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Density-Independent Moisture Measurements in Polymer Powders Using MM-wave Quasi-Optical Resonator

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ABSTRACT. A quasi-optical resonator is implemented to characterize low loss polymer powders at mm waves. Based on suggested resonator configuration, density-independent moisture measurements have been carried out in W-band. The resonant frequencies and Q factor were measured to reconstruct real and imaginary parts of a dielectric constant to be used for density and moisture determinations. The model needed to describe this quasi-optical resonator including the reconstructing algorithm has also been developed. Examples illustrating the method proposed are reported.

1 Introduction

Since many polymer powders being in the natural (not compressed state) have a low loss tangent (10^{-4} or less) and low density, an attenuation of propagating wave becomes too small at microwaves conventionally employed for moisture measurements. Therefore, it is reasonable to develop moisture metering technique at mm-wave range. A shift toward mm-waves allows increasing accuracy of moisture and density determinations due to higher attenuation and better resolution when free-space technique is used. Density-independent moisture method [1,2] is an efficient approach for improving accuracy of the moisture content determinations of variety materials such as solids, grain, sands, tobacco etc. at microwaves. Extension of this method toward mm-waves is considered in the paper.

We have suggested employing a section of overmoded rectangular waveguide filled with powders. Waveguide is closed by a metallic plate (short) on its output port and the array of wire grids operating as a coupler on its input. Such a configuration forms short-circuited quasi-optical resonator that is excited by horn antenna. Measured reflection coefficient data are downloaded to PC via GPIB cable and employed then for a determination of real and imaginary parts of powder's dielectric constant using proper reconstructing procedure. Since both the real and imaginary parts of complex permittivity are obtained from the same measured data, density invariant moisture function $A(\psi)=(\varepsilon'-1) / \varepsilon''$ [2] can be directly determined.

The paper presents the model describing a behavior of a quasi-optical resonator filled with powder. The model links an input reflection coefficient with attenuation constant and power reflectance of the wire grid coupler. Based on this model reconstructing algorithm has been realized. Effects of asymmetric shape of the resonance curve are discussed. Since the reconstructing procedure involves solution of non-linear equations, its stability has also been investigated. Experimental setup and its calibrations are considered. Examples of moisture measurement of low loss polymer powder at W-band are reported.

2 Description of experimental setup

The low loss powder characterization has been performed using experimental setup shown in Fig.1a. The section of short-circuited overmoded guide is filled with a powder under test. Polarizing wire grid at the input of this section is used as a coupling element. It has a large power reflectance, about 0.9. As a result, the short-circuited quasi-optical resonator filled with powder is formed.

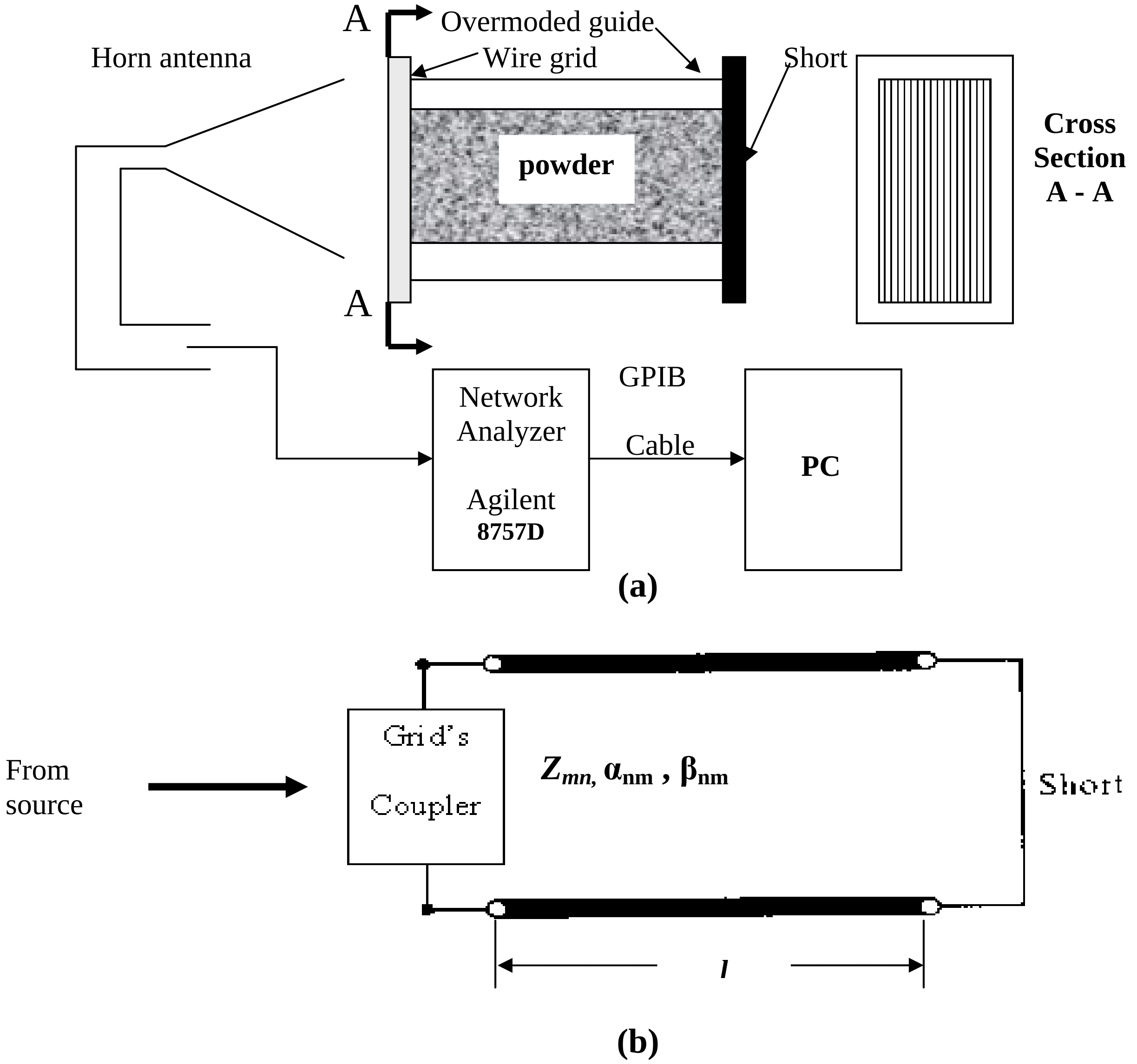

Fig. 1 Experimental setup (a) and equivalent circuit (b) of quasi-optical resonator.

The horn antenna connected with W-band Network Analyzer excites the resonator. Measured reflection coefficient data are transmitted to PC and stored for a further processing. Frequency synthesizer Agilent-83623B being as a part of Agilent-8757D Network Analyzer provides frequency resolution 1 MHz and up to 1600 sampling points within a fixed sweeping range. It is sufficient to observe rather narrow resonance curves of low loss materials under test.

3 Description of the model

A quasi-optical resonator used for characterization of powder at mm-waves can be presented by its equivalent circuit depicted in Fig 1b, where Z_{mn} is the impedance of a resonating waveguide mode TE_{mn}, α_{mn} is an attenuation constant; $\beta_{mn} = 2\pi f_{mn} /c;$ m , n are integer numbers. The resonance frequencies f_{mnp} of ideal resonator can be calculated [3, p.430] using formula:

$$f_{mnp} = \frac{0.3}{\sqrt{\varepsilon_r}} \sqrt{(\frac{2l}{\lambda_c})^2 + p^2}, \quad \text{GHz} \tag{1}$$

where: ε_r is relative dielectric constant of medium filled a resonator, λ_c is critical wavelength, p = 1,2,… are integer numbers, l is the length of resonator. Input coupling element causes a shift of a resonance frequency which is not easy to predict at mm waves due to complexity of coupler. As a result, uncertainty in identification of true values of m, n and p may occur in a practice. The procedure discussed below permits to avoid this undesirable effect.

Using polarizing properties of grid's coupler we can provide an excitation of some selected modes which demonstrate a regular distribution of resonant frequencies as a function of longitudinal number p. In this case, the resonance frequency, $f_{mn(p+1)}$ corresponding to the next number $p+1$ is written as:

$$f_{mn(p+1)} = \frac{0.3}{\sqrt{\varepsilon_r}} \sqrt{(\frac{2l}{\lambda_c})^2 + (p+1)^2}, \quad \text{GHz} \tag{2}$$

Assuming that both frequencies f_{mnp} and $f_{mn(p+1)}$ are known from measurements we can write the following equation derived from (1) and (2) that determines longitudinal number p:

$$r = \frac{q + (p+1)^2}{q + p^2} \tag{3}$$

where: $r = (f_{mn(p+1)} / f_{mnp})^2$ *and* $q = (2l/ \lambda_c)^2$. The solution of (3) yields the two values of p

$$p_{1,2} = \frac{1 \pm \sqrt{r - q + 2rq - qr^2}}{r-1} \tag{4}$$

There is no universal rule for distinguishing these roots formally. However, in many practical situations associated with quasi-optical resonators $p_1 >> p_2$ and the first solution should be used for reconstructing ε_r from measured data resulting in the following expression:

$$\varepsilon_r = (\frac{0.3}{f_{mnp}})^2 [(\frac{1}{\lambda_c})^2 + (\frac{p_1}{2l})^2] \tag{5}$$

In order to reconstruct dielectric loss associated with ε'' from the measured data it is necessary to evaluate behavior of the reflection coefficient in vicinity of resonance frequency of the specified mode. Let's assume that the grid's coupler can be characterized by power reflectance R_p. It can be derived that the input reflection coefficient of the shorted resonator shown in Fig.1 is written as follows [4]:

$$\Gamma(R_p,\alpha,f)=\frac{(1-2i\sqrt{\frac{R_p}{1-R_p}})\tanh(\alpha+i\beta)l-1}{(1+2i\sqrt{\frac{R_p}{1-R_p}})\tanh(\alpha+i\beta)l+1} \qquad (6)$$

It should be pointed out that (6) is valid for any loss and can be used for direct reconstruction of ε'' from measured values of $\Gamma(R_p, \alpha, f)$.

4 Reconstructing algorithm

To apply the procedure for reconstructing ε'', the two measurements of Γ must be done at some specified frequencies, namely, $\Gamma(R_p, \alpha, f_0)$ and $\Gamma(R_p, \alpha, f_1)$, where f_0 is the resonance frequency and f_1 is an arbitrary frequency located just near the resonance one. In standard measurements the f_1 corresponds to a 3 dB level assuming that the resonance curve has symmetrical shape relatively its resonance frequency. Since, the measured resonance curve may be asymmetric we do not use the 3dB criterion. If the measured values A_0 and A_1, correspond to frequencies f_0 and f_1, the following system of two nonlinear equations can be written for unknown R_p, and α:

$$\Gamma(R_p, \alpha, f_0) = A_0$$
$$\Gamma(R_p, \alpha, f_1) = A_1 \qquad (7)$$

The system (7) can be solved numerically. In practice, the resonance curve becomes asymmetrical due to excitation of parasitic modes, and measurements of a reflection coefficient must be done on both sides of the resonance curve (below and above of the resonance frequency). The value of the frequency detuning $\Delta f = f_1 - f_2$ depends on asymmetry of a resonance curve. The real trade-of corresponds to almost the same resonator parameters determined for both detuned frequencies – f_1 and f_2. The averaged value of R_p, and α estimated for these detuned frequencies are used later as a final result of the measurement carried out. It should be pointed out that the basic assumptions used in the model formulations are:

- the resonant frequency of the selected mode is determined by a real part of dielectric constant;
- the width of resonant curve of the same mode is determined by imaginary part of dielectric constant.

Hence, strictly speaking, the above assumptions are valid for low loss powders which are the subject of this paper.

5 Calibration of experimental setup

In measurements described below we used a section of overmoded rectangular guide with dimensions 10.5x25x189.8 mm^3 fabricated from Al-alloy. The input coupler was as an array of one dimension wire grids with the period 0.5mm and wire diameter 0.02mm resulting in power reflection of about 0.9 that was sufficient to form a quasi-optical resonator. To provide an excitation of quasi-T_{01p} mode the wires were oriented parallel to the wall having a width of

25 mm. Agilent-8757D Network Analyzer was employed for measurement of input reflection coefficient. Its output was connected with a rectangular horn antenna illuminating the grid coupler. The distance between them was adjusted to provide the best suppression of undesirable modes. First of all, the calibration of experimental setup must be done in order to estimate unloaded Q_{metal} – factor of quasi-optical resonator associated with loss energy in metal walls. Such a calibration has been performed for the air-filled resonator.

Typical measured reflectance (return loss in dB) as a function of frequency corresponding to air filled resonator is shown in Fig.2. There are several observable resonances within measured frequency range 97 –100 *GHz* with averaged frequency shift between the two nearest resonances $\Delta f = 0.7725$ *GHz*. Lets select from Fig.2 value $f_1 = 97.33 GHz$ as a resonance frequency of the selected mode with index p and $f_2 = f_1 + \Delta f = 98.1025$ *GHz* as a resonance frequency of the nearest mode with index $p+1$. From (4) we can calculate the two values of p: $p_1 = 122.37$ and $p_2 = 2.161$. The first one after rounding to nearest integer number $p_1 \approx 122$ corresponds to real experiment conditions and should be used for reconstructing dielectric constant while the second one must be neglected. Based on (5) we can determine relative dielectric constant of a medium filled with the quasi-optical resonator, $\varepsilon_r = 1.01$. This result is very close to the air's dielectric constant proving that the assumptions introduced into the model were valid.

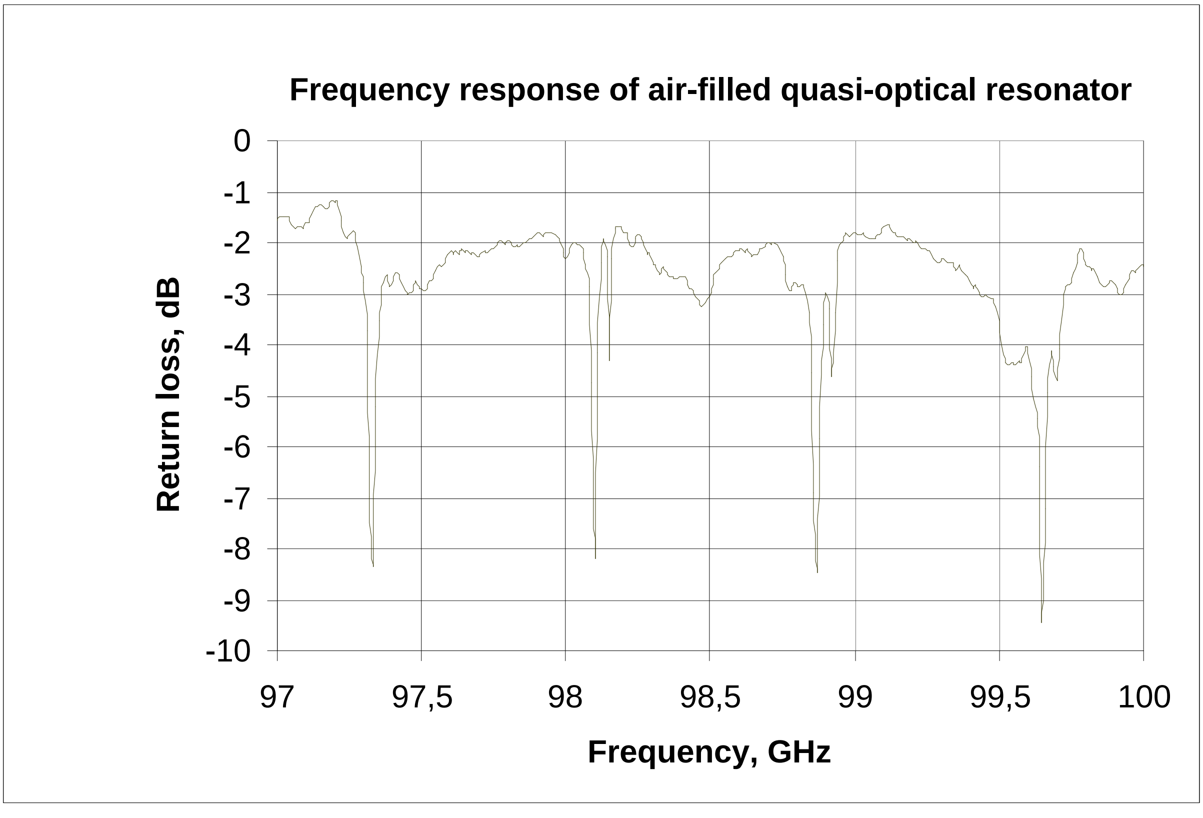

Fig. 2 Measured return loss of air-filled quasi-optical resonator as a function of frequency.

To estimate unloaded Q_{metal} it is necessary to consider (6) and (7). The solution of (7) at the resonance frequency f_2 yields $R_p = 0.89$ and $\alpha = 0.0526$ *(1/m)*. Now, we can calculate resulting Q_{metal} using formula $Q = \beta/2\alpha$ [3, p.420]. In our case we have determined that $Q_{metal} = 19520$. Then, we can employ (6) to validate an accuracy of reconstructing procedure. Figure 3 shows return losses *RL in dB* measured (dotted line) and reconstructed (solid line) from (6) for the calibrated resonator. Neglecting excitation of parasitic modes we can state that reconstructing procedure works rather effectively.

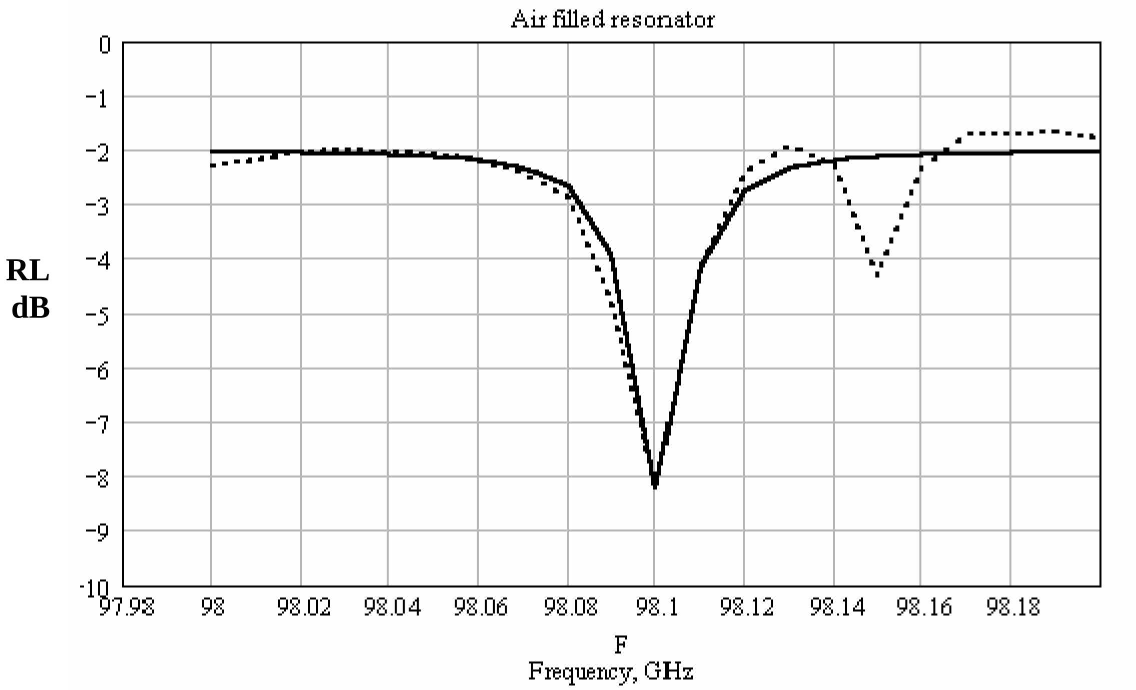

Fig. 3 Measured (dotted line) and reconstructed from (6) (solid line) values of return losses *RL (dB)* for the calibrated resonator.

The following range of guess values providing the same solutions of the system (7) have been determined: $0.01 < \alpha < 0.5$ [1/m] and $0.8 < Rp < 0.99$. So that, a stability of reconstructing procedure is quite well.

6 Low loss powder characterization

The same resonator as described in section 5 has been filled with low loss polymer powder (Fluoropolymer Resine). Its measured reflectance (return loss in dB) is depicted in Fig.4. Now we can reconstruct both real and imaginary parts of complex permittivity the powder under test.

Reconstructing real part of complex permittivity: Repeating calculations of the section 5 we have determined from measured data, Fig.4:

- averaged frequency shift between the two nearest resonances is $\Delta f = 0.685$ GHz;
- index p_1 corresponding to the given experiment conditions is $p_1=140$ at the resonance frequency $f = 97.44$ GHz;
- real part of the dielectric constant reconstructed from (5) is $\varepsilon'_r = 1.286$ that corresponds to density $d = 0.8 g/cm^3$.

It should be pointed out that a real part of dielectric constant is a function of powder density and may vary in a wide range depending on a compressed powder state.

Reconstructing imaginary part of complex permittivity: At first, it is necessary to estimate the unloaded Q_{filled} -factor of the resonator filled with a powder using well known expression:

$$\frac{1}{Q_{filled}} = \frac{1}{Q_{metal}} + \frac{1}{Q_{powder}} \tag{8}$$

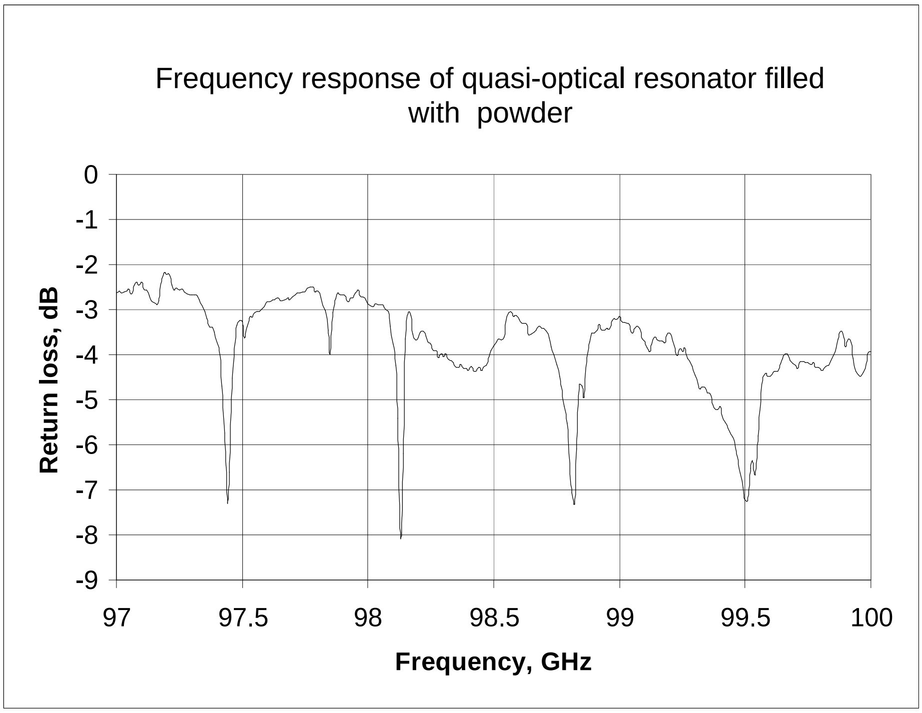

Fig. 4 Measured return loss of quasi-optical resonator filled with powder as a function of frequency.

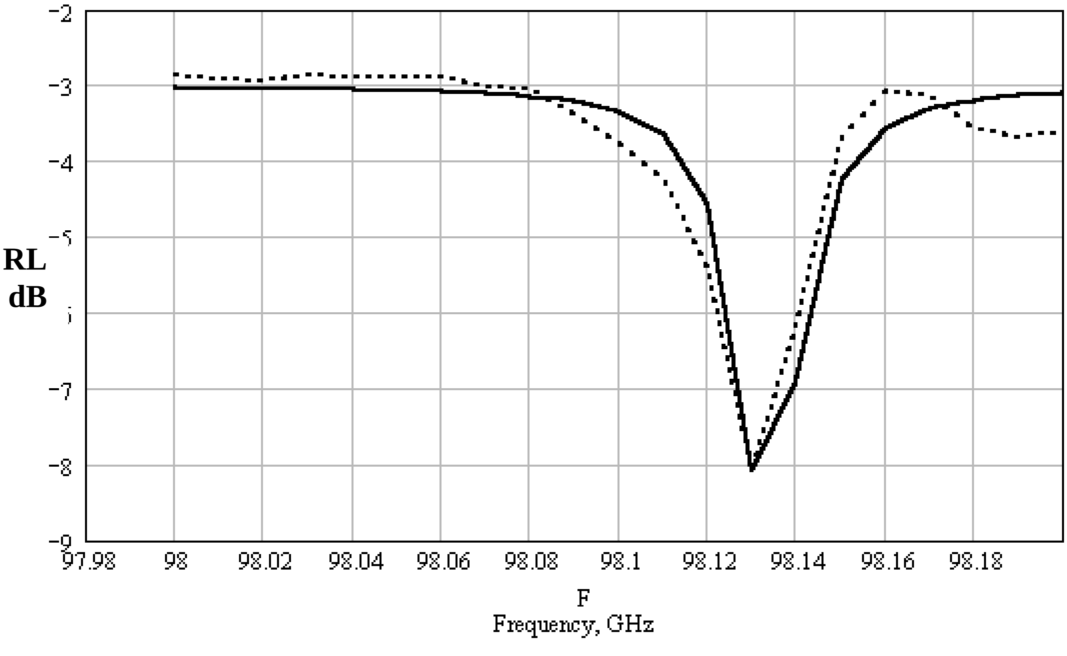

Fig. 5 Measured (dotted line) and reconstructed (solid line) from (6) values of return loss *RL (dB)* for the resonator filled with powder.

For ε'_r determined above the following parameters were found out at the resonance frequency 98.13 GHz: $R_p = 0.863$, and $\alpha = 0.0688$ (1/m) resulting in $Q_{filled} = 16920$. The Q_{powder} determined from (8) is approximately 127000 that corresponds to $\tan\delta = 1/Q_{powder} \approx 0.8x10^{-5}$ resulting $\varepsilon''_r = 1.013x10^{-5}$.

Figure 5 demonstrates both measured result (dotted line) and reconstructed from (6) return losses *RL dB* (solid line) for the resonator filled with the measured polymer powder. Calculation has been made for the resonance mode located near frequency 98.13 GHz. We can see that measured and reconstructed curves are rather close to each other proving an efficiency of the method proposed.

Based on the above measurements we can determine density invariant moisture function $A(\psi)=(\varepsilon'-1)/\varepsilon''$ for the given material, namely, $A(\psi) \approx 2.8x10^4$. Other results are presented in the Table for different compressed state (density) of the powder under testing.

Table: Some results of measurements density independent moisture Ψ %

d [g/cm^3]	0.8	0.85	0.9	0.95	1.0	1.05
ε'_r	1.286	1.321	1.34	1.379	1.42	1.46
$10^5\,\varepsilon''_r$	1.013	1.14	1.21	1.36	1.51	1.62
Ψ %	0.49	0.5	0.51	0.5	0.52	0.51

7 Conclusion

In this paper, we have suggested density-independent moisture method base on an application of mm-wave quasi-optical resonator. Measurements of real and imaginary parts of dielectric constant of low loss powders filling this resonator can be easily performed. The model required for doing the reconstruction procedure has been suggested. Examples illustrating measurement technique have validated the basic assumptions used in the method developed.

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