A Computer Controlled Microwave Resonant Cavity used for the Dielectric Studies of Liquid Crystals

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Abstract— A cylindrical cavity in TE_{011} mode is used to study the microwave dielectric response of liquid crystals cyclopentanol and 4, 4'-di-n-heptyloxyazoxybenzene (HAB). The dielectric behavior of these liquid crystals is studied as a function of temperature at a microwave frequency of 9.12 GHz. A computer controlled resonant cavity is used to study the dielectric relaxation behavior in these liquid crystals. In cyclopentanol there is a very interesting behavior and the dielectric relaxation process seems to be a function of cooling rate. This type of behavior is not seen in liquid crystal 4,4’ –di-n-heptyloxyazoxybenzene (HAB).

Index Terms—dielectric relaxation, liquid crystals, microwave frequency, resonant cavity.

I. INTRODUCTION

Dielectric behavior of a material is a very important process and it reveals the electrical nature of that material. In a dc or low frequency ac field the dipoles are able to follow the field very nicely and there is no lag between the dipoles and the field. As the frequency of the applied field increases some of the dipoles will not be able to follow the field. This behavior becomes very distinct at microwave frequencies. The permanent dipoles have a difficult time following frequencies of the order of GHz. This is the process of dielectric relaxation at microwave frequencies. A number of different types of experiments have been performed to study the microwave dielectric response of materials [1-6]. These and other investigators have used various techniques including wave guides and even resonant cavities in different modes. A microwave resonant cavity in the form of a cylinder has become a standard tool in terms of identifying the dielectric nature of the material. It is a very sensitive cavity with high Q-value and can count the dipoles lining up with the applied field very precisely. The TE_{011} mode is very sensitive in terms of the perturbations of electric field inside the resonant cavity. It has proven to be a very interesting experiment to find the dielectric response of a material near a phase transition region, i.e., going from an ordered to a disordered phase. There is a dramatic change in the dielectric response of a material near its freezing point. Water shows a very rapid change in the dielectric response near its freezing point. This behavior was studied by Dahiya et al [7]. The drop in the dielectric constant of water at the freezing point is caused by the partial or complete loss of rotational polarization, while the electronic and vibrational polarizations remain relatively unchanged.

II. THEORY AND EXPERIMENTAL TECHNIQUE

Dielectric studies of the above mentioned liquid crystals are performed using a microwave spectrometer as shown in Figure 1.
A klystron in the x-band of frequencies (8.0-12.4 GHz) is used to produce a microwave signal at 9.12 GHz. The modulated absorption signal after being reflected from the resonant cavity and amplified by a tuned amplifier at 31 KHz appears on the oscilloscope in the form of a butterfly as shown in Figure 2.

A series of markers are produced after mixing a part of the microwave signal with a standard signal produced by a generator. A radio receiver is used to detect the difference in these two signals and in turn produces markers at a fixed frequency interval. One of these markers is chosen to monitor the change in frequency as well as the width change of the signal as the sample to be studied is inserted into the resonant cavity. The resonant frequency shifts and the Q-changes of the signal are related to the real and imaginary parts of the dielectric constant through Slater’s equations as follows.

\[ \frac{\Delta f}{f_o} = \frac{\varepsilon' - 1}{2} \int \frac{E^0 \cdot E^0}{E \cdot E_s} dv \]  
\[ \Delta \left( \frac{1}{Q} \right) = \varepsilon'' \int \frac{E^0 \cdot E^0}{E \cdot E_s} dv \]

where \( \Delta f \) and \( \Delta \left( \frac{1}{Q} \right) \) are the frequency shifts and Q-changes of the signal, \( \varepsilon' \) and \( \varepsilon'' \) are the real and imaginary parts of the dielectric constant, \( E_o, E_s \) and \( E \) are the applied field, field inside the sample and the total field of the system respectively.

\[ \Delta \left( \frac{1}{Q} \right) = \frac{\sqrt{3} \Delta W}{f_o} \]

where \( \sqrt{3} \) is introduced after modulation correction.

The real and imaginary parts of the dielectric constant are also related to the relaxation time using Debye’s equations, [8],

\[ \frac{\varepsilon_s - \varepsilon'}{\varepsilon''} = \omega \tau \]

where \( \varepsilon_s \) is the static dielectric constant and \( \tau \) is the relaxation time. With the help of the computer interface circuit the flow of air cooled to liquid nitrogen temperature is controlled. It is very important to do this, as the change in the dielectric behavior of a material is significant near the phase transition temperature. The temperature needs to be changed by tenth of a degree during this time and be maintained to a certain value for a few seconds. The flow of air is adjusted to the desired value using a controller circuit. The thermal bath designed for this type
of experiment is very sensitive and can be seen in Figure 3.

A controlled flow of air goes through a mixture of sodium carbonate and calcium carbonate. This helps in absorbing moisture from air coming out of the desired source. The air then goes through a couple of cold traps where any moisture left in air condenses and one hundred per cent dry air is circulated around the cavity. The sample of material under study inside the resonant cavity comes to thermal equilibrium with the temperature of the cavity. This happens very quickly and the material under study attains the liquid nitrogen temperature. The flow of air is then adjusted using the computer-controlled system for the temperature to rise. Temperature is then allowed to rise periodically and dielectric data taken for each temperature change. As the material under study goes through a phase change, data is taken more frequently for up to a tenth of a degree change in temperature. This process allows maximum number of data points taken during the phase change of the material. In order to effectively compute the perturbation response of the probing material a resonant cavity fine tuned in TE_{011} mode was employed in this investigation. A resonant cylinder with diameter to length (D/L) ratio kept less than 0.5 was swept in a frequency range of 9.1-9.2 GHZ , and the loss factor measurements were monitored by measuring the width at half-power maxima as described through equation 3. Slater’s perturbation model was used for analytically plotting the shift in the resonant profile of the cavity, and the data measurements were made with respect to the length of the liquid crystal strip being introduced into the system. It was found using a standard mode-chart that the operational frequency of 9.1-9.2 GHz was favorable with the geometry of our resonant cylinder, thereby assisting in minimizing the effects of other spurious modes that remain present at such high frequencies.

III. RESULTS AND ANALYSIS

There is a dramatic change in the dielectric behavior of the material near its phase change. This behavior can be seen for liquid crystals cyclopentanol and heptyloxyazoxy benzene in Figures 4-8.

In case of cyclopentanol the dielectric behavior is a function of the cooling rate. The cooling rates were adjusted to 1°C/min for slow cooling to 4°C/min for moderate cooling to 7°C for fast cooling. As can be seen in Figures 5-6 there is a dramatic change in the dielectric behavior of cyclopentanol as we go from slow to fast cooling rates.
At the moderate rate of cooling there seems to be another phase change at –42°C that did not show up at slow as well as fast cooling rates. At fast cooling rates the dielectric constant becomes more than double its value at the slow cooling rate. In liquid crystal HAB the observed process of dielectric relaxation is seemed to be linked with the orientation of molecules about their long axes. There is a very strong transition-taking place near 74°C. This can be seen in Figures 7 and 8.

Figure 7 is the measure of the imaginary part of dielectric constant, which in turn is the dielectric loss of the material. In Figure 8, we can see the behavior of the real part of the dielectric constant as a function of frequency at a resonant frequency of 9.12 GHz. In both cases there is a dramatic change in the dielectric behavior near the transition temperature of this material, which is 74°C.

IV. CONCLUSION

The microwave resonant cavity technique as applied in this investigation is very successful in monitoring the phase changes in liquid crystals cyclopentanol and heptyloxyazoxy-benzene (HAB). The microwave dielectric response of these materials shows a very distinct dielectric behavior near the phase transition temperatures of these materials. The dielectric relaxation behavior in cyclopentanol seems to depend on the cooling rate. The heating and cooling methods involved in this experiment are very effective in maintaining a certain temperature. Data is taken at a microwave frequency of 9.12 GHz and the experiment is repeated several times to observe the dielectric behavior of cyclopentanol and HAB.

REFERENCES