## Advanced Liquid Crystals with Low Loss Tangent and Fast Response for Intelligent Reflecting Surface Antennas

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**Abstract** We are focusing our research attention on the improvement of performance for the intelligent reflecting surfaces (IRS) using liquid crystals. By realizing characterization of dielectric properties in liquid crystals using coaxial line in a wide range of frequency from 1 to 50 GHz, it is revealed that the 3-ring or 4-ring compounds possessing a high nematic-isotropic transition temperature exhibit the effects of reduction for loss tangent. For improving response time and loss tangent of IRS antenna using liquid crystals, low loss tangent reaching closely to 0.01 at frequency over 40 GHz band and shortening decay time for the response in almost half were achieved simultaneously by introducing para-terphenyl liquid crystal compounds without polar groups.

Keywords: Loss tangent, Relative permittivity, Frequency dependence, Intelligent reflecting surfaces, Gigahertz band.

## 1. Introduction

Liquid crystals have been paid attention to an attractive material being a controllable propagation even for gigahertz wave or terahertz wave by variation of permittivity to be derived from properties of anisotropy for permittivity. Components using liquid crystals such as phase shifter and antennas in telecommunications are extensively investigating from fundamental research to their applications  $^{1)}$ .

We have been investigating the intelligent reflecting surfaces (IRS) using liquid crystals possessing controllable reflective direction of gigahertz waves by adjusting phase change at each antenna element to be arrayed on the glass substrate  $^{2/3)}$ .

For research of liquid crystals in the telecommunications, understanding their behavior of dielectric anisotropy associated with permittivity and loss tangent in the range of radio frequencies (RF) is

essential maters to progress the properties of the components. Widespread information displays using liquid crystals employ electro-optical properties to be depended on dielectric anisotropy on tens Hz of low frequency and optical refractive index anisotropy. On the other hand, the devices in telecommunications utilize the phase change at a RF band, where the phase depends on reorientation of liquid crystals to be occurred with an applied voltage at a low frequency range <sup>4</sup>).

Liquid crystals as dielectric layer using to the IRS antenna are sandwiched between the two plates consisting of the top substrate arranging antenna elements and the bottom of a ground electrode substrate. This structure is similar to Liquid crystal displays (LCDs) whose liquid crystals are sandwiched between the pixel electrodes substrate and the ground electrode. The difference against LCDs is that the reflective phase on each antenna elements on the array in gigahertz region is controlled by a reorientation of rod-like shaped liquid crystalline molecules occurred with applying low- frequency voltage to each antenna element. In addition, the shape of patterned electrode of antenna elements to be formed at the top and the variation of permittivity as dielectric layer at the middle determines resonance frequency in gigahertz region against incident radio waves <sup>5)</sup>.

For altering reflection direction of incident waves by the antenna elements arranged on the front substrate, attaining a large continuous phase change by the LC

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dielectric layer is an essential function of the IRS antenna. Our experimental results of the antenna has been demonstrated that the IRS antenna requires a thick dielectric layer more than 50  $\mu$ m to attain a sufficient phase change by optimal design of pattern in electrode of antenna elements. Such an increasing the thickness of layer causes an insufficient speed for altering reflective direction due to an increase of switching time derived from an inherent property of LCs. In addition, an increase of loss tangent in LC layer with a thick LC layer leads to a decrease of intensity on the reflection.

Since such performances of antenna closely relate to the physical properties of LCs, improvement of properties for LCs in the gigahertz region plays an important role to progress the performance of the IRS antenna.

In this paper, the characterization for liquid crystals in the range of gigahertz, and the physical properties in liquid crystals to be suitable for IRS antenna are described.

## 2. Experiment

The method for characterization of liquid crystals in the range of RF is incompatible with the method for that of dielectric anisotropy in the range of low frequencies less than ten of kHz at least. The reason is that the wavelength to be shortened than the line length of the device under test (DUT) arises various propagations of electromagnetic waves such as multiple reflected waves at boundaries between media and generation of standing waves. To avoid occurrence of such complex electric field causing errors of measurements, the method using transverse electromagnetic (TEM) transmission lines such as coaxial line, which are designed for propagating a single electromagnetic mode along the transmission line, is effective methods.

Figure 1 shows the block diagram of the device for the measurement in the range of gigahertz band. The setup is composed of two functions. One is for measurements of propagation in RF signals, and the other is for applying a low frequency bias voltage to the DUT. The equipment for RF signal is composed of a vector network analyzer (VNA) P5008A manufactured by Keysight, a coaxial line for the DUT to use liquid crystals as dielectric layer, pair of bias tees (Bias T) to prevent a low frequency bias voltage to be intruded into ports of VNA. The other equipment is composed of a function generator for the low frequency, and DC amplifier. These setups allow us to measure S-parameters associated with changes of waves within a coaxial line in



Fig. 1 Equipment to measure S-parameter and wiring connection to apply a low frequency voltage to liquid crystals as dielectric layer in DUT.

the range of frequency from 100 kHz to 50 GHz. The Sparameters representing the scattering matrix indicate the relation between the forward waves and the backward waves on the two-ports to be equipped for VNA. The matrix elements  $S_{11}$ ,  $S_{12}$ ,  $S_{21}$ , and  $S_{22}$  are referred to as the scattering parameters or the Sparameters. The parameters for  $S_{11}$  and  $S_{22}$  have the meaning of reflection coefficients, and  $S_{21}$  and  $S_{12}$  have the meaning of transmission coefficients. In the measurements, the transmitted waves through DUT are detected as  $S_{21}$  with VNA. The relative permittivity and loss tangent for LCs are extracted from the measured  $S_{21}$  with the method of extraction described in the following paragraphs.

The coaxial line filled with LCs shown in Figure 1 features that a low frequency voltage passing through Bias T from the function generator is applied to LCs in the coaxial line without obstructing travelling RF signal. By utilizing structure of coaxial line consisting of cylindrical inner conductors and outer conductors separated by a spacer of LCs, it is possible to measure anisotropy in the relative permittivity in RF due to reorientation of LCs by applying a low frequency AC voltage in the range from 0 to 50 Vrms between the inner conductors and the outer conductors to LCs.

Figure 2 shows the structure of DUT coaxial line that is able to replace a part of polytetrafluoroethylene (PTFE) located between the inner and the outer conductors with liquid crystals. When comparing the normal coaxial line shown in Fig.2 (a) with the customized coaxial line to build LC filling section shown in Fig. 2 (b), it is identified that the center conductor has appeared at the portion removing the dielectric layer using PTFE and the outer conductor from the normal coaxial line. This opening



Fig.2 Experimental setup of coaxial line. (a) Coaxial line using PTFE for reference, (b) DUT coaxial line using unknown liquid crystals, (c) Cross sectional view for DUT, (d) Overview of complete DUT.

space is used for filling with LCs and is covered with the metal attachment possessing the function of the container and the outer conductor as shown in Fig. 2(c). The metal attachment to form the DUT coaxial line as shown in Fig. 2(c) holds steady to align the inner conductor of opening space shown in Fig. 2(b) with semi-cylindrical groove on the surface of metal attachment. The DUT on completion shown in Fig. 2(d) can be assembled by overlapping the groove surface of two pieces in the metal attachment to the coaxial line for the DUT shown in Fig. 2 (b) so that a cylindrical space is formed.

For the rise time in switching response in the real IRS antenna, occurring a continuous change of capacitance at the transition of alignment from a parallel to a vertical by the applied voltage of bursting square wave at 1 kHz was measured with the elapsed time. The apparatus for measurement was Keysight LCR meter E4980AL, and the change of capacitance was acquired with a 70 msec of sampling rate. For the decay time, the change of capacitance occurred at the transition of alignment from the vertical to the parallel was measured.

# 3. Principle of Characterization for LCs in Gigahertz

The fundamental method of measurement using coaxial line concerning relative permittivity and loss tangent is discussed.

Frequency dependence of magnitude of S21 shown in Fig.3 represents the magnitude of transmitted wave through coaxial line for the reference using PTFE and for the DUT filling with LCs, respectively. There is tendency that the magnitude decreases as frequency increases in both coaxial lines. The DUT line using LCs as dielectric layer exhibits a large attenuation on



Fig.3 Example of frequency dependence of magnitude of  $S_{21}$  for each DUT line and PTFE line.

magnitude as a function of frequency in comparison with the PTFE coaxial line. Such a large attenuation by DUT coaxial line suggests that an insertion loss caused by loss tangent in LCs is larger than that of PTFE line possessing low loss tangent ranging from 0.002 to 0.005 in gigahertz band.

This attenuation for magnitude of S21 is evaluated by the attenuation constant  $\alpha$  given by equation (1).

$$\alpha = \frac{|S_{21}|_{dB} \ln 10}{-20 z} \qquad [\text{Nepers/m}] \tag{1}$$

where, z denotes the line length of the coaxial line.  $|S_{21}|_{dB}$  indicates the magnitude of  $S_{21}$  in dB.  $\alpha$  has units of Nepers/m.

Fig. 4 shows frequency dependence of phase in  $S_{21}$  with DUT coaxial line and reference coaxial line using PTFE, respectively.

The difference for the variation of phase between the DUT and the reference coaxial line shown in Figure 4 is caused by a large relative permittivity of LCs used to DUT in comparison with that of the reference coaxial line using PTFE. As the phase is denoted by the equation (2), the relative permittivity  $\varepsilon_r$  is determined by using equation (2), where  $\mu_0$  denotes the vacuum permeability, and  $\varepsilon_0$  denotes



Fig.4 Example of frequency dependence of phase of  $S_{21}$  for DUT line and PTFE line.

the vacuum permittivity.

$$\phi = 2\pi f z \sqrt{\varepsilon_r \varepsilon_o \mu_o} \tag{2}$$

Phase constant  $\beta$  represents the variation of phase in travelling wave per unit length along the coaxial line. By substituting the  $\varepsilon_r$  determined by equation (2) into equation (3), the phase constant  $\beta$  is obtained.

$$\beta \approx k_0 \sqrt{\mu_0 \varepsilon_r} \tag{3}$$

The complex permittivity  $\varepsilon_c$  is given by equation (4). The real part and the imaginary part in the  $\varepsilon_c$  given by equation (4) are related to respective equation (5) and (6), where  $\omega$  is the angular frequency, and  $c_o$  is the speed of light in vacuum.

$$\varepsilon_c = \varepsilon_r' - j\varepsilon_r'' \tag{4}$$

$$\varepsilon_r' = -\frac{(\alpha^2 - \beta^2)c_0^2}{\omega^2} \tag{5}$$

$$\varepsilon_r^{\prime\prime} = \frac{j(2\alpha\beta)c_0^2}{\omega^2} \tag{6}$$

By substituting  $\alpha$  given by equation (1) and  $\beta$  given by equation (3) into equation (5) representing the real part in  $\varepsilon_c$  and equation (6) representing the imaginary part in  $\varepsilon_c$ , the complex permittivity  $\varepsilon_c$  is obtained. The relative permittivity  $\varepsilon_r$  is determined by the real part in  $\varepsilon_c$ .

The loss tangent of tan  $\delta$  is obtained from the ratio of  $\varepsilon_r$ " to  $\varepsilon_r$ ' in the complex permittivity.

## 4. Results and Discussion

To adapt LCs for the IRS antenna, the improvement of physical properties for LCs in the low frequency and the gigahertz regions are important issues.

In this section, the dielectric constant and the loss tangent in a RF are discussed. The switching response driven by AC voltage at a low frequency is also presented.

## 4.1 Frequency Dependence of permittivity for LCs in Gigahertz Band

The frequency dependence of relative permittivity  $\varepsilon_r'$ , i.e., the real part of complex permittivity for LCs determined from S<sub>21</sub> of S-parameters is shown in Figure 5. The comparison of permittivity with two different liquid crystal mixtures is shown in Figure 5. E7 in Figure 5 is well-known liquid crystals. TD1020 is developed so that a decay time in switching is lowered even in a thick cell gap at 50µm. The classification of dielectric anisotropy is denoted inside the brackets in Figure 5.

As the orientation of LCs in the DUT is varied with reorientation from the random orientation at no applied voltage to the homeotropic orientation with applied voltage at 50 Vrms, the relative permittivity  $\varepsilon_r//$  in a homeotropic alignment is obtained from the



**Fig.5** Frequency characteristics of relative permittivity  $\varepsilon_r$ ' for dielectric anisotropy. (//) denotes  $\varepsilon_r$ ' in parallel component of respective LCs for TD1020 and E7, ( $\perp$ ) denotes ( $\Xi$ µr'in perpendicular component for respective those of LCs.

measurement of  $S_{21}$  which is saturation state at applying 50 Vrms to the DUT coaxial line. In the case without applying any voltage to the DUT, a random orientation occurs because of no coat of the homogeneous alignment layer on the surface of the inner and the outer conductor contacting LCs in the DUT. The relative permittivity  $\varepsilon_{avr}$  in a random orientation of LCs is obtained from the measurement of  $S_{21}$  at applying 0 Vrms to the DUT coaxial line. The perpendicular component  $\varepsilon_r \perp$  in dielectric anisotropy is expressed as equation (7) including the term for  $\varepsilon_r //$  and  $\varepsilon_{avr}^{-6}$ .

$$\varepsilon_r \perp = \frac{3\varepsilon_{avr} - \varepsilon_r \|}{2} \tag{7}$$

The  $\varepsilon_r'$  of PTFE for the reference of coaxial line is almost independent of the frequency in the range from 1 GHz to 50 GHz, whereas the  $\varepsilon_r'$  of TD1020 and E7 decreases monotonously as frequency increases. Moreover, the different variation based on the dielectric anisotropy of the relative permittivity for TD1020 and E7 is observed in Figure 5.

The frequency dependence of loss tangent, i.e., the ratio of  $\varepsilon_r$ " to  $\varepsilon_r$  in complex permittivity is shown in Figure 6.

Frequency dependence of the loss tangent decreasing gradually toward higher frequency is observed. In addition, the loss tangent becomes a minimum in the region of 40 GHz.

It is suggested that these frequency dependence are derived from a dipole relaxation occurred in the lower frequency band less than 1 GHz. This is because it is presumed that the influence of dipole relaxation



**Fig.6** Frequency dependence of loss tangent  $\tan \delta$ . (//) denotes  $\varepsilon_r$ ' in parallel component of respective LCs for TD1020 and E7, ( $\perp$ ) denotes  $\varepsilon_r$ ' in perpendicular component of respective LCs for TD1020 and E7.

decreases gradually as frequency increases.

As the result of the measurements, it is revealed that TD1020 exhibits excellent properties for IRS antenna with low loss tangent and large dielectric anisotropy, when compared with E7.

## 4.2 Influence of Nematic-Isotropic Transition Temperature on Loss tangent

Factor for reduction of loss tangent was considered to improve the performance of antenna.

When we take notice of that loss tangent increases with increasing temperature <sup>7</sup>, it is speculated that a thermal vibration of liquid crystalline molecule influences on a loss tangent.

When assuming that liquid crystal state is exhibited by the competition between molecular interactions and thermal vibration effects, it is considered that the nematicisotropic transition temperature (Tni) becomes one of indicator representing the strength of molecular interaction.

In the case of that the strength of thermal vibration is beyond the strength of molecular interactions in liquid crystals, an isotropic state appears at the side of a high temperature in nematic temperature range. In the case of that the strength of thermal vibration is below the strength of molecular interactions in liquid crystals, a crystal state appears at the side of a low temperature in nematic temperature range. In the case of the strong molecular interactions, a higher Tni is required in order for the strength of thermal vibration to exceed the strength of molecular interactions. In other words, the crystallization temperature increases when the effect of molecular interaction is dominated against the effect of thermal vibration. From this point of view, comparing to connections with Tni and molecular structure for the single liquid crystal compound, it is speculated that 3 or 4-ring liquid crystal compounds with a high Tni exhibits strong molecular interactions. On the other hands, 2-ring liquid crystal compounds having a low Tni exhibit weak molecular interactions. In other words, 3 or 4-ring liquid crystals possessing strong molecular interactions are expected to have the effect of reducing loss tangent.

For probing the effectiveness of reducing loss tangent in 3 or 4 ring liquid crystals, 3 types of the mixture adding the single compound exhibiting high Tni with amount ranging from 5 to 20% into 65CB basic mixture consisted of 2-ring liquid crystal is used.

Mixture 65CB is composed of 4-Cyano-4'pentylbiphenyl (5CB, Tni: 34.4°C) and 4-Cyano-4'hexylbiphenyl (6CB, Tni:28.6°C) with the ratio of 1 to 1. Mixture1 was that 5% of 4, 4'-Bis(trans-4propylcyclohexyl) biphenyl (HPPH, Tni: 280°C) as nonpolar compound was added to 65CB. Mixture2 was that 4-Ethyl-4'-(trans-4-propylcyclohexyl) biphenyl (HPP, Tni: 169°C) as non-polar compound was added to 65CB with the ratio of 1 to 9. Mixture 3 was adding 4-Cyano-4''-pentyl-p-terphenyl (5CT, Tni: 240°C) as polar compound into 65CB with the ratio of 2 to 8.

Frequency dependence of loss tangent shown in Figure 7 exhibits the different curves for 65CB of the basic 2-ring compounds mixture and for 3 types of mixtures containing separately the single compound having Tni more than 169 °C.

It is revealed that effects reducing loss tangent are observed by the addition of liquid crystal compounds having high Tni against loss tangent of 65CB in 2 ring system.

In this fundamental examination using the high Tni compounds, it is confirmed that the loss tangent is reduced by introducing a high Tni liquid crystal compound, whereas using 2-ring liquid crystal tends to increase the loss tangent.

If it were possible to increase amounts of the high Tni compounds, the effect reducing loss tangent would be significant, however amounts of the compounds are limited due to a high crystallization temperature of the high Tni liquid crystal compounds.

From these points of view for the reducing loss tangent, amounts of 3 ring system are increased as compounds for TD1020 used to the IRS antenna in order to balance between the suppressing increase of crystallization temperature and the reducing loss tangent.



Fig.7 Comparison with 3 types of mixtures adding respective single compound having high Tni into the basic mixture of 65CB consisting of 2-ring compounds in frequency dependence of loss tangent tan  $\delta$ .

## 4.3 Physical Properties for LCs in Low Frequency

The subject matter of liquid crystals for IRS antenna is to become extremely slow phase shifting speed on decay time in switching because of a necessary of thick LC layer over 50  $\mu$ m ensuring sufficient phase variation in IRS antenna.

As IRS antenna requires an adjustment of relative permittivity in the gigahertz band with external voltage at the low frequency, physical properties of liquid crystals such as viscosity, and dielectric anisotropy at the low frequency are significant properties to determine the performance related to a beam steering of the antenna<sup>8) 9)</sup>.

Well-known liquid crystal mixture E7 of composition, which compose of 2-ring and 3-ring compounds having polar groups being cyano groups, results in an increase of rotational viscosity providing for the slow decay time in the switching. To decrease the viscosity, concept of design in mixtures should be changed. The amounts of polar compounds should be reduced as possible to minimize an increase of viscosity. From a point of view for reduction of loss tangent with explanation above, it is necessary to maximize amount of non-polar compounds having 3-ring to satisfy a low loss tangent, instead of adding low viscosity of 2 ring compounds that cause an increase of loss tangent. By this design for liquid crystal compositions, the lowering rotational viscosity without the increase of loss tangent is attained.

Physical properties in 45 GHz and 1kHz for both E7 and TD1020 are listed in Table 1. As the lowering viscosity and loss tangent are priority issues for the IRS antenna, TD1020 of liquid crystals is selected.

3-ring of p-terphenyl compounds without a cyano group was used to achieve the low viscosity and a reduction of loss tangent in TD1020, whereas E7 using 8 wt% of 4-Cyano-4"-pentyl-p-terphenyl (5CT) compounds with a high Tni and 92 wt% of n-alkylcyanobiphenyl compounds with a low Tni causes the increasing both rotational viscosity and loss tangent. From a point of view with respect to the difference for amounts of compounds and for polarity of compounds between TD1020 and E7, it is revealed that 3-ring non-polar compounds of p-terphenyl used for TD1020 exhibit the effectiveness of reduction for the rotational viscosity and the loss tangent.

Moreover, by exceeding 70 wt% of amounts in the pterphenyl compounds without a cyano group, a slight dielectric anisotropy of 0.61 for TD1020 arises in comparison with the dielectric anisotropy for E7 being 12.24 at 1 kHz. As the rise time for TD1020 with 50 µm of cell gap exhibits a 1 second or less at the applied voltage more than 12 Vrms, an influence of the rise time on a total switching time comprising the rise time and the decay time is a negligible value in the comparison with the decay time in a few seconds. On the other hand, in order to improve the decay time, the reduction of rotational viscosity  $\gamma$ 1 from 252 [mPa s] for E7 to 107

Table 1 Physical properties of LCs in 45 GHz and 1 kHz.

| LC Mixture | 45GHz                    |                      |                           |                       |      | 1kHz                      |                       |       | $V_{th}$ | $\gamma_1$ | Δn   | Tni   |
|------------|--------------------------|----------------------|---------------------------|-----------------------|------|---------------------------|-----------------------|-------|----------|------------|------|-------|
|            | $	an \delta_{\parallel}$ | $	an \delta_{\perp}$ | $\varepsilon_{\parallel}$ | $\varepsilon_{\perp}$ | Δε   | $\varepsilon_{\parallel}$ | $\varepsilon_{\perp}$ | Δε    | [Vrms]   | [mPa s]    |      | [C°]  |
| E7         | 0.0151                   | 0.0364               | 3.18                      | 2.62                  | 0.56 | 14.4                      | 4.2                   | 12.24 | 0.82     | 252        | 0.21 | 58    |
| TD1020     | 0.0095                   | 0.0195               | 3.25                      | 2.48                  | 0.77 | 3.78                      | 3.11                  | 0.61  | 5        | 107        | 0.25 | 108.1 |

[mPa s] for TD1020 by the decreasing amounts of polar compounds leads to an advancement of the decay time from 22 seconds at 100  $\mu$ m of cell gap for E7 to 10 seconds for TD1020 at the same cell gap. In addition, the decay time at 2.5 seconds is improved with a 50  $\mu$ m of cell gap in the IRS antenna, which is confirmed to ensure a sufficient phase change to operate a beam steering.

By increasing amount of non-polar p-terphenyl compounds having 3-ring without cyano groups on TD1020, the low loss tangent at 0.0095 for TD1020 in the region of 45GHz as shown in Table 1 is attained in comparison with that for E7. It is demonstrated that reducing viscosity and loss tangent are attained simultaneously.

## **5.** Conclusion

It is important to develop the method for evaluation of LCs in gigahertz region.

The examined method is characterized with that frequency dependence of dielectric constant and loss tangent is determined directly from the difference of propagation in a travelling wave for the coaxial line of DUT using liquid crystal and for the coaxial line using PTFE.

Consequently, characterization of both loss tangent and relative permittivity for liquid crystals in a wide range of frequency from 1 to 50 GHz of have been achieved with the coaxial line method.

In the frequency band over 40 GHz, it is clarified that tendency of minimizing loss tangent is suitabel for the properties in IRS antenna operating in the region of 40 GHz.

In relation between loss tangent and liquid crystal compounds, it has revealed that 3-ring or 4-ring liquid crystal compounds exhibiting a high nematic-isotropic transition temperature tend to be a low loss tangent. Furthermore, 2-ring liquid crystal compounds exhibiting a low nematic-isotropic transition temperature tend to be the contrary. It is found out that decrease of amounts of 2-ring compounds having a high loss tangent contained in liquid crystal mixtures is necessary for attaining low loss tangent of liquid crystals. From characterization for composition on the liquid crystal mixtures in the low frequency and gigahertz band, it is found out that liquid crystal mixtures with the maximizing amount of non-polar 3-ring compounds having high Tni are useful for method that improve simultaneously the rotational viscosity and the loss tangent.

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