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Rheological Characterization of Systems with Plastic Flow Behavior

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Abstract: Model suspensions with plastic flow behavior were investigated rheologically. Shear stress and first normal stress difference were measured simultaneously with shear flow start-up experiments. After the shear deformation is stopped, the stress relaxation begins, residual shear stress can appear. The relative residual shear stress τ_{RR} is the ratio of the residual shear stress to the steady state shear stress. The relative residual shear stress is considered as a measure of the structure of the system. In the case of τ_{RR} = 0.98, we assume that an elastic to plastic deformation is occurred, which is the yield point. The system with 7.5% Aerosil 380 has achieved with τ_{RR} = 0.95 in the first approximation the transition to plastic flow, this is the yield point for the system, with a yield stress of 23 Pa. This is the only one known system with yield point. The systems with τ_{RR} > 0.30 and τ_{RR} < 0.95 at the first point, have no transition from elastic to plastic flow, but the structure is strongly destroyed. Analogous to the above, the shear stress at this point will be called yield stress. One requirement is that the start-up experiment is determined with a very low shear rate —0.0007 1/s or 0.00096 1/s. The associated normal stress to the yield stress is called normal yield stress. The dependence of the normal stress of the systems with Aerosil 380 on the shear stress reached, after a short increase/decrease in the values, the straight line of the normal stress/shear stress of the liquid phase M20000. A linear relationship between normal stress and shear stress was found in the systems with Bentone 27 in Araldite GY260. With a certain measured normal stress / shear stress point, you can draw a straight line with 45° in the double logarithmic plot —a way to receive the normal stress / shear stress points.

Key words: Rheology, yield point, yield stress, normal stress-shear stress.

1. Introduction

The main feature of the systems with plastic flow behavior is the existence of a yield stress τ_y. The yield stress (according to DIN standard 13342, part 10) is the stress below which the substance is an elastic solid and above which is a liquid with a plastic viscosity. According to Wikipedia, “The yield point is the point that indicates the limit of elastic behavior and the beginning of plastic behavior. Below the yield point, a material will deform elastically and will return to its original shape when the applied stress is removed”.

We know of only one system [1] with a measured yield point.

Barnes [2, 3] wrote “that all liquids show Newtonian behaviour at low enough shear rate”.

The yield stress is often determined by flow curves [4, 5], which at lower shear rates have a section with shear stress that does not change much with shear rate. The flow curve is extrapolated to the deeper shear rates. The detected shear stress is assumed to be yield stress.

The shear stress and the first normal stress difference were measured simultaneously during shear flow start-up experiments [1]. After the shear deformation is stopped, the stress relaxation begins. The residual shear stress shows the remaining structure after the shear. The relative residual shear stress is the ratio of the residual shear stress to the steady state shear stress. The relative residual shear stress is considered [1, 6, 7] as a measure of the structure of the system.

A plastic system must have a certain structure [1], which can be expressed in terms of relative residual shear stress τ_{RR}. If τ_{RR} is greater than 0.3, a plastic flow behavior is assumed [6].

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2. Experimental

Model suspensions with Aerosil 380 in the silicone oil M20000 and Bentone 27 in Araldite GY 260 were prepared in a 1.5 L reactor with anchor stirrer (100 rpm) at 40 °C in vacuum.

The rheological measurements were carried out with a WRG (Weissenberg Rheogoniometer), Model R18, Sangamo Ltd., in an air-conditioned room at 25 ± 0.2 °C. A cone-plate arrangement was used with 5 cm diameter. All measurements were performed with a strip bar 8. The Rheogoniometer was modified in accordance with Meissner [8] for measurement of the first normal stress difference N_1. The shear stress τ and the first normal stress difference were measured simultaneously during shear flow start-up experiments with constant shear rate γ = const. (Fig. 1).

All start-up experiments were started with the lowest possible shear rate for the device—0.0007 1/s (cone-plate 4°) for the systems with Aerosil 380 in the silicone oil M20000 and 0.00096 1/s (cone-plate 6°) for the systems with Bentone 27 in Araldite GY 260.

The points of the flow and the normal stress curves represent the steady state values from the shear flow start-up experiments. Just after the steady state τ_S value is reached, the shear deformation is stopped, and the stress relaxation begins. In the stress relaxation, after cessation of the shear deformation, the strip bar (torsional spring) tries to return to the starting point. If the starting point is reached, there is no residual shear stress $\tau_{\mathbf{R}}$. But if an existing remaining structure opposes against the elastic springing back of the strip bar to the starting point—a residual shear stress remains. The residual shear stress shows the remaining structure after shear.

The relative residual shear stress $\tau_{RR} = \frac{\tau_R}{\tau_S}$ is the ratio of the residual shear stress to the steady state shear stress τ_S. The relative residual shear stress is considered [1, 6] as a measure of the structure of the system.

If the relative residual shear stress $\tau_{RR} = 1$, there was only an elastic deformation during the shear. We assume that $\tau_{RR} = 0.98$ is the first indication of the transition from elastic to plastic deformation or can be regarded as a yield point.

The yield point and the yield stress have to be measured with the lowest possible shear rate in start-up experiments—0.0007 1/s (cone-plate 4°) for the systems with Aerosil 380 in M20000 and 0.00096 1/s (cone-plate 6°) for the systems with Bentone 27 in GY260.

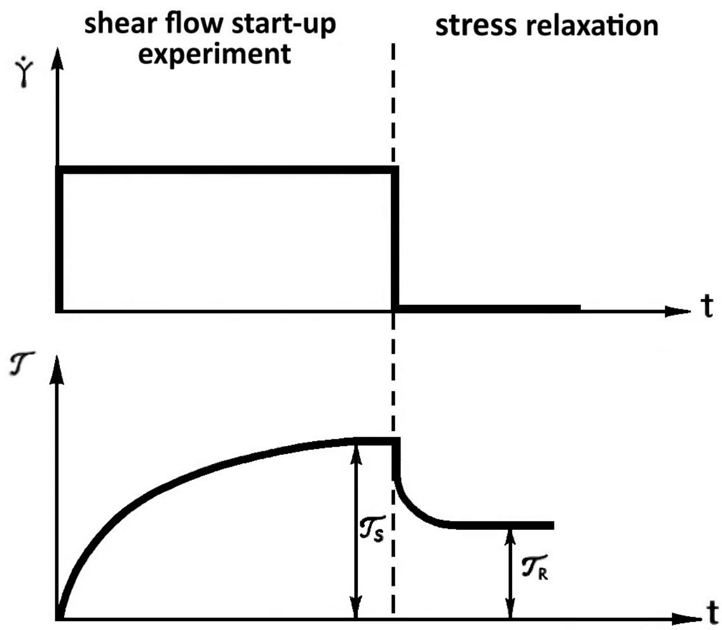

Fig. 1 Shear flow start-up experiment—stress relaxation with residual shear stress τ_R.

3. Results and Discussion

3.1 Systems with Aerosil 380 in the Silicone Oil M20000

Systems were measured with 3 wt%, 4 wt% and 7.5 wt% Aerosil 380 in the silicone oil M20000. The flow curve of the system with 7.5 wt% Aerosil 380 (Fig. 2) shows a range where the shear stress hardly changes with the shear rate. Based on the shape of the flow curve, one could maintain that this system has a yield stress and yield stress region of 1,000 Pa.

According to the flow curve, the systems with 3 wt% and 4 wt% do not have a distinct area where the shear stress does not change with the shear rate (Fig. 2), or one would say that there is no yield stress.

Fig. 3 shows the dependence of relative residual shear stress on shear stress. The 3 wt% and 4 wt% Aerosil 380 systems have a τ_{RR} of 0.78 and 0.80, respectively.

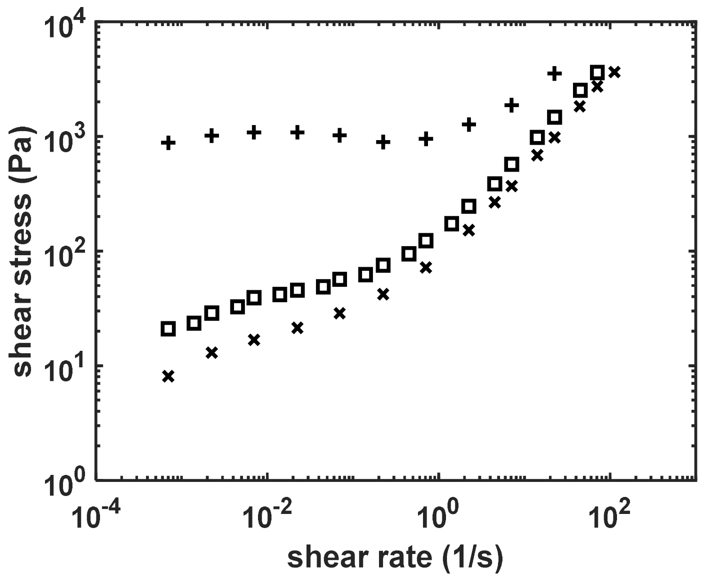

Fig. 2 Flow curves of the systems with 3 wt% (×), 4 wt% (□) and 7.5 wt% (+) Aerosil 380 in the silicone oil M20000 (WRG, cone-plate, 4°, 25 ± 0.2 °C).

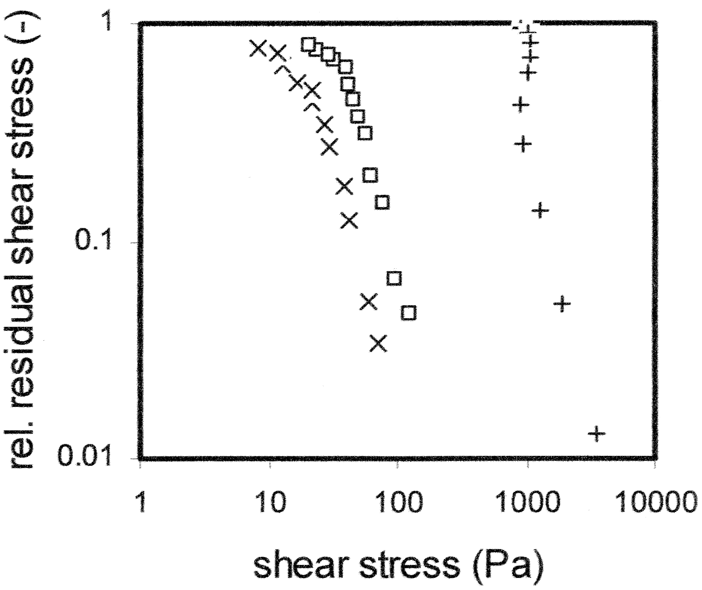

Fig. 3 Dependence of the relative residual shear stress on the shear stress of the systems with 3 wt% (×), 4 wt% (□) and 7.5 wt% (+) Aerosil 380 in the silicone oil M20000 (WRG, cone-plate, 4°, 25 ± 0.2 °C).

These two systems show a τ_{RR} greater than 0.30 and can therefore be regarded as systems with plastic flow behavior according to the definition [6]. In the first shear flow start-up experiment with a shear rate of 0.0007 1/s, there is no transition from elastic to plastic flow for the systems with 3 wt% and 4 wt% Aerosil 380, but the structure is strongly destroyed—τ_{RR} decreases by 0.22 and 0.20 respectively. As an analogy, the shear stress at this point will be called yield stress. The yield stress is therefore 8.1 Pa for the 3 wt% system and 21 Pa for the 4 wt% Aerosil 380 system.

The system with 7.5 wt% Aerosil 380 shows a relative residual shear stress of 0.95 in the first start-up experiment. With the shear deformation of 0.0007 1/s, only 5% of the structure is destroyed. At this point, in the first approximation, the transition from elastic to plastic deformation takes place, or this is the yield point for this system, which seems to be the first and only system [1, 6] with measured yield point and yield stress. The yield stress at this point is 879 Pa.

The dependence of the first normal stress on the shear stress of the liquid phase M20000 is shown in Fig. 4 (with Δ). It is a straight line with a slope $m = 2$:

$$N_1 = K\tau^m \qquad m = 2 \qquad K = 0.0000972$$

The dependence of the first normal stress difference on shear stress of the systems with Aerosil 380, first increases slightly, then the values decrease slightly and land on the straight line of the normal stress-shear stress of the liquid phase.

Analogous to yield stress, one can also define a normal yield stress—the normal stress that corresponds to yield stress. The normal yield stress corresponds to 139 Pa for the system with 7.5 wt% Aerosil 380. For the systems with 3 wt% the normal force appears at 4.5 1/s and for the system with 4 wt% Aerosil—at 1.42 1/s and can therefore not be considered as normal yield stress.

3.2 Systems with Bentone 27 in Araldite GY 260

According to the shape of the flow curves, there is no area, where the shear stress changes insignificantly with the shear rate (Fig. 5). Consequently, the systems with Bentone 27 should not have yield stress.

However, one would have to start from the strength of the structure [1, 6]. The change in relative residual shear stress with shear stress is shown in Fig. 6. The system with 15 wt% Bentone 27 has a relative residual shear stress of 0.29 at 0.00096 1/s, which is at the limit of the definition [6]. It is nevertheless considered to be a system with plastic flow behavior, with a shear stress or yield stress of 13 Pa at 0.00096 1/s.

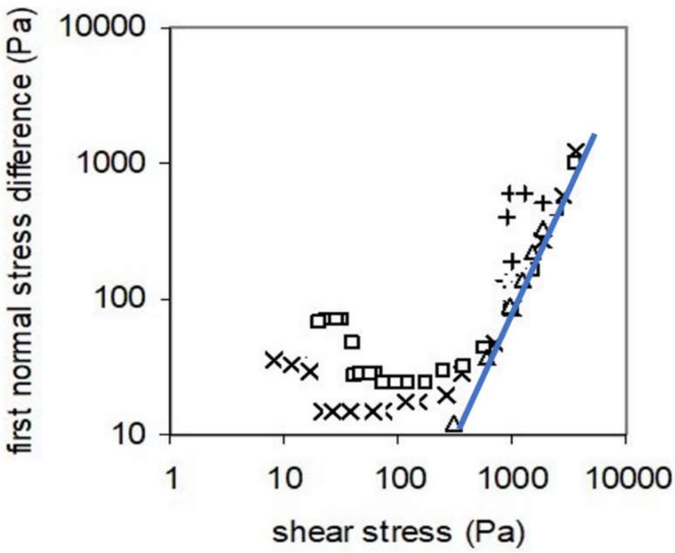

Fig. 4 Dependence of the first normal stress on the shear stress of the systems with 3 wt% (×), 4 wt% (□) and 7.5 wt% (+) Aerosil 380 in the silicone oil M20000 (WRG, cone-plate, 4°, 25 ± 0.2 °C).

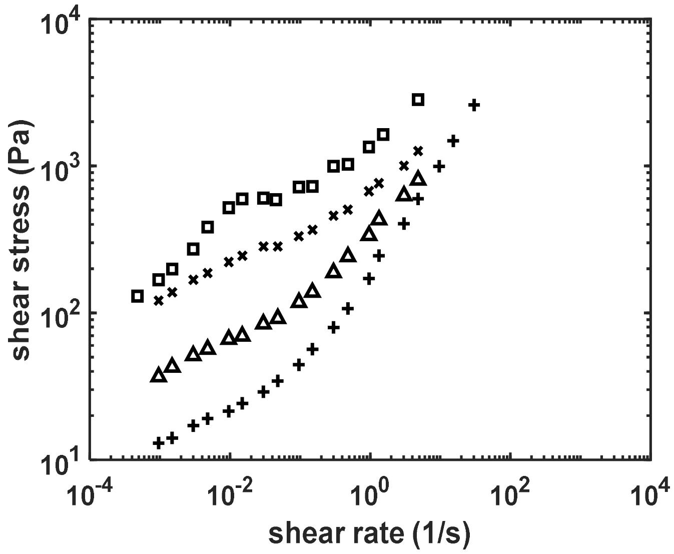

Fig. 5 Flow curves of the systems with 15 wt% (+), 17.5 wt% (Δ), 20 wt% (x) and 25 wt% (□) Bentone 27 in Araldite GY 260 (WRG, cone-plate, 6°, 25 ± 0.2 °C).

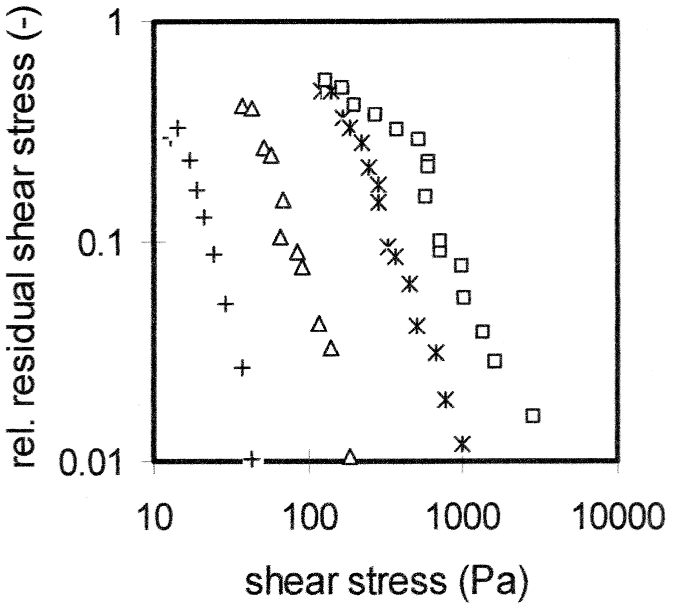

Fig. 6 Dependence of the relative residual shear stress on the shear stress of the systems with 15 wt% (+), 17.5 wt% (Δ), 20 wt% (x) and 25 wt% (□) Bentone 27 in Araldite GY 260 (WRG, cone-plate, 6°, 25 ± 0.2 °C).

The relative residual shear stress of the systems with 17.5 wt% is 0.42, with 20 wt%—0.48 and with 25 wt%—0.54. These three systems apparently have [6] a plastic flow behavior with yield stress at 0.00096 1/s corresponding to 36.7 Pa, 121 Pa and 130 Pa.

The corresponding normal stress or normal yield stress has the following values—95.6 Pa for the 20 wt% system and 107 Pa for the 25 wt% Bentone 27 system. The normal stress of the system with 15 wt% with 26.5 Pa appears at the shear rate of 9.6 1/s and in the system with 17.5 wt% with 2.4 Pa—at 0.048 1/s. They cannot be considered as normal yield stress, as they appear at higher shear rates. It is evident that the stronger a structure is built, the sooner the first normal stress appears.

Fig. 7 shows the relationship between first normal stress difference and shear stress.

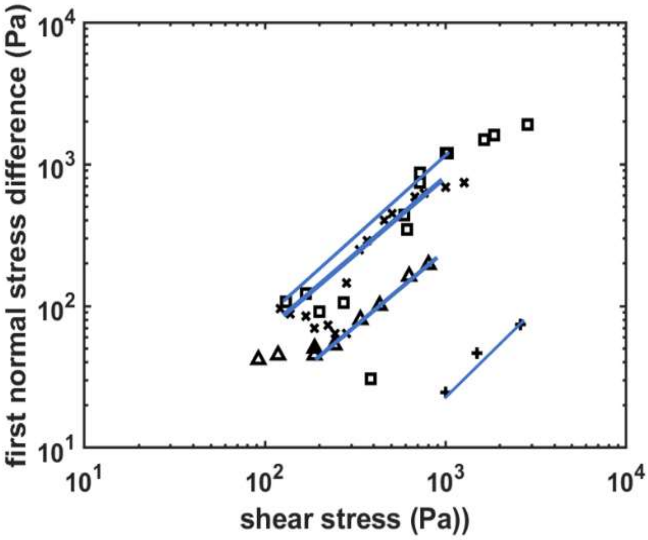

Fig. 7 Dependence of the first normal stress difference on the shear stress of the systems with 15 wt% (+), 17.5 wt% (Δ), 20 wt% (x) and 25 wt% (□) Bentone 27 in Araldite GY 260 (WRG, cone-plate, 6°, 25 ± 0.2 °C).

The relationship between normal stress and shear stress forms straight lines with 45°. The points of the systems with 20 wt% and 25 wt%, which move out of line (see Figs. 9 and 10 in Ref. [1]) are related to the rearrangement of the structure.

There is a linear relationship between normal stress and shear stress. With one measured normal stress/shear stress point, you can draw a straight line with 45° in the double logarithmic plot. This is the way to determine the normal stress/shear stress points.

4. Conclusion

All start-up experiments were started with the lowest possible shear rate for the device—0.0007 1/s (cone-plate 4°) for the systems with Aerosil 380 in the silicone oil M20000 and 0.00096 1/s (cone-plate 6°) for the systems with Bentone 27 in Araldite GY 260.

Just after the steady state value is reached, the shear deformation is stopped, and the stress relaxation begins, and residual shear stress remains.

The relative residual shear stress τ_{RR} is the ratio of the residual shear stress to the steady state shear stress. The relative residual shear stress is considered [1, 6] as a measure of the structure of the system. For a system with plastic flow behavior a relative residual shear stress τ_{RR} more than 0.30 is expected.

In the case of τ_{RR} = 0.98, we assume that there occurred an elastic to plastic deformation, which is the yield point.

The system with 7.5% Aerosil 380 has achieved with τ_{RR} = 0.95 in first approximation the transition to plastic flows or this is the yield point for the system, with a yield stress of 23 Pa. This is the only one known system with yield point.

In the systems with $\tau_{RR} > 0.30$ and $\tau_{RR} < 0.95$ at the first point, there is no transition from elastic to plastic flow, but the structure is strongly destroyed. As an analogy, the shear stress at this point will be called yield stress. One requirement is that the start-up experiment is determined with a very low shear rate—0.0007 1/s or 0.00096 1/s.

The associated normal stress to the yield stress is called normal yield stress. The dependence of the normal stress of the systems with Aerosil 380 on the shear stress reached, after a short increase/decrease in the values, the straight line of the normal stress/shear stress of the liquid phase M20000.

A linear relationship between normal stress and shear stress was found in the systems with Bentone 27 in Araldite GY260. With a certain measured normal stress/shear stress point, you can draw a straight line with 45° in the double logarithmic plot—a way to receive the normal stress/shear stress points.

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