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Ultrasonic and Viscoelastic Properties of Small-Volume Mesogen Samples at the Phase Transition

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Abstract The ultrasonic method is very informative for research of viscoelastic properties of mesogens at changing thermodynamic parameters of state. At phase transitions, anomalies of the velocity of propagation and coefficient of absorption of ultrasound waves as well as of viscoelastic properties are observed. These anomalies for liquid crystals are most pronounced at frequencies lower that 1 MHz. Up to now acoustic resonators with volumes of about 5 cm^3 have been used, which considerably prevents the application of this method for science-based newly synthesized mesogenic compounds. This article presents experimental results obtained by means of a new resonator method with samples with volumes of 0.06 cm^3 to 0.15 cm^3. The velocity and coefficient of absorption of ultrasound were measured at frequencies from 0.68 MHz to 1.63 MHz for four mesogens: esters of alkyloxyphenylcyclo-hexane-2-carbonic acid and *n*-amylphenol. It has been shown that temperature dependencies of ultrasonic parameters obtained in small-volume cells correspond to those established previously by traditional methods using measuring cells with larger volumes. It is also shown that the temperature dependencies of the bulk viscosity and the bulk elasticity modulus derived from our ultrasonic measurements, in general features, duplicate the corresponding dependencies obtained by standard methods. It confirms that the proposed acoustic method is suitable for routine investigations of viscoelastic properties of small-volume samples of mesogenic compounds.

Keywords Dispersion systems · Liquid crystal small-volume samples · Mesogenic compounds · Ultrasonic parameters · Viscoelastic properties

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1 Introduction

Ultrasonic methods can be considered as very useful for the study of viscoelastic properties of liquid crystals (LCs) at changeable thermodynamic parameters of state [1–3]. Interesting results were achieved in research studies of phase transitions where a velocity (c) and an absorption coefficient (α) of ultrasonic waves showed anomalies that could be considered as anomalies in the viscoelastic properties [4,5]. As was shown for a number of liquid crystals, these effects are most well pronounced in the low frequency range, typically at frequencies lower than 1 MHz [6,7]. Until now, such measurements were carried out in acoustic resonators with a minimal volume of about 5 cm^3. It essentially compromises the application of ultrasonic methods for studies of newly synthesized (and rather expensive) mesogenic compounds. In this article, we consider both some theoretical aspects important for an interpretation of the results of ultrasonic studies of viscoelastic properties of LCs, and describe a new method of low-frequency ultrasonic measurements applicable to small-volume samples (of 0.06 cm^3 to 0.15 cm^3). We also present examples of an ultrasonic study of viscoelastic properties at phase transitions in some mesogenic compounds. Additionally, we discuss the possibility of ultrasonic investigations of LCs confined by droplets of micron and sub-micron sizes immersed in an isotropic liquid. The overall objective of this study is to confirm the potential of absolute and relative measurements (at phase transitions) of ultrasonic parameters to extract information similar to that available from standard ultrasonic studies using samples of large volume. Possible variations of viscoelastic properties due to strong confinement (in LC droplets) are also considered.

2 Materials

In this study, some complex mesogen compounds of the esters of alkyloxyphenylcyclohexanecarboxylic acid are investigated. These mesogens have been synthesized at Ivanovo State University [8]. The structure of 6–9th homologues (I–IV, accordingly) is shown in Fig. 1. According to thermal polarizing microscopy [9] on heating samples of compounds I–IV at a rate of 0.4 $\mathrm{K \cdot min^{-1}}$, polymorphism of the following kind is observed: in compound I—Cr 313 K, N 333 K, Sm 328 K, I; II—Cr 309 K, Sm 335 K, N 339 K, I; III—Cr 318 K, Sm 340 K, N 341 K, I; IV—Cr 342 K, Sm 339 K, I (Cr, N, Sm, and I—crystal, nematic, smectic, and isotropic phases, respectively).

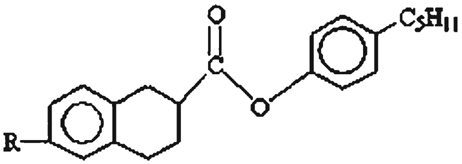

Fig. 1 Structural formulas of compounds I–IV: I: R= $-OC_6H_{13}$; II: R= $-OC_7H_{15}$; III: R= $-OC_8H_{17}$; IV: R= $-OC_9H_{19}$

3 Theoretical Background

Liquid crystals are referred to as partly ordered condensed media, showing complex rheological behavior. One of the simplest classes of such media is represented by nematic liquid crystals (NLC) which differ from isotropic liquids by the additional orientational degree of freedom described in terms of a unit vector (director n). It characterizes a preferable direction of long molecular axes and has to be incorporated into hydrodynamic equations of NLCs. The standard hydrodynamic theory of nematic liquid crystals usually applied for solving of different physical problems was derived by Leslie and Erickson and is well described in a number of books dedicated to physics of liquid crystals [10–12]. In most cases of interest for display applications, NLCs can be considered as incompressible liquids described by six dissipative parameters, α_i (Leslie‘s coefficients). Contrary to it, a propagation of an ultrasonic wave is directly connected with a compressibility. So the three additional dissipative coefficients μ_i (so-called bulk viscosities) have to be added to take into account the dissipative processes arising due to density changes. The solution of a linearized system of hydrodynamic equations results in the dependence of an absorption coefficient α on the angle θ between a wave vector and a director:

$$\alpha/f^2(\theta) = \alpha/f^2(\theta = \pi/2) + a_\alpha \cos^2\theta + b_\alpha \cos^4\theta, \tag{1}$$

$$\alpha/f^2(\theta = \pi/2) = (2\pi^2/\rho c^3)(\mu_1 + \alpha_4), \tag{2}$$

$$a_\alpha = (2\pi^2/\rho c^3)(\mu_2 + \mu_3 + \alpha_5 + \alpha_3 - \gamma_1\lambda^2), \tag{3}$$

$$b_\alpha = (2\pi^2/\rho c^3)(\alpha_1 + \gamma_1\lambda^2), \tag{4}$$

$$\lambda = \gamma_1/\gamma_2, \quad \gamma_1 = \alpha_3 - \alpha_2, \quad \gamma_2 = \alpha_3 + \alpha_2. \tag{5}$$

Here, f is the ultrasonic frequency, ρ is the density of the LC, and c is an ultrasonic velocity, which in a hydrodynamic approach, is not dependent on the orientation of a liquid crystal. Experimental investigations of the anisotropic propagation of ultrasound in mono-domain samples of NLC [1–3] have confirmed a type of angular dependence of the ultrasound absorption coefficient (Eq. 1), predicted by theory and the isotropic character of the ultrasonic velocity in the range of low frequencies. Nevertheless, a strong frequency dependence was found for both the absolute value of the ultrasound absorption coefficient and its anisotropy normalized by the frequency squared. It was referred to as the influence of an acoustic relaxation. Formally, this influence can be taken into account within the framework of generalized hydrodynamics by introduction of the complex modulus of elasticity [13]. The imaginary part of the given modulus determines the relaxation contributions to the absorption coefficient of ultrasound, and the real part to the velocity of ultrasound. In terms of the above-mentioned expressions, it means that factors of bulk viscosity depend on the frequency of the ultrasound. For an individual relaxation process with a characteristic time τ, this dependence looks like

$$\mu_i = \mu_i^o(1 + \omega\tau)^{-1}. \tag{6}$$

In this approach, the velocity of ultrasound also includes the anisotropic part, as determined by

$$[c(\theta) - c(\theta = \pi/2)]/c(\theta = \pi/2) = a_c \cos^2 \theta + b_c \cos^4 \theta \qquad (7)$$

The parameters a_c and b_c include only relaxation contributions and go to zero within the limits of low frequencies. The presented above conclusions have also received a more strict microscopic background [14]. The existence of anisotropy of the ultrasound velocity was confirmed by direct experiments [15]. This relative velocity anisotropy defined by Eq. 7 does not exceed 10^{-3} at a frequency $f = 3$ MHz and may be neglected in many cases.

The dynamic processes responsible for relaxation contributions to viscoelastic parameters of NLCs can be subdivided into the "normal" process, connected, for example, with conformation transformations into lateral molecular chains and the "critical" ones, caused by a relaxation of the order parameter or a relaxation of the fluctuations of the order parameter in the vicinity of a nematic–isotropic transition. Critical contributions to viscoelastic parameters were analyzed within the framework of a general theory of critical phenomena in the isotropic approach [16]; anisotropic contributions from a relaxation of the order parameter were obtained in the framework of microscopic models [14]. Both the approaches predict the strong temperature dependence of the critical contributions arising from the drastic slowing of relaxation processes on approaching the clearing point. The experimental results obtained up to now [6,17,18] are in qualitative agreement with the theoretical conclusions. In particular, the low-frequency asymptotes of acoustic parameters are described by simple power lows with critical indexes close to those predicted by theory.

In summary, we conclude that ultrasonic investigations of oriented samples of nematic liquid crystals provide a lot of information about the bulk elastic modulus, anisotropic viscous parameters of liquid crystals, and the nature of critical processes at phase transitions in liquid crystals. It demands a realization of acoustic measurements over a wide frequency range with variations of the temperature as viscoelastic characteristics show a strong dependence on the specified parameters. Previously, such investigations were carried out for monodomain bulk samples of nematics, oriented by magnetic fields.

Expansion of ultrasonic studies on liquid crystal systems with strong spatial restrictions give enhanced results as they provide detailed information on both phase diagrams and viscoelastic properties of LCs at phase transitions which can be modified by confinement. We can point to such systems suitable for ultrasonic studies, namely, layered systems (a number of liquid crystal and solid layers) and liquid crystal emulsions (drops of a liquid crystal of micron and submicronic sizes in isotropic liquids). Layered systems are convenient from the point of view of the creation of a given orientation of a LC by the appropriate surface treatment of solid plates. Besides, in thin layers of liquid crystals, it is convenient to change orientation with the help of electric fields. Generally, the combination of the orienting action of fields and surfaces results in a spatially non-uniform distribution of the director within the limits of a liquid crystal layer. The theory of such deformed structures is rather well developed in connection with the use of layered systems in liquid crystal displays. Up to now, a distribution of elastic waves in the layered environment of the specified type is practically not investigated. In this article, we report results of a study of a unique LC layer of a liquid crystal of 2 mm thickness, confined by two plane surfaces.

In this case, the influence of surfaces is rather small and can be neglected. So, we consider a nematic layer with an isotropic distribution of local orientations of a director. In this case, Eq. 1 can be averaged and the absorption coefficient of a non-oriented sample can be expressed as

$$\frac{\alpha}{f^2} = \frac{2\pi^2}{\rho c^3}\mu^* \tag{8}$$

where μ^* is the dynamic viscosity—a combination of bulk viscosities and Leslie coefficients [19]. The latter essentially do not contribute in the low frequency range. So the value of μ^* can decrease with a growth of the frequency in accordance with Eq. 6. We used Eq. 8 for the analysis of viscoelastic properties of liquid crystals. To derive information about Leslie coefficients, measurements of oriented samples are needed. Such measurements are beyond the scope of this article.

In addition to the bulk viscosity, it is possible to determine the adiabatic compressibility, β_s, and the bulk modulus of elasticity, K, from ultrasonic velocity measurements in accordance with

$$\beta_s = \frac{1}{\rho c^2} = \frac{1}{K}. \tag{9}$$

These elastic parameters can be considered as equilibrium ones in the low frequency limit, $\omega\tau \ll 1$.

4 Experiment

Measurements of low-frequency ultrasonic characteristics in small-volume samples of LCs were carried out using the experimental setup (Fig. 2) described in detail in [20]. This setup is intended for absolute and relative measurements of c and α in cells of various volumes by pulse, pulse-phase, and resonant methods [21–23]. In this study, three resonant cells were used which provided the frequency range from 0.68 MHz to 1.63 MHz. The cylindrical chamber of 70 cm^3 volume (Fig. 2c) was used for control measurements and for acoustic studies of binary systems (solutions and emulsions) of LC-isotropic liquids (water, glycerin, etc.) [20]. The cell of 0.15 cm^3 volume was made of two planar-convex transducers and a metal spacer ring of 2 mm thickness (Fig. 2b). It was used for absolute measurements of the ultrasonic velocity and absorption coefficient c and α in the samples of LCs at phase transitions [24,25]. The open acousto-optic cell of the smallest size (Fig. 2f) was used for relative measurements (c/c_0) and (α/α_0) at phase transitions in LC samples of 0.06 cm^3 volume and also for direct visual control of phase transitions.

The piezoceramic transducers with 2.7 MHz resonance frequency were used for ultrasonic measurements. The acoustic characteristics were defined in various ways, dependent on the particular type of setup. It was possible to extract these parameters:

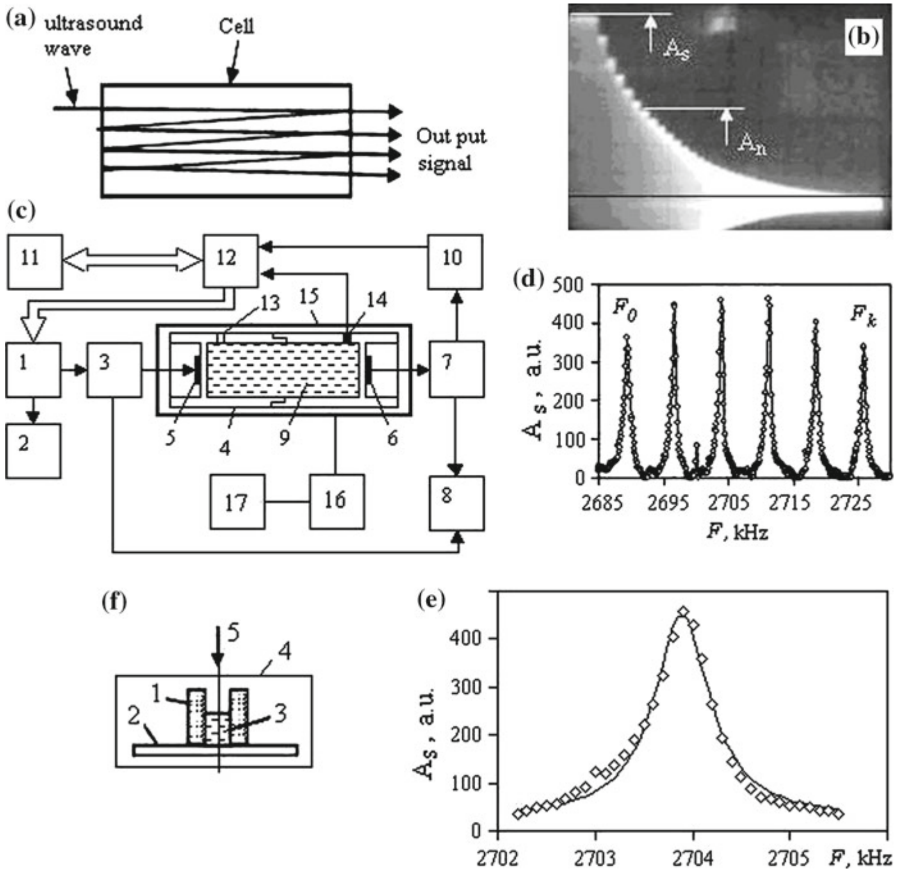

Fig. 2 Schematic diagrams of the ultrasonic setup: (**a**) geometry of experiment; (**b**) form of output signal; (**c**) diagram of the setup: *1* generator, *2* frequency meter, *3* "long" pulse generator, *4* acoustic cell of 70-cm^3 volume; *5*, *6* piezoelectric transducers, *7* amplifier, *8* oscilloscope, *9* sample, *10* voltmeter, *11* computer, *12* interface, *13* inlet, *14* thermometer, *15* thermostat, *16* controller, *17* device for control of temperature changes; (**d**) amplitude–frequency dependence; (**e**) form of a single resonant peak; (**f**) acousto-optic cell of 0.06-cm^3 volume: *1* transducers, *2* glass plate, *3* sample, *4* thermostat, *5* optic channel

(1) from an analysis of the recession (Fig. 2b) of a long radio-pulse signal at small values of α in the cell with a large volume of 70 cm^3; (2) from the frequency dependencies (Fig. 2d, e) of the amplitude of an acoustical signal far from the phase transition, in the cells with a volume of 0.15 cm^3; and (3) from an analysis of the resulting resonant signal in the vicinity of the phase transition—for the resonant cell with a volume of 0.06 cm^3. The following expressions were used in such calculations:

$$c = 2L(f_k - f_0)/k; \quad c/c_0 = f/f_0(1 + b(T - T_0)) \tag{10}$$

$$A_n = A_s \exp(-\alpha_s 2Ln); \quad \alpha = \alpha_s - \alpha_k, \tag{11}$$

$$\alpha_s = \pi \Delta f/c; \quad \alpha = \alpha_s - \alpha_k, \tag{12}$$

$$\alpha_s = \alpha_{s0}(A_{s0}/A_s), \tag{13}$$

where L is the acoustic base of the cell, f_0 and f_k are the frequencies of resonant conditions with numbers k (Fig. 2d); A_s and A_{s0} are the current and initial values, respectively, of the resulting signal on an output of the acoustic cell for frequencies at a resonance depending on an external parameter—temperature; A_n is the ordinate of a signal for a "step" of the oscillogram of a signal with number n (Fig. 2b); α_s and α_{s0} are the current and initial values, respectively, of the attenuation factor of an acoustic signal in the system (the cell + a sample), α_k represents the losses of the cell; c_0 and f_0 are the initial values of the ultrasound velocity and frequency, respectively, of a resonant condition for relative measurements; Δf is the half-width of the resonant condition for relative measurements, i.e., of the resonant peak (Fig. 2g); and $\alpha_s = \alpha_{s0}(A_{s0}/A_s)$. Losses of the cell, α_k, are defined by measurements of the attenuation of the signal α_s in the cell filled with the reference sample with known acoustic properties.

5 Results

The temperature dependencies of the acoustic (c and α) and viscoelastic (μ^* and K) parameters of liquid crystals (compounds I–IV) obtained for the samples of volumes 0.15 cm^3 are shown in Figs. 3, 4, 5, and 6.

The ultrasonic velocity c and the bulk modulus of elasticity K calculated via Eq. 9 show an average decrease with increasing temperature for all investigated compounds (Figs. 3, 4). Some peculiarities of the dependencies $c(T)$ and $K(T)$ can be assigned to the next phase transitions: "Steps" at 219 K and 331 K (in compounds III and IV)—to the transition crystal–smectic phase (Cr–Sm); breaks at 228 K and 300 K (in compounds I–III)—to the transition smectic–nematic (Sm–N); local minima at 325 K

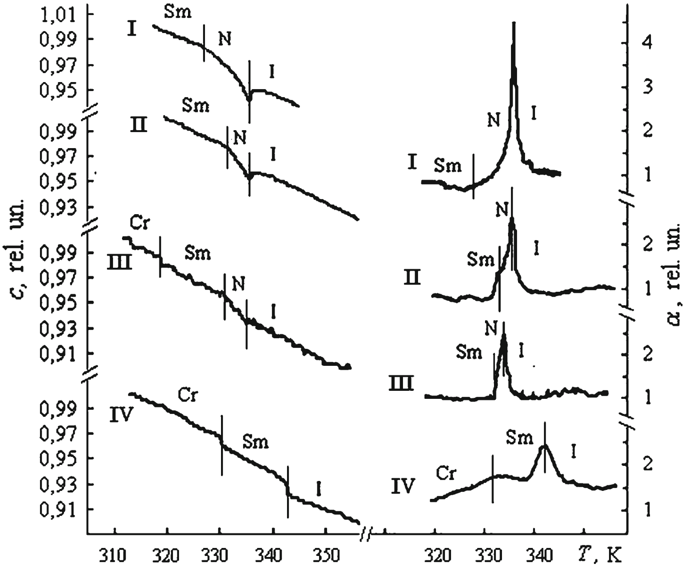

Fig. 3 Temperature dependencies of the sound velocity c and absorption coefficient α at a frequency 0.68 MHz for compound: (I) of volume 0.15 cm^3 and (II–IV) of volume 0.06 cm^3

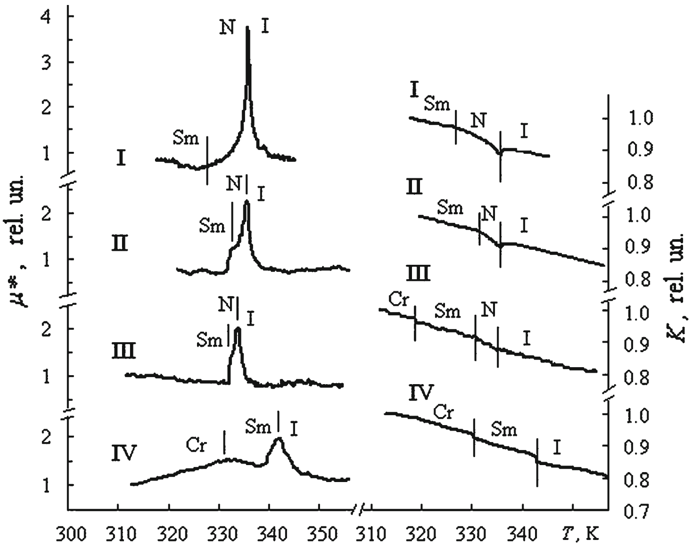

Fig. 4 Temperature dependencies of the effective bulk viscosity μ^* and the bulk modulus of elasticity K for samples of compounds: (I) of volume 0.15 cm^3 and (II–IV) 0.06 cm^3 (calculated on the basis of the acoustic data)

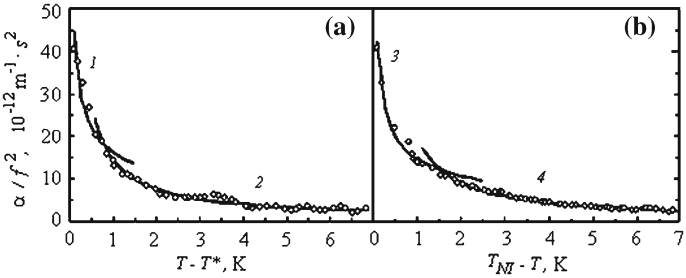

Fig. 5 Temperature dependencies of the absorption coefficient for the **a** isotropic and **b** nematic phases. *Solid curves* correspond to various values of the critical index β: (*1*) 0.44 ± 0.05 (statistical characteristics, $R^2 = 0.902$, $\chi^2 = 12.6$); (*2*) 0.93 ± 0.03 ($R^2 = 0.949$, $\chi^2 = 0.27$); (*3*) 0.46 ± 0.02 ($R^2 = 0.978$, $\chi^2 = 1.8$); and (*4*) 1.08 ± 0.02 ($R^2 = 0.988$, $\chi^2 = 0.07$)

(in compounds I–III)—to the transition nematic–isotropic liquid (N–I); and the weakly seen minimum which looks like a "step" at 343 K (in compound IV)—to the transition smectic–isotropic liquid (Sm–I). The depth of the minimum of the ultrasound velocity and the bulk modulus of elasticity K in compounds I–IV at a constant frequency $\omega = 2\pi f$ decrease with a growth of the number of atoms of carbon in the alkyl chain and can be assigned based on increases of growth of the relaxation time τ which can be extracted from the frequency dependence of the bulk viscosity [1,16].

The critical behavior of the absorption coefficient α in the vicinity of a nematic–isotropic transition established for the compound I (in samples with volumes of 0.15 cm^3) at frequencies less than 1 MHz is shown in Fig. 5. It corresponds to well-known properties of other liquid crystals with samples with a volume of about 5 cm^3

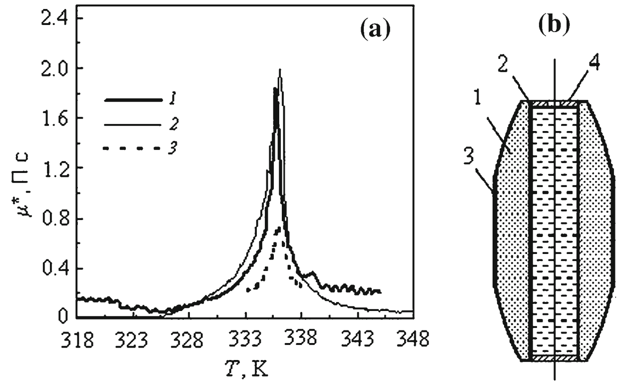

Fig. 6 (**a**) Plots of the effective bulk viscosity μ^* (*T*) for various frequencies (MHz): (*1*) 0.68; (*2*) 1.37; (*3*) 1.63 and (**b**) Schematic diagram of the cell with an inner volume of 0.15 cm^3: *1* piezoelectric transducer: *2*, *3* electrodes, *4* metal spacer ring

[6]. Dependencies of α/f^2 on temperature, for isotropic and nematic phases, are well described (Fig. 6) by simple power laws:

$$\alpha/f^2 = \alpha/(T - T^*)^\beta \quad \text{and} \quad \alpha/f^2 = \alpha/(T_{\text{NI}} - T)^\beta \tag{14}$$

where T_{NI} is the temperature of the nematic–isotropic transition, $T^* = T_{\text{NI}} - \Delta T^*$—the temperature corresponding to the limit of stability of the isotropic phase, and $\Delta T^* \approx 1$ K—the temperature interval of overcooling at the phase transition. The values of the critical indexes for both phases ($\beta \approx 1$ at temperatures less than 1.5 K from the phase transition temperature and $\beta \approx 0.45$ in the vicinity of the phase transition) are also close to those obtained in ultrasonic studies of bulk samples of liquid crystals [6,16–18]. The value $\beta \approx 1$ is consistent with the theoretical result ($\beta = 1$) obtained in a low-frequency hydrodynamic limit $\omega\tau \ll 1$ [16–18]. The value of the critical index near the phase transition (about 0.45) is also consistent with results obtained earlier for samples with a volume of about 5 cm^3 [6].

The data shown in Fig. 6 also confirm that the hydrodynamic limit was achieved at frequencies less than 1 MHz, where is consistent with Eq. 6 which shows that a bulk viscosity does not depend on frequency.

It is obvious that the sensitivity of an acoustic method essentially decreases with a decrease of the sample volume to 0.06 cm^3. However, as apparently seen from Figs. 3 and 4, the mentioned-above sensitivity is sufficient to detect anomalies of acoustical parameters at phase transitions (especially in the vicinity of the N–I phase transition) based on measurements of relative values of c and α. In particular, it is possible to detect an increase of the width for an absorption peak and the dependence $\alpha(T)$ from 332 K to 334 K (in compounds II–III), which can be attributed to the influence of the Sm–N phase transition. A decrease of the height of the maxima on the dependence $\alpha(T)$ and $\mu^*(T)$ is also seen in the vicinity of the N–I transition in a number of compounds I–IV with an increase in the number of carbon atoms in alkyl chains. It can be attributed to the growth of the relaxation time, as the relaxation absorption in LCs decreases with an increase of the product $\omega\tau$ [1,16]. The basic features in the dependencies of $c(T)$ and $\alpha(T)$ are well reproduced in the calculated dependencies of $K(T)$ and $\mu^*(T)$. Thus, the acoustical method can be considered as being informative for studies of the viscoelastic properties of liquid crystal samples of extremely small volumes.

6 Conclusions

The acoustic method and the experimental setups were modified to adapt them for ultrasonic studies of the viscoelastic properties of liquid crystal samples of very restricted volumes.

It was found that the temperature dependencies of the acoustic parameters c and α, determined in cells of volumes of 0.15 cm^3 to 0.06 cm^3 are consistent with the well-known acoustic properties of liquid crystals in bulk samples. It is also true with regard to the temperature dependencies of the bulk viscosity and the bulk modulus of elasticity calculated using acoustic data which show agreement with the main features of the expected temperature dependencies of the acoustic parameters. Thus, the viscoelastic characteristics of liquid crystals can be determined directly via measurements of the temperature dependencies of the acoustic parameters of small-volume samples of liquid crystals. The proposed method provides high sensitivity to the variation of ultrasonic absorption in the vicinity of phase transitions when the latter parameter critically increases at low frequencies. It makes it possible to use such a technique to study both the thin layers of liquid crystals and the submicron composite materials such as liquid crystal dispersions. In particular, it will be interesting to study liquid crystal droplets with sizes comparable with the correlation length of order parameter fluctuations. In this case, one can consider an essential modification of critical dynamics which can be detected by ultrasonic measurements. Such an opportunity is under consideration now.

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