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**TESI DI LAUREA**

in

Infrastructure Systems

# STUDY OF THE BITUMEN AGING EFFECTS ON THE RHEOLOGICAL PROPERTIES AND FATIGUE BEHAVIOR

CANDIDATO:
Mahdi Ali

RELATORE:
Chiar.mo Prof. Andrea Simone.

CORRELATORI:
Prof.Eng. Cesare Sangiorgi.
Dott.Ing. Francesco Mazzotta.

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Sessione I

إلى والدتي الحبيبة ووالدي الحبييب، و الى اختي
الغالية مرام، و الى زوجتي الحبيبة نور.

مَنْ فَضْلُهُمْ قَدْ كَانَ دَوْمًا مِثْلَمَا
ظِلّي الظَّلِيل

*A miei genitori*
*A mia sorella Maram*
*A mia moglie Nur*
*Grazie per aver creduto sempre in me.*

***KEYWORDS:***

*Bitumen*

*Ageing*

*Rheology*

*Fatigue life*

*Healing*

*Polymer modified bitumen*

*SBS*

*Crumb rubber*

*Dynamic Shear Rheometer (DSR)*

*RTFOT*

*Laboratory testing*

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This Thesis is dedicated to my dear family, my father Munther, my mother Mariam, my sister Maram and my sweet beautiful wife Nur. I would like to thank them for supporting me emotionally and morally throughout my life. Thank you for always believing in me.

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# INTRODUCTION

Use of recycled materials has been gaining popularity all over the world to provide a sustainable future for next generations. Crumb Rubber (CR) has been the mostly recycled product in the asphalt industry. The use of shredded tire rubber (i.e., crumb rubber – CR) in asphalt pavements is one of the sustainable recycling approaches. Increasing the amount of CR integrated into asphalt pavements reduces various risks involved with stockpiling, burning, damping into landfills and other undesirable disposal methods of scrap tires.

Another benefit of using CR is being an alternative for polymer modification. Polymer modification of asphalt binders has gained quite a large popularity in many transportation agencies, primarily due to the superior crack and rut-resistant performance. However, added cost of polymer modification results in an appreciable increase in the initial cost of an asphalt pavement. Use of Recycled Tire Rubber (RTR) is a more economical and sustainable alternative to polymers.

Five polymer modified bitumen with or without rubber were made by wet process, were provided by an oil company to be tested. The main purpose of this thesis is the studying of the bitumen aging effects on the rheological properties and fatigue behavior.

The ageing phenomenon is related to oxygen presence, temperature, ultraviolet radiation and time. All these factors lead to chemical, physical and rheological changes of bituminous materials. This thesis has wanted to compare effects of short term aging which simulates aging at high temperature during asphalt mixing, storage and laying, and long term aging at ambient temperature during in-service.

Bituminous materials are used in multiple areas especially in road pavements. Repeated loadings in pavement layers cause damage. Durability of road pavement materials is a recurring problem which is an important research topic. This thesis presents the experimental device, the Dynamic Shear Rheometer (DSR). The testing procedure and specific results are developed. Moreover, fatigue criteria, for life duration, are introduced. Then information about the fatigue characteristic of bitumen are discussed.

It is emphasized that the entire experimental and study phase was carried out in the laboratory of School of Engineering and Architecture of Bologna, DICAM (Department of Civil, Chemical, Environmental and Materials Engineering), Road section. The results were discussed and analyzed in detail in chapter 06.

# CHAPTER 01:
# BITUEMEN AND AGING.

# 1. CHAPTER 01: BITUEMEN AND AGING.

## 1.1. Overview

### 1.1.1. What Is Bitumen?

Bitumen is a black or dark-colored (solid, semi-solid, viscous), amorphous, cementitious material that can be found in different forms, such us rock asphalt, natural bitumen, tar and bitumen derived from oil, which is referred to as petroleum bitumen.

Currently most of the roads globally are paved with bitumen. Today the world's demand for bitumen accounts for more than 100 million tons per year which is approximately 700 million barrels of bitumen consumed annually.

Petroleum bitumen is typically referred to as bitumen or asphalt. In Europe for instance bitumen means the liquid binder. In North America, on the other hand the liquid binder is referred to as asphalt, or asphalt cement.

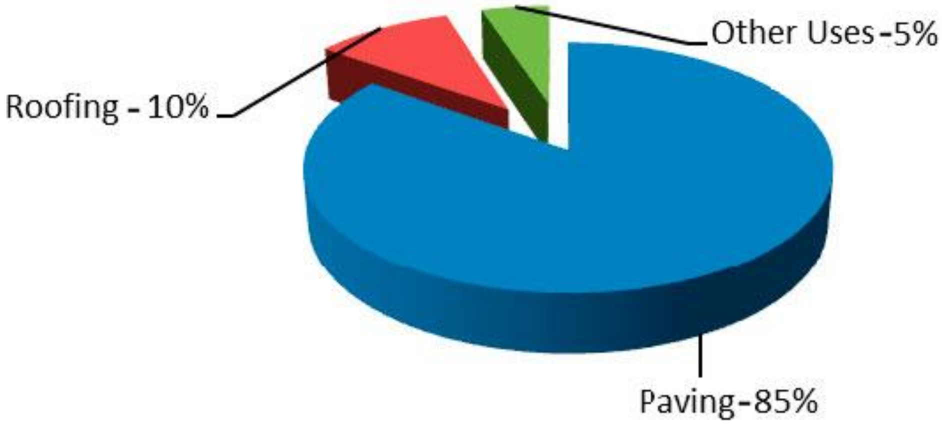


*Figure 1.1 -Percentage of usage of bitumen.*

### 1.1.2. Origin of Bitumen.

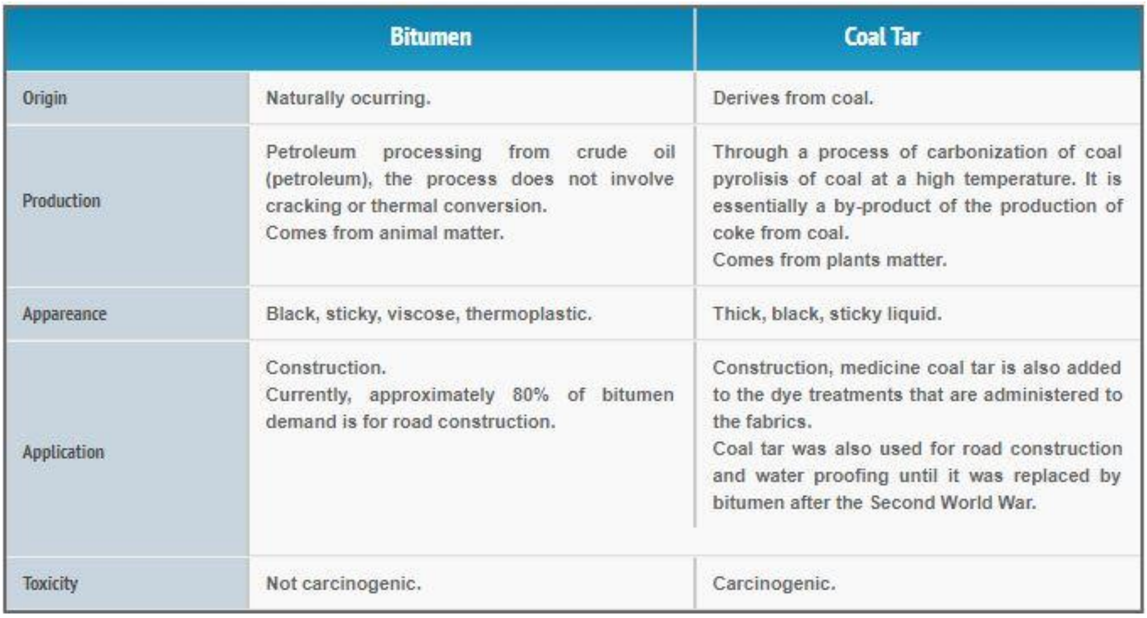
In general the term "bituminous materials" is used to denote substances in which bitumen is present or from which it can be derived. Bituminous substances comprise of primarily bitumen and tars.

Bitumen occurs in nature in several forms: hard one - easily crumbled bitumen in rock asphalt and softer, more viscous material which is present in tar sands and asphalt 'lakes'.

Another way in which bitumen can be obtained is through petroleum processing in this manner the bitumen is essentially the residue yielded through a distillation process of petroleum. Although bitumen can be found in natural form, the world currently relies for all purposes on petroleum. The material has been produced in this way for over a hundred years.

Tars on the other hand do occur in nature. Tars derive as condensates from the processing of coal (at very high temperatures), petroleum, oil-shale, wood or other organic materials. Pitch is produced when a tar is partially distilled so that the volatile components have evaporated.

Often coal tar is confused with bitumen however they are two entirely chemically different products and should not be mistaken. For the distinction drawn between petroleum bitumen and coal tar see the distinction table below:



| | Bitumen | Coal Tar |
|---|---|---|
| **Origin** | Naturally ocurring. | Derives from coal. |
| **Production** | Petroleum processing from crude oil (petroleum), the process does not involve cracking or thermal conversion.<br>Comes from animal matter. | Through a process of carbonization of coal pyrolisis of coal at a high temperature. It is essentially a by-product of the production of coke from coal.<br>Comes from plants matter. |
| **Appareance** | Black, sticky, viscose, thermoplastic. | Thick, black, sticky liquid. |
| **Application** | Construction.<br>Currently, approximately 80% of bitumen demand is for road construction. | Construction, medicine coal tar is also added to the dye treatments that are administered to the fabrics.<br>Coal tar was also used for road construction and water proofing until it was replaced by bitumen after the Second World War. |
| **Toxicity** | Not carcinogenic. | Carcinogenic. |

*Table 1.1 - Distinction between petroleum bitumen and coal tar.*

## 1.1.3. Bitumen Crude

### 1.1.3.1. UNDERSTANDING BITUMEN FEEDSTOCK & TECHNOLOGY

- There are approximately 1,500 different crude oil in existence
- The natural bitumen yield ranges from ca. 60% to practically 0%

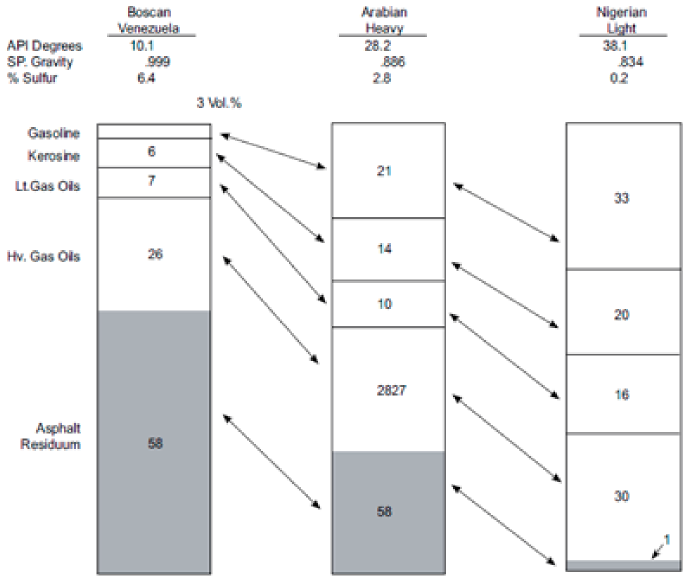


*Figure 1.2 -Crude Oil*

As soon as you start to examine further into bitumen chemistry you will realise that bitumen, is a wide mixture of different substance groups which are in short described by the acronym SARA:

( Saturated, Aromatics, Resins, Asphaltenes )

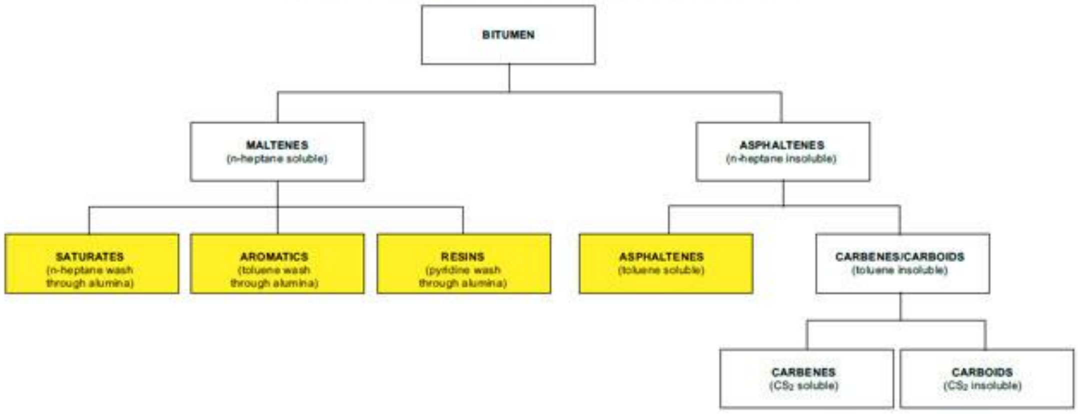
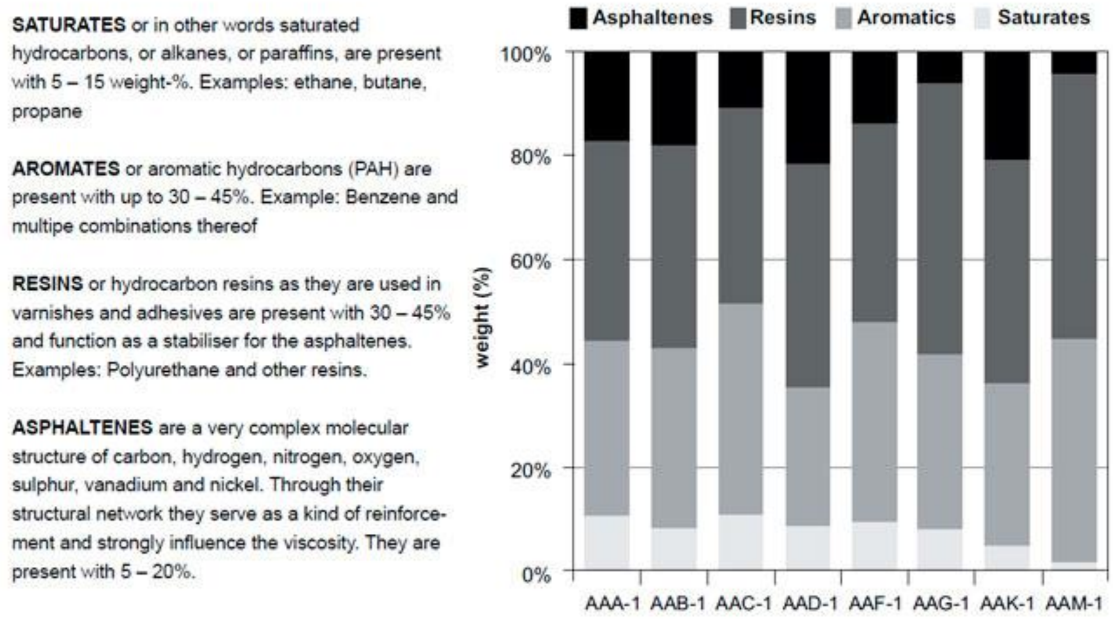


*Figure 1.3 -Bitumen Chemistry.*

#### 1.1.3.2. *S.A.R.A.*



**SATURATES** or in other words saturated hydrocarbons, or alkanes, or paraffins, are present with 5 – 15 weight-%. Examples: ethane, butane, propane

**AROMATES** or aromatic hydrocarbons (PAH) are present with up to 30 – 45%. Example: Benzene and multipe combinations thereof

**RESINS** or hydrocarbon resins as they are used in varnishes and adhesives are present with 30 – 45% and function as a stabiliser for the asphaltenes. Examples: Polyurethane and other resins.

**ASPHALTENES** are a very complex molecular structure of carbon, hydrogen, nitrogen, oxygen, sulphur, vanadium and nickel. Through their structural network they serve as a kind of reinforcement and strongly influence the viscosity. They are present with 5 – 20%.

*Figure 1.4 - S.A.R.A.*

It is fundamentally insufficient to define the quality of a bitumen by its physical properties only. That is by normally defined properties of penetration, viscosity, softening point etc.

You have good quality bitumen if the substance group (S.A.R.A) are presented in the right balance to each other.

Asphaltenes are important (10-20) but there should not be too many. A well-balanced ration and embedment by, the resins is at least equally important.

### 1.1.4. Bitumen Applications

Known for its adhesive and cohesive assets, bitumen is mostly utilised in the construction industry. Bitumen is applied on road paving because it is viscous when hot, but solid once it cools down. Therefore, Bitumen operates as the binder/glue for pieces of the aggregate.

Bitumen is applied in construction and maintenance of:

Highways
Airport runways
Footways / Pedestrian Ways

Car parks
Racetracks
Tennis courts
Roofing
Damp proofing
Dams
Reservoir and pool linings
Soundproofing
Pipe coatings
Cable Coatings
Paints
Building Water Proofing
Tile underlying waterproofing
Newspaper Ink Production
And many other applications

In order realize the complexity of bitumen as a product an in-depth knowledge and detailed understanding for one of the way the roads are built is crucial. Specialists in bitumen know bitumen as an advanced and complex construction material, not as a mere by-product of the oil refining process. The ultimate paving material (also referred to hot mix asphalt concrete – HMAC or HMA) consists of about 93 – 97% mineral aggregate (stone), sand and filler. The remaining percentage is bitumen.

### 1.1.5. How is bitumen Produced?

Petroleum Bitumen, normally called “Bitumen” or “Asphalt” is produced by refining crude oil. Used as a binder in road-building products, it is a very viscous, black or dark brown material.

The crude oil is pumped from storage tanks, where it is kept at about 60°C, through a heat exchanger system where its temperature is increased to typically 200°C by exchanging heat gained from the cooling of newly produced products in the refining process. The crude is then further heated in a furnace to typically 300° C where it is partly vaporized into an Atmospheric Distillation Column. Here the physical separation of the components occurs. The lighter components rise to the top and the heaviest components (the atmospheric residue) fall to the bottom of the column and pass through a second heat exchanger prior to treatment in a vacuum distillation column. Finally, Bitumen is obtained by vacuum distillation or vacuum flashing of atmospheric residue from the vacuum distillation column. This is "straight run bitumen”. This process is called bitumen production by straight run vacuum distillation.

An alternative method of bitumen production is by precipitation from residual fractions by propane or butane-solvent deasphalting. The bitumen thus obtained has properties which derive from the type of crude oil processed and from the mode of operation in the vacuum unit or in the solvent deasphalting unit. The grade of the bitumen depends on the amount of volatile material that remains in the product: the smaller the amount of volatiles, the harder the residual bitumen.

Specialists in bitumen view bitumen as an advanced and complex construction material, not as a mere by-product of the oil refining process.

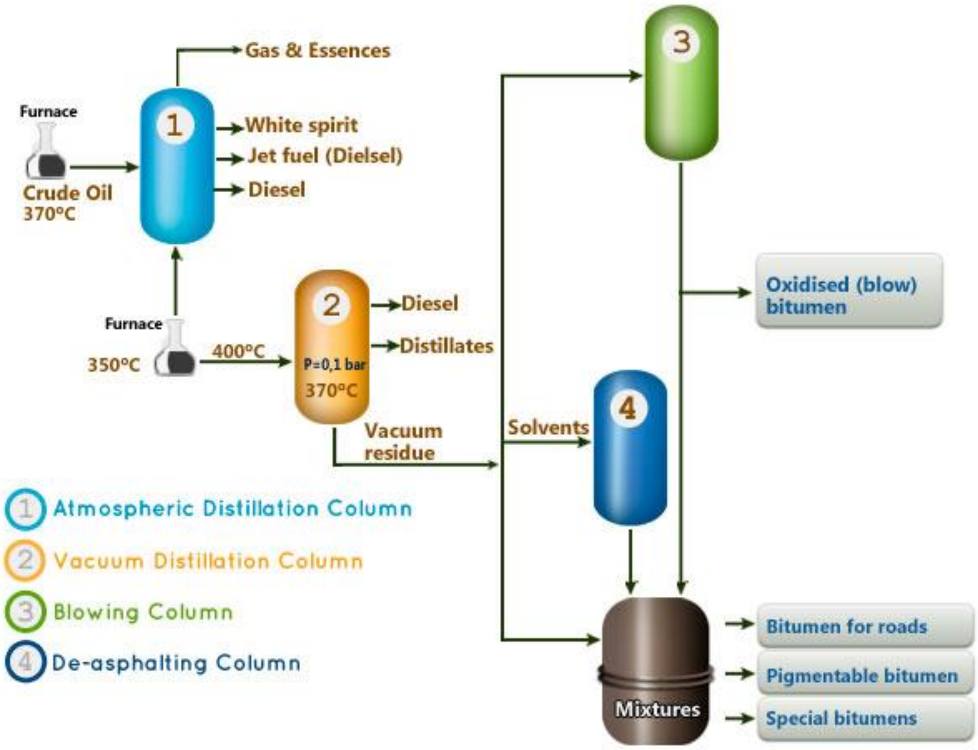


*Figure 1.5 -Standard Bitumen Production Scheme*

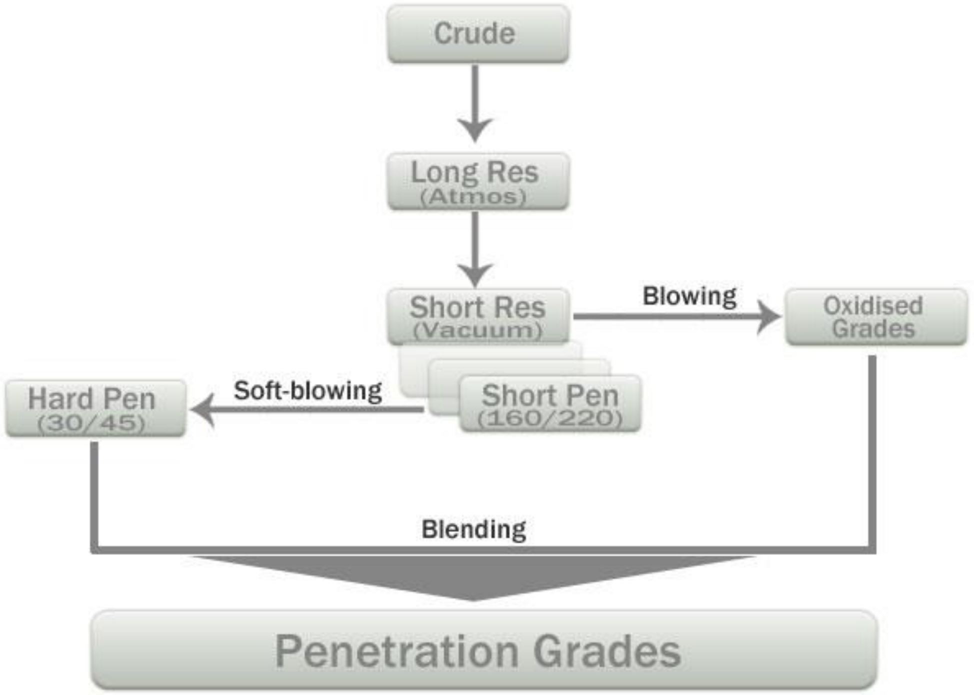


*Figure 1.6 -Typical Bitumen Production Scheme*

## 1.1.6. Bitumen Standards & Grades

### 1.1.6.1. Standardizing Bodies

In different regions and countries, different Standards and Grading systems are used for determining the quality of petroleum bituminous Binders. The most recognized standards for petroleum bitumen are published by

- **European Committee for Standardization (CEN)**
  - Deutsche Industrie Norm (DIN EN)
  - Association Française de Normalisation (AFNOR – NF EN)
  - BSI Standards - the UK's National Standards Body (NSB - BS EN)
- **American Society for Testing and Materials (ASTM)**
- **American Association of State Highway and Transportation Officials (AASHTO)**

- **South African Standard Organization (SABS)**
- **Standards Australia (AS)**

### 1.1.6.2. Most Commonly Used Bitumen Specifications.

**STANDARD PETROLEUM BITUMEN GRADES**

- ASTM D 946 and AASHTO M 20 (Penetration @ 25°C)
- ASTM D 3381 and AASHTO M 226 (Viscosity @ 60°C)
- ASTM D 6373 and AASHTO M 320 (PG-Graded Binder)
- EN 12591 (Penetration @ 25°C) → NF EN; BS EN; DIN EN; etc.
- AS 2008 (Viscosity @ 60°C)
- SABS 307 (Penetration @ 25°C; Viscosity @ 60°C after RTFOT)
- IS 73 (India) changed in 2006 from PEN grades to VG Grades (based on Viscosity @ 60°C); however, CRMB modified bitumen is still graded based on penetration.

**POLYMER MODIFIED BITUMEN**

- ASTM D 5976; ASTM D 5840; ASTM D 5841 (based on polymer type; now outdated and not used in the USA, but still used in some developing countries)
- ASTM 6373 and AASHTO M 320 (PG-Graded Binder; applicable to Unmodified and Polymer Modified Bitumen)
- EN 14023 (Framework Standard for Polymer Modified Binder)
- AUSTROADS Framework Specifications (Australia)

**BITUMEN EMULSIONS (ANIONIC; CATIONIC; UNMODIFIED; POLYMER MODIFIED)**

- USA Anionic Emulsions: ASTM D 977 and AASHTO M 140
- USA Cationic Emulsions: ASTM D 2397 and AASHTO M 208; Polymer Modified Cationic Emulsion: AASHTO M 316
- Europe: Harmonized Framework Standard EN 13808 for Cationic Unmodified and Polymer Modified Emulsions; many different national

- Annexes. *(Note: Internationally there exist a very broad range of emulsion specifications; many of them are technically inaccurate and confusing.)*

**CUTBACK BITUMEN**

- ASTM D 2026, D 2027 and D 2028 for Slow, Medium and Rapid Curing Cutback
- AASHTO M 81, M 82 for Rapid and Medium Curing Cutback
- EN 15522 Cutback and Fluxed Bituminous Binder [1.1].

### 1.1.7. Engineering properties of bitumen

The use of bitumen as such, or as a binder in diverse composite materials, is based on its mechanical properties and on the way these properties depend on the loading time and temperature. For the structural design of pavements, it is essential to be able to measure and predict the fundamental mechanical characteristics of paving materials (Bonnaure et al., 1977). The stiffness modulus of an asphalt depends on the stiffness modulus of the bitumen and the volume fractions of the different mixture components (aggregates, bitumen and air). The stiffness modulus of bitumen is also a suitable parameter for the breaking properties of the bitumen itself and of the asphalts prepared with it (Heukelom, 1966).

#### 1.1.7.1. Stiffness Modulus

A viscous material is one that is semi-fluid in nature. When stressed, it will deform or tend to deform, any deformation being permanent because it is not recovered when the loading is removed. Elastic materials also deform or tend to deform when stressed, but, when the loading is removed, any deformation is fully recovered. Bitumens are visco-elastic materials. The degree to which their behavior is viscous and elastic is a function of both temperature and the period of loading.

In order to define the visco-elastic properties, the concept of the stiffness modulus is a fundamental parameter to describe the mechanical properties of bitumens.

At high temperatures or long loading times they behave as viscous liquids, whereas at very low temperatures or short loading times they behave as elastic (brittle) solids. The intermediate range of temperature and loading times, more typical of conditions in service, results in visco-elastic behavior.

In the case of visco-elastic materials such as bitumen, a tensile stress σ applied at a loading time t = 0 causes a strain ε that increases, but not proportionately, with the loading time. The stiffness modulus St at a loading time t is defined as the ratio between the applied stress and the resulting strain at the loading time t. It follows from the above that the value of the stiffness modulus is dependent on the temperature and the loading time that is due to the special nature of bitumen. Consequently, it is necessary to state both the temperature T and the loading time t of any stiffness modulus measurement.

$$S_{T,t} = \frac{\sigma}{\varepsilon_{T,t}}$$

The methods used to measure the stiffness modulus of bitumen are often based on shear deformations. The resistance to shear is expressed in terms of the shear modulus G, which is defined as:

$$G = \frac{shear\ stress}{shear\ strain} = \frac{\tau}{\gamma}$$

In dynamic tests, the shear stress is usually applied as a sinusoidally varying stress of constant amplitude and fixed frequency. The deformation of the material under test also varies sinusoidally with the same frequency as the applied stress. Tests are carried out at various values of frequency, and the ratio of the stress to strain can be plotted against the inverse of the angular frequency. The degree of elasticity of the bitumen under the test conditions is given by how much the deformation response is out of phase from the applied shear stress, and this is referred to as the phase angle. For purely elastic materials the phase angle is zero. For purely viscous materials the phase angle is 90°. **[2]**

### 1.1.8. Different forms of bitumen

#### 1.1.8.1. Cutback bitumen

Normal practice is to heat bitumen to reduce its viscosity. In some situations preference is given to use liquid binders such as cutback bitumen. In cutback bitumen suitable solvent is used to lower the viscosity of the bitumen. From the environmental point of view also cutback bitumen is preferred. The solvent from the bituminous material will evaporate and the bitumen will bind the aggregate. Cutback bitumen is used for cold weather bituminous road construction and maintenance. The distillates used for preparation of cutback bitumen are naphtha, kerosene, diesel oil, and furnace oil. There are different types of cutback bitumen like rapid curing (RC), medium curing (MC), and slow curing (SC). RC is recommended for surface dressing and patchwork. MC is recommended for premix with less quantity of fine aggregates. SC is used for premix with appreciable quantity of fine aggregates.

#### 1.1.8.2. Bitumen Emulsion

Bitumen emulsion is a liquid product in which bitumen is suspended in a finely divided condition in an aqueous medium and stabilised by suitable material. Normally cationic type emulsions are used in India. The bitumen content in the emulsion is around 60% and the remaining is water. When the emulsion is applied on the road it breaks down resulting in release of water and the mix starts to set. The time of setting depends upon the grade of bitumen. The viscosity of bituminous emulsions can be measured as per IS: 8887-1995. Three types of bituminous emulsions are available, which are Rapid setting (RS), Medium setting (MS), and Slow setting (SC). Bitumen emulsions are ideal binders for hill road construction. Where heating of bitumen or aggregates are difficult. Rapid setting emulsions are used for surface dressing work. Medium setting emulsions are preferred for premix jobs and patch repairs work. Slow setting emulsions are preferred in rainy season.

#### 1.1.8.3. Bituminous primers

In bituminous primer the distillate is absorbed by the road surface on which it is spread. The absorption therefore depends on the porosity of the surface. Bitumen primers are useful on the stabilized surfaces and water bound macadam base courses. Bituminous primers are generally prepared on road sites by mixing penetration bitumen with petroleum distillate.

#### 1.1.8.4. Modified Bitumen

Certain additives or blend of additives called as bitumen modifiers can improve properties of Bitumen and bituminous mixes. Bitumen treated with these modifiers is known as modified bitumen. Polymer modified bitumen (PMB)/ crumb rubber modified bitumen (CRMB) should be used only in wearing course depending upon the requirements of extreme climatic variations. The detailed specifications for modified bitumen have been issued by IRC: SP: 53-1999. It must be noted that the performance of PMB and CRMB is dependent on strict control on temperature during construction. The advantages of using modified bitumen are as follows

- Lower susceptibility to daily and seasonal temperature variations
- Higher resistance to deformation at high pavement temperature
- Better age resistance properties
- Higher fatigue life for mixes
- Better adhesion between aggregates and binder
- Prevention of cracking and reflective cracking

### 1.1.9. Requirements of Bitumen

The desirable properties of bitumen depend on the mix type and construction. In general, Bitumen should possess following desirable properties.

- The bitumen should not be highly temperature susceptible: during the hottest eather the mix should not become too soft or unstable, and during cold weather the mix should not become too brittle causing cracks.
- The viscosity of the bitumen at the time of mixing and compaction should be adequate. This can be achieved by use of cutbacks or emulsions of suitable grades or by heating the bitumen and aggregates prior to mixing.
- _ There should be adequate affinity and adhesion between the bitumen and aggregates used in the mix.

### 1.1.10. Tests on bitumen

There are a number of tests to assess the properties of bituminous materials. The following tests are usually conducted to evaluate different properties of bituminous materials.

- 1. Penetration test
- 2. Ductility test
- 3. Softening point test
- 4. Speci_c gravity test
- 5. Viscosity test
- 6. Flash and Fire point test
- 7. Float test
- 8. Water content test
- 9. Loss on heating test

#### 1.1.10.1. Penetration test

It measures the hardness or softness of bitumen by measuring the depth in tenths of a millimeter to which a standard loaded needle will penetrate vertically in 5 seconds. BIS had standardised the equipment and test procedure. The penetrometer consists of a needle assembly with a total weight of 100g and a device for releasing and locking in any position. The bitumen is softened to a pouring consistency, stirred thoroughly and poured into containers at a depth at least 15 mm in excess of the expected penetration. The test should be conducted at a specified temperature of 25o C. It may be noted that penetration value is largely influenced by any inaccuracy with regards to pouring temperature, size of the needle, weight placed on the needle and the test temperature. A grade of 40/50 bitumen means the penetration value is in the range 40 to 50 at standard test conditions. In hot climates, a lower penetration grade is preferred. The Figure 1.1 shows a schematic Penetration Test setup.

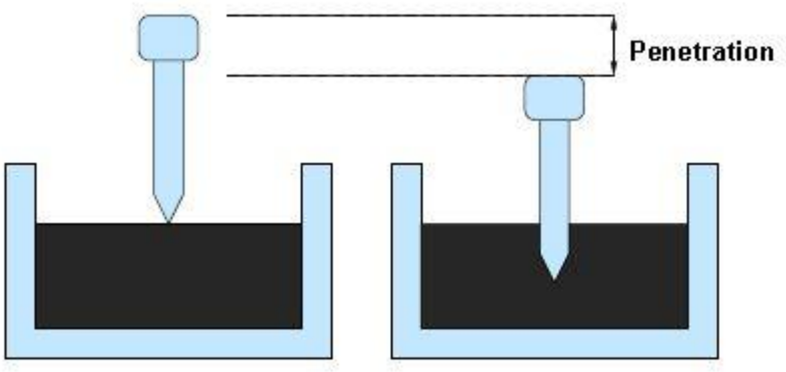


*Figure 1.7 -Penetration Test Setup*

### 1.1.10.2. Ductility test

Ductility is the property of bitumen that permits it to undergo great deformation or elongation. Ductility is defined as the distance in cm, to which a standard sample or briquette of the material will be elongated without breaking. Dimension of the briquette thus formed is exactly 1 cm square. The bitumen sample is heated and poured in the mould assembly placed on a plate. These samples with moulds are cooled in the air and then in water bath at 27o C temperature. The excess bitumen is cut and the surface is leveled using a hot knife. Then the mould with assembly containing sample is kept in water bath of the ductility machine for about 90 minutes. The sides of the moulds are removed, the clips are hooked on the machine and the machine is operated. The distance up to the point of breaking of thread is the ductility value which is reported in cm. The ductility value gets affected by factors such as pouring temperature, test temperature, rate of pulling etc. A minimum ductility value of 75 cm has been specified by the BIS. Figure 1.1 shows ductility moulds to be filled with bitumen.

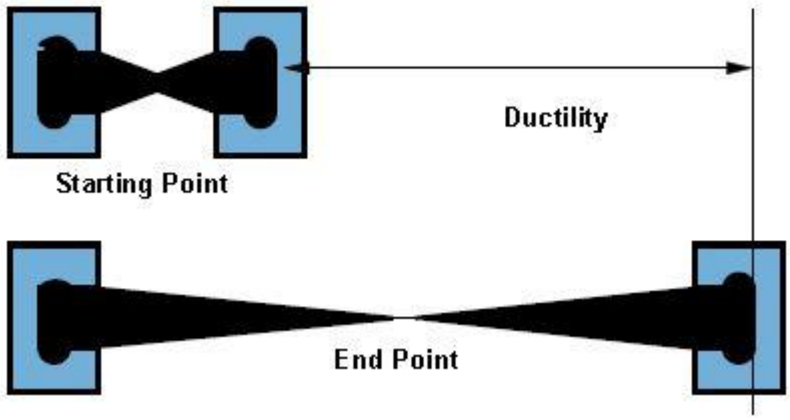


*Figure 1.8 -Ductility Test*

#### 1.1.10.3. Softening point test

Softening point denotes the temperature at which the bitumen attains a particular degree of softening under the specifications of test. The test is conducted by using Ring and Ball apparatus. A brass ring containing test sample of bitumen is suspended in liquid like water or glycerin at a given temperature. A steel ball is placed upon the bitumen sample and the liquid medium is heated at a rate of 5o C per minute. Temperature is noted when the softened bitumen touches the metal plate which is at a specified distance below. Generally, higher softening point indicates lower temperature susceptibility and is preferred in hot climates. Figure 1.3 shows Softening Point test setup.

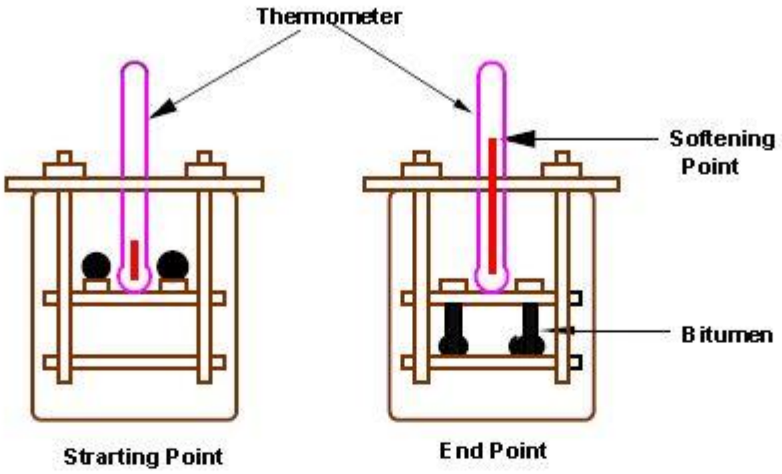


*Figure 1.9 -Softening Point Test Setup*

#### 1.1.10.4. Specific gravity test

In paving jobs, to classify a binder, density property is of great use. In most cases bitumen is weighed, but when used with aggregates, the bitumen is converted to volume using density values. The density of bitumen is greatly influenced by its chemical composition. Increase in aromatic type mineral impurities cause an increase in specific gravity.
The specific gravity of bitumen is defined as the ratio of mass of given volume of bitumen of known content to the mass of equal volume of water at 27o C. The speci_c gravity can be measured using either pycnometer or preparing a cube specimen of bitumen in semi solid or solid state. The specific gravity of bitumen varies from 0.97 to 1.02.

#### 1.1.10.5. Viscosity test

Viscosity denotes the fluid property of bituminous material and it is a measure of resistance to flow. At the application temperature, this characteristic greatly influences the strength of resulting paving mixes. Low or high viscosity during compaction or mixing has been observed to result in lower stability values. At high viscosity, it resist the compactive effort and thereby resulting mix is heterogeneous, hence low stability values. And at low viscosity instead of providing a uniform film over aggregates, it will lubricate the aggregate particles. Orifice type viscometers are used to indirectly find the viscosity of liquid binders like cutbacks and emulsions. The viscosity expressed in seconds is the time taken by the 50 ml bitumen material to pass through the orifice of a cup, under standard test conditions and specified temperature. Viscosity of a cutback can be measured with either 4.0 mm orifice at 25°C or 10 mm orifice at 25 or 40° C.

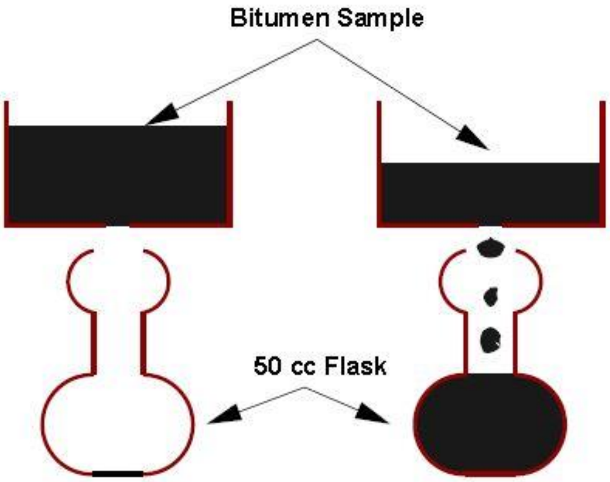


*Figure 1.10 -Viscosity Test*

#### 1.1.10.6. Flash and fire point test

At high temperatures depending upon the grades of bitumen materials leave out volatiles. And these volatiles catches fire which is very hazardous and therefore it is essential to qualify this temperature for each bitumen grade. BIS defined the flash point as the temperature at which the vapour of bitumen momentarily catches fire in the form of flash under specified test conditions. The fire point is defined as the lowest temperature under specified test conditions at which the bituminous material gets ignited and burns.

#### 1.1.10.7. Float test

Normally the consistency of bituminous material can be measured either by penetration test or viscosity test. But for certain range of consistencies, these tests are not applicable and Float test is used. The apparatus consists of an aluminum float and a brass collar filled with bitumen to be tested. The specimen in the mould is cooled to a temperature of 5°C and screwed in to float. The total test assembly is floated in the water bath at 50°C and the time required for water to pass its way through the specimen plug is noted in seconds and is expressed as the float value.

#### 1.1.10.8. Water content test

It is desirable that the bitumen contains minimum water content to prevent foaming of the bitumen when it is heated above the boiling point of water. The water in a bitumen is determined by mixing known weight of specimen in a pure petroleum distillate free from water, heating and distilling of the water. The weight of the water condensed and collected is expressed as percentage by weight of the original sample. The allowable maximum water content should not be more than 0.2% by weight.

#### 1.1.10.9. Loss on heating test

When the bitumen is heated it loses the volatility and gets hardened. About 50gm of the sample is weighed and heated to a temperature of 163°C for 5hours in a specified oven designed for this test. The sample specimen is weighed again after the heating period and loss in weight is expressed as percentage by weight of the original sample. Bitumen used in pavement mixes should not indicate more than 1% loss in weight, but for bitumen having penetration values 150-200 up to 2% loss in weight is allowed. **[1]**

## 1.2. Ageing

### 1.2.1. Introduction

The aging of bituminous binders is one of the key factors determining the lifetime of an asphalt pavement. The process of aging involves chemical and/or physical property changes that usually make bituminous materials harder and more brittle, thus increasing risk of pavement failure. The aging-related pavement failure modes include cracking (thermal or traffic induced) and ravelling. Cracks on pavement surface may increase aging of the binder because of increased exposure area to atmospheric oxygen.

In general, bitumen aging takes place in two stages, namely short-term aging at high temperature during asphalt mixing, storage and laying, and long-term aging at ambient temperature during in-service. The mechanisms of aging include oxidation, evaporation and physical hardening. Physical hardening is a reversible process, which changes the rheological properties of bitumen without altering its chemical composition. At ambient temperatures, physical hardening normally is very slow, but it can speed up at low temperatures. For bituminous binders, loss of volatile components (evaporation) is also considered as an aging mechanism. However, today's penetration grade bitumens are relatively involatile, thus during pavement in-service this type of aging is negligible.

As a principal mechanism, oxidative aging is an irreversible chemical reaction between components of bitumen and oxygen. It may occur through different reactions, such as photo-oxidation and thermal oxidation [3]. The light (mainly ultraviolet, UV) catalyzed reaction occurs rapidly and generally takes place within the top 5 μm of the exposed binder film, since bitumen is a good light absorber [3] [4]. In spite of the limited penetration into bitumen, the photo-oxidation induced aging may have an influence on durability, particularly for some polymer modified binders [5]. Nowadays, most of aging on the road is still regarded as thermally induced. The rate of thermal oxidation of bitumen is approximately doubled for every 10°C rise in temperature. Thus aging rate in service depends to a large extent on pavement temperature [6] [7]. Certainly, the oxidative aging is influenced by the chemical nature of bitumen. It has been shown that different bitumens have very different increase of viscosity with aging time, and the temperature-dependence of aging kinetics is strongly dependent on the bitumen [8] [9] [10].

Another important factor affecting bitumen aging on the road is the void content of asphalt mixture. Much work has indicated that asphalt mixtures of low voids show a low degree of bitumen aging while higher void content facilitates the aging process [3] [6] [11] [12] [13]. Presumptively, void content determines the rate of aging by controlling oxygen access to the bitumen. Thus, the oxidative aging of bitumen as function of depth in the pavement is closely related to void content of the mixture.

To simulate field aging in laboratory, different types of test may be used, including conduction of accelerated aging on bituminous binders, loose asphalt mixture, or on compacted asphalt specimen. For bituminous binders, there are three European standardized tests [14] for short-term aging at high temperatures, namely Rolling Thin-Film Oven Test (RTFOT, EN 12607-1), Thin Film Oven Test (TFOT, EN 12607-2), and Rotating Flask Test (RFT, EN 12607-3). These tests reasonably simulate aging particularly during mixing process in an asphalt mixing plant.

For long-term aging during in-service, laboratory simulation is rather difficult. Ideally, a laboratory test should be able to predict chemical and physical property changes in the bitumen which occur after certain years on asphalt pavement. This may be achieved by conducting an aging test at artificially severe conditions, e.g. at temperatures higher than pavement service temperature and at pressures higher than ambient pressure. Two European standardized long term aging tests are Pressure Aging Vessel (PAV) [15] and Rotating Cylinder Aging Test (RCAT) [16]. Although numerous investigations have been carried out, solid data, especially field data for different types of binders under different climatic conditions, are still not sufficient to support if these laboratory aging tests are relevant or if natural aging occurred in the pavement can be properly predicted. It is also believed that aging of bitumen at a higher temperature may be fundamentally different from aging at lower temperature that is more accurately simulating pavement temperature [7].

### 1.2.2. Influence of ageing on bitumen

*Figure 1.11 -Pavement cracking*

*Figure 1.12 -Loss of aggregate*

Aging or Age hardening of bitumen is one of the main factors that can significantly affect the durability of bituminous paving materials. When the bitumen is age hardened, the asphalt mixture will become brittle and its ability to support traffic-induced stresses and strains may significantly decrease. Pavements may be deteriorated due to cracking. In addition, excessive hardening can also weaken the adhesion between the bitumen and aggregate, resulting in loss of materials at the surface layer and generate weakening of the asphalt mixture.

Bitumen is manufactured from crude oil. Bitumen is obtained as the last residue in fractional distillation of crude petroleum. Crude petroleum is different molecular weights. In the petroleum refineries the individual components like LPG, naphtha, Kerosene, Diesel etc. are separated through the process of fractional distillation. The heaviest material Obtained from the fractional distillation process is further treated and blended to make different grades of paving grade bitumen. Bitumen is defined as “A viscous liquid, or a solid consisting essentially of hydrocarbons and their derivatives, which is soluble in tr-chloro-ethyelene and is substantially non-volatile and softens gradually when heated. It is black or brown in color & possesses waterproofing and adhesive properties. It is obtained by refinery processes from petroleum, and is also found as a natural deposit or as a component of naturally occurring asphalt, in which it is associated with mineral matte.

It is generally agreed that aging is primarily associated with bitumen oxidation and the loss of volatile/oily components from the bitumen to the air and/or aggregates during asphalt mixture production (short-term aging) and in-place service period (long-term aging). Both factors cause an increase in viscosity (or stiffness) of the bitumen and consequential stiffening of the asphalt mixture.

Bitumen, like any organic matter, is affected by factors like presence of oxygen, ultraviolet rays and changes in temperature. These factors are responsible for hardening of bitumen. Hardening results in decrease in penetration increase in softening point and increase in penetration index (PI).For increased life of bituminous pavement it is essential that excessive hardness does not take place.

The aging of bituminous binders is one of the key factors determining the lifetime of an asphalt pavement. The process of aging involves chemical and/or physical property changes that usually make bituminous materials harder and more brittle, thus increasing risk of pavement failure. The aging-related pavement failure modes include cracking (thermal or traffic induced) and ravelling. Cracks on pavement surface may increase aging of the binder because of increased exposure area to atmospheric oxygen.

### 1.2.3. Short and long term ageing

In general, bitumen aging takes place in two stages, namely short-term aging at high temperature during asphalt mixing, storage and laying, and long-term aging at ambient temperature during in-service. The mechanisms of aging include oxidation, evaporation and physical hardening. Physical hardening is a reversible process, which changes the rheological properties of bitumen without altering its chemical composition. At ambient temperatures, physical hardening normally is very slow, but it can speed up at low temperatures. For bituminous binders, loss of volatile components (evaporation) is also considered as an aging mechanism. However, today's penetration grade bitumen's are relatively in volatile, thus during pavement in-service this type of aging is negligible.

As a principal mechanism, oxidation aging is an irreversible chemical reaction between components of bitumen and oxygen. It may occur through different reactions, such as photo-oxidation and thermal oxidation. The light (mainly ultraviolet, UV) catalysed reaction occurs rapidly and generally takes place within the top 5 μm of the exposed binder film, since bitumen is a good light absorber. In spite of the limited penetration into bitumen, the photo-oxidation induced aging may have an influence on durability, particularly for some polymer modified binders. Nowadays, most of aging on the road is still regarded as thermally induced. The rate of thermal oxidation of bitumen is approximately doubled for every 10°C rise in temperature. Thus aging rate in service depends to a large extent on pavement temperature. Certainly, the oxidative aging is influenced by the chemical nature of bitumen. It has been shown that different bitumen has very different increase of viscosity with aging time, and the temperature dependence of aging kinetics is strongly dependent on the bitumen. [2.2]

Ageing process that occurs first is faster compared to that is due during the in service life, in fact, the short-term ageing can also be called primary ageing. Rates of ageing phase are shown in figure 1.13. [17]

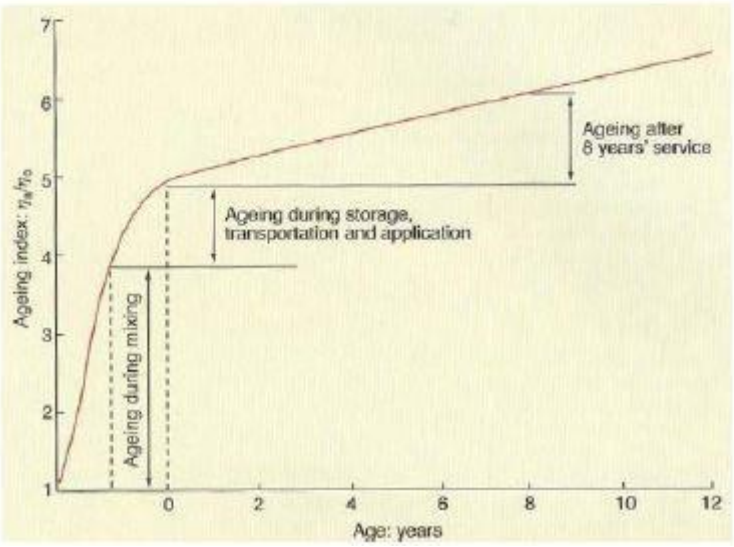


*Figure 1.13 -Rate of asphalt pavement hardening (Shell Bitumen Handbook, 1990)*

### 1.2.4. -6 Factors influencing aging of bitumen

Bitumen, like any organic matter, is affected by factors like presence of oxygen, ultraviolet rays and changes in temperature. These factors are responsible for hardening of bitumen. Hardening results in

- Decrease in penetration value
- Increase in softening point temperature and
- Increase in penetration index (PI).

For increased life of bituminous pavement, it is essential that excessive hardness does not take place.

Hardening of bitumen takes under the influence of external factors in the following ways:

**OXIDATIVE HARDENING:**

When bitumen is exposed to atmosphere for a prolonged period the oxygen starts reacting with the bitumen constituents and higher molecular weight molecules are formed. Larger molecules results in lesser flexibility and hence increased hardness. The degree of hardness is dependent on factors like ambient temperature, exposure time & thickness of bitumen film. It is observed that for $10^0$C increase in temperature above $100^0$C, the rate of oxidation doubles.

**HARDENING DUE TO LOSS OF VOLATILES:**

Over a period of time the volatile components in bitumen evaporate. The rate of evaporation is dependent on temperature only. The volatiles in bitumen are relatively very low and hence hardening due to loss of volatiles is relatively small.

**PHYSICAL HARDENING:**

At ambient temperatures bitumen molecules slowly reorient themselves. This results in physical hardening. This process is an extremely slow process and hence actual hardening due to the above factor is very low.

## EXUDATIVE HARDENING:

Exudative hardening takes place due to the movement of oily components out of bitumen over a period of time. The rate of hardening due to this process is dependent on the type of bitumen and also on the porosity of the aggregate.

## HARDENING OF BITUMEN DURING STORAGE:

Bitumen is stored in above ground tanks at high temperatures and presence of oxygen are the two primary factors responsible for hardening of bitumen. Hence it is very important that bitumen be handled at the lowest possible temperature, consistent with efficient use. Also the storage tanks should have low surface to volume ratio so as to minimize the exposed surface area.. Lower exposed surface area would mean lower oxidation rate.

While designing the tanks it should be ensured that the recirculation pipelines always enter the tank below the bitumen surface. This will reduce splashing during recirculation. When the recirculation line enters the tank above the product surface all the three factors which promote oxidation viz. high temperature, access to oxygen and high exposed surface to volume ratio, are present. Therefore bitumen quality deteriorates very fast.

If handled properly the hardening in tanks can be insignificant as the product is stored for shorter durations. If bitumen is to be stored for long durations (4 to 5 days) then the temperature should be reduced to $20^0$C to $25^0$C above softening point.

In case where bitumen is to be reheated to increase the temperature adequate precautions have to be exercised. Bitumen should not be heated continuously in the beginning. Continuous heating can result in very high localized temperatures in area close to the heating source.

## HARDENING OF BITUMEN ON ROAD:

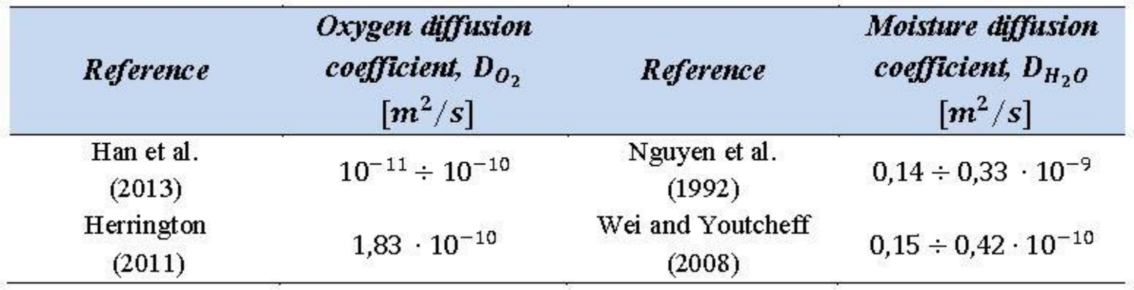
Some hardening of bitumen can also take place on the road due to oxidation. The level of oxidation is purely dependent on the access to oxygen. If the pavement is well graded and well compacted the hardening is nominal as the void content will be low. [HP Bitumen Handbook] [3.3]

### 1.2.5. Combined effects of ageing and moisture diffusion on bitumen

Many laboratory ageing tests can be performed in order to simulate the short and long-term ageing on bitumen. The material may be submitted to the effect of oxygen, heat and pressure, but conditions that occur during the service life of asphalt pavements are different and they combine several factors together. During their in-service life, many different weathering factors, especially water in forms of rainfall, mist, hail or dew affected the pavement performance causing their prematurely failure. In general, laboratory ageing leads to achieve higher levels of oxidation compared to results of samples coming from actual field pavement. It is due to the combined effects of several parameters. Variability of asphalt paving on the permeability, so on the various air voids content and differences in maximum pavement temperatures in different environment. Also minerals contained in aggregates affect bitumen oxidation, albeit slightly, because they act as catalysts of the more non-polar fraction, i.e. naphthene aromatics (Petersen, 2009). Moreover, film thickness influences the oxidation (Karlsson and Isacsson, 2003). Thus, for a better understanding of bitumen ageing process in the field, it is necessary change the standard protocol of tests to more accurately characterise the ageing effect of field factors [32].

As oxygen diffuses into mixtures, binders and bitumen, in the same way also moisture infiltrates into these materials. In general, water is transported in a porous material, not only through diffusion process, but also due to capillary flow and hydraulic flux. The controlling process depends on the relative humidity content (RH) and the driving potential. When RH is less than 95% diffusion is the main mechanism. Capillary transportation occurs only if the RH is higher than 95%. Finally, when a pavement is super-saturated the flux is regulated by the law of hydraulic flux, the so-called Darcy’s law (Apeyagyei et al., 2013). Among all, we focus only on diffusion process because it governs the infiltration of water into the element of mixtures. Similarly to oxygen, moisture diffusion is studied with the first and second Fick’s laws. The key parameter of the process remains the diffusion coefficient. Main studies has focused their attention on diffusion in asphalt mixtures. Many factors that affect oxygen diffusion and mixture oxidation, also influence moisture diffusion. The main effect of moisture diffusion is the softening of binder and further cohesive failure of film. The diffusion coefficient is also influenced by temperature and viscosity (Arambula et al., 2010).

In table 1.2 data referring to coefficient diffusion for both oxygen and moisture are reported. Only representative values are listed in order to compare the magnitude of the diffusion coefficients for the two processes.



| *Reference* | *Oxygen diffusion coefficient,* $D_{O_2}$ $[m^2/s]$ | *Reference* | *Moisture diffusion coefficient,* $D_{H_2O}$ $[m^2/s]$ |
|---|---|---|---|
| Han et al. (2013) | $10^{-11} \div 10^{-10}$ | Nguyen et al. (1992) | $0,14 \div 0,33 \cdot 10^{-9}$ |
| Herrington (2011) | $1,83 \cdot 10^{-10}$ | Wei and Youtcheff (2008) | $0,15 \div 0,42 \cdot 10^{-10}$ |

*Table 1.2 - Diffusion coefficients of oxygen and moisture*

Both oxygen and moisture diffusion coefficients widely vary, because they are strictly connected to bitumen source, temperature and viscosity. Moreover, they also depend on the techniques used to evaluate the diffusivity. Thus, it is very important specify all detailed conditions because there is not a general value.

Huang et al. (2008) investigated the influence of both, moisture and ageing on bitumen, in detail, they compared the chemical and rheological properties of bitumen aged with or without the presence of water. Analyzing the chemical properties of bitumen, results suggested a disruption of colloidal structure of their molecules due to the water during the oxidative ageing process. On the other hand, the construction of the master curve showed a decrease of stiffness, but this was not the rule. It was found that the source of bitumen affected its response.

Also, Tao et al. (2011) [32]; analyzed the response of bitumen subjected to both moisture and ageing conditioning, by comparing the results obtained from penetration tests, Ring-and-Ball test and investigating chemical and rheological properties of bitumen with and without the presence of water. The values of penetration, at 25°C, for neat bitumen decreased with a longer ageing time, instead the opposite behavior was observed by the softening point as shown in figure 1.14.

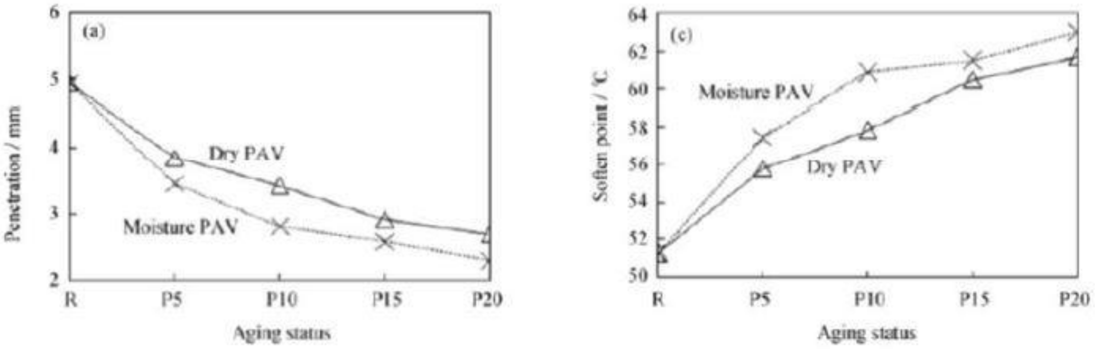


*Figure 1.14 -Penetration Grade test results for aged bitumen at different PAV ageing [32].*

Moreover, the figures show a worsening of the performance due to moisture conditioning. Also, the viscosity and the ratio between complex modulus and sine of phase angle due to ageing were analysed. Moisture accelerated the effects of natural bitumen ageing. On the other hand, polymer modified bitumen had a better moisture ageing resistance. The spectrum of materials showed an increase of carbonyl groups, such as ketones or carboxylic acids [32]. concluded that various factors like heat, oxygen, pressure and water have mutual effects on bitumen ageing processes, which can cause an increase in stiffness and embrittlement for both, neat and modified bitumen. Additional effects due to the combined effects of moisture and ageing result in more low-temperature cracking propensity.

### 1.2.6. Methods of laboratory ageing

Several methods of laboratory ageing were developed during the years in order to simulate the effects of bitumen oxidation. These can be divided in two main categories, that is tests able to simulate the short-term ageing and methodologies for simulating the long-term effects of oxidation process. The most common European method, which simulates the ageing during the storage, mixing with aggregates, transportation and laying, is the Rolling Thin Film Oven Test (RTFOT). Other tests, like Rotating Cylindrical Ageing Tests (RCAT), are performed following the American standards. On the other hand, Pressure Ageing Vessel (PAV) is the most widely used laboratory test for the simulation of long-term ageing.

All of them are based on the variation of three main factors: temperature, pressure and time. Temperature is strongly related to the oxygen diffusion coefficient. The raising of pressure and the extension of ageing time can surely apply greater ageing, speed up the process of oxidation, and consequently, results in major deterioration. The correct balance between these parameters in each ageing test was established through much comparison between samples aged with the laboratory devices and the specimens extracted in field. Also, the use of simple devices like oven, or a pressurized machinery allows the observation of the single effect of high temperature and pressure respectively [17].

#### 1.2.6.1. RTFOT ageing

The Rolling Thin Film Oven Test (RTFOT) was developed during the SHRP (Behera et al., 2013) in order to simulate the short-term ageing of bitumen which occurs especially during mixing in asphalt plan. Bitumen suffers high temperature, 163°C, combined with a constant and specific supply of air, following the standard procedure described in standard EN 12607-1:2014. The apparatus consists of an oven which inside is equipped with an aluminium carriage. It has eight opening and springs clips for firmly holding the corresponding eight glass containers in a horizontal position. Moreover, an air jet is placed in a specific location. Figure 1.15 shows the outside and inside of the device. [17]

*Figure 1.15 -Rolling Thin Film Oven Test equipment*

During the test, the carriage rotates about the horizontal axis with a certain speed, which allows the air jet to enter in each glass. In detail, the air shall blow along the main axis of each glass container. In the same time, high temperatures lead the material to liquefy. The rotation together with the applied temperatures lead bitumen to cover the entire surface of each container forming a thin film, which also rotates following the carriage movement.

Temperature is continuously monitored with a thermometer positioned inside the device. Figure 1.16 shows pictures of bitumen containers before and after the test.

*Figure 1.16* -Samples pre- and post- RTFOT

The glass containers should be filled with 35,0±0,5 g of bitumen, a specific amount that ensure that the material does not leak from the containers during rotation. The number of necessary glass depends on the planned characterization tests of the operator [17].

The RTFOT was performed one time for each type of modified bitumen that we are focus on in this research which are:

- PMB 45/80-70 Hard+ recycled tires powder + viscous reducer.
- PMB 45/80-70 Hard+ recycled tires powder.
- PMB 45/80-70 Hard.
- PMB 45/80-55 Soft+ recycled tires powder.
- PMB 45/80-55 Soft.

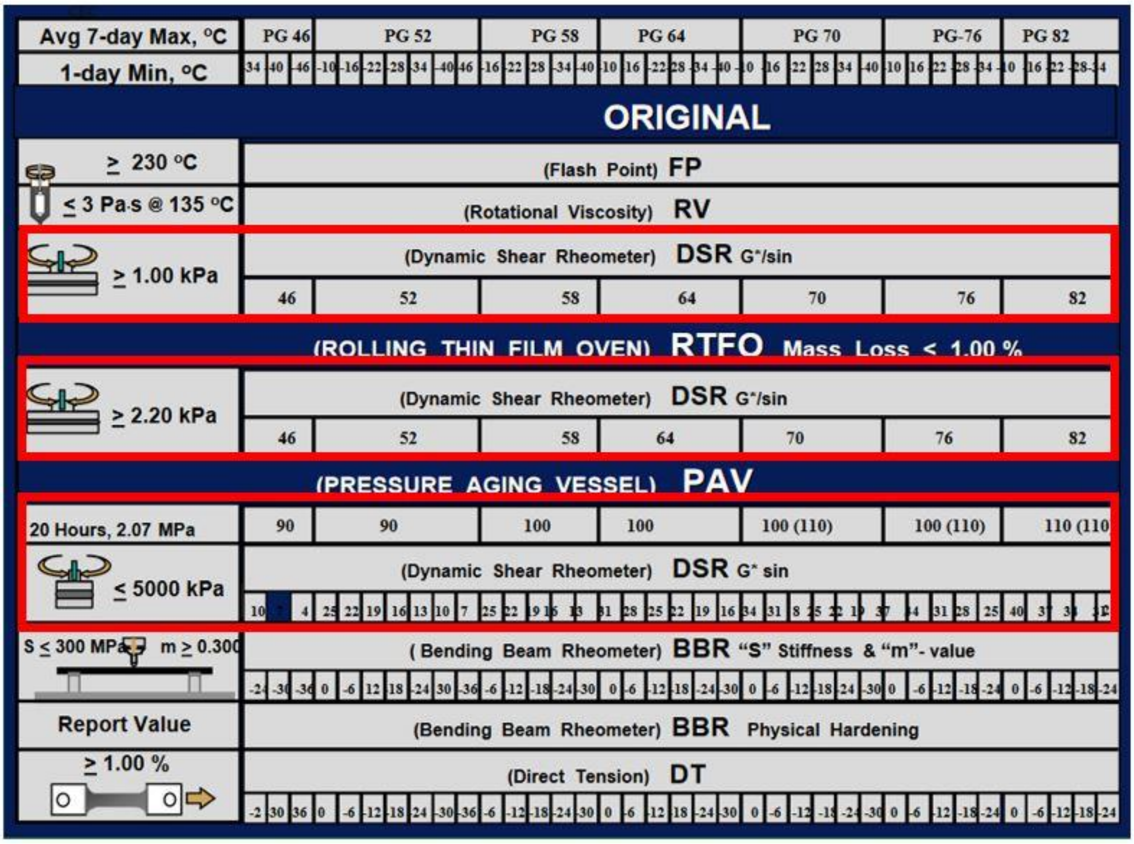
## 1.3. SUPERPAVE PERFORMANCE GRADING (PG SYSTEM)

Recognizing the limitations of the empirical testing, the Strategic Highway Research Program (SHRP) sponsored a US$ 50 million research effort to develop performance based tests and specifications for binders and hot mix asphalt mixtures in 1987 [33].

The main features of the Superpave tests and specifications are: [33]

- Instead of performing a test at a constant temperature and varying the specified value, the temperature at which the specified criteria (kept constant) must be met varies in consideration of the binder grade selected for the prevalent climatic conditions
- physical properties measured are directly related to field performance by engineering principles
- the testing regime simulates the three critical stages of a binder's life. Refer to Section 1.3.3 for further details.
- the entire range of pavement temperatures experienced at the project site is considered
- the tests and specifications are designed to eliminate or control three specific types of pavement distresses i.e. rutting (high temperatures), fatigue cracking (intermediate temperatures) and thermal cracking (low temperatures)
- SI units are used for the specifications developed and not imperial units, as previously.

In the Superpave performance specification (M320-05), SHRP introduced two important properties to determine the performance grading of a binder over a range of temperatures and loading rates (angular frequency) i.e. complex modulus (G*) and phase angle (δ), see Table 1.3. These two properties can be measured using a Dynamic Shear Rheometer (DSR). [33]



| Avg 7-day Max, °C | PG 46 | | | PG 52 | | | | | | | PG 58 | | | | | PG 64 | | | | | | PG 70 | | | | | | PG-76 | | | | | PG 82 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1-day Min, °C | -34 | -40 | -46 | -10 | -16 | -22 | -28 | -34 | -40 | -46 | -16 | -22 | -28 | -34 | -40 | -10 | -16 | -22 | -28 | -34 | -40 | -10 | -16 | -22 | -28 | -34 | -40 | -10 | -16 | -22 | -28 | -34 | -10 | -16 | -22 | -28 | -34 |
| | ORIGINAL | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ≥ 230 °C | (Flash Point) FP | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ≤ 3 Pa·s @ 135 °C | (Rotational Viscosity) RV | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ≥ 1.00 kPa | (Dynamic Shear Rheometer) DSR G*/sin | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | 46 | | | 52 | | | | | | | 58 | | | | | 64 | | | | | | 70 | | | | | | 76 | | | | | 82 | | | | |
| | (ROLLING THIN FILM OVEN) RTFO Mass Loss < 1.00 % | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ≥ 2.20 kPa | (Dynamic Shear Rheometer) DSR G*/sin | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | 46 | | | 52 | | | | | | | 58 | | | | | 64 | | | | | | 70 | | | | | | 76 | | | | | 82 | | | | |
| | (PRESSURE AGING VESSEL) PAV | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 20 Hours, 2.07 MPa | 90 | | | 90 | | | | | | | 100 | | | | | 100 | | | | | | 100 (110) | | | | | | 100 (110) | | | | | 110 (110 | | | | |
| ≤ 5000 kPa | (Dynamic Shear Rheometer) DSR G* sin | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | 10 | 7 | 4 | 25 | 22 | 19 | 16 | 13 | 10 | 7 | 25 | 22 | 19 | 16 | 13 | 31 | 28 | 25 | 22 | 19 | 16 | 34 | 31 | 28 | 25 | 22 | 19 | 37 | 34 | 31 | 28 | 25 | 40 | 37 | 34 | 31 | 28 |
| S ≤ 300 MPa m ≥ 0.300 | ( Bending Beam Rheometer) BBR "S" Stiffness & "m"- value | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | -24 | -30 | -36 | 0 | -6 | -12 | -18 | -24 | -30 | -36 | -6 | -12 | -18 | -24 | -30 | 0 | -6 | -12 | -18 | -24 | -30 | 0 | -6 | -12 | -18 | -24 | -30 | 0 | -6 | -12 | -18 | -24 | 0 | -6 | -12 | -18 | -24 |
| Report Value | (Bending Beam Rheometer) BBR Physical Hardening | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| ≥ 1.00 % | (Direct Tension) DT | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | -24 | -30 | -36 | 0 | -6 | -12 | -18 | -24 | -30 | -36 | -6 | -12 | -18 | -24 | -30 | 0 | -6 | -12 | -18 | -24 | -30 | 0 | -6 | -12 | -18 | -24 | -30 | 0 | -6 | -12 | -18 | -24 | 0 | -6 | -12 | -18 | -24 |

*Table 1.3 -The Performance Grade Table Results*

### 1.3.1. Interpretation of G* and δ

G* is defined as the ratio of stress developed and the strain imposed. By using sinusoidal loading in the DSR, the stress and strain developed varies periodically and the frequency of loading can be varied within a test. G* indicates the stress resulting from the strain. Phase angle (δ), which is expressed in degrees, is the phase shift between the applied stress and strain response of the material. This property ranges from 0° for a simple elastic material to 90° representing a simple (Newtonian) viscous material.

Combinations of G* and δ are used to characterise the visco-elastic properties of a binder. G* is made up of two components i.e. viscous component (G'') and elastic component (G').

To derive (G') and (G'') from G* and δ:

Elastic component, (storage modulus) $G' = G^* \cos \delta$

Viscous component, (loss modulus) $G'' = G^* \sin \delta$

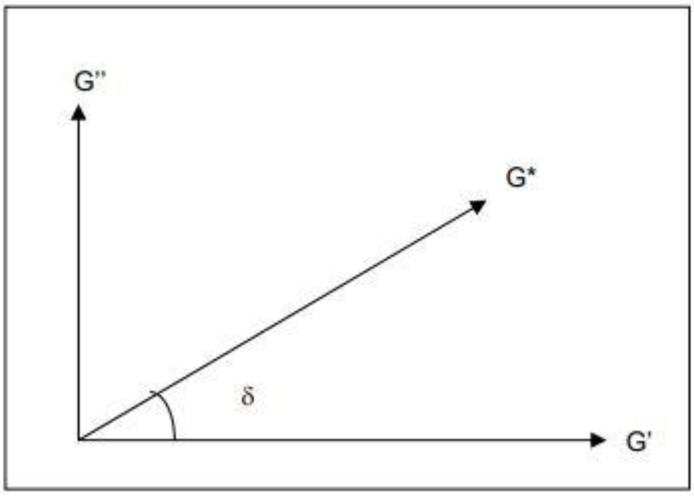


*Figure 1.17 - Relationship between G* and the components G' & G''*

### 1.3.2. SHRP Properties

The PG grading system covers both the high and low service temperatures likely to be experienced by a binder, refer to Table 1.4.

For high service temperature, the PG system defines the specification starting from 46°C through to 82°C in 6°C intervals. To meet the specification requirement, the value of $G^*/\sin\delta$ needs to be a minimum of 1 kPa at the grading temperature and not exceed the requirements of the next grade.

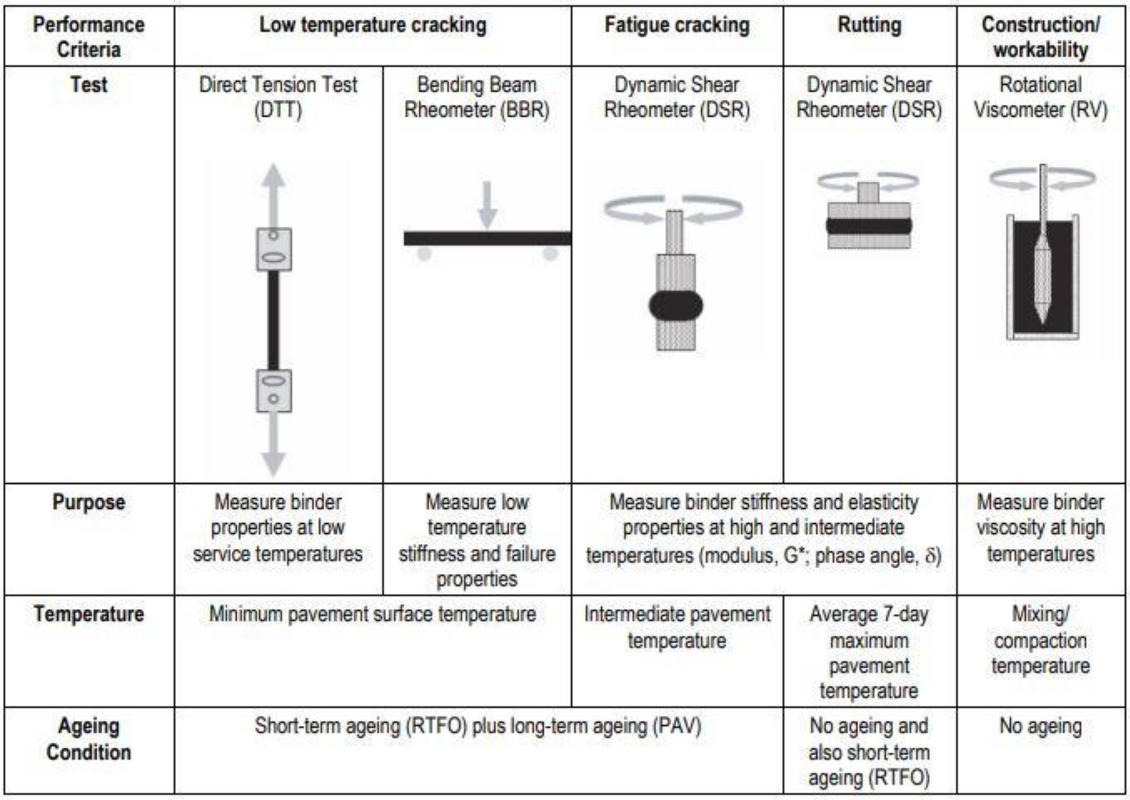
At low service temperature, PG defines the specification requirement as $G^*\sin\delta$.

### 1.3.3. PG Tests and Specifications

The PG system measures the physical properties of binders through tests that are intended to be directly related to field performance by engineering principles. These tests are conducted under the temperature extremes a pavement experiences to simulate the three critical stage of a binder's life, namely: [33]

- first stage – transportation, storage and handling prior to mixing with aggregate.
- second stage – after mixing production and construction (RTFO treatment is used to approximate this stage of short-term ageing).
- third stage – binder ageing on the road (the pressure ageing vessel (PAV) is used to simulate long-term ageing of the binder).

Table 1.4 lists some of the test equipment, temperatures, performance related parameters and ageing conditions of a binder. [33]



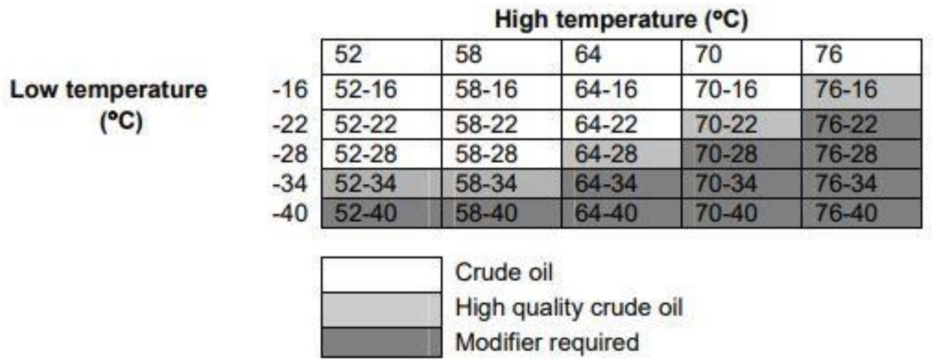
| Performance Criteria | Low temperature cracking | | Fatigue cracking | Rutting | Construction/ workability |
|---|---|---|---|---|---|
| **Test** | Direct Tension Test (DTT) | Bending Beam Rheometer (BBR) | Dynamic Shear Rheometer (DSR) | Dynamic Shear Rheometer (DSR) | Rotational Viscometer (RV) |
| **Purpose** | Measure binder properties at low service temperatures | Measure low temperature stiffness and failure properties | Measure binder stiffness and elasticity properties at high and intermediate temperatures (modulus, $G^*$; phase angle, $\delta$) | | Measure binder viscosity at high temperatures |
| **Temperature** | Minimum pavement surface temperature | | Intermediate pavement temperature | Average 7-day maximum pavement temperature | Mixing/ compaction temperature |
| **Ageing Condition** | Short-term ageing (RTFO) plus long-term ageing (PAV) | | | No ageing and also short-term ageing (RTFO) | No ageing |

*Table 1.4 -PG graded criteria, tests, temperatures and ageing conditions.*

### 1.3.4. Performance Grade Nomenclature

Superpave performance grading is reported using two numbers. The first number indicates the average seven day maximum pavement temperature (°C) while the second shows the minimum pavement design temperature (°C). For example, a PG 64-28 is intended for use in an environment where the average seven day maximum pavement temperature is 64°C and the expected minimum pavement temperature is -28°C.

The working temperature range determines whether a given grade of bitumen requires special manufacture or addition of a modifier. For example, a PG 64-28 has a working range of 92° (64 + 28 = 92). Usually, binders with a temperature difference of 92° or more would generally require some sort of modification (Table 1.5) [33].



| **Low temperature (°C)** | **High temperature (°C)** | | | | |
|---|---|---|---|---|---|
| | 52 | 58 | 64 | 70 | 76 |
| -16 | 52-16 ☐ | 58-16 ☐ | 64-16 ☐ | 70-16 ☐ | 76-16 ▒ |
| -22 | 52-22 ☐ | 58-22 ☐ | 64-22 ☐ | 70-22 ▒ | 76-22 ▓ |
| -28 | 52-28 ☐ | 58-28 ☐ | 64-28 ▒ | 70-28 ▓ | 76-28 ▓ |
| -34 | 52-34 ▒ | 58-34 ▒ | 64-34 ▓ | 70-34 ▓ | 76-34 ▓ |
| -40 | 52-40 ▓ | 58-40 ▓ | 64-40 ▓ | 70-40 ▓ | 76-40 ▓ |

☐ Crude oil
▒ High quality crude oil
▓ Modifier required

*Table 1.5 -PG grades for different bitumen blends.*

# CHAPTER 02: Polymer Modified Bitumen (PMB) and Use of Crumb Rubber in Road Pavement.

# 2. CHAPTER 2: Polymer Modified Bitumen (PMB) and Use of Crumb Rubber In Road Pavment.

## 2.1. Polymer Modified Bitumen (PMB*)*

### 2.1.1. Introduction

Polymer modification of bitumen is the incorporation of polymers in bitumen by mechanical mixing or chemical reaction. During the last 40 years, more and more researchers began to concentrate themselves on polymer modification of bitumen and a rapidly increasing number of research articles have been published since 1970s. In these, the various investigated polymers included plastomers (e.g. polyethylene (PE), polypropylene (PP), ethylene-vinyl acetate (EVA), ethylene-butyl acrylate (EBA)) and thermoplastic elastomers (e.g. styrene-butadiene-styrene (SBS), styrene-isoprene-styrene (SIS), and styrene-ethylene/ butylene-styrene (SEBS)), although none of these were initially designed for bitumen modification. These polymers were reported to lead to some improved properties of bitumen, such as higher stiffness at high temperatures, higher cracking resistance at low temperatures, better moisture resistance or longer fatigue life [20].

Optimize and select appropriate materials for specific applications have been the main goals of materials processing and design engineers worldwide for a long time. Polymer modified bitumens (PMB) are not an exception. Several studies have shown that addition of polymers to bitumen can improve the final properties of PMB.

The two main failure mechanisms suffered by asphalt mixtures are cracking and rutting (permanent deformation). Cracking is usually associated to brittle fracture of bitumen at low temperatures, while rutting is associated to plastic or viscous behavior of bitumen at high temperatures. In order to reduce cracking, bitumen has been typically modified by addition of elastomeric polymers; usually requiring mixing and shearing at high temperatures in order to uniformly disperse the polymers into the blends. In general, the elastomeric polymers added to bitumen build a physical network assisted by bitumen’s phases but do not create a chemical reaction, leaving bitumen as a continuous phase. On the other hand, thermoplastic polymers have been also added to bitumen pursuing improvements on rutting resistance at high temperatures, making use of the hardening effect of these polymers at high temperatures. [18]

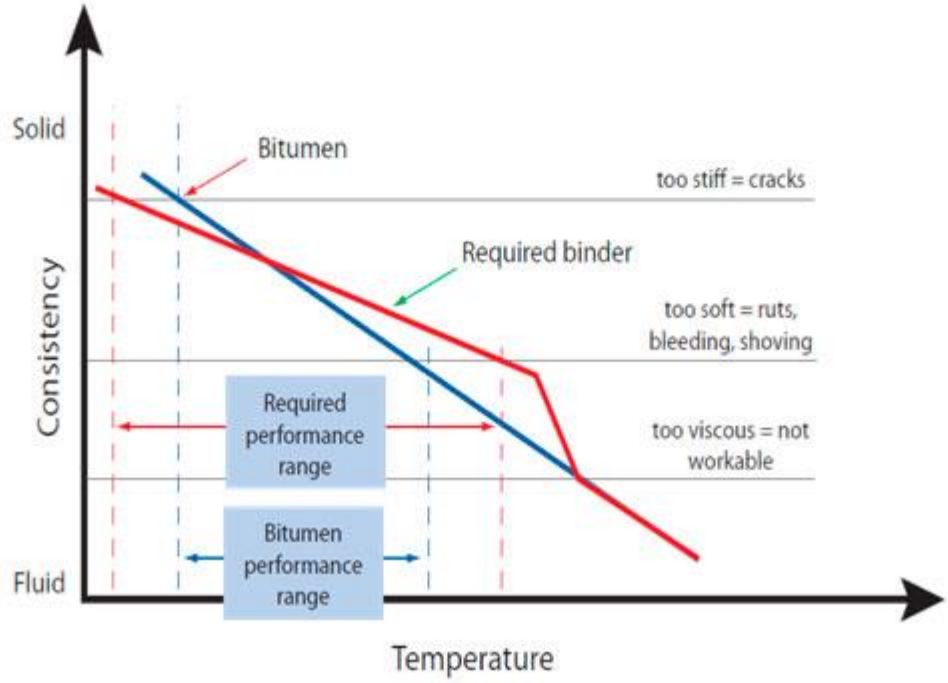


*Figure 2.1 - Typical Viscosity of Binder overlooking temperature. [19]*

### 2.1.2. PMB's qualities & usage:

The main qualities of PMB are:

- Greater Rigidity
- Better resistance to permanent deformation
- Better moisture resistance or longer fatigue life
- Much higher durability
- higher stiffness at high temperatures
- higher cracking resistance at low temperatures [19].

An extensive summary was given that an effective polymer modification results in a thermodynamically unstable but kinetically stable system in which the polymer is partially swollen by the light components of bitumen [20].

The PMB is used for:

- Very stressed pavements
- High traffic volume
- High loading
- High temperature amplitude

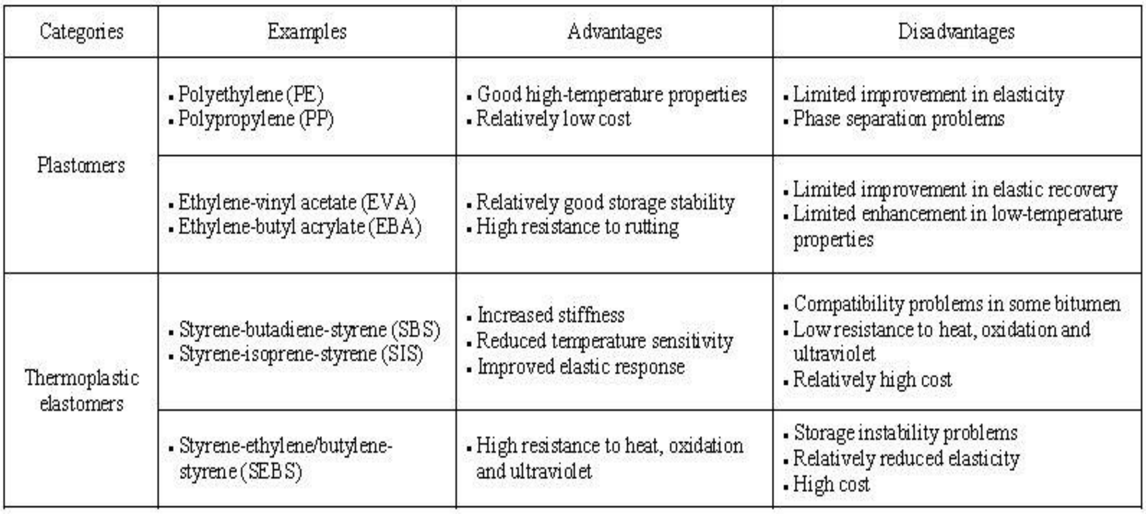
- More durable pavements
- Draining pavements [19].

In addition to the reported advantages, researchers also encountered various challenges, including high cost, some PMBs' high temperature sensitivity, low ageing resistance, poor storage stability and the limited improvement in elasticity. In this, the combination of bitumen oxidation and polymer degradation was reported to cause PMB's ageing propensity, which seems especially challenging for some unsaturated polymers, e.g. SBS. The poor storage stability of some PMBs usually results from the poor compatibility between polymer modifiers and bitumen which is controlled by polymers' and bitumen's different properties such as density, molecular weight, polarity and solubility. The chemical structure and reactivity of polymers, however, are also supposed to affect their compatibility with bitumen, which may have a direct relationship with the resulting PMB properties. In order to conquer these challenges, researchers have tried different categories of solutions, such as saturation, sulfur vulcanization, adding antioxidants, using hydrophobic clay minerals, functionalization and application of reactive polymers (which also can be considered as new functionalized products) [20].

### 2.1.3. Popular polymers for bitumen modification

As mentioned in the above, after World War II ended, synthetic polymers began to be used to modify bitumen. Over the years, researchers developed various polymer modifiers. Today, widely used polymers for bitumen modification can be classified into two categories: plastomers and thermoplastic elastomers. plastomers have little or no elastic component, usually resulting in their quick early strength under load and the following permanent deformation or brittle failure. As for thermoplastic elastomers, they soften on heating, harden on cooling and are able to resist permanent deformation by stretching under load and elastically recovering once the load is removed, which leads to their greater success than plastomers as bitumen modifiers. Some popular polymers for bitumen modification are listed in Table 1 with their advantages and disadvantages. Among them, SBS attracted the most attention due to its relatively good dispersibility (or appropriate solubility) in bitumen as well as the relatively excellent properties and acceptable cost of SBS modified bitumen. Of course, besides these listed polymers, some others like styrene-butadiene rubber (SBR, random copolymers), styrene-butadiene diblock copolymers (SB) and ethylene-propylene-diene monomer rubber (EPDM) were also popular for bitumen modification. In addition, some small molecule organic materials, such as PPA and paraffin wax, were also widely used as modifiers for bitumen [20].

It is worth to note that even for a given polymer modifier, selection of base bitumen still has some important effects on the resulting PMB, as each bitumen has its own particular chemical composition and structure. Additionally, base bitumen usually composes over 90% of the PMB by weight, which could introduce overriding influences on the final properties of the PMB. Good-quality base bitumen helps to enhance the effects of polymer modification, while poor-quality one may make the modification futile. Regarding the compatibility between polymer and bitumen, selection of base bitumen is usually completed by laboratory experiments. However, some theoretical trends were also highlighted based on the SARA (saturates, aromatics, resins and asphaltenes) fractions of bitumen: for example, high asphaltenes content may decrease the compatibility between polymer and bitumen and the aromaticity of the maltenes needs to fall between certain values to reach a good level of compatibility. Some other researchers even gave the components distribution of base bitumen with the optimum compatibility with SBS [20].



| Categories | Examples | Advantages | Disadvantages |
|---|---|---|---|
| Plastomers | ▪ Polyethylene (PE)<br>▪ Polypropylene (PP) | ▪ Good high-temperature properties<br>▪ Relatively low cost | ▪ Limited improvement in elasticity<br>▪ Phase separation problems |
| | ▪ Ethylene-vinyl acetate (EVA)<br>▪ Ethylene-butyl acrylate (EBA) | ▪ Relatively good storage stability<br>▪ High resistance to rutting | ▪ Limited improvement in elastic recovery<br>▪ Limited enhancement in low-temperature properties |
| Thermoplastic elastomers | ▪ Styrene-butadiene-styrene (SBS)<br>▪ Styrene-isoprene-styrene (SIS) | ▪ Increased stiffness<br>▪ Reduced temperature sensitivity<br>▪ Improved elastic response | ▪ Compatibility problems in some bitumen<br>▪ Low resistance to heat, oxidation and ultraviolet<br>▪ Relatively high cost |
| | ▪ Styrene-ethylene/butylene-styrene (SEBS) | ▪ High resistance to heat, oxidation and ultraviolet | ▪ Storage instability problems<br>▪ Relatively reduced elasticity<br>▪ High cost |

*Table 2.1- Popular polymers for bitumen modification.*

#### 2.1.3.1. Plastomers

As an important category of plastomers, polyolefin is one of the earliest used modifiers for bitumen. Various polyolefin materials, including high-density polyethylene (HDPE), low density polyethylene (LDPE), linear low-density polyethylene (LLDPE), IPP and APP, have been studied for application in bitumen modification due to the relatively low cost and the benefits they might bring. Typical Structures of the popular PE and PP are given in Figure 2.2. After polyolefin materials are added into bitumen, they are usually swollen by the light components of bitumen and a biphasic structure is formed with a polyolefin phase (dispersed phase) in the bitumen matrix (continuous phase). As the polyolefin concentration increases, phase inversion occurs in the modified bitumen. Two interlocked continuous phases are ideal for polyolefin modified bitumen, which could improve the properties of bitumen to some extent. Those used materials were usually found to result in high stiffness and good rutting resistance of modified bitumen, although they are quite different in chemical structure and properties.

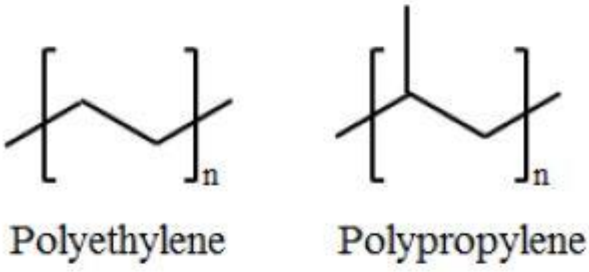


*Figure 2.2 -Structures of polyethylene (PE) and polypropylene (PP).*

However, those used polyolefin materials failed to significantly improve the elasticity of bitumen. In addition to this, the regular long chains of those polyolefin materials give them the high tendency to pack closely and crystallize, which could lead to a lack of interaction between bitumen and polyolefin and result in the instability of the modified bitumen. Furthermore, some researchers claimed that the compatibility of polyolefin with bitumen is very poor because of the non-polar nature of those used materials. As a result, the limited improvement in elasticity and potential storage stability problems of polyolefin modified bitumen restrict the application of polyolefin materials as a bitumen modifier, whereas they are popular in production of impermeable membranes.

More used plastomers in bitumen modification are ethylene copolymers, such as EVA and EBA. Due to their similar chemical structures, EVA is discussed here as an example of ethylene copolymers. As seen in Figure 2.3, EVA copolymers are composed of ethylene-vinyl acetate random chains. Compared with PE, the presence of polar acetate groups as short branches in EVA disrupts the closely packed crystalline microstructure of the ethylene-rich segments, reduces the degree of crystallization and increases the polarity of the polymer, which were both believed to be beneficial to improving the storage stability of modified bitumen by some researchers. However, the properties of EVA copolymers are closely related to the vinyl acetate content. When the vinyl acetate content is low, the degree of crystallization is high and the properties of EVA are quite similar to those of LDPE. As the vinyl acetate content increases, EVA tends to present a biphasic microstructure with a stiff PE-like crystalline phase and a rubbery vinyl acetate-rich amorphous phase. The higher the vinyl acetate content, the higher the proportion of amorphous phase. But the degree of crystallization should be controlled carefully when EVA is used as a bitumen modifier, because neither too low (getting easy to be disrupted) nor too high (causing the lack of interactions with bitumen) degree of crystallization is good for bitumen modification [20].

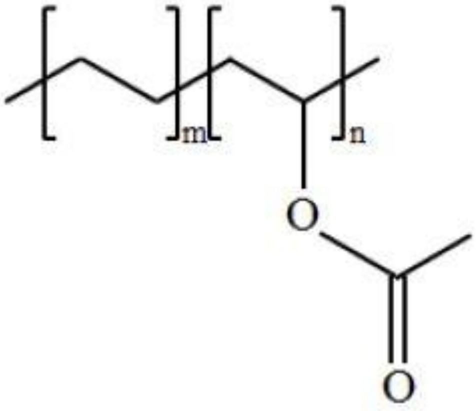
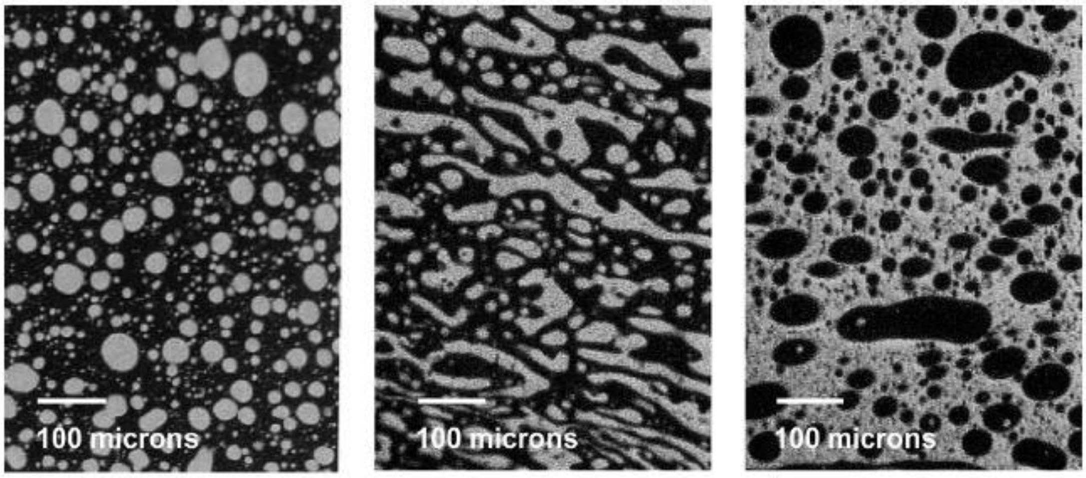


*Figure 2.3 -Structure of ethylene-vinyl acetate (EVA).*

After EVA copolymers are added into bitumen, the light components of bitumen usually swell the copolymers. At low EVA concentrations, a dispersed EVA-rich phase can be observed within a continuous bitumen-rich phase. As the EVA concentration increases, phase inversion occurs in modified bitumen and the EVA-rich phase becomes a continuous phase. The process of phase inversion in EVA modified bitumen was presented by fluorescent images as Figure 2.4. If two interlocked continuous phases form in the modified bitumen, the properties of bitumen could be improved to a large extent. EVA was found to form a tough and rigid network in modified bitumen to resist deformation, which means that EVA modified bitumen has an improved resistance to rutting at high temperatures.



Bitumen B + 3% EVA Bitumen B + 5% EVA Bitumen B + 7% EVA

*Figure 2.4 -Fluorescent images of EVA modified bitumen with various contents (by weight) of EVA. [20]*

Although some properties of bitumen are enhanced by EVA modification, there are still some problems limiting its application. One large limitation is the fact that EVA cannot much improve the elastic recovery of bitumen due to the plastomer nature of EVA. Furthermore, the glass transition temperature (Tg) of EVA copolymers, which strongly depends on the vinyl acetate content, is not low enough to significantly improve the low-temperature properties of bitumen. It was reported that (Tg) of EVA copolymers with 28.4 wt % of vinyl acetate is -19.9 °C [129], which is even quite close to (Tg) of some base bitumen. As a result, EVA's ability to improve the low-temperature properties of bitumen is rather limited, especially at high EVA concentrations [20].

#### 2.1.3.2. Thermoplastic elastomers

Thermoplastic elastomers are usually more effective than plastomers for bitumen modification. The most popular thermoplastic elastomers as bitumen modifiers are SBS copolymers and SIS copolymers. Due to their similar chemical structures, SBS is discussed here as an example of thermoplastic elastomers. SBS copolymers are composed of styrene-butadiene- styrene triblock chains with a biphasic morphology of rigid polystyrene (PS) domains (dispersed phase) in the flexible polybutadiene (PB) matrix (continuous phase), shown as Figure 2.5.A. The chemical linkages between PS and PB blocks can immobilize domains in the matrix. Tg of PS blocks is around 95 °C and Tg of PB blocks is around -80 °C. Under the usual service temperatures of paving bitumen, PS blocks are glassy and contribute to the strength of SBS while PB blocks are rubbery and offer the elasticity. Furthermore, the incompatibility between PS and PB blocks makes it possible to physically crosslink PS blocks as uniformly distributed domains by intermolecular forces at ambient temperatures. This aggregation of PS blocks disappears at high temperatures when the kinetic energy of molecular thermodynamic movements is greater than the energy of intermolecular forces. However, as shown in Figure 2.5, the physical crosslinking among PS blocks can be reformed and the strength and elasticity of SBS can be restored after cooling, which is very important for SBS to be a popular bitumen modifier.

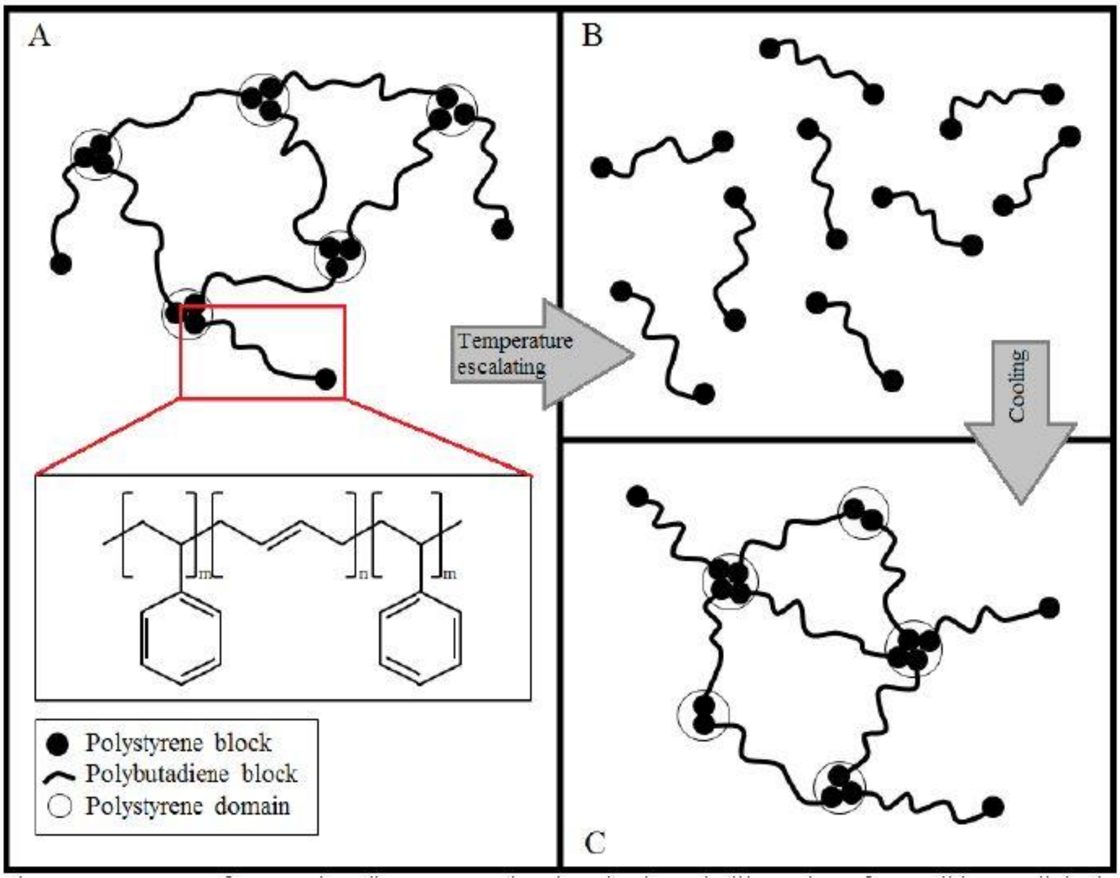


*Figure 2.5 -Structure of styrene-butadiene-styrene (SBS) and schematic illustration of reversible crosslinks in SBS.*

After SBS copolymers are added into bitumen, some interactions happen between bitumen and SBS. Intermolecular interactions between bitumen and the PB blocks are stronger than those with the PS blocks. They believed that PB blocks interact with positively charged groups in bitumen through their π-electrons, whereas PS blocks interact with electron-rich groups in bitumen through their aromatic protons. Mixed with bitumen, PS blocks in SBS copolymers absorb some saturated branches and a few rings in light components of bitumen, which leads to the swelling of PS blocks and the hardening of bitumen. When the polymer content is low, SBS is dispersed as a discrete phase in the bitumen. As the SBS concentration increases, phase inversion starts in the modified bitumen. The process of phase inversion in SBS modified bitumen was presented by fluorescent images as Figure 2.6. It is ideal to form two interlocked continuous phases: bitumen-rich phase and SBS-rich phase. Within the SBS-rich phase, there are two subphases: swollen PB matrix and essentially pure PS domains. Once the SBS-rich phase forms, a rubbery supporting network is created in the modified bitumen, which results in the increased complex modulus and viscosity, improved elastic response and enhanced cracking resistance at low temperatures of SBS modified bitumen.

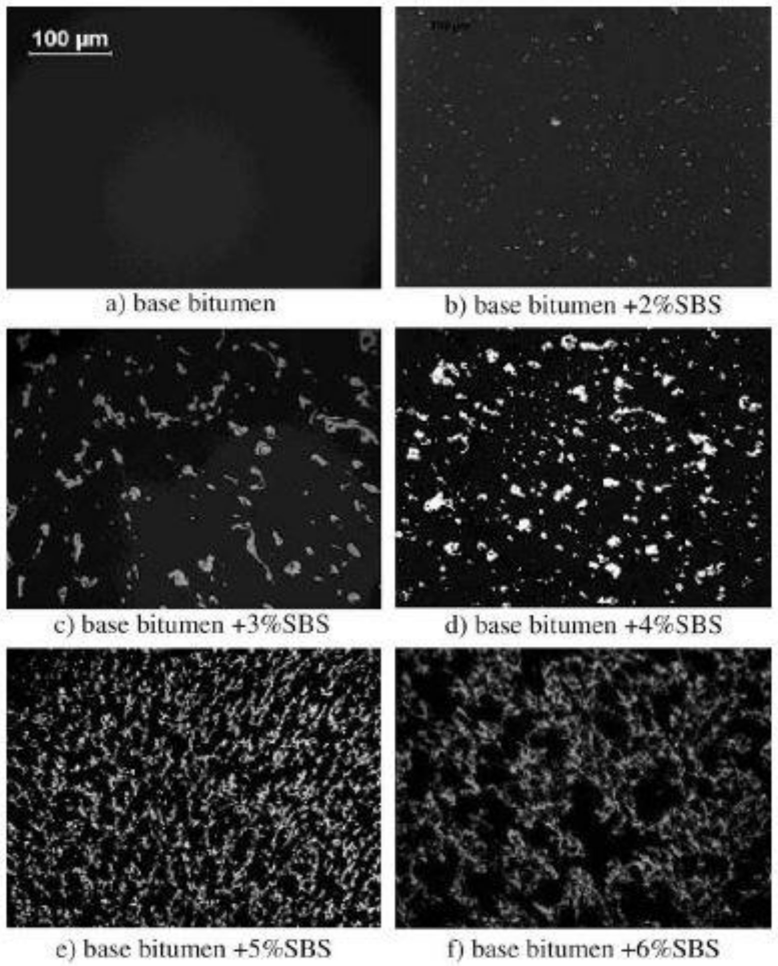


*Figure 2.6 -Fluorescent images of SBS modified bitumen with various contents (by weight) of SBS.*

The repeatedly reported excellent properties, relatively good dispersibility (or appropriate solubility) in bitumen and acceptable cost have made SBS popular as a bitumen modifier.

However, SBS copolymers are far from perfect. For example, the compatibility between bitumen and SBS is not that good. Storage instability of SBS modified bitumen was reported with images as Figure 2.7. Thermoplastic elastomers and asphaltenes compete to absorb the light components of bitumen in SBS bitumen blends. If these light components are insufficient, phase separation could occur in modified bitumen. It was noted that bitumen with high aromatics content can be helpful in producing a compatible and stable SBS modified bitumen and addition of aromatic oils can improve the compatibility between SBS and some bitumen with low aromatics content. Too high aromatics content in modified bitumen, however, may lead to the swelling and anti-plasticization of some PS blocks, which is not good for the resulting properties of the modified bitumen [20].

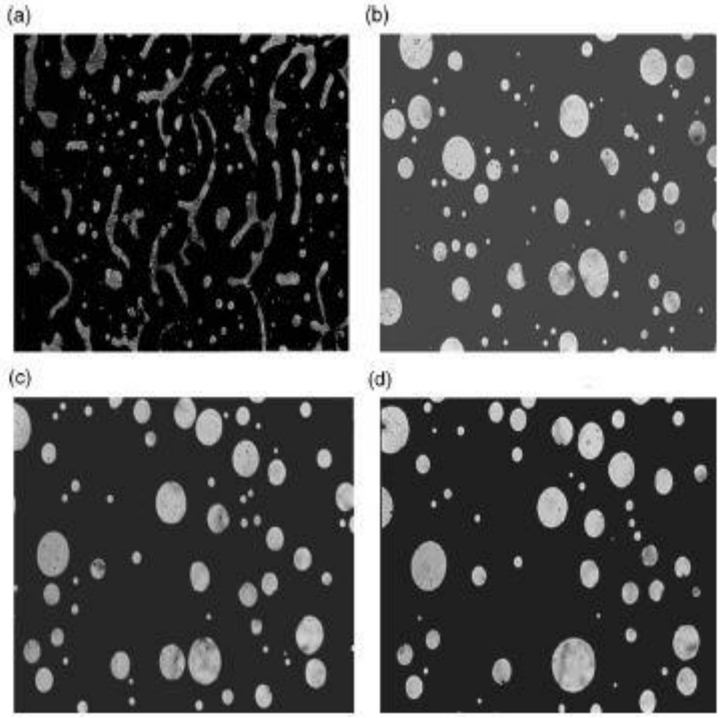


*Figure 2.7 -Morphology development with the storage time of a SBS modified bitumen at 160 °C.*

Another problem with SBS modification of bitumen is its low resistance to heat, oxidation and ultraviolet (UV) because of the presence of double bonds and $\alpha$-H in PB blocks. In fact, the instability of SBS copolymers is mainly due to the high activity of $\alpha$-H and low bond energy of the $\pi$-bond in double bonds. Undesired chemical reactions (e.g. formation of peroxy radicals and hydroperoxides) make them sensitive to heat, oxidation and UV. In order to overcome this disadvantage, researchers firstly paid much attention to saturated thermoplastic elastomers such as SEBS.

SEBS copolymers, which can be obtained by hydrogenation of SBS, consist of triblock styrene-ethylene/butylene-styrene chains. The chemical saturation makes them highly resistant to heat, oxidation and UV. However, as the double bonds disappear, some researchers claimed that the polarity of the copolymers is considerably reduced. Meanwhile, the ethylene/butylene blocks in SEBS have a trend to crystalize. So the compatibility between SEBS and bitumen was believed to become even worse. According to the research by Polacco et al. [21], stable SEBS modified bitumen can only be prepared at a low polymer content (below about 4 wt% of the total mass) when SEBS acts just as filler and does not improve the viscoelastic properties of bitumen significantly. On the contrary, when SEBS content is high enough to really modify bitumen, the prepared PMB is unstable and tends to phase separate. Additionally, extra cost involved by the hydrogenation process and poorer elastic properties were observed in SEBS modified bitumen, which further limits its application as a bitumen modifier [20].

In order to avoid drawbacks of SEBS modification, researchers from Mexico attempted to use partially saturated SBS copolymers in bitumen modification. They prepared styrene-butadiene-ethylene/butylene-styrene (SBEBS) copolymers with various degrees of saturation by partial hydrogenation of SBS copolymers and found that SBEBS modified bitumen has better mechanical properties (e.g. higher rutting resistance and better elasticity) than SBS modified bitumen. Although partial hydrogenation may also cause weaker polarity and possible crystallization of the copolymers, it was claimed that SBEBS dispersed better in bitumen and led to improved storage stability of modified bitumen in the research. An explanation for this phenomenon was given in terms of solubility parameters of copolymers in aromatic compounds. However, no further reports on the application of SBEBS are found to support its success in bitumen modification.

Another attempt for enhancing the ageing resistance of SBS modified bitumen was to transfer the double bonds from the backbone to branches, i.e. using high vinyl content SBS copolymers. From 1,3-butadiene, people usually prepare SBS copolymers with the structure as Figure 2.5.A by 1,4-addition mechanism. Some researchers claimed that a novel class of SBS copolymers, called high vinyl content SBS copolymers, can be obtained from 1,3- butadiene by 1,2-addition mechanism with special additives and processing conditions. This SBS copolymer has the double bonds on the branches, which was believed to result in lower viscosity and better compatibility with bitumen. As heat, oxidation and UV preferably attack double bonds on branches, the backbone tends to be left intact. So it was claimed that the ageing resistance of SBS modified bitumen modified could be improved by using high vinyl content SBS copolymers. In addition to this, when employed to modify hard bitumen for base layers, this SBS copolymer was believed to reduce the layer thickness by as much as 40% and material cost by some 25%. A representative patent, which relates high vinyl content diblock copolymers, linear triblock copolymers, multiarm coupled block copolymers and mixtures thereof, was granted to Scholten and Vonk of Kraton Polymers. However, people currently do not have much experience with high vinyl content SBS copolymers. It is still necessary to carry out more research and field tests to find out to what extend they work for bitumen modification, especially in service. Care should still be taken now when introducing high vinyl content SBS copolymers to bitumen modification.

### 2.1.4. Recyclability of PMB.

Almost 30 years have passed since PMB began to be increasingly used in the late 1980s. Many of the early-constructed PMB pavements have reached the end of their service life and need resurfacing. It complies with the principle of sustainable development to recycle PMB after its service life ends. Researchers tried to investigate the recyclability of PMB, especially the most widely-used SBS modified bitumen. Although some of these investigations concluded that it is technically feasible to recycle aged PMB by adding rejuvenators or virgin bitumen, there is still no widely-accepted PMB recycling technique available today, which also affects the popularization of PMB in turn. Additionally, the mechanism of PMB ageing and rejuvenating is still not well understood. So, in the future, more research should be focused in this direction. As for developing new-type polymer modifiers, the concept of sustainable design should be introduced. Many of the current problems with recycling result from the fact that the property of recyclability was not involved when most products were designed. If a modifier is initially designed with recyclability in mind, it will lead to products with better evaluation of life cycle and its popularization will be much easier [20].

## 2.2. Modified bitumen with rubber powder

### 2.2.1. Introduction

Crumb rubber (CR) derived from grinding of end-of-life tyres (ELTs) may be successfully used as a bitumen modifier or as a supplementary component in the production of bituminous mixtures employed for the construction and maintenance of road pavements. However, CRs deriving from different sources and production processes yield effects on performance of corresponding paving mixtures under traffic loading and on gaseous emissions produced during laying on site which may change considerably depending upon their physical and chemical properties. In order to quantitatively assess the possible variability of CR characteristics [22].

Management of end-of-life tyres (ELTs) has become a critical problem worldwide due to the increasing number of vehicles circulating in the road network and to the crucial role that mobility has assumed in society development. Since landfill disposal has been banned in most Countries, alternative final destinations have been sought, with a major effort being placed in trying to exploit in the most efficient manner the high energy potential of ELTs. Nevertheless, due to the fact that rubber employed in tyre fabrication is the result of specialized materials' selection, recycling and reuse seem to be preferable options for such a high-quality waste material [22].

Practical experience and research have shown that crumb rubber (CR) derived from grinding of ELTs may be successfully used as a bitumen modifier or as a supplementary component in the production of bituminous mixtures employed for the construction and maintenance of road pavements. Available technologies can be grouped into two main categories which are associated to the so-called "wet" and "dry" production processes. Within each group, different versions of the technology have been conceived and subjected to trials either in the laboratory or at the industrial scale, in the constant attempt of exploiting more efficiently the performance-related benefits of CR.In the "wet" process, CR is preliminarily mixed with bitumen, thus obtaining a ductile and elastic modified binder, known as “asphalt rubber” (ASTM D6114-09), that is then combined with aggregates in the hot mix plant. Resulting mixtures are generally of the gap-graded (GG) or open-graded (OG) type, characterized by a very high binder content (of the order of 7.5–10% b.w. of dry aggregates) and by a non-continuous particle size distribution that allows CR to be accommodated within the composite material. GG and OG mixtures are employed for the formation of surface courses and have earned a satisfactory reputation with respect to field performance [22].

In the “dry” method, CR is introduced in the production flow of bituminous mixtures as a supplementary component, substituting part of the aggregates and providing enhanced elastic response under loading. Mixtures are usually of the dense-graded (DG) type, with a continuous particle size distribution and an optimal binder content (usually of the order of 5–6%) which is only slightly higher than that adopted for standard mixtures containing no recycled rubber. Unfortunately, the performance record of these mixtures has been quite inconsistent, with the frequent occurrence of early ravelling phenomena and moisture-related damage. This also explains the limited diffusion of such a technology, with full-scale applications that have been generally carried out locally rather than at the network level.

For both the abovementioned technologies, concerns have been raised on the use of CR in bituminous mixtures with respect to its potential contribution to gaseous emissions during production and laying, and to the possible consequences which it can cause on the health of construction workers. However, a limited number of experimental studies have been carried out on this specific topic, with no clear quantification of the actual hazardous effects of CR [22].

### 2.2.2. CR production

Processing of ELTs is carried out in various phases during which rubber is separated from other materials (textile fibers and metals), mechanically reduced in granular form and finally divided into particle size fractions. When shredding, shear and abrasion operations occur with no specific temperature conditioning, the process is known as “ambient size reduction”. In some plants, however, ELTs are brought below rubber glass transition temperature with adequate cooling systems and the resulting process is referred to as “cryogenic”. Moreover, novel processing methods are continuously developed in order to optimize plant efficiency. As an example, size reduction has also been attempted by means of the “high pressure waterjet” system, based on the abrasive effects caused by water jets at 3.000 bar which pulverize ELTs.

Depending upon the type of treatment process and on the origin of ELTs fed to it, CR may have different physical and chemical characteristics. In particular, particles deriving from ambient size reduction generally have irregular shape and rough surface; moreover, it has been postulated that in some cases heat generated during mechanical processing may induce a partial devulcanization of rubber. By comparison, cryogenically produced CRs are mostly made of cuboid-shaped particles with a smooth surface. With respect to ELT origin, it is well known that tire producers employ different rubber formulations and that truck tires generally have a higher natural rubber content than car tires. However, in practice only the second factor may influence CR production since there are plants that treat only truck (or car) tires, but none are dedicated to a single tire producer.

### 2.2.3. CR-bitumen interaction

In the asphalt rubber “wet” production process, CR is thoroughly mixed with bitumen at a temperature in the 175–225 °C range. The resulting binder is then kept in agitation at high temperature (150–215 °C) for the time period (of the order of 45–60 min) which is necessary for interaction phenomena between the components to occur. In particular, CR particles are partially digested in the bituminous matrix and absorb part of the aromatic fraction of bitumen, with a resulting volume expansion and formation of a gel-like surface coating which gives the binder its peculiar physical and rheological characteristics. In this form, CR particles are still visible in the composite binder which has a distinctive granular-like appearance. If curing is carried out at an excessive temperature and/or for a too long time period, degradation phenomena become prevalent and CR is totally digested in bitumen: as a consequence, the resulting binder does not have the typical characteristics of asphalt rubber and may exhibit unsatisfactory performance.

Asphalt rubber binders usually have a CR content comprised between 18% and 22% (b.w. of total binder), with a high viscosity at storage/mixing temperatures and enhanced elastic properties in service. Binder characteristics are dependent not only upon chemical composition of employed components, but also on CR dosage, particle size and morphology. In such a context, it has been proven that the intensity of the above described interaction phenomena tends to increase with CR dosage and specific surface area. Therefore, CRs which are considered more reactive are those which are finer, constituted by rough, irregular particles.

In the production of “dry” mixtures CR is usually employed with a dosage comprised between 1% and 3% (b.w. of dry aggregates). Depending upon the type of plant, either batch or drum-mix, CR can be introduced in the production flow of bituminous mixtures by means of different methods but is always added to the heated aggregates before coming in contact with bitumen. When this condition occurs, even though CR particles are not digested in bitumen, they do absorb part of its aromatic fractions. However, such an interaction takes place in non-controlled conditions, starting from the mixing process in the plant and progressing throughout the early phases of service life. Studies performed in the past have shown that in this respect beneficial effects can be obtained by pretreating CR by means of function-specific catalysts or extender oils [22].

### 2.2.4. CR selection and acceptance

Selection and acceptance of CR for use in asphalt rubber classically relies upon the requirements that have been set in ASTM D6114. These are expressed in terms of cleanliness (fiber content <0.5%; metal content <0.01%), moisture content (<0.75%), density (equal to 1.15 ± 0.05) and maximum particle size (2.36 mm). However, the standard also specifies that the exact size distribution of CR should be agreed upon between producer and end-user.

Based on experience and local tradition, in other contexts requirements may be slightly different. As an example, technical specifications of the California State Department of Transportation require the use of natural and synthetic rubber in given proportions and provide size distribution acceptance intervals which should be satisfied by single CR components [22].

In the case of the “dry” production technology, depending upon the desired effect on the resulting bituminous mixture, CR can be employed with different size distributions, ranging from “ultrafine” (entirely passing the 0.3 mm sieve) to “coarse” (passing the 6 mm sieve and totally retained on the 2.36 mm sieve). Since it has been recognized that size distribution (and related specific surface area) is the main factor controlling performance properties of “dry” mixtures, specific requirements are usually not set on other CR characteristics.

Physical and rheological properties of asphalt rubber are also subjected to acceptance requirements in technical specifications. This is done either by referring to the results of classical empirical tests (penetration, softening point, etc.) or by considering viscoelastic properties measured at representative temperatures, frequencies and ageing conditions. All evaluation systems also include acceptance criteria referred to apparent viscosity, which is typically required to be comprised between 1500 and 5000 mPa s. Such a property, which refers to the flow behavior of asphalt rubber, is considered as a good quality indicator that can be also measured on site, from storage tanks, with portable hand-held viscometers.

## 2.3. The Use of Recycled Tire Rubber (Crumb Rubber) to Modify Asphalt Binder and Mixtures.

Crumb rubber (CR), from waste tires (Figure2.8) has been used in asphalt by the paving industry since the 1960’s. CR has been used as an asphalt binder modifier and asphalt mixture additive in gap-graded and open-graded asphalt mixtures and surface treatments [23].

*Figure 2.8 - Picture of a waste tire pile.*

Until recently the routine use of CR in pavements has been limited to a few states. While performance is generally good, CR cost has been high when compared to conventional practices. Asphalt binder costs have increased over the past several years due to the rising cost of crude oil. In addition, polymers, such as styrene-butadiene-styrene (SBS), have also seen an increase in cost due to other market demands and fluctuations in availability. In contrast, over this same time period CR from car and truck tires has experienced a relatively stable market price. Local, State, and Federal regulations have also created an increase in the availability of recycled tire rubber. This has driven a renewed interest in CR as an asphalt binder modifier and mixture additive – with the goal of providing a long-life, cost-competitive, environmentally-responsible pavement system [23].

In 2006 the FHWA Recycled Materials Policy was established and is located at: http://www.fhwa.dot.gov/legsregs/directives/policy/recmatpolicy.htm

The FHWA policy states:

1. Recycling and reuse can offer engineering, economic and environmental benefits (Figure 2.9).
2. Recycled materials should get first consideration in materials selection.

3. Determination of the use of recycled materials should include an initial review of engineering and environmental suitability.
4. An assessment of economic benefits should follow in the selection process.
5. Restrictions that prohibit the use of recycled materials without technical basis should be removed from specifications.

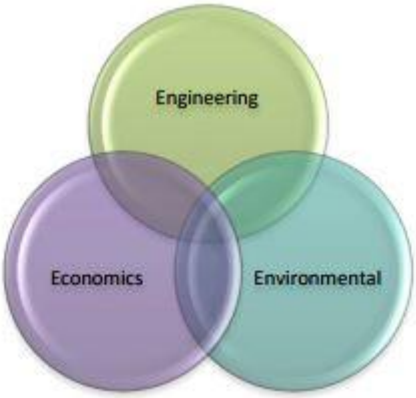
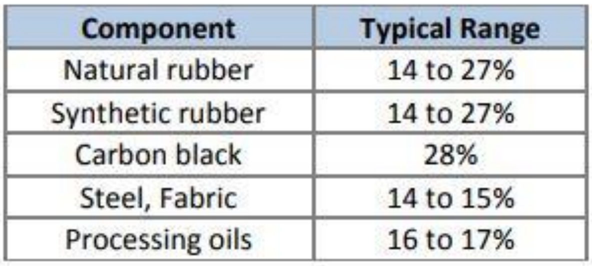


*Figure 2.9 - The three key benefits of recycled/reused materials.*

This Technical Brief provides background on the various CR processes used in asphalt pavements and information on how to incorporate CR into the Superpave design system [23].

### 2.3.1. Tires

The make-up of tires varies depending on the type, truck or passenger, and manufacture. However, the basic components are about the same and are provided in Table 2.2 below.



| Component | Typical Range |
|---|---|
| Natural rubber | 14 to 27% |
| Synthetic rubber | 14 to 27% |
| Carbon black | 28% |
| Steel, Fabric | 14 to 15% |
| Processing oils | 16 to 17% |

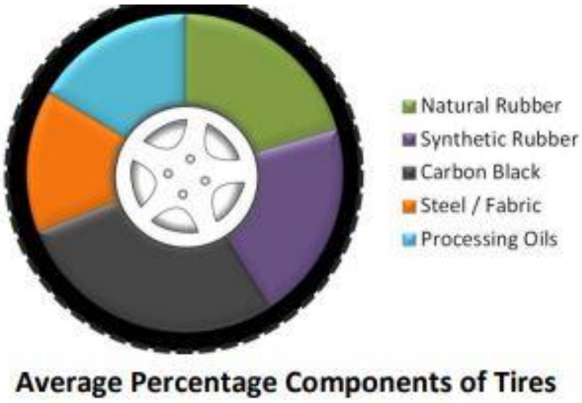


*Table 2.2 - Basic Components of Tires*

In the past it was believed that different types of tires or different portions of the tires produced better material for blending with asphalt binder. However, with modern tires there is little difference between truck and passenger tires. There are slight variances in the percentage of natural and synthetic rubber, but this is not believed to cause differences in CR modified binder performance.

**Tire Processing:**

Processing is required to make tires usable as a modifier or additive. The steel and fiber must be removed from the tires and then the remaining tire must be reduced in size to small particles for blending into the asphalt binder or mixture. Several different processes are used to reduce the CR size for asphalt modification. The two primary processes are ambient grinding and cryogenic fracturing. Processing the tires using cryogenic fracturing involves cutting up the larger tire pieces into smaller, typically 50 mm particles, using sharp steel cutters. These smaller pieces are then frozen and fractured. The fracturing process produces a large variety of sizes from very small, passing the 75 µm sieve, to larger 4 or 5 mm size particles. The rubber particles produced by cryogenic fracturing have a tendency to be cubical with a smooth surface. The ambient grinding process starts the same way as the cryogenic process; the tires are cut into smaller pieces with sharp cutting blades. The smaller pieces are then passed through shredders that grind and tear the rubber into smaller particles. Similar to cryogenic fracturing, the ambient grinding produces sizes ranging from small, passing the 75 µm sieve, to larger 4 to 5 mm size particles. The main difference between the two processes is the surface texture of the rubber particles. Ambient grinding produces a rough texture with increased surface area due to the tearing process. Figure 2.10 below shows the two different types of particles. On the left is the ambient grind with the rough surface texture and on the right is cryogenic fractured with smoother surface texture.

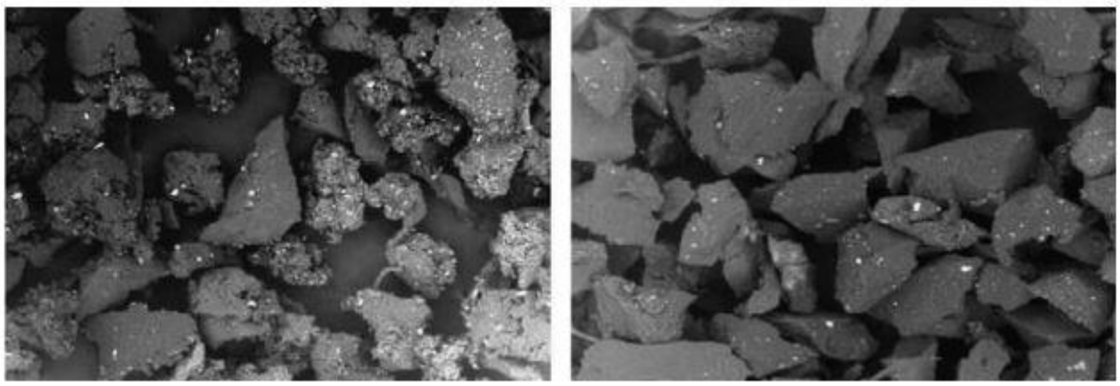


*Figure 2.10 -Ambient grind rubber (left) and Cryogenic fractured rubber (right).*

The surface area of the rubber particles, in addition to blending temperature, controls how fast the particles will react with asphalt binder where the rubber particles absorb some of the asphalt binder and swell. The greater the surface area the faster the reaction time with asphalt binder. It is critical to know the size of the rubber to control the reaction process. Using a different size in production than used in the mix design may not produce optimal results [23].

### 2.3.2. Various Processes for Creating CR Binders and Mixtures

Figure 2.11 shows the three general processes used for creating CR asphalt pavements.

*Figure 2.11 -General processes for creating CR binders and mixtures.*

Each of these processes will produce AR pavements with different properties and different performance. Agencies need to understand these differences so they can make a choice on the type of process that will perform best for their desired application. Agencies are encouraged to determine their goals when using CR so the correct process can be selected for their needs. Further, each of these processes has different risks in terms of success. Agencies need to understand the testing and inspection resources necessary to commit to each of these types of processes to ensure success.

#### 2.3.2.1. Dry Process

CR used in the dry process is considered to be an aggregate replacement in the mix as opposed to a binder additive. Dry process asphalt rubber is the least commercially significant type of asphalt rubber. Dry CR is added similar to reclaimed asphalt pavement

(RAP) at the mixture production plant. The rubber is typically larger size particles between 4 to 18 mesh or 4.75 to 1.00 mm. Cryogenic rubber is typically used in this process. Gapgraded aggregate mixtures are required to provide space for the rubber particles. Figure 2.12. shows CR being auger fed into the RAP collar on the drum of a mixture production plant.

*Figure 2.12 -Feed system to add dry CR into mixture production plant.*

#### 2.3.2.2. Wet Process – On-site Blending

The AR wet process with on-site blending has the longest history of use, Figure 2.13. The CR is field blended in a mixing tank and allowed to react with the asphalt binder for a set time. CR is typically field blended at 350 to 400°F (175 to 200˚C) for 45 to 60 minutes. The temperature and time depend on the base asphalt binder grade, percentage, and particle size of the CR. During this reaction time the rubber particles absorb some of the light fractions of asphalt binder and swell. This absorption and swelling causes an increase in the viscosity of the AR-asphalt binder blend. With extended reaction times the viscosity will then decrease slightly. This has typically been called “digestion” of the rubber in the asphalt binder.

*Figure 2.13 -Wet Process On-site.*

The typical CR addition is 15 to 22 percent by weight of the asphalt and rubber blend. A minimum of 15 percent was initially set to maximize use of recycled tires and has not changed. This initial minimum percentage was not set for performance-related rationale. This is a recipe formulation and may not necessarily produce the optimum performance for traffic or environment at the project. A course graded CR material, 10 to 14 mesh or 2.0 to 1.4 mm maximum size is used. The larger CR particle size requires a gap-graded or open-graded aggregate in the mixture to allow room for the rubber particles. If this is not done, compaction is difficult to achieve because the rubber particles push the aggregate particles apart as they are compressed during rolling and expand when the compaction force is removed.

The increase in viscosity that CR provides to the asphalt binder also requires an appropriate increase in production temperatures for producing and placing mix. Increased temperatures can create unique odors and the potential for smoke. Worker health and safety issues need to be considered. Warm mix asphalt (WMA) technologies have been successfully used to help reduce AR mixture production and placement temperatures [23].

**Best Practice:** The City of Phoenix has a small geographic area. By moving field blending of the RTR to a terminal, they have found more consistent quality control.

**Best Practice:** In California strategically located “depots” exist for just-in-time supply of RTR to small and medium projects. The “depots” have a focus on quality control.

**CAUTION:** Quality control during field blending is a significant factor that Agencies need to consider. In the past, Agencies have made major changes to their AR program to ensure quality control concerns.

#### 2.3.2.3. Wet Process - Terminal Blend

Terminal blend CR modified AR asphalt binder is produced at a supplier's terminal as shown in the left image of Figure 2.14 and shipped to the mixture production plant similar to standard asphalt binder. CR used in this process is typically a smaller particle grind, sized to minus 30 mesh or smaller than 0.6 mm. The smaller rubber particles are used to help improve storage stability and minimize CR particle settlement. In some systems, rubber is completely digested in asphalt with no particulate matter present. The terminal blend CR binders used alone or with polymers can be formulated to produce Superpave performance graded (PG) binders, typically using 5 to 10 percent CR by weight of the total binder.

*Figure 2.14 -Terminal Blend CR (left); Mixture production plant with vertical binder storage tanks which allow for better agitation and storage (right).*

Smaller CR particles and polymers are used in a terminal blend to produce an AR binder that is similar to standard polymer modified asphalt binder. It is shipped to the mixture production plant, stored in in the plant's binder storage tanks and mixed with the aggregate, similar to standard asphalt mixture. It may be used in dense-graded mixes with no modification to the job mix formula.

Depending on the technology used, storage stability can be a problem with terminal blend AR binders. If CR is simply mixed with the binder it will settle with time; because rubber is heavier than asphalt binder. Settlement time will vary depending on the size of the CR particles and other additives or methods used to reduce separation. To avoid separation transport vehicles and storage tanks with agitation capability may be employed. Even with higher solubility AR binders, cleaning of tanks is recommended. Several patented methods have been developed to reduce separation and newer methods are in development. Continuous agitation in the storage tanks using stirring paddles and recirculation pumps will help reduce separation [23].

#### 2.3.2.4. A Hybrid Terminal Blend - Dry Crumb Rubber Modified Asphalt Mixture – CRHY

Benefits of CR modified asphalt pavements (CRMAPs) have been acknowledged by numerous researchers (Way, 1999; Carlson et al., 1999; Hicks et al., 1999). Most CRMAPs are made with the following three major methods: wet process (CRWet), dry process (CRDry), and terminal blend (CRTB) process. In CRWet process, crumb rubber (CR) is added to liquid asphalt at temperatures around 325-400 °F (163-205 ºC) and about 15% - 22% CR by weight of the binder is utilized (1-1.5% by total weight of the mix) (Caltrans, 2005; Caltrans, 2005). The terminal blend (CRTB) process is similar to the wet process, except that less amount of CR is used (~10-12%) and a polymeric additive is used to keep the CR particles suspended in the binder. The characteristics of CRWet and CRTB modified asphalt binders depend on the type of rubber, binder type, size of the CR particles, duration and temperature of the reaction and modification method (Carlson et al., 1999; King et al., 1999). The CR particles react with asphalt binder in two different ways during modification process: swelling and/or degradation (Abdelrahman et al., 1999). Depending on the reaction time and temperature, CR particles swell approximately two to three times of their original size (Jamrah et al., 2015). CRDry process is the method where the CR particles are added to the mix as a replacement of fine aggregates. In addition, there are several treated dry rubber technologies where the crumb rubber particles are pre-mixed with low viscosity petroleum-based products or aromatic oils compatible with the lighter fractions of asphalt binder. These treated rubber technologies are used as a CRDry process where they are added as a fine aggregate to the mixture [36]. By adding about 0.5% Dry CR (by weight of the mix), the amount of the rubber in the mixture can be doubled as compared to the conventional CRTB mix. The relative performances of CRTB and CRTB+dry process (herein called CRHY) [36].

CRHY is a new crumb rubber modified asphalt method, which is a combination of terminal blend (CRTB) and dry process (CRDry). The goal of such combination is to increase the CR content of CRTB modified asphalt mixtures by adding about 0.5% CR (by weight of the mix) via dry process. The CRHY method approximately doubles the rubber content in CRTB method by adding dry crumb rubber particles [36].

# CHAPTER 03: Rheology of Bitumen.

# 3. CHAPTER 03: Rheology of Bitumen

## 3.1. Introduction

Understanding bitumen rheology is a major concern since the mechanical properties of binders are closely linked to the service behaviour of actual flexible pavements.

Rheology involves the study and evaluation of the flow and permanent deformation of time-temperature dependent materials, such as bitumen, that are stressed (usually shear stress or extensional stress) through the application of force. The word rheology is believed to originate from the Greek words "ρεω", which can be translated as "the river, flowing, streaming", and "λογοο" meaning "word, science" and, therefore, literally means "the study of the flow" or "flow science" [24].

In the Rheology Bulletin, Morrison [25] translated the word rheology as, "Rheology is the study of the flow of materials that behave in an interesting or unusual manner. Oil and water flow in familiar, normal ways, whereas mayonnaise, peanut butter, chocolate, bread dough, and silly putty flow in complex and unusual ways. In rheology, we study the flow of unusual materials". The rheology of bitumen can be defined as the fundamental measurements associated with the flow and deformation characteristics of bitumen.

Therefore, understanding the flow and deformation (rheological properties) of bitumen in an asphalt mixture is crucial for pavement performance. Asphalt mixtures that deforms and flows too readily may be susceptible to rutting and bleeding, while those that are too stiff may be exposed to fatigue and cracking.

The study of bitumen rheology is not a new field and has been extensively studied all over the world. It is so well established that the most famous rheology tests on bitumen was started in 1927 at the University of Queensland in Brisbane, Australia (Figure 3.1). Professor Thomas Parnell initiated "the pitch drop" experiment, a test that consists of a bituminous-like pitch, slowly dripping out of a funnel at room temperature.

This test is considered as the longest rheology experiment and, up to the present time, only eight drops have fallen. The pitch has an estimated viscosity of approximately 230MPa.s [26]. Considerable efforts have been undertaken by bitumen and paving technologists over the last five decades to study the rheological properties of bitumen [27].

The rheological properties of bitumen are measured using conventional tests including softening point, viscosity (at 65oC and 135oC), elastic recovery (at 25oC by using a ductilometer), storage stability (penetration point, softening point), flash point and tests after thin film oven ageing (softening point, viscosity, elastic recovery). However, these measurements are insufficient to properly describe the rheology and failure properties that are needed to relate bitumen properties to asphalt mixture performance. The reliability of the tests is also often questionable. In addition, these tests do not quantify the time-dependent response of the binder and are not suitable to measure the rheological properties of modified binders.

Nowadays, the rheological properties of bitumen are usually determined using an oscillatory type testing apparatus known as a dynamic shear rheometer (DSR). This method was initially introduced during the Strategic Highway Research Program (SHRP) in 1993. The DSR is a very powerful tool used to determine the elastic, viscoelastic and viscous properties of bitumen over a wide range of temperatures and frequencies, often using the testing configuration shown in Figure 3.2.

*Figure 3.1 -Pitch drop experiment*

*Figure 3.2 -Dynamic shear rheometer (DSR)*

## 3.2. Rheology, deformation and flow

Rheology is concerned with the study of the internal response of real materials to stresses. Rheology can be considered to be the study of materials that exhibit both solid and liquid characteristics. [5]

A solid or a liquid retains its form; under load the material deforms, and when the load is removed this deformation can be either irrecoverable (viscous) or the deformation can be recoverable (elastic). In the simplest of terms, ideal solid materials behave in an elastic manner, and the deformation is recovered when the force or load is removed, whereas liquids behave in a viscous manner, and the load is not recovered but results in a permanent deformation of the material. Between these two ideal states, materials can exhibit what is referred to as visco-elastic behavior, in which the response to stress is partially viscous and partially elastic.

Rheology involves the study and evaluation of the time-temperature dependent response of materials that are stressed or subjected to an applied force. Rheological properties of bitumen consist of age hardening, temperature susceptibility, shear susceptibility, stiffness, penetration, ductility, and viscosity.[2]

### 3.2.1. Elasticity, plasticity and the behavior of solids

The ability of a deformed material body to return to its original shape and size when the forces causing the deformation are removed is referred to as elasticity, and materials exhibiting this behavior are referred to as elastic. Most solid materials exhibit elasticity, and the response to relatively small stresses results in a directly proportional strain: this relationship was observed by Robert Hooke in 1660, and is referred to as Hooke's law.

Hooke's law states that the applied force F = a constant "k" multiplied by the displacement or change in length: $F = kx$.

The value of k depends not only on the kind of elastic material under consideration but also on its dimensions and shape. Bitumen exhibits elastic behavior at low temperatures.

The limit of proportionality is defined as the point beyond which Hooke's law is no longer true when deforming a material, and the elastic limit is the point beyond which the material under load becomes permanently deformed so that the material does not return to its original length when the force is removed. It marks the onset of plastic behavior. For most brittle materials, stresses beyond the elastic limit result in fracture with almost no plastic deformation.

### 3.2.2. Viscosity and the behavior of liquids

Viscosity (η) is the measure of the resistance to flow of a liquid, and is defined as the ratio between the applied shear stress (τ) and the rate of shear strain (γ).

The viscosity of all simple liquids, including bitumen, decreases with increasing temperature.

#### 3.2.2.1. Newtonian behavior and non-Newtonian behavior.

Newtonian behavior is referred to where stresses arising are linearly proportional to the local strain rate, and the viscosity is independent of the rate of deformation

If η is constant, the fluid is known as Newtonian viscosity, and only varies as the temperature varies.

There are several 'non-Newtonian' effects, such as shear thinning which are termed 'pseudo-plastic' (viscosity decreases as the shear rate increases) also shear thickening termed 'dilatant' (the viscosity increases as a function of the shear rate). Most polymer modified bitumen can be considered to be shear-thinning liquids. Another effect is the yield stress (Bingham Fluid). Fluid with yield stress, flow only when the applied stress is higher than the threshold stress, and this is one of the main characteristics in road asphalt. Many other effects are related to the non-Newtonian flow behavior such as Weissenberg effect, die swell and fluid memory.

The following figure shows the shear stress dependence upon the shear rate for non-Newtonian and Newtonian fluids behavior.

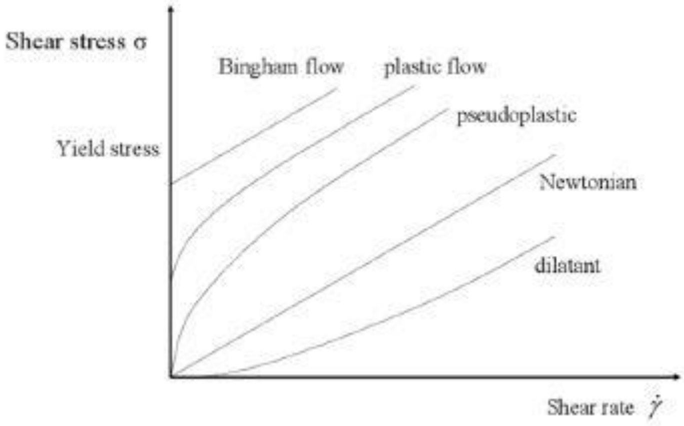


*Figure 3.3 -Different types of flow curves*

### 3.2.3. Visco-elasticity

The term visco-elasticity refers to the mechanical properties of a material that, in two limiting extremes, can result in the material behaving either as an elastic solid or a viscous fluid, depending on the temperature and the time of loading. Visco-elasticity differs from plasticity in that viscoelastic materials exhibit a time-related recovery when a load is removed: this is often referred to as a delayed elastic response. A plastic material does not return to its original form after the load is removed.

Under load, a material can exhibit a mixed visco-elastic and plastic response in which a proportion of deformation occurs; this is an important characteristic of bitumen and asphalts where accumulated non-recovered deformation manifests itself in the development of ruts in asphalt pavements. Reducing this phenomenon has resulted in the adoption of ways to reduce the permanent deformation and includes the modification of bitumen using polymers.

Bitumen can be considered a visco-elastic material where, typically, at low temperatures the elastic properties dominate, while at high temperatures the material behaves as a viscous fluid.

The degree to which a material displays visco-elastic behavior can be quantified; the Deborah number (De) as a measure of a material’s visco-elasticity.

$$De = \frac{\lambda}{t_D}$$

Where:

$\lambda$ = relaxation time

$t_0$ = observation time

If the Deborah number tend to be larger than one (De»1) the time that the stress is applied is much shorter than the relaxation time then the material will behave more elastic than viscous, in the case of viscoelastic behavior De ≈ 1 and for the limit of Newtonian liquid De →0..[2]

## 3.3. Rheological Properties of Bitumen Binders

The bitumen shows viscoelastic properties; these properties are usually measured where the relationship between stresses and strains is influenced only by the temperature and loading time and not by their magnitude (linear viscoelastic condition, LVE).

The LVE rheological properties of bitumen are normally presented in the form of both the complex modulus (|G*|) and phase angle (δ) master curves. |G*| is defined as the ratio of maximum (shear) stress to maximum strain when subjected to shear loading and δ is the phase difference between stress and strain in harmonic oscillation.

The study and understanding of these variables takes considerable importance in view of performance of binders. A binder which under the application of a load manifests high deformations may give rise to phenomena of rutting in the conglomerate. On the contrary, a very hard bitumen may give the pavement a greater sensitivity to fatigue phenomena. Today, the rheological properties of the binders are usually determined using equipment that operates both in oscillatory and continuous regime. The main important laboratory equipment to determine these rheological properties is the Dynamic Shear Rheometer (DSR). [2]

### 3.3.1. Dynamic Shear Rheometers (DSR): oscillatory tests

This type of test imposes sinusoidal stress, as an oscillatory shear force to a bitumen sample sandwiched between two parallel plates. The typical arrangement of DSR is of a fixed lower plate and an oscillating upper plate through which the shear force is applied to the specimen as shown in figure 3.4.

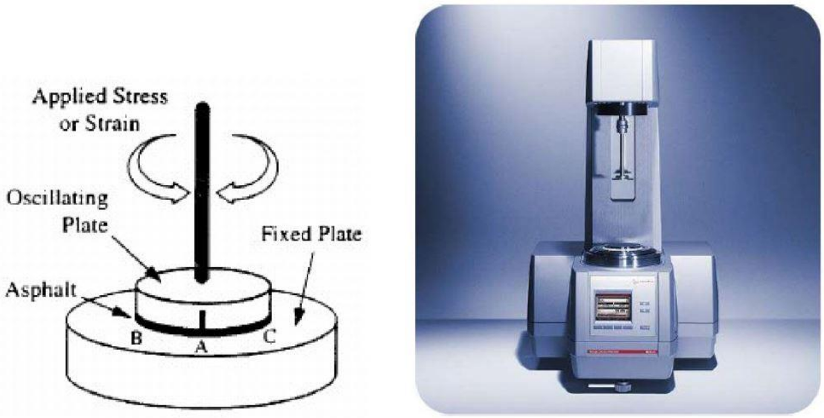


*Figure 3.4 -Dynamic Shear Rheometer*

The amplitude of the responding stress is measured by determining the torque transmitted through the sample in response to the applied strain. (Fig. 3.5)

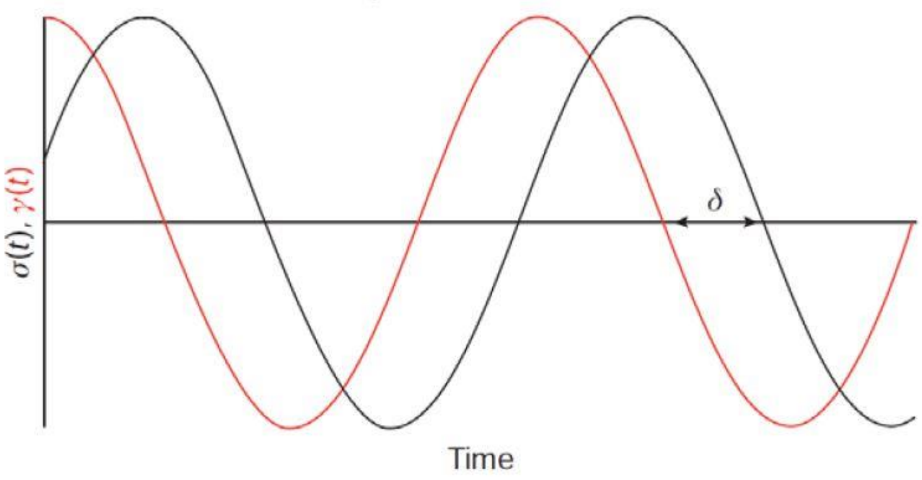


*Figure 3.5 -Stress-Strain response during an oscillatory testing*

The loading frequency (ω) is known as the angular frequency, rotational frequency or radian frequency and is defined as:

$\omega=2\pi f$

Where:

ω= angular frequency (rad/s)

f = frequency (Hz).

The response of the applied strain is the development of a stress that, for linear visco-elasticity is sinusoidal, and the response of the material is out of phase with the applied strain, and is referred to as the phase angle δ. This is defined as the phase difference between the stress and the strain, and is also called the loss angle or the phase lag. The phase angle represents the time lag (Δt) between the applied shear stress and the resulting shear strain converted into degrees:

$$\delta = \frac{\Delta t}{t} * 360$$

It is shown graphically in figure 3.6, a purely elastic material would not show any phase difference between the stress and strain where as a pure viscous material shows a phase angle of 90°.

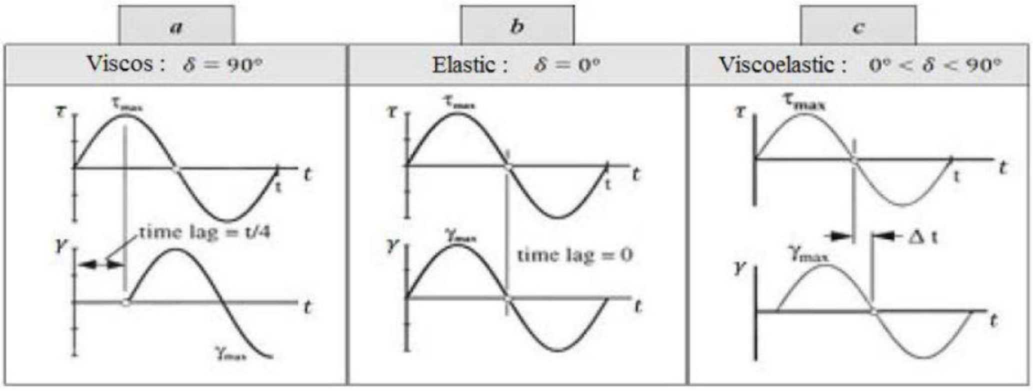


*Figure 3.6 -phase angle determination*

The ratio of the resulting stress to the applied strain is called the complex shear modulus, G*, and is also referred to as the complex modulus, shear modulus or, simply, the stiffness.

G* is an indicator of the stiffness or resistance to deformation under load of bitumen and is defined by the following equation:

$$G^{*} = \frac{\tau_{max}}{\gamma_{max}}$$

Where:

$\tau_{max}$= shear stress

$\gamma_{max}$ = shear strain

The norm of the complex modulus is analogous to the magnitude of a vector (Figure 3.7), as the value is calculated from the square root of the sum of the squares of the components.

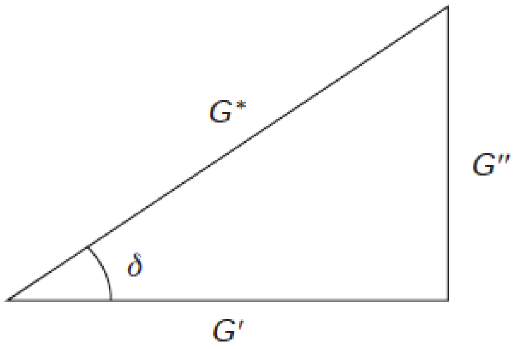


*Figure 3.7 -Relationship between Shear Modulus and phase angle.*

The in-phase component of G* is called the shear storage modulus (G'), or more commonly the storage modulus. The storage modulus equals the stress that is in phase with the strain divided by the strain, or:

$G' = cos\delta.G^*$

The shear loss modulus G", or simply the loss modulus, is the out-of-phase component of G*. The loss modulus equals the stress 90° out of phase with the strain divided by the strain, or:

$G" = sin\delta.G^*$

Materials with higher storage moduli have greater ability to recover from deformation, and materials with higher loss moduli have greater ability to resist deformation at any prescribed frequency.

### 3.3.2. Representation of rheological data

One possible representation of the data from the tests in oscillatory regime sinusoidal, alternative to the representation of viscoelastic functions in the frequency domain, is formed from the Black Diagram. (Fig. 3.8)

A Black diagram is a graph of the magnitude (norm) of the complex modulus ([G*]) versus the phase angle (δ) obtained from a dynamic test. The frequency and the temperature are therefore eliminated from the plot, which allows all the dynamic data to be presented in one plot without the need to perform time–temperature superposition manipulations of the raw data. A smooth curve in a Black diagram is a useful indicator of time–temperature equivalency, while a disjointed curve indicates the breakdown of the time–temperature superposition, and potentially the presence of a high asphaltene structured bitumen, high wax content bitumen or a highly polymer modified bitumen.

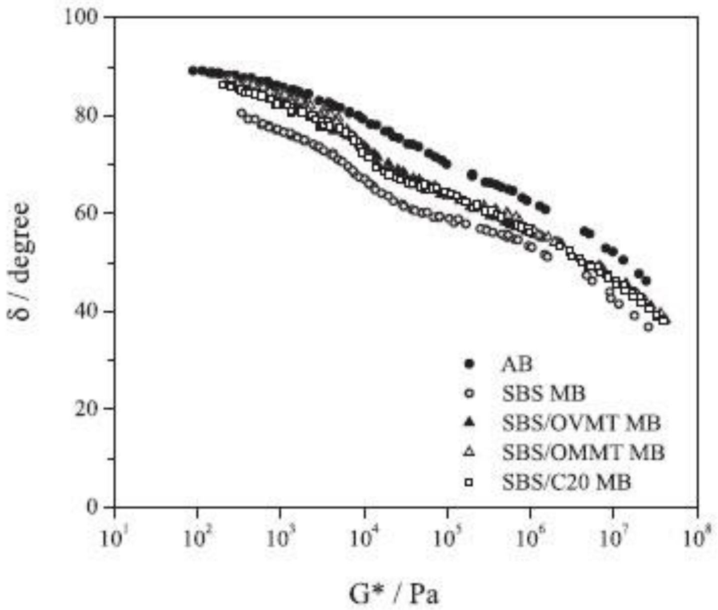


*Figure 3.8 -Black Diagrams Representations*

Another possible representation is a Cole-Cole Diagram (Fig. 3.9) which is a graph of the loss (viscous) modulus (G”) as a function of the storage (elastic) modulus (G’). The plot provides a means of representing the visco-elastic balance of the bitumen without incorporating frequency and/or temperature as one of the axes.

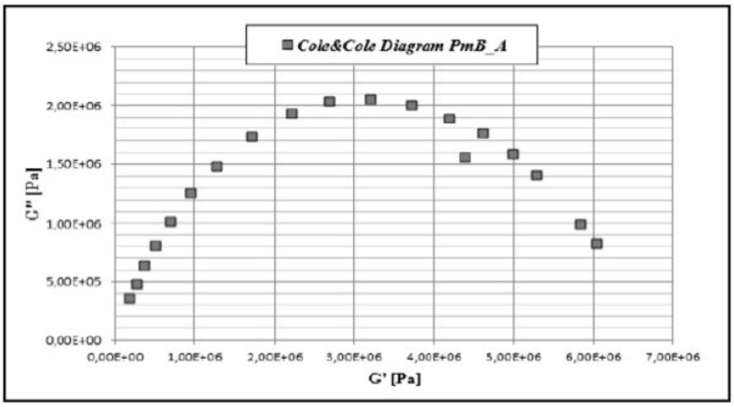


*Figure 3.9 -Cole-Cole Diagram Representation.*

### 3.3.2.1. Principle of Time-Temperature Superposition (TTS)

Under a regime of static stress as in a dynamic regime of stress, there is experimental evidence that the effects of the loading time and the temperature are superimposable. This means that increases the time of loading (or reductions of the oscillation frequency) produce effects on the response of the viscoelastic material completely similar, and therefore superimposable, to those of an increase in test temperature; also applies vice versa.

It follows that a viscoelastic function is measured for a certain charging time $t_1$ and for a certain temperature $T_1$ is identically obtainable for a different pair of values of the time $t_2$ and the temperature $T_2$ connected to the preceding by precise relations.
Viscosity–temperature equations are used to characterize the temperature dependency of bitumens, and therefore to determine the shift factors needed for the time–temperature superposition principle.

Based on the previous the principle of Time-Temperature Superposition (TTS) provides that between the temperature changes and the time (or frequency) there is a relationship, and therefore the effects of time and temperature found to overlap and it is interchangeable.
Several studies have shown that the modules G' and G" recorded at different temperatures can be gathered together into a single curve, called master curve, by means of the principle of Time-Temperature Superposition (TTS).

On the basis of what has been mentioned it can be said that the variation of a mechanical quantity, such as the complex modulus G* obtained by varying the temperature at fixed frequency, can be obtained by varying the frequency and keeping constant the temperature. Immediate consequence is the possible display on a single curve of the viscoelastic behavior of the test material in a frequency range much wider than that available experimental apparatus to set temperature.

In particular, it is assumed that a temperature variation corresponds to a variation, equal to a same factor called "shift factor", of all the relaxation times $\lambda_i$, namely that all the relaxation times of the same system have the same dependence by the temperature. Therefore, if $\tau_1$ ($T_0$), $\tau_2$ ($T_0$), $\tau_3$ ($T_0$) ... are the relaxation times of the system at the reference temperature $T_0$, then the effect of varying the temperature to a different value T will be changed to these times: $\tau_1$ ($a_T$), τ2 ($a_T$), τ3 ($a_T$) ... where $a_T$ is the shift factor depending on temperature and is 1 for $T = T_0$.

Therefore:

$$\tau_1(T) = a_T * \tau_i(T_0)$$

The Williams, Landel and Ferry (WLF) equation (Williams et al., 1955) has been widely used to describe the relationship between the shift factors and temperature, and thereby determine the shift factors of bitumens. The equation is theoretical, based on the free volume theory (Ferry, 1971), and makes use of temperature differences, which makes it suitable for practical manipulations. The equation has also been found to be applicable to bitumen results.

The WLF equation is:

$$\log(a_T) = \log\frac{\eta_0(T_0)}{\eta_0(T)} = \frac{C_1(T-T_0)}{C_2+T-T_0}$$

Where a $_{(T)}$ is the shift factor at a temperature $T_0$, $\eta_0(T_0)$ is the Newtonian viscosity at a temperature $T_0$, $\eta_0(T)$ is the Newtonian viscosity at the reference temperature T, and $C_1$ and $C_2$ are empirically determined coefficients; which depend on the material or fitting. The WLF equation requires three constants to be determined, namely $C_1$, $C_2$ and T. The temperature dependency of bitumens can be described by one parameter, T, if universal constants are used for $C_1$ and $C_2$ in the WLF equation.

An alternative equation that can be used to describe the relationship between the shift factors and temperature is the Arrhenius equation:

$$\log\,(a_T) = \frac{E_f}{R}\left(\frac{1}{T} - \frac{1}{T_0}\right)$$

Where $a_{(T)}$ is the horizontal shift factor, $E_f$ is the activation energy, R is the universal gas constant, $T_0$ is the temperature and T is the reference temperature.

P.E.Rouse proposed a theory that can explain the origins and deduce the implications of time-temperature superposition principle. It examines the motion of a polymer chain represented as a series of masses connected by springs (Fig. 3.10). The masses undergo, then, the effect of viscous forces. The problem is studied researching the normal modes of oscillation. The results include the time-temperature superposition principle.

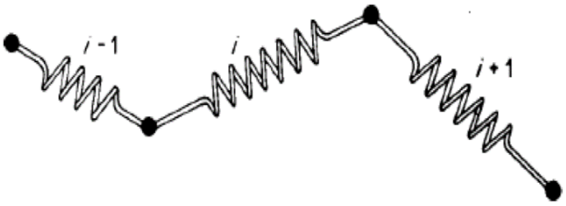


*Figure 3.10 -Rouse Model*

### 3.3.2.2. Master Curves

The development of the master curves are based on the Time Temperature Superposition Principle (TTS) explained above. The master curves can be used to represent the behavior of the material in a wide range of time or frequencies where the trend of load is determined by the only experimental curve that encloses both effects of time and temperature.

These master curves can be constructed either in the time domain or in the temperature domain. In their simplest form, master curves are produced by manually shifting modulus versus frequency plots at different temperatures along the logarithmic frequency axis to produce a smooth master curve.

The shape of the master curve of the complex modulus as a function of the reduced frequency, on a log–log scale, resembles the shape of a hyperbola. (Fig. 3.11)

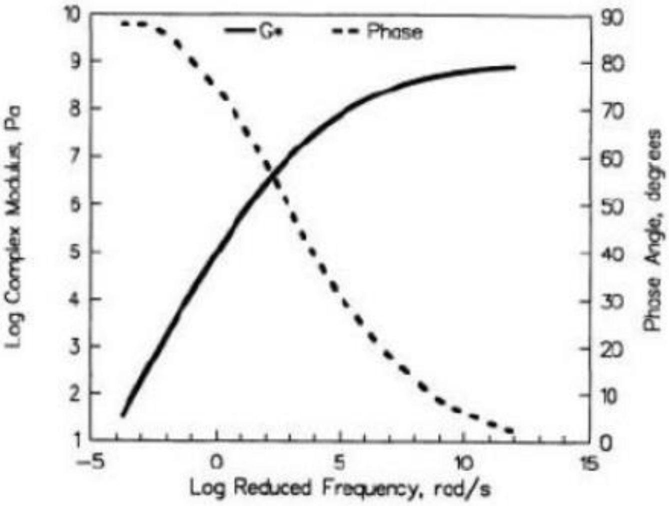


*Figure 3.11 -Master Curve*

The curve has a horizontal asymptote (glassy modulus) at high frequencies and an asymptote at an angle of 45° (viscous part) at low frequencies, with a transition range in between. (Fig. 3.12)

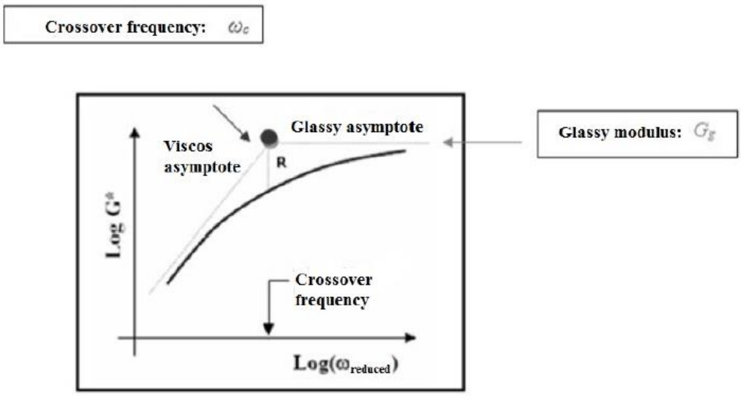


*Figure 3.12 -Parameters Master Curve*

Some general consideration and parameters of the master curve are:

- ***Glassy Modulus***: defines the limit behavior of the material at low temperatures and at high frequencies. The value of G* is said in correspondence with the glassy asymptote called $G_g$ "Glassy Modulus": this is the value asymptote horizontal at which the complex modulus tends to low temperatures and high frequency load.

Gg is a constant value for each type of bitumen and the phase angle δ =0° when the bitumen tends to the glassy regime.

- ***Viscous part***: defines the limit behavior of the material at high temperatures and at low frequencies. In correspondence with the viscos asymptote, it is defined the viscosity of the steady state $\eta_{ss}$ that for small amplitudes can be put equal to $\eta_{0}$.

$$\eta_{ss} = \lim_{w \to 0} \frac{G^*}{\omega}$$

- ***Crossover frequency ωc [rad/sec]:*** corresponds to the intersection between the glassy asymptote and the viscos asymptote. Its inverse is called "crossover time". This value indicates the transition from zone to zone viscoelastic viscose, for which is an indicator of the consistency and hardness of the material.

- ***Rheological index R:*** This parameter indicates the "velocity" with which the curve reaches the glassy asymptote, and it is configured as a parameter of fundamental importance, as it is directly proportional to the amplitude of the spectrum of relaxation, which gives it the property of being a good indicator of the type of rheological material. In practice, this parameter characterizes the performance of the rheological curves. It is independent from the reference temperature but not by the type of bitumen.

- ***Linear Visco Elastic (LVE) limit***: It is in the field of linear viscoelasticity (LVE) if the stress-strain relationship, τ / γ, is constant at every point. The model determines the linear limit on the curve of frequency-elastic modulus defining the limit at the point at which the modulus drops to 95% of the maximum value obtained. It is also suggested to perform the tests with a tension not greater than 75% that of the linear limit.

- ***Viscosity stationary area***: the area is configured as Newtonian viscosity reached at high temperatures and low frequency loading. It is also called asymptote viscous and is represented by a straight line with slope equal to 1. The phase angle is very close to δ = 90°.

As a summary the behavior of bitumen can be addressed in three regions:

- A low temperature linear elastic region
- A high temperature viscous region
- An intermediate temperature visco-elastic region.

Linear behavior is fulfilled at low temperatures and short loading times (high frequencies), where the bitumen behaves as an elastic solid. The linearity is also maintained (for unmodified bitumen) at high temperatures and long loading times (low frequencies), where the material behaves almost entirely as a Newtonian fluid. The area where non-linearity is prominent is therefore in the range of moderate temperatures and moderate loading times. These temperatures and loading are determined from the condition experienced in the field.

- ***Intermediate Temperature visco-elastic region.***

It is noticed that pavement in service has properties that are in the visco-elastic region. At these temperatures the bitumen shows elastic and viscous properties, and a time-dependent relationship between the applied stress or strain and the resultant strain or stress as well.

The two most common means of determining the visco-elastic properties of bitumen are by creep and recovery measurements and dynamic oscillatory measurements (alternating stress and strain of constant amplitude and frequency).

- ***Creep and recovery tests:***

Steady state creep measurements can be used to calculate an apparent viscosity at intermediate pavement service temperatures.

To conduct such measurements, it is necessary to apply a shear stress to the bitumen until the strain rate becomes constant. At low temperatures, longer times are required for delayed elasticity to be expended and steady state flow to occur.

The time–temperature superposition principle can be applied to creep test data to produce a master curve from the creep data. [2]

## 3.4. LINEAR VISCOELASTIC ANALYSIS

### 3.4.1. Amplitude Sweep test

Amplitude Sweep test are oscillatory test performed at variable amplitudes, keeping the frequency and also the measuring temperature at a constant value. (Mezger, 2006)

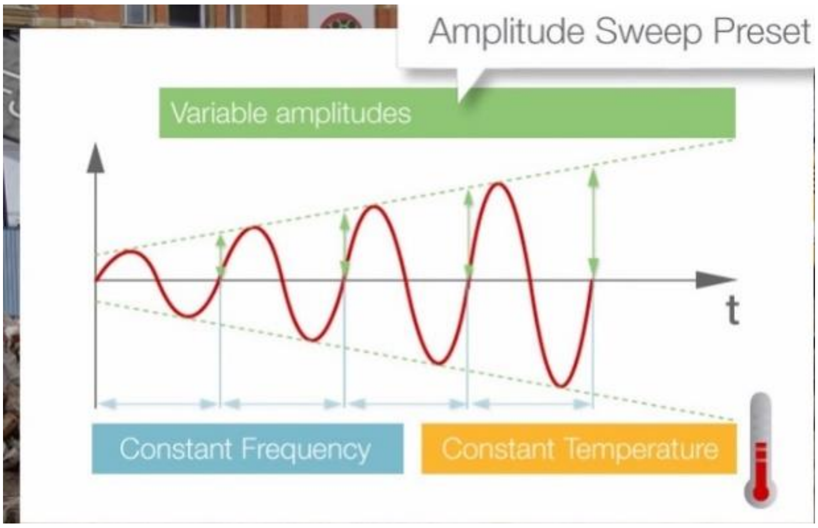


*Figure 3.13 -Preset of a shear strain amplitude sweep*

The test uses cyclic loading with systematically increasing load amplitudes to accelerate the damages and provide sufficient data in less than thirty minutes. (Hintz, 2011).

For test with controlled shear strain:

$$\gamma(t) = \gamma_A * sin\omega(t)$$

With ω=constant and a variable strain amplitude $\gamma_A = \gamma_A(t)$.

For test with controlled shear stress:

$$\tau(t) = \tau_A(t)$$

With ω=constant and a variable strain amplitude $\tau_A = \tau_A(t)$.

It is often selected the angular frequency ω= 10 rad/s. Since ω=2πf, this value correspond to a frequency of 1.59Hz.

Usually for strain sweeps, log (γ) is presented on the x-axis (and log (τ) for stress sweep)' and log G' and log G" are shown on the y-axis both in the same scale.

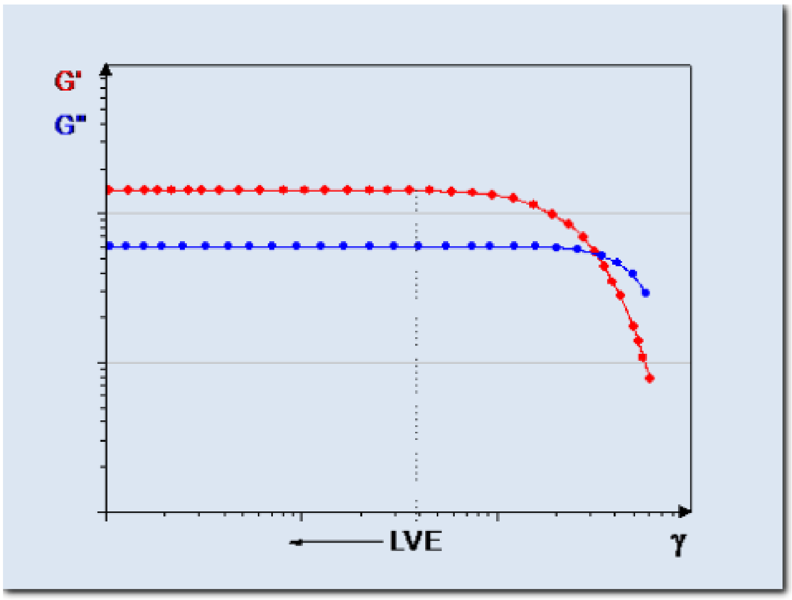


*Figure 3.14 - Output amplitude sweep test.*

The amplitude sweet test is perform with the objective to determine the linear viscoelastic range by keeping the frequency constant(10 rad/s) and the amplitude of the shear strain varied (starting from 0.01% to 100%).

The length of the linear viscoelastic range is a measure of stability. For example in Fig. 4.3, as long as the γ-amplitude are still below the limiting value $\gamma_L$, the G' and G" curves are remaining constant (the structure of the sample is stable under those conditions) (Mezger, 2006). After the $\gamma_L$ is exceeded the LVE range is also exceeded and finally the sample is broken.

There are several ways to determine the LVE ranges most commonly the G' curve is the one taken for the analysis because it is the first one to show the tendency to leave the LVE range. LVE range could be define either by a visual or manual analysis or automatic analysis with the help of the software analysis programs.

The limiting values of $\gamma_L$ may be considered as a value of the permissible maximum strain, with every unknown sample an amplitude sweep test should be done as the first oscillatory test at all.

Another way to determine LVE range is when the software from apparatus (DSR) generates a plot (fig. 16) where the complex modulus is placed on the x-axis while the strain γ is on the y-axis

The limiting values of $\gamma_L$ in this case is given by the same frequency, for which the LVE range is defined as the range in strange where the complex modulus takes a value equal to 90% of the initial one. (fig.4.4)

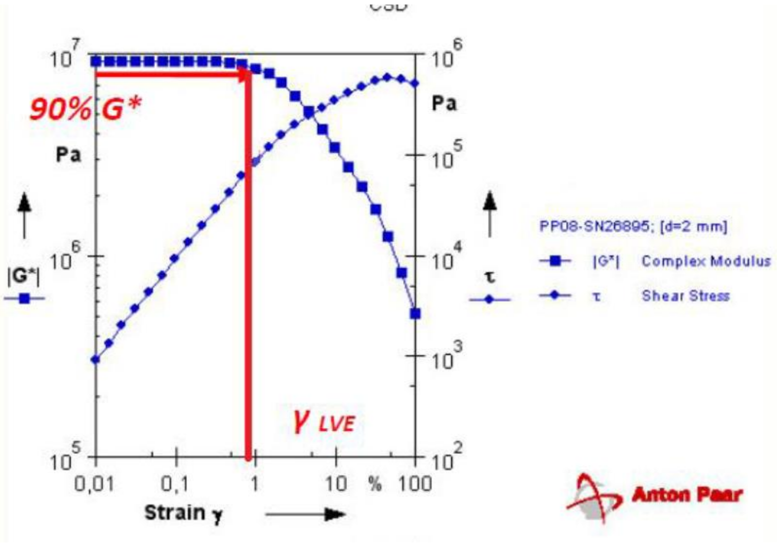


*Figure 3.15 -Output Amplitude Sweep test DSR software.*

***Amplitude Sweep test on Dynamic Shear Rheometer (DSR).***

In accordance with the AASHTO T315-10 “Determining the Rheological Properties of Asphalt Binder using a Dynamic Shear Rheometer (DSR)” the test sample can be 1mm thick by 25 mm or 2 mm by a diameter of 8 mm. The strain γ ranges from 0.01% - 100% and ω= 10 rad/s

During the test, one of the parallel plates from the DSR is oscillated with respect to the other at pre-selected frequencies and rotational deformation amplitudes (strain controlled) or torque amplitudes (stress controlled). (American Association of State Highway and Transportation Officials AASHTO, 2010)

After the LVR is determined a frequency sweep test is performed in order to determine the nature of the material.

### 3.4.2. Frequency Sweep Test

During the frequency sweep the frequency is varied while the amplitude of the deformation - or alternatively the amplitude of the shear stress - is kept constant.

The amplitude is set within the Linear Viscoelastic (LVE) range, for this reason it is advisable to perform an amplitude sweep test to stablish the LVE.

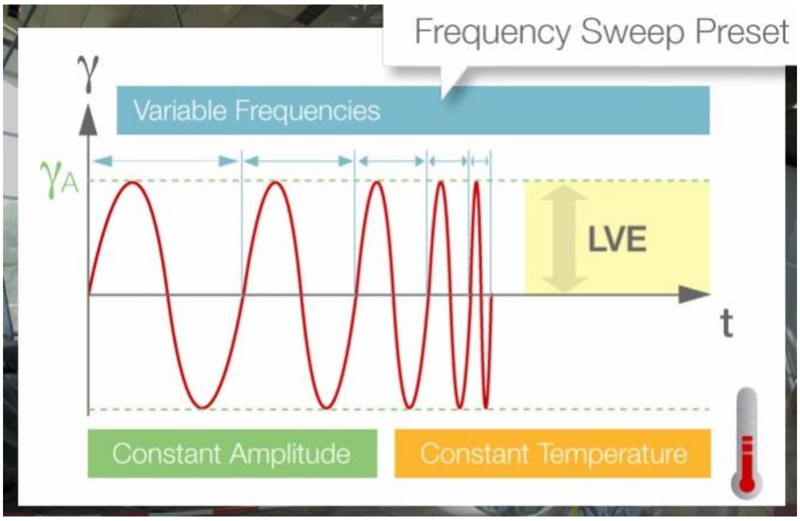


*Figure 3.16 -Preset of a frequency sweep controlled strain*

The frequency test is used to investigate time-dependent shear behavior since the frequency is the inverse value of time (*1/f*). Short term behavior is simulated by rapid motion (i.e., at high frequencies) and long term by slow motion (i.e., at low frequencies). (Mezger, 2006)

For test with controlled shear strain:

$$\gamma(t) = \gamma_A * sin\omega(t)$$

With γ =constant and a variable strain amplitude $\omega = \omega(t)$.

For test with controlled shear stress:

$$\tau(t) = \tau_A(t)$$

With τ=constant and a variable strain amplitude $\omega = \omega(t)$.

The information of the maximum permissible deformation (the limiting value of the $\gamma_L$) is given by the amplitude sweep test discussed in the previous pages.

The final output is usually the frequency is shown on the x-axis while the complex modulus G* is shown on the y-axis on the same scale. (fig.4.6) additional parameter is display like the phase angle δ on the y-axis

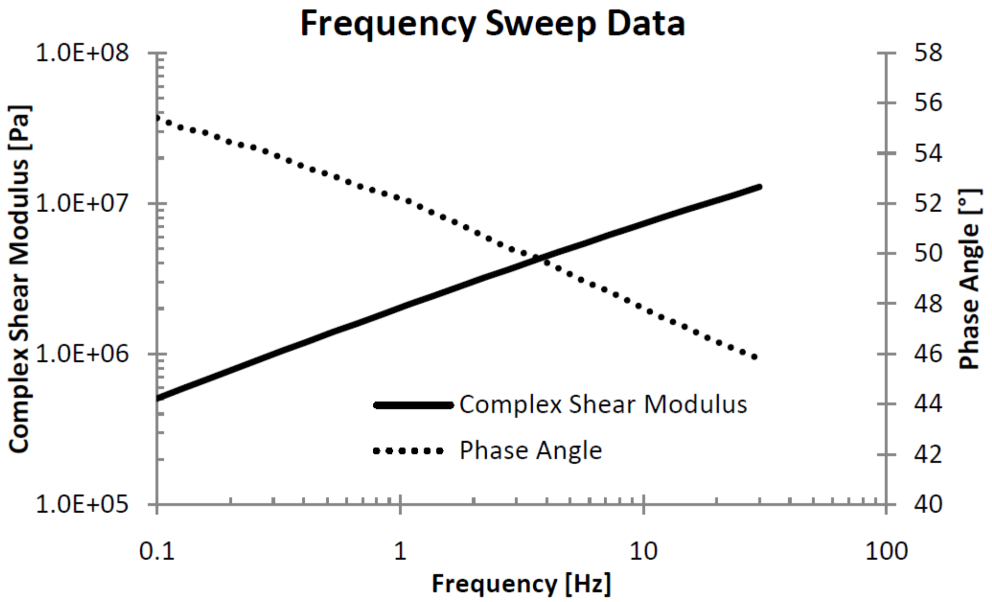


*Figure 3.17 -Example output of the frequency sweep test*

The frequency sweep is very important for polymer melts: By measuring frequency sweeps at different temperatures the characteristics like melting point, glass transition, rubbery-elastic characteristics, and entanglement density. The measurement results can be superposed to significant master curves.

### *Dynamic Shear Rheometer (DSR) test setting*

The standard for this test is AASHTO T315-10 “Determining the Rheological Properties of Asphalt Binder using a Dynamic Shear Rheometer (DSR)” stated that the standard is suitable when the dynamic shear modules range between 100 Pa and 10 MPa. This range in modulus is typically obtained between 6 and 88°C at angular frequency of 10 rad/s depend on the grade, test temperature, and conditions of the asphalt binder.

The test sample can be 1mm thick by 25 mm or 2 mm by a diameter of 8 mm and the setting of the parallel plates can be either stress controlled or strain controlled.

Oscillatory loading frequencies are set between 100 to 0.1 rad/s using a sinusoidal waveform and the maximum permissible deformation γ-value of the LVE range after a previously performed amplitude sweep test.

According to the specification AASHTO T 315-10 testing is performed at a frequency of 10 rad/s.

The test evaluates the rheological parameters using the DSR computer software:

- The complex modules G*
- The phase angle δ.
- Storage modulus or module of elastic restitution G';
- Loss modulus or module of viscous dissipation G";

### Temperature Sweep Test

In a temperature sweep test, the frequency and the oscillation amplitude were kept constant, while the temperature was increased in some progression.

The test is aimed to give the melting point for temperature dependent material.

The temperature sweep test approximates a temperature at which will be satisfied the rutting factor and provide a better understanding about the dependence of the complex modulus G* and the phase angle δ with or without polymer and rubber.

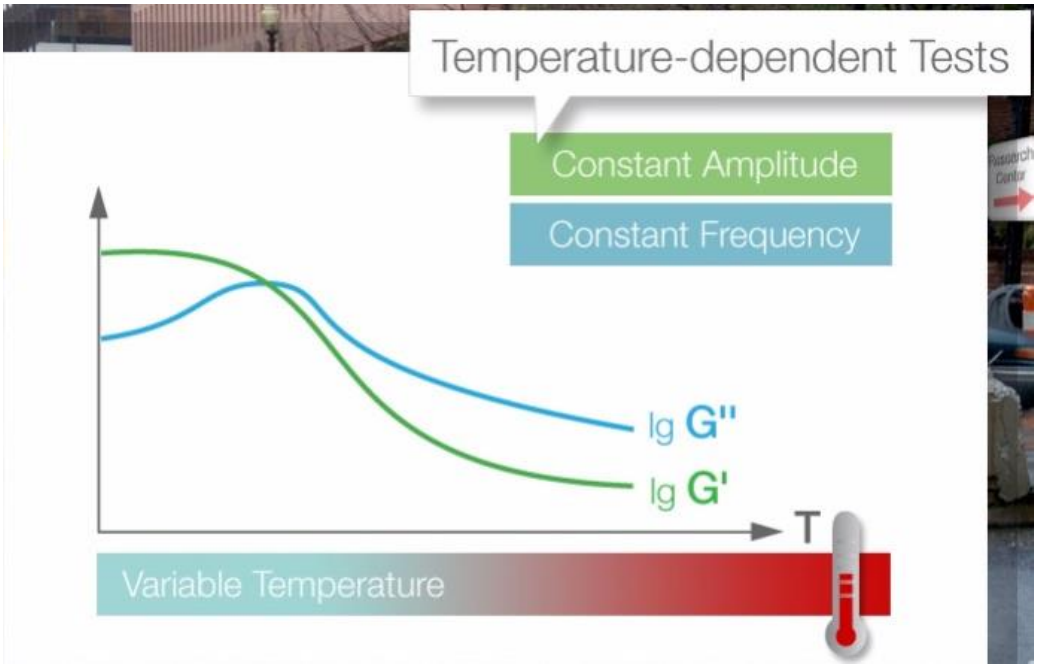


*Figure 3.18 -Preset Temperature Sweep Test*

# CHAPTER 04: Rutting and Fatigue Cracking

# 4. CHAPTER 4: Rutting and Fatigue Cracking

## 4.1. Introduction

The American Association of State Highway and Transportation Officials (AASHTO) published its first asphalt binder specification based on penetration in 1931. The penetration test is commonly performed at 25 °C with a 100 g weight needle allowed to penetrate for 5 s and measured at 0.1 mm increments. Capillary viscosity is used to define viscosity of asphalt cements at 60 and 135 °C. These traditional binder properties are then used as a substitute indicator for predicting the pavement performance.

However, traffic flow has increased tremendously. Overloading vehicles with 12,000 kg single axle loads are rampant, with 8,000 kg being the maximum tolerance. The high tire pressure pumped by truck drivers to carry more goods is close to 1.4 MPa (200 psi). Climatic effects such as temperatures and precipitation directly contribute to the deterioration of pavement performance. These demanding changes have troubled highway engineers all over the world. As a result, pavements are subjected to premature rutting and fatigue cracking. [28]

Fatigue and rutting are the two major failure distresses in flexible pavement that significantly affect the serviceability of pavement. The properties of bitumen have a direct effect on controlling the fatigue and rutting distresses. Because of the increase in vehicular loading and repetitions, the modification of neat bitumens becomes a widespread practice to improve their mechanical properties. Any improvements obtained from developing modified binders need to be reflected by fundamental testing parameters. The empirical testing methods and Superpave grading procedure that were developed mainly for unmodified bitumens have failed in many cases to predict the performance of modified bitumens. Evaluation the influence of such modifiers needs be based on characterizing accurately the inherent resistance of binders to fatigue and rutting damage. A detailed description of the main elements associated with the DSR are presented in this Research [4.4].

## 4.2. Rutting.

### 4.2.1. Description

Surface depression in the wheelpath. Pavement uplift (shearing) may occur along the sides of the rut. Ruts are particularly evident after a rain when they are filled with water. There are two basic types of rutting: mix rutting and subgrade rutting. Mix rutting occurs when the subgrade does not rut yet the pavement surface exhibits wheelpath depressions as a result of compaction/mix design problems. Subgrade rutting occurs when the subgrade exhibits wheelpath depressions due to loading. In this case, the pavement settles into the subgrade ruts causing surface depressions in the wheelpath.

*Figure 4.1 -Severe mix rutting.*

*Figure 4.2 -Mix rutting.*

*Figure 4.3 -Outside wheelpath rutting.*

### 4.2.2. Problem

Ruts filled with water can cause vehicle hydroplaning, can be hazardous because ruts tend to pull a vehicle towards the rut path as it is steered across the rut.

### 4.2.3. Possible Causes

Permanent deformation in any of a pavement’s layers or subgrade usually caused by consolidation or lateral movement of the materials due to traffic loading. Specific causes of rutting can be:

- Insufficient compaction of HMA layers during construction. If it is not compacted enough initially, HMA pavement may continue to densify under traffic loads.
- Subgrade rutting (e.g., as a result of inadequate pavement structure)
- Improper mix design or manufacture (e.g., excessively high asphalt content, excessive mineral filler, insufficient amount of angular aggregate particles).

Ruts caused by studded tire wear present the same problem as the ruts described here, but they are actually a result of mechanical dislodging due to wear and not pavement deformation.

### 4.2.4. Repair

A heavily rutted pavement should be investigated to determine the root cause of failure (e.g. insufficient compaction, subgrade rutting, poor mix design or studded tire wear). Slight ruts (< 1/3 inch deep) can generally be left untreated. Pavement with deeper ruts should be leveled and overlayed [5.5]

### 4.2.5. Performance Tests for Rutting

For asphalt pavements, rutting is one of the most serious forms of pavement distress, and severe rutting is a significant cause of pavement failure. To prevent rutting, the pavement structure needs to provide sufficient support for the roadway surface, while the design and construction of the asphalt mix must create a pavement that can resist deformation. Here we'll look at some of the laboratory test devices used to evaluate asphalt mix designs and predict rut resistance.

Rutting refers to permanent deformation of the asphalt surface that accumulates in the wheelpaths. It is primarily the result of repeated traffic loading cycles. Rutting may be accompanied by fatigue cracking and other distresses, making it a serious concern and potential indicator of pavement failure. The stability of the asphalt mix is an important element in its ability to resist rutting and thus a key factor to evaluate. At the same time, however, the performance of a particular mix design depends on environmental conditions including traffic, temperature, and humidity. Since these factors are often seasonal and mix designs can be exposed to a variety of environments across different projects, this makes it a challenge to reliably predict the rut resistance of a given mix.

This is one reason the Superpave mix design method does not require a specific performance test for rut resistance. Earlier approaches like the Hveem method and the Marshall method do incorporate simple mechanical tests, such as the Hveem stabilometer or the Marshall stability and flow test, in which a load is applied and the resistance of the mix is measured, along with its displacement or deformation. Many agencies still use these established older methods to evaluate rut resistance during the mix design process. However, this is often done on a pass-fail basis without attempting to predict performance, or attempting to factor in changes in loads or environmental conditions.

Newer sophisticated test methods use accelerated loading to test mix performance by simulating the cumulative effect of traffic loading within a short period of time. They may also be able to test at different load levels or specific temperature and humidity conditions, thus providing a more realistic evaluation. Some of the most common devices used include:

- The Asphalt Mixture Performance Tester (AMPT), formerly known as the Simple Performance Tester (SPT)
- The Hamburg Wheel Tracking Device (HWTD)
- The Asphalt Pavement Analyzer (APA), formerly known as the Georgia Loaded-Wheel Tester

#### 4.2.5.1. Asphalt Mixture Performance Tester

The Asphalt Mixture Performance Tester (AMPT) assesses the rut resistance of an sphalt mix based on a flow number. The test is conducted by subjecting a prepared specimen to repeated compressive loads at a specific test temperature. The flow number is determined by the point at which the specimen exhibits tertiary flow, which is shear deformation at constant volume (similar to the deformation that produces rutting in the field).

*Figure 4.4 -Asphalt Mixture Performance Tester.*

In addition to the flow number, the AMPT can also be used to help determine the dynamic modulus of the asphalt. This property can be useful to know if you are implementing mechanistic-empirical pavement design, since it is one of the inputs for that procedure.

#### 4.2.5.2. Hamburg Wheel Tracking Device

The Hamburg Wheel Tracking Device (HWTD) tests rut resistance by tracking a loaded steel wheel back and forth across an asphalt pavement sample. By repeating this for thousands of cycles, it simulates the effect of traffic loading on the pavement over time. The device measures the rut depth in the sample continuously during the test.


photo courtesy of FHWA

*Figure 4.5 -Hamburg Wheel Tracking Device.*

HWTD testing is commonly performed on a sample while it is submerged in water. This allows it to be used to evaluate resistance to moisture damage as well. After the sample is consolidated by the initial load cycles, the rate at which rutting develops will be determined by the stability of the mix at first. Then, once the load cycles reach a certain point (which varies depending on the moisture susceptibility of the mix), rut development will accelerate as damage from stripping kicks in.

#### 4.2.5.3. Asphalt Pavement Analyzer

The Asphalt Pavement Analyzer (APA) operates in a manner similar to the Hamburg Wheel Tracking Device but uses different equipment. The APA uses an aluminum wheel that is loaded onto a pressurized linear hose and tracked back and forth over the asphalt pavement sample. The amount of rutting is measured after the wheel has been tracked for a set number of cycles at a constant load and hose pressure.

*Figure 4.6 -Asphalt Pavement Analyzer.*

When evaluating an asphalt mix design, ideally, we want to know not just its physical characteristics, but how it will perform as well. Resistance to rutting is a critical part of performance in the field, and testing for it is an important consideration. Increased experience with these devices, and ongoing research to connect their test results with empirical pavement performance, will make it easier to adapt testing to local conditions so that the results have more predictive value. With asphalt pavements, the solution isn’t to simply get out of the rut, it involves creating mixes to prevent ruts from happening in the first place [6.6].

### 4.2.6. Creep Test

Rutting can be caused by plastic movement of the asphalt mix either in hot weather or from inadequate compaction during construction. Significant rutting can lead to major structural failures and a potential for hydroplaning and also will not only affects the drivers comfort but also threats the traffic safety.

There are two basic rutting mechanisms. The first one to limit the vertical compressive strain on top of the subgrade, the other to limit the total accumulated permanent deformation on the pavement surface based on the permanent deformation properties of each individual layer. (Huang, 2004)

In the Asphalt Institute and Shell design methods, the allowable number of load repetitions $N_d$ to limit rutting is related to the vertical compressive strain $\varepsilon_c$ on top of the subgrade by:

$$N_d = f_4\left({}^{1}/_{\varepsilon_c}\right)^{f_5}$$

Where:

$N_d$ = *number of load repetitions.*

$f_4, f_5$ = *coefficients of subgrade strain criteria.*

$\varepsilon_c$ = compressive strain

Most of rutting problems result from plastic deformation of the surface course. It is characterized by shear deformation inside the asphalt mixture.

When asphalt mixtures have low shear strength, rutting accumulates after each vehicle loading during the first months or few years of the pavement service life until it becomes noticeable and hazardous. Rutting failure leads to poor serviceability of the pavements, making vehicles ride rough and unsafe. This phenomenon is more critical during the hot season of the earlier period of pavement life because the asphalt binder is less aged and softer.

The first rutting parameter introduced for the dynamic shear rheometer (DSR) was $G^*/\sin\delta$; rutting occurs as a consequence of a larger value of this parameter.

Several researchers have stated that exists a poor relationship between this parameter and rutting, since it is inadequate to demonstrate the rutting performance for certain binders specially modified binder.

In 2001 Bahia et al suggest that it is better to use repeated creep loading in order to simulate the effect of traffic flow.

#### 4.2.6.1. Repeated Creep Test (RCR)

The repeated creep test is proposed as a method of separating the dissipated energy and estimating the resistance to accumulation of permanent strain for asphalt binders.

The Repeated creep test is performed through the dynamic shear rheometer (DSR) to evaluate the resistance of asphalt binders to permanent deformation, by applying shear stress in the range of 30Pa to 300Pa for 100 cycles where each cycle is compose by 1 second of loading time and 9 seconds for the unloading, a parameter “$G_v$” which is the viscous component of the creep stiffness which is consider an improvement characterization of the resistance to rutting for asphalt binder,

The main advantages of RCR can be summarized as follows: (Delgadillo R., 2006)

- Repeated creep loading represents the actual loading on the pavement better than fully reversed load from dynamic testing

- RCR allows an easy identification of the permanent deformation of binders when loaded. This is especially important in modified binders, in which delayed elasticity can be significant, and the binders can recover much of their deformation when unloaded. The parameter $|G^*|/\sin\delta$ does not allow direct evaluation of delayed elasticity.

The repeated Creep test offers valuable information about the susceptibility of asphalt mixtures to rutting when changes in temperature occur (Centeno, 2001). However, in order to fully characterize the asphalt binder behavior, multiple stresses are required.

#### 4.2.6.2. Multiple Stress Creep Recovery test (MSCR)

The Multiple Stress Creep Recovery (MSCR) test is the latest improvement to the Superpave Performance Graded (PG) Asphalt Binder specification. This new test and specification – listed as AASHTO TP70 ‘‘Standard Method of Test for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binders using a Dynamic Shear Rheometer’’, AASHTO MP19 and AASHTO M320. Provide the user with a new high temperature binder specification that more accurately indicates the rutting performance of the asphalt binder and is blind to modification.

A major benefit of the new MSCR test is reduces the number of samples needed for each stress level, also eliminates the need to run tests such as elastic recovery, toughness and tenacity, and force ductility, procedures designed specifically to indicate polymer modification of asphalt binders. A single MSCR test can provide information on both performance and formulation of the asphalt binder. (Federal Highway Administration's (FHWA's) Asphalt, U.S. Department of Transportation, 2011).

The MSCR test uses the well-established creep and recovery test concept to evaluate the binder's potential for permanent deformation. Using the Dynamic Shear Rheometer (DSR), the test uses an one-second creep load is applied to the asphalt binder sample. After the 1-second load is removed, the sample is allowed to recover for 9 seconds, for different stress levels at 10 cycles for each stress level.

Figure 4.7 shows typical data for a polymer modified binder. Normally two stress level are selected 0.1 kPa and 3.2 kPa; ten cycles for each stress for a total of 20 cycles.

The test is started with the application of a low stress (0.1 kPa) for 10 creep/recovery cycles then the stress in increased to 3.2 kPa and repeated for an additional 10 cycles. (Federal Highway Administration's (FHWA's) Asphalt, U.S. Department of Transportation, 2011)

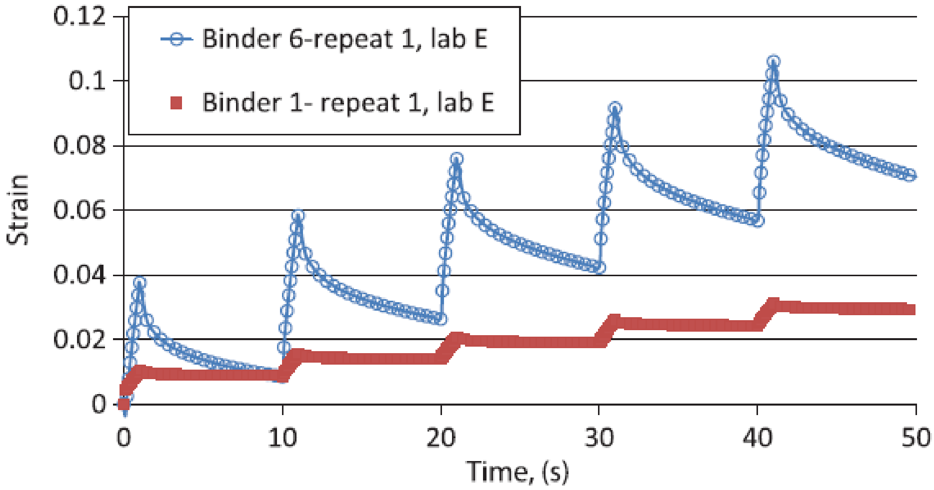


*Figure 4.7 -MSCR response curve. (Federal Highway Administration's (FHWA's) Asphalt, U.S. Department of Transportation, 2011).*

The non-recoverable compliance (Jnr) and the percent recovery after ten cycles at 0.1 kPa and 3.2 kPa will be studied in following chapters. The non-recoverable creep compliance $J_{nr}$ value is obtained by dividing the average non recoverable strain for 10 creep and recovery cycles, and the applied stress for those cycles.

$$J_{nr} = \frac{\gamma_{10}}{\tau}$$

Where

$\gamma_{10}$ = non recoverable strain for 10 creep and recovery cycles

$\sigma$ = applied stress to those cycles

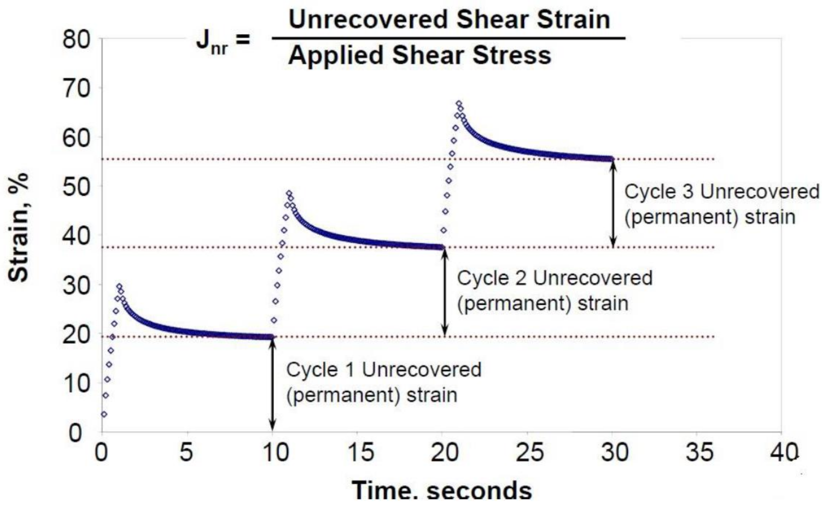


*Figure 4.8 -Typical Data MSCR and Jnr Definition. (Federal Highway Administration's (FHWA's) Asphalt, U.S. Department of Transportation, 2011)*

The Jnr parameter was suggested as a measure of the binder contribution to mixture permanent deformation (D'Angelo et al., 2006).

The material response in the MSCR test is significantly different than the response in the existing PG tests. In the PG system, the high In the PG system, the high temperature parameter, G*/sinδ, is measured by applying an oscillating load to the binder at very low strain. Due to the low strain level, the PG high temperature parameter doesn't accurately represent the ability of polymer modified binders to resist rutting (Federal Highway Administration's (FHWA's) Asphalt, U.S. Department of Transportation, 2011).

Under the very low levels of stress and strain present in dynamic modulus testing, the polymer network is never really activated. In the existing PG specification the polymer is really only measured as a filler that stiffens the asphalt. In the MSCR test, higher levels of stress and strain are applied to the binder, better representing what occurs in an actual pavement. By using the higher levels of stress and strain in the MSCR test, the response of the asphalt binder captures not only the stiffening effects of the polymer, but also the delayed elastic effects (where the binder behaves like a rubber band). (Federal Highway Administration's (FHWA's) Asphalt, U.S. Department of Transportation, 2011).

The compliance value $J_{nr}$ from the MSCR test provides the rut resistance and the amount of recovered strain from the test identifies the presence of polymer and also the quality of the blending of the polymer in the binder as shown in figure 4.9 [2].

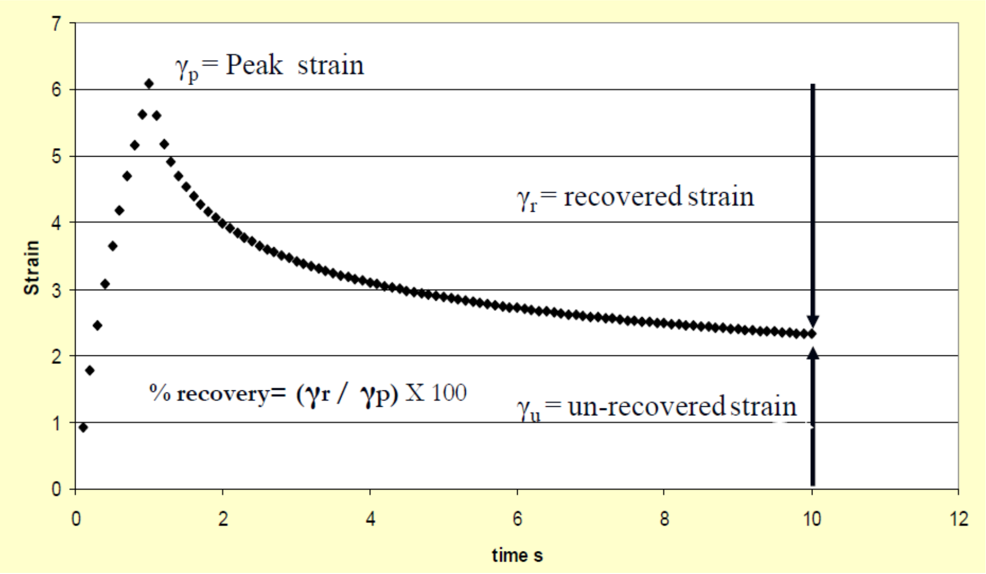


*Figure 4.9 -Determination of % Recovery in the MSCR test.*

## 4.3. Fatigue Cracking in Asphalt

### 4.3.1. Description

Series of interconnected cracks caused by fatigue failure of the HMA surface (or stabilized base) under repeated traffic loading. In thin pavements, cracking initiates at the bottom of the HMA layer where the tensile stress is the highest then propagates to the surface as one or more longitudinal cracks. This is commonly referred to as “bottom-up” or “classical” fatigue cracking. In thick pavements, the cracks most likely initiate from the top in areas of high localized tensile stresses resulting from tire-pavement interaction and asphalt binder aging (top-down cracking). After repeated loading, the longitudinal cracks connect forming many-sided sharp-angled pieces that develop into a pattern resembling the back of an alligator or crocodile.

*Figure 4.10 - Fatigue cracking as a result of frost action*

*Figure 4.11 -Very severely fatigued cracked pavement*

*Figure 4.12 -Fatigue cracking from a lack of edge support*

### 4.3.2. Problem

Indicator of structural failure, cracks allow moisture infiltration, roughness, may further deteriorate to a pothole.

### 4.3.3. Possible Causes

Inadequate structural support, which can be caused by a myriad of things. A few of the more common ones are listed here:

- Decrease in pavement load supporting characteristics
- Loss of base, subbase or subgrade support (e.g., poor drainage or spring thaw resulting in a less stiff base).
- Stripping on the bottom of the HMA layer (the stripped portion contributes little to pavement strength so the effective HMA thickness decreases)
- Increase in loading (e.g., more or heavier loads than anticipated in design)
- Inadequate structural design
- Poor construction (e.g., inadequate compaction)

### 4.3.4. Repair

A fatigue cracked pavement should be investigated to determine the root cause of failure. Any investigation should involve digging a pit or coring the pavement to determine the pavement's structural makeup as well as determining whether or not subsurface moisture is a contributing factor. Once the characteristic alligator pattern is apparent, repair by crack sealing is generally ineffective. Fatigue crack repair generally falls into one of two categories:

- Small, localized fatigue cracking indicative of a loss of subgrade support. Remove the cracked pavement area then dig out and replace the area of poor subgrade and improve the drainage of that area if necessary. Patch over the repaired subgrade.
- Large fatigue cracked areas indicative of general structural failure. Place an HMA overlay over the entire pavement surface. This overlay must be strong enough structurally to carry the anticipated loading because the underlying fatigue cracked pavement most likely contributes little or no strength (Roberts et. al., 1996[1]) [7.7].

## 4.4. Fatigue Mechanisms

There are two mechanisms of fatigue cracking in asphalt pavements: top-down and bottom-up (Huang, 2004) .Bottom-up cracks initiate at the bottom of an asphalt surface or asphaltic base layer as a result of high strains associated with flexure and propagate to the surface. Bottom-up cracking is the most common form of fatigue cracking. Top-down cracks initiate at the surface under the wheel path and propagate downward. Top-down cracking manifests as longitudinal cracks in the wheel path whereas bottom-up cracking leads to an inter-connected network of cracks, often referred to as alligator cracking.

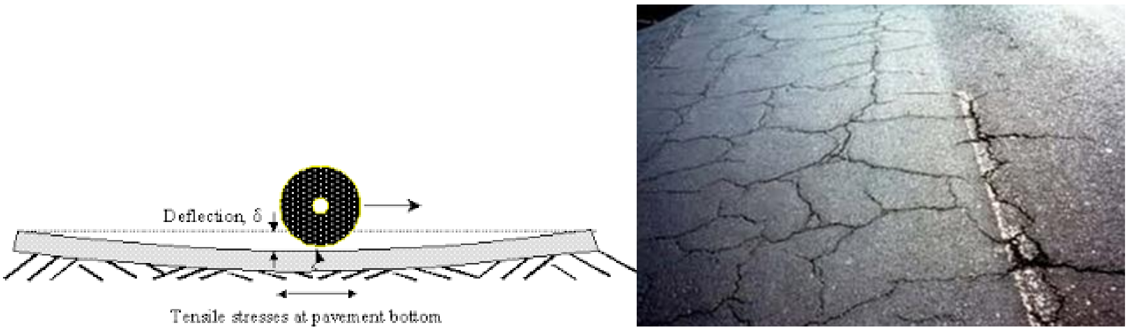


*Figure 4.13 -Bottom-up cracking.*

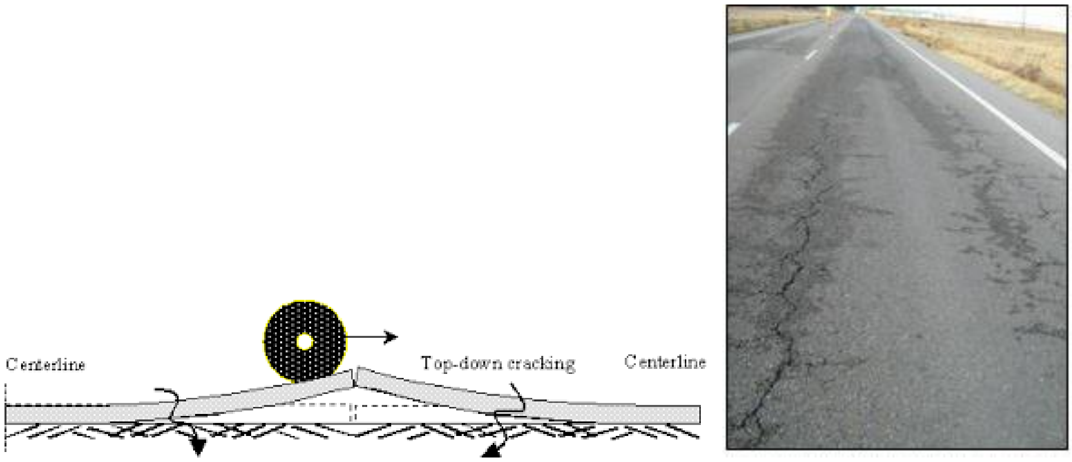


*Figure 4.14 -Top-Down cracking.*

Fatigue is one of the major distresses of flexible pavements and is mainly related to the rheological properties of the bituminous components of mixtures. (Francesca Frigio, 2016).

There are two types of loading modes usually controlled in the laboratory fatigue testing: constant stress (controlled–stress) mode and constant strain (controlled–strain) mode. In the constant stress loading mode, the stress is maintained constant in all the loading cycles and the strain increases with load cycles. In the constant strain mode of loading, the strain is maintained constant in all the loading cycles and the stress decreases with load cycles

## 4.5. Approaches to Characterize Fatigue Behavior

Four approaches are usually used in characterizing fatigue behavior of HMA: the traditional phenomenological approach; the continuum damage mechanics approach; the fracture mechanics approach; and the energy and dissipated energy approach.

### 4.5.1. Traditional Phenomenological approach

It is a combination of the phenomenological observation and the laboratory-tabulated data derived from designated fatigue tests.

August Wöhler studied the fatigue characteristics under a cyclic loading which resulted in development of S-N fatigue curves for shear stress.

S-N curves are two-dimensional curves, plotting cycles of failure (N) and shear stress (S) on X-Y axis on a logarithmic scale. The prerequisite of plotting these curves is that the stress has to be cyclic in nature. Every material, when subjected to alternate or cyclic loads, will fail if the loading surpasses its resistance. However, if rupture occurs at a load less than the projected resistance of the material, then it will be termed as fatigue. Major factors affecting the resisting force of a material, known as the "endurance limit," include:

- Corrosive environment
- Temperature variations.
- Surface finish and exposed area.
- Notches.

The main reason behind fatigue rupture is the development of minor cracks due to successive loading. With every loading, these cracks grow bigger, resulting in untimely failure. Wöhler curves help in the determination of upper limit of shearing stress that can be exerted on a material.

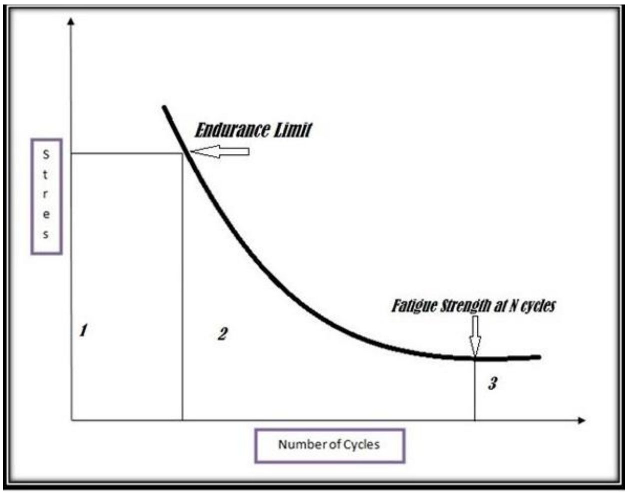


*Figure 4.15 -Wohler Curve*

In figure 24 the region 1, rupture occurs at very small alternations and the material is often subjected to plastic deformations. While in the region 2 denotes the area of limited endurance. *Region 3* is known as the security zone under low constraint.

The mechanism of fatigue failure can be described as a three-stage process involving crack initiation, propagation, and final fracture failure (true failure).

Also, Miner's (1945) cumulative damage concept has been widely used to predict fatigue cracking. It is generally agreed that the allowable number of repetition is related to the tensile strain at the bottom of the asphalt layer. The amount of damage is expressed as a damage ratio between the predicted and the allowable number of load repetitions. (Huang, 2004)

Results of the fatigue tests are formulated according to the mode of loading:

For controlled strain

$N_f = A\left({}^{1}/_{\varepsilon}\right)^{B}$

For controlled stress

$N_f = C\left({}^{1}/_{\sigma}\right)^{D}$

Where:

$N_f$ = *the number of load cycle to failure*

$\varepsilon$=*tensile strain repeatedly applied*

$\sigma$= *tensile stress repeatedly applied*

*A, B, C, D, = Material coefficients*

(Pell, 1967)He introduced the approach to relate the initial strain to load repetition, even in controlled stress mode of testing:

$$N_f = K_1\left(\frac{1}{\varepsilon_t}\right)^{K_2}$$

$N_f$ = *the number of load cycle to failure*

$\varepsilon_t$= *initial strain*

$K_1$, $K_2$= *fatigue coefficients.*

While in the Asphalt Institute and Shell design methods, the allowable number of load repetitions $N_f$ to cause fatigue cracking is related to the tensile strain $\varepsilon_t$ at the bottom of the asphalt layer and to the modulus $E_1$ by: (Huang, 2004)

$$N_f = K_1\left(\frac{1}{\varepsilon_t}\right)^{K_2}\left(\frac{1}{E}\right)^{K_3}$$

Where:

$N_f$ = *the number of load cycle to failure*

$\varepsilon_t$= *initial strain*

*E= stiffness of the material*

$K_1$, $K_2$ *and* $K_3$= *fatigue coefficients. (Laboratory regression coefficients)*

### 4.5.2. Fracture Mechanics Approach

Majidzadeh (1971) stated that the general form of Paris' crack growth law was applicable to asphalt mixtures. This fracture mechanics based approach characterizes fatigue cracking into three stages: crack initiation, stable crack propagation, and unstable crack fracture. The size of the plastic zone (crack opening and length) is critical in obtaining stress intensity factors at the crack tip and analyzing fatigue life during crack propagation stage. (Majidzadeh, 1971)

Fracture mechanics model based on Paris Law is shown below, which is proposed for stage II because this stage consumes most of the fatigue life.

$$\frac{da}{dN} = A(K_I)^n$$

Where:

*da/dN = rate of crack growth*

*a= the crack length*

*N= the number of load replication*

*A, n= parameters according to the material and experimental conditions*

$K_I$ = *Stress intensity factor in mode I*

### 4.5.3. Dissipated Energy Approach

The dissipated energy approach has been used for fatigue analysis in pavement engineering over the last few decades.

When a material is sustaining external loading, the area under the stress – strain curve represents the energy being input into the material. For a non-pure elastic material, a loading and unloading process will always associate with energy dissipation since the loading and unloading path does not overlap.

The energy dissipated during each loading cycle captures the effects not only of the imposed strain but also of the dynamic mixture properties, which makes it key in the development of surrogate models for fatigue life prediction (Shen, Airey, Carpenter, & Huang, 2006).

Regarding to the pavement area, the total dissipated energy up to the number of cycles to failure and other cumulative dissipated energy representations were predominantly used as the energy parameter relating to fatigue failure. However, the relationships established based on these approaches were either material or loading mode dependent, or both. They assumed that all the dissipated energy represents damage done to the material (SHRP, 1994; Van Dijk, 1975, Pronk et al., 1990). In reality, only a portion of the total energy is responsible for the micro crack extension and the structural damage of the materials. Although cumulative dissipated energy indirectly recognizes the fact that not all dissipated energy is inducing damage, it cannot directly determine the value of the damage being done to the material (Ghuzlan, 2001). This requires an improved energy parameter to describe the progression of fatigue damage. (Shen, Airey, Carpenter, & Huang, 2006)

The total dissipated energy consist in three components:

- The crack separation energy (the energy spent in the actual creation of new surface due to micro-separation processes.
- The plastic dissipated energy
- The residual strain energy.

If crack growth occurs, it will cause, besides a change in separation energy, a change in the residual elastic energy. However, the plastic energy dissipation before the crack extends may influence the crack initiation but does not contribute to the crack extension (Turner and Kolednik, 1994).

### 4.5.4. Initial Dissipated Energy Approach

Initial dissipated energy (IDE) is the dissipated energy measured at initial loading cycles.

Usually in fatigue testing, the first 50 cycles are regarded as the conditioning cycles and the dissipated energy at the 50th loading cycle is considered as the initial dissipated energy

Strategic Highway Research Program (SHRP-A-404, 1994) used the following surrogate model to relate initial dissipated energy to fatigue life:

$$N_f = 6.72e^{0.049VFB}(w_0)^{-2.047}$$

Where:

$N_f$ = *fatigue life*

*VFD= percentage of voids with bitumen*

$w_0$ = *initial dissipated energy*

One disadvantage of the initial dissipated energy approach is that it is not appropriate for the whole loading range, especially when dealing with low strain fatigue tests.

### 4.5.5. Cumulative Dissipated Energy Approach

The cumulative dissipated energy is the summation of the dissipated energy experienced by the material during the fatigue test, which relates the fatigue behavior to both initial and final test cycles. A relationship between the cumulative dissipated energy and the number of loading cycles to failure is characterized as:

$$W_N = A(N_F)^z$$

Where:

$W_N$ = *Cumulative dissipated energy to failure*

*A, z = experimentally derived mix coefficient*

$N_f$ = *Number of load cycle to failure*

Van Dijk (1975, 1977) found that there is a strong relationship between the cumulative dissipated energy and the number of loading cycles to failure. This relationship is not affected by the loading mode (controlled-stress or controlled-strain), the effects of frequency (between 10 Hz and 50Hz) and temperature (between 10ºC to 40ºC), and the occurrence of rest periods.

However, it is highly material dependent and has to be mix specific to be constructed and applied.

In 1992 Tayebali et al. introduced two terms: the stiffness ratio which is the ratio of the stiffness at load cycle (i) to the initial stiffness; and the dissipated energy ratio which is defined as the ratio of cumulative dissipated energy up to load cycle (i) to the cumulative dissipated energy up to fatigue life. His work suggested there is a unique relationship between the stiffness ratio and the dissipated energy ratio, but not necessarily between cumulative dissipated energy and fatigue life, which is also verified by Strategic Highway Research Program (SHRP A-404,1994) and Fakhri (1997). This relation was found to be mix and temperature dependent.

Carpenter and Jansen (1997), use the dissipated energy ratio (DER) as a parameter to relate to fatigue life. This approach believes not all the dissipated energy is responsible for material damage. For each cycle, the loss of energy due to material mechanical work and other environmental influence remains almost unchanged. Therefore, if the dissipated energy starts to change dramatically, it could be explained as the development of damage. The NCHRP Report 459 (Bahia et al. 2001) provided a new method to predict bitumen fatigue properties by performing repeated loading time sweep tests with the DSR (Dynamic Shear Rheometer) equipment. The corresponding evaluation criterion is not based on the analysis of a single parameter but on the study of the fatigue damage development by computing the total dissipated energy during loading cycles. In particular, during fatigue tests the complex modulus (G*) and the phase angle (δ) can be obtained as a function of loading cycles and the corresponding dissipated energy per cycle $W_i$ can be written as a function of the stress $\sigma_i$ and the strain $\varepsilon_i$ amplitudes at the ith cycle, as follows. (Francesca Frigio, 2016)

$$W_i = \pi \sigma_i \varepsilon_i Sin(\delta_i)$$

Where $\delta_i$ is the phase angle at the ith cycle.

In the case of test is performed in control strain or stress the $W_i$ will be as follows:

<u>Control Strain</u> $\sigma = G_i * \varepsilon_i$

$W_i = \pi \varepsilon_i^2 Sin(\delta_i)$

<u>Control Stress</u> $\varepsilon = {}^{\sigma_i}/_{G_i}$

$$W_i = \frac{\pi \sigma_i^2 Sin(\delta_i)}{G_i}$$

Then, the damage accumulation can be evaluated through the change in the Dissipated Energy Ratio (DER) over loading cycles:

$$DER = \frac{\sum_{i=1}^{n} W_i}{W_n}$$

Where $W_n$ is the dissipated energy at the nth cycle.

In strain-controlled tests, the curve of DER versus number of cycles (Fig. 4.16) shows an initial linear part that is indicative of no damage as the energy dissipated per cycle is constant. Then, when cracks form and start to propagate, the dissipated energy per cycle tends to rapidly increase experiencing no recoverable fatigue damage. From this curve, two points can be conventionally selected as fatigue failure parameters: $N_{P20}$ is defined as the number of cycles to achieve 20 % deviation of DER from the no damage stage (Bonnetti et al. 2002) and represents the crack initiation condition, whereas $N_P$ is defined as the number of cycles where crack propagation starts. (Francesca Frigio, 2016)

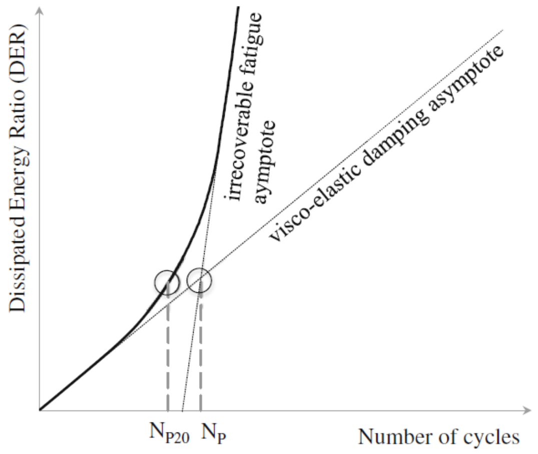


*Figure 4.16 -Typical plot DER as a function of number of cycles in strain controlled test.*

### 4.5.6. Work Ratio Approach

This approach was first introduced by Van Dijk and Visser (1977) and further developed by Rowe (1993). The work ratio, $\psi_{N1}$, is defined as the ratio between the product of the initial dissipated energy in cycle 1 and N1 divided by the cumulative dissipated energy, as shown:

$$\psi_{N1} = \frac{w_0 N1}{W_{N1}}$$

Where:

$w_0$ = *initial dissipated energy*

*N1= number of load cycles to crack initiation and*

$W_{N1}$ = *Cumulative dissipated energy at cycle N1*

Rowe (1993) found that the work ratio can be used effectively to predict the fatigue life to crack initiation. The crack initiation is assumed to occur at 60% reduction of original extensional complex modulus according to the following equation:

$$N1 = 205 * \mathrm{V}_b^{6.44} * w_0^{-2.01} * \psi_{N1}^{1.64}$$

Where:

$V_b$ = *volume of* binder (%)

### 4.5.7. Improved Use of Dissipated Energy: Ratio of Dissipate Energy Change (RDEC) approach

One big disadvantage of these dissipated energy approaches is that the energy fatigue life relationship is either material dependent, or loading mode dependent, or both. For different mixture types or different loading modes, the relationship must be generated individually since the relationship in itself does not include the material and load information. These dissipated energy approaches assume that all the dissipated energy represents damage done to the material. In fact, a crack will only grow when there is a difference in energy dissipation from one cycle to another. Such change can be considered as the amount of damage done to the sample. (Shen, Airey, Carpenter, & Huang, 2006)

From this point the dissipated energy ratio (DER) approach was improved in the subsequent years leading to the Ratio of Dissipated Energy Change (RDEC) which is based on a change in DE between load cycles, provides a true indication of the damage being done to the mixture from one cycle to another as a function of how much dissipated energy was involved in the previous cycle, thus presenting a fundamental mechanistic picture of how damage accumulates in the material during fatigue tests. The energy parameter, Plateau Value (PV), which is obtained using this approach represents a comprehensive material and load related parameter that can be uniquely related to the number of loading cycles to failure, regardless of material type, loading mode, test condition, etc. (Shen, Airey, Carpenter, & Huang, 2006).

In conclusion the energy dissipated in a cycle depends on the energy dissipated in the previous cycles. The Ratio of Dissipated Energy Change (RDEC) can be:

$$RDEC = \frac{DE_{n+1} - DE_n}{DE_n}$$

Where:

*RDEC = the ratio of dissipated energy change per load cycle.*

$DE_n$ = *the dissipated energy produced in load cycle n*

$DE_{n+1}$ = *the dissipated energy produced in a load cycle n+1*

RDEC eliminates the dissipated energy that does not produce crack extension damage. This provides a true indication of the damage being done to the mixture from one cycle to another by comparing the previous cycle’s energy level and determining how much of it caused damage.

As introduced by (Carpenter et al., 2000, 2003), the RDEC develops a plateau after the initial unstable period which then increases dramatically giving a sign of true fatigue failure.

The damage curve given by de RDEC can be divided in three stages; such damage curve develops a plateau after the initial period. This plateau stage (stage II), an indication of a period where there is a relatively constant percentage of input energy being turned into damage, will extend throughout the main service life until a dramatic increase in RDEC, which gives a sign of true fatigue failure (stage III). The RDEC curve is representative for fatigue behavior. RDEC curves with similar trend: a rapid decrease, followed by a plateau region for the majority of the fatigue cycles. (Shen, Airey, Carpenter, & Huang, 2006)

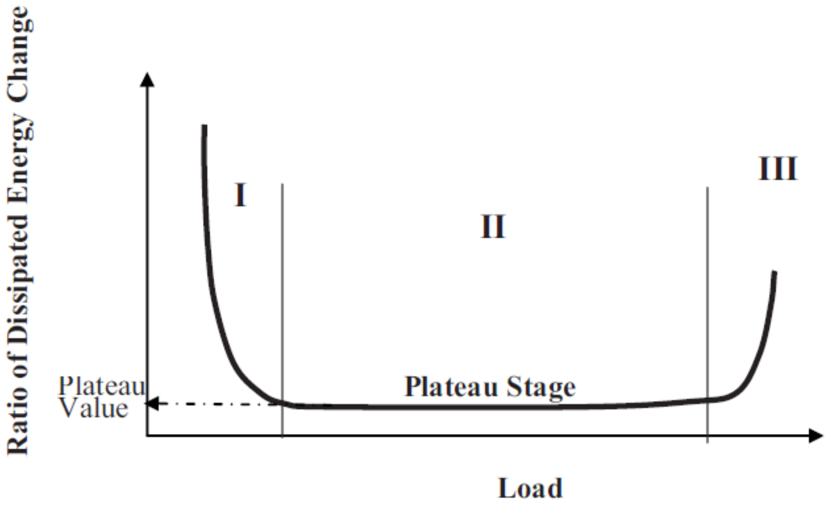


*Figure 4.17 -RDEC curve with 3 behavior zone (Carpenter et al.2003)*

In the plateau stage (stage II), the RDEC value is almost constant, characterizing period where there is a constant percentage of input energy being turned into damage.

A plateau value, PV, is defined as the RDEC value corresponding to the 50% stiffness reduction load cycle ($N_{f50}$). This typical value is selected because at this load cycle ($N_{f50}$), the RDEC is always in the plateau stage and the $N_{f50}$ is the defined failure point. It has been found that the plateau value (PV) is mixture and load/strain input related. For any one mixture the PV is a function of the load inputs, and for similar load inputs, the PV varies with mixture types (Ghuzlan, 2001). The PV value is significant because it provides a unique relationship with fatigue life for different mixtures, loading modes and loading levels (Ghuzlan et al., 2000 and 2001).

In summary, the main advantages of the RDEC approach are:

- It provides a new means of defining a true fatigue failure point which is directly related to the increase in damage.
- The unique fatigue relationship (PV-$N_f$) that it presents is independent of mixture type (bitumen grade, aggregate type, gradation, etc.), mode of loading (controlled

strain or stress) and loading (test) conditions. It is therefore a fundamental relationship.

- The uniqueness of the PV-$N_f$ relationship provides a way to study both fatigue endurance limit and healing, both of which have a great influence on asphalt material fatigue behavior. (Shen, Airey, Carpenter, & Huang, 2006)

### 4.5.8. Healing phenomenon

In different literature over the years has considered the healing phenomenon as the capability of self-recovery of the material.

In 1990 Kim et al propose two mechanics of healing for asphalt materials that occurs during rest period:

1) Relaxation of the stresses in the system due to the viscoelastic nature of asphalt concrete.
2) The chemical healing across micro crack and macro crack faces (Shen & Carpenter, 2007).

It was proposed that the healing mechanism is related to the flow properties of the asphalt, which are influenced by molecular interactions within the asphalt. (Shen & Carpenter, 2007).

Phillips (1998) proposed that the healing of binders is a three step process consisting of:

1) The closure of micro cracks due to wetting (adhesion of two crack surfaces together driven by surface energy), this step occurs faster resulting as a recovery of the stiffness.
2) The closure of macro cracks due to consolidating stresses and binder flow, and
3) The complete recovery of mechanical properties due to diffusion of asphaltene structures. This step along with the step 2 occurs much slower but results in an improvement of the stiffness and the material strength.

Healing can be measured by intermittent loading plus inserting rest periods and measuring the increase in fatigue life; or by storage recuperation and the recovery of mechanical properties over a period of time when the sample is not subject to loading. (Shen & Carpenter, 2007).

Jacobs in 1995 found that the introduction of rest period has a beneficial effect on the fatigue life [2].

## 4.6. Bitumen Fatigue

### 4.6.1. Introduction

Bitumen plays an important role in asphalt pavement fatigue. For normal paving grade bitumen fatigue performance generally improves with increasing penetration. However, this general rule does not hold for special binders like high stiffness binders, polymer modified bitumen and Multigrade bitumen [34].

Concerns have been raised with the fatigue analysis of a bituminous binder. Attempts have been made to include a binder parameter in the fatigue prediction of asphalt mixtures with the current Superpave fatigue criterion, G_ sin δ. However, G_ sin δ is determined as the binder property performs in linear viscoelastic range at low-strain/stress levels within a few cyclic loadings. This Superpave criterion generally is not in good agreement with establishing a comprehensive correlation with the fatigue life of an asphalt mixture [34]. In addition, because bituminous binders exist as thin film in asphalt mixtures, the strains of the binders could be performing in the nonlinear range owing to the significant difference between the stiffness of the mixtures and that of the binders. To overcome the fact that G_ sin δ does not take account of nonlinearity behavior of bituminous binders, direct testing in fatigue has become common, with generation of fatigue characteristics under larger strains/stresses for the binders by means of time sweeps using a dynamic shear rheometer (DSR). A number of research groups have successfully generated load-associated fatigue properties for plain and modified bitumen [34].

### 4.6.2. Bitumen Fatigue Criteria

Fatigue behavior is one of the least understood phenomena in binders, especially defining a criterion that can satisfactorily explain a fatigue failure. In Superpave binder specification AASHTO M320 G*sin(δ) (< 5MPa) an intermediate temperature was selected to establish fatigue criterion (AASHTO M320-10). Several studies have been conducted to better understand the fatigue behavior of binders. In a study by Johnson et al. 2007, the fatigue life of asphalt mixes was compared to the fatigue life of the binders as defined by G*sin(δ). The results from the study show that alternate methods seem to be more accurate based on field studies (Johnson et al. 2007) [35]. The method Johnson investigated involved the decrease in the G* value for a strain controlled DSR test. Another method that has become the interest of researchers takes into account the dissipated energy of binders undergoing fatigue tests. Dissipated energy describes the amount of energy that escapes with each cycle during the test. The dissipated energy ratio (DER), which is the value of the cumulative dissipated energy over the dissipated energy at a particular cycle n, has been suggested as a criterion for fatigue failure. The 50% drop in G* value and the DER from sweep-test using the dynamic shear rheometer (DSR) have been used to determine the number of cycles to failure. The ways in which failure is described are:

1. The number of cycles it takes to cause the initial G* value to drop 50%, and
2. The number of cycles before the DER diverges from a linear trend (Anderson et al. 2001, Bonnetti et al. 2001, Johnson et al. 2007, and Planche et al, 2004) [35]. Though neither method has been defined as the most accurate measure of the fatigue life of binders, both have proven to be more accurate than traditional methods.

### 4.6.3. Stiffness of Binder in Fatigue Testing

In order to measure the fatigue life of an asphalt binder, a specific initial modulus (G*) value must be attained. Instability flow is a type of failure which can occur during sweep tests in the DSR. It is a condition in which the binder begins to flow, causing the surface area of the sample in the DSR to decrease. This type of failure is not true fatigue failure and is avoided by making the binder sufficiently stiff at the beginning of testing. Anderson et al. 2001 recommended testing at temperatures so that the initial G* value at least 15 MPa. In order to achieve the proper stiffness, the temperature of the binder has to be low in comparison to typical fatigue tests (Anderson et al. 2001). This was confirmed in a study by Planche et al. 2004., noting that under repeated shear, the initial complex modulus can significantly affect the behavior of the binder [35].

### 4.6.4. Impact of Modified Binders

Several studies (Bonnetti et al. 2001 and Buisson et al. 2010) have shown that modified binders experience longer fatigue lives than neat binders. In a study by Bonnetti et al. 2001 in which the impact of modified binders on fatigue life was analyzed, it was determined that these binders have improved fatigue life. This was also confirmed by Anderson et al. 2001 and Planche et al. 2004 [35].

### 4.6.5. Strain Amplitude

The strain amplitude in the DSR testing procedure is a measure of the amount of deformation the binder undergoes and can be related to load intensity of the traffic, especially for thin overlays. As the strain amplitude increases, it leads to lower fatigue life of the binder. Over several loading cycles, this deformation weakens the binder and eventually it will break down, or crack (Masad et al. 2001) found that the strain within the binder comprises about 90 times the average throughout the hot mix asphalt sample [35].

### 4.6.6. Impact of Steric Hardening and Temperature

An asphalt binder which has been heated to a fluid consistency will stiffen at ambient temperatures (around 20°C). This reversible phenomenon is called steric hardening and can occur even during testing. Evidence of steric hardening can be observed by an increase in modulus usually during the beginning of the test (Anderson et al. 2001 and Bonnetti et al. 2001) [35]. Steric hardening can most simply be described as thixotropy (molecular structuring) which can be reversed by working the material mechanically or by applying heat. Increased levels of steric hardening occur in samples which are cooled slowly and can affect the stiffness of the binder (Bell). While steric hardening is occurring, the temperature of the binder does not change [35].

Most tests conducted to research fatigue life were done on RTFO-aged binders (neat, polymer, and chemically modified). Successful studies have evaluated the fatigue lives of neat and polymer-modified asphalt binders. It is accepted that polymer modified samples have a longer fatigue life than neat samples. This was proven in tests by Bonnetti et al. 2001 using the DER analysis method on RTFO-aged binders [35].

Studies performed in recent years typically use either the 50% drop in G* method or the DER method to define fatigue. By providing a comparison of these two methods, previously executed tests can be analyzed more effectively by researchers using the other method. Comparisons could be made from study to study and a more accurate definition of fatigue can be determined.

# CHAPTER 05:

# Experimental Program

# 5. CHAPTER 05: Experimental Program.

## 5.1. Introduction

The purpose of this research is to investigate how bitumen properties change as a result of ageing. In particular, the rheological properties of five samples of bitumen, produced by an oil company, and to determine the influence of strain amplitude and recycled rubber powder on short and long term-aged polymer modified bitumen. This research will compare the fatigue life of the modified short and long-aged bitumen, and determine the influence of strain amplitude, and recycled rubber powder on fatigue life. The lesser the strain amplitude, the longer is the fatigue life.

The materials under study were subjected to laboratory tests in the oscillatory regime, made with the Dynamic Shear Rheometer. The oscillatory regime used by the equipment DSR is of harmonic sinusoidal type and, given the variability in continuous of tensions and deformations, the tests in these conditions are commonly defined dynamic analysis (Dynamic Mechanical Analysis, DMA).

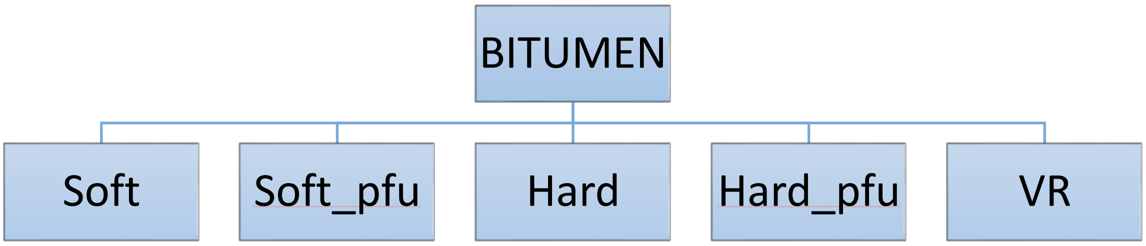
In this chapter will be presented in detail the rotational rheometer DSR used in experimentation and test procedures performed. It will be analyzed, finally, the results obtained in terms of the complex modulus G* and phase angle δ. It is emphasized that the experimental phase concerning the implementation of the specimens and the rheological characterization and performance of the materials under study was conducted at the School of Engineering and Architecture of Bologna, in the laboratory of DICAM (Department of Civil Engineering, Chemistry, Environmental and materials).

## 5.2. Materials

The materials were provided by an oil company, The table below shows the composition and the identification code of the tested bitumens.

| Bitumen | Modifier | Recycled rubber powder | ID |
|---|---|---|---|
| PMB 45/80-70 Hard + Viscous Reducer. | (4-5) % SBS | 10% | VR |
| PMB 45/80-70 Hard | (4-5) % SBS | 10% | Hard_pfu |
| PMB 45/80-70 Hard | (4-5) % SBS | - | Hard |
| PMB 45/80-55 Soft | (2-3) % SBS | 10% | Soft_pfu |
| PMB 45/80-55 Soft | (2-3) % SBS | - | Soft |

*Table 5.1 - bitumen types used in this study*



## 5.3. Overview of experimental research program

Five polymer modified bitumen were used, all samples were tested without ageing conditioning in DSR to establish the deformation ($\gamma_{LVE}$) where the specimen is kept in LVE range from Amplitude Sweep Test, and to obtain the master-curves from Frequency Sweep Test. After that, In order to simulate the short-term ageing of bitumen which occurs especially during mixing in asphalt plan, the five sample were aged by using the Rolling Thin Film Oven Test (RTFOT). Then, Amplitude and Frequency sweep test were done by DSR. After that, in order to simulate the long-term aging during in-service, the samples were aged by using also the RTFOT (3-cycles) instead of Pressure Aging Vessel (PAV), then Amplitude and Frequency sweep test were done for the long aged samples.

After that, for the short term aged bitumen, to be able to develop the relation between shear strain amplitude ($\gamma$) and fatigue life (N) each bitumen is tested at minimum three different strain amplitude levels (Fatigue Test). Then, Time sweep test with rest period one hour was established in order to define the material recovery for the introduction of the rest period plus the increasing in the fatigue life. Then, to make a comparison of the material recovery and the fatigue life between the five short aged bitumen when we have the same initial stiffness G*, time sweep test were done at different temperatures and at one stain level (4%).

After that, for the long aged bitumen, the fatigue tests and Time sweep tests were done at just one strain level (4%).

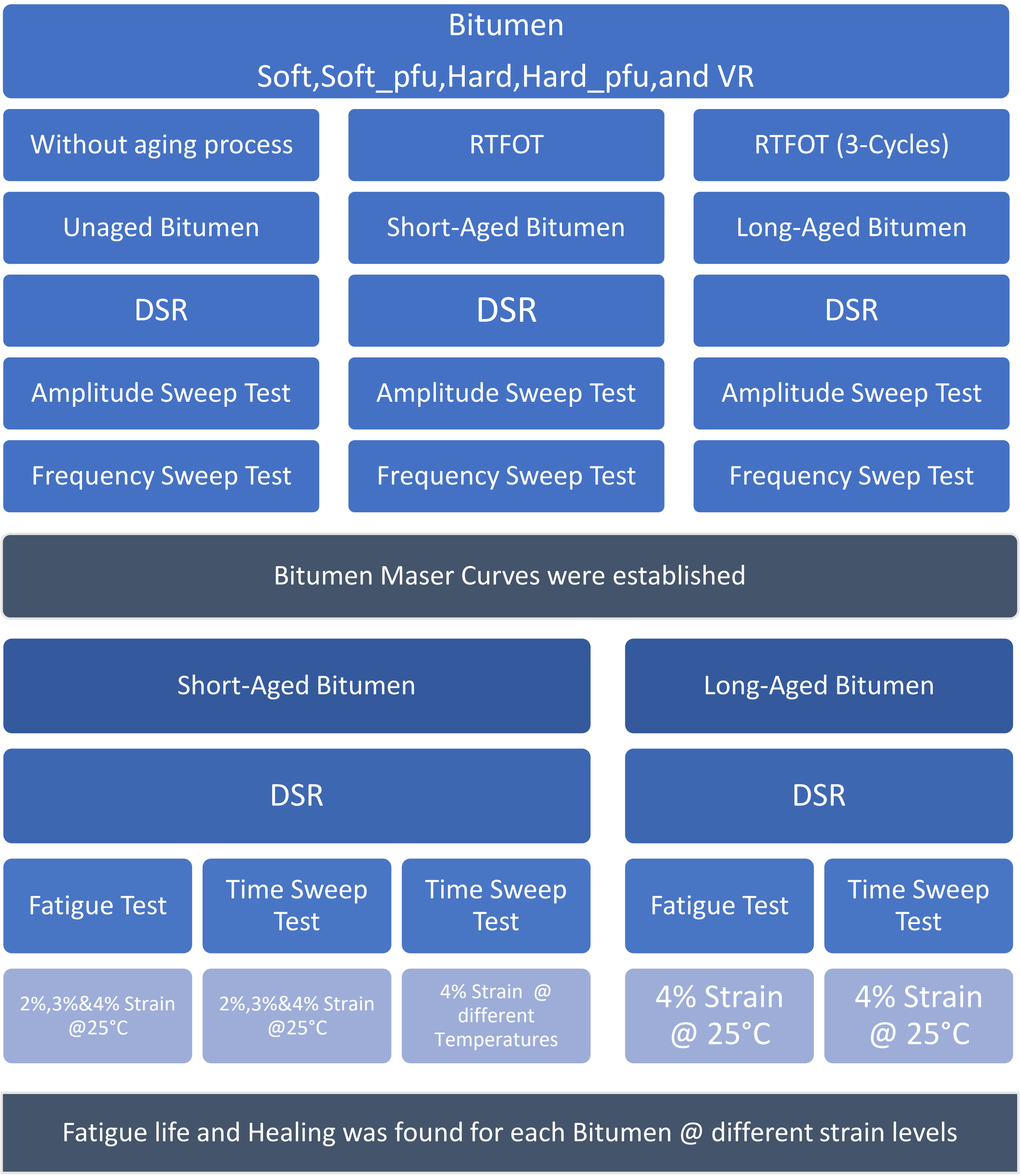


*Figure 5.1 -Experimental research program flow chart*

## 5.4. Preparation of modified binders

The preparation of the modified binders includes a series of operations necessary to provide a representative sample of the material under examination. The correct execution of sampling is necessary condition to obtain reliable results from laboratory tests to which the samples will be subjected. As the bitumen is a thermally sensitive material, therefore the packaging temperature greatly influences the characteristics of the modification; in particular, in order to homogenize the polymer-binding mixture, it will be necessary to operate at a temperature of 180 ° C. This problem does not exist in the case of additives due to the chemical-physical characteristics of the waxes which have low melting points. In addition to the thermal requirements, it is appropriate to ensure the correct mixing conditions in the sampling phase, in order to avoid phase separations. In particular, the samples were mixed with a Silverson L4R mixer (figure 5.2) which has the particularity of having a static component (working head) and a dynamic bladed component (rotor), thanks to which it is possible to generate a high shearing action. Due to this feature, the L4R model lends itself to a wide variety of applications such as mixing, emulsions and dissolutions. The rotation speed of the impeller creates a suction action of material, liquid and solid, from the bottom of the vessel inside the working head (Figure 5.3). For the centrifugal effect, the mixture is pushed towards the outside of the impeller and conducted through the working head. The constructive precision between the inner surface of the stator and the rotor blades, in addition to stator geometry itself, is responsible for a high shearing action, also capable of shredding solid elements such as SBS granules. The mixture created is then expelled radially with a forced passage through the working head frame. The combination between the outgoing current and incoming current from the impeller creates a recirculation motion thanks to which the air supply from the external surface is minimized [30].

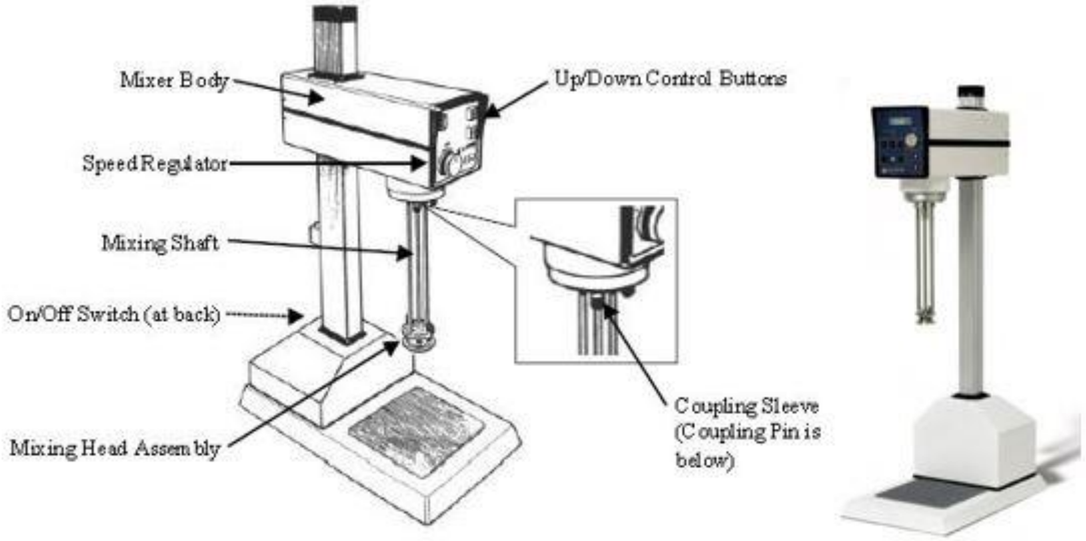


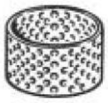
*Figure 5.2 -Silverson L4R High Shear Mixer.*

The efficiency of the motions and the ability to involve the whole mass enclosed by the vessel, depends both on the vessel itself and the characteristics of the mixture. in the case of PMB, it is necessary to vary the height of the impeller to also involve that the surface mass, because of the cooling or the same type of circulatory motion created, it may not be incorporated and then processed. The motor develops a power of 250 W and has a nominal maximum speed of 8000 rpm; at full load it is possible to reach a maximum speed of 6000 rpm [30].

Several accessories are supplied with the mixer, which can be interchanged depending on what mixing conditions are required [see below].

**General Purpose Disintegrating Head**
Suitable for general mixing or disintegration of solids.

**Square Hole High Shear Screen**
High shear screen suitable for emulsion and fine colloid suspension preparation.



**Emulsor Screen**
Lower shear screen suitable for liquid/liquid and emulsion preparations.

**Axial Flow Head**
Used in addition to one of the above screens to force liquid flow upwards
This can reduce aeration or help maintain large suspensions, but high mixing speeds can cause liquid to be ejected from the mixing vessel.

## 5.5. Methods of laboratory ageing

The binder properties of asphalt mixtures change over time during mixing, compaction and service. In these phases chemical and physical characteristics of bitumen change, in particular oxidation occurs involving the increase of material stiffness. This phenomenon is known as aging and it is one of the key factors affecting the lifetime of an asphalt pavement. Several methods of laboratory ageing can be divided in two main categories, that is tests able to simulate the short-term ageing and methodologies for simulating the long-term effects of oxidation process. The most common European method, which simulates the ageing during the storage, mixing with aggregates, transportation and laying, is the Rolling Thin Film Oven Test (RTFOT). Other tests, like Rotating Cylindrical Ageing Tests (RCAT), are performed following the American standards. On the other hand, Pressure Ageing Vessel (PAV) is the most widely used laboratory test for the simulation of long-term ageing.

All of them are based on the variation of three main factors: temperature, pressure and time. Temperature is strongly related to the oxygen diffusion coefficient. The raising of pressure and the extension of ageing time can surely apply greater ageing, speed up the process of oxidation, and consequently, results in major deterioration.

### 5.5.1. RTFOT ageing

The Rolling Thin Film Oven Test (RTFOT) was developed during the SHRP [29] in order to simulate the short-term ageing of bitumen which occurs especially during mixing in asphalt plan. Bitumen suffers high temperature, 163°C, combined with a constant and specific supply of air, following the standard procedure described in standard EN 12607-1:2014. The apparatus consists of an oven which inside is equipped with an aluminum carriage. It has eight opening and springs clips for firmly holding the corresponding eight glass containers in a horizontal position. Moreover, an air jet is placed in a specific location. Figure 5.3 shows the outside and inside of the device.

*Figure 5.3 -Rolling Thin Film Oven Test equipment.*

During the test, the carriage rotates about the horizontal axis with a certain speed, which allows the air jet to enter in each glass. In detail, the air shall blow along the main axis of each glass container. In the same time, high temperatures lead the material to liquefy. The rotation together with the applied temperatures lead bitumen to cover the entire surface of each container forming a thin film, which also rotates following the carriage movement.

Temperature is continuously monitored with a thermometer positioned inside the device. Figure 5.4 shows pictures of bitumen containers before and after the test.

*Figure 5.4 -Samples pre- and post- RTFOT*

The glass containers should be filled with 35,0±0,5 g of bitumen, a specific amount that ensure that the material does not leak from the containers during rotation. The number of necessary glass depends on the planned characterization tests of the operator.

In order to simulate the short-term ageing of bitumen which occurs especially during mixing in asphalt plan, we used the Rolling Thin Film Oven Test (RTFOT):

- 35 g of bitumen
- Temperature: 163 °C
- Air flow: 4000 ml/min.
- Time: 75 minutes

Since the fatigue cracking generally occurs late in the life of the pavement, and therefore the bitumen needs to be tested after appropriate long-term ageing, so in order to simulate the long-term aging during in-service, we used also the RTFOT instead of Pressure Aging Vessel (PAV):

- 35 g of bitumen
- Temperature: 163 °C
- Air flow: 4000 ml/min.
- Time: 3cycles (one cycle=75 minutes).

The preparation of the modified and additioned binders includes a series of operations necessary to provide a representative sample of the material under examination.

## 5.6. Test Methods.

### 5.6.1. Dynamic Shear Rheometer (DSR)

The DSR apply a torque on the sample, correspondingly is measured the angular rotation on the sample. The determination of the properties of the material (|G*|, G', G", δ) is done using a relationship between the torque to the relative angular velocity or rotation and going by these tensions and deformations tangential $\tau$ and $\gamma$.

*Figure 5.5 -Dynamic shear rheometer (DSR).*

One of the advantages that offer these tools is the great versatility, which is the possibility to vary, on a same machine, the measuring system according to the type of investigation to be performed. It is possible in fact the use of the systems coaxial cylinders, couplings plate-plate and plate-cone, each of which is available in different sizes that may be standard or less (custom made). Other important prerogatives are commonly most of these devices have the ability to perform the tests in continuous regime and in oscillatory regime and the possibility to operate both in the control of stress (stress control) that strain (strain control). In stress control, apply a certain desired tension and measure the resulting deformation to them, while in the control strain, the torque applied to the sample is adjusted to a value that allows to obtain certain velocities and amplitudes of deformation.

### 5.6.2. Plate-plate system DSR

The measuring system is formed by a lower plate, usually fixed to the machine, while the upper plate is attached to the motor shaft where the rotation or oscillation is applied.

The geometric quantities that define the system are the radius R and the so-called gap which is the distance between the plates, upper and lower measurement position. In general, two geometries (plates) are commonly used with the DSR. An 8 mm diameter spindle with a 2 mm testing gap and a 25mm diameter spindle with a 1 mm testing gap. The selection of testing geometry depends on the operational conditions; with the 8 mm plate generally used for low temperatures and the 25 mm plate for high temperature.

The DSR employs the following equation to obtain the torque T, the rotation angle ϴ and the angular frequency ω.

$$\tau = \frac{2T}{\pi r^3}$$

$$\gamma = \frac{\theta r}{h}$$

*Where:*

$\tau$= *applied stress*
$\gamma$= *applied strain*
$T$=*torque*
$h$= *specimen height*
$r$ = *specimen radius*

Then the complex modulus G* and the phase angle δ can be determined.

Figure 5.6 shows in oscillating test plate-plate systems how the shear stress and strain in determinate.

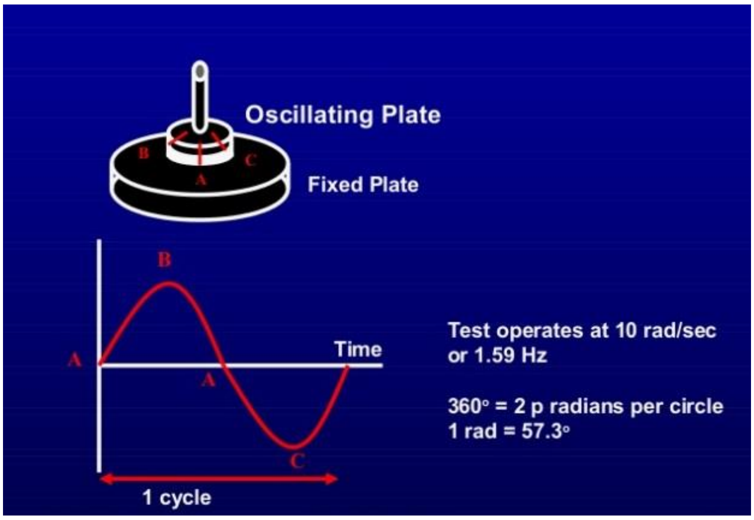


*Figure 5.6 -Scheme of oscillatory test one cycle*

### 5.6.3. Amplitude Sweep Test

The purpose for this test is to establish the deformation ($\gamma_{LVE}$) where the specimen is kept in LVE range. Generally, the deformation $\gamma_{LVE}$ is defined at the strain where the complex modulus G* is 90% of the initial value.

A configuration of parallel plates with a diameter of 8 mm with 2 mm gap (measurement system) for the DSR (Anton Paar MCR 302). The strain $\gamma$ ranges from 0.01% - 100% and $\omega$= 10 rad/s in accordance with the AASHTO T315-10.

Firstly, the DSR must be initialize then the measurement system is attached to the machine (8mm spindle). Afterwards the temperature is set, in this case the test will be done at low temperature which is 10°C, and later through the option Set Zero Gap the testing gap of 2mm for an 8 mm plate is set. (Fig. 5.7)

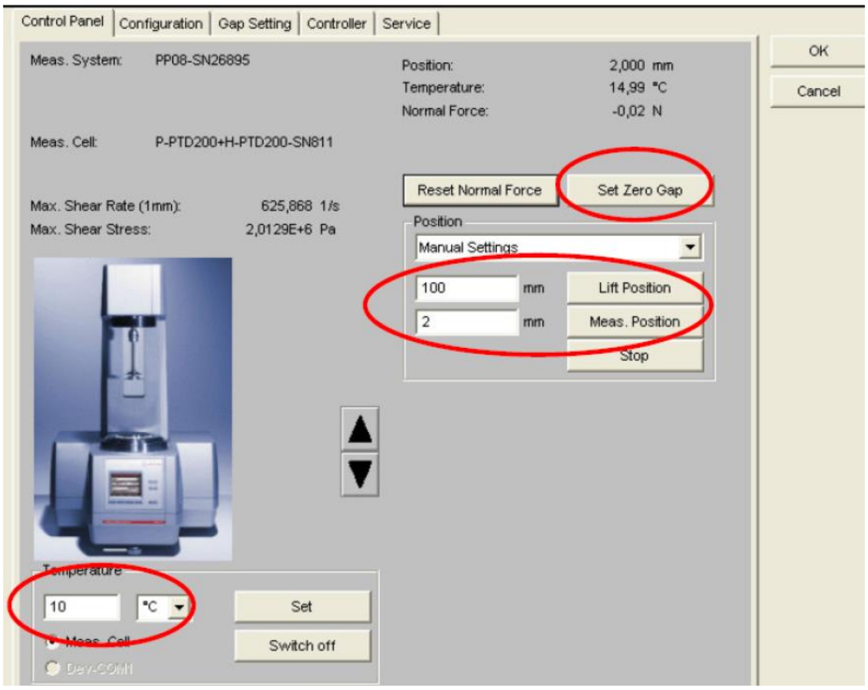

Meas. System: PP08-SN26895
Meas. Cell: P-PTD200+H-PTD200-SN811
Max. Shear Rate (1mm): 625,868 1/s
Max. Shear Stress: 2,0129E+6 Pa
Position: 2,000 mm
Temperature: 14,99 °C
Normal Force: -0,02 N
Position: Manual Settings
100 mm
2 mm
Temperature: 10 °C
● Meas. Cell
○ Dev-COM1

*Figure 5.7 -Set up of temperature and Zero Gap DSR*

Normally the specimen is place in the lower plate, but for this test the specimen will be place in the upper plate for this procedure the temperature is increase to 90°C for modified bitumen. For putting the specimen in the upper place, the option Lift position is used; once the specimen is placed with the command Measurement Position the 2mm gap is reached, during this point the specimen is trimmed in order to remove the excess of the material and avoid that this excess will react during the test. (Fig. 5.8). Subsequently the temperature is set again to the original 10°C which is the one select to realize the test.

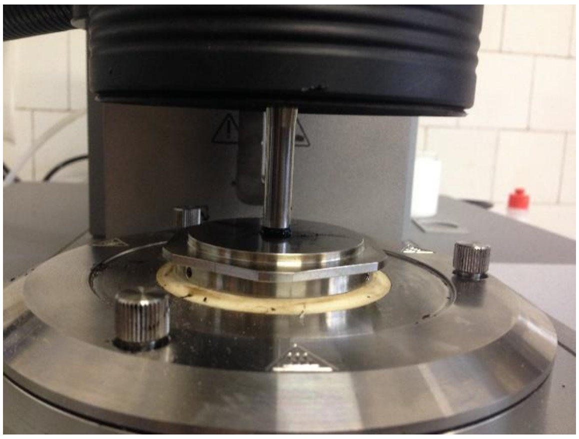
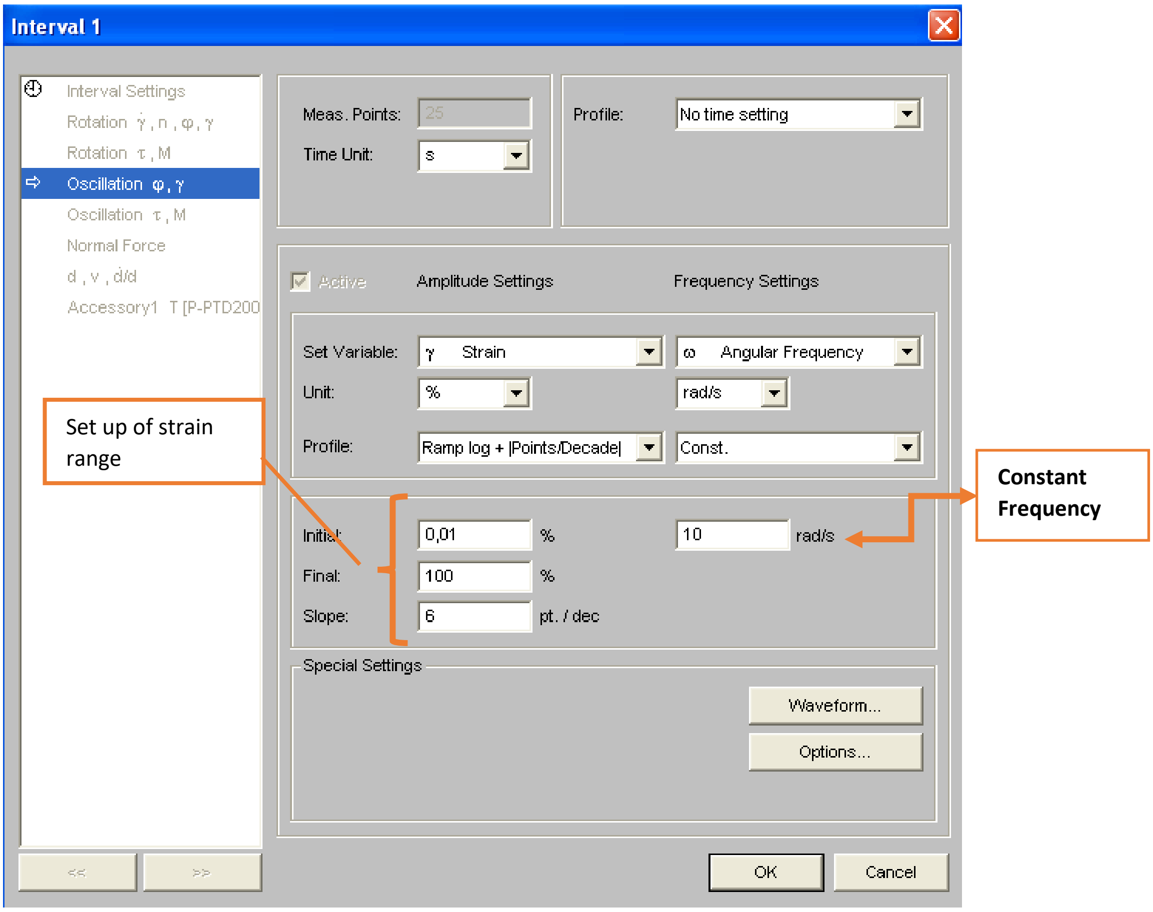


*Figure 5.8 -Example of trimmed specimen*

Once the specimen is ready the range for the strain is introduce 0.01% as initial strain and 100% as final strain until the specimen is damaged, since it is a strain-controlled test the shear stress will increase gradually, and the frequency will be constant at 10 rad/s (1.59Hz); plus six values will be taken as measurement points of the complex modulus for each unit in logarithmic deformation.



Interval 1

Meas. Points: 25

Time Unit: s

Profile: No time setting

Amplitude Settings

Frequency Settings

Set Variable: γ Strain | ω Angular Frequency

Unit: % | rad/s

Profile: Ramp log + |Points/Decade| | Const.

Initial: 0,01 % | 10 rad/s

Final: 100 %

Slope: 6 pt. / dec

Set up of strain range

Constant Frequency

Figure 5.9 -Set up of the strain for Amplitude Sweep

Finally, a diagram with complex modulus G* and the strain is generated. Fig 8 illustrate the G* plotted for the the short term aged sample (PMB 45/80-70 Hard+ Recycled Tires Powder.)

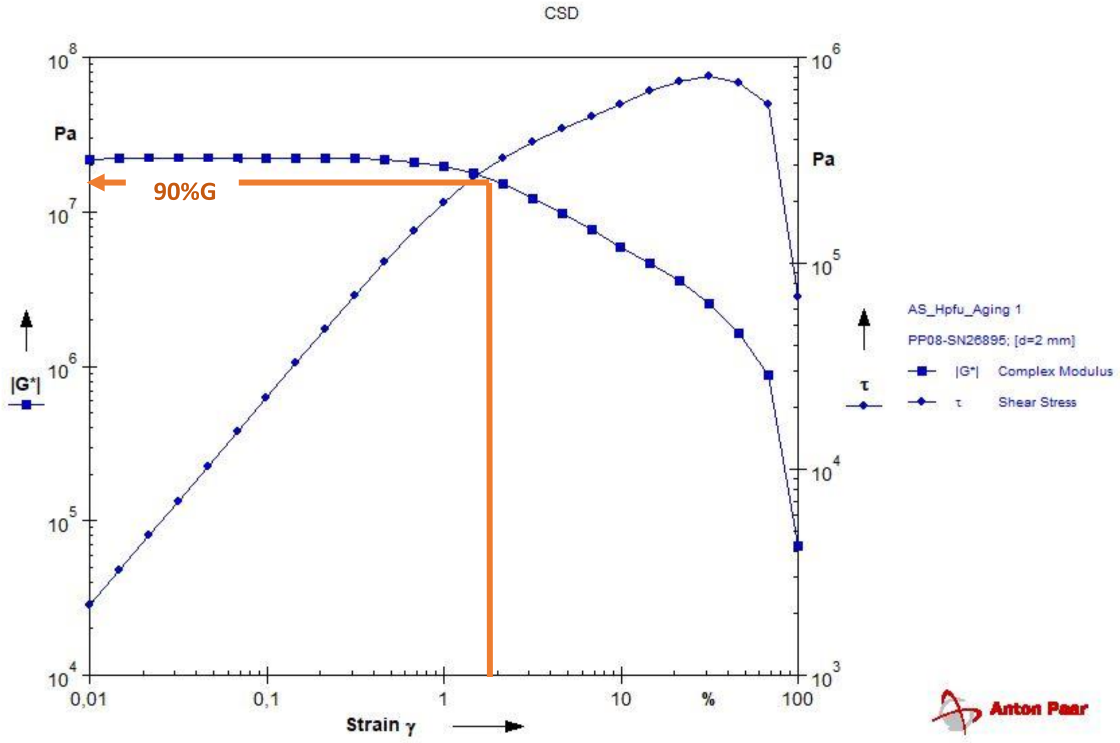


*Figure 5.10 -DSR Diagram Amplitude Sweep Test for the short term aged sample (Hard_pfu.)*

For this test the value that correspond to the limit of the LVE range (90% of the initial G*) will be determined as the 90% of the average of the firsts values of the complex modulus G*. In the table below the $\gamma_{LVE}$ values obtained are shown for each specimen.

| Item | Specimen type | $\gamma_{LVE}$ % |
|---|---|---|
| **1.** | VR | 1.80 |
| **2.** | Hard_pfu | 2.00 |
| **3.** | Hard | 2.50 |
| **4.** | Soft_pfu | 2.30 |
| **5.** | Soft | 3.00 |

*Table 5.2 -Results of γLVE from Amplitude Sweep on tested materials. (Unaged Bitumen).*

| Item | Specimen type | $\gamma_{LVE}$ % |
|---|---|---|
| **1.** | VR | 1,25 |
| **2.** | Hard_pfu | 0.90 |
| **3.** | Hard | 2.30 |
| **4.** | Soft_pfu | 1.65 |
| **5.** | Soft | 2.20 |

*Table 5.3 -Results of γLVE from Amplitude Sweep on tested materials. (Short Term Aging).*

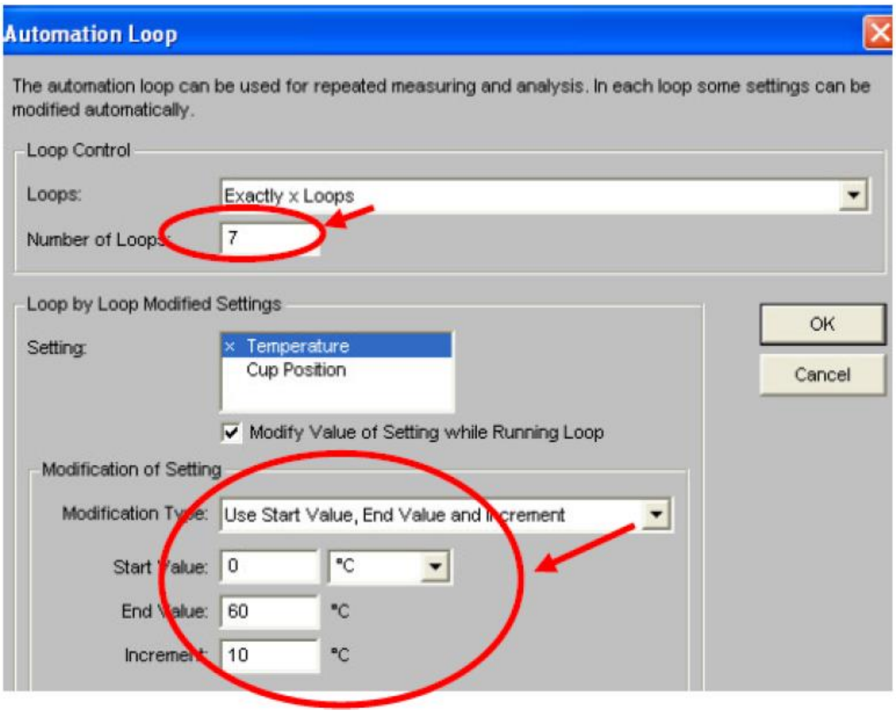
| Item | Specimen type | $\gamma_{LVE}$ % |
|---|---|---|
| **1.** | VR | 1.30 |
| **2.** | Hard_pfu | 1.20 |
| **3.** | Hard | 2.60 |
| **4.** | Soft_pfu | 1.85 |
| **5.** | Soft | 2.50 |

*Table 5.4 -Results of γLVE from Amplitude Sweep on tested materials. (Long Term Aging).*

### 5.6.4. Frequency Sweep Test

Since the value at which the material behave within the LVE range is obtained by the amplitude sweep test, a frequency sweep test is subsequently perform to our five samples, with this test is possible to obtain the master-curves of the tested material using the principle of Time-Temperature Superposition (TTS).

Like the amplitude sweep is necessary to set the zero gap and place the specimen on the upper plate and be trimmed in the DSR. An 8mm plate with a 2 mm gap is selected as a measurement system. The test is made under strain-controlled conditions, the temperature were set from 0°C, 10°C, 20°C, 30°C, 40°C, 50°C and 60°C this means that 7 loops are necessary for the test with an increase of 10°C between them.



The automation loop can be used for repeated measuring and analysis. In each loop some settings can be modified automatically.

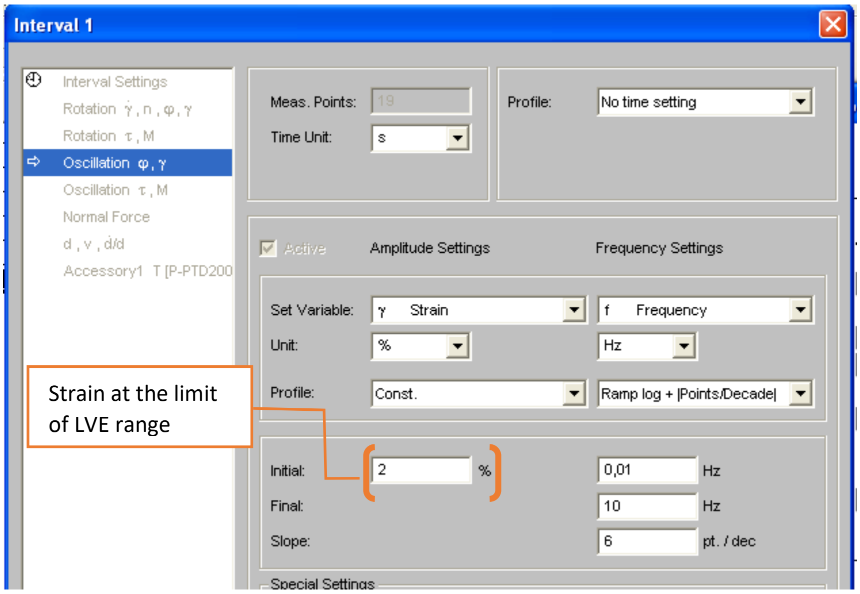
Loop Control
Loops: Exactly x Loops
Number of Loops: 7

Loop by Loop Modified Settings
Setting: × Temperature
Cup Position
☑ Modify Value of Setting while Running Loop

Modification of Setting
Modification Type: Use Start Value, End Value and Increment
Start Value: 0 °C
End Value: 60 °C
Increment: 10 °C

*Figure 5.11 -Setting up Temperature DSR*

The G* and the δ are plotted in six points for each unit (6 points/decade), in total 19 points will be display. The test is made in a strain controlled, where the maximum amplitude is fixed to the limit of the linear viscoelastic range (LVE) determined by the amplitude sweep test. (Fig.10)



Meas. Points: 19
Time Unit: s
Profile: No time setting
☑ Active

| | Amplitude Settings | Frequency Settings |
|---|---|---|
| Set Variable: | γ Strain | f Frequency |
| Unit: | % | Hz |
| Profile: | Const. | Ramp log + \|Points/Decade\| |
| Initial: | 2 % | 0,01 Hz |
| Final: | | 10 Hz |
| Slope: | | 6 pt. / dec |

Strain at the limit of LVE range

*Figure 5.12 -DSR screen setting up limit LVE range*

The final output from the DSR software is a diagram where the G* modulus and the phase angle δ is plotted for the 7 loops corresponding to each tested temperature.

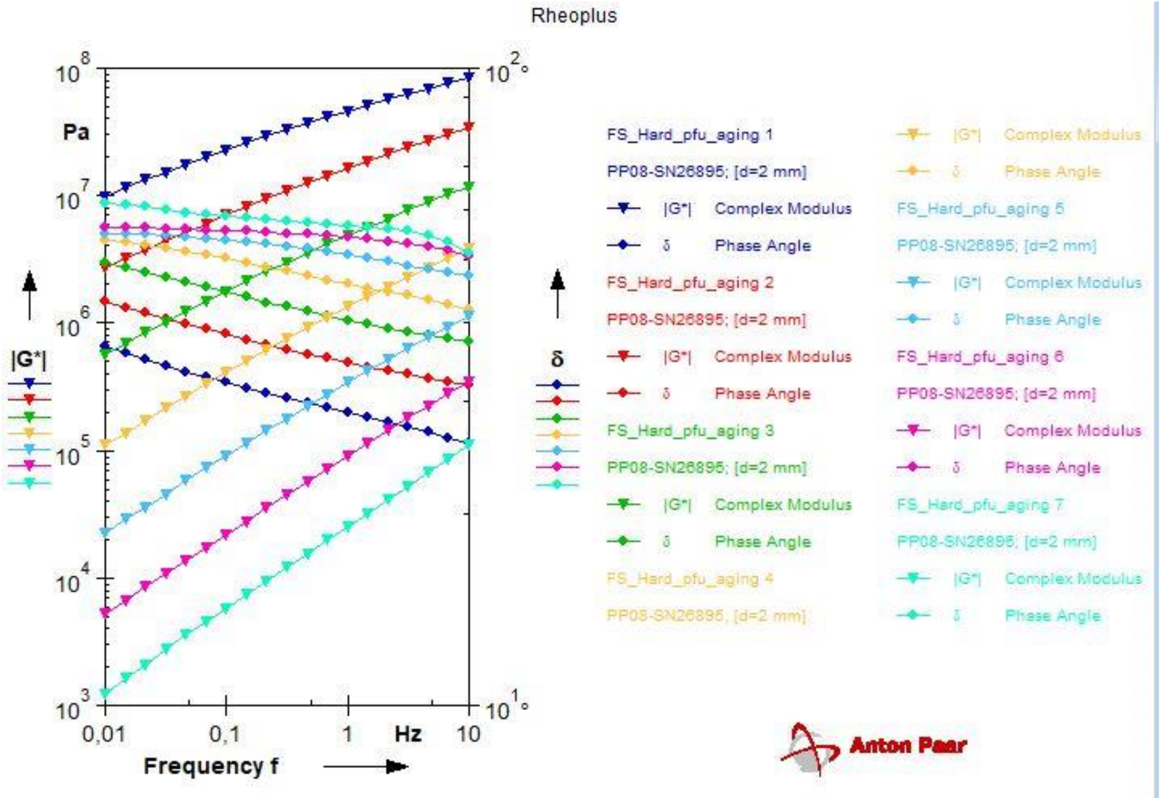


*Figure 5.13 -DSR software output Frequency Sweep Test for the short term aged (Hard_pfu).*

The rheological parameter obtained from the frequency sweep test (G*, δ, G’, G”) it is possible to generate the master curves by imposing the Time Temperature Superposition (TTS) principle.

Thanks to the master curves it can be studied the performance of each of the five samples in terms of rheological parameter (G*, δ, G’, G”) and frequency. The construction of the master curve was achieved by transferring the obtained data at the different temperatures horizontally by using the shift factor $a_T$.

In order to find the shift factor $a_T$, for each test temperature (0°C-60°C) is plot the trend of the complex modulus G* at the different frequency range (for this research 0.01-10Hz) (Fig.12)

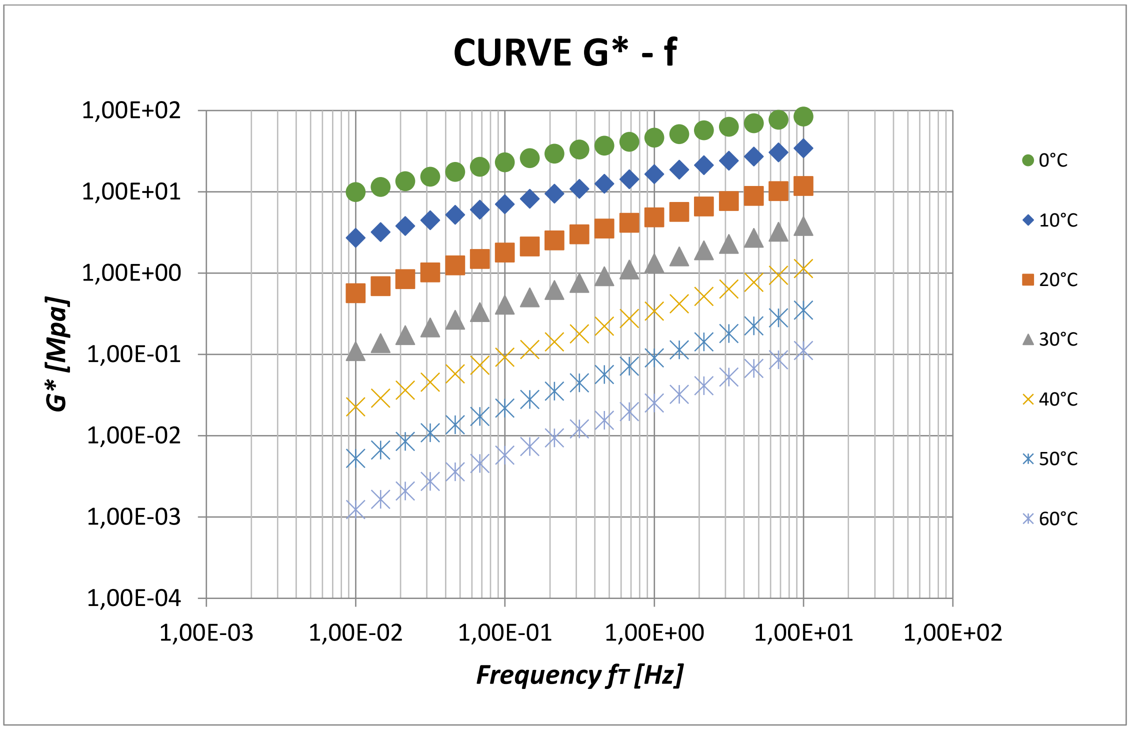


*Figure 5.14 -G*-f Curve for the short term aged sample (Hard_pfu)*

Subsequently the trend of each curve is compared to a general trend *"$y=ax^b$"* and the parameter "a" and "b" is defined for each curve. Later the generic equation *"$y=ax^b$"* is inverse at this point the value of *"x"* is obtained and explaining the generic "*y*" set at a minimum complex modulus capture by the DSR. Finally, the shift factors are calculated for each curve.

After the reduced frequencies are obtained by multiplied the normal frequencies and the shifts factors, afterwards the curves are moved according to its shift factor and finally the master curve is obtained. (Fig.13)

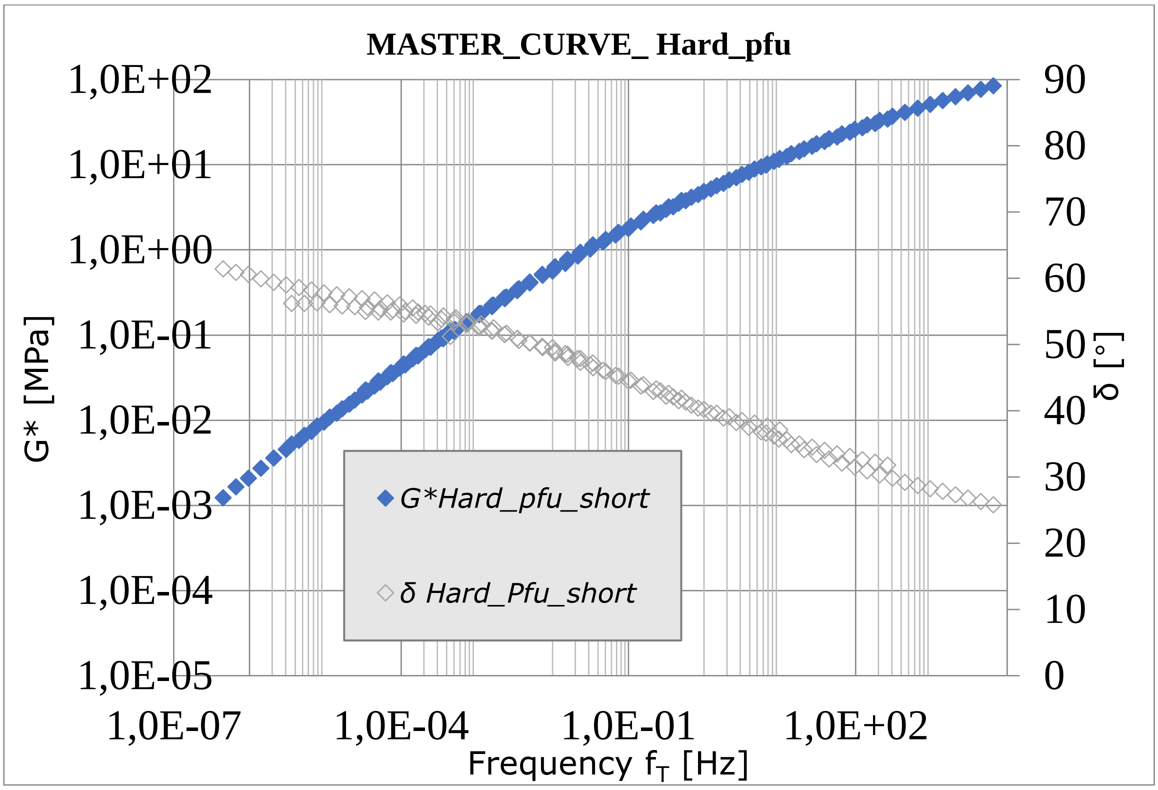


*Figure 5.15 -Master Curve for the short term aged sample (Hard_pfu.)*

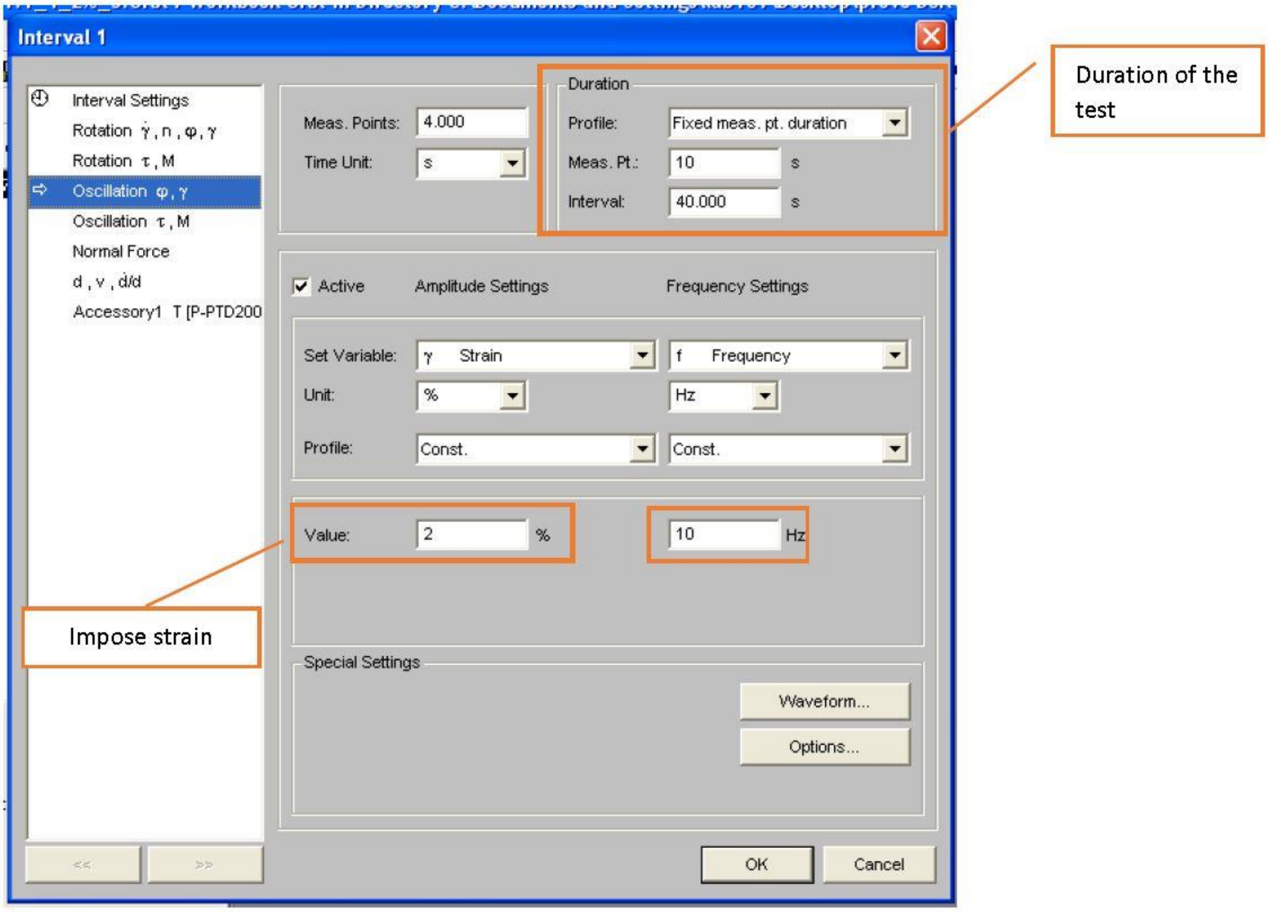
### 5.6.5. Fatigue Test

The test is performed at 25°C with an 8 mm plate 2 mm gap in strain controlled with a frequency of 10 Hz; the level of strain choose for this test is 4%, 3%, 2%.

| **Sample** | **Short Aged** | | | **Long Aged** | | |
|---|---|---|---|---|---|---|
| | Strain Level % | | | Strain Level % | | |
| | 4% | 3% | 2% | 4% | 3% | 2% |
| **VR** | X | X | X | X | | |
| **Hard_pfu** | X | X | X | X | | |
| **Hard** | X | X | X | X | | |
| **Soft_Pfu** | X | X | X | X | | |
| **Soft_Pfu** | X | X | X | X | | |

*Table 5.5 -Experimental plan for Fatigue Test*

Once the parameters are chosen, the test is carry out through the DSR instrumentation by first setting the zero gap, next the temperature is increase to 100°C and the specimen is place in on the upper plate, once the specimen is placed the trimming of the sample will take place. Then, the temperature was maintained at 25 °C for all tests. Afterwards the level of strain is determined in the DSR software. The test is an oscillatory with constant frequency (10 Hz).



Meas. Points: 4.000

Time Unit: s

Duration

Profile: Fixed meas. pt. duration

Meas. Pt.: 10 s

Interval: 40.000 s

☑ Active

| | Amplitude Settings | Frequency Settings |
|---|---|---|
| Set Variable: | γ Strain | f Frequency |
| Unit: | % | Hz |
| Profile: | Const. | Const. |
| Value: | 2 % | 10 Hz |

Duration of the test

Impose strain

Figure 5.16 -DSR screen setting up of Fatigue test.

From this test we can obtain the the complex modulus G*, the phase angle δ, the angular frequency ω, the fatigue parameter G*sinδ and the dissipated energy during the test.

The 50% drop in G* value and the DER from sweep-test using the dynamic shear rheometer (DSR) have been used to determine the number of cycles to failure. The ways in which failure is described are:

1. The number of cycles it takes to cause the initial G* value to drop 50%, and

2. The number of cycles before the DER diverges from a linear trend.

### Fatigue Criteria 1: A 50% Drop in Modulus Method

This method requires the simple calculation which involves the initial G* value (Gi* ) and the G* value at the nth cycle (Gn*), as shown in the following equation:

$$\% \, drop = \frac{(G_i^* - G_n^*)}{G_i^*} * 100$$

When the percent drop has reached 50%, the sample is considered to have failed.

### Fatigue Criteria 2: The Dissipated Energy Ratio Method

This method calculates the amount of energy dissipated per cycle. The cumulative dissipated energy (WΣn) over the dissipated energy at each cycle (Wi) is called the dissipated energy ratio (DER). Where εi is strain, δi is phase angle in radians, and σi is stress, the dissipated energy at a given cycle is as shown in the following equation:

$$W_i = \pi * \varepsilon_i * \sin(\delta_i) * \sigma_i$$

Cumulative energy and DER are calculated using the following equations:

$$W_{\Sigma_n} = \sum_{i=1}^{n} W_i$$

$$Dissipated\ Energy\ Ratio = \frac{W_{\Sigma_n}}{W_i}$$

This value, when plotted against the number of cycles produces a graph which moves linearly upward, then undergoes a nonlinear region which moves into a second linear region, more steep than the first.

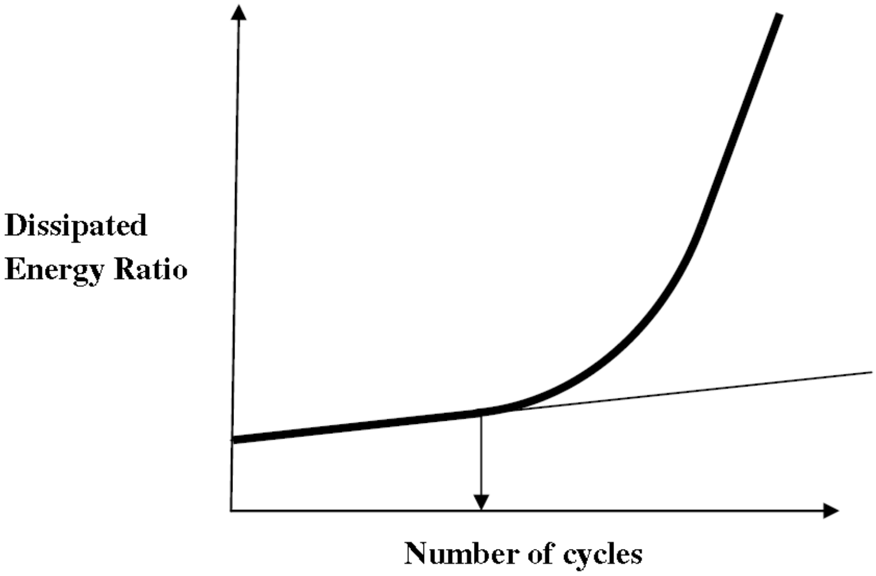


*Figure 5.17 -Schematic of Dissipated Energy Ratio versus Number of Cycles*

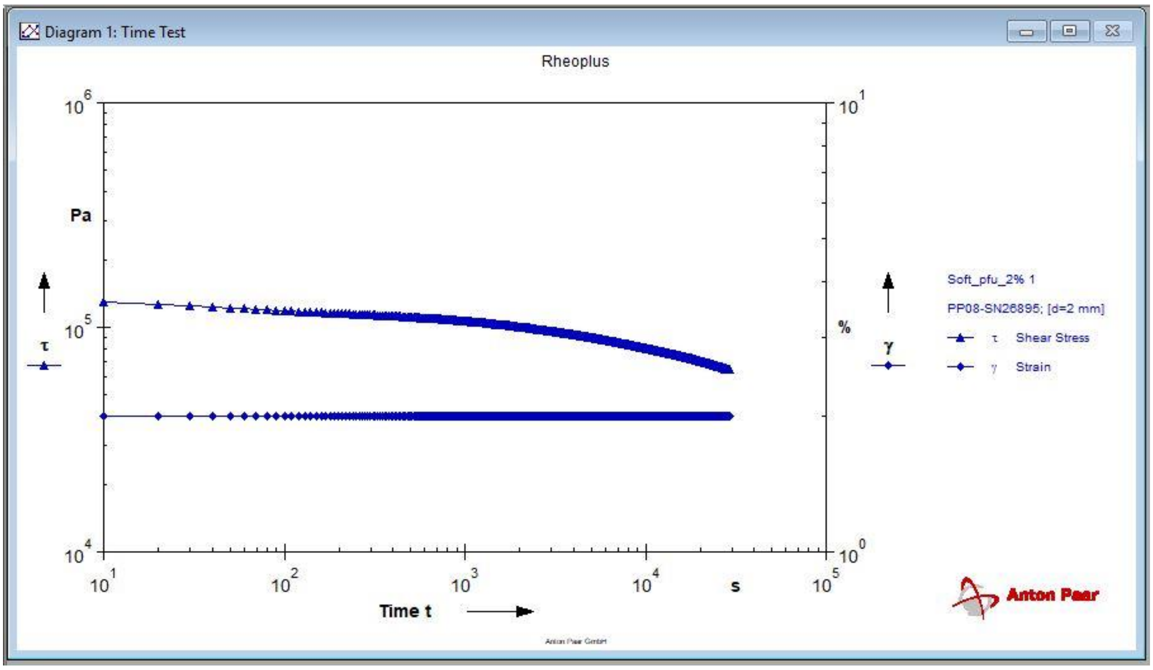


*Figure 5.18 -Graph from fatigue test Soft_pfu at 2% strain DSR software.*

### 5.6.6. Time Sweep Test for Fatigue and Healing.

Healing is defined as the extension in fatigue life resulting from rest, which includes contributions from viscoelasticity, thixotropy, and crack closure [31].

For the tested five bitumen, the healing can be defined as the material recovery due to the introduction of the rest period plus the increasing in the fatigue life. There are several interpretations for the introduction of rest period while some researcher introduces short loading time alongside with short rest periods and other introduce long loading time with a single rest period or very few of them.

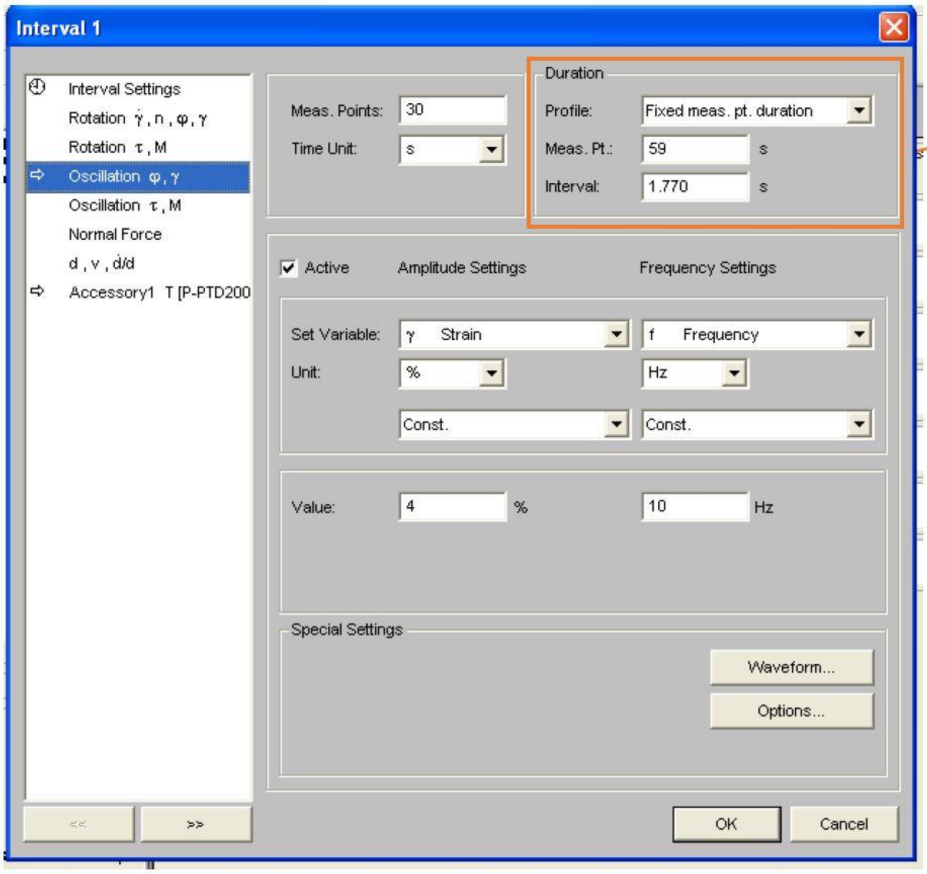
For the evaluation of healing on this research a strain controlled time sweep test is carried out with the introduction of a single rest period of one hour (1h). The measurement system selected is an 8mm plate diameter with 2 mm gap. The temperature was maintained at 25 °C for all tests. The time sweep test method uses the DSR to apply repeated cyclic loading at a fixed load amplitude to a specimen.

For introducing the rest period that allow the healing of the tested samples,it will be fixed at the number of cycles where the Complex Modulus G* reach a reduction of the 35% of the initial value. This value is obtained with the previous fatigue test without a rest period from section.

The time sweep test is carried out in the same strain level as previous fatigue test 4%, 3% and 2% and with a constant frequency of 10Hz.

| **Sample** | **Short Aged** | | | **Long Aged** | | |
|---|---|---|---|---|---|---|
| | Strain Level % | | | Strain Level % | | |
| | 4% | 3% | 2% | 4% | 3% | 2% |
| **VR** | X | X | X | X | | |
| **Hard_pfu** | X | X | X | X | | |
| **Hard** | X | X | X | X | | |
| **Soft_Pfu** | X | X | X | X | | |
| **Soft_Pfu** | X | X | X | X | | |

*Table 5.6 -Experimental plan for Time Sweep Test.*



Meas. Points: 30
Time Unit: s

Duration
Profile: Fixed meas. pt. duration
Meas. Pt.: 59 s
Interval: 1.770 s

☑ Active
Amplitude Settings
Set Variable: γ Strain
Unit: %
Const.
Value: 4 %

Frequency Settings
f Frequency
Hz
Const.
10 Hz

Special Settings

Set up of the first interval where the rest period will be introduce

*Figure 5.19 -DSR screen for time sweep test.*

### 5.6.7. Time Sweep Test with Fixed Initial Complex Modulus

In order to make a comparison of the material recovery and the fatigue life between the five short aged bitumen when we have the same initial stiffness G*, time sweep test were done at different temperatures and at one stain level (4%).

An initial complex modulus ranges between (6.13-6.86) MPa, temperatures adopted for measurements are reported in Table 5.7.

| Sample (Short Aged) | G* Initial - MPa | Test Temperature | Starin Level % |
|---|---|---|---|
| **VR** | 6,39 | 25°C | 4% |
| **Hard_pfu** | 6,80 | 24°C | 4% |
| **Hard** | 6,13 | 20°C | 4% |
| **Soft_pfu** | 6,33 | 24°C | 4% |
| **Soft** | 6,76 | 22°C | 4% |

*Table 5.7 -Temperatures adopted for Time Sweep Test with fixed initial G*.*

# CHAPTER 6:

# Results Analysis.

# 6. CHAPTER 06: Results Analysis.

## 6.1. Amplitude Sweep Results

The purpose for this test is to establish the deformation ($\gamma_{LVE}$) where the specimen is kept in LVE range. Table 6.1 shows the deformation $\gamma_{LVE}$ defined at the strain where the complex modulus G* is 90% of the initial value.

| Specimen type | $\gamma_{LVE}$ % | | |
|---|---|---|---|
| | Unaged | Short aged | Long aged |
| VR | 1.80 | 1,25 | 1.30 |
| Hard_pfu | 2.00 | 0.90 | 1.20 |
| Hard | 2.50 | 2.30 | 2.60 |
| Soft_pfu | 2.30 | 1.65 | 1.85 |
| Soft | 3.00 | 2.20 | 2.50 |

*Table 6.1 -Results of γLVE from Amplitude Sweep on tested materials.*

The results shown in Table 6.2 shows that the linear visco-elasticity limit of the bitumen tested in AS configuration at 10 ° C decreases as the bitumen strength increases in the case of unaged bitumen When moving from unaged to short aged γLVE % decreases, while when moving from short to long aged γLVE % increases. In particular, it can be observed that the presence of the rubber powder increases the bitumen stiffness and consequently reduces the linear viscoleasticity range.

## 6.2. Frequency Sweep Test Results.

### 6.2.1. Master Curve

Through the rheological parameters obtained as output of the tests performed with the DSR, the curves for each bitumen were generated. In particular, thanks to the application of the time-temperature superposition principle (TTS), it was possible to study the trend of the complex modulus | G * | and phase angle δ as a function of frequency. The curves were carried out by translating the data obtained from tests at different temperatures, using the shift factor previously described in section 5.6.4.

#### 6.2.1.1. Study and comparison of the bitumen master-curves before and after aging.

Fig. 6.1 shows the master curves for VR bitumen before aging, short and long aged. At low frequencies, high temperatures, the long aged bitumen reaches values of complex modulus greater than the short aged and unaged sample. For the phase angle δ at high temperature is almost 60° for the long aged and that indicates less elastic behavior (more viscous behavior) for the long aged with respect to short aged and the unaged (50° and 40° respectively), at the other part for low temperature the δ equals to 25° for both long aged and short aged, whereas δ equals to 30 for the unaged. In-between at intermediate temperatures δ changes to be around 60° for the unaged, 50° for the short aged and 40° for the long aged (the long aged has more elastic behavior at intermediate temperatures). It can be observed also that at high and intermediate temperatures G* increases when moving from the unaged to short to long aged. The phase angle δ at high temperature 60°C increase when we move from unaged to the long term aged, which means that the bitumen becomes less elastic.

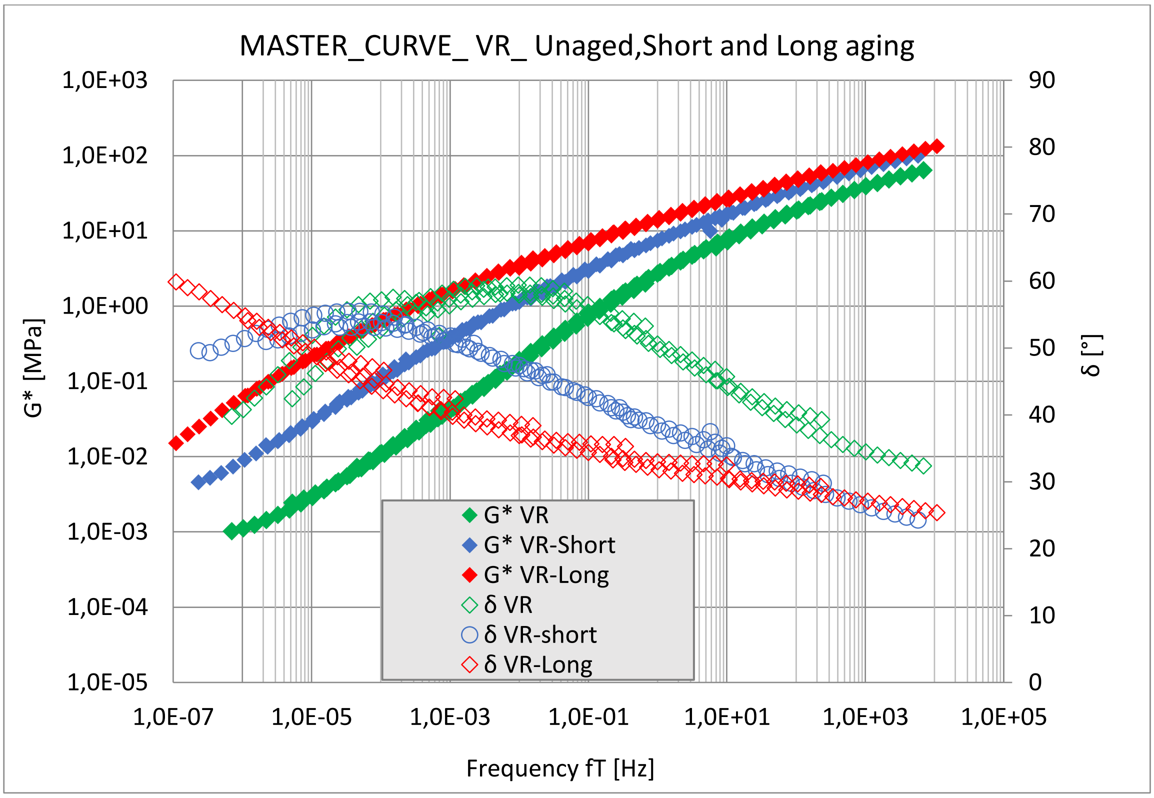


*Figure 6.1 -Master Curve VR (Unaged, Short and long aged).*

Fig. 6.2 shows the master curves for Hard_pfu bitumen before aging, short and long aged. At low frequencies, high temperatures, the long aged bitumen reaches values of complex modulus greater than the short aged and unaged sample. For the phase angle δ at high temperature is almost 55° for the long aged and 60° for the short aged and that indicates less elastic behavior (more viscous behavior) for the short aged with respect to long aged, the unaged at high temperature has a phase angle δ similar to the long aged. At the other part for low temperature the δ equals to 25° for both short and long aged, whereas δ equals to 35 for the unaged. In-between at intermediate temperatures δ changes to be around 60° for the unaged, 50° for the short aged and 40° for the long aged (the long aged has more elastic behavior at intermediate temperatures). It can be observed also that at high and intermediate temperatures G* increases when moving from the unaged to short to long aged. The phase angle δ at high temperature 60°C increase when we move from unaged to the short aged, but it is the lowest for long aged.

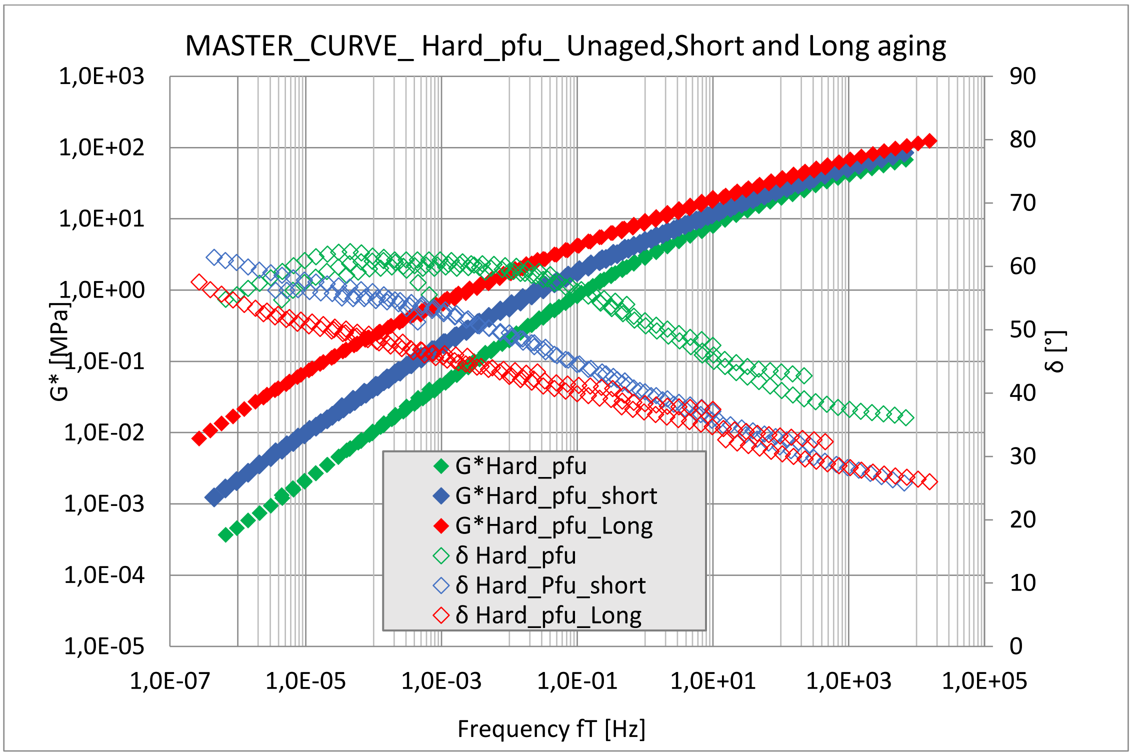


*Figure 6.2 - Master Curve Hard_pfu (Unaged, Short and long aged)*

Fig. 6.3 shows the master curve for Hard bitumen, it can be observed that there is no significant difference between the unaged and the aged bitumen, and it is almost the same G* at high temperatures between short and long aged; which means that ageing leads to less stiffening in the case of hard bitumen without rubber. The phase angle of the bitumen after aging has higher values at high temperatures especially for the long aged, and it is almost the same at low temperatures.

Fig. 6.4 shows the master curves for Soft_pfu bitumen before aging, short and long aged. At low frequencies, high temperatures, the long aged bitumen reaches values of complex modulus greater than the short aged and unaged sample. For the phase angle δ at high temperature is almost 70° for the long aged and 75° for the short aged and that indicates less elastic behavior (more viscous behavior) for the short aged with respect to long aged, the unaged at high temperature has a phase angle δ similar to the long aged. At the other part for low temperature the δ equals to 25° for both short and long aged, whereas δ equals to 35° for the unaged. In-between at intermediate temperatures δ changes to be around 70° for the unaged, 60° for the short aged and 50° for the long aged (the long aged has more elastic behavior at intermediate temperatures). It can be observed also that at high and intermediate temperatures G* increases when moving from the unaged to short to long aged. The phase angle δ at high temperature 60°C increase when we move from unaged to the short aged, but it is the lowest for long aged.

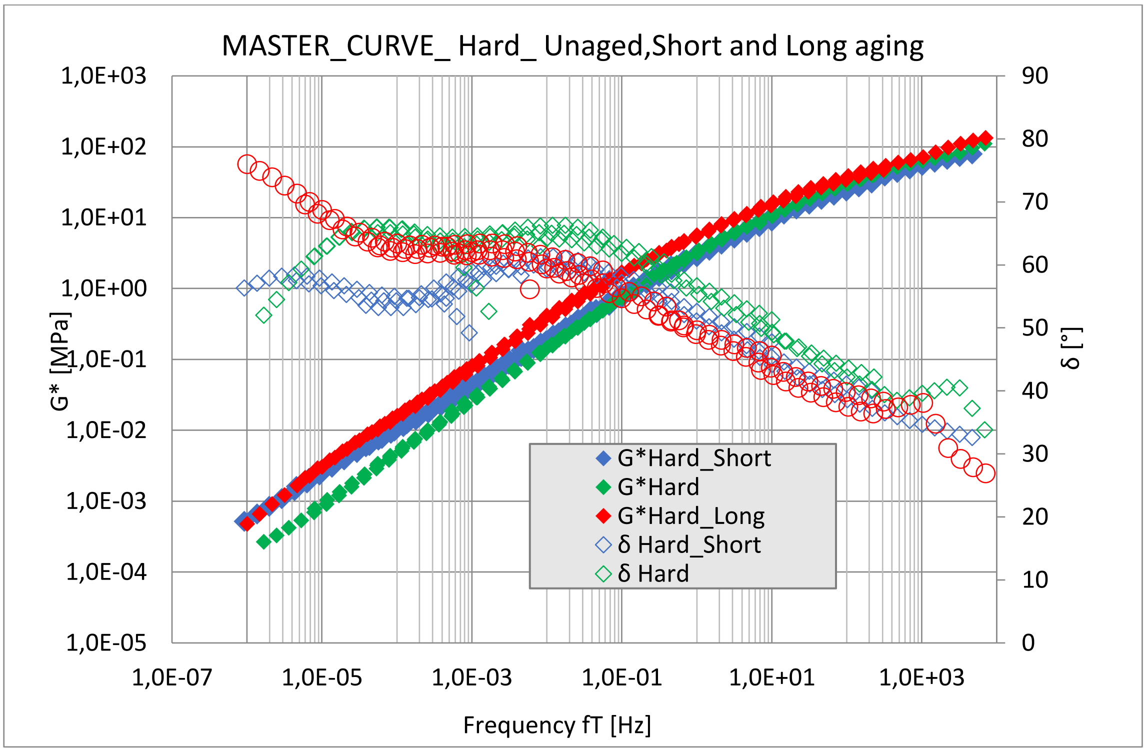


*Figure 6.3 -Master Curve Hard (Unaged, Short and long aged)*

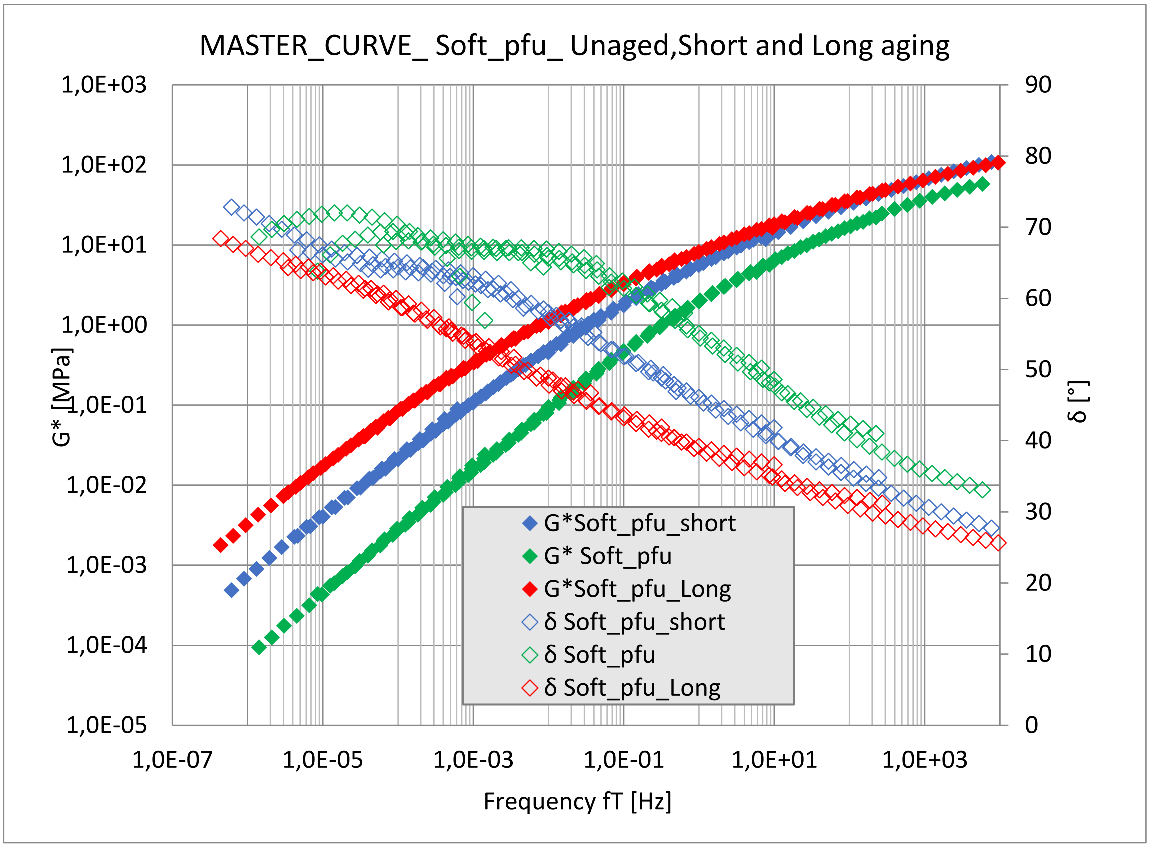


*Figure 6.4 -Master Curve Soft_pfu (Unaged, Short and long aged).*

Fig. 6.5 shows the master curve for Soft bitumen, it can be observed that the increasing of G* at high temperatures moving from unaged to aged bitumen is lower than the soft bitumen with rubber. which means that ageing leads to less stiffening in the case of Soft bitumen without rubber. The phase angle of the bitumen after aging has higher values at high temperatures especially for the short aged, and it is almost the same at low temperatures for short and long aged, while it is higher for the unaged.

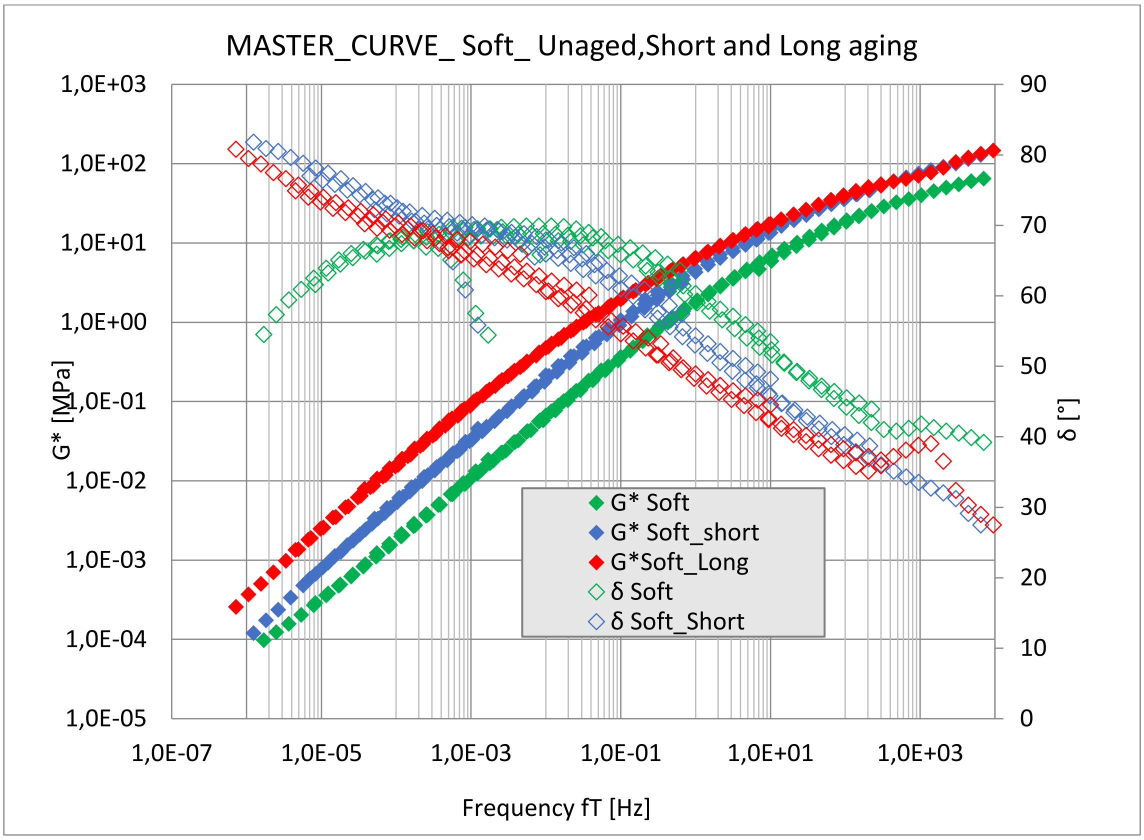


*Figure 6.5 -Master Curve Soft (Unaged, Short and long aged).*

### 6.2.1.2. Study and comparison of the master-curves according to the presence of the additives before aging.

The Master curves for the all unaged bitumen are shown in the following figure 6.6 in order to campare the rheological parameter for all of them. Bitumen with the highest complex module values at high temperatures is the bitumen called VR (hard bitumen contains rubber and visco-reducer) and Hard_pfu bitumen (hard bitumen contains rubber). These bitumen are the least thermosensitive among the five studied and, at increased stiffness at high temperatures, there is a significant reduction in the phase angle relative to the other bitumen. Especially for these two bitumen at high temperatures, the elastic response increases by detecting phase angle values around 45 ° (perfectly visco-elastic response). Hard bitumen, when compared Hard_pfu bitumen contains rubber powder, has lower G * complex module values and higher phase angles at high temperatures. It can be stated that Hard bitumen with the presence of rubber increases the elastic range of the bitumen by increasing its resistance to cutting efforts and consequently to the bulging phenomena. In the case of Soft bitumen there is no significant increasing in G* at high temperatures, and the presence of rubber powder decreases the elastic response at high temperatures.

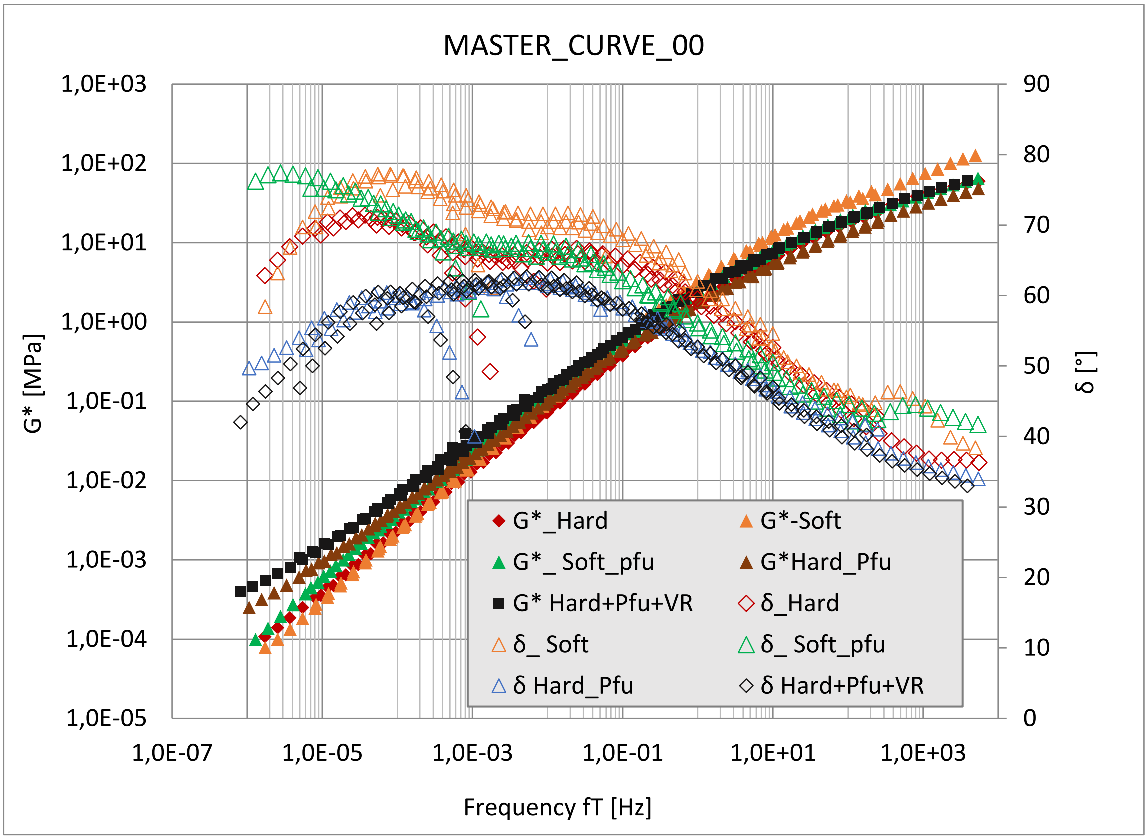


*Figure 6.6 -Master Curve (All tested bitumen - unaged).*

### 6.2.1.3. Study and comparison of the master-curves according to the presence of the additives after short aging.

The Master curves for the all short aged bitumen are shown in the following figure 6.7. Bitumen with the highest complex module values at high temperatures is VR and Hard_pfu. These bitumen are the least thermosensitive among the five studied and, at increased stiffness at high temperatures, there is a significant reduction in the phase angle relative to the Soft and Soft_pfu bitumen. Here it can be observed also that the Hard bitumen has almost the same elastic behavior as VR and Hard_pfu at high temperatures, but it becomes significantly less elastic at intermediate and low temperatures. Hard bitumen, when compared with Hard_pfu bitumen; has lower G * complex module values at high temperatures. It can be stated that Hard bitumen with the presence of rubber increases the elastic range of the bitumen by increasing its resistance to cutting efforts and consequently to the bulging phenomena. In case of Soft bitumen, the presence of rubber increases G* more significantly and increases the elastic response also.

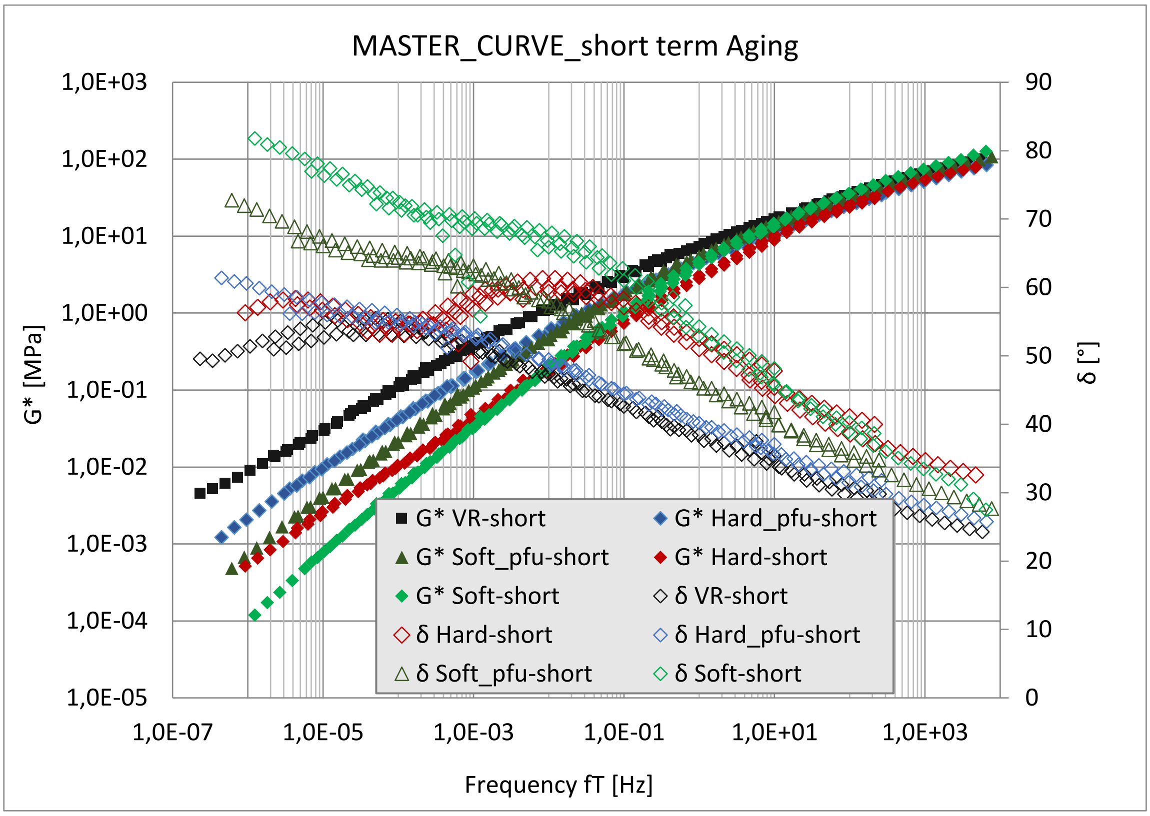


Figure 6.7 -*Master Curve (All tested bitumen – short term aged).*

### 6.2.1.4. Study and comparison of the master-curves according to the presence of the additives after long aging.

Master curves for the all long aged bitumen are shown in the following figure 6.8. Bitumen with the highest complex module values at high temperatures is VR and Hard_pfu. These bitumen are the least thermosensitive among the five studied and, at increased stiffness at high temperatures, there is a significant reduction in the phase angle relative to the hard, Soft and Soft_pfu bitumen. Here it can be observed also that the Hard bitumen less elastic behavior than VR and Hard_pfu at high temperatures and because the long aging effect the difference in G* between Hard and Hard_pfu becomes greater with respect to the short aged bitumen. There are no differences between the master curves of the Hard and Soft.It can be stated that Hard bitumen with the presence of rubber increases the elastic range of the bitumen by increasing its resistance to cutting efforts and consequently to the bulging phenomena. In case of soft bitumen the presence of rubber increases G* more significantly and increases the elastic response also. It shows how for the VR bitumen there is a discontinuous phase angle trend due to the presence of the visco-reducer.

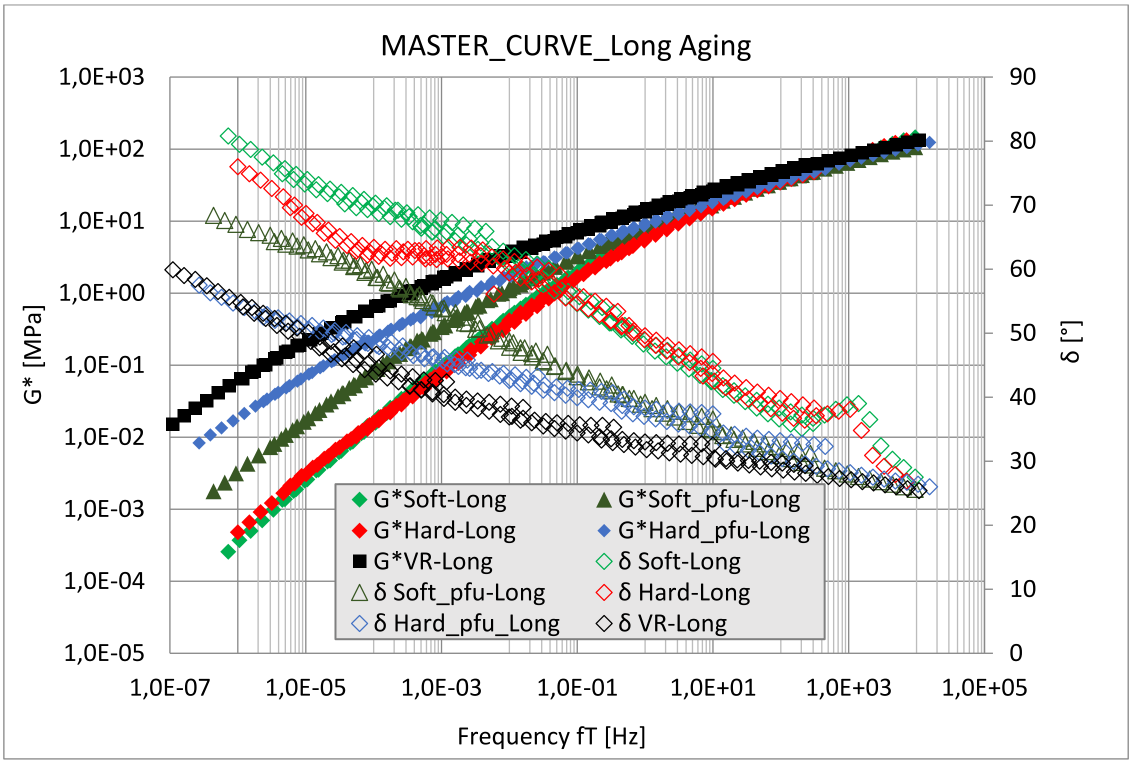


*Figure 6.8 -Master Curve (All tested bitumen – Long term aged).*

### 6.2.2. Cross-over temperature method.

In order to better show the binder rheological properties, the oscillatory tests were analyzed in terms of tan δ. In particular the cross-over temperature, defined as the temperature at which the value of tan δ is equal to 1, was determined for each binder [37]. When this temperature is reached, the elastic component (storage modulus G’) of the complex modulus is equal to the viscous component (loss modulus G’’), whereas for temperature values where tan δ is lower than 1, G’ is higher than G’’ and vice versa [37]. The test results (Fig. 6.9) shows that the presence of rubber increases the cross-over temperature values. (Fig. 6.10) confirms that the rubber has preserved the elastic component of the modified bitumen also after the short aging process. (Fig. 6.11) shows that SBS increases the cross-over temperature values after long aging process.

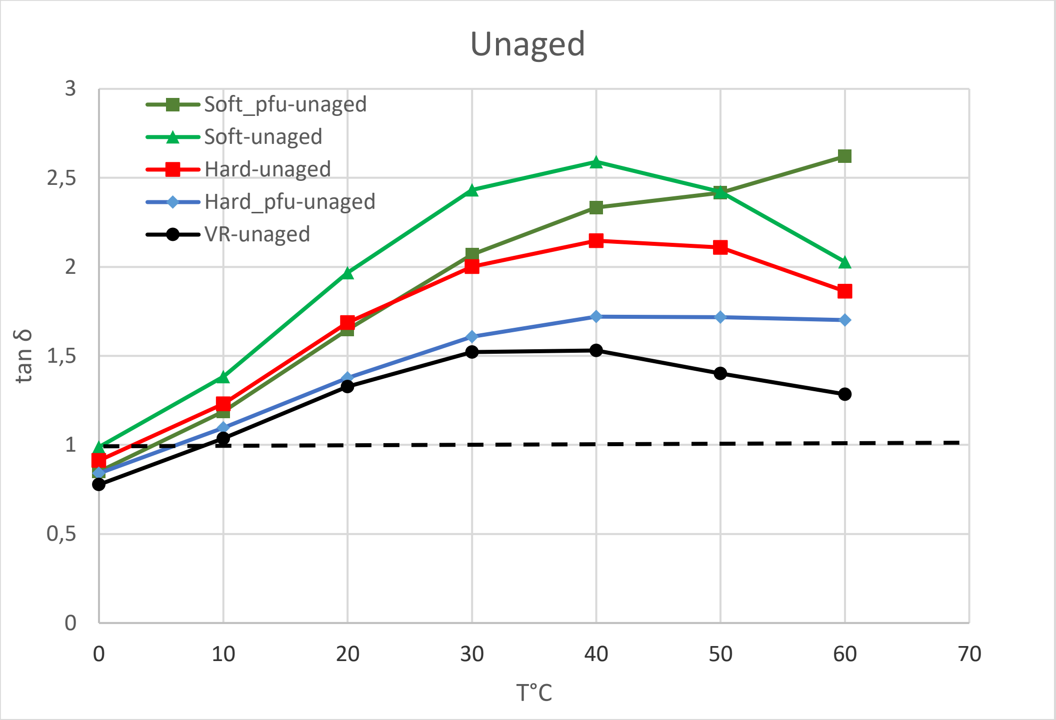


*Figure 6.9* - Tan $\delta$ vs temperature for Unaged Bitumen.

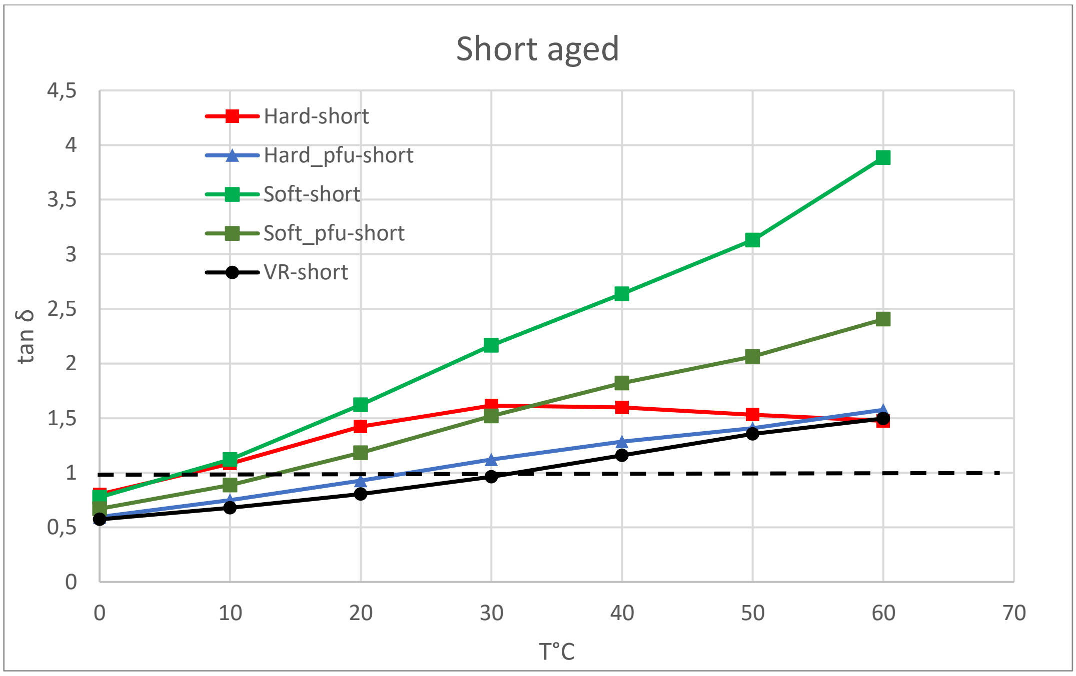


Figure 6.10 -*Tan δ vs temperature for short aged Bitumen.*

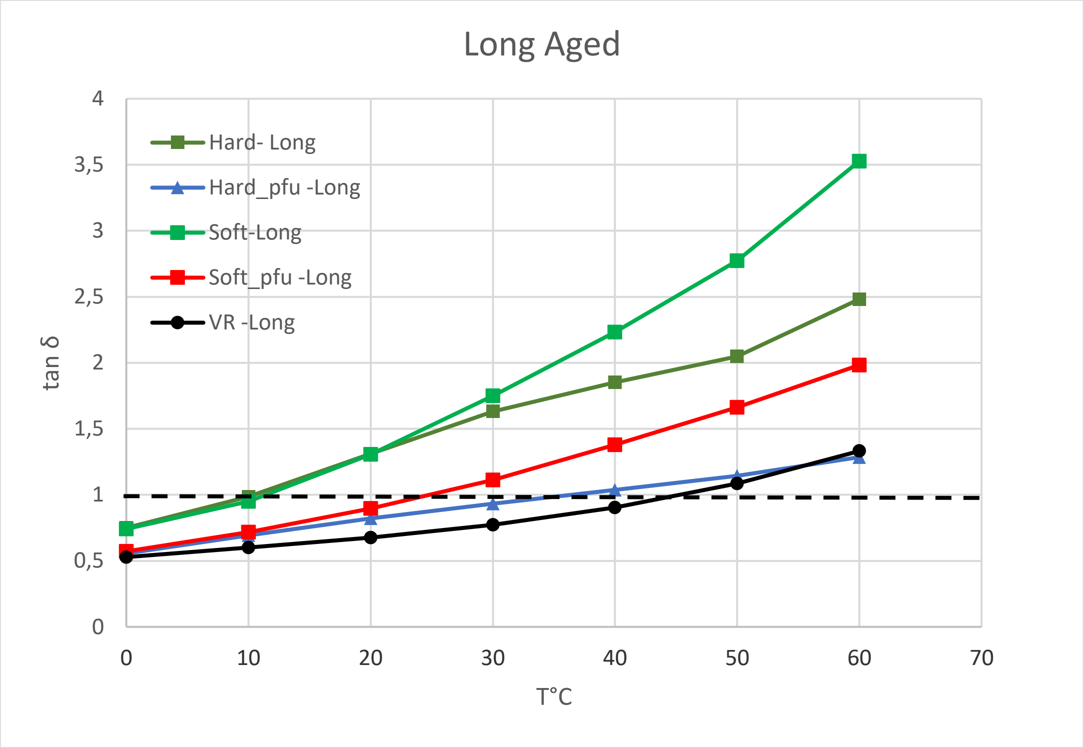


Figure 6.11 - *Tan δ vs temperature for Long aged Bitumen.*

### 6.2.3. Performance Grade

#### 6.2.3.1. Performance grade at 60°C (PG max) (Results from unaged bitumen).

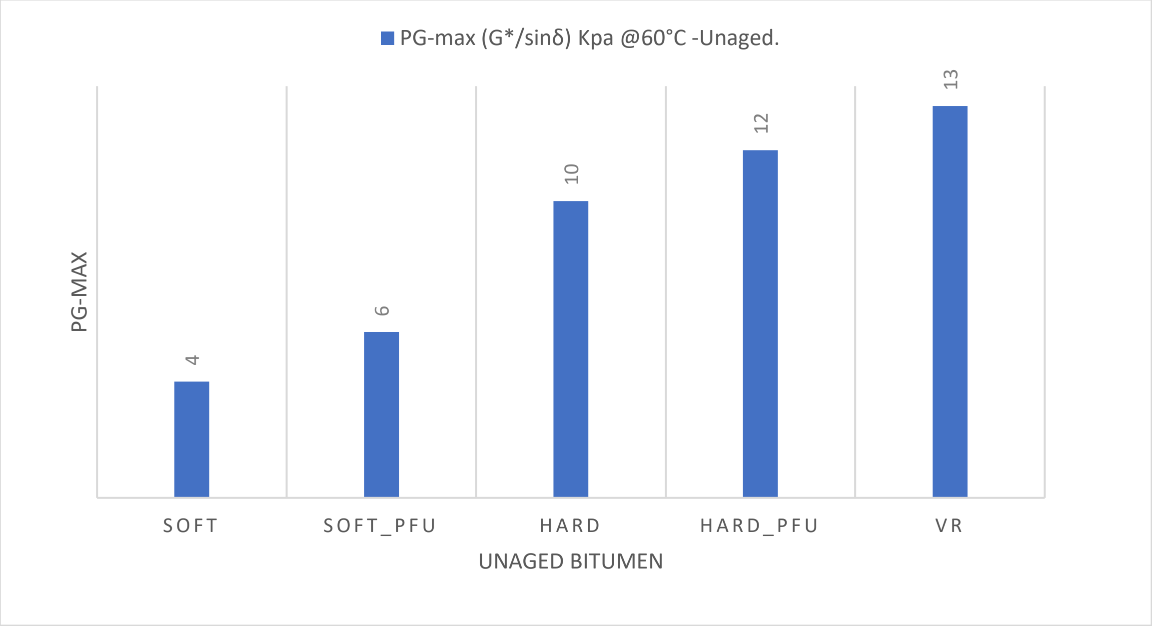


Figure 6.12 -*Performance grade of bitumen at 60° C – Unaged bitumen.*

#### 6.2.3.2. Performance grade at 60°C (PG max) (Results from short aged bitumen).

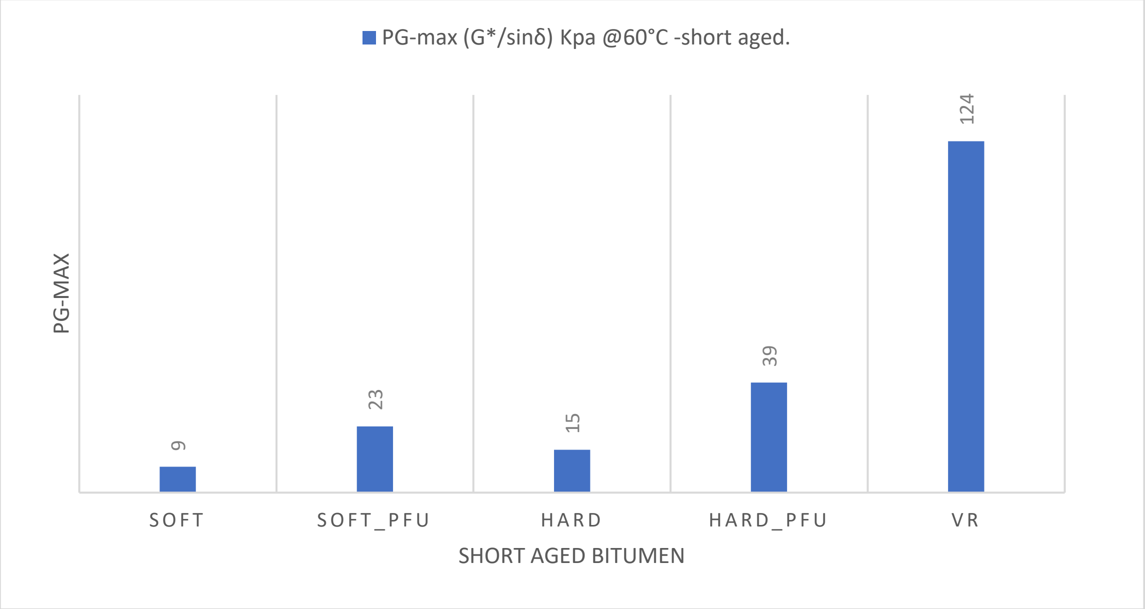


*Figure 6.13* -Performance grade of bitumen at 60° C – Short aged bitumen.

#### 6.2.3.3. Performance grade at 10 C ( PG min) (Results from long aged bitumen)

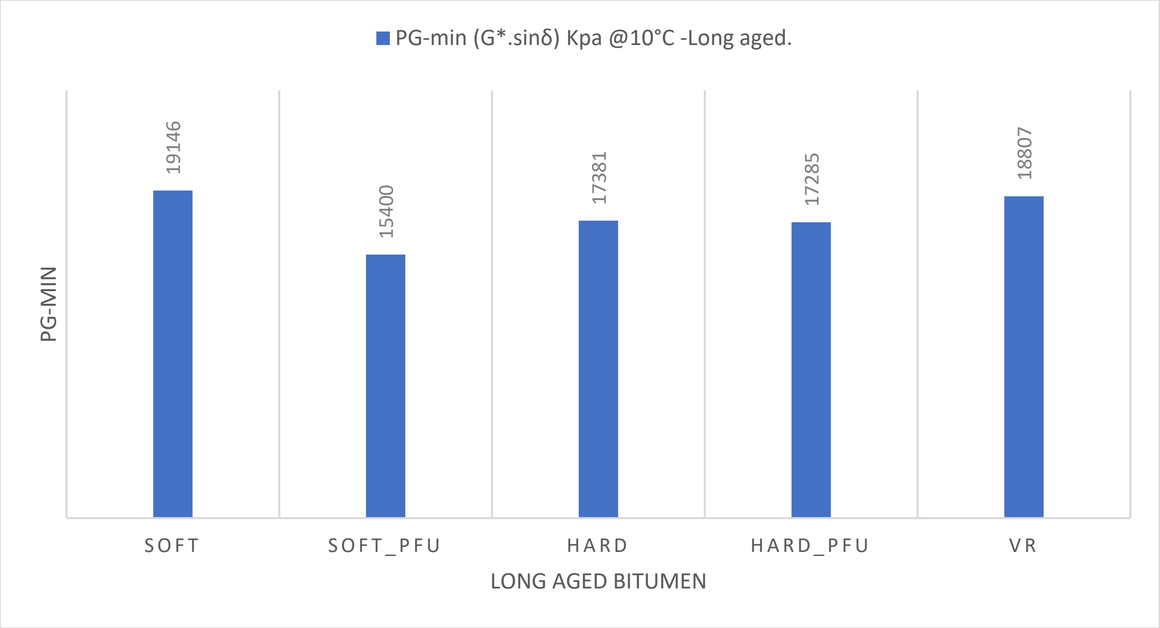


*Figure 6.14 -Performance grade of bitumen at 10° C – Long aged bitumen.*

## 6.3. Fatigue Test.

### 6.3.1. Fatigue Tests for Short Aged Bitumen

Firstly, the five short aged bitumen were tested without a rest period, at different strain levels 4%, 3% and 2%. From these tests it was obtained the Nf (number of cycles to failure) at each stain level for each bitumen except Hard bitumen which tested at strain levels 4%,5% and 6%, because the test takes a long time for it at low strain levels. Figure 6.14 shows the average initial complex modulus G* for each tested bitumen. It can be seen that the presence of the rubber gives higher initial stiffness G*, and the same consideration for the the viscous reducer.

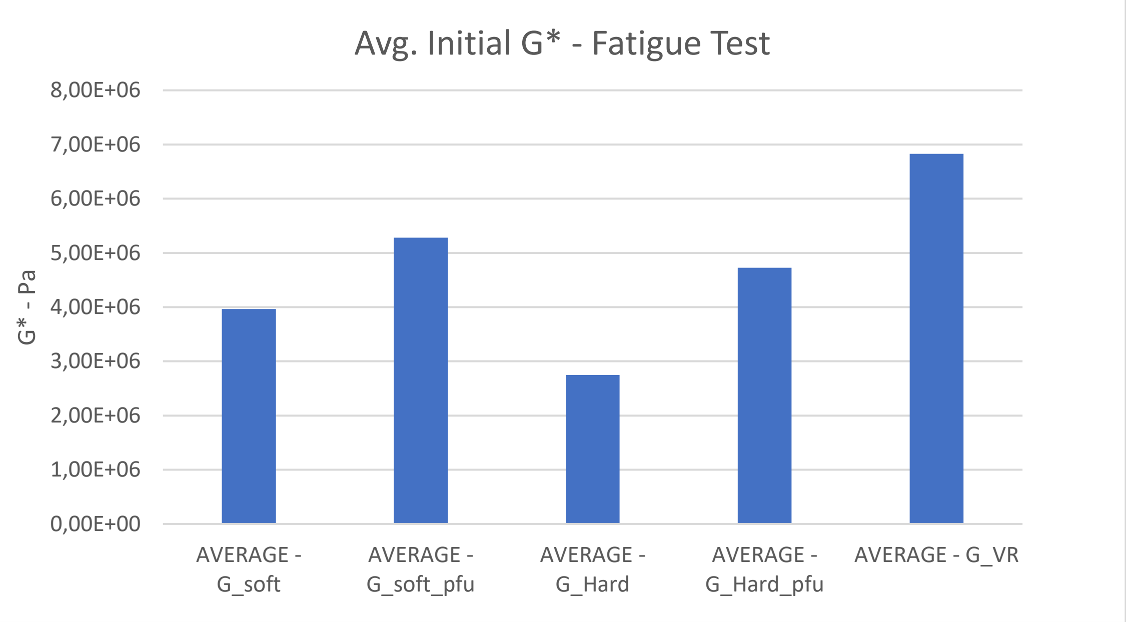


Figure 6.15 -*Average initial G* for each short aged bitumen.*

Then in order to obtain the same initial G*; the five short aged bitumen were tested at 4% strain level at different temperatures. Table 5.7 from the previous chapter shows that as the temperature decrases, the initial stiffness G* increases.

The 50% drop in G* value and the DER from sweep-test using the dynamic shear rheometer (DSR) have been used to determine the number of cycles to failure.

#### 6.3.1.1. Considered fatigue life criteria

Fatigue behaviour is classically characterised by a correlation between applied amplitudes and number of cycles at failure (Wöhler curve). "Ftaigue life" is the number of cycles that the sample can resist before rupture. It is a key point to find a correct criterion for fatigue life. Two criteria (i.e. Two definition of rupture) are considered in this study.

- **Classical criterion (N50)**

This criterion is widely used. N50 corresponds to a 50% decrease of the equivalent complex modulus. A representation of the classical criterion is given Figure 6.15.

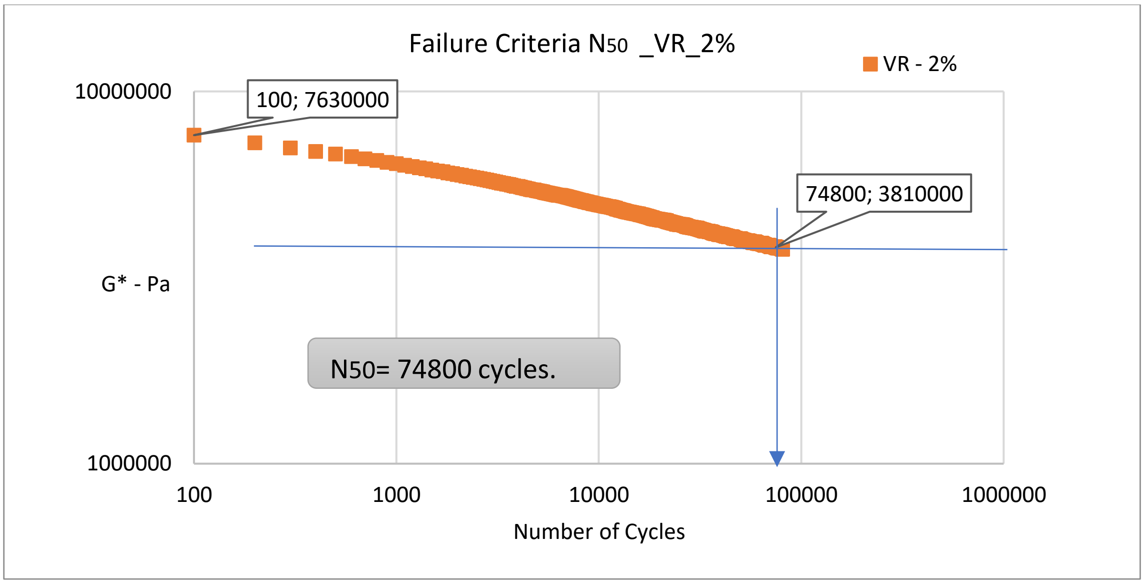


Figure 6.16 -*Failure Curve N50 - Short aged bitumen (VR) @2% strain level.*

- **Dissipated energy ratio (N-DER)**

This criterion corresponds to change in the slope of the Wc /Wi versus N curve , as illustrated in Figure 6.16.

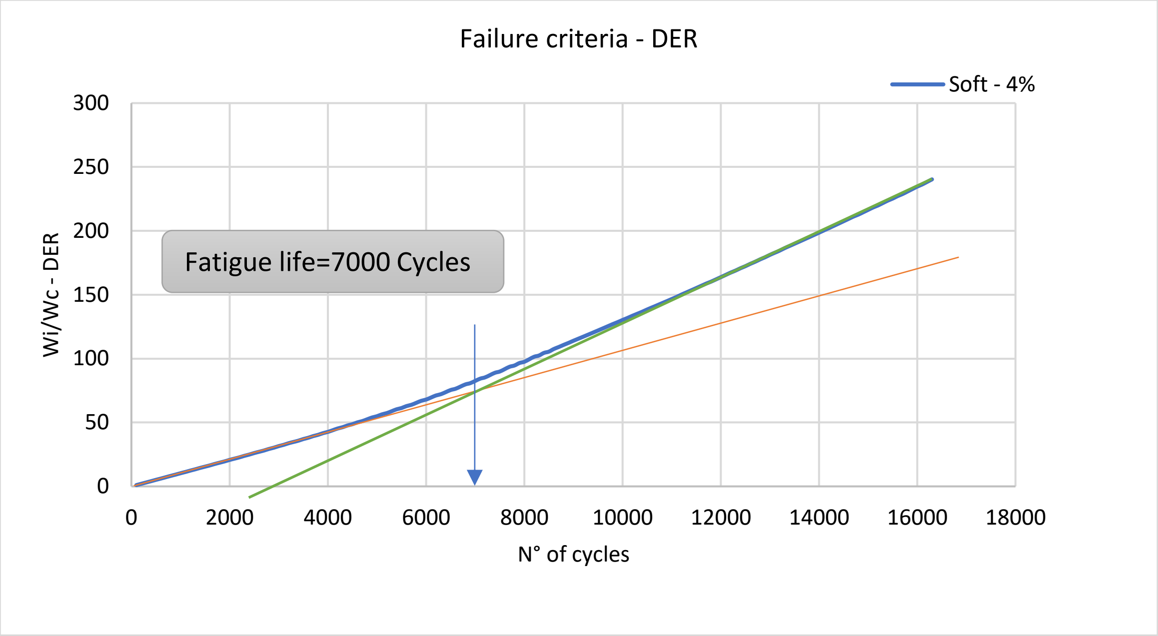


Figure 6.17 -*Failure Curve DER Approach.*

### 6.3.1.2. Wöhler curves for short aged bitumen.

A Wohler curve is a graphical representation of the power relation between fatigue life and the applied load amplitude. The Wohler Curve from each bitumen tested without a rest period and using the classical approach is obtained through N50 at each stain level. (Figure 6.17).

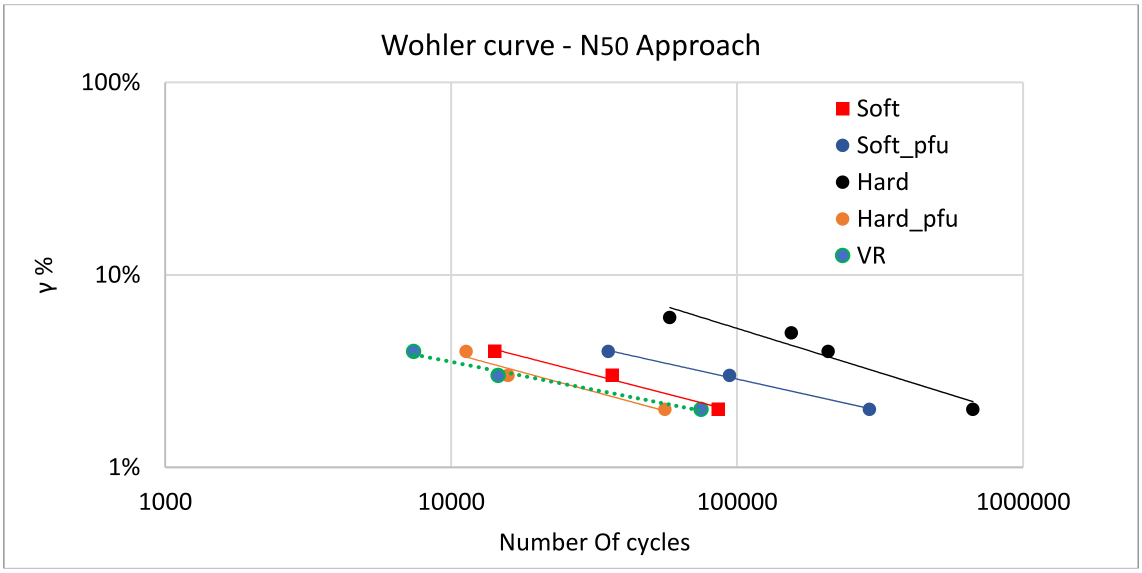


Figure 6.18 -*Comparison Wohler from N50 approach - without rest period (short aged bitumen).*

Fatigue life of Hard bitumen is the best (Figure 6.17). The presence of rubber increases the stiffness but decreases the fatigue performance as it can be seen in Figure 6.17. This observation is not valid for the soft bitumen as the presence of rubber increases the fatigue life. As the Hard, Hard_pfu and VR have the same percentage of SBS, it can be observed that the presence of SBS alone without rubber improves the fatigue life, but SBS with the presence of rubber gives negative effect.

Indeed the 50% decreased is arbitrary and failure could happen far after this limit. The criterion does not take into account phenomena occurring in phase I ((adaptation phase) This phase is characterised by a rapid decrease in stiffness ratio (or stiffness) due to repetitive excitation of the test specimen) , such as heating and thixotropy [38] that can cause a significant modulus drop, especially at high strain amplitude.

The Wohler curve generated from DER approach is shown in the following figure 6.18. It can be seen also here that Hard Bitumen has the best fatigue life,whereas Soft and VR bitumen have worst fatigue life at high strain levels. Also by DER approah can be seen that in the case of soft bitumen, the presence of rubber increases the fatigue life, whereas in the case of hard bitumen the opposite is correct.

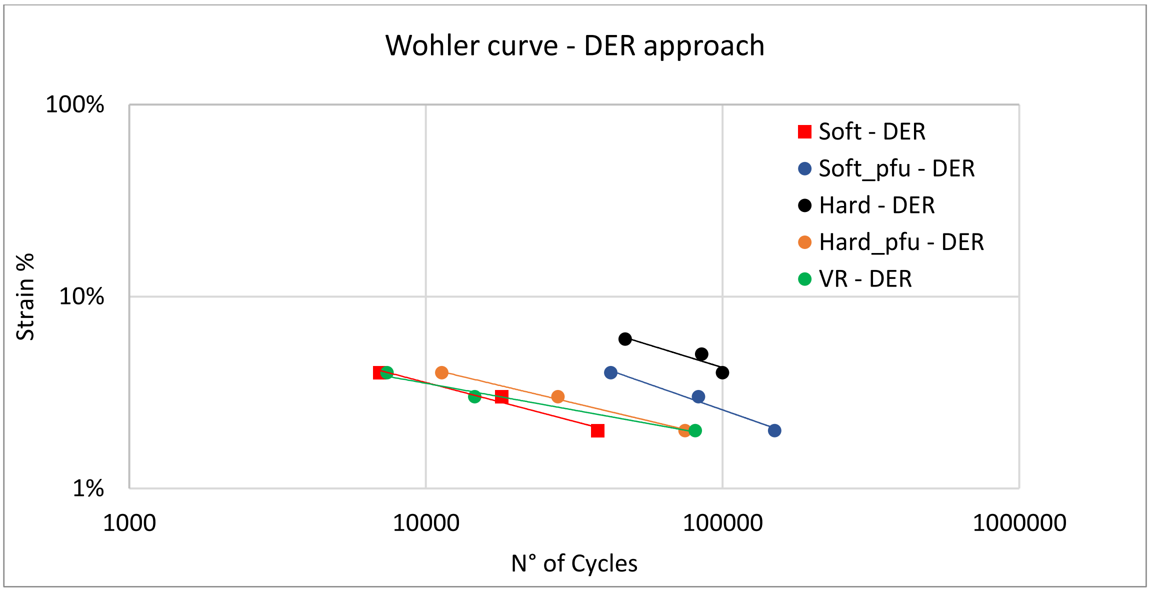


Figure 6.19 -*Comparison Wohler from DER approach* - *without rest period (short aged bitumen).*

### 6.3.2. Fatigue Tests at different temperature.

Then in order to obtain the same initial G*; the five short aged bitumen were tested at 4% strain level at different temperatures. Figure 6.19 shows a fatigue life comparison between different bitumen types obtained by N50 and DER approches.

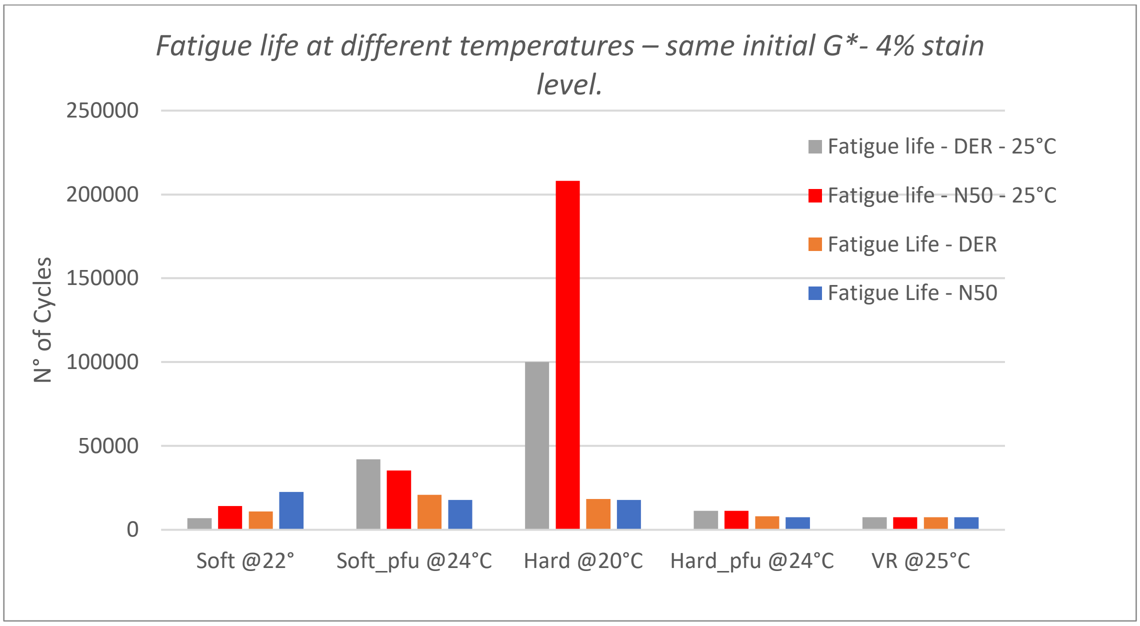


Figure 6.20 -*Fatigue life at different temperatures – 4% stain level.*

It can seen from figure 6.19 that the fatigue life obtained from both N50 and N-DER decreases as the test temperature decreases, except the Soft bitumen which has a fatigue life increses as the temperature decreases.

### 6.3.3. Fatigue Tests for Long Aged Bitumen

The same five bitumen were tested by DSR after long aging process, the tests were carried out at 4% strain level.it can be seen from figure 6.20; fatigue life decreases as a result of long aging process fatigue life decreases except Soft bitumen which has a better fatigue life after long aging process.

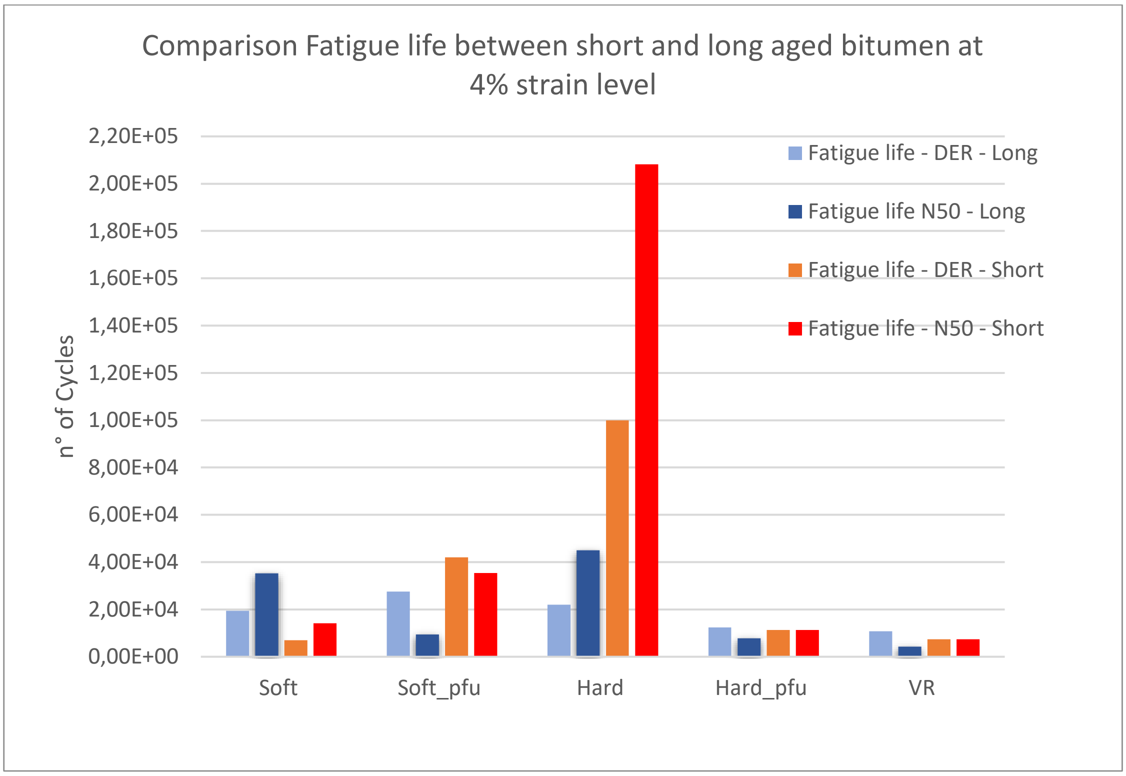


*Figure 6.21-Comparison Fatigue life between short and long aged bitumen at 4% strain level*

## 6.4. Time Sweep Test (Healing Test).

### 6.4.1. Healing Properties.

In order to study the healing properties of the bitumen; a time sweep test is carried out. The tests were made by introducing a rest period for 1 hour at the number of cycles corresponding to the 35% reduction of the G* modulus which was obtained from the previous fatigue test without a rest period.

Figure 6.21 displays a typical result obtained from healing tests. During the first loading phase, the complex modulus of the material decreases from the initial value G0* to the final value GF*. The complex modulus GR* recorded at the beginning of the re-loading phase is greater than GF*, indicating the occurrence of healing during rest time. In some cases GR* exceeds the “true” initial modulus G0*, too. Since healing may involve only the recovery of damage experienced by the material in the first loading phase, the additional stiffness gain is evidently related to other phenomena, such as steric hardening [39]. During the re-loading phase, a rapid stiffness decrease is observed with the complex modulus reaching the value obtained before loading removal (GF) as a result of the loss of both healing and steric hardening effects. Once such a value is attained, the modulus curve exhibits a shape which appears to be coherent with the evolution of stiffness recorded in the first phase of loading, thus revealing that the material has memory of its past damage path.

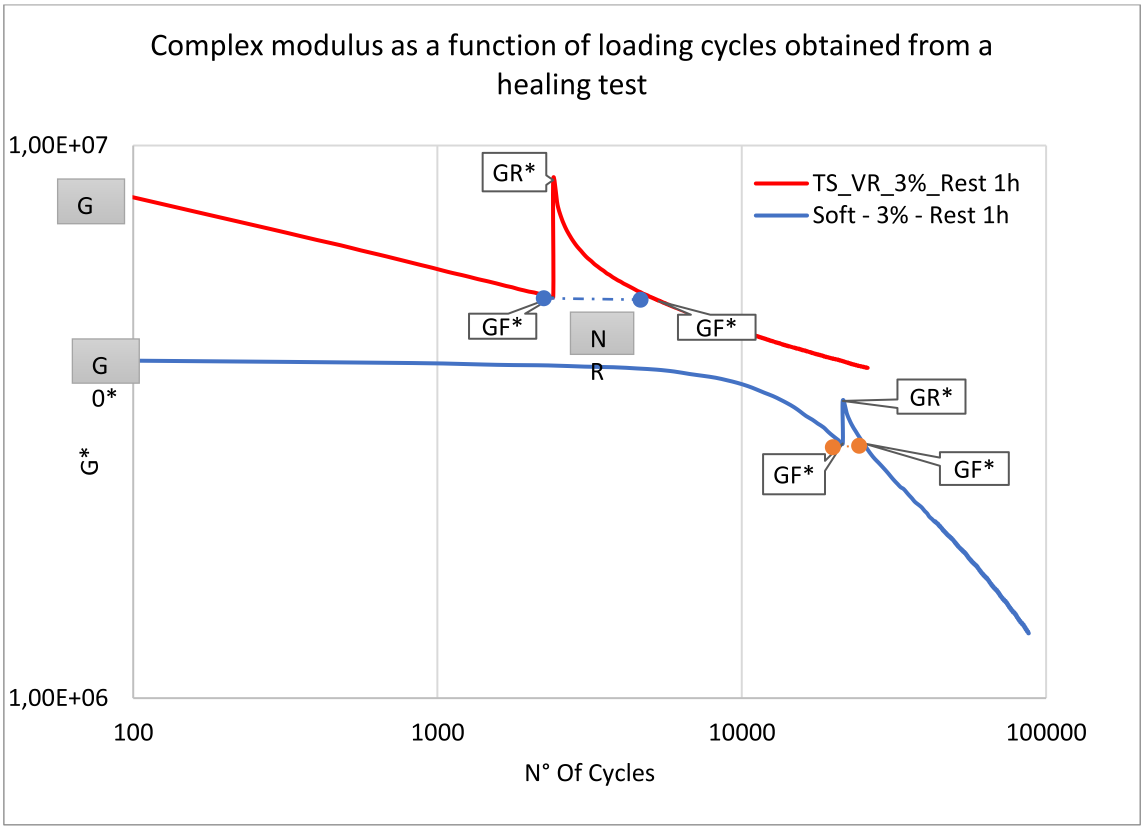


Figure 6.22 -*Complex modulus as a function of loading cycles obtained from a healing test.*

The number of loading cycles NR corresponding to the drop of complex modulus from GR* to GF* in the second phase gives a quantitative measure of the increment of fatigue life of the material due to the rest period. Such a parameter is strongly affected by the the rest period and tested bitumen type. This can be observed by considering the data plotted in Figure 6.22-a, which indicate, for short aged bitumen at different strain levels, an overall increase of NR as the strain level decrease.Figure (6.22-a) shows also that Hard bitumen has the best fatigue peformance, the presence of rubber in soft bitumen increses the fatigue performance, while the presence of rubber in the hard bitumen decrease the fatigue performance. As it can be seen from figure (6.22-b) after a long aging process the presence of rubber decreases the fatigue performance for both soft and hard bitumen. Figure (6.22-c) shows that for a test temperature less than 25°C; the fatigue performance decreses except Soft bitumen without rubber which has a better fatigue performance at test temperature equals 22°C.

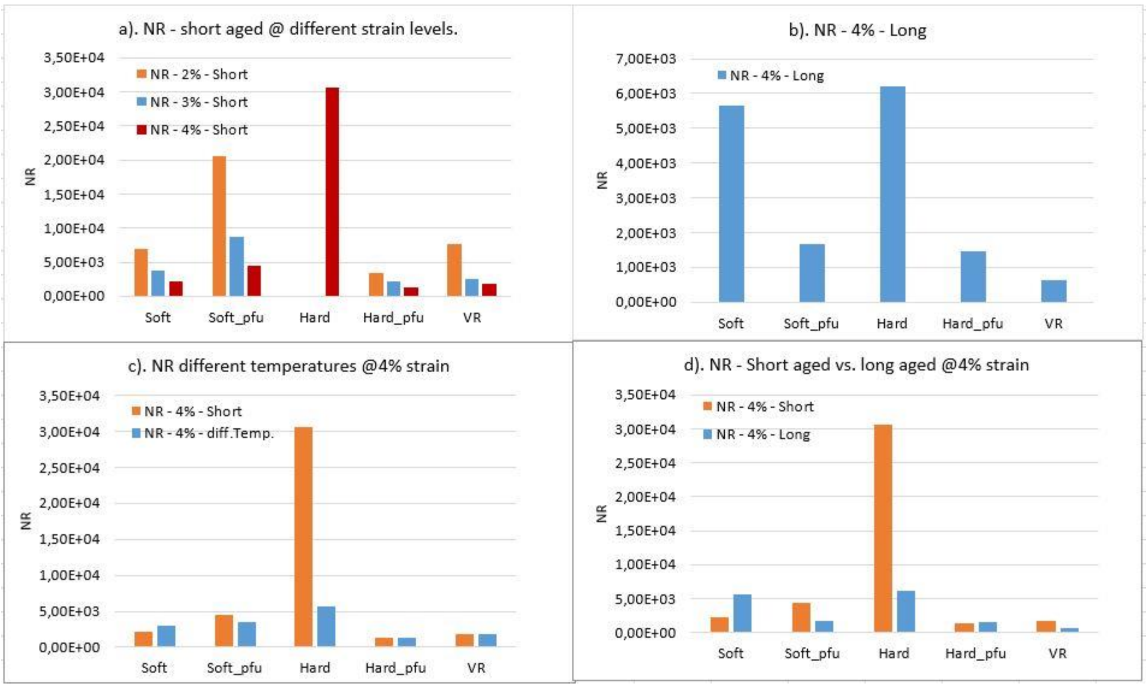


*Figure 6.23 - Variation of NR.*

Figure (6.23-a) shows the healing results for the short aged bitumen, it can be seen that the presence of rubber increases the material recovery, a higher percentage of SBS with the presence of rubber gives a better material recovery as it can be seen for Hard_pfu and VR, whereas a higher percentage of SBS gives a healing negative effect as it can be seen in the case of Hard bitumen. The same healing considerations can be seen after long aging process (figure 6.23-b). Figure (6.23-c) shows that as the test temperature decrease the healing gets better. Figure (6.23-d) shows that after long aging process the healing gets better for the five bitumen.

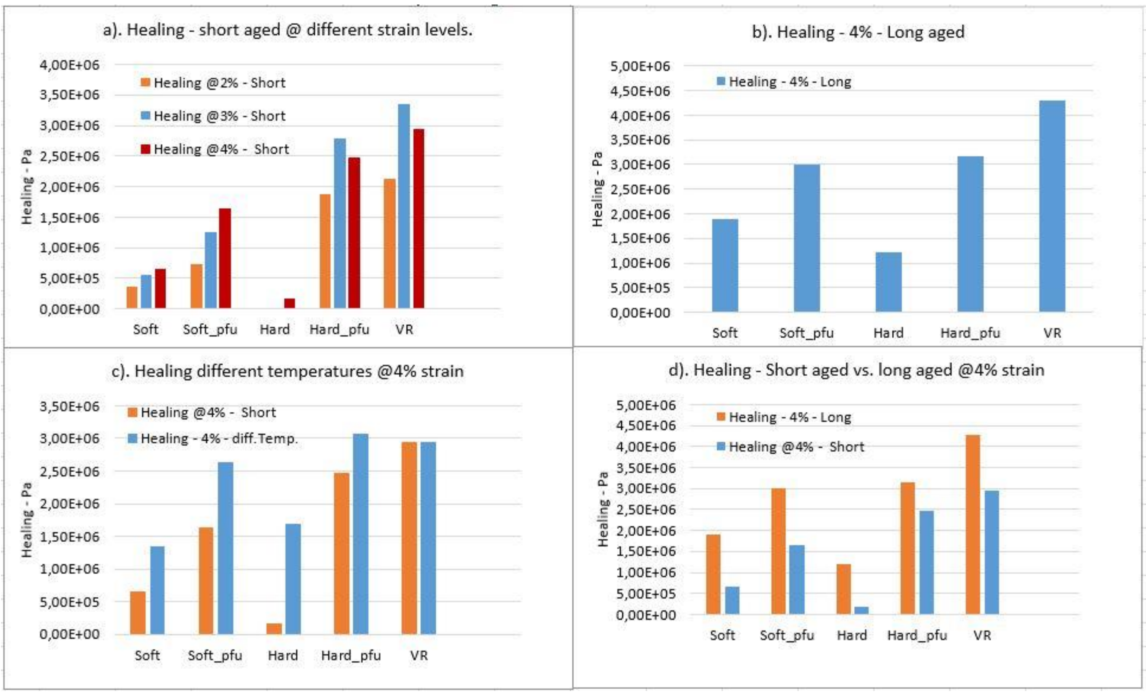


*Figure 6.24* -Variation of Healing.

### 6.4.2. Fatigue life with rest period for short aged bitumen.

Two Wohler curve is generated by the results obtained from time sweep tests were carried out at different strain levels for short aged bitumen, one by using N50 approach (figure 6.24) and the other by DER approach (figure 6.25).

It can be seen that the fatigue life of Hard bitumen is the best, The presence of rubber increases the stiffness but decreases the fatigue performance as it can be seen in Figure 6.24. This observation is not valid for the soft bitumen as the presence of rubber increases the fatigue life. As the Hard, Hard_pfu and VR have the same percentage of SBS, it can be observed that the presence of SBS alone without rubber improves the fatigue life, but SBS with the presence of rubber gives a negative effect for the fatigue life. Same results obtained also from fatigue test without rest period (N50 approach).

By using DER approach figure 6.25, it can be seen that Hard bitumen has the best fatigue life. The presence of rubber in soft btumen decrease the fatigue life at high strain levels (3% and 4%). Hard_pfu has a better fatigue life than VR, in particular at low strain level.

DER approach results from time sweep test with a rest period are more accurate than both the results from N50 approch and DER appproach results from fatigue test without rest period, because the slope changes as it can be seen from figure 6.26.

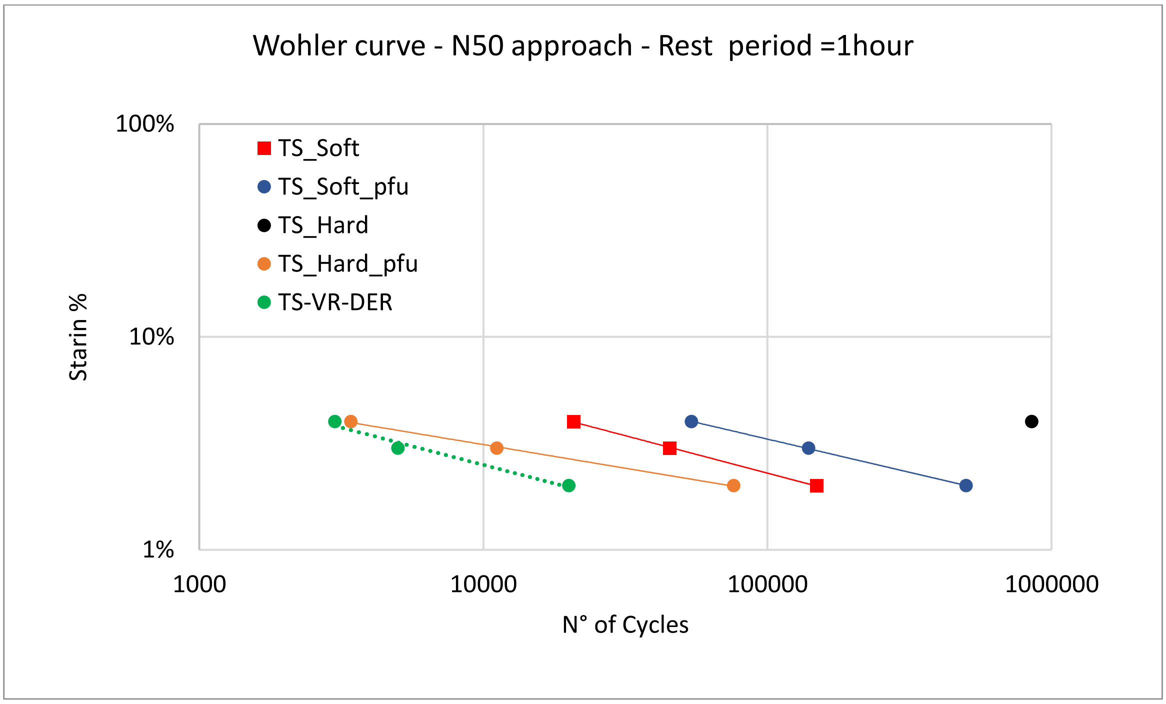


*Figure 6.25 - Wohler curve - with rest period – N50 approach.*

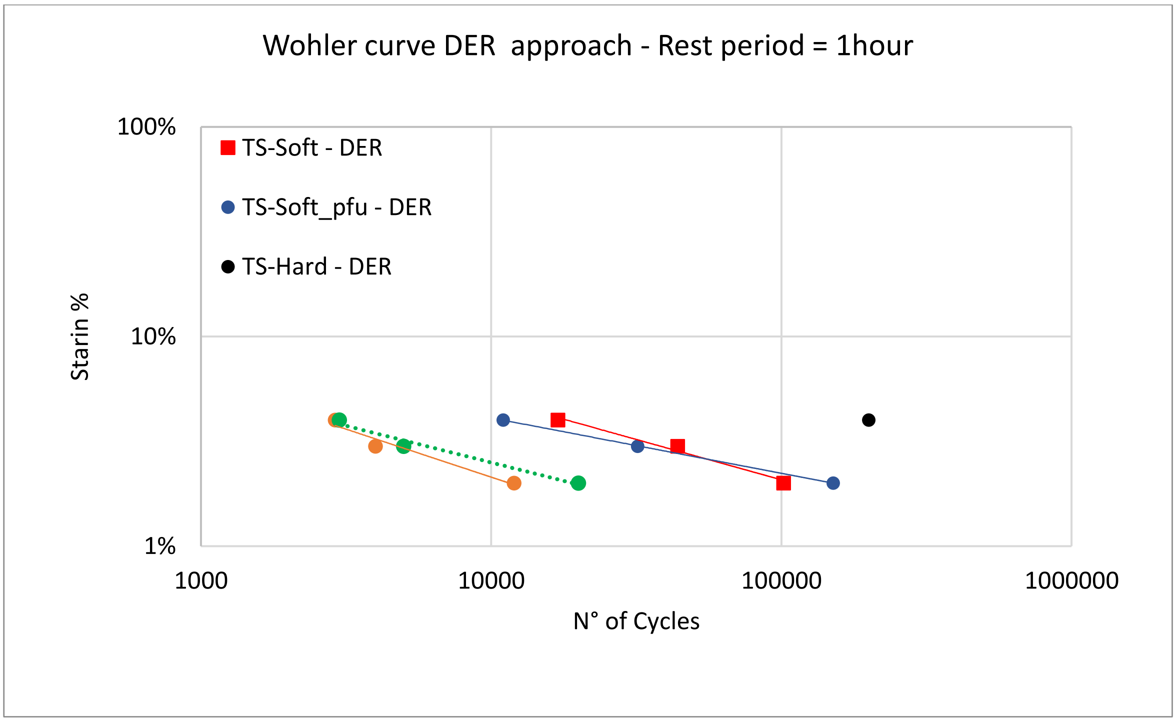


*Figure 6.26 -Wohler curve - with rest period – DER approach.*

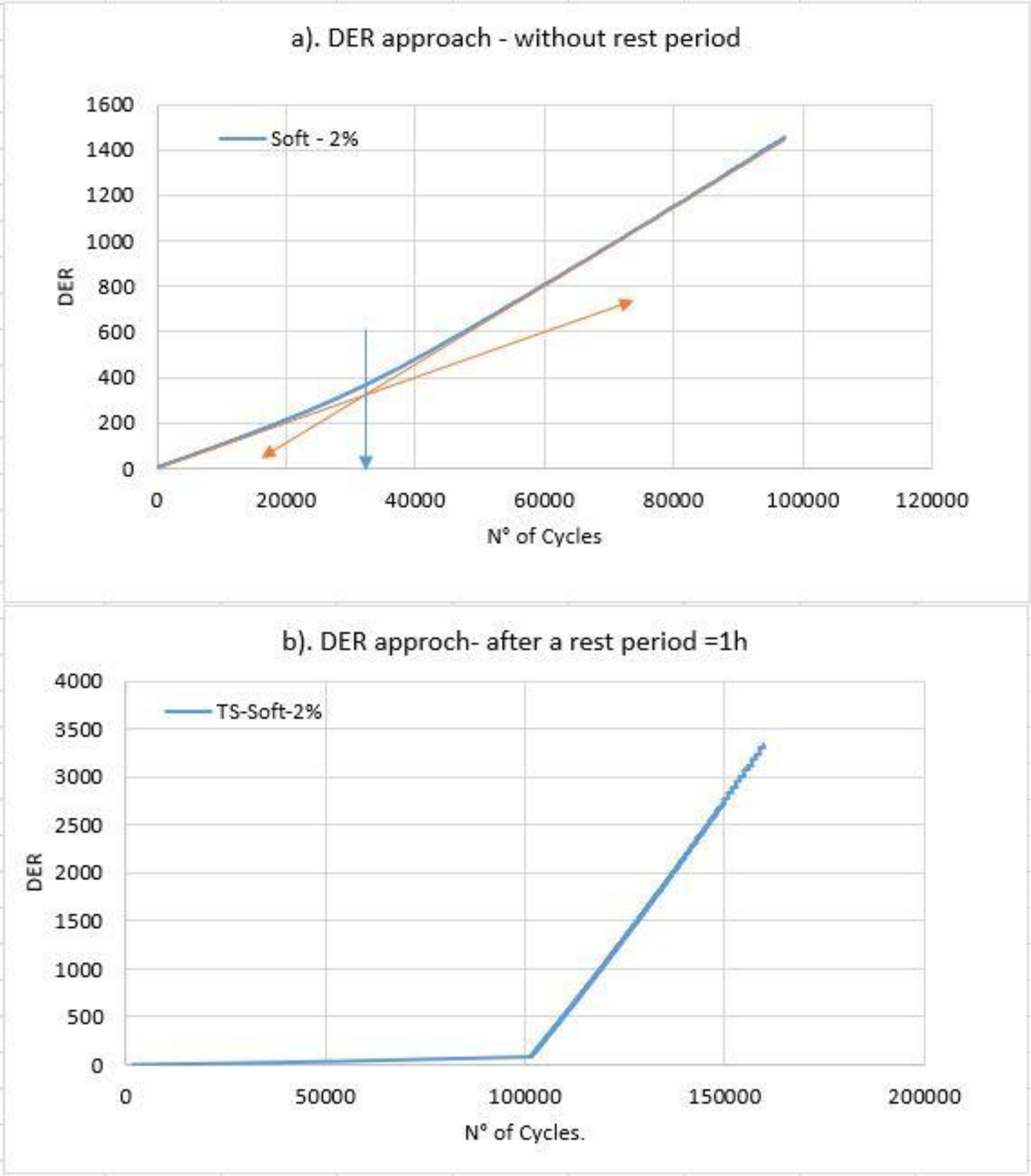


*Figure 6.27 - Comparison DER approach – with and without rest period.*

### 6.4.3. Fatigue life with rest period at different temperatures.

Time sweep tests were carried our at 4% strain level and at different temperature than 25°C, in order to make a fatigue life comparison between the five bitumen when we have the same initial G*. Fatigue life obtained by using both N50 and DER approaches for each bitumen as shown in figure 6.27. It can be seen that always Hard bitumen has the best fatigue life, DER approache shows confirms that the presence of rubber decreae the fatigue life.

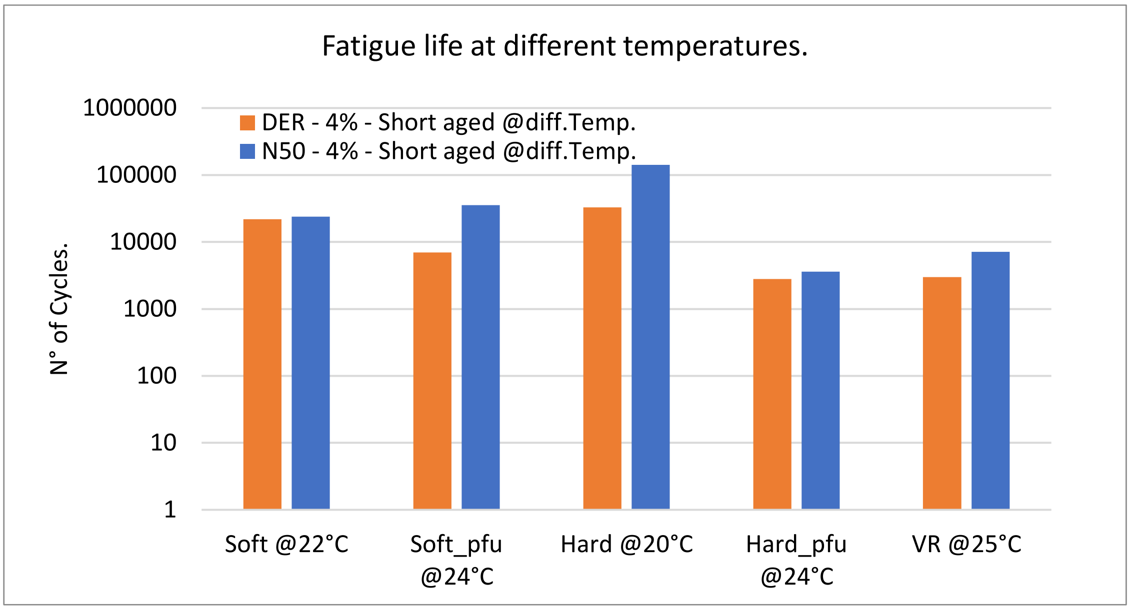


Figure 6.28 -Fatigue life at different temperatures – 4% strain level.

### 6.4.4. Fatigue life with rest period for Long aged bitumen.

Time sweep test were carried out for the long aged bitumen at one strain level (4%). By applying N50 and DER approach, it is obtained the fatigue life for the five bitumen, It can be seen in figure 6.28 that Hard bitumen has the best fatigue life also after long aging process. The presence of rubber decreases fatigue life as it can be seen by DER approach.

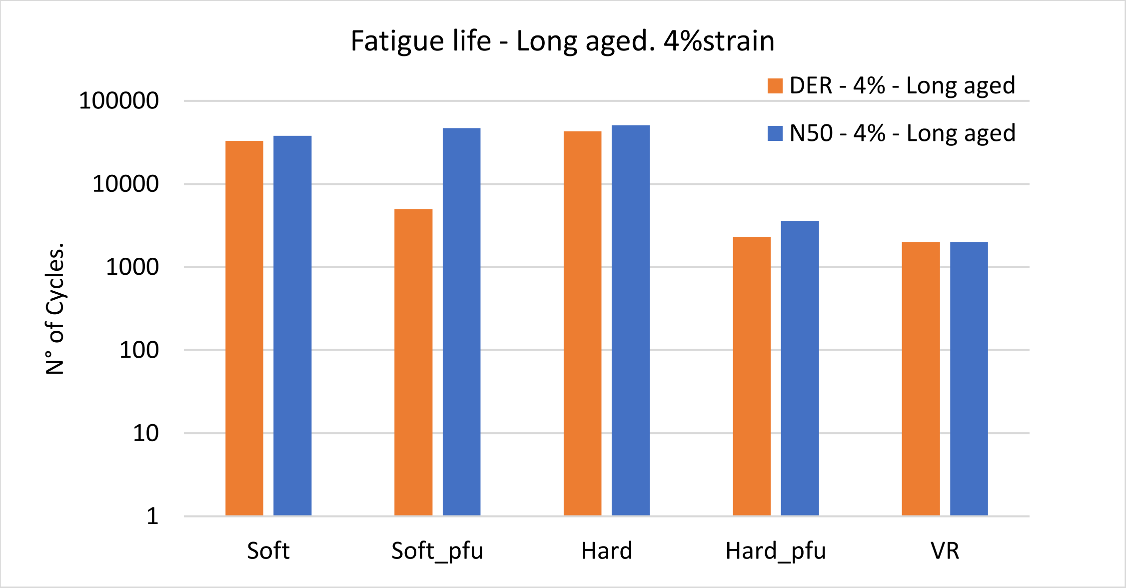


*Figure 6.29* -Fatigue life for long aged bitumen at 4% strain level.

# CHAPTER 07: CONCLUSIONS

# 7. CHAPTER 07. CONCLUSIONS.

The main objectives of this study was to investigate the bitumen aging in short term and long term. In particular, it was studied how this phenomenon affects the rheological properties of bitumen. DSR oscillatory testing were used to determine the fatigue properties of five modified bitumen with SBS and with or without crumb rubber at a temperature of 25°C.

The SBS increases the values of complex modulus throughout the frequency range and reduces the values of phase angle at high temperatures. The master curves have the s-shape typical of asphalt, showing how the presence of SBS gives greater shear stress resistance at the high temperatures.

The presence of rubber increases the elastic response of the bitumen at high temperatures even after aging.

VR bitumen is the best which can resist rutting and thermal cracking.

Ageing effects were discussed by comparing the results obtained from a single bitumen tested in the unaged state with those obtained after short and long aging treatments. It was observed that ageing produces an increment of rutting and thermal cracking resistance with the presence of rubber.

The higher percentage of SBS in Hard bitumen proved to be the material exhibiting the highest fatigue resistance with respect to the other bitumen. Healing properties are enhanced by the presence of the rubber. The introducton of a rest period decreases fatigue life.

The presence of rubber decrease the fatigue life, as evident from the comparison between all five bitumen. The presence of rubber with a higher percentage of SBS decrease the fatigue resistance, as evident from the comparison between soft and hard bitumen. This would indicate that there is some form of chemical reaction occurring between the SBS and rubber.

The fatigue life based on the DER method consistently under-predicted the fatigue life based on the 50% drop in modulus method. Overall life duration of the different criteria are similar indicates coherence between the two approaches.

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