GENERALIZED RELATION OF BITUMEN STRESS AT SHEAR TO ITS PENETRATION IN A RANGE FROM SOFTENING POINT TEMPERATURE TO BREAKING POINT TEMPERATURE

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Abstract. The problem of assessing the quality of bitumen is in the fact that the common quality indicators are empirical. They do not allow the objective characterization neither the consistency of bitumen no its transition from one state of matter to other. Goal. The solution to this problem is based on the principle that the resistance of bitumen to the penetration of the needle is a shear characteristic under conditions of complete adhesion to the surface of the needle. Methodology. The practical confirmation of this approach is based on the Core & Laurent method, which takes into account the rate of the bitumen layer and the resistance force to this displacement based on the geometric parameters of the needle and the force applied to it. Attempts to estimate bitumen viscosity with the depth of needle penetration have failed for two reasons. Firstly, bitumen viscosity is principally a technological characteristic. Secondly, in the simple conversion of the penetration value into viscosity neither the shear rate nor flow anomalies and a units ratio were taken into account. Results. In this research work the shear stress at penetration at the softening point temperature and breaking point temperature is taken as a mechanical characteristic of the states of matter. This statement of the problem is based on the linearity of the relationship between the test temperature and log of penetration, established by Hekelom. Later, this relationship was supplemented with a temperature of equal penetration $T_{31}$ as a midpoint of the plasticity interval of bitumen with the same penetration but different penetration indexes. Practical value of this work is results in finding the relation between penetration and the reverse pliability $G^*/\sin \varphi$, which is applied in the Superpave system to evaluate the bitumen component in the rutting resistance of asphalt concrete. Keywords: bitumen, penetration, softening point temperature, isopenetration, breaking point temperature, complex modulus, phase angle, stress.

“This paper discusses the relation between a number of conventional mechanical tests for asphaltic bitumen and its mechanical properties.”

N. J. Saal [1]

Introduction

The object of this research work was formed almost 70 years ago and presented in the epigraph of this article [1]. The paradigm proposed here is grounded on an assumption that, for each depth of standard needle penetration into bitumen, there is an inherent specific value of shear stress. This implies that in the frame of this work the depth of needle penetration is considered as a shear characteristic, contrary to the conventional point of view of it as a viscous characteristic of bitumen.

Publications analysis

This latter is found in many publications that have had no use in practice, mostly for the reason that the viscosity is considered to characterise the bitumen flow at high temperatures, not at service temperatures. This leads exactly from the research work by Van der Poel [2], where a graph for finding the bitumen stiffness modulus with a known depth of needle penetration, the softening point temperature, and the Penetration Index that is calculated from both of these is presented. The stiffness modulus is used in [2] because it was impossible with the equipment at that time to provide the bitumen deformation in the linear range where the proportionality between stress and strain is constant (in other words, where the stiffness modulus in the specific region of deformation amplitude remains unchanged). In his research, Van der Poel highlights that the
reliability of the stiffness modulus increases when the deformation amplitude and shear stress become lower. The values of the main rheological characteristics (modulus and viscosity) depend on the level of force (shear or normal) that induces the corresponding stresses in the material. In research by [3], a first attempt was made to calculate the bitumen viscosity with penetration, depending on the shear rate specific to each penetration value. This research takes into account the shear rates at penetration of the bitumen with the same penetration but a different Viscosity Anomaly, which is in close correlation with the Penetration Index.

In the 1990s in the frame of Superpave system development, it became clear that finding the mechanical characteristics of bitumen, such as the stress resistance, complex modulus and phase angle, is necessary to estimate the influence of the bitumen on asphalt rutting resistance. These mechanical characteristics are a base for the criteria of asphalt rutting resistance in various PG regions in the USA. The linear semi-logarithmic dependence between penetration and temperature (BTDC) was shown earlier in 1975 by Hekelom [4]. This dependence includes three temperatures at least. It was extrapolated on $T_{800}$ (the temperature at which penetration reaches 800 dmm), even though at that time this temperature was considered as a softening point temperature. Also it was extrapolated on $T_{1.25}$, at which bitumen becomes brittle. This is why nowadays the two penetration indexes currently exist. One can be found with the penetration and softening point temperature ($P$ and $T_{R&B}$), the other with the temperature penetration relation ($P$ and $T_{800}$). This relation connects with the temperature not only penetration, but also viscosity. This article considers this dependence in a range from the breaking point temperature to the $T_{800}$ temperature.

Extrapolation of this dependence on the penetration 1.25 dmm lets us find the Fraas breaking point temperature ($T_{Fr}$) on the plot. Later experiments proved a good correlation between $T_{Fr}$ and $T_{1.25}$. The feature of this plot is that it takes into account the temperature susceptibility of bitumen binders, and as a result finds the temperatures of the rheological state of bitumen of various consistencies, sources, technological and structural types.

In [5], generalized temperature–penetration dependences (Figure 1) were proposed for bitumen with different penetration indices, which intersected at the pole point corresponding to a penetration of 31 dmm and in the middle of the plasticity interval (from $T_{800}$ to $T_{Fr}$). With this temperature, it became possible to calculate the brittle point temperature ($T_{Fr}$) or softening point temperature by the equation: $T_{Fr} = 2T_{31} - T_{800}$.

Presentation of the main material
Formulation of Research Goal
Taking this into account, the modified Hekelom's diagram is reduced to a straight-line relationship between $T_{800}$ (softening), $T_{25}$ (standard temperature for penetration test), $T_{31}$ (isopenetration - pole) and $T_{1.25}$ (transition to the brittle state). Consequently, the depth of needle penetration makes it possible to distinguish various aggregate-thermomechanical states of bituminous binders: brittle (below $T_{Fr}$), viscoelastic (above $T_{Fr}$), and fluid (above $T_{800}$).

![Fig. 1. Influence of the penetration index on the generalized temperature-penetration dependences](image)

The principle and method for finding the relationship between penetration and shear stress used here as an axiom are confirmed by comparing the values of the shear stress ($E$) obtained by calculation, viscometrically and as a result of the one-plane shear scheme tests [6].
This made it possible to formulate the problem of determining the shear stress of bitumen at each of the above-mentioned temperatures.

To obtain a diagram linking the shear stress with the temperatures of characteristic states ($T_{800}$, $T_{25}$, $T_{31}$, $T_F$), it is necessary to find the shear stress values at each of them and prove that they all belong to one semi-logarithmic straight line ($\tau = BTDC$), similar to the dependence of the logarithm of penetration on temperature according to Hekelom. But in contrast to this diagram, the one proposed here should relate the stresses at each of the indicated temperatures instead of the penetration values.

**Stresses at the Softening Point Temperature (according to $T_{800}$)**

The softening point temperature is the most convenient for finding the shear stress. In this case, the shear stress can be calculated with the viscosity and shear rate at 800 dmm [3]. In the data from numerous publications, this shear stress varies within fairly narrow limits: 1300 Pa at $\dot{\gamma} = 1.15\cdot10^{-1}$ according to (Carre & Laurent, 1963); 1200 Pa at $\dot{\gamma} = 1\cdot10^{-1}$ according to [7]; 1500 Pa according to [8] 1260 Pa according to [9]; 1350 Pa according to [10]; 1350 Pa according to [11]. On average, it is close to 1320 Pa.

In the standard EN 12591, prepared for approval, the value of the complex viscosity is taken to be 1500 Pa·s. In this case, the shear stress $\tau$ is also 1500 Pa. According to the developers' opinion, this value obtains at $T_{800}$. This is what is accepted in this work as the isothermal stress at $T_{800}$.

**Stress at Penetration of the Tested Bitumen**

This stress is calculated according to the method based on the concept by [12], in which penetration is considered as a shear stress of the bitumen layer. With the penetration depth of the needle and the temperature $T_{800}$, the penetration index $P_{800}$ is calculated. With the penetration index, the bitumen flow anomaly index ($C$) is determined; from the dependences of equal shear stresses (log $\tau_c = -1.3 \log P_{25}+6.78$) and equal shear rates (log $\dot{\gamma}_c = 1.053 \log P_{25}-2.87$) on penetration, the value of this characteristic is determined for the assessed bitumen.

**Determination of Shear Stress at Penetration 31 dmm**

To find this stress, the temperature dependences of cohesion were experimentally obtained for 5 bitumens of different penetration (Fig. 2) [5]. According to the data, the values of penetration at 31 dmm correspond to the values of bitumen cohesion: for bitumen with a penetration at 25 ºС of 54 dmm, it is 0.2 MPa; for 63 dmm it is 0.21 MPa; for 103 dmm it is 0.21 MPa; for 155 dmm it is 0.215 MPa; and for 207 dmm it is 0.22 MPa. The experiments were carried out at a shear rate of 1s⁻¹. In accordance with the results obtained, the isopenetration values for bitumen of different consistencies are very close (the average value is about 0.21 MPa). A stress value of 0.21 MPa is obtained for each bitumen at its specific temperature. These temperatures decrease with the increasing bitumen penetration. Thus, in the transition from bitumen with a penetration of 54 dmm to bitumen with a penetration of 200 dmm, $T_{31}$ decreased by 13.5 ºС. At the same time, the breaking point temperature decreased by 9 ºС, and the softening point temperature decreased by 13.4 ºС. Thus, the isostress values for the diagram are taken as equal: at $T_{800}$ it is 1.5·$10^3$ Pa; at $T_{25}$ the values correspond to the penetration (calculated according to [12]): at $T_{31}$ it is 2.1·$10^4$ Pa.

**Mechanical Characteristics of Winter Cracking Resistance**

The breaking point temperature of bitumen is the least available for mechanical interpretation. Its purpose is to be a criterion for the winter cracking resistance of asphalt concrete. Traditionally, the Fraas breaking point temperature is used for this. Like any other empirical characteristic, it is not an objective index that refers to the essence of crack formation globally.
In the academic interpretation [13], the breaking point temperature is the temperature of brittle fracture by exceeding the elastic deformations limit. Is the Fraas breaking point temperature linked to this? This is still not clear - most likely it is not. With decreasing temperature, amorphous low molecular weight polymers transform into a truly brittle state, characterised by the glass transition temperature (T_g). Glass transition takes place due to a decrease in the energy of thermal motion of molecules, their association, the formation of branched structures, or a micellar structure in the case of bitumen. It is accompanied by an increase in the density of the structure, an increase in viscosity to 10^{12} Pa·s [14], and a decrease in free volume to 3%.

The main method for determining the glass transition temperature (T_g) is the dilatometric method [15] based on obtaining the temperature at which the rate of decrease of the volume changes. The glass transition temperature determined in this way, like the change in other characteristics (viscosity, storage or loss moduli), also depends on the test conditions. It increases with the acceleration of loading, with an increase in the frequency of deformation.

The relationship between the breaking point (T_F) and glass transition (T_g) temperature of bitumen is extremely poorly studied. One of the few works, perhaps the only one of its kind, is the article by [15], who interprets the transition to glass state from the classical standpoint of free volume, in this case the immobilisation of the structural components of bitumen. A comparison is made between the breaking point temperatures by Fraas and the glass transition temperatures obtained by the classical dilatometric method with a cooling rate of 1 °C per minute for distilled and oxidised bitumen of different penetration. The practical parallelism of the dependences of the glass transition temperatures on the penetration of bitumen obtained by these technologies, as well as significant differences in the absolute values of these temperatures, have been established for Venezuelan oils, for which the temperature T_g is 12–13 °C lower than the temperature T_F. For bitumen with a high value (of more than +2.2) of penetration index (PI), the difference increases to 23–24 °C.

In road practice, attempts are being made to change the criterion and methods for assessing the fracture temperature of bitumen and thus omit the need for the empirical Fraas breaking point temperature. With this, for a clearer understanding of the subject under discussion, one should resort to the generalized term temperature of cracking of bitumen. This temperature is determined in various ways. The most common, other than Fraas, are methods for determining the fracture temperatures based on the determination of the first crack under the cooling in specimens of a fixed length – the TSRS method [16] and ring specimens with a fixed inner diameter – the ABCD method [17,18]. The data on bitumen with different penetrations, summarised together, are shown in Table 1. The temperatures of cracking, obtained on the Fraas device for all three bitumens presented in the work, are 9.4–13.2 °C higher than the temperatures obtained by the TSRS method, and 13.6–17.8 °C higher than those obtained by the ABCD method. The smallest difference is in the breaking point temperatures obtained by Fraas and by the BBR-300 method BBR m = 0.3 is 5.3/6.3; 5.6/7.2; 2.5/5.2 (°C).

The high cracking resistance temperature according to Fraas can be explained by the combination of the influence of two factors: temperature stresses inside the film and forced bending stresses. The reason for this may also be a large bending deformation - about 1.64%. This is evidenced by the dependencies obtained under constrained tensile temperature stress and mechanical bending destruction of asphalt concrete beams (determination of the maximum tensile strength at different temperatures) [19, 20 and 21]. In this case, the temperatures according to TSRST (thermal destruction) are 8–17 °C lower than the temperatures of the maximum strength in free bending, since in the last case two factors of destruction take effect: external load and internal temperature stresses caused by the difference in the coefficients of thermal expansion of the components.

In the case of asphalt concrete, this applies to mineral aggregates and bitumen, and in the case of bitumen, to oils, resins and asphaltenes. In addition, Fraas breaking point temperatures can be higher due to fatigue phenomena that accompany fracture during multi-cyclic bending of plates [22].

![Table 1 – Breaking point temperatures for bitumen [16, 17 and 18](image)](image)
The cracking temperatures determined by BBR [23] are close to the Fraas breaking point temperatures due to the fact that the assigned limits of the stiffness modulus (300 MPa) and creep coefficient \( m = 0.3 \) do not sufficiently meet the brittle fracture conditions. With such values of the criteria, the bitumen is rather far from the truly brittle state [19] and they still exhibit a viscous component of deformation, while truly brittle fracture, as mentioned above, is the destruction of an elastic body. Even asphalt concrete with an exponent \( m = 0.3 \) demonstrates a noticeable dependence on the deformation rate. The “\( m \)” values in their meaning are close to the tangent of the angle of mechanical losses. For asphalt concrete, especially bitumen, to be close to an elastic body, this angle should be 5–10\(^\circ\), and \( \sin \varphi \) should approach 0.1 [24]. In this case, simply put, the complex modulus approaches the elastic modulus. In this regard, it seems that the norms “300 MPa” and “\( m = 0.3 \)” should probably be revised towards tightening by reducing the value of \( m \) to 0.1–0.15. The data in Table 1 convincingly indicate that the Fraas breaking point temperature is much higher than the breaking point temperatures obtained by other methods.

At the same time, the temperatures of fracture formation by the ABCD method [16] and dilatometry [15] differ much less. If we determine the relative tensile deformation on the surface of the bitumen ring, based on the fact that the crack width is in the range from 0.0040 to 0.0045 cm [19], and the circumference is 33.9 cm, then the relative deformation is 0.012 %. Meanwhile the deformation in bending according to Fraas is much higher and reaches 1.64 %. This is consistent with the data [25], according to which the difference between the temperature of fracturing according to TSRST and the temperature of the maximum strength for bitumen 60/70 is 8 \(^\circ\)C, and for bitumen modified with polymer is 12 \(^\circ\)C.

With such a variety and difference in the temperatures of the assumed cracking resistance, it cannot be assumed that the stresses leading to cracks will be the same. For this reason, it is difficult to determine the stress value that is objectively linked with \( T_{1.25} \). There is no doubt that \( T_{1.25} \) is the most common low-temperature characteristic of bitumen, since, with a high degree of reliability, it belongs to the straight line in BTDC space originating from the temperature \( T_{300} \), passes through a penetration at 25 \(^\circ\)C, and through a temperature of penetration of 31 dmm. As shown by Hekelom [4], this inclined straight line intersects with isopenetration 1.25 at a temperature that is very close to the Fraas temperature.

To identify the stress at the breaking point temperature (\( T_{Fr} \) and \( T_{1.25} \)), two approaches were used. The first method adopted here for determining the stress at \( T_{1.25} \) is based on calculations [3 and 26] similar to those used in the resistance of materials to determine the stresses in a solid material that appear when an indenter of any shape is immersed in it (Brunel is a ball, Rockwell is a cone, Vickers is a tetrahedral pyramid). In accordance with this calculation, the stress in bitumen at \( T_{1.25} \) is close to a range from 15.6 to 15.9 MPa. In this case, it can be assumed that the stress state is shear. If we proceed from the classical concepts of the ratio of shear and tensile stresses (which is close to bending) and take into account that, during the Fraas test, the bitumen film bends (stretches), then it can be expected that \( \sigma_{bend} \) can be close to 48 MPa. Naturally, this is possible when Poisson's ratio is 0.5.

With regard to bitumen in a solid state, this is not an indisputable fact. Comparison of the moduli of elasticity in shear by bending and bending of cantilever beams of asphalt concrete in [24] showed that the complex modulus in bending is 2.2–2.5 times greater than in shear. This means that Poisson's ratio is close to 0.25.

At the same time, the review of the temperature dependences of Poisson's ratios of bitumen given in [27] shows that this coefficient decreases from 0.48–0.50 to 0.15–0.18 with a decrease in temperature from 60 \(^\circ\)C to 4 \(^\circ\)C. In addition, data on the dependence of Poisson's ratio on frequency are given in [28] for asphalt concrete, and its value decreases to 0.2. Meanwhile, Filippov [29] admits that at temperatures close to glass transition, the shear and tensile stresses of bitumen can be taken as equal. However, the editor of the Russian translation [29] claims that the author is mistaken. Thus, the probable values of the bending resistance at \( T_{1.25} \) can be 16 MPa and 48 MPa. The literature contains numerous data on various values of the shear moduli of bitumen on the plateau, their dependence on temperature from 600–700 MPa [21] to 1800 MPa [28] and 3000 MPa [2]. Naturally, such a variance of the shear moduli and, accordingly, the shear stresses corresponding to them makes the task of choosing an objective stress value practically unresolved.

In this regard, the second approach was chosen to identify the stress at \( T_{1.25} \). It is based on
the assumption of the glass transition theory that the temperature corresponding to the transition of a viscoelastic body to the solid state can be determined from the temperature–frequency dependences of the rheological characteristics, since the glass transition is associated with relaxation processes. It is the relaxation mechanism of deformation of rheological bodies that forms the basis of the WLF superposition method [30], which is ideally adapted to bitumen. This opens up the possibility of using the results of dynamic tests of bitumen available in the literature to substantiate the stresses at their glass transition temperature.

For this, it is advisable to consider typical temperature–time dependences. They reach a plateau of maximum modulus values at low temperatures and high frequencies. At the same time, according to Van der Poel [2], a plurality of bitumens with different penetrations, softening point temperatures and penetration indices come to one plateau of stiffness moduli. In [2], 47 bitumens were considered with softening point temperatures from 39 °C to 116 °C and penetration indices from –2.6 to +5.5. Each of these bitumens, at a single frequency of 200 Hz, reached the same plateau at different temperatures. These temperatures were subsequently considered as one of the criteria for assessing the glass transition temperature of bitumen [14].

The stiffness modulus set in [2] in this way was equal to 3·10⁹ Pa. The features of the experiment that could affect this critical value of the modulus include: nonharmonic, but cyclic loading and sufficiently large stresses that transfer the object to a nonlinearly deformed state, which was noted by the author himself. In order to calculate the stresses at the breaking point temperatures of bitumen, Van der Poel took the relative deformation that bitumen undergoes when the plate of the Fraas device is bent, equal to 0.0164. These data can be used to determine stresses at $P = 1.25$ dmm. With a stiffness modulus of 3000 MPa and an assumed bending deformation of the plate of 0.016, the corresponding stress is 48 MPa. To solve direct and inverse problems of predicting the viscoelastic behaviour of asphalt concrete in [28], the value of the complex shear modulus found with the DSR method was taken as equal to 1800 MPa.

To be convinced of the validity of the application for the assessment of the stress corresponding to the plateau of temperature–time deformation given in different literature sources, 57 values of complex moduli were selected. The average value of the complex moduli at shear is close to 33 MPa.

Thus, it became possible to decide which of the stresses corresponds to the breaking point temperature to a greater extent. For this purpose, logarithmic stress isolines were plotted on the shear stress temperature diagram (Figure 3). Corresponding to temperatures: the softening point according to $T_{800}$ is 1.5·10¹³ Pa, $T_{25}$ is for the assessed bitumen, $T_{31}$ is 2.1·10⁵ Pa, $T_{Fr}$ is 48 MPa and the average of 57 stress values corresponding to the exit of complex modulus to a plateau is equal to 33 MPa. To illustrate the procedure for using the diagram, the data on bitumen of three structural types given in [28] are plotted on it. The use of data from an independent source is undertaken in order to ensure the objectivity of the proposed solution.

![Graph](image-url)
The temperature–shear straight line of each bitumen crossed the stress isolines at two temperatures spaced by 2–3 °C, depending on the bitumen penetration index: the higher the penetration index (the straight line is flatter), the greater the temperature difference at the intersection points. The values of the cracking resistance temperature obtained at these points were compared with the temperatures experimentally found with the Fraas method (Figure 4, Table 2). This comparison showed that the smallest discrepancy between the experimental and calculated breaking point temperatures corresponds to an iso-temperature stress of 33 MPa. The relative position of the Fraas breaking point temperatures and those obtained from the diagram is shown in Figure 4 and in Table 2.

Table 2 – The comparison of the breaking point temperatures, obtained with experimental and settlement-graphic methods

<table>
<thead>
<tr>
<th>Bitumen</th>
<th>Penetration at 25 °C, dmm</th>
<th>Temperature at 800°C</th>
<th>Temperature at penetration 31 dmm, °C</th>
<th>Fraas breaking point temperature, °C</th>
<th>Temperature at 33 MPa, °C</th>
<th>Temperature at 48 MPa, °C</th>
<th>Stress at penetration at 25 °C by method [12], MPa</th>
<th>Stress at penetration at 25 °C by Figure 3, MPa</th>
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<tr>
<td>Золотарев [29]</td>
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<td>−24</td>
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<td>32</td>
<td>63</td>
<td>49.5</td>
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</table>

Fig. 4. Dependency between Fraas breaking point temperature (T_F) and estimated temperatures of cracking resistance at 33 MPa (Δ) and 48 MPa (▲)

The table contains the data of the bitumen studied by the author and given in [28]. It presents the breaking point temperatures obtained by the Fraas method, temperatures at stresses of 33 MPa and temperatures at 48 MPa. In all cases, the breaking point temperatures at the average stresses on the plateau of temperature–frequency dependences (33 MPa) are very close to the Fraas temperatures. This stress is very close to the 30 MPa taken as calculated for bitumen in [28]. In addition, the table includes stresses at penetration at 25 °C, calculated according to the method of [3 and 12], and found with Figure 3. Their values differ in the range from 9 % to 26 %.

Conclusions

(1) At present, the traditional technical characteristics of bitumen and bituminous binders are recognized as empirical and do not have a convincing mechanical meaning. The most meaningful and promising in this respect is the depth of the needle penetration, if we approach it as a characteristic of the resistance of bitumen to the immersion of an indenter of a given shape and size into it. The conversion of the penetration index into the dimension of shear stress, undertaken in this and previous works of the author, became possible thanks to the studies in the 1960s by Carre and Laurent and their conversion of penetration into shear viscosity.

(2) The present work is based on the fact that all the temperature characteristics of bitumen refer to a certain shear stress: the softening point temperature by T_{500} refers to a shear stress close to 1500 Pa; the temperature 25 °C is a stress at the penetration value; temperature T_{31} is the stress, which is the same for bitumen of the same penetration, but of different structural types with
different penetration index; temperature $T_{1,25}$ is the stress of brittle cracking. The stress at $P = 1.25$ dmm requires a special approach.

(3) Currently, there are many methods for determining the cracking temperature of bitumen. For bitumens of the same consistency (for example 35/50), these temperatures, depending on the method, are arranged in the following sequence: according to Fraas (-11 °C), BBR (-16 °C), TSST (-24 °C), ABCD (-27 °C), dilatometry (-27.5 °C). The most objective and physically justified methods are dilatometry, TSST and ABCD. However, the Fraas breaking point temperature is the most common and well-studied. Consequently, its assessment by the parameter of mechanical stress at the present time remains relevant and expedient. It is shown in the work that the shear stress of bitumen at the Fraas temperature ($T_{1,25}$) is close to 35 MPa, which refers to a complex modulus of 2000 MPa on the plateau of its temperature–frequency dependence.

(4) On the basis of ideas about the temperature states of bitumen, estimated by the shear stress, a generalised linear semi-logarithmic–shear dependence is developed, on which all the stresses at the softening point temperature $T_{50}$, temperature 25 °C, equi-penetration temperature $T_{31}$ and Fraas breaking point temperature fit. With this dependence, knowing any of the characteristic temperatures, it is possible to find the referred shear stress.

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des enrobés bitumineux à basse température. 


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