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Rheology of twist-grain-boundary-*A* liquid crystals

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We report studies on the rheological properties of a liquid crystalline analog of Abrikosov phase in type-II superconductors known as twist-grain-boundary-*A* (TGB_A) phase. The TGB_A phase shows a large apparent yield stress compared to the cholesteric (N*) phase. The storage modulus (G') of the TGB_A phase is significantly larger than the loss modulus (G''). The dynamic relaxation measurements indicate a solid-like behavior of N*, TGB_A, and smectic-C^* phases. The complex shear modulus of the TGB_A phase exhibits a power-law behavior $G^*(\omega) \sim \omega^\alpha$ with $\alpha \simeq 0.5$. The relative amplitude of G' and G'' at various temperatures indicate that the enhanced elasticity of TGB_A phase is due to the structural defects.

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I. INTRODUCTION

Structural disorder or defect in general affects the mechanical properties of all materials. In solid materials the disorder or the defects are created or pinned down by impurities for desired mechanical properties. Many anisotropic fluids such as liquid crystals possess topological defects. The network of defects in such systems can respond to shear, and hence their contribution to the rheological properties is significant. The density of defects in liquid crystals can be enhanced by adding colloidal particles in cholesteric (N*) liquid crystals, aerosil in smectic-*A* (Sm*A*) liquid crystals [1–3]. Recently the enhancement of elasticity due to the network of defect lines entangled with colloidal particles in liquid crystals has been reported [4]. Generally, the viscoelastic properties in such composite systems are enhanced and their dynamics can be explained based on the soft-glassy-rheology or the theory of rubber elasticity. However, soft materials with long-range periodic structural disorder due to the defects (dislocations) and the grain boundary composed of an array of dislocations are rare and their viscoelastic properties are still unexplored. One such very unusual soft material is type-II smectic-*A*, known as the twist-grain-boundary-*A* (TGB_A) liquid crystal. The TGB_A is a liquid crystalline analog of the Abrikosov phase exhibited by type-II superconductors [5]. The analogy between superconductors and smectic liquid crystals was proposed by de Gennes, who predicted the intermediate phase with a lattice of dislocations in smectics. Renn and Lubensky showed that the intermediate phase of highly chiral type-II materials consists of a twisted arrangement of blocks of Sm*A* liquid crystals, separated by twist grain boundaries, which are made of an array of screw dislocations [6,7]. A typical structure of the TGB_A phase is shown in Fig. 1.

The interacting dislocations form the grain boundary and the interaction among the grain boundaries stabilizes the TGB_A structure. A typical distance between two dislocations (l_d) in a grain boundary and the intergrain boundary distance (l_b) is in the range of $l_d \sim l_b \sim 20$ to 30 nm [8]. The smectic-*A* blocks with these dislocations form a helical structure. In this phase, well-defined Sm*A* blocks are separated by regularly spaced planar arrays of parallel screw dislocations (Fig. 1). The normal layer of the successive slabs rotate relative to each other by an angle. The contrary structure of smectic-like layering and cholesteric-like twist was experimentally discovered in highly chiral liquid crystals by Goodby *et al.* [9] and was characterized by several experiments [8,10]. Soon after the discovery there were some reports on the observation of several variants of TGB_A, such as a TGB_C, TGB_{C^*} [10–12]. In a binary mixture of chiral and nonchiral compounds, Pramod *et al.* [12] reported a three-dimensionally modulated chiral smectic-*C* phase called UTGB_{C^*} which has a two-dimensional (2D) undulation of SmC^* blocks in the form of a square lattice. There are several experimental reports on the structure, electro-optic, and dielectric properties of various TGB liquid crystals [13,14]. However, the viscoelastic properties of these phases have remained unexplored. In this paper we report rheological measurements on a binary mixture exhibiting a large temperature range (≃18 °C) of the TGB_A phase. The experimental results are analyzed based on the known theoretical model.

II. EXPERIMENT

We prepared a binary mixture of two compounds namely, 4-(2′-methyl butyl phenyl 4′-n-octylbiphenyl-4-carboxylate (CE8) and 2-cyano-4-heptyl-phenyl-4′-pentyl-4-biphenyl carboxylate [7(CN)5]. CE8 is a chiral and 7(CN)5 is a nonchiral compound and both were synthesized in our laboratory. The detailed phase diagram of the binary mixtures was reported by Pramod *et al.* [12]. Based on the phase diagram, we prepared a mixture of 63.6 wt% of CE8 and 36.4 wt% of 7(CN)5 to get a large temperature range of the TGB_A phase. The mixture was prepared by weighing each component in a glass container. The sample was then heated above the isotropic phase and mixed physically by using a thin glass rod. Polarized optical microscope shows following phase transitions on cooling: I 120.2° C BP 119° C N* 80° C TGB_A 62.1° C UTGB_{C^*} 59.8° C SmC^*. It may be noted that the temperature range of TGB_A is about 18°C and UTGB_{C^*} is very short, about 2° C. The temperature range of TGB_A is much larger than that usually observed in a single liquid crystal compound. Rheological measurements were made by using

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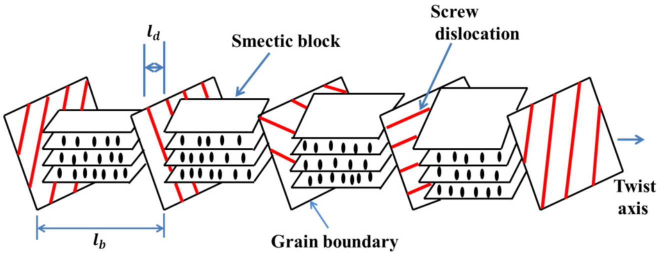

FIG. 1. (Color online) Schematic representation of structure of the TGB$_A$ phase. The distances between two dislocations and two grain boundaries are l_d and l_b, respectively.

a stress-controlled rheometer (Anton Paar MCR 501) with the cone and plate measuring system of diameter 25 mm, cone angle 1°, and with a minimum gap of 50 μm (the gap between the plate and the truncated cone). The temperature of the sample was controlled with an accuracy of 0.1° C by a Peltier temperature controller fitted with a hood for the uniformity of the sample temperature. The calibration of the temperature controller was checked by measuring the phase transition temperatures of some standard liquid crystalline materials [15–18]. The rheological properties of many soft materials, especially liquid crystals, when freshly loaded into a rheometer are usually not reproducible because a large degree of macroscopic disorder and inhomogeneity that may get introduced during the loading process [19]. Hence we heated the sample to the isotropic phase and cooled down to the liquid crystalline phase. The sample is then presheared for about 400 s with a shear-rate of 10 s^{-1} before the measurements.

III. RESULTS AND DISCUSSION

A. Texture and physical observations

The typical textures of various phases observed under optical polarizing microscope in a planar cell (a cell treated for planar alignment of the director, i.e., the average direction of molecular alignment) is shown in Fig. 2. The N* phase exhibits the typical defect network of the "oily-streak" [Fig. 2(a)]. The TGB$_A$ phase shows almost homogenous texture and that the slight nonuniformity in the color is due to the nonuniform alignment of the helix axis at different regions ([Fig. 2(b)]. There is also a short temperature range of the UTGB$_{C*}$ phase below the TGB$_A$ phase with a characteristic square-grid pattern. As the temperature is lowered, SmC^* appears to be keeping to the square grid pattern [Fig. 2(c)], and it can be removed by slightly shearing one of the plates. The physical appearance of the sample under mechanical disturbance such as pulling up by a glass rod at various temperatures (after mounting it on the rheometer) is also shown below the respective textures in Fig. 2. It appears that N* tends to flow easily like a liquid. The TGB$_A$ phase appears like a sticky fluid and SmC^* is kind of a semisolid that does not flow easily.

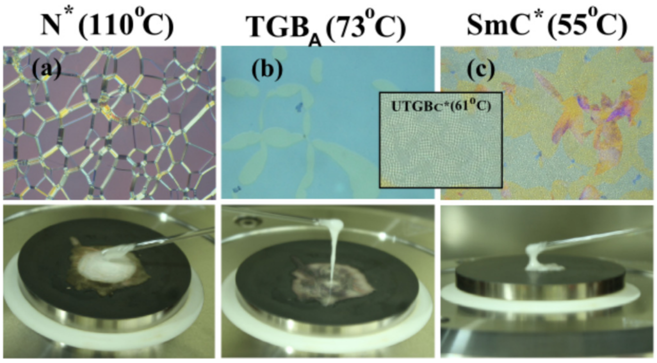

FIG. 2. (Color online) Textures observed in polarizing optical microscope (POM) at different temperatures (a) 110° C (N*) (b) 73° C (TGB$_A$), and (c) 55° C (SmC^*). A square grid pattern characteristics of UTGB$_{C*}$ appears between TGB$_A$ and SmC^* is also shown in the box. The physical appearance of the sample while trying to pull it up by a glass rod at corresponding temperatures are also shown.

B. Yield-stress measurements

The determination of true yield stress is rather difficult and it depends on the measuring techniques [20–22]. However, the apparent yield stress can be estimated from the shear-rate-dependent shear-stress for relative comparison in the different phases of the same sample. Figure 3 shows the shear-rate-dependent effective shear stress (σ_{eff}) at different temperatures. At the low shear rate ($\dot{\gamma} < 0.1$ s^{-1}), the stress tends to reach almost a constant value as $\dot{\gamma} \longrightarrow 0$, showing an apparent yield stress (σ_y). At 110° C (N*), σ_y is negligibly small ($\simeq 10^{-2}$ Pa) and it increases as the temperature is lowered in the TGB$_A$ and SmC^* phases. For example, at 68° C (TGB$_A$) $\sigma_y \simeq$10 Pa and it further increases as the temperature is decreased to SmC^* (57° C) phase. Very small σ_y in the N* phase is due to the residual oily-streak defect network of the presheared sample. Thus, σ_y is three orders of magnitude larger in TGB$_A$ than that of N* phase. The apparent yield stresses have also been reported in many other smectic materials [23,24]. However, the apparent yield stress in TGB$_A$ and in SmC^* phases of the present system is much larger than that known in low molecular weight smectic liquid crystals [23,25]. The N* phase shows shear thinning followed by a Newtonian behavior at high shear rates (>10 s^{-1}). In the intermediate shear-rate range, i.e., $0.1 < \dot{\gamma} < 100$ s^{-1}, it shows shear thinning behavior

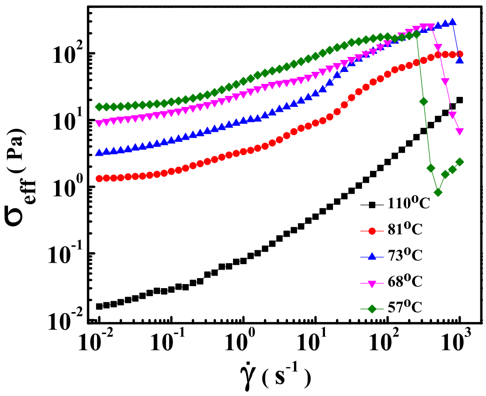

FIG. 3. (Color online) Shear-rate-dependent shear stress (σ_{eff}) at different temperatures N*: [110° C (squares)], near N*-TGB$_A$ transition: [81° C (circles)], TGB$_A$: [73° C (uptriangles)], TGB$_A$: [68° C (downtriangles)], SmC^*: [57° C (diamonds)].

in all phases. The shear-rate-dependent viscosity of nematic or cholesteric liquid crystals is usually characterized by the Ericksen number [26], which is given by the ratio of the flow-induced viscous stress ($\eta\dot{\gamma}$) to the Frank stress (K/h^2), where K is the average curvature elastic constant and h is the length scale of the flow geometry. Theoretically it has been shown that in the intermediate range of the Ericksen number (10 to 400), the shear viscosity shows a shear thinning behavior [27]. Assuming a typical average value of average curvature elastic constant $K \sim 10^{-11}$ N and measured apparent viscosity of the N* phase $\eta \simeq 10$ mPa, the estimated Ericksen number is $\simeq$300, which is in the theoretically predicted range. At very high shear rate ($\dot{\gamma} > 100$ s^{-1}), σ_{eff} in both the TGB$_A$ and SmC^* phases fall suddenly to a much lower value. This sudden decrease could be connected to the wall slip or the plastic deformation of the material.

C. Dynamic relaxation measurements

To determine the linear regime of viscoelasticity by oscillatory measurement, we measured the strain amplitude dependence of storage ($G^{'}$) and loss ($G^{''}$) moduli at various temperatures. The measurement is performed every time on a fresh sample and the results are shown in Fig. 4. The data in the N* phase (110° C) at low strain amplitude are noisy and are attributed to the lower limit that our rheometer can reproducibly measure. Nevertheless, the average values of both $G^{'}$ and $G^{''}$ are independent of strain up to $\gamma \simeq 0.8$ in all phases. In the TGB$_A$ phase, (73 and 68° C) in the linear viscoelastic region $G^{'} > G^{''}$ and $G^{'}$ of TGB$_A$ is more than 1000 times larger than that of the N* phase. Similarly, in the SmC^* phase (57° C), $G^{'} > G^{''}$ and $G^{'}$ is about 2.5 times larger than that of the TGB$_A$ phase. The viscoelasticity of the N* phase is mostly contributed by the three-dimensional network of residual oily-streak defects [1]. The SmA liquid crystals will have a larger viscoelastic response than nematic or N* merely because of the positional order. For example, in our previous studies, in cholesterol nonanoate, (exhibits N* to the SmA transition) we found $G^{'}_{\text{Sm}A} \approx 80\ G^{'}_{N^*}$ [28]. In the present sample, we find $G^{'}_{\text{TGB}_A} \approx 1000\ G^{'}_{\text{N}^*}$. Hence, the enhanced elasticity of the TGB$_A$ phase can be attributed to the bulk sample with structural defects present in the system. In this phase the enhancement in the viscoelasticity may also be due to the energy cost for the deformation of grain boundaries. In addition, the surface tension of the smectic layers which acts against the deformation plays a vital role for increasing the shear modulus. In the SmC^* phase, there is no grain boundary, however, the individual dislocations and their motions contribute to the viscoelastic properties. In fact, it has been suggested that the shear modulus is strongly influenced by the line tension of screw dislocations because it acts against the Peach-Koehler force which controls the motion of the dislocations [29]. When the force exerted on the dislocations dominates over the line tension, then the system overcomes its elastic deformation and undergoes plastic deformation. The drastic fall of σ_{eff} shown in Fig. 3 for both the TGB$_A$ and SmC^* phases could be due to the plastic deformation.

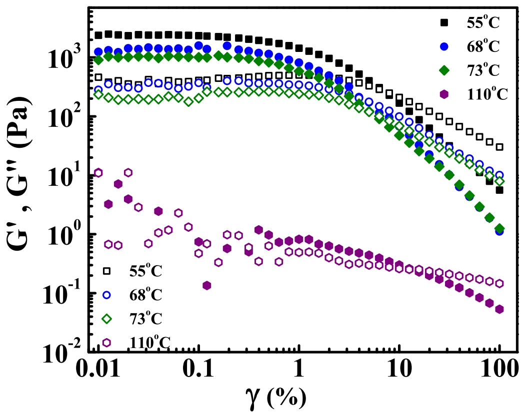

FIG. 4. (Color online) The strain amplitude dependence of the storage $G^{'}$(solid symbols) and the loss $G^{''}$(open symbols) moduli at different temperatures. 110° C (hexagons) [N*], 73° C (diamonds) [TGB$_A$], 68° C (circles) [TGB$_A$], and 59° C (squares) [SmC^*] at $\omega = 1$ rad/s.

The role of defects and disorder on the viscoelasticity of randomly oriented type-I smectic liquid crystals has been discussed by many authors [2,3,29–35]. In those experiments, the random orientation and defects were either stabilized by quench disorder or by putting colloidal particles in the samples. Basappa *et al.* [3] showed in the lyotropic lamellar system that adding a particle enhances the moduli and the dissipation and the elasticity arises mainly due to the defect network. Bandyopadhyay *et al.* [2] showed that the elasticity of 8CB (octyl cyanobiphenyl) liquid crystal in the SmA phase, confined in the aerosil network, was enhanced compared to the pristine sample. The shear modulus of such systems show a power-law behavior

$$G'(\omega) = G_0 + \beta\omega^{\alpha}, \tag{1}$$

where G_0 is the plateau modulus arising from the zero frequency shear modulus. In the present sample, TGB$_A$ is composed of rotating SmA blocks, and hence the random orientation of layers is inherent to the structure, which cannot be removed by the effect of shear. Thus the dynamic response of our sample can be analyzed based on this model. In Fig. 5 we show some representative frequency dependence of storage and loss moduli in all phases. In the N* phase, $G^{'} > G^{''}$, and at the low frequency region ($\omega \leqslant 2$ rad/s) both are constant. They increase with frequency beyond this range. For example, beyond $\omega > 2$ rad/s they vary as $G^{'} \sim \omega^2$ and $G^{''} \sim \omega$. It may be mentioned that solid-like behavior in the low frequency region ($\omega = 1$ rad/s) was also observed in colloidal-particle-dispersed cholesteric liquid crystals. But at higher frequency the same sample showed fluid-like behavior ($G^{'} < G^{''}$) [1].

The fit parameters at different temperatures are shown in Fig. 6(a). In the N* phase (90 to 110° C), α obtained from both the G' and $G^{''}$ data are almost close to 2 and 1, respectively. It is interesting to note that the exponent α obtained from both the fittings of $G^{'}$ and $G^{''}$ decreases rapidly (below 90° C) and is almost equal, i.e., $\alpha \simeq 0.5$. Ramos *et al.* [1] showed that the storage modulus of liquid crystalline systems with a defect network has contributions both from the disoriented part of the sample [$G^{'}(\omega) \propto \omega^{1/2}$] and regions of the sample

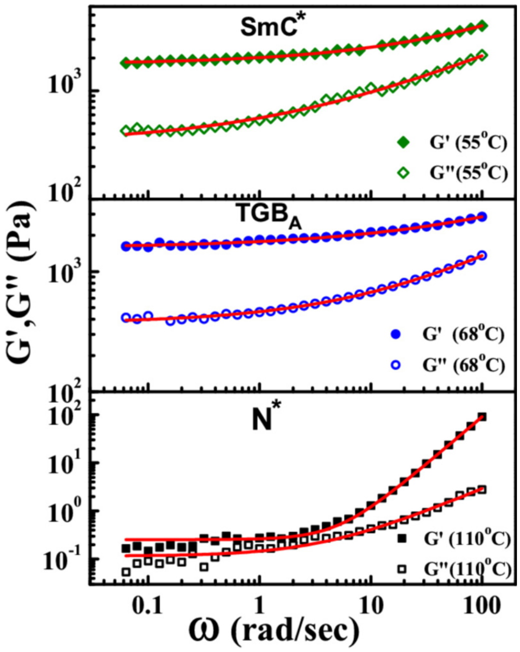

FIG. 5. (Color online) The frequency dependence of (a) storage $G^{'}$(solid symbols) and (b) loss $G^{''}$ (open symbols) moduli at three representative temperatures in all the phases. The solid lines are theoretical fits to Eq. (1).

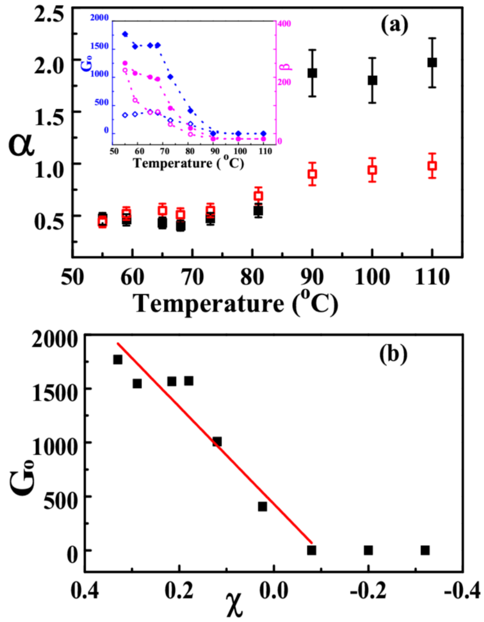

FIG. 6. (Color online) (a) The exponent α characterizing the power-law contribution to $G^{'}(\omega)$(solid squares) and $G^{''}(\omega)$ (open squares) as a function of temperature. (Inset) The inset displays the fit parameters G_o and β obtained from $G^{'}(\omega)$ and $G^{''}(\omega)$ data. Diamonds (solid and open) and circles (solid and open) are obtained from $G^{'}(\omega)$ and $G^{''}(\omega)$ data, respectively. (b) Variation of G_o with reduced temperature χ. The solid line showing the best-fit result $G_o \approx \chi^{\gamma}$, with $\gamma = 1$.

where the layers are parallel to the shear direction [$G^{'}(\omega) \propto \omega^2$]. Similarly, the loss modulus also has contribution from disoriented parts of the sample ($G^{''}(\omega) \propto \omega^{1/2}$) and Maxwell-fluid type a contribution [$G^{''}(\omega) \propto \omega$]. Comparing to our results, it appears that the storage and loss moduli of TGB$_A$ has a significant contribution from the disoriented layers of the sample only. The temperature dependence of G_o and β obtained by fitting $G^{'}(\omega)$ and $G^{''}(\omega)$ is also shown in Fig. 6(a). For example, at 110° C, G_o obtained from both the fits are 0.25 and 0.12, respectively. At the same temperature, β values are 0.01 and 0.03, respectively, and similar values were also reported in other cholesteric samples [1]. The plateau modulus usually arises from the elasticity of static defects in lamellar systems [1,2]. In the present system G_o is much larger ($\simeq$10 times) than the low molecular weight thermotropic SmA liquid crystal, such as 8CB [2]. The variation of G_o with reduced temperature $\chi \equiv (T_{\text{N}^*-\text{TGB}_A} - T)/T_{\text{N}^*-\text{TGB}_A}$ is shown in Fig. 6(b). In the TGB$_A$ and SmC^* phases, G_o is more than three orders of magnitude larger than the N* phase [1]. In analogy with rubber elasticity, the contribution to the elastic response of a static defect network should vary as $G_o \approx \tau/d^2$, where τ is the line tension and d is a typical average spacing between defects [1]. In the case of screw dislocations the defect line tension can be written as $\tau = Bb^4/128\pi^3 r_c^2$, where $b = md_o$ is the Burger's vector of integer strength m and r_c is the defect core radius [36]. Measurements on many smectic systems show that $B \sim \chi^{0.4}$ [37] and $r_c^{-2} \sim \psi^2 \sim \chi^{0.5}$, where ψ is the smectic order parameter [38]. Thus $\tau \sim \chi^{0.9}$, in close agreement with $G_o \sim \chi^{\gamma}$ with $\gamma \approx 1$ [Fig. 6(b)], suggesting that the defects (screw dislocations) significantly contribute to the shear response. It may also be noted that the exponent α in the TGB$_A$ is $\simeq$0.5, and further it tends to decrease in the SmC^* phase. This is somewhat less than that of the lamellar block copolymer $\alpha = 0.6$ [39]. Within the soft glass rheology model, the glass transition is characterized from the power-law exponent $G^{'} \sim \omega^{x-1}$. In this model x, is an effective noise temperature and in the range $1 < x < 2$, $G^{'}$ and $G^{''}$ have a constant ratio [19]. The system approaches the glass transition as $x \longrightarrow 1$, i.e., $\alpha = (x-1) \longrightarrow 0$. In the N*, α obtained from the fitting of $G^{'}$ and $G^{''}$ is very different and it decreases with decreasing temperature and merges to a single value ($\simeq$0.5) upon entering the TGB$_A$ and SmC^* phases. Though the TGB$_A$ and SmC^* are thermodynamically stable phases, the experimental x value is about 1.5, indicating that their rheological responses are similar to many soft glassy materials. It may be mentioned that the adapted model does not include the effect of defect nucleation and annihilation. Theoretically, shear-induced defect nucleation and annihilation in a flow-aligned nematic liquid crystalline polymer has been reported by Grecov *et al.* [40]. Experimentally, the effect of such a process on the shear rheology of low molecular weight liquid crystals is not well established. Nevertheless, this effect is expected to be less important in the TGB$_A$ as this phase is stabilized by interacting dislocations and the dislocations are inherent to the structure.

IV. CONCLUSION

In conclusion, we have studied the rheology of a binary mixture showing a large temperature range of the TGB$_A$ phase. All the phases in the mixture, i.e., N*, TGB$_A$, and SmC^* exhibit solid-like behavior at low frequencies and low strain amplitude. The elasticity of the TGB$_A$ phase is discussed based on the theory in lamellar systems that accounts for the contribution of defects. The power-law dependence of the complex shear modulus and the analysis of the experimental result suggests that the elasticity of the TGB$_A$ phase is due to the structural defects. A more quantitative assessment should be able to provide the prominence of screw dislocations and perhaps the grain boundaries. The effective noise temperature approaches 1, suggesting TGB$_A$ liquid crystals and its low temperature phases are a defect-mediated soft solid whose dynamics is similar to soft glassy materials.

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