet similar requirements as regards the maximum failure temperature. The maximum failure temperature of surface layers may vary between -30 and -20 °C depending on the mix. The maximum failure temperature of binder layers is allowed to vary between -25 and -20 °C.

## 3 COMPOSITION OF ASPHALT MIXES AND PROPERTIES OF POLYMER-MODIFIED BITUMEN SORTS

For the studies, six types of mastic asphalt 0/11 S and asphalt concrete 0/22 S each were produced with the polymer-modified bitumen sorts 10/40-65 A and 25/55-55 A made by four

different manufacturers (Roos et al. 2009). The composition of the asphalts according to ZTV Asphalt-StB 01 (2001) is obvious from Table 2. The bitumen sorts used in the asphalts are evident from Table 3.

Table 2: Composition of the asphalts according to ZTV Asphalt-StB 01 (2001)

| Type of Asphalt                      |  |                      | Mastic Asphalt<br>0/11 S | Asphalt Concrete 0/22 S |  |
|--------------------------------------|--|----------------------|--------------------------|-------------------------|--|
| Type of Aggregate                    | Filler 0/0.09                                | [-]                  | Limestone                | Limestone               |  |
|                                      | High-quality crushed sand 0/2                | [-]                  | Moraine                  | Moraine                 |  |
|                                      | Natural sand 0/2                             | [-]                  | Moraine                  | -                       |  |
|                                      | Grain class 2/5                              | [-]                  | Moraine                  | Moraine                 |  |
|                                      | Grain class 5/8                              | [-]                  | Moraine                  | Moraine                 |  |
|                                      | Grain class 8/11                             | [-]                  | Moraine                  | Moraine                 |  |
|                                      | Grain class 11/16                            | [-]                  | -                        | Syenite                 |  |
|                                      | Grain class 16/22                            | [-]                  | -                        | Syenite                 |  |
| High-quality Crushed                 | High-quality Crushed Sand/Natural Sand Ratio |                      | 2:1                      | 1:0                     |  |
| Fraction of                          | Grain class < 0.09                           | [wt%]                | 26.4                     | 6.5                     |  |
|                                      | 0.09 - 2.0                                   | [wt%]                | 23.6                     | 20.5                    |  |
|                                      | > 2.0  | [wt%]                | 50.0                     | 73.0                    |  |
| Raw Density of Aggregates            |  | [g/cm <sup>3</sup> ] | 2.668                    | 2.693                   |  |
| Bitumen Sort/Volume 10/40-65A        |  | [wt%]                | 7.3                      | 4.3                     |  |
| 25/55-55 A                           |  | [wt%]                | 7.0                      | 4.2                     |  |
| Void contents of the types 10/40-65A |  | [vol.%]              | -                        | 4.3 5.0                 |  |
| with the PmB of various 25/55-55 A   |  | [vol.%]              | -                        | 4.6 5.8                 |  |
| manufacturers                        |  | . ,                  |                          |                         |  |

To maintain a constant grain size distribution, the aggregate mixtures were prepared in exactly the same manner. Due to the use of PmBs of various manufacturers, void contents of the asphalt concretes and static die penetration depths of mastic asphalts varied in small ranges and resulted in the selection of the same bitumen volume. The twelve asphalts tested were selected, such that every manufacturer was represented at least once with every product (Table 3).

Table 3: Asphalts types – PmBs used in the asphalts and PmB manufacturers

| Asphalt Type and Sort      | Bitumen Sort | Bitumen Manufacturer  Asphalt Type No. |    |   |    |
|----------------------------|--------------|--|----|---|----|
|                            |              | 1                                      | 2  | 3 | 4  |
| Mastic Asphalt<br>0/11 S   | 10/40-65 A   | 1                                      | 2  | 3 |    |
|                            | 25/55-55 A   |  | 4  | 5 | 6  |
| Asphalt Concrete<br>0/22 S | 10/40-65 A   | 7                                      |    | 8 | 9  |
|                            | 25/55-55 A   | 10                                     | 11 |   | 12 |

Selected properties or features of the PmBs in the delivered state are obvious from Table 4. The standard characteristics of needle penetration and softening point R & B do not reveal any peculiarities. All characteristics of the PmBs were in compliance with the requirements made in the regulations. The partly large differences of certain characteristics obtained for a single PmB sort, e.g. for force ductility, have to be attributed among others to the variable composition and cross-linking of the polymers and the variable basic bitumen. This was confirmed by gel permeation chromatography and infrared spectroscopy of the PmBs.

Table 4: Selected bitumen features or properties in the delivered state

| 1 1  |                   |                     |                     |       |       |                     |       |       |       |       |
|--|-------------------|---------------------|---------------------|-------|-------|---------------------|-------|-------|-------|-------|
|  |                   | Bitumen Sort        | 10/40-65 A          |       |       | 25/55-55 A          |       |       |       |       |
| Property   | Unit              | Manufacturer        | 1                   | 2     | 3     | 4                   | 1     | 2     | 3     | 4     |
|  |                   | Grade               | 0 (Delivered State) |       |       | 0 (Delivered State) |       |       |       |       |
| Needle Penetration   | 0,1 mm            | DIN EN 1426         | 26                  | 35    | 35    | 28                  | 30    | 46    | 44    | 45    |
| Softening Point Ring and Ball                                  | °C                | DIN EN 1427         | 74.0                | 67.2  | 64.4  | 63.8                | 58.5  | 56.8  | 58.6  | 62.0  |
| Force Ductility, Deformation                                   | J                 | TL PmB/ DIN 52013   | 0.813               | 0.824 | 1.135 | 0.866               | 0.674 | 0.373 | 0.516 | 0.749 |
| Force Ductility, Maximum Force F <sub>max</sub>                | N                 | TL PmB/ DIN 52013   | 14.1                | 6.3   | 8.9   | 8.4                 | 6.9   | 4.7   | 3.5   | 2.4   |
| Force Ductility Deformation at 5 and 10 °C                     | J/cm <sup>2</sup> | DIN EN 13589/ 13703 | 3,89                | 6,43  | 7,44  | 7,38                | 3,88  | 2,92  | 4,87  | 10.5  |
| Force Ductility, Maximum Force F <sub>max</sub> at 5 and 10 °C | N                 | DIN EN 13589/ 13704 | 113.9               | 56.9  | 75.4  | 90.3                | 179.0 | 112.3 | 114.6 | 80.1  |
| Elastic Recovery 20 cm/ 25 °C                                  | %                 | DIN EN 13598        | 66                  | 74    | 78    | 78                  | 57    | 54    | 77    | 83    |

#### 4 LOADING OF THE ASPHALT MIX AT THE LABORATORY

To study cryogenic flexibility of asphalts as a function of variable bitumen properties in further detail, the asphalt mix was subjected to specific loading tests at the laboratory. Loading was not intended to simulate aging of the asphalts versus service life in-situ, but to model potential loads acting on the asphalts under various manufacturing and storage conditions and during transport. To consider specific manufacturing conditions of the asphalts, different types of loading were selected.

After mixing (step 1) according to the Technical Standards, mastic asphalts were subjected to a temperature of 250  $^{\circ}$ C and a low rotating speed in a closed mixer in order to prevent air from entering

- in loading step 2 for 60 minutes and in
- loading step 3 for 165 minutes.

The asphalt concretes were granulated after mixing (step 1) and placed onto a wire grating. The specimens were subjected to a thermal load of 180  $^{\circ}$ C at defined air exchange (11 m<sup>3</sup>/h) as follows:

- Step 2, 120 minutes
- Step 3, 180 minutes.

Hence, loading of mastic asphalts predominantly is thermal, while that of the asphalt concretes is oxidative and thermal.

The PmBs recovered from the asphalt mix after mixing (step 1) and loading (steps 2 and 3) were used to determine various bitumen characteristics, such as needle penetration, softening point R & B, force ductility, and elastic recovery. Selected results are obvious from the figures in the following section.

#### 5 TSRST RESULTS

TSRST were performed in steps 1 (after mixing) and 3 (after extended loading), and for selected types also in step 2 (after loading).

### 5.1 Asphalt Concrete

Failure temperatures of asphalt concretes with 10/40-65 A in step 1 (after mixing) reach an average value of -15.8 °C. Asphalt concretes with 25/55-55 A show a lower failure temperature of -17.9 °C, as had been expected due to the smaller stiffness (Fig. 3).

Thermal and oxidative loading of the asphalt concrete types in steps 2 and 3 caused embrittlement, which was reflected by higher softening points R & B and higher maximum forces  $F_{max}$  of force ductility measurements. In all asphalt concrete types, loading caused a significant increase in failure temperature. In asphalt concretes with 10/40-65 A, temperature increased by 9.1 K on the average from step 1 to step 3. In asphalt concrete types with 25/55-55 A, the temperature increased by 4.7 K. Type 8 shows a pronounced increase in the failure temperature from -17.9 °C to -3.2 °C. This strong increase is due to the strong embrittlement of the PmB with a softening point of 99.5 °C in step 3.

Failure temperatures in step 3 depend on the changed properties of the loaded PmBs. Due to the specific compositions of the different PmBs, every PmB reacts differently to thermal and oxidative loading. Initial values of the PmB characteristics also vary significantly. Consequently, the sensitivity to loading of every PmB varies, and this influences the asphalt properties in various ways.

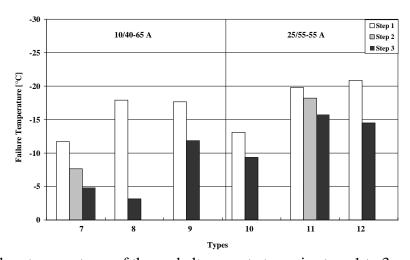


Figure 2: Failure temperatures of the asphalt concrete types in steps 1 to 3

Failure temperatures of the PmB sorts in step 1 show that the use of PmBs of various manufacturers (in the "state" of type testing) affects the TSRST results and, hence, cryogenic flexibility of asphalt concretes. Failure stress does not exhibit the same tendency (increase/decrease) in the different asphalt concrete types.

#### 5.2 Mastic Asphalt

Average failure temperature of mastic asphalts containing 10/40-65 A in step 1 amounts to -22.1 °C. Mastic asphalts containing 25/55-55 A reach a higher failure temperature of -25.2 °C in spite of a binder content reduced by 0.3 wt.% (Fig. 4). The mainly thermal loading in steps 2 and 3 causes the failure temperature to drop by an average value of 3.2 K for types 1 to 3 and by 3.3 K for types 4 to 6. The increase in failure temperature varied depending on the changed properties of the PmBs. In analogy to the asphalt concretes, it was found that every PmB has a different loading sensitivity that has variable effects on the asphalt properties.

Based on Table 1, the mastic asphalts can be assigned to frost impact zones. Mastic asphalts containing 10/40-65 A and permitted for use in frost impact zone II due to their failure temperature only fulfill the requirements of frost impact zone I in step 3. Under loading, mastic asphalts 4 and 6 deteriorated in quality from a suitability for use in frost impact zone III to suitability for use in zone II. The frost impact zone of mastic asphalt 5 did not change. This means that an asphalt subjected to thermal and/or oxidative loading in a mixer usually can no longer meet the requirements of the respective frost impact zone and premature damage of the road layer has to be expected.

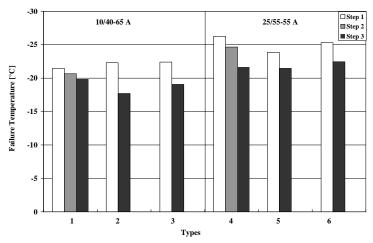


Figure 3: Failure temperatures of mastic asphalt types

The failure stresses did not reveal any uniform behavior over the loading steps. Variation of failure stress from step to step mainly depends on the density properties of the specimens.

Another characteristic to describe cryogenic flexibility is cryogenic stress at a certain temperature, which is independent of the cracking of the specimen and, hence, of the end of the experiment. Cryogenic stresses allow for a better differentiation of specimens (Büchler et al. 2008, Roos et al. 2009) than failure stress at the end of the test. For asphalt concretes, a temperature of -15°C was selected (Fig. 5).

Cryogenic stresses at -15 °C increased in all mastic asphalt types from step 1 to step 3.

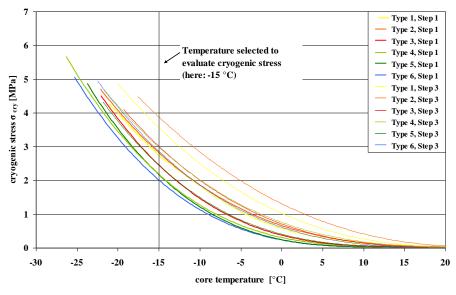


Figure 4: TSRST curves of mastic asphalts in steps 1 and 3

# 6 RELATIONSPHIP BETWEEN TSRST RESULTS AND BITUMEN CHARACTE-RISTICS

### 6.1 Asphalt Concrete

To study the influence of PmB properties on the cryogenic flexibility results, the maximum force  $F_{max}$  of force ductility measurement was determined. In analogy to the TSRST results, comparison of the bitumen studies in steps 1 to 3 revealed the loading sensitivities of the individual PmBs. Accordingly,  $F_{max}$  of force ductility measurement increased to a variable degree.

The relationship between failure temperature and maximum force  $F_{max}$  of force ductility measurement in steps 1 to 3 shows a high coefficient of determination of  $R^2 = 83.1$  % (Fig. 4). Comparison of the failure temperatures with the softening points R & B in all steps reveals a moderate coefficient of determination of  $R^2 = 69.6$  %.

No additional relationships were found. It must be assumed that the viscosity of the PmBs, expressed by the maximum force  $F_{\text{max}}$  of force ductility measurement, has a high influence on failure temperature.

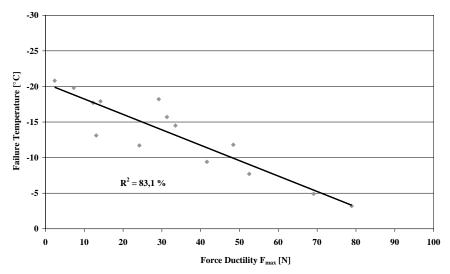


Figure 5: Relationship between the maximum force  $F_{max}$  of force ductility and failure temperature of asphalt concrete types in steps 1 to 3

#### 6.2 Mastic Asphalt

To study the impacts of PmB properties on the cryogenic flexibility results, the softening points R & B were determined.

Again, loading sensitivities of the individual PmBs varied in steps 1 to 3. From various initial values, the softening points R & B increased to a variable extent.

According to the investigations, the failure temperature is closely related to the bitumen characteristics of needle penetration, softening point R & B, and the maximum force  $F_{max}$  of force ductility (Table 5), the coefficients of determination being high. A very close relationship with a high coefficient of determination is also found between cryogenic stress at -15 °C and the bitumen characteristics (Table 5).

Table 5: Coefficients of determination for the relationships between selected bitumen characteristics and failure temperature and cryogenic stress at -15°C

| Characteristic                   | Failure Temperature | Cryogenic Stress at -15 °C |
|----------------------------------|---------------------|----------------------------|
| Needle Penetration               | $R^2 = 75.9 \%$     | $R^2 = 81.0 \%$            |
| Softening Point R & B            | $R^2 = 85.4 \%$     | $R^2 = 88.0 \%$            |
| Force Ductility F <sub>max</sub> | $R^2 = 84.2 \%$     | $R^2 = 87.6 \%$            |

Further investigations of the relationships between bitumen characteristics and failure stress did not yield any results.

#### 7 CONCLUSIONS

According to the investigations performed, PmB sorts of various manufacturers have properties that vary over a wide range and result in variable cryogenic flexibilities of the asphalts. When selecting an appropriate bitumen, the properties of the different products in terms of cryogenic flexibility should therefore be considered.

Oxidative or thermal and oxidative loading of the asphalts usually results in increased failure temperatures and increased cryogenic stresses. Variation of these characteristics due to loading depends on the typical loading sensitivity of the PmB. This was demonstrated by extensive studies after certain loading steps. However, loading was not found to always modify failure stress in the same direction.

Comparison of selected bitumen characteristics with the TSRST results using asphalt concrete types revealed relationships between the maximum force  $F_{max}$  of force ductility measurement and failure temperature. For mastic asphalts, relationships were found between the softening point R & B and failure temperature as well as cryogenic stress at -15°C. It must therefore be assumed that the viscosity of the PmBs has a considerable influence on failure temperature and cryogenic stress.

It remains to be found out in the future how further thermal or oxidative and thermal loading beyond step 3 will affect the bitumen and cryogenic properties of the asphalts. Loading sensitivities of commercial PmBs and their effects on other usage properties (fatigue and deformation properties) also have to be studied and documented.

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