EFFECT OF CARBON BLACK TYPE ON THE MECHANICAL BEHAVIOUR OF ELASTOMERIC MATERIAL UNDER DYNAMIC LOADING

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ABSTRACT

The mechanical behavior of filled elastomeric (rubber and rubber-like) materials is known to be incompressible, or nearly-incompressible, hyperelastic and time-dependent, or viscoelastic. This complex behavior of rubbery materials needs more understanding, and good knowledge is required for such behavior in order to achieve a good constitutive modeling for better design of a rubber component for a specific application. This work concentrated on studying the effect of carbon black type on the mechanical properties of rubbery material characterization. To do this, different tests were performed on filled rubber with three different kinds of carbon black N326, N375, and N660. All tests were performed at room temperature. The tests include rheometer tests, hardness tests, tensile tests, specific gravity tests, compression tests, relaxation test, and cyclic loading tests. Tensile tests were done with different strain rate, relaxation tests done under different mean strain.

Tensile stress-stretch curves were generated from the test data at strain rate ranging from 10 to 500 mm/min and several transitions associated with strain-induced crystallization were observed in all materials. The filled rubber became stiffer when the strain rate increased from 10-200 mm/min, and became more compliant when the strain rate increased from 200 to 500 mm/min.

Hardness and specific gravity tests showed that rubber filled with carbon black N375 is harder and has specific density more than other two types of filled rubber.

The mechanical compression set tests, which performed on rubber with three kinds of carbon black and found that compression set for rubber filled with carbon black N375 is more than the other two kinds of filled rubber.

Relaxation stress-time curves were generated from test data at varying mean strain ranging (50% to 200%) from the effective length of the specimen, at constant strain rate (200 mm/min). Generally, it was observed that the stress reduces with time when the specimen hold at specific strain. This reduction is faster at strain between 50% and 100% than the strain between 150 to 200% till reaches steady state.

Series of cyclic tension tests were carried out at room temperature on a rubber compound under strain rate 200 mm/min. All these cyclic strain-controlled experiments showed that the filled rubber materials, used in the present work are time-dependence with hysteresis. It came out that hysteresis for rubber with carbon black N375 is more than the rubber with other two kinds of carbon black.

تأثير نوعية اسود الكاربون على التصرف الميكانيكي للمواد الايلاستومير تحت التحميل الحركي

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الخلاصة :

يعرف التصرف الميكانيكي للمواد الايلاستومير (المطاط و المواد المشابهة للمطاط) المدعمة على انه غير قابل للانظغاط او انه قريب من ذلك , شديد المطيليه ويعتمد على الوقت او انه مرن لزج . هذا التصرف المعقد للمواد المطاطية يتطلب فهم اكثر , ويتطلب معرفة جيدة من اجل الوصول الى نمذجة جيدة لتصميم افضل لمركبات المطاط عند استخدامها في مجال معين . هذا العمل يركز على دراسة تأثير نوع اسود الكاربون على خواص المواد التي توصف بانها مطاطية . لعمل هذا اختبارات مختلفة اجريت على المطاط المدعم بثلاث انواع مختلفة من اسود الكاربون (N660,N326,N375) . جميع الاختبارات اجريت بدرجة حرارة الغرفة . الاختبارات تضمنت , اختبار الريوميتر , اختبار الصلابة , اختبار الشد , اختبار الثقل النوعي , اختبار الانضغاطية , اختبار الاسترخاء , اختبارات التحميل الدوري . اختبارات الشد اجريت تحت معدلات انفعال مختلفة , اختبارات الاسترخاء اجريت عند متوسط انفعال مختلف .

من بيانات اختبارات الشد عند معدلات انفعال مختلفة تتراوح بين (10-500 ملم/دقيقة) تم توليد منحنيات الاجهاد-الاستطالة حيث لوحظ ولجميع المواد عدة انتقالات ترتبط مع (الانفعال يحث التبلور) . المطاط المدعم يصبح اصلب عندما يزداد معدل الانفعال من 10 ال 200 ملم/دقيقة بينما يكون اكثر مطاوعة عند زيادة معدل الانفعال من 200 الى 500 ملم/دقيقة .

اظهرت اختبارات الصلابة والثقل النوعي ان المطاط المدعم باسود الكاربون (N375) اكثر صلابة وثقل نوعي من النوعين الاخرين الاخرين من المطاط المدعم .

من اختبارات الانضغاطية الميكانيكية التي اجريت للمطاط بثلاث انواع من اسود الكاربون اوجدت ان المطاط المدعم باسود الكاربون (N375) له انضغاطية اعلى من النوعين الاخرين من المطاط المدعم .

من البيانات التي تم الحصول عليها من اختبارات الاسترخاء تم توليد منحنيات الاجهاد-الوقت عند مستويات انفعال متغيرة تتراوح بين 50% الى 200% من الطول الفعال للعينة وعند معدل انفعال ثابت هو 200 ملم/دقيقة . بصورة عامة لوحظ ان الاجهاد يتناقص مع الزمن عند مسك العينة عند انفعال محدد . وهذا التناقص يكون اسرع عند انفعال يتراوح بين 50% الى 100% من الانفعال الذي يحدث عند انفعال بين 150% الى 200% حتى الوصول الى حالة الاستقرار .

سلسلة من اختبارات الشد الدوري اجريت بدرجة حرارة الغرفة على المطاط تحت معدل انفعال (200 ملم/دقيقة) . كل التجارب التي تسيطر على الانفعال الدوري اظهرت ان المواد المطاطية المدعمة هي تعتمد على الوقت مع الهسترة . النتيجة كانت ان الهسترة للمطاط المدعم باسود الكاربون (N375) كانت اكثر من النوعين الاخرين .

Keywords: elastomer, large deformation, viscoelastic, carbon black, strain rate.

1. INTRODUCTION

Rubber and rubber like materials are generally called elastomers. Rubber has excellent properties that make it an excellent choice for tire industries, rubber has high mechanical strength and can be compounded to have excellent elasticity, good abrasion resistance, low relative cost, good dynamic mechanical properties, very good low temperature resistance [Ciesielski 2000], down into the region of -57°C, inherent weather (UV light and ozone) resistance, and electrical insulation is very good. Rubber also capable of adhering to textile fibers and metals, such as rayon, polyamide, polyester, glass, and steel cord.

Recently, many researches about elastomer materials revealed different properties, contrary for all real materials elastomer is incompressible or nearly incompressible material and exhibit a unique property that is a hyperelastic is extensible when subjected to a load with high deformation and could reach from 500% to 1000% and back to the original or almost original dimensions after unload depending on the time. In addition, carbon black is usually used as an additive for improving the rubber's strength, by inducing cross-links to form between the polymer chains in order to limit the chain's ability to move independently [Makul & Rattanadecho 2010].

The complex behavior of elastomer makes it impossible for its properties to be characterized by simple stress-strain relation which is useful in hard solids materials. The typical stress-strain curve shows nonlinear behaviour under large strain, so it is impossible to determine definite value to the Young modulus unless obey to Hooke`s law except in a region for small strain which show almost linear behaviour. Elastomer materials properties need to be described by strain energy function. The principal point in modeling of elastomer material is to select the appropriate constitutive model which gives adequate parameters [Hossa & Marczak 2010].

The main objective of the present research is to study and investigate the effect of carbon black type on the mechanical behaviors of the three tread rubbery blends for farm tire which already used in Babylon tires factory. Three types of carbon black will be used; the testing results and the mechanical response will be the key factor for determining the best blend choice for the farm tire industry.

2. EXPERIMENTAL

2.1 Materials and Sample Preparation

The materials and samples preparing processes were done in Babylon Tire Factory laboratories. The gum material and additives were performed using the calendering machine, the mixing process continues till reaching a homogenous blend. The sulfur and accelerators are added at the end of the mixing process to avoid curing during calendaring processes. Electrical piston was used to cure the blend, when the piston temperature reach (145C°) the (70 gr) from the blend, Table 1, pressed by mold for (45 min) to produce a thin sheet of rubber which can be used later to make the dumbbell specimen. **Table (1)** shows the blend with carbon black N375, the two other recipes are the same but with carbon black N326 and N660.

Dumbbell specimens were obtained under ASTM D412 specifications for tensile test. Same process but different molds were used to produce the specimen in order to use in hardness and density investigations. During this process the mold filled with the above blend and pressed under electrical piston for (15 min) with temperature up to (160 C°) as show in **Figure 1.**

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2.2 Rheometer Test.

This test have been done by using Monsanto rheometer (ODR 2000E). The computerized printer reveals curves for the torque required to oscillate the rotor as a function of time. Test results show in **Figures 2, 3 and 4** for the three blends.

Curve values printed out as abbreviated terms. These important terms can be explained as follows. ML (Minimum Torque): It is the lowest value of Torque recorded when the compound heated under pressure in which the viscosity decreases and the torque fall. Basically ML is a measure of the stiffness and viscosity of unvulcanized compound [Future Foundation 2003].

MH (Maximum Torque): As the curing starts, the torque increases proportionately. Depending upon the type of compound, the slope of rising torque varies. After a while the torque typically attains maximum value and it plateaus out. It is called “Plateau Curve”. MH (Max. torque) is the highest torque recorded in plateau curve.

Ts2 (Induction time): After attaining minimum torque, during cure phase, as the torque rises, ts2 is scorch time for viscosity to raise 2 units above ML. Is measure of initial slope of curing phase of rheograph i.e. this is measure of processing safety. Scorch is premature vulcanization in which the stock becomes partly vulcanized before the product is in its final form and ready for vulcanization. Scorching is the result of both the temperatures reached during processing and the amount of time the compound is exposed to elevated temperatures. This period before vulcanization starts is generally referred to as “Scorch time”. Since scorching ruins the stock, it is important that vulcanization does not start until processing is complete.

T90 (Optimum Cure time): It is the time at which 90% of cure has taken place. **Table 2** summaries the values of these terms for the three blends used in the current analysis.

2.3 Hardness Test

As hardness is probably the most widely used cured property of rubber so the scientist efforts were considerably concentrated on making the hardness test more easily and practical and regard this test is somewhat subjective. In present work Wallace units I.R.H.D instrument was used for hardness test (shore A). **Table 3** shows the results obtained from hardness test for the same blend with different kinds of carbon black used in the current work.

2.4 Tensile Tests

These tests have been carried out by using the instrument showing in **Figure 5** type Monsanto Tensometor 10. To starting the test the instrument must be fed by input data, thickness, width and the strain rate. The experiment has been repeated with different strain rates for all three blends starting from 10 mm/min then increased to 25, 50,100, 200 and 500 mm/min.

2.5 Compression Set Tests

These test methods cover the testing of rubber intended for use in applications in which the rubber will be subjected to compressive stresses in air or liquid media. They are applicable particularly to the rubber used in machinery mountings, vibration dampers, and seals.

The specimen used in this test showing in **Figure 1b** which is a disk of rubber have diameter (29±1) mm and thickness (12±1) mm. The device used in the test is consisting of a force application spring and two parallel compression plates assembled by means of a frame or threaded bolt in such a manner that the device shall be portable and self-contained after the force has been applied and that the parallelism of the plates shall be maintained. The force may be applied in accordance calibrated spring force application. The required force shall be applied by a screw mechanism for compressing a calibrated spring the proper amount. The spring should be of properly heat-treated spring steel with ends ground and perpendicular to the longitudinal axis of the spring. The spring should be calibrated at iron box with temperature 23 ± 5°C.

Calculation process needed to find compression set as a percentage of the original thickness as follows [ASTM International Committee 2008]:

$$CA = ((t_o - t_i)/t_o) * 100 \qquad (1)$$

where *CA* is the compression set as a percentage of the original thickness, t_o is the original thickness, and t_i is the final thickness.

Table 4 shows the compression set tests of the three blends, from this table it can be recognized that the blend with carbon black N375 have higher compression set than that in blends with carbon black N326 which is in turn more than N660 compression set.

2.6 Specific Gravity Test

This property is fully defined by the composition of the material. The specific gravity is the mass per unit volume and is measured by weighing the sample in air and in water according to the following formula [Rubber precision parts 2011]:

$$G_s = \frac{w_{air}}{(w_{air} - w_{water})} \qquad (2)$$

where G_s is the specific gravity, w_{air} is the weight in air, and w_{water} is the weight in water.

The test done by using Monsanto-Densitron instrument, the sample showed in **Figure 1c**. The instrument consists of a closed room as a container. The inside of this room has a drawer which is divided for many places and slide horizontally to put and remove the samples. Below this slide there is a basin filled by water. The instrument has clamps to carry the sample from the slide to the basin water and return it back to its position in slide automatically. The operation of the instrument is totally programmed and the operation start immediately after putting the sample on the slide in the specific place and close the room's door. **Table 5** shows the results for three blends.

2.7 Stress Relaxation Test

Rubber is classified as viscoelastic materials, viscoelastic materials appear both elastic solid and a viscous fluid response when deformed. The main important method to study and compare the viscoelastic compounds properties is stress relaxation experiment method [Karrabi & Mohammadian-Gezaz 2011]. Stress relaxation can be defined as continued decreasing in stress needed to maintain a given deformation or loss of stiffness with time. This test has been carried out by using Monsanto Tensometor 10 instrument, showed in **Figure 5**, which the same instrument used in tensile test before. Dumbbell specimen used in the relaxation test is shown in **Figure 1a.**

The procedure started by holding both ends of the sample via clamps of the instrument. As in tensile test the effective length of the sample is the distance between the two holders which fixed at length (25 mm), then fed the instrument by input data which like, thickness, width of the sample and strain rate. Strain rate was fixed at 200 mm/min for all experiment duration. After completing all those steps the tensile stress was applied and continued till the deformation reached 200% and then stopped. The decreasing in stress was recorded by using video camera. The period of recording the data were continued for 5 minutes and the values taken were used for drawing the relationship between time and stress. Same steps were repeated for each blend used in this work. All the above steps and procedures were repeated for deformations 150%, 100% and 50%.

2.8 Cyclic Loading Tests

In order to validate the cyclic tension test, uniaxial stretching experiments are performed at room temperature. These uniaxial cyclic tests are conducted on dumbbell specimens shown in **Figure 1** under controlled strain, for different strain rate, three loading cycles at constant strain level were carried out for all kind of blends. Machine test was used for this process, show in **Figure 6**, which is Testometric AX-M500-25kN. Each test can be managed by using a computer connected to the machine which fully control the test using winTest™ Analysis universal testing software. This is a multi-functional and fully customizable software package that supports all industry standards including ISO, ASTM and BS EN specifications. Test specifications supported include tensile, compression, flexural, peel, tear, burst, adhesion, shear, cyclic and hardness. Additional flexibility is provided by user-defined multistage step testing for highly specialized testing requirements. Data input to the program to perform the test are specimen length, width, thickness, strain level and strain rate.

Results obtained from the test were force and elongation. The engineering stress was calculated by dividing the force to original area.

Depending on strain value, the stretch was calculated as follows:

$$\lambda = 1 + \frac{\Delta l}{l_o} \tag{3}$$

where λ is the stretch, Δl is the strain and l_o is the original length.

3. RESULTS AND DISCUSSION

The recorded values were used later to draw the stress-stretch curve. The shapes of the curves are shown in **Figure 7 through 9.**

From the above curve it can be notes that the tensile strength for three blends increased with strain rate increasing, but when the strain rate reached high value about 500mm/min the tensile strength tend to decline to lower value.

Curves in **Figures 10 through 12** show the relationship between stress and time for different blends with different deformations which represent the relaxation phenomena.

Three cyclic curves for three blends were plotted at strain rate 200mm/min as show in **Figure 13 through 15.**

4. CONCLUSIONS

The objective of this research is to determine the deformation and viscoelastic behavior for filled elastomeric material under mechanical and cyclic tests and study the effect of the variation of strain rate. Another purpose is to study the effect of the different three types of filled carbon black (N326, N375 and N660) on the mechanical and dynamic behavior of the rubber.

Hardness test for three blends gave results, showing that the hardness for the blend with carbon black N375 is more than that in blend with carbon black N326 which in turn more than hardness in blends with carbon black N660. The reason for that is the surface area for carbon black N357 is larger than the surface area for carbon black N326 which in turn has a surface area larger than that in carbon black N660. The higher amount of surface area gives a high degree of cross linked. The degree of cross-linking has a great effect on the hardness of the elastomer. Also, it has the same effect on the compression set and the specific gravity

Material tension was performed on three blends. The material tension tests were done in order to characterize material stiffness and strength according to strain rate. All tests were done in room temperature and for deformation 200%. In general, and for all strain rates blend with carbon black N375 is stiffer than the others. Also blend with carbon black N326 is stiffer than blend with carbon black N660 till 100% strain, contrary for strain 100 to 200% blend with carbon black N660 is stiffer than the blend with carbon black N326.

All three blends became stiffer when the strain rate increased. For blend with carbon black N375 the stiffness increase from 10 to 100 mm/min, and it became more compliant when the strain rate increased from 100 to 500 mm/min. For blends with carbon black N326 and N660 stiffness increase from 10 to 200 mm/min and it becomes more compliant when the strain rate increased from 200 to 500 mm/min. It was proposed that the increase in stiffness at high rates of loading resulted from the lack of relaxation time in the chain molecules [Imaoka 2008] and due to strain –induce crystallization and the time is insufficient under high strain rate [Ali et.al 2010].

Stress relaxation test was carried out for three blends at room temperature and strain rate (200 mm/min) with different deformations (50, 100, 150 and 200% strain). Relaxation at 200% strain, Filled compounds containing finer carbon black, i.e., blend with carbon black N375 showed higher initial relaxation stress which drops more rapidly as time passes, which subsequently stabilized. This attributed to the loss of viscous motions which are related to carbon black interactions trapped in the cross-links and cannot be broken down [Hussain 2005].

Filler aggregates in a polymer matrix have to form a filler network. During dynamic strain, if the filler network cannot be broken down, the elastic modulus would increase substantially due to the rubber trapped in the network [Meng-Jiao 1998]. It was found that the amount of trapped rubber increases with filler loading and low hysteresis. When the network of the filler breakdown and reformation happened the additional energy dissipation occurs and then higher hysteresis would be expected during cyclic strain. That exactly occurred for blend with carbon black N375 because it had higher filler network.

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Table 1: The recipe of the blend used in tread of farm tire

	Quantity in gm	***PHR****
SBR 1502	**85.5**	**100**
SBR 1712	**142.5**	
BR cis	**56.4**	
ZnO	**7.2**	**2.53**
Stearic acid	**4.2**	**1.47**
6PPD	**5.7**	**2**
TMQ	**3**	**1.05**
Wax	**5.7**	**2**
Paraffin oil	**56.7**	**19.93**
Sulfur	**4.5**	**1.58**
MBS	**3**	**1.05**
Carbon Black N375	**183.6**	**64.55**
Reclaimed Rubber	**42**	**14.76**

***** Parts per Hundred Rubber

Table 2: Summarize Rheometer tests results for the three blends

	ML N-m	MH N-m	Ts2 m.m	t90 m.m
Blend with N375	1.169	4.210	0.88	2.47
Blend with N326	0.962	3.488	0.95	2.84
Blend with N660	0.766	3.237	0.99	2.73

Table 3: I.R.H.D hardness test results

Blend with carbon black	N375	N326	N660
Hardness	57	57	51

Table 4: Compression set for three blends

Blend with different carbon black	Original thickness (t_o) mm	Final thickness (t_i) mm	Compression set (CA)
N375	11.89	11.14	6.30
N326	11.86	11.15	5.986
N660	11.85	11.23	5.23

Table 5: Specific gravity test results

Blend type	N375	N326	N660
Specific gravity	1.131	1.122	1.112

(*a*) (*b*) (*c*)

Figure 1: **(***a***)** dumbbell specimens, (*b*) compression set specimen, (*c*) hardness and density specimens

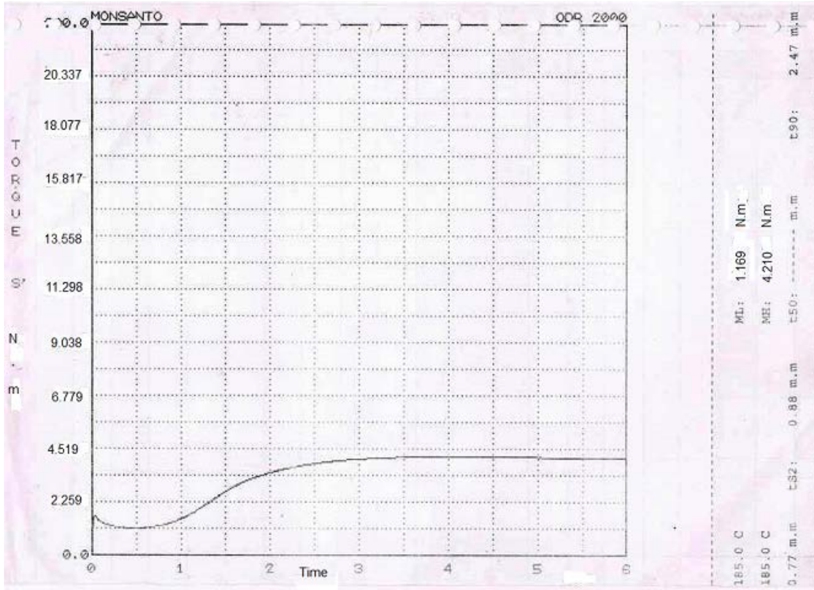

Figure 2: Rheograph for blend with N375

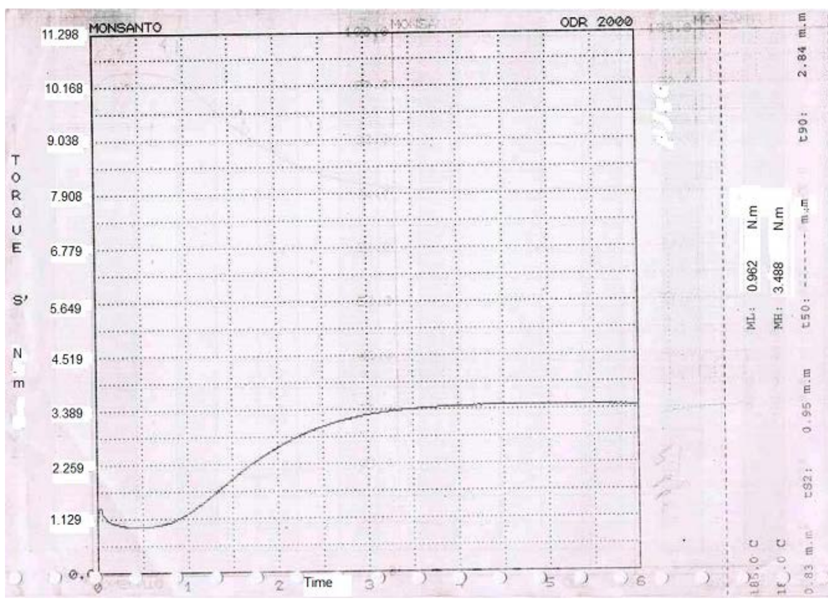

Figure 3: Rheograph for blend with N326

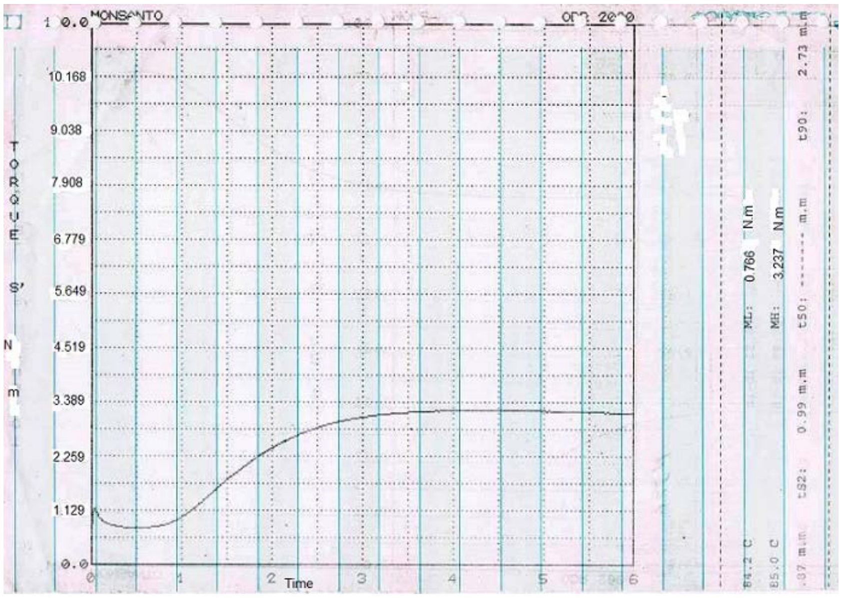

Figure 4: Rheograph for blend with N660

Figure 5: Tensile test instrument, Monsanto Tensometer 10

Figure 6: Cyclic tension test machine Testometric AX-M500.

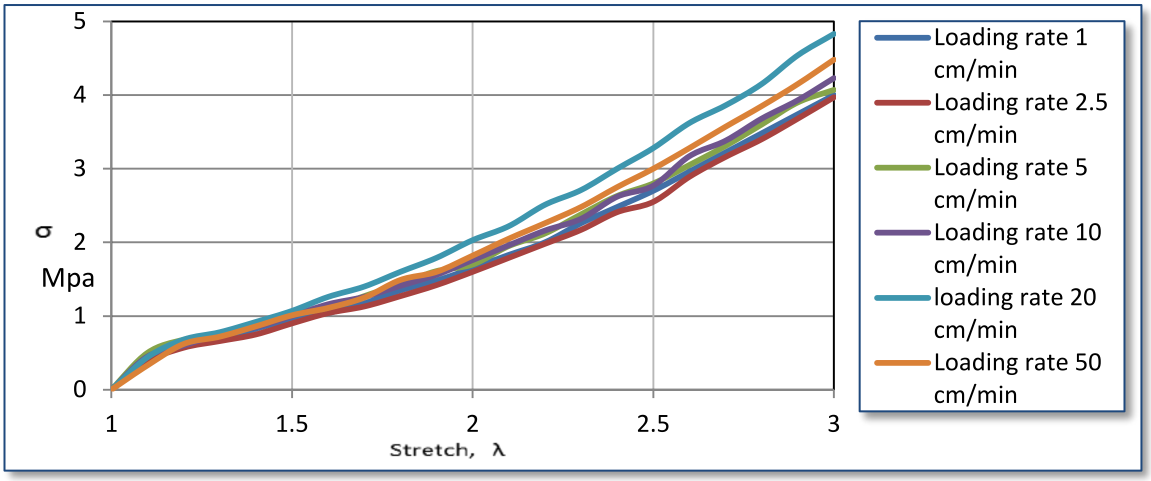

Figure 7: Stress-strain relationship for blend with N375 at different strain rate

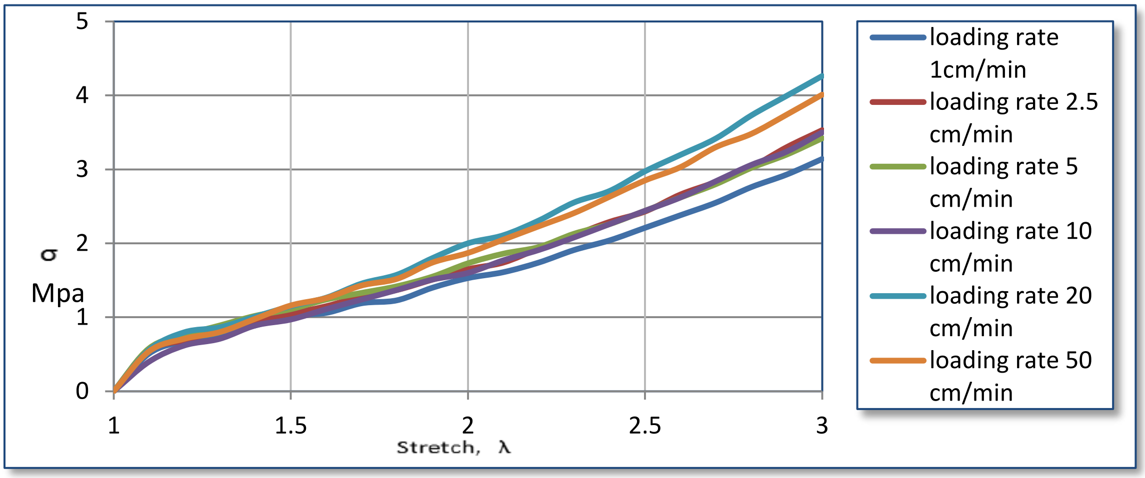

Figure 8: Stress-strain relationship for blend with N326 at different strain rate

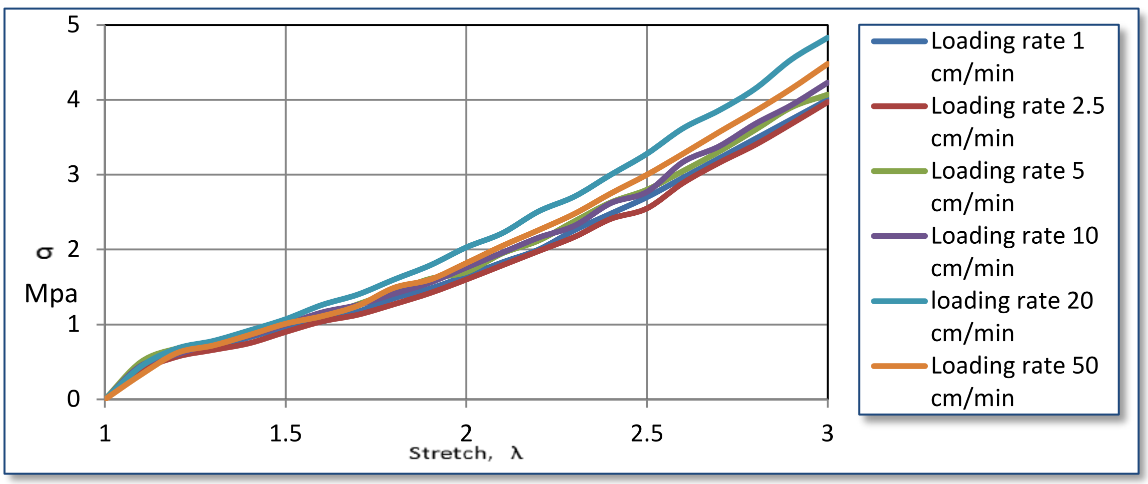

Figure 9: Stress-strain relationship for blend with N660 at different strain rate

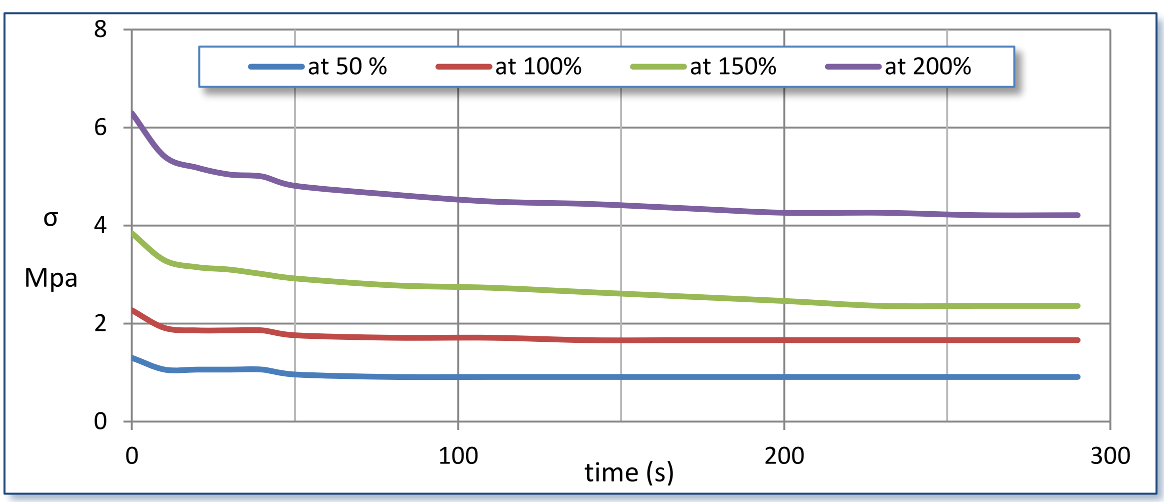

Figure 10: Stress relaxation for blend with carbon black N375 at different deformation

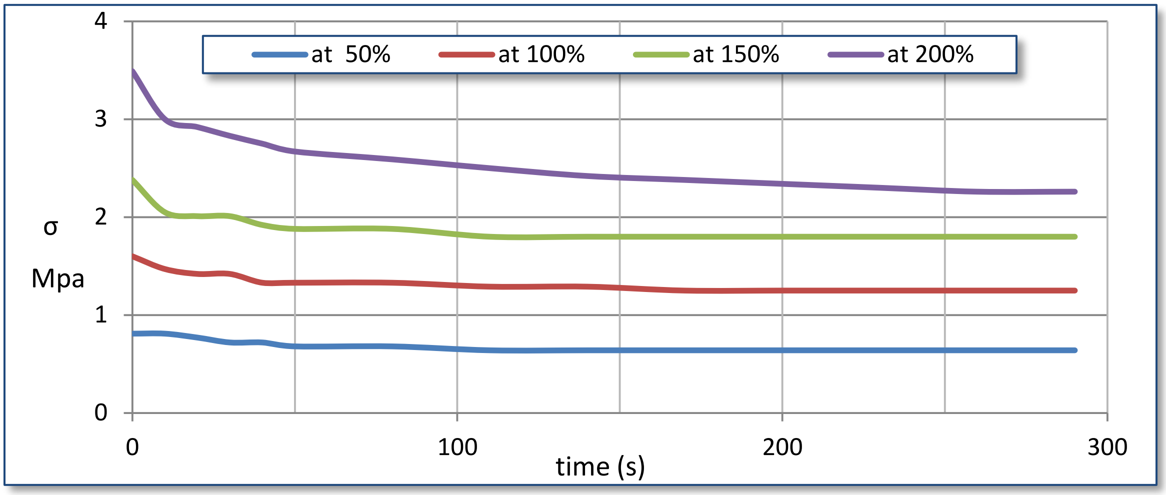

Figure 11: Stress relaxation for blend with carbon black N326 at different deformation

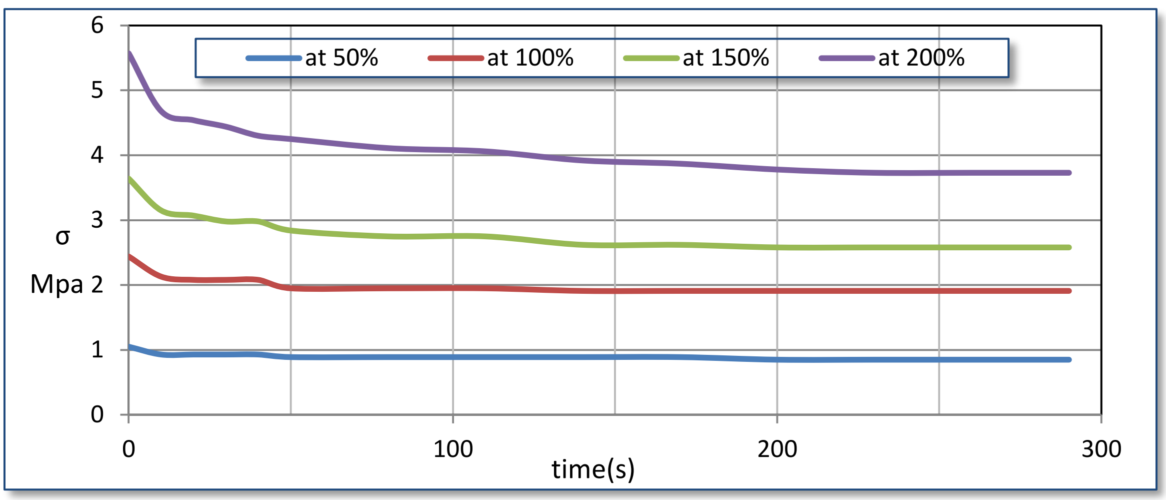

Figure 12: Stress relaxation for blend with carbon black N660 at different deformation

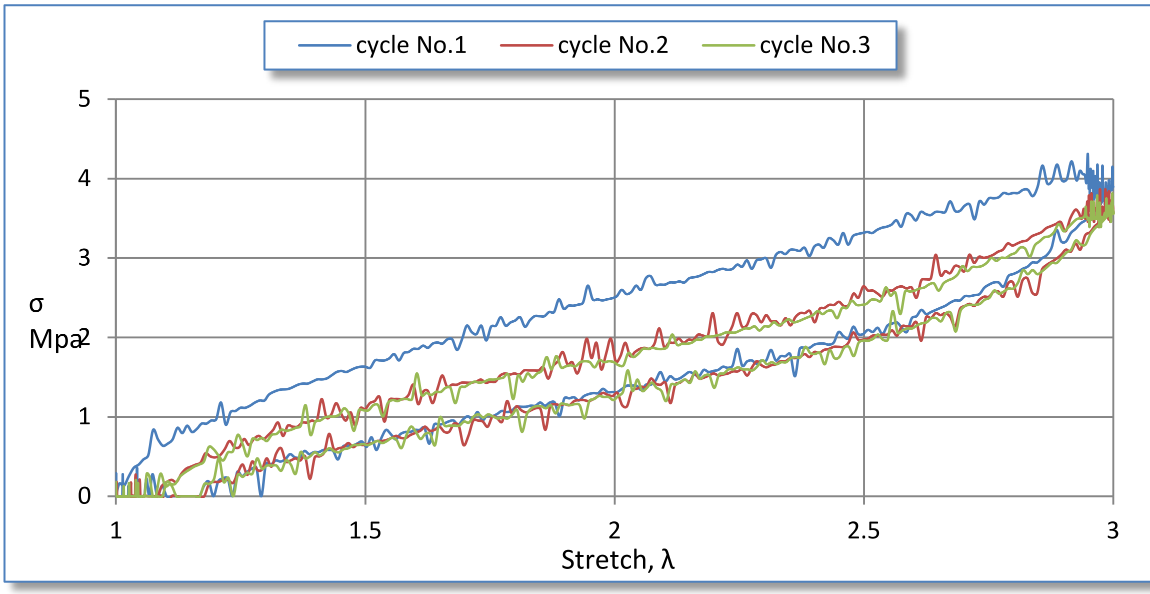

Figure 13: Three cycles for blend with carbon black N326 at strain rate 200 mm/min.

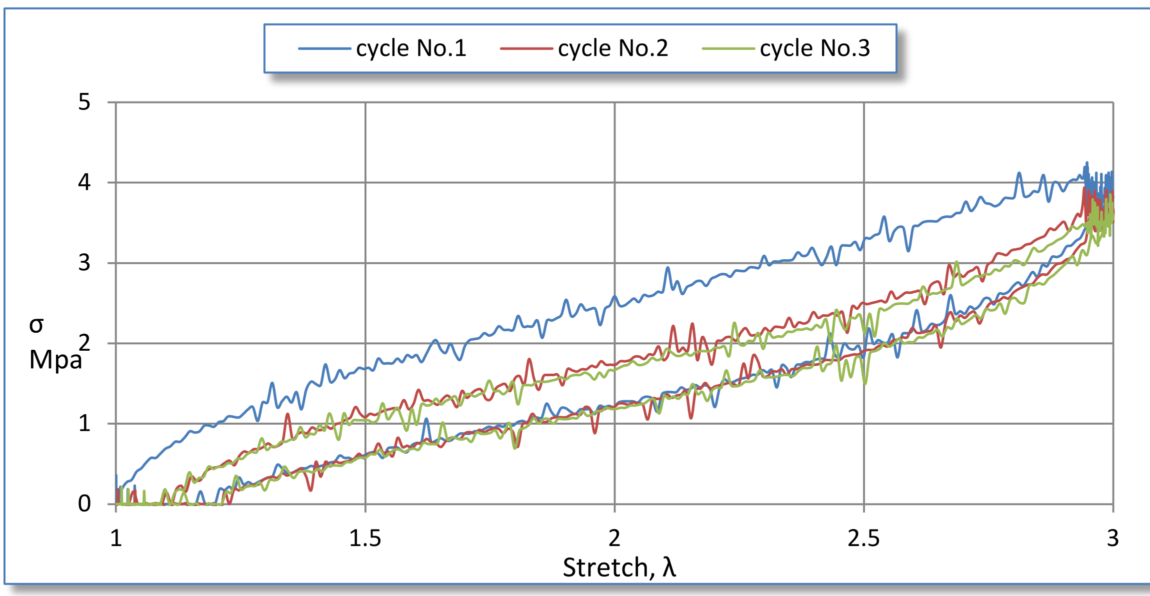

Figure 14: Three cycles for blend with carbon black N375 at strain rate 200 mm/min.

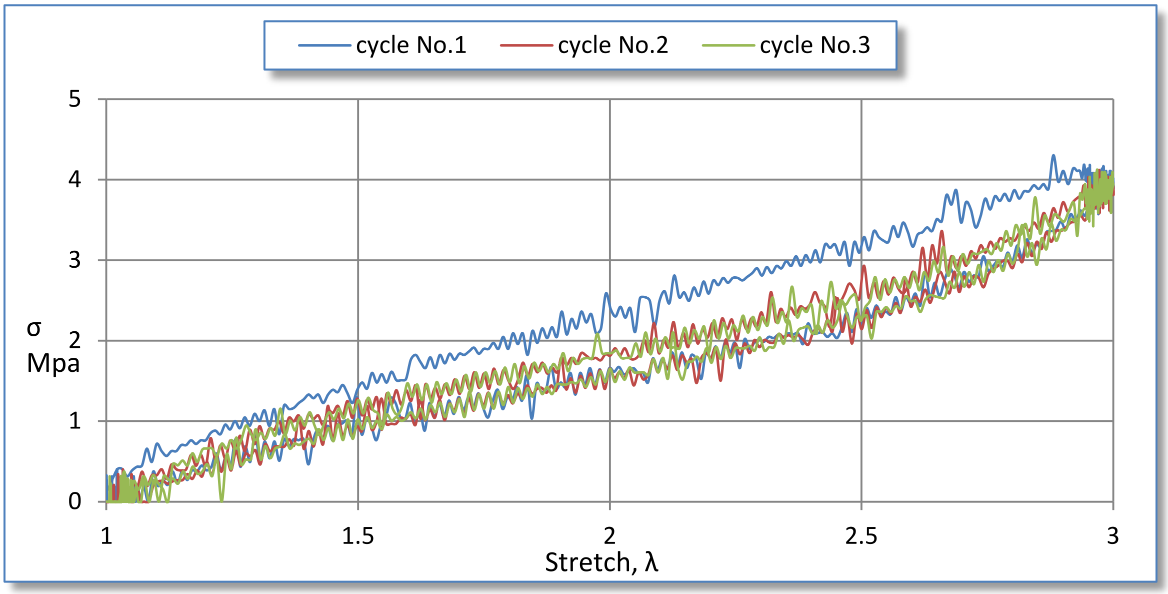

Figure 15: Three cycles for blend with carbon black N660 at strain rate 200 mm/min.