A study on influence of surface roughness of flow cell on yield in α-gels

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Abstract
We had examined the two type of the yield-like behavior in the α-gels, which are observed by the stress-ramp test in the stress-controlled rheometer. We called them the first yield and the second yield from a lower stress, respectively. The influence of the surface roughness of the flow cell in the yield-like behavior is examined in this study. The parallel plate made from the glass, the stainless steel and the glass with the sand-paper of the particles size #100 (Ra125) and #2000 (Ra20) are used in the experiments. The glass plates have the surface roughness in the order of a nanometer and the stainless plates are in the order of submicron scale. The first yield point, which is observed in the low shear stress, is affected by the surface roughness and the gap between the plates. The first yield stress measured by the glass plates is small and the first yield point is unclear compared with the results by the stainless plates. The stress difference between the upper plate and the under plate occurs clearly in the stainless plate in the different stress-ramp rate. It is considered that the uniform velocity distribution in the glass plate is formed and the shear-layer in the stainless plate gives the big influence to the flow field because it is formed stably at long intervals. On the other hand, the second yield point is influenced by the gap but not the surface roughness.

Keywords: α-gel, Yield stress, Surface roughness, Stress-ramp test, Twin-drive rheometer

1. Introduction
Some of the complex fluids show the yield like behavior that can keep their shape under a weak stress like the gravity. Such materials are often used in daily life, for example, mayonnaise, cream, jells etc. The chemical bond of their microstructure has a weak strength that can keep the macro figure under the gravity in the static condition and the strength of the bond suddenly decreases when the stress excesses a critical value. The transition from the elastic property to the plastic property at the boundary of the yield is similar to the yield of the metal. In the case of the complex fluids with the yield-like behavior, the viscoelastic property exhibits before and after the yield point, but the dominant property changes from elastic to viscoelastic. Additionally, the complex fluid has another feature, that is the recovery that can return the original mechanical property with some recovery time before the yield behavior. Since the sample changes from a high viscosity state to a low viscosity state at the boundary of the yield point, it is expected that the yield can add a product higher functionality. For example, cosmetics such as skin-care cream can be handled with ease by keeping the shape when taking out the sample from the container or before the coat, it can be widely spread with weak force due to the yield [Nikko Chemicals Co. Ltd. et.al. 1996]. However, research on the mechanism of the yielding behavior related to the functionality of soft matter is still under study. Previously, Nabata has studied a rheological property of the α-gels which in small strain region [Nabata et. al. 2014]. Watanabe has focused on a Chemical composition and structure [Watanabe et. al., 2012]. Ovarlez has observed the flow after the yield by MRI-velocimetry in a simple yield stress fluid. [Ovarlez et. al., 2012, Ovarlez et. al., 2013]. Garcia has carried out the creep-recovery-creep tests to determine the yield stress in gellan gum [Garcia et. al., 2016]. Coussot has reviewed the wide range of measurement method in a yield stress fluid [Coussot et. al., 2014]. Dinkgreve has shown the evaluation method in yield stress fluid and how the date changes depending on the measurement [Dinkgreve et. al., 2016]. Boujel has shown the method to measure accurately the yield stress from relaxation test [Boujel et. al., 2012].

As the previous study, there are some researches about the influence of the plate surface roughness to the complex fluid. Rajinder has carried out the oscillatory test in different size of the emulsion [Rajinder et. al., 2000]. The author has...
reported that the serrated plate geometry shows a solid-like behavior and the smooth cone-plate show a fluid-like behavior. Sanchez has investigated using geometries with smooth and rough surfaces in oil-in-water emulsions [Sanchez et al., 2001]. Christian has demonstrated the slip velocity of the yield stress fluid like Xanthan gum solution [Christian et al., 2012]. Pérez-González has analyzed the velocity distribution of the yield stress fluid like Carbopol gel [Pérez-González et al., 2012]. Nabata has found that the flat plate shows the step-like behavior of shear stress in low shear rate region in the creamy sample [Nabata et al., 2007]. The plate that has a rough surface by the sand-blast processing does not show the step-like behavior of shear stress. The results of both the flat plate and the plate with the rough surface matched well each other in the high shear rate. The author has considered that the shear-layer disappeared in the high shear rate made in the neighborhood of the flat plate and the sample interface.

In this study, the influence of the plate surface roughness in α-gels and the shear-layer are investigated experimentally. We used four kinds of the plate with different surface roughness: The plate made of the glass, the glass made of the stainless and the glass plate with two different sand-paper. In order to clarify the influence of the plate surface roughness on the gap of the plate, we have carried out the stress-ramp test which gradually increases the shear stress from zero to a certain value in each gap by MCR301. As Nabata has considered the shear-layer, we predicted that the plate surface roughness changes the behavior of the formed shear-layer. Thus, we have carried out the stress-ramp test by twin-drive rheometer and evaluate the occurrence of the shear-layer in each plate surface roughness. From these results, we discussed the influence of the plate surface roughness on the yield in α-gels.

2. Nomenclature

| Symbol | Unit | Description |
|--------|------|-------------|
| \( \alpha \) | [Pa/s] | stress-ramp rate |
| \( r \) | [mm] | radius of the plate |
| \( M_{\text{under}} \) | [Nm] | torque of the under plate |
| \( M_{\text{upper}} \) | [Nm] | torque of the upper plate |
| \( \gamma \) | [-] | strain |
| \( \dot{\gamma} \) | [1/s] | shear rate |
| \( \tau_{\text{under}} \) | [Pa] | shear stress of the under plate |
| \( \tau_{\text{upper}} \) | [Pa] | shear stress of the upper plate |
| \( \tau_{\text{difference}} \) | [Pa] | stress difference between the upper and the under plate |
| \( \tau_{1} \) | [Pa] | stress of the first yield |
| \( \tau_{2} \) | [Pa] | stress of the second yield |
| \( \gamma_{1} \) | [-] | critical strain of the first yield |
| \( \gamma_{2} \) | [-] | critical strain of the second yield |

3. Experimental procedure

3.1 Sample

α-gels which are widely used in the cosmetic field for the coordination of the flow property consist of the lamellar structure which is arranging aggregates by the interaction of a surface active agent and a higher alcohol. The sample used in this study is made by the behenyl alcohol (5.12wt%) as the higher alcohol, the stearoyl taurine natrum (2.38wt%) as the surface active agent, the humectant (10.5wt%), the liquid paraffin (20wt%) as the oil and the ion water. Figure 1 shows the microscope image of the sample, the circle with white is an emulsion particle and the size is about from 1 to 10μm. The sample includes a large amount of water. The surface of the flow cell is wet well. When the force applies to the sample, the shape of the sample is destroyed quickly because the sample is very soft. There is No-slip condition in this study. The influence of a stick-slip is not concerned in this study. It is considered that the result of this study is affected by the larger layer like a shear-layer rather than a stick-slip.

Fig.1 The microscope image of α-gels. The particle size is from 1 to 10μm approximately.
3.2 Plate surface roughness

The parallel plate with the stainless (JIS : SUS316L) whose surface roughness is micron order (Ra0.8) and diameter is 25mm. The parallel plate with the glass (JIS: BK-7) whose surface roughness is nanometer order (Ra0.015: assumption from the literature) and diameter is 43mm. As a pre-experiment, the same test was carried out using the glass plate conducted a hydrophobization by the chemicals and the glass plate which does not conduct any process. There is no difference between both glass plates. The results of the glass are not influenced by the wettability of the plate. The glass parallel plate with the sand-papers of the grain size #100 (Ra125) and #2000 (Ra20) are used respectively.

3.3 Gap dependence measurement by MCR301

The stress-ramp test was carried out by stress-controlled rheometer MCR301 (Anton Paar). In MCR301, the upper plate controlled the stress measures the angular displacement. The parameter is changed as the parameter as shown Table.1. One of the examples of the stress-ramp test is shown Figure 2 as the stress-strain curve. where the point at which the curve levels off is defined as the yield points. It is found that the yield occurs two times in Fig.2. These yield points are defined as the first yield and the second yield from a lower stress, respectively. The stress-ramp test (stress-ramp rate 0.5Pa/s) as a pre-shear was conducted to eliminate the influence of the loading sample in each test.

Table 1 The experimental condition in MCR301

| Measurement machine | Gap [mm]        | Stress maximum [Pa] | Stress-ramp rate $\alpha$ [Pa/s] | Temperature [°C] |
|---------------------|----------------|--------------------|---------------------------------|-----------------|
| MCR301              | 0.1, 0.2, 0.3, 0.4, 0.5, 1.0 | 100                | 0.5                             | 25              |

Fig.2 The stress-strain curve in the stress ramp test. The definition of the yield is the point where strain increases suddenly in the increase of stress. There are two yields, which called the first yield and the second yield respectively.

3.4 Measurement of stress acting the upper and the under plate by MCR702

The shear layer at the yield-like behavior is evaluated by the twin-drive rheometer, which is equipped conventional measurement system in the upper and the under plate. Each of the plates can control independently the torque applied to the sample and measure the strain generated. Thus, it is possible to measure simultaneously the shear stress acting on the upper plate while applying the shear stress to the under plate. Conventionally, although the evaluation of the shear-band and the shear-layer is needed the special technique, machine and facility [Habibi et. al., 2016, Coussot et. al., 2009, Ito et. al., 2016], we have already reported that the formation of shear layer can be evaluated using the twin-drive rheometer for complex fluid having yield behavior [Sato et. al., 2018]. We evaluated the occurrence of the shear layer in each plate surface roughness using the twin-drive rheometer. In a steady shear flow field, Couette flow generally occurs. Therefor, the same shear stress acts on the upper and the under plate. Even if the shear band is formed, there is no difference in shear stress acting on the upper and the under plate in the steady flow field. On the other hand, there is the stress difference between the upper and the under plate in the transient flow field like the stress-ramp test because of the delay in the development of the flow field. Furthermore, there is a possibility to generate the stress difference when the velocity non-uniformity like the shear-band and shear layer occurs. Thus, we carried out the stress-ramp test to evaluate the occurrence
of the shear-layer in each plate surface roughness in each stress-ramp rate as the parameter. By substituting torques $M_{upper}$ and $M_{under}$ acting on the upper and the under plate during flow into equations (1) and (2) which are based on Newtonian fluid, the stress acting on each of the upper and the under plate was calculated.

$$
\tau_{under}(M_{under}) = \frac{4M_{under}}{3\pi r^3}
$$

$$
\tau_{upper}(M_{upper}) = \frac{4M_{upper}}{3\pi r^3}
$$

where $\tau_{under}$ and $\tau_{upper}$ are the shear stress acting on the under plate and the shear stress acting on the upper plate, respectively. $r$ is the radius of the plate used for the measurement. The parallel plate has a distribution to the radial direction. Like the conventional rheometer, we defined 2/3 position to the radial direction in each plate as the nominal value.

The stress-ramp test (stress-ramp rate 0.5Pa/s) was carried out as a pre-shear to eliminate the influence of the loading sample in each test. After that, the sample recovers up to the mechanical property to show the yield behavior again by the static time. The measurements were carried out multiple times under the same conditions including this pre-shear as a preliminary experiment, although the result differs depending on the condition of a pre-shear. All result showed the same behavior below shown. As a pre-test, we have evaluated the result by 25mm stainless plate and 50mm stainless plate. There is no difference between both. To save the amount of sample, we used 25mm stainless plate. The inertia effect in the plate diameter is eliminated by the inertia moment measured in advance. Therefore, The influence of plate diameter is much less in this study.

| Measurement machine | Gap[mm] | Stress maximum [Pa] | Stress-ramp rate $a$ [Pa/s] | Temperature [°C] |
|---------------------|---------|---------------------|-----------------------------|-----------------|
| MCR702              | 0.5     | 100                 | 0.01, 0.1, 0.5, 5.0, 10     | 25              |

### 4. Results and discussion

#### 4.1 Stress-ramp test using MCR301

Figure 3 shows the stress-strain curve of the stress-ramp test in gap 0.5mm. While all plate surface roughness agrees over about 20Pa, the behavior totally differs in each surface roughness below 20Pa. The stainless plate is not plotted below 1Pa because the strain shows minus. The stainless plate shows the first yield clearly in about 5 Pa. The sand-paper plate shows the first yield clearly in about 2 Pa as well and the strain increases in a constant rate below the first yield stress. The first yield stress of the sand-paper plate is lower than the stainless plate. It is predicted that the sand-paper plate having a sufficiently large surface roughness to a particle diameter move such that particles climb over the surface roughness. It is considered that the propagation of the fracture for the first yield is easy for the sand-paper plate even small stress. The glass plate does not show clear first yield behavior and the strain increases linearly up to the occurrence of the second yield. In the glass plate with very small surface roughness to the particle size, it is considered that the friction between the plate and the sample is small due to a small surface area. It is conceivable that the local slip surface generated from the crack propagation is hardly formed and the first yield is unclear. To observe these different behaviors in detail, the stress-shear rate curve of the same data shows Fig.4. The stainless plate flows hardly with a weak force before the first yield stress, and only the stress increases like a step up to about 5 Pa. The shear rate increases suddenly once the first yield occurs. It is considered that the fracture for the first yield starts from the weakest part of the sample, the sample flows locally and the viscosity decreases suddenly. In the sand-paper plate, the behavior like the step of the stress is disappeared and the shear rate increases suddenly after the first yield like the result of the stainless plate. The behavior of the glass plate is totally different from these results. In low stress region, the glass plate shows the shear rate corresponding to the value after the first yield of the stainless plate and sand-paper plate even small stress like 0.5 Pa. It means that the sample on the glass flows even below the yield stress measured by ordinal rheometer’s plate like the stainless plate. It is found that the flow property in the low shear rate is totally different in the plate surface roughness.
4.2 Yield stress and strain calculated by stress-strain curve

Figures 5(a) and (b) show the influence of the gap of the flow plate on the first yield stress and the critical strain of the first yield point, respectively. The results in each surface roughness are compared in them. The vertical axis is the yield stress or strain. Here, the first yield stresses and the critical strain in the glass plate is zero because the glass does not show the first yield behavior except gap 0.1mm. The gap of 0.1mm is too narrow to measure accurately due to the parallelism of plates or the particle size. The too smooth plate like the glass is not influenced by the gap. Both the yield stress and the strain of the stainless plate increase with decreasing the gap, that is, the surface roughness dominates the flow between in the narrow gap. In the case of the sand-paper with the grain size #2000, the yield stress increases as same as the case of the stainless plate, the increase of strain is slower than the stainless. In the sand-paper with grain size #100, the yield stress does not almost increase although the effect of surface roughness increases. Only the strain increases with decreasing the gap. Figures 6(a) and (b) show the second yield stress and the critical strain in each condition. The second yield stress becomes almost constant except the case of the gap 0.1mm of the stainless plate and the glass plate. The critical strain of the second yield point is in the almost same curve in any surface roughness. From these results, the existence or non-existence of the occurrence of the first yield depends on the surface roughness. It is expected that the second yield does not depend on the surface roughness and is influenced by the domain size in gaps.
At high stress ramp rate, the behavior of the stress difference should be different depending on the stress rate measured by the rheometer. Therefore, it was considered that a thick shear layer was formed and the detectable stress difference was the formation of the shear layer, the particles flow not only in the flowing direction but also in the direction of the stress path gap. When the surface is rough with respect to the particle diameter, the particles may climb up the roughness of the surface with the velocity distribution like the metal fracture after the first yield generated. To observe the behavior in detail, Fig.9 shows the graph which the stress difference obtained by subtracting the upper stress from the under stress applying as input condition in all surface roughness. While there is almost no stress difference between the upper and the under plate during the test in the glass plate, the stainless plate shows that the deviation of the stress acting the upper and the under plate occurs clearly after the second yield. To observe the behavior in detail, Fig.9 shows the graph which the stress difference obtained by subtracting the upper stress from the under stress applying as input condition is the vertical axes. While the stainless plate occurs the stress difference from the start of the flow, the stress difference between the upper and the under plate almost does not occur and shows almost zero on the glass plate. It is considered that the shear layer is formed due to the crack propagation like the metal fracture after the first yield generated the fracture from a weak part in the stainless plate and the velocity distribution is almost uniform like Couette flow up to the second yield in the glass plate. When the surface is rough with respect to the particle diameter, the particles may climb up the roughness of the surface with the flow. In the formation of the shear layer, the particles flow not only in the flowing direction but also in the direction of the flow path gap. Therefore, it was considered that a thick shear layer was formed and the detectable stress difference was measured by the rheometer. In both plates, although the stress difference increases after the second yield, it is due to the sudden increase of the shear rate.

If there is a stress difference due to the delay in the development of the shear layer with respect to the stress-ramp rate, the behavior of the stress difference should be different depending on the stress-ramp rate. To confirm this assumption, we measured the stress-ramp test in different stress-ramp rate. Figure 10 shows the stress difference in each stress-ramp rate. The stress difference in both of the plate is almost zero at 0.01 Pa/s. Because the small stress-ramp rate such as 0.01 Pa/s makes the flow field closely steady state regardless of the yield behavior and the roughness of the plate. At high stress-ramp rate such as 5 or 10 Pa/s, the behavior of the stress difference changes depending on the plate surface roughness.

4.3 Stress difference occurring between the upper plate and under plate in stress-ramp

From the result of chapter 3.1, it is a possibility for generating shear-layers on the surface and a plug-band between them. The shear-layer is usually very thin with higher shear rate than the plug-band and it would be affected by the surface roughness. Additionally, the behavior of the stainless plate and the glass plate is totally different from each other. We focused on these two plates and conducted some experiments. Fig.7 shows the stress-strain curve of the stress-ramp test using the twin-drive rheometer. The stainless plate shows the second yield by continuing the flow after showing the clear first yield behavior. In the glass plate, the strain increases at the almost same constant rate up to before the second yield. The results of the stress-ramp test obtained by the twin-drive rheometer qualitatively agree with the results obtained by the MCR301 as described above (Fig.3). Figure 8 shows the time change of the stress acting the upper and the under plate to focus on the stress acting the upper plate fixed in MCR702. Here, τ\textsubscript{1} and τ\textsubscript{2} are the first yield stress and the second yield stress, respectively. The stress acting the upper plate which is rotating is applied as the input condition in all surface roughness. While there is almost no stress difference between the upper and the under plate during the test in the glass plate, the stainless plate shows that the deviation of the stress acting the upper and the under plate occurs clearly after the second yield. To observe the behavior in detail, Fig.9 shows the graph which the stress difference obtained by subtracting the upper stress from the under stress applying as input condition is the vertical axes. While the stainless plate occurs the stress difference from the start of the flow, the stress difference between the upper and the under plate almost does not occur and shows almost zero on the glass plate. It is considered that the shear layer is formed due to the crack propagation like the metal fracture after the first yield generated the fracture from a weak part in the stainless plate and the velocity distribution is almost uniform like Couette flow up to the second yield in the glass plate. When the surface is rough with respect to the particle diameter, the particles may climb up the roughness of the surface with the flow. In the formation of the shear layer, the particles flow not only in the flowing direction but also in the direction of the flow path gap. Therefore, it was considered that a thick shear layer was formed and the detectable stress difference was measured by the rheometer. In both plates, although the stress difference increases after the second yield, it is due to the sudden increase of the shear rate.
roughness. Although the stress difference before the second yield in the glass plate is almost zero in any stress-ramp rate, the stress difference increase after the first yield in the stainless plate. The stress difference is bigger as the stress-ramp rate increases in the stainless. The rough plate like the stainless might generate clear shear-layer due to climbing up the particles. In the case of a low stress-ramp rate, the flow field is close to the static state with regardless of the formation of the shear-layer. There is no stress difference in the stainless plates. In the case of a high stress-ramp rate, there is a delay of the development between the shear-layer and other layer. Due to the delay, there is a big stress difference between the stainless plates. On the other hand, the sooth plate like the glass plate might not generate clear shear-layer. In the case of a low stress-ramp rate, the flow field is close to static state like Couette flow. There is no stress difference in the glass plate. In the case of a high stress ramp rate, the state of the flow filed is almost same to the case of the low stress-ramp rate. There is no stress difference between the glass plates. Additionally, it is considered that the stable shear-layer in the glass plate is formed and the shear-layer in the stainless plate give the big influence to the flow field because it is formed stably at long intervals.

Fig. 7 The stress-strain curve in MCR702. (a) stainless plate (b) glass plate. The stress acting the under plate and the upper plate shows yellow circle and blue circle, respectively. Here, the result of each plate surface roughness is almost same to the result of MCR301 in gap0.5mm. The stainless plate shows the first yield behavior clearly. The glass plate shows only the second yield behavior.

Fig. 8 The time change of the stress (a) stainless plate (b) glass plate. The under stress decreases with the second yield in the stainless plate. On the other hand, the under stress is almost same to the upper stress in the glass plate.
Fig. 9 The stress difference during the stress-ramp test at 0.5 Pa/s. The stress difference occurs after the first yield in the stainless plate. Furthermore, the stress difference increases greatly with the second yield. The stress difference in the glass plate is almost same before the second yield. After the second yield, the stress difference increases slightly in the glass plate.

Fig. 10 The stress difference in each stress-ramp rate (a) 0.01 Pa/s (b) 0.1 Pa/s (c) 5 Pa/s (d) 10 Pa/s. In the case of small stress-ramp rate, the stress difference in both of the plate does not occur during the test. In the case of big stress-ramp rate, the stress difference in the stainless plate increase with the first yield, while the stress difference in the glass plate is almost same.
5. Conclusion

We investigated the influence of the plate surface roughness on the yield in α-gels and carried out the stress-ramp test using MCR301 in a different gap and MCR702 to measure the stress acting the upper and the under plate. The result obtained from the experiments is summarized below.

The first yield which occurs in low shear-rate is strongly influenced by the plate surface roughness. The stainless plate and the sand-paper plate show the clear first yield. The first yield in the glass plate is unclear. In the stainless plate, the stress difference change depending on the stress-ramp rate. On the other hand, the stress difference in the glass plate is almost zero regardless of the stress-ramp rate. It is considered that the stable shear-layer in the glass plate is formed and the shear-layer in the stainless plate give the big influence to the flow field because it is formed stably at long intervals. The second yield which occurs in high shear rate is influenced by the gap regardless of the plate surface roughness. It is assumed that the occurrence of the second yield depends on the domain size of the sample.

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