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Microwave Diagnosis of Rubber Compounds

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Abstract—The results of a study investigating the dielectric properties of rubber compounds and their constituents in the frequency range of 5 to 24 GHz are presented. A completely filled short circuited waveguide technique was used to conduct these dielectric measurements. The influence of carbon black content in rubber was investigated for carefully prepared rubber samples. The results showed that for all frequencies, the dielectric constant increases as a function of increasing carbon black content. The variation of dielectric constant values decreases as frequency increases. The presence of curatives in uncured rubber samples was also detected, which indicates the sensitivity of microwaves to the chemical reaction triggered by curatives.

I. INTRODUCTION

DIELECTRIC properties of rubber have been investigated during microwave curing process [1], [2]. The objectives of these studies have been to choose the optimum rubber composition as a function of microwave absorption (heating), and to show that microwave curing produces rubber compounds with similar properties as those produced by the standard curing process. These measurements were conducted using cavity techniques. Other investigators have reported tensor dielectric constant measurement of anisotropic rubber samples as it pertains to microwave shielding [3], [4].

Recently, microwave techniques have been used as diagnostic tools for investigation of chemically reacting materials (i.e., epoxy) and studying polymerization process [5], [6]. These studies show the potential of microwaves for monitoring chemical reactions associated with the formation of cross-linked molecular networks.

Very little is known about rubber dielectric properties as a function of microwave frequency. Also there is a lack of information about the behavior of rubber compound constituents (ethylene propylene diene rubbers or EPDM, oil, mineral filler, etc.) as a function of microwave frequency. Furthermore, there is very little to none reported on the influence of constituent volume fraction variation and chemical reactions among these constituents on the rubber relative dielectric constant. These types of information may lead to important practical applications such as controlling rubber mixing process and introduction of new materials with predictable properties (i.e., microwave absorbers).

In this paper we discuss the results of a study of the dielectric properties of various rubber stocks as a function of microwave frequency. To perform this task, first we studied the dielectric properties of rubber constituents namely: EPDM, mineral filler, oil, zinc oxide and curatives. Subsequently, the influence of carbon black volume percentage on the dielectric properties of rubber was investigated. The ability of microwaves to detect the presence of curatives in uncured rubber, and the role of frequency in this detection is presented. The impact of chemical reactions on the dielectric constant is also discussed.

II. MEASUREMENT PROCEDURE

There are various well established techniques available for dielectric constant measurement of materials [7]. However, we had to consider a technique that possessed the following characteristics:

1) be able to measure low and high loss dielectric materials such as EPDM and carbon black loaded rubber with good accuracy,
2) be inexpensive and simple so that it could be reproduced easily and be operated by those who are not trained in this area,
3) be suitable for measuring powders, fluids and bulk materials.

Initially we considered a dual arm waveguide bridge [8] and partially filled waveguide techniques [9]. These were soon abandoned as the former technique requires quite a few microwave components which must possess very good frequency characteristics, and the latter technique is only suitable for sheet type materials. Finally, the well known completely filled short circuited waveguide technique was used [10], [11], which was the best compromise considering our measurement requirements. This method is based on the measurement of the complex reflection coefficient whose magnitude and phase are used to extract dielectric properties of the samples.

Fig. 1 shows the measurement apparatus that was used to measure the dielectric properties of rubber samples and their constituents. Five different waveguide set-ups operating in 3.95–5.85 GHz (R-48), 5.85–8.2 GHz (R-70), 8.2–12.4 GHz...
(R-100), 12.4–18 GHz (R-140), and 18–26.5 GHz (R-220) were used. The oscillator generates a microwave signal at the desired frequency. This signal is then fed through a precision attenuator followed by an isolator into a slotted waveguide. The standing wave characteristics inside the waveguide are measured via a detector and a sensitive voltmeter. The measurements were performed using precisely machined sample holders to accommodate samples of different thicknesses. For liquid and powdered constituents the sample holder was filled completely and a piece of clear tape was used to hold them in place.

The relationship between the measured complex reflection coefficient, \( \Gamma \), and the complex dielectric constant \( \varepsilon_r = \varepsilon'_r - j\varepsilon''_r \) is given by

\[
\varepsilon_r = \frac{(\frac{\Gamma}{1 - \Gamma})^2 + (\frac{\lambda_0}{2a})^2}{1 + (\frac{\lambda_0}{2a})^2}
\]  

(1)

where \( x \) is a solution of the following transcendental equation

\[
\tan x = \frac{1 - \Gamma}{jkL(1 + \Gamma)}
\]

(2)

and \( k \) is the wavenumber inside the waveguide given by \( 2\pi/\lambda_0 \). \( \lambda_0 \) is the waveguide wavelength, \( L \) is the sample thickness and \( a \) is the broad dimension of the waveguide.

It is known that (1) has an infinite set of complex roots \[11\]. Thus, to find the correct root we either must have some idea of the dielectric constant value, or make two or more measurements for different sample thicknesses. Hence, not only for obtaining the correct root, but also for increased measurement confidence, \( \Gamma \) was measured for several samples with different thicknesses. \( \Gamma \) as a function of dielectric constant 

\[ \Gamma(\varepsilon_r, L) = \frac{\sqrt{Y} - j \tanh(kL\sqrt{Y})}{\sqrt{Y} + j \tanh(kL\sqrt{Y})} \]

(3)

where \( Y \) is given by

\[
Y = \left[ 1 + \left( \frac{\lambda_0}{2a} \right)^2 \right] \varepsilon_r - \left( \frac{\lambda_0}{2a} \right)^2
\]

(4)

From the measurements we obtained an array of values, \( \Gamma(\varepsilon_r, L_n) \), where \( n \) is the number of samples with different thicknesses. The unknown dielectric constant, \( \varepsilon_r \), is then determined by finding the best fit to (3). The two measured parameters are

\[
\text{SWR} = 20 \log(\text{V SWR}) = 20 \log \left( \frac{1 + |\Gamma|}{1 - |\Gamma|} \right)
\]

(5)

and the position of the standing wave null (which is related to the phase of \( \Gamma \)) in the slotted waveguide. The discrete points shown in Figs. 2 and 3 represent typical results for two rubber samples (the type of the materials used to obtain these figures will be discussed later) of various thicknesses at 5 GHz. These values were used to calculate the dielectric constant of the rubber samples using the best fit (3). The lines represent the results obtained from (3) using the calculated value of the dielectric constant. It is important to note that more sample thicknesses render results with higher level of confidence.

Additional improvements in the measurement accuracy can be obtained by using samples whose thicknesses are around the phase transition (minimum SWR) as shown in Figs. 2 and 3. The position of the phase transition may be calculated either from prior approximate knowledge of the dielectric constant or preliminary measurement with random thicknesses.

III. DIELECTRIC PROPERTIES OF RUBBER COMPOUND CONSTITUENTS

To fully understand the dielectric properties of various rubber compounds, one must first have a complete knowledge of the dielectric characteristics of each individual rubber compound constituent, namely EPDM, mineral filler, zinc oxide, oil and curatives. Table I lists the volume content (by percentage) and dielectric properties of main constituents for the rubber compound which we consider our basic sample throughout this investigation. Due to the very high conductivity of the carbon black, its dielectric properties were not measured. This table shows the mean value of \( \varepsilon'_r \) and

![Figure 2: Null Position in the Slotted Waveguide as a Function of Sample Thickness at 5 GHz. Solid and dashed lines pertain to uncured sample with and without curative, respectively.](image)

![Figure 3: SWR, dB as a Function of Sample Thickness at 5 GHz. Solid and dashed lines pertain to uncured sample with and without curative, respectively.](image)
loss tangent \( \tan \delta = \varepsilon'' / \varepsilon' \) of the constituents measured at several frequencies in X-band (8.2–12.4 GHz), using the technique described earlier. Measurements in other frequency bands resulted in similar values of dielectric constant for these constituents. The accuracy of these measurements is relatively less (about 5% for \( \varepsilon' \) and 10% for \( \tan \delta \)) compared to all other measurements reported later in this paper, since only two random thicknesses of the material were used. The measurement accuracy is discussed in detail later. The results indicate that the dielectric constants of EPDM, oil and mineral filler are for all practical purposes equal. Zinc oxide and curatives have slightly higher dielectric constants but they have small volume percentages in the total composition of a typical rubber compound. Through only physical mixing (not considering any chemical reaction), the contribution of curatives to the rubber compound dielectric constant should be negligible. However, in the course of our investigations it was discovered that the chemical reaction triggered by curatives in uncured rubber samples causes a detectable change in the dielectric properties of rubber at ambient temperature. This particular issue will be discussed separately later.

IV. CURED RUBBER DIELECTRIC CONSTANT DEPENDENCE ON CARBON BLACK

Our primary objective was to investigate the influence of carbon black volume content (%) on the dielectric properties of rubber compound. Consequently, measurements were performed on fourteen rubber samples with varying degrees of carbon black concentration. The rubber compounds were prepared by carefully measuring the ingredients in a precision laboratory scale. They were loaded in a laboratory Banbury mixer by upside down method and mixed for five minutes at maximum temperature of 130° C. Then the compounds were mixed for another five minutes at ambient temperature in an open two-roll mill. The rubber sheets were vulcanized in molds at 150°C for a predetermined time. The carbon blacks used for this study were aggregates of particles ranging in diameter from 5 to 125 nanometers. During the mixing process the aggregates are usually broken in smaller sizes and distributed uniformly throughout the mass. The rubber samples were prepared starting from the basic formulation given in Table I. Then, the volume percentage of carbon black was varied between 8.4% to 35.6%. This was done in such a way that any change in the amount of carbon black percentage was at the expense of EPDM, and the percentages of all other constituents were kept constant (same percentages as shown in Table I). The goal was to isolate the influence of carbon black on rubber compound dielectric constant. Table II lists the carbon black volume content for the measured rubber samples (sample #5 is the basic sample). These samples were prepared in sheet forms with thicknesses of 0.5, 1, 2, 3, and 5 mm to accommodate the completely filled waveguide technique, and their dielectric constants were measured at 5, 7, 10, 16, and 24 GHz. Figs. 4 and 5 show the results of these measurements for the real part and loss tangent at 5 and 24 GHz, respectively (results for other frequencies fall in between these two extremes). The results show that the dielectric constant of rubber increases as the carbon black volume percentage increases. Clearly, dielectric constant variations are more pronounced at 5 GHz which renders this frequency (or lower) useful for dielectric constant variation measurement.

It has been reported [3] that the rolling effect associated with rubber sheet production process of rubber sheets causes anisotropy. To alleviate this problem, our samples were prepared in such a way that rolling effect was minimized by performing the rolling process in many different directions. To check the success of this manipulation, we measured the
dielectric constant of several samples for several arbitrarily oriented (with respect to the electric field vector in the waveguide) pieces. Two open-ended rectangular waveguide measurement techniques were used: "infinite" sample method [12] and short-circuited sample method [13]. The results indicated no systematic deviations in dielectric constant values (i.e., within the measurement errors) which shows the absence of anisotropy in our samples.

V. DETECTION OF CURATIVES IN UNCURED RUBBER

The presence of curatives was checked for two types of rubber, namely carbon black filled and mineral filler rubber. The goal of these experiments was to investigate the possibility of using microwaves to detect the presence (or absence) of curatives prior to product preforming stage and curing. All of the following measurements were performed at ambient temperature.

A. Curatives in Carbon Black Filled Rubber

Uncured version of samples #4, 5, 7, 9, 11, and 14 (referring to Table II) were prepared with and without curatives. These samples were chosen to represent the entire range of carbon black percentages used in practical applications. It was soon determined that the dielectric constant variations for these samples were most pronounced at 5 GHz (as anticipated based on the previous results). We also learned that the dielectric constant difference between these two types of rubber samples was usually small. Therefore, as already discussed, the sample thicknesses are best to be around the phase transition regions. Figs. 2 and 3 show typical results of such measurement (sample #5 at 5 GHz). It is obvious that for sample thickness between 15 mm and 16 mm a difference of more than 10 dB is measured for SWR values. The opposite is true for measurements between 17 mm and 18 mm. These examples illustrate the importance of choosing optimum sample thicknesses to detect this difference for a given frequency.

Figs. 6 and 7 show the real part of dielectric constant and the loss tangent of uncured samples with and without curative at 5 GHz as a function of carbon black volume percentage. From Fig. 6 it is apparent that the addition of curatives does not affect the real part of the dielectric constant in a uniform fashion for all carbon black volume percentages. However, in spite of the low losses exhibited by curatives themselves (Table I), the imaginary part of the dielectric constant of these samples tends to increase with the addition of curatives for all carbon black volume percentages. Furthermore, in the presence and absence of curatives, the difference in tand is consistently detected as shown in Fig. 7. The detected difference is attributed to a chemical reaction triggered by the curatives at room temperature. It is well known in the rubber industry that a rubber compound containing curatives will cross link (cure) in due time even at room temperature. The rate of curing will depend upon the type and the amount of curatives, the polymer and the carbon black. It is apparent from these measurements that the initial stage of curing can be detected. As it was discussed earlier, the difference in the dielectric properties of rubber compound in the presence of curatives can not be described by a simple physical mixing effect (curatives occupy only 1.8% of the total rubber volume with dielectric properties close to those of EPDM). Another observation is that after the carbon black percentage increases beyond 25%, the difference between the samples with and without curatives becomes smaller. It may be hypothesized that at these carbon black percentages the effect of the chemical reaction caused by the presence of the curatives is masked by the overwhelming presence of unlinked carbon black.

B. Relationship Among EPDM, Carbon Black and Curatives

The measurements performed thus far involved a constant percentage of curatives while the ratio of carbon black to EPDM was changed. This may not reflect the composition of a real rubber product. Subsequently, having noticed the effect of curatives on dielectric constant, a new sample (named sample #15) was prepared. The dielectric properties of this sample when cured and not cured (with and without curatives) were then measured. Based on the cured results it was predicted that the carbon black content was close to that of sample #11 (25%). The measured dielectric properties of both samples are listed in Table III. However, a glance at the uncured compound compositions, listed in Table IV, indicates that samples #15 and #11 are not similar which may suggest the following. It is apparent that the volume percentage of carbon black in sample #15 is higher than that in sample #11. This is confirmed only if we focus on the dielectric constant of the uncured
samples. Then, if the uncured dielectric constant values for sample #15 and Figs. 6 and 7 are used, we may closely predict the carbon black percentage. Thus, for the samples without curatives only a physical mixing process may be considered. However, once cured, the chemical reaction will have changed the properties of the rubber sample and hence its dielectric properties. The volume percentages of EPDM and curatives are considerably higher in sample #11 than in sample #15. Therefore, we may hypothesize that similarity of the dielectric constants of the cured samples is due to the fact that the lack of carbon black in sample #11 is compensated by the increased reaction involving EPDM. What was not expected is the importance of this reaction (cross-linking) which in this case is compensating for the influence of carbon black on the dielectric constant. There still remains a question regarding the reduction in uncured with and without curatives loss tangent in the opposite directions between sample #15 and sample #11. Obviously, here we encounter some phenomenon which decreases the losses, and may be due to formation of cross-linked molecular network in such a way that dipoles rotate less freely [5]. Most likely there are two opposite mechanisms influencing the dielectric constant of uncured material when curatives are added depending on the carbon black, EPDM and curatives volume percentages. Clearly, further studies are needed to fully understand the impact of these mechanisms on dielectric constant.

C. Effect of Curatives in Mineral Filled Rubber

Similar measurements were also performed on uncured samples with and without curatives in which carbon black was replaced by mineral filler. As expected, this sample exhibits different dielectric properties than its carbon black counterpart. The results for this measurement are presented in Table V. From this table one may conclude that for such a low-loss rubber, the presence of curatives is difficult to be detected. The difference between the two samples is comparable with the accuracy we report in the next paragraph. However, we believe that this difference may still be detected if samples with optimum thicknesses are used. For thicknesses just after the second phase transition (between 29 mm and 32 mm) at 5 GHz a repeatable difference of about 3 dB in the value of SWR was detected. This difference will be more pronounced for thicknesses after the third phase transition. This part of the study confirms that the effect of curatives on carbon black loaded rubber dielectric constant may not be limited to a chemical interaction involving EPDM (cross linking), but carbon also plays a significant role in this chemical process.

VI. MEASUREMENT ACCURACY

The accuracy of the measurement technique used in this study is well established [14],[15]. However, our accuracy and sensitivity have been improved by measuring multiple sample thicknesses around the phase transition region and direct fitting of the measurement results to obtain the dielectric constant. We believe this was the reason for the consistent ability to detect curatives in uncured rubber samples particularly for low-loss materials.

The measurement technique reported here consisted of determining two parameters (SWR and relative null position) for various sample thicknesses. The accuracy of these measurements is increased if we use a rotary-vane attenuator and a precision slotted line. For high SWR cases we used the following correction factor

\[
SWR_c = -20 \log(10^{-\frac{SWR}{20}} - 10^{-\frac{SWR_0}{20}}) \quad (db)
\]

where \( SWR_c \) is the corrected value of the parameter shown in (5). \( SWR \) in dB, is the measured value, and \( SWR_0 \) in dB, is the value for the short circuited waveguide. The use of this correction factor is justified because a lossless waveguide and perfect short circuit termination were assumed. Equation (6) shows that there is a limitation in the measurement of low-loss materials (SWR approximately equal to \( SWR_c \)). However, the use of thicker samples of low-loss materials can reduce this problem.

Specific precautions must be considered for low and high-loss materials. For low-loss materials the real part of the dielectric constant may be precisely obtained from the multiple thickness null location information, but the imaginary part may not be deduced with reasonable accuracy. Thus, for this case the SWR measurement is very important as this is the sole indication of losses. For high-loss materials a different problem exists. The null location curve quickly degenerates to a line with certain slope as a function of sample thickness. This is why the precise value of dielectric constant can not be extracted from the null location measurements alone. In this case the SWR measurement plays a crucial role as well. Thus, for high-loss materials, measurements should be performed.
close to the first SWR minimum, because beyond that the SWR oscillates negligibly as a function of sample thickness.

It is difficult to estimate our measurement accuracy directly, because we do not use a straightforward approach in determining the dielectric constant of each sample (see (3)). However, from the repeatability of our results, especially when detecting small changes in loss due to the presence of curatives, it is clear that the accuracy is better than the usually accepted for a waveguide method [14, 15]. A numerical approach was used to estimate our apparatus measurement error. The following sources of error were considered: uncertainties due to the resolution error of the rotary-vane attenuator (±0.25 dB), the position of the standing-wave minimum (±0.05 mm), and the thickness of the sample (±0.1 mm). The error due to the frequency instability was neglected as a very stable oscillator was used. The analysis consisted of the following steps. The uncertainty associated to measured quantities for all sample thickness was added (or subtracted). Then dielectric constant, and subsequently the percentage difference for the real part and the loss tangent were calculated. Finally, the maximum error (worst case) for a given measurement was chosen. This procedure was performed for all the measurements reported in previous section. The results in all cases was less than 1% for the real part of dielectric constant and less than 3.5% for the loss tangent. This accuracy is worse if measurement points are not in the vicinity of the phase transition.

VII. CONCLUSION

A completely filled waveguide approach was used to conduct extensive dielectric constant measurement of rubber compounds. The measurement apparatus was comparatively simple, inexpensive and may be put together for a large range of frequency bands. Moreover, this technique offered very good repeatability and measurement accuracy characteristics particularly after some refinements of the measurement procedure. Subsequently, the dielectric constant of individual rubber compound constituents, namely EPDM, oil, mineral filler, zinc oxide and curatives were measured. The results showed that all of these constituents have similar dielectric properties.

Next, we measured the dielectric constants of many rubber compounds with different carbon black volume percentages at 5, 7, 10, 16, and 24 GHz. These samples were carefully prepared such that an increase in carbon black volume percentage was compensated by a reduction in the volume percentage of EPDM, while the volume percentage of all other constituents were kept constant. The results showed that the real and imaginary parts of rubber dielectric constant increase as a function of increasing carbon black percentage.

We also investigated the influence of curatives in uncured rubber compounds with different carbon black volume percentages. The goal was to establish whether the presence of curatives which is very small in volume content causes any detectable changes in the dielectric constant of the rubber compounds. The results of measurements showed that the presence of curatives can be detected. The sensitivity of detection increases with decreasing frequency.

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REFERENCES


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