Permittivity and temperature characterisation of rubber compounds under high power microwaves

J.J. Mallorquí, A. Aguasca and A. Cardama

A waveguide reflectometer, in combination with a fibre optic temperature probe, is used to characterise the permittivity changes of small rubber samples heated with high power microwaves. The molecular changes of rubber during vulcanisation can be clearly seen in the evolution of the measured complex dielectric constant against time and temperature.

Introduction: Rubber, natural or synthetic, is a complex material that must be mixed with several other ingredients, not only to achieve the performance levels of the finished product but also to facilitate manufacture and reduce costs. From the electromagnetic point of view, each agent modifies the dielectric properties of the mix. The industrial vulcanisation chains use a microwave oven to pre-heat the profiles up to 100°C, so the performance of the whole facility will strongly depend on their dielectric properties. In addition, as rubber is heated its dielectric properties change with both temperature and the complex chemical process associated with the vulcanisation. Despite its great industrial interest, very little is known about the dielectric properties of rubber as a function of power and temperature. Previous studies have used completely filled waveguides near a shorting plate to measure the rubber permittivity [1], but the field distribution of the TE_{10} mode causes a non-uniform heating of the sample and makes the method unsuitable for this application. A new method employing a partially filled waveguide is presented in this Letter, where the small sample dimensions and its positioning in the waveguide ensures an almost quasi-homogeneous power distribution.

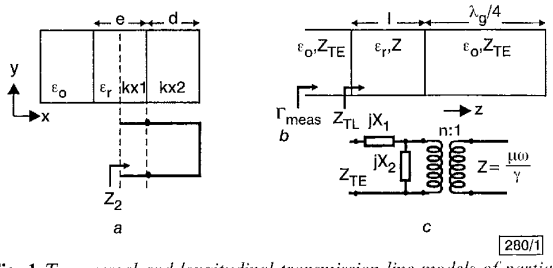

Fig. 1 *Transversal and longitudinal transmission line models of partially filled waveguide and equivalent circuit of dielectric discontinuity*

a Transversal model
b Longitudinal model
c Equivalent circuit

Measurement method: In partially dielectric-filled waveguides, it can be shown that linear combinations of transverse electric TE^z and magnetic TM^z modes can be solutions that satisfy the boundary conditions of such waveguides. The modes are referred to as hybrid modes, or longitudinal section electric (LSE) and longitudinal section magnetic (LSM) modes [2]. The fields must satisfy the transverse wave equation in each homogeneous section, as shown in Fig. 1*a*

$$\gamma^2 = k_{x1}^2 + k_{y1}^2 - k_0^2 = k_{x1}^2 - k_0^2 \quad \text{(empty part)}$$
$$\gamma^2 = k_{x2}^2 + k_{y2}^2 - \varepsilon_r k_0^2 = k_{x2}^2 - \varepsilon_r k_0^2 \quad \text{(filled part)} \quad (1)$$

where γ is the mode complex propagation constant, k_0 the wavenumber in empty space, k_x and k_y are the propagation constant components in the x and y directions and ε_r the complex relative permittivity of the material. Since the feeding waveguide has been chosen to operate in the TE_{10} mode, k_y is zero. The subscript 1 refers to the empty part of the waveguide and the subscript 2 to the filled one. Using the transverse resonance method (TRM) [3], the cross section of the waveguide is represented as a transmission line system that enables the input impedance (Z_2) at the waveguide centre, $a/2$, to be calculated

$$Z_2 = \frac{\omega\mu}{k_{x2}} \frac{k_{x1}^{-1}\tan(k_{x1}d) + jk_{x2}^{-1}\tan(k_{x2}e/2)}{k_{x2}^{-1} - k_{x1}^{-1}\tan(k_{x1}d)\tan(k_{x2}e/2)} = \frac{E_y(a/2)}{H_z(a/2)} \quad (2)$$

The geometrical parameters of the slab, d and e, are defined in Fig. 1*a*. Assuming a symmetrical field distribution excited by a TE_{10} mode, there is an electric field maximum, E_y, and a null magnetic field, H_z, in the centre of the waveguide. Then, Z_2 must be infinite, a condition easily achieved if the denominator of eqn. 2 is zero. This condition combined with eqn. 1 can be rewritten as a trascendental equation depending only on the propagation constant (γ), which can be solved numerically.

The equivalent circuit for a dielectric slab, Fig. 1*c*, was developed in [3]. In the particular case of thin samples and relatively low permittivities the series reactance (X_1) is very small, while the shunt reactance (X_2) is large and the turns ratio (n:1) is found to be equal to unity. The sample is loaded with a short circuit at a distance $\lambda_g/4$ to maximise the electric field at the sample. Then, a good approximation of the value of the input impedance at the reference plane (Z_{TL}) can be obtained using the simplified transmission line model (STLM) of the waveguide, shown in Fig. 1*b*, which does not include the effect of evanescent modes in the dielectric discontinuities.

If the reflection coefficient for the TE_{10} mode, Γ_{meas}, from a sample of known dimensions placed inside the waveguide is measured, an 'inverse' procedure can be applied in order to calculate the unknown dielectric constant. The sequence is as follows: first, the input impedance at the samples reference plane, Z_{meas}, is computed from Γ_{meas}. Secondly, the propagation constant in the partially-filled waveguide is obtained from the input impedance, assuming $Z_{TL} = Z_{meas}$. Finally, the unknown permittivity is obtained from the complex propagation constant, by solving eqns. 1 and 2. However, the accuracy of measurements obtained using the STLM decreases as the dielectric constant increases. Results can be improved if the effects of the evanescent modes are considered. The true measured input impedance (Z_{meas}) and the approximate input impedance based on the STLM (Z_{TL}) can be related via an error function (F)

$$Z_{meas} = Z_{TL} \cdot F(\varepsilon' - j\varepsilon'', d, e, l) \quad (3)$$

F depends on the material dielectric constant and geometry and is a function of the propagation constants and coupling coefficients of the different propagating and evanescent modes required to satisfy the continuity of both the transverse electric and magnetic fields. These coefficients can be obtained using the mode-matching (MM) method [4]. An iterative method has been implemented to refine the initial STLM estimation. The initial permittivity value is used to calculate a first estimate of the error function, and from eqn. 3 up-date Z_{TL} to a more accurate value. Then, the dielectric constant is recalculated and the process repeated until convergence is reached. Table 1 shows the improvement of the permittivity estimation with the iterative process for two different materials (both samples are 15 mm wide by 10 mm long). The value of Γ_{meas} for the TE_{10} mode has been computed using the MM formulation and validated with the Agilent HFSS electromagnetic simulator. The first iteration corresponds to the STLM formulation, which causes a relative error of ~7% in the permittivity estimation. However, in only two more iterations the error function is able to adjust the permittivity to its correct value.

Table 1: Improvement of measured permittivity values with iterative procedure from initial STLM

Sample ε_r	PTFE: $2.5 - (7.5 \times 10^{-4})j$	Rubber: $10.0j - 1.0j$
ε_r (1st iteration, SLTM)	$2.34 - (7.56 \times 10^{-4})j$	$9.29 - 1.10j$
ε_r (2nd iteration, MM)	$2.49 - (7.56 \times 10^{-4})j$	$10.0 - 1.02j$
ε_r (3rd iteration, MM)	$2.50 - (7.50 \times 10^{-4})j$	$10.0 - 1.00j$

Experimental setup: The complex reflection coefficient is measured by using a double waveguide directional coupler (in WR340 at 2.45 GHz) combined with a mixer. The IF signals corresponding to the incident and reflected waves are acquired with a digital oscilloscope connected to a PC. A fibre optic thermometer capable of measuring up to 100°C supplies the temperature reading of the sample with no electromagnetic interference. The system has been validated by measuring well-known materials such as PTFE and PVC.

Results: The evolution of both the real part of the permittivity ($\Re(\varepsilon)$) and loss tangent ($tg\delta$) as the rubber samples are heated

show the evolution of its chemical properties. All rubbers are basically large polymer chains with molecules presenting permanent dipoles. When the temperature rises, the chain structure becomes lighter and the different molecules are allowed to rotate along their axis. The movement of all molecules presenting permanent dipoles helps to raise both the real and imaginary parts of the permittivity. If the dielectric losses rise, so does the power dissipation and consequently the temperature. This 'positive feedback' causes exponential behaviour in both permittivity and temperature. When the rubber starts to vulcanise, its chemical properties change because the long chains are fixed by sulphur bridges reducing the freedom of movement of the permanent dipoles. As a consequence, the dielectric constant is much less sensitive to temperature. Two different rubber compounds have been measured. The first sample, MCS-EPS55DI, is natural rubber while the second one, FER-CN3, is neoprene rubber. Both samples are 19 mm wide by 10 mm long. Fig. 2 shows the evolution of both $\Re(\varepsilon)$ and tgδ and temperature as the samples are heated for 500 s at a power of 12 W. MCS shows the positive feedback phenomenon exponentially increasing both permittivity and temperature. This material starts to cure at higher temperatures than the ones reached during the measurement. However, FER behaves differently. Despite its positive feedback at the beginning, at around 58°C the rubber starts to cure and, consequently, its dielectric properties remain constant despite the temperature continuing to rise.

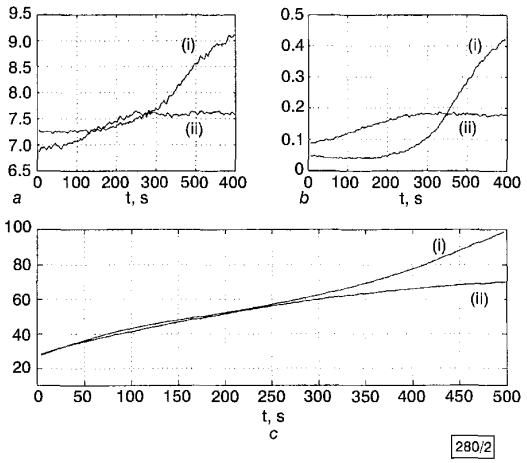

Fig. 2 *Permittivity, loss tangent and temperature time evolution of MCS-EPS55DI and FER-CN3*

a Permittivity
b Loss tangent
c Temperature
(i) MCS-EPS55DI
(ii) FER-CN3

Conclusions: A vectorial reflectometer, capable of automatically obtaining the permittivity and characterising the dynamic behaviour of rubber as RF power is absorbed, has been presented. The potential capacity of the system for monitoring chemical reactions associated with the formation of cross-linked molecular networks during vulcanisation has been shown. The instrument can help in determining the optimum rubber composition and oven dimensions to improve the efficiency of the applicators of industrial vulcanisation chains.

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References

1 GANCHEV, S.I., BHATTACHARYYA, J., BAKHTIARI, S., QADDOUMI, N., BRANDENBURG, D., and ZOUGHI, R.: 'Microwave diagnosis of rubber compounds', *IEEE Trans. Microw. Theory Tech.*, 1994, **42**, (1), pp. 18–24
2 BALANIS, C.A.: 'Advanced engineering electromagnetics' (John Wiley & Sons, 1989)
3 COLLIN, R.E.: 'Field theory of guided waves' (IEEE Press, 1991), 2nd edn.
4 FENG, Y.C.: 'Complex permittivity and permeability measurements for high-loss material in a waveguide'. Microwave and Millimeter Wave Technology Proc., 1998. ICMMT'98. 1998 Int. Conf. Microwave and Millimeter Wave Technology, 1998, pp. 201–204

Binary array set with zero correlation zone

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The zero correlation zone (ZCZ) concept is extended from one-dimensional sequences to two-dimensional arrays. A class of binary arrays with ZCZ properties, based on two-dimensional mutually orthogonal complementary codes, is presented.

Introduction: In various CDMA systems, orthogonal spreading sequences such as Walsh sequences are employed as channelisation codes to reduce the multiple access interference. As the frequency selectivity of the propagation channel increases, the orthogonality between different users tends to diminish because of the increasing inter-cell interference. To maintain orthogonality between different users, a new concept of generalised orthogonality, or zero correlation zone (ZCZ), has been defined and the corresponding ZCZ sequences obtained [1]. By employing ZCZ sequences in a CDMA system, the co-channel and multipath interference can be reduced significantly [2].

In this Letter, the ZCZ concept is generalised to the two-dimensional case, and hence the ZCZ sequences are extended to ZCZ arrays. Based on the two-dimensional complementary codes presented by Lüke [3], classes of binary arrays with rectangular zero correlation zone are derived.

Two-dimensional ZCZ arrays could be used in a phased array antenna, arrays of sound sources, time-frequency-coding, spatial correlation or map matching, built-in tests of VLSI circuits, two-dimensional measuring techniques (optical system), etc.

ZCZ array set: Given a binary arrays set $\{A^{(r)}\}$ with family size M, $r = 1, 2, ..., M$, each array $A^{(r)}$ with order of $L_1 \times L_2$. Let $A^{(s)}$ and $A^{(t)}$ be two arbitrary arrays, $R_{s,t}$ denote their periodic correlation function

$$R_{s,t}(\tau_1, \tau_2) = \sum_{i_1=0}^{L_1-1} \sum_{i_2=0}^{L_2-1} A^{(s)}_{i_1 i_2} A^{(t)}_{i_1+\tau_1 \bmod L_1, i_2+\tau_2 \bmod L_2} \tag{1}$$

We define zero correlation zone Z_{cz} as

$$Z_{cz} = \max\{(T_1, T_2) | R_{s,t}(\tau_1, \tau_2) = 0, \quad \text{where } |\tau_1| < T_1, |\tau_2| < T_2 \text{ and } (\forall s \neq t \text{ or } ((\tau_1, \tau_2) \neq (0,0) \text{ and } \forall s = t))\} \tag{2}$$

If the zero correlation zone of the set A is $Z_{cz} \neq (0, 0)$, the set A is said to be a ZCZ-$((L_1, L_2), M, Z_{cz})$ set.

Construction of ZCZ array set: Consider M mutually orthogonal complementary array sets B, each set containing N arrays of order $L_1 \times L_2$, be arranged in matrix form as

$$B = \begin{bmatrix} B_{11} & \cdots & B_{1N} \\ \vdots & \ddots & \vdots \\ B_{M1} & \cdots & B_{MN} \end{bmatrix} \tag{3}$$

where the i,jth entry $B_{i,j}$ is the jth array in the ith set, satisfying