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A Statistical Method for Computer Treatment of Elastomer Flex Data*

GEORGE C. DERRINGER, *Rubber Chemicals Research, Research Laboratories, PPG Industries, Inc., Chemical Division, Barberton, Ohio 44203*

Synopsis

In flex testing of elastomers, the length of a cut is measured periodically as a function of the number of flexing cycles. In most cases a large number of measurements is accumulated for each test piece which would lead one to believe that the results should have a high degree of precision. This, however, is not the case, mainly because not all of the measurements are used to express the final result. Instead of being a precise test, flex testing is in fact notorious for its irreproducibility. A new method is proposed for computer treatment of flex data in which *all* of the flex measurements are fitted to an empirical equation by means of nonlinear regression analysis. From the fitted equation, more precise estimates of rate of growth and growth initiation point can be obtained than is possible using the data treatment procedures proposed by ASTM. Also a new index of flex life is defined which is conveniently scaled from 0-100. This so-called flex index is a convenient index of overall flex behavior.

INTRODUCTION

Interpretation and reporting of flex results have always been problems. For example, ASTM D813 gives three ways in which results can be reported for the DeMattia flex test: (1) mean rate of crack growth over entire test period; (2) average rate of crack growth over any chosen portion of the test; (3) number of cycles to reach an arbitrary crack length, say 0.5 in. None of these, however, is entirely satisfactory.

The form used to record the raw DeMattia flex data in our laboratories is shown in Table I. Depending upon when failure occurs, up to 26 individual measurements are recorded for each compound, a rather time-consuming and expensive procedure. Clearly, then, a satisfactory way of reporting the results should employ all of the measurements.

The first method proposed by ASTM is unsatisfactory because rates would have to be computed for all pairs of adjacent points and averaged. If this is done for both of the proposed duplicate runs, 24 calculations are required for each compound, a tedious task. If, however, only the initial and final crack lengths are used to calculate the average rate, only 4 of the 26 measurements are used and the other 22 are wasted.

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TABLE I
Raw DeMattia Flex Data

		Crack growth of 0.01 in.												
Compound no.	Cure, min	500	1,000	2,000	3,000	4,500	9,000	13,500	18,000	36,000	54,000	72,000	90,000	100,000 cycles
45694	20	10	10	10	10	10	10	10	10	10	11	10	10	11
45694		10	10	10	10	10	10	10	10	10	10	10	10	12
45695	15	30	55	78	88	C								
45695		32	56	75	85	88	C							
45696	17	10	10	10	14	18	34	41	42	51	56	59	62	66
45696		10	12	19	28	38	46	51	55	65	68	72	76	78
45697	17	10	10	10	10	11	16	21	24	33	34	34	42	43
45697		10	10	10	10	11	16	23	29	32	38	42	43	45
45698	15	11	11	18	20	22	26	31	32	40	48	50	52	52
45698		10	11	15	17	20	26	30	32	42	43	42	50	54
45699	15	24	41	65	77	96	C							
45699		22	42	55	76	90	C							
45700	17	11	18	25	27	30	40	43	46	50	56	78	85	85
45700		10	12	21	27	35	52	53	56	57	60	62	76	78
45701	17	10	10	10	10	10	11	12	13	22	23	29	33	35
45701		10	10	10	10	10	11	13	14	22	25	29	34	35
45702	15	11	22	35	42	53	72	75	80	90	92	C		
45702		12	23	38	43	45	52	66	67	72	C			

The second proposed method is unsatisfactory for the same reasons as the first except that the number of measurements involved is smaller. Furthermore, the portion of the data to use is completely arbitrary.

The third proposed method is unsatisfactory because it employs only 2 or 4 of the 26 recorded measurements. Furthermore, if the chosen crack length lies between readings at two adjacent cycles, interpolation to arrive at the required number of cycles is inaccurate unless the relationship for relating crack length to cycles is known—which usually is not. For example, if we must find the number of cycles required to reach an 0.5-in. crack length and we have, say, a reading of 0.3 in. at 18,000 cycles and of 0.7 in. at 36,000 cycles, it is inaccurate to say that 27,000 cycles is the appropriate number. This figure is correct only if the cycle–crack length relationship is linear—and it usually is not.

In rubber technology, as in all areas of science, new tools continually become available to simplify once tedious and even impossible tasks. Such a tool is the electronic computer. Availability of a computer puts at our disposal mathematical and statistical tools which, prior to the computer era, could not be implemented owing to the tremendous amount of computation involved. With the present wide accessability of computers, it is imperative that we reexamine our methods of data collection, interpretation, and analysis to determine if they can be improved upon with mathematical and statistical tools now available.

APPLICATION TO DEMATTIA FLEX DATA

DeMattia flex is notorious for its irreproducibility. This is a rather strange state of affairs because more measurements are actually made per DeMattia test than for most other rubber tests. The real problem lies in the fact that not all of the test measurements are used in the determination of the characteristic of interest. What is needed is a model relating crack growth to number of test cycles. If a model is known, the precision of a predicted cut length is increased as the number of measurements is increased in the same way that a mean becomes more precise as the number of measurements upon which it is based increases. For example, with a known model, increasing the number of measurements in the vicinity of, say, 80% cut growth will actually increase the precision of all other cut growth predictions, even as far removed as 5%. Without a model, a thousand measurements at the 80% cut growth level will not increase knowledge of cut growth at 10% one iota. The principle can be summarized as follows:

If a well-fitting model is known which relates test measurements, an increase in the number of measurements anywhere within the range of validity of the model will increase the precision of the entire fitted model.

What is needed, then, is clearly a model relating cut length to the number of flexing cycles.

MODEL DEVELOPMENT

Consideration of the shape of cycle-versus-crack length curves led to early realization that an exponential curve of some type was required and also that ln cycles would, more than likely, be the appropriate scale of measurement. After an extensive amount of model building, the following equation was developed:

$$Y = a\left\{\exp\left[-\left(\frac{b}{\ln X}\right)^c\right]\right\} \quad (1)$$

where a, b, and c = fitted parameters, X = number of cycles, and Y = fractional cut growth = $\frac{\text{(cut length at time } t - \text{initial cut length)}}{\text{(sample width} - \text{initial cut length)}}$, and where initial cut length was 0.1 in. and sample width was 1 in. This equation is nonlinear in the parameters so that standard multiple linear regression analysis could not be employed. For such a model, nonlinear regression techniques are required.[1] In nonlinear regression analysis, the residual sum of squares which is a function of the parameters is minimized over the parameter space. A minimization problem, however, can be handled by a maximization algorithm simply by maximizing the quantity to be minimized multiplied by -1. This then reduces to a maximum seeking method in three dimensions for eq. (1). The nonlinear regression program used in this study employed the so-called pattern search method of maximization developed by Hooke and Jeeves,[2] and good estimates were generally found in fewer than 1000 iterations.

The pattern search method was employed simply because it was available in programmed form. It is quite possible that other methods such as Marquardt's algorithm[3] or steepest ascent (descent)[4] would be more efficient. On the other hand, it is very likely that the pattern search program which was employed could be improved by more efficient programming to decrease the number of iterations required.

EXPERIMENTAL

To evaluate the model developed above, it was fitted to the DeMattia flex results of an acceleration study of SBR filled with an experimental silica. The formulations are shown in Table II and constitute a 3^2 factorial design. The flex results have already been given in Table I. The test was run at room temperature, and two replications were made for each compound. In this study, the two replications were run on different days since only eight compounds could be tested at one time. A better method would have been to randomly select test pieces for each run. In this way, true test error is not confounded with any block effects resulting from running the test at widely separated times.

TABLE II
Formulations in Parts per Hundred by Weight

Compound	45694	45695	45696	45697	45698	45699	45700	45701	45702
SBR 1500	100								
N-Phenyl-β-naphthyl-amine	1								
Zinc Oxide	5								
Experimental silica	80								
Naphthenic oil	5								
N-*t*-Butyl-2-benzothiazole sulfenamide	0.8								
Tetraethylthiuram disulfide	0.4								
Sulfur	1.4	2.2	1.8	1.8	1.4	2.2	2.2	1.4	1.8
Polyethylene glycol	0.4	1.2	0.8	0.4	1.2	0.8	0.4	0.8	1.2

ESTIMATION OF PARAMETERS

After the flex results were collected, the cut length measurements were transformed to fractional cut growth. A nonlinear regression computer program was then employed to estimate the parameters a, b, and c in eq. (1) for each compound. The results are shown in Table III. Plots of the equations fitted to the data points are shown in Figures 1 to 8, where the dotted portions of the curves represent extrapolations.

TABLE III
Parameters for Equation (1)

Compound No.	a	b	c
45695	1.174	6.713	6.323
45696	0.933	9.247	4.895
45697	0.535	10.038	6.824
45698	2.069	13.963	2.077
45699	7.568	12.244	1.989
45700	3.715	15.238	1.702
45701	1.567	13.049	4.418
45702	1.611	9.151	3.163

The point scatter for these nine figures looks reasonable, with two possible exceptions. First, the two test pieces for compound 45696 shown in Figure 2 exhibited different crack-growth initiation points. The cause of this was probably poor technique in making the initial cuts. As a result, the two sets of points actually represent two near-parallel curves. The fitted curve is approximately the average of the two individual curves.

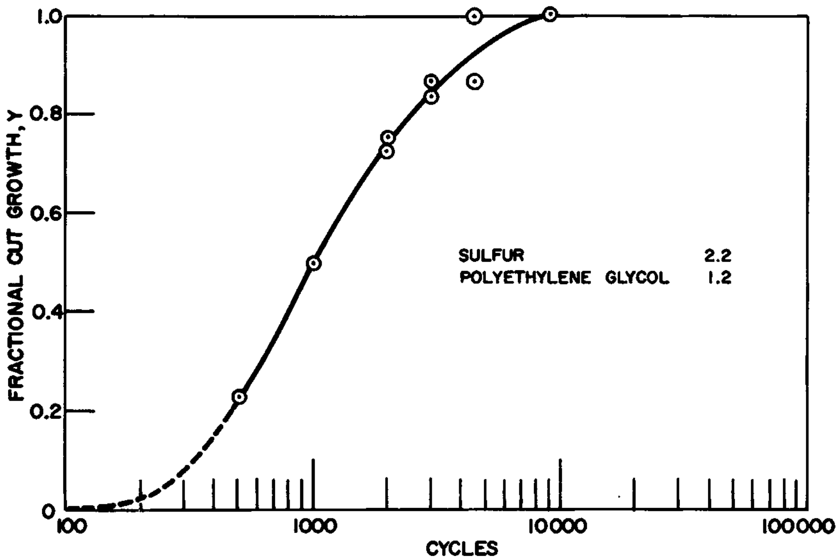

Fig. 1. Data from compound 45965 fitted to eq. (1).

Secondly, the data points for compound 45700 shown in Figure 6 exhibited an unexpected decrease in cut growth rate starting at 10,000 cycles. Such a phenomenon could have been caused by a change in environmental conditions during the testing period or innumerable other factors.

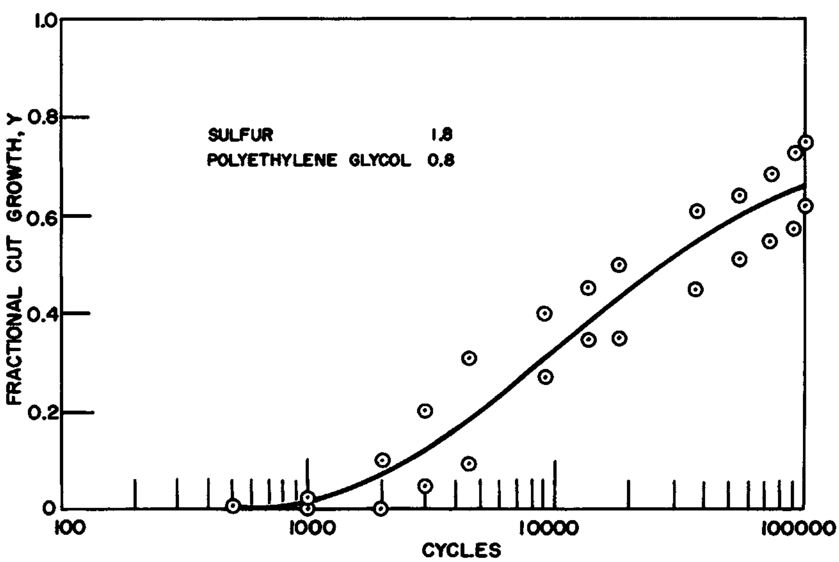

Fig. 2. Data from compound 45696 fitted to eq. (1).

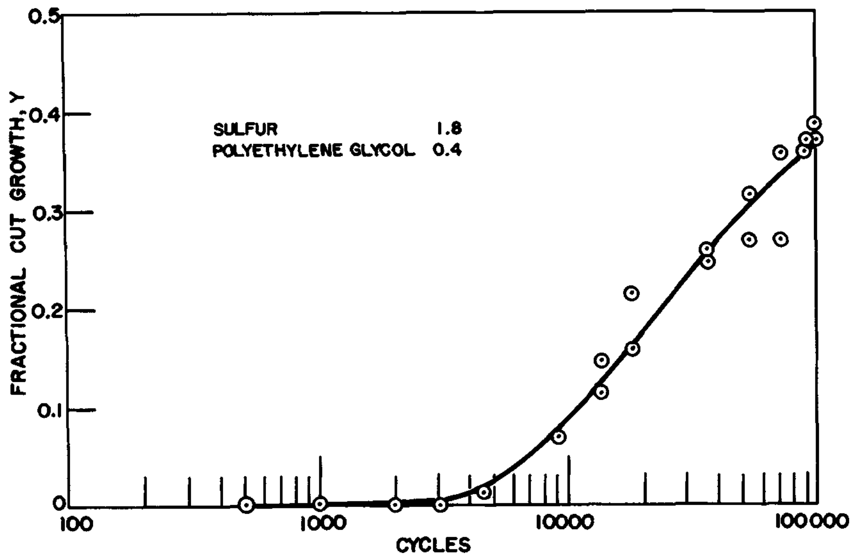

Fig. 3. Data from compound 45697 fitted to eq. (1).

Erratic behavior such as described above can only be checked by running confirmatory tests. Unfortunately, in this study, confirmatory tests could not be run owing to the limited availability of experimental silica.

Use of Fitted Equations

Once the equation has been fitted, the three parameters *a*, *b*, and *c* provide a good representation of the data in highly condensed form. From

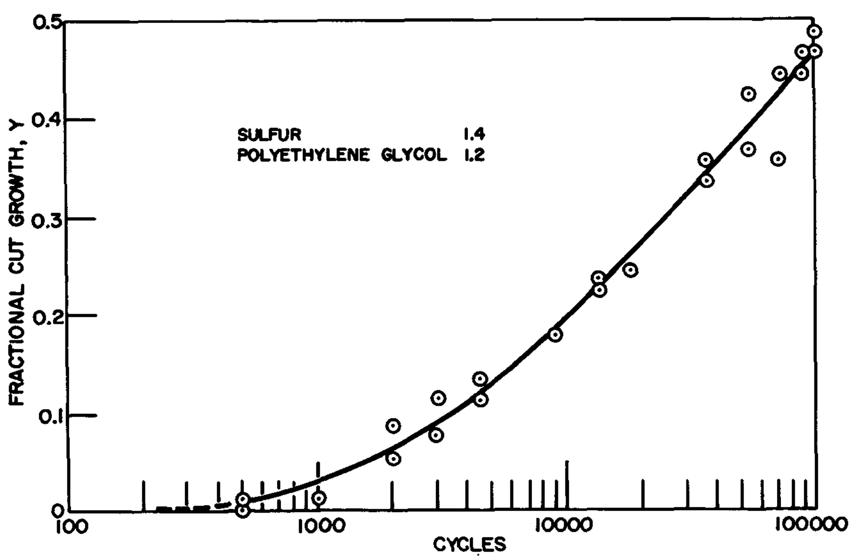

Fig. 4. Data from compound 45698 fitted to eq. (1).

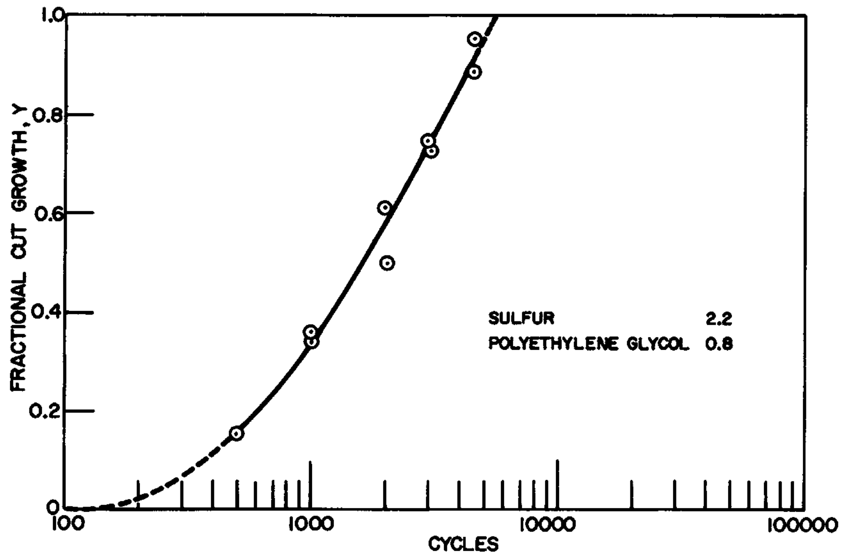

Fig. 5. Data from compound 45699 fitted to eq. (1).

these parameters and the model equation, we can calculate any flex characteristic we like. Furthermore, the result will be more accurate than any of the ASTM criteria because all of the data were employed in estimating the parameters a, b, and c.

Two obvious things we would like to know about flex behavior are (1) when cut growth commences and (2) some index of the rate of growth after

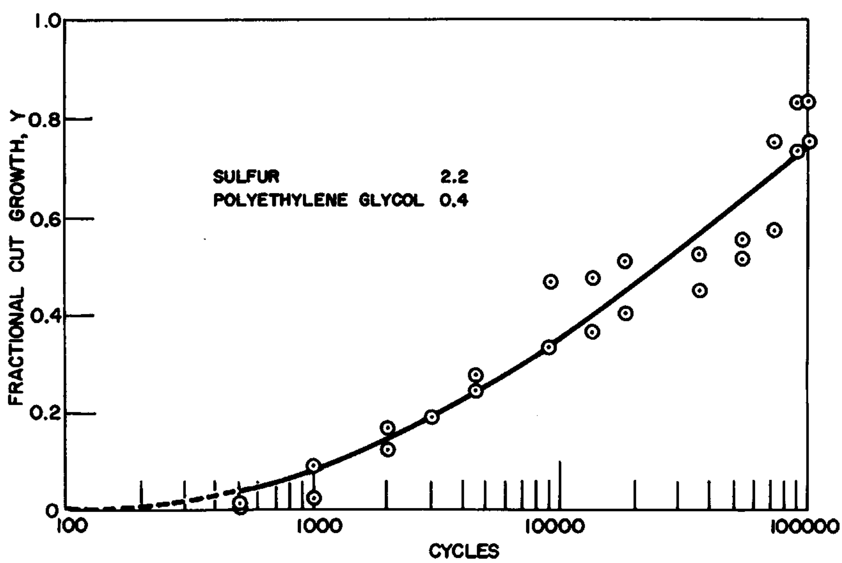

Fig. 6. Data from compound 45700 fitted to eq. (1).

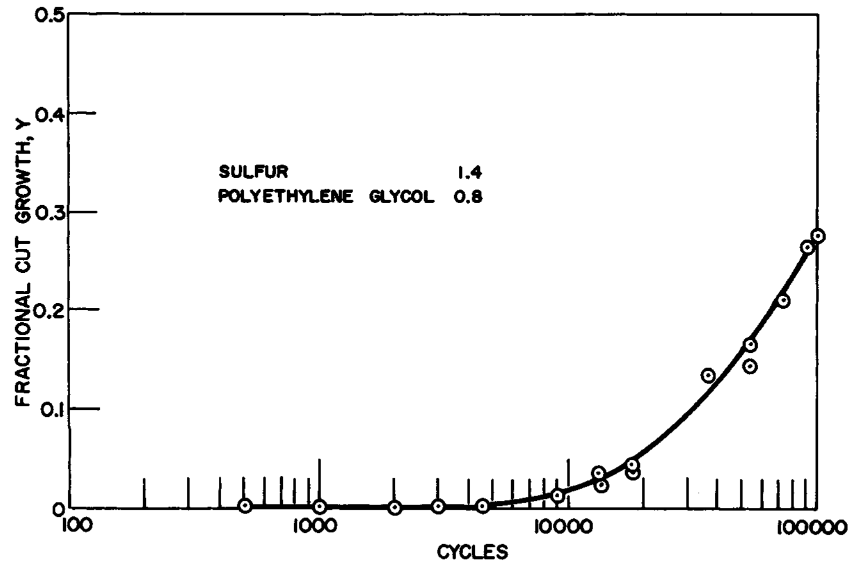

Fig. 7. Data from compound 45701 fitted to eq. (1).

growth commences. From (1), the number of cycles required for any given fractional cut growth is

$$X = \exp\left\{\exp\left[\frac{1}{c}\ln\left(\frac{b^c}{\ln(a/Y)}\right)\right]\right\} \tag{2}$$

In this study, the number of cycles required to give 1% cut growth was taken as the initiation point, X_0, or

$$X_0 = \exp\left\{\exp\left[\frac{1}{c}\ln\left(\frac{b^c}{\ln(a/0.01)}\right)\right]\right\}. \tag{3}$$

This is a convenient indication of time or cycles required for cut growth to begin. In some instances, X_0 will fall in the extrapolated portion of the curve. This will not give unreasonable results because Y approaches zero as $\ln X$ approaches zero in eq. (1).

The rate of cut growth after growth initiation is a little more complex. For rate of growth on a per cycle basis we get

$$R_1 = \frac{dY}{dX} = \frac{ac}{bX}\left[\exp\left(-\left(\frac{b}{\ln X}\right)^c\right)\right]\left[\frac{b}{\ln X}\right]^{c+1} \tag{4}$$

and on a per ln cycle basis we get

$$R_2 = \frac{dY}{d\ln X} = \frac{acb^c}{(\ln X)^{c+1}}\exp\left[-\left(\frac{b}{\ln X}\right)^c\right]. \tag{5}$$

From these two expressions, we can calculate a rate of growth either on a per cycle or per ln cycle basis at any given number of cycles, X. It may also be of interest to calculate rate of growth at any given fractional cut growth,

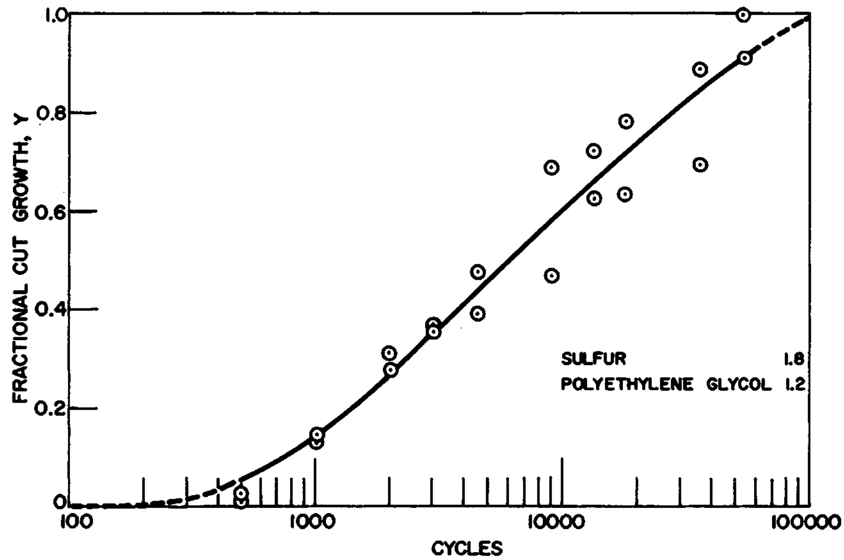

Fig. 8. Data from compound 45702 fitted to eq. (1).

Y_c. In this case, we can calculate the number of cycles X_c corresponding to a growth of Y_c from eq. (2) and substitute X_c in eq. (4) or (5). The decision of what to use as an indication for rate of growth for the purpose of comparing compounds, however, is somewhat arbitrary. Should we use rate at 10,000 cycles, rate at 50% cut growth, average rate between 20% and 50% cut growth, or what? To get a better picture of the situation, the rate on a per decade basis is plotted along with the cut growth curve for compound 45695 in Figure 9 where the rate curve is dotted. The most

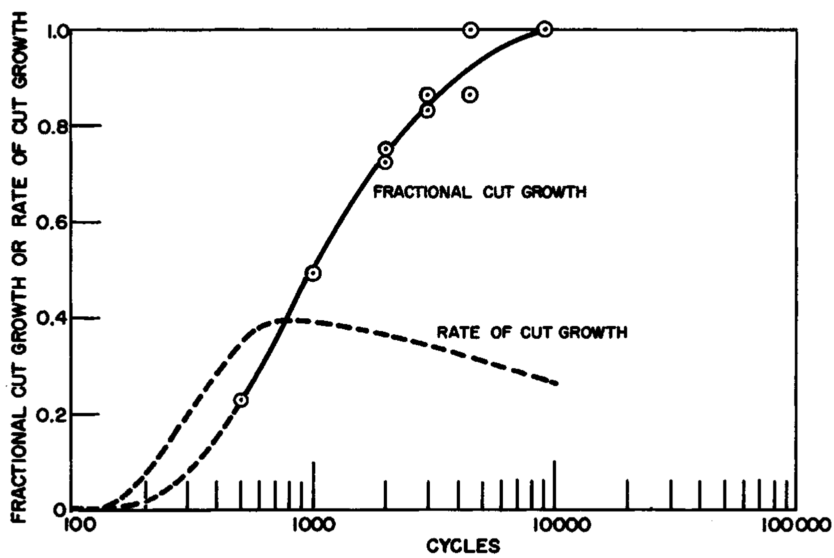

Fig. 9. Cut growth and rate of cut growth for compound 45695.

interesting feature of this curve is that it exhibits a maximum. The maximum occurs at X_m cycles, where

$$X_m = \exp\left[b\left(\frac{c}{c+1}\right)^{1/c}\right]. \tag{6}$$

Furthermore, from eqs. (4) and (5) we can get the actual maximum rates, say, R_{m_1}, R_{m_2}, by substituting X_m for X.

It may appear at first sight that X_m, R_{m_1}, or R_{m_2} may provide good bases for comparison of rates. There is, however, one problem. To use these expressions, X_m must be less than the total number of cycles of the test. In the present study, the test was terminated at 100,000 cycles so that four of the nine compounds have values of X_m greater than 100,000 cycles. If the test is run to complete failure (i.e., 100% cut growth), however: X_m, R_{m_1}, and R_{m_2} may provide good indices for comparing rates.

If the test is run to complete failure, a better index of growth rate would be some average value for growth rate. If we let X_∞ be (the number of cycles required to reach) 99% cut growth, where

$$X_\infty = \exp\left\{\exp\left[\frac{1}{c}\ln\left(\frac{b^c}{\ln(a/0.99)}\right)\right]\right\}, \tag{7}$$

a good average rate of growth would be R_{a_1}, where

$$R_{a_1} = \frac{1}{X_\infty - X_0}\int_{X_0}^{X_\infty} R_1 dX$$

$$= \frac{1}{X_\infty - X_0}\left\{a\left[\exp\left(-\left(\frac{b}{\ln X_\infty}\right)^c\right) - \exp\left(-\left(\frac{b}{\ln X_0}\right)^c\right)\right]\right\}. \tag{8}$$

The corresponding average based on ln X would be

$$R_{a_2} = \frac{1}{\ln X_\infty - \ln X_0}\int_{\ln X_0}^{\ln X_\infty} R_2 d\ln X$$

$$= \frac{1}{\ln X_\infty - \ln X_0}\left\{a\left[\exp\left(-\left(\frac{b}{\ln X_\infty}\right)^c\right) - \exp\left(-\left(\frac{b}{\ln X_0}\right)^c\right)\right]\right\}. \tag{9}$$

Running the test to complete failure, then, X_0 and either R_{a_1}, or R_{a_2} would give an excellent, highly condensed, and very meaningful description of flex behavior.

In many instances, however, because of the amount of time involved, it is not possible to run each compound to failure. As a matter of fact, it is common practice to run the test for a given number of cycles regardless of the extent of crack growth. This obviously creates problems and makes comparisons rather difficult. For example, in the present study, compound 45694 showed no crack initiation by the end of the test, i.e., 100,000 cycles. For such cases, the flex index I was defined as follows:

$$I = \left[\int_{\ln 500}^{\ln 10^5} a\exp\left(-\left(\frac{b}{\ln X}\right)^c\right)d\ln X \Big/ \int_{\ln 500}^{\ln 10^5} d\ln X\right] \times 100. \tag{10}$$

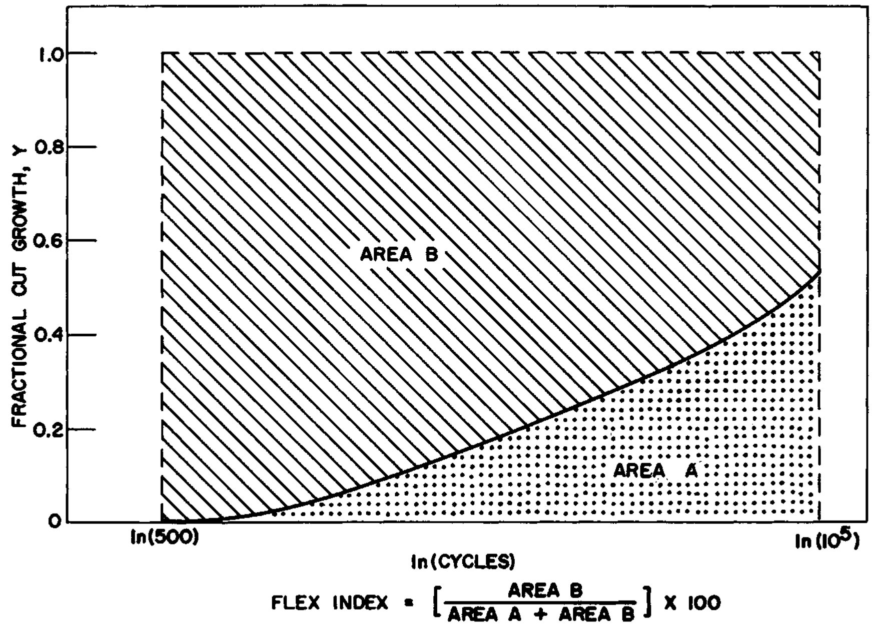

Fig. 10. Illustration of flex index when failure does not occur during test period.

This index is illustrated in Figure 10, where the index is equal to area B divided by the sum of areas A and B multiplied by 100. Obviously, the index will range from 0 to 100, a rating of 100 indicating no crack growth during the entire testing period and a rating near zero indicating rapid failure. For this study, the integration limits were set at 500 and 100,000 cycles. These limits are arbitrary for any given study; but, should the method become widely used, it would, of course, be advisable to standardize them. The integral in the numerator of eq. (10) was too complex to evaluate in closed form, and so was evaluated by iteratively measuring the area under the curve (1) with the aid of a computer. Flex index is obviously a function of both initiation point and rate of growth and thus gives an overall index of flex behavior.

Equation (10) is satisfactory only if complete failure does not occur before the end of the test. If failure does occur, however, say, at X_f cycles, the flex index would be calculted as follows:

$$I = \left\{\left[\int_{\ln 500}^{\ln X_f} a\exp\left(-\left(\frac{b}{\ln X}\right)^c\right) d\ln X + \int_{\ln X_f}^{\ln 10^5} d\ln X\right] \Big/ \int_{\ln 500}^{\ln 10^5} d\ln X\right\} \times 100. \quad (11)$$

This situation is illustrated in Figure 11. It is obvious that the flex index is a very convenient overall index of flex behavior, whether the test is run to complete failure or not, and should always be calculated.

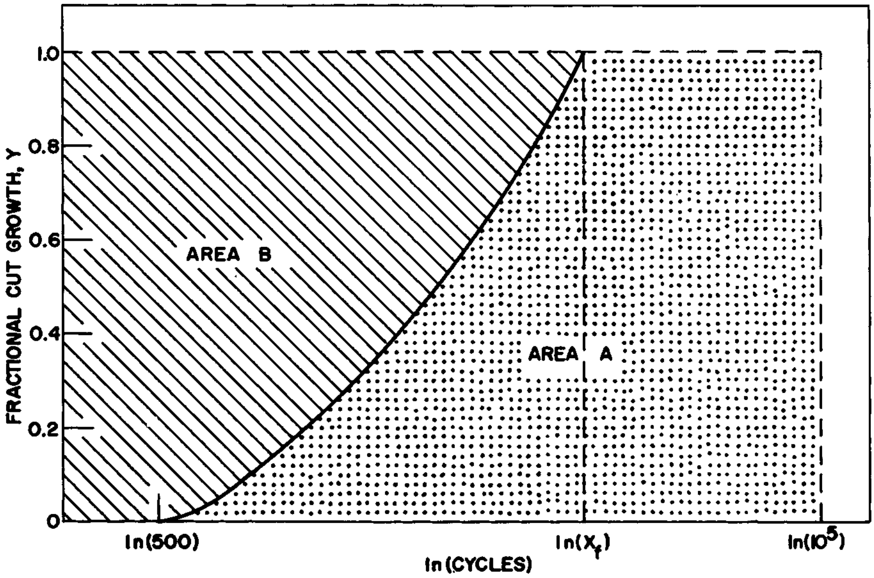

Fig. 11. Illustration of flex index when failure occurs before end of test period.

RESULTS

The flex results of the present study were summarized by: (1) flex index; (2) initiation point X_0; (3) maximum rate of cut growth (log scale) over the testing period (i.e., 0 to 10^5 cycles); (4) mean rate of cut growth (log scale) between X_0 cycles and end of test (10^5 cycles). All of these properties were calculated by direct computer iteration rather than by direct evaluation as outlined above. For compound, 45694 which showed no cut growth over the entire testing period, a value of 10^5 was assigned for the initiation point X_0. This, of course, is only a minimum value but is more preferable than assigning no value at all. The numbers for properties (3) and (4) are, of course, 0 and 0 since no growth occurred over the entire test period. Properties (1) through (4) for all compounds are given in Table IV.

TABLE IV
Four Indices of Flex Behavior from Computer Program "Flex"

Compound No.	Flex index	Maximum rate of growth	Mean rate of growth	Initiation point, cycles
45694	100	0	0	100,000
45695	15.88	0.41	0.27	189
45696	71.14	0.19	0.14	888
45697	89.02	0.14	0.11	3635
45698	81.46	0.13	0.09	510
45699	21.31	0.50	0.26	113
45700	67.01	0.18	0.12	213
45701	94.95	0.18	0.11	8460
45702	47.65	0.21	0.16	238

TABLE V
Summary of Statistics from Regression Analysis

Index of flex behavior	$R^2 \times 100$	F-ratio for regression	Standard error
Flex index	96.5	16.8	9.4
Maximum rate of growth	91.1	6.2	0.073
Mean rate of growth	94.2	9.8	0.033
Ln (initiation point)	99.5	121.8	0.026

A regression analysis was then run to fit a full second-degree polynomila equation in two variables to these four properties. This equation is as follows:

$$\hat{Y} = b_0 + b_1X_1 + b_2X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{12}X_1X_2 \qquad (12)$$

where X_1 = sulfur in design units; X_2 = glycol in design units; and b_i and b_{ij} = fitted regression coefficients. The important statistics from the regression analysis are given in Table V.

Contour plots from the fitted equations are shown in Figures 12 to 15. Figure 12 for flex index indicates that this property decreased with both increased sulfur and increased polyglycol. This probably indicates that flex index decreased with increased cure state.

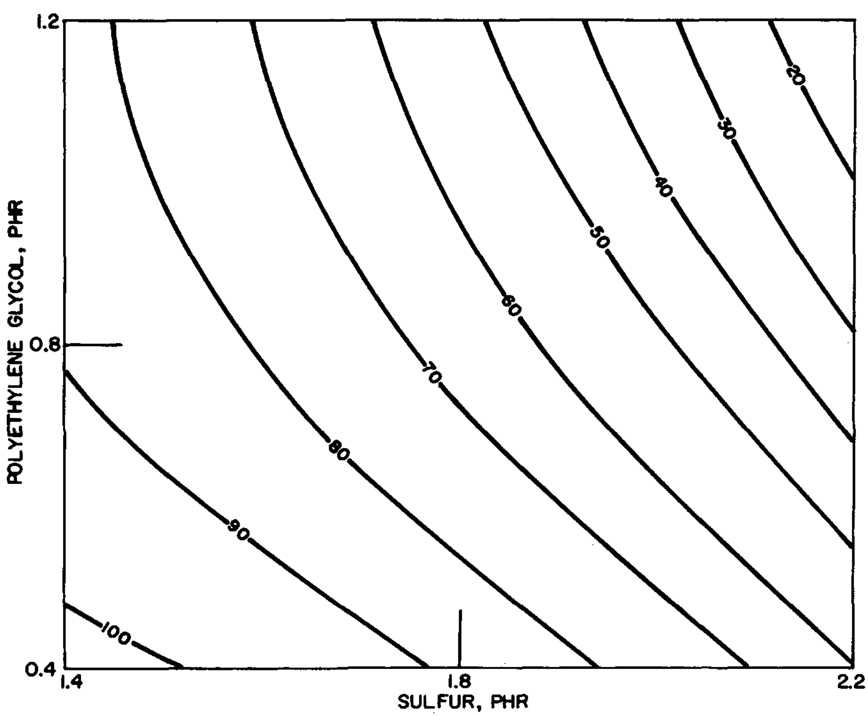

Fig. 12. Flex index as a function of sulfur and glycol.

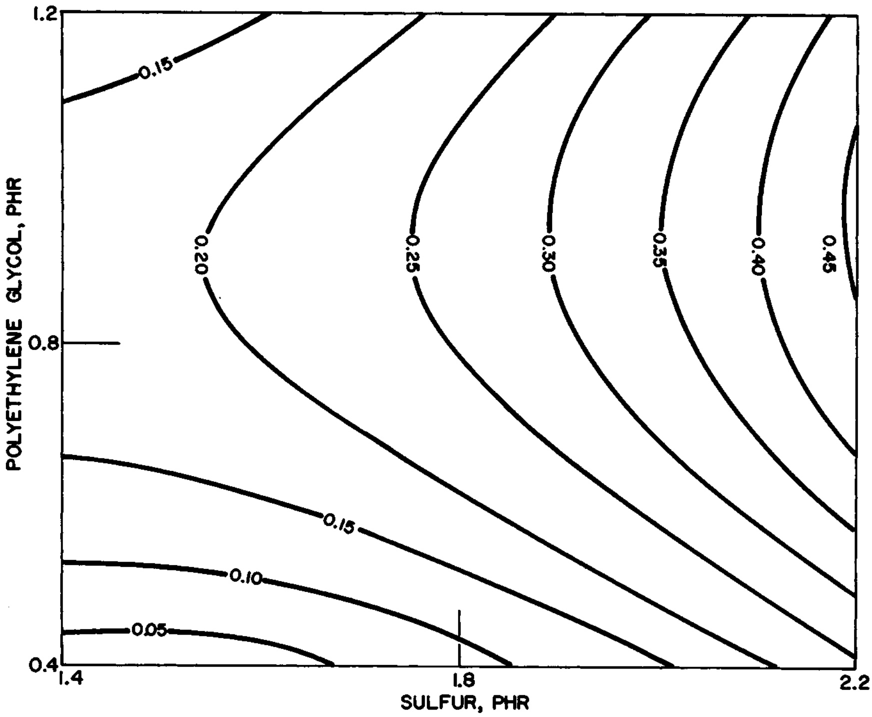

Fig. 13. Maximum rate of growth as a function of sulfur and glycol.

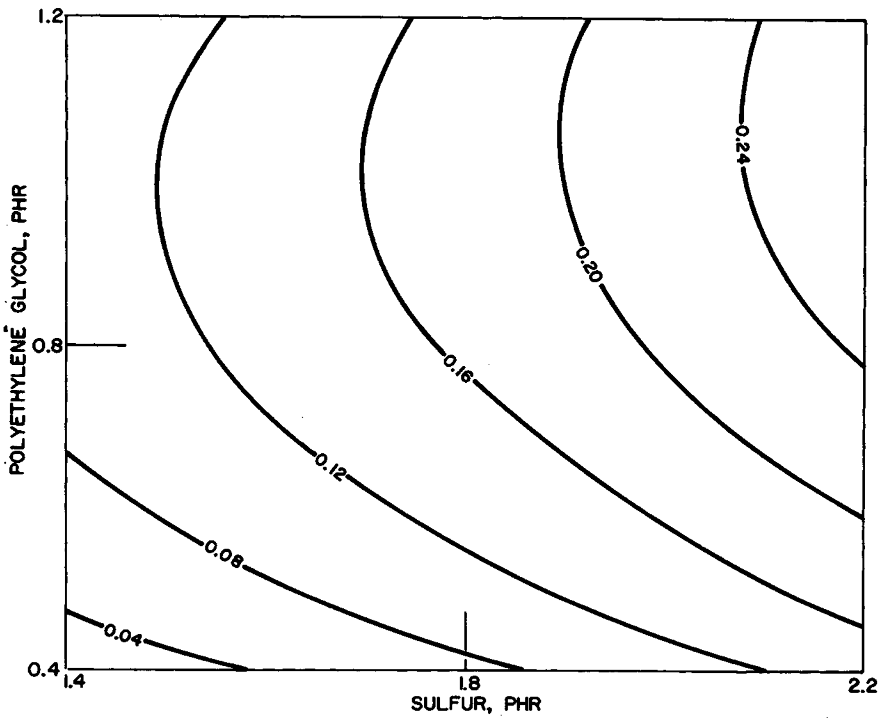

Fig. 14. Mean rate of growth from initiation point as a function of sulfur and glycol.

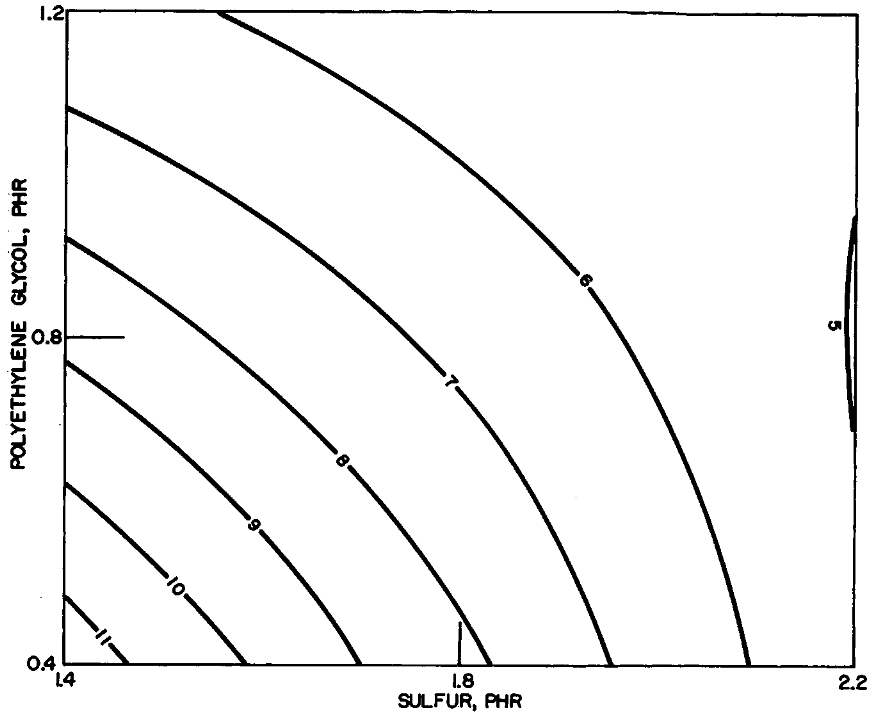

Fig. 15. Log of initiation point as a function of sulfur and glycol.

Figures 13 and 14 for maximum and mean growth rate, respectively, indicate that growth rate was strongly dependent upon sulfur concentration but only slightly dependent upon glycol concentrations.

Finally, Figure 15 for ln (initiation point) indicates a decrease with both increased sulfur and increased glycol. The natural log was used to fit the polynomial (12) to initiation point because the range of this response covered several orders of magnitude. A polynomial is not a flexible enough equation form to fit such wide variations, so the log must be taken to get an adequate fit.

DISCUSSION

The methods for treating elastomer flex data proposed in this paper are far superior to those proposed by ASTM D813 because the results are more meaningful and have greater precision. Furthermore, the method can be programmed for a computer so that, from input consisting only of the original data, any number of flex characteristics can be obtained in one pass through the computer.

The most meaningful results are obtained when all test pieces are run to failure. In this instance, the preferred flex characteristics are the initiation point X_0 and the average rate of growth between X_0 and X_∞. When the test is terminated at a fixed number of cycles, the initiation point X_0 is a valuable characteristic if most specimens have begun to crack. Furthermore, if all of the samples have not failed, an average rate of growth between, say, 1% and 10% cut growth could be calculated. The flex index is a very convenient, overall index of flex behavior which should be calculated whether all of the specimens are flexed to failure or not.

The development of eq. (1) is not the significant feature of this work. Rather, it is the general principle of fitting a model to the data and drawing conclusions from the equation rather than from the raw data. Although eq. (1) is quite flexible and is expected to fit most elastomer flex data, there may be some instances where it does not fit. This would not diminish the significance of this study, but would only necessitate the finding of a more flexible model.

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