Chapter 24
Influence of Thermal Ageing Process on the Crack Propagation of Rubber Used for Tire Application

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Abstract The present work studies the influence of thermal aging behaviour of rubber compounds based on natural rubber (NR), styrene–butadiene rubber (SBR) and their blend NR/SBR 60/40 on dynamic-mechanical and fatigue crack growth properties. The thermal ageing at the temperatures 70 and 110 °C has been applied, which closely simulates the real service conditions of tires. Dynamic mechanical analysis (DMA) and fatigue crack growth (FCG) properties were characterised for the aged samples. The loss compliance J'' has been determined as a parameter, which effectively is detecting the embrittlement or softening/hardening behaviour of the aged rubber. Finally, the FCG studies revealed that increase in aging temperature has deleterious effect on crack growth resistance as was reflected in all the aged materials.

24.1 Introduction

Tire is a key component in a car and performs multiple functions. These include carrying vehicle weight, transmitting tractive and braking forces, cushioning against terrain as well as road surface irregularities and many more. In service, tires are subjected to highly dynamic loading, varying road asperities as well as are exposed to physical processes caused due temperature fluctuation, ozone concentration, solar radiation, fluid medium, etc. These extreme requirements have a significant effect on the fatigue and failure of tire material and lead to the degradation of the

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mechanical properties of rubber matrix. The initiation of local instability in tire due to fatigue is particularly caused by the nucleation of micro-crack. Its propagation may cause dangerous consequences because of the catastrophic failure of tires leading to fatal accidents due to loss of vehicle control. Hence the understanding of the crack initiation and its propagation in rubber with respect to the applied varied physical processes as well as loading conditions is a subject of high scientific interest. The better understanding of the subject will improve the safety, higher durability and life service of the tire as well. Tires generally are made of rubber compounded with various additives and filled with different types of reinforcing fillers (primarily carbon black and silica). It is now a very common practice to use blends of two rubbers and sometimes three to achieve desired performance of the product. The combination of natural rubber (NR), butadiene rubber (BR) or styrene–butadiene rubber (SBR) finds vast application in various tire components. Therefore the present work was focused on the study of NR/SBR blend, which is commonly used as tread and sidewall component in tire.

It is well known that the mechanical properties, especially fracture behaviour depend on the structure of rubber matrix. Particularly the characteristic behaviour are based on the filler network build with the nanoparticles, size of filler cluster, interaction of filler–filler and polymer–filler bonds and the specific surface of filler under the identical compounding conditions [1, 2]. The effect of rubber reinforcement caused due to rubber modification with various fillers on crack or fatigue resistance has been studied by several researchers [1, 3, 4] and are well described. Lake and Lindley [5] investigated the effect of carbon black on the fatigue behaviour of rubbers. They found that the addition of carbon black considerably reduces the temperature dependence of fatigue behaviour of SBR, but no such effect was observed in NR. In NR, hysteresis occurs mainly from crystallisation at high strain and is relatively unaffected by changes in temperature or frequency while the hysteresis of SBR is due to internal viscosity which is continually varying. Further, the addition of carbon black into NR compounds reduces the crack growth because of crystallisation occurring in regions of high constraint due to the filler particles and thus in the vicinity of the crack tip, where high strain locally occurs [6].

Tire rubber components are very much subjected to thermal ageing due to internal heat generation because of viscoelastic losses during rolling. For instance, as reported in [7], tread and sidewall experience temperatures of about 100 and 60 °C, respectively. Several researchers [8–10] have studied the thermal ageing characteristics of rubber. Studies of ageing behaviour using dynamic-mechanical analysis can also be found in earlier publications [1, 11–13]. However, the investigation on the relationship between dynamic properties of aged rubber and fatigue crack growth is rarely done.

The main factors influencing the rubber ageing behaviour are thermal processes with the simultaneous oxidative attack. In vulcanisates, the bond energy between sulfur crosslink atoms and the polymer backbone has highest influence on resistance against thermal effects. If the rubber is subjected to thermal ageing, it is necessary to observe the influence of oxidation and diffusion of oxygen. In the case of relatively low temperature, it has been observed that for cured rubber diffusion

predominates and therefore there is slow oxidation throughout the rubber material. On the contrary, as the thermal activity increases, the rate of oxidation increases significantly than the rate of diffusion. Thus a substantial oxidation occurs on the surface at higher temperature and a brittle oxidised surface is formed. The critical stress of polymer chains in an oxidised rubber surface occurs in a lower strain compared to the non-oxidised material which indicates the degradation of mechanical properties of rubber. The mechanical property degradation is reflected in important dynamic properties like complex modulus, E^* and loss factor, tan δ. The increase or decrease of E^* with ageing indicates hardening or softening of the rubber. The variation of loss factor reflects in terms of ductility and embrittlement of the rubber surface as well as matrix.

Generally the ageing is interconnected to loss of mechanical properties over the whole lifetime of rubber matrix or tire components [14]. Most of the efforts in this field of research were mainly concentrated to the observation of crack propagation or rupture of rubber test specimens under quasi-static loading conditions with no relationship to the fatigue behaviour of rubber matrix. Huang et al. [8] studied the phenomenon of cyclic ageing on NR. They found that for NR, ageing at lower temperatures leads to a decrease in modulus, while at higher temperatures it leads to an increase in modulus. Bauer et al. [15] studied the mechanical properties of skim tire based on butadiene rubber (BR) under the oxidative ageing in the environment of 50/50 blend of N_2/O_2 at various temperatures in the range of 50–70 °C. They have observed a decrease in elongation at break and increase of modulus with time in the whole range of temperature. This degradation of mechanical behaviour is caused due to micro-crack initiation. The micro-cracks in tire component are caused due to natural physical ageing in the tire steady-state condition, while in rolling, the high stress concentration under dynamic loading condition has a predominant effect.

Thus the aim of this study is to determine the influence of thermal ageing processes in air environment and in the temperature range of 70–110 °C which closely resembles the tire application environment in the field. In the present study, fracture crack growth (FCG) behaviour of NR and SBR blends suitable for tire applications have been investigated. A new parameter based on loss compliance factor has been identified for describing the embrittlement or softening/hardening of aged rubber matrix by using of dynamic-mechanical analysis (DMA). These evaluated data have been compared with the fracture crack growth parameters observed by the use of using of a dynamic testing machine, Tear Analyzer. The correlation between loss compliance parameter and fracture crack growth data has been evaluated.

24.2 Theoretical Background

24.2.1 Dynamic-Mechanical Analysis (DMA)

The dynamic-mechanical behaviour of rubber vulcanisates at small dynamic strain depends on temperature and frequency. When a linear viscoelastic rubber material is exposed to a sinusoidal tensile stress σ, the strain ε will also alternate sinusoidally but will be out of phase, the strain lagging the stress [16]. Than the ratio (stress/strain) defines the complex tensile modulus:

$$E^* = \frac{\sigma(t)}{\varepsilon(t)} = \frac{\sigma_0}{\varepsilon_0} e^{i\delta}. \tag{24.1}$$

The complex modulus E^* can be divided into real E' (storage modulus) and imaginary E'' (loss modulus) components:

$$E^* = E' + iE'', \tag{24.2}$$

while the loss factor $\tan \delta$ is defined as follow:

$$\tan \delta = \frac{E''}{E'}. \tag{24.3}$$

Heinrich and Klüppel [16] showed that the loss factor $\tan \delta$ is also a measure of the ratio of energy lost to energy stored in a cyclic deformation. They defined the energy loss density ΔE during one cycle of strain as follow:

$$\Delta E = \pi \cdot \sigma_0^2 \cdot \frac{E''}{|E^*|^2} = \pi \cdot \sigma_0^2 \cdot J'', \tag{24.4}$$

where J'' is the loss compliance which is defined as $E''/|E^*|^2$.

Because of the relatively small value of phase shift δ, it could be numerically taken into the account the following presumption: $E'' \ll E'$. Thus (24.2) can from the numerical point of view be modified as follow:

$$i^2 = -1 \rightarrow E^* = E' + iE'' \rightarrow |E^*|^2 = E'^2 + E''^2 \approx E'^2 \rightarrow |E^*| \approx E'. \tag{24.5}$$

The loss compliance J'' can be substituted due to using of (24.5) and thus it can be written:

$$J'' = \frac{E''}{|E^*|^2} = \frac{E''}{|E^*| \cdot |E^*|} \approx \frac{E''}{E'} \cdot \frac{1}{|E^*|} \rightarrow J'' \approx \frac{\tan \delta}{|E^*|}. \tag{24.6}$$

The loss compliance, J'' which is a function of tan δ and E^*, can be used to represent the dependence of dynamic properties of rubber with respect to influence of ageing temperatures, cure variation and antioxidants. The variation of J'' over a wide temperature range can detect the embrittlement or softening/hardening of rubber materials.

24.2.2 Fracture Crack Growth (FCG)

An important quantity for fracture mechanical investigations is the tearing energy T, i.e. the energy released per unit area of crack surface growth. It was first introduced by Griffith [17] for metallic materials and later Rivlin and Thomas [18] formulated the tearing energy for elastomers. It proposes that the strain energy release rate is the controlling parameter for crack growth and it is mathematically defined as

$$T = -(\partial W / \partial A), \tag{24.7}$$

where T is the tearing energy, W is the elastic strain energy, A is the interfacial area of crack and partial derivative denotes that no external work is done on the system.

The commonly used FCG specimens are single-edge-notched tension (SENT) and pure-shear (PS) test specimens. The tearing energy for these specimens can be computed using separate mathematical expressions. For PS test specimen geometry, which is used in this study, the tearing energy T is considered by Rivlin and Thomas [18] to be independent of the crack length:

$$T = w \cdot L_0, \tag{24.8}$$

where w is the strain energy density stored in the unnotched test specimen and L_0 is the sample length at not-strained state.

Because of the dynamic fatigue loading conditions which is applied on tire tread, Gent et al. [19] determined experimentally the crack growth rate da/dn in dependence on the tearing energy T for rubber materials. Whereas the most important state for the dynamic fracture behaviour is the stable crack propagation, which is defined as follows:

$$\frac{da}{dn} = B \cdot \Delta T^m, \tag{24.9}$$

where B and m are material constants.

24.3 Experimental Details

24.3.1 Material Preparation

The detailed compositions of the blends studied are given in Table 24.1. The rubber compounds were prepared using a laboratory Banbury mixture of 1.5 L capacity in three stages. In the first stage, rubbers and all chemicals except curatives were mixed at 60 rpm for 4 min at 140 °C. The mixed compound was then re-milled at 30 rpm for 3 min at about 120 °C. The final batch containing curatives was mixed at 30 rpm for 3 min at about 100 °C. The rheometric properties were measured using a moving die rheometer (Monsanto MDR 2000E) at 141 °C for 1 h. The test specimens used for all the measurements were prepared by curing rubber compounds for 60 min at 141 °C in a compression mould.

Table 24.1 Composition of analysed rubber materials

Ingredients	A1 (phr)	B4 (phr)	B5 (phr)
Natural rubber	100	60	0
Styrene–butadiene rubber	0	40	100
Carbon black[a]	50	50	50
Zinc oxide	2.50	2.50	2.50
Stearic acid	2.50	2.50	2.50
Antioxidant[b]	2.25	2.25	2.25
Sulfur	2	2	2
Accelerator[c]	0.50	0.50	0.50

[a]Carbon black type was N339
[b]N-(1,3-dimethylbutyl), N'-phenyl-p-phenylenediamine (6 PPD)
[c]N-oxydiethylene, 2-benzothiazole sulfenamide
phr—parts per hundred rubber

24.3.2 Ageing

To evaluate the effects of ageing on the dynamic-mechanical properties as well as fatigue crack growth of rubber matrix, the samples were aged for 72 h at varied temperatures of 30, 70, and 110 °C under aerobic conditions in a thermal chamber. The test specimens were placed on a thin aluminium sheet, and therefore the opposite surfaces of the test specimens were intentionally covered by identical aluminium sheets. Thus identical ageing conditions were accomplished for all of the test specimens. The test specimens are denoted according to the ageing temperature and provided in Table 24.2.

Table 24.2 Test specimen denotation according to the ageing temperature

Ageing temperature (°C)	Rubber compound		
	A1	B4	B5
30	A1-30T	B4-30T	B5-30T
70	A1-70T	B4-70T	B5-70T
110	A1-110T	B4-110T	B5-110T

24.3.3 DMA

The complex modulus E^* and loss factor tan δ in the temperature range 30–200 °C were measured using a dynamic-mechanical analyser (TMA/SDTA840, Mettler-Toledo) in tensile mode at 0.33% strain and 1 Hz frequency. The heating temperature gradient was set on 2 K/min. The test specimens with the goemetry $2 \times 10 \times 50$ mm^3 (thickness × width × length) based on all of rubber compounds aged in accordance with the Table 24.2 analysed.

24.3.4 FCG

FCG measurements were carried out using a Tear Analyzer (Coesfeld GmbH) under sinusoidal loading conditions. The details of testing equipment used are given in previous publications by the authors [20–22]. In this study, two double-notched and one unnotched PS test specimens were simultaneously analysed, whereas the unnotched test specimen was used for determination of tearing energy [18]. The standard PS test specimen with the geometry ratio L_0 (length)/Q (width) = 1:8 was used [20]. Two of the PS test specimen were notched with notch length $a_0 = 21$ mm on both sides of test specimen. The value a_0 was calculated from the definition of minimal notch length a_{0min} in dependence on geometry ratio L_0/Q [20].

Table 24.3 lists the detailed test conditions used in the FCG investigation. The loading frequency was set at 10 Hz. The strain was varied between 13 and 25%. The influence of thermal ageing on FCG was observed for all of the mentioned rubber materials aged at 30 and 70 °C. The temperature of testing conditions was 30 °C.

Table 24.3 Test condition details

Denotation	Type of loading condition	Frequency (Hz)	Strain (%)	Ageing temperature (°C)	Ageing duration (h)	Testing temperature (°C)
x-30T	Sinusoidal	10	13–25	30	72	30
x-30T-70T				30		70
x-70T				70		30

x—denotes the varied type of rubber material

Additionally, FCG measurement of B4 compound was also done at 70 °C to see the influence of test temperature which directly affected the surface due to fatigue condition.

24.4 Results and Discussion

24.4.1 DMA

The variation in E^* (complex modulus) and tan δ (loss factor) with temperature of ageing can be seen for the rubber material A1 based on pure NR in Fig. 24.1 (left). It has been observed that with an increase of ageing temperature the complex modulus E^* decreases over the complete range of temperature. It can also be observed that there is a convergence of E^* curves of all ageing conditions (A1-30T, A1-70T and A1-110T) near to 150 °C which could be purely attributed to thermal degradation at this high temperature. However, in the case of tan δ variation, it shows an increasing trend up to a certain temperature, passing through a maxima and then reflects a decreasing trend for all ageing conditions. It is interesting note that tan δ of samples aged at 70 °C is lower than those aged at 30 °C, indicating softening. Whereas, for 110 °C, it reported higher value than 30 °C which implies that material becomes more brittle.

The variation of loss compliances J'' of aged materials with temperature is shown in Fig. 24.1 (right). It is observed that J'' increases with increase in ageing temperature which clearly demonstrates softening of the material. It is also observed that there is a transition point (temperature) above which softening occurs and appearance of this point is shifted to lower temperature as the ageing temperature increases. This transition point can be termed as softening point.

From Fig. 24.2 (left), it was seen that the complex modulus E^* and loss factor tan δ for the material B5 based on pure SBR exhibited different behaviour in
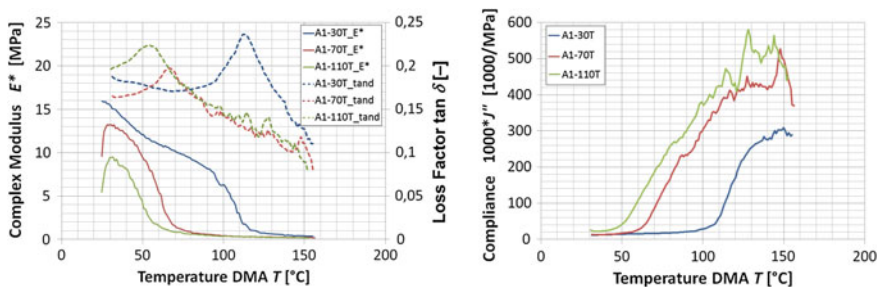

Fig. 24.1 Dynamic-mechanical properties of rubber based on A1 compound in dependence on varied ageing temperature, where the complex modulus E^* and loss factor tan δ as a function of temperature are visualised in the *left diagram* and the loss compliance J'' as a function of temperature are shown in the *right diagram*
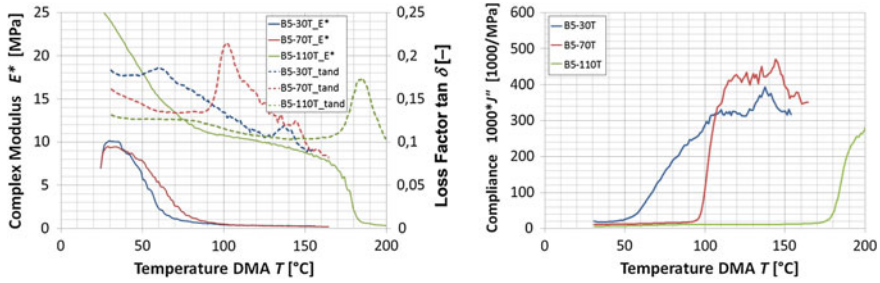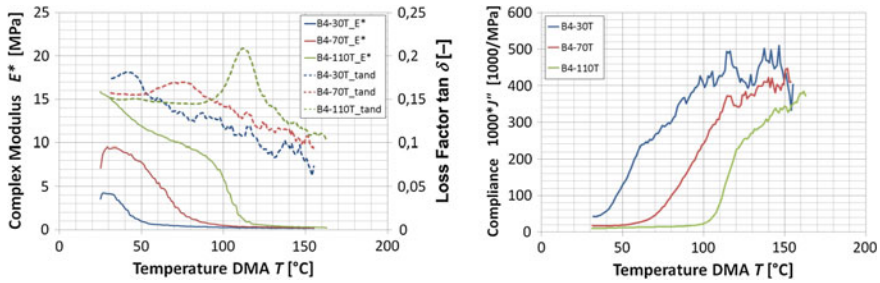
Fig. 24.2 Dynamic-mechanical properties of rubber based on B5 compound in dependence on varied ageing temperature, where the complex modulus E^* and loss factor tan δ as a function of temperature are visualised in the *left diagram* and the loss compliance J'' as a function of temperature are shown in the *right diagram*

Fig. 24.3 Dynamic-mechanical properties of rubber based on B4 compound in dependence on varied ageing temperature, where the complex modulus E^* and loss factor tan δ as a function of temperature are visualised in the *left diagram* and the loss compliance J'' as a function of temperature are shown in the *right diagram*

comparison to the material A1. The complex modulus E^* behaves identically for the ageing temperature 30 as well as 70 °C, whereas E^* significantly increases at 110 °C ageing condition. Thus the material become harder. The functions tan δ in dependence on temperature shown in the Fig. 24.2 (right) demonstrate, that the increase of ageing temperature induces an embrittlement of rubber material B5 in the range up to the 90 °C of temperature, where the behaviours differs from their previous state.

The diagram of loss compliances J'' in dependence on thermal ageing process shown in Fig. 24.2 (right). It demonstrates the embrittlement of the rubber material B5 with increase in ageing temperature. On the contrary to the observation made for A1 material, the softening point for B5 material shifted towards higher temperature with increase in ageing temperature.

Data obtained from DMA over the given temperature for the material B4 based on NR/SBR blend in the dependence on ageing temperature demonstrate the total inverse behaviour in comparison to the material A1 [see Fig. 24.3 (left)]. The

complex modulus $E*$ increases in accordance with the rise of ageing temperature. Thus the rubber blend comes through the hardening phase with respect to the increased ageing temperature. The function tan δ in dependence on temperature has demonstrated the more or less identical values over the complete analysed temperature range.

The diagram of loss compliances J'' in dependence on thermal ageing process shown in the Fig. 24.3 (right) demonstrates the embrittlement of the rubber material B4 in dependence on increased ageing temperature, whereas the increase of ageing temperature causes the higher point of temperature, where the softening of rubber material starts as observed in the case B5 material. The similarity in dynamic properties of B4 and B5 material can be attributed to the presence of SBR in these rubber compounds.

24.4.2   FCG

Figure 24.4 represents the double logarithmic plot of tearing energy versus crack growth rate (da/dn) of the materials in dependence of thermal oxidative ageing. The figure is divided into 3 separate diagrams according to the analysed rubber material, where (a) represent rubber material A1, (b) shows the results for rubber material B5 and in the diagram (c) the results are visualised for the rubber material B4. These plots clearly show that crack growth behaviour is entirely dependent on tearing energy and Power law dependency is observed. FCG parameters, B and m for all the materials under investigation are reported in Table 24.4.

In accordance with the general observation for rubber materials aged with low thermal influence (30 °C), it was found that rubber A1 based on pure NR has a higher resistance against fatigue crack growth rate in comparison to the rubber material B5 based on pure SBR. It is evident from the higher crack growth exponent, m for B5 material compared to A1 as reported in Table 24.4. It was found that the crack growth exponent m in the case of rubber blend B4 (60–40 NR/SBR) slightly increases in comparison to the A1 and B5 compound which are made of pure NR and SBR respectively. It was also observed that the FCG dependence trend is shifted to the range of higher tearing energies. Thus the material B4 based on NR/SBR compound can be denoted as a material with improved resistance against crack initiation but with reduced resistance against crack propagation in comparison to the basis rubber materials.

It is clearly shown that the increased thermal ageing condition has a significant influence on the crack growth behaviour. For all the rubber materials under investigation, the higher crack growth rate has been observed in test specimens aged at 70 °C in comparison to the rubber materials aged at low temperature of 30 °C. From Table 24.4 it is visible that identical crack growth exponent m was exhibited by both rubber materials A1 and B5 aged under varied thermal condition. However, the rubber blend B4 showed a slightly lower crack growth exponent m in dependence on increased ageing temperature, which is more or less identical with
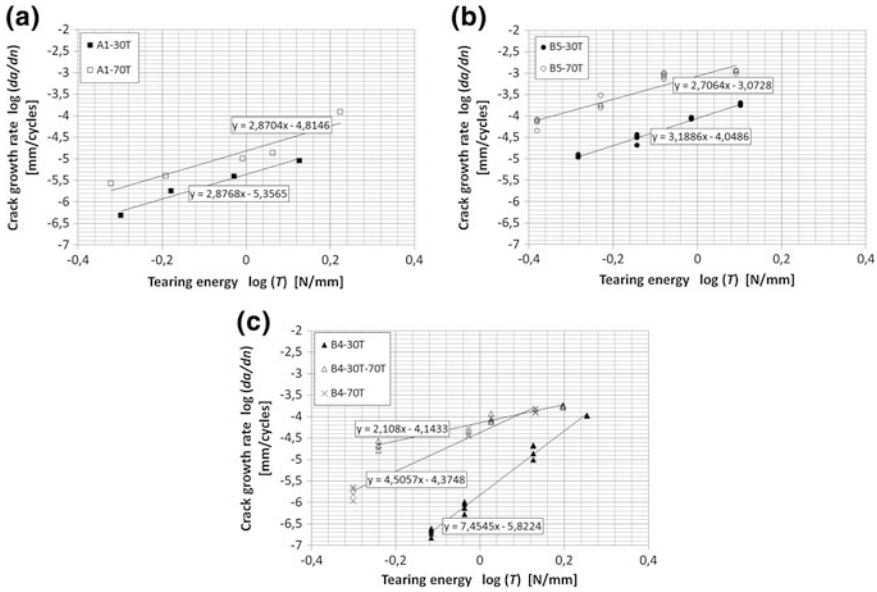
Fig. 24.4 Variation of crack growth rate with tearing energy (log-log plot) with respect to the different rubber material and the ageing temperature, where: diagram describing the FCG behaviour of rubber A1 (**a**), diagram describing the FCG behaviour of rubber B5 (**b**) and diagram describing the FCG behaviour of rubber B4 (**c**)

Table 24.4 The list of evaluated parameters of material constant B and m, which define the stable crack growth rate given by (24.9)

Parameters	Type of rubber material used for FCG analysis						
	A1-30T	A1-70T	B4-30T	B4-30-70T	B4-70T	B5-30T	B5-70T
B	−5.36	−4.81	−5.82	−4.14	−4.37	−4.05	−3.07
m	2.88	2.87	7.45	2.11	4.50	3.19	2.71

the crack growth exponent m observed in materials A1 and B5. The next observation shows that the values of tearing energies at given strain decreases in dependence on increased thermal ageing condition.

Finally the fracture investigation of rubber material B4 based on blend NR/SBR shows the significant influence of direct thermal influence of the rubber test specimens during the FCG analysis. However the rubber material tested at higher temperature (70 °C) shows a significantly lower crack growth exponent m (see Table 24.4). Thus it can be concluded, that the material B4 based on NR/SBR compound in dependence on direct thermal effect during the analysis has a lower resistance against crack initiation but behaves more or less stable in fatigue crack propagation process.

Generally it can be concluded that the point of temperature, which defines the start of rubber softening, is found to be around 50 °C or higher. Therefore the ageing process which could firstly be taken into account is in the range over the mentioned temperature, which generally tires experiences in service.

While the rubber material A1 behaves more soft with increased ageing temperature, the increase of FCG parameters was observed. The increase of FCG parameters is caused due to high strain which is the reason for inducing of crystalisation in the vicinity of the crack tip. However, the divergence of FCG in dependence on thermal ageing for material A1 could be considered as fractional. Secondly, it can be assumed, that the slight softening of the rubber material A1 during the thermal ageing correlates with the slight change in a tearing energy for identical loading amplitudes.

The results for the material B5 shows an embrittlement of the rubber matrix with increased ageing temperature. Hence, significantly higher FCG parameters were observed. Because of the embrittlement of the rubber matrix, the micro-cracks were initiated in rubber material and thus the tearing energy rapidly decreased at given strains for the material aged with higher temperature.

Similar dominating effect of rubber matrix embrittlement and micro-crack initiation have an influence on FCG behaviour in rubber material B4 based on NR/SBR blend. Therefore, the significantly higher FCG parameters were found for the rubber material aged under the higher temperature.

24.5   Conclusion

The present work discussed the influence of thermal ageing behaviour of rubber compounds made of NR (A1), SBR (B5) and their blend of 60–40 proportion (B4) on dynamic-mechanical and fatigue crack growth properties. The samples were thermally aged in the temperature range from 70 to 110 °C which closely simulates the real service conditions of tires. DMA and FCG properties were characterised for the aged samples. It was demonstrated that the loss compliance J'' can be effectively used to detect the embrittlement or softening/hardening point of the aged rubber material. It was also observed that the softening point appears near about 50 °C and was of interest for considering the ageing temperature for studying FCG characteristics. It was observed that dynamic properties of B4 and B5 were quite different from A1, which could be attributed to the presence of SBR in the former compounds. The FCG studies revealed that increase in ageing temperature has deleterious effect on crack growth resistance as was reflected in all the aged materials.

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