Microwave Characterization of Rubber Composites Using Rectangular Dielectric Waveguide (RDWG)

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Abstract

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This paper describes a non-destructive measurement system for microwave characterization of rubber composites using RDWG system at X-Band. The set-up is able to detect the various fillers and particle sizes of the fillers in rubber composites.

Introduction

Natural rubber (NR) in its untreated form has poor properties and of little use. The addition of fillers such as carbon black (CB) and a number of other compounding ingredients that act as cross linking agent, accelerator, activator and anti degrading agent will enhance and improve its properties such as tear strength, abrasion resistance, stiffness, service life and hardness [1] and hence improve its usefulness. To ensure good quality rubber composites, it is of utmost important that these ingredients are incorporated properly with optimum amount of fillers and uniformly dispersed.

In the rubber industry, the various physical property testing is destructive in nature. The most common method of assessing filler dispersion is very qualitative and is based on visual inspection and subjected to human error. In view of such limitations, the present study explores the applicability of microwave non-destructive measurement technique in the characterization of rubber composites.

Microwave characterization of materials involves calculation of the complex permittivity and permeability properties based on the measured scatterring parameters of the materials under the influence of microwave signal [2]. These measured parameters can then be correlated to material parameters of interest such as the type and the particle size of the filler.

There are many microwave measurement techniques used to measure complex permittivity of the Device Under Test (DUT) such as using resonant cavity, free space, microstrip, open-ended coaxial probe or waveguide measurement techniques as cited by various authors [3]-[4]. The use of microstrip and in-waveguide measurement system requires meticulous sample preparation. A free-space measurement system incorporated spot focusing antenna was used by [5] but this technique requires tedious calibration and large transverse dimension of DUT.

In this study, an NDT rectangular dielectric waveguide measurement system operating at X-band was developed for complex permittivity measurement of rubber composites. This set-up required small size of samples while the calibration and measurement could be done fast and efficiently. The chosen DUTs were rubber composites with three types of tiller - china clay, CB and aluminium silicate and composites filled with five sizes of CB particles.

Theory

Every material has a unique set of electrical characteristics that are dependent on its dielectric properties. Permittivity is a quantity used to describe dielectric properties of materials under the influence of electromagnetic waves with reflection at interfaces and the attenuation of wave energy within those materials. In frequency domain, the complex relative permittivity ε^* of a material to that of the space can be expressed as Equation (1) [2]: -

$$\varepsilon^* = \varepsilon^- - j\varepsilon^{"} = D/E \text{ and } \varepsilon^{"} = \sigma/\varepsilon_0 \omega$$
 (1)
 $c_1 = c_2 + c_1 + c_2 + c$

rubber

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where ε' is the relative permittivity of the material, ε' - dielectric constant ε' - loss factor ε_{σ} - the permittivity of the space, σ - the conductivity of the material, ω - angular frequency of the field, D - electric flux density or displacement. E - electric field intensity

The real part, ε is referred to as the dielectric constant and represents stored energy when the material is exposed to an electric field, while the dielectric loss factor, ε , which is the imaginary part, determine the energy absorption and attenuation. When a linearly polarized, uniform plane wave is normally incident on the sample of thickness. d_s as shown by Fig.1, the incident wave is partially reflected, transmitted and absorbed by the sample. The reflected signal and transmitted signal are comprised of an infinite number of components due to multiple reflections between the air/sample. The DUT is assumed to be planar of infinite extent laterally so that diffraction effects at the edges can be neglected, thus the total reflected signal, S₁₁ and transmitted signals, S₂₁ are given respectively by Equation (2) and (3) [5]: -



Fig.1. Schematic diagram of planar sample

$$S_{11} = \frac{\Gamma - \Gamma \exp(-2\gamma_s d_s)}{1 - \Gamma^2 \exp(-2\gamma_s d_s)}$$
(2)
$$S_{21} = \frac{(1 - \Gamma^2) \exp(-\gamma_s d_s)}{1 - \Gamma^2 \exp(-2\gamma_s d_s)}$$
(3)

where γ_s is the propagation constant in the sample and Γ is the reflection coefficient of the sample/air interface. Both are functions of the complex permittivity of the sample, ε and given by Equation (4): -

$$\varepsilon^{*} = \frac{\gamma_{*}}{\gamma_{0}} \left(\frac{1 - \Gamma}{1 + \Gamma} \right) \tag{4}$$

where $\gamma_0 = (j2\pi/\lambda_0)$ represents the propagation constant of free space, and λ_0 is the free-space wavelength. Equations (1) to (4) will be the basis to the calculation of complex permittivity of rubber composites in this research. By using (1) through (4), calculated S^e₁₁ and S^e₂₁ can be expressed in terms of ϵ^* but ϵ^* cannot be expressed explicitly in terms of S₁₁ and S₂₁, so it is necessary to find it iteratively by assuming a guess value for the complex permittivity of the sample. This is achieved by using a zero finding technique, which finds the zeros of the error function. The error function is defined by Equation (5);

$$\mathbf{E} = |\mathbf{S}_{21}^{m} - \mathbf{S}_{21}^{c}| + |\mathbf{S}_{11}^{m} - \mathbf{S}_{11}^{c}|$$
(5)

where S^m and S^c are the measured and calculated values of the complex transmission coefficients, respectively. The Muller method with deflation is used for calculation of zeros of the error function [7].

Measurement System

The RDWG measurement system based on transmission and reflection measurement is as shown in Fig. 2. It consists of a Wiltron 37269B vector network analyzer (VNA), a pair of coaxial cables, coaxial to waveguide adapter, standard metallic waveguide (WR-90) and dielectric filled standard-gain horn antennas. The WR-90 carrying TE₁₀ mode is used as a launcher. The dielectric material used for RDWG is made of poly-tetra-fluoro-ethylene (PTFE) and its length beyond the horn antenna is 4.5 cm. Its cross-sectional area is 22.86 mm x 10.16 mm, which is of the same cross-sectional area as the metallic waveguide. The PTFE material is chosen because it has low dielectric constant loss and very low loss. For the purpose of launching electromagnetic wave into RDWG, the PTFE is double-tapered [8] at one end and inserted into the WR-90 waveguide through standard-gain horn antennas.

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A. Calibration

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The internal TRL (*through, reflect, line*) calibration model of VNA is used for calibration of the RDWG measurement system. The *through* standard is realized by keeping the distance between the two RDWGs equal to zero. *Reflect* standards for port 1 and port 2 are obtained by mounting a metal plate between transmit and receive RDWGs. The *line* standard is achieved by separating two RDWGs by a distance which is approximately equal to quarter wavelength at the mid-band frequency. After TRL calibration, the through standard is metal plate, the amplitude and phase of S₁₁ (or S₁₂) is within 0.00 ± 0.06 dB and $0.00 \pm 0.8^{\circ}$, respectively. For the metal plate, the amplitude and phase of S₁₁ (or S₁₂) are 0.00 ± 0.06 dB and $180 \pm 1.9^{\circ}$ respectively.

B. Validation

Measurement results obtained by RDWG measurement system is validated by measuring complex permittivity of commonly used reference samples. Modelling and simulation of the measurement system is carried out using CST Microwave Studio software package.

Results and Discussion

After performing TRL calibration for the RDWG measurement system, DUT of 5cm by 5cm was sandwiched between the RDWGs and scattering parameters S_{11} and S_{21} were measured. Based on the measured S_{11} and S_{21} the values of the complex permittivity were then extracted using MATLAB program.



Fig. 3 illustrates a graph of dielectric constant for various amounts of filler in parts per hundred (pphr) rubber content. The result shows that the values of dielectric constant increases as the amount of CB in rubber composites is increased.

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For the case of china clay and aluminum silicate, although the values of the dielectric constant were not the same, there was no significant increase in both values of dielectric constant and loss factor. This is due to the fact that carbon black is a conductor and hence electromagnetic waves are absorbed and stored whereas both china clay and aluminum are insulators. Fig. 4 shows a plot of complex permittivity at 10 GHz for five different particle sizes of CB whereby the values of complex permittivity increases as the particle size of the CB decreases. This is due to the smaller the particle size, the surface area will increase and thus will enhance the ability of the composite to absorb and store electromagnetic waves.



Fig 5. Graph of dielectric constant and loss factor for three particle size of CB

Fig. 5 depicts that the values of complex permittivity of rubber composites differ significantly for three different particle sizes of the CB. The finer the CB the greater will be the number of the particles present and the more will be the chains, channel or paths for the wave.

Conclusion

A novel method was developed using RDWG for microwave characterization of rubber composites at X-Band. The measurement system is very convenient, effective and reliable and easy to use for repetitive measurement. This approach and set-up can be applied to other samples in the form of solid and liquid and can be extended for hazardous liquid.

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