Studies on Dynamic Ageing Conditions for Life Cycle Prediction of Tyre Tread

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Abstract

Rubber material properties and useful life estimation are very important in design procedure to assure the safety and reliability of tire. The Tire tread compound undergo various stringent condition during its application like strain, temperature etc. In general Arrhenius techniques have been used to predict the life of rubber component using static ageing technique. But such techniques shall not reveal actual use of the application. An attempt has been made to simulate thermal ageing along with dynamic condition in order to match with actual application. Testing equipment has been designed and fabricated to suit this requirement of dynamic ageing of dumbbell specimens which closely matches with the real application. Study also deals with determining the changes in physical properties of tire rubber vulcanizates under different dynamic heat ageing condition and thereby predicting life of component. Mixing was carried out in open mill and compounded rubber was vulcanized by compression molding at 160°C based as per rheometric studies. Physico-mechanical and other properties were determined as per requirement for tread rubber compounds. Morphological studies of the fracture samples were done using SEM to study the changes if any, in the micro structure failure. Accelerated heat aging tests under dynamic condition were carried out to predict the life of tire rubber vulcanizates using Arrhenius technique which predicted the life of rubber vulcanizates of 17.01 days for threshold value and activation energy found to be 197.56kJ/mol.

Keywords: Tire tread, Dynamic heat ageing, Life prediction, Arrhenius technique

1. Introduction

Elastomers (natural and synthetic rubber) are amorphous polymers to which various ingredients are added, creating what the rubber chemist refers to as a compound. After heating and reaction (vulcanization), these materials become "rubber". While they are elastic and rubbery, they also dissipate energy because of their viscoelastic nature. Their strength is high, especially under shear and compressive deformations. But, as with any mechanically loaded component, failure can occur as a result of fatigue. Thus the long-term durability of rubber has to be predictable.Throughpolymerization, a long-chain molecule is created (the primary structure of any polymeric material) from simple molecules, known as monomers. Polymer molecules can be either amorphous rubbery, amorphous glassy or crystalline materials. Elastomers are typically amorphous polymers with their molecules in random motion. Thus, they are essentially viscous liquids. By bonding (crosslinking) the long molecules together at relatively large distances, a flexible molecular network is created with the component molecular strands still in rapid motion. But the material has now a fixed shape and size – it has become a soft elastic solid. As an engineered product, the material at some point will be subject to an external force. When a solid body is deformed, an internal reactive force called stress, acting across a unit area, tends to resist this deformation. The measure of deformation is called strain [10].Rubber is unique in being soft, highly extensible, and highly elastic. Considering rubber as an engineering material, we can employ the term shear modulus $G = N k T$, where N is the number of molecular network chains in a unit volume, k is Boltzmann's constant, and T is temperature in Kelvin. Many rubbery materials have a similar modulus G or hardness at equivalent temperatures above their glass transition temperature. There are two basic mechanical properties of any material: elastic modulus (stiffness) and damping (ability to dissipate energy). Typically, some energy is lost (converted to heat) in any deformation.

No material is perfectly elastic – they all exhibit some dissipation of the energy expended in deforming them. When subjected to a stress cycle, the stress strain curve is a hysteresis loop instead of a reversible curve, with the

deformation lagging behind the stress as the stress increases and again as the stress decreases. The difference between the loading and unloading curves reflects mechanical energy lost in internal dissipation processes, such as viscous flow or internal bond-breaking induced by stress. All elastomers show some viscous behavior. While this is desirable for shock damping applications, many industrial problems are a consequence of an excessive viscous response. Such common phenomena as stress relaxation, creep, compression set, and unrecovered deformations in general, mechanical irreversibility and energy losses during a deformation cycle ("hysteresis"), limited rebound, heat generation, and temperature rise during flexing are all manifestations of the viscous properties of elastomers. These processes are usually strong functions of test temperature and frequency of oscillation.

Life of an elastomer products depend upon its aging resistance and also other environmental / application conditions. During the ageing process certain chemical and/or physical changes occurs in its microstructure which in turn result in changes in its mechanical properties. "Ageing" is a collective term for alteration in properties of materials that occur when it is used or stored for long period of time that led to partial or complete degradation. Life prediction of rubber compound is very complex in nature as the rubber degradation and the rate of degradation depends on various factors such as temperature, chemical environment, loading conditions and type of rubber and many more. Design of rubber components against fatigue failure is one of the most critical issues and is challenging also. Life prediction and evaluation under simulated conditions closure to the real application are essential. The present work deals with the effect of different simulated conditions such as strain, temperature, time of the tire rubber vulcanizates subjected to dynamic ageing under constant frequency of 100 cpm to predict the life of tire rubber vulcanizates by Arrhenius technique.

2. Materials and methods
2.1. Materials
NR (RMA 1X),PBR 1220, SBR 1502 , HAF – Carbon black – N330, Stearic acid, ZnO, Sulphur,N-cyclohexyl benzothiazyl sulfonamide (CBS), N-1,3-dimethyl-N-phenyl-p-phenylenediamine (4020), TDQ, Wax were received from Samir Suppliers, Wadala, Mumbai .

2.2. Compounding
Mixing of rubber was carried out in two roll mill at a friction ratio of 1:1.2. The compounding ingredients were added followed by the mixing and the mixing time as per procedure given in ASTM D 3184-89 (2001). The formulations are given in *table 1*.

Table 1: Formulation of tire tread compound

Compounding ingredients	Phr
Natural Rubber	70
Styrene Butadiene Rubber 1502	20
Butadiene Rubber 1220	10
Zinc Oxide	5
Stearic acid	2
HAF black	60
Aromatic oil	5
1,2-Dihydroxyquioline	2
Wax	1
Sulphur	2.5
N-cyclohexyl benzothiazyl sulfonamide	0.8
N-1,3-dimethyl-N-phenyl-p-phenylenediamine	2

2.3. Rheometric Study
The cure characteristics of the compound were determined using Monsanto Rheometer (R 100) at 160°C for 30 min. From the rheometric study minimum torque, maximum torque, time to achieve 90% cure (t90), scorch time were calculated and noted in *table 2*.

Table 2: Rheometric Characteristics at 160°C

Properties	Observed values
Maximum Torque (lbs.in)	82.37
Minimum Torque (lbs.in)	9.95
Optimum cure time t90 (min)	5.44
Scorch time t2 (min)	2.55

Vulcanization of test slab was moulded in an electrically heated hydraulic press at 160°C and 200 kg/cm^2 pressure to their respective cure time (t90). Vulcanized test slab (200mm × 200mm × 2mm) were made by compression molding technique.

2.4. Physio-Mechanical Properties
Samples for the test were cut from the molded sheet. Tensile strength, modulus at 300% strain and elongation at break were carried out according to the ASTM D412 using dumbbell shaped specimens in Zwick universal tensile testing machine (model-1445) tested at crosshead speed of 500 mm/min; tear strength was determined as per ASTM D624. Abrasion resistance index is calculated as per IS 3400 part 3. Hardness was measured by using Shore-A Durometer model as per ASTM D2240.

Table 3: Physical Properties of Compound Cured at 160°C (Before Ageing)

Properties	Observed Values
Tensile Strength (kg/cm^2)	249
Elongation at break (%)	490
Modulus at 300 % strain	146
Hardness (Shore A)	71
Abrasion Resistance (ARI)	112
Tear Strength (kg/cm)	66

Most of the properties required for Tread Rubber compound including Physical properties, abrasion Resistance Index were evaluated and tabulated in *table 3* and found in line with general requirements.

2. 5 Dynamic life prediction – Methodology

2.5.1 Equipment Description
A special testing equipment is designed and fabricated along with an attached ageing oven and fixtures to mount the dumbbells as shown in *figure 1*. A set of dumbbells of rubber vulcanizate were mounted in the fixtures at a desired rate of strain in the specimen by moving the movable plates. Dynamic heat aging of the specimens was carried by subjecting the specimen to constant cyclic deformation at 100 cpm. The specimen holder is maintained inside the oven which was set at a desired ageing temperature. In this study, the service life of the tire tread compound was carried out by subjecting the rubber vulcanizates to dynamic ageing condition using the above mentioned equipment. The factors like strain, temperature and frequency were considered for predicting life of rubber component. Arrhenius method was used to determine the service life of the rubber vulcanizates, by way of experimenting the dynamic ageing of dumbbells and thereby testing them. The general criteria such as the degradation of important / key properties to 50 % or more of its original property known as "threshold value". The dynamic ageing conditions such as temperatures ranging from 40°C, 50°C, & 60°C and strain [2, 5 & 8 %] under constant frequency of 100 cpm were used for ageing the specimens and then reduction of properties like Tensile strength and Elongation at break were calculated. The reduction of properties was carried out till it reaches a threshold value i.e. 50 % of its original properties. The temperatures covering a adequate range to establish the life time estimation by extrapolation with the required degree of accuracy.
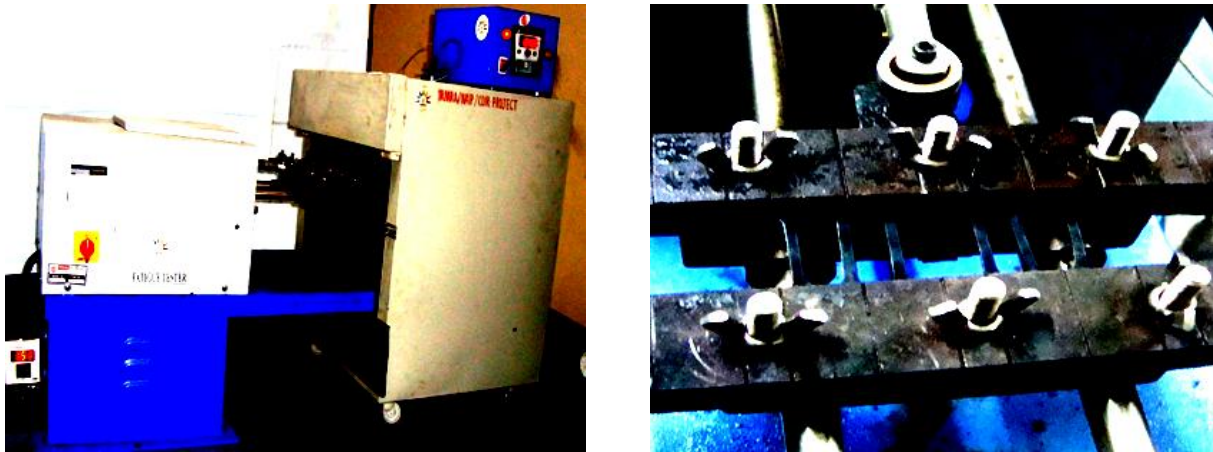
Figure 1- Dynamic ageing oven with fixtures for Dynamic ageing study

2.5.2 Arrhenius Procedure for life prediction
The reduction in the physical properties of the tread compound is mainly due to the temperature or heat generated during rolling of tire. Arrhenius equation is used to determine the activation energy required for the degradation to start in the rubber vulcanizates. General equation for the Arrhenius is given below,

$k = A \exp(-E_a./RT)$

Simplifying the above equation gives,

$$k = A \exp\left[-\left(\frac{E_a}{RT}\right)^{\beta}\right]$$

where β is a unit less number of order 1
Taking the natural logarithm of the Arrhenius equation yields:

$$\ln(k) = \frac{-E_a}{R}\frac{1}{T} + \ln(A)$$

As seen from the above equation that the temperature is the factor which has most effect on the overall equation. In case of our study, the strain factor also was considered along with dynamic frequencies, which also have significant effect on the physical property of the rubber vulcanizate. Considering the real application, the above equation can be modified by incorporating the strain in the denominator equation will yield as below:

$$\ln(k) = \frac{-E_a}{R}\left[\frac{1}{T}\ \frac{1}{S}\right] + \ln(A)$$

Where S is termed as % strain (at given frequency)

3. Results and discussion
3.1. Physical Properties
The *fig. 2a and 2b*, describes the results of changes in physical properties of tread Rubber vulcanizates after dynamic ageing conditions of different durations at ageing temperatures of 40°C, 50°C and 60°C, at a constant frequency and strain of 100 cpm and 2 % respectively.
The *Fig. 2(a)* describes the trends of reduction of Tensile Strength at different dynamic ageing temperatures like 40°C, 50°C & 60°C. The slope of the curves are found to be more for 60°C compared to 40°C and 50°C at a constant frequency and strain of 100 cpm and 2 % respectively. The *Fig. 2(b)* describes the trends of reduction of % Elongation at break at different dynamic ageing temperatures like 40°C, 50°C & 60°C. As seen in the fig. 2 a and 2 b, the changes in physical properties found to follow a trend of decreasing order at fixed temperatures [40°C, 50°C, & 60°C] with increase in durations [say 2, 4, 6 hrs….and so on] of dynamic ageing conditions of dumbbells at a constant frequency and strain of 100 cpm and 2 % respectively. Decreasing trend of tread rubber vulcanizates became more severe as the temperature of dynamic ageing conditions increases from 40 to 60°C. For example reduction of Tensile strength as tabulated in *table 5*, after 4 hours of duration of dynamic ageing of dumbbells at 40°C, 50°C & 60°C found to be – 1.6 %, -6 % & -58 % respectively, at a constant frequency and

strain of 100 cpm and 2 % respectively. Same trend was observed in the case of % Elongation at break of tread rubber vulcanizates subjected to a specific dynamic ageing condition.
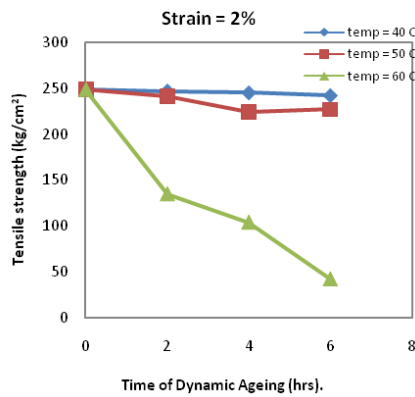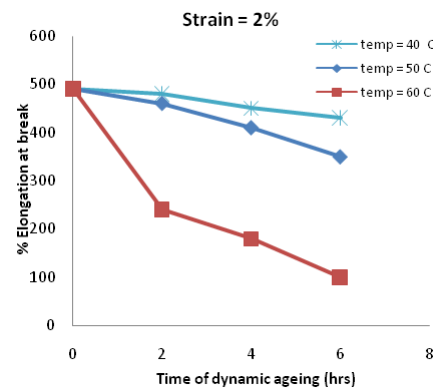

Figure 2a

Figure 2b

Figure 2- Changes in physical properties (a) tensile strength, (b) elongation at break, of tread Rubber vulcanizates after dynamic ageing conditions of different durations at ageing temperatures of 40°C, 50°C and 60°C, at a constant frequency and strain of 100 cpm and 2 % respectively

For example, the reduction of % elongation at Break as tabulated in *table 5*, after 4 hours of duration of dynamic ageing of dumbbells at 40°C, 50°C & 60°C found to be – 8 %, -16 % & - 63 % respectively, at a constant frequency and strain of 100 cpm and 2 % respectively. This is due to the known fact that the rubber degrades faster at high temperature at a faster rate degradation and it becomes more severe after certain temperature especially under dynamic ageing temperature [in this case it is found to be above 50°C.

The *fig. 3* describes the results of physical properties of tread Rubber vulcanizates after dynamic ageing conditions of different durations at ageing temperatures of 40°C, 50°C & 60°C, at a constant frequency and strain of 100 cpm and 5 % respectively.
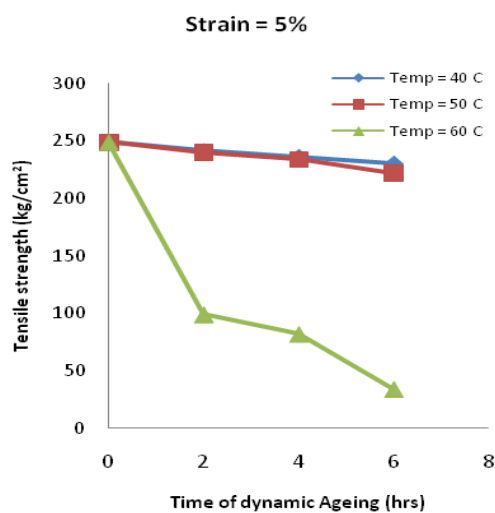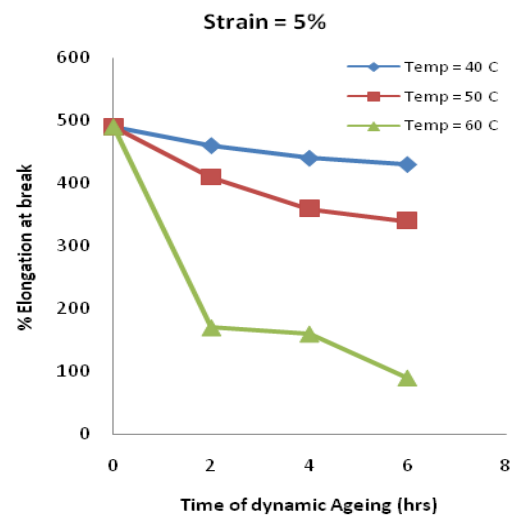

Figure 3a

Figure 3b

Figure 3- Changes in physical properties (a) tensile strength, (b) elongation at break, of tread Rubber vulcanizates after dynamic ageing conditions of different durations at ageing temperatures of 40°C, 50°C and 60°C, at a constant frequency and strain of 100 cpm and 5 % respectively

The changes in physical properties found to follow a trend of decreasing order at fixed temperatures [40°C, 50°C & 60°C] with increase in durations [say 2, 4, 6 hrs….and so on] of dynamic ageing conditions of dumbbells at a constant frequency and strain of 100 cpm and 5 % respectively. Decreasing trend of tread rubber vulcanizates became more severe as the temperature of dynamic ageing conditions increases from 40 to 60°C. For example reduction of Tensile strength as tabulated in table 5, after 4 hours of duration of dynamic ageing of

dumbbells at 40°C, 50°C & 60°C found to be – 5 %, -7 % & -67 % respectively, at a constant frequency and strain of 100 cpm and 5 % respectively.

Same trend was observed in the case of % Elongation at break of tread rubber vulcanizates subjected to a specific dynamic ageing condition. For example, the reduction of % elongation at Break as tabulated in *table 5*, after 4 hours of duration of dynamic ageing of dumbbells at 40°C, 50ºC & 60°C found to be – 10 %, 27 % & -68 % respectively, at a constant frequency and strain of 100 cpm and 5 % respectively. This is due to the known fact that the rubber degrades faster at high temperature at a faster rate degradation and it becomes more severe after certain temperature especially under dynamic ageing temperature [in this case it is found to be above 50°C. The *Fig. 3(a)* describes the trends of reduction of Tensile Strength at different dynamic ageing temperatures like 40°C, 50°C & 60°C. The slope of the curves are found to be more for 60°C compared to 40°C and 50°C at a constant frequency and strain of 100 cpm and 5 % respectively. The *Fig. 3(b)* describes the trends of reduction of % Elongation at break at different dynamic ageing temperatures like 40°C, 50°C & 60°C.

Figure 4a   **Figure 4b**
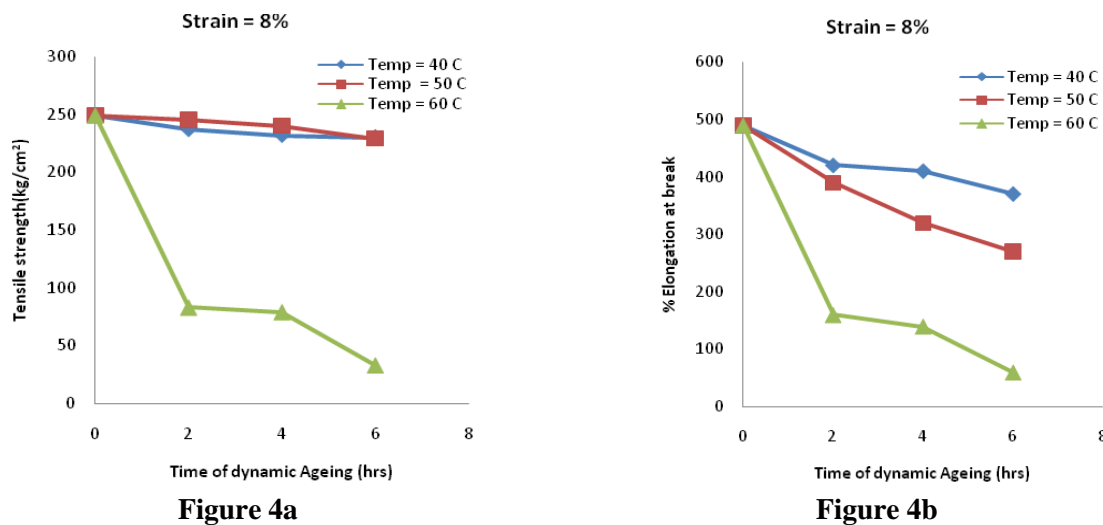
Figure 4- Changes in physical properties (a) tensile strength, (b) elongation at break, of tread Rubber vulcanizates after dynamic ageing conditions of different durations at ageing temperatures of 40°C, 50°C and 60°C, at a constant frequency and strain of 100 cpm and 8 % respectively

Fig 4 describes the results of physical properties of tread Rubber vulcanizates after dynamic ageing conditions of different durations at ageing temperatures of 40°C, 50°C & 60°C, at a constant frequency and strain of 100 cpm and 8 % respectively. The *Fig. 4(a)* describes the trends of reduction of Tensile Strength at different dynamic ageing temperatures like 40°C, 50°C & 60°C. The slope of the curves are found to be more for 60ºC compared to 40ºC and 50ºC at a constant frequency and strain of 100 cpm and 8 % respectively. The *Fig. 4(b)* describes the trends of reduction of % Elongation at break at different dynamic ageing temperatures like 40°C, 50°C & 60°C. The changes in physical properties found to follow a trend of decreasing order at fixed temperatures [40°C, 50°C & 60°C] with increase in durations [say 2, 4, 6 hrs….and so on] of dynamic ageing conditions of dumbbells at a constant frequency and strain of 100 cpm and 8 % respectively. Decreasing trend of tread rubber vulcanizates became more severe as the temperature of dynamic ageing conditions increases from 40°C to 60°C. For example reduction of Tensile strength as tabulated in table 6, after 4 hours of duration of dynamic ageing of dumbbells at 40°C, 50°C & 60°C found to be – 6.8 %, 7.6 % & -68.2 % respectively, at a constant frequency and strain of 100 cpm and 8 % respectively. Same trend was observed in the case of % Elongation at break of tread rubber vulcanizates subjected to a specific dynamic ageing condition. For example, the reduction of % elongation at Break as tabulated in table 6, after 4 hours of duration of dynamic ageing of dumbbells at 40°C, 50°C & 60°C found to be – 16 %, -35 % & - 72 % respectively, at a constant frequency and strain of 100 cpm and 8 % respectively. So, this is due to the known fact that the rubber degrades faster at high temperature at a faster rate degradation and it becomes more severe after certain temperature especially under dynamic ageing temperature [in this case it is found to be above 50°C.

Fig. 5 describes the results of physical properties of tread Rubber vulcanizates after dynamic ageing conditions of different strain conditions [2, 5 & 8 %] of dumbbells at ageing temperature of 40°C, at a constant frequency of 100 cpm. The changes in physical properties found to follow a trend of decreasing order at a given

temperature of 40ºC, with increase in durations [say 2, 4, 6 hrs….and so on] of dynamic ageing conditions of dumbbells at 2, 5 and 8 % strain and at a constant frequency of 100 cpm. Decreasing trend of tread rubber vulcanizates became more severe as the strain of tire tread increases. For example, Tensile strength as tabulated in *table 7*, after 4 hours of duration of dynamic ageing of dumbbells at 40ºC, is reduced to 245, 236, and 232 respectively, at the strain of 2, 5 & 8 % respectively and at constant frequency of 100 cpm. Same trend was observed in the case of % Elongation at break of tread rubber vulcanizates subjected to specific dynamic ageing conditions. This is due to the known fact that the rubber degrades faster at higher strain which represent over loading conditions of a tire, undergo faster rate of degradation and it becomes more severe after higher strain conditions especially under dynamic ageing temperature.
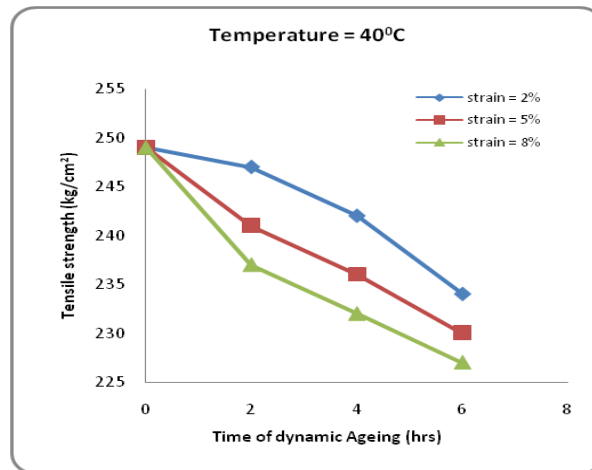

Figure 5: Trends of reduction of Tensile Strength at dynamic ageing condition of different strain condition like 2, 5 & 8 % and at a temperature of 40°C.

Fig. 5 describes the trends of reduction of Tensile Strength at dynamic ageing condition of different strain condition like 2, 5 & 8 % and at a temperature of 40°C.

Fig. 6 describes the results of physical properties of tread Rubber vulcanizates after dynamic ageing conditions of different strain conditions [2, 5 & 8 %] of dumbbells at ageing temperature of 50°C, at a constant frequency of 100 cpm.
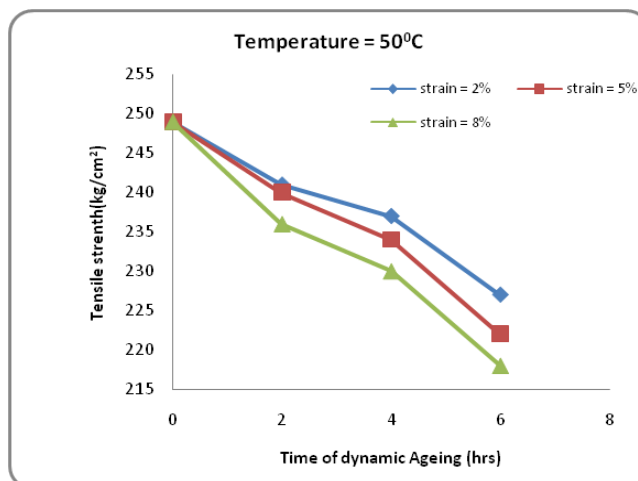

Figure 6: Physical properties of tread Rubber vulcanizates after dynamic ageing conditions of different strain conditions [2, 5 & 8 %] of dumbbells at ageing temperature of 50°C

The changes in physical properties found to follow a trend of decreasing order at a given temperature of 50°C, with increase in durations [say 2, 4, 6 hrs….and so on] of dynamic ageing conditions of dumbbells at 2, 5 and 8 % strain and at a constant frequency of 100 cpm. Decreasing trend of tread rubber vulcanizates became more

severe as the strain of tire tread increases. For example, Tensile strength as tabulated in *table 8*, after 4 hours of duration of dynamic ageing of dumbbells at 50°C, is reduced to 234, 232, and 230 respectively, at the strain of 2, 5 & 8 % respectively and at constant frequency of 100 cpm. Same trend was observed in the case of % Elongation at break of tread rubber vulcanizates subjected to a specific dynamic ageing condition. This is due to the known fact that the rubber degrades faster at higher strain which represent over loading conditions of a tire, undergo faster rate of degradation and it becomes more severe after higher strain conditions especially under dynamic ageing temperature.

The *Fig. 6* describes the trends of reduction of Tensile Strength at dynamic ageing condition of different strain condition like 2, 5 & 8 % and at a temperature of 50°C.

Table 4: Dynamic Heat Ageing at various strain % under constant frequency of 100 cpm at 60°C

	Strain = 2%	Strain= 5%	Strain = 8%
Time(hr)	Tensile strength(kg/cm^2)	Tensile strength(kg/cm^2)	Tensile strength(kg/cm^2)
0	249	249	249
2	135	99	83
4	104	82	79
6	42	34	33

The *table 4* states the results of physical properties of tread Rubber vulcanizates after dynamic ageing conditions of different strain conditions [2, 5 & 8 %] of dumbbells at ageing temperature of 60°C, at a constant frequency of 100 cpm. The changes in physical properties found to follow a trend of decreasing order at a given temperature of 60°C, with increase in durations [say 2, 4, 6 hrs….and so on] of dynamic ageing conditions of dumbbells at 2, 5 and 8 % strain and at a constant frequency of 100 cpm. Decreasing trend of tread rubber vulcanizates became more severe as the strain of tire tread increases. For example, Tensile strength as tabulated in *table 8*, after 4 hours of duration of dynamic ageing of dumbbells at 60°C, is reduced to 104, 82, and 79 respectively, at the strain of 2, 5 & 8 % respectively and at constant frequency of 100 cpm. Same trend was observed in the case of % Elongation at break of tread rubber vulcanizates subjected to a specific dynamic ageing condition. This is due to the known fact that the rubber degrades faster at higher strain which represent over loading conditions of a tire, undergo faster rate of degradation and it becomes more severe after higher strain conditions especially under dynamic ageing temperature
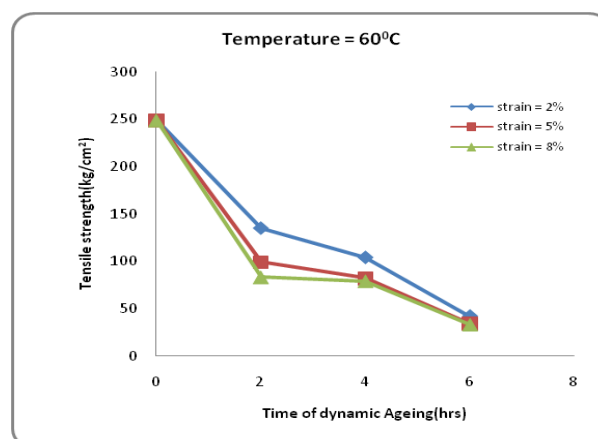

Figure 7: Trends of reduction of Tensile Strength at dynamic ageing condition of different strain condition like 2, 5 & 8 % and at a temperature of 60°C.

The *Fig. 7* describes the trends of reduction of Tensile Strength at dynamic ageing condition of different strain condition like 2, 5 & 8 % and at a temperature of 60°C.

3.2. Morphological Studies

Figure 8 shows SEM images of tread rubber vulcanizates (a) before ageing (b) after ageing of 6 hrs at 40°C under 2 % strain (c) after ageing of 6 hrs at 50°C under 2% strain (d) after ageing of 6 hrs at 60°C under 2 % strain. It can be observed that as the temperature is increased under constant frequency of 100 cpm and constant strain of 2 % from room temperature to 60°C, the rubber vulcanizates starts to detoriate. Under dynamic heat ageing condition formation of so called holes in the rubber matrix can be observed in SEM images. As the tire rotates under certain weight it experiences repeated cycles of deformation and recovery and after one or two hour heat is generated, which cause rapid reduction of the antidegradants in rubber stock and result in reduction in mechanical properties.
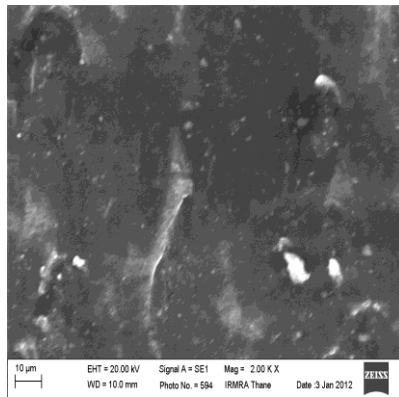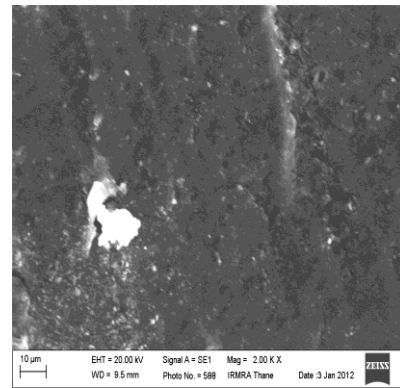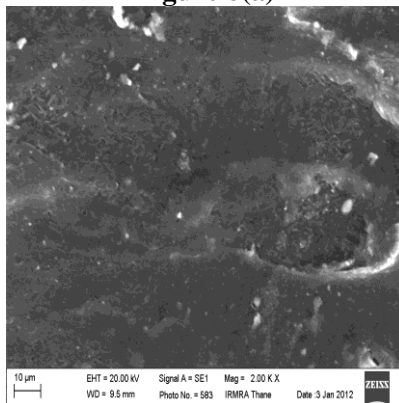

Figure 8(a)

Figure 8(b)
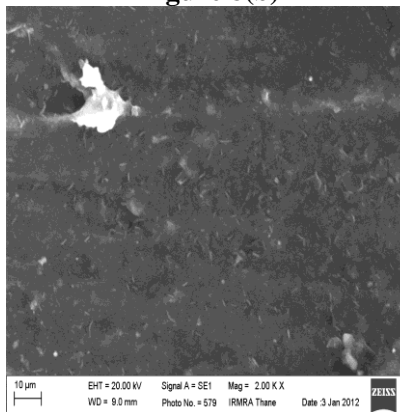

Figure 8(c)

Figure 8(d)

Figure 8: SEM images of fractured rubber vulcanizates with 2 % strain for 6 hrs duration ageing.
(a) Before ageing (b) Dynamic aged 40°C (c) Dynamic aged 50°C (d) Dynamic aged 60°C.

3.3. Life Estimation

Table 5: Threshold value at Various Temperatures under Constant frequency of 100 cpm at 2% strain

Temperature(°C)	Time of Ageing (min.)	% Elongation at break
R.T	0	490
40	20160	240
50	2880	230
60	120	220

The above *fig. 9* describes the Arrhenius plot of life prediction of tread rubber vulcanizate under dynamic ageing condition at severe conditions of strain [2% strain], temperature at a constant speed of 100 cpm.

Through the plot predicted the life of vulcanizate is 17.01 days for threshold value and activation energy found to be 197.56 kJ/mol. In actual application it will be much favourable conditions i.e. the strain will be very low compared to what has been used for prediction. The advantage of this method is that we can predict the life of compound within short duration of testing at laboratory level using vulcanizates as specimens. This could be

corroborated with accelerated ageing testing of tyres and correlated for actual use under normal conditions of road conditions.
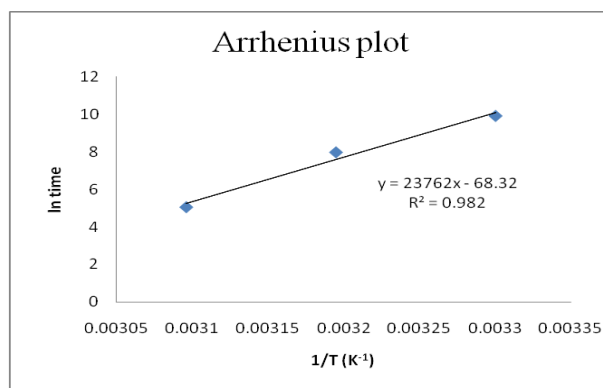

Figure 9: Arrhenius plot of life prediction of tread rubber vulcanizate

Conclusion

A new rapid technique of dynamic ageing of tread rubber vulcanizates using severe test conditions in order to simulate the actual application. An attempt was made to predict the dynamic life of tread rubber compound / vulcanizate using this rapid technique which will reduce the laboratory testing duration. The trend of reduction of physical properties such as Tensile strength and Elongation at Break of rubber vulcanizate were monitored at different dynamic ageing duration and studied. It has been found that the rate of degradation was more at higher temperature at a given strain and frequency of dynamic ageing condition compared to the lower test temperatures. This technique can be used for assessing the life of rubber compound, at a shorter duration in laboratory level, with more reliability and closer to the end use application.Note that in the study of internal energy dissipation a sample is assumed to deform homogeneously, with every part undergoing the same cycle of stress and deformation. But this is not the case when the stress is changed abruptly and a stress pulse is transmitted. The deformation is then markedly non-uniform, with one part of the sample being stressed more than a neighboring part. Some features of the propagation of stress waves in rubber are yet to be discussed in the future work, before we turn to energy dissipation and visco-elastic losses in particular, in homogeneously-deformed rubbery solids.

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