RHEOMETRY OF FLUIDS WHICH EXHIBIT WALL DEPLETION IN SIMPLE SHEAR

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The paper is dedicated to the rheological investigations of materials which exhibit wall depletion phenomena in viscometric flows, such as real or apparent slip or shear banding formation. The rheometry of the tested soft matter materials is characterized by an unstable constitutive relation with non-monotonic steady flow curve. As consequence, the samples show a yield state, defined by a critical value of the shear strain. In the absence of a general model to explain and to correlate the experiments with theory, the steady viscosity function is experimentally undetectable for the shear rates belonging to the domain of instability.

Keywords: rheometry, rheology, viscosity, material instability, yield state

1. Introduction

Wall depletion is a general concept associated with phenomena such as: (i) wall slip (apparent or not), (ii) shear banding, (iii) presence of yielding, observed mainly during the rheological shear tests of some complex materials, generic called soft matter. In this category of materials are included complex fluids characterized by a concentrated phase (solid or liquid) dispersed in a homogeneous liquid (as pastes and creams) [1]. Samples as entangled polymers, dense suspensions, wormlike micellar solutions, lubricated greases, metastable colloidal systems (e.g., gels) and soft particle glasses are also considered from the rheological point of view soft solids, [2-5].

The shear banding and yield stress are very generous and challenging topics, offering researchers and scientists from different fields multiple directions to be considered for theoretical and applied studies. The works related to these subjects and published in the last decades are issued from numerous and diverse sources.

In this respect, several papers [3, 6-16] and reviews [17-24] have a relevant impact on the subject. Since the presence of shear banding cannot be

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directly observed during the rheometry of the samples by using commercial rheometers, constitutive instability and the presence of plateau in the measured flow curves (i.e. the shear stress dependence on the shear rate) are often considered spurious phenomena, induced by the real material slip at the boundary, [3, 14]. However, many measured flow curves of materials adhering to the boundary indicate the existence of a yield state, which is consistent with the measured shear stress plateau in the flow curve, [1-3].

The aim of the present paper is to test whether the models with non-monotonic flow curves can match the experimental data measured with commercial rotational rheometers in simple shear flows. In particular, we are looking to find the answer to a direct question with relevance for experimental rheology: *Can the viscosity function for this category of materials be measured?*

2. Material instability and non-monotonic flow curve

In a steady isochoric viscometric flow the extra-stress tensor $\mathbf{T}$ is characterized by one single shear stress, $\sigma = \sigma(\dot{\gamma})$, which is an odd function (called flow curve) of the shear rate $\dot{\gamma} := \sqrt{4|I_2|}$, where I_2 is the second invariant of the stretching $\mathbf{D}$, with $tr\,\mathbf{D} = 0$.
The shear viscosity material function,

$$\eta(\dot{\gamma}) := \frac{\sigma}{\dot{\gamma}} \tag{1}$$

is considered to be locally defined and steady.

It is important to make distinction between the viscosity function (1), as a material function, and the transient shear viscosity function $\eta_t\left(\dot{\gamma}(t), t\right)$, which is a function of the time dependent shear rate (in general) and the time t. The formal definition of the transient shear viscosity is the same with (1), but with at least one time-dependent quantity (σ or $\dot{\gamma}$, or both). The transient shear viscosity function at constant shear rate, assuming zero inertia (i.e. the Reynolds number $Re \to 0$), is associated with viscoelasticity, since the presence of time as independent variable is introduced by the time dependence of the stress tensor [16].

Analytical expressions for the viscosity function can be normally obtained using either the molecular network theories [25, 26] or the formulations of the continuum constitutive relations; semi-empirical models are also very popular, and much used in applications [27]. A representative example for the viscosity function is the Carreau-Yasuda model. It is considered one of the most versatile expression for the viscosity function which can fit many of the experimental data:

$$\frac{\eta(\dot{\gamma})-\eta_\infty}{\eta_0-\eta_\infty}=\left[1+(\lambda\dot{\gamma})^m\right]^{\frac{n-1}{m}} \tag{2}$$

where n [-] is the shear index, m [-] and λ [s] the material constants. Here the coefficients η_0 and η_∞ are the zero and infinite viscosities, respectively. With $n=1$ the fluid is Newtonian; if $n<1$ the behavior is shear thinning. The parameter $m=2$ defines the Carreau model [25-27].

A negative shear index determines the non-monotonicity of the steady flow curve associated with (2), i.e. to a given value of the shear stress σ_0 it corresponds three values of the shear rates, see Fig. 1.

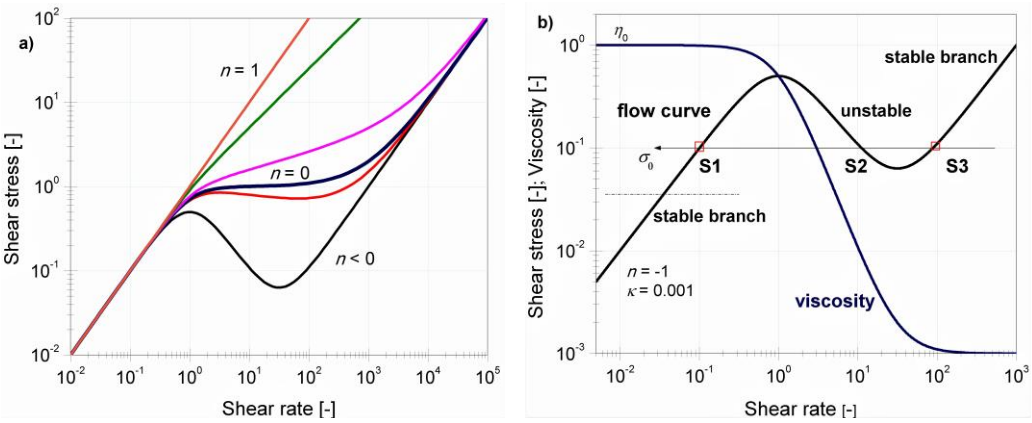

Fig. 1 Non-dimensional representation of the flow curve and viscosity function of the Carreau model (2): a) The flow curve as function of the shear index; b) The unstable flow curve

Similar non-monotonic flow curves with that shown in Fig. 1 are obtained for different models. One classical example is the Oldroyd 3-constants model:

$$\lambda_1\frac{D_i\mathbf{T}}{Dt}+\mathbf{T}=2\eta_0\lambda_2\frac{D_i\mathbf{D}}{Dt}+2\eta_0\mathbf{D}\,, \tag{3}$$

where

$$\frac{D_i\circ}{Dt}=\frac{\mathrm{d}\circ}{\mathrm{d}t}-\mathbf{\Omega}\circ+\circ\mathbf{\Omega}-a_i\left(\mathbf{D}\circ+\circ\mathbf{D}\right) \tag{4}$$

are the objective Gordon-Schowalter time derivatives with $\mathbf{\Omega}$ the spin tensor and $\frac{\mathrm{d}\circ}{\mathrm{d}t}$ the material derivative, λ_1 and λ_2 being the relaxation and the recovery times.

The additional "slipping parameters", $a_i\in[-1,\ 1], i=1,2,$ define the type of objective derivatives for the extra-stress and for the stretching, respectively.

The convected derivatives are defined for $a = a_i = \pm 1$ (classical Oldroyd models with constant viscosity) and $a_i = 0$ defines the Jaumann (corotational) derivative [26-28]. The original model with non-monotonic flow curve proposed by Johnson and Segalman [29] for polymer solutions with non-affine deformation uses a single slipping parameter, $a = a_i \neq \pm 1$. Different objective derivatives for the extra-stress and stretching have been introduced by Balan and Fosdick [11].

One can observe that for $a = 0$ the steady viscosity (1) is identical with that given by the Carreau model (2) ($m = 2$ and $n = -1$), Fig. 1. Both shear thinning viscosity functions are associated with the non-monotonic flow curve:

$$\sigma = \eta_0 \dot{\gamma} \frac{1 + \kappa \lambda^2 \dot{\gamma}^2}{1 + \lambda^2 \dot{\gamma}^2}, \tag{5}$$

for $\kappa < 0.1$, with $\kappa = \lambda_2 / \lambda_1$ and $\lambda_1 = \lambda$, see Fig. 1b.

In viscometric flows (i.e. simple shear), for $a_1 = a_2 = a = 0$ and constant shear stress, relation (3) is an equivalent in non-dimensional form with the differential equations:

$$\begin{aligned} &\dot{\bar{N}}_1(\bar{t}) + \bar{N}_1(\bar{t}) = 2\dot{\bar{\gamma}}(\bar{t})\bar{\sigma} - 2\kappa \dot{\bar{\gamma}}(\bar{t})^2 \\ &\bar{\sigma} = \kappa \ddot{\bar{\gamma}}(\bar{t}) - \dot{\bar{\gamma}}\left(\frac{\bar{N}_1(\bar{t})}{2} - 1\right) \end{aligned} \tag{6}$$

where $\bar{\sigma} = \dfrac{\sigma\lambda}{\eta_0}$, $\dot{\bar{\gamma}} = \lambda\dot{\gamma}$ and $\bar{N}_1 = \dfrac{\lambda(\sigma_{11} - \sigma_{22})}{\eta_0}$ is the first normal stresses difference, [28].

The system (6) describes the dynamics of the constitutive relation (3) in viscometric flows, respectively in any configuration used in rheometry (plate-plate, cone-plate, double cylinders geometries), [30].

In the flow curve (5), at $\sigma = \sigma_0$, the steady solutions S1 (node) and S3 (foci) are stable and S2 is unstable (saddle), see Fig. 1b. Therefore, the region of the flow curve between S1 and S3 is unstable; it is sometimes called the „plateau" in shear rate, corresponding to the constant imposed shear stress σ_0. In this case, the time evolution of the system to the steady solutions is determined by the initial values of the shear rate and the normal stresses, [28].

3. Experimental non-monotonic flow curves

In a rheological test performed with commercial rotational rheometers, there are two possibilities for the input and output respectively (depending upon the test type - strain or stress controlled): the rotational velocity ω of the upper plate or the torque T on the upper plate. Normally, the lower plate is always at rest, the control and measurement being performed only on the upper plate (not the case for the Anton Paar MCR 702 which offers data for both plates).

Therefore, we never control or measure the local shear rate or the corresponding shear stress, in one particular fixed point within the material. For a complex fluid, inside the gap the steady velocity distribution can take any of the configurations shown in Fig. 2, without noticing which one is real (in the absence of a visualization system).

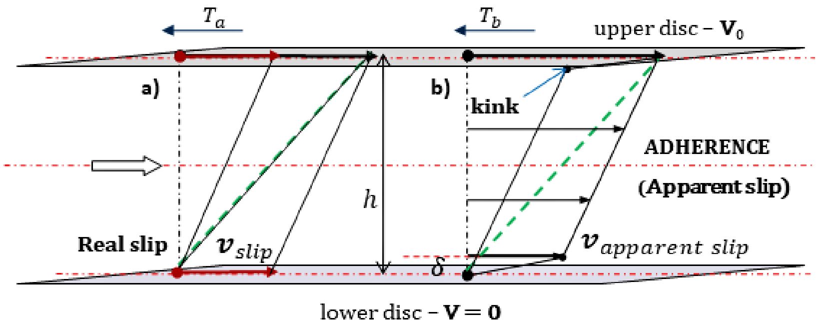

Fig. 2 Possible steady flow configurations in the gap h of a rheometer with the upper plate velocity V_0 and the measured torques T_a, respectively T_b; a) real slip, and b) apparent slip

A priori, all conditions are assumed to be met in order to sustain the validity of the classical formulas (implemented in the software of the rheometer) which relate the mentioned kinematic and dynamic quantities measured on the moving surface with the dimensions of the working geometry:

$$\dot{\gamma} = \kappa_1 \omega \text{ and } \sigma = \kappa_2 T , \tag{7}$$

where κ_1 and κ_2 are constants for each geometry, [30].

Neither $\dot{\gamma}$ nor σ from (7) are locally defined. So, we cannot be sure if the data provided by experiments represent the same quantities as those from the theory. For this reason, the measured shear rate and shear stress are sometimes called “apparent” (especially the shear rate).

In our opinion, to distinguish the two cases shown in Fig. 2 we have to analyze the experimental data in the framework of the theoretical results previously presented.

The tested materials are a lubricating grease (dense concentration of Lithium micro-fibers in a viscous mineral oil) at two temperatures (LG1 at 25° C and LG2 at 30° C) and one commercial cosmetic cream. The measurements have been performed with Paar-Physica MCR 301, Anton Paar MCR 702 and TA Instruments AR-G2 rheometers using the original smooth plate – plate (PP) and cone – plate (CP) configurations.

An important issue related to the experiments is the possible existence of the real slip, since some rheologists may argue that lack of adherence can't be avoided for these samples, slip being "an intrinsic feature of the response of disperse systems in a rheometer", [14], see also [31, 32]. The influence of the gap magnitude in the plate – plate configuration is not investigated in the present paper, since the gap was fixed at a relative high value, respectively 200 μm (for details on this subject, see [33]). Each test was repeated several times with new samples and the measurements marked by edge effects, ejection form the gap or suspicion of real slip were eliminated.

One main difficulty in interpreting the measured data is the dependence of the results of the input parameters. Since a steady state is hard to be reached in a finite time, [2], the measured flow curves seem to be chaotic in a certain range of the shear rates. Fig. 3 depicts the flow curves of a cosmetic cream measured at different slopes of the input parameter, in this case the shear stress.

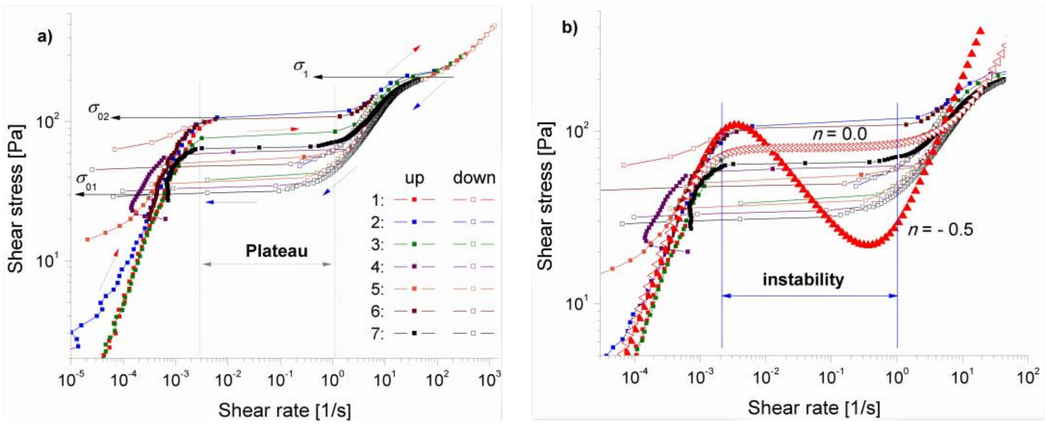

Fig. 3 a) Transitory flow curves for a cosmetic cream at different inputs of the shear stress sweep. The plateau is found to be in the shear stresses range $(\sigma_{01}, \sigma_{02})$; also, a secondary plateau is observed for high shear rates at σ_1. b) The qualitative fitting of the data with the Carreau model (Paar-Physica MCR 301, PP geometry)

These flow curves are not steady in all the measured points and they show different values for the range of shear rates between $3\cdot 10^{-3}$ s^{-1} and 1 s^{-1}, Fig. 3a. However, from a qualitative point of view, all the curves are similar and prove the existence of a plateau in that region of shear rates, which actually corresponds to the instability domain of the non-monotonic steady flow curve, Fig. 3b. We mention that quantitative fitting of the experimental data usually needs 3-4 similar models (5) connected in parallel [34].

The results for the transitory flow curves of the lubricating grease LG1 are shown in Fig. 4. In Fig. 4a the input ramp for the shear stress (i.e. the creep test) was linear (from 0 Pa to 1000 Pa in 200 s) and for the rate the input ramp was logarithmic (from 10^{-3} to 10^{2} in 200 s).

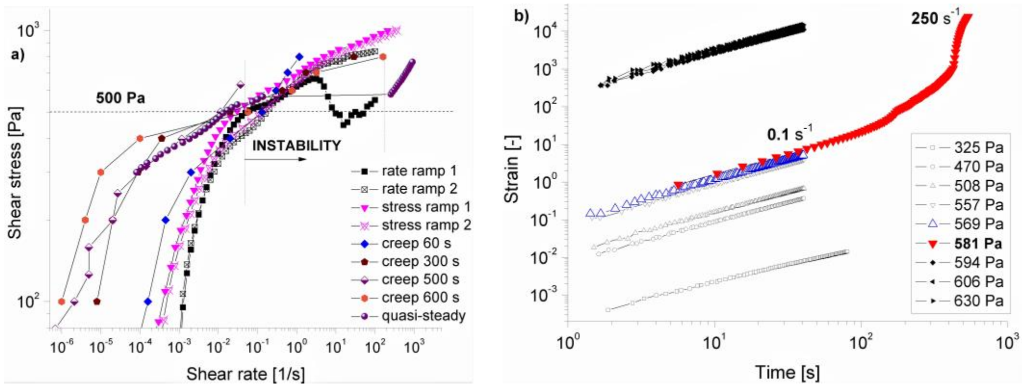

Fig. 4 a) Flow curves for LG1 sample. b) The time dependence of strain corresponding to some constant values of the shear stress in the quasi-steady experiment from (a). The jump at 581 Pa was observed between the rates of 0.1 s^{-1} and 250 s^{-1} (Anton Paar MCR 702, PP geometry)

The values of shear rate corresponding to the imposed constant value of the shear stress in creep test are recorded at different experimental times. In the quasi-steady test, a step-ramp in shear stress is imposed, but each point is measured at a constant shear rate, or for an imposed time limit. It is interesting to remark that equilibrium was not obtained for the stress of 581 Pa, Fig. 4b, which in this case corresponds to the onset of the jump between the two metastable branches of the non-monotonic flow curve from Fig. 1b. Therefore, this yield stress is located in the vicinity of this value.

The time variation of viscosity shown in Fig. 5a is a proof of the non-monotonicity flow curve and for the existence of the two stable attractors, S1 and S3, see also Fig. 1b. The resulting viscosity curve for the sample LG1 from the creep tests at 300 s and the quasi-steady tests are shown in Fig. 5b; one can observe: (i) the shear thinning behavior (expected), and (ii) the tendency to have a limit of viscosity at very low shear rates (i.e. the zero shear viscosity).

We continue the investigations on LG1 sample by applying a series of creep tests at 500 Pa, using different geometries of the AR-G2 rheometer: plate – plate and cone – plate (25 mm diameter and angle of 0.1 rad). The time interval is longer for these experiments, up to two hours, Fig. 6. The results indicate that tests performed with plate – plate geometry are finally attracted by the first steady solution (S1) and the cone – plate data are asymptotically oriented to the second stable solution S3. We also recorded a critical value for the strain (at the shear stress of 500 Pa) which characterizes the onset of the plateau. These experiments give confidence in the interpretation of the data using the framework of the constitutive model with non-monotonic steady flow curve, Fig. 7.

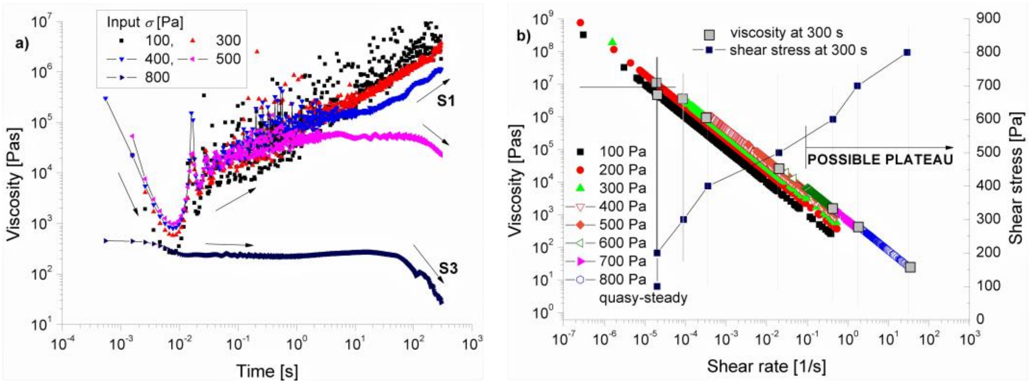

Fig. 5 a) Transient viscosity at constant shear stress. b) Data show the possible existence of a plateau in steady flow curve around 500 Pa, see also Fig. 4 (Paar-Physica MCR 301, PP geometry)

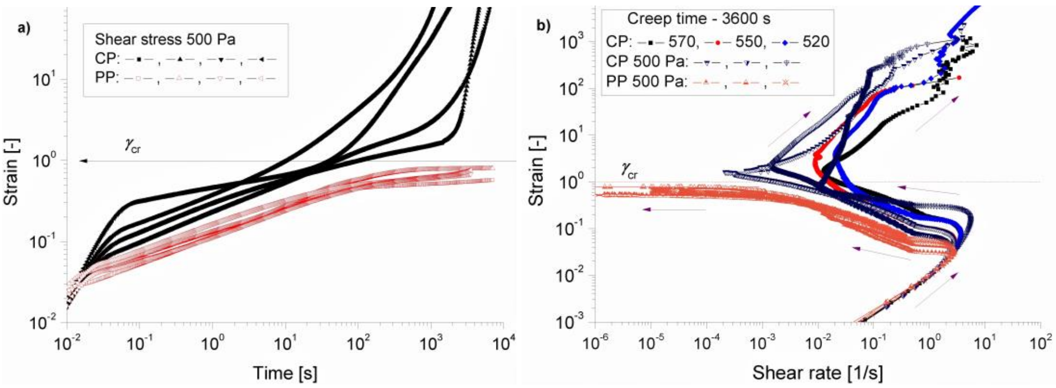

Fig. 6 The measured strain as function of time (a) and as function of the shear rate (b)

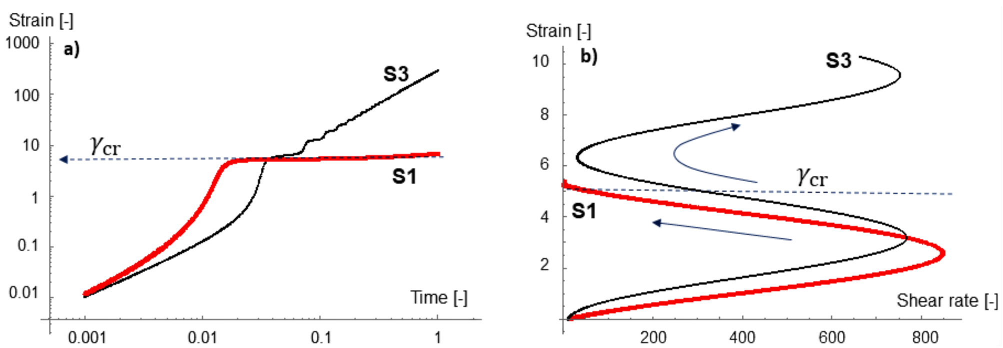

Fig.7 Numerical solutions of relation (6) at different initial conditions for the normal stresses. The dynamics of deformation is qualitatively similar with the experiments from Fig. 6.a and Fig. 6.b

It is important to remark that the value of critical strain at the onset of the plateau is constant, even if the magnitude of the shear stress at the plateau depends on the time slope of the input, as can be observed in Figs. 6 and in Fig. 8.

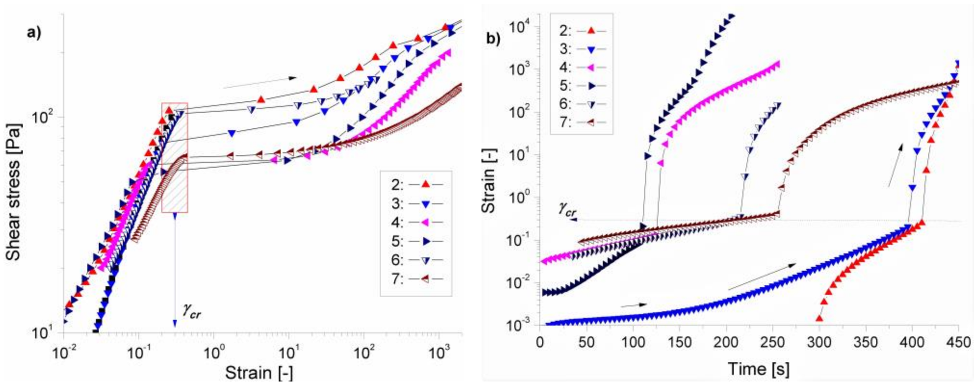

Fig. 8 The critical strain value γ_{cr} corresponding to the onset of the plateau is not dependent on the input or on the value of the shear stress σ_0 (experimental up-curves from the Fig. 3a).

The measured transitory viscosity shows that the steady viscosity function is discrete, with a jump corresponding to the plateau in the flow curve, as is shown in Fig. 5b and Fig. 9.

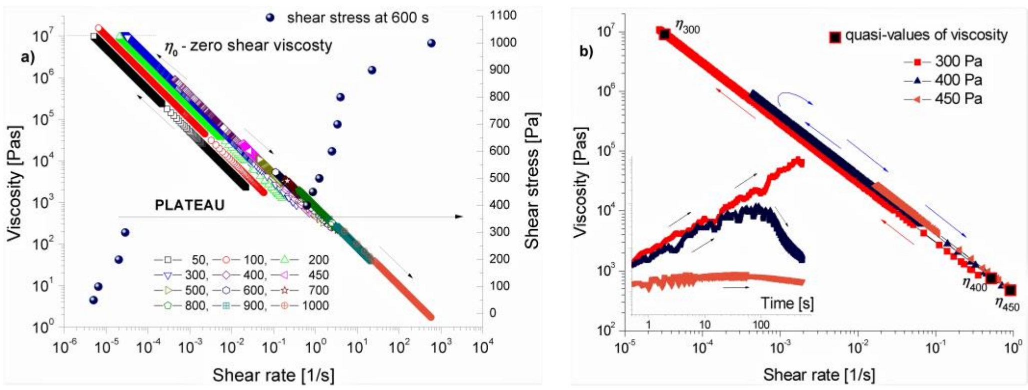

Fig. 9 a) Transient viscosity and the flow curve at experimental time of 600 s for LG2 sample at different values of the applied shear stress. b) The plateau in flow curve corresponds to the interval $\sigma_0 \in (300, 400)$ Pa, (TA Instruments AR-G2, PP geometry)

As we mentioned before, slip might affect the measurements of these types of samples. It is very difficult to perform visualization of the flow field in small gaps for glassy materials (soft solids) in the absence of a MRI/RMN technique [10, 13, 35], particle-tracking velocimetry [36] or ultra-sonic velocimetry [37].

Therefore, neither slipping nor shear banding could be directly observed in our experiments.

The results obtained with classical rheometry suggest that we are dealing with an “apparent slip” generated by the constitutive instability, phenomenon intrinsically related with the existence of shear banding in the very vicinity of the moving wall and the corresponding “kink” (i.e. discontinuity) in the velocity derivative in the gap of the rheometer, Fig. 2b (proved also by numerous experiments [10, 13, 15, 37]).

4. Final remarks and conclusions

The paper summarizes the results obtained by the authors during the investigations on the rheology of soft materials, which exhibit wall depletion phenomena. The theoretical framework, based on constitutive relations with non-monotonic steady flow curve, is correlated to the measured data obtained with commercial rotational rheometers.

The steady and transitory results are in agreement not only with the theory and our experimental data (in the asymptotic limit of the steady state), but also with numerous experimental investigations of the flow field, in particular steady or transient velocity distributions in the gap e.g. [13, 35, 38]. The measurements are consistent with only two possible interpretations: (i) the samples are slipping at the wall, or (ii) the samples are characterized by a structural instability, generated by the existence of a steady flow curve in simple shearing. We believe the second explanation is the right one, but real slip cannot be excluded as a factor which might influence the rheometry of these materials [14, 36].

This remark is based on the analysis and modeling of the transitory shear regime. The recorded dynamics of the measurements is qualitatively reproduced by a theoretical model described by a non-linear system of differential equations with multiple steady solutions. In conclusion, the constitutive relations with non-monotonic flow curve is most probably qualified for the best candidate framework to explain wall depletion of complex fluids in rheometers.

Rheometry is the main tool to characterize the rheology of complex fluids, but real information related to shear banding formation and the existence of the “plateau region” are given by the modelling and simulation of the dynamics inside the gap, corroborated with direct flow visualization (if they are available!).

Therefore, if complex fluids characterized by non-monotonic flow curve exist, then their viscosity function cannot be measured exclusively with the today’s commercial rheometers in the domain of shear rate, where the flow curve is not stable and shear banding is formed.

Acknowledgements

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