# Testing and Analysis

FatigueCrackGrowthV1, July, 2001

## Application of Energy Based Fatigue Crack Growth to Elastomer Compound Development

By Don G. Young, Independent Consultant

### Introduction

This paper will discuss the use of energy based fatigue crack growth testing to evaluate various rubber compounds. It is intended to be useful to those interested in rubber research, development and technical service in a wide variety of mechanical goods, automotive and tire fields. Primary emphasis will be on the application of fracture mechanics based fatigue results to model crack growth under different types of deformations experienced by rubber parts.

The major subjects that will be covered include: why a fracture mechanics approach should be used; factors influencing crack initiation in rubber; some brief theory related to fatigue crack growth (FCG); the experimental approach that has been used by the author; some

results which illustrate the full range of FCG testing; various ways the results can be applied; example data from several programs and how they were analyzed vs field data.

### About the Author

Don Young is currently available for consulting with clients in tire, mechanical goods and other rubber fields concerning fatigue testing and modeling of service requirements. He has worked on rubber applications and technical service at Exxon Chemical Co. for 34 years. During the last 15 years he was especially involved in applying energy based fatigue technology to a wide variety of service environments. Don can be reached at: dgyoung1@comcast.net.

### Why use fracture mechanics?

Compound and Geometry Factors - Conventional fatigue testing of rubber strips or parts is frequently dominated by a number of uncontrolled variables. The most troublesome variable is usually the wide distribution of initial flaws which are present. Since many tests implicitly depend on such flaws to initiate failure of the sample or part, the results can be quite variable. Fracture mechanics based tests, however, measure the rate of crack growth of a preinitiated cut, thus avoiding the uncertainties of variable initial flaw sizes. Further, they can be readily done on servohydraulic equipment, allowing the use of high strain rates, precise strain levels, and accurate data

collection. This leads to high quality data in minimum time because it is not necessary to test many replicates and conduct statistical studies to assess whether there are differences among mean values of many samples.<sup>1</sup> This reference (1) reviews major prior work as well as three studies by the author.

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Axel Products, Inc. 2255 S Industrial Ann Arbor MI 48104 Tel: 734 994 8308 Fax: 734 994 8309 info@axelproducts.com www.axelproducts.com Wide Applicability of Results - The other major reason why fracture mechanics based tests are preferred to conventional fatigue studies is that the results are fundamental material properties which can be applied to a wide range of rubber applications and types of deformation. Conventional tests usually obtain failure data for various rubber compounds under specific testing conditions, and then rely upon experience based correlations to rank the compounds in order of preference. While such ranking can also be done from FCG results, the net result of most FCG tests and analyses is an actual quantitative appraisal of the rates that a crack will grow under conditions which best describe the range of deformations the part experiences (from a consideration of mechanics). In this respect, FCG testing should be considered a natural partner to finite element modeling, because the FCG tests can supply data for the model, and the results of the modeling can supply information on the magnitude of the critical deformations to allow the best possible FCG analysis.<sup>2</sup> Obviously, this would be best done in a step-wise manner for critical applications.

### **Crack Initiation**

It should be understood at the outset that the science of fracture mechanics deals with crack growth, and it only considers crack initiation in an indirect manner. However, there are some well known factors that relate to crack initiation which should be reviewed before discussing crack growth. They can be grouped into four general categories as shown below:

Compound: Undispersed carbon black agglomerates, polymer gel

**Processing:** Contamination, voids, molding flaws, rough cut edges

**Geometry:** Sharp radii, lettering, deformation under load

**Service:** Cuts, nicks, tears from use; also attack from environmental factors

such as ozone, oxygen, or UV

Early crack initiation is obviously to be avoided, since many rubber parts are subjected to loadings which will accelerate the rate of crack growth with repeated deformation cycles. Thus, in virtually all cases one would want to minimize the above factors to slow the rate of crack formation.

However, despite careful efforts, all rubber parts experience these factors to some degree, so it is only a matter of time (deformation history) until small cracks will form. The subsequent growth of the cracks can be modeled using fracture mechanics principles.

### **Fracture Mechanics Theory**

The key parameter that controls crack growth rate has been shown to be the strain energy release rate. This was termed the Tearing Energy by Rivlin and Thomas<sup>3</sup> and it is defined by

$$T = -(\partial U/\partial A)$$

where U is the total elastic strain energy stored in the sample, A is the area of one fracture surface of the crack (in the unstrained state), and the partial derivative indicates that the sample is considered at a fixed deformation so the external forces do no work (strain

controlled rather than load controlled deformation). The tearing energy may be dependent on strain rate, temperature, and strain level similar to other material properties. The early work in this field was discussed by A.G. Thomas in his Goodyear Medal address, April 1994.<sup>4</sup> Lee and Donovan have also shown that the J-Integral may be applied to rubber in a similar manner as the tearing energy.<sup>5,6</sup> They also demonstrated that the J-Integral and tearing energy give equivalent results for pure shear and single edge notched (tensile) specimens.<sup>7</sup>

The tearing energy, T, also known as the "strain energy release rate," has been evaluated for a wide variety of sample geometries. For the most commonly used geometries, it is equal to the "Strain Energy Density,"  $W_{\rm O}$ , times certain geometric factors which may or may not involve the crack length itself. The strain energy density is

simply the area under the stress-strain curve obtained under representative conditions of deformation (not necessarily the usual laboratory tensile test conditions). Thus, for a given compound, higher deformations lead to higher levels of  $W_0$ , and this leads to faster rates of crack growth per cycle of deformation. In essence, that is all the fracture mechanics theory one needs to know other than the appropriate way to calculate T for the specific geometry one wishes to employ for testing or for modeling a specific application. For a discussion of various sample geometries, see references 1 and 8.

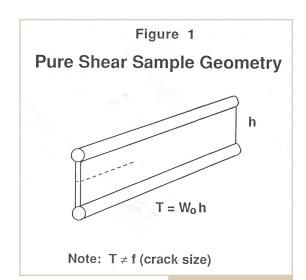
The sample geometry used in the work discussed herein is shown in Figure 1. It is known as a pure shear sample, and it is characterized by dimensions which are very wide vs. the height. Typical dimensions the author has used are 156 mm wide, 12.5 mm high, and 2.1 mm thick. These are also typical of the dimensions that most others have used who have done similar tests. A width-to-height ratio of at least 10 to 1 is desirable to maintain a condition of "pure shear" in the central part of the sample undisturbed by end effects. For this geometry, it has been shown that the tearing energy can be simply calculated from the relationship

$$T = W_0 h$$

where h is the original (unstretched) height of the sample inside the gripping beads. Not only is this very easy to calculate, it has the further advantage that T is not related to the length of the crack. Thus, for a given deformation (strain),  $W_0$  is fixed, and therefore the crack growth rate will be constant with repeated strain cycles (of the same magnitude). This has great experimental convenience, since:

- the data accuracy is increased because the crack growth process is stable, and
- rechecks can be conducted at any time at a given strain level, even after other levels of strain have been applied, to obtain accurate replicates if any points appear questionable.

Finally, it should be understood that, in addition to the tearing energy, environmental factors can play a large role in affecting crack growth rates, especially if the compound is susceptible to oxidation at the crack tip during deformation. An in-depth review of the recent work on the energetics approach to fatigue and fracture, including oxidative effects, environmental cracking, cutting, abrasion and other phenomena was given by Lake.<sup>8</sup>



### **Experimental Procedure**

Extremely precise FCG data can be obtained by employing careful sample preparation, utilizing the stable crack growth process in a pure shear sample, and by using a servohydraulic testing system to apply pulsed loading deformations to precise strain levels. This allows the acquisition of high quality data in a reasonably short time per sample (2 hrs. to one day depending on the detail desired). The essential steps employed are as follows:

- 1. Extrude each compound to give a cross section similar to the molded part; control sample dimensions to careful tolerances to give good pressure but minimize flow in molding; carefully mold to avoid imperfections.
- 2. Insert sample in holders contained within an oven; allow 1 hour thermal equilibration; apply break-in conditions using the servohydraulic tester to apply pulses to intermediate then maximum strain level that will be employed for testing (eliminates Mullins' Effect).
- 3. Using pulsed loading, collect stress vs strain data at each strain level of interest (e.g., 5 to 80%); obtain  $W_0$  by integrating under the loading or unloading curves using data stored in a digital oscilloscope or by computer based data analyses.
- 4. Initiate a cut in one side of the sample using moistened razor blade; apply intermediate strain cycles to break-in crack and overcome initial fast growth due to cut sharpness.
- 5. Test at each strain level by applying known number of deformation cycles,  $\Delta n$ , and measuring the change in crack length,  $\Delta c$ , using a long focal length microscope (looking through the oven window) and a light reflective surface. When measuring the crack, include the length of any major branches.
- 6. Calculate T from  $W_0$  and h data, and calculate  $\Delta c/\Delta n$  for each testing condition using a computer application to automate the process.

The use of a pulsed loading system, which extends the sample at a rate similar to that which is experienced in service, followed by a rest period (for sample recovery) of at least 8 times the pulse time, has the following major benefits:

- Minimizes viscoelastic effects upon  $W_0$  and  $\Delta c/\Delta n$ .
- Prevents samples from gradually changing due to stress relaxation during the testing of successive deformation levels.
- Ensures that the properties are being measured under conditions which are as close as possible to those that are experienced in the field.
- Gives time for heat transfer to avoid excessive sample heatup.
- Replicates the pattern of many typical deformations found in actual service, e.g., tires, belts.

For some materials, e.g., gum natural rubber, viscoelastic effects are a little less prominent, but with typical carbon black loadings, and especially with elastomers such as butyl, and halobutyls, increased hysteresis make these testing precautions mandatory if data are expected to be representative of the results experienced in the field.

### **Typical Results**

Figure 2 shows FCG results obtained on vulcanized gum SBR samples. Results are given for two samples, and they are clearly in excellent agreement. Each result consists of a  $\Delta c/\Delta n$  value, in nm/cycle, and a corresponding tearing energy (T) value, in J/m². At low levels of T, i.e., below 100 J/m², Figure 2 shows that the FCG vs T relationship becomes nearly vertical and asymptotic to a value of T labeled,  $T_0$ . This is called the threshold value of T, since below that level no discernible crack growth occurs.

For values of T in the range of ~800 to ~5000 J/m² for this gum SBR vulcanizate, the FCG relationship is a straight line of slope, m. This is the "power law" region where

$$\Delta c/\Delta n = AT^m$$

where A is merely a scaling constant for a given material and conditions of evaluation. Values of m range from 1.0 to 4.0 for common elastomer compounds, with values of 1.0 to 2.0 typical for NR, 1.5 to 3.0 for SBR, and 1.5 to 3.5 typical for BR, IIR, BIIR, CIIR, or EPDM with the wide ranges caused by the effects of variables such as strain rate, temperature, and oxidation. The low value of m for NR has been ascribed to strain crystallization effects which can inhibit crack growth (due to energy dissipation near the crack tip) under some conditions.

At high levels of T, the dc/dn vs T relationship becomes much steeper, signifying rapidly increasing crack growth rates with increases in T. This begins at a point labeled  $T_C$  because this represents the onset of "catastrophic tearing." To ensure that a part does not experience premature failure, it is important that the fatigue history which it will experience does not result in tearing energy levels that fall in the catastrophic tearing region. This can be accomplished by careful choice of:

- 1. design parameters to minimize stress levels and stress concentrations,
- 2. elastomer and compound variables, and
- 3. compound processing and part handling.

Several of these were discussed in some detail in the Crack Initiation section. FCG testing as described herein allows the development chemist and engineer to check item 2 and some aspects of item 3 independent of the part design aspects. After the best choices of elastomer, compound and processing have been determined, it is then possible to check the performance of various part designs in field evaluations to ensure that performance will be satisfactory.

### **Modes of Deformational Control**

In analyzing FCG data with a given part in mind, it is critical that the appropriate mode of deformation control is considered.

Figure 2 Results for SBR Illustrate all Three Regions of FCG 10<sup>6</sup> Conditions: 27° C, 40 Hz Pulse, 105 5 Hz overall cycle 104 dc/dn, nm/cycle 103 102 10 1 100 Sample 1 Sample 2 10 -1 -2 1000 10000 T. J/m<sup>2</sup>

From this perspective, there are three primary modes of control which need to be considered. They are strain, load (or stress) and energy control. The two which require the largest difference in analysis are strain vs load control, so they will be compared and discussed. Energy control is very easily dealt with in comparing results because the most common means of comparing FCG data is to prepare a plot of  $\Delta c/\Delta n$  vs T. By simply comparing crack growth rate at a given level of T, which has been estimated from part geometry and fracture mechanics principles, one is performing an appropriate analysis for a part which is subject to energy controlled deformation.

What is meant by deformation control is probably best understood by considering some examples shown below (some of which are debatable depending on the specifics of a given part):

<b>Deformation Control</b>	Example Applications
Strain	Belts, hoses, bellows, seals, tire sidewalls, innerliners, bladders, treads (some aspects)
Load (or stress)	Auto exhaust hangers, body mounts, tire treads, belt skims, tank track pads
Energy	Auto engine mounts, suspension bumpers

In cases where strain is the controlling parameter, the part usually has some reinforcement or other deformation limiting feature which dictates the degree to which the rubber will deform. Hence the load level, or the modulus of the compound, has little or no influence on the degree of deformation. Load controlled applications are exactly the opposite. Here the load applied, and the modulus of the rubber, determine the extent of deformation. These two cases require very different analyses, as will be illustrated by Figures 3 and 4.

In Figure 3 are shown schematically the stress-strain relationships of a low vs a high

Low Modulus Compound Has Low Woin Strain Controlled Deformation

Low Mod.

High Mod.

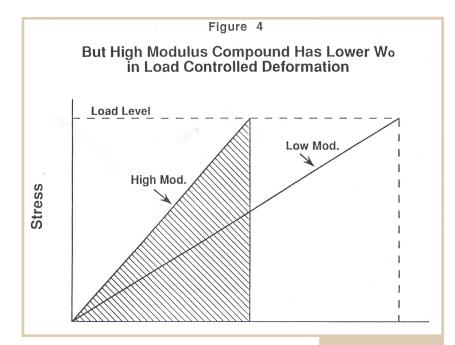
Strain

Figure 3

modulus compound for an application which experiences strain controlled deformation. The vertical dashed line represents the typical or maximum strain level to which the part is subjected. The strain energy density, Wo, as discussed earlier, is a primary material parameter which directly affects crack growth rate of a material. Wo is determined by the area under the stress-strain curve of the rubber. It is easily seen that the area under the stress-strain curve of the low modulus compound (shaded area) is much smaller than the area under the stressstrain curve of the high modulus compound when the two are considered at the same maximum strain level. Thus, if one wants to minimize FCG by minimizing Wo for a strain controlled part, assuming other factors such as rubber type, oxidation resistance and crosslink type are equal, it is preferable to use the lowest modulus

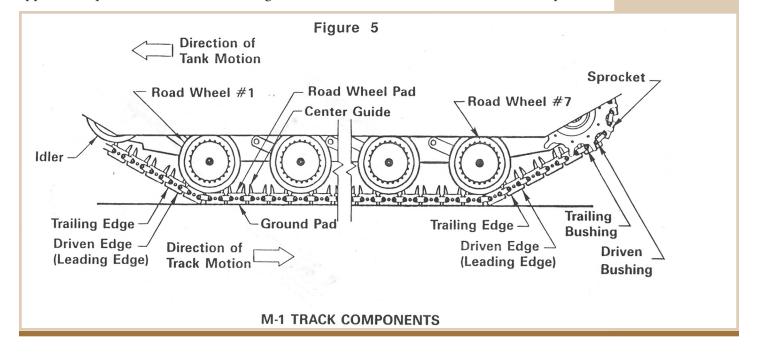
compound possible, consistent with satisfying other performance criteria. This is one reason why most rubber compounds for bellows, seals, tire sidewalls and innerliners are relatively soft (low modulus) vulcanizates. The slope of the fatigue function may be affected somewhat by compounding changes to achieve low modulus. However, the slope, m, is primarily affected by the elastomer type, so modulus changes will often have the major (desired) effect.

Figure 4 represents the same low and high modulus compounds, but in this case the parameter controlling the deformations is the load applied to a given cross section (i.e., the stress level). The load level is represented by the horizontal dashed line. Here



the compound with the lower level of  $W_O$  is the *high* modulus compound because it will experience a much lower level of deformation simply because of its resistance to deformation, i.e., "modulus." This is seen by comparing the shaded area for the high modulus compound vs the much larger area (triangle) representing the  $W_O$  of the low modulus compound. It should now be obvious why one should be clear about the mode of deformation control that a given part experiences. The rubber choice which is most likely to lead to success, where minimum crack growth rate is the primary criteria, is exactly opposite for strain vs load controlled deformations. This principle was illustrated by the author by the data and (load controlled) analysis in reference 1 on tire belt skim compounds. An energy based analysis was shown in the same reference for a basic material study comparing blends of CIIR and NR compounds with and without carbon black, and a strain controlled analysis was used to compare FCG performance of developmental grades of BIIR for innerliner use.

Regardless of the controlling mode of deformation, and therefore the analysis approach required, the method of testing remains the same. Each material (rubber sample)



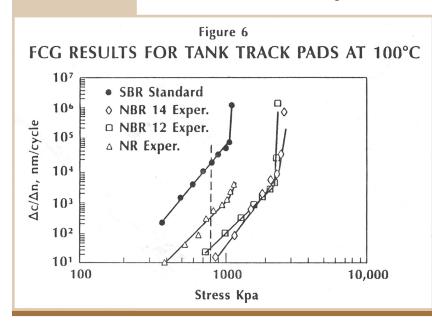
is evaluated at specific strain levels spanning the widest possible range that the rubber might experience. Why use strain control exclusively for testing when many parts experience load or energy control? Why not subject materials being considered for a load controlled application to load controlled testing, since that is relatively easy to do with servohydraulic equipment? There are three very good reasons for always using strain control for testing:

- 1. That is the way the tearing energy is defined. Use of load control for testing would subject the sample to added energy of deformation as the crack grew, and this would make the determination of T much more difficult.
- 2. Servohydraulic equipment is safest when operated in this manner. If the sample breaks (at high strains/loads) the crosshead will not continue to drive to the end points of the actuator in order to try to achieve a programmed load level, it will be limited by the strain level programmed.
- 3. It is no problem at all to apply the results using fixed load or fixed energy criteria even though the data were obtained at specific strain levels.

Point 3 assumes that one has obtained enough data to allow interpolation between load or energy levels that each compound will experience. In other words, it is possible to conduct FCG tests of a series of compounds being considered *for a strain controlled application* where only a single data point is obtained, provided one is very confident of both the strain level and the quality of the FCG results. However, tests being conducted *for load or energy controlled applications* require that appropriate ranges of strain levels are used for the FCG testing to ensure that the results will cover sufficient ranges of load (stress) or energy to allow direct comparison of performance when the analysis is conducted. The following example will illustrate this approach.

### FCG Testing of Tank Track Pad Compounds

In cooperation with the US Army Materials Technology Laboratory, FCG tests were performed on 4 compounds which had been employed in tank track pads and tested on an M-1 tank. Figure 5 shows a schematic of the M-1 Track Components. Each metal "shoe" on the track has two rubber pads, each of which is hexagonal in shape and roughly 6x8x2



inches in dimension. The rubber pads serve to dampen some of the ride vibration and reduce noise, but their main function is to prevent excessive damage to roads. The SBR-based standard production track pad compound has shown a high rate of wear due to severe loading (stress levels) and high temperatures requiring replacement of the pads in as short as 500 miles under severe road operational conditions. Research and testing was conducted to improve the wear performance and allow a greater useful life of the track pads, thereby improving range and reducing maintenance.

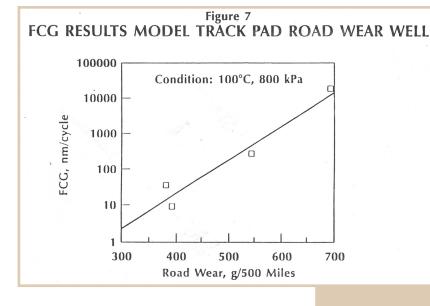
The four (proprietary) compounds evaluated were as follows:

The SBR-based standard production control.

An experimental NR-based compound.

Two experimental HNBR-based compounds, designated NBR-12 and 14.

Samples were prepared and molded in the standard manner and tested at 100° C using a 40 Hz pulse and a 1 Hz overall cycle. This gave a deformation period of 0.025 sec, fol-



lowed by a rest period of 0.975 sec. Such a cycle gives ample time for viscoelastic recovery of the sample after each loading, and also gives time for heat transfer to prevent excessive temperature buildup. Since several of these compounds were very high in modulus, the above testing protocol was considered important to avoid excessive heat and stress relaxation effects during testing.

The FCG results are given in Figure 6. Since each tank track pad is subjected to a given stress (tank weight/total pad area), regardless of compound modulus, this was viewed as a load (stress) controlled application. Thus, the  $\Delta c/\Delta n$  results were plotted as functions of the peak stress level experienced at each (strain) condition. This plot allows direct comparison of compound performance at any given stress level within the range of conditions evaluated. Clearly, at all stress levels evaluated, the SBR standard production compound is poorest, the NR-based experimental intermediate, and the two HNBR-based compounds are best. This is not surprising since HNBR has improved heat resistance vs SBR and NR, but also because the two HNBR compounds were very high in modulus vs the SBR and NR.

Based on the stress levels the pads experience in service, and the ranges of results available, a stress level of 800 kPa was selected as the primary condition for comparison. Figure 7 shows the FCG results at a stress level of 800 kPa plotted vs the wear results for these track pad compounds obtained in a paved road test on an M-1 tank at the Yuma Proving Grounds in Arizona. The wear results were previously published by Pergantis et al, 10 and also by Medalia et al. 11 The Pergantis paper focused on the surface temperatures, while the Medalia paper dealt with the type of abrasion experienced. The two HNBR compounds gave road wear of 400 gm/500 miles or less, whereas the SBR control gave a result of nearly 700 gm/500 miles. The agreement of the 4 FCG results to the road wear is very good, and probably due to the fact that the abrasion in this case was

### **TABLE 1**

### **BIIR Innerliner Compound**

BIIR 2222 N660 Black Naphthenic Oil Processing Aid <sup>1</sup> Phenolic Resin <sup>2</sup> Stearic Acid Magnesium Oxide <sup>3</sup>	2	100 60 8 7 4 0.15
Zinc Oxide MBTS Sulfur		3 1.5 0.5
MS, T <sub>3</sub> at 135°C ML, 1+4 at 100°C		18 54

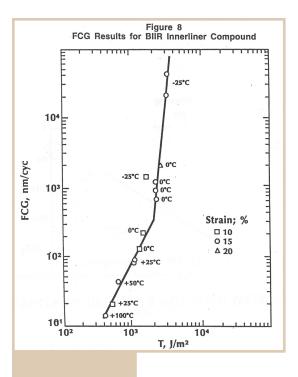
### ODR Cure: 3° arc at 160C

$M_H-M_I$ , $dN-m$	16
ts <sub>2</sub> , min.	6.3
ť90, min.	22.3

### Physical Properties: Cured 25' at 160°C

Hardness, Shore A	55
300% Modulus, MPa	2.5
Tensile Strength, MPa	9.0
Elongation, %	880

- 1) Struktol 40 MS
- 2) SP 1068 Resin
- Maglite K



primarily by a "pattern abrasion" mechanism which is well known to involve crack growth. Thus, it appears that the FCG test can be predictive of the fatigue and fracture occurring in a highly severe abrasion process when the analysis is conducted appropriately.

### **Combined Strain and Temperature Effects**

Previous research studies by the author on CIIR, BR and NR employed fixed strain rates, and found unique relationships of FCG vs T at various temperatures. A later study was done to assess performance of BIIR vs epichlorohydrin (ECO) and high styrene SBR elastomers. The success of halobutyls as innerliner elastomers has hinged on their unique permeability, Q, vs  $T_g$  relationship. This results in FCG results for a given strain level or Tearing Energy which are 1 or 2 magnitudes lower (better) for halobutyl innerliner compounds vs those of high  $T_g$  polymers such as ECO or HS SBR.

To assess the general ability of the FCG vs. T relationship to encompass both strain and temperature effects, a series of tests were

conducted using the 100% BIIR innerliner compound shown in Table 1. The FCG results are given in Figure 8. With the exception of one data point (10% strain at -25°C), the data are extremely consistent and show the steady and catastrophic crack growth regions very well. The fact that the catastrophic crack growth region may be involved with the BIIR compound at 10% strain and -25°C can be put into perspective by observing that the level of FCG found was only 1.3 x 10<sup>3</sup>nm/cycle, or 1.3 x 10<sup>-4</sup> cm/cycle. Thus, it would require 10<sup>4</sup> (10,000) cycles for a crack to grow 1.3 cm (if the tearing energy imposed was high enough). Since this is approximately 5% of the total cycles that most passenger tires experience in their total lifetime, and since the tire warms up quickly with use, the chance of a BIIR tire innerliner experiencing flex fatigue failure due to startup under winter

conditions of -25°C are extremely remote. In fact, some special low-temperature tire tests conducted several years ago showed no appreciable BIIR liner cracking at starting temperatures as low as -40°C, whereas tires with a high  $T_{\rm g}$  liner showed significant cracking at temperatures as high as -22°C.

# The strong relationship of FCG with T shown in Figure 8 was anticipated, but these results were significantly different from those reported by the author previously. The earlier results, generated under constant strain rate conditions for CIIR, NR and BR compounds, showed a family of lines which were different for each temperature in the range of 0 to 75° C. The only testing difference with the current work was that constant frequency conditions (40 Hz pulse, 5 Hz overall cycle) were used rather than constant strain rate. Strain rate effects had been shown in the previous work to be strong for the CIIR compound, but weak for the NR compound at low tearing energy levels (low strains). Thus, since the BIIR compound of the current work was not greatly different from the CIIR compound of the earlier work, it was very surprising to find the single relationship shown in Figure 8. To assess the general-

# TABLE 2 NR Control Compound

NR, SMR CV 50	100
IRB #6 Black	50
ASTM #1 Oil	10
Stearic Acid, NBS 372i	2
Zinc Oxide, NBS 376e	5
Sulfur, NBS 371g	2
MBTS, ASTM, IRM-002	1.5
MS, T10 at 135°C	9.8
ML, 1+8 at 100°C	49.6
ODR Cure: 1° arc at 160°C M <sub>H</sub> - M <sub>L</sub> , dN-m ts2, min. t'90, min.	22.3 2.2 6.1

Physical Properties: Cured 8' at 160°C

Hardness, Shore A	56
300% Modulus, MPa	7.1
Tensile Strength, MPa	21.4
Elongation, %	610

ity of this relationship an additional series of tests were conducted on a NR compound used in this laboratory for control charting to ensure consistent system performance. The formulation is given in Table 2. The results of FCG tests conducted at temperatures of -25 to 100°C and strain

TABLE 3
Hysteretic Energies Measured for NR Control Compound

Strain, %:	train, %: 10		15		20	
Temp., °C	W <sub>H</sub> 1	$\% W_H^2$	$W_{H}$	% W <sub>H</sub>	$W_{H}$	% W <sub>H</sub>
100 50 26 0 -25	6.5 9.8 12.0 18.5 72.9	31 35 36 47 92	11.9 18.4 22.3 36.2 137.0	29 35 35 47 89	18.5 28.9 34.4 56.0 193.0	28 35 34 46 90

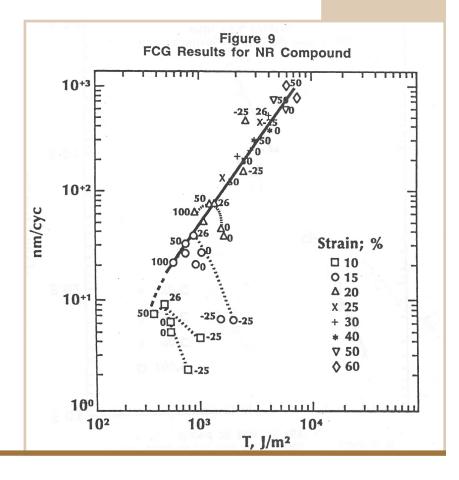
1) W<sub>H</sub> = hysteretic energy (area within stress-strain loop), J/m<sup>2</sup> 2) % W<sub>H</sub> = (W<sub>H</sub>/total area under loading curve, W<sub>O</sub>) x 100

levels ranging from 10 to 60% are given in Figure 9.

In general, the magnitude and slope of the results for NR are very similar to those in Figure 8 up to a Tearing Energy level of 2500 J/m<sup>2</sup>. The really interesting results for NR, however, are at low strains and at 0 and -25°C. Here the behavior is more complex, with clear deviations from the general body of results. At a given strain level (10, 15, or 20%), as the temperature was decreased (using different samples), there is a sudden deviation from the general line for temperatures of 0 or -25 at 10 and 15% strain. At 20% strain the deviation occurs at 0°C, but at -25°C the results (2 data points at higher T) are close to the general trend line.

The reason for this complex behavior of NR at low temperatures and low strains is believed due to its well-known ability to strain crystallize.<sup>13</sup> The data in Table 3 show the hysteretic energies measured at each of the test conditions, and the percent of total loading energy that was hysteretic. At temperatures of 26°C and above, the hysteretic energy is about 35% of the total. However, at 0°C, it increases to 47% and at -25°C it has increased to  $90 \pm 2\%$ . This is clear evidence of strain crystallization taking place, and it is not surprising that the FCG rates decline over what was experienced for the amorphous polymer. As previously reported, strain crystallization for NR is most efficient at -24°C, and it occurs at low strains in the presence of a filler such as carbon black.14

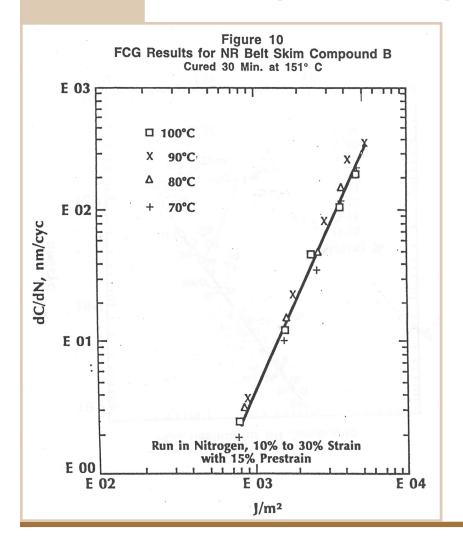
What about the two results for -25°C conducted at 20% and 25% strain as well as



0°C at 30, 40, and 50% strain? It must be realized that these were not constant strain rate tests, and that strain crystallization requires a significant amount of time to develop. It has been shown that strain crystallization in gum NR produces large increases in tearing energy only over certain ranges of rates and temperatures; outside these ranges the strengthening effect virtually disappears. 15 At the higher strain levels, the strain rate was proportionally higher since the frequency of the haversine pulse used was 40 Hz in all cases. Also, the strain was fully relaxed after each cycle. Thus, this data suggests that, even though one would expect strain crystallization under certain conditions, e.g., -25°C and 20% strain, the strain rate is rapid enough that the crack growth occurs too quickly for the strain crystallization to impede it. Although strain crystallization is accelerated by stretching, it is also retarded by vulcanization. 16 This is obviously very interesting behavior which has implications for a number of strain crystallizable elastomers as well as plastics. For example, the fatigue behavior of (high ethylene) EPDM rubber compounds, or of PE plastics, at various strain rates and temperatures encompassing their strain crystallization region could be very interesting to assess. The use of a constant strain rate testing protocol as shown previously9 would be one way to approach this.

### **Results for NR Belt Skim at Elevated Temperatures**

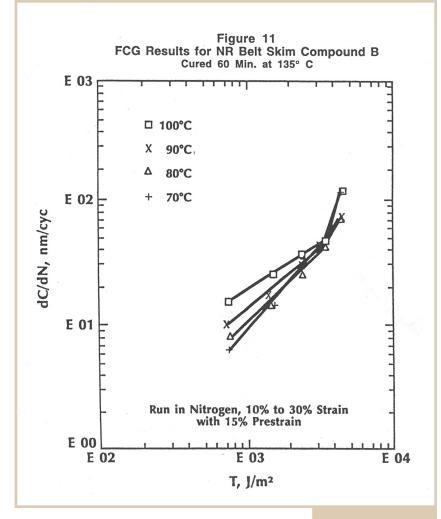
A cooperative testing program was conducted with a major tire manufacturer on two NR belt skims used in truck tires. 12 The temperature range employed was 70 to 100°C, and all tests were run on unaged samples in a nitrogen atmosphere, using 10 to 30% strain and a 15% prestrain. Results for Compound B, cured at 151°C, are in Figure



10. These will be compared with results from the same compound cured at a lower temperature to illustrate effects of undercure on FCG performance. The formulation was proprietary, and not revealed to this laboratory.

The FCG results vs. Tearing Energy follow a linear relationship, with very little experimental scatter despite the variety of strain and temperature conditions employed. In Figure 11 are results for Compound B cured 60 min. at 135°C. This is obviously an undercure compared with the 30 min. at 151°C used for results in Fig. 10, since 60 min. at 141° C would be an equivalent cure (to 30 min. at 151°C) based on generally accepted temperature coefficients of vulcanization. The results in Fig. 11 show a much more complex response of FCG with increasing temperature. In general, especially at lower strains (lower tearing energies), the FCG rates were significantly increased as testing temperature increased.

Also, the FCG rates in Fig. 11, at the 2 lowest strain levels, are significantly higher than those at the corresponding strains in Fig. 10. Clearly, the lower cure state of Compound B used for the Fig. 11 results had a major influence on the nature of the crack growth process. The substantially better results in Fig. 10 at low tearing energies (10% strain and 100°C) suggest that the achievement of a good state of cure in the NR belt skim may be a critical aspect of minimizing FCG and the development of belt edge separations. This is another area of R&D where additional FCG testing could be very helpful to the development of optimum formulations, especially in view of the never-ending desire of rubber manufacturers to reduce molding times by using higher curing temperatures. Further data on known compositions and known states of cure would be highly desirable.



### Summary

Brief theory and background were provided to show the importance and relevance of

using a fracture mechanics approach to model and evaluate rubber materials for diverse applications. The experimental approach was briefly discussed, and alternate forms of analysis were described to properly model strain, stress and energy controlled deformations. It was shown how the preferred compound properties are usually opposite for compounds being evaluated for a strain controlled vs a load controlled application. Examples of alternate analyses given in past work were cited. FCG results for M-1 tank track pad materials illustrated the power of this technology to model even such severe rubber fatigue and fracture as high rates of road wear. Results were presented to show the ability of energy based FCG to define crack growth behavior under wide ranges of temperature and strain conditions. Beneficial effects of strain crystallization were shown under certain conditions for a NR compound. Deleterious effects of undercure on FCG performance were also illustrated.

### **Acknowledgements**

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### References

- 1. D.G. Young, Rubber Chem. Technol. 63, 567 (1990)
- 2. D.G. Young, Tire Sci. & Technol., 19, Number 2 (1991)
- 3. R.S. Rivlin and A.G. Thomas, J. Polym. Sci., 10, 291 (1953)
- 4. A.G. Thomas, Rubber Chem. Technol., 67, No. 3, G50 (1994)
- 5. R.F. Lee and J.A. Donovan, Rubber Chem. Technol., 59, 787 (1986)
- 6. R.F. Lee and J.A. Donovan, Rubber Chem. Technol., 60, 674 (1987)
- 7. H. Liu, R.F. Lee and J.A. Donovan, Rubber Chem. Technol., 60, 893 (1987)
- 8. G.J. Lake, Rubber Chem. Technol., 68, No. 3, 435 (1995)
- 9. D.G. Young, Rubber Chem. Technol., 59, 809 (1986)
- 10. C.G. Pergantis, T. Murray, J.L. Mead, R.J. Shuford, and A.L Alesi, Rubber World 200, (1), 31 (April 1989).
- 11. A.I. Medalia, A.L. Alesi, J.L. Mead, and R. Simoneau, Paper no. 34 at meeting of Rubber Division, ACS, Cincinnati, October 18-21, 1988.
- 12. D.G. Young, Rubber Chem. Technol., 67, 137 (1994)
- 13. G.J. Lake, et al, Polymer, 32, 2963 (1991)
- 14. L. Bateman, "The Chemistry and Physics of Rubber-Like Substances," p. 228, 309, John Wiley & Sons, NY (1963)
- 15. Ibid., pg. 313
- 16. Ibid., pgs. 243, 239

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