Structure of the foggy phase of liquid crystals: transmission and diffraction spectra in an electric field

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The foggy phase (BPIII) of liquid crystals in an electric field is investigated by diffraction measurements, both in a liquid crystal film and for various directions of the scattered wave vector with respect to the electric field direction in bulk samples; in addition, the dependence of the index of refraction on field is measured. A significant change is observed in the transmission spectra of the liquid crystal an electric field, which is associated with the BPIII type of orientational ordering. The orientational distribution function in the BPIII phase is recovered from the experimental data. These data are compared with existing models of local order in liquid crystals. The results obtained agree neither with the body-centered cubic model, the icosahedral-ordering model, nor the double-twist cylinder model; however, they can be explained using a model with simple cubic structure. © *1995 American Institute of Physics.*

1. INTRODUCTION

In recent years, there has been much interest among condensed-matter physicists in structures with competing interactions. In these systems, atomic or molecular configurations that are energetically advantageous locally are nevertheless unable to continuously occupy a macroscopic volume. As a result, compromise structures can occur with defect distributions that exhibit three-dimensional ordering or disordering. The blue phases (BPI, BPII, BPIII) of liquid crystals[1,2] are one of the most interesting examples of such systems.

A large number of chiral liquid-crystal materials form blue phases in the temperature interval between isotropic liquid behavior and the cholesteric phase. In this past year, considerable progress has been made in the experimental and theoretical study of these blue phases. The most remarkable characteristic of the BPI and BPII phases is Bragg diffraction in the visible or ultraviolet wavelength regions, which indicates three-dimensional periodic ordering. It has been established that BPI possesses a body-centered cubic structure with symmetry space group $O^8(I4_132)$, while BPII has a simple cubic structure with the space group $O^2(P4_232)$.[3-5] A unit cell of the blue phases contains $\sim 10^7$ molecules. Two approaches have been successfully used to describe the three-dimensional crystallization and structure of the blue phases theoretically: the Landau theory of phase transitions, where the anisotropic part of the dielectric tensor $\varepsilon_{ij}(r)$ is used as an order parameter, and dislocation theory, which treats the BPI and BPII structures as ordered defect lattices.[1,2] Despite the complexity both of the theory and the structures of the blue phases, a large amount of experimental data for the phases with three-dimensional periodic order parameters (BPI, BPII) has been successfully described at the quantitative level.

BPIII, which is also called the foggy phase,[6,7] has been studied theoretically and experimentally in parallel with BPI and BPII. A whole series of theoretical models has been proposed to explain why BPIII arises, and what its structure is: (1) According to the double-twist cylinder model,[8] BPIII consists of elongated cylinders in which adjacent molecules are twisted together in the radial directions of the cylinders; (2) In the body-centered cubic model,[9] the BPIII phase is assumed to consist of arbitrarily oriented domains with O^5 structure; (3) In the simple cubic model,[10] it is assumed to consist of domains with O^2 symmetry; (4) Several theoretical groups have independently predicted a quasicrystalline structure for BPIII[11-14] with local quasiperiodic icosahedral symmetry, similar to the symmetry observed in binary metallic quasicrystals. In Ref. 14 a theory was developed that predicted a reconstruction of the quasicrystalline structure of BPIII in an electric field.

The mechanism for the BPI–BPII and BPII–BPIII phase transitions implies an anomalous increase in fluctuations[16-19] and Kosterlitz–Thouless transitions.[20] It was shown theoretically that as the temperature increases BPIII can become unstable against spontaneous thermal distortion of the lattice, breaking the translational symmetry while the cubic orientational ordering of the crystal lattice is preserved. It is possible for such structures to form when ordinary crystals melt; this was discussed previously by Nelson and Toner (their "phase with orientationally ordered bonds").[21]

There are experimental problems in determining the structure of BPIII, connected with its large diffraction width. The BPII–BPIII transition is accompanied by an order-of-magnitude increase in the diffraction width, i.e., the phase possesses only short-range order.[22,23] In determining the type of local order of BPIII, the researcher is faced with the same problems as in the case of amorphous materials. As a rule, information about the local order of atoms or molecules in an amorphous material is obtained by comparing the relative positions and intensities of several reflections. In BPIII most of the diffraction intensity is concentrated in a single wideband maximum, so that no unambiguous conclusions can be drawn about the presence of other weaker reflections. The theoretical models (1)–(4) also imply that these reflections

should either be absent [in the case of (1)] or that their intensities should be considerably lower than the primary diffraction maximum [in the case of (2)–(4)]. For this reason, no one has succeeded yet in determining the structure of the BPIII phase, despite considerable effort, and it has become clear that other methods must be found to distinguish experimentally which of the structures predicted by the theories (1)–(4) is correct.

The goal of this paper is to identify which of the structures (1)–(4) is correct by estimating the magnitude of the duplication index N (i.e., the number of wave vectors τ with a given $|\tau|$) for the primary diffraction maximum of the foggy phase. This can be done by orienting BPIII in an electric field. Previous workers[24,25] have shown that in materials with a negative dielectric anisotropy $\Delta\varepsilon$ the intensity of the diffraction increases in an electric field, while the half-width of the diffraction curve decreases. In the present work the transmission spectra were measured along with the optical diffraction for various orientations of the scattered wave vector with respect to the direction of the electric field and the change in the index of refraction induced by the electric field. The transmission spectra were found to be much altered by an electric field. The experimental data were used to reconstruct the orientational distribution function of the wave vectors τ connected with diffraction in BPIII. A comparison of the experimental data with existing theoretical representations revealed that the results are incompatible with the double-twist cylinder, body-centered cubic, or icosahedral ordering models, but can be explained using a model with simple cubic local order.

2. SAMPLES AND EXPERIMENTAL METHOD

The measurements were made on a mixture of the chiral material S811 (30% by weight) and the nematic liquid crystal EN18 (70% by weight).[25] The mixture formed three blue phases: BPI (41.80–42.15 °C), BPII (42.15–42.32 °C), and BPIII (42.32–42.45 °C), and possessed negative local dielectric anisotropy. The samples were placed in planar optical cuvettes with fixed slits having widths between 16 and 30 μm. A transparent conducting coating was deposited on the inner surface of the cuvettes. For liquid crystals in the BPII and BPIII phases, some of the diffraction measurements were made on films with thicknesses of ≈ 1 μm, deposited on a glass surface treated with polyvinyl alcohol. After this type of treatment, BPII is oriented with its [100] direction perpendicular to the surface. The opposite surface of the film was free. The temperature was maintained and regulated to within 0.005 °C. In the electric-field measurements, a sinusoidal electric field at $4 \cdot 10^3$ Hz was used in order to prevent electrodynamic effects. The transmission spectra were measured with the electric field directed parallel to the light passing through the sample. Diffraction measurements were made at a fixed diffraction angle ($\varphi = 90°$) and various directions of the scattering wave vector τ with respect to the direction of the electric field. The spectra given in this paper consist of the true spectrum of light passing through the sample or diffracted by it normalized by the lamp spectrum. The change in refractive index in an electric field was measured according to the shift of interference maxima in the
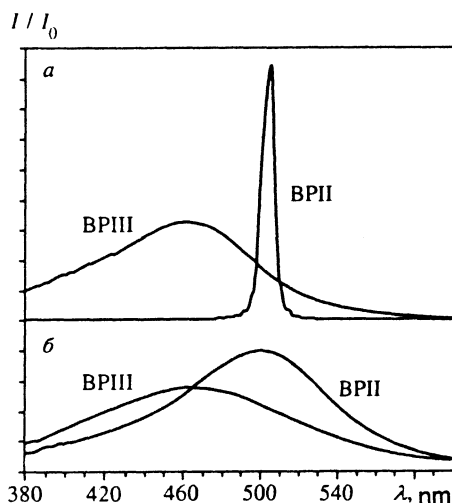

FIG. 1. Diffraction spectrum of BPII (42.28 °C) and BPIII (42.40 °C): a) sample thickness 16 μm, b) liquid-crystal film with thickness 1 μm on a glass surface.

reflection spectrum from a plane parallel cuvette containing the blue phase. The quantity Δn was measured outside the regions of diffraction and absorption by the sample to an accuracy of 10^{-4}.

3. EXPERIMENTAL RESULTS AND DISCUSSION

In the absence of an electric field, the diffraction (Fig. 1a) and transmission (Fig. 2) spectra for the samples studied here had a form typical of the BPIII phase:[22–24,26] the wideband diffraction maximum of the BPIII phase was shifted towards the short-wavelength region of the spectrum relative to the narrow (100) reflection of BPII, and the transmission spectrum was structureless and varied smoothly in intensity over a wide spectra interval. As proposed earlier,[22,23] the wideband nature of the diffraction from BPIII is associated with a short coherence length $\delta L \approx 0.5-2\mu$m. The mea-

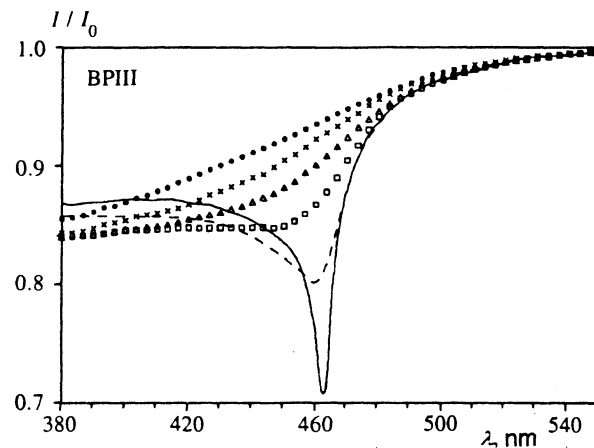FIG. 2. Transmission spectrum of BPIII (42.40 °C) in an electric field. Electric field intensities (in V/μm): 0.00 (●), 1.92 (×), 2.54 (△), 2.87 (□), 3.06, and 3.12. The sample thickness was 31.5 μm.
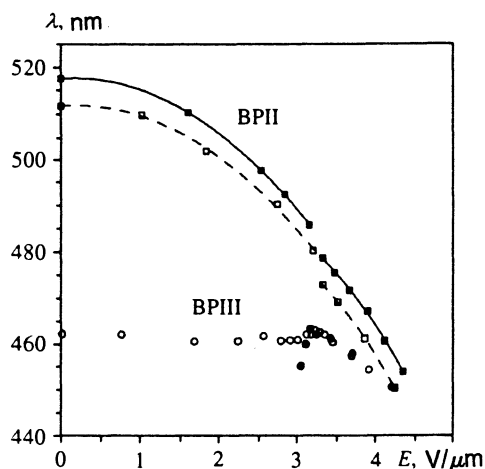
FIG. 3. Spectral functions for the position of the (100) reflection in the BPII phase at 42.20 °C (■) and 42.24 °C (□). (O) marks the diffraction maximum for BPIII, (●) marks the transmission spectrum maximum for BPIII.

FIG. 4. Diffraction spectra of BPIII and BPE (42.40 °C) in an electric field. Electric field intensities (in V/μm): 0.00 (●), 1.67 (x), 2.79 (△), 3.01 (□), and 3.12 (--). The intensity of the BPIII spectra (—, E=3.18 V/μm) and BPE spectra (—, E=3.91 V/μm) are decreased by a factor of 20.

sured diffraction spectra for films of liquid crystal (Fig. 1b) confirm this fact. For a film thickness $d \sim 1\,\mu$m, the width of the diffraction band for BPII is the same as that of BPIII in a bulk sample. The diffraction for the BPI phase has an analogous form. For BPIII the diffraction is somewhat broader than that of BPII (by $\sim$ 30%). This additional broadening is apparently associated with local disorder in BPIII, which is preserved in the film. It should be noted that samples with $d \sim \delta L$ exhibit the same sequence of phase transitions (BPI–BPII-BPIII) as bulk samples; in the BPII–BPIII transition the same discontinuous change in the position of the diffraction maximum takes place. In particular, this implies that the BPII–BPIII transition is not associated with a simple shrinking of the region of coherent scattering or formation of small-sized domains, but rather is a more complicated phenomenon.

There are significant differences in the way the position of the band depends on electric field in the BPIII and BPII (BPI) phases. In the BPI and BPII phases, a considerable shift in the bands is observed. This shift is the same in both the transmission and diffraction spectra, and is connected with electrostriction. In BPII, the maximum of the transmission spectrum initially shifts towards the long-wavelength side in an electric field (Fig. 3). This shift is connected not with electrostriction, but rather with a transformation of the distribution function of the planes in an electric field. In BPIII, the position λ_0 of the band in the diffraction spectrum is determined by the interplanar spacing; in contrast to BPII (BPI), it remains practically constant over a wide interval of fields. In our view, the constancy of λ_0 at low fields is associated with the small size of the BPIII domains and thermal reorientation of domains that are not fixed by the surface of the cuvette. In the blue phases, the time for establishing the equilibrium value of the lattice constant[24] $\tau_e \sim 10$ s in an electric field; furthermore, the signs of the electrostriction are opposite for planes perpendicular and parallel to the field direction. Characteristic times for formation and decay of domains, and also their reorientation time τ_r, are smaller
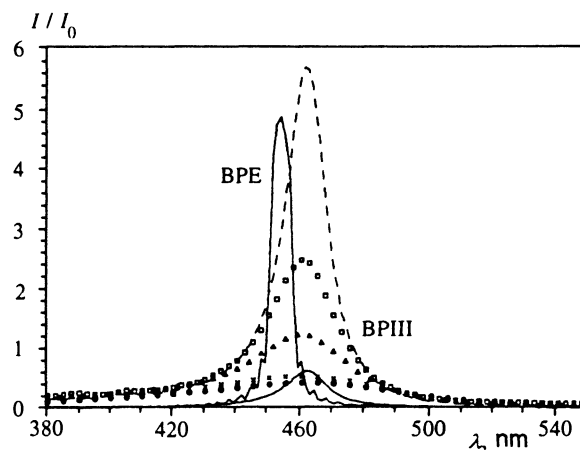
than 100 μs, i.e., $\tau_r \ll \tau_e$, so that the lattice of the blue phase cannot be transformed. As the field and the size of the domain L increase, the reorientation time increases $(\tau_r \sim L^3)$, and for $\tau_r \gtrsim \tau_e$ we should observe a distortion of the cells in the electric field. A considerable ultraviolet shift in the diffraction band is observed for $L \sim 10\,\mu$m, i.e., when the sizes of the domains have increased by an order of magnitude.

A significant change in the transmission spectrum $T(\lambda)$ of the foggy phase was observed in an electric field (Fig. 2). This change in the spectrum was reversible in character; in intermediate fields, the spectra did not appear to be superpositions of the initial spectrum (E=0) and the spectra at high values of the field. The diffraction measurements were made in parallel with the measurements of the transmission spectrum in an electric field (Fig. 4). Previous measurements have shown that a transition occurs in an electric field from the blue phases to a three-dimensional hexagonal phase or to a distorted BPII (BPE phase).[24,27] Figure 3 shows the electric field dependence of the position of the (100) reflection from BPII and the diffraction maximum from BPIII. The phase transition to BPII takes place at E_c=3.25 V/μm, and is accompanied by a discontinuous change in the cell parameter. In the BPIII phase, no discontinuous change was observed in the position of the diffraction band λ_0, although a change took place in the function $\lambda_0(E)$ at $E \sim E_c$. The feature at $E \sim E_c$ could also be associated with a phase transition in BPIII; therefore, in what follows only fields $E < E_c$ will be used to interpret the transmission spectra.

The change in the transmission spectra of BPIII in an electric field could have two causes: a change in the half-width of the diffraction peak (i.e., growth of the region of coherent scattering), or in the orientation of BPIII in the electric field. In phases with long-range translational order (BPI, BPII) the shape of the transmission spectrum depends on the orientation of crystallites with respect to the light incident on the sample. In a polycrystalline sample, each reflection corresponds to a step in the transmission spectrum. A discontinuous decrease in the intensity of the light trans-
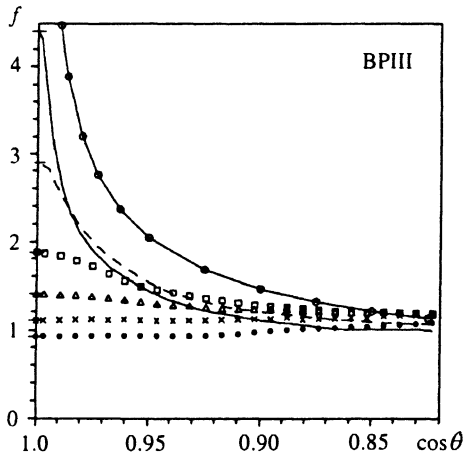
FIG. 5. Orientational distribution function of planes in BPIII. Electric field intensities (in V/μm): 0.00 (●), 1.92 (x), 2.54 (△), 2.87 (□), 3.06 (− −), and 3.12 (—). (—●—) shows the orientational distribution function $f_c(\cos\theta)=(2/\pi)(1-\cos^2\theta)^{-0.5}$.

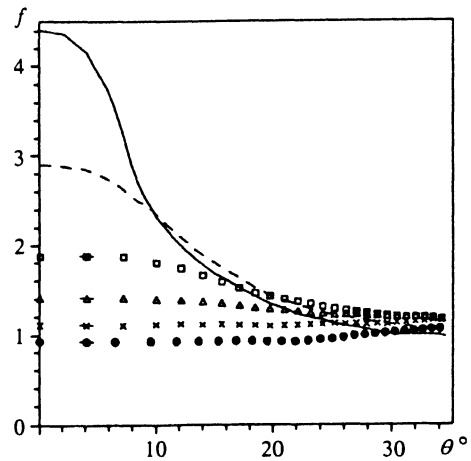FIG. 6. Orientational distribution function of planes in BPIII. Electric field intensities (in V/μm): 0.00 (●), 1.92 (x), 2.54 (△), 2.87 (□), 3.06 (− −), and 3.12 (—).

mitted through the sample takes place for $\lambda_0=2nd$ (where d is the interplanar spacing and n is the refractive index), i.e., λ_0 is the maximum wavelength for which diffraction is possible. λ_0 is realized for planes perpendicular to the direction of the light beam. The spectral function $T(\lambda)$ for $\lambda<\lambda_0$ is determined by a superposition of Bragg reflections with $\lambda=2nd\cos\theta$, where θ is the angle the light beam makes with the normal to the plane. It should be noted that for wavelengths not too far displaced from the steps ($\lambda/\lambda_0 > 0.8$) the transmission spectrum[28] $T(\lambda/\lambda_0)\approx f(\lambda/\lambda_0)g(\lambda/\lambda_0)$ as a function of $\cos\theta=\lambda/\lambda_0$ turns out to be almost proportional to the orientational distribution function for the planes $f(\cos\theta)$, since[28] $g(\lambda/\lambda_0)=(\lambda/\lambda_0+\lambda_0/\lambda)^2$ is a weak function of λ. For a polycrystalline sample with unoriented crystallites $f(\cos\theta)=$ const, the smearing, of the steps is determined by the width of the reflections or the size of the crystallites.

In the translationally disordered BPIII phase, the transmission spectrum is a superposition of broad diffraction bands is associated with the increased transmission in the range $\lambda<\lambda_0$ due to the reflections that arise from the long-wavelength side. The changes in the transmission spectrum that take place in this case are largely analogous to those that occur when spectral measurements are made with a detector having a finite-width instrument function. In order to estimate the effect of the electric field on the distribution function in BPIII, the decrease in reflection intensity for $\lambda<\lambda_0$ was compensated using the long-wavelength wing of the transmission spectrum:

$$f(\cos\theta)=f(\lambda/\lambda_0)\sim T(\lambda/\lambda_0) \quad [\text{for } \lambda<\lambda_0]$$

$$+T(2-\lambda/\lambda_0) \quad [\text{for } \lambda>\lambda_0] .$$

The distribution function obtained in this way is shown in Figs. 5, 6. The wavelength λ_0 was taken from diffraction measurements (Fig. 3). Taking into account the dependence of $g(\lambda/\lambda_0)$ on λ did not lead to an appreciable change in $f(\cos\theta)$. The distribution function for $E=0$ was found to be

almost independent of $\cos\theta$, which we should expect for orientationally disordered BPIII. An insignificant increase in $f(\cos\theta)$ as $\cos\theta$ decreases is apparently related to an increase in the refractive index and Δn in the short-wavelength spectrum close to the intrinsic absorption edge of the material, which is not taken into account in obtaining $f(\cos\theta)$.

Before analyzing how the distribution function changes in an electric field, we must make certain that the finite width of the diffraction band does not significantly distort the measured function $f(\cos\theta)$. Figure 7 shows the dependence on E of the integrated diffraction intensity $I(E)$ ($\varphi=90°$; $\theta=0°$; $\theta=10°$; $\theta=14°$) and the function $f(E)$ obtained from the transmission spectra. The qualitative difference between the function $I(E)$ at $\theta=0$ and $I(E)$ for finite values of θ is associated with the change in the angular distribution function in the electric field. It is impossible to compare the diffraction intensities for different values of θ, because the
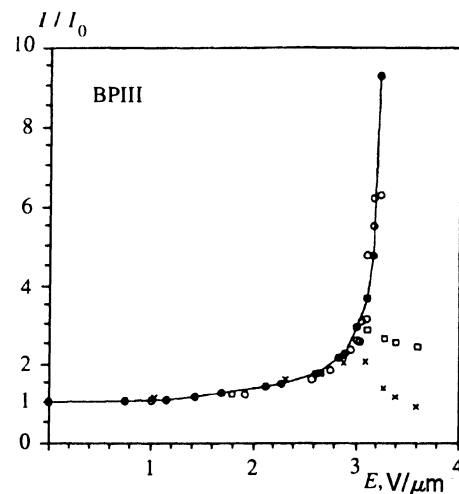

FIG. 7. Electric field intensity dependence of the integrated diffraction intensity of BPIII: $\theta=0°$ (●), $\theta=10°$ (□), $\theta=14°$ (x). (○) is the function $f(E)$ for $\theta=0°$.

reflection of incident and diffracted light from the surface of the cuvette and thermostatic apparatus changes. All the data in Fig. 7 were normalized to the corresponding values in zero field. The change in the integrated intensity of the diffraction band is proportional to $f(E)$ at the corresponding θ. The agreement between $f(E)$ and $I(E)$ implies that the finite width of the diffraction curves does not introduce to appreciable errors in $f(\cos\theta)$; thus, the curves in Figs. 5, 6 correctly reflect the way the distribution function changes in an electric field.

The possible structures of BPIII were analyzed using the measured distribution functions. For structures characterized by the set of reciprocal lattice vectors $\mathbf{r}$ we compared $f(\cos\theta)$ with the values implied by the models (2)–(4). The average value of the distribution function (Fig. 5) for orientationally disordered structures is taken to equal 1, i.e., $\int f(\cos\theta)d(\cos\theta)=2$, $(0 \leqslant \theta < \pi)$.

For the body-centered cubic structure O^5, the long-wavelength reflection is associated with the (110) lattice planes. According to the calculations of Refs. 3, 9, this reflection should be the most intense. If the model (2) with a local O^5 structure is correct, the maximum in the distribution function should be associated with one of the (110) lattice planes, while diffraction from the other planes should give a maximum for $\cos\theta=0.5$ or $\cos\theta=0$ (i.e., $\theta=60°$ or $\theta=90°$). For this structure, the duplication index is $N_{110}=12$, i.e., when the structure is orientationally ordered the integrated intensity in the neighborhood of one peak should not exceed 0.167 (2/12). The distribution function for the disordered sample $(E=0)$ has the same area from $\cos\theta=1$ to $\cos\theta=0.833$. It is clear from Fig. 5 that for structures with orientational order the areas under the distribution functions in the range of $\cos\theta$ connected with one (110) plane actually exceeds 0.167. This result therefore contradicts the cubic O^5 model. Furthermore, the maximum of the distribution function for $\cos\theta=1$ cannot be associated with any other reflections of O^5, because the duplication indices for these reflections are greater than 12.

Let us compare the experimental data on the foggy phase with the characteristics of a quasicrystal structure. For the quasicrystal model the same considerations as for the O^5 model apply. The icosahedral structure should have reflections at wavelengths $\lambda_i/\lambda_3=1.62$, 1.05, 1, 0.85, 0.73, 0.71, 0.62, 0.59, 0.58 ($\varphi=90°$) (where λ_3 is the wavelength of the corresponding reflection from the icosahedral edges).[11–14] According to the theoretical calculations of Refs. 11–14, the primary contribution to the diffraction comes from harmonics of the order parameter with wave vectors from the center to a vertex of the icosahedron ($\lambda_2/\lambda_3=1.05$) and along its edge (λ_3). For an icosahedron, the reflection with smallest duplication index $N=12$ is λ_2 (for the wave vector λ_3 the duplication index $N=30$). The same arguments used for O^5 structures lead us to conclude that the distribution function does not correspond to the quasicrystal model. An additional argument against the icosahedral ordering of BPIII is the presence of only one maximum in the distribution function over the measured interval of θ (Fig. 6). The angle between the most intense structural reflections for icosahedral ordering (λ_2 and λ_3) is 32°. Although an additional maximum

does appear at the edge of the range of angles θ accessed by these measurements, we can say with confidence that in the ordered phase there is no second maximum in $f(\cos\theta)$ like the one at $\theta=0$.

In the double-twist cylinder model (1) the duplication index N cannot be used to analyze the experimental data, since the wave vectors are along the cylinder diameters and are distributed continuously in the plane perpendicular to its axis. In this case, the data can be compared with the model by comparing the shape and absolute values of the experimental and theoretical distribution functions. For materials with a negative local dielectric anisotropy along the cylinder axis, the dielectric permittivity is smaller than in the perpendicular direction. Thus, the electric field should lead to an orientation of the cylinder axes in directions perpendicular to the field.[24] For an orientationally ordered sample, the distribution function for wave vectors $f_c(\cos\theta)$ $=(2/\pi)(1-cos^2\theta)^{-0.5}$ remains nonzero for any θ. This distribution function is shown in Fig. 5. In the range of θ where the measurements were made, orientational ordering should lead to an increase of $f(\cos\theta)$. In fact, however, this increase is observed for only small E; further increases in E reduce $f(\cos\theta)$ over a large portion of the angular interval of θ. This result contradicts the double-twist cylinder model.

Let us consider the agreement between the model of a simple cubic structure and the experimental data. In an electric field, O^2 domains are oriented in the direction (100) along E, i.e., the peak of the distribution function for $\theta=0$ should be associated with one of the (100) planes. Other maxima of the distribution function are found at $\theta=90°$. The duplication index is $N_{100}=6$, i.e., for orientational ordering the area under the distribution function peak should not exceed 0.333 (2/6). From Fig. 5 it is clear that the experimental data do not contradict a cubic O^2 model.

Thus, of the possible structures (1)–(4) for BPIII discussed in the literature, only simple-cubic ordering (model (3)) corresponds to the measured distribution functions. The possibility that BPIII phases could arise with the orderings (2)–(4) was based on the Landau theory of phase transitions (models (2)–(4)), and on an energy minimum associated with locally chiral ordering for molecular double-twist cylinders (model (1)). However, these experimental data on the distribution function together with the structure O^2 do not formally contradict a model of BPIII with a cholesteric structure of small-size domains. Although this model of BPIII has no theoretical basis, let us discuss it as another possibility.

There is many experimental data that contradict cholesteric ordering: (a) Figure 8 shows the change in refractive index $\Delta n/n$ in an electric field. For $E \approx 5$ V/μm the function $\Delta n(E)$ is analogous to functions observed previously in blue phases with three-dimensional ordering.[29] At an electric field $E \approx 5$ V/μm a phase transition is observed to the cholesteric phase; this field value is considerably larger than the field $E=3.25$ V/μm at which the structure is already ordered and the region of coherence corresponds to the sample size. (b) An additional argument against cholesteric ordering in BPIII is the field dependence of the position of the diffraction peak: it is well known that an electric field increases the pitch of
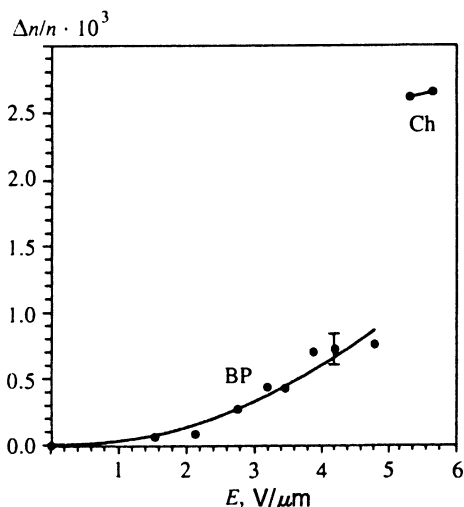
FIG. 8. Change in the index of refraction $\Delta n/n$ in an electric field for BPIII and at the phase transition to a cholesteric.

the cholesteric spiral, whereas we observed a decrease in λ_0 up to the transition at $E \approx 5$ V/μm.

The foggy phase (BPIII) possesses a number of unusual macroscopic and microscopic characteristics, and for the last ten years it has been one of the most puzzling of the liquid-crystal phases. The results of this paper show that an electric field causes a profound change in the transmission spectrum, which is associated with the orientational ordering of BPIII. Possible structures for BPIII were analyzed by comparing the orientational distribution function of wave vectors τ with existing models of BPIII. Of the proposed theoretical models, only simple cubic ordering agrees with the results obtained here. However, the reason why a structure possessing only local cubic order forms near the transition temperature to an isotropic liquid cannot be understood without further theoretical and experimental investigation.

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