Structure and fracture behaviour of rubber

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Doctoral Thesis Summary

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Struktura a lomové chování pryže

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Content

Abstract

The thesis deals with fracture behaviour of rubber and methodology associated with its characterization. Understanding particular mechanisms of fracture is important in prediction of rubber compound performance under real loading conditions and increase of rubber products service life. The theoretical part summarizes published approaches used to characterize fracture behaviour of rubber. In addition, a part of theoretical background is devoted to the analysis of fatigue crack growth (FCG) and describes the effect of the structure and test conditions. The experimental part focuses on the preparation of rubber compounds based on natural rubber (NR), butadiene rubber (BR) and styrene butadiene rubber (SBR) as well as combinations of them. Subsequently, rubber test specimens are prepared for characterizing the fracture behaviour throughout their service life under cyclic and static loading. Last but not least, functional prototype of device for characterization of the fracture behaviour of stressed rubber in tension simultaneously being penetrated by sharp object is introduced.

Keywords: rubber, rubber compound, fracture mechanics, crack initiation, crack propagation, fracture, fatigue, wear, mechanical loads

Abstrakt

Předložená práce se zabývá problematikou lomového chování pryže a metodikou spojenou s jeho charakterizací. Pochopení mechanismů jednotlivých stádií lomu je důležité z hlediska predikce a zvýšení doby životnosti pryžových výrobků v reálných podmínkách. V rešeršní části práce jsou shrnuty teoretické přístupy užívané pro charakterizaci lomového chování pryže. Mimo to je pozornost věnována rozboru únavového lomu a popisu vlivu struktury a zkušebních podmínek na jeho průběh. Experimentální část je zaměřena na přípravu kaučukových směsí na bázi přírodního (NR), butadienového (BR) a styren butadienového (SBR) kaučuku, tak jako jejich kombinací. Z připravených kaučukových směsí byla vyrobena pryžová zkušební tělíska pro zkoušky charakterizující lomové vlastnosti v jednotlivých stádiích životnosti při cyklickém a statickém zatěžování. Mimo to byl představen funkční prototyp zařízení schopného charakterizovat lomové chování pryžového tělesa namáhaného tahem, do něhož současně vniká ostrý předmět.

Klíčová slova: pryž, kaučuková směs, lomová mechanika, iniciace trhliny, propagace trhliny, lom, únava, oděr, mechanické zatížení

1. INTRODUCTION

Rubber-like materials are widely used in engineering applications because of their unique properties that combine high extensibility, high strength, high energy absorption and high resistance to fatigue and good environmental resistance. [1] Thousands of products such as tires, hoses, belts, seals, bearings and bushings are manufactured using such types of rubber. Indeed, rubber is crucial for a modern industrialized economy because of its wholesale usage *e.g.* in the automotive industry. In fact, a modern passenger car contains over 600 rubber parts. [4] Rubber products are exposed to large static and time-varying strains over a long time. Long-term durability is therefore a critical issue. The mechanical fatigue and the nucleation and growth of cracks in the rubber are very important aspects influencing products' service life. For effective and economical solution of mechanical fatigue issues, engineers need to model and design the product development process in accordance to the state of the art requirements for rubber products. [5] The theoretical background of this work gives a summary of the various theoretical approaches describing fracture mechanics, especially approach based on the energy balance of the system. Experimental part deals with the description of fracture behaviour during various stages of tearing level. In order to describe fracture properties of commonly used elastomers and their blends, several fundamental mechanical methods based on the energy balance were used. Moreover, new methodology and testing prototype able to characterize minimal energy for crack growth has been introduced.

The aim of this work is a thorough description of several types of fundamental testing methods and their application. The methods are based on varied loading conditions in order to investigate complex fracture properties from crack initiation up to total rupture of basic commonly used vulcanizates, which are based on natural and synthetic elastomers. However, the main mission is to elaborate simple but very complex overview over state of the art testing methodologies and equipment leading to the predicative description of fracture behaviour of rubber products with respect to the real loading conditions occurring in service life and thus summarize a prescription for simple, smart and efficient fracture characterization in a laboratory.

2. THEORETICAL BACKGROUND

2.1 Fatigue of rubber-like materials

Mechanical fatigue in rubbery materials is demonstrated by a progressive weakening of physical properties as a result of crack growth during application of dynamic loads or deformations. [6] The process of mechanical fatigue can be divided into four stages, which characterize different phases of tear behaviour. The initial process of tearing describes a generation of high stress concentration around area of natural defects or flaws. In the second stage, the crack initiation

occurs due to formation of defects at a molecular level. In another stage, the formed crack grows due to additional load. The mechanical fatigue in a limiting case leads to failure. [7] Many factors influencing the mechanical fatigue life of rubber components are known. The four major aspects are effect of mechanical loading history, environmental effect, effect of rubber formulation, and effect due to dissipative aspects of the constitutive response of rubber. [8] Recent research into the fatigue of rubber has focused on the mechanics of fatigue failure as well as on the development of the complete fracture process. [9] The primary significance has description of local stress concentrations around structural flaws. Hence, the fracture mechanics approach has been evolved. [6]

2.1.1 Crack Initiation

Crack initiation is a localized deformation process that is necessary for crack propagation. One of the necessary premises of fracture mechanics is the presence of natural defects or flaws, which form highly localized stress concentrations that initiate failure at fatigue loading. [10] Under sufficient magnification, every solid body contains heterogeneities of composition or structure, each of which may serve as the precursor to a crack. [2] For natural rubber, the size of flaws which can cause initiation of crack is estimated to be about 25 μm. Another precursor to mechanical fatigue is a crack initiated by ozone at surface areas where tensile stress is present. [6] Two parameters are widely used for a detailed prediction of crack nucleation in rubber i.e. maximum principal strain or stretch, and strain energy density. The strain analysis is a common investigative tool because it can be directly determined from an immediate tensile test measurements of displacements.

2.2 Fracture mechanics

The fracture mechanics is a branch of science that deals with description of material behaviour containing a notch. It includes methods for calculating mechanical characteristics such as crack driving force and resistance to fracture. Fracture mechanics is an important tool for mechanical performance improvement of materials and components that defines relationship between stress and strain by using the theories of elasticity and plasticity of materials. Indeed, it is a quantitative analysis for evaluating structural strength depending on applied stress, crack length, and specimen geometry. [3]

Fundamentals of fracture mechanics were laid by an English aeronautical engineer, Griffith, during World War I. In 1920 Griffith quantitatively connected the strength and crack size for brittle materials. Griffith's theory is based on energy-balance approach that describes the slow propagation of a crack as a conversion of elastic energy stored in the bulk to surface energy. Griffith postulated that the strain energy of the material decreases at constant deformation due to creation of new fracture surfaces. [9, 11, 12]

$$U = U_0 - U_a + U_\gamma \tag{1}$$

Energy balance is given by Equation 1 where U represents potential energy of the system, U_0 is potential energy of the system before introducing the crack, U_a (strain energy) and U_γ (surface energy) decreases in potential energy due to deformation associated with introduction of the crack and increase in surface energy due to the newly created crack surfaces, respectively. [13]

Griffith's original work dealt with very brittle materials, specifically glass rods. Therefore, it cannot be applied to describe the behaviour of ductile materials e.g. metals. Irwin and Orowan independently proposed extensions of the Griffith's theory to make it usable for ductile materials almost 30 years after Griffith. They pointed out that for engineering materials the strain energy is not absorbed only to create new surfaces but the energy is also dissipated due to plastic flow in the material close to the crack tip. They suggested that crack growth occurs when enough strain energy for both processes required to crack propagation in ductile materials is released. [8, 14, 15] However, fracture behaviour is characterized by relationship between crack driving force and crack resistance.

2.2.1 Crack driving force

Crack driving force G is proportional to the energy available for fracture, and is equal to the mechanical work necessary to increase crack area per unit and can be written in following form,

$$G = -\frac{dU_a}{dA} \tag{2}$$

where U_a is strain energy and A is area of newly created crack surface.

2.2.2 Crack growth resistance

The crack resistance R is a material constant which characterizes the amount of energy necessary to allow propagation. The degree of resistance against crack growth is given by value of material surface energy U_γ.

$$R = \frac{dU_\gamma}{dA} \tag{3}$$

Generally, the crack driving force increases with crack length so that after initiation, unless there is a similar increase in resistance, there is a positive difference between driving force and resistance and more energy is available for fracture than is required simply to create the new fracture surfaces. [16] There are two ways, depending on the ratio between value of driving force and crack resistance, how crack can spread after exceeding the critical value.

Steady state crack propagation

$$\frac{dG}{dA} \leq \frac{dR}{dA} \tag{4}$$

Unsteady state crack propagation

$$\frac{dG}{dA} > \frac{dR}{dA} \tag{5}$$

2.3   The fracture mechanics of rubber

The Griffith's concept for a crack growth inside brittle materials does not represent exactly the fracture mechanics of rubber due to its viscoelastic behaviour. The stored elastic energy in the bulk serves as a driving force G of crack spreading in rubber and its amount required to growth a fracture surface by one unit area characterizes mechanical strength of elastomers. Viscoelastic zone in vicinity of crack tip leads to the fact that amount of G does not depend only on energy necessary to break bonds at crack tip, but also on energy dissipated in vicinity of crack tip during crack propagation.

According to this relation, it is obvious that the effects of crack velocity and temperature on crack propagation in rubber materials are due to viscoelastic processes in the bulk. [18]
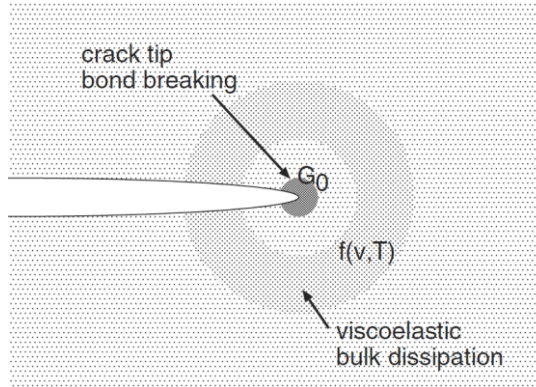

Figure 1. The crack propagation energy G is a product of a term G_0 derived from the bond breaking at the crack tip, and a term f (v, T) derived from the bulk viscoelastic energy dissipation in front of the tip. [17]

Stresses induced at the tip of a crack in viscoelastic solid cause large local deformations which lead to dissipation of energy. The innermost region at the crack tip zone is associated with highly nonlinear processes like cavity formation and bond breaking. This region is in glassy state for high frequencies $\omega = v / r$ (high crack velocities v and short distance from crack tip r) and is described phenomenologically via the term $G_0 = 2\gamma_0$ where for rubber-like materials γ_0 is much larger than the surface energy γ. The elastic modulus of glassy region is $E_{(\omega)} \approx E_\infty$. The second contribution comes from the viscoelastic dissipation in the polymer in the linear viscoelastic region in front of the tip which is given by factor

f(v,T) and it is referred to as transition region which exhibits the highest values of loss factor. For a fast moving crack this may be very far away from the crack tip. The last region which is represented by rubbery behaviour where $E_{(\omega)} \approx E_0$ is indicated for large distance *r*. When a crack propagates in a viscoelastic material, heat is produced in the vicinity of the crack tip, which will increase the temperature close to the crack tip. For fast crack propagation flash temperature effect leading to increase of temperature close to the crack tip is visible. This effect is caused due to a movement of a crack tip from the glassy region which contributes very little to the total energy dissipation towards the transition region where viscoelastic energy dissipation occurs. [17]

The crack will propagate when energy required to create the new surface plus the energy required by the viscoelastic dissipation processes near the crack tip is supplied to the system. The strain energy release rate criterion is still valid for rubber materials, when the energy dissipation is confined to a small zone at the crack tip. [6, 18] Rivlin and Thomas were first to apply Griffith's theory to the tearing of rubber successfully. [19] Rivlin and Thomas developed a criterion for the tearing of a rubber vulcanizates, which is analogous to the Griffith's characteristic energy, describing the amount of energy released per unit area of crack surface growth, i.e. the tearing energy *T* that is defined by Equation 10 [20, 21]

$$T = -\left(\frac{dU}{dA}\right) \quad (6)$$

where *U* is the elastic energy of specimen and *A* is the area of the created fracture surface.

The energy necessary to grow the crack is supplied either from the strain energy in the deformed rubber, or as energy applied by mechanical loading or both. It is evident from the experimental results that value of the tearing energy *T* is independent on the geometry and dimensions of the specimen and can be considered as a material property. [20]

Because of the nonlinear behaviour and large deformation, it is difficult to determine the tear energy using applied forces or deformations. However, by suitably choosing the specimen geometry the tearing energy can be determined without detailed knowledge of strain energy density. Thomas showed that the tearing energy is given by Equation 11,

$$T = W_b \cdot d_n \quad (7)$$

where W_b and d_n are work required to break and diameter of notch tip, respectively. [22]

3. AIMS OF THE WORK

The aim of the work is contribute to the field of characterization of fracture behaviour of rubber. Rubber compounds contain a wide range of ingredients which influences their final properties. Moreover, at real conditions of usage, rubber products are mechanically loaded in various ways that affect their fracture behaviour. For a fundamental prediction of lifetime of rubber products is necessary to understand interrelations between structure and mechanical loading and their influence on fracture behaviour.

Thus, the main goal of the study is experimental determination of the interrelations between structure and loading condition on fracture behaviour. Natural rubber and two wide used synthetic elastomers were used for determination of fracture behaviour.

The aims of this work have been summarized in the following major points:

- Preparation of carbon black filled rubber compounds based on NR, BR, SBR and their combination.

- Determination of mechanical properties using basic testing methods.

- Creation of overview of basic test methods characterizing the fracture behaviour of rubber in quasi-static, dynamic or cyclic loading, supplemented by results.

- Introduction of functional prototype of a device which is able to characterize the fracture behaviour of rubber, supplemented by results.

4. EXPERIMENTAL PART

During service life, rubber products are rarely visibly loaded in tension. However, from the mechanical point of view the tension is one of the main stress vectors of a loaded element. Figure 2 schematically shows bending of beam where the tension is clearly illustrated. Therefore, for the fundamental understanding of mechanical as well as fracture behaviour of material it is important to perform experimental investigation in tension.
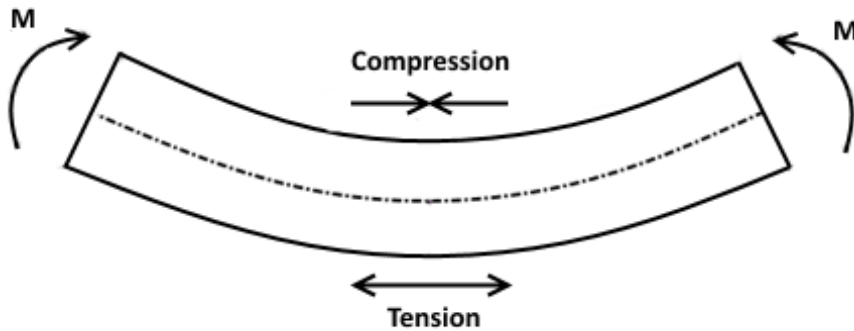

Figure 2. Stress distribution of bending beam.

In Figure 3 the diagram of cyclic loading in tension of commonly used sinusoidal waveform shape is illustrated.
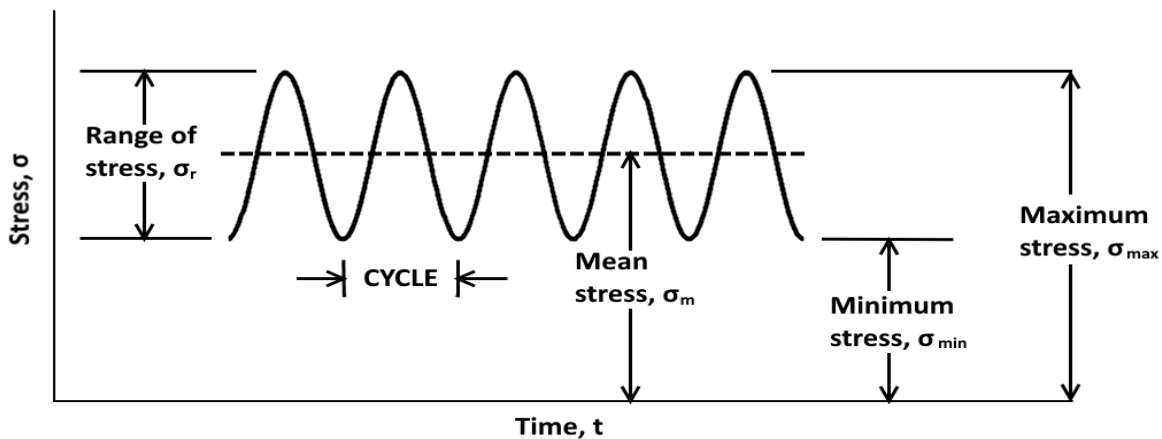
Figure 3. Schematic diagram of symmetric cyclic loading.

In detail, loading force in one cycle may have a different course over time, either it can be constant in time or generate quasi static or dynamic grow over time as is illustrated in Figure 4.
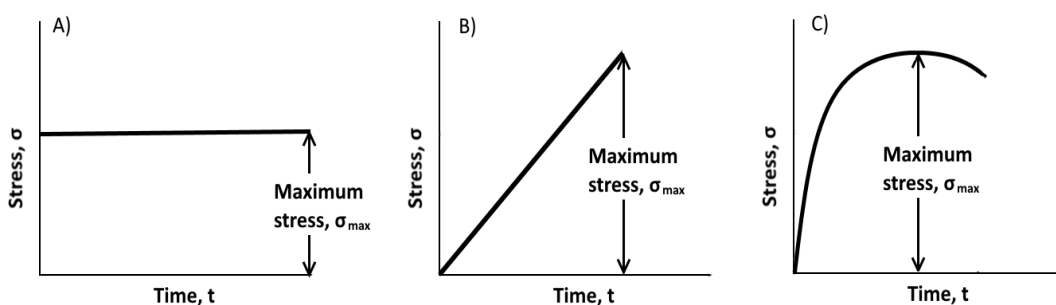
Figure 4. Schematic diagrams of the various loading conditions; A) constant force loading, B) quasi static loading, C) dynamic loading.

For this thesis, two experimental sets each including two compounds with varying concentration of BR and SBR elastomers were prepared. The composition of compounds is shown in Table 1.

Table 1. Composition of characterized rubber compounds.

	NR	**NR/BR**	**BR**	**NR/SBR**	**SBR**
Natural Rubber	100,0	50	-	50	-
Butadiene Rubber	-	50	100	-	-
Styrene Butadiene Rubber	-	-	-	50	100
Carbon Black	50,0	50,0	50,0	50,0	50,0
Zinc Oxide	3,0	3,0	3,0	3,0	3,0
Stearic Acid	1,0	1,0	1,0	1,0	1,0
Antioxidant	1,5	1,5	1,5	1,5	1,5
Accelerator	2,5	2,5	2,5	2,5	2,5
Sulfur	1,7	1,7	1,7	1,7	1,7

All rubber additives were compounded with raw elastomers in two steps. Internal mixer Banbury mixer (Pomini Farrel) was used for the first step and whole process was completed by adding of curing system consisting of an accelerator and sulphur on two roll mill Farrel G-2603 150 x 330 mm. Detailed compounding procedure is given by Table 2.

Table 2. Compounding procedure.

Order	**Ingredients**	**Time [min]**	**Equipment**	**Temperature [°C]**
1	Elastomer	2	Internal mixer	80
2	Stearic acid + Zinc oxide + IPPD	1		
3	Carbon black	5		
4	CBS	1	Two roll mill	60
5	Sulfur	5		

After mixing, conditioning of the mixed compounds was applied for 24 hours. Vulcanisation characteristics were determined using a rotorless curemeter MDR 3000 Basic (MonTech, Germany) in accordance with ASTM D 5289 at 160 °C. For vulcanization of test specimens optimum cure time t90 was used.

4.1 Measurement of mechanical properties of vulcanizates

In order to determine basic mechanical properties of tested rubber materials, measurements of hardness, quasi-static tensile test and dynamic mechanical analysis were performed.

4.1.1 Hardness Shore A

For fundamental characterization of rubber hardness properties Shore durometer was used. For rubber vulcanizates Shore A method according to ISO 7619 is the most appropriate. The principle of determination Shore hardness is based on measurement of penetration resistance of pin into the material.
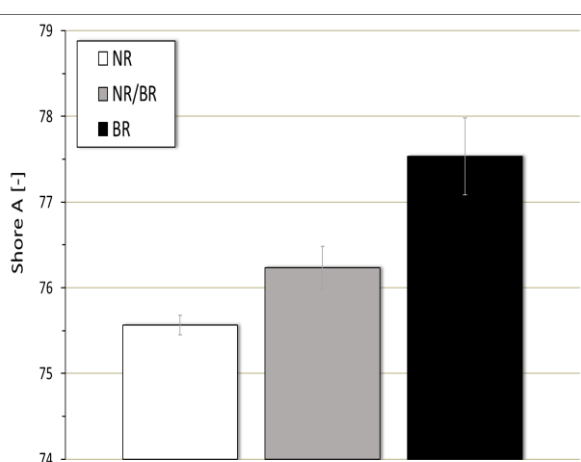
Figure 5. Results of Shore A hardness of NR, NR/BR and BR.
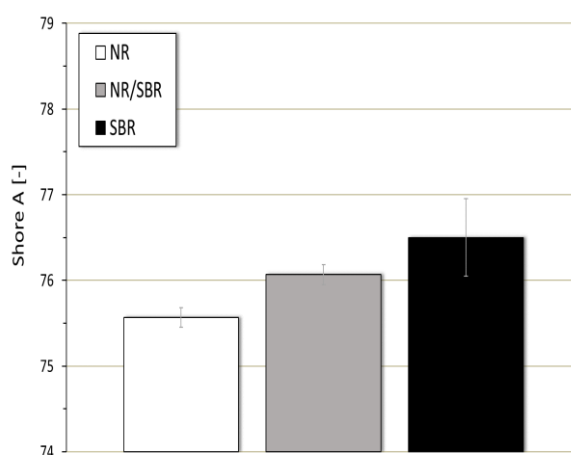
Figure 5. Results of Shore A hardness of NR, NR/SBR and SBR.

For both sets of materials, it is visible in Figures 5 and 6 that addition of BR or SBR increased value of Shore A hardness which is characteristic for rising level of material elasticity.

4.1.2 Tensile test

Displacement controlled tensile test in accordance with ISO 37 using dumbbell specimen type B was used as another method for characterization of mechanical properties. The tensile strength, elongation and modulus were plotted as functions of the changes of mechanical properties in dependence on percentage representation of each elastomer in rubber compound. From results, which are represented in Figures 7 and 8 it is visible that strength and elongation in tension reached the highest value for reference material prepared only based on NR.
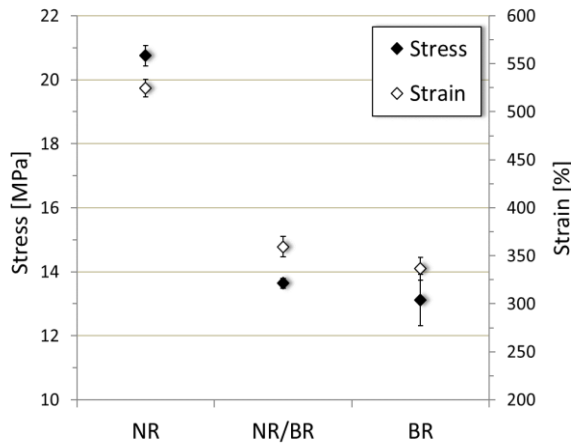
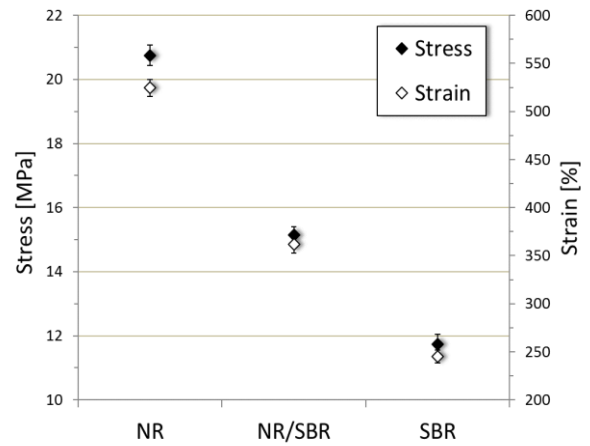*Figure 7. Results of stress and strain of NR, NR/BR and BR.*

Figure 8. Results of stress and strain of NR, NR/SBR and SBR.

Figures 9 and 10 depict values of stress that were measured at 50, 100 and 200 % of elongation. SBR containing styrene sections in polymeric chain are characterized by increased mechanical stiffness.
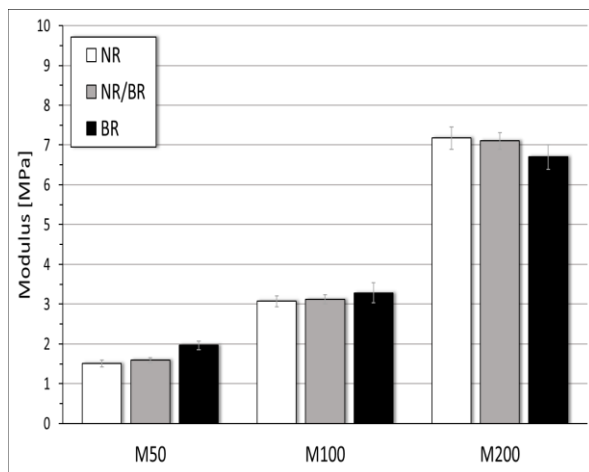

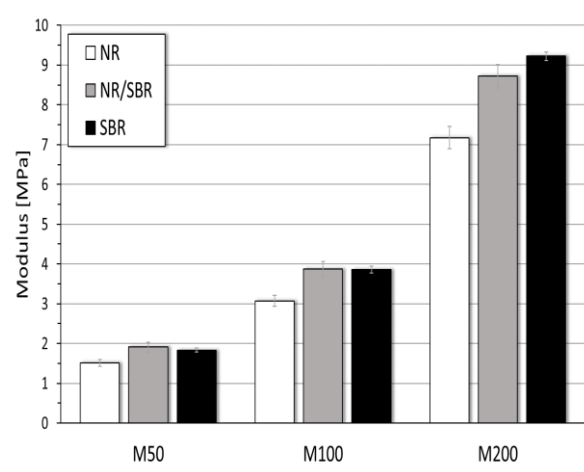*Figure 9. Results of stress necessary to elongation of 50, 100 and 200 % of NR, NR/BR and BR.*

Figure 10. Results of stress necessary to elongation of 50, 100 and 200 % of NR, NR/SBR and SBR.

4.1.3 Dynamic mechanical analysis

Dynamic mechanical analysis (DMA) was used to examine effect of temperature on viscoelastic properties of rubber compounds which is shown in Figures 11 and 12. Using DMA progress of Tan δ depending on temperature and material formulation was determined. Increase in Tan δ indicates that material has bigger energy dissipation potential thus material exhibits higher degree of damping. Measurement was performed in shear mode by DMA (Mettler Toledo, Switzerland) according to ISO 4664. Damping of rubber materials was determined in the temperature range, which is corresponded to the conditions of the measurement of fracture behaviour by tensile impact test.
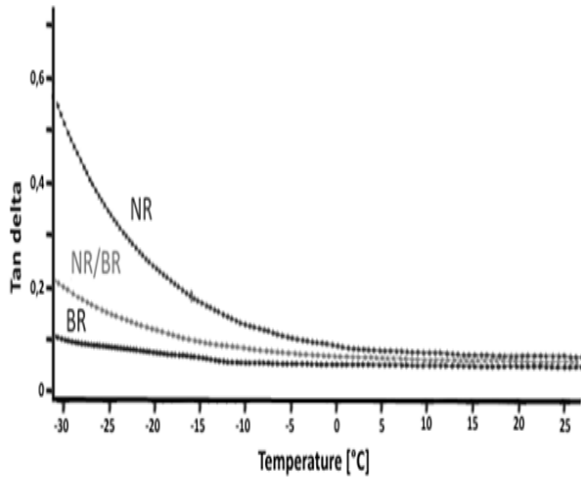
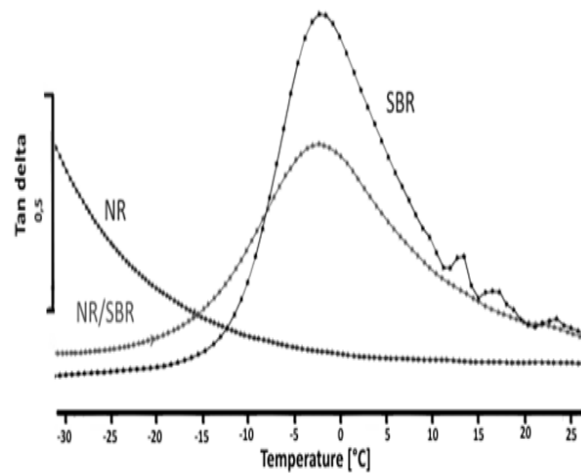*Figure 11. Dependence of Tan delta vs. temperature of NR, NR/BR and BR.*

Figure 12. Dependence of Tan delta vs. temperature of NR, NR/SBR and SBR.

The progress of Tan δ signal displayed in Figure 11 shows that rubber based only on NR exhibits the highest degree of damping at temperatures below freezing point for set of materials with BR addition. Ability to damping decreases with rising concentration of BR. And Figure 12 illustrates dependence of Tan δ signal on temperature for materials containing SBR. From these results it is visible that Tan δ signal of NR/SBR and SBR exhibited peaks which indicated value of *Tg* where rubber changes from glassy state when material is brittle to rubbery state.

4.2 Determination of wear and fracture properties of vulcanizates

In order to describe wear and fracture behaviour a few mechanical methods of testing both dynamical and cyclic were performed. Using these tests, it is possible to characterize influence of type and rate of loading on fracture behaviour of each used elastomer. Interpretation of results is focused on definition of influence of commonly used synthetic elastomers on durability at each stage of rubber product failure.

4.2.1 Determination the wear resistance behaviour using the abrasion resistance test

The wear properties were investigated by abrasion resistance test represented by moving the tested specimen across the surface of abrasive sheet mounted to revolving drum as this principle is well known and standardized. This testing method could fundamentally simulate process of tire sliding on the road surface when a rubber specimen is sliding on sandpaper with hard rough surface. Normal length of abrasion path used for standard method according to ASTM D5963 is 40 meters. For deeper determination of wear resistance, the abrasion

length was set to the 200 meters. Dependence of weight loss on the length of abrasion path is given as a result which are shown in Figures 13 and 14.
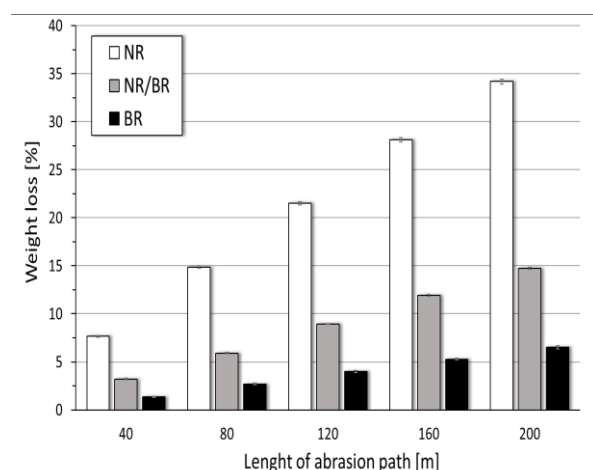

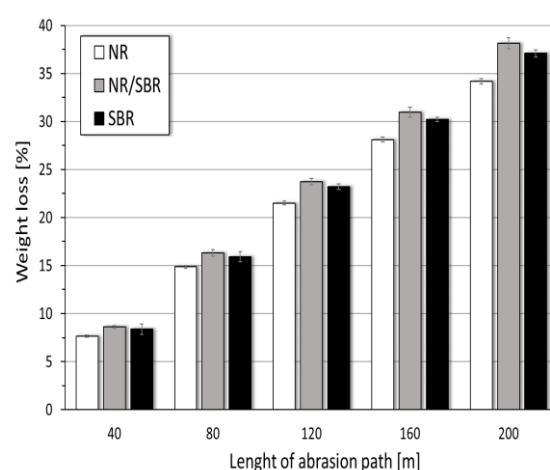*Figure 13. Results of abrasion test of NR, NR/BR and BR.*

Figure 14. Results of abrasion test of NR, NR/SBR and SBR

As it is seen in Figure 13 the weight loss was the lowest for pure BR throughout the test period. It is a feature of a good resistance against crack nucleation. This property is due to low amount of shear stress generated in contact area with asperities thanks to low loss modulus of BR at testing conditions. Results also indicate that added content of BR in compound with NR improves resistance to abrasion by more than a half. Figure 14 shows results of materials with SBR content. In comparison with NR values, slight decline of wear resistance is visible. It is caused by higher amount of friction between rubber and abrasive surface of SBR due to viscoelastic behaviour. Increment of friction results in stronger shear stresses and higher temperatures in contact area, thus higher rate of crack initiation. The 50/50 NR/SBR compound possess the wear resistance which could be caused by bad cohesion of NR and SBR segments in matrix.

4.2.2 Determination of impact resistance by free falling dart method

For characterization of resistance against penetration of unnotched specimen at high rate of deformation free falling dart method which is described by ISO 6603-2 was used. The principle of this method consists in penetration of clamped test specimen by a lubricated striker at a nominally uniform velocity. The force – extension diagram which is shown in Figure 15 could be divided into two parts which determine fracture behaviour of unnotched specimen under impact. Cracking energy E_{cr} corresponds to area under force – deformation diagram bounded by the origin to F_{cr} and provides information about resistance to crack initiation. Energy necessary to penetration of cracked specimen is described by area between F_{cr} and maximal penetrant force F_{pen} and

17

characterizes resistance to crack propagation. Results of these parameters are given by Figures 16 and 17.
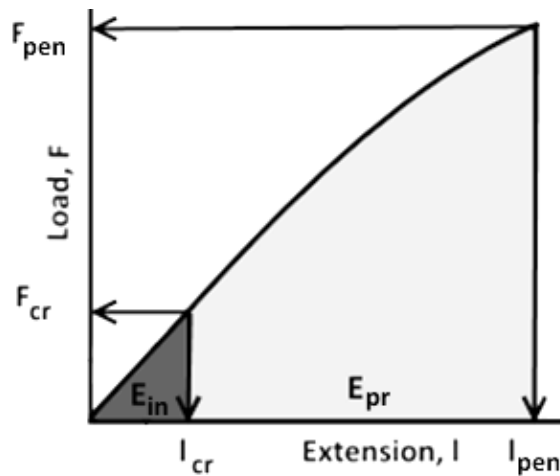

Figure 15. Schematic diagram of load – extension progress obtained by free falling dart test.
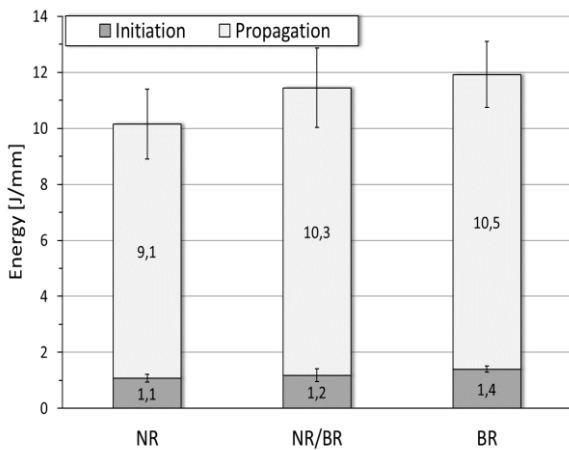

Figure 16. Summary of energy necessary to initiation and propagation of cracks in unnoched specimens of NR, NR/BR and BR.
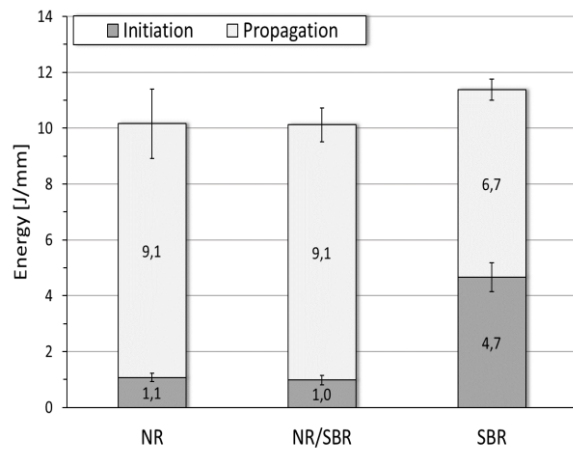
Figure 17. Summary of energy necessary to initiation and propagation of cracks in unnoched specimens of NR, NR/SBR and SBR.

Figure 16 shows the energy necessary to penetration of specimen which is divided into two stages representing crack initiation and propagation. From these results it is visible that presence of BR in compounds improves crack initiation resistance and also resistance to penetration. Furthermore, it is obvious that value of energy required to break grows with rising concentration of BR. As can be seen in Figure 17 material based on NR and its compound with SBR exhibits similar fracture properties as NR/BR compound. Material consisting solely from SBR exhibited the highest resistance to formation of crack by striker at high rate of deformation. Value of energy necessary to crack formation was approximately 4,5 times higher than for other materials. This could be caused by

both presence of styrene parts in molecules of SBR which carry strength and fact that energy necessary to crack propagation is lower in comparison with other materials due to brittle behaviour of SBR in this phase. Furthermore, the increased mechanical modulus of SBR was reflected by higher value of energy necessary to penetrate the sample thickness completely.

In conclusion, both commonly used synthetic rubbers have higher degree of resistance against penetration compared to NR.

4.2.3 Determination the crack resistance behaviour using instrumented notched tensile impact test

The instrumented notched tensile-impact test is an extension of the conventional tensile-impact test according to ISO 8256 generally used determine toughness properties of polymeric materials. In this case application of the instrumented Charpy impact test is not possible because of the specimen thickness and low material stiffness. [24] This method should be applied especially within testing of polymeric sheets and elastomers. [25]

The determination of crack resistance was performed by pendulum device according to ISO 13802 with impact energy 7,5 J and falling angle 150°. Double-edge-notched tension (DENT) specimens were used for testing.

Ten test specimens were analysed for each material. DAS4WIN software that displays force – extension diagram which schematic shape typical for elastomer is depicted in Figure 18 was used for analysis data. Area under force – extension curve is divided into two parts: A_{max} determines energy up to maximum load which characterize resistance to crack propagation; A_p represents crack propagation energy. The area under A_p gives information about ratio between stable and unstable crack propagation. When the area of A_p is small, the process of unstable crack propagation when crack spreads without supplying any additional energy is dominant.
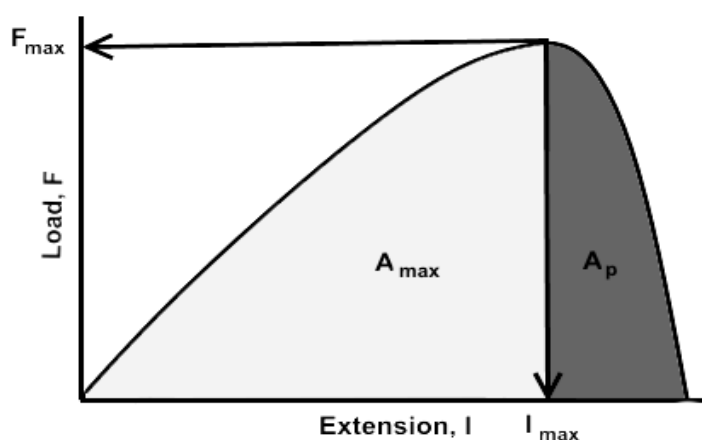

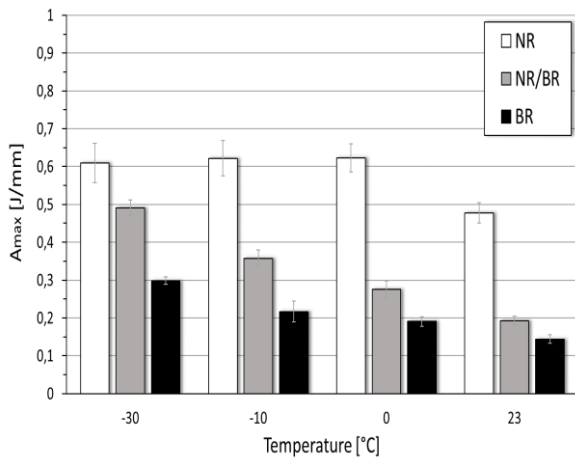*Figure 18. Schematic diagram of load – extension progress obtained by impact tensile test.*

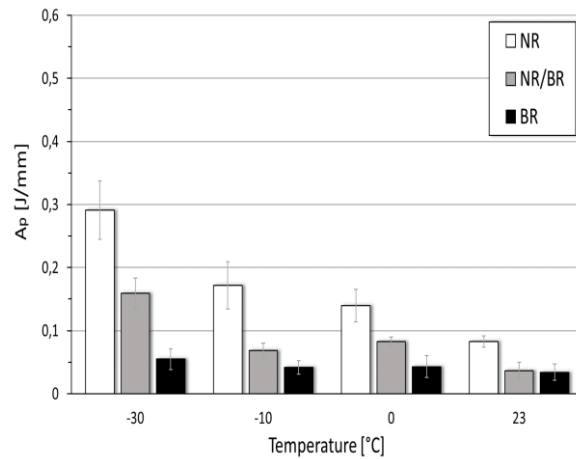*Figure 19 Results of energy associated with resistance crack growth of NR, NR/BR and BR.*

Figure 20. Results of energy associated with crack growth of NR, NR/BR and BR.

Energy necessary to growth of created notches which summary is given by Figure 19 decreases with rising content of BR, whereas material based only by BR exhibits the lowest level of crack propagation resistance. Furthermore, the effect of temperature shows that energy required for crack propagation increases with decreasing temperature. This trend is caused by increasing degree of material stiffness and necessity to reach higher energy needed to crack propagation. Second part of area under force – extension curve indicates how much energy is necessary to add to spread crack leading to rupture and it is shown in Figure 20. Results indicate that energy required to crack propagation is higher for NR than NR/BR and BR material, thus NR exhibits the highest ratio of stable crack propagation. On the other hand, crack in BR spreads unstably at lower energy values. The decline of temperature leads to improvement of fracture resistance.
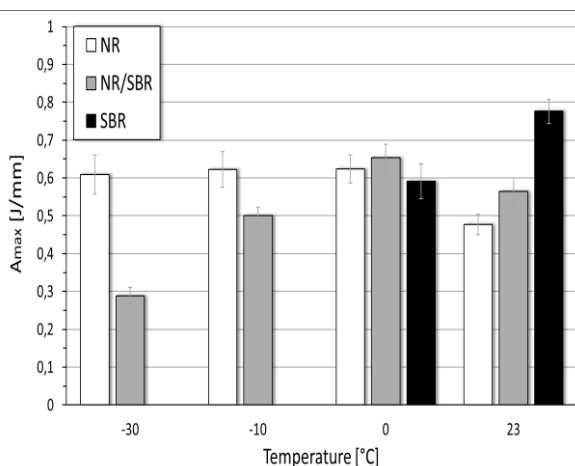

Figure 21. Results of energy associated with resistance to crack growth of NR, NR/SBR and SBR.
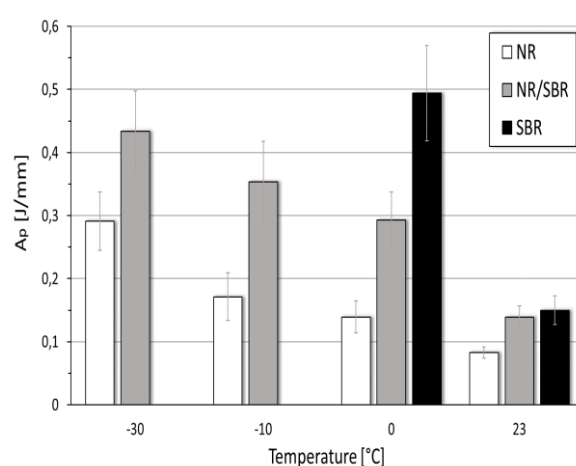
Figure 22. Results of energy associated with crack growth of NR, NR/SBR and SBR.

From results which are shown in Figure 21 it is evident that presence of SBR improves level of crack propagation resistance but only for temperature to the freezing point. With temperature approaching to the temperature of glass transition decline of energy absorption is visible. The energy absorption can be supplied before rupture occurs. For NR/SBR better resistance to crack propagation at 0 °C and for temperatures under zero together with huge decline of fracture properties is visible. Also effect of decreasing temperature on increase of energy *Ap* shown in Figure 22 is visible. For temperatures under glass transition material formed by pure SBR exhibits brittle behaviour therefore it was not possible to complete characterization due to fracture in a different part of specimen.
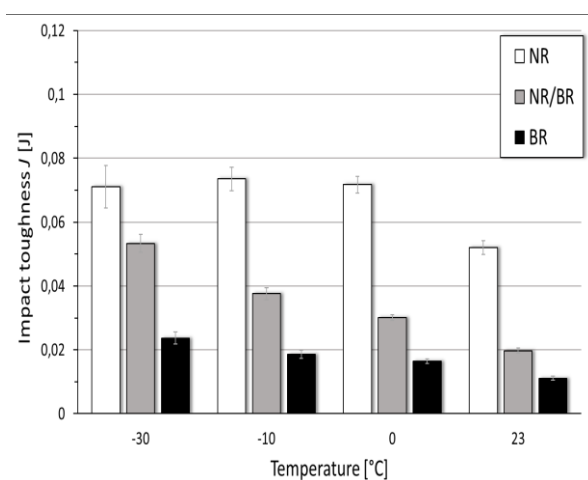

Figure 23. Results of impact toughness of NR, NR/BR and BR.
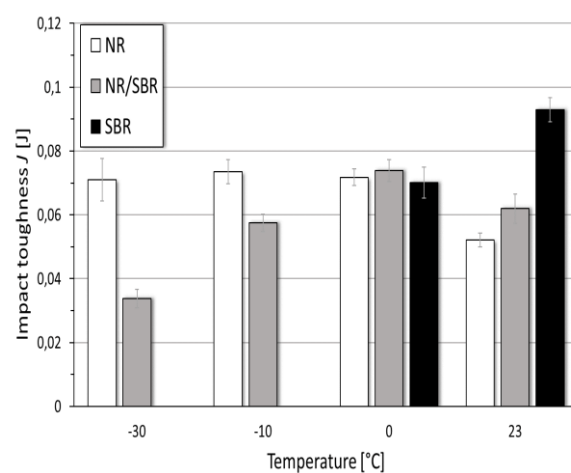
Figure 24. Results of impact toughness of NR, NR/SBR and SBR.

In general, NR has good impact toughness as can be seen in Figure 23 where is comparison with BR which exhibits the lowest impact toughness. Blend of NR and BR reaches values between NR and BR. Temperature has an influence on rate of impact toughness value. With decreasing temperature, the ability to absorb energy without rupture increases. Under zero degree of Celsius NR exhibits only slight improvement. SBR and their compound with NR achieve better impact toughness at room temperature than NR in comparison with BR as it is shown in Figure 24. But ability to absorb energy rapidly decreases with decreasing temperature. This phenomenon is given by reaching the temperature of glass transition of SBR at higher temperature than NR. At temperature near the glass transition material becomes more brittle and loses its ability to absorb energy and deforms without rupturing.

From results of crack resistance determination under impact in tension influence of viscoelasticity is evident. BR is a material with Tan delta peak occurring at temperature far below freezing. Due to this property it can be assumed that energy necessary to crack propagation will be lower for BR and on the other hand SBR should exhibit the best resistance to crack grow which have

the temperature of glass transition slightly under zero degree of Celsius. Both assumptions were confirmed experimentally using DMA analysis.

4.2.4 Crack propagation resistance under cyclic loading condition

Fatigue crack growth properties of vulcanizates were measured using a Tear and Fatigue Analyser (Coesfeld, Germany) which is an instrument allowing a quantitative analysis of fatigue crack growth processes on test specimens.

Fatigue crack growth measurements under sinusoidal loading with frequency 10 Hz for strains from 10 to 20 % were performed for vulcanizates. For materials containing BR were chosen deformation up to 15 % due to high velocities of FCG at higher values of strain. For each strain, three pure shear specimens were analysed. Results analysed with TFA are represented by double logarithmic plot of tearing energy, *T* versus crack growth rate (*da/dn*) that is shown in Figures 25 and 26.
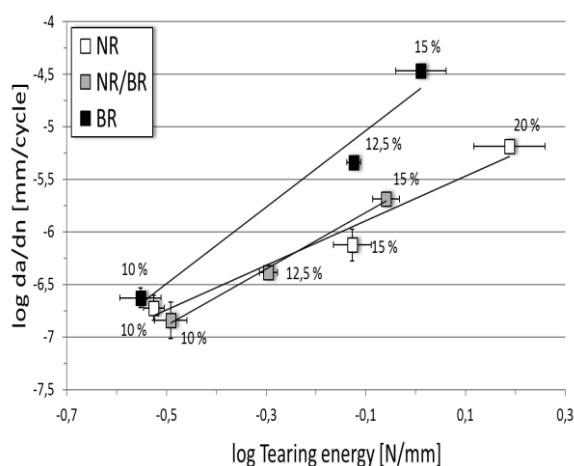

Figure 25. Dependence of fatigue crack growth rate vs. tearing energy of NR, NR/BR and BR.
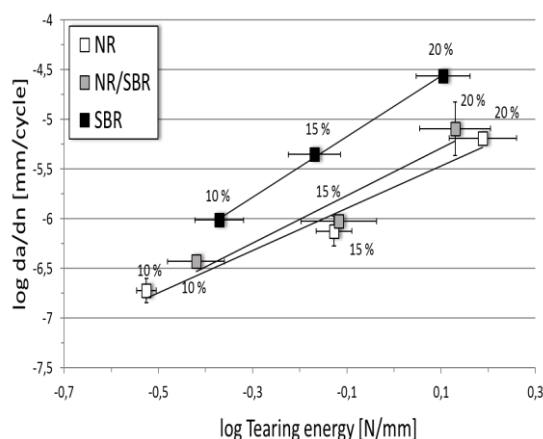
Figure 26. Dependence of fatigue crack growth rate vs. tearing energy of NR, NR/SBR and SBR.

From Figure 25 representing comparison of crack growth behaviour of NR, BR and their compound it is visible that vulcanizates based on NR exhibit the lowest crack growth rate at strain of 15 %. That indicates NR having higher fatigue crack growth resistance compared to BR and compound of NR with BR. Furthermore, NR has a lower crack growth rate at higher values of tearing energy than BR. Influence of SBR presence is seen in Figure 26 where results of NR, NR/SBR and SBR are shown. These measured data indicate that SBR elastomer exhibits lower resistance to crack growth in comparison with NR and NR/SBR which are characterized by using the same conditions. However, NR exhibits slightly better resistance to crack growth at higher level of strain than SBR and compound of NR and SBR.

4.2.5 Determination of crack growth resistance at rubber specimen which is cut by a sharp object

For characterization of resistance against crack propagation at pre-stressed rubber test specimen which is cut by sharp object newly developed device made in cooperation with Co. PRL Polymer Research Lab., s.r.o. (Zlín, Czech Republic) was used. The system uses the theoretical background and cutting method of Lake & Yeoh. [26] Principle of measurement is based on application of fully instrumented and electrically driven testing equipment, which is able to apply various loading force. Visualization of testing equipment is illustrated in Figure 38. The test specimen (6) fixed in a clamping system (7) and (8) is pre-stressed due to actuator of the axis Y (1) by given force which is controlled by load cell (3) in a strain direction Y. The actuator (2) provides movement of tip of razor blade (5) in an orthogonal direction to main loading stress applied to specimen. The process of cutting by razor blade tip is used as simulation of crack initiation by sharp object. Movement of razor blade through test specimen is provided by cutting force which is observed and controlled by load cell (4) throughout duration of analysis. The fracture mechanics phenomena were described on pure-shear test specimens. Measured data were used for calculation of the characteristic fracture values such as tearing energy T, which is responsible for rubber test specimen tearing process according to Rivlin & Thomas energy criterion. [27]
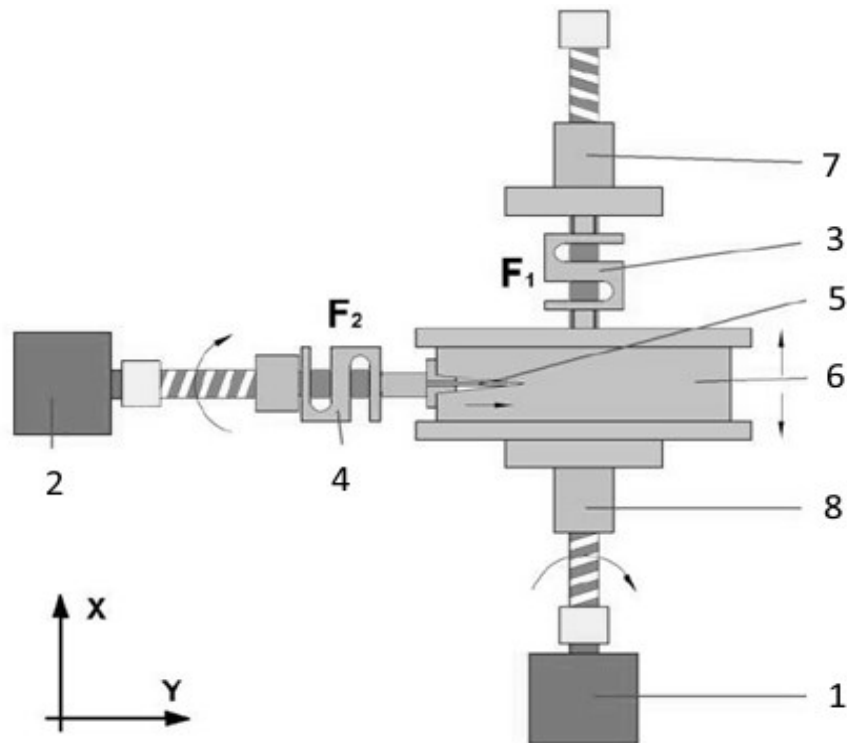

Figure 27. Functional principle and visualization of testing equipment for experimental characterization of crack initiation, where: 1 – actuator of the axis Y; 2 – actuator of the axis X; 3 – loading cell of the axis X; 4 – loading cell of the axis Y; 5 – razor blade; 6 – test specimen; 7 – upper clamping system of test specimen; 8 – bottom clamping system of test specimen. [2]

Influence of applied amount of cutting velocity, v_x; pre-force, F_y; and thickness of razor blade, d_b on change of tearing energy were evaluated from obtained data. Testing conditions, which were applied, are provided in Table 3.

Table 3. List of applied testing conditions used for crack propagation resistance.

Cutting velocity, v_x [mm/min]	Pre-force of test specimen, F_y [N]	Thickness of razor blade, d_b [mm]
25	50	0,4
100	100	0,1

Three standard pure-shear geometry specimens with length to width ratio of 8:1 were analysed for each pre-set condition and average values are plotted as results.

From results describing dependence of tearing energy increment ΔT that has been calculated by Equation 6 on crack length durability of material which is represented by rate of strain energy necessary to crack propagation can be deduced. Moreover, the ratio between stable and unstable crack propagation and value of critical tearing energy increment ΔT_{cr} causing rupture of specimen can be determined. Schematic progress of ΔT is shown in Figure 41.
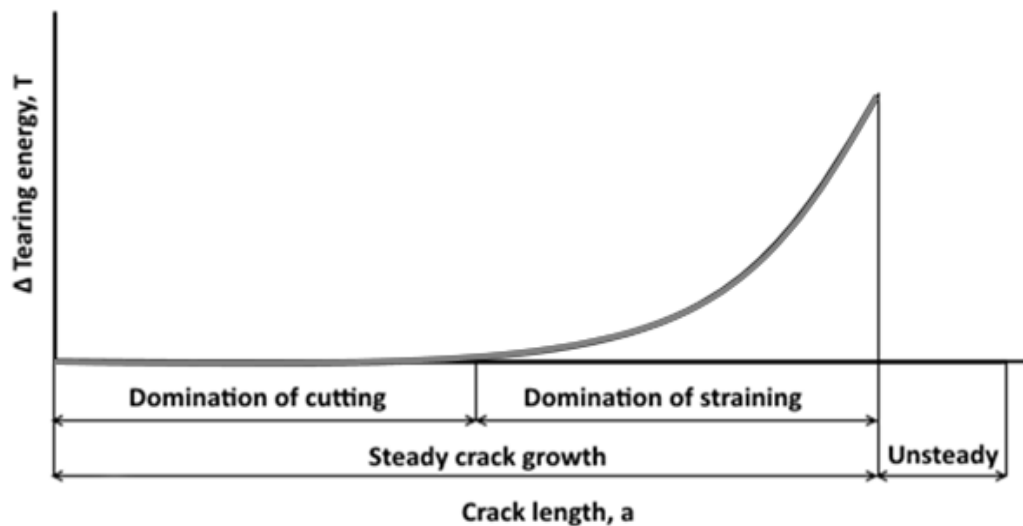

Figure 28. Schematic graph of dependence of tearing energy increment vs. crack length.

Tables 4 and 5 gives a summary of measured and calculated fracture characteristics.

Table 4. List of measured fracture characteristics of NR, NR/BR and BR.

	Pre-stressed force, F_y [N]	50				100			
	Cutting velocity, v_x [mm/min]	25	100	25	100	25	100	25	100
	Thickness of razor blade. d_b [mm]	Rupture [%]		Critical ΔT [N/mm]		Rupture [%]		Critical ΔT [N/mm]	
BR	0,1	88,5	89,7	14,5	8,4	77,4	75,8	22,9	13,1
		± 4,6	± 3,8	± 0,9	± 0,5	± 3,5	± 5,1	± 2,1	± 1,2
	0,4	89,8	91,7	19,7	11,6	81,1	80,0	27,2	17,5
		± 3,7	± 5,7	± 1,5	± 0,8	± 4,4	± 6,4	± 2,5	± 2,1
NR/BR	0,1	89,9	90,0	21,1	12,8	77,5	78,0	22,9	17,8
		± 3,2	± 4,4	± 1,3	± 1,8	± 6,5	± 4,9	± 0,9	± 2,5
	0,4	89,2	92,5	23,1	15,4	77,8	79,7	25,3	21,3
		± 4,5	± 5,8	± 2,1	± 1,1	± 7,5	± 5,2	± 1,7	± 1,4
NR	0,1	91,3	93,7	35,2	15,9	80,5	82,4	39,9	37,8
		± 3,4	± 3,9	± 2,8	± 2,7	± 4,8	± 6,7	± 3,8	± 2,9
	0,4	93,1	95,0	38,7	16,9	83,8	85,0	52,2	41,4
		± 4,9	± 4,8	± 3,4	± 2,1	± 3,7	± 4,1	± 4,1	± 4,9

Table 5. List of measured fracture characteristics of NR, NR/SBR and SBR.

	Pre-stressed force, F_y [N]	50				100			
	Cutting velocity, v_x [mm/min]	25	100	25	100	25	100	25	100
	Thickness of razor blade. db [mm]	Rupture [%]		Critical ΔT [N/mm]		Rupture [%]		Critical ΔT [N/mm]	
SBR	0,1	90,4	92,1	23,8	14,6	81,1	85,0	30,5	33,9
		± 8,1	± 7,7	± 1,9	± 1,1	± 3,4	± 5,4	± 2,1	± 2,1
	0,4	90,3	94,6	25,3	16,5	81,0	83,7	36,0	36,9
		± 7,2	± 9,2	± 2,0	± 1,4	± 5,4	± 4,2	± 3,8	± 1,7
NR/SBR	0,1	90,8	93,9	30,4	15,7	83,1	82,3	38,0	33,7
		± 8,8	± 9,5	± 1,8	± 1,6	± 4,5	± 8,2	± 2,2	± 3,1
	0,4	91,0	95,5	33,3	17,6	81,1	84,7	43,0	35,7
		± 6,3	± 6,2	± 0,9	± 1,8	± 6,8	± 6,7	± 4,8	± 2,7
NR	0,1	91,3	93,7	35,2	15,9	80,5	82,4	39,9	37,8
		± 8,4	± 5,9	± 3,1	± 2,3	± 6,6	± 6,1	± 3,3	± 3,7
	0,4	93,1	95,0	38,7	16,9	83,8	85,0	52,2	41,4
		± 8,2	± 7,1	± 2,8	± 1,3	± 7,2	± 7,4	± 3,8	± 3,4

From obtained results, it is visible that BR and its compound with NR exhibit the lowest crack propagation resistance in comparison with NR, SBR and their compounds. Moreover, it is seen that strain energy necessary to crack growth is higher for cutting using thicker razor blade which corresponds with Equation 7. For higher velocity of razor blade movement value of tearing energy is lower due to greater contribution of cutting energy on crack propagation.

4.2.6 Determination of resistance against crack initiation by cutting with sharp object

Characterization of crack initiation resistance was performed using improved version of device measuring stress states in rubber specimen which was cut by a sharp object. This improved version is able to measure applied cutting force and movement of razor blade more accurately and therefore it can be used for crack growth rate characterization at small area. In table 6 conditions which were used for observation of movement velocity of razor blade through the sample to a length of 10 mm are shown. For a purpose to describe differences in crack growth velocity during cutting process, the distance of 10 mm was divided into five points with 2 mm length. The change of crack growth rate, da/dt depends on tearing energy, T is represented by double logarithmic plot.

Table 6. List of applied testing conditions used for crack initiation resistance.

Designation	Cutting force, F_x [N]	Pre-force of test specimen, F_y [N]	Thickness of razor blade, d_b [mm]
XX_2_50	2	50	0,1
XX_2_100	2	100	0,1
XX_3_50	3	50	0,1
XX_3_100	3	100	0,1

XX – BR, NR/BR, NR, NR/SBR, SBR

Three standard pure-shear geometry specimens with length to width ratio of 8:1 were analysed for each pre-set condition and average values are plotted as results in following Figures 29-33.

The designation of each test specimen set is given by the code:
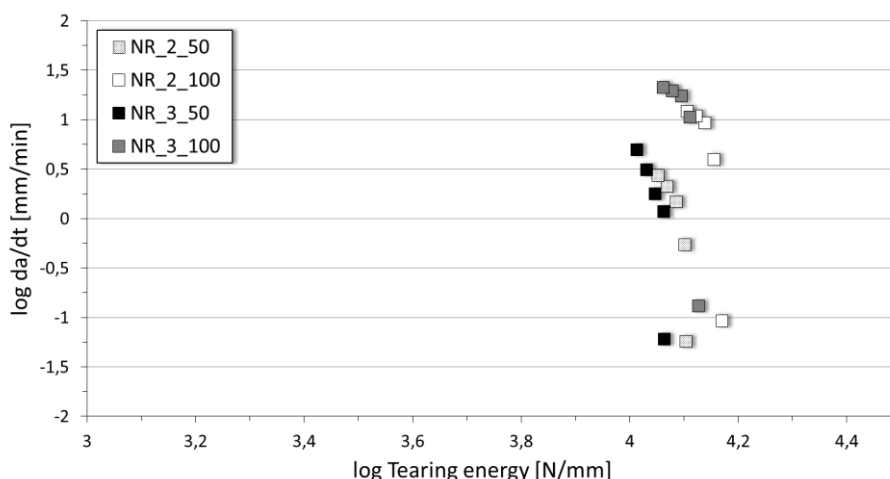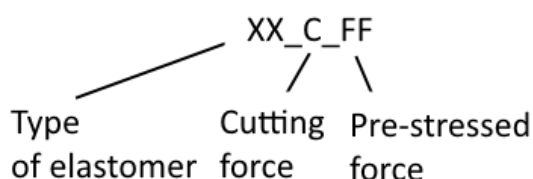
Figure 29. Dependence of crack growth rate vs. tearing energy of NR pre-stressed by force 50 N and 100 N and notched by 0,1 mm thick razor blade by force 2 and 3 N.

From results of NR it is visible that crack growth rate at the first 2 mm of crack length is significantly lower than its values of further points of crack length. It is given by increased initial resistance to cut. Reduction of initial resistance with increasing crack length resulting in decrease of energy required for crack extension of 2 mm, and simultaneously, increase of crack growth rate is detectable. Nevertheless, it is evident, when a specimen is cut by lower force, amount of energy necessary to crack propagation slightly growing.
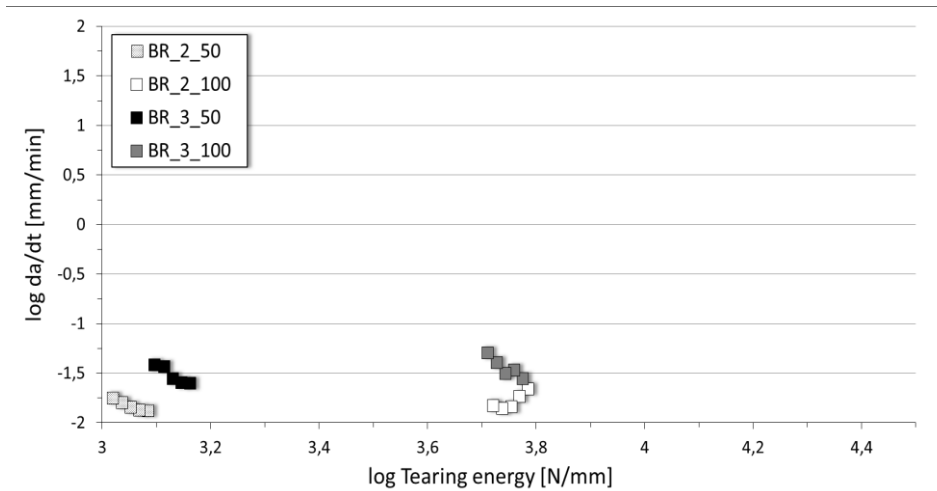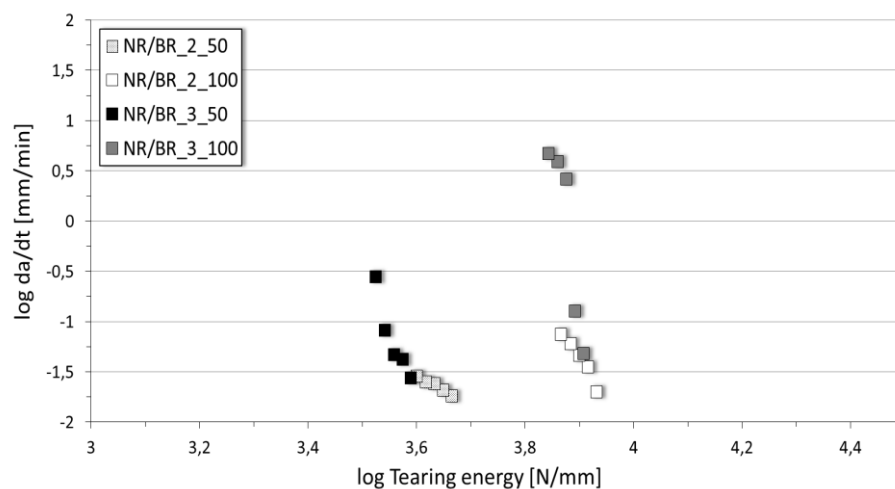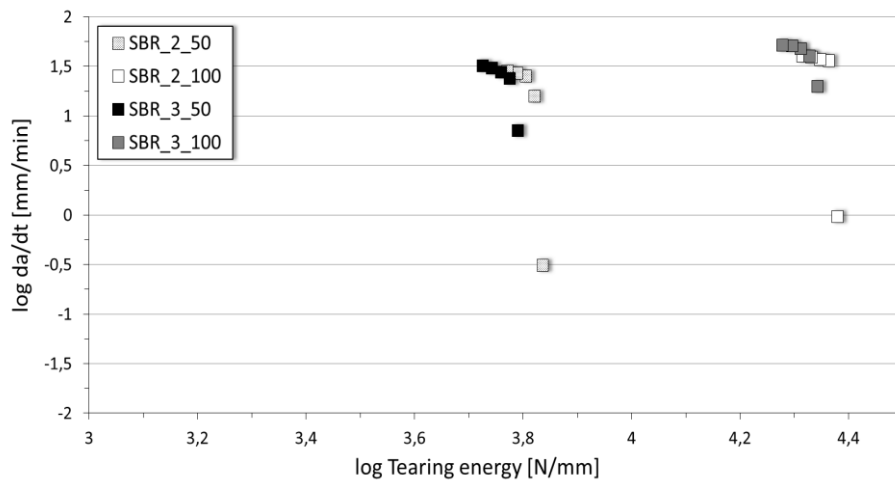
Figure 30. Dependence of crack growth rate vs. tearing energy of BR pre-stressed by force 50 N and 100 N and notched by 0,1 mm thick razor blade by force 2 and 3 N.

In contrast, BR exhibits low crack growth rate indicating good crack initiation resistance compared to the NR. Gradual increase of crack growth rate is visible for all specimens except specimens that were loaded by 100 N and cut by 2 N. Specimens studied at these conditions were characterized by gradual decrease of crack growth rate in dependence on crack length.
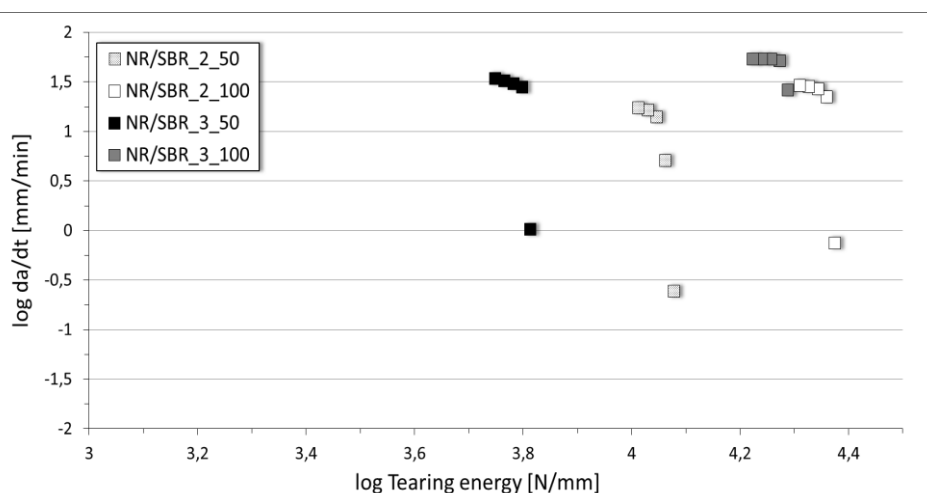

Figure 31. Dependence of crack growth rate vs. tearing energy of NR/BR pre-stressed by force 50 N and 100 N and notched by 0,1 mm thick razor blade by force 2 and 3 N.

Compound consisting NR and BR is represented by fracture behaviour influenced by contribution both elastomers. NR in compound improves crack growth resistance. That is demonstrated by increasing amount of energy necessary to crack growth. On the other hand, there is visible decline of crack growth rate in comparison with NR that is noticeable influence of BR.

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Figure 32. Dependence of crack growth rate vs. tearing energy of SBR pre-stressed by force 50 N and 100 N and notched by 0,1 mm thick razor blade by force 2 and 3 N.

According to the results published in Figure 32 it is visible, that SBR exhibits the highest values of crack growth rate. It is a feature of low resistance to propagation of crack formed by sharp object. Energy necessary to crack propagation is slightly higher than for NR except specimens loaded by force of 50 N. Resistance to crack initiation and propagation is low, therefore there is not enough time for creep process leading to bigger amount of energy absorption than in case of NR.

Figure 33. Dependence of crack growth rate vs. tearing energy of NR/SBR pre-stressed by force 50 N and 100 N and notched by 0,1 mm thick razor blade by force 2 and 3 N.

In Figure 33 results of material which was formed by NR and SBR can be seen. This material has approximately the same values of crack growth rate like SBR. However, effect of NR content is visible for specimens which were loaded by 50 N where NR evidently improves crack initiation resistance. Values of crack growth rate are lower than for SBR and moreover energy necessary to crack growth achieves higher values.

5. CONCLUSION

Presented thesis was conducted to make a theoretical summary focused on fracture mechanics of rubber. The theoretical background was supplemented with experimental part containing a collection of basic mechanical testing methods usable for characterization of mechanical and fracture behaviour of rubber. Rubber compounds based on NR, BR, and SBR filled by carbon black were used as standard materials for investigation of fracture behaviour.

A few mechanical testing methods working quasi-statically and cyclically were performed in order to describe fracture behaviour at different shape of loading waveform. The initial process leading to failure of rubber product is represented by formation of crack being able to propagate when value of supplied energy is high enough. In this work, methods describing crack initiation resistance at low and high velocities were performed.

Conventional abrasion test was used to determine crack initiation resistance of rubber at low velocities. It is evident that BR increases resistance against crack initiation whereas SBR has the opposite effect.

In contrast, free falling dart method was used to characterize durability of rubber specimens without any macro cracks at high speed. The first phase of specimen penetration describes resistance to crack formation. From these results it is visible, that addition of BR also leads to improve crack initiation resistance at high velocities, and however material prepared only from SBR exhibits the highest energy necessary to crack formation. Furthermore, the data obtained by free falling dart test could be used to determine amount of energy necessary to break specimen, thus propagation of crack. Data analysis has shown that growing concentration of BR leads to increase of resilience.

When the energy supplied in specimen with presence of flaws and macro cracks is sufficiently large, crack propagation occurs. Rubber resistance to crack propagation was measured in impact and cyclically in tension using notched specimen. The impact tensile test was used to evaluate crack propagation resistance at high velocities, especially the effect on ratio between stable and unstable crack growth region. Moreover, influence of temperature was investigated. It is visible from the obtained results, that BR exhibits low resistance to crack propagation at both regions. However, the resistance increases with decreasing temperature. SBR exhibits an improvement of crack propagation resistance in comparison with NR. But this property is noticeable only for temperatures higher than temperature of glass transition which is given by peak of Tan delta. Furthermore, influence of elastomer type on rate of ability to absorb strain energy without failure was characterized. It is shown that BR exhibits the lowest level of impact toughness from tested materials and the property is positively influenced by decreasing temperature. On the contrary SBR has positive effect on impact toughness.

In case of fatigue crack propagation resistance, sinusoidal cyclic loading in tension was carried out. From dependence of tearing energy on crack growth rate it is visible that NR achieves the lowest level of crack growth rate which is a sign of good crack propagation resistance whereas the worst resistance is detected for BR.

In addition, as part of published research a new device to investigate stress states into specimen which is loaded in tension and simultaneously cut by sharp object has been introduced. Resistance to crack initiation and propagation was described using this device. From obtained data it is confirmed that BR exhibits the best resistance to crack initiation in comparison with NR and SBR which exhibits the worst resistance. In the case of propagation, the best properties were described for NR whereas BR has the lowest durability. In addition, it was confirmed that the energy necessary to crack propagation decreases with decreasing radius of crack tip.

In conclusion, the experimental work summarizes various common testing methods and demonstrates the ability of the simple methodology to characterize the complex fracture properties of rubber materials to predict the final rubber product behaviour. It was found out that published testing methods are useable for description of processes leading to mechanical failure of rubber as well as for fracture properties description of tested rubber compounds. Further, the device able to characterize resistance against penetration of sharp object into pre-stressed rubber test specimen was introduced. Using this device, good crack initiation resistance of BR and good durability of NR was confirmed. Thus it could be said that the newly developed device can be used for characterization of crack initiation resistance and is able to fully substitute the abrasive test device. Moreover, the crack propagation resistance can be promptly measured using this device.

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List of figures

List of tables

List of symbols and acronyms

a	crack length
A	area of one fracture surface
ASTM	American Society for Testing and Materials
BR	butadiene rubber
d	thickness of specimen
d_b	thickness of razor blade
DMA	dynamic mechanical analysis
d_n	diameter of notch tip
E	elastic modulus
DENT	double-edge-notched tension
E_{cr}	cracking energy
FCG	fatigue crack growth
F_{cr}	cracking force
F_{pen}	penetration force
F_x	cutting force
F_y	pre-stressed force
G	crack driving force
ISO	lnternational Organization for Standardization
J	impact toughness
K_I	stress intensity factor in tension
MDR	moving die rheometer
NR	natural rubber
SBR	styrene butadiene rubber
t	time
T	tearing energy
T_{cr}	critical tearing energy
T_g	glass transition temperature
TFA	Tear and Fatigue Analyzer
U	elastic energy of the system
U_0	elastic energy of the system without crack
U_a	strain energy
U_γ	surface energy
v_x	cutting velocity
w	strain elastic energy density
W	mechanical work
W_b	work required to break

List of publications

Publications

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2012
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Curriculum Vitae

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Work experience

Dates	2014–present
Occupation or position held	Research staff – Centre of Polymer Systems
Name of employer	Tomas Bata University in Zlín, University Institute, Centre of Polymer Systems

Education

Dates	2010–2012
Title of qualification awarded	Ing.
Principal branch	Polymer Engineering
Organization providing education	Tomas Bata University in Zlín, Faculty of Technology
Dates	2007–2010
Title of qualification awarded	Bc.
Principal branch	Polymer Materials and Technology
Organization providing education	Tomas Bata University in Zlín, Faculty of Technology

Training Abroad

Dates	September – December 2013 (3 months) Study stage at Chemnitz University of Technology, Faculty of Mechanical Engineering, Professorship of Plastics, Chemnitz (Germany)
Field of study	Initiation and propagation of microcracks in vulcanizates
Dates	May – July 2014 (2 months) Study stage at Chemnitz University of Technology, Faculty of Mechanical Engineering, Professorship of Plastics, Chemnitz (Germany)
Field of study	Characterization of the dynamic properties of polymer materials
Dates	November – December (1 month) Study stage at Polymer Service GmbH Merseburg, Germany Merseburg (Germany)
Field of study	Characterization of rubber material under high dynamic loading conditions

Expertise

Fields	Polymer materials, rubber materials, fracture and fatigue behaviors

Other languages

	Level
English	C1
German	A2

Ing. Ondřej Kratina

Structure and fracture behaviour of rubber
Struktura a lomové chování pryže

Doctoral Thesis Summary