

# A Fractal Approach to the Mixing–Microstructure–Property Relationship for Rubber Compounds

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Received 10 March 2000; accepted 2 May 2000

**ABSTRACT:** This research is concerned with exploration of the utility of fractal methods for characterizing the mixing treatment applied to a rubber compound and for characterizing the filler dispersion developed during mixing. Fractal analysis also is used for characterization of the fracture surfaces generated during tensile testing of vulcanized samples. For the purposes, the maximum entropy method, to rubber mixing, and the box counting method are applied to analyze the mixing treatment and the filler dispersion, respectively. These methods are effectively used, and it is found that fractal dimensions of mixer power traces and fracture surfaces of vulcanized rubber decrease with the evolution of mixing time while the fractal dimension of the state-of-mix also decreases. The relationship of the fractal dimensions thus determined with conventional properties such as tensile strength, electrical resistance, and fracture surfaces are then explored. Finally, the utility of fractal methods for establishing mixing–microstructure–property relationships is compared with more conventional and well-established methods such as electrical conductivity and carbon black dispersion. It is found that the characterization by the fractal concept agrees with the conclusions from these conventional methods. In addition, it becomes possible to interpret the relationships between these conventional methods with the help of the fractal concept. © 2000 John Wiley & Sons, Inc. *J Appl Polym Sci* 78: 1555–1565, 2000

**Key words:** fractal; rubber mixing; carbon black dispersion; maximum entropy method; box counting method

## INTRODUCTION

The study of rubber technology is interesting because of the large number of potential applications and range of new concepts. The study of rubber technology includes rheological characterization of polymer flow, chemical reactions such as vulcanization and bound rubber formation, the material sciences of ingredients, and many engineering subjects such as fracture mechanism and adhesion.

These rubber technologies can be classified into three fields:

1. the materials and ingredients of rubber compounds,
2. polymer processing, and
3. the properties of rubber compounds.

In addition, the uniqueness of polymer technology lies in the fact that polymer processing and the properties of the final products are more closely related to each other than other materials. Thus extensive consideration of the mechanism of processing and the behavior of materials are required if accurately specified final products are to be obtained. To obtain this broad perspective, ranging from rubber processing to material science, it is necessary to “focus attention on underlying engineering and scientific principles, which are also the basics of the unifying elements to all

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*Journal of Applied Polymer Science*, Vol. 78, 1555–1565 (2000)  
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processes."<sup>1</sup> This research paper is an examination of the possibility that fractal concepts could be used to characterize and unify rubber processing and the material sciences.

Four aspects from within the field of rubber technology were chosen to define the research with regard to the application of fractal technology to rubber processing, and these are as follows:

### **Characterization of Polymer Mixing**

This topic was developed directly from K. Nishimoto's<sup>2</sup> work. Mixing is very important and the first step in rubber processing, and the quality of mixed stock seriously affects the processability downstream. To characterize a mixing process, a mixer power trace is analyzed by the fractal concept to represent industrial parameters for a mixing process.

### **Evaluation of the State of Mix of Carbon Black in Rubber Compounds**

In the case of rubber compounds, the most important requirement at the mixing stage is to achieve uniform mix of carbon black, as the other ingredients of a compound can be distributed uniformly with a diffusion mechanism downstream. Although the importance of the state of mix of carbon black has been recognized for a long time, research has not been succeeded in explaining the complicated rubber properties with enough accuracy. Therefore, the characterization of the state of mix was chosen as the second topic for this research paper. This topic can be regarded as an evolution of the work initiated by P. K. Freakely.<sup>3</sup>

### **An Application of the Relationship Between the Fractal Concept and the Rubber Fracture Mechanism**

The third topic is the characterization of the fracture surface of vulcanized rubber sample. It is necessary to evaluate the validity of Medalia's<sup>4</sup> mechanism or to propose a potential experimental method. This topic is an attempt to characterize the fracture surface with the fractal concept.

### **The Relationship Between Mixing, the State of Mix, and Fracture Mechanism**

In addition to an examination of the applicability of the fractal concept to these three topics, it is also necessary to consider the relationship be-

tween operating conditions, the state of mix, and the tensile strength at break. Fractals may provide an effective framework for dealing with this problem. In this paper, the application of fractals to characterize the relationship between rubber mixing and the ultimate properties of the resulting compound is explored. Because of the lack of a consistent explanation for both unvulcanized and vulcanized properties, it is very difficult to obtain an optimum operating condition for rubber processing that satisfies many factors (processability, vulcanizate properties, and process cost). The application of fractals is expected to describe how these requirements can be optimized. Much research is currently being undertaken to characterize rubber properties from the fractal point of view. However, a systematic fractal application to rubber technology is not available at the moment. Such a systematic fractal application has the potential to connect existing research within a comprehensive theoretical framework.

## **EXPERIMENTAL**

### **Material Preparation**

For comparison purposes, two recipes were adopted based on different matrix polymers by referring to the American Standard for Testing and Materials (Tables I and II). In the case of the styrene-butadiene rubber (SBR) recipe, SBR1502 was used. In the natural rubber (NR) recipe, SMR 20 (Standard Malaysian Rubber) was used as the matrix polymer. The other ingredients were as used for the SBR recipe.

### **Mixing and Operating Conditions**

A Francis Shaw K1 Internal mixer (chamber volume 5.5 L) was used for the mixing. The conditions were as follows: Three rotor speeds (30, 50, 70 rpm) were used. And the fill factor was 0.5. Circulating cooling water temperature of rotor was 40°C and the mixing pattern was two-stage mixing.

To change the state of mix of carbon black, various mixing times after adding the carbon black into the polymer were adopted (90, 180, 300, 360, 420 s) for three rotor revolution speeds (30, 50, 70 rpm). At first, polymer was added to the internal mixer and masticated for 2 min. Then the carbon black was added and mixed for the selected time to obtain samples that had various

**Table I Experimental Recipe for SBR Compounds (ASTM D3186)**

No.	Ingredients	Amount (phr)	Supplier
1	Styrene–butadiene rubber	100	Goodyear
2	Carbon black N330	40	Cabot
3	Zinc oxide	3	Schill Seilacher
4	Stearic acid	1	Schill Seilacher
5	CBS <sup>a</sup>	2	Schill Seilacher
6	Sulfur	2	Schill Seilacher

<sup>a</sup> *N*-cyclohexyl-2-mercaptobenzothiazolyl sulfenamide.

states of mix. When a certain mixing time had passed, the rubber compounds were discharged from the mixer and passed once through the two-roll mill to minimize any further mixing. These rubber compounds were left at room temperature for 2 h to cool them down to room temperature. Then mixed stocks were cut into strips for refeeding into the mixer for the second mixing. At the beginning of the second stage, sulfur and accelerator were added, and mixed for 90 s. After the second mixing stage, the mixed rubber compounds were passed once through the two-roll mill to avoid further mixing.

#### Fourier Transform Analysis of the Mixer Power Chart

The duration after adding carbon black and before dumping in the first stage was used to characterize the mixing behavior of the carbon black with the matrix polymer. The Fourier transform was applied to the power charts and the results were analyzed by using fractal method. In this experiment, the maximum entropy method (MEM) was used to obtain precise data. Figure 1 shows the result of the Fourier transform analysis. There are two peaks in the chart. Each peak

corresponds to the rotation of the tips of the rotor. There is another peak in the vicinity of zero area. This peak corresponds to the total shape of the power chart and this peak was used to obtain a fractal dimension because it characterizes the mixing behavior.

#### Measurement of Electrical Resistance of Unvulcanized Compounds

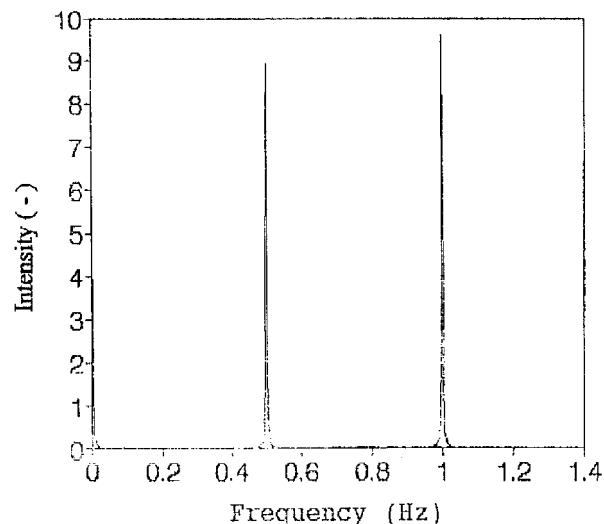
Electrical resistance was measured to correlate the fractal method for evaluating the state of mix to existing data. This electrical resistance measurement is a widely accepted method for evaluating the state of mix of carbon black. The main ideas and construction of the apparatus are based on Boonstra's method.<sup>5</sup>

Rectangular samples of unvulcanized rubber with 50 × 50 × 20 mm were cut out from the mixed rubber compound by means of a knife or

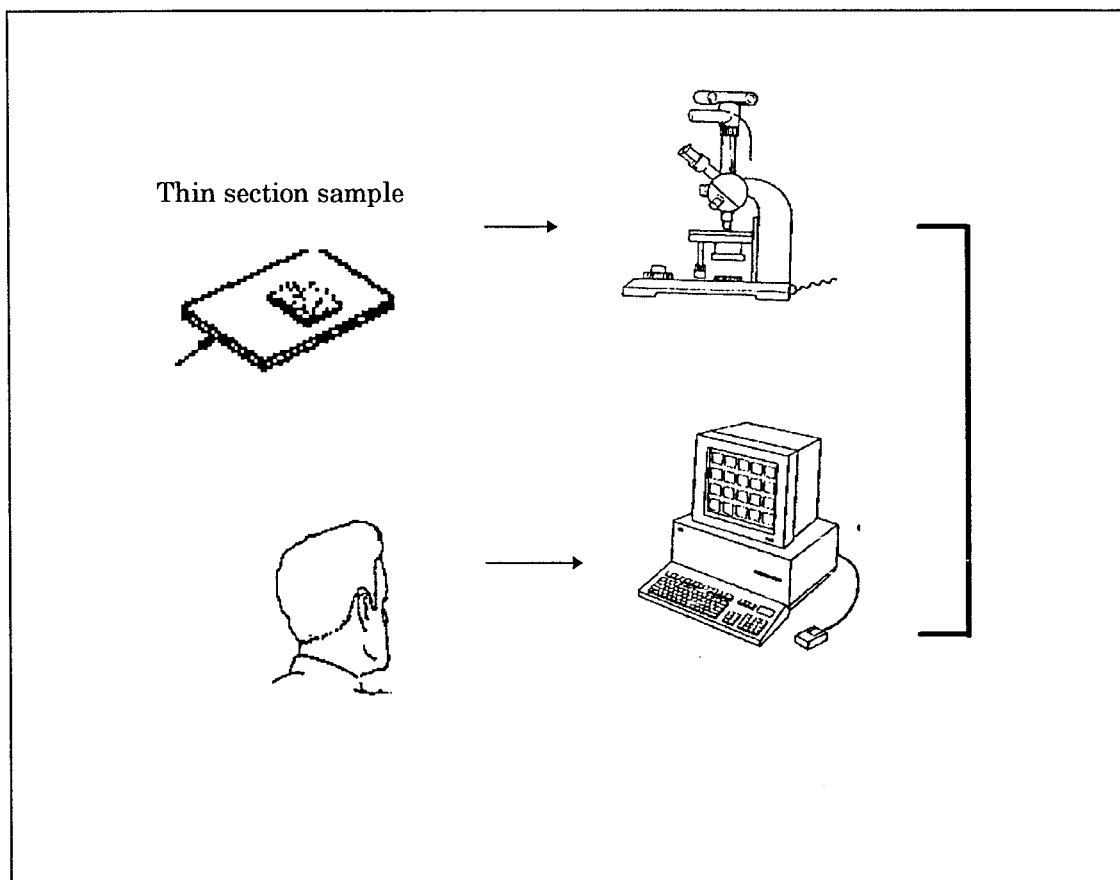
**Table II Experimental Recipe for NR Compounds ASTM (D3184)**

No.	Ingredients	Amount (phr)	Supplier
1	Natural rubber	100	
2	Carbon black N330	40	Cabot
3	Zinc oxide	5	Schill Seilacher
4	Stearic acid	1	Schill Seilacher
5	TBBS <sup>a</sup>	1	Schill Seilacher
6	Sulfur	2.5	Schill Seilacher

<sup>a</sup> *N*-*tert*-butyl-2-mercaptobenzothiazole sulfenamide.



**Figure 1** Power spectrum of a mixer power trace obtained by the Fourier transform.



**Figure 2** Schematic explanation of experimental procedure for evaluating the state of mix of carbon black.

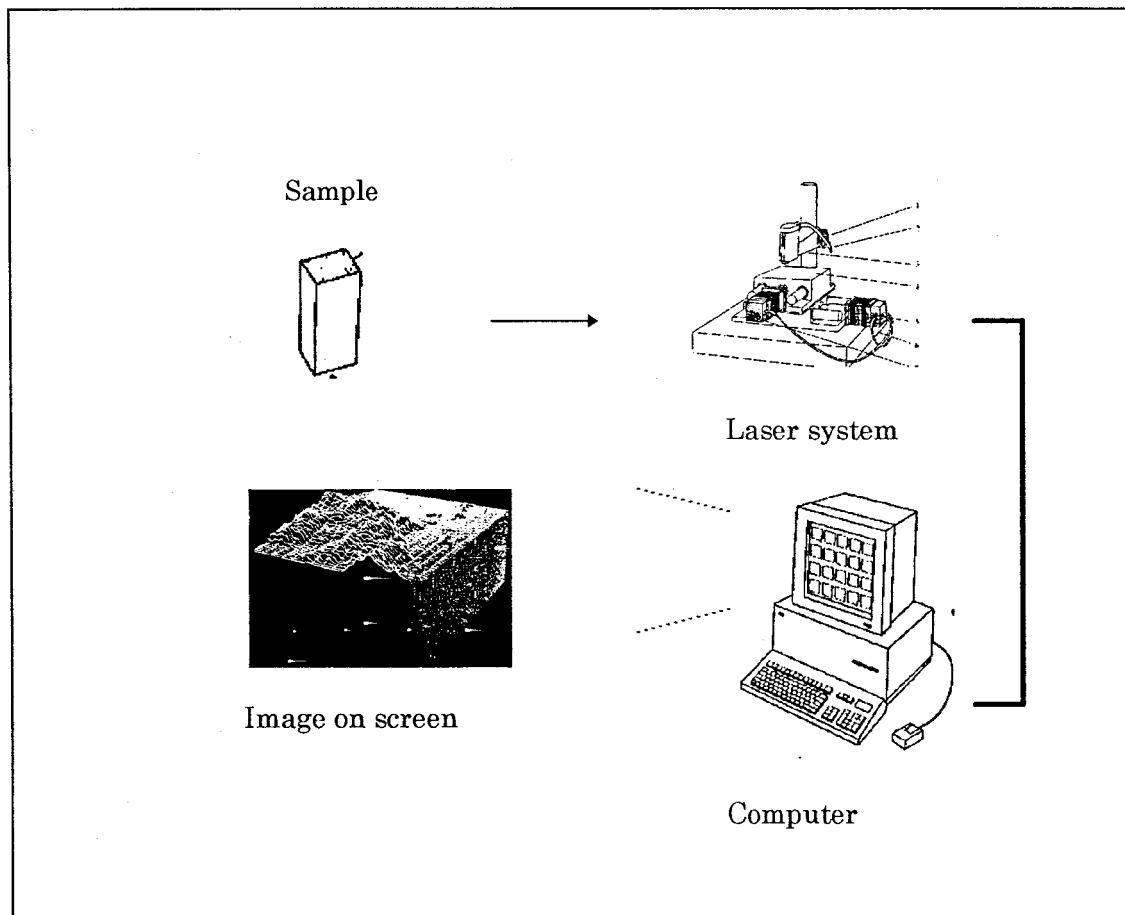
scissors. Before the electrical resistance measurements, the samples were placed in a heating oven at 60°C for 30 min to ensure a temperature equilibrium in the bulk of the samples before they were subjected to the measurements. During the measurements, three readings were taken at 15, 30, and 60 s after starting the test to check the reduction in resistance, but only the value taken at 60 s was used in this experiment.

### Molding

Vulcanization was carried out by using a hydraulic press. Slab sheets with 2 mm thickness were prepared for the experiments. Molding temperature and pressure were 150°C and 9.8 MPa, respectively. The cure time depends on each sample. Thus,  $T_{0.9}$  (90% cure time) cure was measured by a Wallace Precision Cure Analyser (Wallace PCA) with the molding conditions and used as molding time.

### Carbon Black Dispersion Analysis

The box counting method was used to measure the fractal dimension of carbon black dispersion. Figure 2 shows the procedure. By using a microtome, thin section samples from the vulcanized rubber sheet molded for the tensile test were prepared and they were put on to a slide glass. A light microscope and video system were then used to magnify the sample. Total magnification was 720 folds. Four transparency sheets, which have different mesh sizes, were used to count the boxes (lattices on the transparency sheet). The box sizes on the transparency sheets were 1, 2, 3, and 4 cm. These transparency sheets were put on the CRT screen and the boxes that contained the carbon black agglomerates were identified visually. A software package named VID V (Sunpotics, UK) was used to count the number of boxes that contained the carbon black agglomerates.



**Figure 3** Rodenstock RM 600 laser stylus.

### Measurements of the Fracture Surface of Tensile Samples

Figure 3 shows a schematic procedure for measurements of the fracture surface. Rubber samples were fixed on the measuring platform of laser surface detection machine (RM 600 Laser stylus by Rodenstock). The horizontal position of the platform is shifted by two stepping motors, which enable controlled movements on the order of 0.01 mm. The control unit operated these motors and detected the horizontal positions. The laser sensor unit detected the roughness on the sample surface with a resolution of 1  $\mu\text{m}$ . The computer displays the measured roughness on a screen.

A line measurement was used for detecting the fractal dimensions of the fracture surface. For this purpose, a line detection of the surface roughness was carried out at the intervals of 10  $\mu\text{m}$  on at least 10 positions to obtain a statistical mean value. Again, each line spectrum had a resolution power of 10  $\mu\text{m}$  order. These measured data were

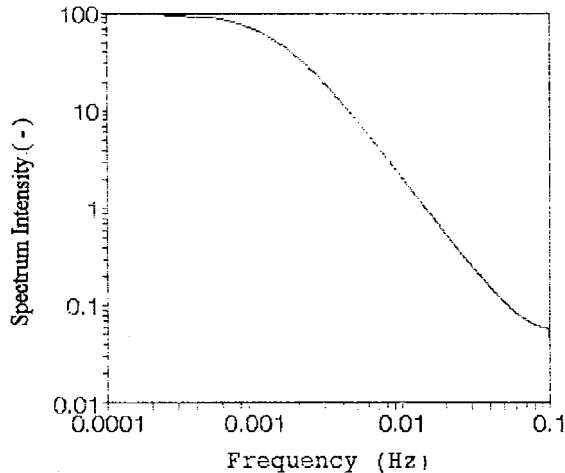
stored by a computer and used later for the fractal analysis by using the maximum entropy calculation program. The samples being subjected to the tensile measurements were used for measuring the fracture surfaces. This enables to correlate the fractal dimension to tensile strength and the state of mix.

### Measurement of Tensile Strength

The vulcanized sample were cut into dumbbell-shaped specimens by using a standard die cutter according to BS 903 type 1. The stress strain tests of these samples were carried out with Hounsfield 500L testing machine at room temperature.

## RESULTS AND DISCUSSION

The validity of fractal applications to rubber technology is discussed in the light of the experimen-



**Figure 4** Determination of fractal dimension. From the slope of this graph, the fractal dimension is calculated.

tal results. The purpose is to evaluate the applicability of fractal to rubber properties, such as rubber mixing, the state of mix, fracture surface, and their relationship.

#### Fractal Analysis of the Mixer Power Trace

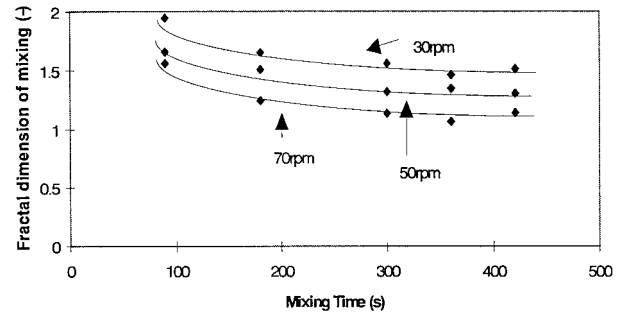
A mixer power trace is one type of power spectrum. The relationship between the power of a mixer power trace and frequency is given by

$$S(f) \propto f^{-\beta}$$

$$D_f = 5 - 2\beta \quad (1)$$

where  $S$  is the intensity of the spectrum,  $f$  the frequency,  $\beta$  a power, and  $D_f$  the fractal dimension.

Figure 4 demonstrates one example of the log-log plot to determine the fractal dimension from the power trace of the mixing process. (Figure 4 is a rescaled graph of the vicinity of zero in Figure 1.) From the slope of the figure, the power is obtained at first, then the fractal dimension is calculated from eq. (1). In this case, the value of  $D_f$  is 1.58. Some literature is available to check the validity of the data. Nishimoto<sup>2</sup> reported the fractal dimension as ranging from about 1.1 to 1.6. The figure is in broad agreement with the result obtained in this study. Unfortunately, a procedure to derive a fractal dimension from the basic equation (Navies-Stoker's equation) of hydrodynamics has not been carried out, so the data



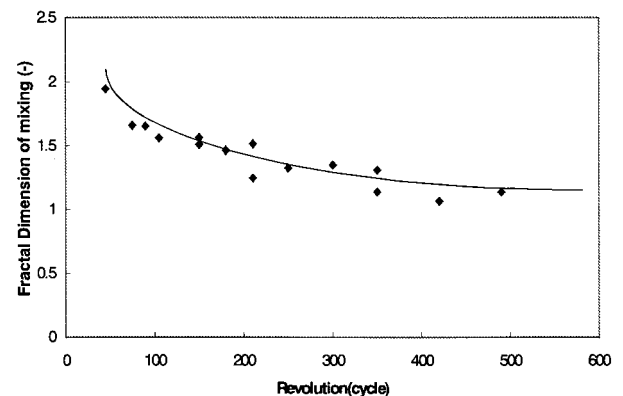
**Figure 5** The relationship between mixing time and fractal dimensions (SBR sample).

obtained here is justified only on the basis of existing data.

The fractal dimensions of mixer power traces are shown in Figure 5 as a function of mixing time. There are two features in the graph:

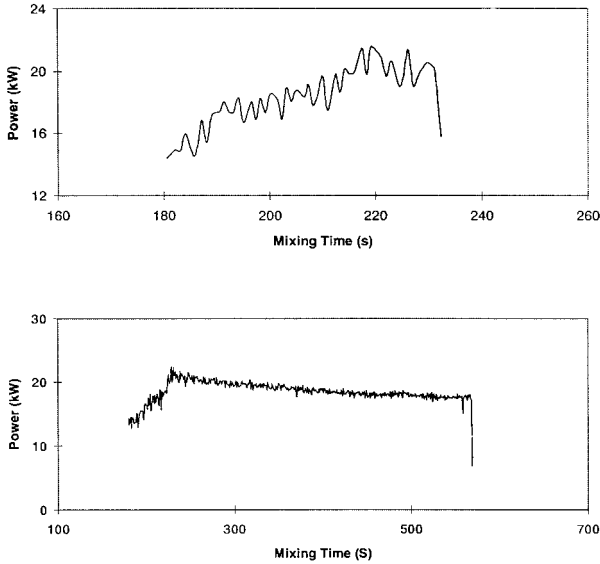
1. The longer the mixing time, the smaller the fractal dimension.
2. The greater the number of revolutions, the smaller the fractal dimensions when compared at a certain mixing time.

These two features can be integrated by introducing a normalizing method. Clark and Freakley<sup>2</sup> proposed that the accumulated rotor revolution number mainly controls the state of mix in a certain condition. Their proposal is applied to the fractal analysis and is shown in Figure 6. In Figure 6, the abscissa is a multiplication of mixing time and a rotor revolution speed. Thus, it stands for the total rotor revolution number during a mixing. It is obvious from the graph that the



**Figure 6** Normalized relationship between mixing time and fractal dimensions. The abscissa is a product of rotor speed and mixing time (SBR).





**Figure 7** Mixer power trace of SBR compound: (a) 90 s mixing; (b) 420 s mixing.

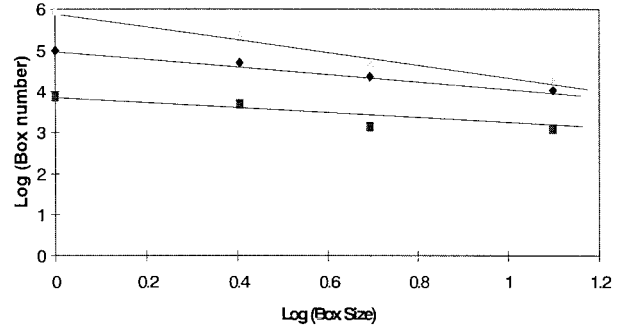
fractal dimension of a mixing is characteristic of a mixing process and it is related to the total rotor revolution number.

The two features arise from the fact that the fractal dimension reflects the total shape of a mixer power chart. The power chart with a big fractal dimension tends to cover a larger two-dimensional surface with its trace while a power chart with a small fractal dimension tends to be a simple line. This explanation is confirmed by checking the original power chart shown in Figure 7. In the case of graph (a) of Figure 7, the fluctuation of the power trace is more prominent than in graph (b); also, the power trace tends to cover a whole plane than the corresponding long time mixing in graph (b). Therefore, the power trace of graph (a) is more complicated than graph (a) and results in a bigger fractal dimension.

**Fractal Analysis of Carbon Black Dispersion**

A box counting method was used to determine the fractal characteristics of the state of mix. Then, a log–log plot was constructed. Figure 8 demonstrates the validity of the fractal concept as a means of evaluating the state of mix, because the graph follows the power rule. In Figure 8, some experimental scattering is detected. Two main factors contribute to this:

1. The lack of objectivity. The experiments were carried out visually, not by using a



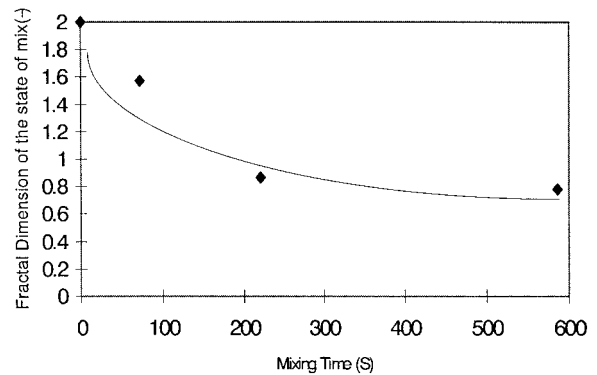
**Figure 8** An example of the box counting method to determine the state of mix of carbon black.

reliable computer software package. Visually, it is very difficult to judge consistently whether a box contains carbon black or not.

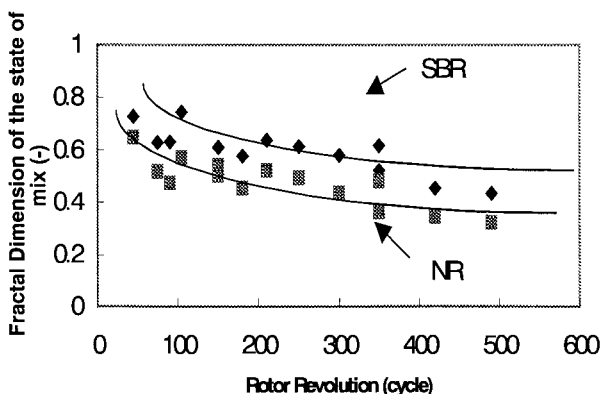
2. The box counting method. In this method, the box size (mesh size) had to be changed for the determination of fractal dimensions.

The precision decreases as the box size increases. In the big box size region, the data points in Figure 8 show the scattering caused by this factor. To overcome this, it is better to adopt a suitable range of microscope magnification, by considering the precision of experimental instruments. Figure 9 shows the relationship between the fractal dimension of carbon black dispersion and mixing time. The fractal dimension of carbon black dispersion becomes smaller and approaches a constant value as a mixing proceeds. This phenomena can be explained by the following mechanism.

Initially, added carbon black in a system simply covers the surface of the matrix polymer in an



**Figure 9** A relationship between fractal dimension of carbon dispersion and mixing time (SBR sample).



**Figure 10** A time dependence of the fractal dimension of the state of mix (◆) SBR and (■) NR have a different recipe).

internal mixer. Then carbon black is incorporated into the polymer matrix. The incorporated carbon black reduces its size by both shear and elongation forces during mixing. This mechanism means that carbon black tends to cover the whole surface of a thin section sample taken after a short mixing process, while the thin section sample will have a scattered carbon black agglomerate after an efficient mixing time. The fractal dimension of carbon black dispersion decreases as carbon agglomerates disappear from a thin section sample. Therefore, the longer the mixing time, the smaller the fractal dimension of carbon black dispersion.

Three characteristic points in Figure 9 have to be considered:

1. The starting point of mixing: In Figure 9, the number "2" is a theoretical value because a surface completely covered by carbon black is a two-dimensional distribution. The extrapolation from the experimental data agrees with the theoretical value. This fact demonstrates the validity of fractal analysis used here.
2. The middle point of mixing: As can be seen from Figure 9, the fractal dimension at 210 s approaches a constant value. It is reasonable to consider that this point corresponds to the starting point of the steady state of the mixing process.
3. The end point of mixing: It is necessary to consider the meaning of a steady state fractal dimension,  $d = 0.78$ . The figure is smaller than one and it means that the carbon black dispersion in the matrix is almost randomly distributed at this stage.

This conclusion is reasonable from our experience, which tells us that well-mixed samples have a random carbon black dispersion.

Figure 10 shows the relationship between mixing time and the fractal dimensions of SBR and NR samples. The SBR and NR samples have different recipes; therefore it is not easy to compare the results between these two formulations.

Two features have to be mentioned here:

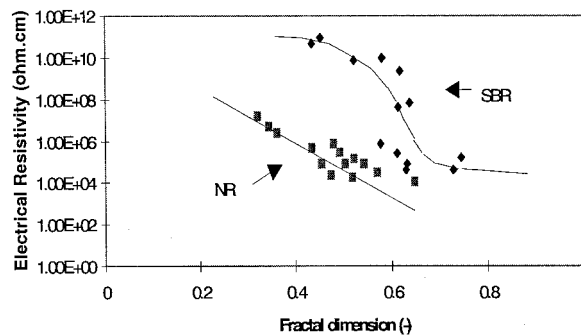
The first feature is the plateau value of the fractal dimension at long mixing times. The surface density of the detected agglomerates in the thin section sample mainly contributes to the difference in the fractal dimension between SBR and NR samples. It is known that NR compounds show a quicker incorporation time than SBR. Thus, an NR compound reduces the number of carbon agglomerate more extensively than SBR compounds. This mechanism results in a smaller value of the fractal dimension of the NR compound.

The second feature is the rate of decay of the fractal dimension. The fractal dimension at zero mixing time is two. If the value shown in Figure 10 is extrapolated to two, it is obvious that NR has a bigger rate of decay than the SBR system. Again, the mechanism mentioned above is applicable to the phenomenon.

In conclusion, these experiments positively shows that the state of mix of carbon black in rubber compounds can be evaluated effectively using the fractal concept.

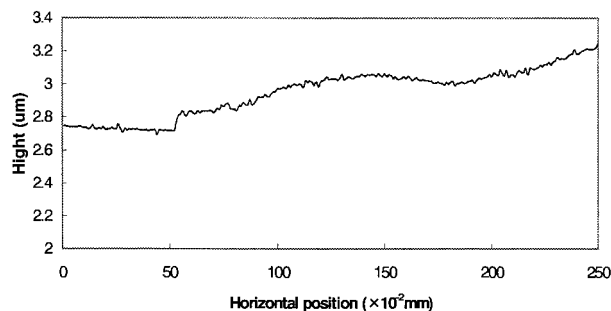
### The Relationship Between the Fractal and Existing Methods for Evaluating Carbon Black Dispersion

To consider the microscopic dispersion effect, the relationship between electrical resistance and the



**Figure 11** Electrical resistance versus fractal dimensions of the state of mix: (◆) SBR; (■) NR.





**Figure 12** A contour profile of a fracture surface.

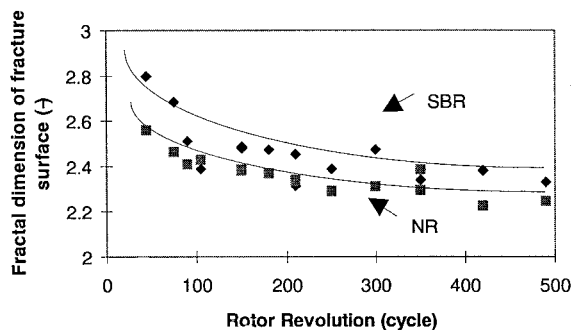
fractal dimension is shown in Figure 11. As can be seen from the graph, the relationship is not simple in the SBR compounds. In addition, there is a threshold around 0.6 of the fractal dimension in the SBR compounds. This point can be regarded as the point where the macroscopic carbon black chunk disappeared. The point naturally depends on the rate of carbon black incorporation into the matrix polymer. In the case of NR compounds, the threshold cannot be seen in Figure 11 because the carbon black incorporation in an NR compound is quicker than in an SBR compound. Therefore, in this experimental range, it was impossible to check the threshold.

The difference of electrical resistance between SBR and NR compounds also has to be considered. In Figure 11, the electrical resistance of NR is smaller than SBR compounds, even in the case of well-mixed samples. It was reported<sup>6</sup> that NR is more sensitive to the amount of carbon black than SBR. Although this sensitivity can be regarded as a main factor of the difference, the mechanism was not suggested even in the original paper.

### Fracture Surfaces of Tensile Strength Samples

To apply the fractal concept to the fracture surface of rubber samples, the contour profile was measured. Figure 12 shows one example of such experiments. To carry out a Fourier analysis, it is possible to apply the same type of software that was used for the analysis of the mixer power trace.

In the case of a fracture surface, the relationship between a fractal dimension ( $D$ ) and a power ( $\beta$ ) is given by  $\beta = 7 - 2D$ . As for the other analyses, normalized data is shown in Figure 13. For both the SBR and NR samples, the fractal dimensions decrease as mixing proceeds. As men-

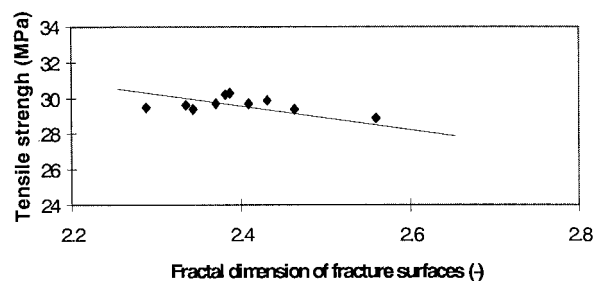


**Figure 13** A normalized fractal dimension of the fracture surface. (◆) SBR; (■) NR.

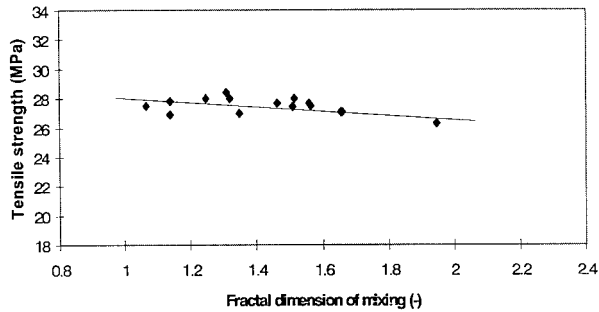
tioned before, the fractal dimension represents the characteristic of the whole measured surface. The smoother the fracture surface, the smaller the fractal dimension. It is easily observed that a badly mixed compound has a rough fracture surface, while a well-mixed compound has a smooth fracture surface. Figure 13 quantifies such observations based on the fractal concept.

In Figure 13, NR has a smaller fractal dimension than SBR. Such a difference may arise from the difference in the formulation or carbon black incorporation speed. Although the mechanisms that account for the differences were not the subject of this research paper, it is possible to propose that residual carbon black agglomerates in the samples control the roughness of the fracture surface.

Because they tend to be both an initiation point of a fracture process and a source of increased fracture energy due to stress intensification. As previously seen, NR compounds have a shorter carbon black incorporation time than SBR compounds. Therefore NR compounds experience a longer dispersion process than SBR compounds when they are compared at the same total rotor revolutions. Through this mechanism, the differ-



**Figure 14** A relationship between tensile strength and the fractal dimension of SBR samples.



**Figure 15** Tensile strength and fractal dimension of mixer power trace (SBR compound).

ence of carbon black incorporation time during mixing resulted in the difference in fractal dimension of fracture surface of SBR and NR compounds in Figure 13.

Figure 14 shows the relationship between tensile strength and the fractal dimension of the fracture surface. The tensile strength is inversely proportional to the fractal dimension of the fracture surfaces. In this experiment, the difference was only mixing time in each series of SBR compounds. The other factors that contributed to tensile strength were kept at minimum level. Therefore, the reason why a relatively straight relationship between the fractal dimension of fracture surface and the tensile strength was obtained arises from the fact that the state of mix mainly controls the tensile strength in each compound. It can be concluded that the smaller the fractal dimension, the higher the tensile strength because a smaller fractal dimension means that a compound is in a well-mixed state.

#### The Relationship Between the Tensile Strength and the Fractal Dimension of Mixing

Figure 15 shows the relationship between a fractal dimension of the mixer power trace and the tensile strength at break. It can be seen that the tensile strength of SBR compounds has a linear relationship with the fractal dimension of mixing. The tensile strength at break is inevitably a function of molecular weight, ingredients, mixing conditions, and cure conditions. In the experiment, the differences among samples in a series of SBR compounds are confined to mixing time and rotor revolution speed. Thus, it is reasonable to suggest that the state of mix of carbon black is the most important factor in this experiment, and this resulted in a linear relationship between tensile strength and the fractal dimension of mixing.

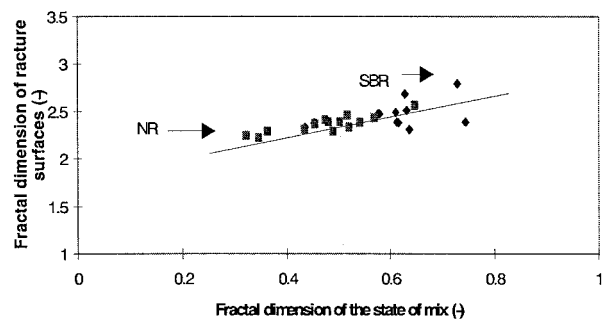
In conclusion, it can be said that a fractal concept can be an elemental concept to understand the relationship between tensile strength and carbon black dispersion.

#### The Relationship of the Fractal Dimension Between the State of Mix and Fracture Surface

Figure 16 shows a fractal relationship between the state of mix and the fracture surfaces using the fractal concept. It is clear from this graph that the fractal dimensions connect both properties quite well. Although only one line is drawn in the graph, it should not be considered that the fractal dimension of fracture surfaces is only a function of the state of mix. An overlap of the plots found here in Figure 16 for SBR and NR compounds is merely a coincidence. Each compound is expected to have its own intrinsic line.

#### CONCLUSIONS

1. The fractal concept has been applied successfully to characterize the following:
  - (a) The mixer power trace: MEM is applied to obtain a fractal dimension of mixing from a mixer power -trace. The fractal dimension decreases steadily as the total number of rotor revolution increases. The mechanisms that contribute to the fractal dimensions are disagglomeration of carbon black.
  - (b) Carbon black dispersion: The state of mix of carbon black is characterized by the fractal concept with the help of a box counting method. The fractal dimension of the state of mix decreases steadily as



**Figure 16** Fractal application for the evaluation of the state of mix and the fracture surfaces. Abscissa and ordinate are fractal dimension of the state of mix and fracture surfaces, respectively: (◆) SBR; (■) NR.

mixing time proceeds due to the disagglomeration of carbon black resulting in the disappearance of carbon black agglomerates.

- (c) Fracture surfaces: The fracture surface of the tensile sample is characterized by using the fractal concept. The fractal dimension of the fracture surface decreases as the state of mix is improved. Tensile strength at break of SBR compounds are proportional to the fractal dimension of the fracture surface.
2. The relationship between mixing treatment (power trace), state of mix (carbon black dispersion), and fracture surfaces: The relationship between the fractal dimensions of mixing, the state of mix and fracture surface are discussed, and relatively straightforward linear and quadratic relationships are found. These simple relationships arise from the fact that the carbon black dispersion and its dispersion process dominate such properties in the experiments in this paper.
  3. Relationships between properties characterized by fractal analysis and characterized by

conventional methods: The results of fractal method of the carbon black dispersion are compared to conventional measurement of electrical resistance. The agreement of the fractal analysis and the conventional analysis gives the confidence of the validity of the fractal methods.

The author would like to express appreciation to Dr. P. K. Freakley for his support and advice to carry out this work.

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