DOI: 10.24352/UB.OVGU-2019-004 TECHNISCHE MECHANIK, 39, 1, (2019), 30 - 38
submitted: November 1, 2018

Shear Layer Generation in Yield Behavior of Gels

Y. Sato, I. Homma, T. Takahashi

The shear layer generation at the yield point and its recovery process are observed by the twin-drive rheometer. When the stress-ramp test or the shear rate-ramp test is applied to the colloidal gels, the stresses acting on the plates show different values under certain conditions. The bottom plate is used as a driving side, and the stress is controlled. Additionally, the upper plate side is a fixed side. The stress acting on the upper plate increases with the bottom plate, but at the occurrence of the yield behavior the different curve from the bottom plate is suddenly revealed. The stress acting on the upper plate becomes low, and the difference between the two stresses increases with an increasing stress or shear rate. This means that the shear stress is not constant over the gap, and a shear layer is generated. In the case of a small stress-ramp rate, the stress on the upper plate becomes the bottom value from the bottom plate stress at the yield point, but it returns to the same curve a few seconds later. The two layers of the shear layer generated at the yield point are adhered by the structure recovery, and the shear layer then disappeared.

1 Introduction

There are many fluids which show this yield-like behavior while having flowability in our daily life. Food products such as margarine and mayonnaise, cosmetics such as skincare cream, and a type of grease are such examples of this yield-like behavior. Oils, particles, and bubbles in the products form the macro structure from the weak binding force and maintain their shape from a weak force like gravity. Furthermore, when the force exceeds a certain value, the fluid suddenly begins to flow. The concept of the yield was presented by Bingham (Bingham ES, 1916). This sudden change is similar to the yield point in the metal, soft matter can recover it up to the mechanical property before the yield by the static time, which is completely different from the yield of metal. These phenomena are closely related to the functionality of the product. For example, the handling property in the cosmetics field such as skincare cream is controlled by the yield stress before taking the sample from the container (Nikko Chemicals Co. Ltd., 1996). However, the research related to the mechanism of the yield behavior on the functionality of the soft matter is still being studied (Okamoto T et al., 2014; Nabata Y et al., 2014; Watanabe K et al., 2012; Ovarlez G et al., 2012; Garcia MC et al., 2016; Dinkgreve M et al., 2016; Ovarlez G et al., 2013; Brown JR et al., 2011; Boujel J et al., 2012).

Although a previous research paper written by Barns claimed the materials considered as “Bingham’s plastic material” did not show the true yield stress (meaning the material continues to flow under the yield stress), it was not considered to be the wall slip (Barns et al., 1985). The apparent viscosity increases up to an infinitely high value over time by using the rough plate (Coussot et al. 2006; Moller et al. 2009). The flow, including the wall slip, shear-banding, and the shear-layer is an important factor when the industrial products are used. It is investigated by using special facilities and techniques. (Pérez-González, J et. al, 2012; Coussot P et. al, 2015).

The author has reported that the shear layer at the yield-like behavior is evaluated by the twin-drive rheometer, which is equipped with a conventional measurement system in the upper and the bottom plates. Each of the plates can independently control the torque applied to the sample and measure the strain that is generated. Thus, it is possible to simultaneously measure the shear stress acting on the upper plate while applying the shear stress to the bottom plate. The stress-ramp test and the shear-rate-ramp test were carried out with various acceleration values, and the stress difference between the upper plate and the bottom plate was measured. Based on these results, we discussed the generation of the shear layer in the gels (Sato Y, 2018). In this research study, we have focused on the generation of the shear layer in yield behavior of gels using the twin-drive rheometer in clay dispersing colloidal gels.

2 Materials and Experimental Methods

2.1 Sample

The colloidal gels used in this experiment consisted of silicone oil, hectorite, and PEO. The concentration of the hectorite (800×80×1~3 nm) is 8wt%, and the PEO concentration is 2wt%. The microscope image is shown in Figure 1. The black lump (seen on the right side) of approximately 0.1 mm is an agglomerate of hectorite. The silicone oil was used as a Newtonian fluid (Shin-Etsu Chemical Co., Ltd.).

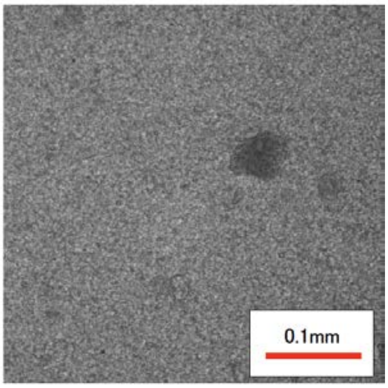

Figure 1. The observed microscope image in clay dispersing colloidal gels.

2.2 Rheometer

In this study, we used the rheometer which is MCR702 (Anton Paar Co., Ltd.) which is twin-drive stress-controlled rheometer. As we have already described above, the rheometer can measure independently each of the stress in the upper and the bottom plates. In this research, “2EC isolation transducer mode” which rotates only the bottom plate while fixing the upper plate was used as shown in Figure 2. Both of the upper and the bottom plates are the flat disk of 25 *mm* in diameter and they consist the parallel plate geometry. Both plates are made from the stainless steel. The gap between the plates is constant at 0.5 *mm* in all test. 0.5 *mm* is enough large to the particle size of the colloidal gels.

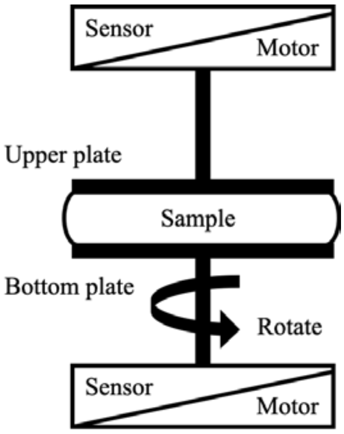

Figure 2. The outline drawing of the twin-drive rheometer.

2.3 Stress-ramp Test

The accurate value of the yield stress cannot be evaluated using the step shear test due to the destroyable discontinuous rapid acceleration that occurs at the start of the test. Therefore, the generation of the yield behavior is observed by the stress-ramp test in this study. The stress-ramp test is a rheological test, which increases the shear stress gradually from zero to a certain value in the same stress-ramp rate. The stress-ramp rate is an important parameter related to the time-dependent characteristics of the samples.

In the stress-ramp test using the twin-drive rheometer, it seems possible to detect the generation of the shear layer by measuring and comparing the stress acting on the upper and the bottom plate, in addition to the yield

behavior. In a steady shear flow field, Couette flow generally occurs. In this steady flow state, the same shear stress should be acting on the upper and the bottom plates. Even if the shear band is formed, there is no difference in shear stress acting on the upper and the bottom plate in the steady flow field. On the other hand, a stress difference between the upper and the bottom plates would be observed in the transient flow field by the stress-ramp test, because of the delay of the development of the flow field. Furthermore, there is a possibility to detect the generation of the non-uniformity in the velocity profile caused by the shear-band or the shear layer, by monitoring the occurrence of the stress difference.
In this experiment, the influence of the transient flow field on the stress of the upper and bottom plates in the stress-ramp test was investigated. By substituting the torques $\boldsymbol{M_{upper}}$ and $\boldsymbol{M_{bottom}}$ that are acting on the upper and the bottom plates during the flow into the equations (1) and (2), the stresses acting on each of the upper and bottom plates were calculated.

$$\boldsymbol{\tau_{upper}}(\boldsymbol{M_{upper}}) = \frac{4\boldsymbol{M_{upper}}}{3\pi d^3} \tag{1}$$

$$\boldsymbol{\tau_{bottom}}(\boldsymbol{M_{bottom}}) = \frac{4\boldsymbol{M_{bottom}}}{3\pi d^3} \tag{2}$$

Here, d is the radius of the plate used for the measurement.
The stress-ramp test with a stress-ramp rate of 0.5 Pa/s was carried out after the pre-shear to eliminate the influence of the loading sample in each test. The static time was set after the stress-ramp test to recovers up to the mechanical property and the sample can show the same yield behavior again by the static time. All tests confirmed the repeatability from multiple experiments.

2.3 Shear rate-ramp Test

In the stress-ramp test, the viscosity decreases suddenly due to the occurrence of the yield behavior. In the case of a stress-controlled test, the plate on the driving side increases the rotation speed with rapid acceleration in order to reach the corresponding shear rate according to a sudden viscosity drop. The stress difference measured at the time of the sudden change in the transient rotation may not only include the yield behavior, but also the effect due to the rheometer such as the inertial force of the plate. The shear rate-ramp test (which increases the rotational speed at a constant ratio) was conducted to confirm the inertial effect. The shear rate increased from zero to 100 s^{-1} for the colloidal gel at a specific ratio. The parameter to consider is the shear rate-ramp rate b, the stress acting on the shear layer, and other layers in the transient test. The pre-shear and the static time are also applied, such as the stress-ramp test. All the tests were able to confirm the repeatability from multiple experiments.

3 Result and Discussion

3.1 Stress-ramp Test

Figure 3 shows the transient behavior of the upper stress at each stress increase ratio. In addition, the value of the yield stress estimated from stress-strain curve (where the slope of the curve suddenly changes) is pointed out by an arrow in each plot in Figure 3. Here, τ_{y1} and τ_{y2} are the first and second yield stress, respectively. The bottom stress acting on the bottom plate is applied as the input condition. The upper stress acting on the upper plate, which is the fixed side for this test, is followed with the bottom stress on the lower plate. However, the upper stress cannot follow the bottom stress when the stress exceeds a certain stress level. The sudden stress difference between the upper and bottom plates is in good agreement with the yield stress estimated from stress-strain curve, which shows that the stress distribution of the sample in the plates became non-uniform due to the sudden viscosity decrease at the yield behavior. Figures 4 shows the time change of the upper and bottom stress for the stress-ramp test for the silicone oil. The upper stress shows a slightly lower stress than the bottom stress as the stress-ramp rate increases, but the stress difference is much smaller than the colloidal gel shown in Figure 3.

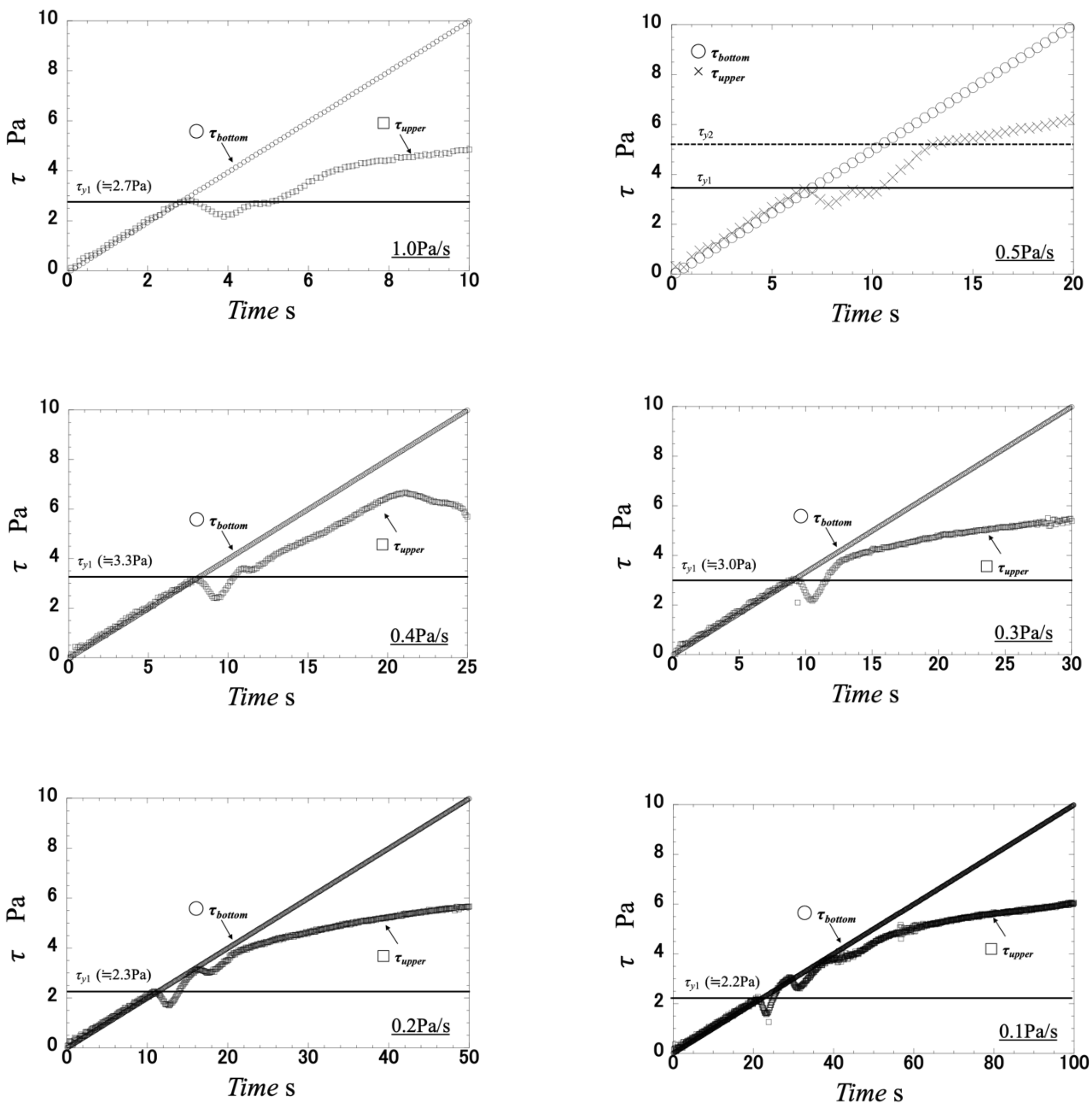

Figure 3. Bottom stress τ_{bottom} and upper stress τ_{upper} during the stress-ramp test by MCR702 on the colloidal gels.

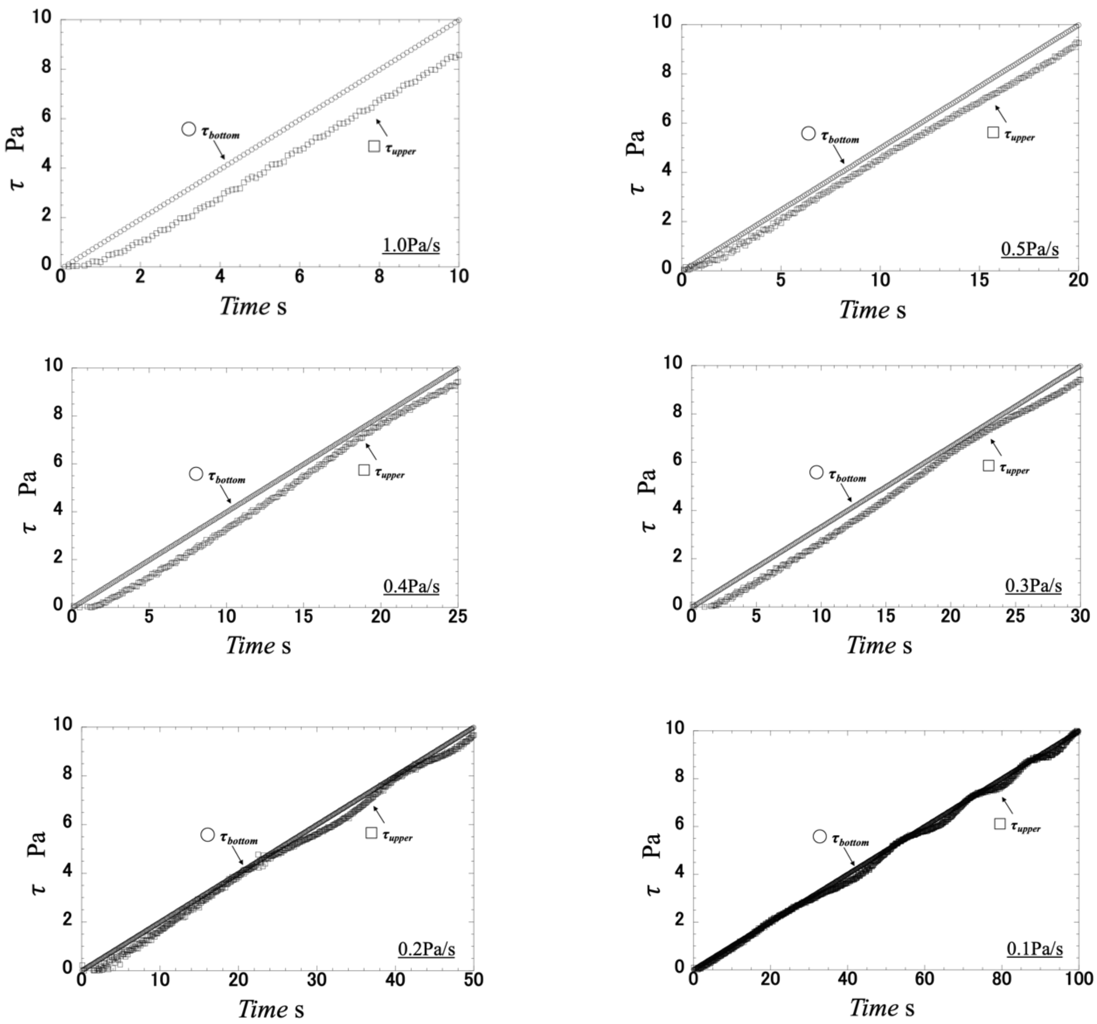

Figure 4. Bottom stress τ_{bottom} and upper stress τ_{upper} during the stress-ramp test by MCR702 on the silicone oil.

The results were observed in more detail in Figure 3. Regarding the behavior after the stress difference occurred between the plates, the stress difference generated at the first yield recovered immediately and exhibited almost the same stress value as the bottom plate once again in the case of the 0.3 or less stress-ramp rate. After that, the upper stress acting on the upper plate decreased at the second yield and increased again after showing the minimum value. In the weak bond which formed the structure, it is considered that the shear layer is formed due to crack propagation, such as the dislocation of the metal fracture after the first yield generated the fracture from a weak part. In the region where the macrostructure is destroyed, the viscosity of the sample decreases and the shear rate increases. This does not occur throughout the entire flow path, but rather just the shear layer. However, when the stress increase ratio is low, the shear layer is thin and the rotation speed is low. The shear layer disappears immediately, and the stress difference between the upper and bottom plates ends only at the moment the yield behavior occurs. After that, the structure breaks incrementally until the second yield, and the viscosity decreases slowly. On the other hand, the shear layer formed by the first yield rapidly increases the rotational speed of the plate and decreases the viscosity of the fluidization when the stress increase ratio is 1.0 *Pa/s*. Therefore, the stress difference between the upper and the bottom plates at a stress-ramp rate of 0.3 or less does not occur. In addition, the second yield was observed only at 0.5 *Pa /s*. The second yield is considered, as some macroscopic / microscopic structure is formed during shear flow and it occurs when it rapidly breaks, even though the formation of the structure related to the second yield is inhibited when the stress increase rate is high. This is also considered as an influence of the shear layer. The authors reported the following as a previous study (Homma I, 2017). The high-speed optical microscopy which can observe the structure within several hundred microns was conducted in order to analyze the velocity of the oil particle. From the start of the flow to 1.5

seconds, the particle close to the stationary plate moves at almost the same velocity as the rotational bottom plate. This means that the shear-layer is generated in the vicinity of the stationary plate, and the sample above the shear-layer moves at the velocity close to the rotational bottom plate as a bulk. Over 1.5 seconds, the velocity decreases suddenly and approaches zero. The shear-layer expands to the entire gap and forms a Couette flow. Eventually the particle velocity approaches zero near the stationary upper plate. It was verified to quantitatively evaluate the occurrence of the shear layer generation, the increase of the thickness of the shear layer, and the development of the velocity distribution over time. This result shows that a thin shear layer was formed in the vicinity of the fixed plate at the beginning of the flow, and is the reason for the above consideration.
Next, we examine the relationship between the upper stress and the strain obtained in Figure 3. The result is shown in Figure 5. All the results of the stress-ramp rate are plotted on the one curve. The upper stress maintains a nearly constant value over a wide strain range. At all stress-ramp rates, the stress increases almost in proportion to the strain in the low strain region and the region dominated by elastic behavior before the first yield have been detected, which was not seen in other results. The upper stress decreases after the first yield and reaches the same curve as the other results. After that, the upper stress starts to increase again for the second yield. Furthermore, the viscosity was calculated based on the shear stresses measured on the upper and the bottom plates, respectively. The relationship between the shear rate and viscosity was investigated. Figure 6 shows the relationship between the upper viscosity, the bottom viscosity, and the shear rate. The elastic behavior of the low strain region observed in Figure 5 is not discussed because the shear rate is too small. A significant viscosity was measured immediately after the first yield, and a much higher viscosity than the other results was obtained. As the stress rate increases, it shows low viscosity from the start of flow. Although it seems that the bottom viscosity increases as the stress increase ratio increases, when evaluated by the shear stress of the upper plate fixed side all results appear to agree very well. Although the detailed reason is still unknown, there is a possibility that the conventional rheometer cannot measure the accurate viscosity in the gels exhibiting the yield behavior.

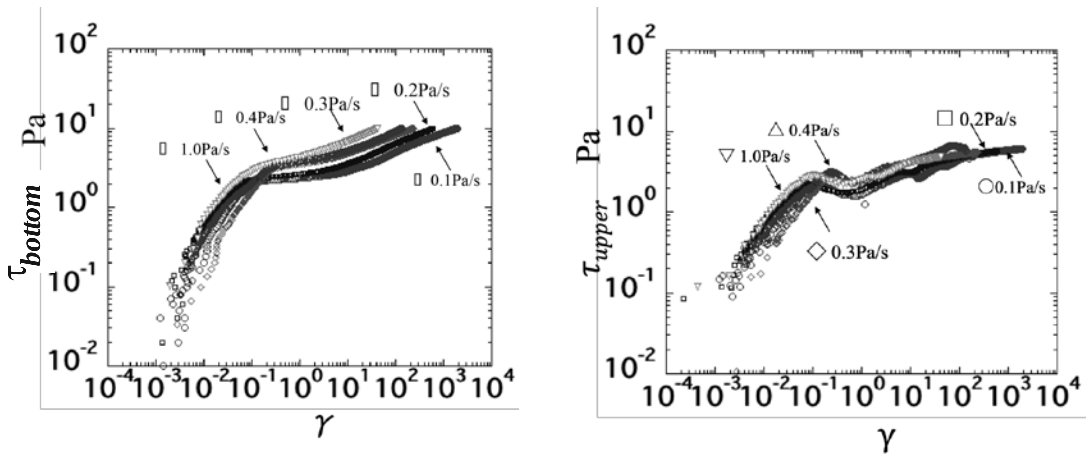

Figure 5. Bottom stress τ_{bottom} and upper stress τ_{upper} -straint by MCR702 on the colloidal gels.

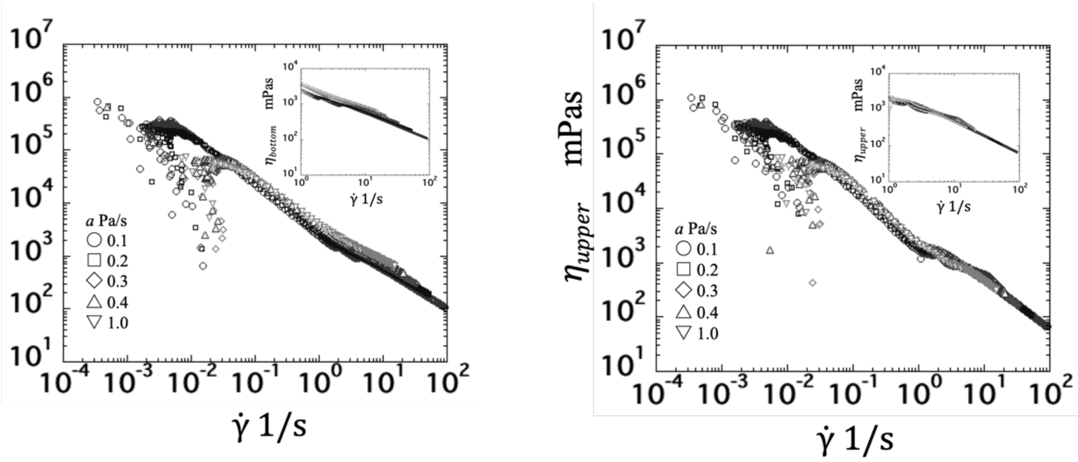

Figure 6. Bottom viscosity η_{bottom} and upper viscosity η_{upper} -shear rate curve on the colloidal.

3.2 Shear rate-ramp Test

In the stress-ramp test, the viscosity decreases and the rotational speed increases suddenly by the formation of the shear layer after the yield behavior. The shear rate-ramp test was carried out to confirm whether the stress difference is caused by the problem of the control system of the rheometer, or the property of the sample. Figure 7 shows the stress-strain curve and the time change of the stress during the test. At a shear rate-ramp rate b = 5.0 s^{-2}, the stress difference between the plates is very small after the occurrence of the yield. It is thought that the stress non-uniformity is present because the strain is greatly smaller than the stress-ramp test, so that the restructuring was influenced relatively strongly and the flow field is in a closely static state. The bottom stress exhibits the periodic fluctuation, but it is due to the control of feedback system to reach the shear rate to a certain value. From the above results, the stress difference during the stress-ramp test is not caused by the sudden change of the rotational speed, but is due to the formation of the shear layer by the yield behavior and the flow after the yield. In addition, the stress difference between the upper and the bottom plates is easier to generate in the stress-ramp test, which applies the strain with an acceleration rather than the shear rate-ramp test which applies the strain at a constant ratio. The shear layer develops due to the accelerated flow, which does not disappear by the restructuring, and results in the stress non-uniformity appearing strongly.

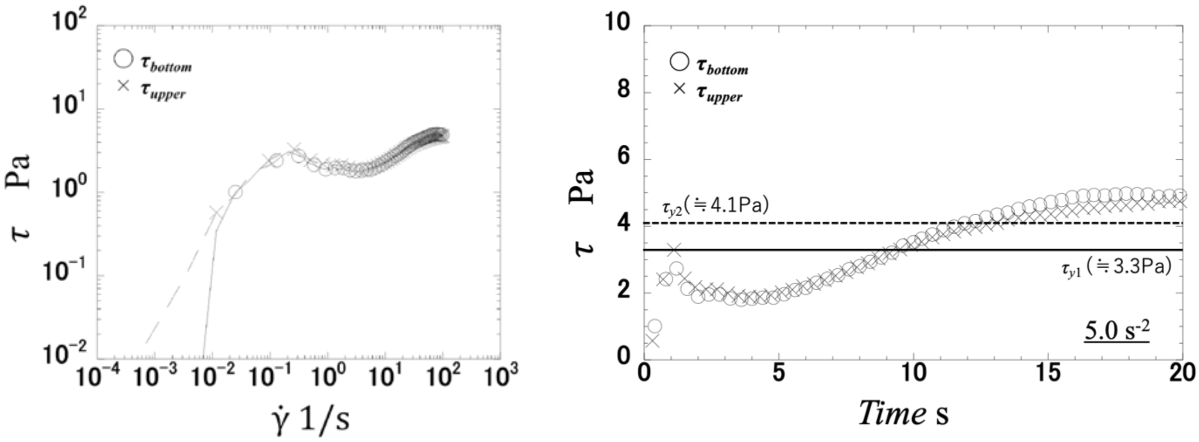

Figure 7. Bottom stress $\boldsymbol{\tau_{bottom}}$ and upper stress $\boldsymbol{\tau_{upper}}$ during the shear rate sweep test by MCR702 on the colloidal gels.

4 Conclusions

In this study, the stress-ramp test and the shear rate-ramp test was conducted using the twin-drive rheometer to the colloidal gel, which results in the yield behavior being recognized. Additionally, the stress acting on the upper and the bottom plate was measured, as well as the evaluation of the formation of the shear layer in the flow. The obtained results are summarized below.

In the colloidal gel, the bottom stress was applied as the input condition during the stress-ramp test, but the upper stress deviated from the bottom stress after the yield behavior. When the stress-ramp rate is sufficiently small, the elastic behavior before the yield was observed and the upper stress reached the same value, even though the stress difference occurred after the first yield. When the stress-ramp rate is high, the elastic behavior was not observed and the relationship between the upper stress acting on the fixed plate and the strain appeared on the same curve at any stress-ramp rate. Furthermore, the bottom viscosity evaluated by the driving plate shows the different value depending on the stress-ramp rate, but the upper viscosity was in good agreement regardless of the stress-ramp rate. It also means that the accurate viscosity could not be evaluated by a conventional rheometer, because of the influence of the shear layer in the case of such fluids that exhibit the yield behavior.

References

Barnes H., Walters K.: The yield stress myth., *Rheol. Acta*, 1985, Vol. 24, 323.

Bingham ES: An investigation of the laws of plastic flow, 1916, *Bull US.*

Brown JR, Callaghan PT: *Soft Matter*, 2011, Vol. 7, 10472.

Boujel J., Coussot P.: Measuring yield stress: A new, practical, and precise technique derived from detailed penetrometry analysis, *Rheol. Acta*, 2012, Vol. 51, 867.

Coussot P., Tocquer L., Lanos C., Ovarlez G.: Macroscopic vs. local rheology of yield stress fluids, *J. Non-Newtonian Fluid Mechanics*, 2015, Vol. 158, 85.

Dinkgreve M., Paredes J., Denn MM, Bonn D.: On different ways of measuring “the” yield stress, *J. Non-Newtonian Fluid Mechanics*, 2016, 238, 233.

Garcia MC, Alfaro MC, Munoz J.: Creep-recovery-creep tests to determine the yield stress of fluid gels containing gellan gum and Na+, *J. Biological Eng.*, 2016, Vol. 114, 257.

Homma I., Takahashi T., Noda K., Sekine T., Kitajima M., Sogabe A.: Macroscopic Structure Change and Optical Anisotropy Induced by Yield Behavior of Clay Dispersing Colloidal Gel, *Nihon Rheoloji Gakkaishi (J. Soc. Rheol. Jpn)*, 2017, Vol. 45, 277.

Moller PCF, Fall A., Bonn D.: Origin of apparent viscosity in yield stress fluids below yielding, *EPL*, 2009, Vol. 87, 38004

Nabata Y., Saito T., Tetsu M.: Rheological Properties of α -Gel Lamellar Samples with Different Morphology, *Proc. 62th Rheol. Symp*., 2014, Vol. 306, (in Japanese).

Nikko Chemicals Co. Ltd., Nippon Surfactant Indusiries Co Ltd.: *Keshouhin Handbook*, Toshiki Pigment Co., Ltd, , Chuo Printing Co., Ltd, Niigata (in Japanese). 1996.

Okamoto T.: *Netsu Sokutei*, 2010, Vol. 37 (3), 124, (in Japanese).

Ovarlez G., Bertrand F., Coussot P., Chateau X.: Shear-induced sedimentation in yield stress fluids, *J. Non-Newtonian Fluid Mechanics*, 2012, Vol.177, 19.

Ovarlez G., Cohen-Addad S., Krishan K., Goyon J., Coussot: On the existence of a simple yield stress fluid behavior, *J. Non-Newtonian Fluid Mechanics*, 2013, Vol.193, 68.

Pérez-González J., López-Durán JJ, Benjamin MM, Rodríguez-González F.: Rheo-PIV of a yield-stress fluid in a capillary with slip at the wall, *Rheol. Acta*, 2012, Vol. 51, 937.

Yasunori Sato, Homma I., Takahashi T., Noda K., Sogabe A.: Evaluation Method of Shear Layer Generation at Yield Behavior of Softmaters, *Nihon Rheoloji Gakkaishi (J. Soc. Rheol. Jpn),* 2018, Vol. 46, 67.

Watanabe K., Inoue H., Teshigawara T., Kimura T.: Gel Prepared in Sodium Methyl Stearoyl Taurate/ Behenyl Alcohol/Water System-Characterization of Structural Changes with Water Concentration, *J. Oleo Sci*, 2012, Vol. 61, (1), 29.

Address: 662 Mechanical Engineering Depertment, 1603-1 Kamitomioka, Nagaoka-city, Niigata, Japan
email: s143039@stn.nagaokaut.ac.jp