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Effect of double network hydrogel film thickness on the run-in tribological properties of a friction system using DN gel

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Abstract

This study aims to investigate the potential of double network hydrogel (DN gel) in a low friction mechanical system. In this study, the effect of the thickness of DN gel films on the friction property are clarified. An indentation test, contact pressure measurement, and series of friction tests in water using three different thickness (1.5, 3, 6 mm) of DN gel film and a SiC disc were carried out.

Friction of DN gel film sliding against SiC in water can be classified into two types as “Friction region I: friction remains substantially unchanged during the test” and “Friction region II: friction decreases due to run-in”. Friction region transition can be related to the Hersey number region, which consists of viscosity of lubricant, sliding speed and apparent contact pressure. Hersey number larger than 1.5×10^{-10} is a necessary condition for friction region II where different lubrication regimes can be found.

1.5 and 6 mm thick DN gel films have exhibit super low friction ($\mu < 0.01$) under a wide range of friction conditions as friction region II, and especially 1.5 mm thick DN gel film, super low friction is achieved by twice run-in phenomena.

Keywords: soft matter, DN gel film, super low friction, run-in

I. Introduction

Hydrogels are generally defined as water-swollen cross-linked polymeric networks produced by the reaction of monomers [1]. Most types of hydrogels are made of highly hydrophilic polymer networks. They have polymeric chains that can be connected physically and/or chemically. Hydrogels can be classified in terms of their structure, composition, synthesis method, type (natural or synthetic), properties (pH or thermo-responsive), and so on. Hydrogels have been used in contact lenses [2], drug delivery systems [3], and biomimetic models for surgery training [4].

Since hydrogels usually exhibit poor mechanical strength, they have not been actively employed in mechanical applications. In the 1990s, a study reported that the mechanical strength of gel materials can be improved by the creation of an interacted network increasing the mechanical resistance of the hydrogel [5]. Among these smart gel materials, the double network hydrogels (DN gels), developed in 2003 [6], especially present a higher mechanical resistance, stress [7–10] compared to conventional hydrogels. The elastic component is dominant in their shear stress [11,12], and they can reach an extremely low friction coefficient [13].

Friction properties of tribological system using soft matter can be affected by contact pressure, sliding speed, temperature [14], or viscoelastic properties [15] and so on. The roughness and wettability of the counter surface are also critical factors affecting friction property [16]. Parameters changing friction properties on simple hydrogels have been investigated. For instance, in this study [17], repulsion adsorption model was proposed and a negative load dependence of friction coefficient with load was found. Another study [18] focused on the influence of the roughness of the hydrogel and concluded that the dependence of friction with roughness decrease with the normal pressure increase. Also, the dependence of friction with elasticity of hydrogel have been studies in conventional way [19] and by using resonance shear measurements [20]. Finally, mainly studied with a light load applied and in a flat configuration, friction of the DN gel need additional investigation.

41 The wear on polymer friction is based on the tensile and a compressive part in the contact, following the direction of friction. Indeed, as
42 explain in this paper, soft matter presents a typical wear. That underline the existence of a tensile or extension stress at the front of the friction
43 force and a compressive stress at the end of the friction direction [21].

44 Moreover, from the viewpoint of practical machine design, the thickness of soft materials should be kept at a minimum, as thick soft
45 materials can break the mechanical alignment due to their easy deformation. Therefore, in this study, we investigated the potential of DN gel as a
46 covering film for a tribo-system application. In addition, to understand how to find a use for DN gel as a cover film, we aim to clarify the role of
47 the DN gel film thickness on the friction property and the evolution of friction coefficient over distance.

49 **2. Material and experimental procedure**

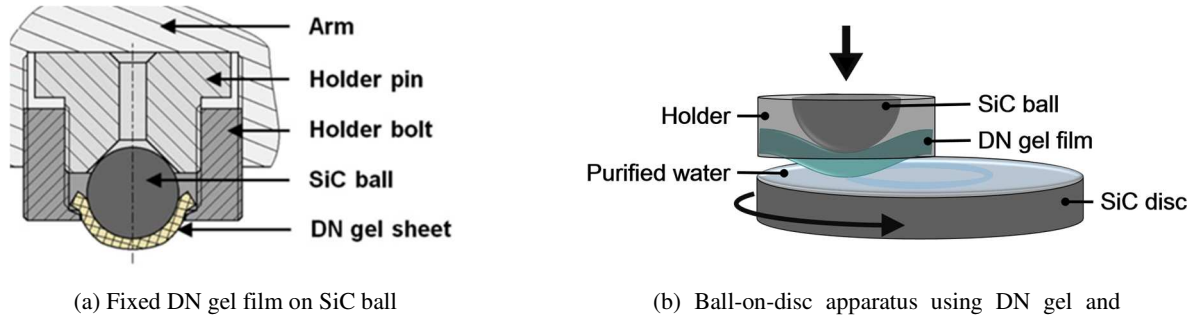
50 **2.1. Synthesis of DN gel film**

51 The synthesis of the DN gel film consisted of the following three steps.

- 52 1. Polymerisation of the first network (18 hours)
- 53 2. Absorption of the second network solution into the first network (48 hours)
- 54 3. Polymerisation of the second network (18 hours)

55 The first network was made from 2-acrylamido-2-methyl-1-propanesulfonic acid (AMPS) (1 mol %), with N, N'-methylene bis-
56 acrylamide (MBAA) as the cross-linker (0.04 mol %), α -ketoglutaric acid (0.001 mol %) as the initiator, and water (43 mol %) as the solvent.
57 The mixed solution was prepared in a mold and irradiated with ultraviolet (UV) light for 18 hours. The thickness of the DN gel film was
58 controlled by changing the thickness of the frame for the first network gel. The second network was a polymer made from N,N-dimethyl
59 acrylamide (DMAAm) (1 mol %) with the same cross-linker (MBAA, 0.0002 mol %), initiator (α -ketoglutaric acid, 0.001 mol %), and solvent
60 (water) (22 mol %). The first network gel was immersed into the second network solution for 48 hours. Finally, absorbed second network
61 solution in first network gel was fixed for 18 h under UV light, thus DN gel film was synthesized. All procedures were performed at 30 °C in a
62 thermostatic chamber.

63 In this study, DN gel films with three different thickness, of 1.5 mm, 3 mm, and 6 mm were produced to investigate the effect of film
64 thickness on the friction property.



(a) Fixed DN gel film on SiC ball

(b) Ball-on-disc apparatus using DN gel and

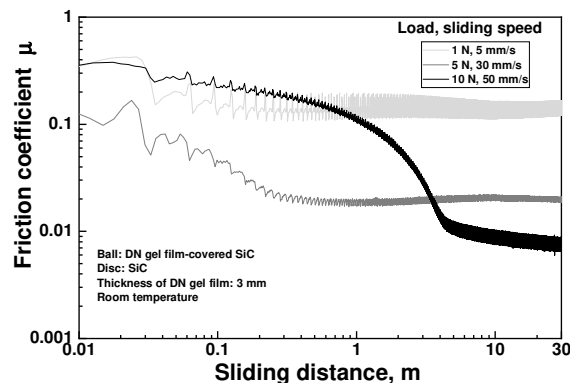
Fig. 1 Schematic of (a) fixed DN gel film on SiC ball, (b) schematic of ball-on-disc rotating tribometer

We used a ball-on-disc type tribometer as shown in Fig. 1. From initial square of DN gel film, specimens of 10 mm diameter were cut out by laser cutter. Then, the DN gel sample was placed in the sample holder and folded by the silicon carbide (SiC) ball. Finally the specimen holder pinches the DN gel film on its edge by screwing the holder bolt on the holder pin, to obtain an 8 mm diameter silicon carbide ball covered by DN gel film. Polished SiC was used as a disc specimen to minimise the effect of the asperities. The average arithmetic measured height of SiC disk is 11 nm. 1 mL of purified water dropped on the disc keeps the DN gel hydrated and serves as a lubricant. Hereafter, the friction system in this research is referred as DN gel/SiC in water.

In the friction tests, the normal load applied was: 0.5, 1, 5, and 10 N, and the sliding speed varied from 5 to 100 mm/s. The total sliding distance was fixed at 30 m. The resulting friction force is recorded by a precise sensor, the model LMA-A-5N from Kyowa Electronic Instruments Co., Ltd.

After friction test, it is necessary to investigate the friction interface by the observation of the wear after rubbing with Scanning Electron Microscopy (SEM). Since the DN gel is mainly made of water, DN gel can be shrunk in vacuum condition in SEM chamber. To avoid this, DN gel sample was first dried then immersed in T-butanol solution [22,23]. Thus detail of wear surface of DN gel after friction test can be observed by SEM.

3. Experimental results

Fig. 2 Representative friction properties of DN gel ($t=3\text{mm}$) / SiC in water under three different friction condition

Representative friction properties of DN gel ($t=3\text{mm}$) / SiC in water under three different friction condition (1 N - 5 mm/s, 5 N - 30 mm/s, 10 N - 50 mm/s) are shown in Fig. 2. Under 1 N - 5 mm/s conditions, there is no significant change in friction regardless of the sliding distance and a value of 0.2 is reached in the steady state. By applying a 5 N - 30 mm/s conditions, the friction coefficient suddenly decreases and reaches a low value of 0.02. Under 10 N - 50 mm/s conditions, friction progressively decreases over the sliding distance and reaches super low

90 value of friction coefficient, below 0.01. This clearly shows that the high potential of friction system using DN gel to achieve low friction is due
91 to a run-in period.

92 It is also found under all conditions that a stable friction can be reach within 30 m distance. Therefore, average friction coefficient from 20 m to
93 30 m is used for evaluation.

94

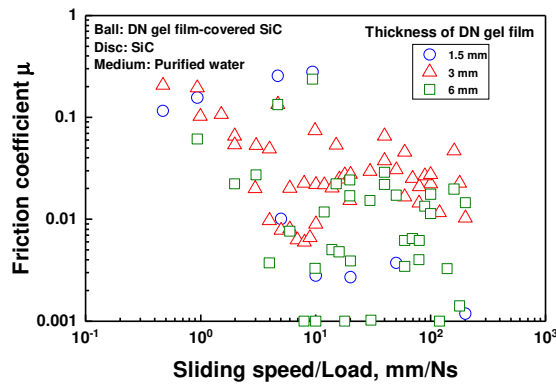


Fig. 3 Tendency of friction coefficient as a function of sliding speed/ load

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96 Friction data obtained with friction pair of SiC and DN gel with three different film thickness under all experimental condition are summarized
97 in Fig. 3 as a function of sliding speed/load.

98 Friction coefficient depends on the load and sliding speed regardless of the DN gel film thickness and is distributed over three orders of
99 magnitude. Moreover, an extremely low value (< 0.01) is reached for a relatively large sliding speed/normal load. This results clearly shows the
100 potential of tribological system using DN gel film to achieve super low friction. However, such extremely low friction is obtained only when the
101 DN gel film thickness is 1.5 and 6 mm, and not when it is 3 mm. In order to properly use DN gel for any tribological application, a clarification
102 of the effect of DN gel film thickness to realize such super low friction is necessary.

103

4. Discussion

4.1. Influence of DN gel film thickness on apparent contact stiffness and contact pressure

We aim to understand the effect of DN gel film thickness on the decreasing friction previously mentioned. Thus, investigation about contact stiffness and contact pressure depending on the film varied thickness is important. Therefore, we introduced following two methods to evaluate both contact stiffness and contact area at the interface of SiC disc and DN gel film covered SiC ball, which is similar to friction test configuration.

(a) Indentation method for evaluation of contact stiffness

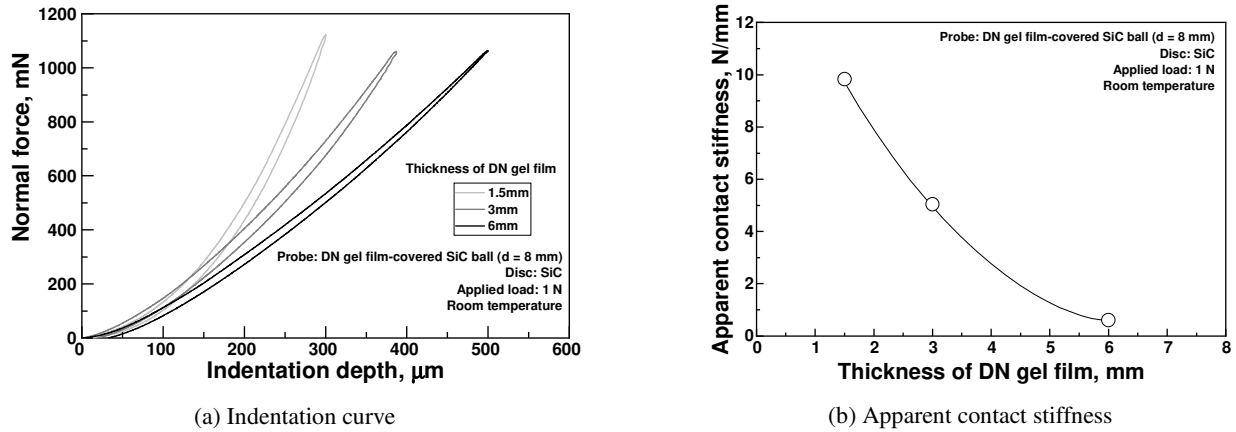


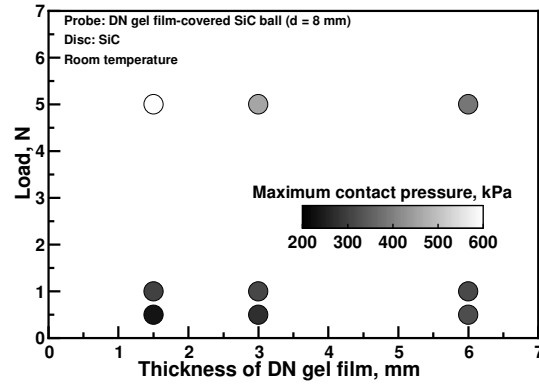
Fig. 4 (a) Indentation curve and (b) apparent contact stiffness at the contact of SiC disc and DN gel film ($t=1.5, 3, 6$ mm) covered SiC ball ($d=8$ mm) obtained under normal load of 1 N

The indentation test apparatus has been used for biological measurements [24]. It can apply or remove load and record the penetration depth, δ , on the surface to be tested with a probe. As the probe, we used our tribo-system probe consisting of a SiC ball covered with the DN gel film. The normal force, F_N , was recorded using a force sensor. From the slope of the indentation curve, we calculated the apparent contact stiffness of the system.

The indentation curve and apparent contact stiffness of the system depending on the DN gel film thickness are shown in Fig. 4 (a) and (b), respectively. The apparent contact stiffness, K_Z , was calculated from the indentation curve using the equation of $K_Z = \frac{\partial F_N}{\partial \delta}$.

When the applied load is 1 N, the apparent contact stiffness of the DN gel film with a thickness of 1.5 mm, 3 mm, and 6 mm was 9.83, 5.04, and 0.61 N/mm, respectively. As shown in Fig. 4, the thinnest DN gel film showed a much higher apparent contact stiffness compared to the other DN gel films. As predicted, the apparent contact stiffness decreases while the thickness of the DN gel increases.

123 (b) Measurement of contact area for evaluation of contact pressure
 124



125 Fig. 5 Maximum contact pressure at DN gel/SiC determined by both load and thickness of DN gel film

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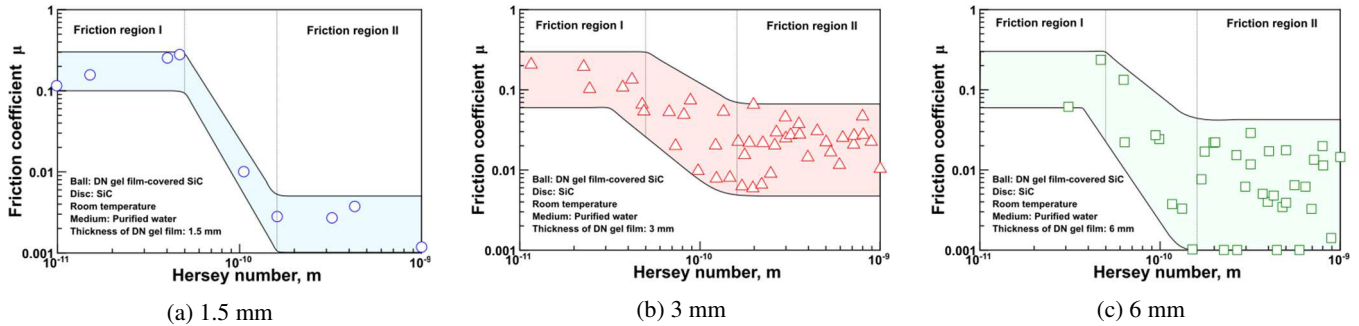
126 To measure the contact pressure of the system, we placed a mesh sensor (I-Scan, NITTA Corporation) on the SiC disc. Contact area and pressure
 127 were recorded when 1 N, 5 N and 10 N were applied for each DN gel thickness.

128 In Fig. 5, apparent maximum contact pressure of DN gel on SiC is plotted depending on both load and thickness of DN gel film. A thick
 129 DN gel film tends to reduce contact pressure by relaxation effect, especially with a load of 5 N. It is clear from these results that thickness of DN
 130 gel film is a driving parameter to manage the contact pressure. Itself being an essential feature for tribological design such as surface damage,
 131 fracture, lubrication region and so forth.

132

133 4.2. Influence of DN gel film thickness on friction property of DN gel / SiC in water

134



135 Fig. 6 Trend of friction coefficient of DN gel/SiC in water as a function of Hersey number when thickness of DN gel film thickness is
 136 (a) 1.5 mm, (b) 3 mm and (c) 6 mm

135

136 Figure 6 shows trend of friction coefficient of DN gel/SiC in water with three different DN gel film thickness as a function of Hersey number H_s
 137 defined by following equation:

$$138 \quad H_s = \frac{\eta V}{p} \quad (1),$$

139 where η ($N \cdot s/m^2$), V (m/s), and p (N/m^2) correspond to the lubricant viscosity, sliding speed, and apparent contact pressure, respectively
 140 [25]. In case of 3 mm thick DN gel, tendency of friction coefficient can be classified by Hersey number as friction region I, friction region II and
 141 transition between regions I and II as follows:

142

- 143 Friction region I: $H_s [m] < 5 \times 10^{-11}$,
- 144 Transition: $5 \times 10^{-11} < H_s [m] < 1.5 \times 10^{-10}$,
- 145 Friction region II: $1.5 \times 10^{-10} < H_s [m]$.

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Fig. 7 Summarized trend of friction coefficient with Hersey number for 1.5 mm, 3 mm and 6 mm cases.

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Summarized trend of friction coefficient with Hersey number for 1.5 mm, 3 mm and 6 mm cases is shown in Fig. 7. Criteria of each region is similar for all thickness cases in this study. The friction coefficient of 1.5 and 6 mm thick DN gel films can pass under 0.01 over a wide range of friction conditions, refer to friction region II. However, the 3 mm thick DN gel film does not show super low friction even under the similar value of Hersey number. It is assumed that such a low friction cannot be explained by the previously observed reduction of apparent contact pressure and different super low friction mechanism lies for 1.5 mm and 6 mm cases.

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The low friction mechanism in friction region II is considered to depend on the film thickness of DN gel. As shown in Fig. 2, a run-in at the beginning of the friction is the trigger to achieve a super low friction thus we focus on the run-in period for all thickness cases.

Fig. 8 Representative run-in behaviour at the beginning of sliding for each DN gel thickness

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Representative run-in behaviour at the beginning of sliding for each DN gel thickness is shown in Fig. 8 for each DN gel film thickness. In the case of 3 and 6 mm thick DN gel film, friction coefficient reduces and stabilizes after first run-in. 1.5 mm thick DN gel shows contrasting behaviour as shown in Fig. 8. Friction coefficient shows around 0.1 in the beginning, then drops lower than 0.01 and minimum value continues to decrease until the end of friction test. Finally, average friction coefficient shows lower value than minimum resolution of the apparatus.

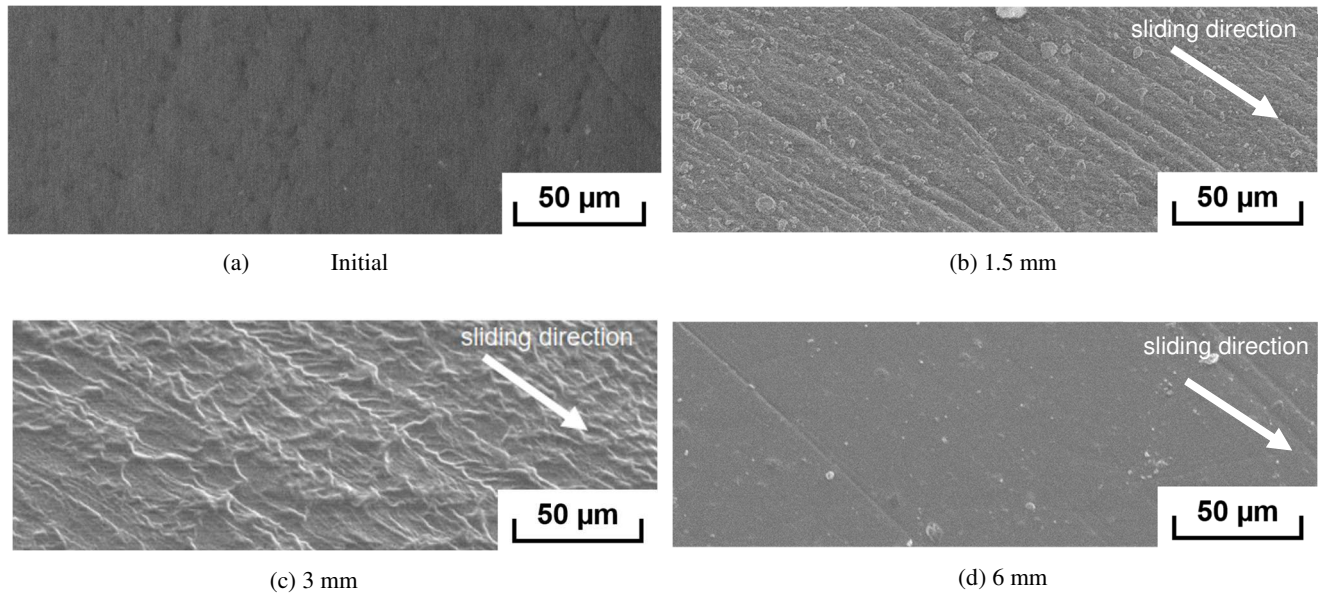


Fig. 9 SEM image of (a) initial DN gel surface and typical wear scars on the (b) 1.5 mm (c) 3 mm and (d) 6 mm thick DN gel films after friction test in friction region II

162

163 SEM image of (a) initial DN gel surface and typical wear scars on the (a) 1.5 mm (b) 3 mm and (c) 6 mm thick DN gel films after friction test in
 164 friction region II is shown in Fig. 9. In the case of the 3 mm thick DN gel film, the surface was fully damaged along sliding direction of counter
 165 surface and fish-scale like wear scars were made in the entire region. In contrast, on the surface of the 6 mm thick DN gel, few linear scars are
 166 seen. Finally, we found out a special linear scare using 1.5 mm thick DN gel.

167 Meanwhile, the scar-filled surface of the 3 mm thick DN gel indicates that high contact stiffness disables the formation of the water film
 168 locally even apparent contact pressure is small. Thus local adhesion between the DN gel and SiC disc prevents reduction of friction.
 169 Unsurprisingly, due to relaxed contact pressure, the tribological system using 6 mm thick DN gel, induces hydrodynamic lubrication regime and
 170 led to reach super low friction as explained by conventional studies using hydrogels against ceramics materials [26]. This is confirmed by the
 171 mild wear scars observed on the DN gel as shown in Fig. 9 (d).

172 The unique wear for 1.5 mm is exclusively observed in this case of double run-in behaviour. Thus, it is assumed that the wear occurring
 173 at the interface is the trigger element for the second decrease of friction coefficient to reach super low friction. The tensile stress on DN gel
 174 surface is already known to cause local yielding [7] and sparse layer where network structure is broken [12]. Due to thin film thickness (1.5 mm),
 175 enhanced tensile stress concentration on top surface can form similar sparse layer. Finally, sparse layer of hydrogel is known to be effective to
 176 form water layer between hydrogel and sliding counter surface [27]. Therefore first decrease of friction is related to the formation process of
 177 sparse layer and second decrease is linked to the formation process of water layer between DN gel and sliding counterpart, possible by
 178 absorption of water to form a water film at the interface. For the 1.5mm thick DN gel, a unique phenomenon of double run-in is observed. An
 179 aggressive stress, due to stiffer contact, forms a damaged layer on the DN gel surface. The damaged layer also called sparse layer become moist
 180 then thin water film is created locally and lower friction coefficient is obtained. This process is the key to design a tribological system using a
 181 thin DN gel film to realize super low friction.

182 The DN gel is a complex material made of a double polymer network, then the friction applied on the material certainly affects other
 183 properties such as its absorption capacity or its chemical structure change. In this study we mostly focused on the micrometrical mechanical
 184 process and how it can be controlled by changing the DN gel thickness.

185

5. Conclusions

Friction tests using a DN gel film and SiC disc were carried out under water-lubricated conditions with various loads and sliding speeds. From the results previously exposed, the following conclusions were obtained.

(1) The friction region can be classified into two types as follows:

Friction region I: friction remains substantially unchanged during the test,

Friction region II: friction decreases due to run-in.

(2) Regardless of the DN gel film thickness, transition condition of friction region can be expressed by Hersey number quantitatively as follows:

Friction region I: $H_s [\text{m}] < 5 \times 10^{-11}$,

Transition: $5 \times 10^{-11} < H_s [\text{m}] < 1.5 \times 10^{-10}$,

Friction region II: $1.5 \times 10^{-10} < H_s [\text{m}]$.

(3) 1.5 and 6 mm thick DN gel films can reach super low friction ($\mu < 0.01$) under a wide range of friction conditions as friction region II.

(4) In the case of 1.5 mm thick DN gel film, friction decreases in two steps by double run-in phenomena.

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