

PERFORMANCE TESTS ON BITUMEN AND BITUMINOUS MIXES

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In this article, the Authors intend to highlight how it is now possible to have a more engineering-based approach to designing and constructing bituminous road paving, intervening in both definition of the bitumen mix and in choice of the binder which is its active component.

The empirical tests used until now - and which have given excellent results over time, keeping under control groupings of requisites obtained from the experience gained over the years - are no longer sufficient for supporting the leap in quality of paving. Paving now has to have increasingly extreme and durable performance over time, due to the continual development of road traffic (in terms of frequency, weight and load time).

Test methods and the relative laboratory equipment currently exist, all complying with standards, which allow performance of tests to identify the performance requisites which are necessary to guarantee extreme and durable quality of the surfacing. This approach, which has been used in the USA for over a decade through the SuperPave system, as part of the SHRP research program, should hopefully also now be introduced into Italy, following definitive publication of the relative European standards.

Three levels of interpretation may be identified in this article. The first, which is the most practical, consists in identifying the limits imposed on the requisites of certain tests, performed on bitumen and its mix, as a function of the minimum and maximum operating temperature, the type and frequency of traffic of the specific road. Deterioration of paving is, in fact, mainly attributable to three things: ruts (permanent deformation due to temperature), fatigue cracking (at moderate temperatures) and cracks due to rigidity at low temperatures.

Certain performance requisites of bitumen and the mix correspond with these phenomena and must therefore be kept under control.

The second level of interpretation consists in understanding and interpreting the laboratory tests conducted and the engineering-based use of the results obtained.



Figure 1 - Dynamic tests: set-up for the four-point bending fatigue test (Standard UNI EN 12697:24 - Annex D)

The third level of interpretation, more specialistic and directed towards laboratory technicians, is useful for anyone who must conduct tests in accordance with the texts of existing standards and the equipment currently available. However, the authors will only make a brief mention of this in the boxes, since the principal purpose of this article is to provide, in a simple, and therefore incomplete manner, obviously for reasons of space, an interpretation destined for designers, firms and works managers.

We will discuss the two topics (mixes and bitumen) separately below, each to be interpreted with the three methods of interpretation indicated above.

Bituminous mixtures

The intention of harmonised European standards EN 13108 on bitumen mixes is that the specifications thereof are given in terms of fundamental properties, identified on the basis of product performance during use. EN 13108 is formed of 11 parts, of which only part 1 (for the time being) is considered here, because it refers to the type of mix most widely used and defines, more completely than the others, how to specify the product¹.

The standard, recognising that making a performance-based approach to the specifications immediately and completely obligatory would not be appropriate, as things stand, in view of the different levels of experience currently present in Europe, allows asphalt concrete to be defined according to two approaches:

- An empirical approach, where the mix is specified and classified with constitutive formulas, with clearly defined dosages and with constituent material requisites and final product requisites. These final product requisites have been identified, following decades of experience, in correlation with basic “performance related” engineering characteristics, in order to be able to predict product performance;
- A fundamental approach, where the mix is specified directly in terms of requisites of basic “performance based” engineering characteristics (such as stiffness, fatigue strength and resistance to permanent deformation), which allow product performance to be predicted. The restrictions on composition and the constituent materials are limited, so a greater degree of freedom in product formulation is possible.

The two approaches have some similar general requisites, of which the most significant ones are: the granulometric composition, the percentage of air voids in the compacted mix, the water-sensitivity (EN 12697 - 12) and the susceptibility to deform, tested using the “Wheel tracking test” method (EN12697 - 22), which is linked to performance.

Fundamental Tests (“performance based”)

Road traffic is continuously developing and paving may be severely tested by the increasingly intense dynamic stress to which it is subject. Consequently, empirical tests on the product may no longer be sufficient to support a leap in quality. As mentioned, in performance-based tests, it is not necessary to comply with a detailed classification of the mix composition, as in the case of the empirical approach. Although the granulometric composition must comply with general requisites, a certain deviation from the limits set is allowed. For example, it is sufficient that the percentage of bitumen be guaranteed with a minimum of 3% or that the oscillation in the percentage of air voids be up to 3% on the declared value.

The specificity of the approach is provided by the introduction of tests which measure the physical magnitudes directly linked with the performance characteristics. The most important are:

- stiffness, EN 12697 - 26;
- resistance to fatigue, EN 12697 - 24;
- cyclic compression test, EN 12697 - 25, for determination of the resistance to permanent deformation.

A short mention to pr EN 12697 - 46 “Test methods for hot mix asphalt. Part 46: Low temperature cracking and properties by uniaxial tension tests”.

The standard still all the status of provisional, considers the resistance of compacted bituminous mixes to cracking due to low temperature.

The general test configuration is uniaxial tension by which various test parameters are achieved as:

- The tensile strength versus temperature, by uniaxial tension stress test (UTST);
- The failure by thermal stress restrained specimen test (TSRST);
- The tensile strength reserve test, by combination of TSRST and UTST;
- The relaxation time test (RT);
- The creep curve for back calculation of the rheological parameters by tensile creep test (TCT);
- The fatigue resistance test by combination of cryogenic and mechanical loads by Uniaxial Cyclic Tension Stress (UCTST).

These dynamic tests are conducted by automatic machines which operate both under load control and strain control (microstrain) and which are contained in a thermostatic cabinet (Figure 1).

It should be emphasised that the bitumen mix, as a viscoelastic material, behaves in a manner particularly dependent upon temperature and load in terms of load type, frequency and application time; these parameters must therefore be well-defined in the tests.

The methods of conducting the tests are therefore described, with boundary conditions defined by the standards. The designer may take further boundary conditions into account, as a function of the specific characteristics of the work and the relative values of the requisites.

EN 12697 - 26: Stiffness test

When it is stressed by dynamic loads, a compacted bitumen mix is characterised, within the limits of deformation, temperature and load application times, by basically linear viscoelastic behaviour, although always accompanied by a plastic component, at the operating ambient temperatures.

For small deformations, of the type to which road pa-

ving is subject, the material, subjected to repeated loads - which may be applied in an alternate time sequence, for example of the sinusoidal type - tend, on release from the load and irrespective of its entity, to return to the original configuration, although with a time delay. This behaviour is dependent upon the temperature and the load application time. At high environmental temperatures, a plastic-type component of deformation, i.e. permanent, is accentuated, and the same applies with prolonged times or at low load application frequencies. With dynamic application of the load, which may be the sinusoidal type, simulating the repetitive loads of traffic in the laboratory, at ambient temperatures, the Complex Modulus E^* is determined, which represents the relationship between the unit load applied and

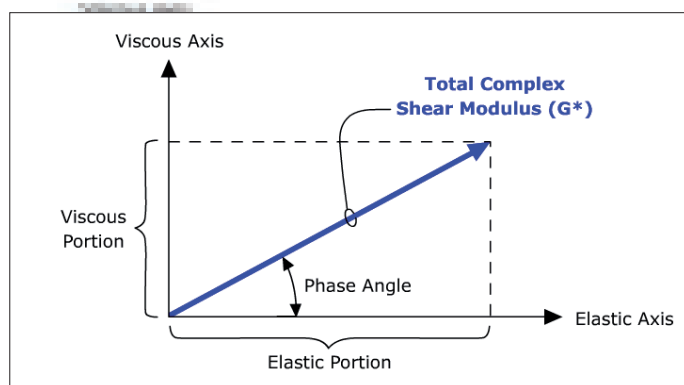


Figure 2 - Complex Modulus E^* (bitumen mixes), G^* (bitumens)

viscoelastic deformation.

With representation of the Complex Modulus on a graph, with Cartesian axes, for immediate evidence, the elastic component $E_1 = |E^*| \cos(\Phi)$ is placed on the x-axis and the viscous component $E_2 = |E^*| \sin(\Phi)$ on the y-axis. Angle Φ , the phase angle, represents the time delay caused by the viscous component E_2 . Complex Modulus E^* is represented by the vector resulting from the two components E_1 and E_2 and the absolute value of its Modulus $|E^*|$ represents stiffness value "S", the subject of the standard (Figure 2).

A common characteristic of this performance-based test is the possibility of application of the principle of overlapping of the stiffness/frequency curves, at a fixed temperature (isotherms) or the stiffness/temperature curves, at fixed frequency or constant load application time (isochrones). Thanks to this possibility, master curves may be created which allow the operator, once a limited number of tests have been conducted under different frequency and temperature conditions, to perform continual assessment of the modulus within a relatively wide range of temperatures and load application times (Figure 3), by shifting the experimental curves (isochrones or isotherms) in a manner to prolong

one of the experimental curves, used as a reference (shifting).

EN 12697 - 26 includes several methods with different test configurations (see Box 1).

As indicated in the scope of the standard, use of these procedures is aimed at classification of the formulations of compacted bitumen mixes in terms of the stiffness modulus as a guide to estimation, by designers, of structural behaviour and, once the test configuration has been chosen, it may be used as a means of control of contractual specifications. It is not expected for the modulus values obtained to be univocal, whatever the procedure. There may, in fact, be considerable differences between the modulus values obtained with different test configurations on the same compacted mix.

This apparent incongruity appears to be justified by the considerable difference in the shape and dimensions of the test specimens, which influences calculation of the modulus. In this regard, studies exist which have identified correlations².

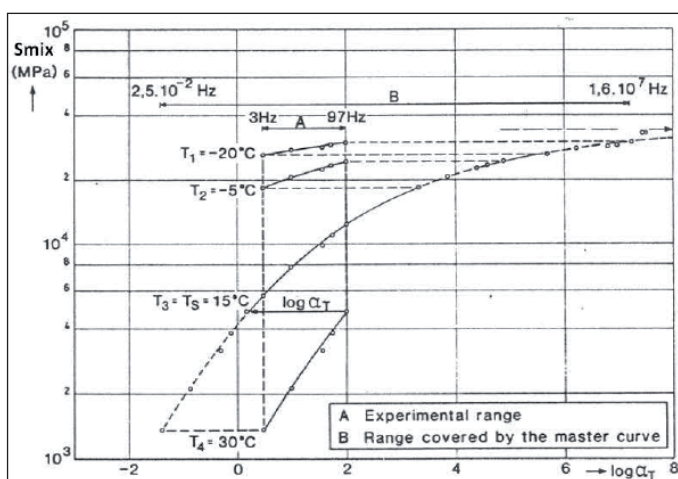


Figure 3 - The stiffness/temperature master curve (isochrone)

EN 12697 - 24: Fatigue strength test

The fatigue strength test consists in assessment of the number of pulses, usually of the sinusoidal type, necessary to achieve a specific reduction in resistance. In the case of dynamic tests conducted with preset deformation (controlled deformation), the criteria consists in counting the pulses, obviously lower than the ultimate tensile stress, necessary to reach a given decrease of the stiffness modulus compared with the initial one of the test, usually 50%; in contrast, for dynamic tests conducted with a preset load (controlled load), the criteria consists in counting the pulses, obviously also in this case lower than the ultimate tensile stress, necessary to reach a specific increase in deflection of the test specimen, compared with the one found at the start of the test, which is usually double.

Currently, as required in the 2010 edition of EN 12697

EN 12697 - 26 deals with several dynamic test configurations, alternatives to each other. These include the type tests contemplated by EN 13108 - 20, which are:

- two-point restricted cantilever bending test (shelf type) on trapezoidal test specimens, “2PB-TR” with application of a sinusoidal load (Figure 4). The sinusoidal load frequencies vary between 3 Hz and 30 Hz (EN13108 - 20 indicates 10 Hz at 15°C) with deflection set at less than 50 microdeformations. The measured parameters are the applied force, the deflection measured at the top of the trapezoidal specimen and the phase angle. The measurements acquired in the last 10 s of the test, the duration of which may vary between 30 s and 2 min., provide the data for calculation of stiffness;
- four-point beam loading test, “4PB-PR”, with application of a sinusoidal load. The prismatic specimen, generally with dimensions 380x50x50 mm, is mounted on an accessory which contains and restricts the beam in four points. Under controlled deformation, a sinusoidal force is usually applied such as to ensure the amplitude of oscillation of deformation of the joist is 50 +/-3 microdeformations. The load

frequencies may vary between 2 and 30 Hz (EN 13108 - 20 indicates 8 Hz at 20°C). The parameters measured are force, deflection and phase angle measured between 45th and 100th of the application cycle of the load for calculation of the stiffness modulus;

- indirect tensile stress test with repeated pulses on cylindrical specimens, “IT-CY”, with application of load pulses of a quarter-wave (Figure 5).

A specific accessory is used to apply vertical diametral compression in pulses to the cylindrical specimen, with a diameter of 100 or 150 mm and thickness varying between 30 and 75 mm. The consequent horizontal diametral expansion is measured. The application time of the pulse is equal to a quarter-wave and the peak is reached in 124 +/-4 ms. The peak values of induced horizontal deformation are around 5/10 microns. EN 13108 - 20 indicates a test temperature of 20°C. The diagram provided in real time shows the trend of five pulses of force applied and the relative horizontal deformation curves (Figure 6) and corresponding stiffness values.



Figure 4 - The test accessory 2-point bending stiffness. EN 12697 - Annex A



Figure 5 - The test accessory. For Stiffness by indirect tensile test UNI EN 12697:26 - Annex C

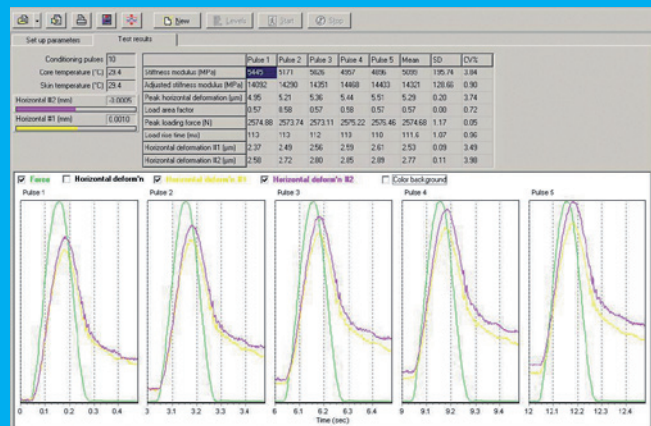


Figure 6 - EN 12697:26 Stiffness - Annex C: indirect tensile test

- 24, the assessment of fatigue strength may also be quantified on the basis of counting of the pulses as a function of dissipated energy. Fatigue strength is usually indicated as the deformation value for which decay of the compacted mix is reached at 10⁶ cycles, indicated as ε⁶ (see Box 2, page 5).

A characteristic of these performance-based tests is being able to construct “Fatigue lines”, diagrams which represent decay of the fatigue strength value as the deformation set increases (Figure 7).

Recent empirical-mechanistic studies have highlighted and rationalised, through fatigue tests, the

phenomenon of self-healing of the micro-cracks which form in the compacted mix (“Healing”).

This contributes to better understanding of development of damage from fatigue and to bringing it closer to the real behaviour of the paving. The Dutch Guideline on road building published by the “CROW National Information and Technology Centre for Transport and Infrastructure, Ede, Holland” currently includes, among the corrective empirical factors for calculation of the project lifetime of bituminous paving, an empirical corrective factor for “healing”, based on the penetration value of the bitumen used.

Resistance to fatigue of the mix Box 2

The fatigue test instruments are basically identical to the ones used for determination of the stiffness modulus (UNI EN 12697 - 26). The accessories and the software installed allow performance of the following fatigue tests:

- two-point bending test, with trapezoidal specimen, with application of a sinusoidal load;
- restricted four-point bending test, with application of a sinusoidal load;
- indirect tensile stress test on a cylindrical specimen.

UNI EN 13108 - 20: "Type-testing" indicates the first two test methods, with the following conditions: the two-point bending test, at temperatures of 10°C and 15°C, with frequency of 25 Hz and 30 Hz respectively, and the four point beam loading test at a temperature of 20°C with frequency of 30 Hz.

EN 12697 - 25: Cyclical compression test (for determination of resistance to permanent deformation)

This determines susceptibility to plastic deformation of the mix. The standard deals with two test methods for determination of resistance to permanent deformation of a cylindrical specimen with a diameter of 150 mm and a height of 60 mm, subjected to repeated cycles of vertical stress. Measurement consists essentially in recording of deformation of the specimen in relation to the number of vertical pulses applied (see Box 3, page 6).

The value considered for classification is the "creep rate" f_c , considered in the median part of the deformation curve, expressed in $\mu\text{m}/\text{m}/\text{n}$, with $f_c = B_1 \times 10^4$, where B_1 represents the gradient of the median section of the curve considered as almost straight.

Indicative values for performance-based specifications of hot bituminous mixes (EN 13108 - 1)

Depending on the empirical or performance-based approach followed by the manufacturer for characterisation of the rolled compacted mix, the CEN TC 227 Commission on "Road materials" has established specifications which contribute to satisfying the required performance characteristics.

These include stiffness, resistance to fatigue and resistance to permanent deformation. These specifications may be defined, in the empirical approach, by empirical requisites correlated with performance, or by performance-based requisites, according to the performance-based approach.

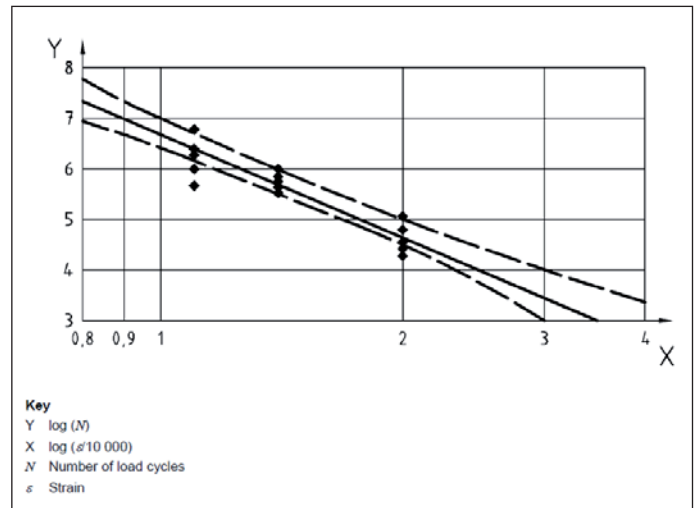


Figure 7 - Fatigue lines of bitumen mixes

These requisites, in EN 13108 - 1, are in turn divided into categories of values, with the exception of temperature, for which minimum and maximum values are established according to the consistency (penetration) of the bitumen used.

In the performance-based approach, together with the general requisites, there is a fundamental requisite based directly on stiffness according to the methodology of EN 12697 - 26. The requisite of "Stiffness" is expressed in categories of minimum values, from 21,000 to 1,500 MPa (13 ranges) and into categories of maximum values, from 30,000 to 7,000 MPa (eight ranges).

The same applies for resistance to permanent deformation, with introduction of the fundamental characteristic linked with measurement of the creep rate f_c , classified into categories, by means of triaxial test according to UNI EN 12697 - 25 and for resistance to fatigue with introduction of values of the performance-based characteristic, divided into categories of decreasing deformation values set at 10^6 cycles (ϵ^6), following EN 12697 - 24. In the laboratory, the manufacturer defines and validates the "mix design" with the formulation of a mix with requisite values which come within those required by the categories indicated.

Table 1 shows the value ranges of the stiffness, fatigue strength and strain strength modulus of several of the types most widely used mixes.

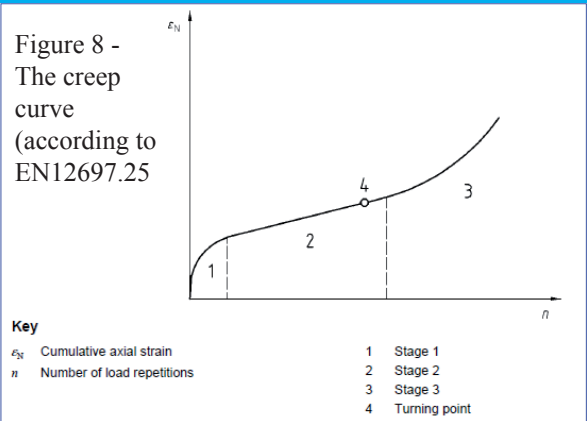
It is emphasised that the values shown, extrapolated from tests and experiments conducted by the laboratories of Elletipi Srl, are statistical data and therefore only indicative. They should therefore only be used as a reference of a dated mix and must take into account the mix design chosen in order to be used as a specification. Table 2 also shows the values of the aforementioned characteristics from the guide cited which adopts the performance-based approach required by EN 13108-1 for hot bituminous mixture.

Of the two methods, A and B, the test set-up “B” is cited, since it is the only one contemplated by UNI EN 13108 - 20. This method involves a real cyclical triaxial test, in which the specimen, introduced into a triaxial cell and confined laterally by pneumatic or hydraulic radial pressure, which may vary between 50 and 200 kPa, is subjected to cyclic axial vertical pressure of the sinusoidal type, with amplitude from 100 to 300 kPa and frequency from 1 Hz to 5 Hz, or pulsating vertical pressure of the block type, load square wave with release phase, with intensity from 100 to 700 kPa and frequency of 0.5 Hz. The number of pulses applied is equal to 10,000 and thermostating varies between 30°C and 50°C.

The “creep” curve is constructed, which shows three phases: phase 1, the initial phase, where the gradient of the curve decreases as the pulses increase; phase 2, the central phase, where the gradient is practically constant and has a concave point of inflection in its final part; phase 3, the final phase, where the gradient of the curve increases with the number of pulses, indicating that the mix is collapsing (Figure 8).

A mix being examined which has a long section and a low gradient of phase 2 indicates good resistance to permanent deformation.

The values of f_c are determined at preset test conditions. For example, according to UNI EN 13108 - 20, for the surface layer, the values indicated are 15°C for conditioning of the specimen, 50°C for the test, 150 kPa confinement pressure in a triaxial cell, 300 kPa axial peak load, 3 Hz for sinusoidal application or 0.5 Hz for application of the load in blocks (1 s load, 1 s release).



Bituminous mix type	Stiffness UNI EN 12697 - 26	Resistance to fatigue UNI EN 12697 - 24	Resistance to permanent deformation UNI EN 12697 - 25
	Test configuration ITCY@ 20°C S	Test configuration 2PB @10°C, 25 Hz 4PB @20°C, 30 Hz ϵ^6	Test configuration dynamic creep @50°C f_c, ϵ_n, E_n
Base-binder-surface with unmodified bitumen	3.500 - 6.000 MPa	$60 < \epsilon^6 < 90 \mu\epsilon$	$0,1 < f_c < 0,8 \mu\epsilon/\text{cycle}$ $0,05 < \epsilon_n < 0,6 \mu\epsilon$ $35 < E_n < 70 \text{ MPa}$
Base-binder-surface with modified bitumen	4.000 - 11.000 MPa	$80 < \epsilon^6 < 130 \mu\epsilon$	$0,1 < f_c < 0,4 \mu\epsilon/\text{cycle}$ $0,05 < \epsilon_n < 0,6 \mu\epsilon$ $15 < E_n < 40 \text{ MPa}$
Binder AM	6.000 - 13.000 MPa	$120 < \epsilon^6 < 220 \mu\epsilon$	-
SMA	2.500 - 4.000 MPa	$50 < \epsilon^6 < 75 \mu\epsilon$	-
Porous bituminous mix	800 - 1.600 MPa	-	-

Table 1 - The value ranges of the stiffness, resistance to fatigue and resistance to permanent deformation of several types of mix, where:

S = stiffness in MPa; ϵ^6 = value of microdeformation through interpolation at 10^6 cycles;

ϵ_n = cumulative deformation at cycle n; E_n = creep modulus; f_c = creep rate in $\mu\epsilon/\text{cycle}$

Bitumens

As known, bitumen is the active binder of the bitumen mix. It is present on average as 5% of the mixes and determines their rheological behaviour to a large extent. The following European standards are currently in force on bituminous binders.

UNI EN 12591:2009 - Specifications for bitumens for road applications, which deals with bitumens by dividing them into three classes: bitumens with a penetration at 25°C between 20 and 330 dmm; bitumens with penetration at 25°C between 250 dmm and 900 dmm; soft bitumens with kinematic viscosity at 60°C between 1,000 and 16,000 mm²/s.

EN 13924:2006 - Specifications for hard bitumens for paving, which deals with bitumens with a penetration range at 25°C of 10/20 dmm and 15/25 dmm. This standard may be considered as an addition to UNI EN 12591:2009.

“EN 14023:2005 - Specifications for bitumens modified with polymers (reviewed standard, with the 2010 edition due for publication).

These three framework standards are structured to express specifications corresponding with the following essential requisites, as required by Mandate EEC M/124 relating to building products, which are:

- consistency at intermediate operating temperatures (ambient temperatures);
- consistency at high temperatures;

- fragile behaviour at low temperatures and durability, intended as resistance to ageing. It should be noted that UNI EN 14023:2005, modified bitumens, also includes the cohesion requisite.

Other requisites are included, such as inflammability; kinematic viscosity at 135°C, solubility (only for hard bitumens EN 12591:2009) and Fraas breaking point (for hard bitumen EN 12591:2009 and for bitumen modified with polymers EN 14023:2005). The plasticity range and storage stability are requisites reserved only to modified bitumen.

The current standards comply with the requisites indicated above with traditional type tests, the acceptance values of which are determined by experience.

New European standards have been published recently and include CEN standards on performance-based test methods which examine several rheological and dynamic magnitudes of the material.

These test methods have contributed to development of models, experiments and validation performed in Europe and the USA. In the near future, this standard will join the empirical standards currently in force and will eventually replace them in the activity of formulations of high-quality mixes, in response to the essential requisites laid down by Mandate EEC M/124.

The CEN is currently defining future specifications for “complex” binders, i.e. binders for which traditional tests are not sufficient for describing the characteristics in a complete way.

Bitumen mix type	Stiffness EN 12697 - 26 Test configuration 4PB @ 20°C, 30 Hz		Resistance to fatigue EN 12697 - 24 Test configuration 4PB @ 20°C, 30 Hz		Resistance to permanent deformation EN 12697 - 25 Triaxial test configuration method B block pulses, 0,4/0,6@50°C confinement 150 kPa, axial load 300 kPa	
	S		ε ⁶		fc	
	Statal Road	Motorway	Statal Road	Motorway	Statal Road	Motorway
Base for traffic speed with VA > 2.500	7.000 - 14.000 MPa	Minimum value to report - 17.000 MPa	≥ 90 με	Value to report	≤ 0,4 με/cycle	≤ 0,4 με/cycle
Binder for fast traffic with VA > 2.500	5.500 - 14.000 MPa	5.500 - 17.000 MPa	≥ 80 με	≥ 70 με	≤ 0,4 με/cycle	≤ 0,4 με/cycle
Surface layer for fast traffic with VA > 2.500	5.500 - 11.000 MPa	5.500 - 14.000 MPa	≥ 100 με	≥ 100 με	≤ 0,6 με/cycle	≤ 0,4 με/cycle
Layer support for draining layer with VA > 2.500	5.500 - 14.000 MPa	5.500 - 17.000 MPa	≥ 80 με	≥ 80 με	≤ 0,4 με/cycle	≤ 0,4 με/cycle

Table 2 - The values of the characteristics for hot bitumen mixes indicated by the Dutch Guideline, where: VA = intensity of lorry traffic defined as the number of lorries per day per carriageway; ε⁶ = value of microdeformation through interpolation at 10⁶ cycles; S = stiffness in MPa; fc = creep rate in με/cycle

Performance-based or fundamental tests

These methods, already in circulation as EN Standards, may be used, in a coming review of the framework Standards, to satisfy the essential requisites listed here:

Consistency at high temperatures (EN 13702:2010)

Consistency, intended as viscosity, must be measured with test methods which take into account the non-linear variability of viscosity as a function of temperature. Standard UNI EN 13702:2010 adopts the “cone and plate” method for determination of dynamic viscosity in a temperature range.

In the test instrument (Figure 9), the sample of bitumen is placed on a lower circular plate; an upper plate with a conical surface is pressed into the sample after the thermal conditioning to the test temperature chosen.

The lower plate rotates at a given speed selected; a torque is generated and the instrument measures dynamic viscosity η which is given by $\tau/\dot{\gamma}$, where $\dot{\gamma}$ is the shear speed expressed in s^{-1} and τ is the shear stress expressed in Pa, which is calculated by:

$$\tau = A \times M_d$$

where:

A = the factor of cone shape selected expressed in m^{-3} ;

M_d = the torque expressed in $N \times m$.

The instrument is equipped with a series of conical plates at various angulations, to be selected together with the rotation speed, in a manner that the torque is included in the measurement capacity of the instrument and it is thereby possible to determine the binder viscosity.



Figure 9 - The cone and plate viscometer (with reference to EN 13702 (GARDCO - Brookfield))

The temperature range for measurement of viscosity is between 60°C and 150°C. Three tests are usually conducted: at 60°C with shear speed $5 \times 10^{-2} s^{-1}$; at 100°C with shear speed $5 \times 10^1 s^{-1}$; at 150°C with shear speed $5 \times 10^2 s^{-1}$. This is a performance-based test, because it satisfies production needs of spreading and rolling of the bitumen mix. Especially for “hard” modified bitumen, or in the case of use of recycled mix containing naturally hardened bitumen, viscosity is relatively high at those temperatures, and therefore requires a precise assessment to optimise the dosages and performance methods, since low viscosity values may assist working. A highly unusual characteristic of the “cone and plate” method is the particularly small quantity of sample to be submitted to the test and the rapidity of performance. For a higher “range” of temperatures, which may be between 40°C and 230°C, a rotary viscometer is used with cylindrical rotating test chamber and spindle, according to EN 13302:2010.

As for the “cone and plate” viscometer, the magnitude measured is dynamic viscosity and the same units of measurement are adopted ($Pa \times s$).

The instrumentation is also equipped with a thermostating unit and a series of rotary cylindrical spindles, each with its own shape factor.

The test is conducted at various temperatures and shearing speeds.

A typical purpose of the test is assessing viscosity of bitumen in the liquid phase to check that “handling” requirements, such as pumping and spraying, are satisfied.

Behaviour at operating temperatures (EN 14770:2006)

The dynamic rheometer DSR (Dynamic Shear Rheometer), EN 14770, examines the bitumen from a rheological viewpoint, as a response to dynamic stress, at normal operating temperatures of the paving, which may range between 5°C and 85°C. Like the compacted bitumen mix, a prevalently linear viscoelastic behaviour is found, although always accompanied by a plastic component, when the bitumen is stressed by dynamic loads at the typical temperatures and stress of road paving, within deformation, temperature and load application time limits. With the application of sinusoidal loads, simulating the respective loads of traffic in the laboratory, at environmental temperatures, the Complex Modulus G^* is determined, which represents the relationship between the unit load applied and the viscoelastic deformation: $G^* = \tau^*/\gamma^*$ where τ^* is the sinusoidal force expressed in Pa and γ^* is the sinusoidal deformation.

With representation of the Complex Modulus on a

graph, with Cartesian axes, the elastic component $G' = |G^*| \cos(\delta)$ is placed on the x-axis and the viscous component $G'' = |G^*| \sin(\delta)$ on the y-axis. Angle (δ) , the phase angle, represents the time delay caused by viscous component G'' . Complex Modulus G^* is represented by the vector resulting from the two components G' and G'' and the absolute value of its modulus $|G^*|$ represents the norm of complex modulus G^* , which is the ratio between the peak unit load and peak deformation (Figure 2).

The DSR equipment consists in a thermal conditioning unit and a system which generates torsional oscillation, under controlled force or under controlled deformation, between two parallel test plates spaced apart from each other, containing the sample to be analysed. There are two diameters for each pair of plates: 8 mm or 25 mm, to be chosen depending on the consistency of the bitumen and the range of measurement of the instrument. Operating at various oscillation frequencies, the instrument can measure G^* in the range from 1 kPa to 10 MPa and the phase angle δ from 0 to 90°.

Performing a series of measurements at temperature steps of 10°C and frequency steps, usually spaced by decades, isotherm curves are constructed, $|G^*|$ and δ versus the frequency, and isochrone curves, $|G^*|$ and δ versus the temperature. Formation of permanent deformation is associated with dissipation of energy during oscillation. Under constant and controlled force, σ_0 , the following is obtained :

$$W_c = \pi \sigma_0^2 \frac{1}{G^* \sin(\delta)}$$

("Dynamic shear rheometer"
 da PAVEMENT INTERACTIVE
 University of Washington)

Therefore, for low values of the elastic component $|G^*| \cos(\delta)$, there will be a large amount of dissipated energy W_c and a clear aptitude for permanent deformation (formation of rut). In any case, an excessive characteristic of stiffness of the bitumen may lead to cracks caused by fatigue, so it is opportune to maintain a minimum viscous component $|G^*| \sin(\delta)$ (see Box 4).

Behaviour at low temperatures (EN 14771:2005 - EN 13587:2010)

The standard EN 14771:2005 - determination of the bending stiffness modulus using the BBR (Bending Beam Rheometer) - allows the stiffness and the aptitude for relaxation of bitumen at very low temperatures to be determined.

The test instrument subjects the specimen, consisting in a prismatic bar with measurements 6.25x12.7x127 mm, under static bending stress on three points of approximately 1,000 mN, with measurement of deflection at the test temperatures. Test temperature obtained by refrigerated liquid, ranges from -36°C to 0°C .

Behaviour of finders at operating temperatures

Box 4

In this regard, it is possible to consider another two standards with test methods based on the DSR:

- CEN/TS 15325 - Determination of viscosity under ZSV ("Zero Shear Viscosity") through shear tests under a constant load. The test consists in the static application of a very small torque, which may be 50 Pa, at temperatures of 45°C or 50°C and measurement of the corresponding deformation after stability of deformation has occurred. The deformation speed $d\gamma/dt = \sigma/\eta$ allows measurement of η_0 , absolute viscosity in Pa x s in a Newtonian field. The test is considered as a useful contribution to prevention of permanent deformation;
- CEN/TS 15324 - Determination of the equiviscosity temperature based on the Low Shear Viscosity (viscosity at low shear gradient) through a dynamic rheometer at low oscillation frequency. The equiviscosity temperature is the typical one of bitumens which have a given value of absolute viscosity, for example 2 kPa x s, measured with a very low given frequency value, for example 0.01 Hz, and with a very small deformation amplitude, for example 0.1. The equiviscosity temperature allows bitumens to be classified directly in relation to thermal susceptibility.

The DSR equipment consists in a thermal conditioning unit and a system which generates torsional oscillation, under controlled force or under controlled deformation. The test may be performed on the virgin sample of bitumen or on bitumen conditioned after ageing operations (RTFOT and PAV). Acquisition of the load and deflection values occurs for a duration of 240 s, with scanning less than 0.5 s. The stiffness modulus is measured on the basis of the values acquired at 8.0 s; 15.0 s; 30.0 s; 60.0 s; 120.0 s and 240.0 s with the formula:

$$S(t) = \frac{PL^3}{4bh^3\delta(t)}$$

where:

S(t) = stiffness on deformation at time t in MPa;

P = the static load in N;

L = the distance between supports in mm (102 mm);

b = the width of the specimen in mm (12.5 mm);

h = thickness of the specimen in mm (6.25 mm);

$\delta(t)$ = deflection of the specimen measured at time t in mm.

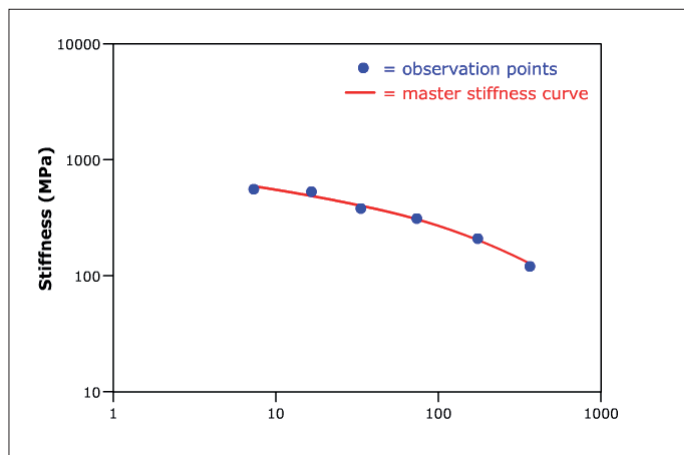


Figure 10 -
The BBR test: the stiffness/time master curve.

A master stiffness curve as a function of load time is obtained (Figure 10).

The gradient of the curve at time t is also expressed, as "m-value". The "m-value" represents a measurement of the rate of release of stiffness of the bitumen with time (see Box 5) The European standard includes another test method for low temperature: EN 13587:2010 determination of the tensile stress characteristics of bitumen with the direct tensile test (DTT) method.

With this method, the resistance and corresponding elongation is measured (ϵ , in percentage deformation) of a specimen in the shape of a handlebar, at a reduced scale of only 75 mm in length, which is subjected to tensile stress at different test speed (1, 10, 50, 100, 500 mm/min.) and conditioned at different temperatures (-10°C, -5°C, 0°C, 5°C, 10°C, 15°C, 25°C). The test ends with fracturing of the specimen or with achieve-

Behaviour of bitumen at low temperature Box 5

The value of the modulus, which is useful for classification, is calculated, by creating a second-degree polynomial curve, using the values of the modulus measured as a function of the acquisition times as follows:

$$\log S_c(t) = A + B \times \log(t) + C \times [\log(t)]^2$$

where:

$S_c(t)$ = bending stiffness calculated at time t, in MPa;

A, B e C = regression coefficients;

t = load maintenance time in s.

The gradient of the curve at time t, "m" (m-value) is calculated by:

$$m(t) = \frac{d \log[S(t)]}{d \log(t)} = B + 2 \times C \times \log(t)$$

where:

B and C = the regression coefficients determined previously;

t = load maintenance time.

ment of elongation of 400%, with measurement of the relative deformation. The deformation energy is then calculated according to standard EN 13703.

Without further specifications, the standard indicates that the test, useful for quality assessment of suitability, may be performed on a virgin sample of bitumen or on bitumen conditioned after ageing operations (RTFOT and PAV).

The DTT supplements the results of the BBR, in order to obtain the most complete picture possible of cold behaviour of bitumens. Bitumen binders which have stiffness modulus S, measured as explained in the previous point and between 300 and 600 MPa, must be subjected to this test.

Durability: resistance to ageing (EN 12607-1: 2007 - EN 14769:2006)

The three framework standards mentioned above currently only take into consideration short-term ageing, with the use of RTFOT "Rolling Thin Film Oven Test" equipment according to EN 12607 - 1 (Figure 12). Resistance to hardening is measured on bitumen subjected to the RTFOT, which is assessed in terms of percentage lowering of penetration, increase in the softening point, change in the penetration index, percentage loss of mass (only EN 13924) and elastic recovery according to EN 13398 (only for bitumens modified with elastomers, EN 14023).

The instrument essentially consists of a forced-ventila-



Figure 11 - The PAV, paving long-term ageing test (according to UNI EN 14769)

tion oven with a vertical turntable, on which eight glass containers are placed, each containing a thin layer of the bitumen to be examined. At the test temperature of 163°C, hot air is blown into the containers, which rotate with the turntable. The partial distillation and oxidation to which the bitumen is subject during mixing at the production plant is approximately reproduced.

In the near future, assessment of short-term ageing will be joined by assessment of long-term ageing, simulated in the laboratory with the PAV (Pressure Ageing Vessel) - EN 14769:2006 (Figure 11).

The instrumentation essentially consist of an autoclave containing a series of trays, on each of which thin layer of the bitumen are placed. Inside the autoclave hot-air pressure is created at 85°C for 65 hours, or at 90°C or 100°C or 110°C for 20 hours, in order to reproduce the ageing action of the paving caused by atmospheric agents as closely as possible. After conditioning, the bitumen is subjected to the BBR and DSR test, to supply the values in terms of $|G^*|/sen(\delta)$ and $|G^*| sen(\delta)$, which indicate the process of hardening with ageing.

Behaviour of bitumen at low temperature

The value of the modulus, which is useful for classification, is calculated, by creating a second-degree polynomial curve, using the values of the modulus measured as a function of the acquisition times as follows:

$$\log Sc(t) = A + B \times \log(t) + C \times [\log(t)]^2$$

where:

$Sc(t)$ = bending stiffness calculated at time t , in MPa

A, B e C = regression coefficients;

t = load maintenance time in s.

The gradient of the curve at time t , "m" (m-value) is calculated by:

$$m(t) = |d \log[S(t)] / d \log(t)| = |B + 2 \times C \times \log(t)|$$

where:

B and C = the regression coefficients determined previously;

t = load maintenance time

Reference values for bitumen performance specifications

The studies performed as part of the SHRP program have shown that the most frequent problems in paving are due to:

- rut;
- fatigue cracking;
- thermal cracking.

These problems are linked with the rheological characteristics of bitumen, which are in turn influenced by temperature and load frequency.

As far as concerns the temperature variable, the following table 3, for example, shows the maximum and minimum temperatures for the main cities of the Italian regions, to be used indicatively as a calculation input by engineers, in standard traffic conditions ($V > 100$ km/h, $N < 107$ equivalent axes of 8 kN). This table

City	Country	T max - T min	City	Country	T max - T min
Genoa	Liguria	58/-10			
Aosta	Valle d'Aosta	46/-22	Milan	Lombardy	58/-16
L'Aquila	Abruzzo	52/-16	Naples	Campania	58/-10
Bari	Puglia	52/-10	Palermo	Sicily	58/-10
Bologna	Emilia Romagna	58/-10	Perugia	Umbria	52/-16
Cagliari	Sardinia	64/-10	Potenza	Basilicata	58/-10
Campobasso	Molise	58/-10	Rome	Lazio	58/-10
Catanzaro	Calabria	58/-10	Trento	Trentino	58/-10
Florence	Tuscany	58/-16	Turin	Piedmont	52/-10

Table 3 - Maximum and minimum reference temperatures for the project of mixes of the surface layer for the main cities of Italian regions

has been extrapolated from a detailed study conducted by Palermo University, to which reference should be made for further details.

Summarised in the article in the magazine
"LE STRADE" OCTOBER 2010
by P. Celauro, C. Celauro, A. Scavuzzo

Rut formation

Several design factors (granulometric curve, aggregate shape and origin, compaction) besides bitumen contribute to this problem.

For prevention of rut, it is necessary for the value of the bitumen parameters obtained by the DSR to be such that, at the maximum design temperature (T_{max}), this is

$$G^*/\sin(\delta) \geq 1,00 \text{ kPa}$$

In fact, the lower the phase delay between the load set and the consequent deformation, the lower will be the phase angle, with the elastic component therefore being higher and the viscous component which causes rut being lower.

Increasing the test temperature decreases the value of the complex modulus, so, in practice, it is a case of identifying an upper temperature limit beyond which the previous relationship is not maintained.

The DSR test is performed with a frequency of 10 rad/sec. (1.59 Hz) and performed on virgin bitumen and bitumen after RTFOT; in this second case, it must be checked that:

$$G^*/\sin(\delta) \geq 2,20 \text{ kPa}$$

Fatigue cracking

For prevention of this type of cracking, at the design intermediate temperature $[(T_{max} - T_{min}) / 2 + 4^\circ\text{C}]$, it must be checked that

$$G^* \sin(\delta) \leq 5.000 \text{ kPa}$$

This parameter corresponds with the viscous component of the complex modulus and is indicative of the energy dissipated under load by the material which causes cracks.

Decreasing the test temperature increases the value of G^* , so, in practice, it is necessary to identify the lower temperature limit, below which the previous relationship is not maintained.

The test is performed on bitumen after PAV.

Thermal cracking

In extremely cold climates, the bitumen cracks in conditions of both repeated traffic loads (fatigue cracking) and in conditions of low temperatures which persist

over time. For prevention of thermal cracking caused by low temperatures, it is necessary to check that the value of the bitumen parameters after PAV, obtained from BBR and DTT at the minimum design temperature ($T_{min} + 10^\circ\text{C}$, approximately) are such that:

$$S(60 \text{ sec}) < 300 \text{ MPa}$$

$$m \geq 0,3$$

and

$$\varepsilon > 1\% \text{ (for } S \text{ between } 300 \text{ and } 600 \text{ MPa)}$$

The BBR and DTT tests are conducted on bitumen after PAV. In the BBR test, the modulus S is calculated 60 seconds after application of the load, as is the angular coefficient of tangent m . The DTT test must be performed with a test speed of 1 mm/min.

The bitumen performance specifications to be taken into consideration include further technological characteristics with the respective values with which it is necessary to comply, referring to conditions of safety and plant processing and workability, and specifically:

- flash point (EN ISO 2592:2010): $\geq 230^\circ\text{C}$;
- loss of mass with ageing RTFOT $\leq 1\%$;
- dynamic viscosity at a temperature of 135°C (EN 12597:2007): $\leq 3 \text{ Pa} \cdot \text{s}$.

Conclusions

From what is indicated above, it is necessary to use a new method of design which proposes achieving in the laboratory characteristics of the mix which are commensurate to the actual conditions of use. That is for designing bitumen mixes capable of satisfying increasingly extreme requirements, guaranteeing performance of the paving through careful selection of the aggregate, the binder and, if necessary, the modifying agent, and their best combination. It is necessary to take into consideration new specifications on the materials, new tests and new test equipment. Thanks to development of the test methods, new equipment is now available, as well as new test protocols and innovative selection criteria based on expected performance during operation. In practice, characterisation procedures are currently of the traditional type essentially, with the advantage, however, of allowing use of equipment which most road-test laboratories already possess and which is well-known to operators, with rapid and simple performance.

For traditional bitumen mixes, the tendency for rut formation is caused more by the characteristics of the stone phase than the binder phase. Despite this, it is

possible to limit this tendency through use of bitumen with more pronounced elastic characteristics and/or higher stiffness moduli (for example, modified bitumens).

In contrast, resistance to thermal cracking is essentially linked to characteristics of the binder phase, to which the functions of relaxation of induced stress are attributed. It is therefore necessary to assess the relationships between the laboratory tests, the characteristics to be measured and the levels of deterioration according to the methods described above.

It is possible then to indicate the values of the specifications associable with the performance of the mix. Bitumen has characteristics which change with ageing. During its lifetime, in fact, the binder goes through three phases; virgin bitumen, on storage, short-term aged bitumen, during production of the mix and construction of the superstructure; long-term aged bitumen, on the road inside the superstructure, due to thermal-oxidation phenomena.

As observed, in the laboratory, the ageing phases are simulated with the RTFOT and PAV tests: tests, as described above, are performed on samples of virgin bitumen and on those conditioned by ageing tests, in order to reproduce the state of the bitumen present in the superstructure as closely as possible.

As far as concerns the mix-design of the mixture, it is necessary to take into consideration a new method of compacting laboratory specimens, EN 12697 - 31, based on the use of a gyratory compactor (Figure 13). The specimen, which is subjected to constant vertical pressure, is rotated at an angle to the axis of rotation; the resulting shearing stress generated inside the speci-



Figure 12 - The RTFOT (Rolling Thin Film Oven Test) equipment - EN 12607-1



Figure 13
The gyratory compactor (according to EN 12697:31)

men provides compaction which approximately reproduces the one to which the bituminous mix is subjected during rolling. The results of the tests, performed on the specimen compacted in this manner, will be closer to the actual behaviour of the mix in operation than those performed on specimens compacted with the impact method.

It should be noted that the bitumens, manufactured by eni, a leading oil company, are tested at eni's laboratories in collaboration with Parma University. Bitumens are tested according to performance-based characteristics with the intention of implementing eni's databank and seeking values associable with both the current characteristics and the performance of the mix.. It should also be noted that all the laboratory equipment for the tests described above is now available in Italy: Controls Srl, a company specialized in the manufacturing and marketing of laboratory equipment, can satisfy all requirements for the supply of equipment, with all the necessary assistance.

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