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Space-adjustable Non-contact 3-electrodes Systems and the Relative Permittivity and Loss Angle Tangent Measurement of Insulating Films

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Abstract: IEC and Chinese national standards recommend the air-medium space-adjustable non-contact 3-electrodes method for the relative permittivity and loss angle tangent measurement of insulating materials. With nearly no technical instructions in the standards, during application of this method, especially to very thin films, many technical matters are unsettled, while they will influence the test results remarkably. In this paper, using the high-precision space-adjustable non-contact 3-electrodes systems developed, relevant influencing factors in the measuring process are thoroughly studied, including the measurement accuracy of sample thickness, the whole thickness of film multi-layers, and the remaining air space between the sample and the electrode, and suggestions for successful experiments are provided. Besides, three methods for film measurements are presented and discussed. At present, the space-adjustable non-contact 3-electrodes systems have been successfully applied to measure relative permittivity and loss angle tangent of plate samples and thin films with measuring error of relative permittivity less than 5% and the order of loss angle tangent up to 10^{-5}.

Key-Words: space-adjustable non-contact 3-electrodes system, plate sample, thin film, relative permittivity, loss angle tangent

1 Introduction

Because of its excellent dielectric performance, organic insulating films are widely used in electrical and electronic equipments. For films thicker than 100μm, the relative permittivity(ε_r) and loss angle tangent($tan\delta$) can be measured by recommended methods of IECpub250-69[1]. While in recent years, films are becoming thinner and thiner. For example, the polypropylene(PP) films used in all-film power capacitors are even no thicker than 5μm. For such thin films, measuring error of ε_r and $tan\delta$ may be as high as 10% to 50%. Thus much attention from all over the world has been focused on the measuring method and technique of thin films.

ASTM, JIS, FN and etc. introduced their methods respectively[2]. IEC recommended the "component method", in which films were made into samples of capacitor component type and measured[3]. Chinese Researchers began to study this problem since 1980[4,5], and reported the space-fixed non-contact electrode method on ICPADM in 1985[6].

In recent 10 years, the development of precision mechanical measuring and processing technique make it possible for the non-contact measuring method to change from space fixed to adjustable. The IEC standard drafting workgroup for film test method recommended the space-adjustable 3-electrodes measuring technique on the basis of the air-medium non-contact method in the appendage specially added to the film measuring method in 2002[7]. With air as the medium, besides conventional samples, the non-contact 3-electrodes system can be used to measure ε_r and $tan\delta$ of expansile materials, papers, thin films and laminates, to which contact electrodes are inapplicable for the lack of appropriate electrode materials to be coated on the sample surface[8].

Two high-precision non-contact 3-electrodes systems are designed on the air-medium space-adjustable principle. One is the electrode-50(2) system whose electrode dimensions are conventional with 50mm, 74mm and 2mm being the measuring electrode diameter, high-voltage electrode diameter, and guard gap width[9]. This system can be used to measure ε_r and $tan\delta$ of common plate samples and thick films. The other one, the electrode-38(0.2) system, with corresponding dimensions 38mm, 50mm and 0.2mm respectively, is specially designed for very thin films. The measuring and testing technique of the space-adjustable non-contact method is thoroughly studied in this paper, and the investigations mainly focus on its application on films thinner than 25μm. Works in this paper are supposed to provide theoretical and technical supports for Chinese national standards to be pushed in the soon future and to generalize the air-medium space-adjustable non-contact 3-electrodes measuring method.

2 Technical Innovations of the Electrode for Thin Film Measurement

The key innovations of the electrode-38(0.2) system especially for thin film measurements are the narrow guard gap which is 0.2mm wide and the small measuring electrode which is 38mm in diameter.

A picture of the electrode-50(2) system, including the electrode and the space indicator, is given in Fig.1. The introductions of its structure and usage can be found in [2]. And it has been proven that ε_r and $tan\delta$ of common plate samples and thick films can be measured accurately by this device[10].

Fig.1 the electrode-50(2) system

When the voltage is applied, the electrical field between the high-voltage and measuring electrode is not absolutely uniform, for at the edge of the measuring electrode, the electric fluxlines still bend outside, which enlarges the effective area of the measuring electrode[11]. What makes things worse is that different electrode space causes different bending extent of electric fluxlines, so as to different effective area of the measuring electrode.

To illustrate this, electrical field in the electrode region is investigated. The profile of the electrode system for field analysis is shown in Fig.2 and this issue is two-dimensional. The thickness of each electrode is 20mm and the width is determined by actual size. The test sample is supposed to be a PP film whose ε_r is 2.2 and thickness is 0.1mm. The sample is placed on the surface of the high-voltage electrode and 5mm wider than it in each side. The relative pemittivity of the air is 1.0. The electrode set is at the center of a square with side length of 200mm, which is the outer earthy boundary of the profile. The potential of the measuring and guard electrode is zero and 100V is applied on the high-voltage electrode. What to be investigated are patterns of equipotentials in the air and the sample, and the region at the edge of the measuring electrode is enlarged and represented.

For the electrode-50(2), when the electrode space is set to be 0.2mm, 5mm and 10mm respectively, the patterns of equipotentials are shown in Fig.3. It can be seen that when the electrode space changes, the pattern of equipotentials is varying, so the effective area of the measuring electrode is different: for 0.2mm space, it is approximately the original size; while for 10mm, it is more exact to take (50+2)mm as the effective diameter. For accurate solution, the effective area of the measuring electrode should be modified by the following equation[9]

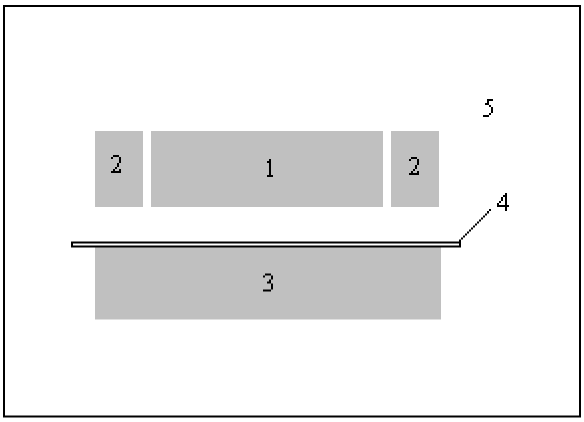
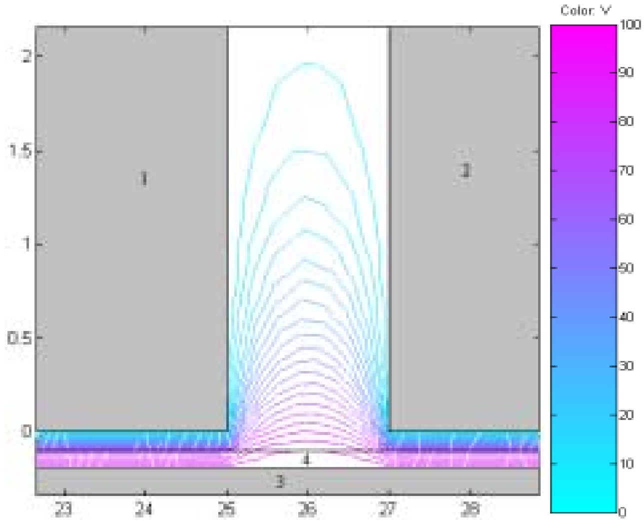
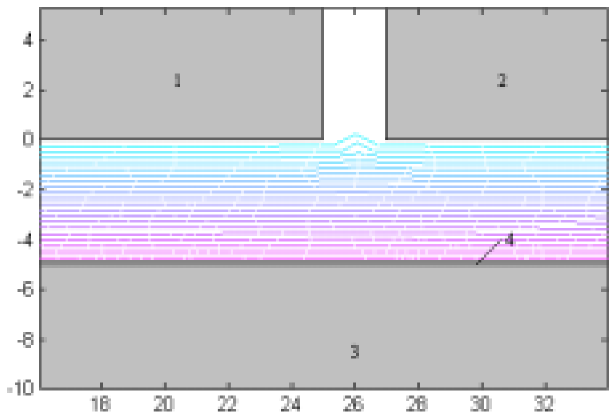


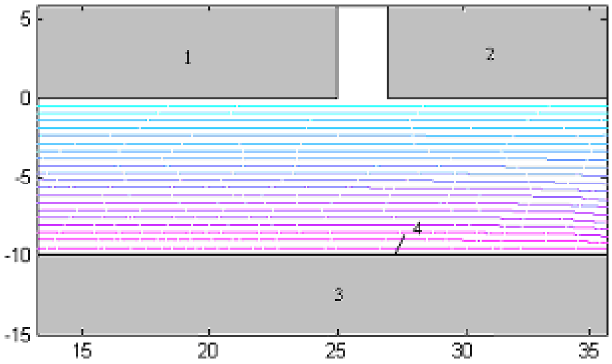
Fig.2. Profile of the electrode system for field analysis. 1, the measuring electrode; 2, the guard electrode; 3, the high-voltage electrode; 4, the test sample; 5, the air.



(a)



(b)



(c)

Fig.3. Equipotential lines of the electrode-50(2) with different spaces between the measuring and high-voltage electrode. a, 10mm; b, 5mm; c, 0.2mm. The color-voltage ruler is shown in (a) and it is the same for (b) and (c).

$$D = D_1 + g - 2\delta = D_1 + Bg \tag{1}$$

where $\delta = h[2/\pi \ln\cosh(\pi g/4h)]$, $B = 1 - 2\delta/g$, D and D_1 are the effective and original diameters of the measuring electrode, mm; g is the guard gap width, mm; and h is the electrode space, mm. Equation (1) shows the influence of guard gap on the effective electrode area, which can be seen more clearly by the air capacitance measurement of the electrode-50(2) system.

Capacitance C and loss angle tangent $tan\delta$ of the air are measured under 100V with the electrode space changing from 1.000mm to 0.020mm as shown in Table 1. In the table, C_m and $tan\delta$ are measured values by an electric bridge, C_u and C_c are capacitance values theoretically calculated by

$$C_u = \varepsilon_0 \frac{S}{h} = \varepsilon_0 \frac{\pi D_1^2}{4h} \tag{2}$$

$$C_c = \varepsilon_0 \frac{S'}{h} = \varepsilon_0 \frac{\pi D^2}{4h} \tag{3}$$

and we define

$$\gamma_{C_U} = \frac{C_m - C_u}{C_u} \times 100\% \tag{4}$$

$$\gamma_{C_U} = \frac{C_m - C_c}{C_c} \times 100\% \tag{5}$$

where ε_0 is the air permittivity and $tan\delta$ of clean air is supposed to be zero. It is shown that after the modification, the deviation of measuring data from theoretical values diminishes. Except for the smallest two spaces, where the measuring precision is limited by system accuracy, the errors are no more than 2%; while before the modification, the biggest error is over 5%. Thus it is necessary to do modification on measuring data from the electrode-50(2).

ε_r and $tan\delta$ of samples are calculated by:

$$\varepsilon_r = \frac{t}{t - (t_1 - t_2)} \tag{6}$$

$$\tan\delta = (\tan\delta_1 - \tan\delta_2)\frac{t_2}{t - (t_1 - t_2)} \tag{7}$$

Table 1. C and $tan\delta$ of the air at different electrode space

Space (mm)	C_m (pF)	C_u (pF)	γ_{Cu} (%)	C_c (pF)	γ_{Cc} (%)	$tan\delta$ ($\times 10^{-4}$)
1.000	18.04	17.39	3.7	17.97	0.4	0
0.900	20.25	19.32	4.8	19.91	1.7	0
0.800	22.68	21.73	4.4	22.33	1.6	0
0.700	25.77	24.84	3.7	25.44	1.3	0
0.600	29.87	28.98	3.1	29.59	1.0	0
0.500	35.61	34.77	2.4	35.39	0.6	0
0.400	44.16	43.46	1.6	44.08	0.2	0
0.300	57.98	57.95	0.0	58.57	-1.0	1
0.200	88.44	86.93	1.7	87.54	1.0	1
0.100	172.32	173.85	-0.9	174.47	-1.2	1
0.090	193.09	193.17	0.0	193.78	-0.4	1
0.080	215.29	217.31	-0.9	217.93	-1.2	1
0.070	247.71	248.36	-0.3	248.97	-0.5	1
0.060	287.03	289.75	-0.9	290.37	-1.1	1
0.050	347.71	347.70	0.0	348.32	-0.2	1
0.040	426.98	434.63	-1.8	435.24	-1.9	2
0.030	547.65	579.51	-5.5	580.12	-5.6	2
0.020	825.76	869.26	-5.0	869.87	-5.1	2

where ε_r and $tan\delta$ are relative permittivity and loss angle tangent of test sample; t is the sample's average thickness, mm; t_1 and t_2 are electrode spaces with and without the sample, mm; and $tan\delta_1$ and $tan\delta_2$ are loss angle tangent with and without the sample.

During the measuring process of the space-adjustable method by the non-contact electrode, the operator needs to balance the electric bridge with and without the test sample, where the electrode space is not the same, resulting in different effective area of the measuring electrode. Especially when the sample is put in the electrode, there is not only the sample but also the air in the space, which makes things even complex. While during the derivation of formula (6) and (7), the areas used to calculate capacitance values for the air and the sample are regarded the same at different electrode space without any modification, which will cause errors to the results inevitably[2].

Theoretically saying, the guard gap is narrower, the influence of electric fluxlines distorting at the edge is smaller, and the result is more accurate. The actual width of the guard gap is restrained by the processing technical level, and 0.2mm is adopted in the electrode-38(0.2), which is the smallest one applied in an electrode system so far. The equipotential lines of the electrode-38(0.2) with the electrode space being 0.2mm are shown in Fig.4.

Compared with Fig.3(a), it can be seen that due to the much narrower guard gap, the bending of the equipotential lines at the measuring electrode edge is much smaller, even at such a small electrode space. Consequently it can be concluded that no matter what the electrode space is, the equipotential lines and the

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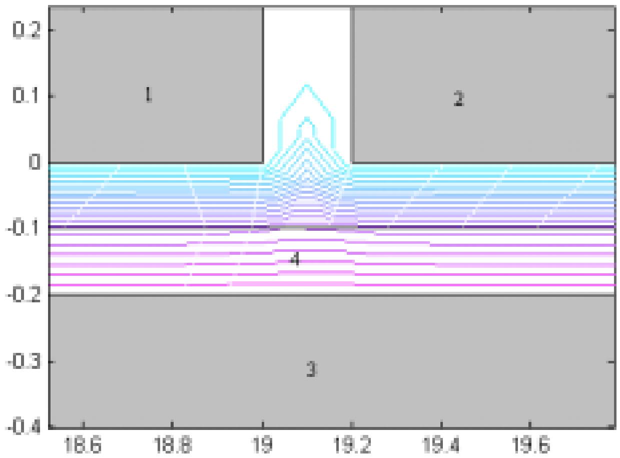

Fig.4. Equipotential lines of the electrode-38(0.2) with 0.2mm electrode space. The color-voltage ruler is the same as that in Fig.3(a).

electric fluxlines in the system region hardly change, enabling the effective area of the measuring electrode to be almost the same as its geometrical size, and as the result the error from the varying effective area of the measuring electrode along with the changing electrode space is eliminated.

Sample capacitance C is theoretically calculated by

$$C = \varepsilon_0 \varepsilon_r \frac{S}{h} = \varepsilon_0 \varepsilon_r \frac{\pi \bullet 38^2/4}{h} \approx 10\varepsilon_r / h \qquad (pF)$$

For the air, ε_r=1, so $C = 10/h$. If the thickness of the sample is 1mm, $\varepsilon_r = C/10$. So data processing is simplified greatly by choosing 38mm as diameter of measuring electrode in the electrode-38(0.2) system.

Besides, the measuring errors augment considerably for very small electrode space as seen in Table 1. It is the result of intrinsic resolution of the optical grating ruler in the system, as well as nonperfect parallel or smooth electrode surfaces, whose influence can be reduced by smaller measuring electrode.

Fig.5 The electrode-38(0.2)

A picture of the electrode-38(0.2) is given in Fig.5. It is vertical type instead of horizontal, which is especially applicable to thin films. The error from folding of films between electrodes for self-gravity is diminished remarkly.

3 Researches and Analyses on Very Thin Film Measurements

Factors to affect the testing results are investigated in this section, including the measuring accuracy of sample thickness, choice of the whole thickness of thin film layers, and choice of the air space remaining between the sample and the electrode. Besides, results from different methods for thin films are presented, as well as comparison between the two non-contact methods. Following experiments on thin films are all carried out by the electrode-38(0.2) with the same space indicator in Fig.1.

3.1 Measurement of Sample Thickness

Accuracy of ε_r and $tan\delta$ tested depends on the measuring accuracy of sample thickness in the space-adjustable non-contact 3-electrodes method. For thin films, the mass-density method can be used to measure sample thickness accurately.

According to formula (6) and (7), if measuring error of sample thickness is the only consideration, then

$$\gamma_{\varepsilon_r} = \frac{\Delta\varepsilon_r}{\varepsilon_r} = (1-\varepsilon_r)\frac{\Delta t}{t} = (1-\varepsilon_r)\gamma_t \qquad (8)$$

$$\gamma_{\tan\delta} = \frac{\Delta \tan\delta}{\tan\delta} = -\varepsilon_r \frac{\Delta t}{t} = -\varepsilon_r \gamma_t \qquad (9)$$

where $\Delta\varepsilon_r$, $\Delta tan\delta$ and Δt are absolute errors of ε_r, $tan\delta$ and t, and γ_{ε_r} , $\gamma_{\tan\delta}$ and γ_t are relative ones. Because ε_r>2 for common dielectrics, γ_{ε_r} and $\gamma_{\tan\delta}$ will be larger than γ_t , and they will rise with larger ε_r. Equation (8) and (9) also show that if $\Delta t > 0$, errors in ε_r and $tan\delta$ will be negative, and vice versa. Thus the sample thickness must be accurately measured in the air-medium space-adjustable non-contact method. Once the accuracy of sample thickness is high enough, $\gamma_t < 0.5\%$, for example, according to equation (8), the relative error of ε_r can be no higher than 0.5% for a material with ε_r=2, still less than 5% even for a material with ε_r=10.

Which are commonly used to measure thickness of thin films are the mechanical thickness-meter and the mass-density method[12,13]. The former one is to directly measure tiered and packed thin film layers by a thickness-meter. In the latter, the mass m, density ρ and area S of film layers are measured, so the thickness t can be obtained by

$$t = \frac{m}{\rho \cdot S} \qquad (10)$$

Monolayer thickness is t/n where n is the number of plies. Experiment data show that the sample thickness values from the two methods differ notably, so ε_r and $tan\delta$ calculated accordingly are not the same, as shown in Table 2.

Table 2. t, ε_r and $tan\delta$ by two methods

PP samples	Thickness-meter method			Mass-density method		
	t (mm)	ε_r	$tan\delta$ ($\times10^{-4}$)	t (mm)	ε_r	$tan\delta$ ($\times10^{-4}$)
1#	0.307	1.94	1.9	0.279	2.13	2.3
2#	0.304	1.95	1.9	0.275	2.17	2.3
3#	0.329	1.88	1.6	0.297	2.08	2.0
4#	0.182	1.92	1.8	0.168	2.08	2.1

1-4# PP samples come from different factories. The data show that ε_r and $tan\delta$ calculated from sample thickness values from mass-density method coincide with referenece quantities of PP films satisfactorily, while those from thickness-meter method are smaller than reference ones. That is to say, sample thickness values from mass-density method are accurate, while positive errors exist in thickness data measured by a thickness-meter. This can be explained by the air remaining between film layers during the mechanical measurement. According to equation (8) and (9), this will bring about negative errors in ε_r and $tan\delta$. So in the air-medium space-adjustable non-contact 3-electrodes measurement the mass-density method is recommended.

3.2 Choice of the Whole Thickness of Thin Film Layers

For each electrode system, there is an electrode space region where the systematic error is the lowest and the tested results are the most accurate. The sample thickness should be chosen to be in this optimal working region.

For very thin films, measurements should be performed on multilayer samples[2]. ε_r and $tan\delta$ of PP film samples with different multi-layer thickness are measured and listed in Table 3. It can be seen that once the whole thickness of film layers is larger than 0.2mm, the result is rather accurate and stable, no matter how thick the monolayer film is and how many layers are tiered. So it is suggested to measure thin films on multilayer samples, and to make sure that the samples are thicker than 0.2mm.

Table 3. ε_r and $tan\delta$ of multi-layer samples

PP sample	Monolayer thickness (μm)	Dielectric parameters	Number of layers 10	20	30
1#	9	ε_r	1.61	2.08	2.13
		$tan\delta$ ($\times10^{-4}$)	2.5	2.1	2.3
2#	13	ε_r	1.85	2.05	2.06
		$tan\delta$ ($\times10^{-4}$)	2.0	2.1	2.1
3#	15	ε_r	1.85	2.20	2.20
		$tan\delta$ ($\times10^{-4}$)	1.7	2.1	2.1
4#	18	ε_r	1.92	2.13	2.11
		$tan\delta$ ($\times10^{-4}$)	1.6	1.5	1.7

3.3 Choice of the Air Space Remaining between the Sample and the Electrode

With air as the medium, for low loss materials, the air space remaining between test sample and the electrode should be small, so as to achieve a high accuracy.

The equation (7) can be written as

$$\tan\delta = (\tan\delta_1 - \tan\delta_2)\cdot\frac{t_1-(t_1-t_2)}{t-(t_1-t_2)} \qquad (11)$$

Because $\frac{t_1-(t_1-t_2)}{t-(t_1-t_2)} > 1$, so $\tan\delta > (\tan\delta_1 - \tan\delta_2)$. For an electric bridge whose accuracy is 5×10^{-5}, the lower limit of $tan\delta$ to be tested is 5×10^{-5}. If one wants to measure a PP sample whose $tan\delta$ is 1×10^{-4} and ε_r is 2.2, then from (11) he gets $\frac{t}{t_1} = 68.75\%$. If $tan\delta$ is decreased to 0.8×10^{-4}, then $\frac{t}{t_1} = 78.57\%$. So generally the sample thickness is required to be no less than 80% of the whole electrode space, which is especially important for $tan\delta$ measurements of low loss materials[9].

3.4 Different Testing Methods for Films and Interpretation of Results

Several methods can be used for film measurements. Among them, the vacuum deposition method is feasible for thick films; the space-fixed non-contact method can be used for ε_r and $tan\delta$ spectrums measurement; and the space-adjustable non-contact method can provide the highest accuracy, especially for $tan\delta$ measurements of low loss materials.

3.4.1 Vacuum Deposition Method

Vacuum deposition method is commonly used for thin films[14]. Now apply it to measure ε_r and $tan\delta$ of PP films and the results are shown in Table 4.

Vacuum deposition method is applied on monolayer film samples. The data from Table 4 show that it can provide rather accurate values of ε_r but $tan\delta$ larger than reference quantities. The positive errors in $tan\delta$ may be the result of metal ions migrating and permeating in the depositing process, and this influence will decline along with increased film thickness. Thus it can be deduced that the vacuum deposition method is feasible for thick films.

Table 4. ε_r and $tan\delta$ by vacuum deposition method

PP sample	Monolayer thickness (μm)	ε_r	$tan\delta\,(\times 10^{-4})$
1#	8	2.29	7.8
2#	8	2.12	6.4
3#	12	2.24	6.0
4#	12	2.28	4.5
5#	15	2.13	3.6

3.4.2 The Two Non-contact Methods

If the maching precision of the electrode system is high enough, it is recommended to perform the space-adjustable measurement; if it is not, while the electric bridge can provide a high accuracy, then the space-fixed measurement is more appropriate.

In this system, the electrode space is designed to be changeable by regulating position of the movable measuring electrode, so as to carry out the space-adjustable non-contact measurement. If one fixes the electrode space, regulating the capacitance to balance the bridge with and without the sample, then he can carry out the space-fixed non-contact measurement. $tan\delta$ of air is considered to be zero, then the testing results can be calculated by

$$\varepsilon_r = \frac{1}{1-(1-\frac{C_2}{C_1})\frac{t_0}{t}} \qquad (12)$$

$$\tan\delta = \tan\delta_1\left[1-\left(1-\frac{t_0}{t}\right)\varepsilon_r\right] \qquad (13)$$

where t_0 is the electrode space, mm; C_1 and C_2 are capacitance values with and without the sample, pF.

Results from the two non-contact methods are shown in Table 5. The testing values of ε_r are approximately

Table 5. ε_r and $tan\delta$ by the two non-contact methods

Sample		Dielectric parameters	Non-contact method: adjustable	Non-contact method: fixed
PP	1#	ε_r	2.30	2.26
		$tan\delta(\times 10^{-4})$	2.2	2.2
	2#	ε_r	2.19	2.25
		$tan\delta(\times 10^{-4})$	2.1	2.5
	3#	ε_r	2.16	2.23
		$tan\delta(\times 10^{-4})$	2.1	2.6
	4#	ε_r	2.01	2.25
		$tan\delta(\times 10^{-4})$	1.9	2.4
PI	1#	ε_r	3.47	3.35
		$tan\delta(\times 10^{-4})$	2.4	2.5
	2#	ε_r	3.41	3.30
		$tan\delta(\times 10^{-4})$	1.0	3.4

the same from the two, but $tan\delta$ from the fixed method are much bigger. Now make a simple analysis. To connect the sample with the testing system, wires are needed, whose resistance and inductance is R_l and L_l. For the sake of simplicity, the test object is represented by series circuit of resistance R_x and capacitance C_x, which is connected to the resistance and inductance of wires in series, as in the Fig.6. Thus the equivalent impedance of the testing system are

$$R' = R_x + R_l$$

$$C' = \frac{C_x}{1-\omega^2 C_x L_l}$$

The measured values of capacitance and loss angle tangent are C' and $tan\delta'$, while the real values of the sample are C_x and $tan\delta_x$, so the differences between the measured and real values are

$$\Delta C = C' - C_x = (\frac{1}{1-\omega^2 C_x L_l} - 1)C_x$$

$$\Delta\tan\delta = \tan\delta' - \tan\delta_x = \omega C'R' - \omega C_x R_x$$

At power frequency, $\omega^2 C_x L_l$ <<1, so $\Delta C \approx 0$, and $\Delta\tan\delta = \omega C_x R_l$.

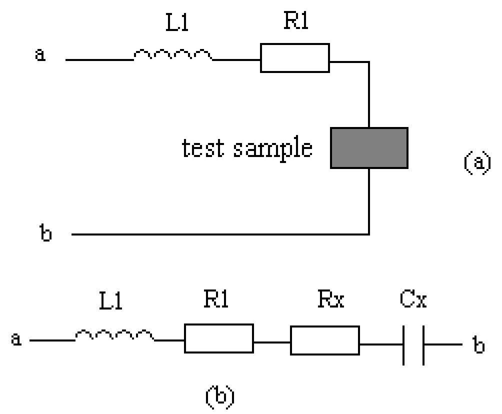

Fig.6. The testing system. a, the connecting sketch; b, the equivalent impedance.

In the space-adjustable method, the capacitance is unchanged during the twi-measurement with and without the sample, so $\Delta\tan\delta$ remains the same. From equation (7), $tan\delta$ is obtained by ($tan\delta_1$– $tan\delta_2$), so the influence from wires is eliminated effectively. While in the space-fixed method, according to equation (13), the calculated $tan\delta$ bears only on $tan\delta_1$, which includes the additional loss of the wires, so consequently tested $tan\delta$ values are bigger than reference quantities. Thus for low loss materials the space-adjustable method is recommended.

However, if one wants to obtain spectrums of ε_r and $tan\delta$ by non-contact method, the space-fixed one is the only choice. The process is as this: with the sample, one gets $C_1 \sim \omega$ and $tan\delta_l \sim \omega$; taking the sample off, one gets $C_2 \sim \omega$; then through appropriate data processing, one gets $\varepsilon_r \sim \omega$ and $tan\delta \sim \omega$. While in the space-adjustable method, the electrode space must be regulated manually to balance the electric bridge at each frequency, making it impossible to achieve an automatic and continuous measurement. Hence in practice the space-adjustable non-contact method is infeasible for spectrum measurement.

4 Conclusions

In this paper, investigations focus on the special technical matters in very thin film measurements, and suggestions for successful measurement are provided. The conclusions are as follows:

(1) Samples can be measured by the air-medium space-adjustable non-contact 3-electrodes system accurately with the measuring error of ε_r less than 5% and the order of $tan\delta$ up to 10^{-5} under the condition that the accuracy of electric bridge on $tan\delta$ reaches 5×10^{-5} and that the sample thickness is accurately measured, by the mass-density method for thin films, for example.

(2) Comparatively saying, the two air-medium space-adjustable non-contact 3-electrodes systems are both applicable to plate samples and thick films, while the electrode-38(0.2) is more appropriate for very thin film measurements because of its special technical designing, including the guard gap 0.2mm wide and the measuring electrode 38mm in diameter.

(3) For films thinner than 25μm, measurements must be employed on multilayer samples whose thickness has to be thicker than 0.2mm to be located in the optimal working region of the electrode system to achieve a high accuracy. Besides, the sample thickness is required to be no less than 80% of the whole electrode space for low loss materials.

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