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Horizontal chevron domain formation and smectic layer reorientation in SmC* liquid crystals stabilized by polymer networks

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The influence of a polymer network, stabilizing an initial texture of horizontal chevron geometry, on the in-plane smectic C* layer reorientation process is studied for different applied electric field conditions. As expected, the reorientation of smectic layers is strongly slowed down and eventually suppressed by the network, even at rather low monomer concentrations. Polymer network formation in a uniformly reoriented smectic layer state reveals that the network acts in two ways: first it gives a biased elastic torque counteracting a field of such symmetry as to cause a change from the templated layer direction; second it introduces an increased effective viscosity counteracting the reorientation in both directions. The behaviour of samples stabilized by two different kinds of polymer networks, created in between the smectic layers (intra-layer) and across them (inter-layer), is then investigated and discussed.

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1. Introduction

Generally a variety of smectic C* layer and director configurations can be observed depending on sample preparation and surface boundary conditions. For thin, parallel rubbed liquid crystal cells we often observe a structure known as the vertical chevron smectic layer configuration [1]. This can usually be identified by characteristic defects, called 'zig-zag' lines [2, 3]. Under symmetric waveforms an electric field with sufficient amplitude causes a straightening of vertical chevron smectic layers to a more upright position in which the local polarization has been oriented to deviate less from the direction of the applied electric field [4–9]. As a consequence of preserving a constant smectic layer thickness, a folding then takes place not across, but in the plane of the cell. Thus horizontal chevrons appear. These are smectic layer structures with the liquid crystal molecules oriented parallel to the rubbing direction, but with the spontaneous polarization in opposite directions in different domains, implying inclined smectic layers with the layer normal oriented at an angle equal to the director tilt angle to either side of the rubbing direction [10–14]. A schematic representaton of this structure is depicted in figure 1. For samples thick enough not to suppress the intrinsic SmC* helix, a line pattern is observed with disclination lines parallel to the smectic layers.

Application of an asymmetric electric field to such a horizonal chevron domain structure may result in a reorientation of smectic layers in the plane of the substrate such that, depending on the direction of the field asymmetry, favoured domains grow at the expense of the others. This has been demonstrated for different chiral smectic phases [15–20] and varying applied electric field conditions [21, 22]. The reorientation of smectic layers has been shown to be strongly dependent on polymorphism [23], ionic contamination [24, 25] and surface treatment, not only quantitatively [26], but

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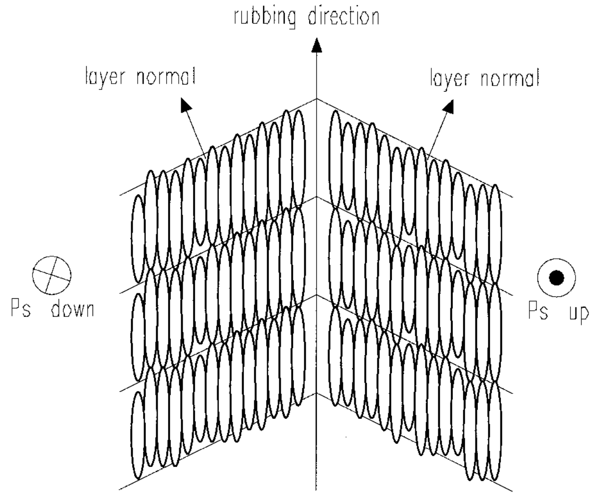

Figure 1. Schematic illustration of the horizontal chevron domain structure, as a top view of the cell, in the surface-stabilized state with unwound helix.

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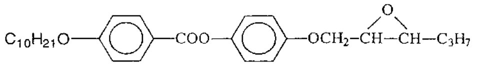
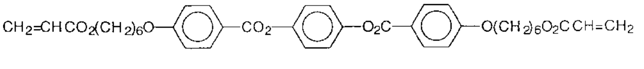
also in a qualitative way [27]. In this study we will report on the influence of a polymer network on the formation process of horizontal chevron structures and the dynamics of the smectic layer reorientation in these systems with respect to pure liquid crystal samples. The polymer network is created in the liquid crystal volume by photopolymerization of a small amount of a photoreactive monomer dissolved in the liquid crystal host. Two different kinds of networks, those situated in between smectic layers and across them, are studied and their influence discussed.

2. Experimental

Samples investigated in these studies were prepared from a commercially available liquid crystal epoxy compound, 4-[(*S*,*S*)-2,3-epoxyhexyloxy]phenyl 4-decyloxybenzoate [28], from Aldrich,



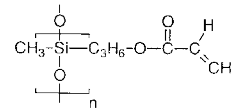
with the general phase sequence on cooling: I–N*–TGBA*–SmC*–SmI*–Cr. For samples studied with respect to monomer concentration and polymerization under different smectic layer configuration conditions, the commercially available photoreactive monomer RM1 [29] from Merck was used:



This compound is a mesogen with the phase sequence Cr 106 N 156 I (°C).

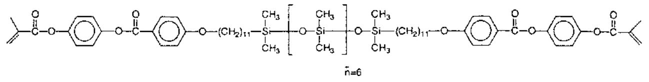
For investigations on the effect of the polymer network created in between smectic layers and across them, two different photoreactive monomers were used.

(i) A commercially available polysiloxane acrylate (ABCR GmbH, Karlsruhe, Germany)



allowing only polymerization within a smectic layer (intra-layer polymerization), and thus giving network formation in between the smectic layers.

(ii) A polysiloxane dimethacrylate (T42) [30]



which permits the polymerization across the smectic layers (inter-layer polymerization), as the end groups and mesogenic units are long enough to extend over a smectic layer distance.

The material from ABCR is non-mesomorphic and a liquid at room temperature, while T42 is a liquid crystal material with the phase sequence SmX 20 SmA 45 N 65 I (°C). The liquid crystal material from Aldrich used in this study has a rather similar molecular constitution to that of the inter-layer polymerizing photoreactive monomer and approximately the same molecular length as one of the side groups. The fact that for monomers (i) and (ii) intra- versus inter-layer polymerization is observed has its reason in the microphase separation of the polysiloxane backbone. This has been demonstrated by several authors [31–33]. With the polysiloxane accumulated between smectic layers, the side group length of monomer (i) is far too short to extend into the next smectic layer. Therefore only intra-layer polymerization is permitted and a network created in between the smectic layers. This is not the case for monomer (ii), because its photoreactive side group is of comparable length to the liquid crystal matrix layer spacing. Here inter-layer polymerization can be obtained.

The horizontal chevron formation process and smectic layer reorientation were optically followed by texture observation with a Nikon OPTIPHOT2-POL polarizing microscope, equipped with a Sony Hyper HAD model SSC-DC38P digital video camera and imaging software from Bergström Instruments AB. The sample temperature was controlled within 0.1 K by a Mettler FP52 hot stage and a FP5 temperature controller. For all studies, time asymmetric square wave fields were applied by an OR-X model 410 function generator in combination with a model F400D high voltage amplifier (FLC Electronics), as schematically depicted in figure 2.

Commercially available sandwich cells (EHC, Japan) of cell gap 8 μm were used in the study of the dependence of horizontal chevron formation and layer reorientation on RM1 polymer concentration in the liquid crystal matrix.

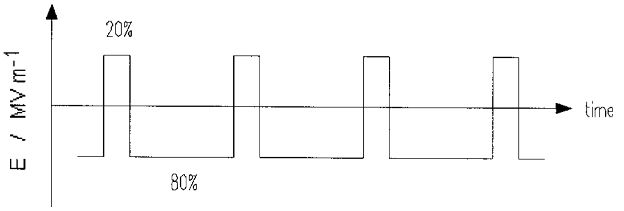


Figure 2. Time asymmetric, square wave, electric waveform applied to induce the smectic layer reorientation. Positive: negative time asymmetry is 1:4 (20 pos. to 80 neg.).

General measurement conditions were set to frequency $f = 200$ Hz, electric field amplitude $E = 4$ MV m^{-1}, field asymmetry 20:80 and reduced temperature $T_c - T = 1$ K below the TGBA*–SmC* transition. In different measurement series one parameter was varied at a time. To avoid the influence of domain nucleation processes and to be able to compare data obtained for samples polymerized under different molecular orientations, the layer reorientation time is defined as the time needed for the one domain type to reorient into the opposite direction. Each individual measurement was performed on a virgin horizontal chevron sample, obtained by heating onto the cholesteric phase, successive cooling into the SmC* phase to a reduced temperature $T_c - T = 1$ K and induction of the horizontal chevron domain structure by a symmetric electric field. Due to the strong effect of the polymer network on the layer reorientation [34], we could only investigate samples with a rather low monomer content, less than approximately 1 wt %. To investigate the effect of a polymer network formed under conditions of an already rotated layer configuration on the reorientation process, a sample of 0.5 wt % RM1 was used. For the photopolymerization, a Teklite UV curing unit was used, with a maximum intensity at wavelength $\lambda = 365$ nm and powder density of 70 mW cm^{-2}.

Investigations concerning the effect of intra- versus inter-layer polymer network formation were carried out using the liquid crystal epoxide host in E.H.C. cells of 8 μm cell gap with the respective monomers photopolymerized under the same conditions as above. The monomer concentrations were chosen at equal wt % (0.25%) as well as equal crosslinking density. Measurements were carried out with a 200 Hz square wave field at 1:4 time asymmetry, amplitude $E = 4$ MV m^{-1} at $T_c - T = 1$ K.

For all illustrations we use the reciprocal layer reorientation time τ^{-1} across the 4×4 mm^2 electrode area as a measure of the layer rotation velocity.

3. Experimental results and discussion

In a previous investigation [35] we pointed out that for non-stabilized samples the process of horizontal chevron domain formation is not related to a surface memory effect. Domains of opposite smectic layer tilt were formed arbitrarily within the electrode area. This is not the case for polymer network stabilized systems formed in SmC* at monomer concentrations above which smectic layer reorientation is suppressed. This behaviour is demonstrated in the texture observations of figure 3, obtained for a sample polymerized with 3 wt % RM1 dissolved in the epoxy compound. Figure 3 (*a*) depicts the sample in the SmC* phase after horizontal chevron domain induction by a symmetric a.c. field. The photograph was taken on application of a small d.c. electric field, orienting the director of one domain practically along the direction of one of the polarizers, which coincides with the rubbing direction (dark domain). The director of the other domain then has to be oriented at an angle twice the tilt angle with respect to the polarizer and the rubbing direction, thus appearing bright. The epoxy compound used as the liquid crystal matrix in this study shows an inversion of the handedness of the cholesteric pitch at a certain temperature; therefore a uniform director configuration (infinite pitch) in a temperature interval in the vicinity of the cholesteric twist inversion is observed [36]. The effect of the polymer network on the liquid crystal director configuration can clearly be seen in this state under zero field conditions. The domain structure is retained and the director basically is oriented along the polymer network strands, figure3 (*b*). Raising the temperature into the isotropic phase, we can visualize the direction of the polymer strands by the weak birefringence due to the anisotropy of the network seen in figure 3 (*c*) (note that for this photograph the light intensity has been greatly increased). From the image it becomes clear that the polymer network is basically directed along the smectic layer normal as reported in [34]. Figure 3 (*d*) shows the SmC* phase after cooling below T_c, but before the induction of the horizontal chevron structure. A very unoriented texture is observed, similar to the one before field application and polymerization. Thus the polymer network does not cause horizontal chevron formation by itself when cooling the polymerized sample below T_c. Application of a symmetric electric field with sufficient amplitude again induces the horizontal chevron domain texture, this time, in contrast to non-polymer network stabilized samples, with domains formed in the same position as those observed after polymer network formation, figure 3 (*e*), compared with figure 3 (*a*). This means, by introduction of a polymer network, we have created a template of the original director configuration [37, 38], now exerting a mechanical field on the liquid crystal, thus representing a memory state. The actual smectic layer orientation is depicted in figure 3 (*f*) at zero electric field with helix lines parallel to the smectic layers.

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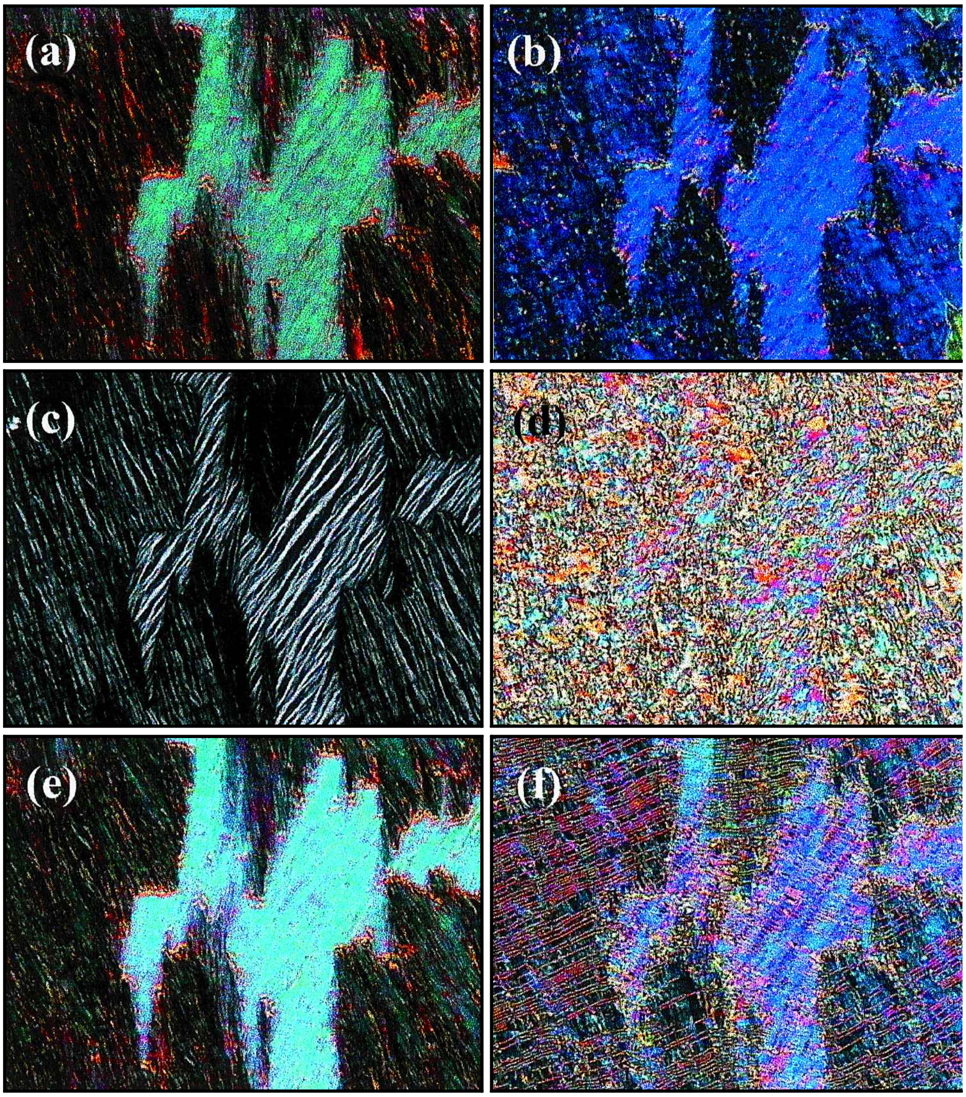

Figure 3. Texture photographs of the epoxy compound stabilized with 3 wt % RM1 by photopolymerization in the SmC* phase. (*a*) SmC* phase after the induction of a horizontal chevron domain structure by a symmetric a.c. electric field. The photograph was taken under d.c. bias conditions to demonstrate more clearly the observed domain texture. One domain type is oriented parallel to the rubbing direction (dark) while the other is oriented at an angle twice the molecular tilt angle (bright). (*b*) The same area at the twist inversion point of the cholesteric phase. The average director orientation is directed along the direction of the former smectic layer normals. (*c*) Liquid crystal material in the isotropic phase. Bright lines are due to polymer network induced birefringence, indicating the directions of the smectic layer normals in the former individual smectic horizontal chevron domains. (*d*) On cooling the sample back from the isotropic to the SmC* phase, a very disordered texture is observed. (*e*) Again applying a symmetric electric field, horizonal chevron domains form in the same positions as those in the sample which was polymerized (depicted under d.c. field conditions). The polymer network represents a template of the original structure and therefore a memory state—compare with (*a*)—in constrast to unstabilized samples, which do not show a (surface) memory effect. (*f*) In the field-free SmC* phase the disclination lines due to the helical superstructure are oriented along the smectic layer planes.

Figures 4(*a*)–(*c*) demonstrate the drastic influence of the introduction of a polymer network on the smectic layer reorientation process. In all cases the general behaviour of in-plane smectic layer reorientaton is observed [21, 22]: increasing reorientation velocities with increasing field asymmetry, increasing field amplitude and increasing temperature. Introducing a polymer network in the virgin horizontal chevron geometry (before any application of an asymmetric electric field), even at very low monomer concentrations, strongly decreases the smectic layer reorientation velocity. This is depicted for varying polymer content in figure 4(*a*) as the dependence of the reorientation velocity on the electric field asymmetry ratio, in figure 4(*b*) as the rotation velocity

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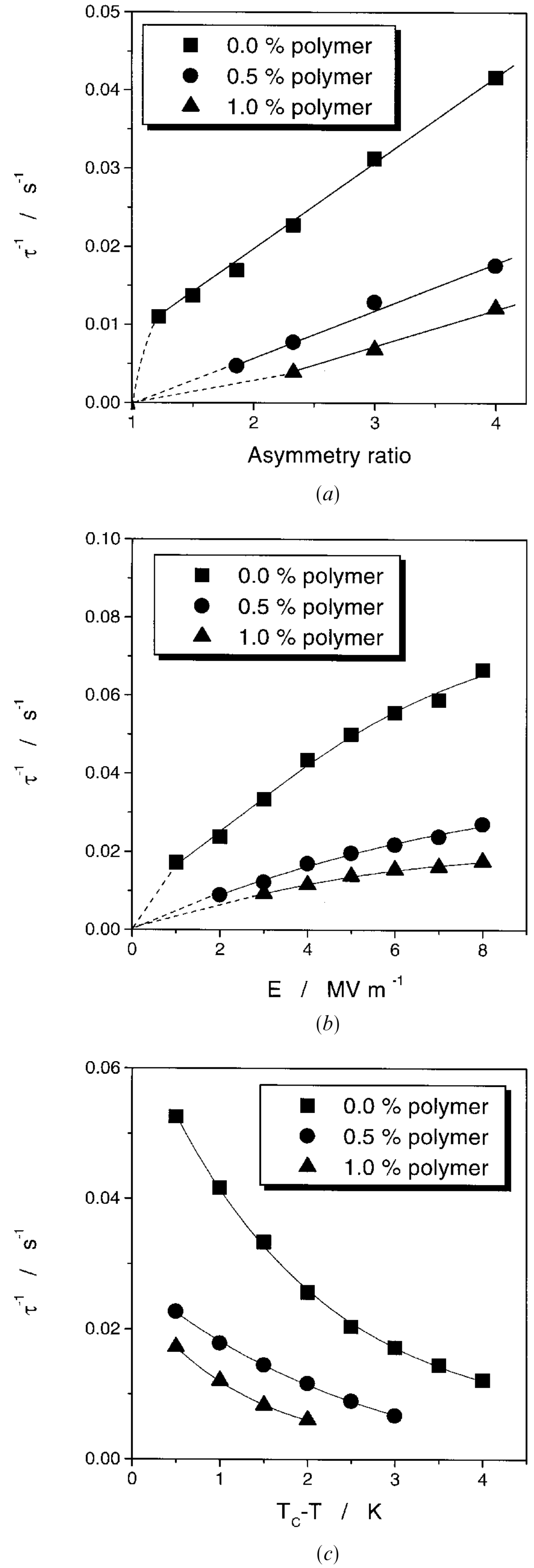
dependence on the electric field amplitude (at asymmetry 20:80), and in figure 4 (*c*) as the dependence on the reduced temperature below the TGBA*–SmC* transition. We attribute this behaviour to an increased effective viscosity counteracting the smectic layer reorientation in both reorientation directions.

Another point of investigation is the smectic layer reorientation behaviour for samples with networks formed in an already rotated smectic layer geometry in the SmC* phase. In this case, smectic layers were rotated to one position by application of an asymmetric electric field after having induced horizontal chevron domain structures with symmetric fields. Photopolymerization was then induced, in a sample containing 0.5 wt % RM1, by UV irradiation in this already reoriented smectic layer geometry. In contrast to the symmetric reorientation behaviour of samples polymerized in the virgin horizontal chevron state (and of course, also samples without a stabilizing polymer network [22]), we now observe an asymmetric dependence of the layer reorientation velocities on different applied electric field parameters, meaning that the dynamic response is different depending upon whether we reorient out of the templated layer structure or into it. This behaviour is depicted in figures 5 (*a*)–(*c*) as a function of electric field asymmetry, field amplitude and reduced temperature, respectively. Again, the general reorientation behaviour is in accordance with investigations on unstabilized samples as pointed out above [21, 22]. But now, we do observe decreased field response for reorientation *out* of the polymer network templated orientation, as compared with that in the opposite direction. This effect is especially pronounced at small reorientation velocities. The observation of different smectic layer reorientation times (applying opposite field asymmetry, i.e. 1:4 and 4:1), for polymer network stabilized samples formed with rotated layer conditions, implies an elastic field acting like a bias electric field. This bias behaviour should of course be dependent on the interactions between polymer network and liquid crystal, and thus be material dependent. In the case investigated here a polymer network formed by 0.5 wt % monomer imposes an elastic bias approximately equal to an electric bias field of 2 MV m^{-1}. This behaviour is rather similar to that found in liquid crystalline elastomers [39]. It should be mentioned however that the layer reorientation velocities for polymer stabilized samples are slower than for the pure



(*a*)

(*b*)

(*c*)

Figure 4. Effect of the polymer network on the dynamics of the smectic layer reorientation as a function of (*a*) electric field asymmetry, (*b*) electric field amplitude and (*c*) reduced temperature. The samples were polymerized in a virgin horizontal chevron state before any asymmetric electric field application.

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epoxy compound, even for the elastically favoured process (switching into the direction templated by the network). This indicates that the material flow, which is necessary for the smectic layer reorientation process [25], is greatly hindered by the interactions of the liquid crystalline material with the polymer network.

In the next part of our investigations we compare the smectic layer reorientation behaviour for intra- versus inter-layer polymer network stabilized samples. Again we do observe the expected behaviour as a function of externally applied conditions for all samples: increasing layer reorientation velocity for increasing field asymmetry, figure 6(*a*), electric field amplitude, figure 6(*b*) and temperature, figure 6(*c*). Due to microphase separation of the polysiloxane backbone, the short monomer from ABCR can only polymerize within a smectic layer and thus creates a network in between the layers when subjected to UV irradiation (intra-layer polymerization). In contrast, the longer monomer T42 can polymerize across the smectic layers (inter-layer polymerization). In all cases the smectic layer reorientation is clearly faster for inter-layer (squares) than for intra-layer (circles) polymerization, as can be seen in figures 6(*a*)–(*c*) for samples of equal wt% (compare squares to closed circles), as well as for equal crosslinking density (compare squares to open circles), accounting for the different molecular masses of the two monomers. It should also be pointed out that the layer reorientation velocities are of course much lower than for the non-stabilized system. The observed behaviour supports our assumption [25] of the layer reorientation process being strongly dependent on material flow along the preferred alignment direction, causing a domain growth perpendicular to it. Material transport along the rubbing direction (permeation across smectic layers) is expected to be much easier for inter-layer than for intra-layer polymerized networks, as molecules are expected to slide along the preferred network direction more easily than to overcome a barrier formed between successive smectic layers. This behaviour is schematically depicted in figure 7.

4. Conclusions

We have investigated the effect of a polymer network formed under different conditions within a smectic C* host on the formation process of horizontal chevron

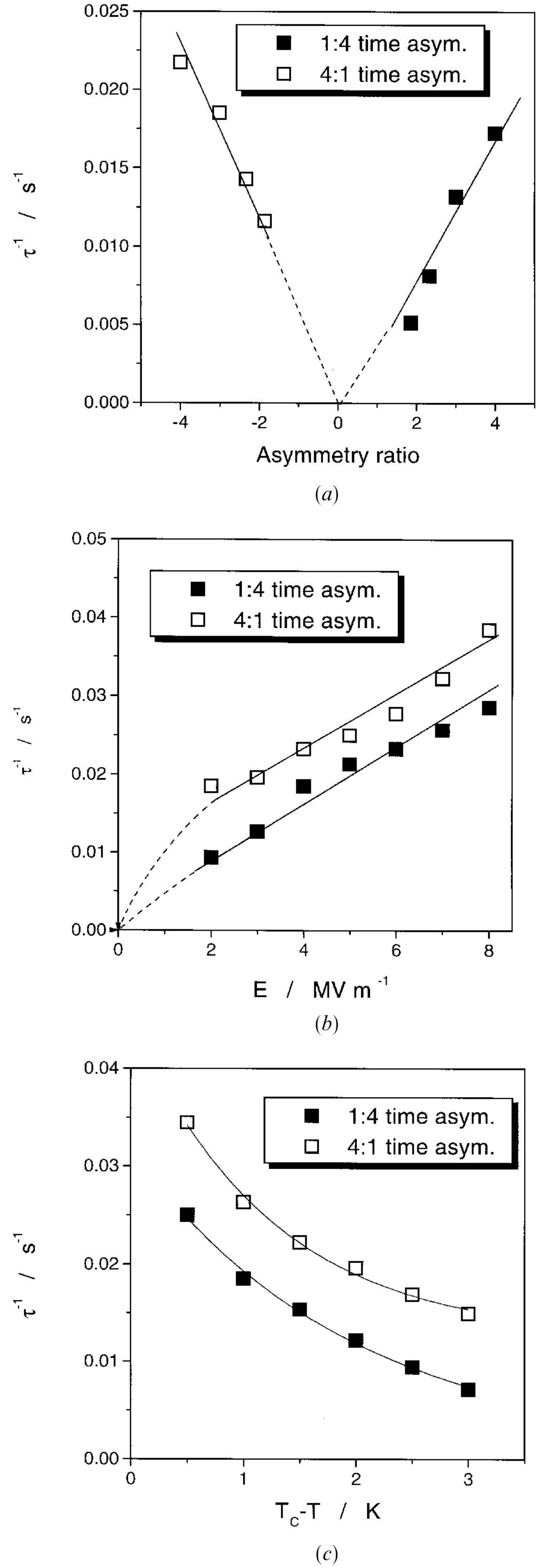

Figure 5. Effect of the polymer network formed under already reoriented smectic layer conditions on the dynamics of the smectic layer reorientation process. Dependence on (*a*) electric field asymmetry, (*b*) electric field amplitude and (*c*) reduced temperature. The elastic field introduced by the polymer network acts like an electric bias field.

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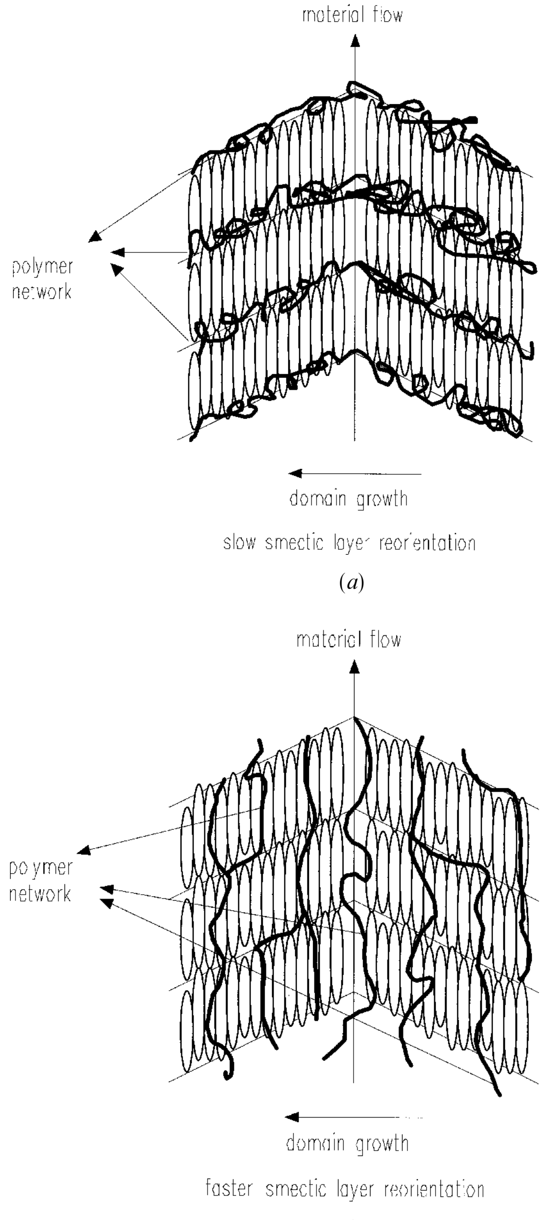

Figure 7. Schematic illustration of the smectic layer reorientation in samples of (*a*) intra- versus (*b*) inter-layer polymerized monomers. Material flow needed for the layer reorientation can be realized more easily in a system with an inter-layer network than for intra-layer polymerization, accounting for faster reorientation velocities.

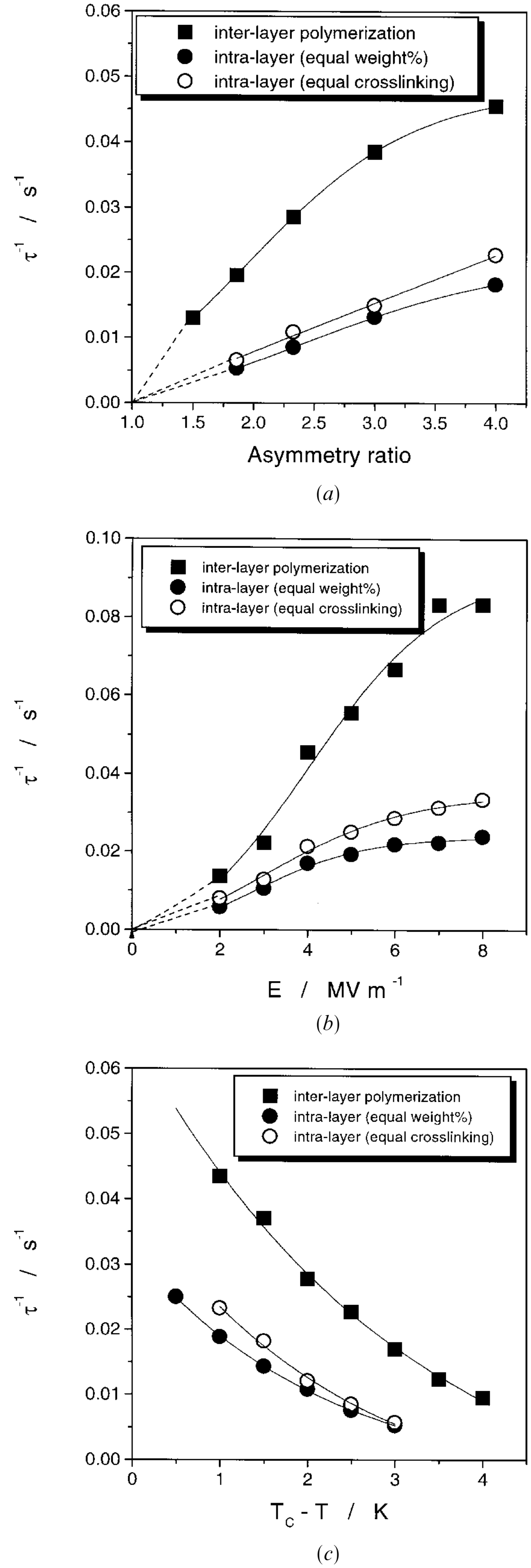

Figure 6. Comparison of the smectic layer reorientation velocity for inter- (squares) vs. intra-layer (circles) polymerization on (*a*) electric field asymmetry ratio, (*b*) electric field amplitude and (*c*) reduced temperature at equal wt % (closed circles) and equal crosslinking density (open circles) of the respective monomers.

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domain textures and the smectic layer reorientation under asymmetric electric fields. In contrast to non-stabilized samples, where the formation of horizontal chevrons does not show a surface memory effect, the polymer network acts like a template of the structure in which it was formed and preserves a memory of the initial domain structure. The polymer network strongly decreases the velocity of smectic layer reorientation when increasing the polymer network content, eventually leading to its suppression [34]. Polymer networks formed in the smectic C* phase of a rotated smectic layer structure cause an asymmetry in the dynamic layer reorientation response, with faster reorientation towards the direction preferred by the polymer network and, in addition, a considerable slowing down of the reorientation process due to an increased effective flow viscosity. When comparing the influence of polymer networks formed by long and short polysiloxane photoreactive monomers, inter-layer polymerization leads to faster layer reorientation than intra-layer polymerization. This is in accordance with an interpretation of the smectic layer reorientation being strongly influenced by material flow across smectic layers (permeation flow). Polymer networks formed in between the smectic layer surfaces are thus more effective in suppressing the layer reorientation process.

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