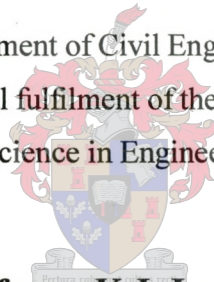# Rheology of Polymer Modified Binders

A comparative study of three binders and three binder/filler systems

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A thesis submitted to the Department of Civil Engineering, Faculty of Engineering, University of Stellenbosch in partial fulfilment of the requirements for the degree Master of Science in Engineering

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Declaration

I, the undersigned, do hereby declare that the work contained in this thesis is my own original work and has not been submitted previously in its entirety or in part at any university for a degree:

Summary

The use of polymer modified binders have increased drastically over the past twenty years. It is generally accepted that current empirical specification tests do not adequately characterise the improvement in binder properties when bitumen is modified with polymers.

At the University of Stellenbosch, a programme was initiated to relate binder properties to asphalt mix performance. This study forms part of that programme.

This study focuses on three binders and is an attempt to characterise binder properties fundamentally. The binders that were tested are:

- 60/70 penetration grade bitumen
- SBS modified 60/70 bitumen
- Bitumen-Rubber

Two rheometers were used to characterise the binder properties:

- Sliding Plate Rheometer (SPR)
- Dynamic Shear Rheometer (DSR)

The Sliding Plate Rheometer was modified to allow testing at elevated temperatures. The modification is described in this thesis.

Three types of testing were used to test the binders:

- Creep tests (SPR and DSR)
- Dynamic tests (Strain and Frequency Sweeps – DSR)
- Flow tests (high temperature viscosity tests – DSR)

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The results of the tests were used to characterise differences between the binders. Shear susceptibility lines were plotted and viscosities calculated from the creep test results; Master Curves and Black Diagrams were plotted from frequency sweep results; flow test results were used to characterise high temperature behaviour.

A further development in the asphalt industry is the move towards the development of performance based specifications. A programme has been initiated in this regard and this study forms part of the programme.

As a step towards the development of performance based specifications filler was included in this study. Binder/Filler systems were subjected to the same test programme the binders were, to characterise the influence on binder properties of filler addition.

Some of the conclusions that were drawn at the end of the study are:
- Polymer modification improves binder properties related to rutting.
- Filler addition does not affect binder properties negatively.
- Shear rate is very important during production of asphalt mixes and construction of asphalt layers containing polymer modified binders.

Samevatting

Die gebruik van polimeer gemodifiseerde bitumen het drasties toegeneem oor die afgelope twintig jaar. Dit word algemeen aanvaar dat die huidige empiriese spesifikasie toetse nie die verbetering in bindmiddeleienskappe van polimeer modifisering aanvaarbaar karakteriseer nie.

'n Program is geïnisieer by die Universiteit van Stellenbosch om 'n verband te vind tussen bindmiddeleienskappe en asfaltmengseleienskappe. Hierdie studie vorm deel van die program.

Hierdie werk fokus op drie bindmiddels en is 'n poging om die bindmiddeleienskappe fundamenteel te karakteriseer. Die bindmiddels wat gebruik is in hierdie werk, is:

- 60/70 penetrasiegraad bitumen
- SBS gemodifiseerde 60/70 bitumen
- Bitumen-Rubber

Twee reometers is gebruik om bindmiddeleienskappe te karakteriseer:

- Glyplaat Reometer (SPR)
- Dinamiese Skuif Reometer (DSR)

Die glyplaat reometer is aangepas om toetse by hoër temperature te doen en die aanpassing wat gemaak is word in die tesis beskryf.

Drie tipes toetse is uitgevoer om die bindmiddels mee te toets:

- Kruiptoetse (SPR en DSR)
- Dinamiese toetse (DSR)
- Vloei toetse (DSR)

Die resultate van die toetse is gebruik om die verksille tussen die bindmiddels te kwalifiseer. Verskillende grafieke en figure is getrek van die resultate om die onderskeie bindmiddels te karakteriseer.

Die beweging weg van empiriese spesifikasies na werkverrigting gebaseerde spesifikasies in die asfalt industrie is 'n verder nuwe ontwikkeling. 'n Program is geïnisïeer ten opsigte hiervan en hierdie werk vorm deel van die program.

As 'n stap in die ontwikkeling van die werkverrigting gebaseerde spesifikasies is 'n vuller ingesluit in die toetsprogram. Bindmiddel/vuller stelsels is onderwerp aan dieselfde toetse waaraan die bindmiddels onderwerp is om die invloed van die vuller op die bindmiddels te karakteriseer.

Sommige van die gevolgtrekkings waartoe gekom is aan die einde van die tesis, is:
- Polimeer modifisering verbeter bindmiddeleienskappe met betrekking tot wielsporing.
- Die toevoeging van vuller affekteer nie die bindmiddeleienskappe negatief nie.
- Die vervormingstempo is van groot belang wanneer asfaltmengsels gemeng en lae gebou word van mengsels wat polimeer gemodifiseerde bitumen bevat.

This work is dedicated to my wife, Anelle.

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1 Introduction

Bitumen specifications that are used today are based on empirical tests. Binder properties are characterised by means of tests such as the penetration test and viscosity measurements.

The abovementioned tests have been developed to characterise normal penetration grade bitumen. These tests, in conjunction with tools such as the Bitumen Test Data Chart (BTDC) and Van der Poel's Nomograph, have been used extensively for the characterisation of binder behaviour and prediction of binder properties for use in asphalt design methods. The material properties and binder performance of normal binders have been well correlated through the use of the tools mentioned.

However, the empirical tests and the viscosity measures fail to characterise the improved performance of polymer modified binders. Polymers are used to modify binders in order to improve binder performance in the field. Binder properties that are improved include resistance to permanent deformation and fatigue cracking – two widely encountered problems where asphalt pavements are used.

Specifications used in the design of asphalt pavements are changing with time. This is because new materials and improved design methods are used for the construction of asphalt pavements. These changes in design methods and the materials are the result of increased traffic loads and higher wheel loads being allowed. In South Africa there is also an effort to improve the design specifications for Hot-Mix Asphalt (HMA). [12] The interim guidelines make special mention of the use of polymer modified binders. However, the tests described for the characterisation of binder properties are the empirical tests and viscosity measures. Thus, it seems that there is a large gap in terms of knowledge that has to be addressed. It is the aim of this study to contribute towards the development of the required knowledge in order that improved binder properties can be characterised effectively.

It should also be mentioned that there is a move in the world today towards the development of performance based specifications. This type of specification has the properties of the constitutive components of asphalt mixtures as input and through predictive functions expected performance is predicted.

The improved performance of polymer modified binders can be characterised by means of fundamental rheological characterisation of the binders. Test methods and apparatus for this type of fundamental characterisation have improved enormously over the past two decades. Due to the relative newness of these tests and the high initial cost for the acquisition of the testing apparatus the binder industry is reluctant to use it. However, the benefits that can be gained from the fundamental characterisation of binder properties are high. For instance, through the use of a Black Diagram the increased resistance to permanent deformation is immediately characterised when a modified and unmodified binder is compared.

Rheological characterisation must be used for the development of new test methods that adequately describe the improved properties of modified binders. On the other hand, knowledge of this type of testing has to be built up in order that binder properties can be related to improved performance. In the next section rheology will be described in more detail.

1.1 Rheology

The word rheology comes from a Greek verb that means "to flow". It was coined in 1920 by a professor of chemistry, Eugene Bingham, at Lehigh University, Pennsylvania, U.S.A. The new word described the budding science of studying the flow behaviour of synthetic polymers. This new science was the result of the development of the chemistry industry at the beginning of the twentieth century, which resulted in a host of new materials with "strange" flow behaviour.

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Thus, rheology means the study of flow and deformation. In principle, rheology would include everything dealing with flow behaviour: aeronautics, hydraulics, fluid dynamics, and even solid mechanics. In practice, rheology has been restricted to the study of fundamental relations (constitutive relations) between force and deformation in materials, primarily liquids.

The rheologist, therefore, focuses on material behaviour using very simple deformations. The mechanicist, on the other hand, studies the forces developed in complex deformation, applying the constitutive relations developed by the rheologist.

Rheology is a very important field of study for bitumen technologists. A thorough study of the behaviour of bitumen under different loading (load and frequency) and temperature domains can go a long way in the prediction of the binder's behaviour in the field.

The binder's characteristics have to be converted into mix behaviour in order that prediction about field behaviour can be made. Predictions are made through the use of predictive functions. These predictive functions are developed through the study of the influence of different aspects on mix behaviour. Thus, to develop predictive functions that are of value to the pavement engineer, the full picture has to be kept in mind and research must revolve around that. (For a full description of this, see section 1.3)

Rheology is not only restricted to the study of pure bitumen. A study at the fundamental level of the rheology of polymer-modified bitumen is invaluable for the characterisation of this type of material. The following section looks at this type of binder specifically.

Two types of apparatus were used in this study to characterise binder properties. These are the Sliding Plate Rheometer (SPR) and the Dynamic Shear Rheometer (DSR). When the empirical measures are used to characterise binder properties (e.g. penetration, softening point, ductility, etc.) a value is obtained at the end of the test. These results are difficult to correlate with binder performance. When, on the other hand, binder characterisation is done fundamentally, there is a direct correlation between property

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measured and binder performance (e.g. the Black Diagram shows phase angle at different complex moduli – this can be used directly to predict binder performance under different load and temperature regimes; high temperature viscosity measurements may show unusual behaviour for polymer modified binders – this knowledge can be used to determine mixing and compaction temperatures). It is for this reason that fundamental characterisation of binder properties is superior to conventional empirical characterisation. Figure 1-1 shows this graphically.
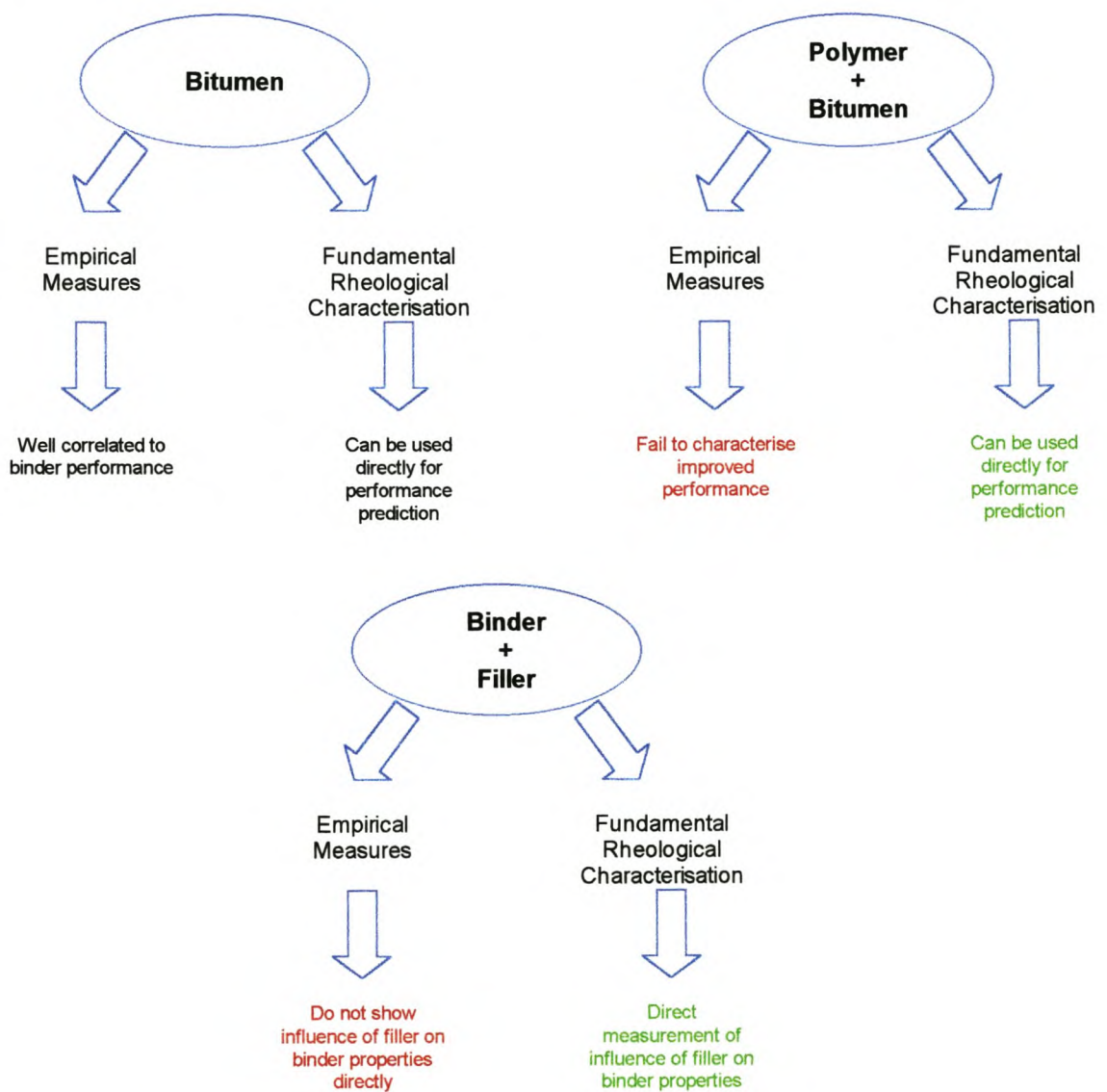
Figure 1-1: Fundamental Characterisation versus Empirical Measures.

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1.2 Polymer Modified Binders

Polymer modification of bitumen has been increasing steadily over the past 15 to 20 years. Every paper that has been published on polymer modification of bitumen has mentioned the need to improve the performance of the binder in the road. This need for better performance is the result of the increase in traffic loads and wheel loads.

Table 1-1 shows typical polymer modifiers and the distress types addressed.

Type	Class	Distress Type				
		PD[1]	FC[2]	LTC[3]	MD[4]	AG[5]
Elastomers	Styrene butadiene diblock (SB)	x		x	x	
	Styrene butadiene triblock/radial block (SBS)	x	x	x		
	Styrene Isoprene (SIS)	x				
	Styrene ethylbutylene (SEBS)					
	Styrene butadiene rubber latex (SBR)	x		x		
	Polychloroprene latex	x	x			
	Natural rubber	x				
	Acrolynite butadiene styrene (ABS)	x				
Plastomers	Ethylene vinyl acetate (EVA)	x	x			
	Ethylene Propylene diene monomer (EDPM)	x				
	Ethylene acrylate (EA)	x				
	Polyisobutylene	x				
	Ployethylene (low density and high density)	x		x		
	Polypropylene	x				

1: Permanent Deformation
2: Fatigue Cracking
3: Low Temperature Cracking
4: Moisture Damage
5: Ageing

Table 1-1: Typical polymer modifiers.

Furthermore, it has been stated numerous times that many of the current specification tests are of an empirical nature and do not reflect the true performance of the binder in the asphalt layer. Therefore, there has been a move towards the development of performance-based specifications for binders. A first result of this is the current SUPERPAVE specification, which makes use of dynamic mechanical analysis to characterise the bitumen.

One of the areas where performance based specifications are of special interest is polymer modified bitumen. It has been shown that the current empirical tests are characterising the base bitumen reasonably, but the improved performance derived from the polymer modification is not quantified. Therefore, there is a need in the asphalt industry to look towards developing new tests that will quantify performance of polymer modified binders.

An example of this move towards performance-based specifications is the resolution adopted at CAPSA '99: "The industry should look towards the harmonisation of specifications in SADC – specifically for bitumen and performance criteria for PMB's." The delegates at CAPSA '99 also expressed the need for the initiation of projects to gain field experience on the performance of polymer modified binders.

1.3 Performance based specifications

When performance based specifications are to be applied it is imperative to keep the full picture in perspective. Material behaviour as well as the factors influencing it should be considered in the overall picture.

Figure 1-2 gives a graphical representation of the factors that affect material behaviour.
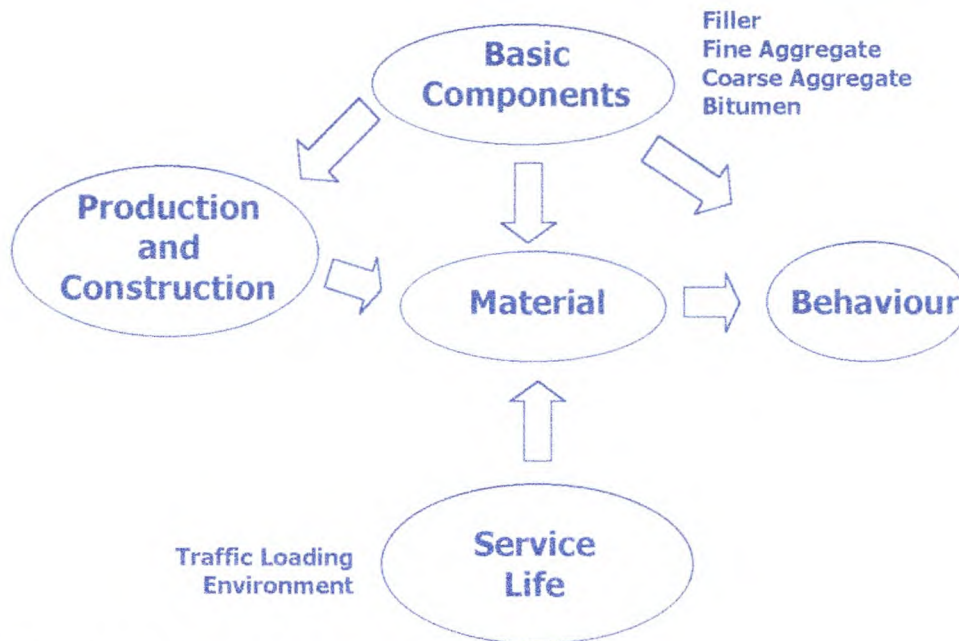
Figure 1-2: Factors influencing material behaviour.

As can be seen on Figure 1-2 the material is constituted by different basic components. Each of these components takes its own intrinsic material properties into the material in which it is used. Thus, it can be said that material behaviour is influenced by the basic components constituting the material.

In the case of asphalt mixtures, the mixing and compaction activities can also influence material behaviour. For instance, if the temperature during mixing is too high, the bitumen may age excessively leading to a very stiff asphalt mixture that may not perform as was expected. On the other hand, if the compaction activity was not monitored well, it may happen that the asphalt mixture has very high voids leading to early rutting and even stripping. Had the voids been too low after compaction, the mix might be unstable and shear failure might occur. Thus, mixing and compaction activities influences the type of material obtained and the material in turn has unique behaviour.

The influence of factors affecting the material during the service life must also be taken into account. The expected traffic load during the service life has to be used when the

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mix is designed to ensure that it will be able to carry the traffic load. Traffic load include the tyre pressures of the vehicles travelling on the road as well as the number of load repetitions.

The environment might also influence the material. For instance, in places experiencing hot days with lots of sunshine, it might be expected that the binder will age quickly due to the influence of the UV radiation and the heat combined with the oxygen in the atmosphere.

Thus, the development of performance based specifications is a very complex exercise where all the factors affecting material behaviour have to be included. This study is a step towards the development of performance based specifications and focuses on two of the basic components that are used to manufacture asphalt mixes: the binder and filler.

1.4 Objective of Study

This study is intended as a step towards the development of performance based specifications. The aim is also to contribute towards the development of a knowledgebase from which tests can be developed for the effective characterisation of binder properties.

The goal at the outset was not the quantification of differences between different binders and binder/filler systems, but rather to serve as an exploratory study into the basic material properties of two components of asphalt. An understanding of the fundamental properties of modified bitumen and the characteristics with which they should be measured, is imperative before performance based specifications can be developed. Understanding the influence of the constitutive components on mix properties is just as important. Therefore binder/filler systems were included in this study.

The purpose of this study was to lay the groundwork for further developments in this field and to contribute towards the building of a knowledgebase of fundamental

rheological testing. Therefore this work could form the basis for further development of research programmes in this field.

1.5 Scope of study

This study focuses on the fundamental rheological characterisation of three binders: a 60/70 penetration grade bitumen; a 60/70 bitumen modified with 3% by mass SBS; and a bitumen-rubber binder.

The fundamental characterisation was done by means of the Sliding Plate Rheometer (SPR) and Dynamic Shear Rheometer (DSR).

The SPR was used to perform creep tests on the three binders and three binder/filler systems. The results of these tests were used to plot shear susceptibility lines and calculate the viscosities of the binders and binder/filler systems at the temperatures that were used.

The DSR was used for four types of testing: strain sweeps; frequency sweeps; creep tests; and high temperature viscosity testing.

- The strain sweeps were done to determine the linear limits of the binders at the temperatures at which the frequency sweeps were performed.
- The frequency sweeps were performed below the linear limits of the binders at eight different temperatures. These frequency sweep results were used to construct Master Curves and Black Diagrams for the three binders.
- Creep tests were performed with the DSR to compare the results with the SPR results.
- High temperature viscosity tests were performed to gain insight into the high temperature and high shear rate behaviour of the binders used in the study.

As a first step toward the development of prediction functions for pavement behaviour from binder properties (performance based specifications), the study was expanded to include binder/filler systems. The binder/filler systems were made up out of the three binders and a mineral filler common to the Western Cape. The binder/filler systems were subjected to the same tests to which the binders were subjected. This was done in order to characterise the effect of the filler on the binder properties.

1.6 Chapter Outline

In chapter 2 an overview of bitumen chemistry is given.

In chapter 3 the rheology of bitumen is discussed. Chapter 3 also contains information on current specification testing and the rheology of polymer modified bitumen.

Chapter 4 contains the test programme followed in this study. The different materials that were used are also described.

In chapter 5 the results of the creep tests performed with the Sliding Plate Rheometer is presented.

In chapters 6, 7 and 8 the results of the testing with the Dynamic Shear Rheometer is discussed.

Chapter 9 contains conclusions and recommendations.

2 Bitumen Chemistry and Chemical Structure

Bitumen can be classified as a polymer – it is made up of hydrocarbon molecules with small amounts of sulphur, oxygen and nitrogen. There are also some trace quantities of metals present such as vanadium, nickel, iron, magnesium and calcium. These occur as inorganic salts and oxides.

It is very difficult to describe the composition of bitumen precisely. Unlike the production of a pure synthetic polymer where the chemical composition is monitored closely, bitumen composition varies with the crude source from which it is manufactured. The chemical composition can be summarised as follows [7]:

Carbon	82 – 88%
Hydrogen	8 – 11%
Sulphur	0 – 6%
Oxygen	0 - 1.5%
Nitrogen	0 - 1%

The precise composition depends on the source of the crude oil and the subsequent modification induced by semi-blowing and blowing during manufacture and subsequent ageing in service.

It is necessary for the civil engineer to have some understanding of the chemical structure of bitumen when studying rheology. Fortunately, he does not need the same level of information that the chemist will be looking for when studying the same bitumen.

Due to the complexity of bitumen, it is too laborious to do a complete chemical analysis. The amount of information will also make it virtually impossible to correlate the chemical composition with the rheological properties.

Fortunately, bitumen composition can be broadly divided into two main groups (see Figure 2-1) – asphaltenes and maltenes. The maltenes can be further subdivided into saturates, aromatics and resins.

Bitumen is regarded as a colloidal system. It consists of the high molecular weight asphaltene micelles dispersed or dissolved in a lower molecular weight oily medium (maltenes). [7] (A colloidal system is a system where some particles/molecules are dispersed/dissolved in a medium. An example of a colloidal system is clay particles dispersed in water. This gives the water a murky colour.)

The micelles are considered to be asphaltenes together with an absorbed sheath of high molecular weight aromatic resins that act as a stabilising solvating layer. As one moves away from the centre of the micelles there is a gradual transition to less polar aromatic resins. These layers extend outward to the less aromatic oily dispersion medium.

The four groups are not well divided and there is some overlap between the groups. However, it does allow bitumen rheology to be compared with the broad chemical composition.
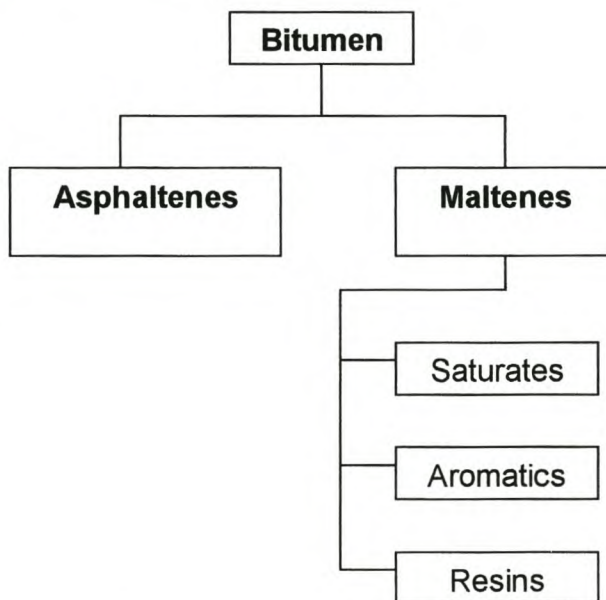
Figure 2-1: Composition of bitumen (broad chemical groups) [7].

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The asphaltenes are black or brown amorphous solids and are insoluble in n-heptane. The chemical composition includes the following substances: carbon, hydrogen, nitrogen, sulphur and oxygen. Asphaltenes are considered as highly polar and complex aromatic materials of high molecular weight. The molecular weights fall in the range 1 000 – 100 000 and the particle size range between 5 nm to 30 nm. Asphaltenes constitute 5 to 25% of bitumen.

The asphaltene content influences the rheological behaviour of the bitumen – increasing the asphaltene content produces a bitumen that is harder, has a lower penetration value, has a higher softening point and higher viscosity. This would translate into a higher complex modulus (G*) when looking at the parameters obtained from dynamic mechanical analysis.

The resins contain similar chemical substances to the asphaltenes (hydrogen, carbon, oxygen, sulphur and nitrogen). Unlike the asphaltenes, the resins are soluble in n-heptane. The resins are coloured dark brown. The resins are solid or semi-solid and are very polar in nature. The polarity of this substance makes it very adhesive. The resins are the dispersing agents or peptisers that disperse the asphaltenes in the bitumen. The proportion of asphaltenes to resins governs to a degree the solution (SOL) or gelatinous (GEL) type character of the bitumen.

The aromatics comprise the substances in bitumen with the lowest molecular weight – the weights range from 300 to 2 000. Aromatics are dark brown, viscous liquids and constitute 40 to 65% of the bitumen.

The aromatics constitute the major proportion of the dispersion medium for the peptised asphaltenes. The aromatics consist of non-polar carbon chains in which the unsaturated ring systems dominate. They have a high dissolving ability for the other high molecular weight hydrocarbons.

Saturates are non-polar viscous oils. The saturates comprise straight and branch-chain aliphatic hydrocarbons as well as alkyl-naphtenes and some alkyl-aromatics. They include both waxy and non-waxy saturates. Saturates are straw or white in colour and constitutes 5 to 20% of the bitumen.

When there are sufficient quantities of resins and aromatics of adequate solvating power the asphaltenes are fully peptised. The resulting micelles have good mobility within the bitumen. These types of bitumen are known as solution or 'SOL' type bitumens (see Figure 2-2).

Asphaltenes

High molecular weight aromatic hydrocarbon

Low molecular weight aromatic hydrocarbon

Aromatic-naphtenic hydrocarbons

Napthenic/aliphatic hydrocarbons

Saturated hydrocarbons
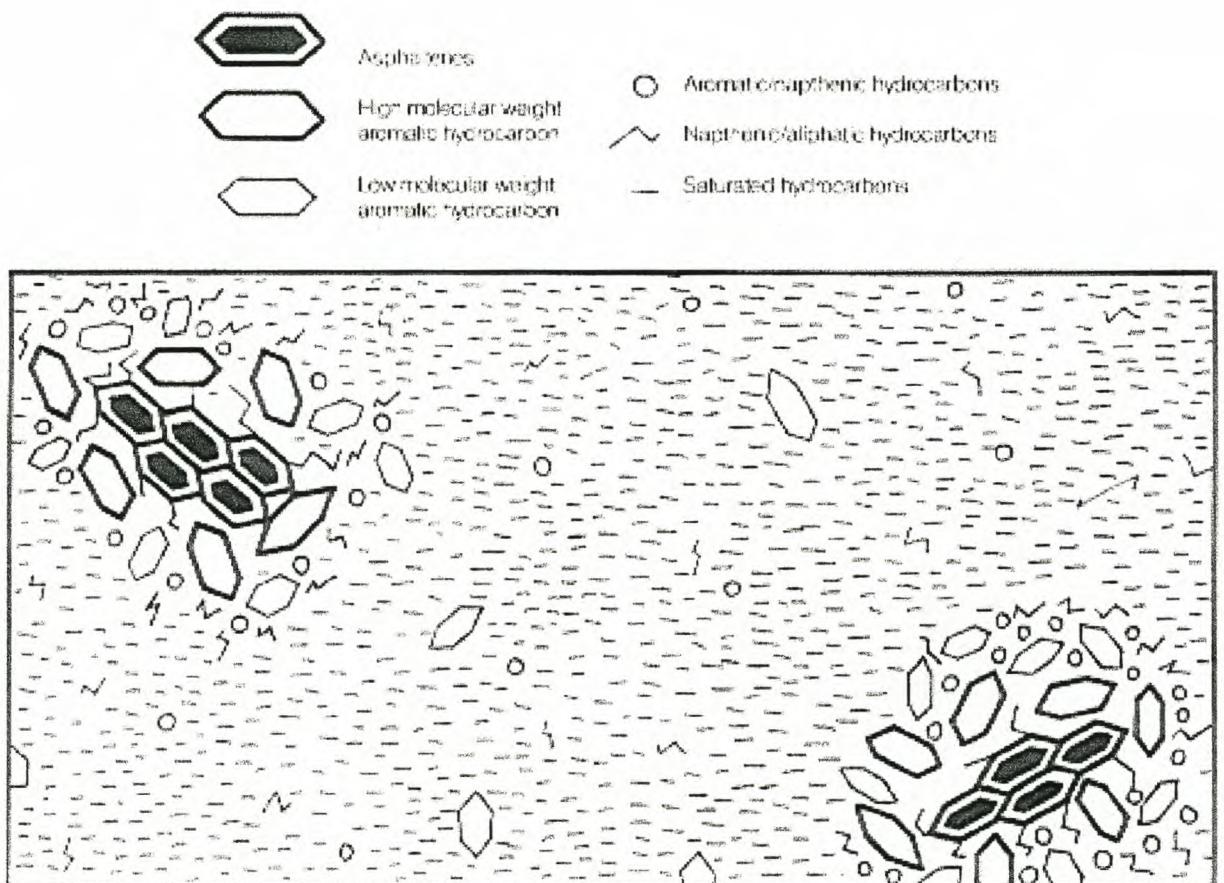
Figure 2-2: "Sol"-type bitumen structure [7].

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When the aromatic/resin fraction is not present in sufficient quantities to peptise the micelles, or has insufficient solvating power, the asphaltenes can associate further together. This association of asphaltenes leads to an irregular open packed structure of linked micelles. The internal voids are filled with an inter-micellar fluid of mixed composition. These types of bitumen are known as gelatinous or 'GEL' type bitumens (see Figure 2-3). The best examples of this type are the blown or oxidised grades used for roofing purposes.
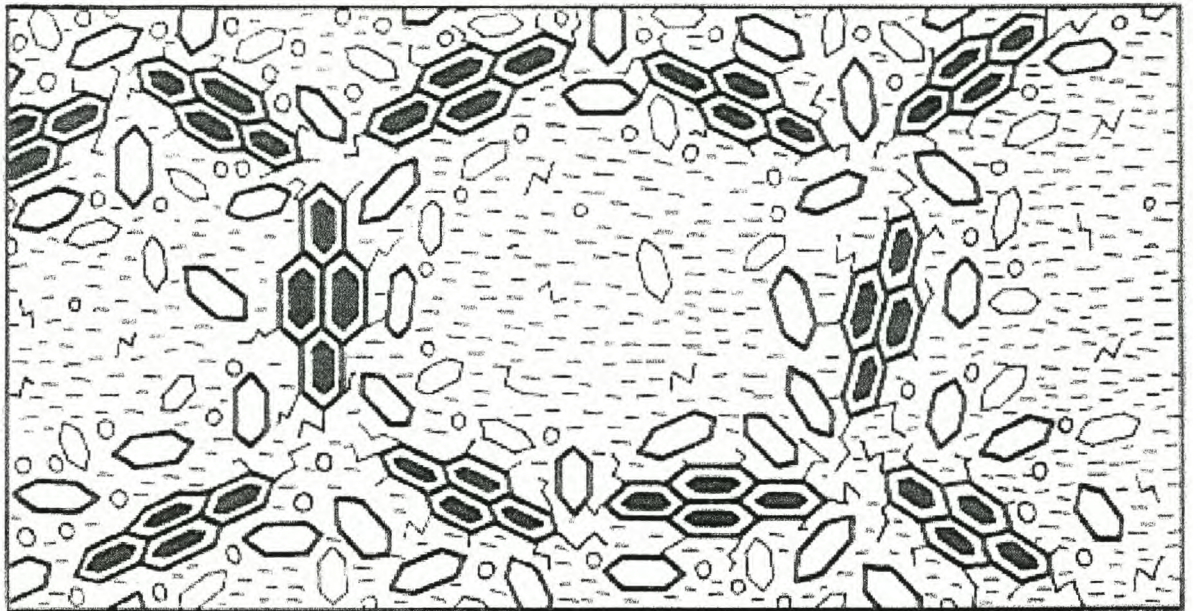
Figure 2-3: "Gel"-type bitumen structure [7].

In general, most bitumens are of intermediate character.

The colloidal behaviour of the asphaltenes in bitumen is a result of aggregation and solvation. The viscosity of the system is influenced considerably by the degree to which the asphaltenes are peptised. These effects decrease with an increase in temperature. The GEL character of certain bitumens may be lost when they are heated to high temperatures. The viscosities of the saturates, aromatics, and resins depend on their molecular weight distributions – the higher the molecular weight, the higher the viscosity.

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The maltenes (continuous phase) imparts an inherent viscosity to the bitumen. This viscosity is increased by the presence of the asphaltenes (dispersed phase).

The ability of the maltenes to solvate the asphaltenes is decreased by the saturates fraction, because a high saturates fraction can lead to marked agglomeration of the asphaltenes. Thus, an increase in GEL character and a lower temperature dependence for bitumens not only results from the asphaltene content but also from the saturates content.

The rheological properties of a bitumen depends strongly on the asphaltene content. At constant temperature the viscosity of a bitumen increases as the concentration of asphaltenes is increased. The increase in viscosity is considerably greater than would be expected if the asphaltenes were spherical, non-solvated entities. This suggests that the asphaltenes can interact with each other and the solvating medium.

Asphaltenes are believed to be stacks of plate-like sheets formed of aromatic/naphtenic ring structures. The viscosity of a solution depends on the shape of the asphaltene particles. The size is important only if the shape changes as the size increases. At high temperature the hydrogen bonds holding the sheets and stacks together are broken. This results in a change in both the size and shape of the asphaltenes. The dissociation will continue until the limiting moiety, the unit sheet of condensed aromatic and naphtenic rings, is reached. Thus, viscosity decreases with an increase in temperature. When hot bitumen cools, associations between asphaltenes occur again to produce extended sheets. These sheets interact with the resins and aromatics and stacking occurs to form discrete asphaltene particles.

As bitumen cools, there is a marked increase in non-Newtonian behaviour. This is a consequence of the inter- and intra-molecular attractions between asphaltenes and other entities. When subjected to shear these extended associations will deform or dissociate in a way not adequately described by classical Newtonian concepts. This leads to the conclusion that, at ambient and intermediate temperatures, the rheology of bitumens is

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dominated by the degree of association of asphaltene particles and the relative amount of other species present in the system to stabilise these associations.

3 Bitumen Rheology

In this chapter the basic concepts of bitumen rheology will be explained. The reader is referred to Appendix E for a discussion of the basic concepts of rheology as it is used here.

3.1 Bitumen as a visco-elastic material in terms of Dynamic Mechanical Analysis

A material that has a time dependence in its elastic response to deformation is classified as a visco-elastic material.

When such a material is loaded, there is an instantaneous deformation followed by a continuous deformation or creep. When the load is removed part of the deformation is recovered instantly (elastic response); part of the deformation recovers after a while (delayed elastic response); and in some materials part of the deformation does not recover (viscous response). Figure 3-1 shows the deformation response of a visco-elastic material.
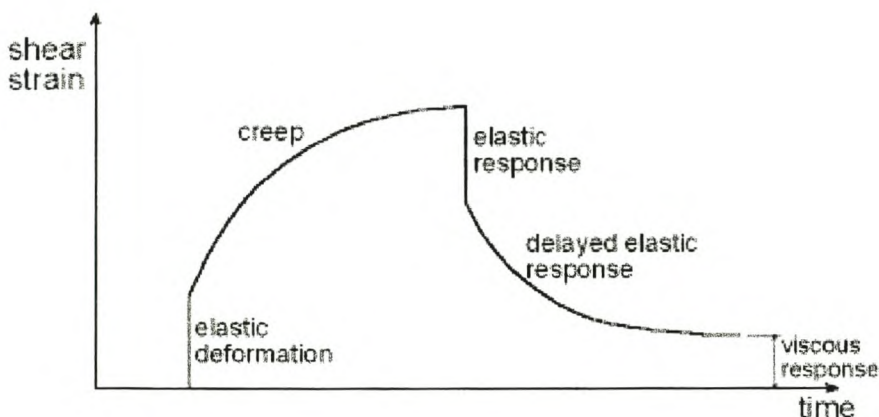
Figure 3-1: Deformation response of visco-elastic material when loaded (constant shear load).

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Bitumen is a material characterised by a time of loading and temperature dependence of the mechanical response to loading. Bitumen is also characterised by time-frequency inter-dependence. This means that for a certain temperature and frequency the material might have a certain response. At a different frequency and temperature it might have the same response. Thus, bitumen does not have unique behaviour at every temperature or frequency point in the real domain. This inter-dependency is characterised by the shift factor. The form of the shift factor is unique for every bitumen.

At any combination of time and temperature, the behaviour of the bitumen must be characterised by at least two properties: the total resistance to deformation and the relative distribution of that response between an elastic part and a viscous part.

Dynamic mechanical testing is the best way to characterise the behaviour of this class of material. Normally, the complex modulus (G*) and phase angle (δ) is measured in shear. This can be done through the application of a sinusoidal shear strain within the linear range.

The complex dynamic modulus is the primary response of interest in dynamic testing [3, 4]:

$$G^* = \frac{\tau(\omega)}{\gamma(\omega)}$$

The complex modulus represents the total resistance to deformation of the material, while the phase angle represents the relative distribution of this resistance between an in-phase component and an out-of-phase component. The phase angle is the measured lag between the application of the strain and the response of the material and it varies between 0° and 90° (purely elastic response to purely viscous response).

The in-phase component of the complex modulus (G' - storage modulus) is the elastic component and it is a measure of the recoverable energy stored in the material with every

loading cycle. The out-of-phase component ($G^{"}$ - loss modulus) is the viscous component and is a measure of the dissipated (lost) energy for every loading cycle.

The relative distribution of the elastic and viscous components, is a function of material composition, loading time and temperature. The relationship between the four parameters can be described graphically (Figure 3-2).
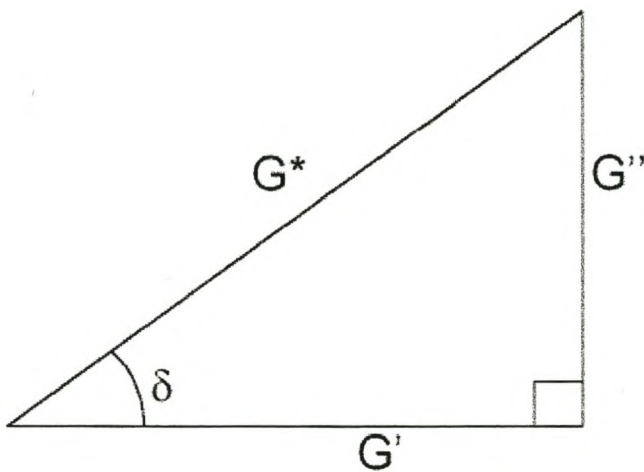
Figure 3-2: Relationship between complex modulus components and δ [1].

From Figure 3-2 the following relations can be written [3, 4]:

$$G^{'} = G^{*}(\omega)\cos\delta$$

$$G^{"} = G^{*}(\omega)\sin\delta$$

$$\tan\delta = \frac{G^{"}}{G^{'}}$$

The rheological properties are represented in the following ways:
a)   variation of G* and δ with frequency at a constant temperature (isothermal or "master" curve – Figure 3-3)
b)   variation of G* and δ with temperature at a constant frequency (isochronal curve)

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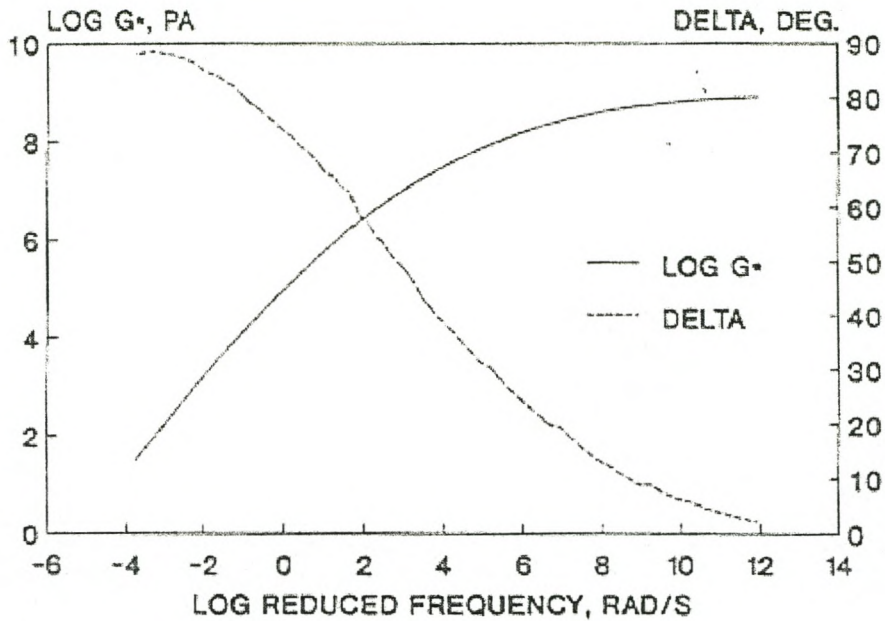
Figure 3-3: Typical master curve. [3]

The time of loading and temperature dependence of the material can be related using a temperature shift function – see Figure 3-4. There are two types of functions that are used for this purpose. [3]

a)   The William-Landel-Ferry (WLF) function:

$$\log a(T) = \frac{-C_1(T - T_r)}{(C_2 + T - T_r)}$$

b)   The Arrhenius function:

$$\ln a(T) = \frac{E}{R}\left(\frac{1}{T} - \frac{1}{T_r}\right)$$

21
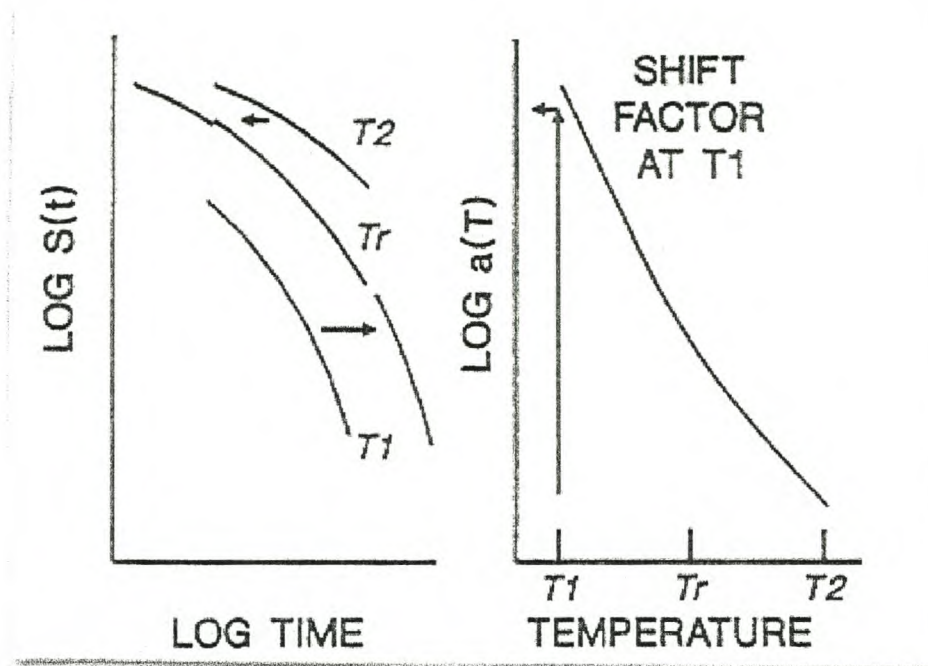
Figure 3-4: Graphical representation of shift factor. [3]

At low temperatures and/or high frequencies, bitumen approaches a limiting value of 1 GPa for G*. δ tends toward 0° for the same conditions. An increase in temperature (or decrease in frequency) results in a decrease in the value of G* while the value of δ increases. At high temperatures and/or low frequencies, the slope of the master curve approaches 1:1. This signifies viscous flow, as the rate of deformation is directly proportional to the applied stress. The bitumen behaves as a Newtonian fluid at low frequencies.

A decrease in the value of G* means a decrease in resistance to deformation. An increase in the value of δ means a decrease in elastic response. The rate of change of the properties (the form of the graph) is dependent on the bitumen composition. Some bitumens show a rapid decline, while others have a more gradual change.

At high temperatures and/or low frequencies, δ approaches a limiting value of 90°. This means that the material response is almost totally out of phase with the load and complete viscous behaviour (complete dissipation of energy) is approached. The bitumen tends

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toward Newtonian fluid behaviour and it is normally characterised in terms of dynamic viscosity. The value of G* varies due to the different consistency properties of the bitumens.

3.2 Bitumen properties related to pavement performance

The performance of bitumen is directly related to the temperature at which it is studied when the frequency is kept constant. This is due to the visco-elastic nature of the material.

There are five temperature zones of interest when looking at the performance of bitumen:

1.) mixing zone – T > 130 °C

2.) compaction zone – 80 °C < T < 150 °C

3.) high in-service temperature zone – 45 °C < T < 85 °C

4.) intermediate temperature zone – 5 °C < T < 45 °C

5.) low temperature zone – T < 5 °C

In the mixing and compaction temperature zones, the consistency of the bitumen is of importance. At the normal mixing temperatures a binder is needed which will adequately cover the aggregate and not run off the aggregate. At the compaction temperature the binder needs to be workable enough to allow adequate compaction. The cooling behaviour should also be such that there is adequate time for the compaction operation to be run.

At temperatures above 100 °C most bitumens behave as Newtonian fluids, thus a measure of viscosity is sufficient to represent the workability of the bitumen during mixing and compaction.

The main distress type in the high in-service temperature zone is rutting. In this zone the total resistance to deformation and the relative distribution of this resistance between an

elastic and viscous part is of importance and should be measured. Thus a measurement of G^* and δ should be made.

A viscosity measurement alone is not enough, since viscosity is measured with the assumption that bitumen has only a viscous response to loading.

A high G^* value is favourable, since this indicates a good resistance to deformation. A low value for δ is favourable, since this indicates a more elastic response for the bitumen to loading.

In the intermediate temperature range, the major distress type is fatigue damage. This is caused by repeated cycles of loading on the bitumen. For this distress type the value of G^* and δ is of importance, since the damage is related to the stress or strain developed in the pavement and how much of the deformation is recoverable.

A softer (low G^*), more elastic (low δ) binder is more favourable to resist fatigue damage. This is due to the fact that the stress developed for a given deformation is lower (low G^*) and the binder can recover more of its deformation (low δ). The resistance of a bitumen to fatigue damage, as with rutting, cannot be described by a single measure of viscosity or hardness.

The major distress type in the low temperature zone is thermal cracking. This is a phenomenon which is not very common in the South African environment.

Thermal cracking results from stresses developed in the bitumen due to shrinkage because of cooling. A lower G^* would be favourable with this distress type because less stress is developed during shrinkage. A higher value for δ would also be favourable because this indicates a more viscous response at lower temperatures – thus the stresses can be relieved through relaxation of the bitumen.

described as a function of temperature on one chart. Figure 3-5 gives an example of a BTDC.
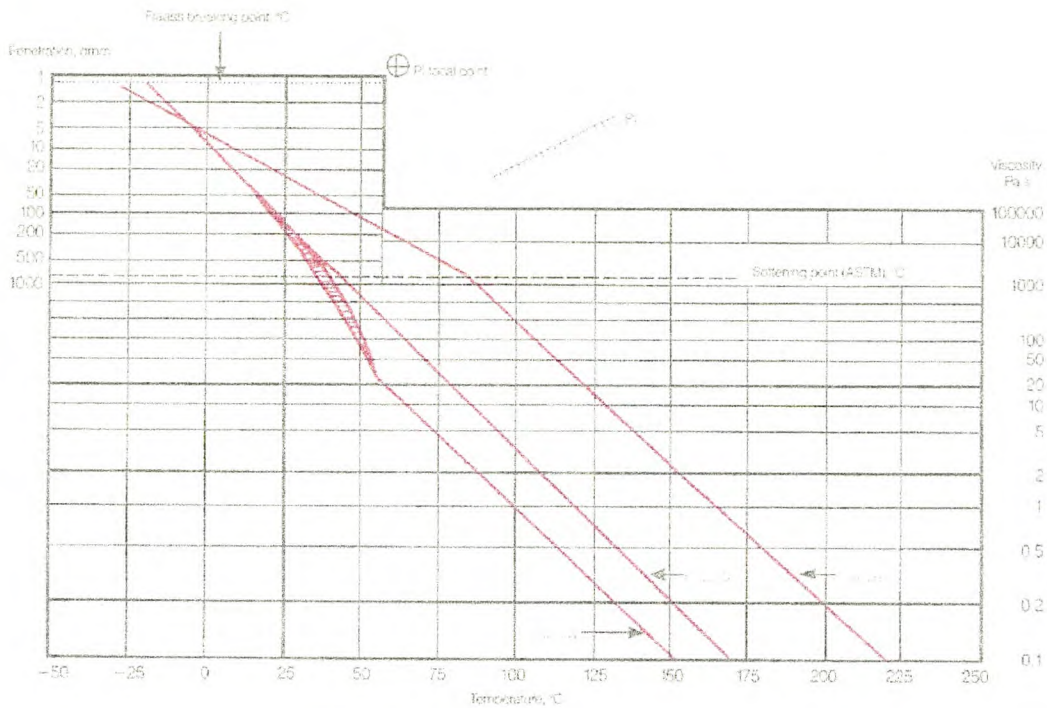
Figure 3-5: Example BTDC. [7]

The BTDC can be used for the characterisation of penetration grade bitumen. The temperature/viscosity relationship can be derived from the BTDC with only the softening point and penetration values known. However, the BTDC cannot be used to characterise polymer modified binders.

Another useful, empirical tool that can be used to characterise penetration grade bitumen, is Van der Poel's Nomograph (Figure 3-6). The nomograph can be used in conjunction with the BTDC to estimate the stiffness of different bitumens. It is possible to predict bitumen stiffness to within a factor of 2. [7]
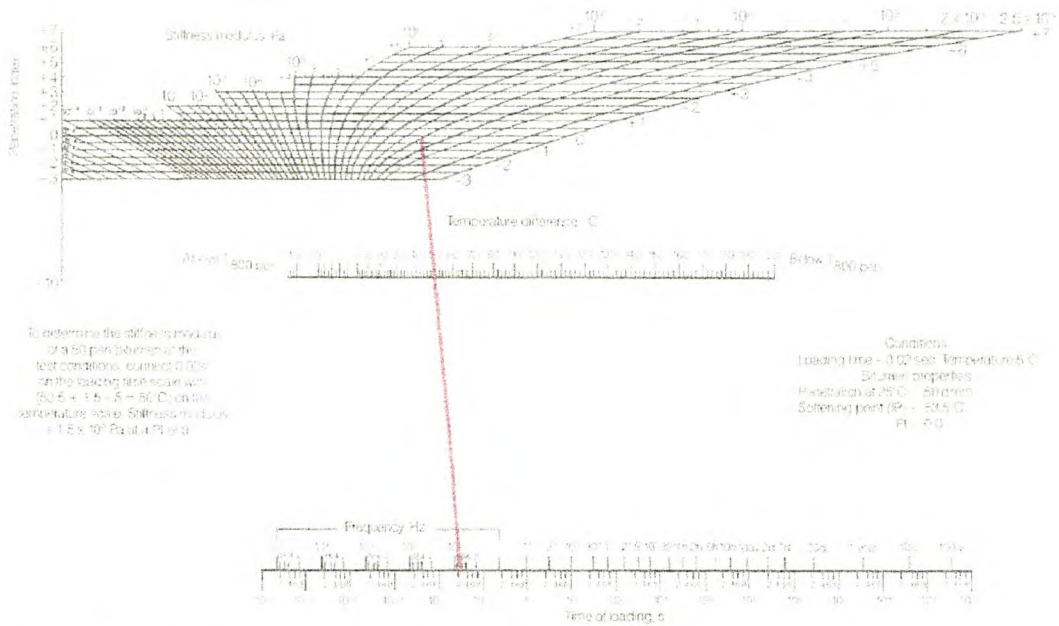
Figure 3-6: Van der Poel nomograph for the estimation of penetration grade bitumen stiffness. [7]

There are several problems related to the empirical measurement of bitumen properties. The following is a list of a few of them:

- These measures cannot be expressed in fundamental engineering units. Thus they cannot directly be related to any of the rheological properties of bitumen.
- They also do not give an indication of the relative distribution of the elastic and viscous response of the bitumen at the test temperature. The frequency dependence of the bitumen is not considered in the measurements.
- The three empirical measures have different loading modes, loading rates and temperatures. Thus, they cannot be combined directly to estimate rheological properties of the bitumen.

Over the years, attempts have been made to correlate the empirical measures with fundamental rheological properties.

Another phenomenon that should be kept in mind when looking at performance is ageing. Ageing of the binder results in a higher G^* and lower δ at all temperatures. For rutting this is a favourable situation, but not for thermal cracking. For fatigue the increase in G^* is not favourable, but the decrease in δ is.

3.3 Conventional Bitumen Characterisation

In the previous section bitumen characterisation and the visco-elastic response to loading was described in terms of dynamic mechanical analysis. This type of characterisation is a fairly recent development (over the last 20 years) in bitumen technology and is still regarded as a high tech method.

The Strategic Highway Research Program (SHRP) has done much for the promotion of dynamic mechanical analysis, because the properties measured can be related directly to pavement performance. There is a move in the world towards performance related specifications and as such dynamic mechanical analysis is an invaluable tool.

For application in pavement problems a complete characterisation of bitumen is needed. The rheological characterisation by dynamic mechanical analysis is not an easy task. It is generally regarded as expensive, complicated and the material is difficult to handle compared to other visco-elastic construction materials. This has led to the simplification of measurement of bitumen properties.

Two groups of conventional measures will be discussed: the empirical measures and the viscosity measures.

The empirical measures are penetration, ductility and softening point. Heukelom developed the Bitumen Test Data Chart (BTDC) in the late sixties. [7] The BTDC was developed to enable penetration, softening point, Fraass and viscosity data to be

The penetration value attracted the most attention due to the ease of the test. The penetration value has been related to viscosity. There exist a few coefficient of viscosity formulae with the penetration value as basis of calculation. [1]

Ductility could not be related to a fundamental property. It has been related in general terms to other materials' properties. Ductility is considered by some as an indication of internal structure, while others showed that it is correlated with shear susceptibility. Another group tried to combine penetration and ductility and relate it to pavement performance. [1]

All these attempts to correlate empirical measures are lacking in that they cannot explain how a simple empirical measurement can describe the complex behaviour of bitumen.

The coefficient of viscosity is a fundamental material property. It is the ratio of shear stress to shear rate and is expressed in engineering units (Pa.s). There are two measures of viscosity that are generally used: absolute viscosity and apparent viscosity.

The absolute viscosity is a measure of Newtonian fluids whose properties are independent of the loading rate or stress level. Bitumen exhibits Newtonian behaviour at high temperatures or very low shear rates.

At intermediate temperatures bitumen does not have Newtonian properties and cannot be described by an absolute viscosity. Many researchers measured absolute viscosity at high temperatures and tried to relate this to low-temperature properties. [1]

Absolute viscosity cannot be used to characterise polymer modified bitumens, because for the majority of the polymer modified bitumens Newtonian behaviour does not exist due to the interaction of the bitumen with the modifier.

For bitumen with its visco-elastic behaviour at intermediate and low temperatures, viscosity is not an absolute value. It varies with the shear rate and shear stress at which it

is calculated. The viscosity calculated in this manner is known as an apparent viscosity. A certain shear rate, shear strain or shear stress is selected and the viscosity is calculated.

The problem with the apparent viscosity measurements is that a constant strain rate is needed to calculate the viscosity. Due to delayed elasticity it takes a long time to reach the constant strain rate, but it will eventually reach a constant value. The time required to reach the constant value is dependent on the temperature, material properties and stress level. It is easy to underestimate the time to reach constant strain rate.

It can be seen that none of the conventional methods discussed – the empirical methods and the viscosity measures – adequately describe bitumen's response to loading. The empirical methods do not give information that can be correlated directly with rheological properties which is useful to determine bitumen response and thus performance. The viscosity measures, on the other hand, could be correlated with resistance to deformation, but there is no indication of the relative distribution of this resistance to deformation between an elastic and viscous part.

Thus, the conventional methods of characterising bitumens cannot be used directly to determine performance related specifications. This is especially important when polymer modified binders are studied.

3.4 Polymer Modified Bitumen Rheology

It has been generally reported that the modification of bitumen by polymers enhances the properties of the base bitumen. The desired effect of the addition of polymer modifiers is that of increasing the molecular association (agglomeration). This higher molecular association may provide more elastic stability at higher temperatures.

An increase in the molecular association may also result in a decrease in the loss tangent value ($\tan(\delta)$) at higher temperatures. This parameter is used by some researchers to

characterise a binder's rut resistance, since it can also be written as G''/G' – the ratio of the loss modulus to the storage modulus. This result is confirmed by the increased rut resistance reported for most polymer modified bitumen when compared to the conventional bitumen from which they were produced.

The loss modulus is a measure of the binder's viscous response, while the storage modulus is a measure of the binder's elastic response. They are not called the viscous and elastic moduli respectively, because both contain a part of the delayed elastic response.

At lower temperatures there does not seem to be a marked difference in loss tangent between the conventional and modified bitumen.

The frequency-temperature equivalence of bitumen is a unique and important characteristic. It is a very important tool when master curves and isochronal curves are being constructed. The usefulness of the isochronal curve, at a frequency close to the traffic loading time, in assessing a binder's response to loading, have been mentioned earlier. Thus, the frequency-temperature equivalency is a fundamental and important characteristic of bitumen.

It has been reported that the modification of bitumen by polymers may result in a binder for which the frequency-temperature equivalence cannot be confirmed. This may be due to the change in degree of crystallisation that the polymer may experience with a change in temperature.

As stated earlier the phase angle tends toward 90° at high temperatures. Modification of bitumen by polymers leads to the result that the loss angle no longer monotonously tends toward 90° at long loading times/high temperatures (Figure 3-7). The binder has non-Newtonian behaviour across the full spectrum of testing temperatures.
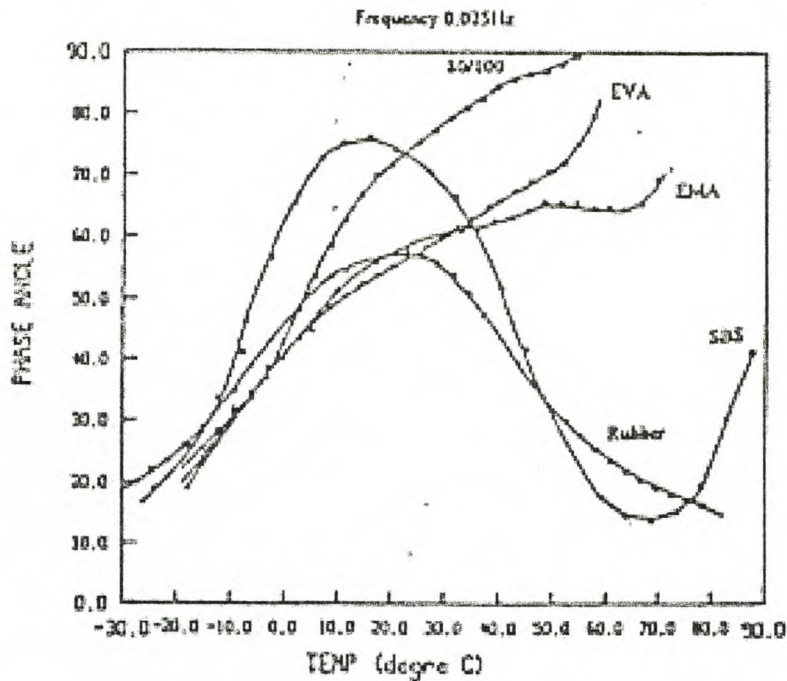
Figure 3-7: Influence of polymer modification on phase angle. [2]

Being able to relate the binder rheology to pavement performance is the object of specifying the binder's characteristics. In this regard, the performance of a polymer modified bitumen will be discussed with respect to rutting resistance and fatigue strength.

There are two schools of thought about the rheological characteristic that should be studied when looking at rutting. The one school focuses on the viscosity of the binder, while the other school focuses on the complex modulus of the binder.

Many authors do not accept viscosity as a meaningful parameter for characterising rut resistance, as they feel that the influence of polymer modification is not adequately qualified by viscosity measures. On the other hand, viscosity has been correlated well with the rate of rutting. Viscosity was discussed in detail in the section on conventional characterisation of bitumen. The problem with viscosity measures and polymer modified bitumen is that the interaction of the polymer with the bitumen molecules results in a binder that do not show Newtonian behaviour at elevated temperatures.

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The characteristic preferred by the authors who do not look at viscosity is the "lossability", J" = J*sinδ. J* is known as the creep compliance of the binder and is approximately equal to the inverse of the complex modulus (J* ≈ 1/G*). Thus, the lossability can be written as: J" = sin δ/G*. The lossability, in the complex modulus form, is the criterion that was adopted by the SHRP researchers to characterise the rut resistance of a binder. This parameter was arrived at by considering the dissipated energy during a load cycle. The dissipated energy during a cycle is:

$$w = \pi \sigma \varepsilon \sin \delta$$

This equation can be rewritten as:

$$w = \pi \tau \gamma \sin \delta$$

with

$$\tau = \gamma G$$

Thus, the equation can be rewritten again to obtain:

$$w = \frac{\pi \tau^2}{\left(\dfrac{G}{\sin \delta}\right)}$$

The dissipated energy during a load cycle has to be minimised in order to minimise the permanent deformation of the bitumen. Thus, the term $\dfrac{G}{\sin \delta}$ has to be maximised to minimise the dissipated energy during a load cycle. Thus, a minimum value for this term was specified by the SHRP researchers.

At 60°C, sinδ ranges between 0.9 and 1 for conventional binders. This would imply almost full viscous response to loading. For a polymer modified bitumen the value of sinδ at 60°C could be as low as 0.7. Thus there is a marked difference in the sinδ values and this is in favour of the polymer modified bitumen.

It is worth noting that the plot of the value of δ with respect to either temperature or frequency is different for a polymer modified bitumen when compared to a conventional bitumen. With the conventional bitumen the value of δ tends monotonously towards 90° in a s-curve shape. Polymer modification might lead to a disturbed s-curve (Figure 3-7).

The other performance parameter is the fatigue strength of the binder. It has been shown that the durability of a mix for a given strain in a constant deformation test will be greater the lower the value of the complex modulus at the testing temperature/frequency. Experience has also shown that in the constant deformation test, the more elastic (lower sinδ) binders performed better.

The SHRP researchers decided on the G*sinδ parameter for the specification purposes. They specified a maximum limit for this parameter. This parameter is valid for both conventional and polymer modified bitumen. To explain the logic used to arrive at this parameter, the disspated energy during a load cycle is again considered. In this case, the strain is kept constant during the test and the dissipated energy equation can be written as:

$$w = \pi \gamma^2 G \sin \delta$$

Here, the term $G \sin \delta$ has to be minimised in order to minimise the disspated energy during a load cycle. For this reason, a maximum value was specified for this term by the SHRP researchers.

The following should be noted with regard to fatigue strength: When looking at a thin layer the imposed strain in the asphalt layer is constant and given by the deformation of

the rest of the structure. Thus, the strain is approximately constant irrespective of the binder properties. For a thick layer, on the other hand, the imposed strain is a function of the binder stiffness – this means that for the same stress conditions in a thick layer a stiffer binder will have a lower strain compared to a less stiff binder.

Thus the specification of $G^*\sin\delta$ with a maximum value, is only valid for thin layers on deformable surfaces (the test is performed in strain controlled mode). For thin layers a lower stiffness (G^*) and more elastic behaviour ($\sin\delta$) are favourable. Most elastomers have the ability to lower the values of both G^* and $\sin\delta$.

4 Test Programme

In this chapter the test programme that was followed will be described. The first part of the chapter will focus on the materials that were used during this study. The second part of the chapter will give a short description of each test method. In the third part the test programme will be described.

4.1 Materials

This part of the chapter gives information on the materials that were used in this rheological study.

4.1.1 Binders

Three bituminous binders were used in this study. These were:
- 60/70 penetration grade bitumen
- SBS modified 60/70 bitumen
- Bitumen-Rubber

The 60/70 penetration grade bitumen was used as the base bitumen from which the SBS modified binder was produced.

These binders were chosen as it was found from interviews that these binders were proposed most often in Southern Africa when solutions had to be found for problems such as rutting on roads with high traffic loads.

In the following sections the certification properties of the binders are reported.

4.1.1.1 60/70 penetration grade bitumen

In the table below the certification properties of the 60/70 binder is reported.

PROPERTY	UNITS	LIMITS	RESULTS	TEST METHOD	
				ASTM	OTHER
Viscosity @ 60 °C	Pa.s	120-250	214	D4402/92	
Viscosity @ 135 °C	Pa.s	0.22-0.45	0.40	D4402/92	
Penetration @ 25 °C/100g/5 sec	0.1mm	60-70	62	D5/86	IP 49
Softening Point (Ring & Ball)	°C	46-56	48	D36/89	
Ductility @ 15 °C	cm	100 min	>150		DIN52013/85
Spot Test	% Xylene	30 max	25		AASHO-T102/90
AFTER RTFOT					
Mass Change	Mass %	0.5 max	+0.07	D2872/88	
Viscosity @ 60 °C	% Original	300 max	164%	D4402/92	
Ductility @ 15 °C	cm	10 min	>100		DIN52013/85
Softening Point (Ring & Ball)	°C	48 min	53	D36/89	
Retained Penetration	% Original	55 min	74	D5/86	IP 49
Increase in Softening Point	°C	9 max	5	D36/89	

Table 4-1: Binder properties 60/70 bitumen.

4.1.1.2 SBS modified 60/70 binder

The same certification tests were performed on the SBS modified binder that were performed on the 60/70 bitumen and the results are reported in Table 4-2.

PROPERTY	UNITS	RESULTS	TEST METHOD	
			ASTM	OTHER
Viscosity @ 60 °C	Pa.s	-*	D4402/92	
Viscosity @ 135 °C	Pa.s	0.81	D4402/92	
Penetration @ 25 °C/100g/5 sec	0.1mm	43	D5/86	IP 49
Softening Point (Ring & Ball)	°C	57	D36/89	
Ductility @ 15 °C	cm	76		DIN52013/85
Spot Test	% Xylene	25		AASHO-T102/90
AFTER RTFOT				
Mass Change	Mass %	+0.10	D2872/88	
Viscosity @ 60 °C	% Original	-*	D4402/92	
Ductility @ 15 °C	cm	-*		DIN52013/85
Softening Point (Ring & Ball)	°C	57	D36/89	
Retained Penetration	% Original	67	D5/86	IP 49
Increase in Softening Point	°C	0	D36/89	
* Tests could not be performed on this binder				

Table 4-2: Binder properties of SBS modified 60/70 binder.

4.1.1.3   Bitumen-Rubber

The binder properties of the Bitumen-Rubber are reported in Table 4-3.

PROPERTY	UNITS	LIMITS	RESULTS	TEST METHOD	
				ASTM	OTHER
Softening Point (Ring & Ball)	°C	46-56	48	D36/89	
Flow	mm	20-70	33		
Compression Recovery: 5min	%	85-100	92		
1 hour	%	70-95	89		
4 hours	%		74		
24 hours	%		54		
Resilience Air	%	10-35	27		
Resilience Oven	%	10-35	27		

Table 4-3: Binder properties of Bitumen-Rubber.

The softening point for the Bitumen-Rubber seems low. However, it must be kept in mind that this product is made with bitumen for which a penetration value of 80 (± 5) is the target value for the base bitumen. Thus, it might lead to a lower softening point than expected.

4.1.2 Filler

The filler that was used in this study is a mineral filler from the Western Cape. The filler was obtained from baghouse fines and is the product of aggregate crushing from Hornfels – the prevalent source of aggregate in the Western Cape.

The particle distribution is given in Table 4-4. [6]

Sieve Size (mm)	% Passing
0.2	100
0.1	98
0.08	97
0.06	87
0.04	72
0.02	52
0.01	35
0.008	31
0.006	25
0.004	18
0.002	10

Table 4-4: Particle distribution of filler used in this study.

4.1.3 Binder/Filler systems

Binder/Filler mixes were made from the three binders and the filler described in the previous two sections. These mixes were tested under the same conditions as the binders to characterise the influence of the filler on the binder.

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A filler content of 50% bulk volume filler was used. It was found that the filler that was used in this study had optimum filler content of between 50% to 55% bulk volume. [6] This translates into a filler/binder ratio of 1.1 by mass for the 50% bulk volume filler case.

The filler/binder ratio is calculated as follows:

- The masses of filler and binder in a binder/filler mix are calculated from the phase diagram (Figure 4-1). The filler used in this study has 42% Rigden Voids and a bulk density of 2 709 kg/m^3.
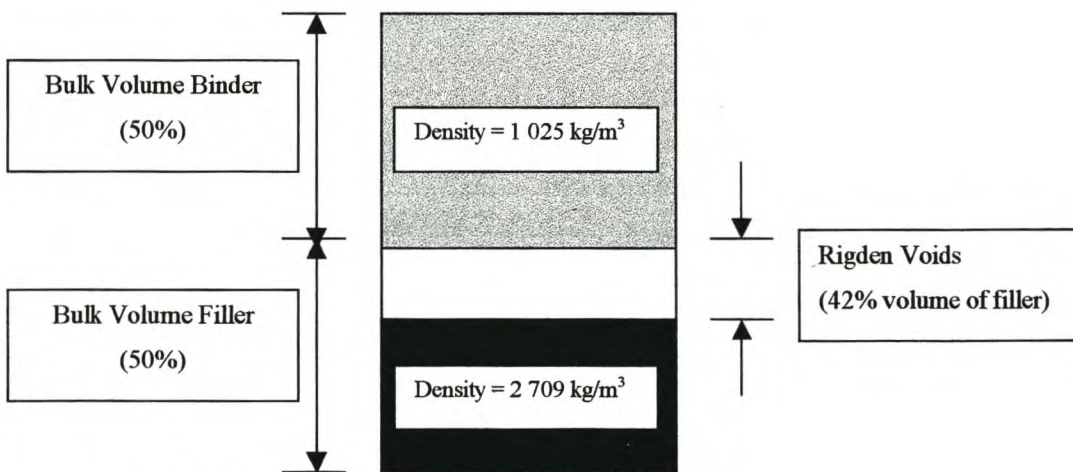
Figure 4-1: Phase diagram of binder/filler mix.

- Mass of filler:

$$MassFiller(g) = 0,5 \times (1 - 0,42) \times 2709 = 786g$$

- Mass of binder:

$$MassBinder(g) = (1 - [0,5 \times (1 - 0,42)]) \times 1025 = 728g$$

- The filler/binder ratio (mass/mass) is therefore 1,08 or approximately 1,1.

The same filler/binder ratio was used for all three binder/filler systems.

4.2 Test Methods

Two rheometers were used in this study. These were:

- Sliding Plate Rheometer
- Dynamic Shear Rheometer

A number of tests were performed with these machines and will be discussed in the following sections.

4.2.1 Sliding Plate Rheometer

One type of test was performed on the sliding plate rheometer: the creep test. A short description of the purpose of this test is given below.

4.2.1.1 Creep Test

The creep test has been designed originally to be performed on bituminous materials such as bitumen and bitumen/filler mixes (mastic). The methodology of this test is to load a sample of the test material in shear and to record the creep curve during the test.

The objective of the test is to reach a constant shear rate at the end of the test. It is assumed that only viscous deformation is present when the shear rate is constant. Using the constant shear rate and the applied load, the viscous component of the material can be calculated. Thus, comparisons can be made between different materials based on the viscous components of the materials for the given load conditions.

From the results of the creep tests, shear susceptibility lines can be drawn under certain conditions. The lines for the different materials are then used to compare the behaviour of

the different materials. A detailed description of shear susceptibility is given in the next section.

4.2.1.2   Sample Preparation, Test Set-Up and Methodology

A sample tested in this apparatus consists of two aluminium plates (20 x 30 mm) with a film of binder sandwiched in between. The film thickness can be varied – the film thickness used in this study varied between 3 and 5 mm.

The samples were prepared in a mould developed in a previous study on Bitumen-Rubber. [5] Figure 4-2 shows the mould used. An aluminium plate was inserted in the bottom plate holder, after this the spacers followed and finally the top plate holder. A quantity of hot bitumen was poured on the bottom plate and the top plate was inserted. The top plate was then pressed down firmly until it touched the sides of the spacer plates. After the sample was removed from the mould, it was placed in a freezer for a few minutes (minimum 10 minutes, maximum 30 minutes). This was done to allow for the easy trimming of excess binder from the sides of the sample.

The aim was to test samples on the same day of preparation. However, samples that could not be tested on the same day were stored overnight in a refrigerator at 5°C and tested the next day.

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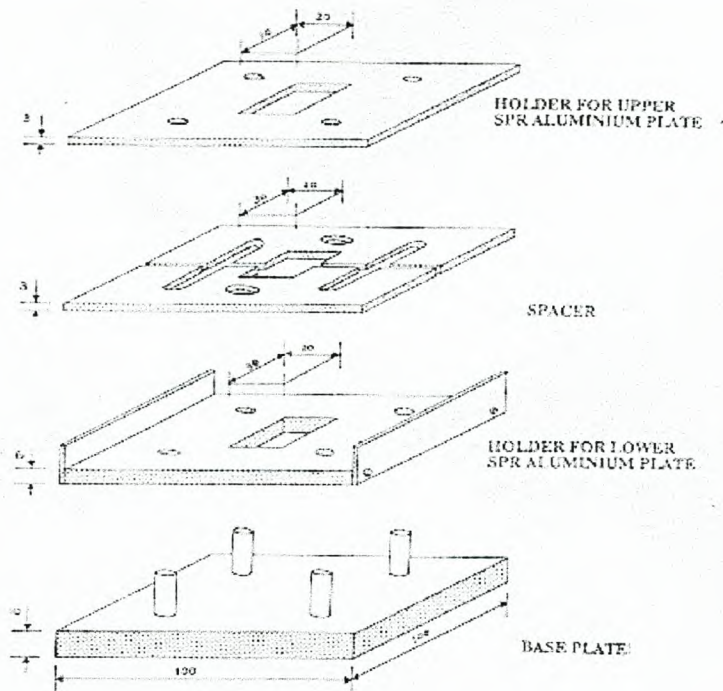
Figure 4-2: Mould for sample preparation [5].

Prior to testing, the samples were immersed in a thermostatically controlled water bath that had been brought to the test temperature. The samples were allowed to achieve temperature equilibrium (minimum of 15 minutes). The temperature of the water bath can be accurately controlled above room temperature. For tests below room temperature, use was made of a temperature chamber for temperature control.

The following equipment were used in this study for the creep tests:

a) Enraf Nonius Delft Sliding Plate Rheometer and Electronic Unit
b) Hewlett Packard DCDT/LVDT
c) Kipp & Zonen strip-chart recorder
d) Thermostatically controlled water bath

When the samples were ready, the first sample was placed in the main frame. Both aluminium plates rest on the specimen support at this stage. The back plate is clamped to the frame (See Figure 4-3). The specimen support is released by means of an electro-magnet and the front plate is allowed to displace freely in the vertical direction.
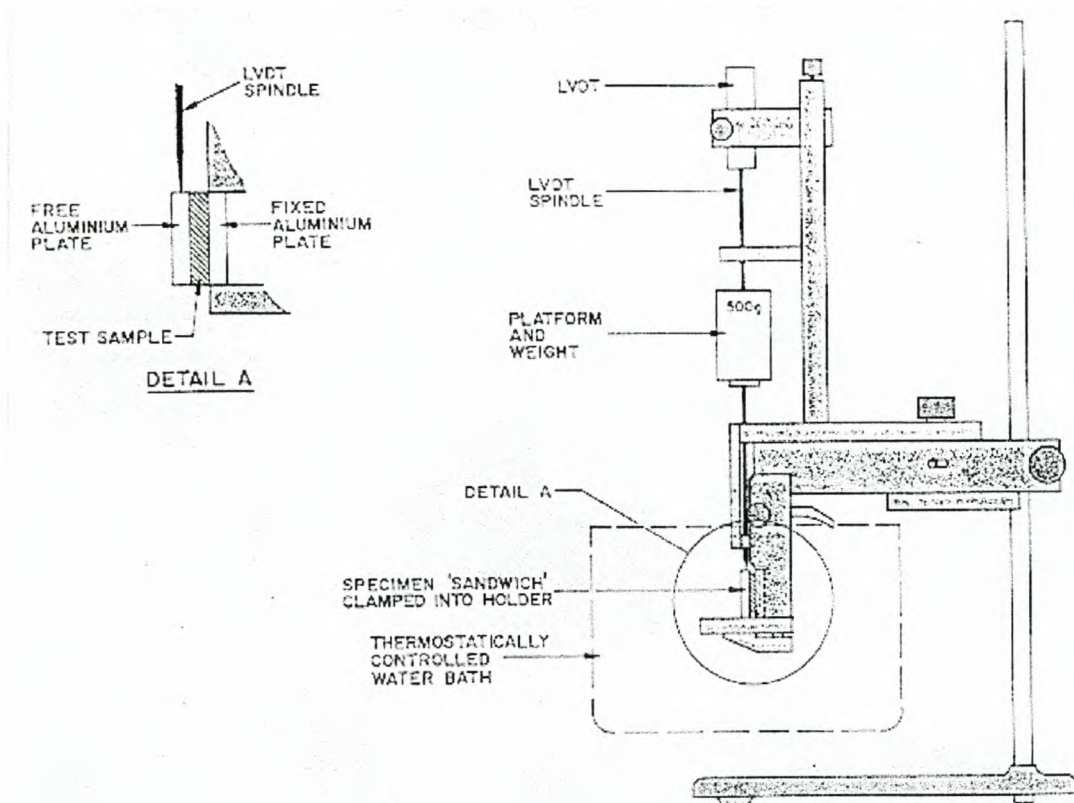
Figure 4-3:Sample configuration in main frame.[5]

The sample is subjected to a constant vertical load and the vertical deformation versus time is recorded by means of a strip-chart recorder. The load is applied by means of a steel needle with a platform, on which the load is placed. A load adaptor is fitted to the front plate. Figure 4-4 gives a qualitative view of the loads applied to the sample when loaded with and without the load adaptor fitted.
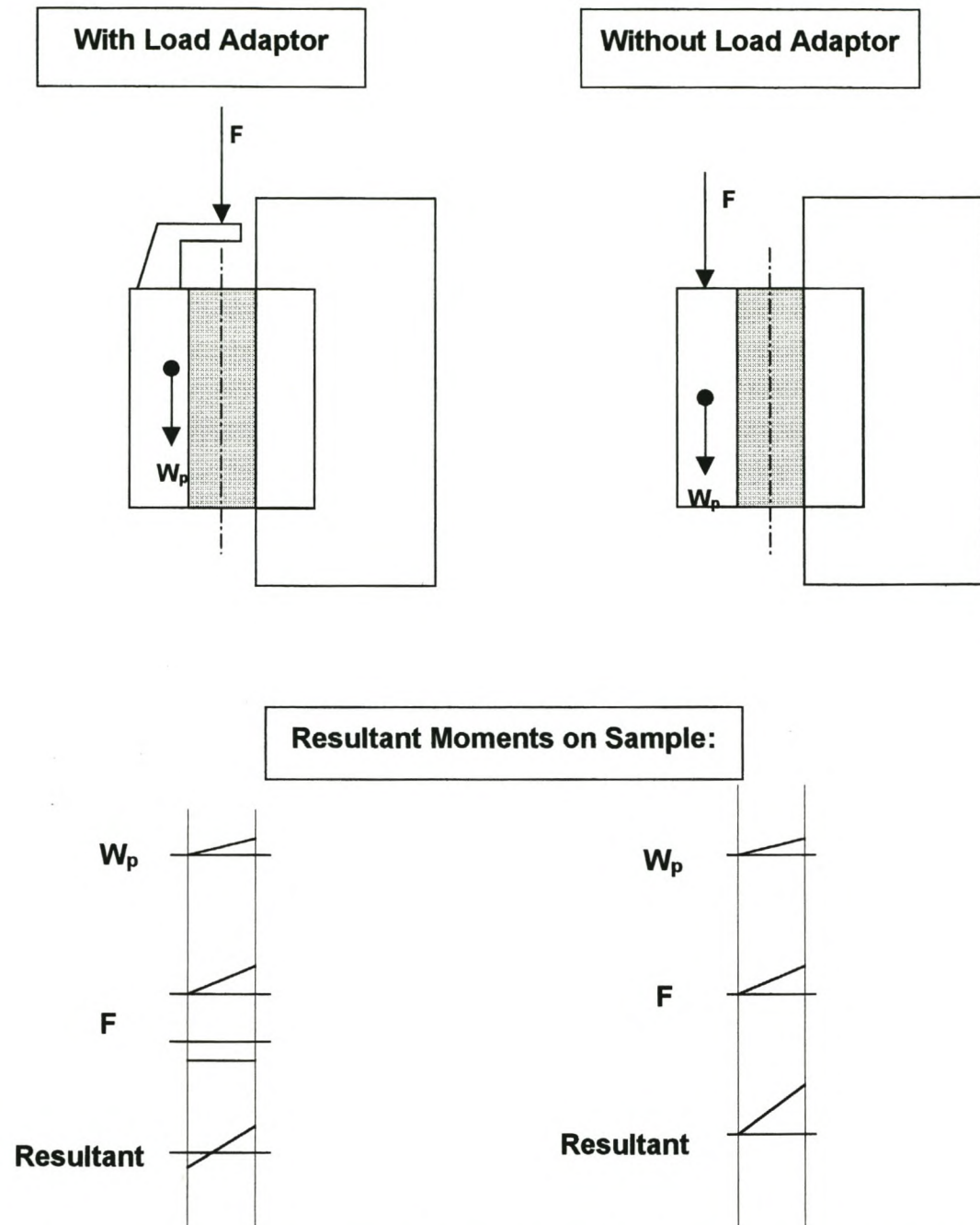
Figure 4-4: Resulting moments from applied loads on sample in SPR. (Counter clockwise moment is positive.)

It can be seen on Figure 4-4 that it is desirable to apply the load through the load adaptor, since this results in a favourable moment distribution over the sample. When the load adaptor is not fitted to the sample, the resulting moment will tend to rotate the sample. In

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the case where the adaptor is fitted, the tendency to rotate the sample is much less compared to the case without the adaptor.

The sample deformation is measured by means of a linear variable displacement transducer (LVDT). The signal received from the LVDT passed through a signal amplifier before it was recorded on the strip-chart. The signal can be amplified to a number of levels (called sensitivities) and the different amplification levels had to be calibrated. The calibration was performed by placing a micrometer in the mainframe. The needle used to apply the load was displaced by an appropriate amount and the displacement recorded. In this way the calibration factors for every sensitivity were calculated, e.g. at sensitivity 4: 1 mm of sample displacement is equal to 48.2 mm on the strip-chart. Table 4-5 gives the results of the calibration.

SENSITIVITY	CALIBRATION FACTOR
1	11.5
2	23.6
4	48.2
8	97.5
16	192.1
32	386.7
64	773.1
128	1545.8
256	3059.2

Table 4-5: Calibration factors calculated for the SPR.

Figure 4-5 gives an example of a typical response curve and the formulas used to do the calculations of the results.
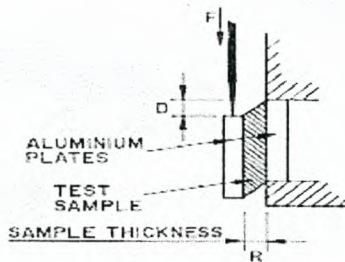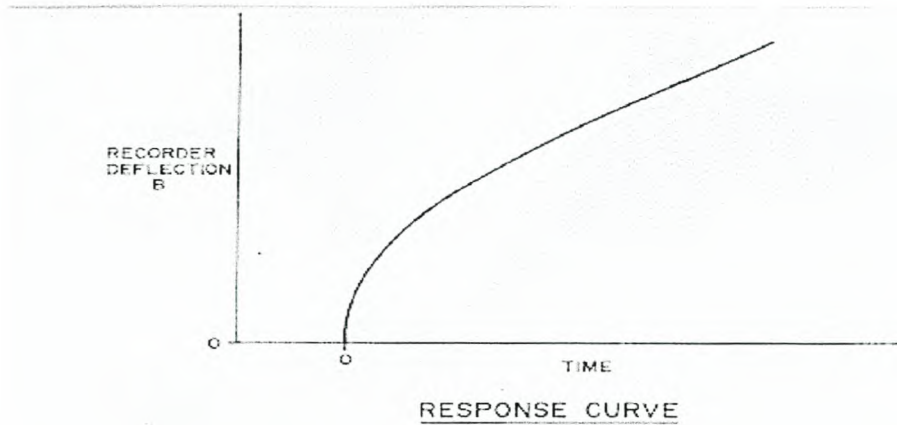
45

Figure 4-5: Calculations for the Sliding Plate Rheometer (after [5]).[1]

The aim of the creep test is to achieve a constant shear rate at the end of the test – this would give an indication of the viscous response of the material to the load it is subjected to. If plots are made of the shear stress vs. shear rate obtained from tests with different stress levels lines can be drawn that gives an indication of how much permanent deformation can be expected. This is a result of the mechanism studied with the creep test – the material is assumed to be under viscous flow conditions when the shear rate reaches a constant value.

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[1] Equation 6 on Figure 4-5 should read: $S = 2 \times (1 + \mu) \times G$

The creep test can be regarded as a viscosity test at ambient temperatures for the bitumen. This follows from the fact that there is no more elastic or delayed elastic response from the material when the shear rate is constant (Figure 3-1). Newton's law applies when the shear rate is constant at the end of the test:

$$\tau = \eta \, \dot{\gamma}$$

Thus, it is clear that the shear rate obtained for different binders at the same stress level is an important indicator of a binder's resistance to flow (permanent deformation). The lower the shear rate, the higher the viscosity and vice versa. A higher viscosity indicates a higher resistance to flow, while a lower viscosity indicates a lower resistance to flow.

Shear susceptibility lines can be drawn from the results of creep tests at different stress levels for the same binder. The stress level is plotted vs. the strain rate on a log-log scale and a line is fitted to the data. The slope of the line is defined as the shear susceptibility of the binder. The binder is said to be less shear susceptible the steeper the slope of the line. Shear susceptibility can be defined as the relative increase in shear rate with an increase in shear stress – the higher the increase with shear stress, the flatter the slope of the shear susceptibility line. Figure 4-6 describes the concept.

log(Shear stress)

Less shear susceptible

More shear susceptible

log(Shear rate)
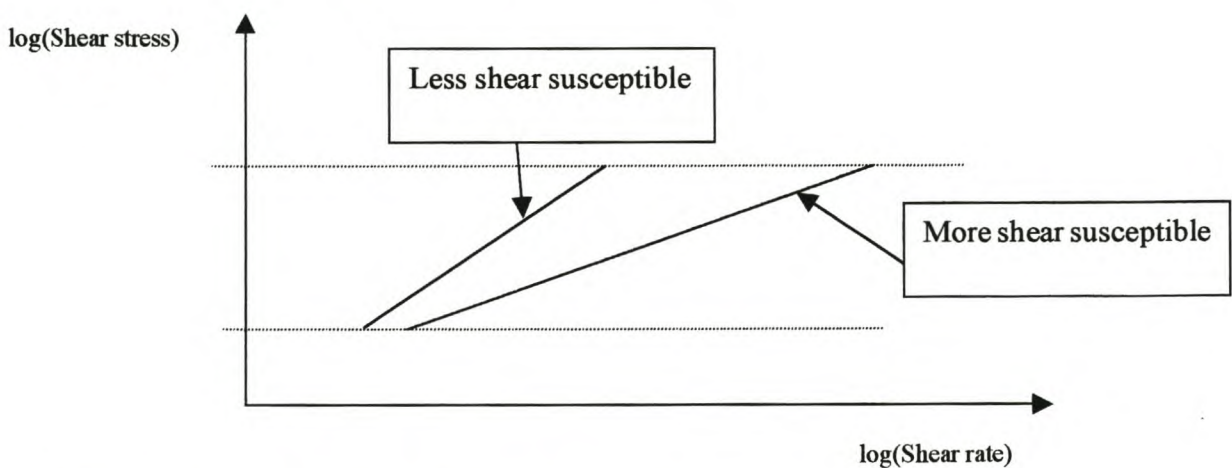
Figure 4-6: Conceptual description of shear susceptibility.

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4.2.1.3   Modification of SPR equipment

The SPR equipment was modified so that tests could be performed at the highest possible temperatures. The modification was made because the load at higher temperatures had to be much lower than those normally used. The unmodified system exerted a load of ± 39 grams on the samples (referred to as the "system load"). It was felt that this load is too high when tests were done at higher temperatures – this would lead to a situation where unsatisfactory results would be obtained.

The problem of the system load was solved in the following way: A counterweight was connected to the LVDT shaft by a piece of very light fishing line. This fishing line was run over a single pulley. Thus, the system load was counteracted by the counterweight.

The counterweight system was designed to have minimum friction in it. If the system load was counter acted by the exact load on the other end of the fishing line, this would mean the needle would be in an unstable equilibrium. The slightest jerk in the system would then cause the needle to lose contact with the load adaptor. Thus, the displacement would not be recorded. For this reason the counterweight was chosen so as to balance the system load with a resultant load in the direction of the system load of ± 2 grams. Figure 4-7 shows the counterweight system.
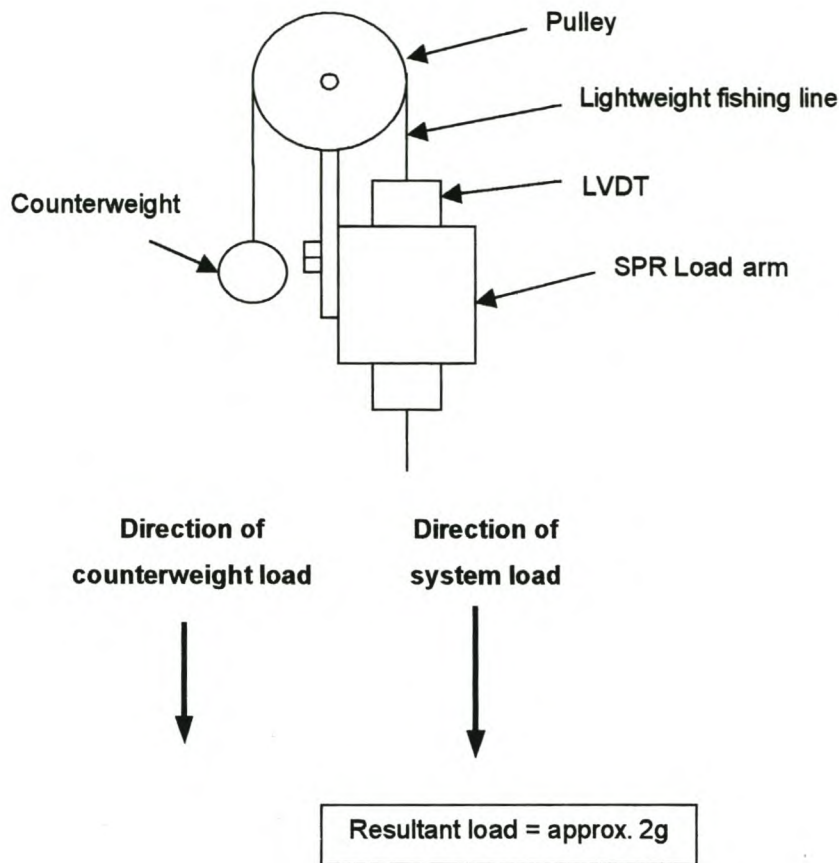
Figure 4-7: Schematic representation of SPR modification.

The resultant load was deemed low enough together with the front plate and load adaptor's weights (± 14 grams) to get accurate measurements with the SPR at high temperatures.

Another property of bituminous binders that can be studied when the system load is counteracted is the elastic and delayed elastic response of the binders. The system load before the modification was too high and the samples kept on displacing under the system load. After the modification of the equipment, it was possible to obtain curves showing the elastic recoil of the binder samples after unloading of the sample. However, the elastic recoil of the binders was not studied in this project. It should also be kept in mind that due to the arrangement of the test set-up (vertical configuration) there is a residual load on the sample that cannot be counteracted with the counterweight system (own weight, front aluminium plate, load adaptor). Due to these restrictions on the vertical test

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set-up, it might be worth the effort to investigate the development of a horizontal set-up to study creep behaviour of bituminous materials.

4.2.2 Dynamic Shear Rheometer

The dynamic shear rheometer is a very versatile machine. Dynamic testing of bituminous binders can be performed over a wide range of testing temperatures. Both stress and strain controlled tests can be performed with this machine. Types of tests that can be performed include rotational and oscillatory tests. The machine that was used for this study is described in detail in the next section.

The tests used in this study include both rotational and oscillatory tests. The tests that were performed are:

- Strain sweeps – Purpose: obtain linear visco-elastic limits
- Frequency sweeps – Purpose: obtain Master Curves, Black Diagrams
- Viscosity tests – Purpose: mixing and compaction parameters
- Creep tests – Purpose: comparison with SPR results

Each of these tests will be described further in the following sections.

4.2.2.1   Equipment used

Use was made of a state of the art dynamic shear rheometer manufactured by Paar Physica of Germany. The system consists of the following components:

- UDS 200 Rheometer and Control unit
- TC10 Temperature Control Unit
- US 200 Testing and Analysis Software

The system can be run in either stress controlled or strain controlled mode.

The system features automated gap control – this means that the system determines the zero gap at a given temperature and then controls the set gap with the zero gap as reference point. The advantage of this is that the operator does not have to watch the gap setting during testing as the system does this automatically. It is important that during the test the gap be maintained at the defined value for the test. This ensures that the strain is applied accurately during the test.

Another feature of the system is the pre-defined test types in the Testing and Analysis Software. [11] The operator chooses the type of test that is going to be run and a workbook is automatically opened that contains the relevant components for testing and analysis. The operator just has to change the relevant test parameters. Types of tests that can be chosen include: Flow Tests, Oscillation Tests and Standard SHRP tests.

The system uses an air bath for temperature control of the sample. Temperature can be controlled accurately to 0,1 °C. At temperatures below room temperature, liquid nitrogen is used to cool the air.

4.2.2.2   Sample Preparation

The binders used in the research project were poured into one litre tins at the start of the project. This was done to ease the handling of the binders.

When the part of research aimed at the Dynamic Mechanical Analysis of the binders was started, it was decided to pour the binders into small tins. These tins contained about 50-80 grams of binder. The binders were handled in these small quantities to prevent continuous reheating of the binders.

Due to the high temperatures to which polymer-modified binders such as SBS have to be heated, care had to be taken about the length of time the binders spent in the oven. The binders spent one and a half hours maximum in the oven. This means that a tin was heated three times. After usage, the tin was discarded.

The test samples were prepared in the following way:

- The tin containing the appropriate binder was heated in the oven for about twenty to thirty minutes until the binder was viscous enough to be poured. The temperature used depended on the binder. Table 4-6 gives the temperatures used.

Binder	Temperature
60/70	120 °C – 130 °C
SBS	150 °C – 160 °C
Bitumen-Rubber	150 °C – 160 °C

Table 4-6: Temperatures used for heating of binder samples.

- An amount of binder was poured onto the test spindle that was pre-heated at the appropriate test temperature. The amount of binder was calculated from the gap setting that was going to be used during the test.
- The test spindle was placed in the rheometer and the spindle was moved to the trim position.
- At the trim position the excess binder was removed and the spindle was moved to the test position.
- At this point the test sample was left to equilibrate at the test temperature for 5 minutes.
- After the equilibration time the test could be started.

The same procedure was used for sample preparation of all the binders and binder/filler systems tested in the DSR during this project.

4.2.2.3   Strain sweeps

Strain sweeps were performed to determine the linear visco-elastic limits of the materials studied. This was done to ensure that the frequency sweeps were performed inside the linear limits of the materials. The linear limit of a material is defined as the range of strains where the measured response of the visco-elastic material is not a function of the applied strain. Thus, it is important that the frequency sweeps are performed inside the linear range to ensure that the results are reliable. The linear limits were determined over the range of temperatures that the frequency sweeps were done.

4.2.2.4   Frequency sweeps

These tests were performed over a wide range of temperatures. The temperatures were chosen to reflect the range of pavement temperatures normally encountered in Southern Africa. The temperature ranges from 5 °C to 75 °C in steps of 10 °C (5 °C, 15 °C,..., 65 °C, 75 °C).

The purpose of the frequency sweeps is to collect data from which Master Curves and Black Diagrams can be drawn. The Master Curves and Black Diagrams are used to characterise the material behaviour of bituminous binders and can be used to compare the binder properties of different binders.

4.2.2.5   Viscosity tests

These tests were performed at high temperatures (105 °C, 135 °C and 165 °C) on two of the binders (60/70 and SBS). The purpose of these tests was to characterise the binder properties under high temperatures and high shear rates.

4.2.2.6   Creep tests

The purpose of the creep tests that were performed with the dynamic shear rheometer was to compare the results with the results of the sliding plate rheometer.

4.3   Summary of test programme

The test programme that was followed in this study is summarised in Table 4-7.

The test programme summarised here was used for the purpose of the rheological characterisation of the three binders and binder/filler systems. The results of the tests were compared for the different binders and on this basis conclusions could be drawn on the effect of polymer modification on binder properties as well as the effect that the filler has on the binder properties.

Rheometer	Test	Temperature	Material		Purpose
			Binder	Binder/Filler	
SPR	CT	10 °C	✓		Compare results of different binders.
		25 °C, 40 °C	✓	✓	
DSR	SS	5 °C – 75 °C	✓	✓	Determine linear visco-elastic limits
	FS	5 °C – 75 °C	✓	✓	Collect data for drawing of Master Curves and Black Diagrams
	VT	105 °C, 135 °C, 165 °C	✓*	✓*	Characterise binder behaviour at high temperatures and high shear rates
	CT	10 °C	✓	✓	Compare with SPR results
		25 °C, 40 °C	✓	✓	

CT: Creep Tests
SS: Strain Sweeps
FS: Frequency Sweeps
VT: Viscosity Tests
* Tests performed only 60/70 & SBS

Table 4-7: Summary test programme.

In the following chapters the results of the different tests and the comparisons made between the different binders and binder/filler systems are presented.

5 Creep Tests with the Sliding Plate Rheometer on Binders and Binder/Filler Systems

The Royal SHELL Research laboratory in Amsterdam in the Netherlands developed the Sliding Plate Rheometer (SPR). The purpose of this apparatus is to determine the creep behaviour of bituminous binders in shear.

5.1 Purpose of Tests

It has been shown in earlier studies that the Sliding Plate Rheometer could be used satisfactorily to identify and characterise differences between different binders. [5] For this reason the SPR was used in this study. The SPR is also relatively inexpensive compared to the DSR and it can be used to characterise certain material behaviour satisfactorily if a DSR is not available.

5.2 Test Programme

Table 5-1 sets out the test programme that was followed on the binders and binder/filler system.

Material		Number of stress levels per temperature			Number of test repetitions per stress level
		10 °C	25 °C	40 °C	
Binder	60/70	5	5	5	3
	SBS	5	5	5	3
	Bitumen-Rubber	5	5	5	3
Binder/Filler	60/70	=	5	5	3
	SBS	=	5	5	3
	Bitumen-Rubber	=	5	5	3
TOTAL NUMBER OF TESTS: 225					

Table 5-1: Test programme for creep tests using SPR.

5.3 Results: Binders

The creep tests were performed on the three binders at three temperatures – 10°C, 25°C and 40°C. The results are reported in the chronological order that the tests were performed.

In the following section, the procedure that was used to obtain the test results is reported. The explanation given here is based on test results at 25°C, but the procedure was used for all the results obtained.

5.3.1 Procedure used to obtain results

The results were obtained in the following way:

- The test was performed as described in the previous chapter. A load was applied and the sample allowed to deform to a total shear strain of approximately one

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(γ =1). (This was done in accordance with the operating instructions of the SPR equipment that states that the samples should not be allowed to deform beyond this point – deformation beyond this point leads to non-linear behaviour being observed for the vertical test set-up (Figure 5-1). The increase in the strain rate after the 100% strain level can be attributed to the sample disintegrating – it was observed that the front plate rotates and pulls away from the fixed plate.) The time to achieve a strain of one varied with type of binder and the load level – it could be anything between thirty seconds to twenty minutes.
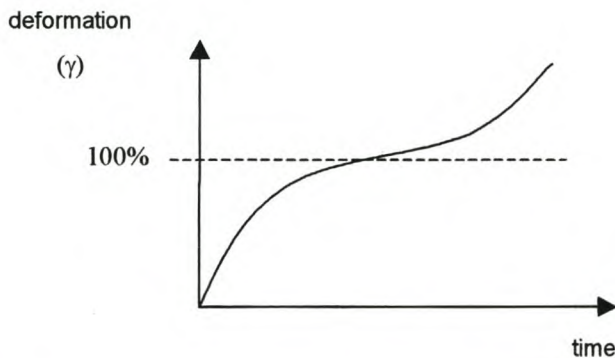
Figure 5-1: Deformation beyond 100% with the SPR creep test.

- As described in the previous chapter the aim of the tests was to obtain a constant shear rate at the end of the test. The resulting graph was analysed and two adjacent periods were compared for a change in slope of the graph. All of the tests had no change between the two adjacent periods in terms of slope – thus it was assumed that the shear rate at the end of a test was constant. (Figure 5-2 gives an example of the creep curve obtained from the creep test data. In Appendix D on the CD the results of the creep tests are given as well as the data that can be used to draw the creep curves for the different binders.)

Creep Curve 25°C - 60/70 set 3 number 5
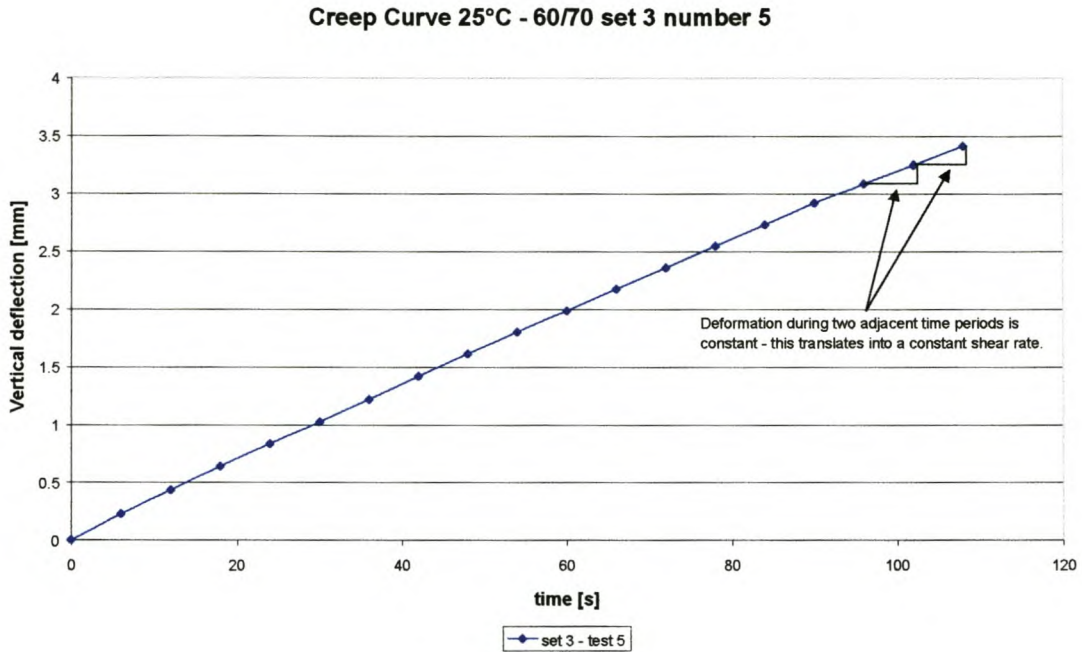
Figure 5-2: Example of creep curve for 60/70 binder at 25°C.

- The load level and resulting shear rate was used to compile a graph (Figure 5-3) that shows the influence of the different modifiers on the behaviour of the binders.

Three tests were done at five stress levels for each of the binders (fifteen tests per binder in total). A line was fitted to the data and the slope of the line obtained. The slopes of the lines are indicative of the binders' shear susceptibility. The steeper the slope, the lower the shear susceptibility of the binder.

5.3.2 Results: 25 °C

The first set of results obtained is for the tests at 25°C. The modification had not been made to the equipment at this stage. Figure 5-3 gives a graphical representation of the results. The data used to obtain these creep test results are contained in Appendix D on the CD supplied with the thesis document. A short description of the procedure followed

for the drawing of graphs such as Figure 5-3 is given in this section – the same procedure was followed throughout for the creep tests performed with the SPR.
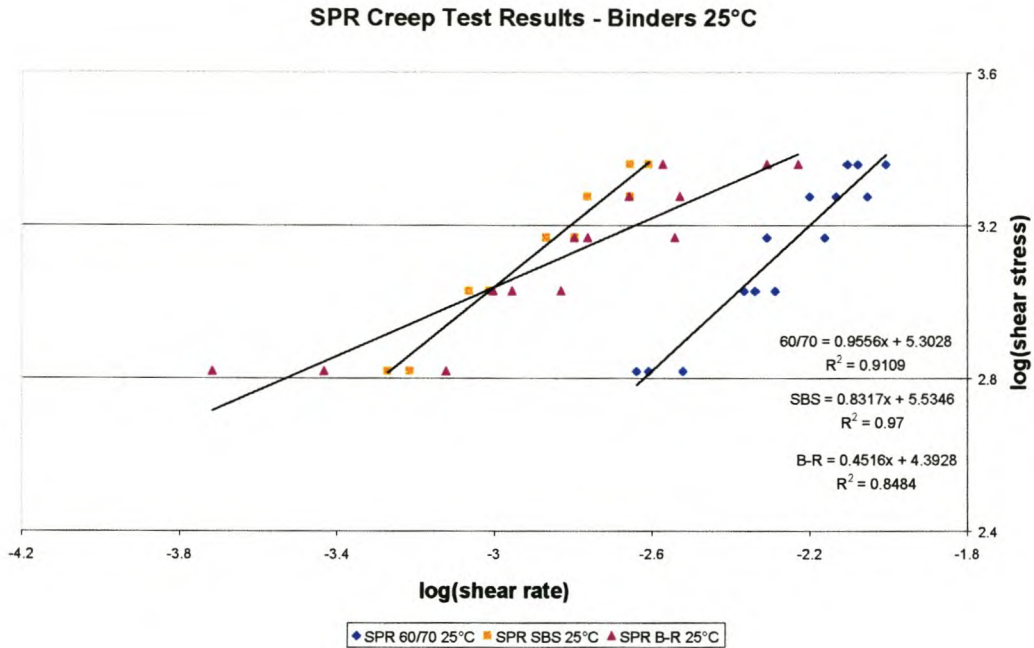
Figure 5-3: Creep test results of SPR tests at 25°C on binders.

Referring to Figure 5-3 the following can be said about the three binders:

- The most important result of the tests is that the modified binders are shifted horizontally to the left of the unmodified binder – this means that the same load level leads to a lower shear rate in the modified binders. As described earlier, a horizontal shift towards lower shear rates means that the viscosity increases.
- The 60/70 is the least shear susceptible of the three binders.
- The SBS modified binder is more shear susceptible than the 60/70, but this is not a very big difference.
- The Bitumen-Rubber is the most shear susceptible of the three binders – this result could be attributed to the large variation in results (low r^2 value) when compared to either of the other two binders.

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- The correlation coefficient for each of the three binders is high, which indicates reliable results. The only exception is the Bitumen-Rubber, but the correlation coefficient is still 84%, which is acceptable.

In Table 5-2 the viscosity values calculated from the creep test results at 25°C are given. It is clear from the table that the addition of the modifier to the bitumen leads to a binder that is much more resistant to permanent deformation (flow) – this is a result of much higher viscosity values. It is also evident from the table that the viscosities of the binders decrease with an increase in the load level.

Binder	Set Number	Test Number				
		1	2	3	4	5
60/70	1	218,229.3	127,300.4	95,475.3	74,258.6	66,832.7
	2	286,426.0	143,213.0	133,665.5	89,110.3	83,540.9
	3	267,330.9	152,760.5	133,665.5	104,154.9	78,626.7
SBS	2	1,233,470.2	763,802.7	486,056.2	381,901.3	297,034.4
	5	1,079,286.4	678,935.7	411,278.4	297,034.4	267,330.9
Bitumen-Rubber	1	3,437,112.0	664,176.2	412,866.3	299,530.5	246,388.0
	2	1,782,206.2	594,068.7	381,901.3	222,775.8	133,665.5
	3	872,917.3	445,551.6	229,140.8		111,387.9

Table 5-2: Viscosity values [Pa.s] from creep tests at 25°C on binders.

5.3.3 Results: 10°C

The tests at 10°C were the first to be obtained with the modified SPR equipment. The tests were performed inside a temperature chamber to control the temperature at 10°C. This was done because with the existing equipment the water in the water bath could not be cooled and the temperature maintained at a constant level.

The same procedure as described in the previous section was used to test the samples. Figure 5-4 gives the results in graphical form. The data from the creep curves are given in Appendix D on the CD.

SPR Creep Test Results - Binders 10°C
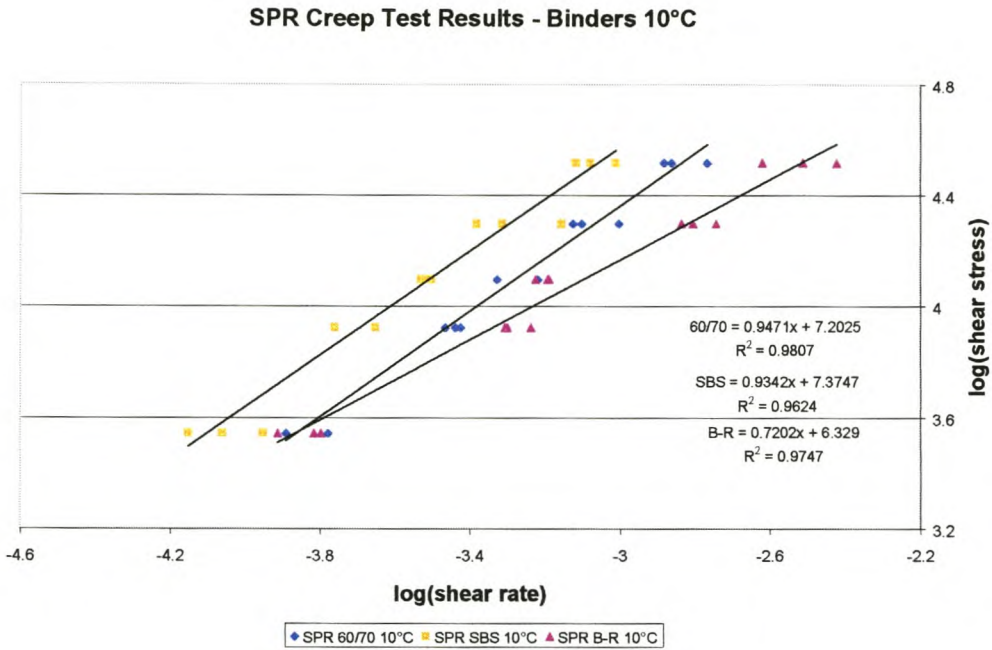
Figure 5-4: Creep test results of SPR tests at 10°C on binders.

The following can be said about this set of results:

- The shift in the shear rates between the three binders is not as clear as with the tests at 25°C. This means that the binders at these low temperatures have viscosities of the same order of magnitude.
- The differences between the modified and unmodified binders are not very clear at the lower temperature. In fact, it seems from these results that the 60/70 binder is more viscous at this temperature.
- The Bitumen-Rubber is more shear susceptible than the other two binders. The 60/70 and SBS are again very close together with the 60/70 having a slightly steeper slope than the SBS.

Table 5-3 contains the viscosity values for the different binders calculated from the creep test results at 10°C. It is clear from the table that the modification of the bitumen does not lead to any improved resistance to flow at this temperature. This confirms the result of

the shear susceptibility lines lying very close together for the three binders. What is also interesting to note is that the Bitumen-Rubber has slightly less viscous response to loading at this temperature compared to the other two binders – this means that the Bitumen-Rubber will flow more readily under loading at this temperature compared to the other two binders.

Binder	Set Number	Test Number				
		1	2	3	4	5
60/70	1	21,063,219.2	9,283,418.8	5,802,136.8	3,524,143.3	2,047,813.0
	2	27,304,173.0	9,636,766.9	7,446,592.6	4,680,715.4	2,685,656.4
	3	27,304,173.0	10,239,064.9	7,446,592.6	4,427,703.7	2,559,766.2
SBS	1	40,355,567.7	15,717,431.6	11,656,781.6	7,216,102.9	4,216,085.5
	2	31,387,663.8	20,177,783.8	11,169,889.0	8,418,786.7	4,592,065.5
	3	49,771,866.8	20,177,783.8	11,945,575.7	5,005,765.1	3,596,159.4
Bitumen-Rubber	1	28,697,292.6	7,086,831.4	5,460,834.6	2,239,795.4	924,281.3
	2	22,012,127.8	6,980,422.5	5,876,332.9	2,409,191.7	1,460,815.3
	3	22,971,630.8	6,053,335.2	5,412,077.1	1,945,422.3	1,137,419.3

Table 5-3: Viscosity values [Pa.s] from creep tests at 10°C on binders.

5.3.4 Results: 40°C

As stated earlier, one of the reasons why the SPR equipment was modified was to obtain acceptable test results at the higher temperature. This had to be done due to the relatively high system load applied to the samples. The creep test results are summarised in graphical form in Figure 5-5. The data obtained from the creep tests at 40°C are given in Appendix D on the CD.

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SPR Creep Test Results - Binders 40°C

60/70 = 0.8276x + 3.7607
R^2 = 0.9695

SBS = 0.6602x + 3.9914
R^2 = 0.9629

B-R = 0.6054x + 3.9544
R^2 = 0.965

log(shear stress)

log(shear rate)

◆ SPR 60/70 40°C ■ SPR SBS 40°C ▲ SPR B-R 40°C
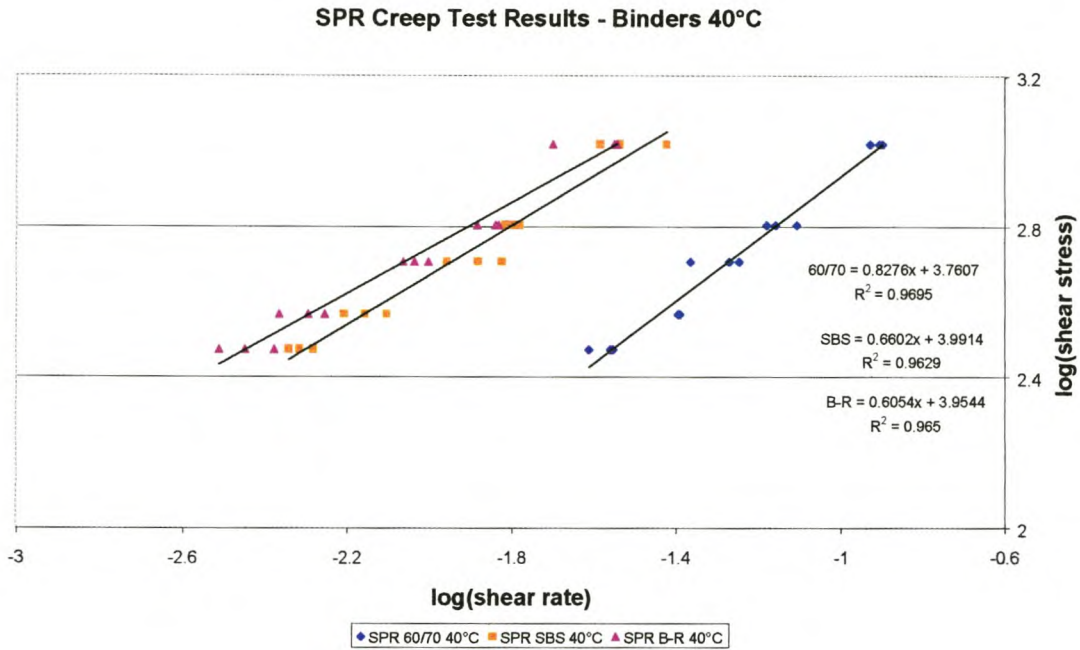
Figure 5-5: Test results of SPR tests at 40°C on binders only.

The following can be said about these results:

- It would seem that the Bitumen-Rubber performs the best of the three binders at the high temperature. The SBS line is very close to the Bitumen-Rubber line.
- There is a clear horizontal shift between the modified binders' and the 60/70's lines – the modified binders are shifted to the left of the unmodified binder. This means that the modified binders have higher viscosities (more resistant to viscous flow) at the higher temperature.
- The 60/70 performs better in terms of shear susceptibility, but the same loading regime results in much higher shear rates than that of the modified binders.

Table 5-4 contains the viscosity values calculated for the different binders from the results of the creep tests. It is clear that the influence of the modification of bitumen is much more pronounced at this temperature. The results given here shows that the SBS

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and Bitumen-Rubber binders have higher viscosity values for the same load levels compared with the 60/70 bitumen. This means that the modified binders have more resistance to flow and should have better performance in the field when permanent deformation is considered.

Binder	Set Number	Test Number				
		1	2	3	4	5
60/70	1	12,174.5	7,304.7	5,549.1	4,282.1	2,520.7
	2	10,635.7	7,304.7	5,256.3	3,806.3	2,388.1
	3	10,779.5	7,358.8	6,898.9	4,499.3	2,345.6
SBS	1	61,597.0	42,543.0	27,040.0	17,925.4	10,231.4
	2	65,614.2	47,908.8	22,693.6	19,455.6	11,395.4
	3	56,977.2	37,728.2	19,856.9	18,479.1	7,859.5
Bitumen-Rubber	1	71,144.5	53,466.2	32,465.2	20,220.8	14,895.3
	2	83,699.4	58,640.3	29,955.6	20,585.3	10,413.4
	3	97,015.3	68,988.6	34,494.3	22,790.9	10,539.9

Table 5-4: Viscosity values [Pa.s] from creep tests at 40°C on binders.

5.3.5 Results: 10°C, 25°C and 40°C combined

The results from the different sets of tests on the binders were combined on one graph. This was done to show the influence of temperature on the response of the three different binders. The graph shows a distinct difference in response for the three binders at the three temperatures (Figure 5-6). The reader is referred to Appendix D on the CD for the data that was used for this discussion.
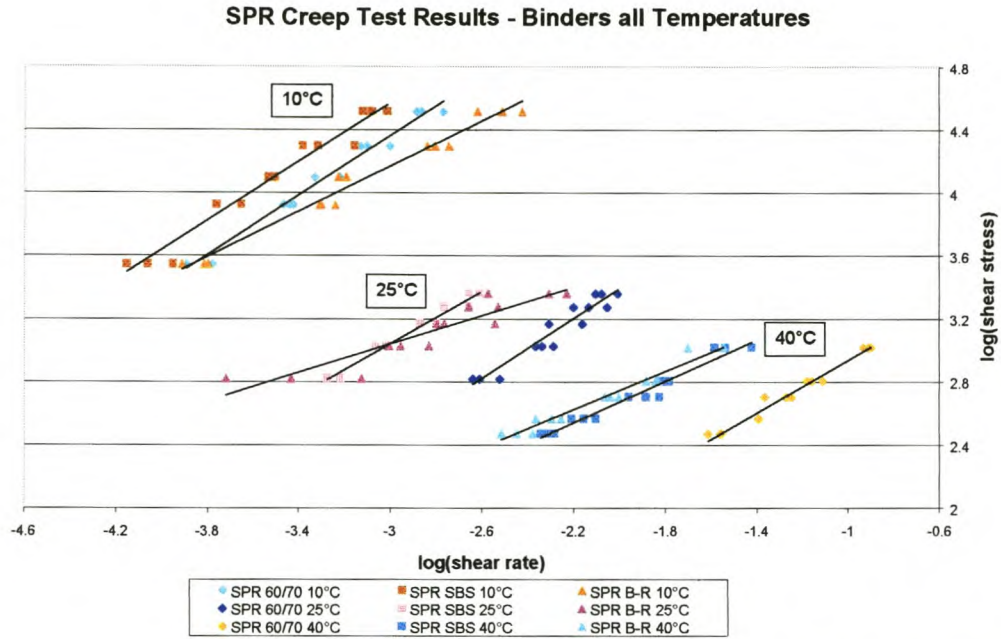
Figure 5-6: Combination of SPR creep test results for the three binders at the three temperatures.

From Figure 5-6, the following comments can be made:

- A distinct shift in the shear rates resulting from the applied loads can be noted between the three temperatures. The lowest temperature (10°C) results in the lowest shear rates, while the shear rates increase with increase in temperature (25°C and 40°C). This is consistent with the generally accepted fact that a binder's resistance to flow decrease with an increase in temperature (viscosity decrease with increase in temperature).

- It is also clear from the graph that the increase in temperature leads to a more pronounced shift in shear rates between the modified and unmodified binders. This means that the effect of polymer modification becomes more significant as temperature increases.

- Binder response to temperature changes is quite clear from this figure. At the low temperature the binders are much stiffer which results in much lower shear rates compared to the high temperature.

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- At 40°C the difference in shear rate for a specific loading regime is very clear. It can be seen that there is a distinct shift towards lower shear rates for the modified binders compared with the 60/70. At 25°C this shift towards lower shear rates for the modified binders is also apparent. At 10°C there is no shift – in fact the 60/70 had lower shear rates than the Bitumen-Rubber at the low temperature.

Table 5-5 summarises the results obtained from the creep tests performed with the SPR.

Material		Temperature		
		10 °C	25 °C	40 °C
60/70	slope	0.9471	0.9556	0.8276
	r^2	0.9807	0.9109	0.9695
SBS	slope	0.9342	0.8317	0.6602
	r^2	0.9624	0.97	0.9629
Bitumen-Rubber	slope	0.7202	0.4516	0.6054
	r^2	0.9747	0.8484	0.965

Table 5-5: Summary of creep test results obtained with SPR for binders.

The Bitumen-Rubber does not follow the trend exhibited by the other two binders in that the slopes of the lines do not decrease with the increase in temperature. This is an unexpected result and could be due to the large variations in the results of the Bitumen-Rubber obtained at 25 °C. If the same (low) variation in test results was observed at 25 °C that was observed at the other two temperatures, there might have been a trend similar to the other two binders visible. The other two binders exhibit a definite trend that can be associated with the increase in temperature.

Looking at the tables of viscosity values (Table 5-2, Table 5-3 and Table 5-4) given in the previous three sections, the trend associated with the decrease in temperature is apparent. It can be seen that the viscosities of the binders decrease with an increase in temperature – this result is consistent with the generally accepted fact that the permanent deformation of a bituminous binder increases with an increase in temperature.

5.4 Results: Binder/Filler Systems

Binder/Filler systems were studied in an attempt to characterise the influence the filler has on the binder properties. The aim was to ascertain whether the addition of filler would have a negative effect on the properties of the binders.

The tests were performed at 25°C and 40°C to compare the results with the results for the binders only. 10°C was not used because it was felt that the binder/filler system would be too stiff at this temperature to acquire acceptable results.

The following sections give the results of these tests on the binder/filler systems.

5.4.1 Results: 25°C

Figure 5-7 gives a graphical representation of the results of the SPR tests on the binder/filler systems at 25°C. The data that was obtained from the creep tests on the binder/filler mixes can be found in Appendix D on the CD. The same procedure was used for the interpretation of the creep curves that was described in the section on the creep tests at 25°C on the binders.

SPR Creep Test Results - Binder/Filler Systems 25°C

Binder/Filler Systems

$60/70+F = 0.8613x + 5.6342$
$R^2 = 0.9875$

$SBS+F = 0.8719x + 6.1822$
$R^2 = 0.9821$

$B-R+F = 0.6329x + 5.5192$
$R^2 = 0.991$

log(shear stress)

Binders

log(shear rate)

| ◆ SPR 60/70+F 25°C | ■ SPR SBS+F 25°C | ▲ SPR B-R+F 25°C |
| ◆ SPR 60/70 25°C | ■ SPR SBS 25°C | ■ SPR B-R 25°C |
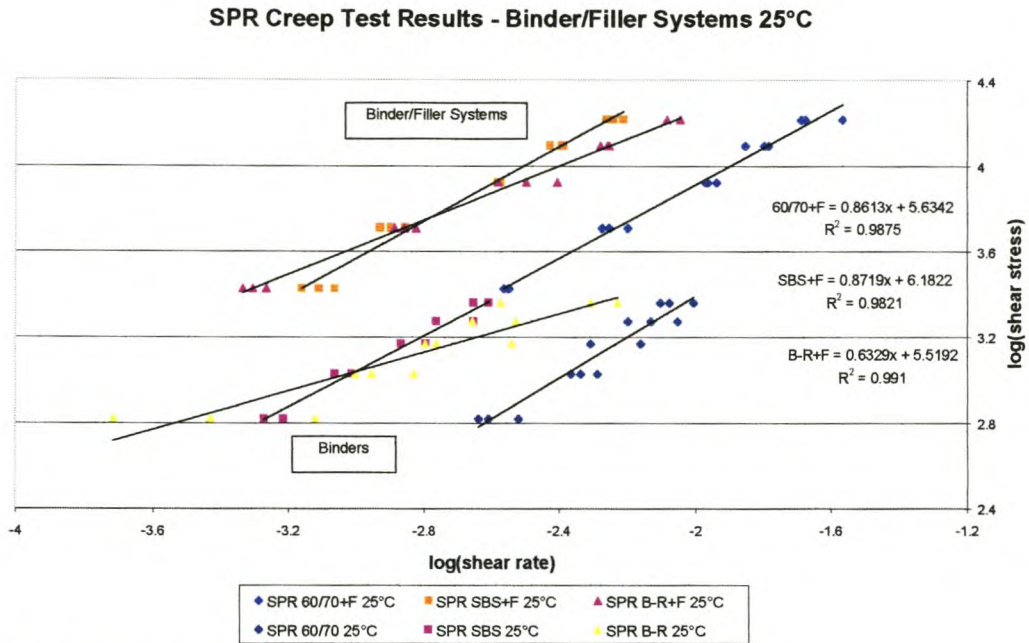
Figure 5-7: Creep test results from SPR tests on Binder/Filler systems at 25°C.

The effect of the addition of filler to the binder is clear from this graph. When the lines for the binders (Figure 5-3) and the lines for the binder/filler systems (Figure 5-7) are compared, the following observations can be made:

- The addition of filler to the binder leads to a less shear susceptible system when compared to the binder only. This is expected due to the stiffening effect the addition of filler has. The only exception to this is the 60/70 that has increased shear susceptibility when filler is added.

- The same trend exhibited by the binders is exhibited by the binder/filler systems. The difference between the binders and the binder/filler systems lies in the slopes of the lines – the addition of the filler leads to a decrease in the shear susceptibility.

- The horizontal shift apparent for the polymer modified binders is again clear on the figure for the binder/filler systems. The polymer modified binder/filler systems still have an improved resistance to deformation compared to the 60/70

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binder/filler system. This means that the increase in viscosity for the binder with the addition of polymer is carried through to the binder/filler system.

- The stiffening effect of the filler is apparent on the figure. A much higher shear stress regime is needed to obtain the same range of strain rates. This is due to the high stiffness of the binder/filler systems compared to the binders.

Table 5-6 contains the viscosity values that were calculated for the different binder/filler systems from the results of the creep tests. The data can be found in Appendix D on the CD. It is apparent from the results that the addition of filler to the binders does not alter the binder properties. The modified binders still show a marked increase in viscosity compared to the 60/70 bitumen. Another result apparent when compared to the results at 25°C for the binders (Table 5-2) is that the addition of filler leads to a system that is much more resistant to flow (stiffer) compared to the binder only.

Binder	Set Number	Test Number				
		1	2	3	4	5
60/70+Filler	1	945,661.1	480,849.3	251,206.4	167,785.7	98,216.0
	2	955,267.8	423,219.9	233,263.1	163,094.5	130,627.3
	3	983,363.9	503,116.4	247,912.4	191,053.6	126,710.8
SBS+Filler	1	3,887,717.7	2,280,794.4	1,029,719.8	716,158.5	466,526.1
	2	3,455,749.1	2,125,285.7	1,020,525.9	652,581.2	490,450.5
	3	3,110,174.2	1,920,989.9	1,003,282.0	662,351.9	437,368.2
Bitumen-Rubber+Filler	1	5,831,576.6	1,788,350.1	684,238.3	480,849.3	298,058.4
	2	5,442,804.8	1,943,858.9	846,658.5	485,964.7	
	3	4,956,840.1	2,073,449.4	1,021,914.4	510,957.2	326,568.3

Table 5-6: Viscosity values [Pa.s] from creep tests at 25°C on binder/filler systems.

5.4.2 Results: 40°C

In Figure 5-8 the results of the tests on the binder/filler systems at 40°C is presented. The data obtained form the creep tests can be found in Appendix D on the CD.

SPR Creep Test Results - Binder/Filler Systems 40°C
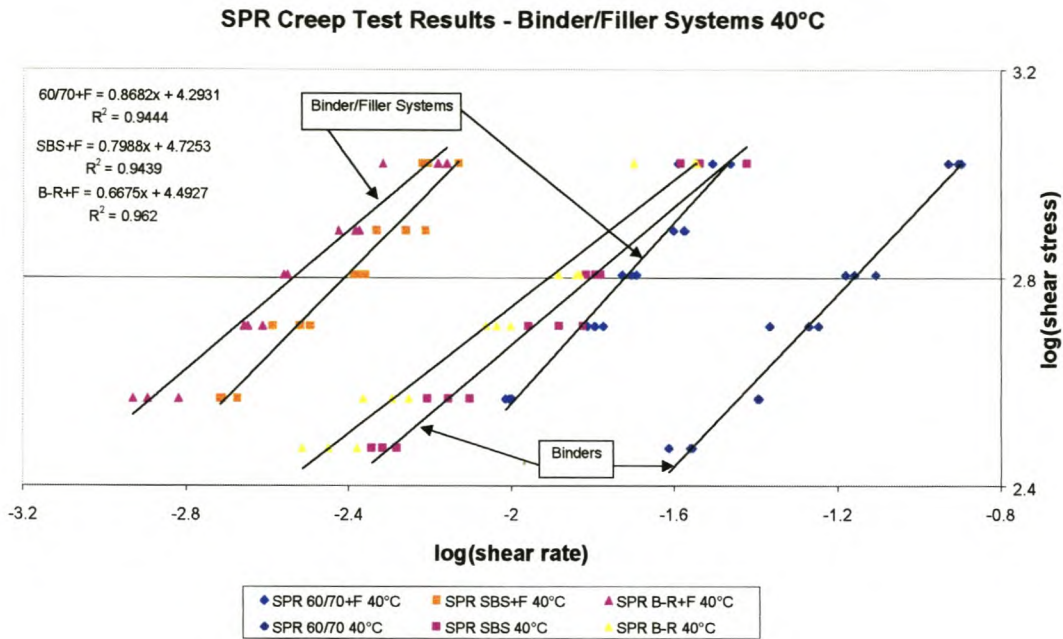
Figure 5-8: Creep test results of SPR tests on Binder/Filler systems at 40°C.

Referring to Figure 5-8 and Figure 5-5, the following comments can be made:

- As with the tests at 25°C, the general trend of the shift in shear rates is preserved with this set of results. The modified binders are shifted to the left (lower shear rates) for the same loading regime – this is due to the stiffening effect of the filler.

- The effect of the addition of filler is the same as with the 25°C results – there is a decrease in the shear susceptibility of the binder/filler systems compared to the binders only. All the binder/filler systems have a decreased shear susceptibility compared to the binders when looking at the slope of the line fitted through the results.

- The interesting result where the Bitumen-Rubber performed better than the SBS is again evident from these results. This is another indication that the addition of filler does not impair the effect of modification on the binders.

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- Another interesting results which is apparent on the figure is that the viscosities of the polymer modified binders (without filler) are even higher than the viscosity of the 60/70 + Filler.

In Table 5-7 the viscosity values calculated from the creep test results are given for the binder/filler systems at 40°C. Again the influence of the filler addition is apparent form the table. As with the results at 25°C for the binder/filler systems, the addition of the filler leads to much higher viscosity values compared to the binders only and the effect of polymer addition is still apparent.

Binder	Set Number	Test Number				
		1	2	3	4	5
60/70+Filler	1	37,288.2	23,966.4	18,691.0	14,724.0	14,389.4
	2	38,097.2	23,013.7	19,668.5	13,817.3	10,708.4
	3	36,810.1	21,980.4	18,157.7	13,838.5	11,835.6
SBS+Filler	1	174,011.5	115,538.0	89,950.6	60,234.7	49,972.5
	2	191,858.8	121,956.8	84,431.6	67,270.7	59,661.1
	3	189,830.7	142,778.7	87,005.7	79,321.5	61,394.8
Bitumen-Rubber+Filler	1	314,952.9	151,177.4	131,177.9	89,236.7	53,542.0
	2	243,372.7	164,450.4	133,855.0	98,517.3	76,743.5
	3	289,756.7	167,764.9	133,855.0	87,451.9	56,219.1

Table 5-7: Viscosity values [Pa.s] from creep tests at 40°C on binder/filler systems.

5.4.3 Results: 25°C and 40°C Combined

Figure 5-9 combines the results of the creep tests at 25°C and 40°C on the binder/filler systems. From this figure and the figures presented in the previous sections, the effect of the addition of filler to the binder is evident. The addition of filler does not affect the binder properties negatively. As seen previously the shear susceptibility lines follow the same trend for the binders and binder/filler systems, with the shift to lower shear rates for the polymer modified binders being carried through to the binder/filler systems.

SPR Creep Test Results - Binder/Filler Systems all Temperatures
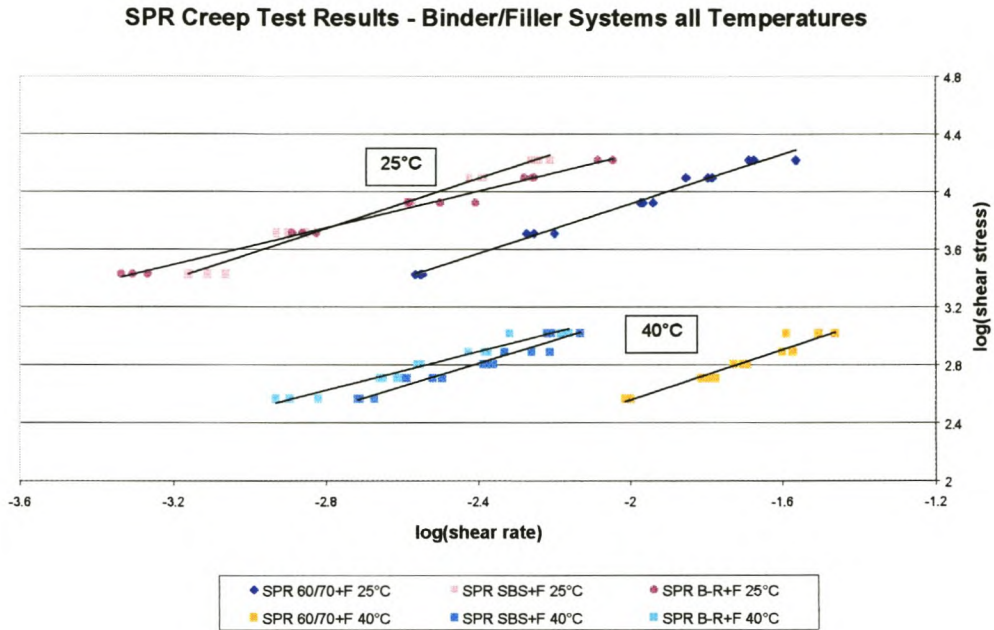
Figure 5-9: Combined test results on SBS for the Binder/Filler system and binder only at 25°C and 40°C.

The other effect of filler addition that is evident when considering the tables of viscosity values reported in each section, is the stiffening effect of the filler on the binders. This can be seen in the increase in viscosity of the binder/filler systems when compared to the binders. It is also clear from these tables of results that the effect of polymer addition is carried through to the binder/filler systems. This means that the addition of filler does not lead to a change in the observed binder properties.

Table 5-8 below summarises the results of the SPR creep tests on the binders and binder/filler systems.

Material		Temperature	
		25 °C	40 °C
60/70	slope	0.9556	0.8276
	r^2	0.9109	0.9695
SBS	slope	0.8317	0.6602
	r^2	0.97	0.9629
Bitumen-Rubber	slope	0.4516	0.6054
	r^2	0.8484	0.965
60/70 + Filler	slope	0.8613	0.8682
	r^2	0.9875	0.9444
SBS + Filler	slope	0.8719	0.7988
	r^2	0.9821	0.9439
Bitumen-Rubber + Filler	slope	0.6329	0.6675
	r^2	0.991	0.962

Table 5-8: Summary of creep test results from SPR on binder/filler systems.

5.5 Comparison with DSR results

Creep tests were performed with the Dynamic Shear Rheometer (DSR) that was used for the second part of this study. These creep tests were performed under the same conditions that the SPR creep test were performed (stress controlled). This was done to compare the results from the DSR with those obtained with the SPR. A detailed discussion of the comparison between the two test methods can be found in the chapter dealing with the DSR tests (Chapter 7).

5.6 Conclusions

Based on the results and discussion presented here the following conclusions may be drawn:

5.6.1 Binders

- From the figures the effect of binder modification was clear. The addition of SBS and Rubber to bitumen leads to a binder that responds more viscous (more resistant to flow) to loading.
- It was clear that the effect of polymer modification becomes more pronounced at higher temperatures (25 °C and 40 °C in this case). At 10 °C there was no evidence of improved binder performance due to polymer modification.
- The SPR qualified the influence of polymer modification well.

5.6.2 Binder/Filler Systems

- The general results were still the same – the binder/filler systems were qualified the same as for the binders in terms of the horizontal shift in shear rates. This leads to the conclusion that the filler does not have a negative effect on the binder properties.
- The addition of filler leads to a mix that is much stiffer than the binder only.

In summary it can be concluded that the SPR proved useful for the study of the differences between different binders. It was also possible to characterise the influence filler addition has on the binder properties. From the results one can expect that properly constructed asphalt mixes containing the two modified binders will perform better than mixes with the unmodified binder when rutting is considered.

The modification of the SPR equipment makes it possible to perform accurate tests at 40°C. It is important to be able to perform this type of testing at higher temperatures in order to relate the binder/filler systems' behaviour to the rutting performance of mixes. Normally, rutting occurs in pavements where the temperature range is in the order of 50°C to 70°C. Thus, it is important that it is possible to perform the creep tests at such high temperatures.

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6 Strain and Frequency Sweeps on Binders and Binder/Filler Systems

The binders and binder/filler systems were tested in the dynamic shear mode using a Dynamic Shear Rheometer (DSR).

This chapter deals with the strain and frequency sweeps that were performed on the binders and binder/filler systems.

Table 6-1 gives an overview of the test programme that was followed for the strain and frequency sweeps on the binders. One test was performed at each of the test temperatures.

Binder		Strain sweeps	Frequency sweeps
60/70	Unaged	8	8
	RTFOT residue	8	8
	+ Filler	8	8
SBS modified 60/70	Unaged	8	8
	RTFOT residue	8	8
	+ Filler	8	8
Bitumen-Rubber	Unaged	8	8
	+ Filler	8	8

Table 6-1: Tests performed during strain and frequency sweeps.

6.1 Purpose and Methodology of Strain and Frequency sweeps

6.1.1 Strain sweeps

6.1.1.1 Purpose

The purpose of the strain sweeps was to determine the linear limit of the binders and binder/filler systems. It is important that the dynamic shear testing be conducted below the linear limit of the materials – this ensures that the response being measured during the test is independent of the applied strain.

The AASHTO provisional standard (TP5-93: "Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)") that deals with dynamic testing of binders, defines the linear region as follows:

> "The linear region is defined as the range in strains where the complex modulus is 95% or more of the zero-strain value."

This requirement was used to determine the linear limit of the different binders and binder/filler systems.

6.1.1.2 Methodology

It was decided to perform strain sweeps at each of the temperatures at which frequency sweeps were going to be conducted. This was done to ensure that the frequency sweeps were conducted within the linear range at all temperatures. The temperatures used were:

- 5 °C, 15 °C, 25 °C, 35 °C, 45 °C, 55 °C, 65 °C, 75 °C

The temperature range was chosen based on the temperature range that can be expected in pavement layers in South Africa. Summer temperatures are very high and encountering pavement temperatures exceeding 60 °C is not uncommon. Winter temperatures on the other hand are quite mild and pavement temperatures seldom drop below 0°C.

The samples were allowed to achieve temperature equilibrium before the tests were started.

The test system was set up for the strain sweeps in the following way:

- A standard oscillation test was chosen.
- The test was set up to increase the strain during the test in a log-ramp (equal amount of points per decade) fashion from 0,01% strain to 100% strain. The frequency used was 10Hz. It was initially decided to run the frequency sweeps from 10 Hz, but after exploratory tests it was decided to increase this frequency to 50 Hz. The linear limits obtained at 10 Hz were used for the frequency sweeps and acceptable results were obtained. Due to the time-temperature interdependence of bituminous materials, the linear limit obtained at a high frequency for a test temperature will fall within the linear limit at the same temperature at lower frequencies for the same material. The concern is when frequencies are used that are higher than the one at which the limits were obtained. The linear limit for a material decreases with an increase in frequency at a temperature (the material response is much stiffer), but it was found that acceptable results were obtained in this test programme.
- Fifty measuring points were taken during the test.
- The temperature was set for the test and kept constant during the test – temperature was controlled to within 0,1 °C of the set temperature.

From the test results a graph could be drawn of the decrease in the storage modulus with the increase in strain. From this graph the linear limit of the material was determined.

6.1.2 Frequency sweeps

6.1.2.1   Purpose

Frequency sweeps are at the heart of what dynamic mechanical analysis of binder behaviour is all about. Frequency sweeps give information about a binder's behaviour at different frequencies. The binder response is measured in terms of the Complex Modulus (G^*) and Phase Angle (δ).

Frequency sweeps are run over a range of temperatures. Making use of the time-temperature superposition principal applicable to bituminous binders, the frequency sweeps at different temperatures can be shifted relative to a reference temperature. This is done to form a smooth curve over a wide range of frequencies known as the Master Curve at the reference temperature.

A master curve can cover frequencies that are impossible to achieve in the laboratory (very high frequencies) or that are impractical to achieve (very low frequencies). Thus, the binder response is characterised over a much wider range of frequencies than can practically be tested in the laboratory at a given temperature.

The Black Diagram is another graph that can be drawn. The Black diagram can be regarded as a fingerprint of the binder because predictions of binder response at any temperature or frequency can be made from it – the only prerequisite is that the complex modulus of the binder be known or that it is accurately predicted. The binder's complex modulus can be calculated from the binder stiffness predicted by a tool such as BANDS for normal penetration grade bitumens.

The Black Diagram is a plot of the phase angle of the material versus the complex modulus (Figure 6-1). From this the phase angle can be determined at a given value for the complex modulus. The phase angle is a measure of the amount of viscous response of

the material – the higher the phase angle, the more viscous the response will be. Thus, binder response can be predicted.
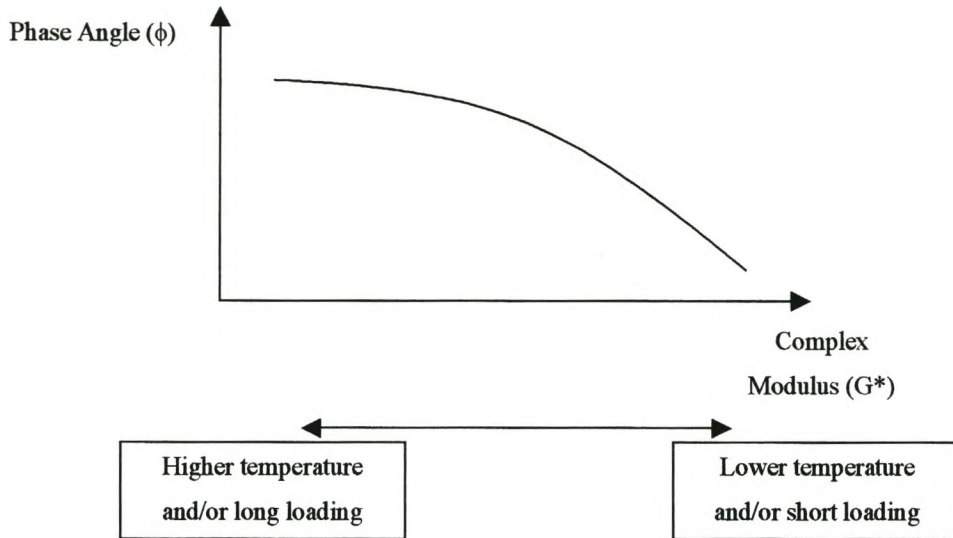
Figure 6-1: Example of a Black Diagram for normal bitumen.

6.1.2.2   Methodology

A standard oscillation test was used for the frequency sweeps. The tests were conducted in strain controlled mode. The strain used was determined from the strain sweeps at the different temperatures. The strain levels are presented in Table 6-2.

When the values in Table 6-2 are compared to those in Table 6-6, it can be seen that the binder/filler systems might have been tested outside the linear limits for the materials at the lower temperatures. However, the results obtained do not suggest that there were any problems.

Temperature	60/70			SBS			Bitumen-Rubber	
	unaged	aged	+ Filler	unaged	aged	+ Filler	unaged	+ Filler
75 °C	5%	1%	0,2%	2%	1%	0,2%	1%	0,2%
65 °C	5%	1%	0,2%	2%	1%	0,2%	1%	0,2%
55 °C	5%	1%	0,2%	1%	1%	0,2%	1%	0,2%
45 °C	5%	1%	0,2%	1%	1%	0,2%	1%	0,2%
35 °C	5%	1%	0,1%	1%	1%	0,1%	1%	0,1%
25 °C	5%	1%	0,1%	1%	1%	0,1%	1%	0,1%
15 °C	2%	0,5%	0,1%	1%	0,5%	0,1%	1%	0,1%
5 °C	1%	0,5%	0,1%	1%	0,5%	0,1%	1%	0,1%

Table 6-2: Table of strain levels used for frequency sweeps at different temperatures.

The test was run in a ramp-log fashion with the highest frequency 50Hz down to the lowest frequency of 0,05Hz.

Before a test was started the sample was first allowed to reach temperature equilibrium at the desired temperature.

The system recorded the material response to the applied deformation. This file was exported as a text file. The text file was imported into Excel and the data manipulated and graphs drawn. The Excel files can be found in Appendix B on the CD. The algorithm used for the drawing of the Master Curve is discussed in Appendix B on the CD.

6.2 Results and Discussion

6.2.1 Strain Sweeps

This section will deal with the results of the strain sweeps that were carried out on the binders.

Every section will report the results of the strain sweeps on a binder and binder/filler system.

6.2.1.1 60/70

In this section the results of the strain sweeps that were carried out on the 60/70 binders and binder/filler system are presented. The data from which the linear limits were determined can be found in Appendix B on the CD.

The results from the strain sweeps on the 60/70 binders are summarised in Table 6-3. The effect of the ageing and addition of filler on the bitumen is clear from the results. The open cells in the table are due to test results that could not be used.

Temperature	Binder		
	unaged	RTFOT residue	+ Filler
75 °C	21,1%		
65 °C		21,1%	
55 °C	32,5%	28,3%	0,83%
45 °C	18,5%	12,2%	0,47%
35 °C	5,6%	4,5%	0,36%
25 °C		2,24%	<0,1%
15 °C	2,0%	1,69%	<0,1%
5 °C	1,5%	1,48%	<0,1%

Table 6-3: Linear limits (% strain) from strain sweeps at 10Hz on 60/70 binders.

The results are presented graphically in Figure 6-2.

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Linear Limit vs. Temperature - 60/70
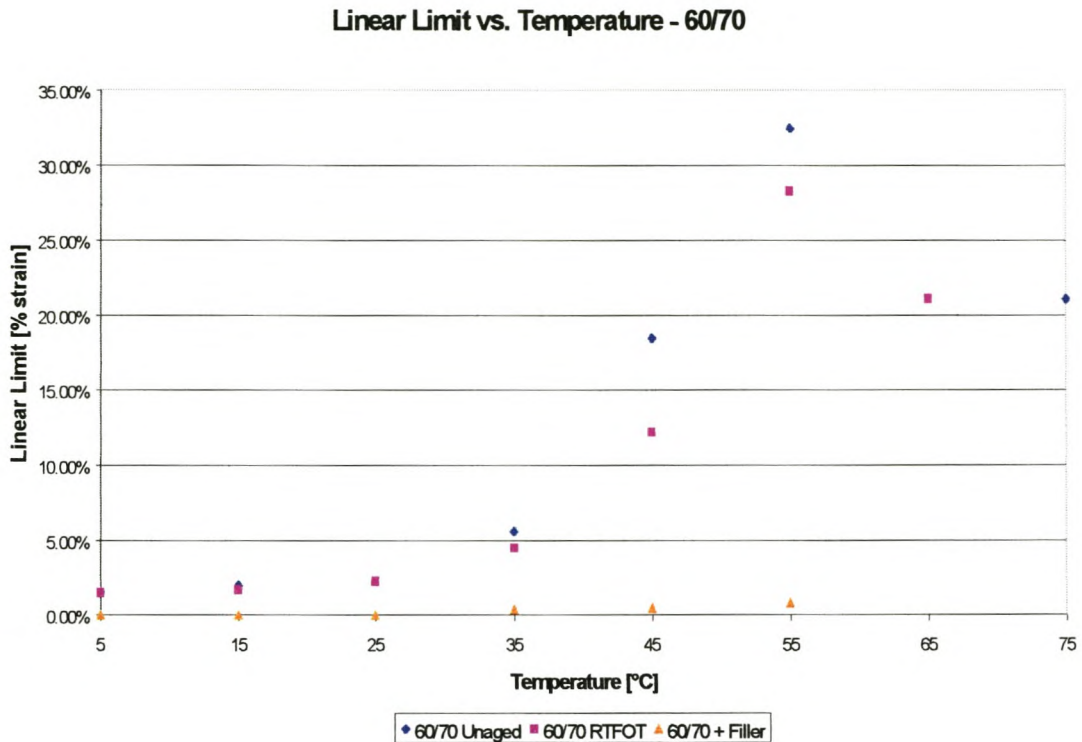
Figure 6-2: Graphical representation of linear limits at 10 Hz for the 60/70 binders.

Comparing the results in Table 6-3 of the three 60/70 binders, the following comments can be made:

- There is a decrease with temperature in the values of the linear limits for each of the three binders.

- At higher temperatures (T > 40 °C) the linear limits are in excess of 10% strain for the normal and aged bitumen.

- The linear limits of the binder/filler system are drastically reduced compared to the normal and aged binders.

- There is a decrease in the linear limits for the RTFOT residue compared to the unaged 60/70 – this could be attributed to the stiffening effect of ageing. This decrease is not as noticeable as for the binder/filler system.

6.2.1.2   SBS

In this section the results of the strain sweeps on the SBS modified binder are reported. The results of the tests on the binder, RTFO residue and binder/filler system are summarised in Table 6-4. Figure 6-3 gives a graphical representation of the results in Table 6-4. The data from which these results were obtained can be found in Appendix B on the CD.

Temperature	Binder		
	unaged	RTFOT residue	+ Filler
75 °C	56,5%	65,4%	2,56%
65 °C	57,6%	37,5%	1,27%
55 °C	37,6%	18,6%	2,96%
45 °C	14,0%	7,97%	1,69%
35 °C	4,65%	2,52%	0,84%
25 °C	2,58%	1,69%	0,36%
15 °C	1,69%	1,47%	<0,1%
5 °C	1,28%	1,11%	<0,1%

Table 6-4: Linear limits (% strain) from strain sweeps at 10Hz on SBS binders.
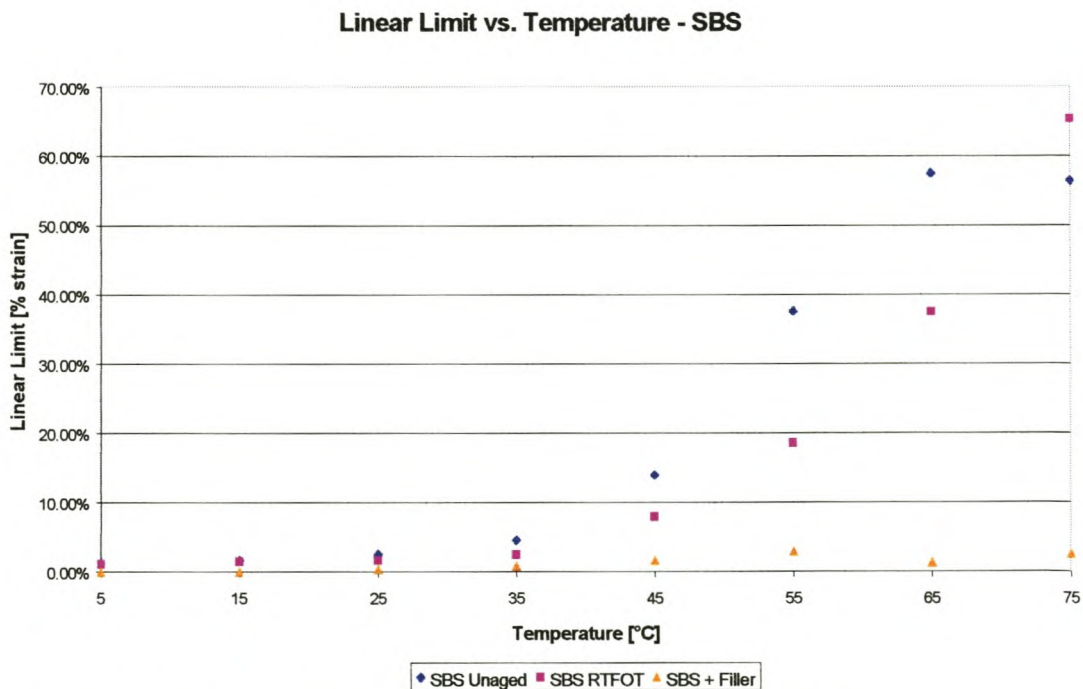
Figure 6-3: Graphical representation of linear limits at 10 Hz for the SBS binders.

Comparing the three binders in Table 6-4, the following comments can be made:

- The difference between the unaged and aged SBS binder is more marked than for the 60/70 binder. The decrease in the linear limits is bigger with ageing. The only exception is for the RTFOT residue at 75 °C, but this point could be an outlier.

- The addition of filler has a big effect on the linear limits, drastically reducing it compared to the unaged and aged binders.

6.2.1.3   Bitumen-Rubber

In this section the results of the strain sweeps on the bitumen-rubber and binder/filler system are reported. Table 6-5 shows the summarised results for the linear limits. The RTFO test was not performed on the bitumen-rubber. The data from which these results were obtained can be found in Appendix B on the CD.

Temperature	Binder	
	unaged	+ Filler
75 °C	12,0%	2,96%
65 °C	13,9%	2,57%
55 °C	12,1%	1,46%
45 °C	9,18%	0,55%
35 °C	6,95%	<0,1%
25 °C	2,97%	<0,1%
15 °C	1,68%	<0,1%
5 °C	1,28%	<0,1%

Table 6-5: Linear limits (% strain) from strain sweeps at 10Hz on Bitumen-Rubber binders.

Linear Limit vs. Temperature - Bitumen-Rubber
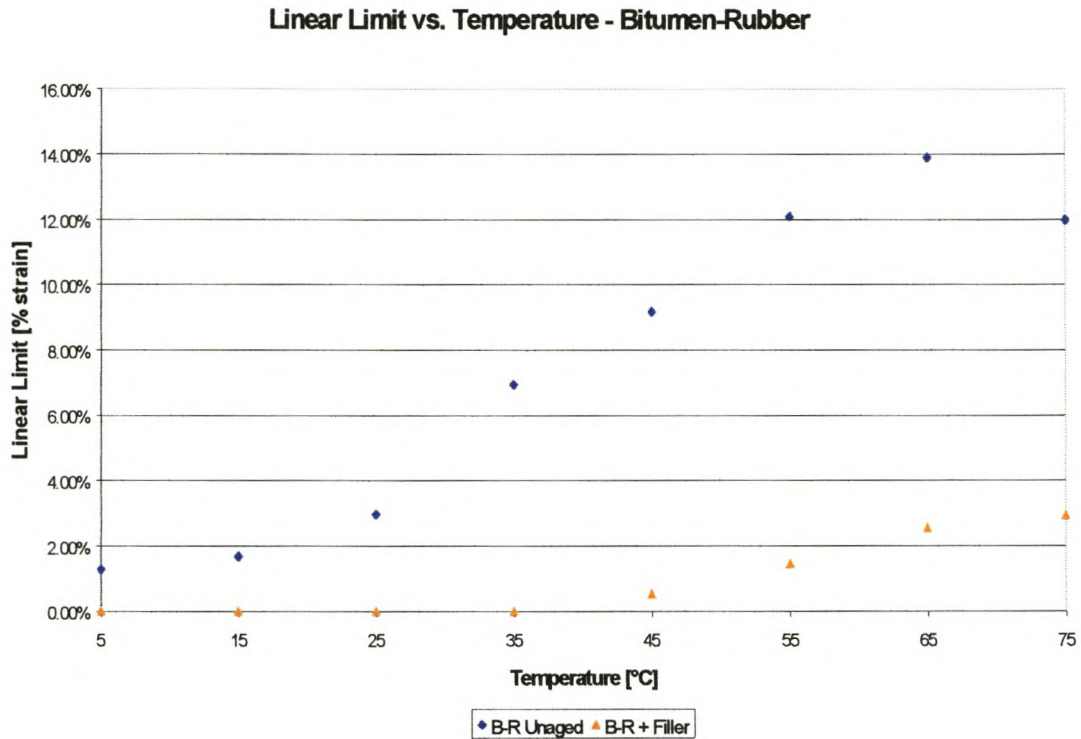
Figure 6-4: Graphical representation of linear limits for the Bitumen-Rubber binders.

The following are a few comments that can be made when looking at the bitumen-rubber's results:

- The linear limits for this binder are much lower at the higher temperatures compared to the other two binders.
- There is a decrease in the linear limits with the decrease in temperature.
- The addition of filler reduces the linear limits drastically.

6.2.1.4 Combined Results

In this section the results of the strain sweeps on all the binders are combined. This is done to compare linear limits of the different binders.

Table 6-6 summarises the linear limits of the different binders.

Temperature	60/70			SBS			B-R	
	unaged	aged	+ Filler	unaged	aged	+ Filler	unaged	+ Filler
75 °C	21.10%			56.50%	65.40%	2.56%	12.00%	2.96%
65 °C		21.10%		57.60%	37.50%	1.27%	13.90%	2.57%
55 °C	32.50%	28.30%	0.83%	37.60%	18.60%	2.96%	12.10%	1.46%
45 °C	18.50%	12.20%	0.47%	14.00%	7.97%	1.69%	9.18%	0.55%
35 °C	5.60%	4.50%	0.36%	4.65%	2.52%	0.84%	6.95%	<0,1%
25 °C		2.24%	<0,1%	2.58%	1.69%	0.36%	2.97%	<0,1%
15 °C	2.00%	1.69%	<0,1%	1.69%	1.47%	<0,1%	1.68%	<0,1%
5 °C	1.50%	1.48%	<0,1%	1.28%	1.11%	<0,1%	1.28%	<0,1%

Table 6-6: Combined linear limits (% strain at 10 Hz) for the three binders studied.

The following three figures (Figure 6-5 – Figure 6-7) are graphical comparisons of the linear limits at the different test temperatures for the three binders.
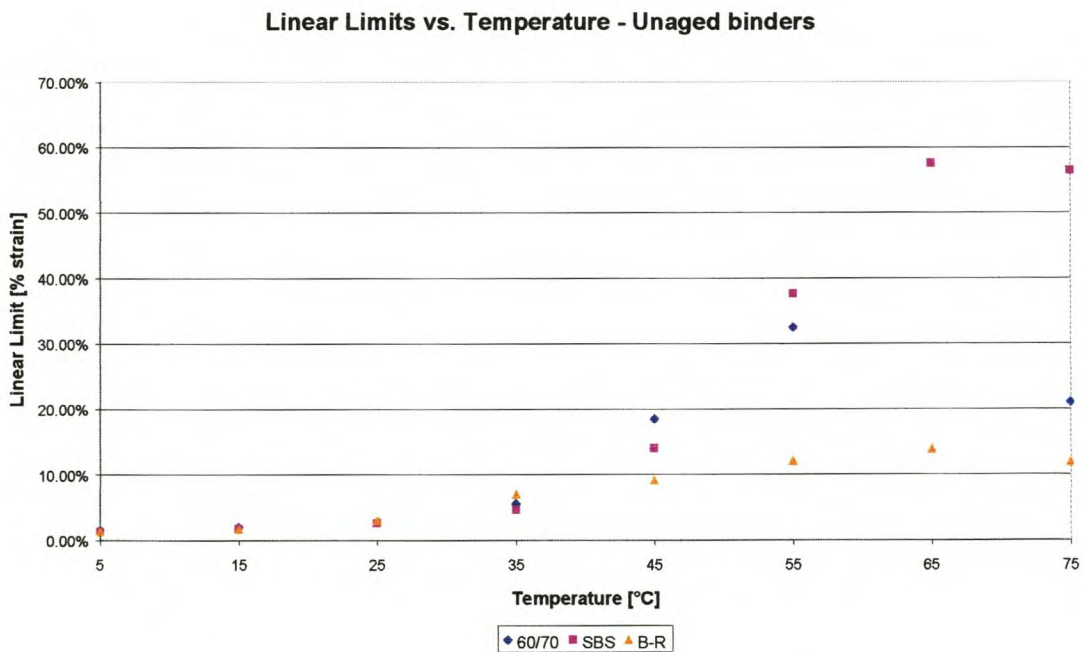
Figure 6-5: Comparison of the three unaged binders' linear limits (% strain) at 10 Hz.

Linear Limits vs. Temperature - RTFOT aged binders
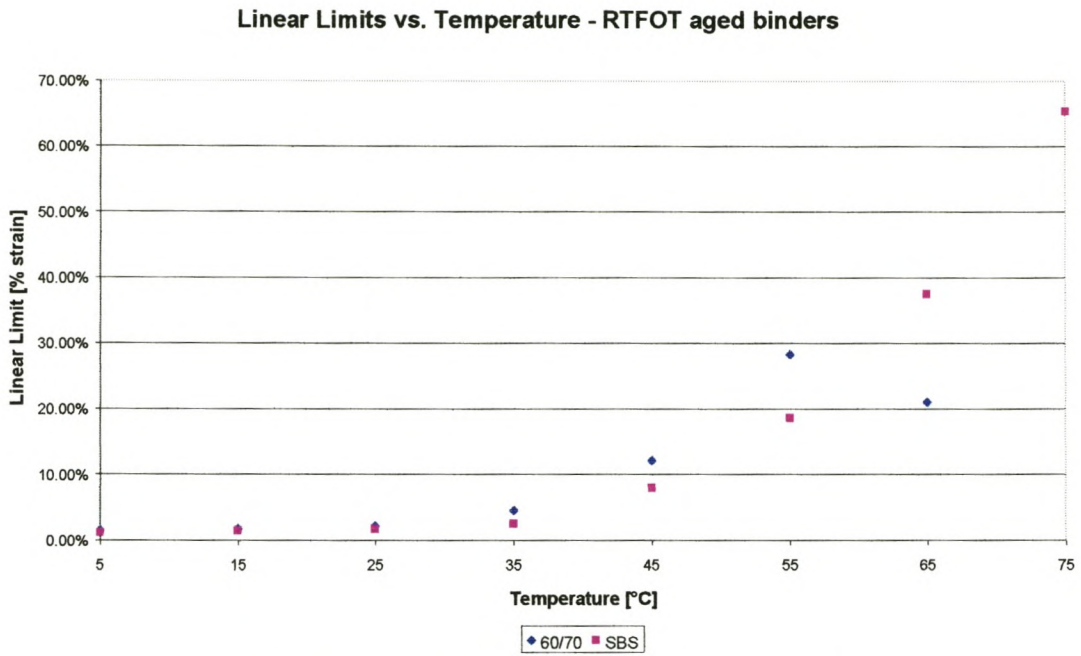
Figure 6-6: Comparison of the two aged binders' linear limits (% strain) at 10 Hz.

Linear Limits vs. Temperature - Binder/Filler Systems
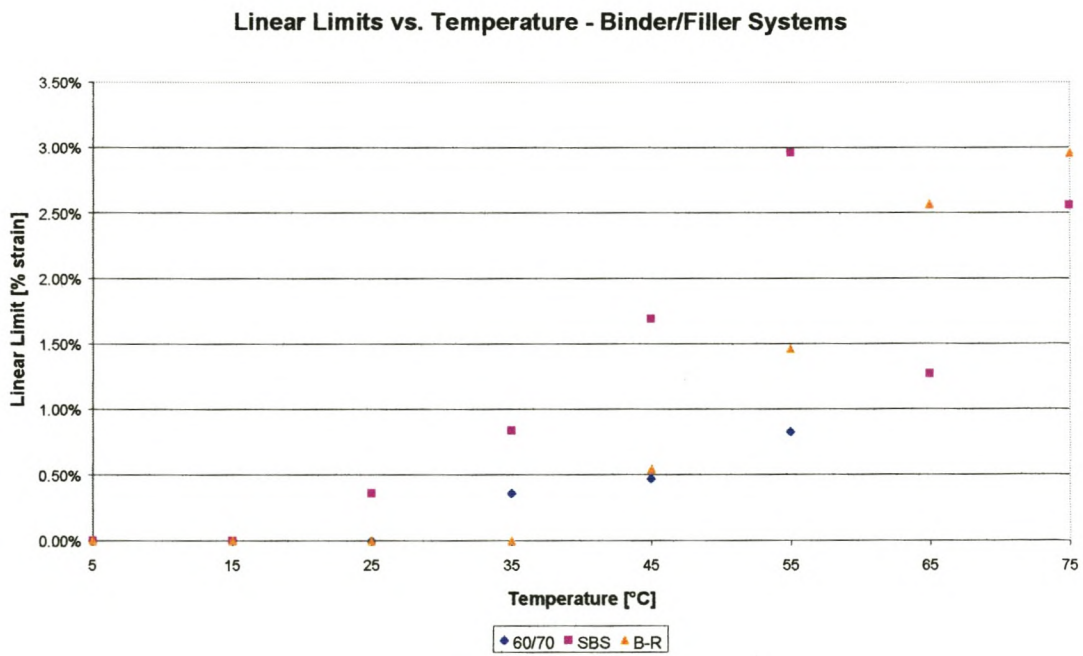
Figure 6-7: Comparison of the three binder/filler systems' linear limits (% strain) at 10 Hz.

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The results in Table 6-6 and from the three graphs presented here show the differences between the three binders clearly. The following comments can be made:

- For the unaged binders, it is clear that the SBS has higher linear limits compared to the 60/70 and Bitumen-Rubber at the higher temperatures. The decrease in temperature affects the SBS more. This is evident from Figure 6-5, when the SBS curve is compared to the 60/70 and Bitumen-Rubber curves.

- It is also evident from Figure 6-5 that the Bitumen-Rubber's linear limits do not increase as rapidly with temperature as the other two binders.

- When the two aged binders are compared, it is again noticeable that the decrease in temperature affects the SBS more than the 60/70 (Figure 6-6). It can also be seen that the ageing affects the linear limits when compared to the limits for the unaged binders. The difference between the unaged and aged binders is more marked for the SBS binder than for the 60/70 binder. The Bitumen-Rubber could unfortunately not be aged.

- When the binder/filler systems are compared, it can be seen that the 60/70 is affected more by the addition of filler. It would seem that the Bitumen-Rubber is affected the most by the decrease in temperature.

In closing, it must be noted that the frequency sweeps were performed with these results as guideline for the selection of strain levels at the different temperatures. The strain levels that were used are reported in Table 6-2.

6.2.2 Frequency Sweeps

In this part the results of the frequency sweeps on the binders will be discussed. As with the discussion on the results of the strain sweeps, this part will be divided into smaller sections dealing with each of the binders. The smaller sections will contain the frequency sweeps on each of the binders as well as the Master Curves and Black Diagrams drawn from the data obtained. At the end of this part there is a section where the different

binders' results will be compared by looking at the Black Diagrams of the binders. A discussion of the algorithm used for drawing the Master Curves is presented on the CD in Appendix B.

6.2.2.1   60/70

This section contains the results of the frequency sweeps performed on the unaged, RTFOT aged binders and the binder/filler system. The data obtained during testing is given in Appendix B on the CD.

This section contains a figure of each of the different binder's (unaged (Figure 6-8), RTFOT aged (Figure 6-9) and binder/filler (Figure 6-10)) frequency sweeps. A figure (Figure 6-11) showing the master curves drawn from the frequency sweep data is also shown. A Black Diagram (Figure 6-12) containing the three binders' data is also shown.
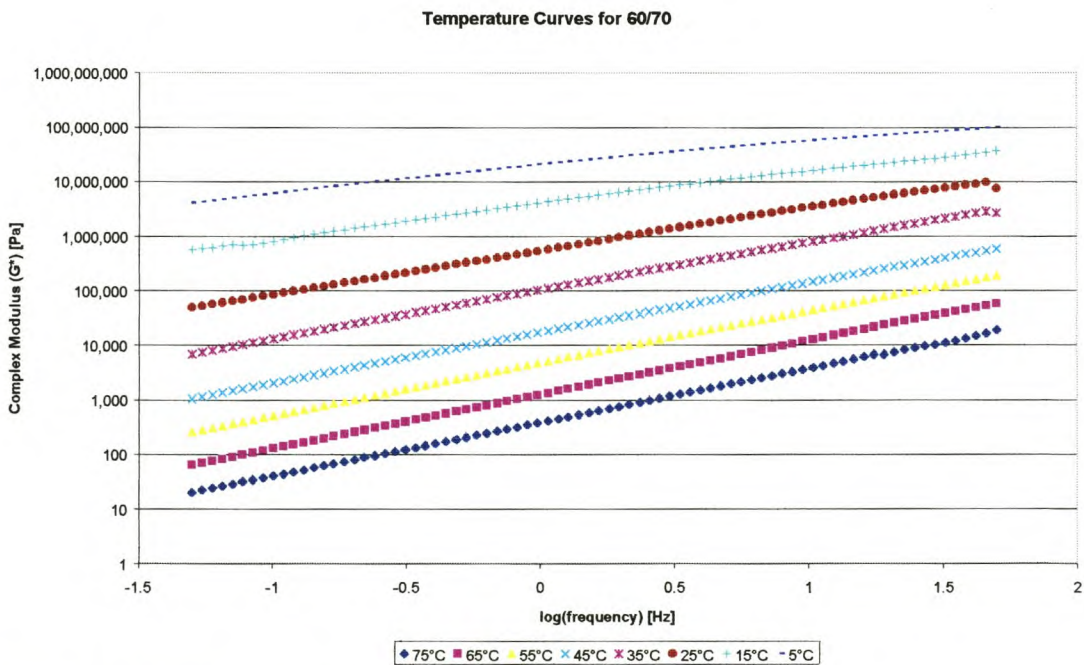
Figure 6-8: Frequency sweeps on 60/70 bitumen.

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Temperature Curves for 60/70 RTFOT residue
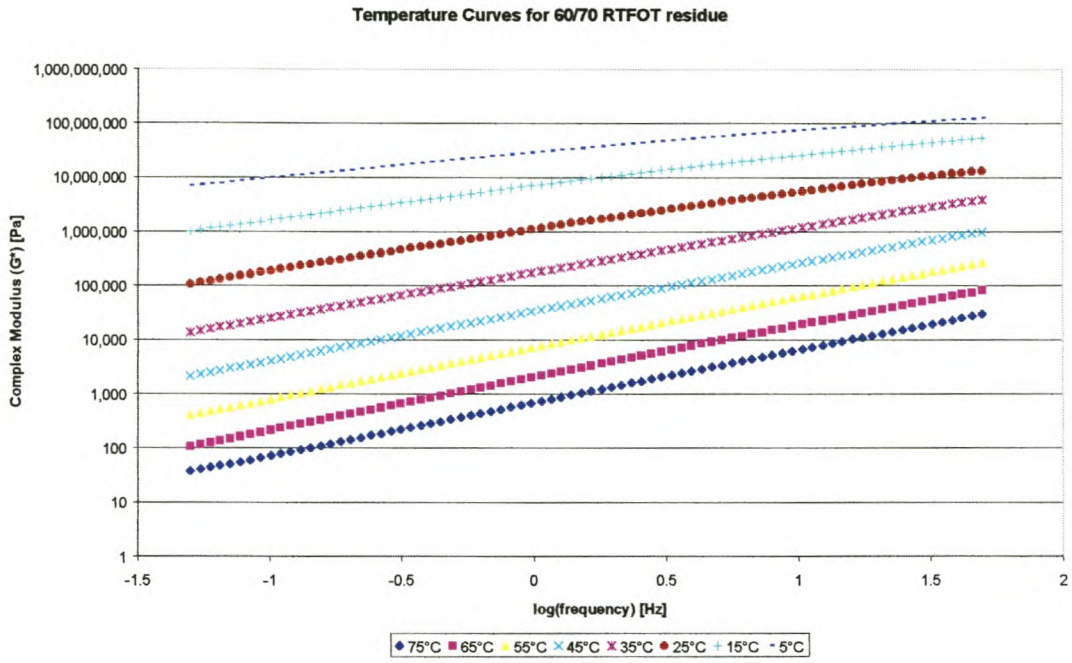
Figure 6-9: Frequency sweeps on 60/70 RTFO residue.

Temperature Curves for 60/70 + Filler
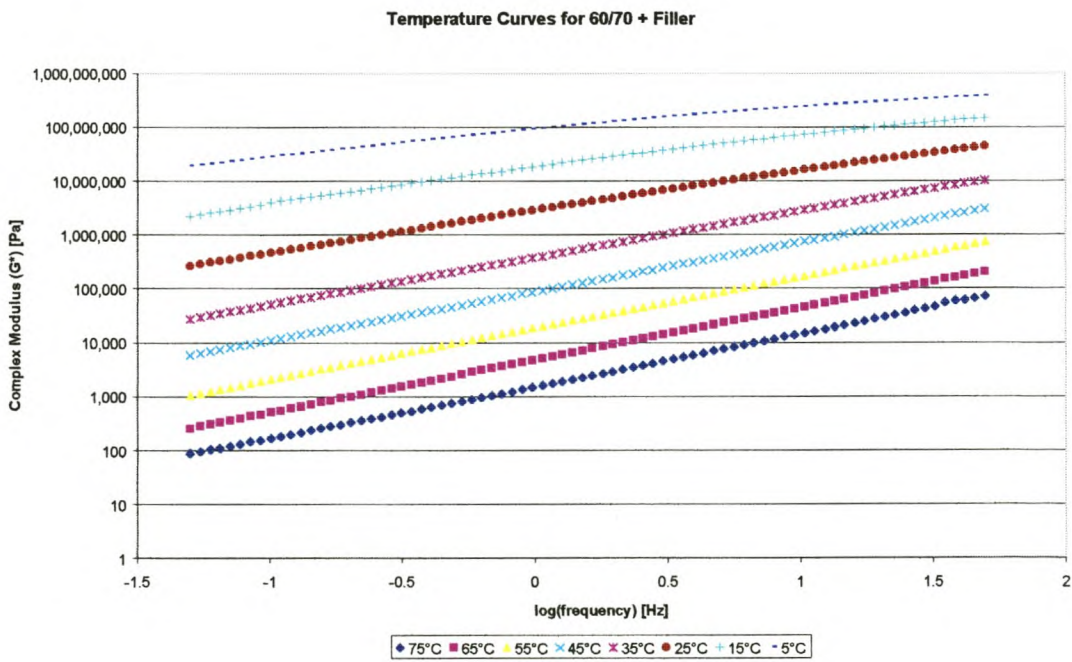
Figure 6-10: Frequency sweeps on 60/70 + Filler.

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Combined Master Curves 60/70
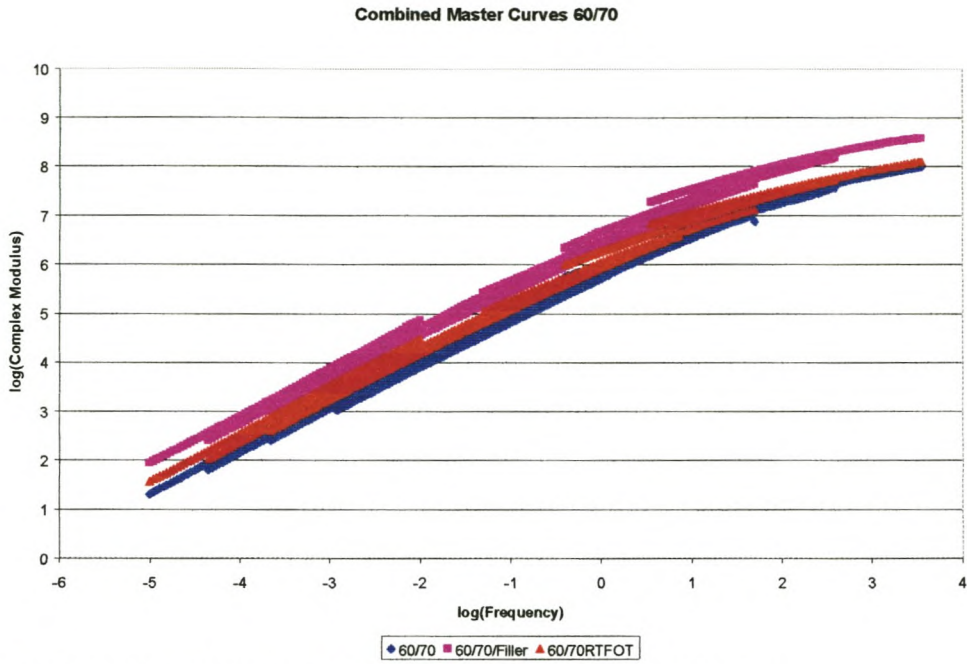
Figure 6-11: Master Curves at 25 °C of the three different 60/70 binders.

Combined Black Diagrams 60/70
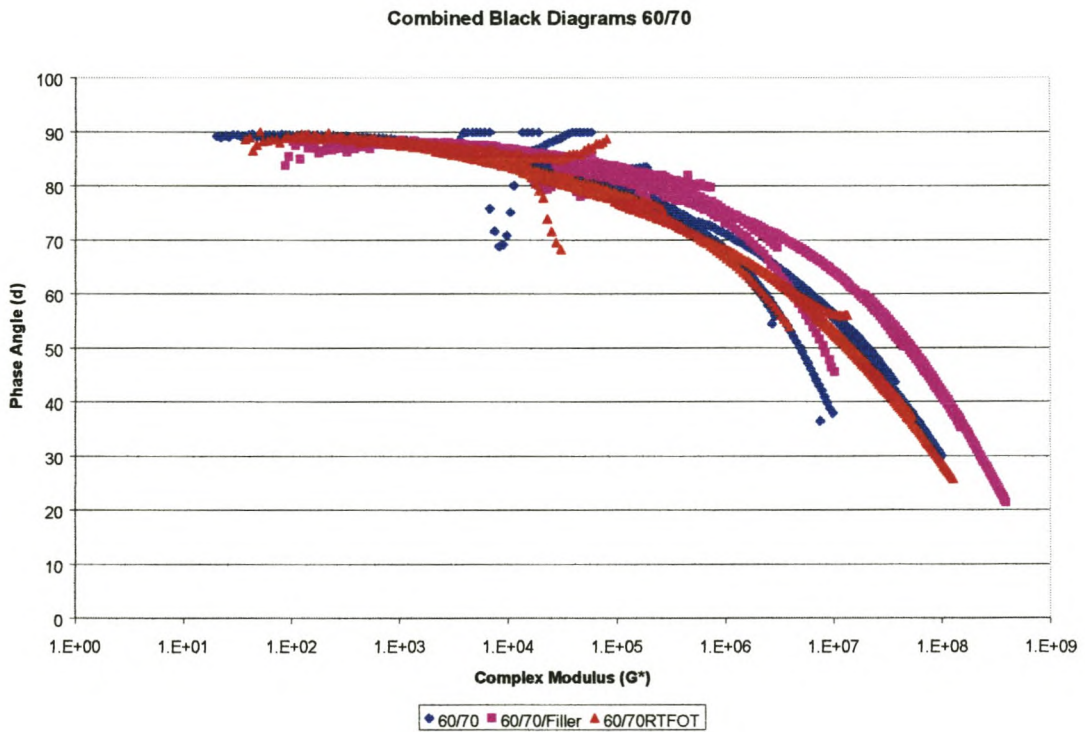
Figure 6-12: Black Diagrams of the three different 60/70 binders.

In reference to the figures presented in this section the following points should be noted:

- The temperature curves (frequency sweeps) for the unaged, aged and binder/filler cases take the same general form. (Figure 6-8, Figure 6-9 and Figure 6-10)
- When looking at the Master Curves (Figure 6-11) the following comments can be made:
  - From the figure, it can be seen that there is a stiffening effect when the binder is aged (RTFOT Master Curve shifted vertically).
  - The influence of filler addition is apparent: There is a stiffening effect (vertical shift of Master Curve) but the form of the curve stays the same – this suggests that the filler does not affect the binder properties.
- Referring to the Black Diagrams (Figure 6-12) the following can be noted:
  - The Black Diagram for the aged binder is shifted to he right (stiffer response), but the form of the curve stays the same – this suggests that the ageing effect does not affect the binder properties.
  - The binder/filler system's Black diagram is shifted more than that of the aged binder's Black Diagram – this means the binder/filler system is much stiffer. The form of the curve stays the same, which suggests that the filler does not influence the binder properties.

6.2.2.2   SBS Modified Binder

This section contains the results of the frequency sweeps performed on the SBS modified binders.

Figure 6-13 to Figure 6-15 show the frequency sweeps on each of the binders. Figure 6-16) shows the Master Curves drawn from the frequency sweeps. Figure 6-17 contains the Black Diagrams of the binders.

Temperature Curves for unaged SBS
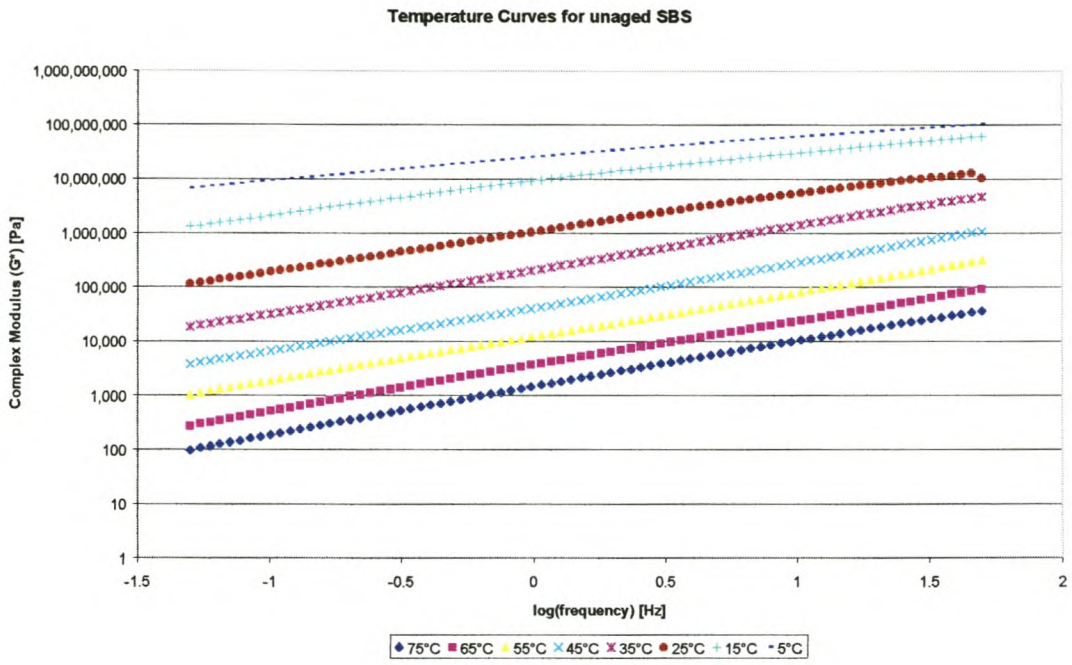
Figure 6-13: Frequency sweeps on unaged SBS modified binder.

Temperature Curves for SBS RTFOT residue
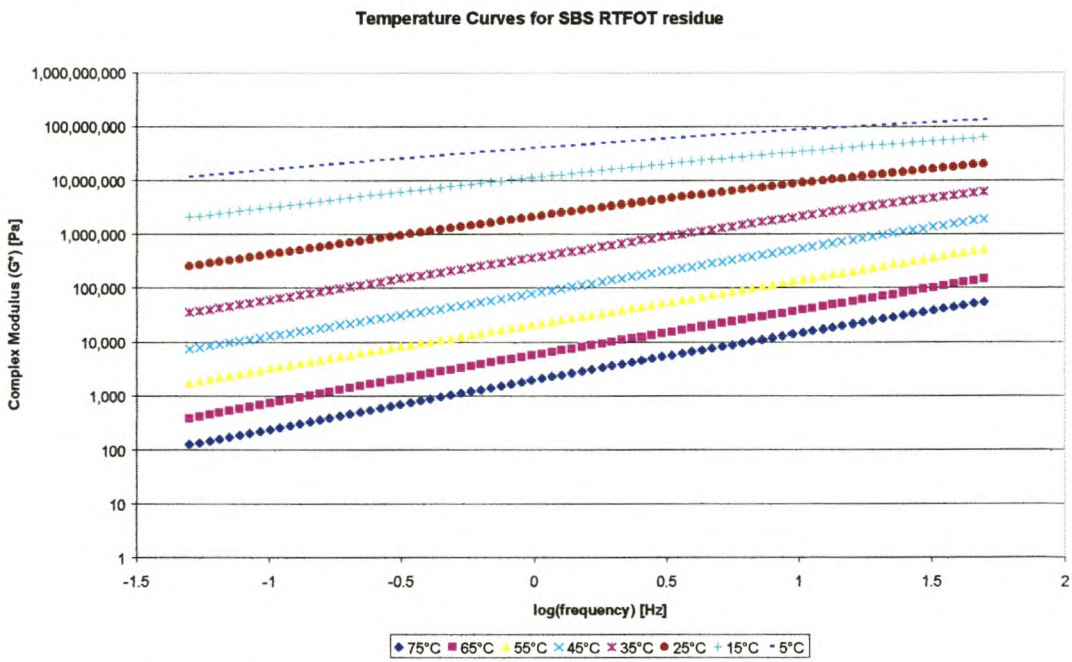
Figure 6-14: Frequency sweeps on RTFO residue of SBS modified binder.

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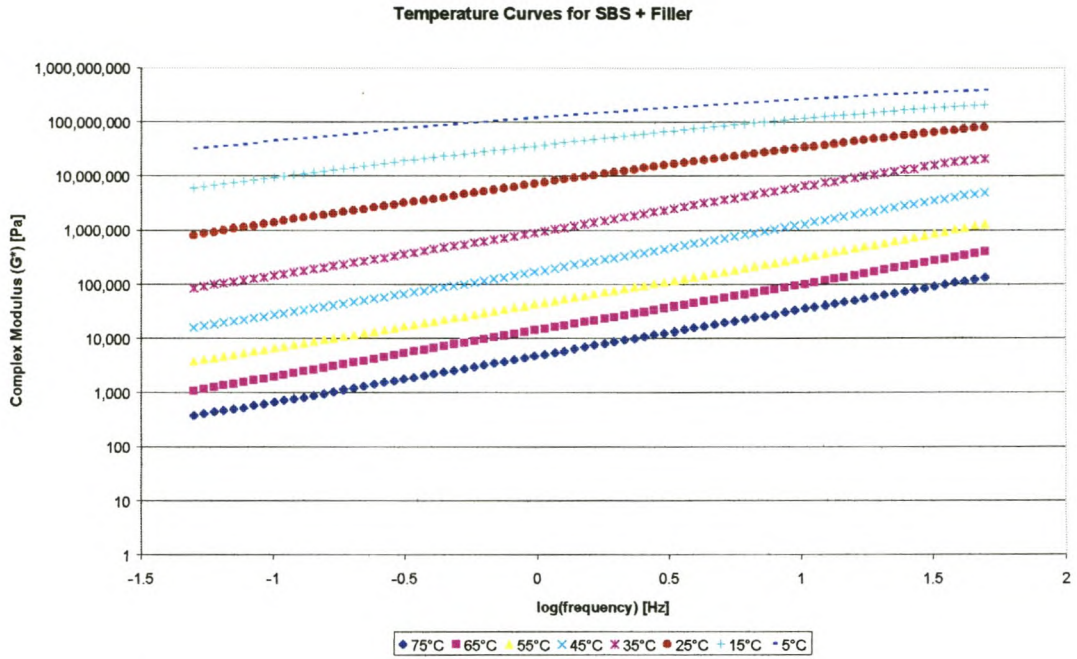
Figure 6-15: Frequency sweeps on SBS modified binder + Filler.
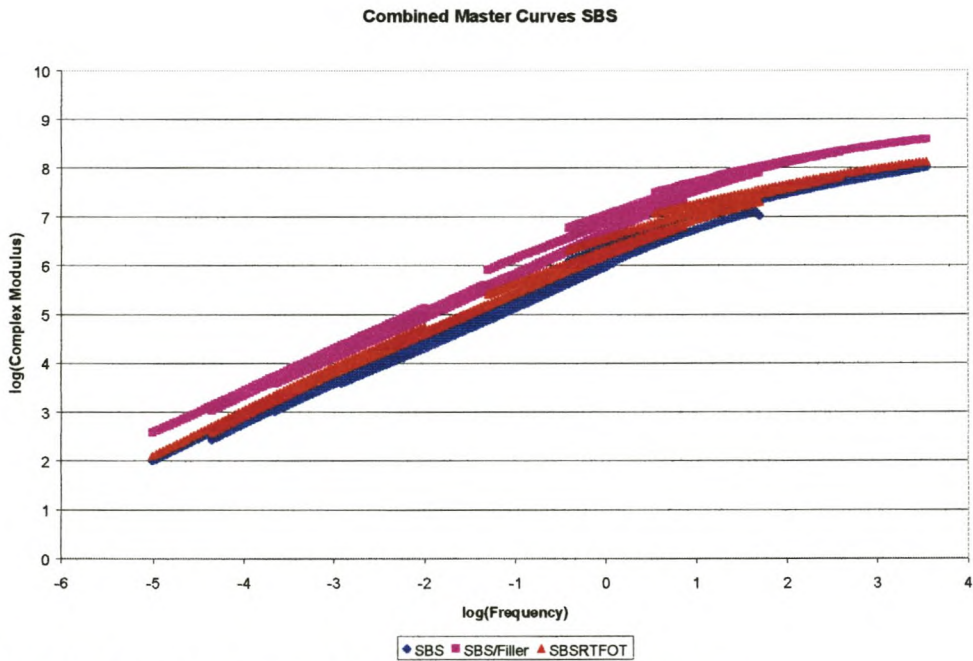
Figure 6-16: Master Curves at 25 °C for the SBS modified binders.

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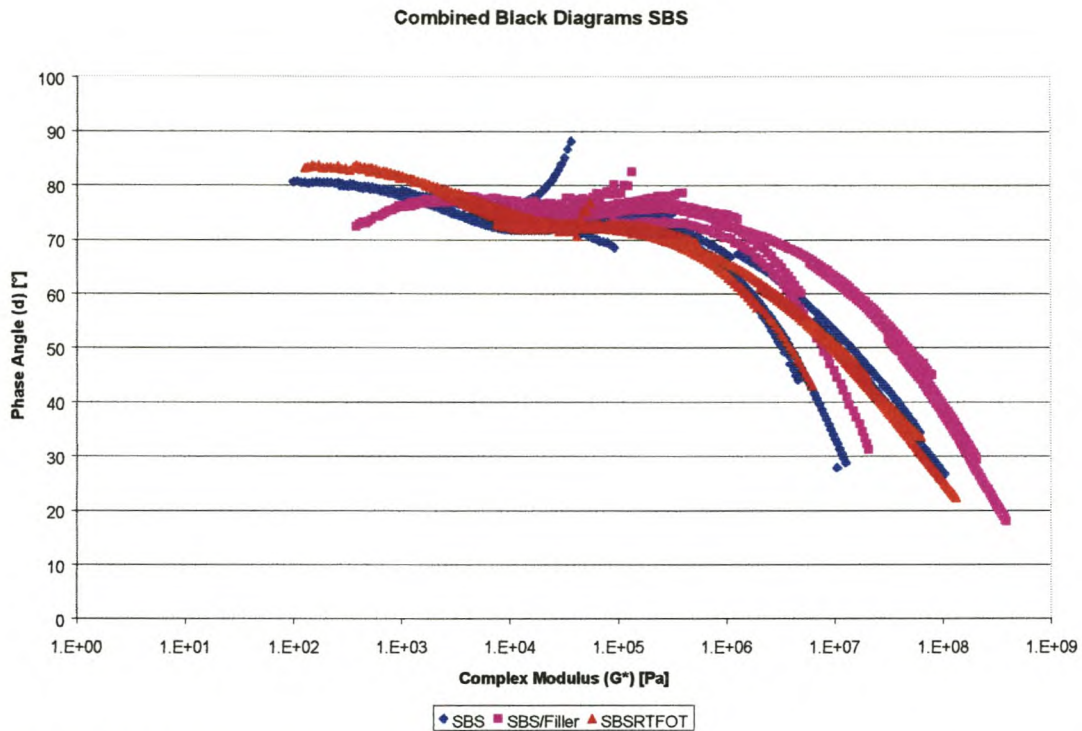
Figure 6-17: Black Diagrams for the SBS modified binders.

With reference to the figures presented in this section, the following comments can be made:

- As was the case for the 60/70 binders, the general form of the frequency sweeps stays the same (Figure 6-13, Figure 6-14 and Figure 6-15) for the different cases.

- From the Master Curves (Figure 6-16) the following comments can be made:
  - Comparing the unaged Master Curve with that of the aged binder, the effect of ageing is clear – there is a slight stiffening effect (slight vertical shift at a frequency).
  - The effect of filler addition to the binder is also apparent on this figure – the addition of filler stiffens the response to loading, but does not change the form of the curve. This suggests that the filler does not affect the binder properties.

- Referring to the Black Diagrams (Figure 6-17) the following can be said:

- o The unaged and aged binders have the same form for the Black Diagram – the aged binder is shifted slightly to the right (stiffer).

- o The binder/filler system has the same form as the unaged binder, but it is shifted to the right of that curve – this suggests that the filler does not affect the binder properties of the SBS binder.

- o The general form of the Black Diagrams for the SBS binders differs from the Black Diagrams of the 60/70 binders. The SBS does not tend monotonously toward a phase angle of 90°, in fact there is a decrease apparent in the value of the phase angle at lower values of the complex modulus. The lower values for the complex modulus would be encountered at high temperatures or slow loading times. This is an improved performance characteristic when compared to the 60/70 binder and would suggest an improved rutting performance for the SBS binder when compared to that of the 60/70.

6.2.2.3   Bitumen-Rubber

In this section the results of the frequency sweeps performed on the Bitumen-Rubber is discussed.

Figure 6-18 and Figure 6-19 show the frequency sweeps on the Bitumen-Rubber and the Bitumen-Rubber + Filler. Figure 6-20 shows the Master Curves drawn from the frequency sweeps. Figure 6-21 shows the Black Diagrams drawn from the results of the frequency sweeps.
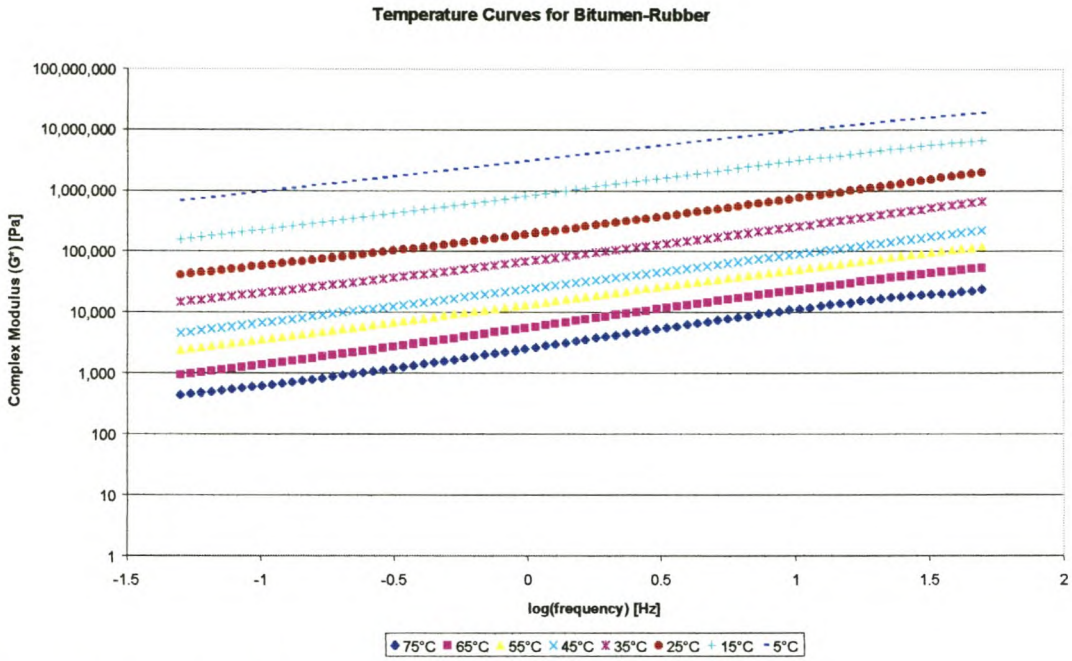
Figure 6-18: Frequency sweeps on Bitumen-Rubber.
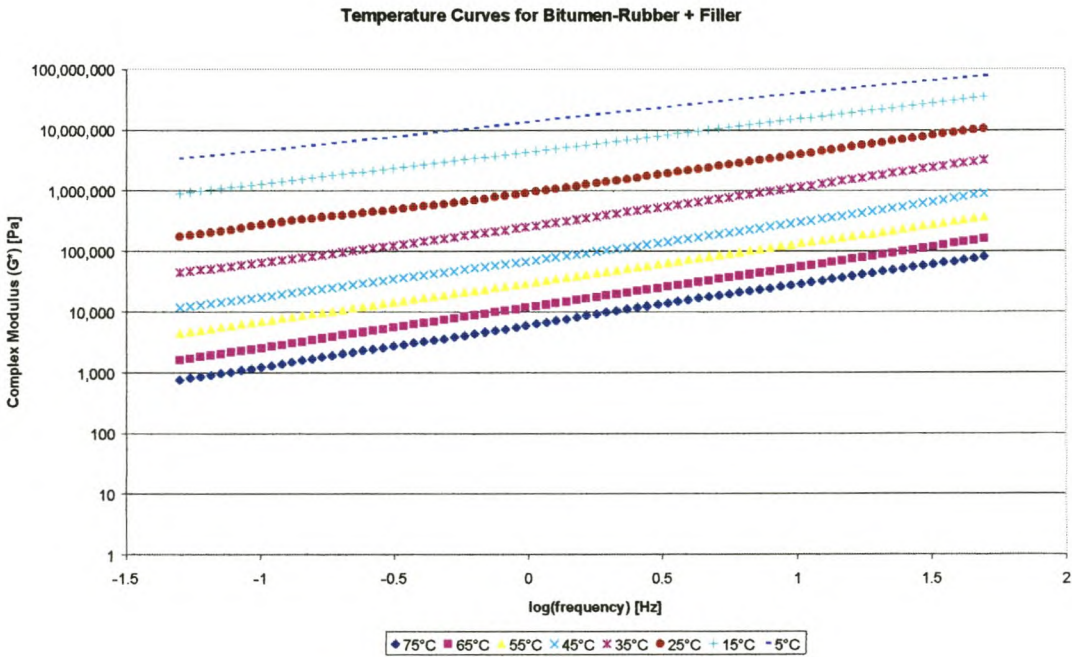
Figure 6-19: Frequency sweeps on Bitumen-Rubber + Filler.

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Combined Master Curves Bitumen-Rubber
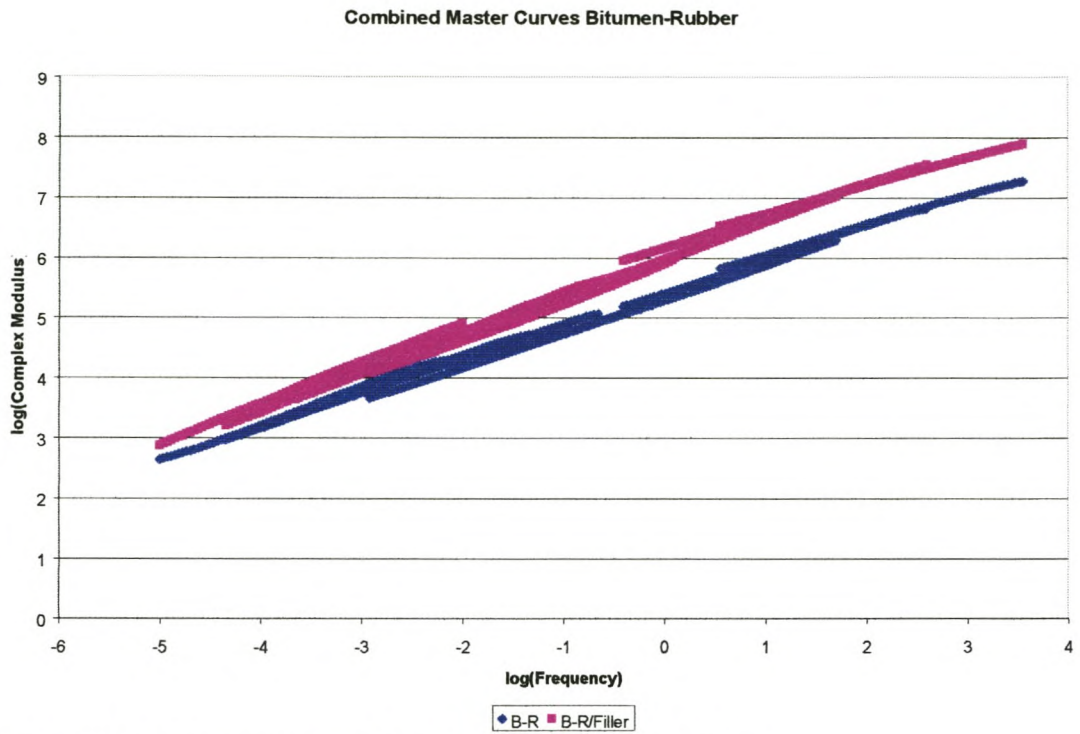
Figure 6-20: Master Curves at 25 °C for the Bitumen-Rubber binders.

Combined Black Diagrams Bitumen-Rubber
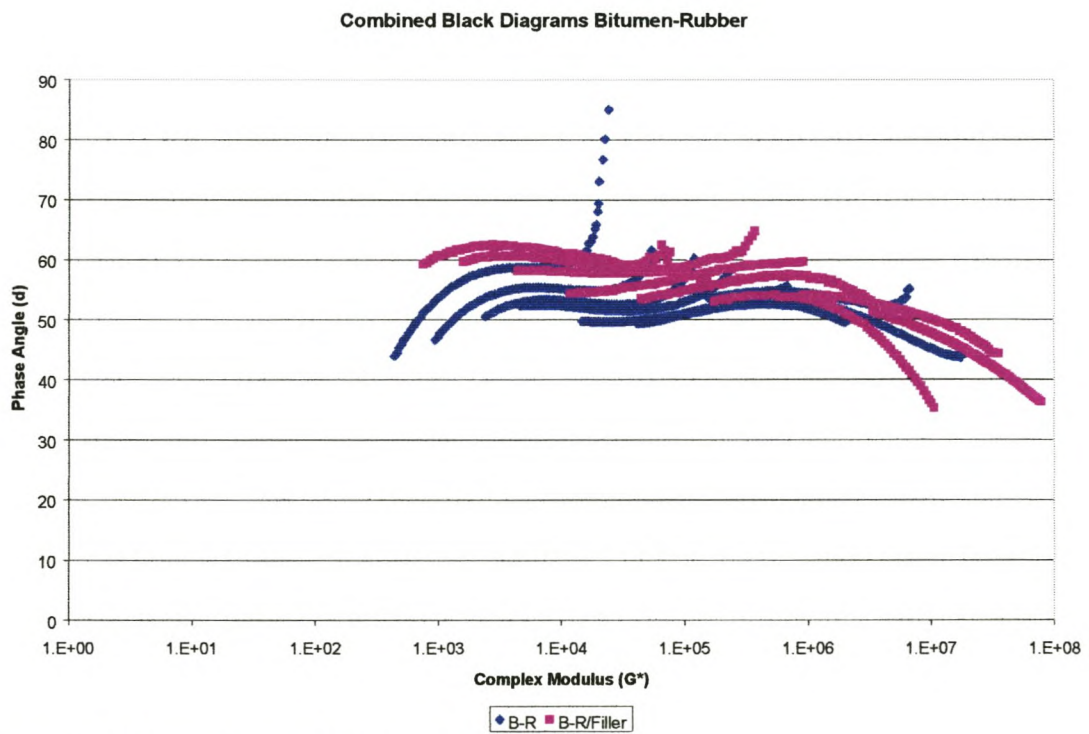
Figure 6-21: Black Diagrams for Bitumen-Rubber.

There is no data for aged bitumen-rubber because the binder could not be aged in the Rolling Thin Film Oven. Therefore, this discussion will be centred on the unaged and binder/filler systems for this binder.

With reference to the figures presented in this section, the following comments can be made:

- The frequency sweeps of the bitumen-rubber and bitumen-rubber/filler systems (Figure 6-18 and Figure 6-19) have the same general form.
- The following comments can be made when looking at the Master Curves (Figure 6-20):
    - From the Master Curves of the bitumen-rubber it is clear that the addition of filler leads to a stiffening effect that is more pronounced at higher frequencies than at lower frequencies.
    - The rate of increase in complex modulus with increasing frequency is higher at higher frequencies for the binder/filler system than for the binder system.
    - At lower frequencies the rate of increase in complex modulus with increase in frequency is basically the same for the two systems.
- From the Black Diagrams (Figure 6-21) the following comments can be made:
    - At the lower values of the complex modulus, it would seem that the filler addition has a negative effect on the binder – the curve shifted vertically.
    - The Black Diagrams for this binder has the same general form than that of the SBS binder. However, the effect of binder modification is more pronounced in this case when compared to the SBS. This is clear when considering the fact that the maximum phase angle is approximately 60° for the binder and for the binder/filler system the maximum value is approximately 65°.

○ The form of the bitumen-rubber's Black Diagram would suggest a very good performance at high temperatures or long loading times (circumstances related to rutting of asphalt mixes).

6.2.2.4   Discussion of Frequency Sweep Results

Figure 6-22 presents the Black Diagrams for the three binders. From this figure, some general comments on the expected performance of the binders can be made when the three curves are compared.

Combined Black Diagrams
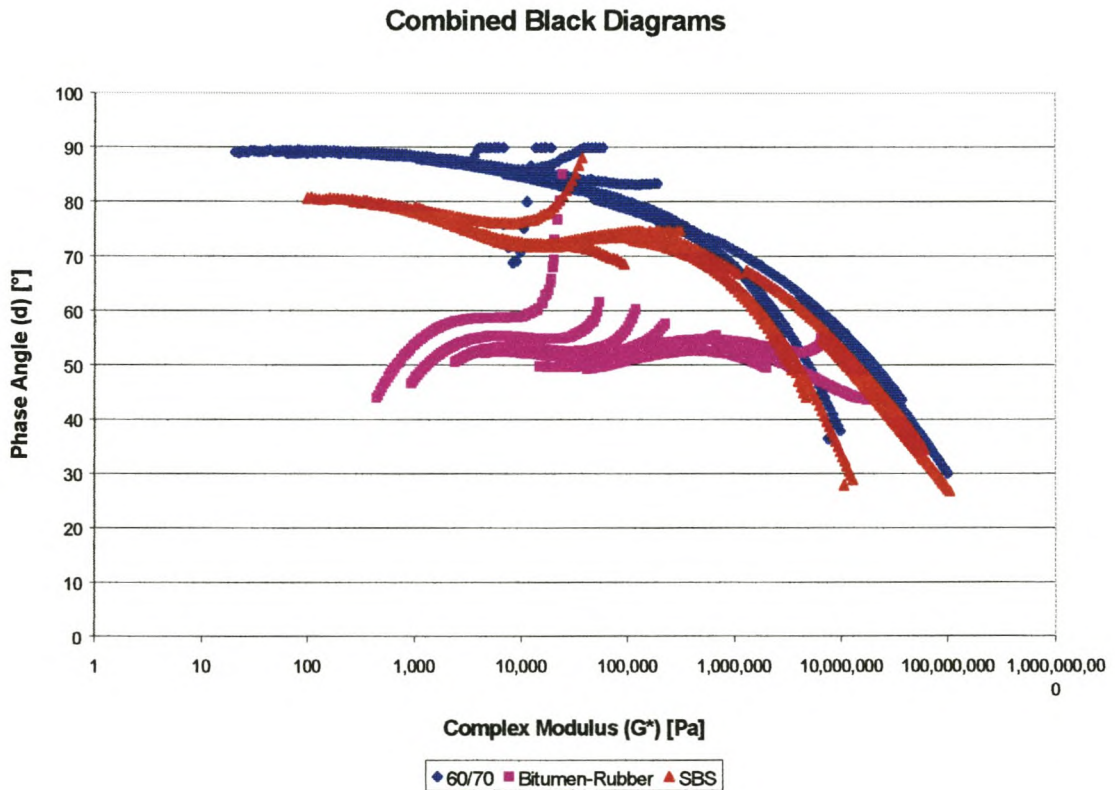
Figure 6-22: Black diagrams of the three binders used for the DSR testing.

The following comments can be made regarding the three curves presented in Figure 6-22:

- From these curves the effect of polymer modification of binders is apparent.

- It is clear that the SBS and rubber modified binders will give an improved performance at the lower values of the complex modulus (G*). This improved performance is due to the much lower values for the phase angle for both the SBS and Bitumen-Rubber binders compared to the 60/70. The lower values for the complex modulus are encountered at high temperatures and/or long loading times (low frequencies) – these are also the prevailing conditions in which rutting of asphalt mixes occur.

- These binders can be ranked in terms of expected rutting rate from these curves as follows, starting with the lowest rutting rate:
    1. Bitumen-Rubber
    2. SBS modified 60/70
    3. 60/70

- From these comments, it would seem that the bitumen-rubber would have a significant impact on the life of an asphalt mix by increasing the expected life of the mix in terms of rutting.

6.2.2.5   Discussion of Black Diagrams

Two unusual phenomena are apparent on the Black Diagrams presented in the previous sections. These two phenomena are pointed out on Figure 6-23 and will be discussed further in this section.

The first phenomenon has to do with the spindles of the measuring system. On each of these curves it can be seen that there is one line that deviates from the curve. It was found that the data for the line that deviates was usually obtained with the 25mm spindle at 35°C. Thus, it might be that the binder was to stiff at this temperature for the chosen spindle size to obtain good measurements. The 8mm spindle was used from 25°C and

lower and the data obtained from those measurements fits in seamlessly with the other data. The suggestion can therefore be made that the 8mm spindle be used from 35°C and lower when frequency sweeps are performed.

The second phenomenon has to do with the high temperature measurements. It can be seen that there is scatter in the data obtained during the frequency sweeps at the high temperatures. This phenomenon is characteristic of the frequency sweeps at the high temperatures (75°C, 65°C and 55°C) and at the high end of the frequency range (f > approximately 20 Hz). It is probably the inertia of the bituminous fluid tested at the high frequency and high temperature that is influencing the measurements at these conditions.
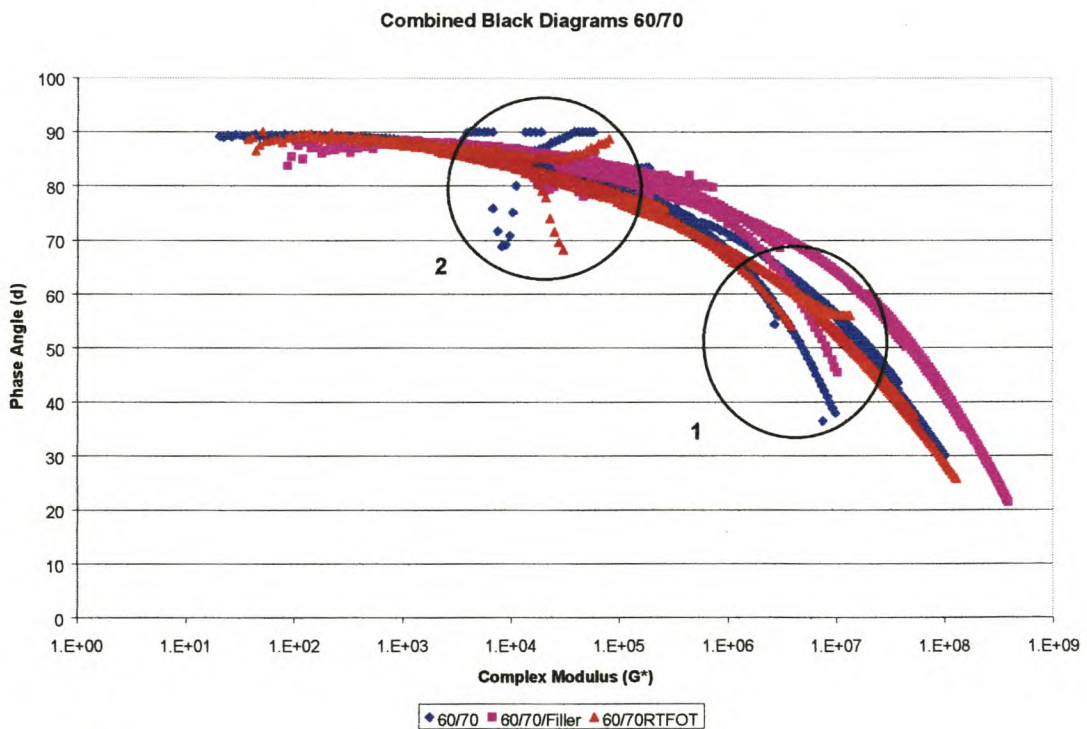
Figure 6-23: Two unusual phenomena on Black Diagrams.

It was decided to leave these phenomena on the Black Diagrams to give a true representation of the results of the frequency sweeps. The Black Diagrams did not prove to be difficult to interpret because of these phenomena and the general trend in the curves

is still clear. The effect of ageing and filler addition is also still clear on these curves. It should also be noted that the phenomena discussed was not unique to a binder but manifested for all three binders studied.

6.3 Conclusions

The strain sweeps showed that the temperature at which a binder is tested has a definite effect on the linear limit of the material. The linear limit is also a function of the frequency at which the strain sweep is done. The higher the frequency, the lower the linear limit will be at a temperature and vice versa. Thus, to determine the linear limit at a relatively high frequency for a specific temperature would ensure that the assumed value of the linear limit would be on the conservative side for all the frequencies lower than the one used. The problem of higher frequencies, where the linear limit would be lower than the determined value, was overcome by using a value for the strain well below the determined linear limit. This was successful to ensure that the material is tested within the linear visco-elastic range of strain (no non-linear behaviour was observed).

The strain sweeps were successful for determining appropriate values of strain below the linear limit for use during the frequency sweeps.

Good results were obtained from the frequency sweeps. The influence of polymer modification, ageing and filler addition were all well described from the results of the frequency sweeps.

Polymer modification was shown to increase a binder's elastic response at high temperatures/low frequencies. High temperatures and low frequencies (slow moving or static traffic) are the prevalent conditions in the road surface when rutting is recorded. It was shown that the addition of rubber to bitumen was the most beneficial mode of modification studied, when aiming at an increased resistance to permanent deformation.

The frequency sweeps on the aged binders showed the effect of ageing. The binders' response to loading was stiffer after ageing. Thus, ageing results in a stiffer binder. This phenomenon has been widely reported in the industry and has been shown fundamentally here.

It was shown that filler addition did not affect the binders negatively, i.e. binder properties were not altered by the addition of filler. The binder/filler systems' response to loading was much stiffer than that of the binders. This shows that the addition of filler results in a mastic that is much stiffer than the binder is.

7 Creep Tests with the Dynamic Shear Rheometer on Binders and Binder/Filler Systems

In this chapter the creep tests that were performed on the binders and binder/filler systems using the DSR are reported. The creep tests were performed with the DSR in order to compare the results with the results from the SPR tests. The temperatures that were used are given in the table below (Table 7-1).

Material	Temperature		
	10 °C	25 °C	40 °C
Binders	✓	✓	✓
Binder/Filler Systems		✓	✓

Table 7-1: Temperatures used for creep tests performed with DSR.

These tests were performed under similar conditions (stress controlled) to that of the SPR tests described in Chapter 5, in order to evaluate the use of the SPR as an instrument to characterise the influence of different additives to bitumen.

The equipment used and sample preparation were discussed in chapter 4. Sample thickness used was 2mm in all cases. In terms of the binder/filler systems, the sample thickness was assumed adequate since the maximum particle size of the filler is 0,1mm.

7.1 Creep Tests

In this section, the creep tests that were performed with the DSR are described. This section is divided into a section describing the purpose of the tests and a section describing the methodology used.

7.1.1 Purpose

These creep tests were performed to compare the results with those obtained with the Sliding Plate Rheometer (SPR). The description of the tests performed with the SPR can be found in Chapter 5.

The comparison was to be done by drawing shear susceptibility lines for the DSR results and comparing these lines with the lines drawn from the SPR results. Calculated viscosity values were also used to compare the results from the SPR with the DSR.

7.1.2 Methodology

A standard flow test (viscosity test) was chosen on the standard test types menu of the DSR software. The standard flow test can be set up to perform a creep test on the binders and binder/filler systems.

To accurately compare the results of these tests with that of the SPR, it was decided to do the DSR tests in the stress controlled mode. This meant that a constant shear stress was applied to the sample and the rate of deformation was recorded during the test. This is the same approach followed when creep tests are performed with the SPR (Figure 7-1).
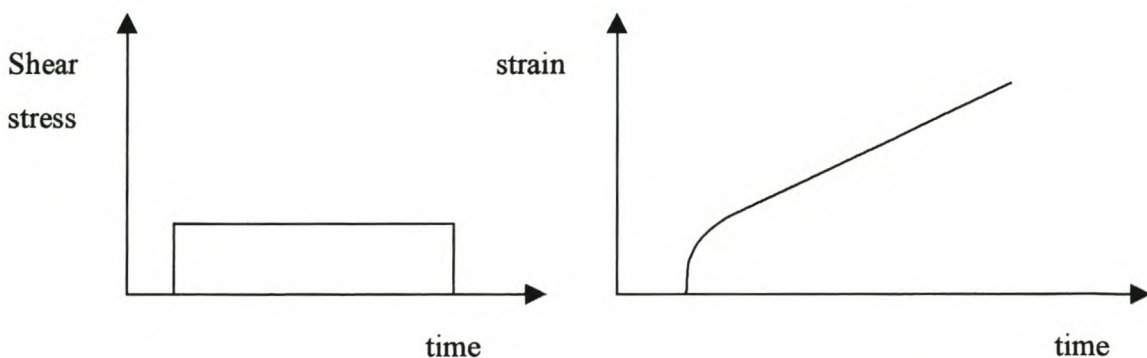
Figure 7-1: Schematic representation of procedure for creep test.

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The deformation behaviour of bituminous materials is studied with the creep tests. The aim of the creep test is to reach a constant rate of deformation at the end of the test.

The results of these tests were plotted as shear stress versus shear rate (shear susceptibility lines) and these lines were compared with the lines obtained with the SPR.

Table 7-2 shows the test plan that was followed. Unlike the SPR tests, only one repetition was made at a stress level for the DSR tests. For the SPR tests three repetitions were made at every stress level used.

MATERIAL	Number of Stress Levels		
	40 °C	25 °C	10 °C
60/70	4	4	4
SBS	4	4	4
Bitumen-Rubber	4	4	4
60/70 + Filler	4	4	==
SBS + Filler	4	4	==
Bitumen-Rubber + Filler	4	4	==
TOTAL NUMBER OF TESTS: 60			

Table 7-2: Test programme for DSR creep tests.

7.2   Results and Discussion

It was mentioned earlier that this part of the work was done to compare the results with the results of the Sliding Plate Rheometer (SPR) work. This section contains the results of the creep tests performed on the DSR as well as a discussion of the results for each of the different binders.

The figures presented here contain a line drawn from the results of the SPR tests and a line drawn from the results of the DSR tests. These lines, together with calculated viscosity values, form the basis of the comparison and discussion that follows.

7.2.1 60/70

The results of the creep tests on the 60/70 bitumen and binder/filler mixes will be presented and discussed in this section. The data from which these results were calculated can be found in Appendix A on the CD.

From the figures presented here (Figure 7-2 and Figure 7-3), it can be concluded that:

- At 10 °C and 25 °C the SPR tests recorded a stiffer response to loading for the binder compared to the results from the DSR – this can be seen in the shift of the lines from the DSR tests to the right of the lines from the SPR.
- In all the cases the slopes of the DSR shear susceptibility lines are steeper than that of the SPR shear susceptibility lines.
- In the case of the binder/filler systems the DSR recorded stiffer responses to loading compared to the SPR.

Combined Creep Results 60/70 - SPR and DSR
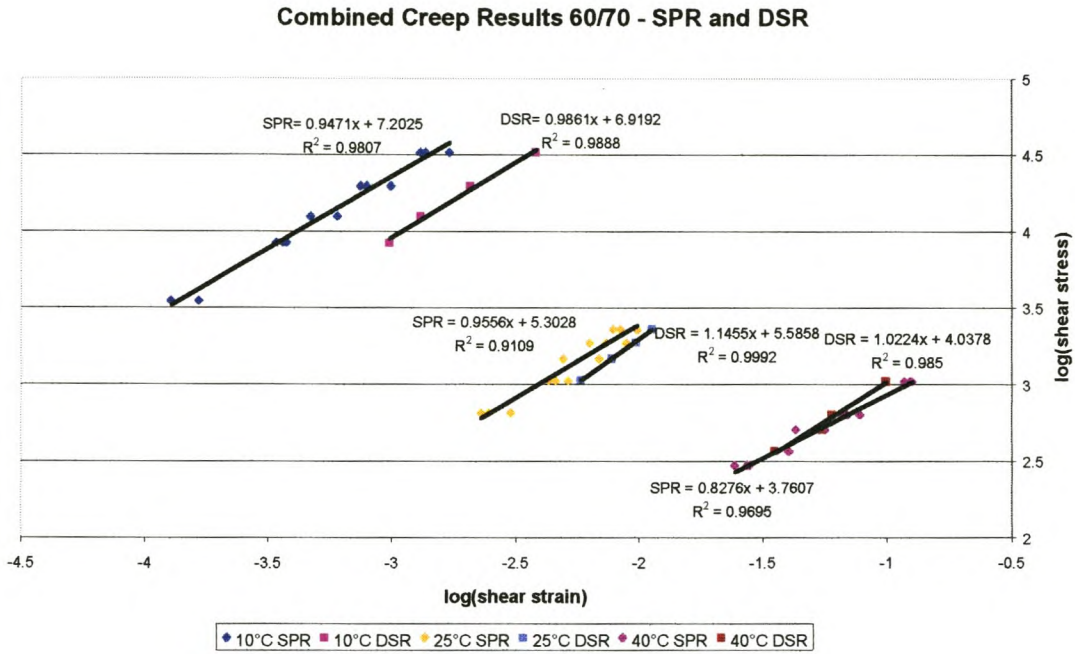
Figure 7-2: Shear susceptibility lines derived from creep test results with SPR and DSR for 60/70.

Combined Creep Results 60/70+Filler - SPR and DSR
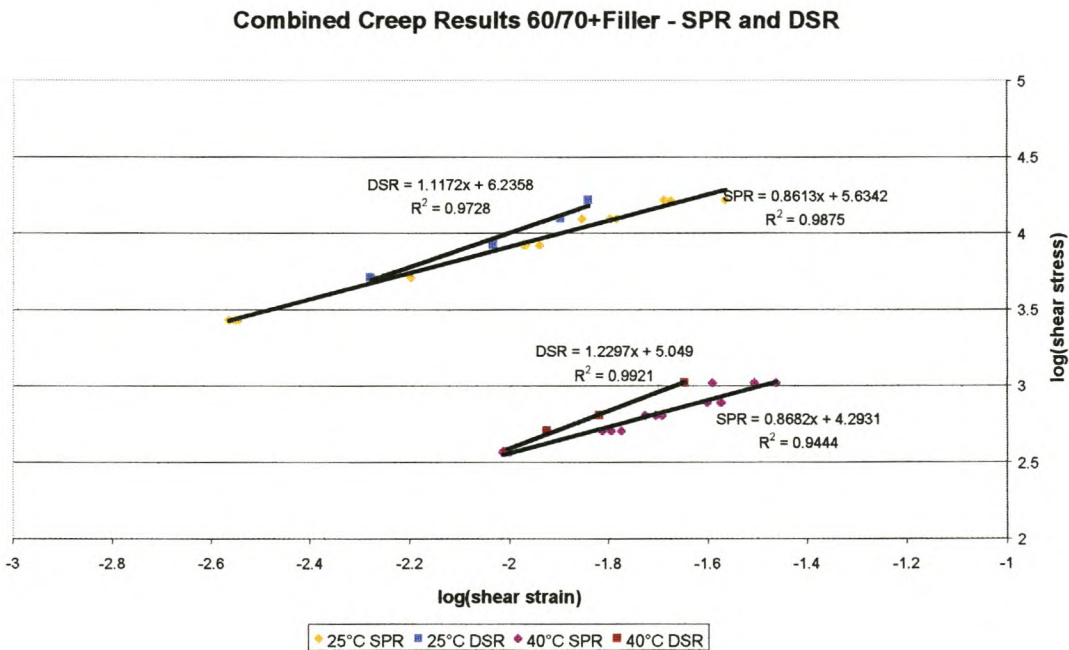
Figure 7-3: Shear susceptibility lines derived from creep test results with SPR and DSR for 60/70+Filler.

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Table 7-3 gives the calculated viscosity values for the results of the creep tests on the 60/70 bitumen and binder/filler system. The viscosities were calculated at the end of the tests assuming a constant shear rate. The average of the values, the standard deviation and the coefficient of variation (COV) are also shown.

Binder	Temperature	Test Number	Calculated Viscosity	Average	Standard Deviation (COV) %
60/70	10°C	1	8,565,059.6	9,077,099.7	570,066.4 (6.3)
		2	9,566,299.7		
		3	9,574,917.1		
		4	8,602,122.4		
	25°C	1	183,170.8	192,356.6	8,283.3 (4.3)
		2	189,291.4		
		3	194,179.8		
		4	202,784.5		
	40°C	1	10,444.6	10,248.6	544.5 (5.3)
		2	9,436.8		
		3	10,590.5		
		4	10,522.4		
60/70+Filler	25°C	1	974,691.6	1,003,652.2	102,135.8 (10.2)
		2	907,259.2		
		3	984,756.9		
		4	1,147,901.3		
	40°C	1	37,402.1	42,235.4	3,771.7 (8.9)
		2	42,767.3		
		3	42,174.6		
		4	46,597.6		

Table 7-3: Calculated viscosity values [Pa.s] for the 60/70 bitumen and binder/filler system (DSR).

The effect of filler addition is clear in this table. The filler addition increases the viscosity of the binder/filler system compared to the binder. Taking this result into account together with the figures presented here, the effect of filler addition is clear: The binder properties are not changed while the binder/filler system is much more resistant to flow as can be seen in the higher viscosity values. However, it seems that the addition of the filler leads to a system with more varied response to loading – as can be seen in the increase for the COV for the binder/filler system compared to the binder. It is also

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interesting to note that the increase in temperature do not lead to more varied response (COV does not increase with increase in temperature).

Looking at the values of the viscosities at the end of the tests it can be seen that an average value is approached. An average value being approached suggests that the binder approaches Newtonian behaviour if loaded for a long enough time. (This could be Newtonian behaviour of the viscous component and is characterised by a constant value for the viscosity at a temperature (viscosity is a material constant for a Newtonian fluid at a temperature), irrespective of the applied load on the material – $\tau = \eta \times \dot{\gamma}$.)

In order to better understand the discussion here, a well-known model used to describe the visco-elastic behaviour of bituminous materials, Burger's model, will be presented. A schematic representation of Burger's model is given in Figure 7-4.
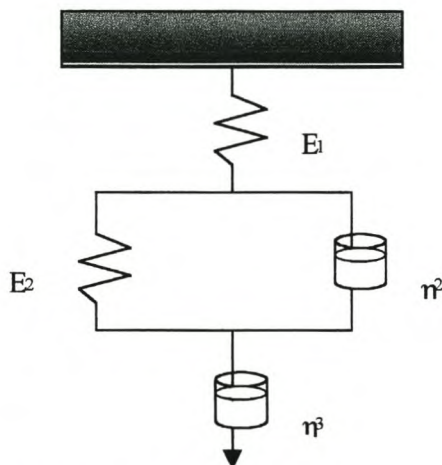
Figure 7-4: Burger's model.

Material response to loading is influenced by three components in Burger's model:
- elastic response (E_1)
- visco-elastic response (E_2 and η_2)
- viscous response (η_3)

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In Figure 7-5 the deformation behaviour of bitumen during a creep test is given. The assumption is made that the behaviour can be described by Burger's model. During period 1 the slope of the line can be assumed to be influenced by the visco-elastic and viscous parts of the load response (E_2 and η_2 in Figure 7-4). If the creep test is run long enough, it can be assumed that only the viscous component of the material response is being recorded (period 2). If the shear rate during period 2 is constant, the viscosity can be calculated . Thus, the test duration of the creep test is an important parameter.
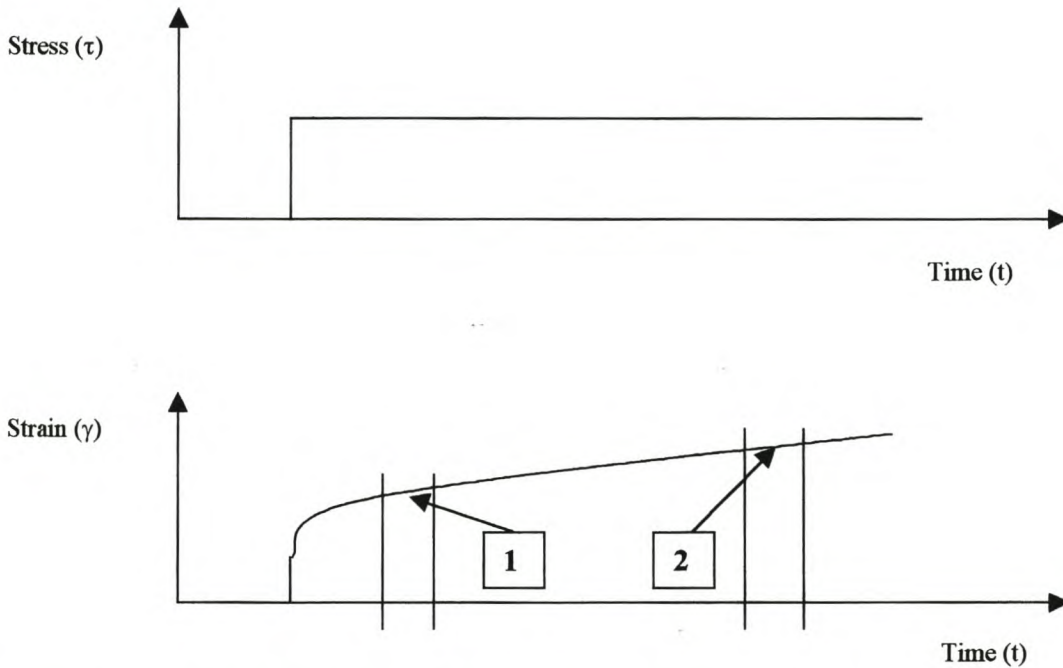
Figure 7-5: Deformation behaviour of bitumen during creep test.

When Table 7-3 is compared with the viscosity values for the 60/70 in Table 5-2, Table 5-3 and Table 5-4, it is clear that the viscosities calculated from the DSR creep test results approached a constant value at each of the temperatures. This is in contrast to the SPR test results, where a constant value was not approached. One would expect the two test methods to give the same general results if all the test conditions are assumed the same. However, there was one fundamental difference between the two test methods – the test duration.

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In the case of the DSR the test duration was ten minutes. On the other hand, the duration of the SPR tests varied between fifteen seconds to twenty minutes. The SPR test duration was influenced by how quickly the sample reached a deformation of 100% strain. When the SPR tests reached a value of 100% strain the test was stopped. The results presented here suggest that the shear rate was not constant at the end of the SPR tests (as was assumed when the results were calculated). The reason for this must be that the load levels used were too high. Thus, if lower load levels were to be used and longer test duration allowed, it can be assumed that the SPR results will also converge on an average value for the calculated viscosity at the end of the test.

7.2.2 SBS Modified Binder

In this section the results of the creep tests on the SBS modified binders are presented together with the results of the SPR tests on the same binder and binder/filler systems. The data from which these results were calculated can be found in Appendix A on the CD.

Combined Creep Results SBS - SPR and DSR
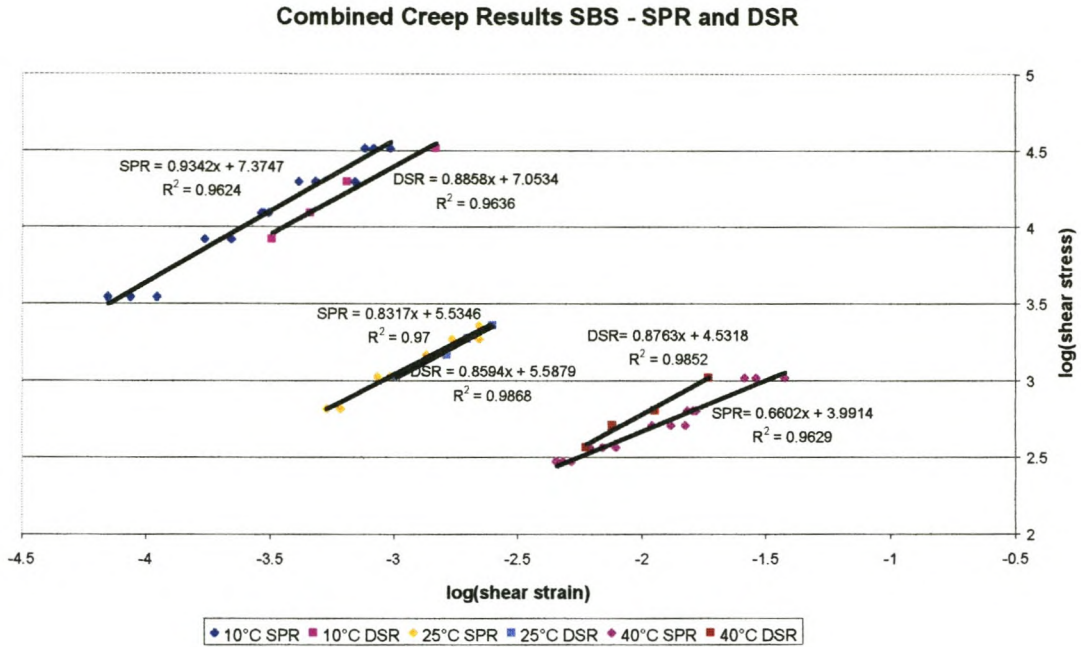
Figure 7-6: Shear susceptibility lines derived from creep test results with SPR and DSR for SBS.

Combined Creep Results SBS+Filler - SPR and DSR

Figure 7-7: Shear susceptibility lines derived from creep test results with SPR and DSR for SBS+Filler.

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Referring to the figures presented in this section, the following comments can be made:

- At 10 °C and 25 °C the SPR results for the SBS binder were stiffer than that of the DSR, although the difference is not as pronounced as for the 60/70 binder.
- In all the figures, the slopes of the DSR shear susceptibility lines are steeper than that of the SPR shear susceptibility lines.
- For the binder/filler systems the DSR results are stiffer than for the SPR results.

Table 7-4 gives the calculated viscosity values at the end of the DSR creep tests – a constant shear rate is assumed at the end of the test.

Binder	Temperature	Test Number	Calculated Viscosity	Average	Standard Deviation (COV) %
SBS	10°C	1	26,067,067.3	26,481,893.2	3,526,564.8 (13.3)
		2	26,998,108.6		
		3	30,724,581.6		
		4	22,137,815.1		
	25°C	1	1,041,887.7	950,341.7	63,881.8 (6.7)
		2	901,374.0		
		3	945,751.0		
		4	912,354.0		
	40°C	1	61,731.0	60,365.3	4,982.7 (8.2)
		2	66,830.5		
		3	56,731.3		
		4	56,168.5		
SBS+Filler	25°C	1	5,220,706.6	5,751,033.5	438,005.2 (7.6)
		2	5,679,054.1		
		3	6,284,276.7		
		4	5,820,096.5		
	40°C	1	350,114.4	320,741.6	63,143.0 (19.7)
		2	228,299.6		
		3	335,483.9		
		4	369,068.5		

Table 7-4: Calculated viscosity values [Pa.s] for the SBS binder and binder/filler system.

In this table the effect of the filler addition is clear – the addition of filler increases the viscosity values. Again, when the figures and table are considered, it can be noted that the

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addition of filler does not alter the binder properties. The binder/filler system is much more resistant to viscous flow compared to the binder. The variability in the results as measured with the COV is again higher for the binder/filler system – in fact the binder/filler at 40 °C has very high variability (almost 20%). The temperature did not influence the variability of the test results.

The same comments that were made about the difference in test duration between the two test methods in the previous section are applicable here.

7.2.3 Bitumen-Rubber

In this section, the results of the creep tests on the bitumen-rubber will be presented and discussed. The data from which these results were calculated can be found in Appendix A on the CD.

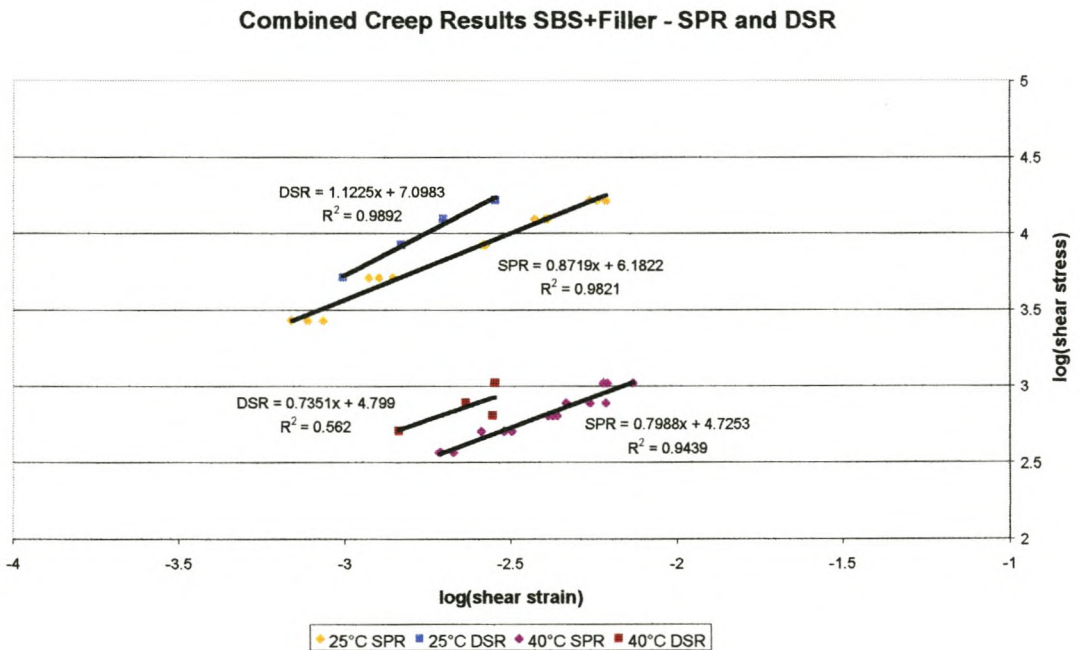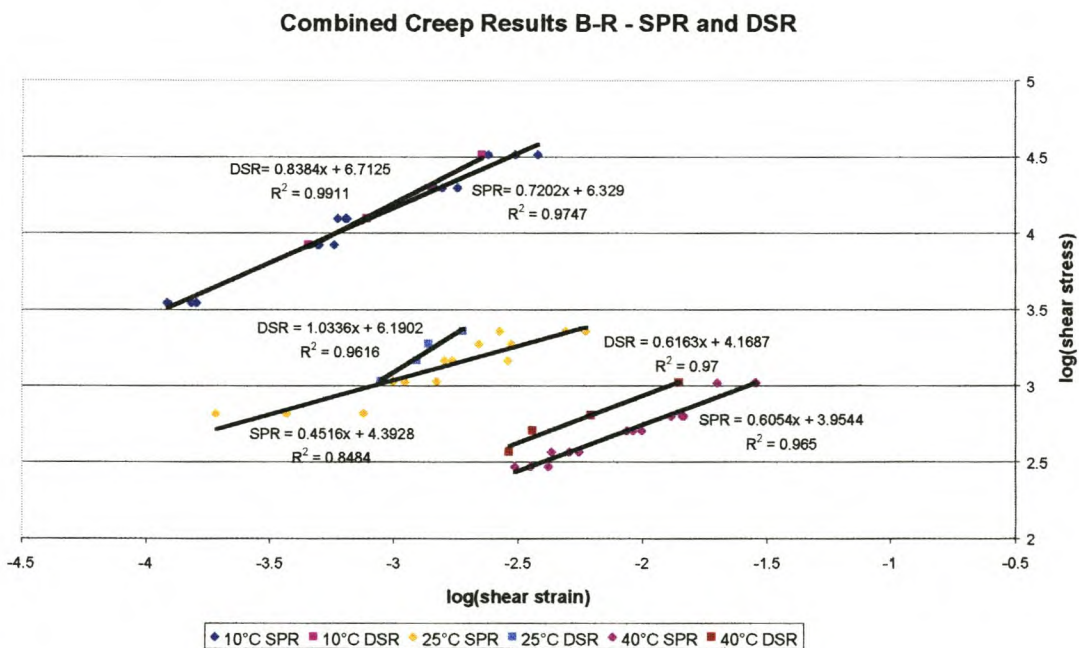Figure 7-8: Shear susceptibility lines derived from creep test results with SPR and DSR for Bitumen-Rubber.

Combined Creep Results B-R+Filler - SPR and DSR
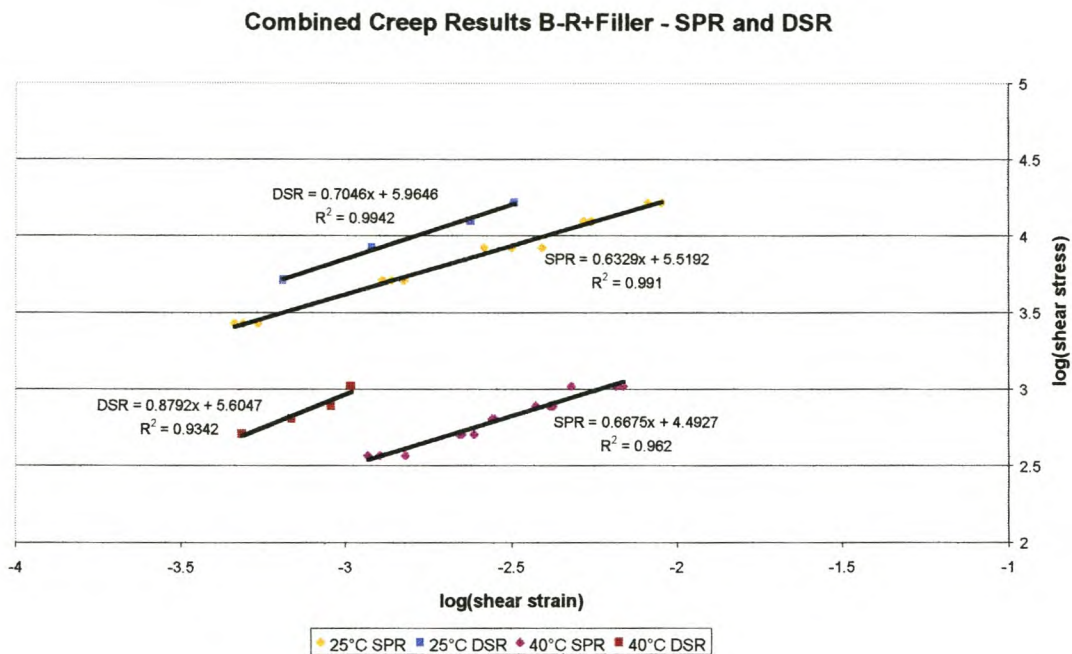
Figure 7-9: Shear susceptibility lines derived from creep test results with SPR and DSR for Bitumen-Rubber+Filler.

With reference to the figures presented in this section, the following conclusions can be reached:

- For the binder at 10 °C and 25 °C the DSR results are in the same region as the results from the SPR tests.

- As with the other binders, the slopes of the shear susceptibility lines derived from the DSR tests are steeper than the slopes of the SPR derived lines.

- For the filler systems, the DSR lines indicate a stiffer response to loading compared to the SPR lines.

Table 7-5 presents the calculated viscosity values for the Bitumen-Rubber binder and binder/filler system. The assumption was made that the shear rate was constant at the end of the creep tests for the calculation of the viscosity values.

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Binder	Temperature	Test Number	Calculated Viscosity	Average	Standard Deviation (COV) %
Bitumen-Rubber	10°C	1	18,531,073.4	15,759,325.2	2,040,015.1 (12.9)
		2	15,993,597.4		
		3	13,917,616.1		
		4	14,595,013.9		
	25°C	1	1,201,349.8	1,241,904.1	83,817.9 (6.7)
		2	1,190,922.8		
		3	1,367,180.4		
		4	1,208,163.3		
	40°C	1	127,696.3	111,680.9	29,090.4 (26.0)
		2	141,030.1		
		3	102,997.4		
		4	75,000.0		
Bitumen-Rubber+Filler	25°C	1	7,946,866.1	6,322,834.4	1,375,482.7 (21.7)
		2	6,979,930.8		
		3	5,231,413.6		
		4	5,133,126.9		
	40°C	1	1,050,103.0	962,376.1	84,181.1 (8.8)
		2	931,021.9		
		3	860,057.0		
		4	1,008,322.7		

Table 7-5: Calculated viscosity values [Pa.s] for the Bitumen-Rubber binder and binder/filler system.

This table shows that the filler addition increases the viscosity of the binder/filler system compared to the binder. The COV for the different test temperatures varies considerably (between 6.7 % and 26 %). This variability might be a characteristic of the material. In this case the addition of filler to the binder did not seem to influence the variability in the test results as measured with the COV.

In this case the comments that were made about the duration of the DSR creep tests applies.

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7.2.4 Discussion of results for different binders

The creep tests performed with the DSR gave results that are comparable to the results obtained with the SPR. These results are summarised in Table 7-6. The table shows that in general the two rheometers gave comparable results. The slopes of the individual lines are not much different, while the correlation coefficient (r^2 value) is greater than 0,9 for all the lines. The only exceptions are the Bitumen-Rubber at 25 °C where the correlation coefficient is 0,8484 (SPR) and the SBS + Filler where the correlation coefficient is 0,562 (DSR). The r^2 value referred to gives an indication of how well a straight-line fit fits the data. It would not make any sense to compare the variance in the test results between the two machines. This is because the SPR line were fitted on data with three results at a load level, while the DSR lines were fitted on data with only one test result at a load level. The SPR results also did not approach an average value for the calculated viscosity.

The slope given in the tables is an indication of the shear susceptibility of the different binders and binder/filler systems at the different temperatures. The shear susceptibility lines give an indication of the viscosity of different binders. As was seen in the tables of calculated viscosity values that were presented in the previous sections, the value of the viscosity tends toward an average value at a temperature for a binder. This is true if the creep test duration is long enough.

The viscosity value is calculated by dividing the applied stress with the shear rate at the end of the test (constant shear rate assumed). Thus, if different binders are studied and the shear susceptibility lines are horizontally shifted for the same load regime at the same temperature, conclusions can be drawn about the viscosity values of the different binders relative to each other. This was clear from each of the figures presented in previous sections. Table 7-7 shows that the modified binders has much higher viscosity values and from the figures we know that the shear susceptibility lines of the modified binders are shifted horizontally towards lower shear rates – this indicates much higher viscosity values for the modified binders.

Material			Temperature		
			10 °C	25 °C	40 °C
60/70	SPR	slope	0.9471	0.9556	0.8276
		r2	0.9807	0.9109	0.9695
	DSR	slope	0.9861	1.1455	1.0224
		r2	0.9888	0.9992	0.985
SBS	SPR	slope	0.9342	0.8317	0.6602
		r2	0.9624	0.97	0.9629
	DSR	slope	0.8858	0.8594	0.8763
		r2	0.9636	0.9868	0.9852
Bitumen-Rubber	SPR	slope	0.7202	0.4516	0.6054
		r2	0.9747	0.8484	0.965
	DSR	slope	0.8384	1.0036	0.6163
		r2	0.9911	0.9616	0.97
60/70+Filler	SPR	slope	=	0.8613	0.8682
		r2	=	0.9875	0.9444
	DSR	slope	=	1.1172	1.2297
		r2	=	0.9728	0.9921
SBS+Filler	SPR	slope	=	0.8719	0.7988
		r2	=	0.9821	0.9439
	DSR	slope	=	1.1225	0.7351
		r2	=	0.9892	0.562
Bitumen-Rubber+Filler	SPR	slope	=	0.6329	0.6675
		r2	=	0.991	0.962
	DSR	slope	=	0.7046	0.8792
		r2	=	0.9942	0.9343

Table 7-6: Summary of creep test results for DSR and SPR on binders.

Material	Temperature	Average Viscosity	Standard Deviation
60/70	10°C	9,077,099.7	570,066.4
	25°C	192,356.6	8,283.3
	40°C	10,248.6	544.5
SBS	10°C	26,481,893.2	3,526,564.8
	25°C	950,341.7	63,881.8
	40°C	60,365.3	4,982.7
Bitumen-Rubber	10°C	15,759,325.2	2,040,015.1
	25°C	1,241,904.1	83,817.9
	40°C	111,680.9	29,090.4
60/70+Filler	25°C	1,003,652.2	102,135.8
	40°C	42,235.4	3,771.7
SBS+Filler	25°C	5,751,033.5	438,005.2
	40°C	320,741.6	63,143.0
Bitumen-Rubber+Filler	25°C	6,322,834.4	1,375,482.7
	40°C	962,376.1	84,181.1

Table 7-7: Average viscosity values [Pa.s] calculated for the different binders and binder/filler systems.

Figure 7-10 and Figure 7-11 show the results of the SPR and DSR creep tests graphically. From these figures it is clear that the DSR results have the same general form as the SPR results, i.e. the qualification of the three binders as obtained from the SPR results are the same for the DSR results. Both rheometers qualified the binders in the same way – the SPR results and the DSR results suggest that the modified binders must have much higher viscosity values compared to the unmodified binder. Thus, it can be concluded that the modified binders must have better permanent deformation performance compared to the 60/70 since they are much more resistant to flow.

However, it is also clear that the DSR gave consistently "stiffer" results compared to the SPR results at 25 °C and 40 °C (compare the different lines on the figures). A "stiffer" response means that the DSR line is shifted horizontally to the left of the SPR line. This effect is most noticeable for the binder/filler lines (Figure 7-11). Referring to the binder/filler figures in the sections for the individual binders (Figure 7-3, Figure 7-7 and Figure 7-9), it is clear that this shift is more pronounced for the modified binders. The reason for this result has been described in detail in the section on the 60/70 results. Basically, this is a result of different test durations being used for the two rheometers – there is some doubt whether the SPR results can be assumed to have reached constant shear rates at the end of the tests.
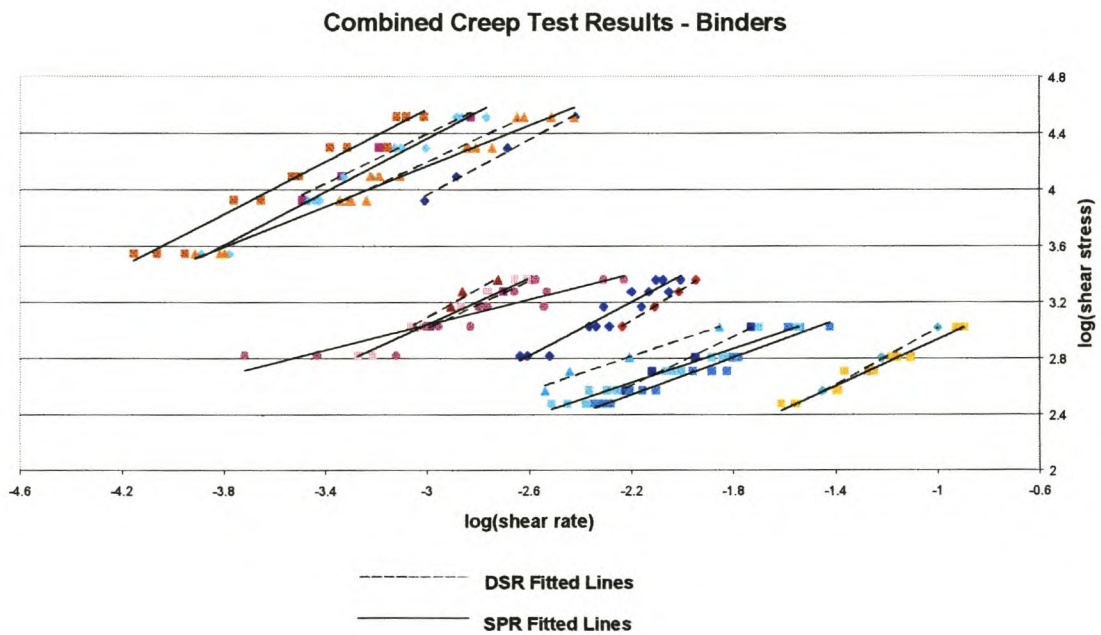
Figure 7-10: Combined creep test results obtained with the DSR and SPR for the binders.

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Combined Creep Test Results - Binder/Filler Systems
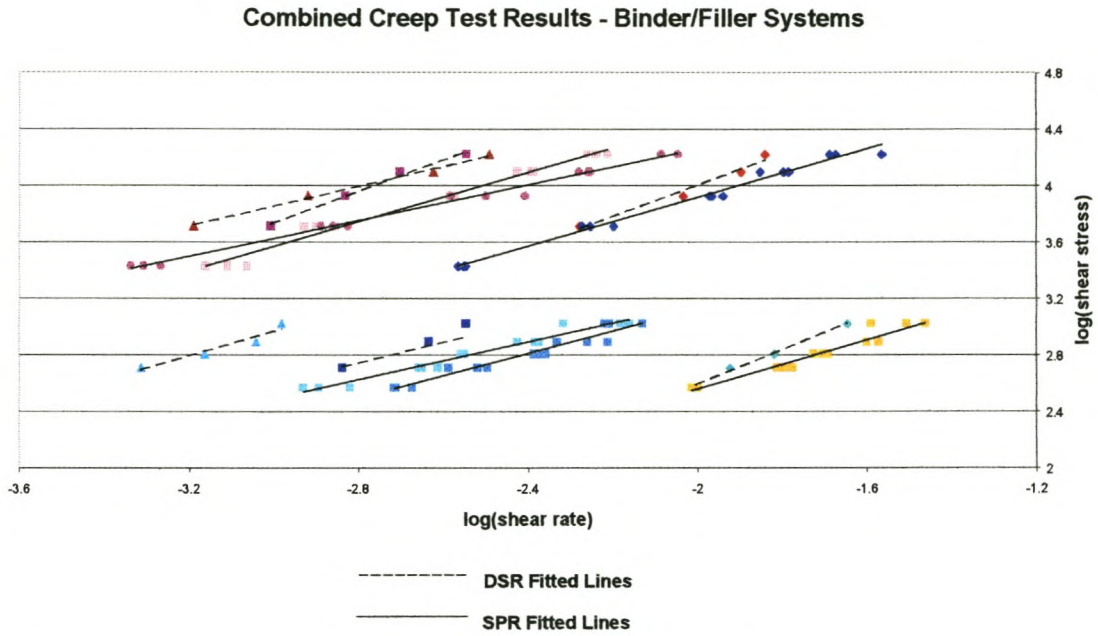
Figure 7-11: Combined creep test results obtained with the DSR and SPR for the binder/filler systems.

Deformation vs. Time - Creep Tests DSR on Bitumen-Rubber+Filler
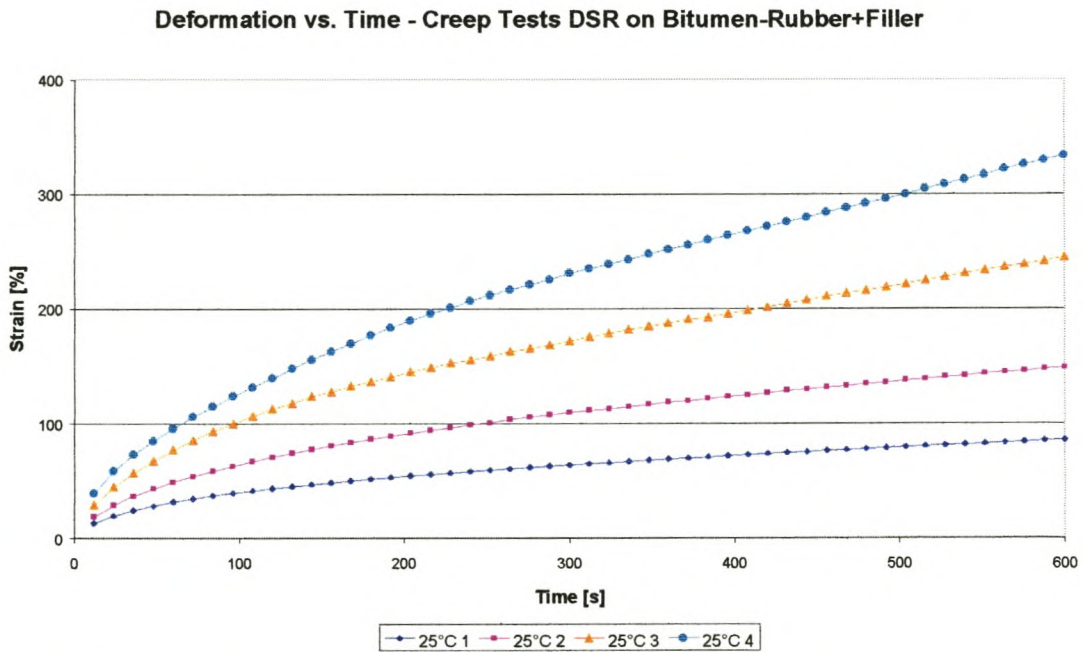
Figure 7-12: Strain vs. time plot for Bitumen-Rubber+Filler at 25 °C - DSR.

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Deformation vs. Time - Creep Tests DSR on SBS+Filler
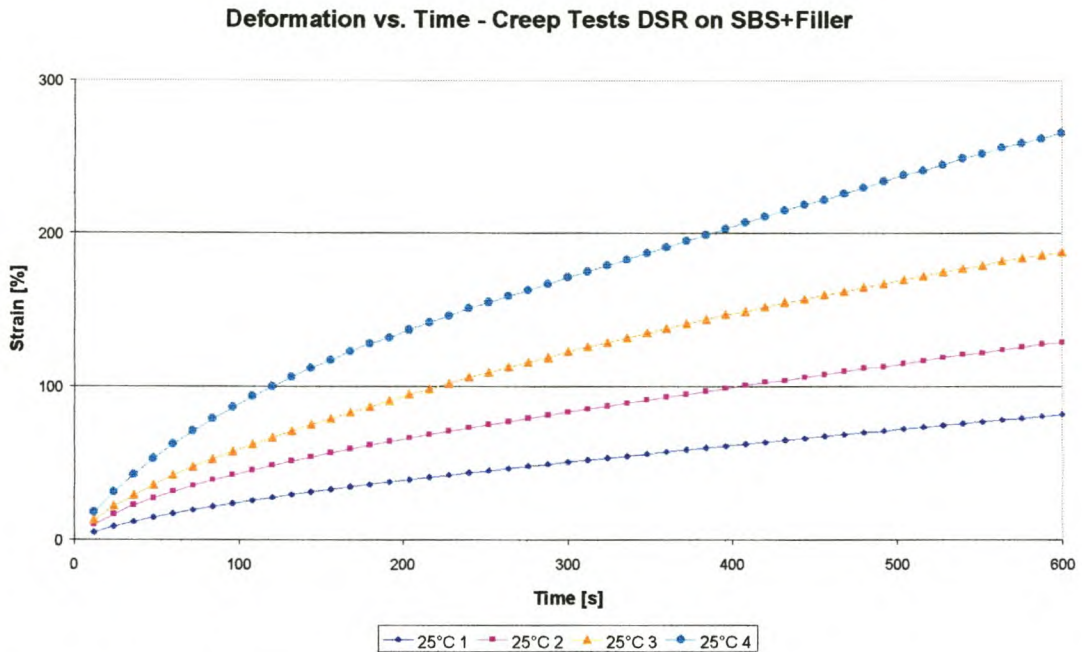
Figure 7-13: Strain vs. time plot for SBS+Filler at 25 °C - DSR.

The test results of the creep tests are reported as plots of shear stress vs. shear rate plots. The aim is to obtain a constant shear rate at the end of the test. When the two strain vs. time figures are studied it is clear that the shear rate at the end of the tests was constant (the slope of the curve is constant).

The user manual for the SPR suggests that the creep test be run until a total shear strain of 100% is reached and to stop the test after this point has been passed (this is due to the end effects that come into play when the sample is displaced further). However, if the test results of the DSR tests are studied (Figure 7-12 and Figure 7-13), it is clear that a constant shear rate was only reached after the shear strain had passed the value of 100%. Closer inspection of these graphs shows that the shear rate at 100% strain is not constant for most of the cases. This result is most clearly demonstrated in Table 7-7 where it can be seen that the DSR results tended toward a constant value for the viscosity calculated at the end of each test at a temperature.

7.3 Conclusions

In essence, the results obtained with the DSR were the same compared to the SPR results. The main differences between the two machines lay in the slopes of the shear susceptibility lines and the calculated viscosities at the end of the creep tests.

It would seem that the methods followed for the determination of the resulting shear rate, led to different results when loaded similarly. For the SPR the aim was to reach a total deformation of 100% strain and to stop the test when this value was reached. [10] From the results of the DSR tests, it was shown that one could conclude that the shear rate was probably not constant at this point (100% strain) and only reached a constant value much later. This is a result of the high load levels being used. If lower load levels were used and the test duration was increased, the SPR will most probably give results comparable with the DSR results.

Even with the differences between the two methods it was shown that the SPR and DSR creep tests gave similar results. The overall qualification of the three binders and three binder/filler systems, as determined with the SPR, was repeated with the DSR. It would seem that the viscosity values calculated from the SPR results are not reliable, but the qualification of the binders is reliable.

8 High Temperature Testing of Binders and Binder/Filler Systems

In this chapter, the results of the high temperature viscosity tests are discussed. The chapter is divided into two parts. The first part deals with the high temperature tests that were performed on the binder systems. In the second part the high temperature tests that were performed on the binder/filler systems are discussed.

The purpose of these tests was to determine the high temperature/high shear rate behaviour of the binders and binder/filler systems used in this study. The binders were studied to further investigate the results of work by another researcher [8], while the binder/filler systems were studied to compare it with the binders.

Two binders were tested during this part of the research. These were the 60/70 penetration grade bitumen and SBS modified 60/70 binder. The Bitumen-Rubber binder was not tested in this part of the research and the reasons that led to the decision are discussed.

High temperature viscosity tests were also performed on the 60/70 + Filler and SBS + Filler systems. As with the pure binders, the Bitumen-Rubber + Filler system was not tested in this part of the research.

In each part, the purpose of the tests and methodology used will be discussed. The results and discussion of the results will be presented after this. At the end of the chapter conclusions will be drawn with regard to the test results.

The test programme that was followed is summarised in Table 8-1. 18 tests were performed on each binder and 9 tests on each binder/filler system. Three test temperatures were used: 105 °C, 135 °C and 165 °C.

Shear Rate	Binders		Binder/Filler Systems	
	60/70	SBS	60/70 + Filler	SBS + Filler
$100s^{-1}$	1	1	1	1
$200s^{-1}$	1	1		
$300s^{-1}$	1	1	1	1
$400s^{-1}$	1	1		
$500s^{-1}$	1	1	1	1
$600s^{-1}$	1	1		
Testing at three temperatures: 105°C, 135°C and 165°C.				

Table 8-1: Table of test programme followed for high temperature tests at the three temperatures.

8.1 High Temperature Viscosity Tests on Binders

In this part of the chapter the high temperature viscosity tests done on the binders will be covered. The purpose and methodology of the tests will be described first and thereafter the results will be presented and discussed. The equipment used and sample preparation are discussed in chapter 4.

8.1.1 Purpose

An attempt was made to describe the behaviour of the binders when subjected to high shear rates at high temperatures (T > 100 °C). This was done as an attempt to define and understand the behaviour of polymer modified binders under these conditions. In general, it is assumed that polymer modified binders behave different compared to normal bitumen in the high temperature domain (during mixing and compaction). The aim was to investigate this.

The procedures (ASTM D2493, Bitumen Test Data Chart (BTDC) developed by SHELL) used today to determine mixing and compaction temperatures have been developed for unmodified binders. If the same procedure is used for modified binders, unreasonably

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high temperatures result for the mixing and compaction temperatures. [8] The ASTM procedure assumes Newtonian behaviour at high temperatures for the binders.

Viscosity vs. Temperature
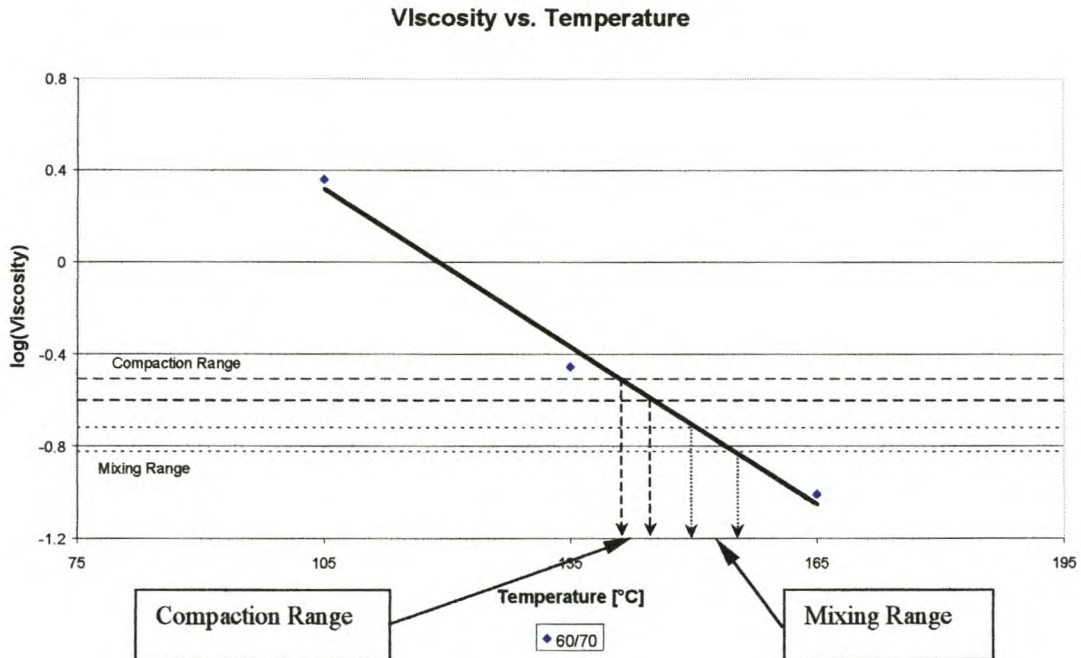
Figure 8-1: Example of Viscosity vs. Temperature curve for use with ASTM D2493 procedure.

Many problems have been encountered with the determination of mixing and compaction temperature ranges for polymer modified binders, especially when the ASTM procedure is used. These problems are due mainly to the fact that the ASTM procedure prescribes that the viscosity of the binders be determined at a shear rate of $6,8s^{-1}$. As mentioned earlier, the ASTM procedure assumes Newtonian behaviour at high temperatures $(T > 100 \ °C)$. It is a well documented fact that normal bitumen exhibit Newtonian behaviour at high temperatures. Polymer modified binders, on the other hand, might not exhibit Newtonian behaviour at these temperatures because the polymer structure might not yet be broken down totally in the binder. Thus, the problem increases in complexity when working with polymer modified binders.

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A recently published paper approached the problem of mixing and compaction temperatures for polymer modified binders in a new way. [8] First, the equiviscous temperatures at which two of the same asphalt mix samples, containing unmodified and modified binders respectively, were compacted to the same density level were determined. (The equiviscous temperatures are defined as the temperatures at which it can be assumed two binders will have the same viscosity in order to obtain the same density level after the same effort of compaction.) This was done by constructing density vs. compaction temperature curves from data obtained with the Superpave Gyratory Compactor. From these curves the equiviscous temperatures were determined. They postulate that if all other factors affecting density (including the compaction effort) are kept equal, only the viscosity of the binder will influence the density reached during compaction. Thus, for two different temperatures with the same density after compaction, the viscosity had to be equal during compaction.
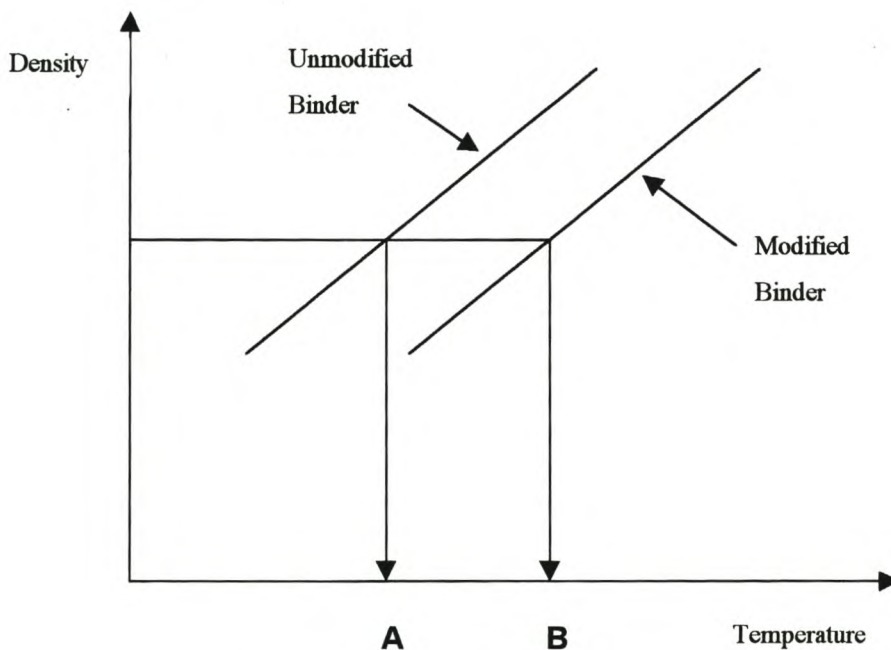
Figure 8-2: Determination of equiviscous temperatures. [8]

Using the equiviscous temperatures obtained from the compaction tests, viscosity curves were drawn for the unmodified and modified binders at these equiviscous temperatures. The unmodified binders exhibited Newtonian behaviour while the modified binders exhibited shear thinning behaviour over the range of shear rates used ($0,1s^{-1} \leq \dot{\gamma} \leq 93s^{-1}$). The two viscosity curves were extrapolated and the shear rate at the intersection of the two curves was taken as the shear rate during compaction. The viscosities of the two binders are equal at the intersection point.
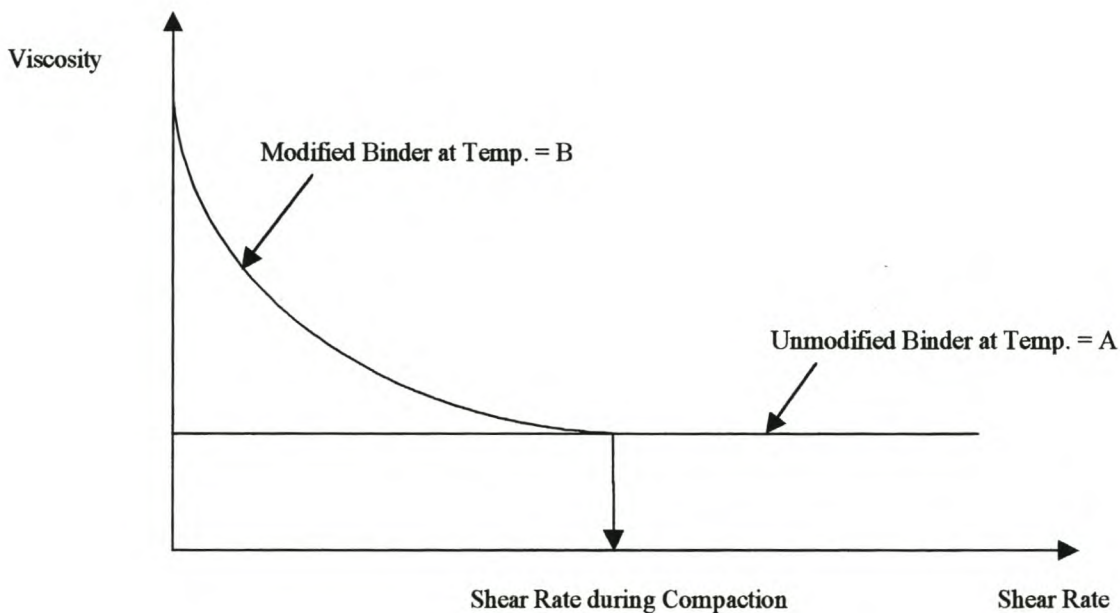
Figure 8-3: Determination of shear rate during compaction. [8]

The shear rate calculated from the intersection point of the two viscosity curves drawn at the equiviscous temperatures were then used to determine the viscosity of the polymer modified binders at the high temperatures (use was made of the regression results obtained from the extrapolation of the viscosity curves). Using viscosity curves drawn at 165 °C and 135 °C and reading off the corresponding viscosity at the determined shear rate determined the viscosity at the high temperature. Using these two points, lines were drawn on the normal viscosity-temperature graphs and mixing and compaction

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temperature ranges were determined from this. By using their procedure, they were able to reduce the temperature windows for mixing and compaction by 10 °C to 30 °C for the modified binders when compared to the temperature windows determined by the ASTM D 2493 procedure.

It was the aim of this research to characterise binder behaviour at high shear rates and high temperature. The results can then be used to determine mixing and compaction temperature windows for the binders.

8.1.2 Methodology

Standard viscosity tests were performed in shear rate controlled mode at three temperatures. The temperatures used were 105 °C, 135 °C and 165 °C. At each of the three temperatures the binders were tested at six shear rates: 100, 200, 300, 400, 500 and 600 s^{-1}.

It should be noted that the test results presented here did not take into account the shear rate region researched previously ($\dot{\gamma} \leq 100 s^{-1}$). The equipment used by those researchers could shear the binders to a maximum rate of 93s^{-1}. [8]

The Bitumen-Rubber was not tested in this part of the research. This was due to a perceived problem that existed due to the inhomogeneous nature of the binder. The decision to not test the Bitumen-Rubber was taken on the grounds of a visual evaluation of the test method that, in retrospect, was erroneous. The evaluation of the test procedure for the Bitumen-Rubber will be described further in a section dealing specifically with it.

8.1.3   Results and Discussion

In this section, the results of the high temperature viscosity tests on the binders will be presented.
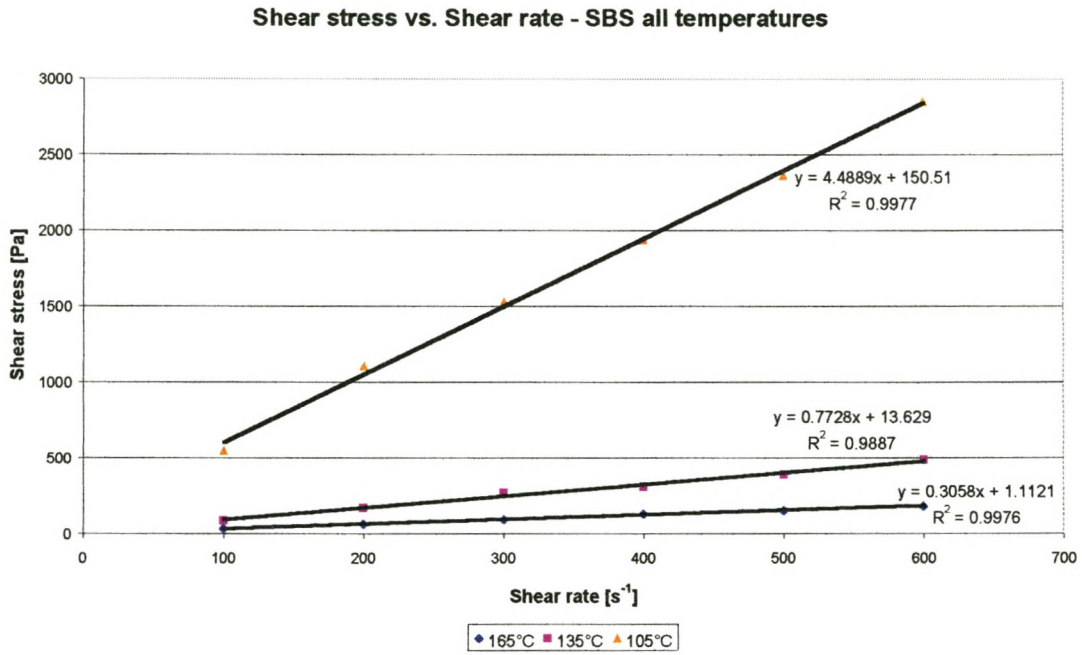
Figure 8-4: Shear stress vs. Shear rate lines for SBS.
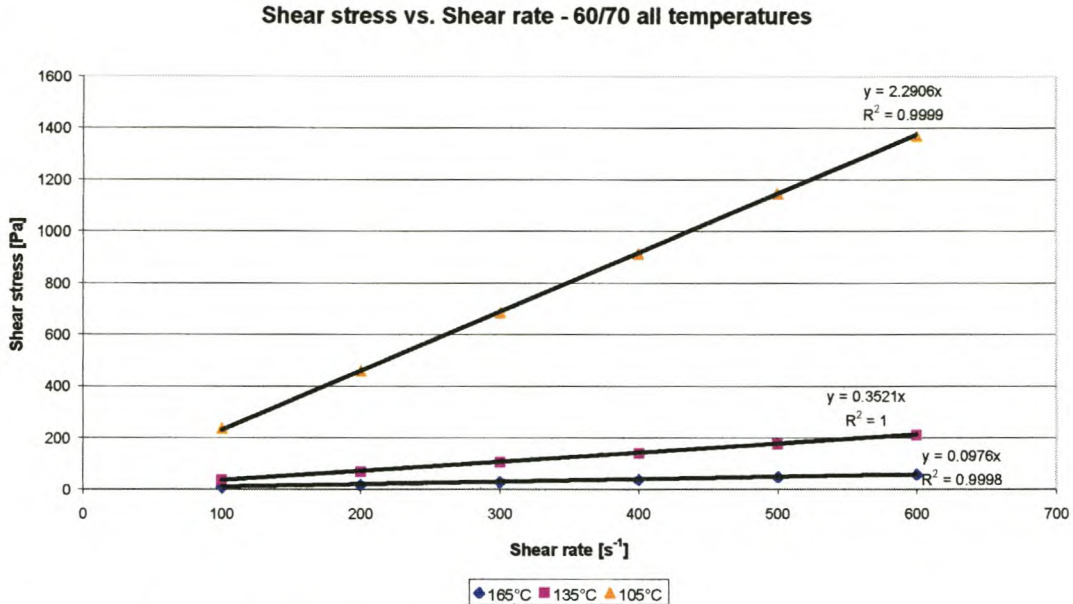
Figure 8-5: Shear stress vs. Shear rate lines for 60/70.

From the results of the viscosity tests shear stress was plotted against shear rate. The curve obtained from the plotted points gives an indication of the rheological behaviour of the material. Regression lines were fitted on the data as can be seen in Figure 8-4 and Figure 8-5. The following comments can be made based on these figures:

- Figure 8-4:
  - The shear stress vs. shear rate line is a straight line.
  - There is a y-intercept in the regression result – this is most probably a result of shear thinning behaviour in the shear rate region not covered.
  - At 165 °C the y-intercept value is not significant – this could be because the polymer structure is broken down due to temperature effects at this temperature. It can be assumed that the SBS binder is a Newtonian fluid at this temperature.
  - As temperature decreases, the y-intercept of the regression lines increase. This suggests that the polymer structure is starting to form and that it plays an increasing role in the binder behaviour.
- Figure 8-5:

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- o A straight line for shear stress vs. shear rate, which passes through the origin, is a characteristic of Newtonian behaviour.
- o The regression lines for the 60/70 binder suggest Newtonian behaviour.
- o The decrease in the slope as temperature increases is an indication of the decrease in the viscosities with increasing temperature.

The figures discussed above were obtained from viscosity tests at the shear rates and temperatures shown. The viscosity/shear stress value used at a strain rate was the value obtained at the end of a test. It was found that the viscosity decreased with time from the start of a test, until a constant value was reached (similar procedure to the creep curves from the SPR). This constant value was then used as viscosity/shear stress value. Fifty points were measured during a test of five minutes and the average of the last twenty points was used. Figure 8-6 shows a typical plot of viscosity vs. time obtained from these tests – note the curves tend toward horizontal lines at the end of the test.
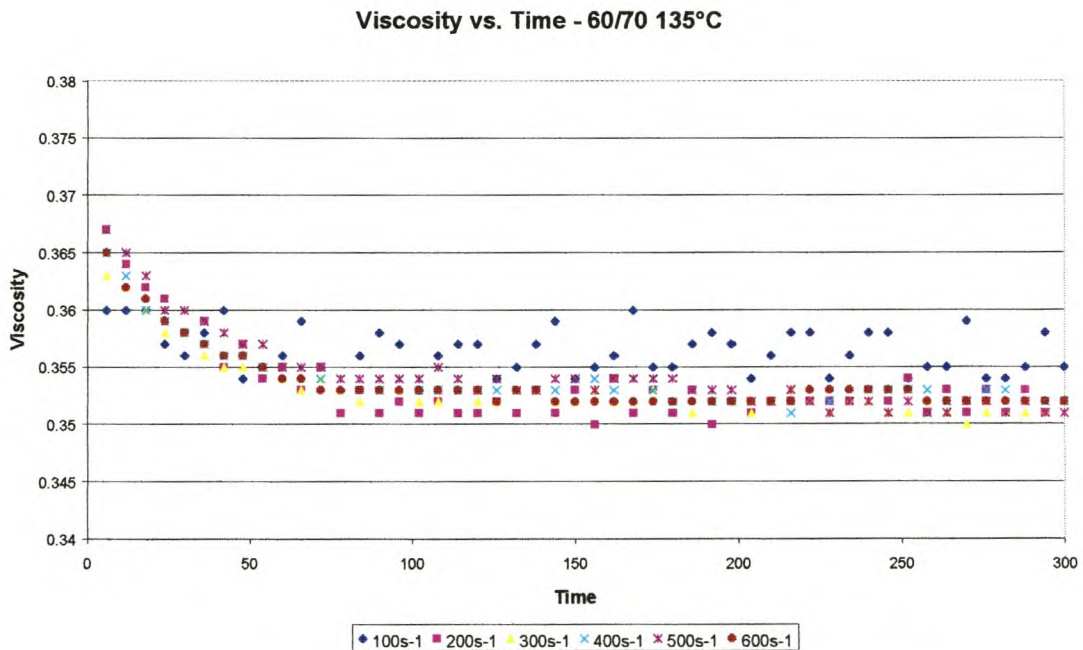
Figure 8-6: Typical plot of viscosity vs. time - 60/70 at 135 °C.

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The results of the regression analyses were used to calculate apparent viscosities for the two binders. The apparent viscosity lines were plotted at the three temperatures at which the tests were conducted. The resulting graphs are shown in Figure 8-7, Figure 8-8 and Figure 8-9. From these figures one can form an impression of the behaviour of the two binders at high temperatures.

It can be seen that the 60/70 binder behaves as a Newtonian fluid at the three temperatures. It is generally accepted that normal, unmodified binders exhibit Newtonian behaviour at temperatures where it is sufficiently fluid to flow.

On the other hand, the behaviour of the SBS binder cannot be described so easily. At 165 °C the polymer structure of the SBS is sufficiently broken down for the binder to approach Newtonian behaviour. As temperature decreases the SBS binder's behaviour shows an increase in the apparent viscosity as shear rate decreases (more noticeable at 105 °C).
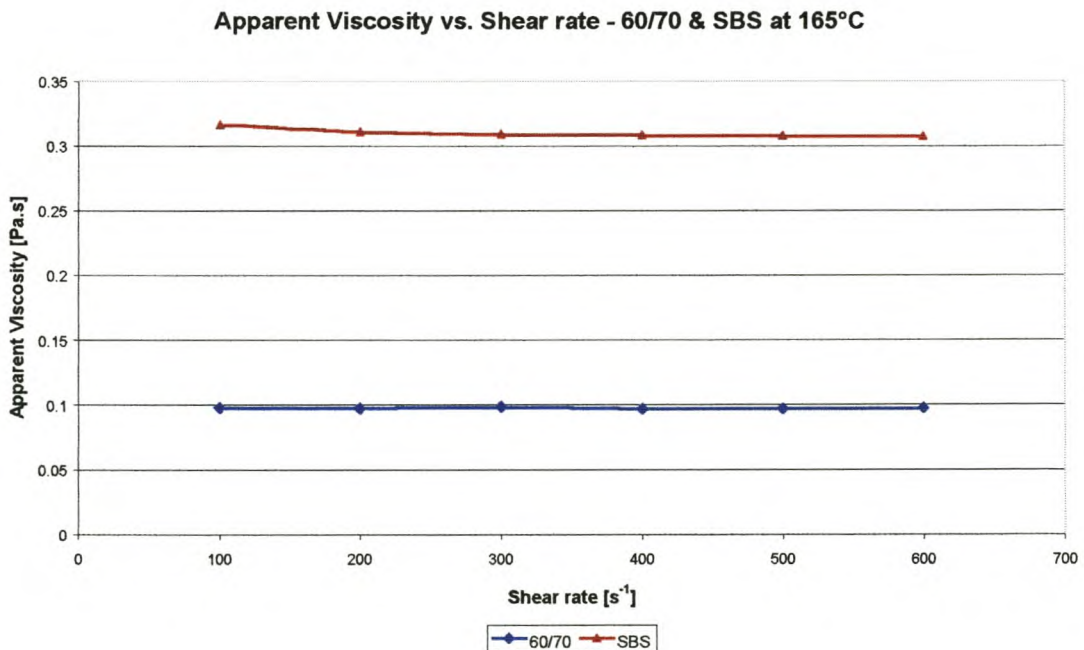
Figure 8-7: Apparent viscosity curves for 60/70 and SBS binders at 165 °C.

Apparent Viscosity vs. Shear rate - 60/70 & SBS at 135°C
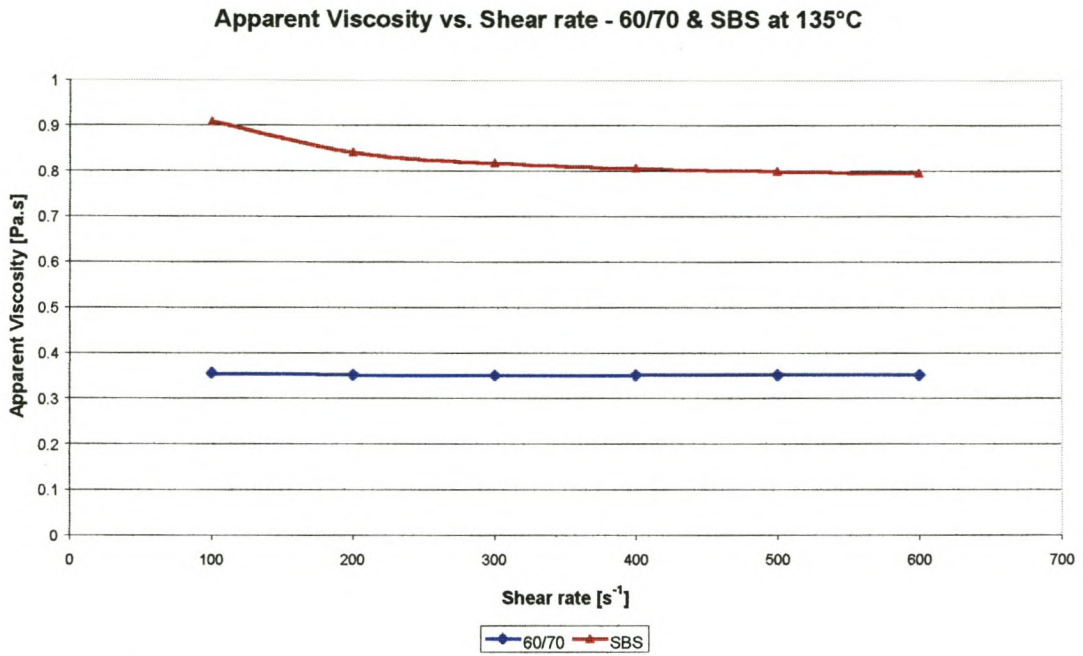
Figure 8-8: Apparent viscosity curves for 60/70 and SBS binders at 135 °C.

Apparent Viscosity vs. Shear rate - 60/70 & SBS at 105°C
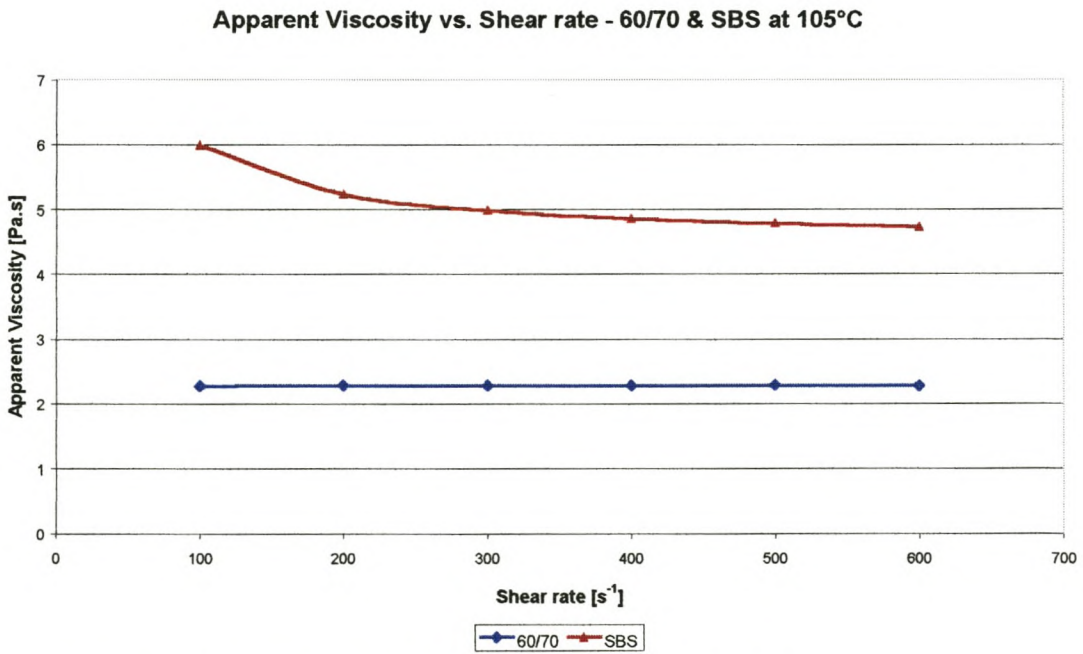
Figure 8-9: Apparent viscosity curves for 60/70 and SBS binders at 105 °C.

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As was mentioned earlier, the SBS could be exhibiting shear thinning behaviour. It should be stressed that this research did not cover the region of shear rates below $100s^{-1}$. Therefore, it is difficult to say with certainty whether it is shear thinning behaviour or not. However, based on recent research that covered the region below $100s^{-1}$, one can say that it seems most reasonable that the SBS binder is exhibiting shear thinning behaviour. [8]

Another important characteristic of the SBS binder that arises out of the figures is the increase in apparent viscosity as shear rate decreases. This is important when considering the establishment of temperature windows for mixing and compaction of polymer modified binders. Some recommendations about the shear rate that should be used when determining viscosities for polymer modified binders were made by other researchers, based on their research results. [8] The shear rate that was recommended by them was determined by extrapolation of viscosity curves for an unmodified and modified binder in the same mix that was compacted to the same density level. Based on the average values for the binders and mixes used by them, they recommended a value of $490s^{-1}$ for the shear rate during compaction.

As is clear from the figures presented here, the accurate prediction/determination of the shear rate is very important. For high shear rates ($\dot{\gamma} > 300s^{-1}$), the apparent viscosity is approximately constant. However, if shear rates are encountered below the constant apparent viscosity region, the viscosity of the binder at a temperature cannot be predicted accurately because of the rapid increase in apparent viscosity with decreasing shear rate.

Thus, if the shear rates encountered during mixing and compaction are indeed as high as suggested, figures such as presented here can be used to determine new temperature windows for mixing and compaction. If, on the other hand, the shear rates are much lower, it is clear that temperatures much higher than used for normal bitumen are necessary for proper manufacture and placing of mixes containing polymer modified binders.

8.2 High Temperature Viscosity Tests on Binder/Filler Systems

In this part the tests on the binder/filler systems will be described and the results will be presented and discussed.

8.2.1 Purpose

These tests were performed in order to characterise the influence the filler has on the binder. The object was to perform the same tests that were performed on the binders. This would then give an indication of the influence of the filler on the system.

8.2.2 Methodology

In essence, the same methodology that was used for the high temperature viscosity tests on the binders was used for the high temperature viscosity tests on the binder/filler systems. The tests were performed at three temperatures (105 °C, 135 °C and 165 °C) and three shear rates were used ($100s^{-1}$, $300s^{-1}$ and $500s^{-1}$).

For the binder/filler systems, a gap setting of 0,2 mm was used instead of the 0,1 mm used for the binders. This was done to allow space for the filler particles – the maximum size of which was 0,1 mm. The gap setting could not be made any bigger due to the high temperatures at which the tests were performed – at the high temperatures the material flowed out from under the spindle of the DSR if the gap setting was too large.

The gap setting was chosen arbitrarily and a sensitivity analysis for different gap settings was not performed.

As with the binder systems, the Bitumen-Rubber + Filler system was not tested. The reasons for this decision are described later in this chapter.

8.2.3 Results and Discussion

The viscosity tests for the binder/filler systems were conducted in the same way as those for the binders. The machine was programmed to rotate the spindle at a constant shear rate and the viscosity of the material was measured. Unlike the binders, the binder/filler systems did not reach a constant value for the viscosity at the end of the test – even after the test duration was doubled to ten minutes. These results are presented in Figure 8-10 to Figure 8-15.

Flow Curve - 60/70+Filler 165°C
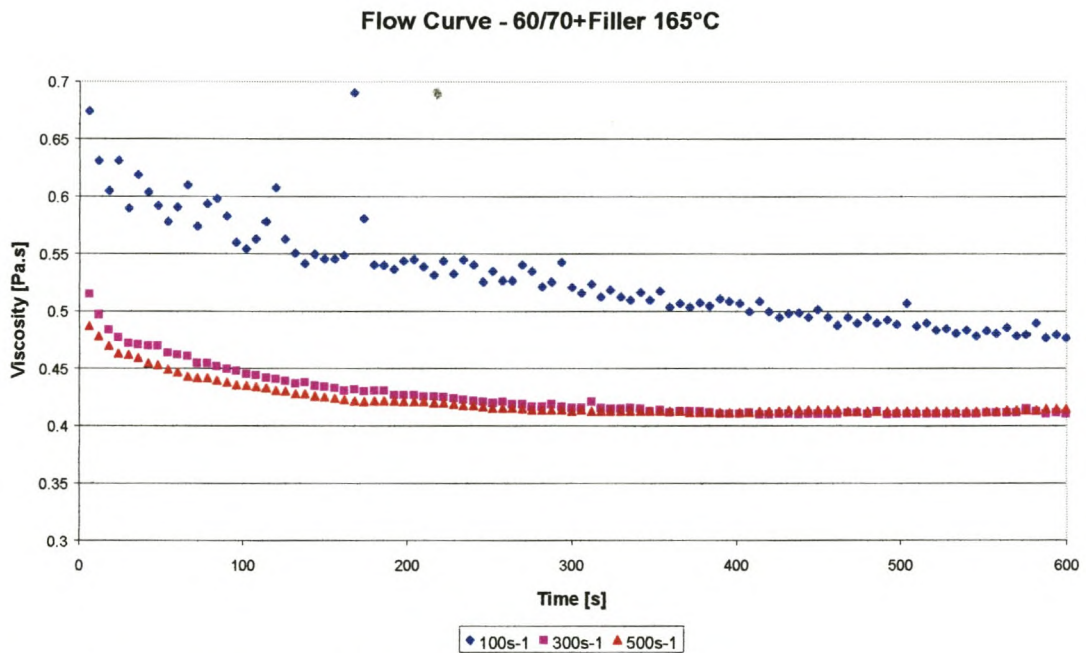
Figure 8-10: Viscosity vs. time curve for 60/70+Filler at 165 °C.

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Flow Curve - 60/70+Filler 135°C
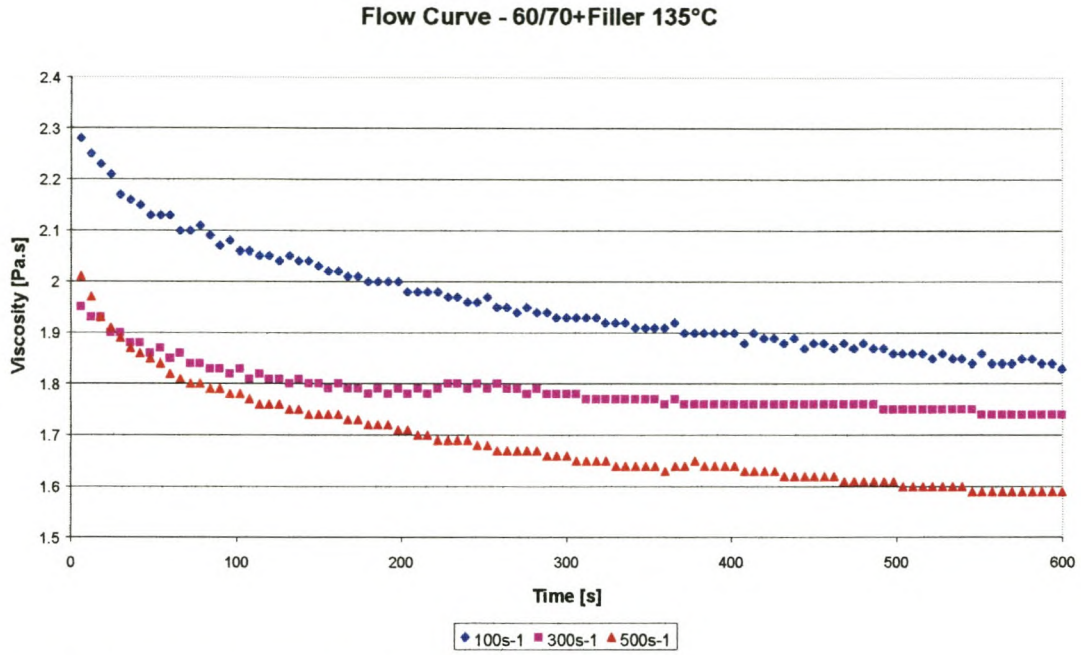
Figure 8-11: Viscosity vs. time curve for 60/70+Filler at 135 °C.

Flow Curve - 60/70+Filler 105°C
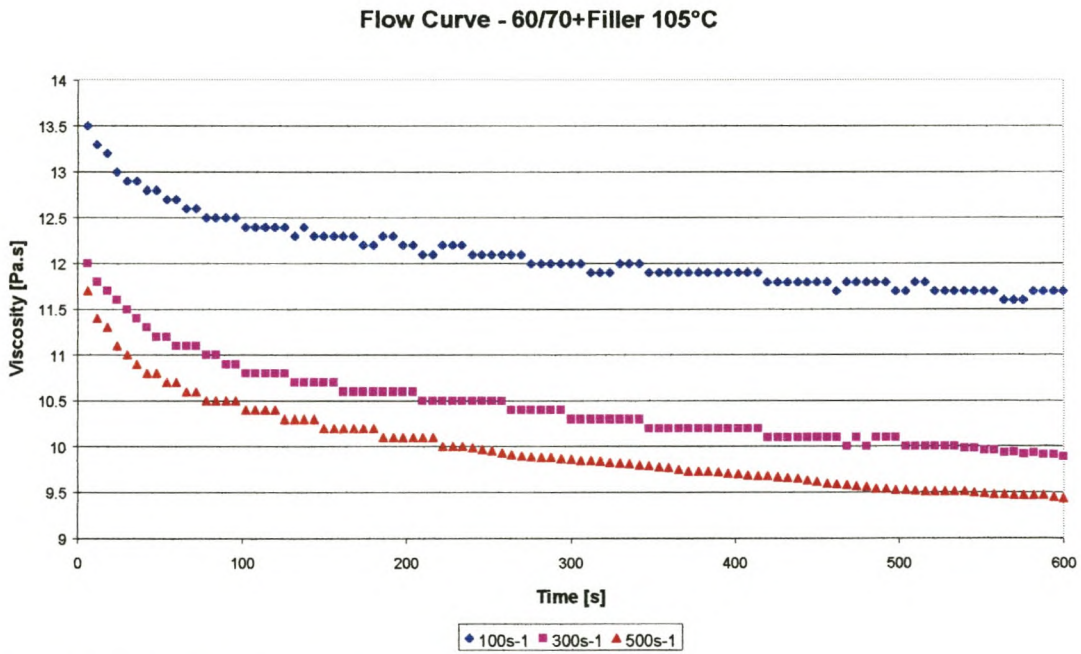
Figure 8-12: Viscosity vs. time curve for 60/70+Filler at 105 °C.

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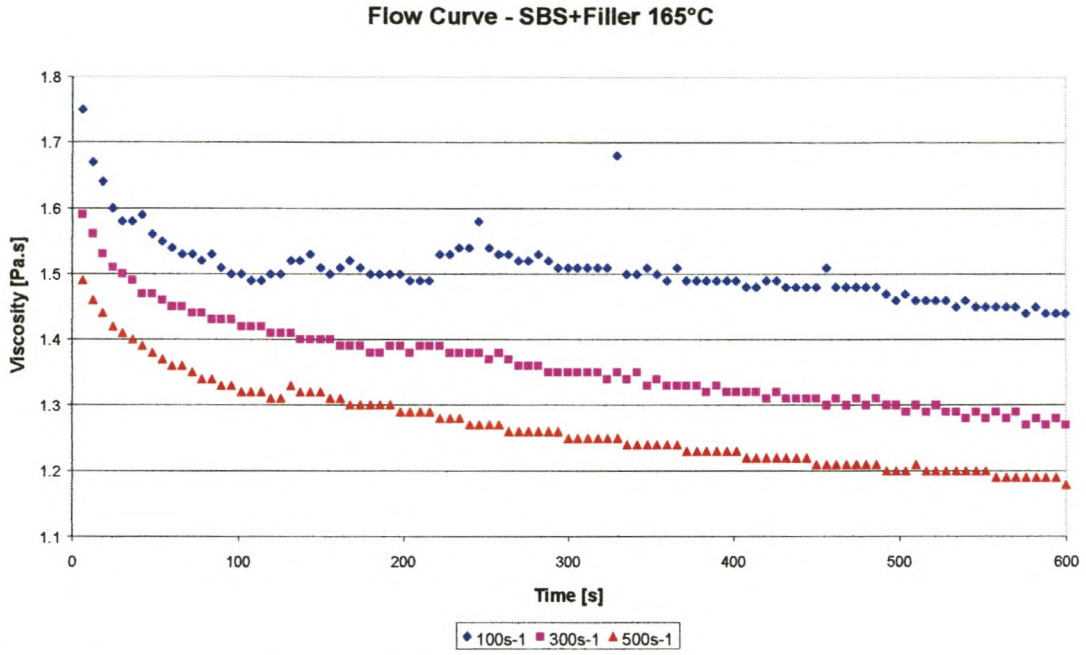
Figure 8-13: Viscosity vs. time curve for SBS+Filler at 165 °C.
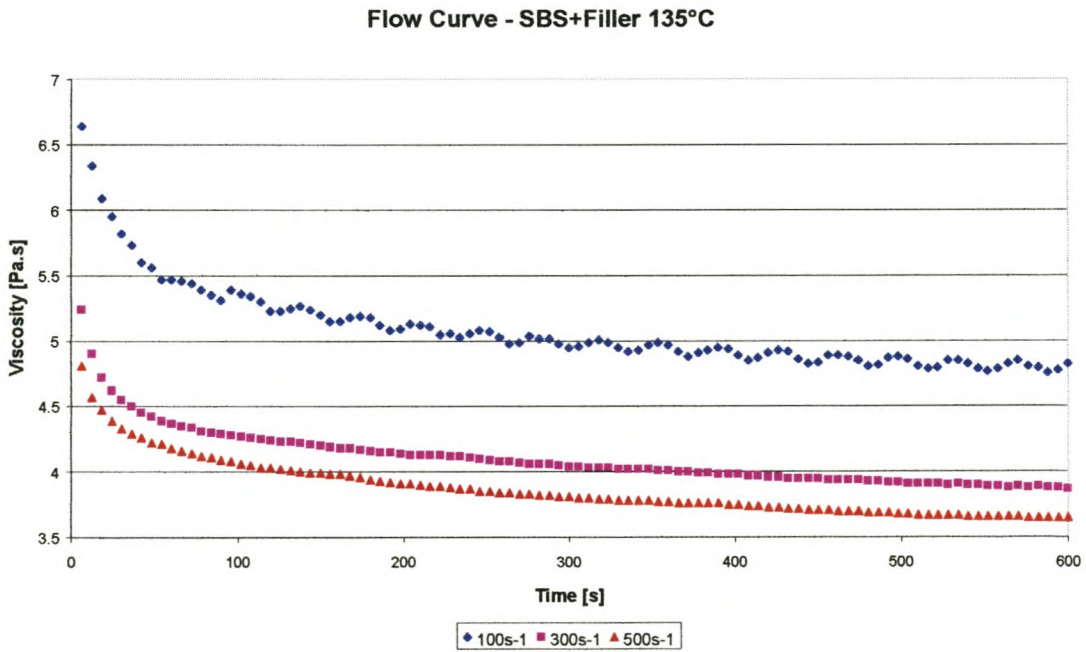
Figure 8-14: Viscosity vs. time curve for SBS+Filler at 135 °C.

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Flow Curve - SBS+Filler 105°C
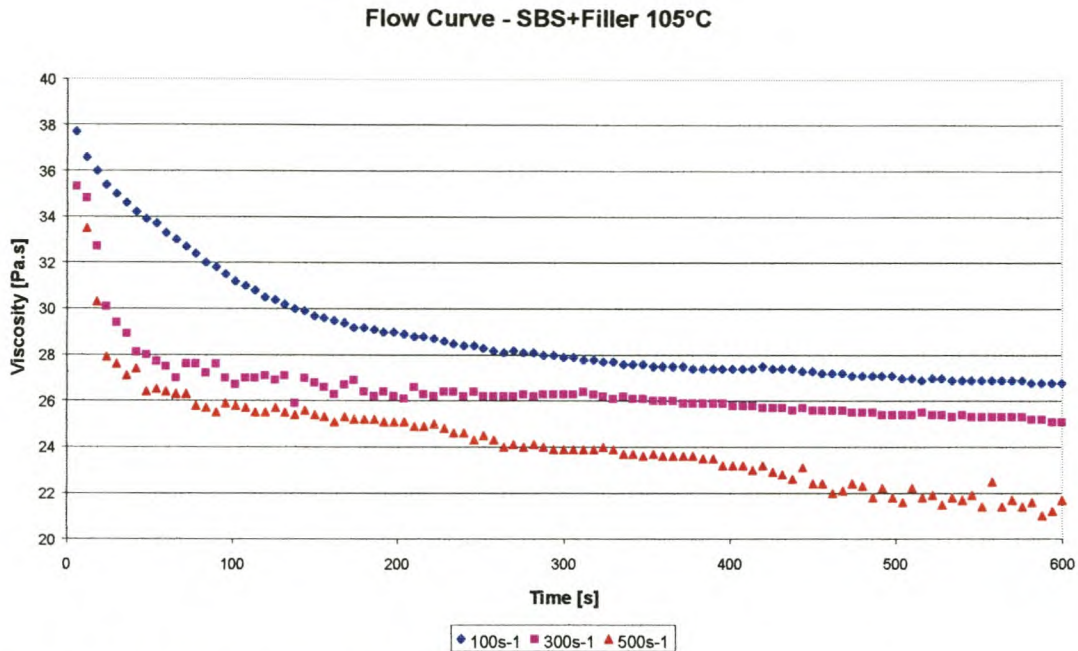
Figure 8-15: Viscosity vs. time curve for SBS+Filler at 105 °C.

All these curves (Figure 8-10 to Figure 8-15) show that the viscosity is decreasing with time – even after ten minutes of testing. This clearly indicates time dependent behaviour. The only exception to this is the 60/70 at 165 °C curve (Figure 8-10) – the two curves at $300s^{-1}$ and $500s^{-1}$ seem to have reached a constant value of viscosity.

The rheological behaviour that is characterised by a decrease in viscosity with time is thixotropic behaviour (Appendix E). As was mentioned in Appendix E the cause for thixotropic behaviour is normally sought in the network structure of the material being broken down. In the case of the binder/filler systems the cause for this behaviour might well lie in the rearrangement of the filler particles.

The gap setting between the spindle surface and the bottom plate of the rheometer (0,2 mm) was chosen so as to leave enough room for the filler particles to move around in. However, a sensitivity analysis was not performed on the gap setting. It could well be that there is a confinement problem with the gap setting that was used (0,2 mm) and that higher viscosities than the true viscosity of the binder/filler systems were measured. A

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filler/spindle interaction could also explain why the viscosity did not tend towards a constant value.

Because the viscosity did not reach a constant value at the end of the test, shear stress vs. shear rate curves similar to those drawn for the binders could not be drawn for the binder/filler systems. It is clear from these results that the addition of the filler results in behaviour dissimilar to that of the binder.

However, in an attempt to characterise the binder/filler systems, viscosity measurements are compared in the following two tables (Table 8-2 and Table 8-3). The viscosity values for the binder/filler systems were obtained from averaging the last five viscosity measurements in the data files. The viscosity measurements presented for the binders are not the calculated apparent viscosities, but are the average of the last twenty points of the measured viscosities during the viscosity tests.

Shear rate	SBS+Filler			SBS		
	165 °C	135 °C	105 °C	165 °C	135 °C	105 °C
100	1.442	4.796	26.820	0.312	0.834	5.464
300	1.274	3.880	25.180	0.309	0.905	5.088
500	1.188	3.650	21.380	0.303	0.775	4.722

Table 8-2: Viscosity measurements for binder and binder/filler system for SBS.

Shear rate	60/70+Filler			60/70		
	165 °C	135 °C	105 °C	165 °C	135 °C	105 °C
100	0.481	1.842	11.680	0.098	0.356	2.280
300	0.412	1.740	9.912	0.099	0.351	2.288
500	0.415	1.590	9.460	0.097	0.352	2.297

Table 8-3: Viscosity measurements for binder and binder/filler system for 60/70.

From these tables it is clear that there is a definite increase in the viscosity of the binder/filler systems compared to the binders. The effect of temperature decrease is also clear from the tables – viscosity measurements increase with decrease in temperature. The SBS has higher values for viscosity in all instances. The SBS + Filler shows the

same trend. The SBS measurements result in viscosities approximately two to three times higher than the 60/70 measurements.

8.3 Bitumen-Rubber

As was mentioned earlier under the description of the methodology followed for the high temperature viscosity tests for the binders, these tests were not performed on the Bitumen-Rubber. This was due to an erroneous decision that was made based on a visual evaluation of the test procedure on the Bitumen-Rubber. It is the purpose of the following discussion to shed some light on the reasons that led to the decision.

When the high temperature viscosity tests were started, it was felt that the particle size of the rubber crumb would have an influence on the tests. In the case of the 60/70 and SBS binders, the structure of the binders is homogenous – the SBS modification is done on a molecular level with the addition of the polymer to the bitumen.

Unlike the SBS, the rubber modification is done through the addition of rubber crumb that forms an inhomogeneous mixture of bitumen and rubber. The particle size is dependent on the reaction time during mixing, as well as subsequent bouts of heating.

The reasons mentioned here led to the feeling that the gap setting of the DSR during the high temperature tests would have an influence on the Bitumen-Rubber's results. For the 60/70 and SBS binders a gap setting of 0,1 mm was used. The Bitumen-Rubber's gap setting would have to be much bigger due to the particle size of the rubber crumb.

To determine the size of the gap setting for the Bitumen-Rubber a series of viscosity tests were performed at a shear rate of 100 rad/s and a temperature of 165 °C. The gap settings that were used were 0,5 mm, 1,0 mm, 1,5 mm and 2,0 mm. The tests were visually assessed for any abnormal behaviour.

Abnormal behaviour was indeed observed during the high temperature viscosity tests on the Bitumen-Rubber samples. An amount of hot binder was placed on the test plate of the DSR and the spindle was lowered to the trim position. The excess binder was removed and the spindle was moved to the test gap setting. The test was then started. It was at this point that the abnormal behaviour started.

When the spindle started to turn, Bitumen-Rubber was "thrown" out from the sides of the sample (Figure 8-16 shows this schematically). This continued until equilibrium was reached when no more material was "thrown" out. At this point a "windrow" started to form on the edge of the sample and interference could be observed between the material in the "windrow" and the material that remained of the sample. As the gap setting was increased, the perceived amount of material "thrown" out decreased and the "windrow" was not as pronounced. The decrease in the amount of material "thrown" out could explain the increase in viscosity with increase in gap setting on Figure 8-17 for the gap settings 1,0 mm, 1,5 mm and 2 mm.

| Start of test | Material "thrown" out and "windrow" starting to form | "Windrow" formed |
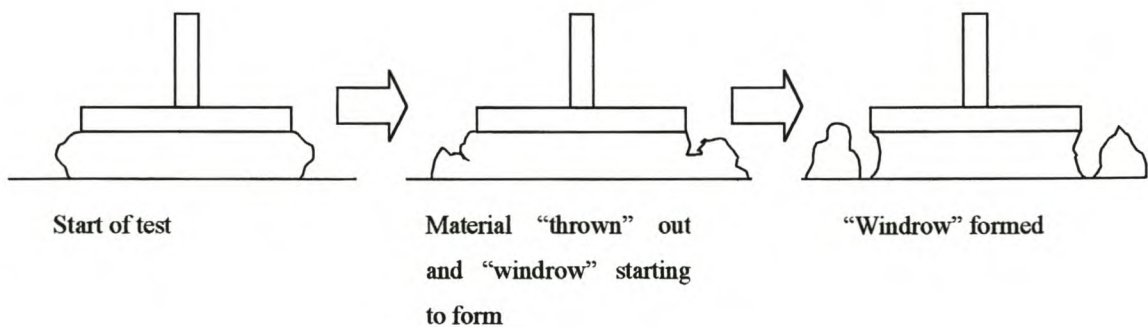
Figure 8-16: Schematic representation of viscosity tests on Bitumen-Rubber.

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Viscosity Curves at Different Gap Settings - Bitumen-Rubber
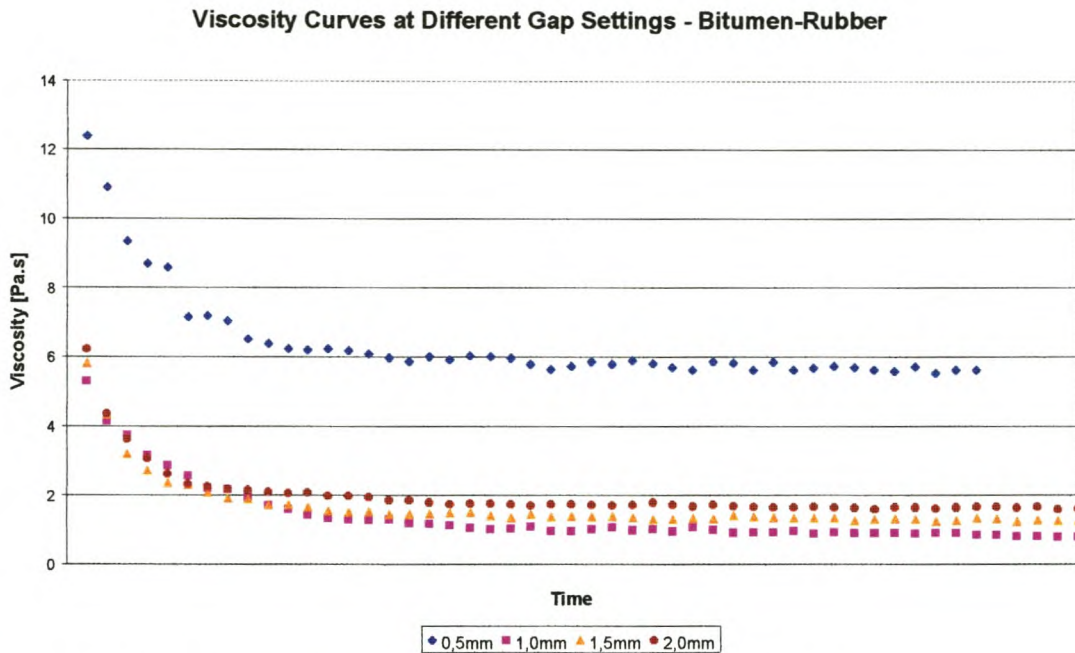
Figure 8-17: Viscosity tests at 100 rad/s shear rate and 165 °C on Bitumen-Rubber.

Evaluating Figure 8-17, it is clear that the decision not to test the Bitumen-Rubber based on the visual assessment of the test was wrong. It can be observed that the 0,5 mm gap setting did indeed lead to a very high viscosity being measured. On the other hand, the results for the 1,0 mm, 1,5 mm and 2,0 mm gap settings are quite close together. It can also be seen that the viscosities measured for the mentioned gap settings increased with increase in gap setting. This can be attributed to the fact that the "windrow" effect was less pronounced at the bigger gap setting than at the smaller. This would translate into a bigger area of binder in contact with the spindle. This in turn would lead to a higher value for the force needed to turn the spindle, which translates into a higher viscosity being measured.

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8.4 Conclusions

When all the results of the high temperature viscosity tests on the binders and binder/filler systems are considered, the following conclusions may be drawn from it:

8.4.1 Binders:

The SBS binder exhibits non-Newtonian behaviour – this can be seen in the y-intercept of the regression lines of the shear stress vs. shear rate plots. Recent research points toward shear thinning behaviour in the region of shear rates not covered by this research $(0,1 \text{ s}^{-1} - 93\text{s}^{-1})$. [8] This behaviour becomes more defined with a decrease in temperature. The viscosity curves drawn also point toward shear thinning behaviour (increase in viscosity with decrease in shear rate) that is most noticeable at 105 °C.

The 60/70 binder exhibits Newtonian fluid behaviour at all temperatures. This is the generally accepted behaviour of unmodified bituminous binders.

For the SBS, plots of the apparent viscosity at the three temperatures (105 °C, 135 °C and 165 °C) show an increase in apparent viscosity with decreasing shear rate. This effect becomes more noticeable with decreasing temperature – specifically at 105 °C. This leads to the conclusion that the shear rate and temperature during mixing and compaction is very important. If the shear rate can be shown to be below a value of 300s^{-1}, the effect of the polymer will result in very high viscosities. At shear rates above approximately 300s^{-1}, the viscosity curve is approximately a horizontal line and the viscosity can be assumed constant.

8.4.2 Binder/Filler Systems:

The binder/filler systems did not reach a constant value for viscosity during the high temperature viscosity tests – even after ten minutes. The viscosity of the binder/filler systems decrease with time and this indicates thixotropic behaviour.

The thixotropic behaviour of the binder/filler systems can be attributed to the rearrangement of the filler particles during the test.

The influence of the gap setting was not studied and this might have shed some light on the thixotropic behaviour of the material. The gap setting used might have been too small and a bigger gap could have given better results.

Tables of viscosity measurements at the end of the viscosity tests on the binder/filler systems, show the increase in viscosity due to the addition of filler when compared to the binders. This points toward a stiffening effect with the addition of filler – a result apparent from all the other tests in this research programme.

9 Conclusions and Recommendations

This study is a step towards the development of performance based specifications for asphalt mixes, based on the properties of the constitutive components.

The objective of this study was the fundamental rheological characterisation of three binders and binder/filler systems. The binders were studied to characterise the effect of polymer modification on the properties of normal bitumen. The binder/filler systems were studied to determine the effect the filler has on the binder properties.

Two rheometers were used to achieve the objective of this study:

- Sliding Plate Rheometer
- Dynamic Shear Rheometer

Some conclusions that were drawn at the end of each chapter are summarised in the following section. After that, recommendations are made based on the conclusions.

9.1 Conclusions

9.1.1 Creep Tests

9.1.1.1 SPR Results

The SPR was used to perform creep tests on the binders and binder/filler systems. It was shown through the drawing of shear susceptibility lines and the calculation of viscosities, that the addition of polymers to bitumen leads to a modified binder that has higher viscosity in the temperature range 10°C to 40°C compared to the normal bitumen. This means that, compared to the normal bitumen, the modified binders are more resistant to flow. This is an important result, as this suggests that the polymer modified binders should be more resistant to permanent deformation.

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9.1.1.2   SPR versus DSR creep test results

The results of the creep tests performed with the SPR were verified with the DSR through creep tests performed under similar conditions. In general, the DSR results and the SPR results were comparable. The DSR results showed that the viscosities calculated for a binder at a temperature tended toward an average value. This was a result not obtained with the SPR. Upon further investigation, the reason for the difference was shown to lie in the duration of the creep tests for the two rheometers. It was also shown through the DSR results that the horizontal shift in the shear susceptibility lines between the modified and unmodified binders, was a measure of the difference in the viscosities – a shift towards lower shear rates at a temperature points toward an increase in the viscosity at that temperature.

9.1.1.3   Influence of filler

The creep test results showed that filler addition leads to higher viscosities being measured, but the overall influence of polymer modification stayed the same. Thus, it was concluded that filler addition does not result in changed binder properties. This was a result obtained with both the SPR and the DSR. However, it must be kept in mind that only one filler type was tested.

9.1.2 *Dynamic Tests*

9.1.2.1   Linear visco-elastic limits

The dynamic testing of the binders gave very interesting results. First, it was shown that temperature has a definite influence on the linear visco-elastic limits of the materials tested. It was also shown that the binder/filler systems have much lower linear limits compared to the binders. The linear limits were used to determine the strain levels at which the frequency sweeps were performed.

9.1.2.2   Frequency sweeps

The frequency sweeps produced good results and the results could be used for the construction of both Master Curves and Black Diagrams. From the two types of curves the effect of polymer addition, ageing and filler addition were determined.

The Black Diagrams were very useful for the characterisation of binder properties. It was shown that the addition of polymer leads to a binder that performs better under high temperatures/low frequencies compared to normal bitumen – the polymer modified binders had a more elastic response under these conditions.

9.1.2.3   Influence of ageing and filler addition

The Master Curves and Black Diagrams showed that both ageing and filler addition leads to a stiffer response from the binders and that the general form of the curves stayed the same. Thus, it was concluded that filler addition does not affect binder properties negatively and that it results in a binder/filler system with much stiffer response to loading compared to the binders.

9.1.3 High (Mixing and Compaction) Temperature viscosity tests

The results of high temperature viscosity tests were used to plot apparent viscosity vs. shear rate for the 60/70 and SBS binders. It was shown that the 60/70 had Newtonian behaviour at the temperatures used and that the SBS had shear thinning behaviour. A decision was made not to test the Bitumen-Rubber binder in this part of the study, which in retrospect was shown to be erroneous. Again, a comparison of viscosity values between the binders and binder/filler systems showed that the filler increases the measured viscosity of the binders.

9.1.4 Overall Conclusions

Overall, the following conclusions may be drawn:

- Polymer addition improves binder response to loading at service temperatures – and thus the expected performance of the polymer modified binders. Ageing and filler addition does not affect this result.
- At high temperatures, care has to be taken when use is made of polymer modified binders as it was shown that these binders exhibit shear rate dependent behaviour.
- Filler addition results in a stiffer mastic compared to the binder, while binder properties are not changed – this is true for the one filler that was tested.

This study showed that the fundamental rheological characterisation of binder properties is superior to the conventional empirical measures. For example, the empirical measures report a value such as penetration and the decrease thereof with ageing or it prescribes a value for ductility. These empirical measures cannot be directly related to the improved binder properties resulting from polymer modification. However, the fundamental rheological characterisation – especially the results of the dynamic tests – could be used directly to predict binder performance (e.g. the Black Diagrams comparing normal bitumen with SBS and rubber modified bitumen).

Through the use of fundamental rheological characterisation, new knowledge regarding the effect of ageing on polymer modified binders and the influence of filler addition on binder properties was gained. Both ageing and filler addition did not alter binder properties as characterised with the Black Diagrams. It was also shown that the shear rate during mixing and compaction of asphalt pavements containing polymer modified binders is an important factor – the shear rate should also be considered when mixing and compaction temperatures are determined for polymer modified binders.

In the following section some recommendations are made pertaining to the results obtained in this study.

9.2 Recommendations

The difference in the DSR and SPR results for the creep tests should be investigated further. The recommendations in this regard are:

- The strip-chart recorder should be replaced with a direct real-time link to a computer system for the displacement recording.
- The load levels used during the tests should be chosen lower than used in this study to allow for longer test durations – this should lead to results for which the calculated viscosities for a binder will converge on an average value at a temperature.

The results of the high temperature viscosity tests must be investigated further. It is recommended that:

- The shear rate during mixing and compaction must be determined to ascertain whether it will have an impact or not.
- The study should be expanded to include shear rates below $100s^{-1}$.
- The Bitumen-Rubber should be included.
- The impact of the gap setting on the results of these tests on the binder/filler systems must be ascertained.

This study was a first step towards the development of performance-based specifications for asphalt pavements. First, the binder properties were characterised. After this, the influence of one type of filler on the binder properties was evaluated. The following recommendations are made with regard to the expansion of the research programme:

- The next step should be to go to full-scale mixes and do performance testing on those.
- An attempt should be made to relate the Master Curves and Black Diagrams for the binders and binder/filler systems to the same curves for mixes.
- The programme should be expanded to include more binders and to use different mixes.
- The programme should be expanded to include more filler types.

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Rheology

In this appendix the basic concepts of rheology are defined and an introductory discussion into the rheology of materials is given. This chapter is largely an adaptation of Dutch notes on rheology obtained from the study leader [9].

E.1. Stress and Strain

In rheology the relationship between stress and strain (deformation) or strain rate (rate of deformation) is studied. Understanding stress and deformation are basic to understanding rheology. For this reason stress and strain will be elaborated on.

E.1.1. Strain

Strain is the quantitive measure of the change in form (deformation) of a material when subjected to a force. The change in form of the material can be reported in a number of ways.

For example the elongation of a material when subjected to a force may be under consideration (Figure E-1).
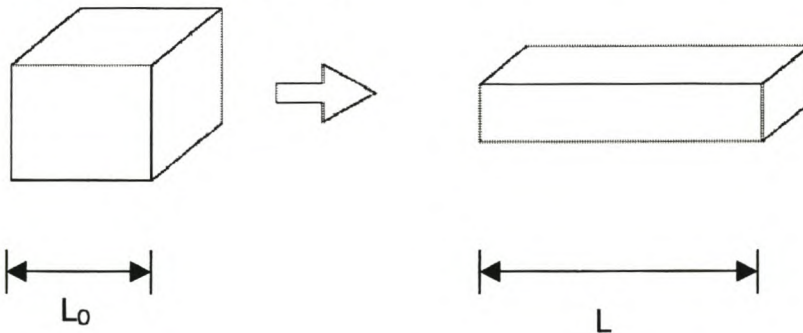

Figure E-1: Simple elongation of an element under a tensile load.

As measure of change in form, the change in length may be reported:

$$Elongation = L - L_0$$

The disadvantage of using this definition is that the elongation is a function of the original length. Therefore, it is better to work with the relative change in form of the material, which may be expressed as:

$$Strain = \varepsilon = \frac{(L - L_0)}{L_0} = \frac{L}{L_0} - 1$$

A second type of deformation is shear deformation (Figure E-2). This type of deformation occurs when a material is placed between two parallel plates. One of the plates is then displaced parallel to the other.
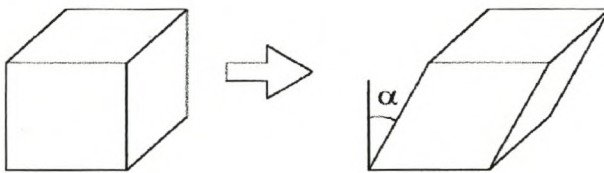

Figure E-2: Simple shear on an element.

Every volume element will undergo the same local deformation if there is no slip at the plate interfaces and the inertial forces can be ignored. Figure E-3 shows the displacement of a sample of material between two parallel plates.
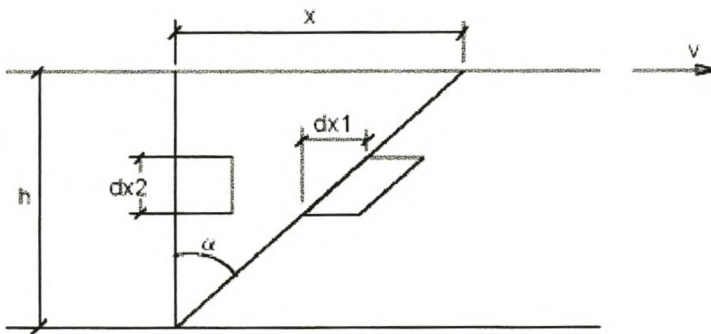

Figure E-3: Shear between parallel plates. [9]

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The measure for deformation in this case is the shear strain γ and is defined as follows:

$$\gamma = \tan\alpha = \frac{dx_1}{dx_2} = \frac{x}{h}$$

$\alpha \rightarrow$ shear angle

When a fluid is being studied it is subjected to a constant shear velocity (v). The velocity of the fluid at a distance x_2 from the bottom plate is given by:

$$v_1 = \frac{x_2}{h}v$$

The local shear rate is given by:

$$\dot{\gamma} = \frac{d\gamma}{dt} = \frac{v}{h}$$

Where

$$\frac{d\gamma}{dt} = \frac{d}{dt}\left(\frac{x}{h}\right) = \frac{1}{h}\frac{dx}{dt} = \frac{v}{h}$$

Flow with a constant shear rate is known as steady simple shear. The unit of shear rate is s^{-1}.

E.1.2. Stress

Stress is force per unit area. Stress is measured in pascal (Pa = N.m^{-2}).
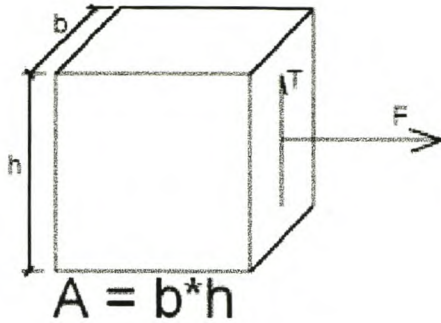
Figure E-4: Two types of stress.

In physics two types of stress are described (Figure E-4):

1.   Normal stress: this is a stress that acts perpendicular to the surface the force is applied to. *(σ = F/A)*

2.   Shear stress: this is a stress which acts parallel to the surface the force is applied to. *(τ = T/A)*

To describe stresses in three dimensions nine quantities have to be specified. (For each of the three independent faces of the element: one normal stress and two shear stresses.) Six of these quantities are independent. None of the calculations in this text is done in three dimensions so the stress calculations will not be developed further in three dimensions.

E.2. Material Function and Material Constant

To explain the difference between the two concepts (material function and constant) we have to go back to the definition of rheology. The relationship between stress and strain or strain rate can be given by one or more material functions of the material being studied. When working with a pure elastic material the stress is proportional to the strain (Hooke's Law). In this case the material function is a constant known as the modulus of elasticity.

E.3. Newtonian Fluid

For a purely viscous material the stress is proportional to the rate of deformation (Newton's Law). In this case the material function is also a constant called the viscosity (η):

$$\tau = \eta \frac{d\gamma}{dt} = \eta \dot{\gamma}$$

In fluids with a low molecular weight the viscosity is only dependent on the pressure and temperature. The rate of shear does not affect the viscosity. Such fluids are known as Newtonian fluids.

One material constant (viscosity) is sufficient in the case of a Newtonian fluid to describe the stresses in the fluid in terms of the deformation.

E.4. Non-Newtonian Behaviour

Suspensions, emulsions, solutions of polymers and polymer melts usually exhibit non-Newtonian behaviour. In such a case one measurement of viscosity is not enough to characterise the material behaviour. When such a material is subjected to shear it will be seen that the viscosity is dependant on the shear rate. The result is that such a material does not have a material constant but a material function:

$$\eta = \eta\left(\dot{\gamma}\right)$$

The cause of non-Newtonian behaviour is the complexity in the structures of the systems named. The structure changes with the shear rate and this causes the viscosity to change.

If the change in the structure with the change in shear rate is dependent on time too we find that the viscosity is not only a function of shear rate but also of time:

$$\eta = \eta\left(\overset{\bullet}{\gamma}, t\right)$$

Unlike the previous definition of viscosity this is not a material function anymore.

E.5. Shear Rate Dependent Behaviour

Different types of shear rate dependent behaviour are given in Figure E-7. These figures are also known as flow curves or viscosity functions.

Viscosity can be defined in two ways:

1. The viscosity at a given shear rate is given by the slope of the flow curve at the shear rate (Figure E-5):

$$\eta = \frac{d\tau}{d\overset{\bullet}{\gamma}} \frac{[Pa]}{[s^{-1}]} \qquad \text{(slope viscosity)}$$

the dimension of η is Pa.s.

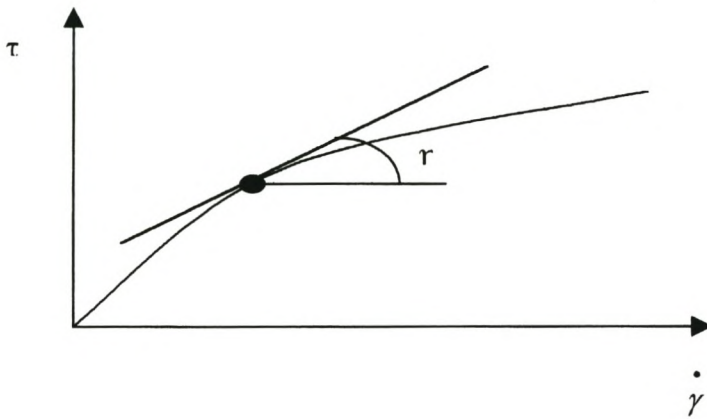**Figure E-5: Slope viscosity.**

2. The viscosity at every point is the ratio of shear stress to shear rate (Figure E-6):

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$$\eta = \frac{\tau\left(\dot{\gamma}\right)}{\dot{\gamma}} \qquad \text{(apparent viscosity)}$$
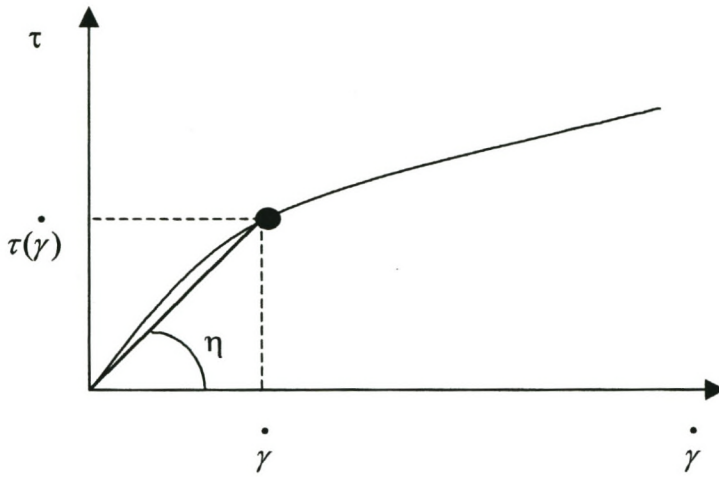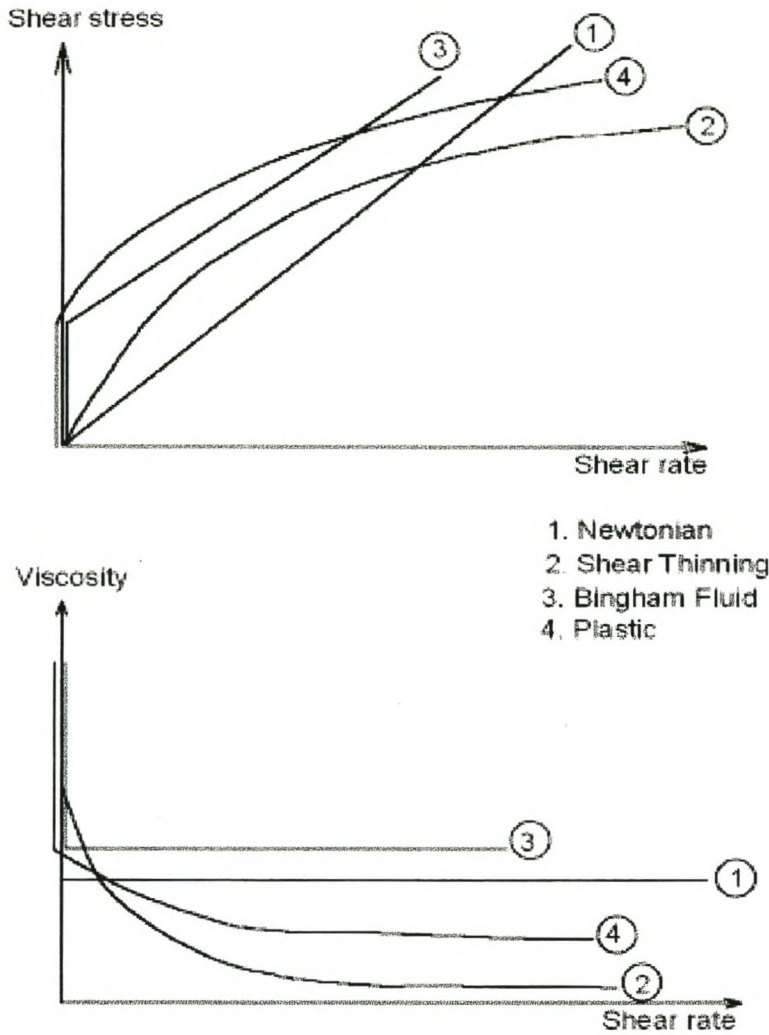

Figure E-6: Apparent viscosity.

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Figure E-7: Different types of shear rate dependent flow behaviour (slope viscosity). [9]

The viscosities in Figure E-7 were derived according to the first definition of viscosity (slope viscosity).

Newtonian flow behaviour has been discussed in detail previously. In the following sections the focus will fall on shear thinning, Bingham and plastic behaviour.

E.5.1. Shear Thinning

Shear thinning is the term used for materials that show a decrease in viscosity with an increase in shear rate. The curve representing this type of material is curve number 2 in Figure E-7.

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This type of behaviour is common for the following types of systems:

- Dispersions (solid particles dispersed in a fluid)

- Emulsions (droplets of a fluid in a different fluid)

- Solutions of polymers

When in rest these systems show a certain type of disorder, but when these systems are subjected to flow the particles re-orientate in a different pattern (Figure E-8).
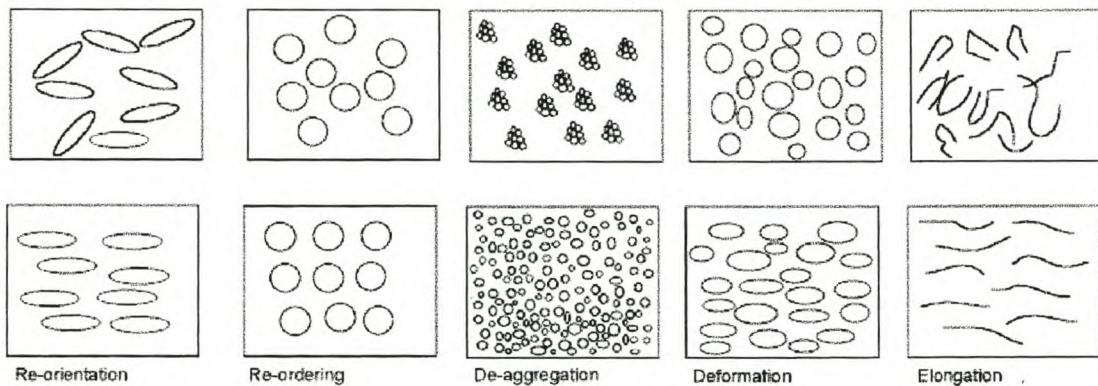

Figure E-8: Types of re-ordering leading to viscosity changes. [9]

The re-orientation in all of these systems leads to a reduction in the viscosity measured.

Shear thinning is a reversible phenomenon. When the source of shear flow is removed the system returns to its previous state.

There are a number of empirical methods used to describe the shear thinning effect. The most commonly used one is the power law equation to describe the relation between shear stress and shear rate:

$$\tau = k \dot{\gamma}^n$$

where k and n are constants that have to be determined for the system being studied.

E.5.2. Material with a Yield Strength

Materials that have a yield strength behave in an elastic fashion when the stress applied is below the yield strength. If the stress increases to above the yield strength the material will flow.

A class of such materials is the Bingham fluids. These materials exhibit linear viscosity above the yield strength. See graph 3 in Figure E-7. The relation between stress and shear rate can be characterised by the following formula:

$$\tau = \tau_0 + k\dot{\gamma}$$

where k is a material constant that has to be determined experimentally.

Materials that exhibit non-linear behaviour above the yield point (graph 4 in Figure E-7) are called plastic or visco-plastic materials. These materials can be characterised by the following formula that makes use of the power law described under shear thinning:

$$\tau = \tau_0 + k\dot{\gamma}^n$$

where k and n are constants that have to be determined experimentally.

Figure E-9 gives a schematic explanation of the behaviour of a material with a yield strength. The first figure shows the system in rest. In the second figure the applied deformation leads to a stress below the yield strength. Thus, the material behaves elastic because the system returns to the rest state when the deformation is removed. In the third figure the stress exceeds the yield strength of the material and the system is broken down. This leads to a decrease in the viscosity with increasing shear rate. Normally these materials return to the rest state after the source of deformation has been removed, but materials exist that do not return to the rest state.
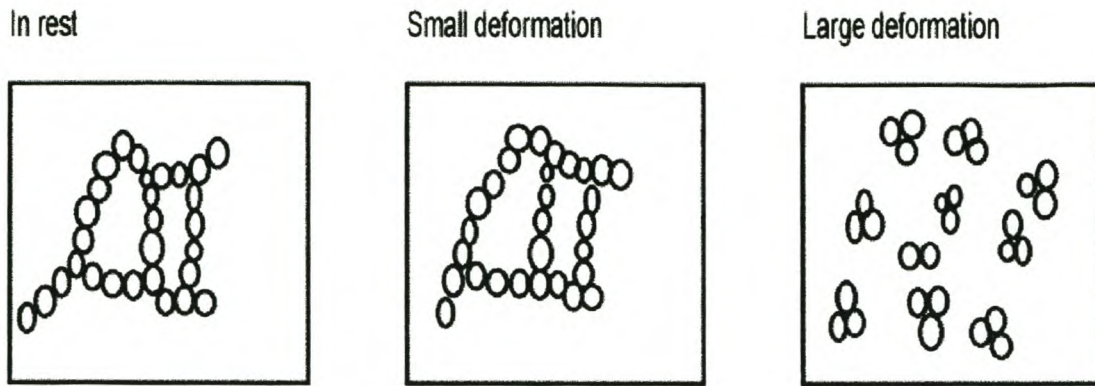
In rest   Small deformation   Large deformation

Figure E-9: Behaviour of a material with yield strength. [9]

E.6. Time Dependent Behaviour

Materials exist for which viscosity is not only shear rate dependent but the viscosity is also dependent on the time elapsed after the start of the experiment (application of deformation). Such behaviour is referred to as thixotropic behaviour.

Strictly speaking the term viscosity may not be used since the term viscosity has been defined only for stationary situations where the stress does not vary with time. Viscosity as it is used in this instance is defined as the instantaneous shear stress divided by the shear rate.

Thixotropy and linear visco-elasticity will be discussed further in this section on time dependent behaviour.

E.6.1. Thixotropy

Thixotropy is the term used to define the behaviour of a material for which the viscosity decreases with time in a stationary shear experiment. When the source of deformation is removed the viscosity returns to its initial value. (Figure E-10)
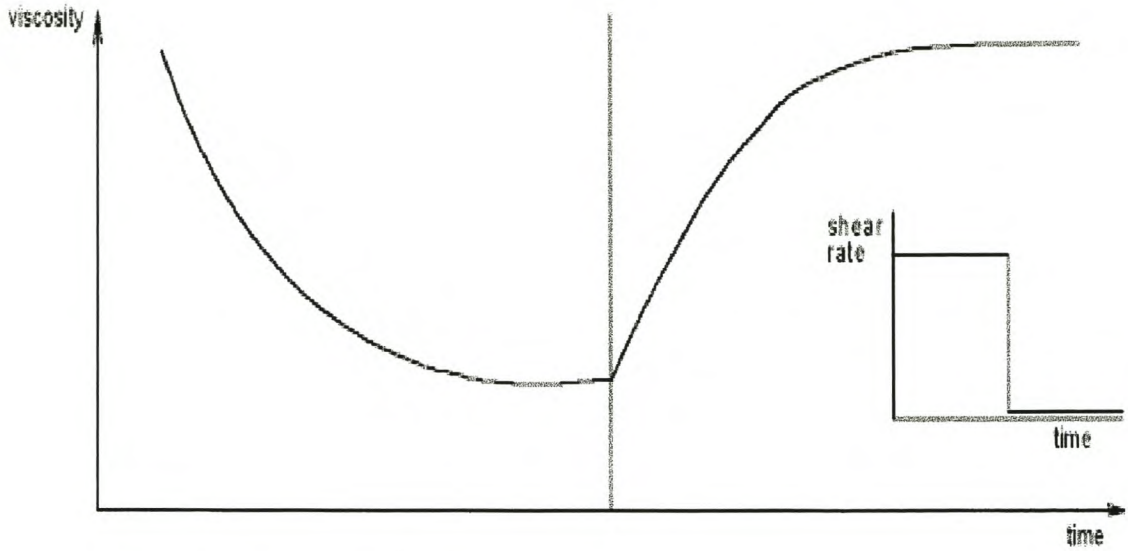
Figure E-10: Thixotropic behaviour – constant shear rate.[9]

When the shear rate is linearly increased from zero to a value and then decreased to zero again, the curves for the shear stress will not run concurrent. The viscosity will also not have concurrent curves for this experiment (Figure E-11).
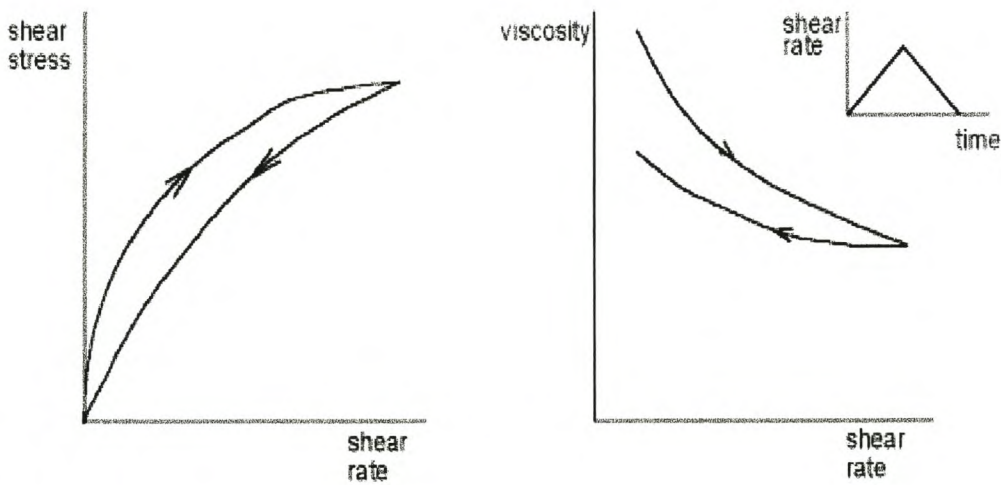

Figure E-11: Thixotropic behaviour - linearly increasing shear rate. [9]

The cause for thixotropic behaviour is normally sought in the network structure of these materials. As the experiment progresses with time, more and more bonds in the structure are being broken and so the viscosity decreases with time. When in rest again the structure returns to the initial conditions.

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Materials exist that exhibits thixotropic behaviour, but these materials do not return to the initial conditions when in rest. Thus, the viscosity does not return to the initial value, but remains at the value at the end of the measurement. An example of such a material is yoghurt.

E.6.2. Visco-elasticity

The viscosity of thixotropic materials is dependent on the shear deformation history of the material. A visco-elastic material has a memory for the deformation history. These visco-elastic materials exhibit two types of behaviour:

- Stress relaxation
- Elastic recoil

The basis for the discussion of these two phenomena is the simple shear experiment. In this experiment a sample of the material studied is placed between two plates. One of the plates is then displaced parallel to the other while the other one is kept stationary. The moving plate is then stopped instantaneously and cannot be displaced further. The response of the material is recorded.

If the material is a viscous material the stress will return to zero instantaneously. A visco-elastic material's stress will return to zero over time. If the material's response can be described by an exponential function, the relaxation time is defined as the time needed for the stress to decrease to *1/e* of the initial value. Figure E-12 shows a typical relaxation curve.
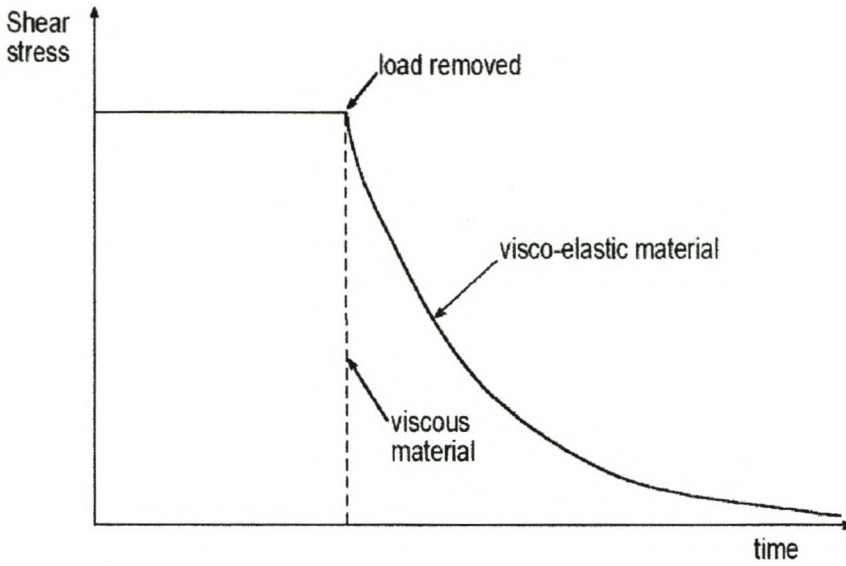
Figure E-12: Stress relaxation curve - visco-elastic material. [9]

In another experiment the plate is not just stopped, but it is also released to displace. If the material is viscous the plate will remain in the same position. In the case of a visco-elastic material, the sample will exhibit elastic recoil (Figure E-13) – the plate will move back in the direction opposite to the direction in which it was displaced earlier. The recoil is not instantaneous but will increase over time until a maximum value is reached. The time needed to decrease the strain to a value of $1/e$ of the initial value is called the retardation time.
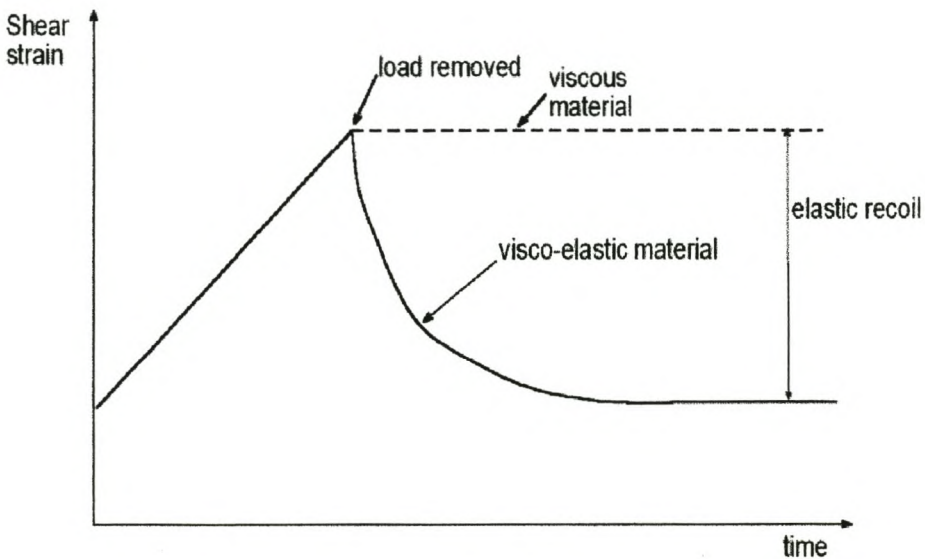

Figure E-13: Elastic recoil - visco-elastic material. [9]

E.7. Linear Visco-elasticity

Linear visco-elasticity is the term used when the material functions being measured seem to be independent of the magnitude of the deformation or the rate of deformation.

Studying the linear visco-elastic behaviour of a material will give insight into the undistorted state of that material. It should be noted that the behaviour of materials at large deformations cannot be described by linear visco-elasticity.

In this section three types of experiments will be discussed: stress relaxation, creep and dynamic experiments.

E.7.1. Stress relaxation experiment

A constant shear rate $(\dot{\gamma})$ is applied over a very short time (t') to a sample of the material studied. For linear visco-elastic materials the following relation will be applicable for times comparatively short to t':

$$\tau(t) = \gamma_0 G(t)$$

In this relation $G(t)$ is called the relaxation function. $G(t)$ is a monotonously decreasing function in time. In the case of a purely elastic material this function is a constant (shear modulus G). Figure E-14 shows the stress relaxation experiment graphically.
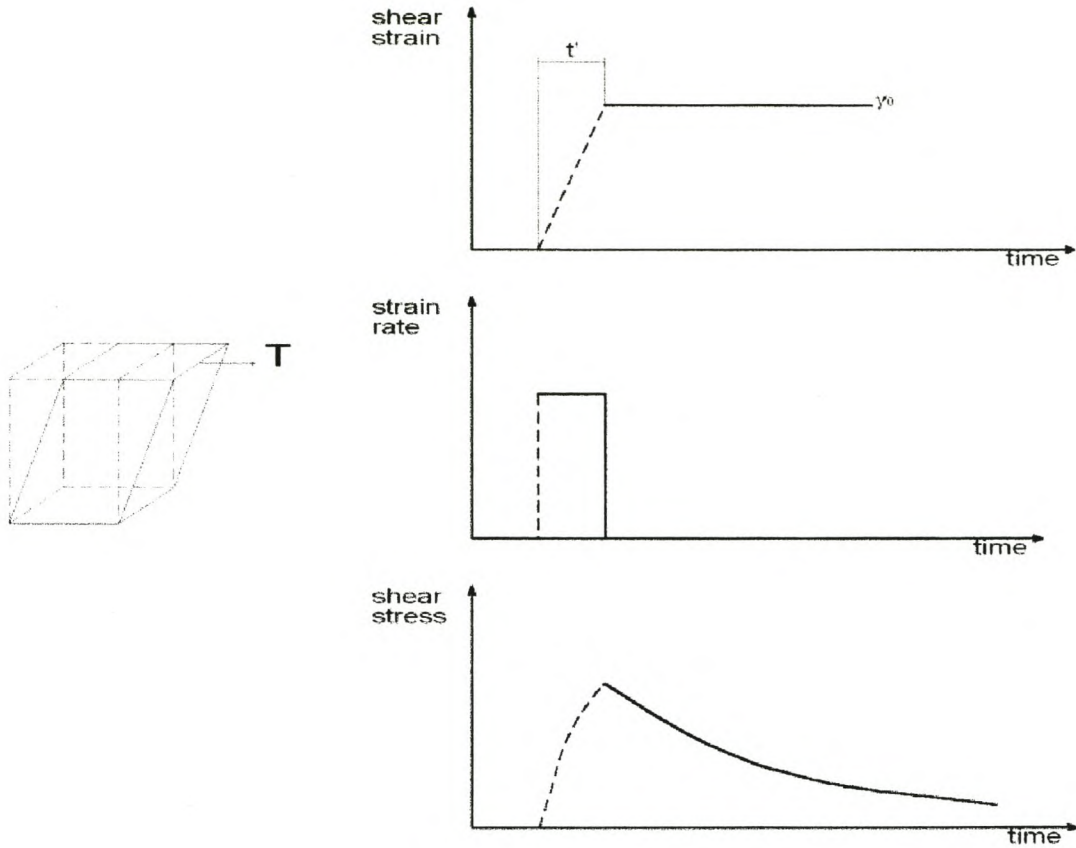
Figure E-14: Stress relaxation experiment - linear visco-elasticity. [9]

E.7.2 Creep experiment

In this experiment a constant shear stress (τ_0) is applied to a sample over a very short time (t') and then it is kept constant. The following relation gives the deformation as a function of time (for time much greater than t'):

$$\gamma(t) = \tau_0 J(t)$$

In this relation J(t) is known as the creep compliancy of the material (or the retardation function). The unit of measurement of the creep compliancy is the reciprocal of that of the modulus (m^2N^{-1}). $J(t)$ increases monotonously with time and in the case of a purely elastic material $J = 1/G$. For a visco-elastic material $J(t) \neq 1/G(t)$. Figure E-15 shows the creep experiment graphically.
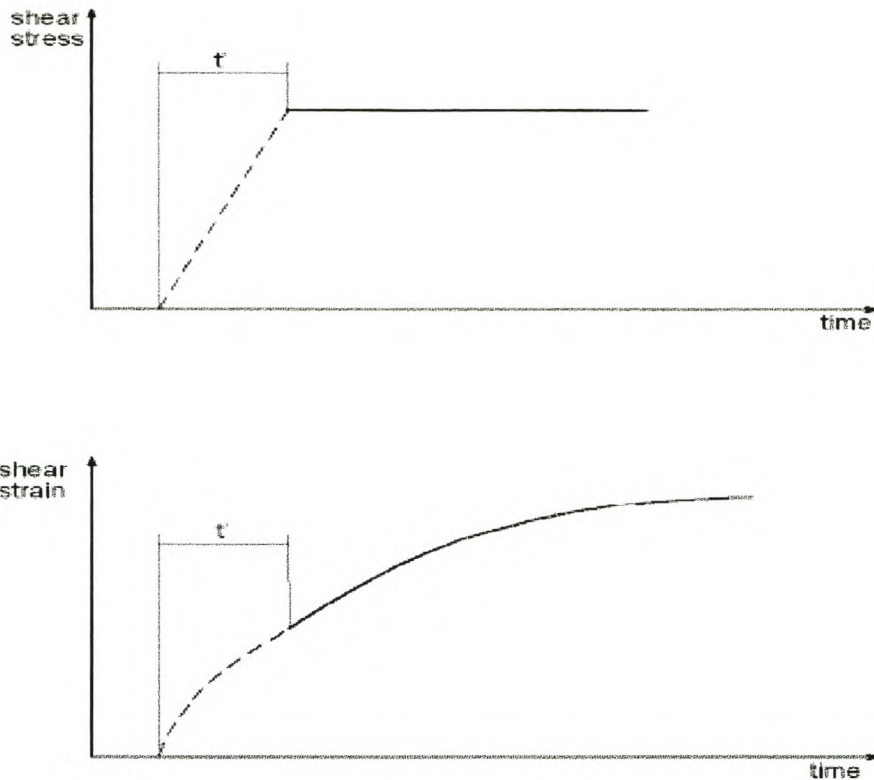
Figure E-15: Creep experiment - linear visco-elasticity. [9]

E.7.3. Dynamic Experiment

Creep and relaxation experiments are very difficult to perform on systems of fluids (such as bitumen at high temperature). Experimentally it is much easier to do dynamic measurements on these materials. Another aspect that increases the difficulty of these experiments is the fact that the displacements have to be very small to stay in the linear range. It is much easier to impose small displacements and measure material response in the dynamic mode. See Figure E-16.
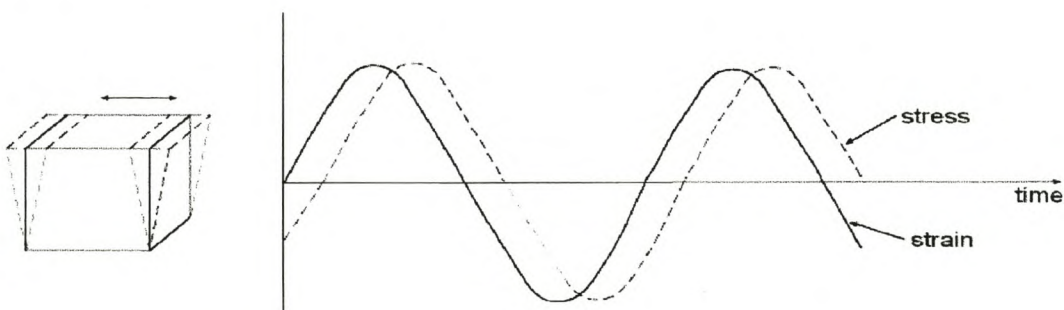

Figure E-16: Dynamic experiment.

In the dynamic experiment the deformation is applied to the sample in a sinusoidal wave. The wave varies in time with an angular frequency ω. The resultant stress also varies in a sinusoidal fashion and is recorded.

Strain in the dynamic experiment can be described as follows:

$$\gamma(t) = \gamma_0 \sin \omega t$$

where γ_0 is the amplitude of the strain.

During the dynamic experiment it is observed that there is a time lag between the applied strain and the measured response (shear stress). The magnitude of the time lag is characterised by the phase angle, δ.

The phase angle has two extremes in magnitude:

$\delta = 0°$ purely elastic material

$\delta = 90°$ purely viscous material

The stress that is measured is described by the following equation:

$$\tau(t) = \tau_0 \sin(\omega t + \delta)$$

When the two extreme values for the phase angle are observed, the following two equations result:

$$\tau(t) = \tau_0 \sin \omega t \qquad \text{purely elastic response}$$

$$\tau(t) = \tau_0 \sin\left(\omega t + \frac{\pi}{2}\right) \qquad \text{purely viscous response}$$

For a visco-elastic material the magnitude of the phase angle varies between the two extreme values ($0° < d < 90°$) and the stress is described by:

$$\tau(t) = \tau_0 \sin(\omega t + \delta)$$
$$= \tau_0 \cos \delta \sin \omega t + \tau_0 \sin \delta \cos \omega t$$

The first term ($\tau_0 \cos \delta \sin \omega t$) in the equation above is the in-phase component of stress, while the second term ($\tau_0 \sin \delta \cos \omega t$) is the out-of-phase component of stress.

The stress can also be described by:

$$\tau(t) = \gamma_0 (G' \sin \omega t + G'' \cos \omega t)$$

where $\quad G' \rightarrow$ the storage modulus

$\quad\quad G'' \rightarrow$ the loss modulus

These moduli are frequency dependent. From the above equation for stress it can be seen that the stress is made up of an in-phase part ($G' \sin \omega t$) and an out-of-phase part ($G'' \cos \omega t$).

The two equations for stress can be compared to form the following relationships:

$$G' = \frac{\tau_0}{\gamma_0} \cos \delta$$

$$G'' = \frac{\tau_0}{\gamma_0} \sin \delta$$

$$\frac{G''}{G'} = \tan \delta$$

A purely elastic material does not have a loss modulus and the stress is in-phase with the strain at every moment in time. A purely viscous material does not have a storage modulus and the stress is always 90° out-of-phase with the strain.

E.7.4. Closing thoughts

In this part of the discussion of rheology, three experiments were described that are used to characterise material behaviour for visco-elastic materials.

Two of these experiments will be used further in this study on the rheology of binders and binder/filler systems:

- Creep experiment
- Dynamic experiment

A third experiment is also used: flow experiment. The flow experiment is a direct measurement of the viscosity of materials at temperatures where the materials are sufficiently fluid to flow.