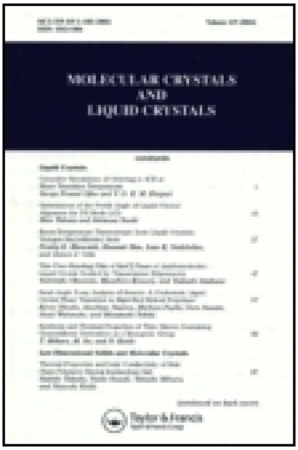

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Molecular Order and Surface Tension for the Nematic–Isotropic Interface of MBBA, deduced from Light Reflectivity and Light Scattering Measurements†

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Abstract—Light reflectivity measurements show that the molecules of MBBA lie parallel to the nematic–isotropic interface, that their orientations in the absence of any external field are correlated in the plane of the interface over distances greater than the optical wavelength and allow a study of the dynamics of the effect of a magnetic field on these molecules. The spectrum analysis of the light scattered by the interface yields the value of the surface tension. This experimental value is used to estimate the correlation length in the isotropic phase.

In our experiments the nematic–isotropic interface of MBBA (methoxy-benzilidene butyl aniline) was obtained by placing the liquid sample in a vertical temperature gradient. We used a small oven formed of three parts (Fig. 1): a copper block at the top,

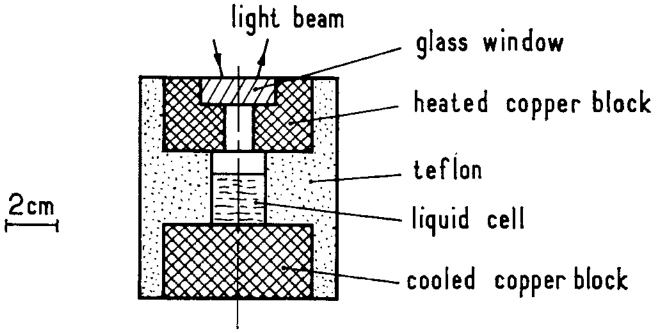

Figure 1. Schematic drawing of the oven used to obtain the nematic–isotropic interface of MBBA.

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† Presented at the Fourth International Liquid Crystal Conference, Kent State University, August 21–25, 1972.

heated to around 100 °C and temperature controlled within ± 0.3°, a teflon spacer surrounding the glass cell which contains the liquid and another copper block at the bottom cooled by a circulation of water. The nematic–isotropic transition temperature of the MBBA we have used, was $t_c = 45.5\,°\mathrm{C}$. The oven was standing on a small table which could be tilted around two orthogonal horizontal directions in order to adjust the temperature gradient in the vertical direction. We found that it was sometimes difficult to keep the interface rigorously plane (i.e., to avoid planeity defects smaller that 1/10°). The reason may be that the surface of constant temperature ($t = t_c$) differs slightly from an horizontal plane so that thermal and mechanical equilibrium cannot be reached simultaneously.

1. Light Reflectivity Measurements

1) MEASUREMENTS IN AN HORIZONTAL MAGNETIC FIELD

In a previous paper⁽¹⁾ we described how light reflectivity measurements can give information about the molecular order at an interface, and we applied this method to the free surface of a nematic liquid crystal. Let us recall briefly the principle of the method: if θ is the angle of the molecules of the nematic phase with respect to the interface and if an horizontal magnetic field **H** is applied to the liquid, the equilibrium configuration is the following: the molecular axes project themselves on the horizontal plane along **H** in the whole sample, but the tilt angle is θ at the interface and 0 far beneath. The thickness of the distorted region is of the order of the magnetic correlation length

$$\xi_M \sim \sqrt{\frac{K}{\chi_a}}\frac{1}{H}$$

(K elastic constant, χ_a anisotropy of the magnetic susceptibility). Since the wavelength λ of the light is small compared to ξ_M, the alignment at the interface is the only one which is important for reflection. By using a laser beam nearly normal to the interface, with a plane polarization **E** which can be rotated, we obtain a reflected beam having an intensity which is proportional to the Fresnel reflectivity coefficient ρ. One has: $\rho = [(n - n_I)/(n + n_I)]^2$ where

n_I is the refractive index of the isotropic phase and n is the refractive index of the nematic phase in the direction of **E**: i.e., $n = n_0$ (ordinary index) when **E** is perpendicular to **H**, and

$$n = n_\theta = n_0\sqrt{(1+tg^2\theta)/(n_0^2/n_e^2+tg^2\theta)}$$

when **E** is parallel to **H**, n_e being the extraordinary index.

The liquid sample was placed in an horizontal magnetic field of 3000 Gauss. We used a laser ($\lambda = 6328\,\text{Å}$) on which a rotator of polarization (Spectra Physics) was mounted. The angle of incidence was 3°. We measured the ratio of the reflectivities, for a polarization **E** parallel ($\rho_\parallel$) and perpendicular ($\rho_\perp$) to **H**, to be:

$$\rho_\parallel/\rho_\perp = 4.0 \pm 0.4$$

Taking the values obtained by Haller⁽²⁾ for the refractive indices at the transition temperature $t = t_c$:

$$n_0 = 1.5708$$
$$n_e = 1.6778$$
$$n_I = 1.6048$$

we derive $\rho_\parallel/\rho_\perp = 4.31$ for $\theta = 0$. This value is compatible with our experimental value, and we feel that we can conclude that the molecules lie parallel to the interface. More precisely, taking into account our experimental uncertainty we deduce

$$0° \leqslant \theta \leqslant 13°$$

2) MEASUREMENTS IN THE ABSENCE OF A MAGNETIC FIELD

In order to verify that the molecular order at the nematic–isotropic interface is not produced by the horizontal magnetic field, we studied also the properties of the reflected light when the sample was in a zero magnetic field. We then found that the reflected intensity was independent of the direction of the polarization **E** and that *the reflected light was depolarized.* As shown below this proves that the molecules project themselves on the plane of the interface in a random way and that the correlation length l in this plane is such that $L \gg l \gg \lambda$, L being the dimension of the illuminated region. This depolarization effect is exactly similar to the one expected for reflection from a random arrangement of many birefringent crystal plates with dimensions much larger than a wavelength.

Let us now justify this assertion and compute what the depolarization ratio is expected to be. Let α be the angle between the incident electric field **E**, and the projection on the interface of the molecular axes, for a small element of the interface having dimensions d smaller than the correlation length l.

We first consider the situation $l,\ d \gg \lambda$. We can apply the usual laws of reflection to a given surface element. For the components of the field $\mathbf{E}_r(i)$ reflected by an element (i) with a direction parallel $(0x)$ or perpendicular $(0y)$ to **E**, we find:

$$\frac{E_{rx}(i)}{E} = r_{\parallel} \cos^2\alpha + r_{\perp} \sin^2\alpha \tag{1}$$

$$\frac{E_{ry}(i)}{E} = (r_{\parallel} - r_{\perp}) \sin\alpha \cos\alpha \tag{2}$$

with $\quad r_{\parallel} = (n_\theta - n_I)/(n_\theta + n_I) \quad$ and $\quad r_{\perp} = (n_0 - n_I)/(n_0 + n_I)$

Two different elements are further than a wavelength apart, so the corresponding reflected fields add uncoherently: the total reflected intensity is the sum of the intensities $|\mathbf{E}_r(i)|^2$ reflected by each surface element. If $L \gg l$, α varies randomly over the illuminated region; this case is schematized on Fig. 2(a). We finally obtain that the reflectivity coefficient is that of a small element averaged over the molecular orientation (α):

$$\rho_x = \langle (r_{\parallel} \cos^2\alpha + r_{\perp} \sin^2\alpha)^2 \rangle = \tfrac{1}{8}(3r_{\parallel}{}^2 + 3r_{\perp}{}^2 + 2r_{\parallel} r_{\perp})$$

$$\rho_y = \langle r_{\parallel} - r_{\perp})^2 \sin^2\alpha \cos^2\alpha \rangle = \tfrac{1}{8}(r_{\parallel} - r_{\perp})^2$$

The variation of ρ_y and of the depolarization ratio ρ_y/ρ_x are represented in Fig. 3 versus the angle θ. We can see that if $\theta = 90°$ (i.e., the molecules at the interface are vertical) the reflected light has the same polarization as the incident one, as expected.

When $l \gg L$, or when an horizontal magnetic field is applied, the molecular alignment does not vary over the illuminated region and the average over α has not to be performed (see Fig. 2(b)). We then find that the plane of polarization of the reflected wave rotates with respect to that of the incident one. This result simply arises from the difference in reflection efficiency for a wave polarized perpendicular or parallel to the direction of molecular alignment. The cases

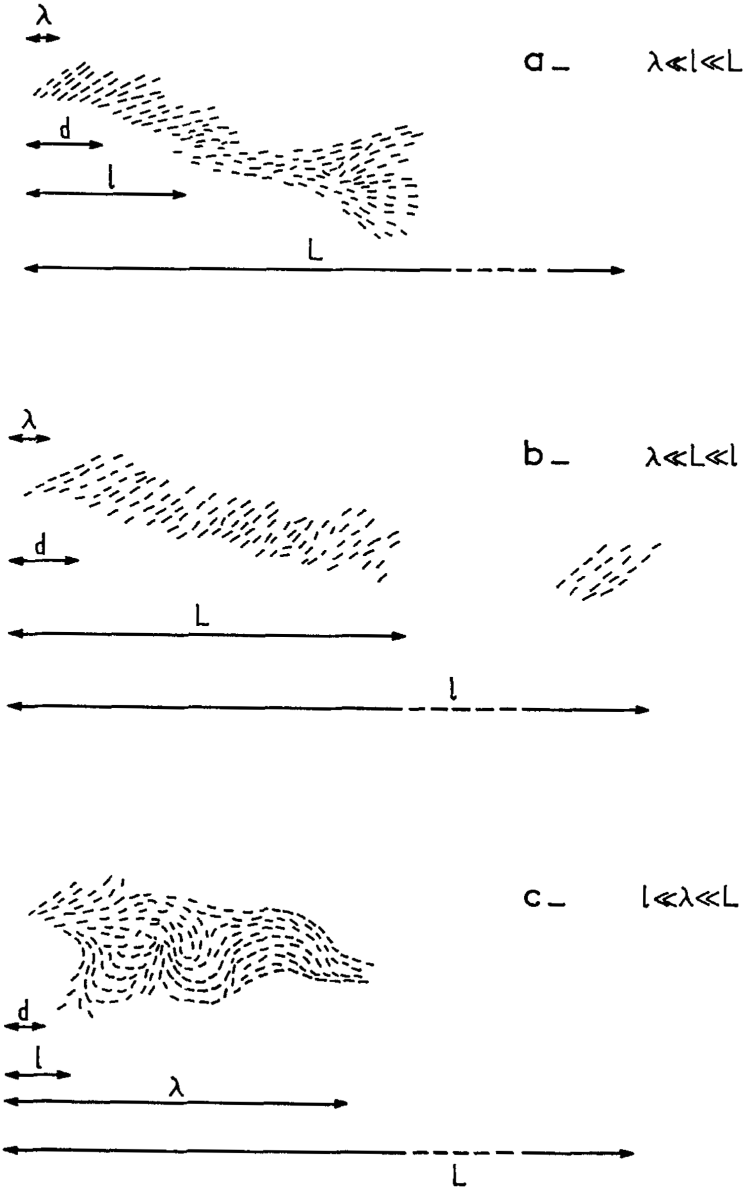

Figure 2. Possible arrangements of the molecules in the plane of the interface Case *a* corresponds to the situation encountered for MBBA in zero field: an incident polarized light beam is depolarized by reflection. Case *b* corresponds to the situation observed for MBBA in the presence of an horizontal magnetic field: there is a rotation of the plane of polarization by reflection, if the incident beam is polarized. In case *c*, light is reflected like from an isotropic medium.

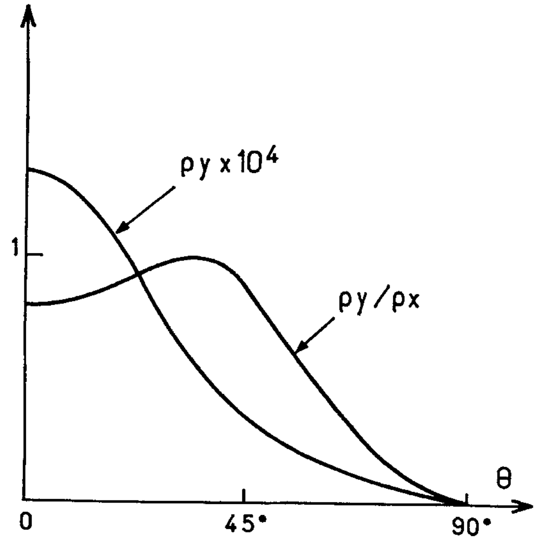

Figure 3. Theoretical variations of ρ_y and of the depolarization ratio ρ_y/ρ_x versus the angle θ of the molecular axes with the horizontal plane, in the case $L \gg l \gg \lambda$ encountered in zero magnetic field.

$\alpha = 0$ or $\pi/2$ when the angle of rotation becomes zero and the ratio of the reflectivity coefficients is 4.3 for $\theta = 0$, has been considered in the preceding paragraph.

We now turn to the opposite case $l \ll \lambda$ (see Fig. 2(c)). Expressions (1) and (2) represent the contribution to the reflected field of the aligned molecules of a small element of dimension $d < l \ll \lambda$. Now the fields coming from different surface elements, less than a wavelength apart, add coherently. Suppose we perform the sum over a fraction of the surface having dimensions D such that $d \ll D \ll \lambda$. Since α varies randomly over this surface, we obtain $\bar{E}_{rx}/E = \langle r_{\parallel} \cos^2 \alpha + r_{\perp} \sin^2 \alpha \rangle$ and $\bar{E}_{ry} = 0$. It follows that the wave reflected by the entire surface is polarized as the incident wave, whatever θ is. The medium looks like an isotropic medium having the reflectivity coefficient $\rho = \frac{1}{2}(r_{\parallel}{}^2 + r_{\perp}{}^2)$.

We conclude from this discussion that our results prove that the correlation length is greater than the optical wavelength but smaller than L, which is of the order of 5 mm in our experiment. We plan to reduce L in order to try to measure l.

The experimental value of the depolarization ratio is $\rho_y/\rho_x = 1 \pm 0.3$ which is compatible with (see Fig. 3).

$$0^\circ \leqslant \theta \leqslant 55^\circ$$

This determination is very rough because of the flat shape of the curve giving ρ_y/ρ_x versus θ, for small values of θ. But a more precise value of θ can be obtained by measuring ρ_y. In fact we did not measure experimentally the absolute value of ρ_y, but the ratio $\rho_y/\rho_\perp$ of the depolarized reflected intensity in zero field and the reflected intensity in an horizontal field **H** perpendicular to **E**. We obtained $\rho_y/\rho_\perp = 1.1 \pm 0.1$ and therefore $\rho_y = (1.3 \pm 0.1) \cdot 10^{-4}$. This corresponds to (see Fig. 3):

$$0 \leqslant \theta \leqslant 16^\circ$$

Within experimental error we can conclude that the molecules lie parallel to the interface even in the absence of an external magnetic field.

Remarks

1) In the preceding discussion we have supposed the correlation length in the direction perpendicular to the interface, ξ_0, larger than λ in zero field, so that the alignment at the interface is the only one important for reflection. Other cases seem excluded since the condition $\xi_0 \lesssim \lambda$ would lead to a smaller depolarization ratio (ρ_y/ρ_x goes to zero in the extreme case $\xi_0 \ll \lambda$).

2) The intermediate cases $l \approx \lambda$ or $l \approx L$ would give theoretical results for ρ_y and ρ_y/ρ_x versus θ which would lie below the values drawn on Fig. 3. In view of the experimental results, they look unlikely.

3) We can also consider the case when the horizontal projections of the molecules are not completely random over the illuminated region, but make an angle β (such that $0 \leqslant \beta \leqslant \beta_L < 90^\circ$) with a given direction. This situation can occur when some weak force, a radial thermal gradient for instance, produces a preferential direction in the plane of the interface but creates an uncomplete alignment of the molecules. One can show that in these conditions, whatever the value of l, the reflected intensity should exhibit a variation with the direction of **E**.

3) Dynamics of the Orientation and Disorientation of the Molecules

As the reflected light on the nematic–isotropic interface is depolarized in a zero magnetic field, and polarized when we apply a magnetic field, it is easy to observe the dynamics of orientation and disorientation of the molecules at the interface by studying the variation of the depolarization ratio of reflected light, for an incident beam polarized parallel to the applied magnetic field. If we plot this ratio as a function of time, we obtain the curve of Fig. 4.

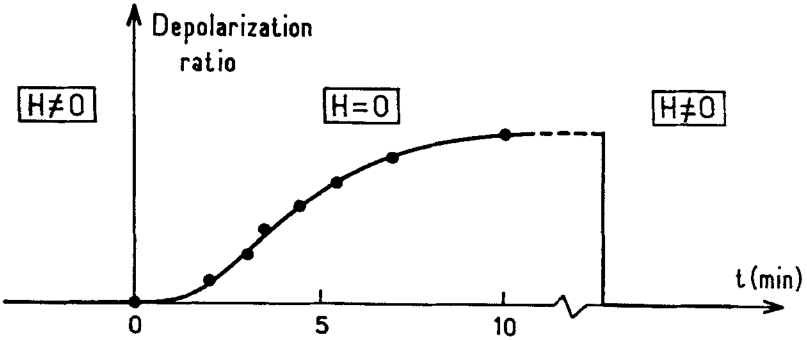

Figure 4. Variation of the depolarization ratio of the light reflected by the nematic–isotropic interface of MBBA versus time, when the horizontal magnetic field is cut off at time $t = 0$ and applied again, after the limit value of the depolarization ratio ρ_y/ρ_x has been reached.

The characteristic time for disorientation is of the order of 5min. On the other hand, when one applies the field again, the orientation looks instantaneous.

Presently we are studying the effect of a vertical magnetic field, in order to see if the molecules near the surface can be tilted.

2. Light Scattering Experiments

We have studied the spectrum of the light scattered by the nematic–isotropic interface using heterodyne spectroscopy techniques.

On a liquid interface thermal motion produces small asperities. The liquid interface is submitted to a capillary restoring force†

† In the range of wave numbers of interest in the present experiment, the gravity forces can be omitted owing to the very small difference of density between the two phases.

and its motion is damped by viscosity. The statistical evolution of the fluctuations can be described by the laws of hydrodynamics. The light scattered by a surface vibration mode of wave vector **q** is concentrated in a well defined direction simply related to **q** by expressing momentum conservation in the surface plane during the scattering process. Experimentally we used a diaphragm to select from the light scattered by fluctuations that part having a wave vector well defined both in direction and in modulus. The light is then received on a photomultiplier and we perform the frequency analysis of the photocurrent power spectrum.

One could expect the spectrum to be anisotropic, and to depend on the relative orientation of **q** with respect to the mean orientation of the molecules $\mathbf{n}_0$, for the two following reasons: the anisotropy of the viscosity as in the case of the free surface of a nematic liquid,(3,4) and the anisotropy of the surface tension which may result from the anisotropy of elastic forces (this was negligible in the case of the free surface(4)). In our experiments, we fixed $\mathbf{n}_0$ by applying a horizontal magnetic field **H** of around 3000 Gauss. As the preferential orientation of the molecules at the interface is horizontal, $\mathbf{n}_0$ is parallel to **H** in the whole sample.

1) Derivation of the Theoretical Spectrum

The hydrodynamic equations for the present problem can be written

$$\operatorname{div} \mathbf{v} = 0, \quad \rho \frac{\partial v_i}{\partial t} = \frac{\partial \sigma_{ij}}{\partial x_j} - \frac{\partial p}{\partial x_i} \tag{3}$$

where **v** is the velocity of the fluid, p the pressure, ρ the density and σ_{ij} the viscous stress tensor:(3)

in the nematic phase

$$\sigma_{ij}^N = \eta_3 n_i n_j A_{kp}^N n_k n_p + 2\eta_2 A_{ij}^N + 2(\eta_1 - \eta_2)(n_i n_k A_{kj}^N + n_j n_k A_{ki}^N),$$

whereas in the isotropic phase $\sigma_{ij}^I = 2\eta A_{ij}^I$,

with $$A_{ij} = \frac{1}{2}\left(\frac{\partial v_j}{\partial x_i} + \frac{\partial v_i}{\partial x_j}\right);$$

n_i is the i-component of $\mathbf{n}_0$, η_1, η_2, η_3 and η are the shear viscosities of the nematic and the isotropic phases.

Introducing the Fourier transforms of the hydrodynamic variables with respect to $\mathbf{r}(x, y)$ and t:

$$\tilde{v}_i(\mathbf{q}, z, \omega) = \int d^2\mathbf{r} \int_{-\infty}^{+\infty} \mathrm{d}t\, v_i(\mathbf{r}, z, t)\, \mathrm{e}^{i\omega t}\, \mathrm{e}^{i\mathbf{q}\mathbf{r}},$$

and taking $0x$ parallel to $\mathbf{q}$, the plane $x0y$ as the equilibrium position of the interface, the solution of Eq. (3) for $\tilde{v}_z$ is of the form:

$$\tilde{v}_z = \alpha\, \mathrm{e}^{m_1 z} + \beta\, \mathrm{e}^{m_2 z}$$

with $m_1 = -q$, $m_2 = -q\sqrt{1 + (i\omega\rho/\eta q^2)}$ in the isotropic phase. In the nematic phase m_1 and m_2 are complicated functions of ω.(3) But one can verify that the real parts of m_1 and m_2 which are positive in the nematic phase, negative in the isotropic phase, are of the order of q. This leads to a considerable simplification of the hydrodynamic problem. Actually, in our experiments $q \gtrsim 1000\ \mathrm{cm}^{-1}$; the surface waves are damped over a distance less than $10\ \mu$ from both sides of the interface. The temperature gradient used to obtain the interface is of the order of 3°/mm. The viscosities and the densities of the two phases can thus be considered to be constant and equal to their limit value for $t = t_c$. Moreover light scattering experiments from the free surface of MBBA(4) have indicated that the anisotropy of the viscosity disappears at t_c and that

$$\eta_1 = \eta_2 = \eta, \quad \eta_3 = 0;$$

the anisotropy of the viscosity in the nematic phase can thus be ignored.† So finally, the anisotropy of the spectrum can only be produced by an anisotropy of the surface tension γ. Apart from this possible effect, we see then that the nematic–isotropic interface behaves practically as the interface between two normal liquids having the same densities and the same viscosities.

At the interface, the velocities must be continuous and the stresses

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† We have also not taken into account the contribution of elastic forces in the nematic phase. This contribution is expected to be negligible because different experimental results tend to show that the viscosity coefficients γ_1 and γ_2 which couple the hydrodynamic motion to elastic forces tend to 0 at the transition point $t = t_c$. More precisely Haller and Litster(5) have deduced from their data that γ_1 is very small at $t = t_c$ for MBBA and Gahwiller(6) has deduced from flow alignment on MBBA that γ_1/γ_2 is finite at $t = t_c$.

must satisfy the following conditions:

$$\begin{aligned} \sigma^N_{zx} &= \sigma^I_{zx} \\ \sigma^N_{zy} &= \sigma^I_{zy} \\ \sigma^N_{zz} &= \sigma^I_{zz} + \gamma_x \frac{\partial^2\zeta}{\partial x^2} + \gamma_y \frac{\partial^2\zeta}{\partial y^2} \end{aligned} \tag{4}$$

γ_x and γ_y are the values of the interfacial tension in the directions $0x$ and $0y$, ζ is the vertical displacement of the interface.

The theoretical power spectrum can be obtained⁽⁷⁾ by using the fluctuation dissipation theorem $P(\omega) = (kT/\pi\omega)Im[\chi(\omega)]$ where $\chi(\omega)$ is the response function to an external pressure $\Pi(\omega)$: $\zeta(\omega) = \chi(\omega)\Pi(\omega)$. The external pressure must be added in Eq. (4):

$$\tilde{\sigma}^N_{zz}\,(z=0) = \tilde{\sigma}^I_{zz}\,(z=0) - \gamma_{\mathbf{q}} q^2 \tilde{\zeta} + \Pi(\omega)$$

$\gamma_{\mathbf{q}}$, is the surface tension for a deformation of the interface in the direction of **q**. After a certain amount of manipulation the limit conditions at the interface can be written:

$$\begin{cases} \alpha_N + \beta_N = \alpha_I + \beta_I = i\omega\tilde{\zeta} \\ \alpha_N q + \beta_N m = -\alpha_I q - \beta_I m \quad \text{with} \quad m = q\sqrt{1 + \dfrac{i\omega\rho}{\eta q^2}} \\ \dfrac{i\omega\rho}{q}(\alpha_N + \alpha_I) + \gamma_{\mathbf{q}} q^2 \tilde{\zeta} = \Pi(\omega) \\ \alpha_N q^2 + \beta_N m^2 = \alpha_I q^2 + \beta_I m^2 \end{cases}$$

One finds finally

$$\chi^{-1}(\omega) = \gamma_{\mathbf{q}} q^2 - \frac{2\omega^2\rho m}{q(m-q)}$$

and

$$P(\omega) = \frac{kT}{\pi\omega} Im\left\{\frac{1}{\gamma_{\mathbf{q}} q^2 - [2\omega^2\rho m/q(m-q)]}\right\}$$

As the interfacial tension of the nematic–isotropic interface is very small, one can approximate the spectrum by

$$P(\omega) = \frac{kT}{\pi\omega} Im\left\{\frac{1}{\gamma_{\mathbf{q}} q^2 + 4i\omega\eta q}\right\} = \frac{kT}{4\pi\eta q}\left\{\frac{1}{\omega^2 + (\gamma_{\mathbf{q}} q/4\eta)^2}\right\}$$

This is a Lorentzian spectrum at zero frequency and of half width

$$\Delta\nu = \frac{\gamma_q q}{8\pi\eta} \tag{5}$$

2) EXPERIMENTAL RESULTS

A description of the experimental set-up can be found for instance in Ref. 8.

In the present measurements we used together with a General Radio spectrum analyser, a real-time spectrum analyser(9) because most of the experimental spectra obtained had a very small width. This last apparatus was tested with very viscous isotropic liquids, such that the surface waves were highly damped as in the case of

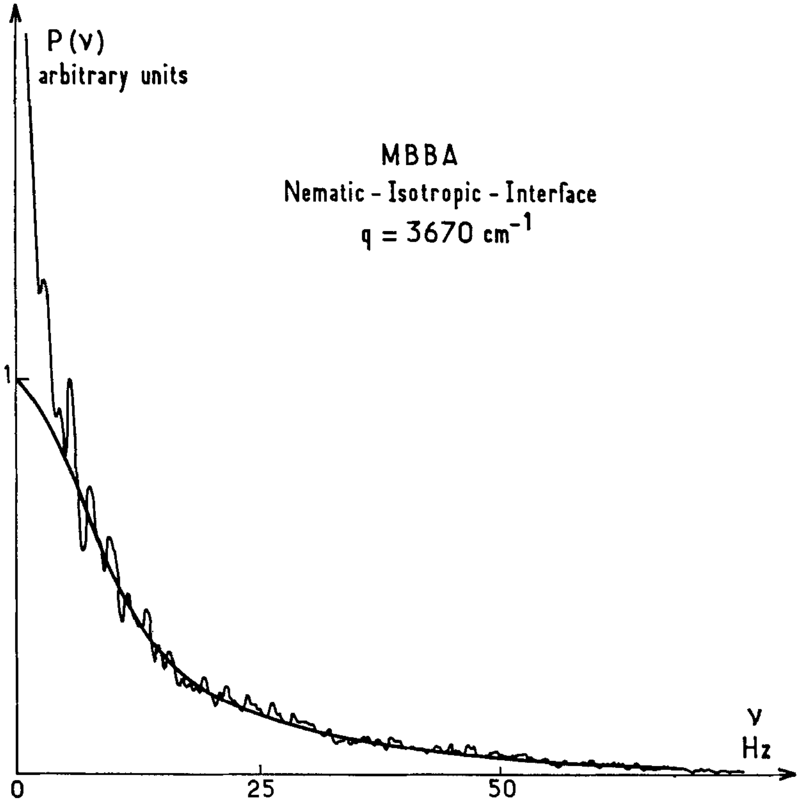

Figure 5. Spectrum of light scattered by the nematic–isotropic interface of MBBA corresponding to a wave vector $q = 3670\ \text{cm}^{-1}$.

the waves at the nematic–isotropic interface. We were able to measure spectral widths down to 0.2 hertz.

We studied the spectrum of the light scattered by the nematic–isotropic interface for wave vectors $\mathbf{q}$ having various orientation with respect to the magnetic field $\mathbf{H}$ (3000 Gauss) in the range $1340 < q < 8000\ \mathrm{cm}^{-1}$. We found *no anisotropy of the spectrum* within experimental uncertainty, that is to say the spectrum remained unchanged when we rotated $\mathbf{q}$ with respect to $\mathbf{H}$. We can thus conclude that the anisotropy of the surface tension is smaller than 20%. Figure 5 shows a typical spectrum corresponding to $q = 3670$ cm^{-1}. The full line curve is the theoretical Lorentzian. The results of the measurements of the half-widths as a function of q are represented in Fig. 6. We find a linear variation of $\Delta\nu$ with q as predicted by Eq. (5). Using the value $\eta = 0.25$ poise,$^{(4)}$ we deduce from

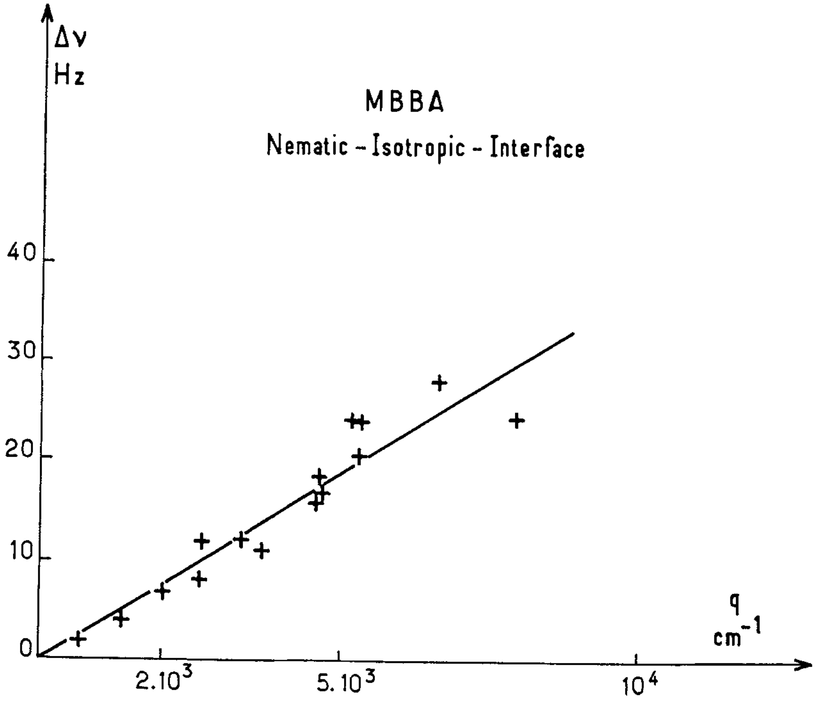

Figure 6. Damping curve of surface waves for the nematic–isotropic interface of MBBA.

the slope of the curve the value of the surface tension,

$$\gamma = (2.3 \pm 0.4)10^{-2} \text{ dynes/cm}$$

3) ESTIMATION OF THE COHERENCE LENGTH

De Gennes(10) has computed the free energy of the nematic-isotropic interface using a mean field approximation. He introduced the elastic part of the free energy of the isotropic phase:

$$F_g = \tfrac{1}{2}L_1 \partial_\alpha Q_{\beta\gamma} \partial_\alpha Q_{\beta\gamma} + \tfrac{1}{2}L_2(\partial_\alpha Q_{\alpha\gamma} + \partial_\beta Q_{\beta\gamma})$$

where $\partial_\alpha \equiv \partial/\partial x_\alpha$; $Q_{\alpha\beta}$ is the tensor order parameter, L_1 and L_2 are the elastic constants in the isotropic phase.

He studied two different configurations: molecules parallel or perpendicular to the interface. He found that the first case is the stable one if $L_2 > 0$. Our experimental results showed that they lie parallel to the interface and consequently indicates that $L_2 > 0$ for MBBA.

The expression of the interfacial tension obtained by De Gennes is

$$\gamma = 2b_t \int_0^{Q^*} F(Q)\, \mathrm{d}Q,$$

with

$$b_t^2 = \tfrac{1}{4}(3L_1 + \tfrac{1}{2}L_2)$$

$F(Q)$ is the total free energy and can be estimated by using an expansion in terms of the order parameter Q, even though the validity of such an expansion is not proved in the ordered phase $(Q = Q^*)$.

Retaining the three first terms

$$F(Q) = \tfrac{1}{2}AQ^2 + \tfrac{1}{3}BQ^3 + \tfrac{1}{4}CQ^4, \quad \text{with} \quad Q^* = \tfrac{2}{3}B/C$$

we obtain

$$\gamma = \sqrt{3L_1 + \tfrac{1}{2}L_2}\,\frac{Q^{*2}}{6}\sqrt{\frac{A}{2}}$$

or, introducing the coherence length

$$\xi = \sqrt{\frac{L_1 + L_2/6}{A}}$$

$$\gamma = \sqrt{\tfrac{3}{2}}\,\xi\,\frac{AQ^{*2}}{6}$$

Using $Q^* = 0.40$,⁽¹¹⁾ $A = 0.062$ J/cm,⁽¹²⁾ and our experimental value of $\gamma = 2.4\ 10^{-2}$ dynes/cm, we find that for $t = t_c$

$$\xi = 120\,\text{Å}$$

This value is in reasonable agreement with the value obtained by Chu, Bak and Lin⁽¹³⁾: $\xi(t_c) = 150\,\text{Å}$. We can conclude that Eq. (8) giving $F(Q)$ is quite satisfactory for the present problem.

Let us close by the final remark: even though these experiments have been performed on quite a clean sample kept in a sealed cell without noticeable change of the transition temperature it is not yet possible to exclude the possibility that some of the properties of the nematic–isotropic interface reported here are affected by contamination, since chemical impurities can be selectively adsorbed at the interface.

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