Shear-banding phenomena and dynamical behavior in a Laponite suspension

F. Ianni,[1,2,*] R. Di Leonardo,[2] S. Gentilini,[1] and G. Ruocco[1,2]
[1]*Dipartimento di Fisica, Università di Roma "La Sapienza," I-00185, Roma Italy*
[2]*SOFT-INFM-CNR c/o Università di Roma "La Sapienza," I-00185, Roma Italy*
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Shear localization in an aqueous clay suspension of Laponite is investigated through dynamic light scattering, which provides access both to the dynamics of the system (homodyne mode) and to the local velocity profile (heterodyne mode). When shear bands form, a relaxation of the dynamics typical of a gel phase is observed in both bands soon after the flow stops. Periodic oscillations of the flow behavior, typical of a stick-slip phenomenon, are also observed when shear localization occurs. Both results are discussed in the light of various theoretical models for soft glassy gels.

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I. INTRODUCTION

Under the influence of a shear flow, many complex fluids exhibit the formation of macroscopic bands, parallel to the flow direction and characterized by different local shear rates. This phenomenon is called *shear banding* and is attributed to the presence of a decreasing branch in the stress-shear rate curve: in a controlled shear experiment, it is observed when the stress falls in the interval where the so-called flow curve is multivalued. Experimentally, shear banding has mainly been observed in a class of complex fluids, like wormlike micelles [1–4], colloidal crystals [5], or lamellar surfactant systems [6,7], where a phase transition associated with the microstructure occurs under flow. As described by various theoretical models [8–12], in such systems an ordered phase coexists with a disordered one in the shear-banding regime.

However, shear banding has also been observed in soft glassy materials [13], like foams [14,15], emulsions [16], or glassy suspensions [16–18], where a homogeneous structure is kept throughout the system. To our knowledge, few experimental results are available in the literature for this class of systems [15–18], while the microscopic origin of the phenomenon is poorly understood. Soft glassy materials are characterized by a viscosity that increases many orders of magnitude as time evolves and the gelation process proceeds (*aging* behavior), and have rheological properties typical of soft solids, such as solidlike behavior below a finite yield stress and the shear-thinning effect [19]. At the microscopic level, the gelation process corresponds to a slowing down of the structural relaxation with the elapsed time, while shear thinning results from the system structural dynamics being accelerated by a shear flow (shear *rejuvenation*).

A shear-banding behavior is expected for such systems, due to the presence of a yield stress, which provides a multivalued region in the flow curve. When heterogeneous flow emerges, a band with null local shear rate, flowing as a solid block, may coexist with a band flowing at a finite local shear rate. This behavior has been called shear localization and, according to numerical and theoretical models [20,21], is associated with a dynamical transition: an arrested dynamics characterizes the unsheared band, while the sheared band exhibits a liquidlike behavior. Moreover, these works have evidenced the emergence of a fluctuating shear-banded flow, a phenomenon also observed in nonglassy shear-banding systems [22]. In order to describe this fluctuating behavior, a model accounting for intermittent plastic and elastic events has been developed [23,24]. At the experimental level, a fluctuating shear-banded flow has been observed in glassy suspensions [17,18] and in emulsions [14,15], while direct investigation of the system dynamics when shear localization occurs is still missing.

Among others, proper candidates for the study of the shear localization phenomenon are suspensions of charged anisotropic colloidal particles such as clay, which have been widely investigated both for their important industrial applications [25] and as prototypes of glassy systems [26]. Shear localization has been observed in such suspensions through magnetic resonance imaging (MRI) [16,27] or visualization techniques [17]. In particular, in Ref. [17], shear localization is studied in relation to the rheological behavior of the sample and by varying the concentration of the suspension, while an oscillating behavior of the shear stress is observed at low shear rates. However, an exhaustive comprehension of shear localization phenomenon in such systems is still lacking. More specifically, a study of the system dynamics would be essential in order to verify if the unsheared band is dynamically different from the sheared band, as suggested in Refs. [20,21]. Moreover, the existence of periodic oscillations in the shear flow, predicted by Ref. [21], still has to be evidenced by direct measurements of the fluid velocity profile.

In this paper, we investigate the shear localization phenomenon in an aqueous clay suspension of Laponite, a highly thixotropic liquid which undergoes structural arrest. We are interested both in the study of the system dynamics when the shear bands form and in the fluctuating flow phenomenon. The technique that we use is dynamic light scattering (DLS), which provides access both to the dynamics of the system after shear cessation (in the homodyne mode) and to the local velocity profile during the flow (in the heterodyne mode). In particular, once a shear-banding profile has been detected, we monitor the evolution of the system dynamics soon after flow cessation in both the flat and the

*francesca.ianni@phys.uniroma1.it

sheared bands. A relaxation of the dynamics typical of a gel phase is observed in both regions, with a qualitatively similar behavior. A more quantitative interpretation of these results, in the light of a theoretical model for anomalous dynamic light scattering in soft glassy gels [28], suggests that a slow microcollapse mechanism characterizes the flat band and a fast microcollapse mechanism the sheared band. For the investigation of the fluctuating flow instead, the heterodyne mode provides higher spatial and temporal resolution than MRI or other techniques previously used [17,18]. We thus observe periodic oscillations in the flow, which are typical of a stick-slip behavior: a layer reversibly fractures and reheals, fluctuating between a frozen state (slip) and a fluidized state (stick). Our results on the dynamics of the shear-banding system and on the fluctuating behavior of the velocity profile show strong analogies with the elastoplastic model of Ref. [23], which describes the flow behavior of a yield-stress fluid.

II. MATERIALS AND METHODS

Aqueous Laponite suspension is a thixotropic yield-stress fluid [29,30], which has been extensively investigated as a model system for glassy suspensions, both from a microscopic and from a rheological point of view [27,31–33]; the rejuvenating effect of a shear flow on the system dynamics has also been studied [34–36]. Laponite particles are disk shaped with a diameter of 25 nm and 1 nm thickness and become negatively charged on the faces when dispersed in a polar solvent. For our experiments, Laponite powder, provided by Laporte Ltd., is dispersed in ultrapure water at 3 wt % concentration and stirred for about 30 min. The obtained suspension, which is optically transparent and initially "liquid," is loaded through a 0.45 μm filter into a homemade, disk-disk shear cell for DLS measurements. The cell has a glass disk, of diameter $d=10$ cm, as the rotating plate, and an optical window as the static plate. The cell gap is $h=7$ mm and we fix the y axis conventionally along this direction, corresponding to the velocity gradient direction under flow.

In the setup we implemented for DLS measurements [37], an incident laser beam (diode-pumped solid-state laser, $\lambda=532$ nm, $P=150$ mW) impinges on the sample passing through the optical window. The scattered light passes through the same window and is collected by a monomode optical fiber. Optionally, it can interfere with a coherent local oscillator field (heterodyne mode) through a fiber collection apparatus. Collected light is then detected by a photomultiplier and analyzed by a homemade software correlator [36]. The scattering geometry is fixed, with a scattering angle $\theta=132°$ and thus a scattering vector $q=22$ μm^{-1}. We placed the shear cell in order to have the scattering volume positioned at a radial distance of 2.1 cm from the rotational axis.

The shift of the cell allows us to select the position of the scattering volume in the cell gap and the possibility of choosing a heterodyne correlation scheme enables direct access to the detailed velocity profile [38]. Due to the Doppler effect, the velocity v of the particles in the scattering volume can indeed be obtained from the frequency ω of the collected oscillating intensity in heterodyne mode: $v=\omega/(q\cos\widehat{\boldsymbol{qv}})$, where $q\cos\widehat{\boldsymbol{qv}}$ is fixed by the scattering geometry. The frequency ω is simply obtained as the peak location in the power spectrum of the intensity fluctuations, acquired in a time interval $0-T$. The maximum time resolution that we can achieve with this measurement, at a fixed position of the scattering volume in the gap, is $T=10^{-2}$ s, while the spatial resolution is determined by the scattering volume dimension ~ 100 μm.

The dynamics of the system can be investigated through the normalized correlation function of the scattered intensity (homodyne mode) $g^{(2)}(t',t)=\langle I(q,t)I(q,t')\rangle/\langle I(q)\rangle^2$, which is easily represented in terms of the particle correlation function. In particular, homodyne DLS directly probes the intermediate scattering function of the colloidal particles, $F_q(t,t')=\langle\rho_{-q}(t)\rho_q(t')\rangle/\langle|\rho_q|^2\rangle$, which plays a central role in both theoretical and numerical approaches to glassy dynamics: $g^{(2)}(t,t')=1+|F_q(t,t')|^2$ [39]. However, due to geometrical decorrelation effects during the flow [40], the intermediate scattering function cannot be detected when the system is under shear. Therefore, we will follow the system dynamics soon after shear cessation and try to deduce some information on its dynamical behavior during the flow. In classical DLS, the correlation function is calculated as an average over the time origin. In aging systems, this is possible when the experimental acquisition time needed to get a good signal to noise ratio is longer than the characteristic slow relaxation time of the system, τ, and shorter than the time one should wait before changes in τ, due to the aging process, are significant. This condition does not hold when the aging dynamics is characterized by a very fast evolution, as happens soon after flow cessation in a shear-banding Laponite sample. Therefore, we cannot obtain the intensity correlation function by time averaging, and an ensemble average over many rejuvenating experiments is used instead [36]: $g^{(2)}(t_w,t)=\langle I(q,t_w)I(q,t_w+t)\rangle_e/\langle I(q,t_w)\rangle_e^2$, where $\langle\cdot\rangle_e$ indicates the ensemble average over several intensity evolutions acquired after cessation of a repeated shear application. The flow stop is taken as the origin of the waiting times t_w. We choose the following protocol: a global shear rate $\dot{\gamma}_1$ is applied to the system for a time interval T_1; after shear cessation, the intensity fluctuations are collected for a time interval T_0 with a time resolution of dt; then a shear rate of the same value $\dot{\gamma}_1$ is applied for T_1 and the cycle starts again. The intensity autocorrelation function is then calculated in the time window $dt-T_0$ as an ensemble average over all the bunches of counts. The stability of the results with respect to variations of T_1 and T_0 has been checked. Aging of the sample under shear [36] is negligible during the whole experiment, as the same results are obtained if we calculate the correlation functions by taking only the first or last group of acquired counts.

III. RESULTS I: INVESTIGATING THE DYNAMICS

After cell loading, we let the Laponite sample age and wait for the dynamical arrest of the system, which is reached after about 18 h when the slow relaxation time diverges [36]. A shear rate of the order of 1s^{-1} is then applied to the arrested sample 20 h after cell loading and the shear localiza-

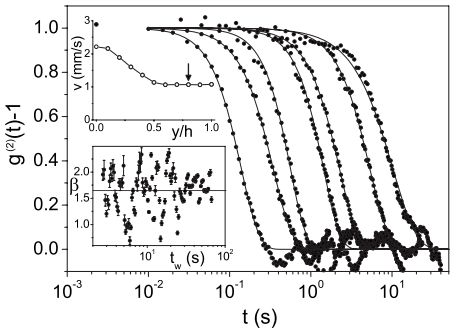

FIG. 1. Normalized intensity autocorrelation functions measured in the flat band soon after shear cessation. 103 correlation functions are calculated through an ensemble average over many shearing experiments in the range of waiting times 3–67 s and seven curves for logarithmically equidistant waiting times in the interval are plotted (from left to right). A compressed exponential fit $\exp[-(t/\tau)^{\beta}]$ is also plotted for each curve. In the bottom inset, the β parameter for the 103 correlation functions is plotted as a function of the waiting time since flow stop: its value fluctuates greatly around the average value 1.7, which is plotted as a line. In the top inset, the velocity profile of the fluid during the experiment is reported, and the arrow represents the position of the scattering volume in which the measurements were performed. The full circle represents the plate velocity and evidences the presence of wall slip.

tion phenomenon is observed. If we let the sample age for a longer time (more than two days), drastic wall slip takes place and the whole system rotates as a solid body, leaving a null shear in the core. But a shear-banding regime can still be reached if the solid band is broken through the application of a high shear rate ($\sim 100\ s^{-1}$) for a few minutes, followed by a few hours of aging. A typical velocity profile observed during the shear-banding regime is shown in the top inset of Fig. 1. Here the velocity profile along the cell gap, measured through heterodyne DLS, is plotted: a flat band forms next to the static window, while wall slip occurs at both sides. According to theoretical models [20,21], soft glassy materials exhibiting shear localization are characterized by an arrested dynamics in the flat band and a fluidlike dynamics in the sheared band. We thus want to investigate the dynamics in both bands of our sample. As we cannot access the dynamical behavior of the system during the flow directly, we will follow the dynamics after shear cessation and then deduce some information on the shear localization regime.

Through the acquisition method explained above, and fixing the global shear rate $\dot{\gamma}_1 = 0.4\ s^{-1}$ and the time intervals $T_1 = T_0 = 120$ s, we first monitor in the flat band the evolution of the correlation functions with the waiting time since flow stop, as shown in Fig. 1. These data seem to evidence an aging behavior after shear cessation, as if the flow had a rejuvenating effect on the dynamics of the flat band. However, a null shear rate is not supposed to modify the structure of the system and induce an acceleration of the structural dynamics [41]. In order to exclude the possibility that the observed behavior is due to any residual shear rate in the flat band, we monitor the dynamics after the application of a small strain to a gelled sample. In this case, the flow should not modify the structure of the system, as it is supposed to

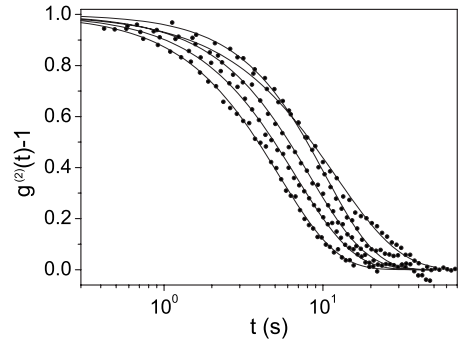

FIG. 2. Normalized intensity autocorrelation functions measured in the flat band soon after a brief strain application: an aging behavior, qualitatively similar to the one observed after a shear flow, is evident. Correlation functions are obtained through the ensemble average procedure for five logarithmically equidistant waiting times t_w between 3 and 17 s after flow cessation. A compressed exponential fit $\exp[-(t/\tau)^{\beta}]$, with $\beta \geq 1$, is also plotted for each curve.

deform the material only elastically. Therefore, a strain $\Delta x/h = 0.3$ of the duration of 1s is applied cyclically to a gelled sample, and the intermediate scattering function soon after strain application is measured as an ensemble average, following the same procedure already described. As evidenced in Fig. 2, an aging dynamics with the same qualitative behavior is still evident after flow cessation, showing that it is not due to a shear rejuvenating effect.

The system dynamics in the sheared band is investigated through the same DLS technique and the evolution of the intermediate scattering functions after a shear flow are plotted in Fig. 3. The same qualitative behavior is observed in the form of the correlation functions and in their evolution with t_w, as also described elsewhere [36].

Interpretation of the results

The investigation of the dynamics in the shear localization regime thus results in an aging behavior in both bands, with

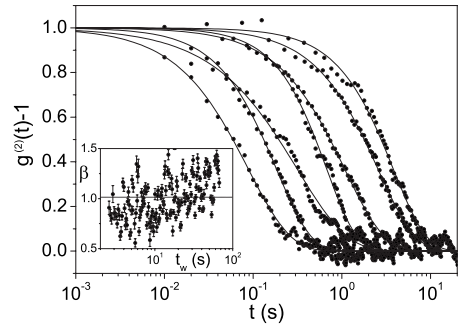

FIG. 3. Normalized intensity autocorrelation functions measured in the sheared band soon after the application of a local shear rate $\dot{\gamma} = 0.5\ s^{-1}$. 163 correlation functions are calculated through an ensemble average over many shearing experiments in the range of waiting times 3–67 s and seven curves, for logarithmically equidistant waiting times in the interval, are plotted with a compressed exponential fit $\exp[-(t/\tau)^{\beta}]$. In the inset, the β parameter for the 163 correlation functions is plotted as a function of the waiting time since flow stop: its value fluctuates greatly around the average value 1.0, which is plotted as a line.

the correlation functions evolving with the waiting time after flow stop and keeping the form $A\ \exp[-(t/\tau)^{\beta}]$. This result is in contrast with the models describing shear localization in soft glassy materials [20,21], where a dynamical transition is expected: there, the unsheared band is characterized by an arrested dynamics, while the sheared band exhibits a liquidlike behavior.

However, the observed behavior of the intermediate scattering function can be interpreted in the light of a model developed to describe anomalous dynamical light scattering in soft glassy gels [28]. There, the system behaves as an elastic medium [42] and force dipoles appear at random in space and time, inducing microcollapses of the structure due to a local stress relaxation mechanism. The decorrelation of light scattered by this model system is not due to the dynamics of single scatterers, but to a drift mechanism of big aggregates of particles, resulting in a normalized correlation function of the form $\exp[-(t/\tau)^{\beta}]$, where the effective exponent β depends on the time scale of these microcollapse events with respect to the experimental time t (which spans the whole time window where the correlation function is plotted). Moreover, an aging behavior is accounted for by the model, where it is interpreted in terms of strain-dependent energy barriers.

In analogy to this model for soft glassy gels, the form and the evolution of the correlation functions collected for the Laponite sample in both bands seem to suggest such elastic medium behavior, in which the system relaxes to equilibrium through microcollapse events after the application of a stress. In order to attempt a more quantitative relation between this model and our results, we have plotted, for both bands, the β parameter from the fit of the normalized correlation functions (insets of Figs. 1 and 3). Though highly noisy, the effective exponent does not seem to depend significantly on the waiting time in the range here investigated and keeps on fluctuating around an average value, corresponding to 1.7 for the flat band and to 1.0 for the sheared band. According to the model described above, an effective exponent $\beta \geq 3/2$ results in the slow collapse regime, i.e., when the experimental time (here scanning about two decades) is smaller than the time scale of the collapse events. Vice versa, an effective exponent $\beta=1$ results in the fast collapse regime.

Therefore, instead of showing a dynamically arrested phase in the flat band and a fluidlike dynamics in the sheared band, as in soft glassy models, the Laponite sample seems to show a gel-like behavior in both bands, but with a different time scale in the microcollapse mechanism. In particular, our data are coherent with the presence of very slow collapse events in the flat band and very fast collapse events in the sheared band with respect to the experimental time [43]. At a first glance, the supposed gel structure in the nonflat band seems to be incompatible with a shear flow. However, a gel-like behavior after the application of a shear flow has already been observed in a Laponite suspension [36]. The problem may be solved by supposing that, though the sample is sheared, aggregates of gel phase larger than the scattering volume ($\sim$100 μm) exist and slip one over the other during the flow. After shear, the dynamics in the scattering volume would thus be characterized by a gel behavior in the sheared band also. Further experiments should be made to clarify whether such nonhomogeneous flow is effectively present in the sheared band.

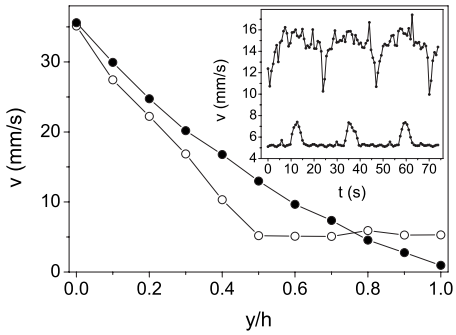

FIG. 4. A global shear rate $\dot{\Gamma}=4.5\ s^{-1}$ is applied to a very old Laponite sample and the velocity profile is observed to oscillate between the two extreme profiles plotted. The time period is $T \simeq 23$s. In the inset, the oscillations of the particle velocity when the scattering volume is at $y/h=0.4$ (top) and 0.7 (bottom) are plotted. The small and fast oscillations visible in the bottom curve of the inset have the same period as the disk rotation $T_{rot} \simeq 4$ s. They are due to a slight misalignment of the rotational axis (with respect to the orthogonal to the window plate), which induces a small oscillation of the gap width ($\sim$8%).

IV. RESULTS II: OSCILLATIONS IN THE FLOW BEHAVIOR

Here we report on the observation of periodic oscillations of the shear-banding velocity profile, exhibited by very old samples. By monitoring the evolution of the particle velocity through heterodyne DLS, at different positions along the gap, we are able to rebuild the two extreme profiles between which the system oscillates. An example is represented in Fig. 4, where it is evident that the velocity profile oscillates between a configuration where the shear is localized next to the rotating plate and a configuration with a linear profile. Most of the time, the system lies in an intermediate profile between the two. The oscillation period is larger than the disk rotation period and remains constant on the time scale of hours.

Periodic oscillations of the flow behavior when shear localization occurs have been predicted by the phenomenological model presented in Ref. [21] and are here observed by directly accessing the velocity profile with a high enough time resolution. In the theoretical model, such behavior is interpreted as a stick-slip phenomenon: when shear localization occurs, a diverging viscosity characterizes the unsheared band, which thus slips on the wall; while in the linear profile configuration, the system has a liquidlike behavior and sticks on the wall.

V. CONCLUSIONS

Summarizing, the investigation of the shear localization phenomenon on a Laponite sample has provided two main results: (i) through homodyne DLS, the relaxation of the structural dynamics has been followed in the two shear bands

soon after flow stop and a stress relaxation behavior typical of a gel phase has been evidenced; (ii) through heterodyne DLS, periodic oscillations of the shear-banding profile, reminiscent of a stick-slip phenomenon, have been observed. Taking into account both the oscillating flow behavior and the stress relaxation mechanism, our experimental results strongly support the elastoplastic model proposed in Ref. [23] to describe the flow behavior of a yield-stress fluid. Two generic ingredients build up the model, leading to a complex spatiotemporal behavior of the system: local plastic events occur above a microscopic yield stress and nonlocal elastic release of the stress follows [44]. At low shear rates, an intermittent flow localization emerges, with spatially correlated structures forming parallel to the walls; while at high shear rates the flow is homogeneous. The stress relaxation behavior and the oscillating flow localization that characterize this model have both been observed in our experiments on a Laponite sample. This suggests that such an elastoplastic mechanism occurs in the sample, and our two main results may be interpreted as two different aspects emerging from this mechanism.

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[43] In our measurements, the experimental time spans about two decades, increasing on average with the waiting time at which the correlation function has been collected (Figs. 1 and 3). However, the β parameter does not seem to depend on the waiting time in both bands, as if the collapse time stays far from the experimental time during the whole experiment and the system remains in the fast (slow) collapse regime. The collapse mechanism thus seems to happen on very different time scales in the two bands, though the evolution of the relaxation time τ with t_w is quantitatively similar in both bands (as shown by the evolution of the correlation functions). The fast collapse mechanism may be compensated by a lower efficacy in decorrelating the structure, thus providing the same relaxation time that results from the slow collapse mechanism. Further experiments should be made in order to verify this conjecture.

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