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Rotation of liquid crystalline macromolecules in shear flow and shear-induced periodic orientation patterns

Received: 18 December 1995
Accepted: 5 January 1996

Dedicated to Prof. Dr. J. Meissner on the occasion of his retirement from the chair of Polymer Physics at the Eidgenössische Technische Hochschule (ETH) Zürich, Switzerland

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Abstract We have measured the shear-induced rotation of the nematic director in a liquid crystalline polymer using poly benzyl glutamate (PBG) as model system. PBG is a well characterized synthetic poly (α amino acid) with rigid chain architecture and well defined conformations. For the experiments it is important to start out with a sample in which the molecules are highly aligned with a uniform director. This so-called monodomain morphology is obtained by use of strong magnetic fields and surface modifications of the sample holders. When shearing the monodomain at a constant rate, the macromolecules rotate initially homogeneously until a periodic director pattern develops. These spatially periodic structures emerge in a narrow range of shear strain

and, as shearing continues, disintegrate into a chaotic texture. By varying the initial monodomain director with respect to the flow direction (but within the shear planes) we could show that the periodic patterns do not depend on the shear direction; they are governed by the director of the initial monodomain. We observe conoscopically that at high shear rates the texture becomes uniformly aligned. The molecules are aligned preferentially with an angle of about 4° to the shear direction (against vorticity direction). Interestingly, this agrees very well with predictions made by Larson (1990).

Key words Rheology – liquid crystal – monodomain – instability – conoscopy – shear

Introduction and background

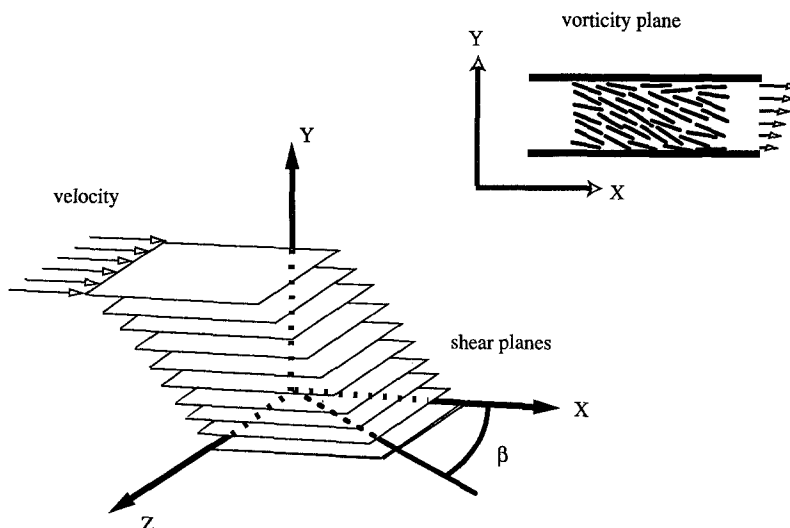
The kinematics of shear flow is defined by fluid planes (shear planes) sliding rigidly past each other as shown schematically in Fig. 1. Nematic liquid crystals in shear have the interesting property that their stiff molecules, even if they are initially all aligned within shear planes (x -

z planes), do not stay in these shear planes¹ but rotate out of them. Molecules of *flow-aligning* liquid crystals rotate against the vorticity of the shear flow and eventually adopt a stable angle with the shear planes, the Leslie angle (Leslie, 1968). Most polymeric liquid crystals are *non-flow-aligning*, having their molecules or semi-rigid sections of their molecules rotating with the vorticity and becoming unstable. Similar instabilities have been found not only in shear but with electric fields, magnetic fields, and temperature gradients (Guyon et al., 1977; Manneville and Dubois-Violette, 1976; Pieranski and Guyon, 1974; Srajer et al., 1989).

The experiments in this study always start out with molecular alignment in the shear plane (so-called planar

¹ We define *shear planes* (x - z planes) being normal to the velocity gradient and *vorticity planes* (x - y planes) normal to the vorticity direction. It should be noted that some of the published work on sheared liquid crystals (mostly outside the rheological literature) refers to the *vorticity planes* as *shear planes*. This will not be followed here.

Fig. 1 Kinematics of shear flow: shear planes move rigidly past each other. LC molecules rotate out of the shear planes; the rotation occurs in the vorticity plane. Angle β denotes the initial angle of the aligned molecules within shear planes



alignment). This excludes a wide range of experiments in which the initial director was chosen normal to the shear planes (so-called homeotropic alignment) (Gu and Jamieson, 1993, 1994; Yan and Labes, 1994; Han and Rey, 1995; Mather et al., 1995).

Shear-induced instabilities have been observed under crossed polars as spatially periodic patterns (bands), some researchers observing them after cessation of shearing and others during flow. The formation of banded structure upon cessation of shear flow has been studied extensively (Graziano and Mackley, 1984; Narvard, 1986; Narvard and Zachariades, 1987; Larson and Mead, 1992; Picken et al., 1992; Gleason et al., 1992; Vermant et al., 1994a). It has been attributed to the relaxation of an aligned structure, however, with an alignment which is somewhat flawed (alignment produced by intermediate shear rates as compared to the more stable alignment from shearing at high rates) (Picken et al., 1992). This study will solely focus on banded structures which develop *during* shear flow, such as the ones observed by Kiss and Porter (1980) and by Vermant et al. (1994b).

Before developing the shear-induced instability, the molecules rotate out of the shear plane. Conoscopy is the ideal method for observing this phenomenon and for measuring the shear induced angle. It can distinguish between rotation of the molecular axis within the shear plane (x - z plane) and out of the shear plane as they occur during flow. They are seen as rotation and translation of the conoscopic interference pattern. Conoscopy has been applied to nematics already in 1911 by Mauguin (1911) who found that the conoscopic pattern represents an alignment angle averaged over the sample thickness provided that the birefringence multiplied by the sample thickness is larger than the wavelength of light. Later, conoscopy was used again by a number of researchers for studying liquid crystals (Cladis, 1972; Srinivasarao and Berry, 1991; Müller et al., 1994; Mather et al., 1995).

In this study we focus on polymeric nematogens with well defined initial alignment (monodomain) and study the transition to a disordered texture during shear. Periodic director pattern occurs as an intermediate between the ordered and disordered structure. We specifically investigate the appearance of bands during flow and its dependence on the initial director alignment angle. The ordered state, also referred to as monodomain, which is characterized by a uniform director alignment, was achieved by applying strong magnetic fields. The average deviation from perfect alignment is commonly defined by an order parameter which is relatively high for our system. Defect-free samples with uniform director alignment allowed us to systematically investigate the effects of shear on the monodomain morphology.

Experimental

Material

The liquid crystalline system studied was poly γ -(benzyl glutamate) (PBG) (Sigma Chemical Company) in *m*-cresol as solvent. PBG is a synthetic polypeptide and belongs to the class of poly (α -amino acids). We prepared a racemic solution with 15% PBG with an average molecular weight of $M_w = 212000$. An about equal amount of right- and left-hand helices is used to suppress the cholesteric character. The birefringence was measured with an Abbe refractometer by using two different polarizations and was found to be 0.0043. PBG was the first synthetic polypeptide and is very well characterized. The persistence length of its α -helix in helicogenic solvents such as *m*-cresol is between 70 nm and 100 nm (Block, 1983).

PBG in *m*-cresol was chosen here because its shear behavior had been studied extensively in the literature.

When producing a monodomain and shearing it parallel to the monodomain director, Burghardt and Fuller (1991) found PBG, like most polymeric liquid crystals, to be non-flow aligning, which means that the nematic director rotates with the vorticity of the shear flow and never reaches a steady state alignment angle.

Experimental conditions

A schematic of the experimental configuration for molecular alignment is depicted in Fig. 2. Two glass slides separated by 500 μm spacers hold the sample, which initially has a multidomain texture. To generate macroscopic alignment of the molecules, the device is mounted on an optical tray inside the bore of a 4.7 Tesla superconducting magnet. The magnet belongs to an NMR imaging/spectroscopy system, but was made available to us by Prof. Sam Patz (Harvard Medical School, Department of Radiology). The tray assembly carries all necessary components to project the conoscopic image on a screen inside the bore and thus to follow the alignment process *in situ*.

We use conoscopy as our primary optical technique since it allows the direct observation of the director alignment. The sample is illuminated with monochromatic polarized light by means of a high numerical aperture condenser. The interference of the ordinary and extraordinary rays in the birefringent material results in a characteristic optical pattern, which can be analyzed to give the spatial orientation of the optic axis. For details of conoscopic optics, we refer to the book by Wahlstrom (1960).

We developed a shearing device for simultaneous optical and rheological measurements. The device is integrated in an optical microscope (Zeiss Universal Stand). A schematic of the device is given in Fig. 3. The shearing unit is mounted on a base plate which is attached to the

microscope stage. A stage adapter allows for easy and reproducible mounting of the device. It can be easily detached and placed on an optical tray inside the bore of a superconducting magnet. All parts are exclusively manufactured from brass or stainless steel and allow the device to be operated in strong magnetic fields, which is applied *in-situ* to produce monodomain texture.

The sample is between two parallel plates. The lower plate is driven in linear motion by an inchworm motor (Burleigh Inc.) with respect to a stationary upper plate. The linear piezo motor is attached to the base plate and allows speeds in the range from below 0.1 $\mu\text{m/s}$ up to 2 mm/s. Total displacement is measured by an optical encoder. A dove tail sled provides very precise and continuous motion of the moving plate. The 25 mm \times 75 mm rectangular glass slides are separated by spacers, available with different thicknesses. We used cell thicknesses between 50 μm and 750 μm . Microscope slide cover glasses can be used as spacers, although their thickness may vary. The device has the capability to measure shearing forces. Figure 3 depicts the design of the force transducer. Two sheet metal springs connect the upper frame with the base plate. They support the weight of the frame vertically, but allow for horizontal displacement in the direction of shear. The displacement is measured with a high sensitivity LVDT (linear velocity differential transducer) from the Schaevitz company. Its output voltage is read by a micro-computer with an *A/D* board. Springs with different thickness can be used to operate the transducer in its optimum sensitivity range for a particular force level. We tested the quality of different springs by shearing a Newtonian fluid (viscosity standard by Brookfield, silicon oils). The fluid was filled in the shearing cell with a thickness of 300 μm . We waited until the transducer signal was

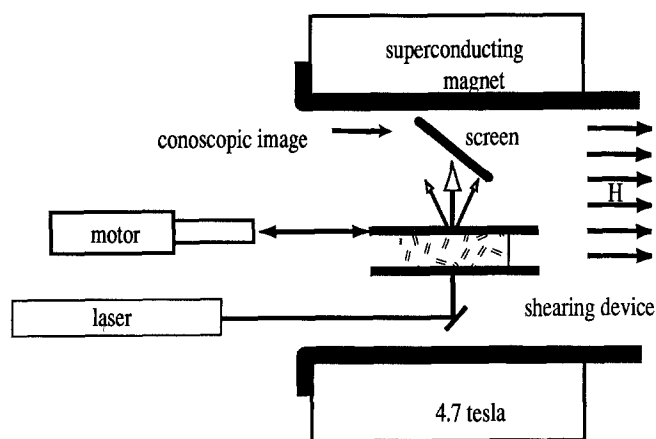


Fig. 2 Experimental set-up for formation of monodomain. Shearing device and conoscopic optics integrated with 4.7 T magnet

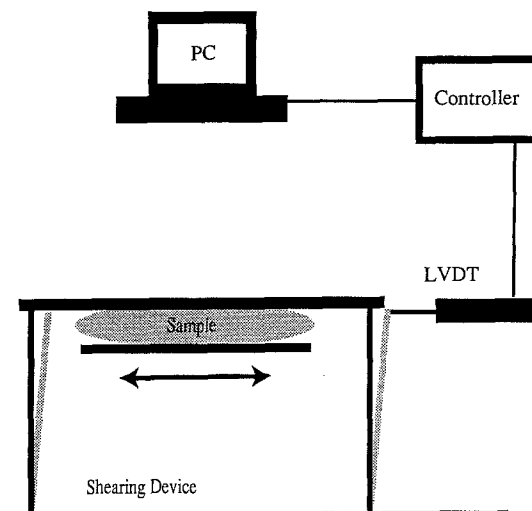


Fig. 3 Design of cantilever beam construction for shear force measurement

steady, which took usually about 30 min after the power had been turned on. We started the motor at a preset velocity at time $t = 0$ and sheared the fluid for about 50 strain units in one direction. The transducer signal has to recover the original level upon cessation of shear, which means that the springs have not recovered their starting position and have a zero slackness and cannot be used for the experiments. We found that razor blade steel has a negligible zero slackness and a high sensitivity for the shear force measurements. The device allowed to measure forces as small as 0.001 N. We used four razor blades on each side of the frame. Two were sandwiched and held with a screw at the bottom and top. Since the razor blades already have holes on the center line, they can be used only with slight modification. The springs were locked in their position during the alignment process to avoid motion of the plates, which could have resulted in destruction of the monodomain. A calibration prior to each measurement was necessary, because the transducer needed to be disassembled before each measurement to load the sample. This was always done with the same standard viscosity fluid at 25 °C.

Results

Shearing in direction of initial molecular alignment

At early stages of shear up to seven strain units (we call this “region I”), we observed a rotation of the nematic director around the vorticity axis with a measured rate of about 2° per strain unit. Sideways rotation could be excluded since the conoscopic figure only translated and not rotated.

The data may be analyzed in terms of the Leslie-Ericksen constitutive equations (Ericksen, 1960; Leslie, 1966) for the coupling of the optic axis with the fluid motion. From six Leslie viscosity coefficients ($\alpha_1, \alpha_2, \dots, \alpha_6$) and three elastic constants, the ratio α_2/α_3 is sufficient to describe the dynamics of the director as long as it is confined in the x - y plane (flow direction – gradient direction). The Ericksen number has to be large enough so that the director responds uniformly and the effects of distortional elasticity are restricted to orientational boundary layers near the surface. For non-flow aligning systems such as PBG/*m*-cresol system, the ratio α_2/α_3 is negative. With conoscopy we found α_2/α_3 to be -44 for a PBG solution (15% by weight) with an average molecular weight of 298 000 g/mol at 20 °C (Müller et al., 1994). In these experiments we found the rotation of the molecular axis is 1) independent of the strain rate and 2) reversible up to 10 strain units. Measurements covered a range of shear rates from 0.002/s to 2/s and used simultaneous conoscopy.

With the initial director aligned in flow direction, we observe that the molecular alignment remains uniform during the early stages of shear induced rotation. Then, a spatially periodic structure develops (denotes as “region II”), which persists for about another five strain units. The nematic director is no longer confined to the x - y plane at the critical strain of $\gamma_{\text{crit}} = 7-8$, but makes periodic excursions (Fig. 4a). The periodicity in the director is shown as a sequence of parallel bands in the polarized microscope. The periodic director pattern gets destroyed when shearing is continued (“region III”, Fig. 4b) and becomes a complex three dimensional structure (Fig. 4c). Figure 4 shows the texture as viewed under crossed polars 45° to the flow direction.

The stress was measured during the start-up of shear from monodomain morphology, see Fig. 5. It rises rapidly when the shear is turned on. At about seven shear

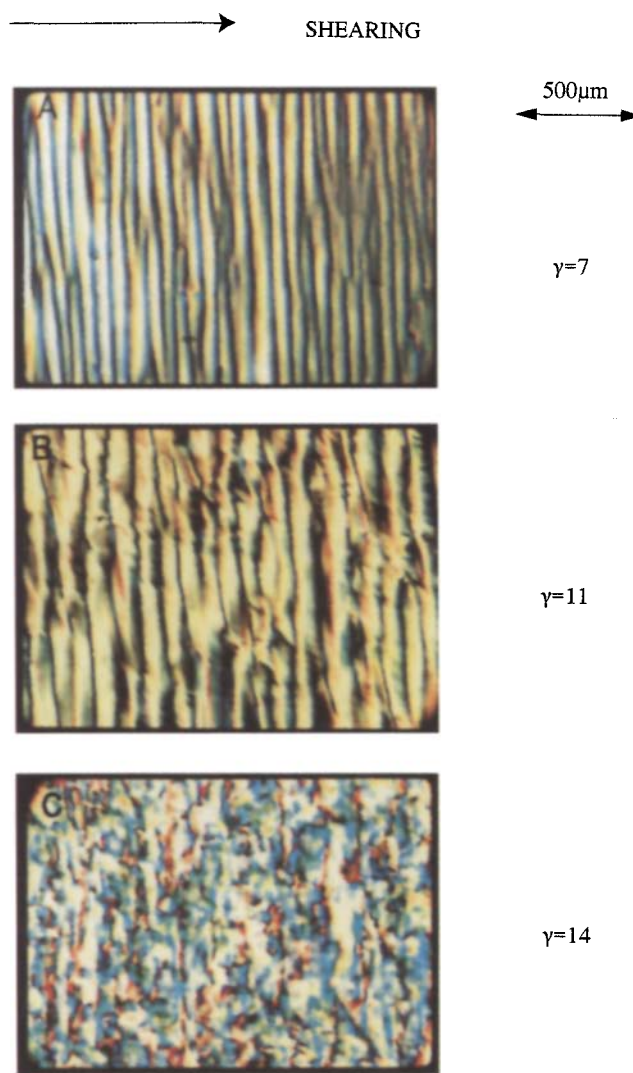


Fig. 4 Optical micrograph of texture viewed under crossed polaroids for different total shear strains. A $\gamma = 8$; B $\gamma = 11$; C $\gamma = 14$

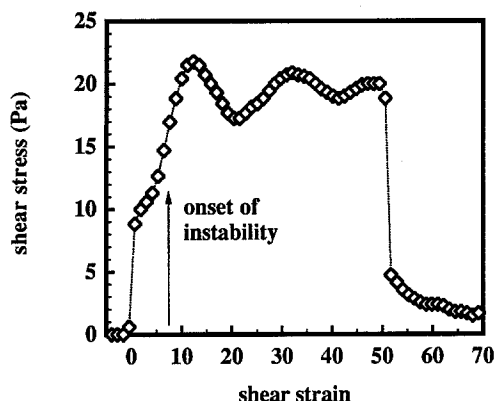


Fig. 5 Stress growth during start-up of shear flow at constant rate, $\dot{\gamma} = 4 \text{ s}^{-1}$. The initial condition is a monodomain with its director aligned in shear direction

units, the periodic instability sets in and the rate of stress growth increases and becomes periodic with decreasing amplitude. The period of the stress is about 22 shear units. The shear rate was equal to 4/s. Experiments at different shear rates are in progress.

Shearing at an angle to the monodomain director

We again prepared monodomain samples with molecules aligned within the shear planes but, by rotating the device inside the magnet bore, we were able to change the initial director alignment anywhere from being parallel to perpendicular with the shear direction ($0^\circ < \beta < 90^\circ$). The angle between the monodomain director and the shear direction, angle β , affects the stability quite substantially.

During the first few shear units of the shear experiments with the monodomains (region I), we found that the conoscopic fringe figure only moves sideways but does not rotate. This means that the director remains at its initial angle β and is only rotating in the x - y plane just as in the preceding experiments in which monodomain and shear direction were parallel ($\beta = 0^\circ$). Then, when the shear reached a critical value, γ_{crit} , the conoscopic figure of the mono-domain vanished and periodic bands emerged, which indicates that the flow has become unstable (region II). The periodic bands were always found to be perpendicular to the initial molecular alignment. Figure 6 shows the periodic pattern for initial alignment of 30° and 40° (micrograph have different magnification). While the orientation is independent of the flow direction, the onset of the instability occurs at lower strain, γ_{crit} , as β of the monodomain is increased from 0° to 90° . Bands do not appear suddenly but within a range of one to two strain units. The onset, γ_{crit} , depends on the alignment angle β in the following way

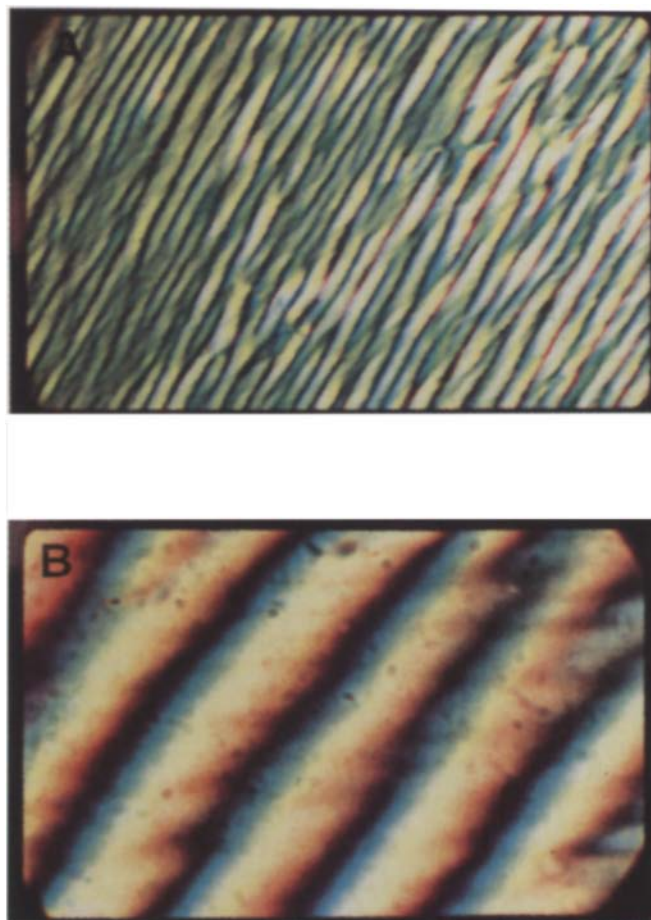


Fig. 6 Periodic structure developed in lyotropic PBG from an initial monodomain at oblique alignment angles. A $\beta = 30^\circ$; B $\beta = 40^\circ$

for $\beta = 0^\circ$	$\gamma_{\text{crit}} \cong 7-8$
for $\beta = 30^\circ$	$\gamma_{\text{crit}} \cong 4-5$
for $\beta = 40^\circ$	$\gamma_{\text{crit}} \cong 4-5$
for $\beta = 90^\circ$	$\gamma_{\text{crit}} \cong 0-1$

For the limiting case of shearing the monodomains sideways ($\beta = 90^\circ$), we observed an immediate instability leading to formation of a periodic pattern within two strain units. This phenomenon is usually referred to as roll instability and has been studied in the case of small molecule nematogens (Pieranski and Guyon, 1974). In roll cells, the director pattern is periodic within the vorticity plane, which will focus incident light if it is polarized along the bands. We did not see any evidence for roll cells when shearing perpendicular to the director (possibly because of the low birefringence of the PBG solution). However, we observe periodic bands under crossed polaroids, indicating a periodic director pattern in the shear plane.

There seems to be a major difference between our experiments with macromolecules and stability behavior of

small molecule LC's, since the magnitude of the strain is the determining parameter for the onset of instability in macromolecular LC's and not the rate of strain. The region of stable flow may be described by a range of alignment angles, which is enveloped by the instability onset γ_{crit} , see Fig. 7.

Steady state molecular alignment at high shear states

Instead of using magnetic fields, uniform alignment might also be introduced by shearing at a sufficiently high rate. We start the flow experiment with unaligned samples. The texture during shear is usually highly defect ridden and very complex. The length scale over which the spatially varying director rotates by about 90° may be used to characterize the texture. Marrucci (1985) proposed that over a range of shear rate, the texture length decreases as the shear rate increases according to $1/\gamma^2$. This simple scaling relation has been confirmed experimentally for a thermotropic LC (Alderman and Mackley, 1985) but does not apply PBG (Burghardt and Hongladarom, 1994). With further increase of the shear rate above a critical value the texture becomes uniform (Kiss and Porter, 1980). The critical shear rate for our PBG solution with $M_w = 212000$ is about 80/s. The birefringence color under crossed polaroids becomes uniform after shearing for about 150 strain units. This indicates a steady state with aligned director. Interestingly, the nematic director is not completely aligned with the shear direction but it rotated by about 4° against the flow vorticity as measured with conoscopy. This rotation direction is opposite to what occurs during the start-up of shearing the monodomains (region I, where the PBG molecules rotate in vorticity direction). Larson (1990) theoretically predicted a steady state alignment angle of 4.5° after a transition from director tumbling to steady state alignment with increasing shear rate.

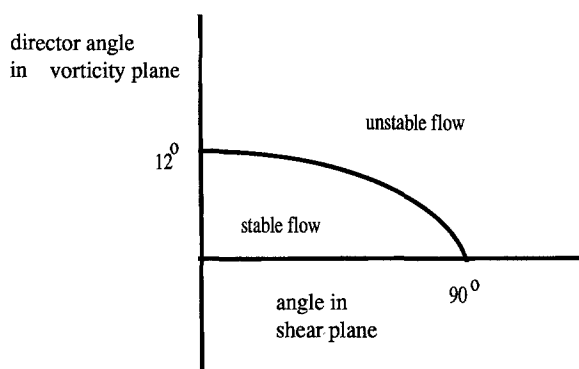


Fig. 7 Schematic of instability envelope as a function of the director angle in the x - y plane (vorticity plane) and x - z plane (shear plane)

Discussion

The shearing of the monodomain morphology may be divided into three regions: 1) A region of stable flow occurs from initial monodomain conditions (region I). The director alignment is only a function of the lateral coordinate (velocity gradient direction) which is an ideal condition for quantitative conoscopic measurements. 2) Region II starts with the onset of an instability, which results in a periodic director pattern, and 3) disintegrates in region III.

The physical origin of spatio-temporal patterns is assumed to be twofold. On the one hand there is strong coupling between flow and director field. On the other hand the viscosities are strongly anisotropic as shown by Miesowicz (1946), who measured three anisotropic viscosity coefficients of *p*-azoxyanisole and *p*-azoxyphenetole. He used a magnetic field to keep the molecular orientation constant. For polymeric LC, the anisotropy was found to be larger than for small molecule systems (Taratuta et al., 1985; Yang and Shine, 1993; Grabowski and Schmidt, 1994).

Large strain produces a defect-dominated LCP structure, except when shearing at sufficiently high rate (here enters the rate dependence!), where the average nematic director adopts a steady state angle which is very small (about 4°). Moldenaers and Mewis (1987) studied the large strain rheological behavior below this critical shear rate. They performed shear stress measurements after a step-up in shear rate. The shear stress shows at least three overshoots and the steady state is achieved only after more than 50 strain units. In their experiments they do not have uniform alignment as a starting condition. Peaks in the shear stress occur at strain values that are independent of the final rate. It is conceivable that the stresses directly reflect a certain distribution of director alignment angle.

The stress growth during start-up of shear from planar monodomains has, because of its periodicity, great similarity with the stress growth in slightly aligned LC samples as observed by Mewis and Moldenaers (1987). However, the first peak was found to be the largest while the first peak in Mewis and Moldenaers' observation is quite small and the second one dominates. These differences might be due to differences in the initial alignment in the two experiments.

Vermant et al. (1994) have reported a very similar shear stress development for a 15% PBG solution in *m*-cresol. Their measurements were taken in a rotational rheometer. It seems that the curvature of the shearlines does not have a significant effect on the stress development.

Gu and Jamieson (1993, 1994) studied monodomains with initial homeotropic alignment in a rotational flow cell. They have measured the transient shear stresses of flow aligning and non-flow aligning materials (5CB and

8CB). The non-flow aligning material shows the double peak in shear stress, which is in excellent agreement with the Leslie Ericksen theory. The distinct features of the stress trace diminish with increasing strain. Interestingly, our data does not show a double peak and this might be due to several reasons. After all, PBG/*m*-cresol is a polymer solution and shows viscoelastic behavior (\rightarrow spectrum), may it be liquid crystalline or not. To solely rely on Frank elasticity and Leslie coefficients may not be enough to explain the macroscopic stresses.

Conclusions

The experimental observations on a model LCP under shear show how the rodlike molecules rotate out of the shear planes and become unstable. The instability is manifested in the formation of a periodic director pattern. The transition occurs at a material-dependent characteristic strain, γ_{crit} , which depends on the angle β between shear direction and nematic director (besides concentration and temperature which has not been addressed

here), but it does not depend on shear rate as one might have guessed from experience with small molecule LC's. By varying the initial director alignment, we found for region I that the director rotates around the vorticity axis, irrespectively of the initial orientation in the shear plane, and that it does not rotate sideways. In further experiments we will study the effect of sample thickness on the observed band spacing.

A main result of this study is that, independent of the shear direction, the periodicity in the nematic director is always along the initial molecular alignment and, hence, the bands are perpendicular to the initial molecular alignment. Reports in the literature have been misleading when they stated that shear-induced bands occur perpendicular to the shear direction. In these studies, it just happened that the initial alignment and the shear direction coincided and the above distinction could not be made.

Acknowledgment We thank Prof. Sam Patz at Harvard Medical School, Department of Radiology, for his kind hospitality when he allowed access to his magnet facility. Financial support from National Science Foundation (NSF grant DMR-9422180) and NSF-MRSEC at the University of Massachusetts is gratefully acknowledged. We thank R.G. Larson for conducting the anonymous review as member of Rheologica Acta's editorial board.

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