

Effect of Physical and Chemical Crosslinking Structure on Fatigue Behavior of Styrene Butadiene Elastomer

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ABSTRACT: The fatigue behavior of two styrene butadiene rubbers, thermoplastic elastomer (SBS) with physical crosslinking structure and vulcanized styrene butadiene rubber (SBR) with chemical crosslinking structure, was investigated. The fatigue lives of SBS and SBR were greatly affected by the fatigue frequency, amplitude, and temperature. The fatigue lives of both SBS and SBR decreased with the increasing fatigue frequency and amplitude. However, the effect of fatigue temperature on the fatigue life of SBS was different from that of SBR. With the increasing fatigue temperature, the fatigue life of SBS first decreased and then increased, however, the fatigue life of SBR showed continuous decreasing trend. The tensile strength of SBS first increased and then decreased with the increase of fatigue time, but the tensile strength of SBR was not significantly influenced by the fatigue time. The fatigue amplitude had greater effect on the tensile strength of SBS than that of SBR. The permanent deformation of both SBS and SBR increased with temperature; however, the parameter for the former rubber was higher than that of the latter. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 2014, 131, 40917.

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INTRODUCTION

Styrene butadiene thermoplastic elastomer (SBS) is a copolymer of butadiene and styrene, which is one of the very important thermoplastic elastomer, possessing rubber and plastics characteristics.¹ SBS has a lot of advantages comparing to the traditional vulcanized rubbers, such as excellent mechanical properties, convenient processing without vulcanization, and better recyclability, thus SBS becomes one of important recyclable and sustainable polymeric materials. SBS is widely used in many products, such as tapes, seals, hoses, and footwear and the likes.² These products in usage are subjected to the action of cyclic dynamic load. There are a lot of studies on the phase behavior of SBS in the previous years,^{3–6} but further investigation is needed on the fatigue behavior of SBS. The phase morphologies of SBS change with its composition and molecular architecture.⁴ The relationship of the morphology and micro-mechanical behavior of SBS was further investigated.⁷ The external conditions such as temperature and stress could influence the self-assembled nanostructures of SBS.⁸ The phase morphology of SBS exhibits that it could deform with the stretched strain, forming different structures under the static loading.^{5,6}

The dynamic fatigue behavior of materials shows different phenomena compared to those under static loading conditions, and their fatigue behaviors are also influenced by different fatigue conditions, such as compressive or tensile stress, and uniaxial or multiaxial fatigue damage.^{9–12} Some damage mechanics and fatigue life prediction models for the vulcanized rubber and fiber-reinforced polymers are established.¹³ The structure of polymer materials has a profound influence on the tensile, thermal transition, and long-term dynamic fatigue behavior.¹⁴ Although many works have been done on the fatigue behavior of the vulcanized rubber and fiber-reinforced polymers in the previous years,^{15–20} there is lack of research on the fatigue behavior of thermoplastic elastomers. The molecular structure of thermoplastic elastomer is different from that of vulcanized rubber. It is known that vulcanized rubber has chemical crosslinking structure through chemical reactions. However, thermoplastic elastomer has physical crosslinking structure through entanglement of molecular chains. The fatigue aging behavior of thermoplastic elastomer might be different from that of the vulcanized rubber, and it is necessary to investigate the fatigue behavior of thermoplastic elastomer.

Table I. Formulation of SBR Compound

Ingredients	Composition (phr)
SBR	100
Zinc oxide	5.0
Stearic acid	1.5
Sulfur	1.5
M	1.5

The purpose of this work is to evaluate the different fatigue behavior of two styrene butadiene rubbers, SBS with physical crosslinking structure and SBR with chemical crosslinking structure. The effect of fatigue frequency, amplitude, and temperature on the fatigue lives of SBS and SBR is discussed. The fatigue time and amplitude on the tensile strength of SBS and SBR after fatigue are analyzed from the percentage retention of tensile strength, the change of molecules, and the permanent deformation.

EXPERIMENTAL

Materials

SBS. The linear molecular structure, a commercial grade (1401-1) was produced by Yanshan Petroleum Chemical of China. Its melt flow index (MFI) was 1.0 g/10 min, which was measured at 190°C under 2.16 kg loading (XNR-400). Its weight average molecular weight M_w and number average molecular weight M_n

Table II. Relationship Between the Amplitude and the Maximum Strain

Amplitude (mm)	ϵ_{\max} (%)
2	15
5	36
10	76
20	140
40	204

were 1.14×10^5 and 1.03×10^5 (as measured by GPC), and the ratio of styrene and butadiene is 40 : 60.

SBR. Commercial grade (1500) with Mooney viscosity of 53 was produced by Lanzhou Petroleum Chemical of China, Zinc oxide and stearic acid were obtained from Kelong Chemical of Chengdu, China; sulfur and mercaptobenzothiazole (M) were produced by Huangyan Zhedong rubber auxiliary agent Chemical of Zhejiang, China. The formulation of SBR compound is listed in Table I.

Samples Preparation

SBS Samples. The pellets of SBS were molten for 10 min using the compression molding equipment (XLB-D-400×400) at temperature 190°C and compression pressure was 14 MPa.

SBR Samples. Compounds were mixed in the two-roll mill at 45°C, and then the mixtures were vulcanized at 150°C and compression pressure was 14 MPa for 12 min using the compression molding. The sheets were machined to the standard dumbbell specimens (115 mm × 6 mm × 2 mm).

Characterization

Fatigue Test. The dynamic fatigue experiment was performed using a multi-function aging testing machine, and the schematic representation is indicated in Figure 1. In fatigue experiment, the fatigue amplitude, frequency, time, and temperature could be precisely controlled.

The strain ratio (R) could be defined as eq. (1):

$$R = \frac{\epsilon_{\min}}{\epsilon_{\max}}, \quad (1)$$

where R is the strain ratio, ϵ_{\min} is the minimum strain, and ϵ_{\max} is the maximum strain.

In our fatigue experiment, the specimens were subjected to the uniaxial tension fatigue. The R is zero, which means that the minimum strain is zero and the maximum strain is changed with the amplitude. The fatigue amplitudes are selected to 2, 5, 10, 20, and 40 mm, and the relationship between the amplitude and the maximum strain is indicated in Table II.

The fatigue frequency is defined as the reciprocating times per second. In our experiment, 5 and 10 Hz were chosen as the fatigue frequencies according to the experimental conditions. The fatigue temperature is referred to the fatigue test ambient temperature, which is precisely controlled by the temperature control equipment (Figure 1). In our experiment, the fatigue temperatures were -40 , 0 , 10 , 23 , 30 , 40 , and 50°C , and fatigue

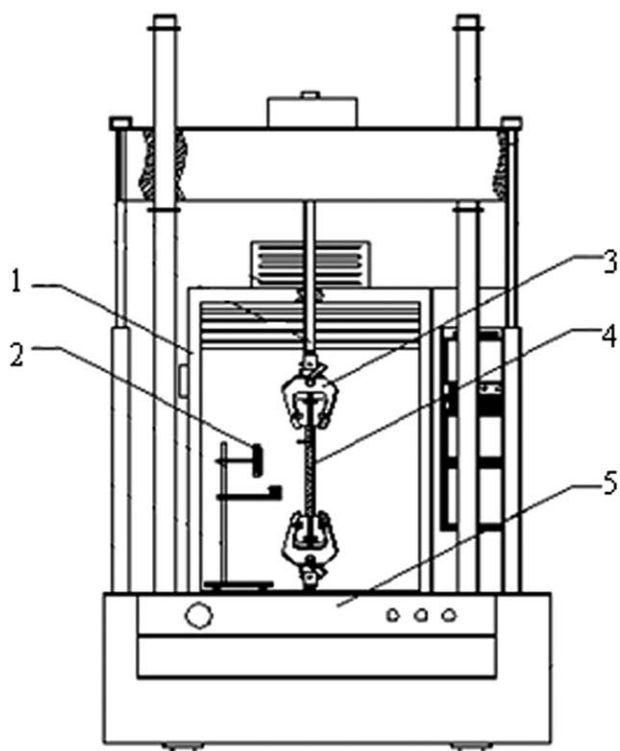


Figure 1. Schematic representation of the multi-function aging testing machine. 1. Temperature control equipment. 2. Ultraviolet light. 3. Clamp. 4. Sample. 5. Reciprocating controller.

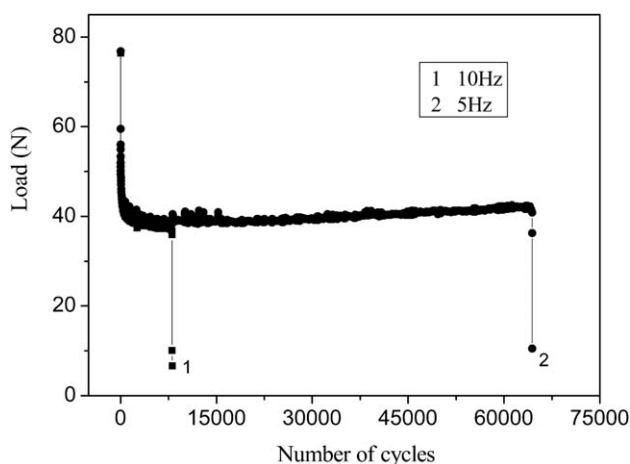


Figure 2. Effect of frequency on fatigue life of SBS (fatigue amplitude: 10 mm, temperature: 23°C).

times (40, 60, 120, 270, and 430 min) were carefully chosen to meet the requirement of our research.

Mechanical Property. The specimens were subjected to different fatigue conditions and then the tensile strength was measured by the universal testing machine (REGER-3010) using 500 mm/min testing speed, and testing temperature was 25°C. Retention of tensile strength is calculated from eq. (2):

$$\text{Retention of tensile strength} = \frac{\sigma'}{\sigma_0} \times 100\%, \quad (2)$$

where σ' is tensile strength after fatigue and σ_0 is tensile strength before fatigue.

Stress Relaxation. Stress relaxation of the samples at different temperatures was performed on a multi-function aging testing machine by a model of stress relaxation. The tensile strain was maintained at 140%, and the change of stress is recorded.

Crack Growth Rate. The crack length of sample pre-cut using a razor blade was detected using a measuring microscope (15 J).

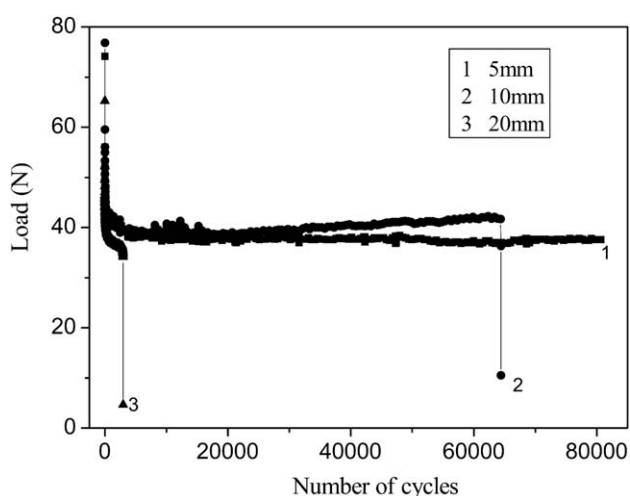


Figure 3. Effect of amplitude on fatigue life of SBS (fatigue frequency: 5 Hz, temperature: 23°C).

The change of the crack length was measured after different fatigue time and the crack growth rate was obtained.

Permanent Deformation. The specimens were stretched at the elongation percentage of 76%, 200%, and 300%, respectively, for 15 min, and then allowed them to recover for 5 min. The permanent deformation could be calculated from the following eq. (3):

$$\text{Permanent deformation} = \frac{L - L_0}{L_0} \times 100\%, \quad (3)$$

where L_0 is original length of specimen, and L is length of specimen after being stretched.

Scanning Electron Microscope. The surfaces of the specimens were measured by the scanning electron microscope (INSPECT F, FEI). The surfaces were coated with gold and then examined by scanning electron microscope (SEM).

Dynamic Mechanical Thermal Analysis. The dynamic mechanical thermal analysis was performed using a TA instrument (Q800). The samples were measured in tensile mode at a heating rate of 3°C/min and a frequency of 1 Hz over the temperature range of -120 to 120°C.

RESULTS AND DISCUSSION

Fatigue life is defined as the number of cycles to fracture a sample under dynamic cyclic loading. The effect of fatigue frequency on the fatigue curves of SBS is shown in Figure 2. The fatigue life of SBS remarkably reduced from 63,940 to 8,072 cycles when the fatigue frequency changed from 5 Hz to 10 Hz, probably because of the viscoelasticity of polymer materials.^{21,22} The energy imposed on the polymer materials might increase with the increasing fatigue frequency, and if the molecular chains could not rapidly move to keep up with the frequency, they may break. Additionally, thermal-oxidation might occur at high fatigue frequency due to the hysteresis of polymers.^{17,23} The effect of fatigue amplitude on fatigue life of SBS is indicated in Figure 3. Fatigue life of SBS decreased with the increase of fatigue amplitude. Similar results on the effect of fatigue amplitude on fatigue life of SBR were obtained in the previous studies.¹⁵ During dynamic fatigue process, the oxidation activation energy decreased with the increase of tensile strain,²⁴ which could accelerate the oxidation degradation of materials. Conversely, the strain energy density and tearing energy increased with the increase of tensile strain, resulting in the increase of crack growth rate.^{25,26}

The effect of fatigue temperature on fatigue life of SBS and SBR is displayed in Figures 4 and 5, respectively. With the increase of fatigue temperature, the fatigue life of SBS decreased at first and then the parameter increased, but the fatigue life of SBR showed a continuous decreasing trend (Figure 5).

Fatigue life is controlled by the combination of the crack initiation and propagation.²⁷ The crack initiation is determined by internal defects or the breaking of molecular chains of the sample. The capability of molecular chains to move plays an important role in the process of crack initiation. If the molecular chains cannot withstand the stress they can not quickly move, the molecular chains are weakened or broken, which forms the

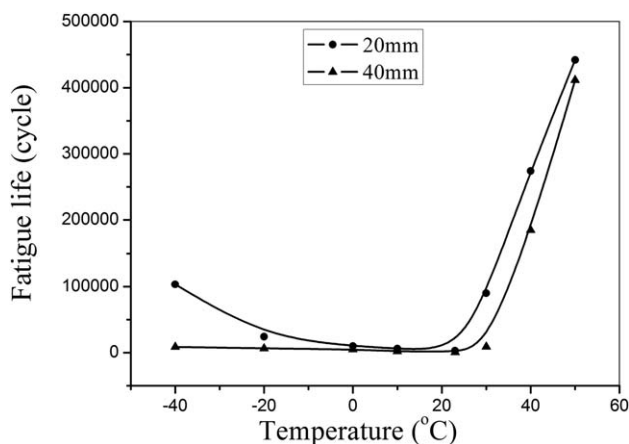


Figure 4. Effect of fatigue temperature on fatigue life of SBS (fatigue amplitude: 20 mm and 40 mm, frequency: 5 Hz).

crack nucleation.²⁸ With the aid of the stress relaxation experiment, the effect of molecular motion on fatigue behavior can be further characterized (Figures 6 and 7). The motion capability of SBS and SBR increased with the elevated temperature. Furthermore, it was found that the effect of temperature on the molecular motion capability of SBS was more significant than that of SBR. This might be related to the different structures of SBS and SBR. SBS has physical crosslinking structures, which could easily move with increasing temperature.²⁹ SBR has chemical crosslinking structures, and the disentanglement could not easily occur with the increasing temperature, unless the temperature is high enough to make the thermal-oxidation aging of SBR molecules.

The crack begins to grow and propagate after the initiation of crack. The effect of temperature on the crack propagation of SBR and SBS is shown in Figure 8. Both crack growth rate of SBS and SBR increased with the increasing temperature. Some researchers have reported similar results for the crack growth rate of vulcanized rubbers,^{30,31} probably because at higher temperature, higher thermal energy led to thermal-oxidative degradation.^{27,32} Additionally, it was found that the crack growth rate

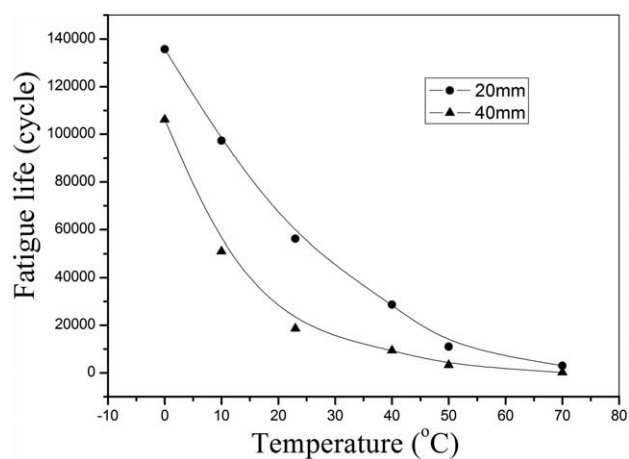


Figure 5. Effect of fatigue temperature on fatigue life of vulcanized SBR (fatigue amplitude: 40 mm, frequency: 5 Hz).

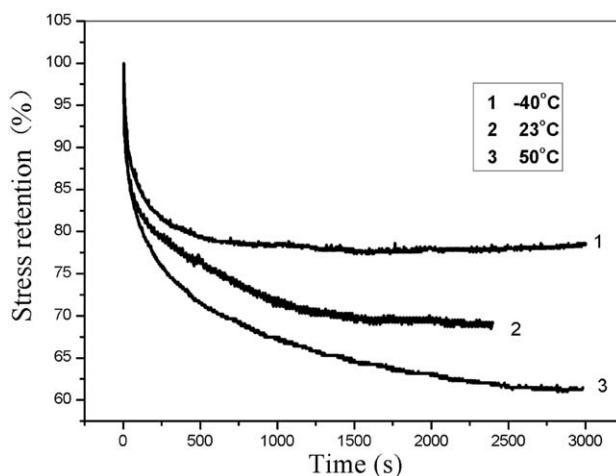


Figure 6. Stress relaxation curves of SBS at 140% strain at different temperatures.

was higher in SBR than that of the SBS. With the aid of SEM, the surfaces after fatigue under different temperatures are measured as shown in Figure 9. The cracks on the samples surfaces of SBS become clear with increment of temperature. An experimental investigation reveals that the elevated temperature environment significantly alters the microscopic fatigue damage evolution.³³

The combined effect of crack initiation and propagation could promote the fracture of sample, in another word, the fatigue life is controlled by the crack initiation and propagation. For SBS materials, the fatigue life at different temperatures could be divided into five regions (Figure 10).

Region I: at high temperature region, it is very difficult to initiate crack in the sample.

Region II: with the decreasing temperature, the motion capability of molecular chains gradually slows down, and could not keep up with external stress applied, which might result in the breaking of molecular chains. It becomes possible that a lot of

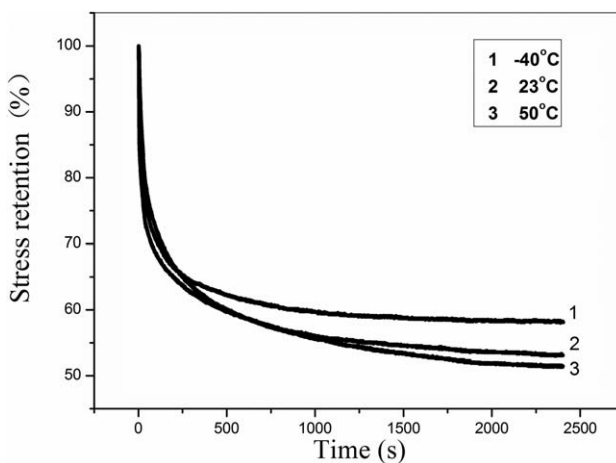


Figure 7. Stress relaxation curves of SBR at 140% strain at different temperatures.

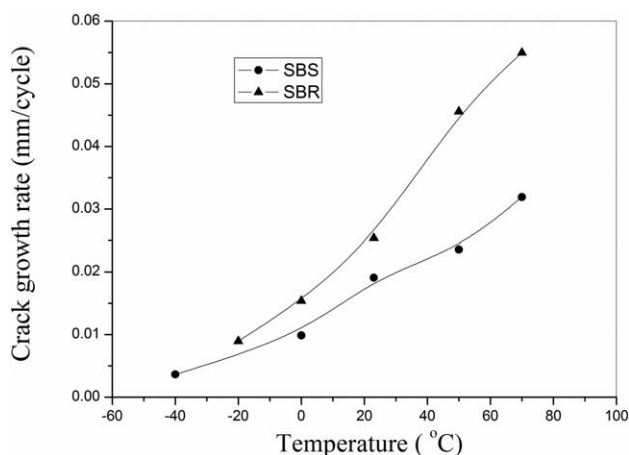


Figure 8. Crack growth rate of SBS and SBR at different fatigue temperatures.

broken molecule chains would initiate the crack, and then the crack propagation is possible.

Region III: the crack initiation and propagation easily happen, and the combined effect of the crack initiation and propagation is the strongest, leading to easy fracture of the sample. Thus, a minimum fatigue life of the sample is appearing in this period.

Region IV: as the temperature continues to decrease, it is easier for crack initiation, but the crack propagation rate is slow.

Region V: at low temperature region, although it is easiest to initiate the crack, the crack propagation is the most difficult in this period.

For SBR materials, the fatigue life of SBR at different temperatures is divided into three regions (Figure 11).

Region I: at high temperature region, it is difficult to initiate crack in the sample.

Region II: with the decrease of temperature, the motion capability of SBR molecules slows down, and the molecular movements could not keep up with external stress applied, which would result in the breaking of molecules. It becomes possible that a lot of broken molecular chains would initiate the crack, and then the crack propagation is possible easily. Therefore, the fracture rate of samples significantly reduced with the decreasing temperature.

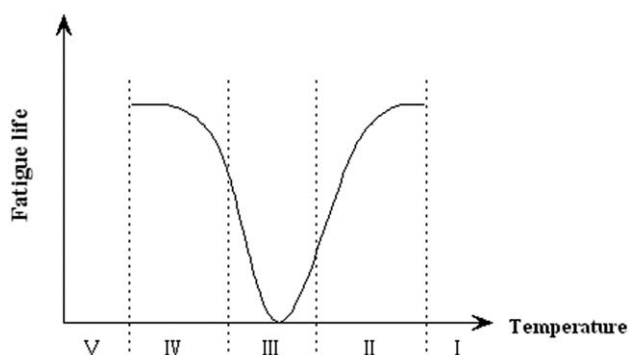


Figure 10. Fatigue life of SBS at varying temperatures.

Region III: at low temperature region, although the crack initiation is the easiest, the crack propagation is the most difficult in this period.

The fatigue lives of SBS and SBR presented the different changes with the increasing temperature, which are consistent with the results obtained in Figures 4 and 5.

The effect of fatigue time on the percentage retention of tensile strength of SBS and SBR is indicated in Figure 12. With the increasing fatigue time, both the tensile strength of SBS and SBR increased at first and then the parameter decreased. Additionally, the behavior of SBS is more significant than that of SBR. It is attributed to the different change of SBS and SBR as seen in Figures 13 and 14. For the thermoplastic SBS elastomer, the change in tensile strength is associated with the orientation of molecular chains of SBS.^{3,4} The molecular chains of SBS oriented along the tensile direction and formed the oriented structure under stress [Figure 13(b)], which would be favorable to the enhancement of tensile strength.⁶ However, with further increase of tension time, SBS molecular chains might break,^{3,4} resulting in the decrease of the tensile strength [Figure 13(c)]. For the SBR elastomer, the chemical crosslinking density changed with the fatigue time.^{17,18} However, the motion of SBR molecules might be restricted due to the existence of chemical crosslinking [Figure 14(b)], and therefore the oriented structures might not significantly form in SBR sample. Thus, the change of SBR in tensile strength was smaller than that of SBS. When the tension time is further increased, the SBR molecular chains were also broken,^{3,4} resulting in the decrease of the tensile strength [Figure 14(c)].

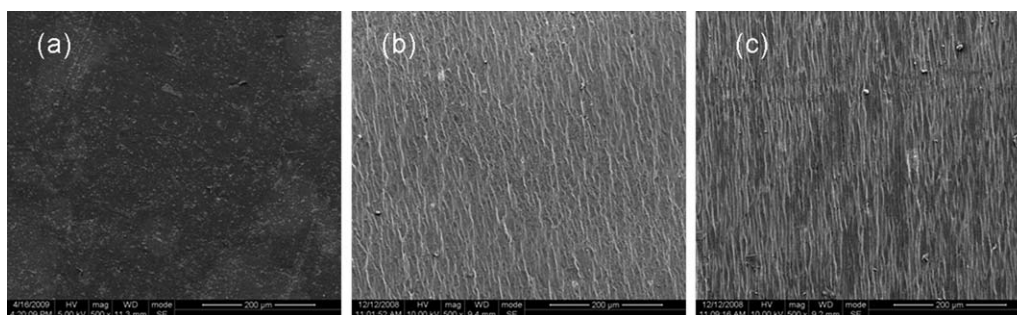


Figure 9. The surface SEM micrograph of SBS sample subjected to the same fatigue conditions (fatigue amplitude: 10 mm, frequency: 5 Hz, time: 40 min) at different temperatures: (a) -40°C , (b) 23°C , and (c) 50°C .

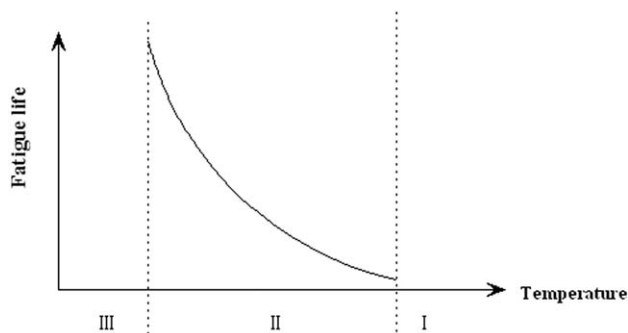


Figure 11. Fatigue life of SBR at varying temperatures.

The effect of fatigue amplitude on the tensile strength of SBR and SBS is displayed in Figure 15. The fatigue amplitude has no significant effect on the tensile strength of SBR, but the tensile strength of SBS remarkably increased with the enhancement of fatigue amplitude. The phase of SBS oriented with the tension direction, and then the oriented structure is favorable to the improvement of mechanical property.^{3,6} In addition, SBS has stronger ability to retain the oriented structure than that of vulcanized SBR.

The oriented structure of SBS was further characterized using the dynamic mechanical thermal analysis (DMTA). Glass transition (T_g) and loss factor ($\tan \sigma$) of SBS after fatigue at different temperatures are indicated Table III. It is found that the T_g and $\tan \sigma$ of polybutadiene (PB) both increased with fatigue temperature. However, there is no remarkable change in the T_g and $\tan \sigma$ of polystyrene (PS). The PB chains, as the soft segments, could provide SBS with flexibility and elasticity. The PS chains, as the hard segments, form the physical crosslinking structures, which could provide SBS with the stiffness and strength.² During deformation the lamellar morphology should give an equivalent stress distribution in both the soft and hard chains. Owing to the lower Young's modulus the applied stress localizes first in the soft phase and therefore the PB chains could orient earlier

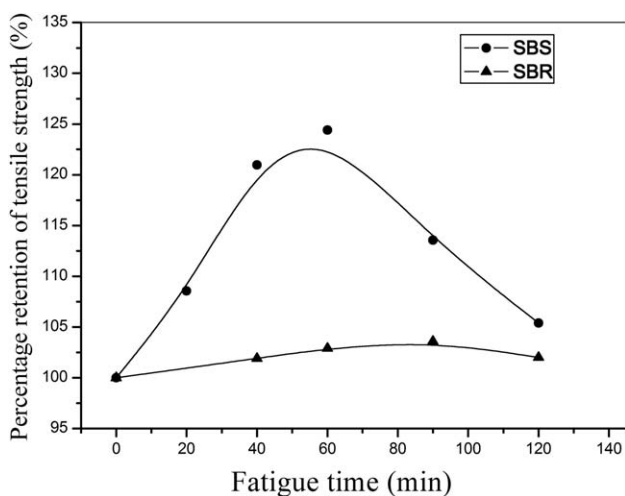


Figure 12. Effect of fatigue time on the percentage retention of tensile strength of SBS and SBR at 23°C (fatigue amplitude: 10 mm, frequency: 5 Hz).

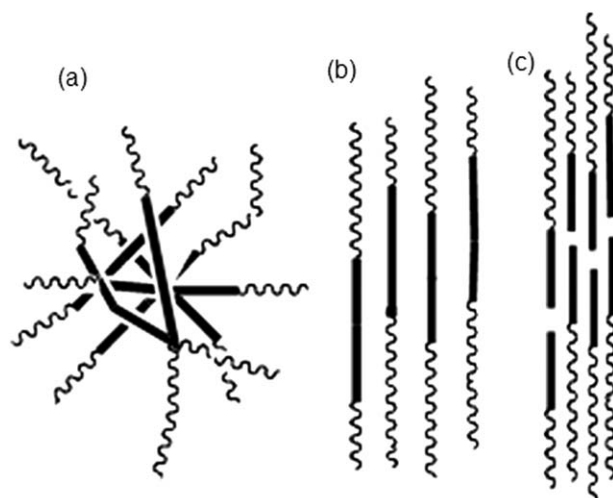


Figure 13. The change of SBS molecular chains under the stress. PB chains; PS chains. (a) Before fatigue. (b) Most of SBS molecular chains oriented along the tensile direction after fatigue. (c) SBS molecular chains further oriented along the tensile direction, even some molecular chains were broken.

and become stronger than the PS chains.⁶ However, the T_g and $\tan \sigma$ of SBR are not significantly influenced by the fatigue temperature, and the similar result of the vulcanized natural rubber was also obtained in the previous research.³⁵

When polymer materials are subjected to stress, the total deformation (ϵ) is evaluated using the following eq. (4)^{34,36}:

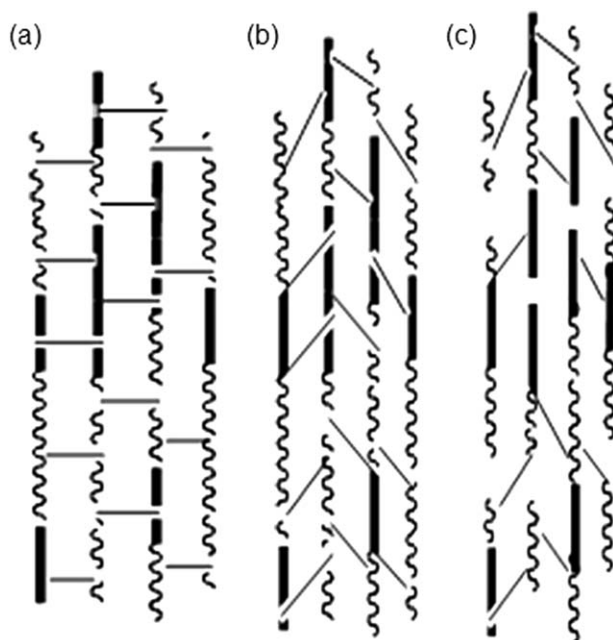


Figure 14. The change of SBR molecular chains under stress. PB chains; PS chains; Chemical crosslinking. (a) Before fatigue. (b) SBR molecular chains oriented along the tensile direction after fatigue. (c) SBR molecular chains further oriented along the tensile direction, even some molecular chains and chemical crosslinking bonds were broken.

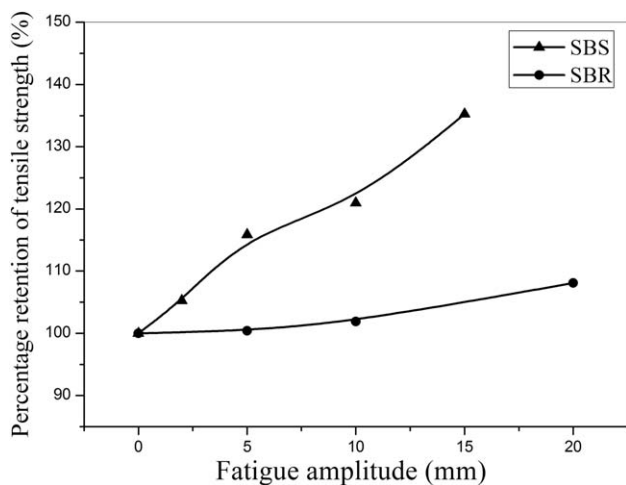


Figure 15. Effect of fatigue amplitude on the percentage retention of tensile strength of SBS and SBR at 23°C (fatigue time: 40 min, frequency: 5 Hz).

$$\varepsilon = \varepsilon_1 + \varepsilon_2 + \varepsilon_3, \quad (4)$$

where ε is the total deformation. ε_1 is the regular elastic deformation, which is small and could be immediately recovered when the external stress is removed. ε_2 is the high elastic deformation, which is larger than ε_1 , and ε_2 is related with the relaxation of molecular chains. It could be gradually recovered when the external stress is removed. ε_3 is the viscous deformation, which mainly exists in the molecules without the chemical crosslinking structure. It could not be recovered when the external stress is removed, thus it is also called irreversible deformation or permanent deformation.³⁷

Figures 16–18 indicate the permanent deformation of SBS and SBR at -40 , 23 , and 50°C . Permanent deformations of SBS and SBR increased with the increase of temperature. Furthermore, the permanent deformation of SBS is remarkably larger than that of SBR. These might be explained from the different molecular structures of SBS and SBR. SBS with the physical crosslinking structure would facilitate the motion of molecules, forming unrecoverable deformation. SBR with the chemical crosslinking structure would be more resistant in forming the unrecoverable deformation. Therefore, the permanent deformation of SBR is smaller than that of SBS. Besides, it was found that the permanent deformation of SBS and SBR both increased with temperature. The molecular motions increased with the temperature, promoting the formation of the oriented structure.

Table III. DMTA Data of SBS After Fatigue at Different Temperatures

Fatigue temperature ($^\circ\text{C}$)	PB		PS	
	T_g ($^\circ\text{C}$)	$\tan \sigma$	T_g ($^\circ\text{C}$)	$\tan \sigma$
-40	-78.55	0.27	106.20	0.97
23	-70.73	0.28	105.44	0.98
50	-68.73	0.34	105.40	0.99

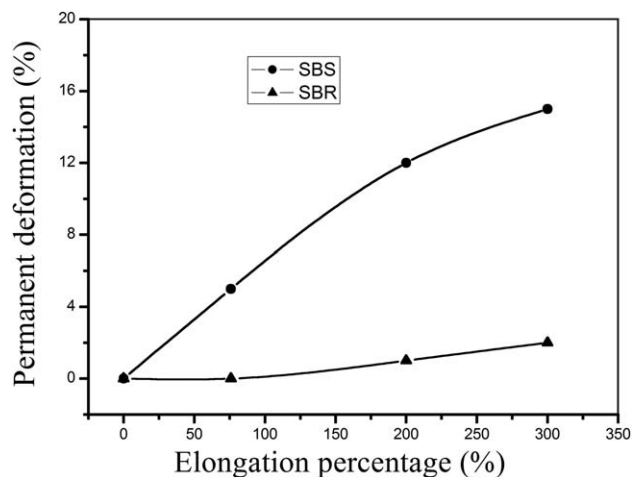


Figure 16. Permanent deformation curves of SBS and SBR at -40°C .

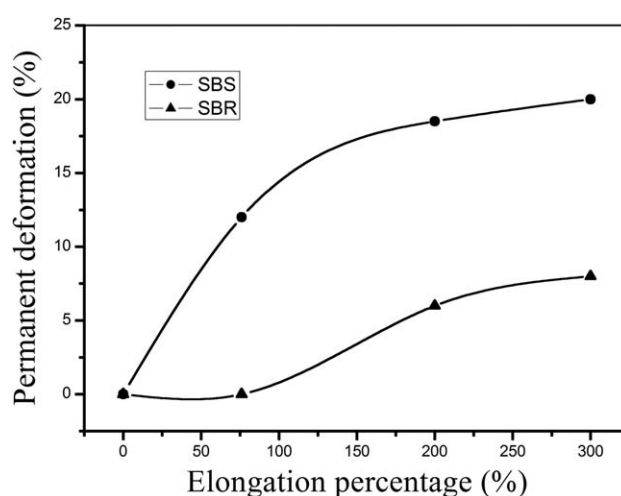


Figure 17. Permanent deformation curves of SBS and SBR at 23°C .

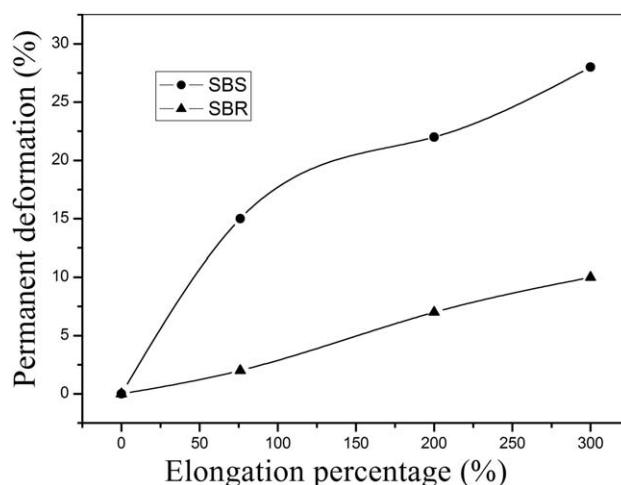


Figure 18. Permanent deformation curves of SBS and SBR at 50°C .

CONCLUSIONS

The fatigue lives of SBS and SBR were greatly affected by the fatigue frequency, amplitude, and temperature. The fatigue lives of both of SBS and SBR decreased with the increasing fatigue frequency and amplitude. However, the effect of fatigue temperature on the fatigue life of SBS was different from that of SBR. With the increasing fatigue temperature, the fatigue life of SBS decreased at first and then increased; however, the fatigue life of SBR showed continuous decreasing trend. The effect of fatigue time and amplitude on the change of tensile strengths of SBS and SBR after fatigue was researched. The tensile strength of SBS increased at first and then decreased with the increase of fatigue time, but the tensile strength of SBR was not significantly influenced by the fatigue time. The fatigue amplitude had greater effect on the tensile strength of SBS than that of SBR. The permanent deformation for both SBS and SBR increased with the increment temperature, and the parameter was higher in the former than that in the latter.

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