

Helically modulated electroclinal effects in twist grain boundary liquid crystals

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Received: 24 April 1998 / Revised: 22 June 1998 / Accepted: 31 July 1998

Abstract. We report the first observation of an electroclinal effect at the TGB_C – TGB_A transition induced by an external DC electric field applied perpendicular to the pitch direction. Upon increasing the field, the smectic layers rather than the director field tilt over relative to the helical axis, allowing to detect the effect by X-ray scattering from well aligned samples. The observations are qualitatively interpreted in the frame of a mean field phenomenological model of a helically modulated electroclinal effect.

PACS. 61.30.Gd Orientational order of liquid crystals; electric and magnetic field effects on order – 64.70.Md Transitions in liquid crystals

In 1977 Garoff and Meyer [1] demonstrated the existence of an electroclinic effect above the smectic A–smectic C* (SmA – SmC^*) transition in a chiral liquid crystal. The electroclinal effect is a continuous rotation of the smectic optical axis in response to an electric field applied parallel to the layer planes. The electric field couples linearly to the local dipole moment by inducing a nonzero director tilt proportional to the electric field amplitude. The electroclinal phenomenon has been extensively investigated in the bulk [2–4] and at surfaces [5–8] of SmA and other layered phases; it has even been observed in cholesteric phases [9–13] but not yet reported in Twist Grain Boundary smectic phases (TGB) for which electric field induced transition to the unwound ferroelectric SmC^* state has instead been described [14].

The TGB state represents the liquid crystal analog to the Abrikosov flux phase appearing in type II superconductors [15]. The TGB phase is made of smectic slabs (*i.e.* superconducting phase in the analogy) regularly stacked in a helical fashion along an axis x perpendicular to the slab boundaries. Adjacent slabs are continuously connected *via* a grain boundary constituted of a grid of parallel equispaced screw dislocation lines analogous to magnetic vortices. Two TGB phases have been predicted [15] and experimentally identified: the TGB_A [16] and TGB_C phases [17]. The TGB_A is characterized by SmA slabs. The nematic director \mathbf{n} is parallel to the normal to the smectic planes \mathbf{N} and both are perpendicular to the pitch direction x . In the TGB_C phase, instead, the slabs have a SmC structure with a SmC tilt angle θ defined by $\cos \theta = \mathbf{n} \cdot \mathbf{N}$. The smectic layers are tilted by an angle ω_L with respect to x [18]. The TGB_C structure has *a priori* one more degree of freedom than TGB_A namely the orientation of the

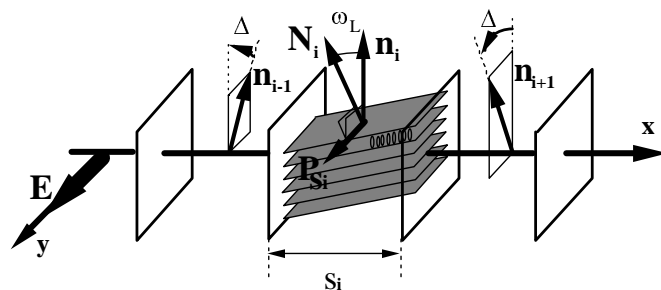


Fig. 1. Helielectric model of the TGB_C phase: local spontaneous polarizations \mathbf{P}_{Si} are perpendicular to the pitch axis x at an angle χ_i relative to the electric field \mathbf{E} (along y). ω_L is the angle of the smectic layer with respect to x .

vector $\mathbf{n} \times \mathbf{N}$ parallel to the spontaneous electric polarization \mathbf{P}_S of each slab. It was suggested in reference [18] that \mathbf{n} in the TGB_C phase remains orthogonal to the helical axis x like in the TGB_A and cholesteric phases. This assumption which minimizes the bend term of the Frank energy is possible since ω_L equals θ within experimental accuracy. As a consequence, the spontaneous electric polarization \mathbf{P}_S of each SmC slab is orthogonal to the pitch axis and precesses in a helical fashion about it (see Fig. 1). Interestingly, such *helielectric* structure can be checked by applying an external transverse electric field \mathbf{E} which couples to the spontaneous polarizations of each smectic slab [19,20]. The electric energy term per unit surface of the i th slab reads:

$$\int_{\text{slab } i}^{\text{thickness}} -\mathbf{P}_{Si} \cdot \mathbf{E} dx = -P_{Si} E s_i \cos \chi_i \quad (1)$$

where s_i is the i th slab thickness, and χ_i is the angle between the spontaneous polarization \mathbf{P}_{S_i} and the electric field direction, chosen along y . In order to minimize the electric energy (1) three different mechanisms are possible: (i) a modulation of the thickness s_i of the slabs: according to the sign of their electric energy, slabs with positive (negative) $\cos \chi_i$ will expand (shrink) (ii) a rotation of the slabs about the screw axis x under the effect of the electric torque in order to increase $\cos \chi_i$ and (iii) a modulation δP_{S_i} of the modulus P_{S_i} of the local polarization, according again to the sign of the electric energy (*i.e.* the product $\delta P_{S_i} \cos \chi_i$ must be positive). The first two effects have been observed and characterized in TGB_C phases [19,20]. The third effect corresponds to an electroclinic distortion as will be discussed in the following.

In the TGB_C phase, the electric polarization modulus P_{S_i} is proportional to $|\mathbf{n}_i \times \mathbf{N}_i| = \sin \theta_i$. An external electric field parallel to \mathbf{P}_{S_i} will increase the local polarization P_{S_i} of slab number i *via* a corresponding variation of the tilt θ_i . This is the well known electroclinic effect. Its magnitude is proportional to the component $E \cos \chi_i$ of the field along \mathbf{P}_{S_i} . It is thus expected to be modulated with χ_i . A similar behavior occurs for the TGB_A phase. The TGB_A has no spontaneous polarization since $|\mathbf{n}_i \times \mathbf{N}_i| = 0$. Nevertheless a polarization δP_{S_i} and the corresponding tilt can be induced by an external electric field. Both should likewise be modulated by the helical structure of the TGB_A . In analogy with the electroclinic effect of a chiral SmA, we call this phenomenon *helically modulated electroclinic effect*. The local SmC tilt θ_i can *a priori* be changed by reorienting \mathbf{n}_i , \mathbf{N}_i or both. A rotation of the director \mathbf{n}_i out of the y - z plane costs a large elastic energy of bend. On the other hand, a \mathbf{N}_i -reorientation costs only a little increase in the grain boundary energy. The latter mechanism is therefore expected to prevail, allowing X-ray diffraction an easy detection (unlike usual electroclinic effect in non twisted chiral smectics).

In this letter we report an experimental study of the helically modulated electroclinic effect in a TGB phase. The experimental results are qualitatively explained by a simple phenomenological model.

The electroclinic effect is known to be maximum close to a SmA–SmC* transition where the tilt susceptibility is large. We therefore chose a compound with a continuous TGB_A – TGB_C transition. The experiment was performed with the *R*-enantiomer of the $n = 11$ homolog of the series studied by the Bordeaux group [17,18], 3-fluoro-4-[(*R*) or (*S*)-1-methylheptyloxy]-4'-(4''-alkoxy-2'', 3''-difluorobenzoyloxy) tolan (nF2BTFO1M7 for short). The phase sequence on cooling is: isotropic (112.4 °C) blue phases (105.7 °C) N* (104 °C) TGB_A (101.5 °C) TGB_C (100.1 °C) S_C* (46.4 °C) crystal. Well aligned samples were prepared as discussed elsewhere [21] between thin (50 μm) flat pieces of polymer coated and unidirectionally buffed glass. The thickness of the liquid crystal film was fixed by two calibrated spacers: we used 25 μm parallel gold wires, 3 mm apart and parallel to the direction of rubbing. The quality of the alignment, with helical axis x perpendicular to the cell walls was checked optically under

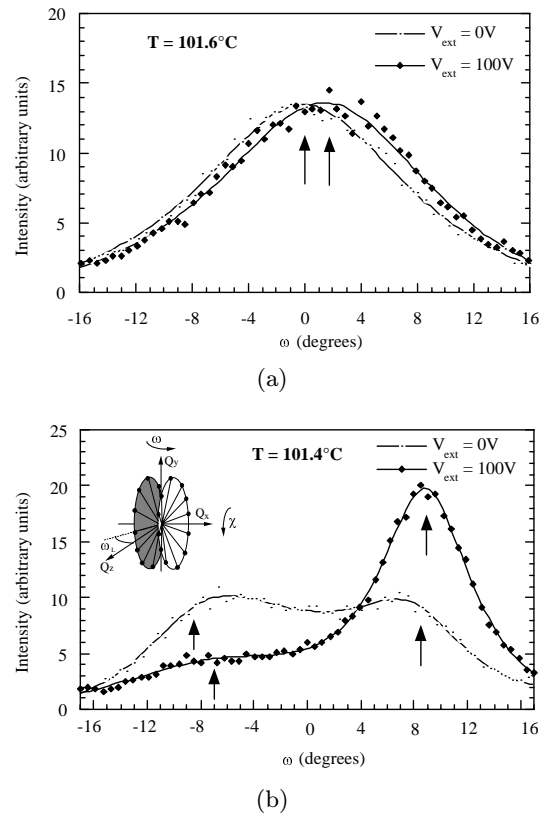


Fig. 2. Rocking curves (ω -scans) recorded at constant wavevector transfer $Q_S = 0.164 \text{ \AA}^{-1}$ across the ring of scattering in the plane (Q_x, Q_z) perpendicular to the field direction ($\chi = 0$) for external voltage $V_{ext} = 0 \text{ V}$ and $V_{ext} = 100 \text{ V}$. (a) $T = 101.6 \text{ }^\circ\text{C}$ in the TGB_A phase: at zero field (dots), the scattering profile is well fitted to a Gaussian lineshape (solid line) centered at $\omega = 0$. In the field (filled diamonds), the Gaussian profile is preserved, but shifted by 1.8 degrees, well beyond the uncertainty on the position of the maximum (of order 0.3 degrees). (b) $T = 101.4 \text{ }^\circ\text{C}$ in the TGB_C phase: at zero field (dots), the profile exhibits two maxima centered at $\omega_L \approx \pm 8.5$ degrees. It is reasonably well fitted to two overlapping Lorentzian functions (solid line). In the field (filled diamonds), the two maxima are shifted both in intensity and angular position (ω). The variation of the intensity are due to field induced translations of the grain boundaries [19]. Arrows indicate the center of the Gaussian or Lorentzian fitting function. Inset shows scattering geometry and reciprocal lattice of the TGB_C structure.

polarizing microscope. The wires were connected to a DC power supply in order to produce an electric field along a direction perpendicular to x . The cells were then introduced in a two stage oven ($\pm 10 \text{ mK}$ accuracy) mounted on the X-ray spectrometer. Experiments were performed using Cu-K α radiation of an 18 kW rotating anode X-ray generator. The samples were mounted on a 4-circle goniometer. The scattered intensity was analyzed by vertical slits and collected by a scintillator. The scattering vector \mathbf{Q} is defined by the axes Q_x, Q_y and Q_z (see inset of Fig. 2b for the TGB_C structure). ω and χ are the rocking angles about the vertical (Q_y) and horizontal (Q_x)

axes respectively. The X-ray optics of the spectrometer was optimized to probe the angular distribution of the layer normal (ω -scans) at constant momentum transfer: the size of the X-ray beam at sample position was set to $1 \times 2 \text{ mm}^2$. The in-plane resolution was then broad enough ($1.0 \times 10^{-2} \text{ \AA}^{-1}$ FWHM) to avoid two-theta (*i.e.* counter position) corrections with electric field or temperature. Samples are mounted with the rubbing direction parallel to the z -axis.

We first performed a series of ω -scans with no applied voltage ($V_{ext} = 0$) [22]. The liquid crystal sample was cooled down slowly by steps of $0.1 \text{ }^\circ\text{C}$ from the N^* phase into the TGB_A phase, then into the TGB_C phase. In the TGB_A phase, the intensity profiles show a broad peak centered at $\omega = 0$ (circles in Fig. 2a). Upon cooling down to the TGB_C phase, the signal splits up into two broad symmetrical peaks close to the TGB_A - TGB_C transition which occurs at temperature T_{AC} (circles in Fig. 2b). Deeper in the TGB_C phase, finally, the two symmetrical peaks get sharper at positions $\pm\omega_L$. ω_L represents the tilt angle of the smectic layers relative to the pitch axis. In the TGB_C phase, the two maxima $\pm\omega_L$ correspond to slabs with scattering vector \mathbf{Q} at angles $(+\omega_L, \chi)$ and $(-\omega_L, \chi)$, the latter orientation being equivalent to $(+\omega_L, \chi + \pi)$ due to the symmetry $\mathbf{Q} \leftrightarrow -\mathbf{Q}$. ω_L can be taken as the order parameter of the TGB_A - TGB_C transition.

We then applied a DC voltage V_{ext} across the gold electrodes to induce a positive electric field along the y -axis. The origin of the χ circle is chosen for vertical external field. For each voltage value, a series of ω -scans at $\chi = 0$ was recorded by increasing the temperature by steps of $0.1 \text{ }^\circ\text{C}$ from $100.9 \text{ }^\circ\text{C}$ in the middle of the TGB_C phase to $102.2 \text{ }^\circ\text{C}$ in the TGB_A phase. Two ω -scans recorded for $V_{ext} = 100 \text{ V}$ are shown in Figure 2 (filled diamonds). Temperature values are the same as for $V_{ext} = 0 \text{ V}$. The symmetry of the profiles with respect to $\omega = 0$ is lost. In the TGB_A phase, the maximum is shifted from $\omega_L = 0$ towards $\omega_L > 0$. At $101.6 \text{ }^\circ\text{C}$ for instance, the shift is $\omega = 1.8$ degrees well beyond the uncertainty on the position of the peak (± 0.3 degrees). A more pronounced shift of ω_L towards positive values is measured close to the TGB_A - TGB_C transition. On the other hand, the position of the peaks remains almost unchanged deep in the TGB_C phase. All these observations indicate that the layer normal tilts over in response to a DC electric field to reach a new equilibrium value $\omega_L(T, V_{ext})$. The effect of a DC electric field is not limited to tilting the smectic layers: the two peaks of the ω -scan recorded at $101.4 \text{ }^\circ\text{C}$ close to the TGB_A - TGB_C transition (filled diamonds in Fig. 2b) have very different intensity. The peak corresponding to slabs with polarization parallel to the electric field ($\omega_L > 0$) is much more intense than its counterpart at $\omega_L < 0$ corresponding to slabs with polarization antiparallel to the electric field. This difference in intensity is a consequence of the other two phenomena discussed from equation (1):

- (i) variation of the thickness s_i of the smectic slabs and
- (ii) χ -reorientations [19, 20].

The variations of the tilt angle (or else TGB_C order parameter) $\omega_L(T, V_{ext})$ with temperature are plotted on

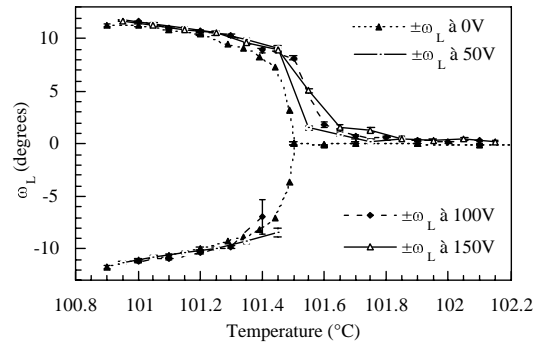


Fig. 3. Experimental results: ω_L versus T by going from TGB_C to TGB_A for $V_{ext} = 0 \text{ V}$, $V_{ext} = 50 \text{ V}$, $V_{ext} = 100 \text{ V}$ and $V_{ext} = 150 \text{ V}$. Estimated error bars on ω are drawn. Note that most of them are less than the symbol size.

Figure 3 for four different values of the applied voltage. The temperature scans go from the TGB_C phase to the TGB_A . In the TGB_C and for $V_{ext} = 0$, $\omega_L(T, 0)$ vs. temperature is represented by two symmetrical branches $\pm\omega_L$ corresponding to positions $(\omega_L, \chi = 0)$ and $(\omega_L, \chi = \pi)$ on the reciprocal rings. ω_L vanishes continuously upon heating at the TGB_C - TGB_A transition with a power law decay t^β with $t = (T_{AC} - T)/T_{AC}$, $T_{AC} \approx 101.56 \text{ }^\circ\text{C}$ and $\beta = 0.20 \pm 0.05$ [22]. When the DC electric field is applied, the behavior of the tilt angle ω_L depends strongly on the sign of the branch. The positive (negative) branch corresponds to scattering from slabs with polarization parallel (antiparallel) to \mathbf{E} (*i.e.* $\chi = 0$ and $\chi = \pi$ respectively). Let us describe the behavior of the positive branch first. Deep in the TGB_C phase, ω_L is not influenced by the field: the electroclinical effect is not detected far from the TGB_C - TGB_A transition, as expected. Upon approaching the TGB_A - TGB_C transition, the tilt susceptibility becomes more important and the spontaneous TGB_C tilt increases with the field. At the transition temperature T_{AC} and above in TGB_A phase, a non zero tilt $\omega_L > 0$ is observed meaning that a layer tilt relative to the helical axis appears in response to the transverse DC electric field. Whatever the voltage, ω_L varies continuously at the TGB_C - TGB_A transition. Note that the positive branch looks very much like the behavior of an order parameter close to a second order transition in presence of a conjugated field (*e.g.* para to ferromagnetic transition under external magnetic field): the critical singularities at $t = 0$ disappear in the presence of the field. The negative branch exhibits a quite different behavior. $\omega_L(< 0)$ is again very close to the zero field value at low temperature in the TGB_C phase, it shifts a little upon heating but disappears abruptly at some temperature T^* below T_{AC} , the difference $(T_{AC} - T^*)$ increasing with the field. For $V_{ext} = 150 \text{ V}$ the negative branch even disappears for all measured temperatures in the TGB_C range.

We show now that the reported experiments can be qualitatively explained in terms of helically modulated electroclinical effect in a simple mean field phenomenological approach. The TGB free energy is constructed as

a Landau expansion in powers of the scalar (Ising) order parameter ω_L . It reads, per unit area in the y - z plane:

$$F_{AC} = \sum_{\text{slabs } i} \left\{ -EPs_i \sin \omega_{Li} \cos \chi_i + \frac{A}{2} \omega_{Li}^2 + \frac{B}{4} \omega_{Li}^4 + O(\omega_{Li}^6) + \frac{C}{2} (\omega_{Li+1} - \omega_{Li})^2 \right\} \quad (2)$$

ω_{Li} is the tilt angle of the smectic layers in slab number i of thickness s_i and orientation χ_i of the local polarization with respect to the external field. The first term in (2) is the electric contribution discussed in equation (1). The phase transition at zero field is controlled by the Landau expansion in even powers of ω_{Li} (symmetry $\omega_L \leftrightarrow -\omega_L$). It occurs for $A = 0$ at T_{AC} and is second order in mean field for $B > 0$. In presence of the field, spatial modulations of ω_{Li} are expected. Their cost in energy is described by the gradient term $(C/2)(\omega_{Li+1} - \omega_{Li})^2$. At zero field, this term is minimum (and null) for a uniform value of ω_{Li} . Since we focus on electroclinic effect, spatial modulations of slab thickness s_i and orientation χ_i are neglected (see Ref. [20] for modelization of these effects) which amounts to postulate $\chi_i = i\Delta$ and $s_i = l_b$. Minimization of the free energy (2) for an infinite stack of discrete slabs is difficult. As usual, a continuous description is easier to handle and captures the same physics. Taking the continuous limit (*i.e.* infinite stack of slabs of infinitesimal thickness $s_i \rightarrow 0$ and orientation $\chi = 2\pi x/P$) we rewrite the free energy (2) per unit area in the y - z plane and per pitch:

$$f = \frac{1}{2\pi} \int_0^{2\pi} \left\{ -\lambda \eta \cos \chi + \frac{r}{2} \eta^2 + \frac{u}{4} \eta^4 + \frac{\nu}{6} \eta^6 + \frac{c}{2} \left(\frac{d\eta}{d\chi} \right)^2 \right\} d\chi \quad (3)$$

$\eta = \sin(\omega_L(\chi, T, V_{ext})) \approx \omega_L$ denotes the new continuous order parameter of the TGB_A-TGB_C transition. The new Landau coefficients r , u and c are proportional to A , B and C in (2). The gradient term is now a simple spatial derivative. The 6th order term has been added to the Landau expansion to include a possible tricritical behavior. At zero field, for $u > 0$, the TGB_A-TGB_C transition is second order at $r = 0$ ($T = T_{AC}$) and η scales as $|T - T_{AC}|^{1/2}$. The transition is first order for $u < 0$ and tricritical for $u = 0$ with $\eta \propto |T - T_{AC}|^{1/4}$. The experimental order parameter exponent measured on our liquid crystal compound $\beta = 0.20 \pm 0.05$ [22] is marginally consistent with a tricritical mean field value; we shall therefore consider the tricritical condition $u = 0$, $\nu > 0$ in the following for numerical calculations. This restriction which may not apply to other TGB_A-TGB_C systems is obviously of minor importance in the present model. Finally, the number of independent coefficients is reduced to two by an appropriate rescaling of the order parameter and of the energy:

$$F = \frac{2\pi f}{c} \left(\frac{c}{\nu} \right)^{-1/2} = \int_0^{2\pi} \left\{ -A\phi \cos \chi + \frac{R}{2} \phi^2 + \frac{1}{6} \phi^6 + \frac{1}{2} \left(\frac{d\phi}{d\chi} \right)^2 \right\} d\chi \quad (4)$$

in which the rescaled order parameter is $\Phi = \alpha\eta$ with $\alpha = (\nu/c)^{1/4}$. The parameters $A = \alpha\lambda/c$ and $R = r/c$ are proportional to the electric field amplitude E and to the distance from the transition temperature $T - T_{CA}$. The stationary solution $\Phi(\chi)$ is obtained by solving the Euler-Lagrange equation of the free energy (4):

$$\frac{d^2\Phi}{d\chi^2} - R\Phi - \Phi^5 = -A \cos \chi. \quad (5)$$

Before we proceed to numerical resolution of (5), let us discuss the solutions $\Phi(\chi)$ in the limit of low electric field $|A/R| \ll 1$. For $R > 0$ (TGB_A), a particular solution of equation (5) reads:

$$\Phi_A(\chi) = \frac{A}{1+R} \cos \chi. \quad (6a)$$

The electric field induces a layer tilt $\Phi_A(\chi) \neq 0$ modulated by the TGB helical structure: $\Phi_A(\chi)$ is maximum for $\chi = 0$ and zero for $\chi = \pi/2$. In reciprocal space, the TGB_A ring is rotated by a weak angle $\omega_L(T, V_{ext}) \approx \Phi_A(\chi)/\alpha$ about the Q_y axis parallel to the field direction. An experimental ω scan at $\chi = 0$ [π] detects the shift of the maximum of scattering towards ω_L proportional to the field ($\Lambda \propto E$) and decreasing upon heating above T_{CA} . Note that a quadratic response (dielectric) would be proportional to $E^2 \propto \cos^2 \chi$ and produce two maxima on an ω scan. In the TGB_C phase in the low field limit ($|A/R| \ll 1$, $R < 0$), two solutions corresponding to the two Bragg rings $\Phi_{C+}(\chi)$ and $\Phi_{C-}(\chi)$ are found:

$$\Phi_{C\pm}(\chi) = \pm(-R)^{1/4} + \frac{A}{1-4R} \cos \chi. \quad (6b)$$

The two TGB_C rings are rotated about the Q_y axis in a symmetrical way preserving the inversion symmetry about the origin of reciprocal space. An ω scan at $\chi = 0$ would detect two peaks at positions $\omega_{L+} = \Phi_{C+}(0)/\alpha$ and $\omega_{L-} = \Phi_{C-}(0)/\alpha$ which are both shifted by the same amount, proportional to E (with the same sign) and decreasing upon cooling far below T_{CA} .

In the general case, the non-linear differential equation (5) can be solved numerically. We used the D02RAF NAG Fortran routine. This routine solves the two-point boundary-value problem with general boundary conditions for a system of ordinary differential equations, using a deferred correction technique and Newton iteration. We impose periodic boundary conditions: $\Phi(0) = \Phi(2\pi)$ and $(d\Phi/d\chi)|_{\chi=0} = (d\Phi/d\chi)|_{\chi=2\pi}$. Figure 4 shows $\Phi_{C+}(0)$, $\Phi_{C-}(0)$ and $\Phi_A(0)$ versus R for different values of A . $\Phi_{C-}(0)$ corresponds to smectic slabs with polarization anti-parallel to the electric field: the polarization decreases in response to the electric field and as consequence $\Phi_{C-}(0)$ and hence the tilt is lower than the equilibrium value in absence of the field. On the other hand, $\Phi_{C+}(0)$ refers to slabs with polarization parallel to the electric field and increases with the field. For weak values of the field $\Phi_{C+}(0)$ and $\Phi_{C-}(0)$ merge together at $R \approx 0$, then, for $R > 0$, only one stable solution $\Phi_A(0)$ exists corresponding

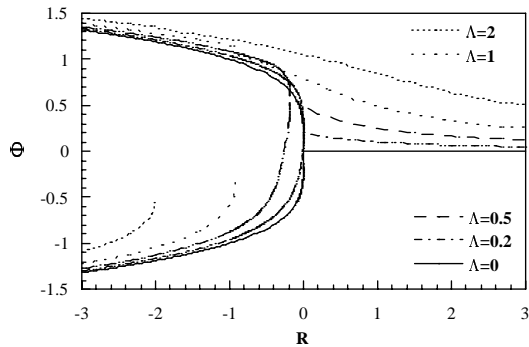


Fig. 4. Numerical results: order parameter Φ versus control parameter R of the TGB_C - TGB_A transition for $\Lambda = 0$, $\Lambda = 0.2$, $\Lambda = 0.5$, $\Lambda = 1$ and $\Lambda = 2$.

to a tilted TGB_A phase. The evolution remains continuous: the single distorted ring splits up continuously into two rings. For higher values of the electric field ($\Lambda = 1$ and $\Lambda = 2$, in Fig. 4) a first order transition occurs at a critical temperature lower than $T_{AC}(R < 0)$, the numerical solution jumps from the field distorted two-branch solution $\Phi_{C\pm}(\chi)$ to the single branch $\Phi_A(\chi)$. The numerical solutions agree qualitatively well with the experimental results $\omega_L(\chi)$ vs. T shown in Figure 3. In particular, the experimental discontinuous jump from two branches solution to one branch solution is well reproduced on the numerical curves. The change in intensity of the Bragg maxima upon increasing the field is however not described by the model since it corresponds to translations of grain boundaries as was discussed in reference [20].

In conclusion, the present paper demonstrates the existence of an electroclinical effect in TGB phases close to a TGB_A - TGB_C transition. Although the phenomenological model is oversimplified, it supports the idea of a helical modulation of this effect and reproduces the main features of the observed behavior. A more accurate modelization should consider slabs of finite size, include translation of grain boundaries and rotation of smectic slabs.

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