MEASUREMENT OF THE COMPLEX PERMITTIVITY OF LOW LOSS POLYMER POWDERS IN THE MILLIMETER-WAVE RANGE

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An improved measurement method of complex permittivity of low loss polymer powders is suggested. The measurements are done in the mm-wave range using a quasi optical resonator. The 2-D corrugated mode exciter is employed to improve suppression of undesirable higher modes. The model used for reconstructing complex permittivity takes into account ohm losses of metal mesh coupling that provide better accuracy of the reconstructing procedure. An example illustrating this method is reported.

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INTRODUCTION

There is a great amount of interest in the use of the millimeter-wave and sub-millimeter-wave portions of the electromagnetic spectrum for some industrial, biomedical and homeland security applications, stimulating efforts in more detailed mm-wave characterization of different materials. Investigation of the complex permittivity spectrum of polymer powders on microwaves and mm-waves is a powerful tool for the estimation of the dielectric relaxation process: the activation energy of the conductivity, the polarization mechanisms and motions of charge carriers. Based on such a study, it is possible to predict the electrical properties of bulky samples fabricated from polymer powders [Murata et al., 2005]. Another area associated with micro-wave characterization of powders is nanoparticle-filled polymers that provide improved electrical and thermo-mechanical properties of dielectric materials [Roy et al., 2005]. For example, the composites fabricated with ~0.1 μm Cabot BT-8 hydrothermal powder showed a broad relaxation over the measured frequency range [McNeal et al., 1996]. The reduction in particle size and subsequent modulation of the domain structure are responsible for the shift in the relaxation frequency. The dielectric properties of ceramics and powders of PbTiO3, composed of paraffin, were measured from 1 MHz to 18 GHz. All samples exhibited evidence of the relaxation phenomena in their dielectric spectra [Jiwei et al., 2001; Yeh et al., 2005]. However, an extension of this approach on low loss polymer powders with tan δ < 10^{-4} cannot be done because of the much higher losses in paraffin, which lead to a screening effect.

Earlier measured polymer powders with low

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density (less than 1 g cm\(^{-3}\)) have demonstrated a loss tangent within the range \(10^{-5} < \tan \delta < 10^{-4}\), depending on the powder’s density [Kapilevich et al., 2007]. The measurements were performed in mm waves using a quasi-optical resonator filled with the powder under test. In this case, there are no screening effects caused by higher losses in paraffin. However, the accuracy of the measurements is greatly dependent on the excitation conditions and the reconstructing procedure.

This paper describes the improved method developed for mm-wave characterization of low loss powders. The following modifications of the previously reported method [Kapilevich et al., 2007] are suggested to determine complex permittivity:

1. Replacing a standard rectangular horn with a 2-D corrugated mode exciter. As a result, a quasi-Gaussian beam is formed providing better suppression of undesirable resonating modes;
2. Modification of the model used for reconstructing complex permittivity by introducing ohmic losses in the coupling metal mesh. Such a modification takes into account the asymmetry of the resonance curve caused by finite conductivity of the input mesh;
3. Implementing multi-points reconstructing algorithm, which is ideally matched with the asymmetrical resonance curves of the selected modes used for the determination of real and imaginary parts of the dielectric constant.

**The configuration of the measurement setup**

The determination of complex permittivity of low loss powder is based on the measurement reflection coefficient of the quasi-optical resonator filled with the powder under test. The configuration of the experimental setup has been reported in detail earlier [Kapilevich et al., 2007]. However, the standard horn illuminating input coupling mesh was not matched well enough with the quasi-Gaussian beam existing in the resonator. To overcome this drawback, we have replaced the rectangular horn with a 2-D corrugated mode exciter in order to form such a beam. The undesirable higher modes of the quasi-optical resonator were suppressed better now. Figure 1 shows a general view of the experimental setup with a reflectometer, which is used for reflectance measurements. The reflection coefficient has been measured by the network analyzer Agilent HP-8757D operating in the 75-110 GHz range using the data acquisition system VEE-Pro.

The quasi-optical resonator consists of a section of an overmoded rectangular guide of the dimensions 10.5x25x189.8 mm\(^3\) fabricated from Al. The input resonator’s port is excited by the 2-D corrugated mode exciter via the metal mesh as shown in Figure 1. The output port is short-circuited. The 75-110 GHz reflectometer is used for the measurement of input reflectance.

Typical measured return losses recorded by VEE-Pro as a function of frequency are depicted in Figure 2 for the air-filled resonator, and in Figure 3 for the resonator filled with polymer powder (PTFE) having the density 0.65 g cm\(^{-3}\). The following algorithm is suggested for the calculation of real and imaginary parts of complex dielectric constants of the powder:

1. Calibrating the air-filled resonator to determine its unloaded Q-factor. The data depicted in Figure 2 should be used in this case.
2. Reconstructing a real part of the complex dielectric constant from the data depicted in Figure 3.
3. Reconstructing an imaginary part of the complex dielectric constant from the data depicted in Figure 3.
4. Comparison of the measured results with a calculated reflectance, using the reconstructed values of complex permittivity to validate the accuracy of the method.
The reconstructing model

Two basic assumptions have been used in the formulation of the reconstructing model:
1. The resonant frequency of the selected mode is determined by a real part of dielectric constants;
2. The width of the resonant curve belonging to the same mode is determined by an imaginary part of dielectric constants.

These assumptions are not universal but are still valid for low loss powders, which are the subject of this paper. According to Kapilevich et al. [2007], the real part of dielectric constants can be written in the form

\[ \varepsilon_r = \left( \frac{0.3}{f_{mnp}} \right)^2 \left[ \left( \frac{1}{\lambda_c} \right)^2 + \left( \frac{p_1}{2l} \right)^2 \right] \]  \hspace{1cm} (1)

where \( f_{mnp} \) is the resonance frequency of the mode with the indexes \((m,n,p)\), \( \lambda_c \) is the critical wavelength for the set of indexes \((m,n)\), \( l \) is the length of the resonator and \( p_1 \) is the maximum root of the quadratic equation

\[ R(q+p^2) = q+(p+1)^2 \]  \hspace{1cm} (2)

where \( R = \left( f_{mnp+1} / f_{mnp} \right)^2 \) and \( q = \left( 2l / \lambda_c \right) \).

The resonance frequencies of the nearest modes with the indexes \((m,n,p)\) and \((m,n,p+1)\) are directly determined from the measured frequency responses shown in Figures 2 and 3.

In order to reconstruct an imaginary part of the dielectric constants from the measured data, it is necessary to evaluate the behavior of the reflection coefficient in a vicinity of resonance frequency of the specified mode. The following three parameters are essential here:
1. attenuation constant \( \alpha \) that determines dissipative losses of the resonator and its unloaded Q-factor
2. normalized reactance of coupling metal mesh, \( B \);
3. normalized conductance of coupling metal mesh, \( G \);

It can be shown that the input reflection

**FIGURE 1.** Photo of the experimental setup and its basic components. The metal mesh between the mode exciter and the resonator has an air cell size of 1x1 mm² and the width of the metal strip is 0.08 mm.
FIGURE 2. Measured return loss (RL) in dB of the air-filled quasi-optical resonator.

FIGURE 3. Measured return loss (RL) in dB of the powder-filled quasi-optical resonator. The powder density is 0.65 g cm$^{-3}$. 
coefficient of the shorted waveguide section can be presented in the form [Kapilevich et al., 2007]

\[
\Gamma = \frac{(1 - Y) \tanh(\gamma l) - 1}{(1 + Y) \tanh(\gamma l) + 1}
\]  (3)

where \( Y = G + jB, \gamma = \alpha + j\beta \) and \( \beta = \sqrt{\varepsilon(2\pi/\lambda)} \).

Since VEE-pro downloads the return loss (RL) in dB, it is convenient to transform \( \Gamma \) to RL, namely, \( RL = -20\log |\Gamma| \text{ dB} \).

The frequency set \([f_0, f_1, \ldots, f_N]\) and set of the corresponding measured parameters \([RL_0, RL_1, \ldots, RL_N]\) must be selected in order to begin the reconstructing procedure. Such a selection was done in the vicinity of the resonance frequency of the chosen mode. The error function is then introduced to carry out minimization

\[
\frac{1}{N} \sum_{i=1}^{N} |RL_{\text{meas}}(f_i) - RL_{\text{mod}}(G_0, B_0, \alpha_0)| \Rightarrow \min(G_0, B_0, \alpha_0)
\]  (4)

where \( N \) is the number of sampling points for which the measured (\( RL_{\text{meas}} \)) and calculated (\( RL_{\text{mod}} \)) values are determined, and \( G_0, B_0, \alpha_0 \) are parameters providing minimization of the error function. If the chosen \( N \) is large enough, the error function is rather smooth and standard unconstrained strategies based on Quasi-Newton or Conjugate Gradient optimizations can be used.

When non-linear optimization is carried out, we need to determine the “starting point” from which the solver has to start the search for the minimization procedure. To create a set of guess values, some preliminary information concerning the behaviour of the reflection coefficient in a space of searching variables is required near the resonance frequency. This can be obtained, for instance, by using the proper approximated analytical model for the resonator and coupling mesh [Kapilevich and Litvak, 2005].

Calibration of the experimental setup

In order to perform the calibration of the experimental setup, we have selected one of the measured resonating modes of the air-filled resonator shown in Figure 4 and the assigned set of guess values employed in minimization of (4) is \( G_{\text{guess}} = 0.2, B_{\text{guess}} = -8 \) and \( \alpha_{\text{guess}} = 0.2[1/m] \). The results of the calculated RL for these guess parameters are depicted in Figure 4 by the dashed line. The minimization according to (4) yields the following values: \( G_{\text{min}} = 0.52, B_{\text{min}} = -5.3 \) and \( \alpha_{\text{min}} = 0.131[1/m] \). The unloaded Q-factor can be estimated from the expression [Rizzi, 1988]:

\[
Q_{\text{un}} = \frac{B}{2\alpha} \approx 7450
\]  (5)

The RL calculated from (3) are depicted by the solid line, while the measured data are depicted by circles. Comparison of both results reveals their agreement, thus proving the validity of the proposed method. The above determined \( Q_{\text{un}} \) is associated with ohmic losses in the resonator’s metal walls, \( Q_{\text{metal}} = Q_{\text{un}} \) and will be used below as the reference parameter in determination of the powder’s loss.

Reconstructing the powder’s characteristics

A) The real part of dielectric constants:

From the measured data we have determined the following parameters needed to estimate \( \varepsilon_r' \):

1. averaged frequency shift between nearest resonating modes: \( \Delta f = 0.696 \text{ GHz} \);
2. index \( p_j \) corresponding to the given experiment conditions: \( p_j = 130 \) at the resonance frequency \( f = 94.113 \text{ GHz} \);
3. the real part of dielectric constants calculated from (1): \( \varepsilon_r' = 1.24 \) corresponding to the powder density 0.65 g cm\(^{-3}\).
B) The imaginary part of dielectric constants:

At first, we need to find the unloaded $Q_{\text{filled}}$ - factor of the powder-filled resonator with powder using the well known expression:

$$\frac{1}{Q_{\text{filled}}} = \frac{1}{Q_{\text{metal}}} + \frac{1}{Q_{\text{powder}}}$$

(6)

For the above determined $\varepsilon'_r$ and the same set of guess values (dashed line in Figure 5), we can determine the following parameters needed to estimate $\varepsilon''_r$:

1. attenuation coefficient: $\alpha_{\text{min}} = 0.173[1/m]$;
2. normalized parameters of the coupling mesh: $G_{\text{min}} = 0.19, B_{\text{min}} = -5.1$;
3. the unloaded $Q_{\text{filled}}$ - factor calculated from (5): $Q_{\text{filled}} = 6350$;
4. $\tan \delta = 1/Q_{\text{powder}} = 2.33 \times 10^{-5}$ calculated from (6).

Figure 5 demonstrates the measured result (circles) and reconstruction from (3) return losses $RL \, dB$ (solid line) for the resonator filled with the measured polymer powder. Calculations have been done using the above determined parameters. We can see that the measured and reconstructed curves are close to each other, thus proving the relevance of the method proposed. The return loss corresponding to the guess values is depicted by a solid curve.

CONCLUSION

In this paper, we have suggested an improved method for determining the complex permittivity of low loss polymer powders on mm-waves using the quasi-optical resonator. Measurements of real and imaginary parts of dielectric constant can be easily performed. The model required for use in the reconstruction procedure has been suggested and tested. Examples illustrating the
measurement technique have validated the basic assumptions used in the method developed and proved its efficiency.

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REFERENCES


FIGURE 5. Frequency response of the powder-filled resonator for the selected mode: dashed line – guess curve, circles – measured results, solid line– minimization according to (4).