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Al_2O_3 Crystal Capacitor를 이용한 유전손실 측정

Dielectric Loss Tangent Measurement Using the Al_2O_3 Crystal Capacitor

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Abstract

The standard capacitor must have not only precise value of the capacitance but also the basic properties of low dielectric loss tangent. In the reforming process of capacitors, the dielectric loss tangent must be also reformed. In this paper, the development of standard capacitors of 10 and 100pF for the dielectric loss tangent standard using Al_2O_3 Crystal and the measurement of dielectric loss tangent are discussed. The dielectric loss tangent depends upon the surface between electrode and dielectric in capacitor. With using the Electric Field Simulator, precise design values of electrode are simulated. For the purpose of measuring capacitance effect just in the dielectric, 3-Terminal and 4-Terminal Pair configuration are applied respectively at the electrode and the connector for the measuring equipment. As stated above method, the standard capacitors of 10 and 100pF for the establishment of the dielectric loss tangent standard using the Al_2O_3 Crystal are made with low dielectric loss tangent less than 10-4 .

1. Introduction

The history of capacitor started with experiment of charge accumulation by J. Desaguliers in 1737 and experiment of spark discharge occurred from charge accumulation by C. Ludolph in 1745. Capacitance is the concept of energy storage in an electric field and is restricted to the area, shape and spacing of the capacitor plates and the property of the dielectric material separating them. As the dielectric of capacitor are gas, liquid, and solid and their dielectric constant have effects to capacitance and dielectric loss tangent of capacitor. The study of capacitor has focused on the safety of dielectric property, processing technology and capacitor using high dielectric constant material. Now a days, with highly integrated and small-sized electronic equipment, the development of highly reliable capacitor is especially required in several pF region. But the such tiny-sized capacitors of small capacitance have large errors which depend on the type of dielectric, design method, and operating configuration. There is a need to consider a countermeasure. So high precision capacitors of small capacitance must be developed for standard equipment[1,2].

The standard capacitors have been developed using the dry nitrogen or mica film as a dielectric, generally at 1592 Hz or 1000 Hz. In the reforming process of the capacitance, the dielectric loss tangent must be also reformed. The dielectric loss tangent which is defined δ of dissipation factor at low frequency A.C., is the ratio of accumulated to loss energy of capacitor by periods and is lumped circuit associated with resistance, inductance, and capacitance. The following facts are reasons of importance. At first, as the A.C. resistance and inductance in the impedance components are respectively decided by Quadrature bridge and Maxwell-Wein bridge that are referenced by absolute measuring equipment of calculable cross capacitor, loss tangent is all of the complex conjugated impedance measurement and basic of their research. Second, the measurement of the loss tangent is indispensable in measurement part for dissipation factor that is electrical property in all materials. Third, as applying the loss tangent measurement technology to air or gas dielectric capacitor, the electrical property measurement for microscopically molecular absorption layer can be possible at surface of the metal electrode metallic corrosion, out gassing property of the metal in vacuum, related basic researches as well as measurement result is important information for choice of standard capacitor[3,4,5,6].

In this study, standard capacitors of 10 pF and 100 pF for the establishment of the dielectric loss tangent standards are made using Al_2O_3 Crystal with low dielectric loss tangent, and then the dielectric loss tangents are measured with precision. Obtained using the Electric Field Simulator, precise design values are in addition to stray capacitances. To obtain precision capacitor, the existence of stray capacitances must be located in the dielectric. So, the following methods, namely the high quality Al_2O_3 Crystal with parallel structure, low loss electrode with silver paste, and installation of Al_2O_3 capacitor in the metal construction with 3-terminal structure to protect from external electromagnetic effects, are used.

2. Manufacture and Properties of Al2O3 Crystal

Verneuil in 1902 developed the high temperature flame above 2000℃ to manufacture the artificial ruby and his idea had been researched to the utility so long time. Verneuil method is single crystal growth method by high temperature using the gas. The principle of Verneuil single crystal growth method is following.

Subtle powder source fills up the reservoir and the hammer tap to the source. Subtle powder in the reservoir must be homogeneously descend by tapping. Powder source is completely mixed with Oxygen applied as delicate partial pressure and then moves to end of burner. Applying hydrogen to the outer of burner separated with dual tube, igniting the mixed with hydrogen, oxygen and powder source at the end of burner makes high temperature above 2100℃. Melted subtle powder falls with flame on the seed and crystal growths. Supporter must descend with the same of crystal growth speed, high temperature part of flame to locate top of crystal growth.

The Verneuil method has many merits that easily obtaining high temperature and not only cost reduced by non melting pot but also protection of injected impurity from melting pot and large sized crystal growth as short time. Verneuil method grown by melted flame restricts the kind of single crystal; Ruby(Al_2O_3+Cr_2O_2), pure Al_2O_3, doped Al_2O_3, Rutile(TiO_2), colored Rutile, Spinel and $SrTiO_3$.

In the study, the dielectric is high pure powder of 99.9% Al_2O_3. Purity is important but particle size is more important to powder. The particle size of Al_2O_3 powder adapts Verneuil method and processing is follows.

1. $2Al + 3H_2SO4 \rightarrow Al_2(SO_4)3 + 3H2$

2. $Al_2(SO_4)\ 3 + (NH_4)\ 2SO_4 + 24H_2O \rightarrow (NH_4)\ 2SO_4 \cdot Al_2(SO_4)3 \cdot 24H_2O$

3. $(NH_4)\ 2SO_4 \cdot Al_2\ (SO_4\ )3 + 24H_2O \rightarrow Al_2O_3 + (NH_4)\ 2\ SO_4 + 3SO_3 + 24H_2O$

Al_2O_3 Crystal is conundrum structure, which shows the hexagonal closest packing with the oxygen and the stratford structure -ABABA. Al3+ ion is located on interstice with 6 oxygen between layers. As crystal axes are same a1, a2 and a3 but c axes are different, physical properties are widely different with each crystal direction. The difference of thermal expansion coefficient occurs biggest crack. For crystal side of 0001 also consist highest density in molecular structure, growth speed and grown crystal boule vary with angle difference from growth direction and crystal side. Cylindrical boule crystal obtains, crystal growth speed is fast and to prevent the crack by thermal expansion is possible, when angle of growth direction and 0001 direction is 60° with crystal structure[7,8,9].

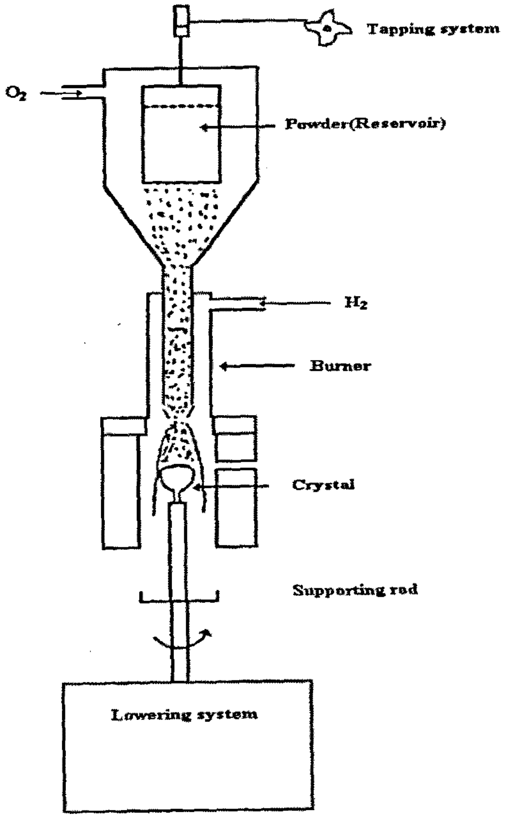


Fig. 1. Principle of Verneuil equipment

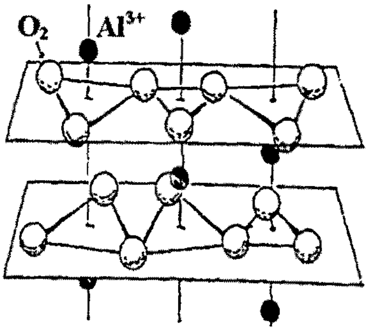


Fig. 2. Corundum structure of Al_2O_3 Crystal

Physical	Density	3.98
	Dislocations density	10^4~10^6 ㎠
	Hardness victors	2500~3000 kg/㎟
	Modulus of elasticity	36000~44000 kg/㎟
	Modulus of rupture	4000~7000 kg/㎠
	Modulus of rigidity	19000 kg/㎟
	Tensile strength	2600 kg/㎠
	Compressive strength	21000 kg/㎠
Thermal	Melting point	2050 ℃
	Specific heat	0.18 cal/g℃
	Thermal conductivity	0.09 cal/cm℃
	Coefficient of linear expansion	4.8~5.3×10^{-6} ㎝/℃
	Maximum operating temperature	1950 ℃
	Resistance to thermal shock	△T>1000 ℃
Optical	Index of refraction(sodium D-line)	1.760⊥C
	Dispersion n_F-n_C	0.11
	Constringency	72
	Optical transmission	>50% 5~6 ㎛
Electrical	Resistivity	10^{16} Ohm/cm
	Dielectric constant	8.5 ~ 10.5
	Dielectric loss tangent	< 0.0001

Table 1. Physical properties of Al_2O_3 Crystal

3. Electrode Design and Electric Field Simulation

across the capacitor, the charged capacitor can regain electron balance, that is, discharge its stored energy.If a potential difference is found between two points, an electric field exists as a result of the separation of unlike charges. The strength of the field will depend on the amount the charges have been separated. Capacitance is the concept of energy storage in an electric field and is restricted to the area, shape, and spacing of the capacitor plates and the property of the material separating them. When electrical current flows into a capacitor, a force is established between two parallel plates separated by a dielectric. This energy is stored and remains even after the input is removed. By connecting a conductor

The value of a parallel-plate capacitor can be found by the equation

$$C = \frac{\varepsilon_0 \varepsilon_r S}{d} = \frac{8.854 \times 10^{12} \varepsilon_r S}{d} \quad [F]$$

where, S d a ε_0 a ε_r are the dielectric constant of air and dielectric material. The dielectric constant of a material determines the electrostatic energy which may be stored in that material per unit volume for a given voltage. The value of the dielectric constant expresses the ratio of a capacitance in a vacuum to one using a given dielectric. The dielectric constant of air is 1, the reference unit employed for expressing the dielectric constant. As the dielectric constant is increased or decreased, the capacitance will increase or decrease, respectively. The dielectric constant of most materials is affected by both temperature and frequency, expect for Al_2O_3, Styrofoam, and Teflon, whose dielectric constants remain essentially constant[10].nd re respectively the surface area of the electrode and the thickness of the dielectric.and

The electrode is designed as a 3-terminal configuration, which means separate main and guard electrode, to obtain trustworthy measurement. To produce the precise 10 pF and 100 pF capacitors, the effect of the dielectric constant, the electrode dimension, the thickness of dielectric and the stray capacitance are studied.

As the dielectric constant of Al_2O_3 Crystal is fixed 3.82, the capacitance distributions of 10 pF and 100 pF capacitor are shown respectively with change in the diameter of electrode and the thickness of Al_2O_3 Crystal in Fig.3 and Fig.4. The needed design values must be confirmed by matched line, which existed between referenced and calculated values in Fig.3 and Fig.4. With foundation of the above methods, A12O3 Crystal capacitors of 10 pF and 100 pF using the 3-terminal configuration are designed as Fig.5 and Fig.6 respectively. But to reduce the loss in the electrode, the thickness of the electrode is limited by below 0.02 mm.

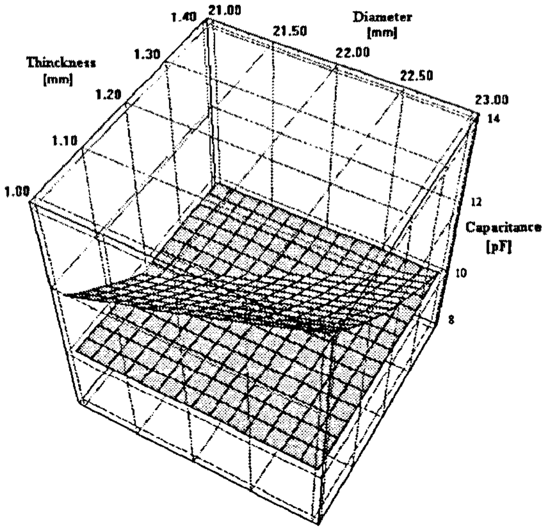


Fig. 3. The capacitance distribution of 10 pF capacitor

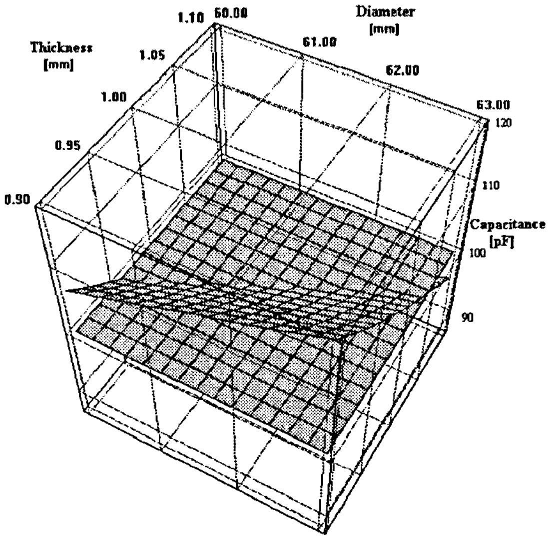


Fig. 4. The capacitance distribution of 100 pF capacitor

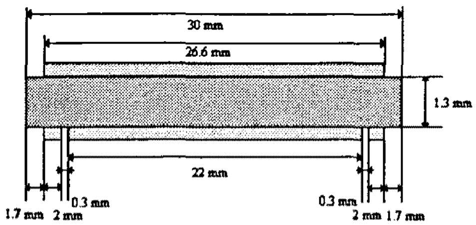


Fig. 5. Design values of 10 pF Capacitor

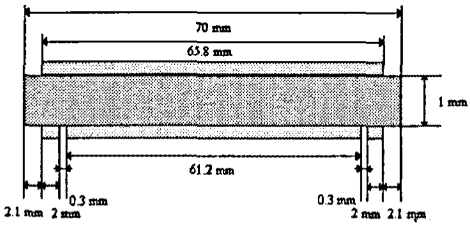


Fig. 6. Design values of 100pF Capacitor

Before fabricating the prototype, the design values of prototype are simulated in R-Z coordinate system in the electric field simulator. The simulated capacitor results are respectively 9.967 pF and 99.83 pF for 10 pF and 100 pF as 10 V is biased. Practically, must be considered the augmentation of the capacitance caused by the stray capacitance. So, the simulated values of needed capacitances must not be high than the required values of 10 pF and 100 pF.

In Fig. 7 and Fig. 8, the equipotential lines and the mesh-plot obtained using the simulated values, which is simulated by Maxwell 2D Electric Field Simulator, in the metal construction are shown respectively[11]. The equipotential lines indicate the stray capacitances, which are located at the gap between main and guard electrode and the edge of electrode in Fig.7. In the design, the stray capacitance must be considered as a summed capacitor.

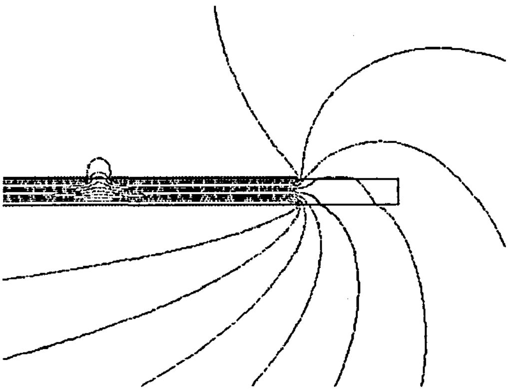


Fig. 7. Equipotential lines in electric field simulator

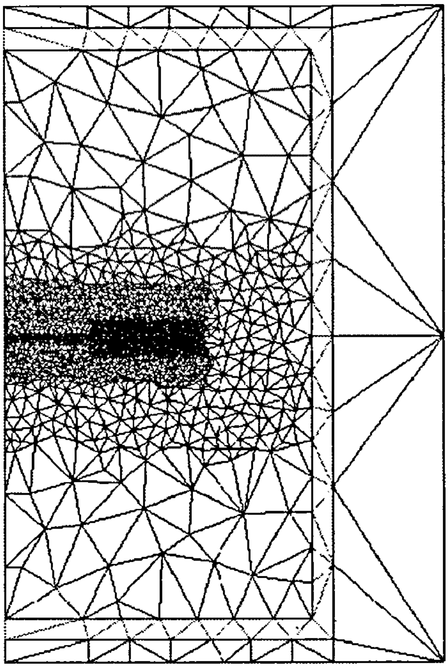
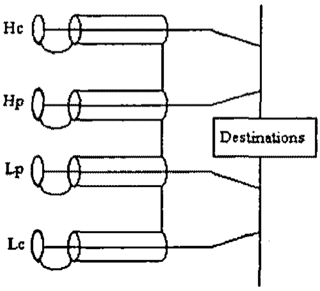


Fig. 8. Mesh-plot in metal construction

4. Experiment and Results

There are many measurement methods to choose from when measuring capacitance; each has advantages and disadvantages. The experimental measurement requirements and conditions must be considered, while choosing the most appropriate method, considering such factors as frequency coverage, measurement range, measurement accuracy, and ease operation. Considering these conditions, the Auto Balancing Bridge method is founded to be the best choice for Al_2O_3 Crystal capacitor. The Auto Balancing Bridge is generally equipped with four coaxial terminals(Hc, Hp, Lp and Lc). To interconnect the destination to the terminals, several connection methods can be used. Because each method has advantages and disadvantages, the most suitable method must be selected based on the required measurement accuracy[13,14]. In this paper, the Four Terminal Pair(4TP) configuration is used from metal construction, which contains the Al_2O_3 Crystal capacitor for protecting the outer electromagnetic effects, to measuring equipment. The 4TP configuration solves the mutual coupling problem because it used coaxial cable to isolate the voltage sensing cables from the signal current path in Fig.9 and Fig.10. Since the return current flows through the outer conductor of the coaxial cable, the magnetic flux generated be the inner conductor is canceled by that of the outer conductor.



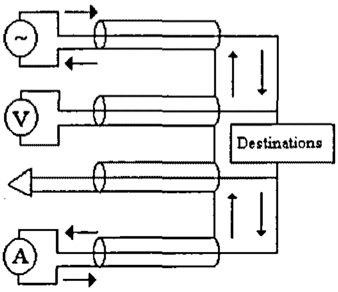


Fig. 9 Fig. 10

Four Terminal Pair Connection Diagram and Schematic Diagram

In the metal construction, the lead line from Al2O3 Crystal to output port is flexible, coated with Ag and covered by teflon with low loss tangent. The connection method to the measuring equipment being 4TP, the potential sensing cable is separated from the current signal path and the related errors from inductance, resistance and stray capacitance in BPO coaxial cable are reduced. Simultaneously, the capacitance and loss tangent are measured at 500~5000 Hz and 1000 Hz with HP 4194A Impedance/Gain-Phase Analyzer. The measured values of the capacitance of 10 pF and 100 pF Al_2O_3 Crystal capacitor are shown in Table 2. Accurate values are obtained by clipping the main electrode. In this process, capacitance is reduced but loss tangent still remains constant.

Table 2 refers to experimental results from the reforming of capacitance. In Fig.11 and Fig.12 respectively, show the capacitance values of Al_2O_3 Crystal for 10 pF are measured at 1000 Hz and from 100 to 5000 Hz. Figure11 indicate that the capacitance and loss tangent at 1000 Hz are almost constant. The small distortion at the low frequency range is attributed to the effect of the silver electrode in Fig.12. Approximately constant capacitance and loss tangent existed in most of the standard measurement range, which is above of the 900 Hz. With the same configuration, the measured values of capacitances of Al_2O_3 Crystal for 100 pF are shown in Fig.13 and Fig.14. But over most of the ranges, the results are constant unlike in the Fig.11 and Fig.12. The fact that Al_2O_3 Crystal has good stable dielectric properties over a variable frequency range is confirmed by Fig. 11,12,13, and 14.

Table 2. The results of 10 pF and 100 pF Capacitor

	10 pF		100 pF	
	C [pF]	tan δ	C [pF]	tan δ
1	11.5	3.6751 × 10^{-4}	101.27	3.6521 × 10^{-4}
2	11.3	3.6603 × 10^{-4}	100.98	3.6587 × 10^{-4}
3	10.9	3.6684 × 10^{-4}	100.12	3.6524 × 10^{-4}
4	10.05	3.6650 × 10^{-4}	100.004	3.6555 × 10^{-4}

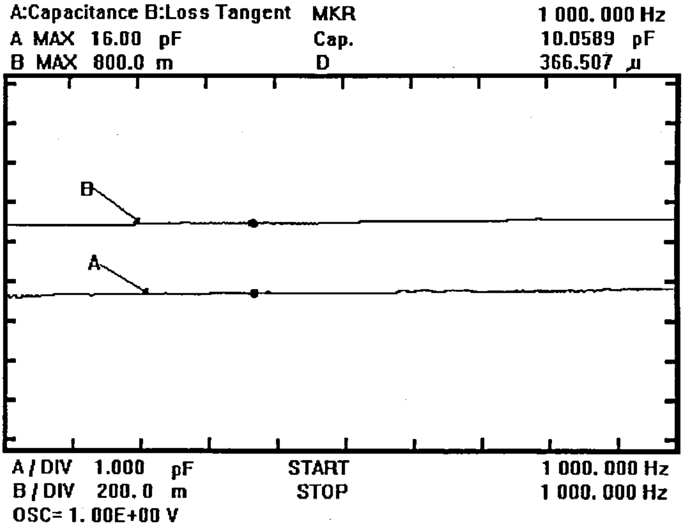


Fig.11. The capacitance and dielectric loss tangent of 10 pF capacitor at 1000 Hz

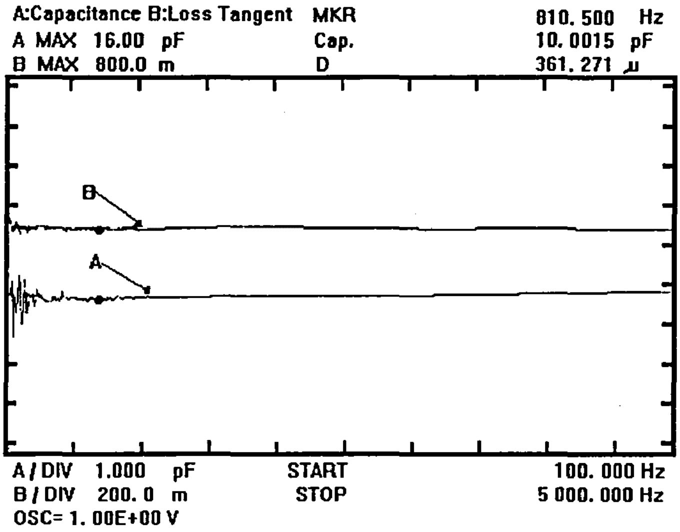


Fig.12. The capacitance and dielectric loss tangent of 10 pF capacitor at 100~5000 Hz

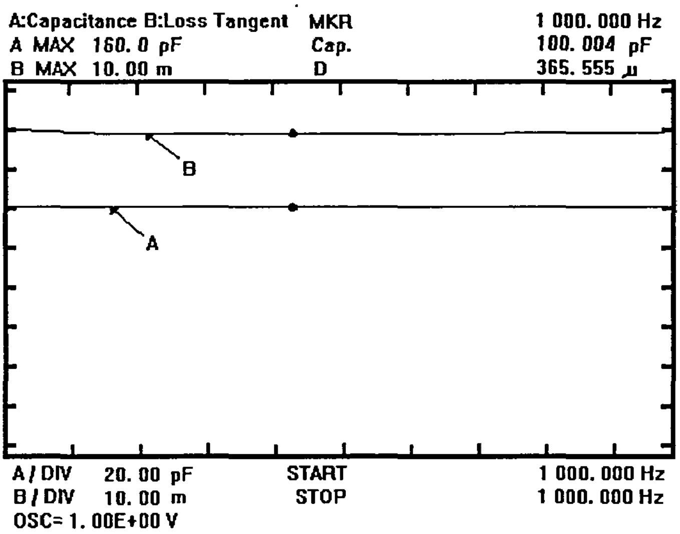


Fig.13. The capacitance and dielectric loss tangent of 100 pF capacitor at 1000 Hz

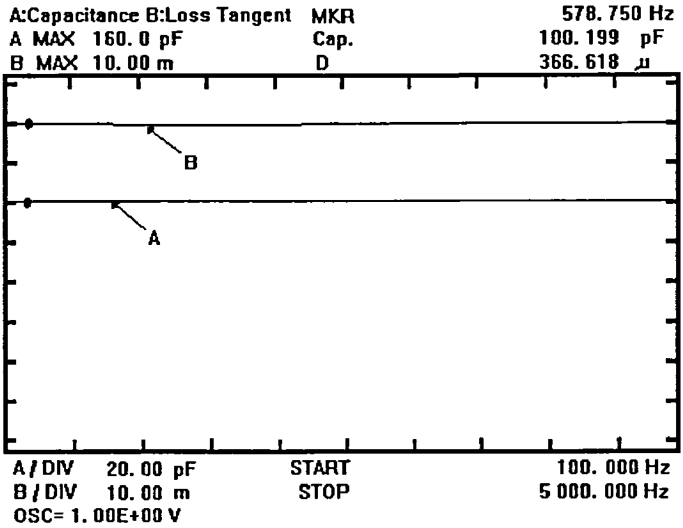

A:Capacitance B:Loss Tangent MKR 578.750 Hz
A MAX 160.0 pF Cap. 100.199 pF
B MAX 10.00 m D 366.618 µ
A/DIV 20.00 pF START 100.000 Hz
B/DIV 10.00 m STOP 5 000.000 Hz
OSC= 1.00E+00 V

Fig.14. The capacitance and dielectric loss tangent of 100 pF capacitor at 100~5000 Hz

5. Conclusions

In this paper, the development of standard capacitors of 10 pF and 100 pF for the establishment of the dielectric loss tangent using Al_2O_3 Crystal with low dielectric loss tangent presented and their dielectric loss tangents were measured with precision.

1. Al_2O_3 Crystal is difficult to process but it gives lower dielectric loss tangent than general dielectric materials and the dielectric loss tangent value is stable to the order of 10-4 over a frequency range.

2. In the electrode design on Al_2O_3 Crystal, Maxwell 2D electric field simulator is used to determine the most suitable electrode configuration.

3. 4-terminal pair connection method is used in the cabling configuration to reduce the errors related with inductance, resistance and stray capacitance in BPO coaxial cable.

The above results show that stable dielectric loss tangents of 3.6650×10-4 and 3.6555×10-4 could be obtained for 10.05 pF and 100.004 pF capacitors respectively. So, Al_2O_3 Crystal standard capacitors can be used for the measurement and evaluation of dielectric material properties and for important information about choice of standard capacitor.

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