# Time-Domain Characterization of Nematic Liquid Crystals Using Additive Manufacturing Microstrip Lines

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*Abstract*— This article presents a method for effectively characterizing the dielectric permittivity of nematic liquid crystals (LCs) across a broad frequency range. These materials show significant potential for reconfigurable devices operating in microwave and millimeter-wave frequencies. To achieve this goal, an additive manufacturing technique is used to create a microstrip line that can be filled with liquid that acts as its substrate. The LC is then biased to modulate its permittivity. After manufacturing, a timegating approach is used to extract the permittivity, eliminating the need for in-fixture calibration, such as thru–reflect–line (TRL). Finally, the approach is validated through simulations and experimental results, which closely align with those reported using other methods in the bibliography.

*Index Terms*— Broadband measurements, liquid crystals (LCs), permittivity characterization, reconfigurability, time gating.

### I. INTRODUCTION

THE rapid development of wireless communication services requires reconfiguring the radiation patterns of HE rapid development of wireless communication serantenna systems efficiently and cost-effectively to adapt themselves to the changing environment. Some examples of

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<span id="page-0-1"></span><span id="page-0-0"></span>applications of tunable antennas are 5G/6G services or automotive radars. Traditionally, fixed-frequency beam steering has been achieved either mechanically, which results in bulky structures, or by implementing phased arrays, which lead to complex and expensive feeding networks. An alternative technology to reconfigurable components, such as varactors or micromechanical actuators, is nematic liquid crystals (LCs) [\[1\]. Th](#page-6-0)ese tunable materials are potential candidates for reconfigurable antennas [\[2\], es](#page-6-1)pecially at high frequencies for which mechanical actuators and integrated devices suffer high transmission losses. In addition, tunable materials have the main advantage of reconfiguring the whole structure at once for modern directive planar antennas, such as reflect arrays [\[3\],](#page-6-2) [\[4\], le](#page-6-3)aky wave [\[5\], or](#page-6-4) metasurface-based antennas [\[6\]. Th](#page-6-5)ey have also been used for other reconfigurable devices, such as filters [\[7\]](#page-6-6) or phase shifters [\[8\].](#page-6-7)

<span id="page-0-8"></span><span id="page-0-7"></span><span id="page-0-6"></span><span id="page-0-5"></span><span id="page-0-4"></span><span id="page-0-3"></span><span id="page-0-2"></span>In their nematic phase, LCs are anisotropic materials with tunable electric permittivity through a biasing voltage [\[9\].](#page-6-8) In [\[10\], a](#page-6-9) dispersion analysis of anisotropic periodic structures applied to LCs was performed. Although LCs for optical applications have been studied extensively, their use is not so extended at microwave frequencies. The broadband characterization of its effective dielectric constant is required to enable the design of reconfigurable antennas using such a technology. In general, dielectric and transmission line characterization methods have been divided into two groups: resonant and broadband. Resonant characterization methods have been presented in the literature, using multilayer structures based on microstrip transmission lines [\[11\]](#page-6-10) or planar patch resonators [\[12\]](#page-6-11) among others, but they are inherently narrowband. Another multilayer structure for the extraction of permittivity as a function of the bias voltage is presented in [\[13\].](#page-6-12) In [\[14\], a](#page-6-13) broadband characterization method was proposed, which needs thru and line standards to carry out the calibration and, thus, requires fabricating and measuring additional structures.

<span id="page-0-13"></span><span id="page-0-12"></span><span id="page-0-11"></span><span id="page-0-10"></span><span id="page-0-9"></span>There has been research on the characterization of LCs in the time domain for many years [\[15\],](#page-6-14) [\[16\]. T](#page-6-15)ime-gating techniques have been traditionally used to avoid undesired effects, such as noise or reflections. In devices whose different section responses are not physically separable, the undesired section contributions can still be mostly removed

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<span id="page-1-2"></span>

Fig. 1. Not-to-scale diagram of (a) stack-up with the LC molecules polarized and (b) top view of the proposed covered microstrip line.

<span id="page-1-6"></span><span id="page-1-5"></span>in the time domain (see [\[17\]\),](#page-6-16) enabling dielectric materials characterization [\[18\]. T](#page-6-17)his contribution proposes a time-gating technique to characterize the relative permittivity of LCs inside microstrip lines fabricated using additive manufacturing. This approach allows the fabrication and measurement of a single prototype while enabling broadband characterization. The main advantage is that it does not require any in-fixture calibration of the analyzer  $[19]$ ,  $[20]$  or the use of more than one transmission line when using differential length characterization techniques [\[21\],](#page-6-20) [\[22\], w](#page-6-21)hich considerably facilitates its application due to the difficulty of manufacturing this type of structure.

<span id="page-1-8"></span>This article is organized as follows. Section [II](#page-1-0) describes the stack-up of the proposed structure. Section [III](#page-1-1) illustrates the proposed method based on time gating to extract the dielectric permittivity of the LC. Section [IV](#page-3-0) shows the validation through simulation and experimental results. Finally, the conclusions are drawn in Section [V.](#page-5-0)

## II. STRUCTURE DESCRIPTION

<span id="page-1-0"></span>The proposed microstrip transmission line stack-up is shown in Fig.  $1(a)$ . As seen, it consists of four layers. The first one from the bottom is made of copper and plays the role of ground plane. The second layer is a thin polyimide (kapton) sheet to ensure the correct LC molecules orientation at rest [\[1\]. Th](#page-6-0)e third one is the pool where the LC is contained, whereas the pool edges are made of high-impact polystyrene (HIPS) using additive manufacturing. Fig.  $1(b)$  shows a top view of the structure described. In the top layer is a strip milled from a  $350$ - $\mu$ m-thick copper sheet and an overlay of HIPS with a groove in which the strip fits and stays correctly suspended above the LC pool. In this layer, two perforations allow the liquid crystal pool to be filled using a syringe. All junctions between layers are performed using screws located outside the pool and nonconductive epoxy to prevent fluid leakages. Fig. [1](#page-1-2) details the structure's main dimensions.

<span id="page-1-9"></span>Regarding HIPS, it was proposed for microwave applications and characterized in  $[23]$ . It is a material for fused filament fabrication with excellent electromagnetic properties due to its low loss tangent and relative permittivity, close to 2.45. In addition, HIPS is durable and possesses good impact resistance together with the fact that remains stable when exposed to moisture. For the proposed experiment, it was printed out using the Ultimaker 3 printer, setting the infill to

<span id="page-1-3"></span>

<span id="page-1-7"></span>

Fig. 2. Fabricated prototype (a) as a side-by-side view of its layers and (b) fully assembled and sealed.

100%. The extrusion and bed temperatures were established to 240 °C and 90 °C, respectively, and the layer height was set to 60  $\mu$ m. The HIPS layers (including structural support), along with the rest of them used in the structure, are shown side by side in Fig.  $2(a)$  and the assembled and sealed prototype in Fig. [2\(b\).](#page-1-3)

## III. CHARACTERIZATION METHOD

## <span id="page-1-4"></span><span id="page-1-1"></span>*A. Time Gating*

The time-domain processing from frequency-domain measurements is used to remove the undesired contribution from the sections of the transitions between the ports and the pool [see Fig.  $1(b)$ ], thus moving the reference planes to the pool outer interfaces. First, the measured *S*-parameters are resampled to present  $2^n$  frequency points evenly spaced along the whole bandwidth, starting from dc. As the original measurements from a vector network analyzer will probably not include dc, a sensible assumption is to consider it a short circuit. So, an inverse fast Fourier transform (IFFT) is adequately applied to every *S*-parameter to obtain their timedomain responses. These are the convolution of the impulse responses with a sinc function due to the measured frequency

response truncating the actual band-unlimited one. Another type of window (Hamming, Blackman, and so on) can be applied before the IFFT to reduce the impulse response widening and, thus, improve its visualization. Furthermore, applying frequency-domain zero padding in the IFFT is recommended to increase time resolution. Then, if the zero-padding order  $n<sub>z</sub>$  represents the factor by which the number of frequency points is increased, the achieved time resolution will be  $dt = 1/(2n_z f_{\text{max}})$ , with  $f_{\text{max}}$  being the maximum measured frequency.

The time interval related to the LC section (when does it start,  $t_{\text{start}}^{(11)}$ , and end,  $t_{\text{stop}}^{(11)}$ ) can be derived from previous knowledge of the structure dimensions and the widened impulse response of the *S*11. This time response will be composed of several widened deltas, among which the  $t_{\text{start}}^{(11)}$  timestamp will correspond with the peak of one of the most relevant ones, and for which a fine time resolution is needed for its proper identification. In addition, as the structure shown in Fig. [1\(b\)](#page-1-2) is symmetric and its delay  $t_{\text{delay}}^{(21)}$  can be obtained as the main peak timestamp in the  $S_{21}$  time response,  $t_{stop}^{(11)}$  directly arises from  $t_{\text{start}}^{(11)}$ , once derived, as follows:

$$
t_{\text{stop}}^{(11)} = 2 \ t_{\text{delay}}^{(21)} - t_{\text{start}}^{(11)}.
$$
 (1)

The time-gate width can then be calculated as follows:

$$
t_{\text{width}} = \left(t_{\text{stop}}^{(11)} - t_{\text{start}}^{(11)}\right) / 2 \tag{2}
$$

which is divided by two to account for the round trips in the reflection signals, as shown in Fig.  $1(b)$ .

This way, the time-gating timestamps for the rest of the *S*-parameters can be geometrically derived from the port-1 timestamps. Transmission parameters time responses will consist of a main pulse (whose peak's timestamp determines the signal delay between both ports) followed by the contributions of several internal reflections. Using the *t*width value also for these parameters, the timestamps for the transmission from port 1 to port 2, for example, would be

$$
t_{\text{start}}^{(21)} = t_{\text{delay}}^{(21)} - t_{\text{width}}/2
$$
  
\n
$$
t_{\text{stop}}^{(21)} = t_{\text{delay}}^{(21)} + t_{\text{width}}/2
$$
 (3)

thus applying the time gate around the main pulse arrival. For reflection parameters, the corresponding timestamps can be geometrically derived from Fig.  $1(b)$ , provided that the time delay between port 1 and the port of interest is already known. For example, port-2 reflection timestamps would be calculated as follows:

<span id="page-2-6"></span>
$$
t_{\text{start}}^{(22)} = 2 \ t_{\text{delay}}^{(21)} - t_{\text{stop}}^{(11)}
$$
  
\n
$$
t_{\text{stop}}^{(22)} = 2 \ t_{\text{delay}}^{(21)} - t_{\text{start}}^{(11)}.
$$
\n(4)

Following the approach from [\[24\], a](#page-6-23) high-order Butterworth filter window is used as the time gate to prevent amplitude distortion within the interval of interest (from  $t_{start}$  to  $t_{stop}$ ) while significantly attenuating the rest of the response without introducing abrupt changes. The corresponding time window is multiplied by each impulse response, and the result is shifted toward the time origin to remove the undesired sections'

contribution to the equivalent transmission line length. The time shift will depend on the specific parameter

<span id="page-2-4"></span>
$$
t_{\text{shift}}^{(ii)} = t_{\text{start}}^{(ii)}
$$
  
\n
$$
t_{\text{shift}}^{(ji)} = t_{\text{delay}}^{(ji)} - t_{\text{width}}.
$$
  
\n(5)

Finally, the impulse responses are transformed back to the frequency domain by applying a fast Fourier transform (FFT), reverting zero padding and dividing by the used frequency window to remove the amplitude distortion, if needed. Thus, an approximation of the LC section's *S*-parameters is obtained.

#### <span id="page-2-5"></span>*B. Permittivity Extraction*

Considering that the processed *S*-parameters have a good impedance matching and represent a transmission line, then its effective permittivity can be estimated from the transmission parameter

<span id="page-2-1"></span>
$$
S_{21} = e^{-jkl} = e^{-\alpha l} e^{-j\beta l} = e^{-j\frac{2\pi f}{c_0}\sqrt{\varepsilon_{\text{eff}}l}}
$$
(6)

where *l* is the pool length [Fig. [1\(b\)\]](#page-1-2). To extract  $\varepsilon_{\text{eff}}$  from the complex exponential without facing problems related to the phase wrapping and phase reference, its value is reconstructed from both the magnitude and the unwrapped phase of  $S_{21}$  as follows:

$$
\sqrt{\varepsilon_{\text{eff}}} = \sqrt{\varepsilon' - j\varepsilon''} = x + jy
$$
\n(7)

$$
|S_{21}| = |e^{-\alpha t}| \Rightarrow y = \frac{c_0}{2\pi f l} \log |S_{21}| \tag{8}
$$

$$
\angle S_{21} = -\beta l \Rightarrow x + f \frac{\partial x}{\partial f} = -\frac{c_0}{2\pi l} \frac{\partial \text{unwrap}(\angle S_{21})}{\partial f} \tag{9}
$$

<span id="page-2-2"></span><span id="page-2-0"></span>
$$
\varepsilon_{\rm eff} = (x^2 - y^2) + j(2xy). \tag{10}
$$

The first-order differential equation from [\(9\)](#page-2-0) can be numerically solved. Thus, by inserting  $(8)$  and  $(9)$  into  $(10)$ , the LC-section effective relative permittivity is calculated. For this process, it is assumed that there is a linear  $S_{21}$  phase variation with frequency, which is the natural behavior in a transmission line.

However, this work aims to characterize the liquid crystal's nominal permittivity  $\varepsilon_r$ , not the whole stack of different materials. Hence, a multilayered microstrip model is considered. First, the HIPS overlay used to seal the LC container is taken into account using the model from [\[25\], w](#page-6-24)hich, although not frequency-dependent, allows the direct calculation of the microstrip substrate permittivity  $\varepsilon'_{r}$  from  $\varepsilon_{\text{eff}}$ . In addition, the substrate comprises both LC and Kapton. The latter is well known and used in microwave applications, having a relative permittivity  $\varepsilon_{r_k} = 3.5$ . The equivalent single-layer permittivity of these materials can be obtained using [\[26\]](#page-6-25)

<span id="page-2-8"></span><span id="page-2-7"></span><span id="page-2-3"></span>
$$
\varepsilon'_{r} = \frac{\varepsilon_{r_k} \varepsilon_r (h_k + h_{\rm LC})}{\varepsilon_{r_k} h_{\rm LC} + \varepsilon_r h_k}.
$$
 (11)

This model can be used considering the liquid crystal as a lossy dielectric, since it only considers the electromagnetic properties of the material  $(\varepsilon_r$  and tan  $\delta$ ) and not its mechanical characteristics, which also change with the bias voltage. Knowing the layers heights [see Fig.  $1(a)$ ], the LC permittivity  $\varepsilon_r$  can be retrieved from the previously calculated substrate permittivity  $\varepsilon'_{r}$  through [\(11\).](#page-2-3)

On the other hand, determining the loss tangent requires reliable information solely about the transmission magnitude of the pool section. Applying the time gate in the middle of an already brief time response induces distortion in the reflection response and results in the main transmission response peak being a composite of inseparable contributions from the entire prototype. Consequently, when time gating the transmission response around the main peak, only the contribution of secondary internal reflections occurring outside the time gate will be eliminated (thus conveniently smoothing the transmission parameter). Nevertheless, the magnitude of the gated peak remains identical to that of the original ungated response. Therefore, deriving a loss tangent from this parameter lacks physical validity, as it would encompass losses from undesired sections outside the pool, thereby distorting the actual poolsection losses.

## IV. VALIDATION

<span id="page-3-0"></span>In this section, both preliminary simulation tests and experiments using a manufactured prototype will be carried out using the GT7-29001 LC from Merck, which offers  $\{\varepsilon_{r_1} = 2.46,$ tan  $\delta_{\perp}$  = 0.0116} and { $\varepsilon_{r_{\parallel}}$  = 3.53, tan  $\delta_{\parallel}$  = 0.0064} at 19 GHz, according to available datasheet.

#### <span id="page-3-3"></span>*A. Simulation Test*

The structure is first simulated in ANSYS HFSS to verify the proper signal processing operation with ideal and controlled data. Three cases are simulated: with the pool filled with LC in ON- and OFF-states and air used as a reference. The whole pool, entirely filled with LC, is assumed to offer the permittivity  $\varepsilon_{r_{\perp}}$  in the electric field main direction for the OFF-state. In the ON-state, however, the LC will not be homogeneously polarized for two main reasons. First, under electric field biasing, the LC molecules orient differently along the LC height, with a behavior modeled by the differential equations governing the nematic LCs physics [\[27\],](#page-6-26) [\[28\]. T](#page-6-27)his effect translates into average LC molecules tilt angle  $\theta_m$ , concerning the OFF-state, that will be lower than 90°. Then, the LC permittivity tensor can be calculated in the chosen coordinates system and biasing field direction as follows:

$$
\overline{\overline{\varepsilon_r}} = \begin{pmatrix} \varepsilon_{\perp} + \Delta \varepsilon_r \cos^2 \theta_m & 0 & \Delta \varepsilon_r \cos \theta_m \sin \theta_m \\ 0 & \varepsilon_{\perp} & 0 \\ \Delta \varepsilon_r \cos \theta_m \sin \theta_m & 0 & \varepsilon_{\perp} + \Delta \varepsilon_r \sin^2 \theta_m \end{pmatrix}
$$
 (12)

where  $\Delta \varepsilon_r = \varepsilon_{r_{\parallel}} - \varepsilon_{r_{\perp}}$ . Nevertheless,  $\theta_m$  can be easily calculated through [\[2\], an](#page-6-1)d from which it can be concluded that for sufficiently high biasing voltages,  $\theta_m \approx 90^\circ$ , allowing this effect to be neglected in simulations.

The second cause for inhomogeneity comes from the bias voltage being applied to the microstrip itself, thus only fully polarizing the LC immediately below it and partially polarizing the LC beyond the microstrip width. Hence, as the microstrip width is not much greater than the pool height, the fringing fields' contribution to the effective permittivity should be considered  $[3]$ . Thus, the ON-state is simulated by stratifying the pool media, where the LC below the line presents a permittivity tensor given by [\(12\)](#page-3-1) with  $\theta_m = 90^\circ$ ,

<span id="page-3-2"></span>

Fig. 3. Original and time-gated simulated *S*-parameters for the ON and OFF LC states.

the LC alongside the microstrip (with a width equal to the microstrip width on each side) has  $\theta_m = 45^\circ$ , and the rest is assumed not polarized at all, just as schematically shown in Fig.  $1(a)$ . From simulations and bibliography, this scenario was chosen as a good estimation.

The *S*-parameters obtained from the simulations for the LC-filled pool cases are presented in Fig. [3](#page-3-2) with dotted lines, where a good matching and a linear transmission phase variation are appreciated, as expected. These are transformed to the time domain after applying a Hamming window to improve visualization, and the resulting  $S_{11}$  and  $S_{21}$  time responses are represented in Fig. [4](#page-4-0) with dotted lines. The first reflection peak, where the three responses begin to differ, indicates the timestamp when the pool section starts  $t_{\text{start}}^{(11)} = 133 \text{ ps}$ (represented with a black dashed vertical line). The structure delay for each case is obtained as the timestamp of the main transmission peak. Then, as explained in Section [III-A,](#page-1-4) due to the structural symmetry, the timestamps when the pool ends  $t_{\text{stop}}^{(11)}$  are automatically obtained for each case (represented with vertical dashed lines colored as their corresponding response), coinciding with the reflection peaks at the exit of the pool section, which is electrically longer as the filling substrate permittivity increases.

<span id="page-3-4"></span><span id="page-3-1"></span>Note that the 20-GHz simulated bandwidth would give a time resolution of  $dt = 25$  ps, too coarse for the proper determination of the time-gating timestamps in such a small device. Consequently, an IFFT zero-padding order of  $n<sub>z</sub>$  = 50 was used, thus giving a smaller step  $dt = 0.5$  ps. From tests with the studied configuration, the steps of  $dt$  in the  $t_{\text{start}}^{(11)}$ value produce variations less than  $\pm 0.02$  in the obtained pool section  $\varepsilon_{\text{eff}}$ .

The time responses are time gated using the obtained times-tamps, resulting in the solid lines in Fig. [4.](#page-4-0)  $t_{\text{start}}^{(11)}$  becomes the new time origin for the reflection response, the transmission one is shifted following [\(5\),](#page-2-4) and the responses are transformed back to the frequency domain, resulting in the time-gated *S*-parameters represented in Fig. [3](#page-3-2) with solid lines, from which the processing described in Section [III-B](#page-2-5) is carried out. The obtained permittivities are shown in Fig. [5,](#page-4-1) from the complete DUT to the extracted pool-media ones. In addition,

<span id="page-4-0"></span>

Fig. 4. Widened impulse responses of the simulated  $S_{11}$  (up) and  $S_{21}$  (down) before and after the time gating, and with their timestamps.

<span id="page-4-1"></span>

Fig. 5. Relative permittivities obtained from the processed simulations for different pool substrate scenarios.

the effective permittivity of the pool section alone has also been simulated, and it is in excellent agreement with the one obtained from the time-gated parameters, thus validating the time-gating approach.

The reference air-filled case gives a  $\varepsilon_r$  close to 1, thus confirming the proper selection of the time-gating timestamps. It can be noted that the OFF-state LC  $\varepsilon_r$  is practically equal to  $\varepsilon_{r_{\perp}}$ , while for the ON-state, the LC permittivity that the microstrip "perceives" is lower than  $\varepsilon_{r_{\parallel}}$  (which is the plot ordinate higher limit) due to the LC anisotropy and the stratified media configuration, which prevents the full polarization of the whole LC filling the pool. These results will serve as a reference for the real experiments.

The choice of the particular frequency window applied before the IFFT must take into account that, although higher time-domain sidelobe suppression could facilitate the main widened deltas recognition, they also significantly suppress higher frequencies, which degrades the permittivity extraction accuracy in that range. This effect is shown in Fig. [6](#page-4-2) for different applied frequency windows, from where it is clear that the majority of the bandwidth remains unaffected. After correct timestamp determination, the frequency window offering less distortion in the band edge should be used. Moreover,

<span id="page-4-2"></span>

Fig. 6. Comparison of simulation extracted  $\varepsilon_r$  for different applied frequency windows.

the time window itself mainly distorts the lowest frequencies, so the results are always shown from 1 GHz onward, except in Fig. [6](#page-4-2) for illustrating the explained artifacts. This should not be an issue for reconfigurable devices designed for microwave frequencies.

#### *B. Experimental Results*

The fabricated prototype is shown in Fig. [2\(b\).](#page-1-3) Southwest 2.4-mm end launch connectors are used, whose contribution will also be removed through time gating. Port 1 of the prototype is connected to a bias T, where the 1-kHz sinusoidal bias signal is input, and port 2 is connected to a dc block. The measurements are carried out with a Keysight PNA-X N5247A network analyzer configured with a frequency sweep of 6401 points from 10 MHz to 20 GHz. The bandwidth of the intermediate frequency (IF) bandpass filter is set at 10 kHz, whereas the port power is −5 dBm.

As in Section [IV-A,](#page-3-3) the *S*-parameters are measured for the same three pool-media cases (air, LC OFF, and LC ON). In addition, for the LC ON-state, two biasing voltages are tested, 20  $V_{pp}$  and 100  $V_{pp}$ , and the measurements are presented in Fig. [7](#page-5-1) as dotted lines, which are saved upon the stabilization of the  $S_{21}$  phase. Worse matching and greater losses concerning simulations are appreciated, which can be attributed to manufacturing imperfections, the applied epoxy, and the connectors. The time responses obtained from applying the same processing as in Section [IV-A](#page-3-3) are shown in Fig. [8.](#page-5-2) In this case, it may not be so evident which  $t_{\text{start}}^{(11)}$  value should be selected, as the responses are expectably not as clean as in simulations. Thus, the air-filled pool case is advantageous to undergo a final calibration, ensuring its extracted relative permittivity is consistently close to 1 along the bandwidth. This is achieved by choosing  $t_{\text{start}}^{(11)} = 235$  ps, higher than in simulations due to the connectors contribution.

The time-gated *S*-parameters are shown as solid lines in Fig. [7.](#page-5-1) Note that although a quantitative assessment of the LC tan  $\delta$  cannot be carried out, the material is qualitatively behaving as expected, offering lower losses as it is increasingly polarized. Fig. [9](#page-5-3) presents the permittivity values extracted from these parameters. It is observed how the time gating and then the multilayered model effectively transform the permittivity values from those of the whole structure to the pool media ones. The results, although not as ideal, are

<span id="page-5-1"></span>

Fig. 7. Original and time-gated measured *S*-parameters for the ON and OFF LC states.

<span id="page-5-2"></span>

Fig. 8. Widened impulse responses of the measured  $S_{11}$  (up) and  $S_{21}$  (down) before and after the time gating, and with their timestamps.

<span id="page-5-3"></span>

Fig. 9. Effective and relative permittivities obtained from the original and time-gated measurements for different pool substrate scenarios.

considerably similar to those from simulations in Fig. [5.](#page-4-1) The fact that the OFF-state presents an  $\varepsilon_{r_{\parallel}} > \varepsilon_{r_{\text{OFF}}} > \varepsilon_{r_{\perp}}$  is mainly due to the high pool thickness, which prevents the rubbed Kapton layer from correctly orienting the LC molecules that are far from it, thus realizing an intermediate permittivity given by a  $\theta_m > 0^\circ$ . For the ON-state, the higher values

<span id="page-5-4"></span>

Fig. 10. Temporal change in the calculated permittivity of the LC from OFF- to ON-states and vice versa for  $20-V_{pp}$  (top) and  $100-V_{pp}$  (bottom) bias voltages.

obtained for the 100-V<sub>pp</sub> bias signal indicate that the 20-V<sub>pp</sub> bias was insufficient to polarize the LC fully. Furthermore, the achieved permittivity value is very similar to the simulation one (taking into account fabrication tolerances) and, so, less than  $\varepsilon_{r\parallel}$ , due to the inhomogeneous LC polarization by the narrow microstrip. These results show the achievable  $\varepsilon_r$  values for this physical configuration.

The results could be different if another type of geometry (for example, other transmission lines, substrate height, or microstrip width) was used, a problem arising from the anisotropic physical properties of the LCs. However, the aim of this work has been focused on extracting the achievable permittivities for any specific structure considered as a transmission line, which can significantly facilitate the work of researchers to know the effective range of variation of the relative permittivity of crystals when designing reconfigurable devices. For instance, if a leaky-wave antenna based on a periodically loaded microstrip line is built, this experiment is of direct utility to have a model for the LC permittivity and be able to design the reconfigurability range of its beam directions.

Using a script to periodically save measurements while the LC is being polarized and processing each of them, the LC permittivity's time evolution is obtained, as shown in Fig. [10](#page-5-4) for both polarization voltages. A higher bias voltage achieves not only a greater permittivity but also a faster polarization response (10%–90% response time of 68 s for 20  $V_{pp}$  and less than 3 s for 100  $V_{pp}$ ), as is the expected behavior for this GT7-29001 LC presenting elastic constants  $K_{11} = 14.3$  pN and  $K_{33} = 18.7$  pN, rotational viscosity  $\gamma = 316$  mPa s, and threshold voltage  $V_{\text{th}} = 0.85$  V. The return to the OFF-state has a similar evolution in both cases, with a response time around 350 s. These values are in close agreement with the theoretical expressions from [\[1\].](#page-6-0)

## V. CONCLUSION

<span id="page-5-0"></span>In this work, an efficient method for characterizing the dielectric permittivity of nematic LCs in broadband has been introduced, contributing to the advancement of reconfigurable devices for microwave and millimeter-wave applications. The approach uses additive manufacturing technology to fabricate a microstrip line that accommodates LCs, making it easy to polarize and achieve a dynamic modulation of its permittivity. Using additive manufacturing and eliminating thru–reflect–line (TRL) calibration in favor of time gating offer a practical and effective approach for efficiently characterizing LC-based reconfigurable devices. The validity of this method has been assessed through a comprehensive evaluation involving a combination of simulations and experimental measurements. The results are reliable, demonstrating the accuracy of the proposed technique, which is more straightforward than others found in the state of the art.

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#### **REFERENCES**

- <span id="page-6-0"></span>[\[1\] D](#page-0-0). C. Zografopoulos, A. Ferraro, and R. Beccherelli, "Liquid-crystal high-frequency microwave technology: Materials and characterization," *Adv. Mater. Technol.*, vol. 4, no. 2, Feb. 2019, Art. no. 1800447.
- <span id="page-6-1"></span>[\[2\] A](#page-0-1). Alex-Amor, Á. Palomares-Caballero, A. Palomares, A. Tamayo-Domínguez, J. M. Fernández-González, and P. Padilla, "Generalized director approach for liquid-crystal-based reconfigurable RF devices," *IEEE Microw. Wireless Compon. Lett.*, vol. 29, no. 10, pp. 634–637, Oct. 2019.
- <span id="page-6-2"></span>[\[3\] G](#page-0-2). Perez-Palomino et al., "Accurate and efficient modeling to calculate the voltage dependence of liquid crystal-based reflectarray cells," *IEEE Trans. Antennas Propag.*, vol. 62, no. 5, pp. 2659–2668, May 2014.
- <span id="page-6-3"></span>[\[4\] R](#page-0-2). Guirado, P. De la Rosa, G. Perez-Palomino, M. Caño-García, E. Carrasco, and X. Quintana, "Characterization and application of dual frequency liquid crystal mixtures in mm-wave reflectarray cells to improve their temporal response," *IEEE Trans. Antennas Propag.*, vol. 71, no. 8, pp. 6535–6545, Aug. 2023.
- <span id="page-6-4"></span>[\[5\] R](#page-0-3). Tchema, N. C. Papanicolaou, and A. C. Polycarpou, "An investigation of the dynamic beam-steering capability of a liquid-crystal-enabled leaky-wave antenna designed for 5G applications," *Appl. Phys. Lett.*, vol. 119, no. 3, Jul. 2021, Art. no. 034104.
- <span id="page-6-5"></span>[\[6\] E](#page-0-4). Abdo-Sanchez, A. Epstein, and G. V. Eleftheriades, "Reconfigurability mechanisms with scanning rate control for omega-bianisotropic huygens' metasurface leaky-wave antennas," *IEEE Access*, vol. 7, pp. 168247–168260, 2019.
- <span id="page-6-6"></span>[\[7\] F](#page-0-5). Kamrath et al., "Bandwidth and center frequency reconfigurable waveguide filter based on liquid crystal technology," *IEEE J. Microw.*, vol. 2, no. 1, pp. 134–144, Jan. 2022.
- <span id="page-6-7"></span>[\[8\] Q](#page-0-6). Wang, Z. Su, S. Li, Y. Su, H. Zhao, and X. Yin, "Electrically tunable phase shifter with improved phase-shifting capability based on liquid crystal," *IEEE Microw. Wireless Technol. Lett.*, vol. 33, no. 5, pp. 527–530, May 2023.
- <span id="page-6-8"></span>[\[9\] J](#page-0-7). Parka, J. Krupka, R. Dąbrowski, and J. Wosik, "Measurements of anisotropic complex permittivity of liquid crystals at microwave frequencies," *J. Eur. Ceram. Soc.*, vol. 27, nos. 8–9, pp. 2903–2905, Jan. 2007.
- <span id="page-6-9"></span>[\[10\]](#page-0-8) A. Alex-Amor, A. Palomares-Caballero, F. Mesa, O. Quevedo-Teruel, and P. Padilla, "Dispersion analysis of periodic structures in anisotropic media: Application to liquid crystals," *IEEE Trans. Antennas Propag.*, vol. 70, no. 4, pp. 2811–2821, Apr. 2022.
- <span id="page-6-10"></span>[\[11\]](#page-0-9) H. Peng, Y. Zhang, S. Zhu, M. Temiz, and A. El-Makadema, "Determining dielectric properties of nematic liquid crystals at microwave frequencies using inverted microstrip lines," *Liquid Crystals*, vol. 49, no. 15, pp. 2069–2081, Dec. 2022.
- <span id="page-6-11"></span>[\[12\]](#page-0-10) M. Yazdanpanahi, S. Bulja, D. Mirshekar-Syahkal, R. James, S. E. Day, and F. A. Fernandez, "Measurement of dielectric constants of nematic liquid crystals at mm-wave frequencies using patch resonator," *IEEE Trans. Instrum. Meas.*, vol. 59, no. 12, pp. 3079–3085, Dec. 2010.
- <span id="page-6-12"></span>[\[13\]](#page-0-11) V. Nova, C. Bachiller, B. Villacampa, R. Kronberger, and V. E. Boria, "Characterization of nematic liquid crystals at microwave frequencies," *Crystals*, vol. 10, no. 12, p. 1106, Dec. 2020.
- <span id="page-6-13"></span>[\[14\]](#page-0-12) P. Deo, D. Mirshekar-Syahkal, L. Seddon, S. E. Day, and F. A. Fernández, "Microstrip device for broadband (15–65 GHz) measurement of dielectric properties of nematic liquid crystals," *IEEE Trans. Microw. Theory Techn.*, vol. 63, no. 4, pp. 1388–1398, Apr. 2015.
- <span id="page-6-14"></span>[\[15\]](#page-0-13) M. El Kadiri, J. P. Parneix, and A. Chapoton, "General time domain analysis of T.D.S. data: Application to liquid crystals," *IEEE Trans. Instrum. Meas.*, vol. IM-34, no. 1, pp. 70–74, Mar. 1985.
- <span id="page-6-15"></span>[\[16\]](#page-0-13) K. M. Fidanboylu, S. M. Riad, and A. Elshabini-Riad, "An enhanced time-domain approach for dielectric characterization using stripline geometry," *IEEE Trans. Instrum. Meas.*, vol. 41, no. 1, pp. 132–136, Mar. 1992.
- <span id="page-6-16"></span>[\[17\]](#page-1-5) B. Archambeault, S. Connor, and J. C. Diepenbrock, "Time domain gating of frequency domain S-parameter data to remove connector end effects for PCB and cable applications," in *Proc. IEEE Int. Symp. Electromagn. Compat.*, Feb. 2006, pp. 199–202.
- <span id="page-6-17"></span>[\[18\]](#page-1-6) A. M. H. Nasr, A. Y. Nashashibi, and K. Sarabandi, "Ultrawideband characterization of complex dielectric constant of planar materials for 5G applications," *IEEE Trans. Instrum. Meas.*, vol. 70, pp. 1–11, 2021.
- <span id="page-6-18"></span>[\[19\]](#page-1-7) H.-J. Eul and B. Schiek, "A generalized theory and new calibration procedures for network analyzer self-calibration," *IEEE Trans. Microw. Theory Techn.*, vol. 39, no. 4, pp. 724–731, Apr. 1991.
- <span id="page-6-19"></span>[\[20\]](#page-1-7) K. J. Silvonen, "Calibration of test fixtures using at least two standards (microwave circuits)," *IEEE Trans. Microw. Theory Techn.*, vol. 39, no. 4, pp. 624–630, Apr. 1991.
- <span id="page-6-20"></span>[\[21\]](#page-1-8) M. Pérez-Escribano and E. Márquez-Segura, "Random errors in broadband characterization of the propagation constant of transmission lines using multiple two-port measurements," *IEEE Access*, vol. 9, pp. 59038–59047, 2021.
- <span id="page-6-21"></span>[\[22\]](#page-1-8) M. Pérez-Escribano and E. Márquez-Segura, "End-launcher repeatability in broadband methods for characterization of the propagation constant of transmission lines using two-port measurements," *IEEE Trans. Instrum. Meas.*, vol. 71, pp. 1–4, 2022.
- <span id="page-6-22"></span>[\[23\]](#page-1-9) M. Pérez-Escribano and E. Márquez-Segura, "Parameters characterization of dielectric materials samples in microwave and millimeterwave bands," *IEEE Trans. Microw. Theory Techn.*, vol. 69, no. 3, pp. 1723–1732, Mar. 2021.
- <span id="page-6-23"></span>[\[24\]](#page-2-6) Cadence®. *TDR Gating Python Script—AWR Scripts*. Accessed: Oct. 27, 2023. [Online]. Available: https://kb.awr.com/display/ awrscripts/TDR
- <span id="page-6-24"></span>[\[25\]](#page-2-7) M. A. Couker and L. J. Kushner, "A microstrip phase-trim device using a dielectric overlay," *IEEE Trans. Microw. Theory Techn.*, vol. 42, no. 11, pp. 2023–2026, Nov. 1994.
- <span id="page-6-25"></span>[\[26\]](#page-2-8) A. K. Verma and G. H. Sadr, "Unified dispersion model for multilayer microstrip line," *IEEE Trans. Microw. Theory Techn.*, vol. 40, no. 7, pp. 1587–1591, Jul. 1992.
- <span id="page-6-26"></span>[\[27\]](#page-3-4) D.-K. Yang and S.-T. Wu, *Fundamentals of Liquid Crystal Devices* (Wiley SID series in display technology). Hoboken, NJ, USA: Wiley, 2006.
- <span id="page-6-27"></span>[\[28\]](#page-3-4) R. Guirado et al., "Dynamic modeling of liquid crystal-based metasurfaces and its application to reducing reconfigurability times," *IEEE Trans. Antennas Propag.*, vol. 70, no. 12, pp. 11847–11857, Dec. 2022.



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