Discussion of Some Experimental Methods for Permittivity Measurements on 12-Tungstophosphoric Acid Hexahydrate in the Frequency Range 8-12 GHz

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Abstract

Three different methods have been used for permittivity measurements of 12-tungsto-phosphoric acid hexahydrate and their relative merits are discussed. Complex permittivity was measured in shorted rectangular waveguide and by the network analyzer in the frequency range of 8-12 GHz at room temperature.

1. Introduction

Complex permittivity of 12-tungsto-phosphoric acid hexahydrate (WPA-$6H_2O$), which belongs to the group of protonic conductors [1], was measured by three different methods in the frequency range of 8-12 GHz.

The first one is a classic method of measurement in a shorted waveguide (the short circuiting method) [2]. This method is reliable for low-loss solid dielectric samples and 12-tungsto-phosphopric acid (WPA-$6H_2O$) is known to be a high-loss dielectric.

The second method is a reflection measurement on the network analyzer, i.e. the short circuiting method applied on network analyzer.

The third method is the well-known Nicolson-Ross method, proposed by the manufacturer of network analyzer.

The same sample was used in all three methods. The sample was made by pressing the hexahydrate powder under pressure of 3 t/cm^2 into a metallic frame whose shape and dimensions matched the cross-section of the rectangular waveguide IEC-100 (22.86x10.16 mm). This procedure produces the solid sample and a tight contact between the sample and the waveguide sides. The sample thickness was chosen so as to ensure the least possible error in this particular frequency range [3].

2. Measurement Method

Applied short circuiting measuring method [2] consists in the following procedure. The curve of the standing wave of the shorted waveguide was measured and then the measured sample is replaced by the short circuit and the standing wave curve was measured again. On the basis of obtained results: the standing wave coefficient S, distance x_m from the standing wave minimum to the sample surface (when the sample is in the waveguide), wavelength λg in the part of the waveguide filled with air, as well as known sample thickness d - impedance in the cross-section of the sample/air boundary surface is determined. Real R_e and imaginary I_m parts of this impedance, normalized with wave impedance of the waveguide in the part filled with air, were calculated by the following formulas:

$$R_e = \frac{S\left(1 + tg^2(\beta x_m)\right)}{S^2 + tg^2(\beta x_m)}, \quad (1)$$

$$I_m = \frac{\left(1 + S^2\right) tg^2(\beta x_m)}{S^2 + tg^2(\beta x_m)}, \quad (2)$$

$$\beta = 2\pi / \lambda_g \quad (3)$$

Real and imaginary parts of the impedance on the sample/air boundary surface may also be expressed by propagation parameter in the part of the waveguide filled with the sample, $\gamma_u = \alpha_u + j\beta_u$, where α_u is attenuation and β_u phase coefficient in the material that is being tested:

$$z_u = R_e + jI_m = \left(j\beta/\gamma_u\right) th(\gamma_u d), \quad (4)$$

By equalizing the real and imaginary parts of the impedance in the sample/air cross-section, expressed by measured values and propagation parameters in the sample, formulas for calculation of attenuation and phase coefficient in the sample α_u and β_u have been obtained, and on this basis values of real and imaginary parts of complex permittivity ε' and ε'' can be calculated:

$$\varepsilon' = \frac{1 - (\alpha_u^2 - \beta_u^2)\left(\frac{\lambda}{2\pi}\right)^2 \frac{1}{1-(\lambda/\lambda_g)^2}}{1 + \left(\frac{\lambda}{2\pi}\right)^2 \frac{1}{1-(\lambda/\lambda_g)^2}}, \quad (5)$$

$$\varepsilon'' = \frac{2\alpha_u\beta_u\left(\frac{\lambda}{2\pi}\right)^2 \frac{1}{1-(\lambda/\lambda_g)^2}}{1 + \left(\frac{\lambda}{2\pi}\right)^2 \frac{1}{1-(\lambda/\lambda_g)^2}}, \quad (6)$$

where λ is the wavelength of the generator.

This measurement method was also performed on the network analyzer, as a reflection measurement. Sample was shorted at one side and placed at one port of network analyzer. Measured parameter $S_{11}\angle\varphi_{11}$ as well as real R_e and imaginary I_m parts of the input impedance on the sample/air boundary can be calculated. If we express measured S parameter in the following form:

$$S_{11}\angle\varphi_{11} = S_r + jS_i, \quad (7)$$

then real and imaginary parts can be expressed as follows:

$$R_e = \frac{(1-S_r^2)-S_i^2}{(1-S_r^2)^2 - S_i^2}, \quad (8)$$

$$I_m = \frac{2S_i^2}{(1-S_r^2)^2 - S_i^2}, \quad (9)$$

The procedure of calculation of complex permittivity is continued by formulas (4)-(6).

The simulation showed and measuring in distilled water proved that the precision of measuring on higher loss dielectrics by the short circuiting method also depends on the sample thickness. If the sample thickness is small, standing wave coefficient is very high and therefore it is difficult to determine its exact value. If the sample thickness exceeds certain value, which is not so big for high loss dielectrics, an effect of "infinite layer thickness" appears, i.e. further increase of thickness does not affect the reflection coefficient. Therefore, it is also necessary to assess the range of thickness in which measuring would be reliable.

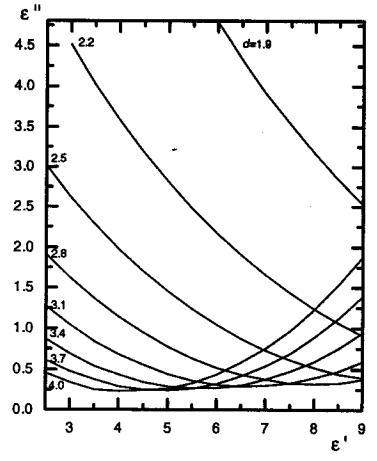

Fig. 1. Dependence ε''(ε') on varying sample thickness (in mm) for S=8.

Fig. 1 represents an example of the simulation results. It was presumed that standing wave coefficient of $S=8$ was measured and then for varying dielectric thickness d (in mm) values of ε'' for determined ε' were calculated.

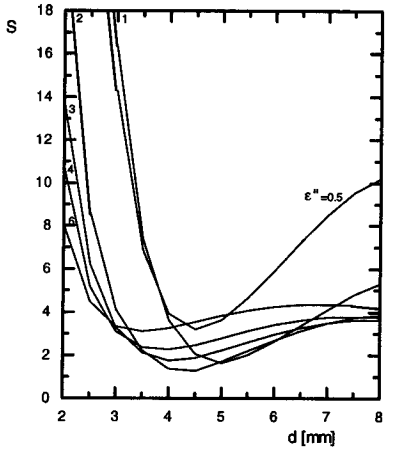

Fig. 2. Dependence S(d) for varying values of ε'', upon ε'=3.7.

The curve shows that in this case the most convenient thickness values were those above 3 mm. Fig. 2 represents results of one of the simulations, too. It was presumed that the real part of the complex permittivity was 3.7, so for various values of the imaginary part of the complex permittivity measured distances between standing wave minimum and the boundary sample/air surface were proposed and thus the corresponding coefficient of the standing wave was calculated.

The simulations showed that the most convenient sample thickness values were those between 3 and 5 mm for expected values of permittivity for 12-tungsto-phosphoric acid hexahydrate (real part between 3 and 12 and imaginary part around 1).

The Nicolson-Ross method was proposed by the manufacturer of network analyzer HP 8410S [4] and it consisted in measuring of $S_{11}\angle\varphi_{11}$ and $S_{21}\angle\varphi_{21}$ parameters, by which reflection coefficient Γ and

transmission coefficient T were found and, consequently, the values of complex permeability and complex permittivity. Between the two waveguides a plate with pressed sample in a frame, whose dimensions matched the cross-section of the waveguide, is laid and S parameters were measured. As this is the standard method, we shall not discuss it in detail but we shall list the formulas used upon our measuring. Γ and T are expressed by the following formulas:

$$\Gamma = K \pm \sqrt{K^2 - 1}, \tag{10}$$

where:

$$K = \frac{\left[(S_{11}\angle\varphi_{11})^2 - (S_{21}\angle\varphi_{21})^2\right] + 1}{2(S_{11}\angle\varphi_{11})}, \tag{11}$$

$$T = \frac{\left[(S_{11}\angle\varphi_{11}) + (S_{21}\angle\varphi_{21})\right] - \Gamma}{1 - \left[(S_{11}\angle\varphi_{11}) + (S_{21}\angle\varphi_{21})\right]\Gamma}, \tag{12}$$

$$\mu_r = \frac{1+\Gamma}{\Lambda(1-\Gamma)\sqrt{\frac{1}{\lambda^2} - \frac{1}{\lambda_c^2}}}, \tag{13}$$

$$\varepsilon_r = \frac{\left(\frac{1}{\Lambda^2} - \frac{1}{\lambda_c^2}\right)\lambda^2}{\mu_r}, \tag{14}$$

where:

$$\frac{1}{\Lambda^2} = -\left[\frac{1}{2\pi d} ln\left(\frac{1}{T}\right)\right]^2, \tag{15}$$

d represents thickness of pressed sample and λ_c the critical wavelength.

3. Results of Measuring

Measurements in 8-12 GHz range were performed on a slotted rectangular waveguide HP809C (cross-section dimensions: 22.86 x 10.16 mm), fed by generator HP 8673C and on the network analyzer HP8410C.

All measurements were performed at room temperature and humidity (t=20°C, relative humidity around 40%).

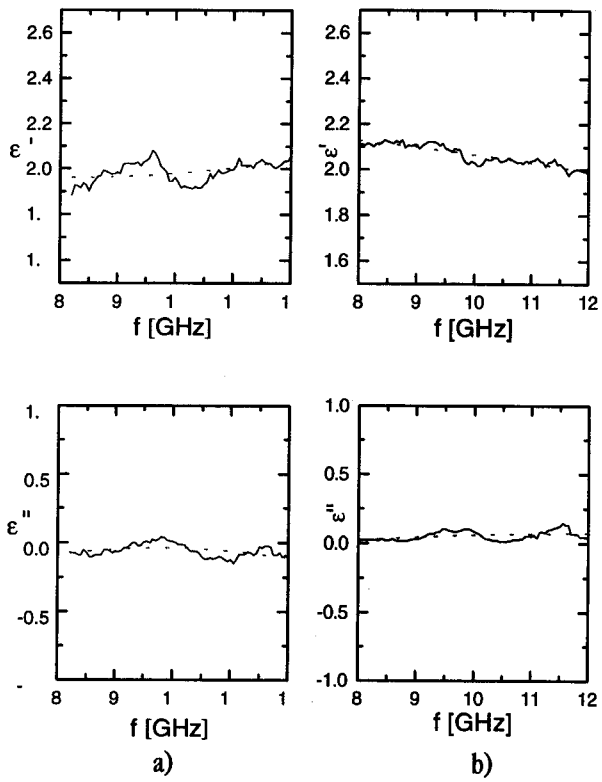

Fig. 3.

In order to test the above mentioned methods, control measurements were performed first on the familiar dielectric - Teflon [5]. The Teflon sample thick 6 mm completely fitted the opening in the frame of 6 mm. Diagrams in Fig. 3a show results of measuring the real and imaginary parts of relative permittivity according to Nicolson - Ross method, and diagrams in Fig. 3b show results obtained by method of reflection measurement by network analyzer.

The sample of pressed hexahydrate was 3 mm thick and placed in the frame of 4.45 mm. Fig. 4a shows results of measuring on hexahydrate by Nicolson-Ross method and Fig. 4b shows results obtained by the reflection measurement on network analyzer. Fig. 4c shows results of measuring on slotted waveguide.

As it is obvious from the diagrams, the Nicolson-Ross method, recommended by the manufacturer, gave more considerable deviation from the average values than the method of short circuiting. Deviations for some frequencies were considerable even with low loss dielectrics, like Teflon, and they became even greater with high loss materials. The short circuiting method represents one of the oldest measuring methods which is rarely used because it requires solving of transcendent equation (4) whose solutions are manifold. However, by using a computer, such difficulties have been overcome and the equation applied on network analyzer proved to obtain good results.

From the measurement results on the slotted waveguide and reflection measurement on network analyzer for ε' and ε'' versus frequency (Fig. 4c), after deduction of conductivity loss [6], a Cole-Cole diagram [7] was drawn. The Cole-Cole diagram allowed calculation of time or times of bipolar relaxation and on the basis of these details identification of protonic entities was performed, which was the aim of these measurements.

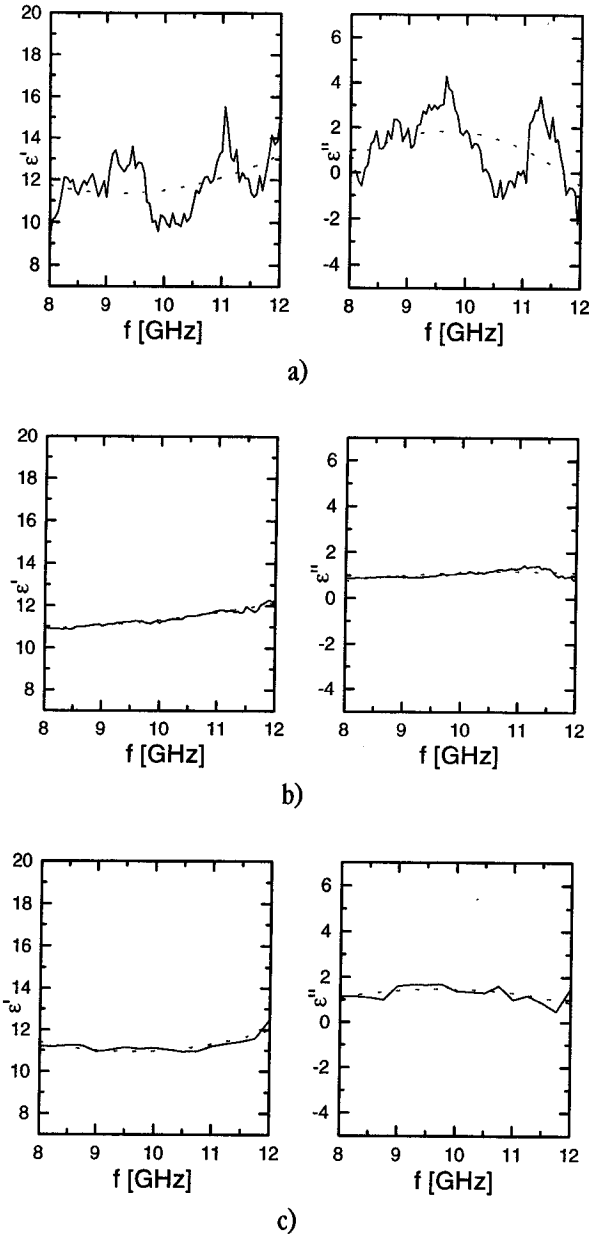

Fig. 4.

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