

## CHAPTER 2 : EXPERIMENTAL

### 2.1 Materials

The materials used in the compound formulation as well as their sources are shown in Table 3.

<b>Material/Ingredient</b>	<b>Supplier</b>
SMR10	Kota Trading
ISAF-N220	Cabot (M) Sdn. Bhd.
Processing oil, Shellflex 250MB	Shell Eastern Petroleum
Silica	Degussa
Silane coupling agent, Si-69	Degussa
PEG	BASF, Germany
Zinc oxide	Approfit Zinc Oxide
Stearic acid	Acidchem (M) Sdn. Bhd.
6PPD	NOCIL, India
Sulphur	Taiko Bleaching Earth
TBBS	Uniroyal Chemical

*Table 3 : List of material and its supplier*

The grade of carbon black used was ISAF-N220 with a mean particle diameter of 21 nm and surface area of 111 m<sup>2</sup>/g. The silica used was an amorphous hydrated type with a surface area of 126 m<sup>2</sup>/g. The silane coupling agent, bis-(3-triethoxysilylpropyl)-sulfane, also known as Si-69, was in the form of yellowish liquid. The PEG used was of an average molecular weight of 4000 as stated in the material specification.

## 2.2 Compound Formulations

The compound used in this study was based on a typical truck tread compound. There were two sets of experiments conducted here. The first set of experiment was to study the effects of varying the level of PEG in a silane-modified-silica compound with 1 pphr silane coupling agent, Si-69. The compound formulations are summarized in Table 4. The second set of experiment was to study the effect of varying the level of PEG in an unmodified silica compound without Si-69. The compound formulations are summarized in Table 5.

In both sets of experiment, the level of PEG was varied at 0, 0.5, 1.5, 3 and 5 pphr. The loading of silica and carbon black remains the same at 12 and 40 pphr respectively for the two sets of experiment mentioned above.

A control compound was also mixed containing only carbon black in the absence of silica, silane coupling agent and PEG. The loading of carbon black used was the total filler loading (12 pphr silica + 40 pphr N220) of the experimental compound mentioned earlier. It was used for physical properties comparison of a silica-carbon black-filled compound to that of a carbon black-filled compound. The compound formulation is shown in Table 6.

*Table 4 : Compound formulations to study the effects of PEG in a silane-modified-silica compound with 1 pphr Si-69*

<b>INGREDIENTS</b>	<b>B/1</b>	<b>B/2</b>	<b>B/3</b>	<b>B/4</b>	<b>B/5</b>
SMR 10	100	100	100	100	100
ZnO	3	3	3	3	3
Stearic Acid	2	2	2	2	2
6PPD	1	1	1	1	1
Silica	12	20	20	20	20
Silane coupling agent, Si-69	1	1	1	1	1
<b>PEG</b>	-	<b>0.5</b>	<b>1.5</b>	<b>3</b>	<b>5</b>
N220	40	40	40	40	40
Processing oil, Shellflex 250MB	4	5	5	5	5
TBBS	1.4	1.4	1.4	1.4	1.4
Sulphur	1.5	1.5	1.5	1.5	1.5
<b>TOTAL</b>	<b>165.9</b>	<b>166.4</b>	<b>167.4</b>	<b>168.9</b>	<b>170.9</b>

*Note : All ingredients are quoted as part per hundred of rubber (pphr)*

*Table 5 : Compound formulations to study the effects of PEG in an unmodified silica compound (without Si-69)*

<b>INGREDIENTS</b>	<b>C/1</b>	<b>C/2</b>	<b>C/3</b>	<b>C/4</b>	<b>C/5</b>
SMR 10	100	100	100	100	100
ZnO	3	3	3	3	3
Stearic Acid	2	2	2	2	2
6PPD	1	1	1	1	1
Silica	12	20	20	20	20
Silane coupling agent, Si-69	-	-	-	-	-
<b>PEG</b>	-	<b>0.5</b>	<b>1.5</b>	<b>3</b>	<b>5</b>
N220	40	40	40	40	40
Processing oil, Shellflex 250MB	4	5	5	5	5
TBBS	1.4	1.4	1.4	1.4	1.4
Sulphur	1.5	1.5	1.5	1.5	1.5
<b>TOTAL</b>	<b>165.9</b>	<b>166.4</b>	<b>167.4</b>	<b>168.9</b>	<b>170.9</b>

*Note : All ingredients are quoted as part per hundred of rubber (pphr)*

Table 6 : All black control compound formulation

INGREDIENTS	Control
SMR 10	100
ZnO	3
Stearic Acid	2
6PPD	1
Silica	-
Silane coupling agent, Si-69	-
<b>PEG</b>	-
N220	52
Processing oil, Shellflex 250MB	4
TBBS	1.4
Sulphur	1.5
<b>TOTAL</b>	<b>164.9</b>

Note : All ingredients are quoted as part per hundred of rubber (pphr)

## 2.3 Preparation of Compounds

### 2.3.1 Weighing of Ingredients

The ingredients used were weighed as accurate as possible. Rubber and carbon black was weighed up to  $0.1 \pm 0.05$  gram while other ingredients were weighed up to  $0.01 \pm 0.005$  gram.

The ingredients in the compound formulation were divided into the following five groups for ease of incorporation during mixing :

- i. Rubber
- ii. Silica, Si-69 and PEG
- iii. Carbon black and processing oil
- iv. Activators and antiozonants
- v. Curatives and accelerator

The individual weight of ingredients were calculated prior to weighing. Due to the small amount of sulphur and accelerator, the effect on batch factor of a laboratory scale internal mixer is negligible. Therefore, calculations for the masterbatch and final mix were carried out together. An example of the calculation for compound B/2 is shown in Table 7.

*Table 7 : An example (Compound B/2) of calculation of ingredient weight for the internal mixer*

<b>Ingredients</b>	<b>pphr</b>	<b>Specific gravity (Sg)</b>	<b>Volume cm<sup>3</sup></b>	<b>*Weight used g</b>
SMR 10	100	0.92	108.696	817.85
Silica	12	1.95	6.154	98.14
Si-69	1	1.095	0.913	8.18
PEG	0.5	1.217	0.411	4.09
N220	40	1.8	22.222	327.14
Shellflex 250MB	4	0.875	4.571	32.71
Zinc oxide	3	5.5	0.545	24.54
Stearic acid	2	0.85	2.353	16.36
6PPD	1	1	1.000	8.18
TBBS	1.4	1.27	1.102	11.45
Soluble sulphur	1.5	2.1	0.714	12.27
<b>Total</b>	<b>166.4</b>		<b>148.682</b>	<b>1,360.91</b>

*Note :*

*\* Adjusted weight of individual ingredients calculated due to the fill factor effect of the internal mixer. The detail computation is shown in the following page.*

### Calculations :

Volume of each ingredient = pphr of the ingredient / Sg of the ingredient

(Example : Volume of SMR 10 =  $100 / 0.92 = 108.696 \text{ cm}^3$ )

Sg of rubber compound = Total pphr / Total volume

=  $166.4 / 148.682$

= 1.119167

Fill factor = 0.76

Volume of mixer =  $1600 \text{ cm}^3$

Batch weight = efficient volume x Sg of rubber compound

= fill factor x volume of mixer x 1.119167

=  $0.76 \times 1600 \times 1.119167$

= 1360.907072 g

Batch factor = Batch weight / Total pphr

=  $1360.907072 / 166.4$

= 8.178528

Weight of each ingredient = pphr of the ingredient x Batch factor

(Example : Weight of SMR 10 =  $100 \times 8.178528 = 817.85 \text{ g}$ )

Calculated discharge weight = Total weight of all ingredients



### 2.3.2 Mixing of Masterbatch and Final Mix

Mixing was carried out in an internal mixer, Farrel BR1600. The volume of the mixing chamber is 1.6 litre and the fill factor used was 0.67. There are two mixing sequence used i.e. for the masterbatch and final mix. The mixing sequence used are shown in Table 8 and 9 respectively. All compounds were discharged by time after completing the entire mixing sequence.

The mixing sequence used for the masterbatch (Table 8) was designed to ensure that the discharge temperature of the compounds are approximately 150°C. This was to ensure that the chemical reaction for in-situ modification of silica with silane coupling agent takes place <sup>10, 11, 15, 36, 37</sup>.

The dump temperature and weight were recorded when the masterbatch was discharged from the internal mixer. It was then milled into sheets on a two-roll mill (Farrel Polymill 150P). All masterbatches were left to cool for a minimum two hours before the final mix was carried out.

Step	Ram Down Time (seconds)	Rotor Speed (rpm)	Cylinder pressure (bar)	Step Instruction
1	0	60	2.00	SMR 10
2	60	60	2.00	Silica, Si-69, PEG
3	120	60	2.00	N220, Processing oil
4	180	60	2.00	ZnO, Stearic acid, 6PPD
5	240	60	2.00	Sweep
6	270	60	2.00	Discharge

Table 8 : Mixing sequence of the masterbatch

The mixing sequence used for the final mix (Table 9) was designed to ensure that the dump temperature is not too high to prevent scorching of the compound. The dump temperature and weight were recorded when the final mix was discharged from the mixer. It was then milled into sheets on a two-roll mill (Farrel Polymill 150P). Weight loss of the compounds were kept below 1% to ensure there was no excessive loss of ingredients during mixing. Weight loss is the percentage difference between the calculated weight and the discharged weight.

Step	Ram Down Time (seconds)	Rotor Speed (rpm)	Cylinder pressure (bar)	Step Instruction
1	0	45	2.00	Masterbatch
2	60	45	2.00	Sulphur, TBBS
3	120	45	2.00	Discharge

Table 9 : Mixing sequence of the final mix

#### 2.4 Preparation of Moulded Test Piece

Moulding of test pieces were carried out using a hot press, Marushichi Hydraulic Press. Moulding temperature used was 150°C. Cure time was taken as  $t_{95}$  read from the rheometer graph obtained from 2.5.

The minimum time between vulcanization and testing shall be 16 hours as stated in ISO 1826. Conditioning of test pieces were carried out in a temperature and humidity controlled room prior to testing. The standard conditions for conditioning the samples are  $23 \pm 2^\circ\text{C}$  and  $60 \pm 10\%$  relative humidity.

## 2.5 Determination of Vulcanization Properties

The vulcanization properties were determined using the Monsanto Moving Die Rheometer, MDR 2000. Tests were conducted in accordance to test procedure ISO 6502:1991. The rheometer type is a torsion shear curemeter with an oscillation amplitude of 0.5° and oscillation frequency of 1.7 Hz.

The sample weight used was approximately 5 grams. The test temperature used was 150°C and the test was stopped once reversion was observed. Only one test was conducted on each compound. The curing characteristics were determined from the rheometer graph. All calculations were computerised.

In the curemeter, the compound test piece is contained in a sealed cavity heated to a constant temperature. A cyclic strain is then applied to the rubber sample. What is recorded is the increase in compound stiffness, as a function of time, which appears as a vulcanization curve. A sample of a rheometer chart is shown in Appendix 1.

The following parameters were obtained from the vulcanization curve.

- i. ML, minimum torque, which indicates the onset of crosslinking
- ii. MH, maximum torque, which indicates the maximum crosslinking attainable by the compound before reversion takes place
- iii.  $t_{s2}$ , time to 2 units of torque increase above ML, which indicates processing safety
- iv.  $t_{cy}$ , cure time in minutes to y percentage of full torque development where

$$t_{cy} = y/100 (MH-ML) + ML$$

The time to achieve  $t_{95}$  read from the vulcanization curve was used for moulding test pieces.

## 2.6 Determination of Rheological Properties

The rheological properties, Mooney viscosity and Mooney scorch, were determined using Monsanto Mooney Viscometer, MV 2000. Tests were conducted in accordance to test procedure ISO 289/2:1994. The viscometer is equipped with a shearing-disc. For both measurements, a large rotor was used. The sample weight used was approximately 25 grams. The test piece was pre-heated for 1 minute in the cavity prior to testing. Only one test was conducted on each compound.

During testing, the rotor turns at a constant rate of  $0.209 \pm 0.002$  rad/s inside the close cavity containing the test piece which is sandwiched between the flat surfaces of the rotor and the walls of the chamber. The torque required to rotate the rotor is monitored by a suitable force transducer.

### 2.6.1 Mooney Viscosity

Viscosity is the resistance to plastic deformation or flow and defined as shear stress/ shear rate. It gives an indication of the amount of energy to mix and form the rubber. The test temperature used was 130°C. Results of Mooney viscosity were expressed as follows.

60 ML (1+4) 130°C

where

60 - rubber having a viscosity of 55 Mooney units

L - indicates the use of a large rotor in this test

1 - pre-heating time of 1 minute prior to the start of the test

4 - running time of 4 minutes and when the viscosity was read

130°C - temperature at which the test was conducted

## 2.6.2 Mooney Scorch

The scorch time is used to determine pre-vulcanization characteristics of a compound. It gives an indication of how long a rubber can be maintained at the processing temperature and still be subsequently processed. The rubber compound must have adequate scorch safety so that it can flow and be shaped before the onset of vulcanization. Mooney scorch was determined as the time when the Mooney units rises by 5 units above the minimum viscosity. The test temperature used was 130°C. A sample of the chart obtained for Mooney scorch is shown in Appendix 2.

## 2.6.3 Cure Rate

Cure rate is a measure of the rate of curing of the compound after the processing safety period had lapsed. Cure rate was obtained from the Mooney scorch curve conducted at 130°C. It is the time required for the torque to rise 30 Mooney units, i.e. ( $t_{35} - t_5$ ). The shorter is the cure rate, the faster is the curing process.

## 2.7 Determination of State of Cure

### 2.7.1 Crosslink Density

Crosslink density is the amount of crosslinks present in the compound per gram mol of rubber hydrocarbon. It is important to distinguish between the apparent concentration of physically manifested crosslink ( $n_{\text{phys}}$ ) and the concentration of chemically discrete crosslinks ( $n_{\text{chem}}$ ). In sulphur vulcanizates, the latter are covalent crosslinks comprised of C-S and S-S bonds; the former include these but also include other forms of inter-chain interaction like chain entanglements <sup>5</sup>.

Methods for determining concentrations of crosslinks have up to now been physical ones based on the statistical theory of rubber-like elasticity. Therefore, a correlation of  $n_{chem}$  and  $n_{phys}$  has to be obtained based on correction factors <sup>5</sup>. In this study the correction for crosslink density was not carried out as the reinforcing fillers consists of both silica and carbon black. The correction factor was not known yet.

The test piece used was a thin strip having a dimension of 5 mm x 36 mm x 1.5 mm. An average of three test results were computed for each compound. The procedure for conducting crosslink density measurement was detailed as follows.

- i. The test piece was weighed and recorded as  $W_o$ .
- ii. The test piece was immersed in n-decane in a test tube and conditioned in a water bath at 25°C for 24 hours to attain equilibrium swelling. The swollen test piece was weighed and recorded as  $W_s$ .
- iii. The test piece was left in the oven at 50°C for 24 hours. The deswollen test piece was weighed and recorded as  $W_d$ .

Crosslink density was calculated using the Flory-Rehner equilibrium swelling measurement as shown below <sup>5, 38, 39, 40</sup>. Data showing that the test pieces had attained equilibrium swelling after 24 hours is found in Appendix 3.

$$-\ln(1-v_r) - v_r - \chi v_r^2 = 2 \rho V_o [X]_{phys} v_r^{1/3}$$

- where
- $v_r$  = volume fraction of rubber in the swollen gel at equilibrium
  - $\chi$  = parameter characteristic of interaction between rubber network and swelling agent, n-decane = 0.42 (for NR in n-decane)
  - $\rho$  = density of rubber = 0.92 (for NR)
  - $V_o$  = molar volume of the swelling agent, n-decane = 195.88 cm<sup>3</sup>
  - $[X]_{phys}$  = physically manifested crosslink

### **2.7.2 Differences in Maximum and Minimum Torque, $\Delta(\text{Torque})$**

The difference in the maximum torque (MH) and minimum torque (ML) from the rheometer curve obtained in 2.5 gives an indication of the amount of crosslinks formed in the compound during vulcanization.

$$\Delta(\text{Torque}) = \text{MH} - \text{ML}$$

### **2.8 Determination of Physical Properties**

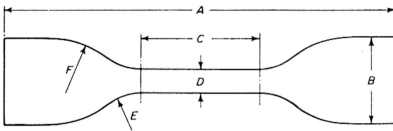
Physical testing of rubber often involves application of a force to a specimen and measurement of the resultant deformation or conversely, application of a deformation and measurement of the required force. The following tests were conducted :

- i. Tensile stress/strain properties
- ii. Hardness
- iii. Resilience
- iv. Tear strength
- v. Heat build-up
- vi. Dynamic mechanical properties
- vii. Cut growth resistance
- viii. Abrasion resistance

### 2.8.1 Tensile Stress-strain Properties

Tensile stress-strain measures the ability of the rubber compound to stretch to several times its original length. Properties obtained were tensile strength, elongation-at-break and modulus.

The tensile properties were measured using an Instron Universal Testing Machine, Model 4301. Elongation was measured using an extensometer. Tests were conducted in accordance to test procedure ISO 37:1994 using Dumb-bell Type 1 test piece. The dimension of the dumb bell is depicted in Figure 12. A gauge length and crosshead speed of 25 mm and 500 mm/min respectively were employed.



<u>Dimension</u>	<u>Type 1 (mm)</u>
A	115 minimum
B	25 ± 1
C	33 ± 2
D	6.0 ± 0.2
E	14 ± 1
F	25 ± 2

Fig. 12 : Dumbell test piece <sup>41</sup>



A pneumatic cutter was used to cut the dumb bell test piece from the tensile slab. The test direction of the test piece was along the grain direction of milling. A total of five dumbbell test pieces were tested for each compound. The value of the median was taken as the stress-strain property of the compound.

The calculations involved in the determination of tensile properties were derived as follows. All calculations were generated by the computer.

$$\text{Tensile strength} = F/A$$

$$\text{Elongation at break} = 100 [(l_1 - l_0) / l_0]$$

$$\text{Modulus} = \sigma/\varepsilon$$

where  $F$  = ultimate load, N

$A$  = original cross-sectional area, mm<sup>2</sup>

$l_0$  = initial gauge length, mm

$l_1$  = ultimate gauge length, mm

$\sigma$  = stress, MPa

$\varepsilon$  = strain

The effect of ageing on tensile stress-strain properties were also conducted. Ageing was carried out according to test procedure ISO 188:1998. The tensile slab was aged at 70°C for 3 days in a normal oven with a rotating carrier.

### 2.8.2 Hardness

Hardness of a rubber implies its resistance to indentation under conditions that do not puncture the rubber. Hardness is expressed as a number referring to the scale of the instrument by which it is measured. Here, results of hardness were expressed as IRHD, *International Rubber Hardness Degree*.

Hardness was determined using Wallace Deadload Hardness Tester. Tests were conducted in accordance to test procedure ISO 48:1994 (Method N). Hardness reading was measured at five different points on one test piece and the value of the median was recorded. Tests were conducted on two test pieces and the average of the median was calculated as the hardness of the compound.

### 2.8.3 Resilience

Rebound resilience is defined as the ratio of the energy of the indenter after impact to its energy before impact expressed as a percentage.

$$\text{Resilience} = \frac{1 - \cos(\text{angle of rebound})}{1 - \cos(\text{angle of fall})} \times 100$$

Rebound resilience was measured using the Wallace Dunlop Tripsometer. Tests were conducted in accordance to test procedure BS903 Pt A8:1990 (Method A). The test pieces were subjected to time and temperature conditioning prior to testing for 30 minutes and 55°C respectively.

During testing, the test piece was held under vacuum in a vertical cavity at a temperature of 55°C. The pendulum was released from an angle of 45° to strike the test piece. The test result was taken after the seventh strike. Tests were conducted on two test pieces and the average was calculated as the resilience of the compound.

### 2.8.4 Tear Strength

Tear resistance in rubber may be described as the resistance to growth of a nick or cut when tension is applied to the cut test piece. Tear strength is defined as the force required to tear a test piece per unit thickness.

Tear strength was determined using the Instron Universal Testing Machine, Model 4301. Tests were conducted in accordance to test procedure ISO 34-1:1994 (Method A). The test piece used was trouser type as shown in Figure 13. It was prepared from the tensile slab using a cutter. A crosshead speed of 100 mm/min was employed during testing. A total of five tests were conducted and the value of the median was recorded as the tear strength of the compound.

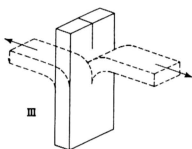


Fig. 13 : Trouser tear test piece <sup>41</sup>

( — Original shape; - - - - - Shape after testing)

### 2.8.5 Heat Build-up

Heat build-up is a measure of heat generation in the compound when subjected to cyclic compression.

Heat build-up was conducted using the Ueshima Compression Flexometer. Tests were conducted in accordance to ISO 4666/3:1982. Each test piece was pre-heated at 55°C for 30 minutes prior to testing. The height of the test piece was measured prior to pre-heating.

During testing, a compressive load of 11 kg and a high-frequency cyclic compression of 4.45 mm stroke were applied to the cylindrical test piece. Temperature of the test chamber was 55°C. The test was conducted for a period of 25 minutes.

The temperature of the test piece during testing was determined by a thermocouple connected to the lower platen. A plot of time-temperature curve was produced throughout the test on the chart recorder. The temperature of the test piece will normally reach a plateau at the end of the test period.

Heat generation of the compound was taken as the difference between the final temperature and the initial temperature of the test piece. The change in the specimen height before and after testing gives an estimate of the degree of stiffening (or softening). Three tests were conducted and the average was calculated as the heat build-up of the compound.

$$\Delta T = T_1 - T_0$$

$$\text{Set after testing} = H_0 - H_1$$

where  $\Delta T =$  Heat build-up, °C

$T_1 =$  final temperature, °C

$T_0 =$  initial temperature, °C

$H_0 =$  initial height of test piece, mm

$H_1 =$  final height of test piece, mm

### 2.8.6 Dynamic Mechanical Properties

Dynamic mechanical properties refer generally to responses to periodically varying strains or stresses. Rubber compound is a viscoelastic material and its response to dynamic stressing is a combination of an elastic response and a viscous response and energy is lost in each cycle. If the rubber compound were an ideal elastic material, the stress would be similarly sinusoidal and in phase with the strain. If the rubber compound were an ideal viscous material, the stress would be  $90^\circ$  out of phase with the strain. However, as rubber is viscoelastic, the stress will not be in phase with the strain but can be considered to precede it by the phase angle  $\delta$  <sup>41</sup>.

It is convenient to consider the stress as a vector having two components, one in phase with the displacement and one  $90^\circ$  out of phase and to define corresponding in phase, out of phase and the resultant modulus <sup>41</sup>. The sinusoidal motion is illustrated in Figure 14 and the vector in Figure 15.

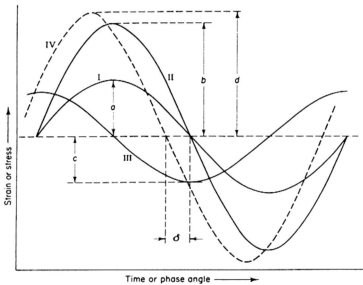


Fig. 14 : Sinusoidal strain and stress cycle. I - strain of amplitude  $a$ ; II - in phase stress of amplitude  $b$ ; III - out of phase stress of amplitude  $c$ ; IV - total stress (resultant of II and III) of amplitude  $d$ ;  $\delta$  is the loss angle <sup>41</sup>

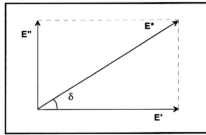


Fig. 15 : Vector stress or strain diagram <sup>41</sup>

The vector modulus in tension mode is defined by <sup>41</sup>:

$$E^* = E' + iE''$$

where  $E^*$  = complex modulus

$E'$  = in phase or storage modulus

$E''$  = out of phase or loss modulus

The storage modulus,  $E'$ , is defined as the stress in phase with the strain in a sinusoidal deformation divided by the strain; it is a measure of the energy stored and recovered per cycle <sup>42</sup>. The loss modulus,  $E''$ , is defined as the stress 90° out of phase with the strain divided by the strain; it is a measure of the energy dissipated or lost as heat per cycle of sinusoidal deformation <sup>42</sup>. The loss tangent, a dimensionless parameter, is a measure of the ratio of energy lost to energy stored in a cyclic deformation where  $\tan \delta = E'' / E'$  <sup>42</sup>.

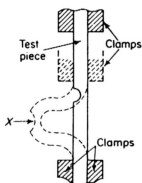
Dynamic mechanical properties were tested using the Rheometrics RSAII Dynamic Mechanical Analyzer. Test were conducted in accordance to in-house test procedure PTL/NSTP02 (Dynamic Temperature Ramp Default Test). The test conditions used were 10Hz frequency, 0.1% strain and a ramp rate of 30°C/min. The test piece was a thin strip having a dimension of 5mm x 36mm x 1.5mm. Tangent delta at 60°C gives an indication of rolling resistance of the compound <sup>43,44</sup>.

## 2.8.7 Cut Growth Resistance

The growth resistance of an insipient cut was determined using the De Mattia Cut Growth Resistance Tester. Tests were conducted in accordance to test procedure ISO 133:1983. The test piece used was approximately 6 inches long, 1 inch wide and 0.25 inch thick with a groove moulded across its width <sup>45</sup>. A cut of an initial length,  $L$ , of approximately 2 mm, was introduced to the test piece prior to testing.

During testing, the test pieces were subjected to flexing. The flex cycle was such that the specimen was bent almost double at the groove, then relaxed and the whole cycle was repeated. This can allow progressively greater strain along the groove and concentrated at the cut. The growth in the length of each test piece was measured after every 2000 cycles. Flexing of the test piece was shown in Figure 16.

A cut length - flexing cycle graph was then plotted for each test piece. The number of cycles for the cut to extend from  $L$  to  $(L+2)$  mm,  $(L+2)$  to  $(L+6)$  mm and  $(L+6)$  to  $(L+10)$  mm were then determined from the graph and recorded for each test piece. A sample of calculation is attached in Appendix 4. Three tests were conducted and the average was calculated as the cut growth resistance of the compound.



*Fig. 16 : Flexing of the test piece during testing <sup>41</sup>  
(X indicates the position of the cut at the groove)*

## 2.8.8 Abrasion Resistance

Abrasion resistance is a measure of the resistance of the compound from being abraded when in contact with an abrasive surface.

This test was conducted using the Zwick Abrasion Tester as shown in Figure 17. Tests were conducted in accordance to test procedure ISO 4649:1985 (Method B). The test piece used was of rotating type. During testing, the test piece was inserted into the sample holder and travels over the surface of the abrasive paper at a rate of 0.32m/second under a load of 10N for a distance of 40m. Upon completion of the test, the sample was removed and weighed to calculate the wear index.

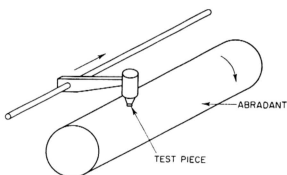


Fig. 17 : Zwick abrasion tester<sup>41</sup>

In the abrasion test, a standard rubber (as stated in clause B.2, Annex B) was tested together with the test piece for each compound. One standard abrasion button was tested together with three test pieces. The average volume loss of the three test pieces was compared against that of the standard rubber to determine the abrasion resistance index (ARI).

$$\text{ARI (\%)} = \frac{\text{Average volume loss of standard rubber}}{\text{Average volume loss of test compound}} \times 100$$



## 2.9 Determination of Regression Analyses

Simple linear regression is the development of a model to exploit the linear relationship between the variable of interest and an independent variable <sup>46</sup>. Linear regression analysis is the process of fitting a straight line through the data points to describe the relationship between the two variables. The best straight line is determined using the least-squared method. It is based on an analysis of the errors in which the sum of squared errors is the smallest.

When investigating the relationship between two variables, the first step should be to produce a scatter plot. A visual inspection can reveal more than a single measure of association. For example, there may be a very good, but non-linear relationship between the two variables - this would not be reflected by the correlation coefficient, which measure linear association. The correlation coefficient is a measure of the amount of 'scatter'; that is the extent to which observations of a pair of variables vary from a straight line relationship <sup>46</sup>.

For the model to be useful for estimation, it must satisfy two conditions. Firstly, the data points must not be too scattered about the line of best fit and secondly, the line must have a significant slope. We determine the degree of scatter by looking at r-squared ( $r^2$ ). The closer the value is to 1, the better the fit. The value of  $r^2$  indicates the percentage of the variability in Y which is explained by the regression line <sup>46</sup>. For example,  $r^2 = 0.91$  indicates that 91% of the variability in the Y-axis is explained by X-axis and the remaining variability may be attributable to other non-related factors.

Linear regression was computed using Lotus 123 spreadsheet. The straight line was expressed as follows :

$$Y = A + BX$$

where

- Y = dependent variable
- X = independent variable
- A = intercept
- B = correlation coefficient