

Study of correlation between grease film formations and mechanical losses on various surfaces

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Defense of the PhD thesis
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JXTG Nippon Oil & Energy



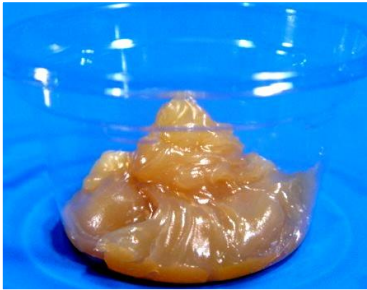
**Institute of Machine
and Industrial Design**

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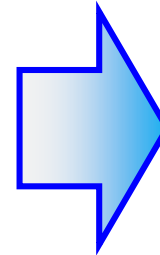
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Introduction - Grease product development

Grease



- ✓ Not leaky
- ✓ No circulation system
- ✓ Low lubricant volume



Bearing lubrication



Grease
lubrication > 90%

Global demand for energy-saving properties
in order to reduce CO₂ emissions.



Requirement for grease development

1. High performance grease reducing bearing mechanical loss
2. Persuasive mechanisms of performance for customers

Mission

Clarifying the relationship between
grease formulations and mechanical losses (bearing torque)

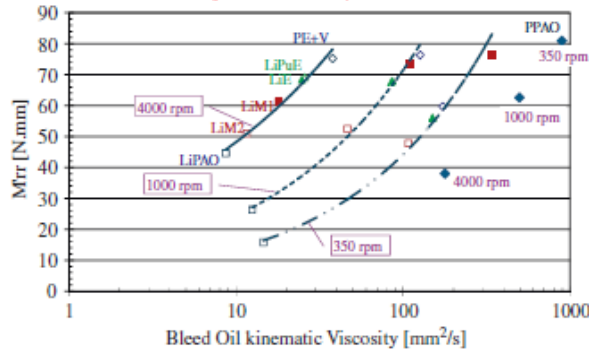
State of the art review and analysis

1. Bearing torque under grease lubrication

1.1 Thrust type

Seabra et al. decomposed a total friction torque into rolling torque and sliding torque.

Relevance; **Rolling torque: base oil viscosity**



High viscosity → High rolling torque

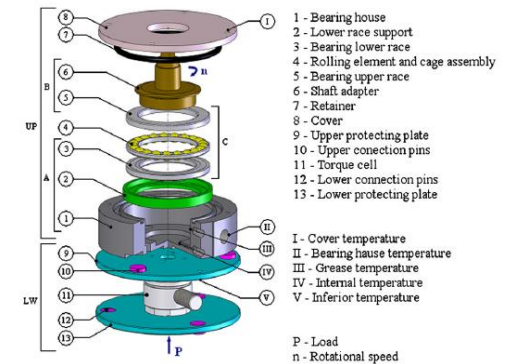
Limitation: thrust type bearing, commercial greases

Cousseau, T., Graca, B., Campos, A., and Seabra, J., "Friction Torque in Grease Lubricated Thrust Ball Bearings," *Tribology International*, 44, 2011, 523-531.

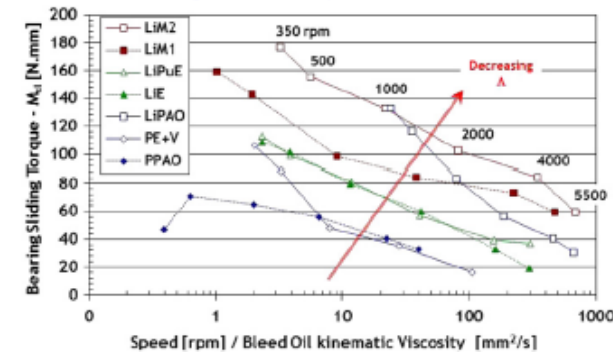
Cousseau, T., Graca, B., Campos, A., and Seabra, J., "Experimental Measuring Procedure for the Friction Torque in Rolling Bearings," *Lubrication Science*, 22, 2010, 133-147.

Cousseau, T., Graca, B. M., Campos, A. V., and Seabra, J. H. O., "Influence of Grease Rheology on Thrust Ball Bearings Friction Torque," *Tribology International*, 46, 2012, 106-113.

Defense of the PhD thesis



Sliding torque: film thickness



Thin film thickness

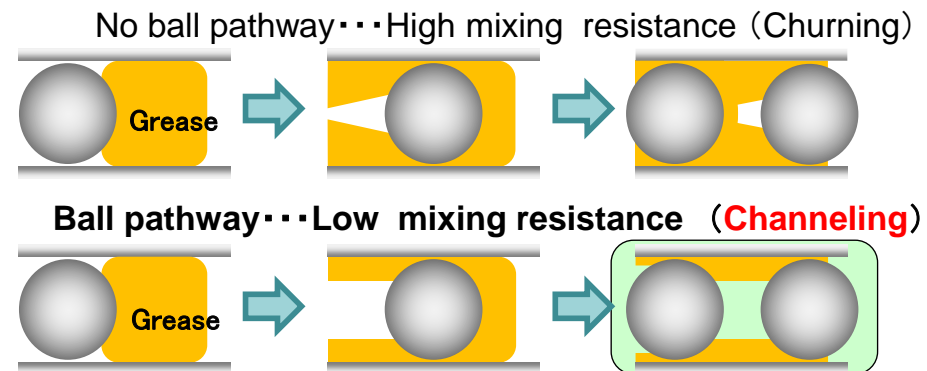
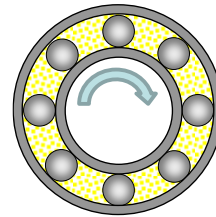
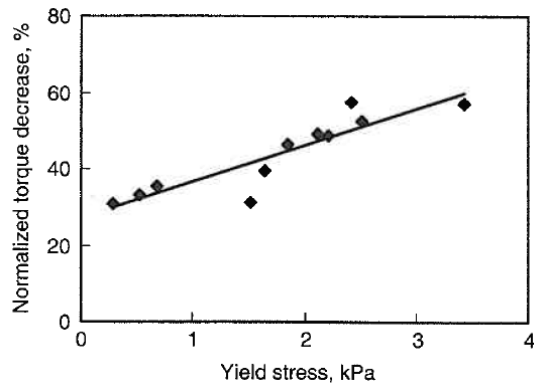
→ High sliding torque

State of the art review and analysis

1. Bearing torque under grease lubrication

1.2 Radial type

Yokouchi et al. indicated relationship between bearing torque and yield stress of greases.



*Channeling and churning of grease

High yield stress → Torque reduction
Due to grease channeling

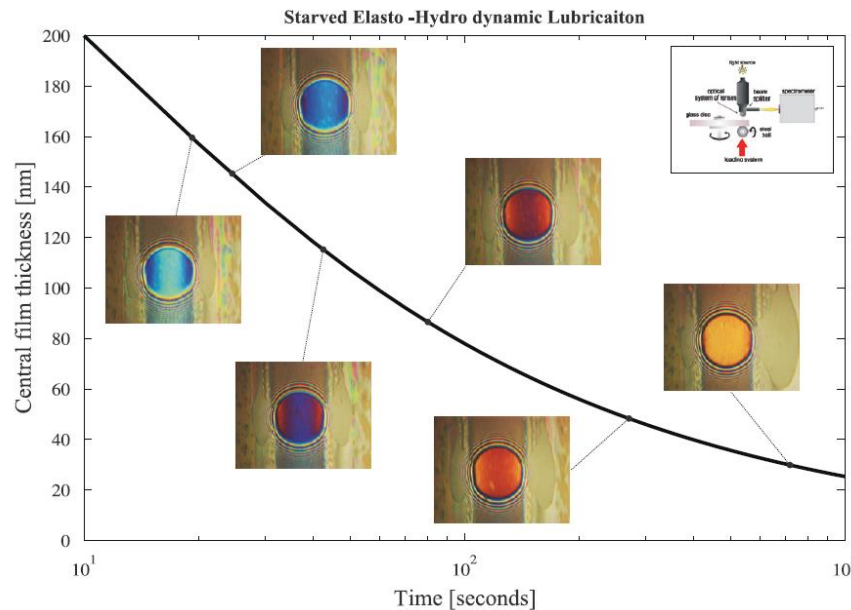
Limitation: one type thickener grease(Li-12OH-stearate)

Oikawa, E., Inami, N., Hokao, M., Yokouchi, A., and Sugimura, J., "Bearing Torque Characteristics of Lithium Soap Greases with Some Synthetic Base Oils," *Proc IMechE Part J: J Engineering Tribology*, 226, 6, 2012, 575-583.

State of the art review and analysis

2. Grease behaviors in a bearing

Venner et al. estimated the reduction of the grease film thickness in bearings by numerical simulations of grease flows.



The transition from flooded region to heavily starved contact

Bearings can be operated in not only flooded but also starved conditions.

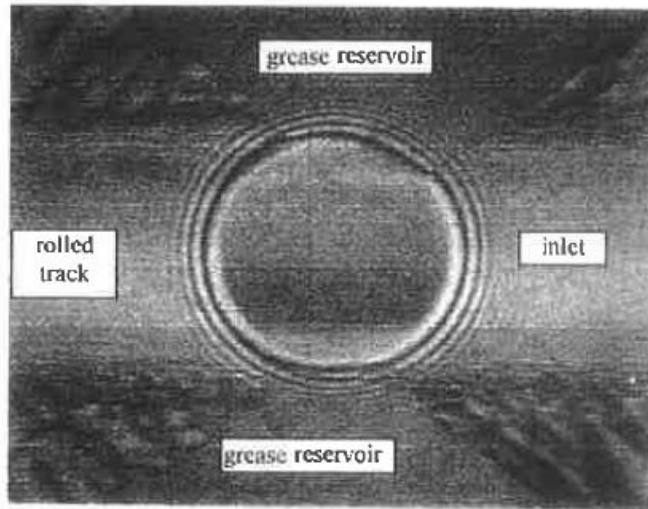
Limitation: no direct information about relationship with bearing torque

Venner, C. H., van Zoelen, M. T., and Lugt, P. M., "Thin Layer Flow and Film Decay Modeling for Grease Lubricated Rolling Bearings," *Tribology International*, 47, 2012, 175-187.

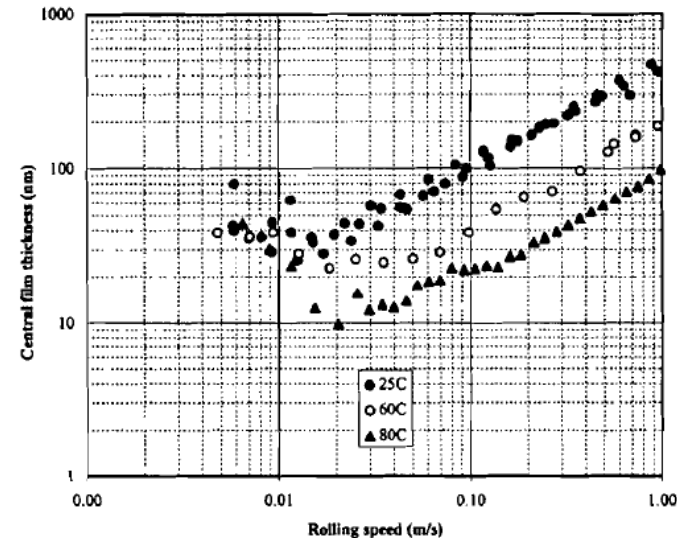
State of the art review and analysis

3. Film thickness under grease lubrication

Cann et al. reported the behaviors of grease film thickness in EHL.



Grease is pushed away with disk rotations.



Under fully flooded condition, greases augment film thickness in slow speed.

Limitation: no direct information about relationship with bearing torque

Cann, P., and Lubrecht, A. A., "An Analysis of the Mechanisms of Grease Lubrication in Rolling Element Bearings," *Lubrication Science*, 11-3, 1999, 227-245.

Cann, P., "Starved Grease Lubrication of Rolling Contacts," *Tribology Transactions*, 42, 4, 1999, 867-873.

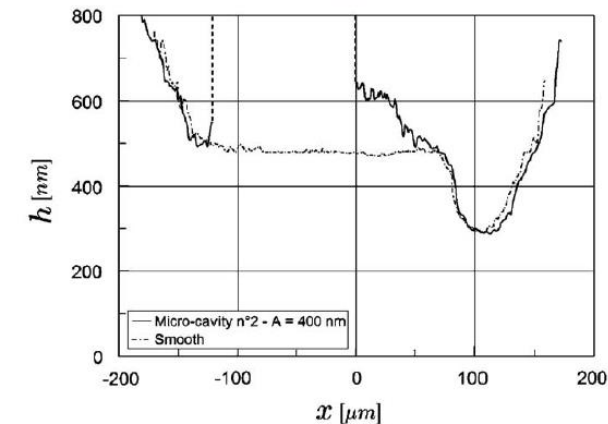
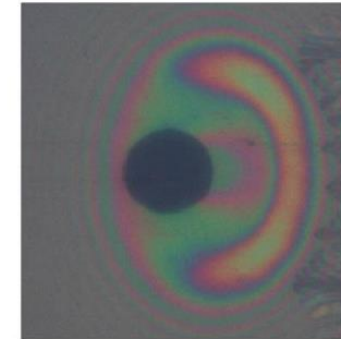
State of the art review and analysis

4. Surface texturing for film thickness (oil lubrication)

Mourier et al. indicated shallow micro cavity can increase Film thickness under rolling/sliding condition.

Krupka et al. showed deep micro dents decrease film thickness but shallow micro dents increase.

Possibility of improvement of film thickness by surface texturing depending on the specific conditions in spite of non-conformal contacts



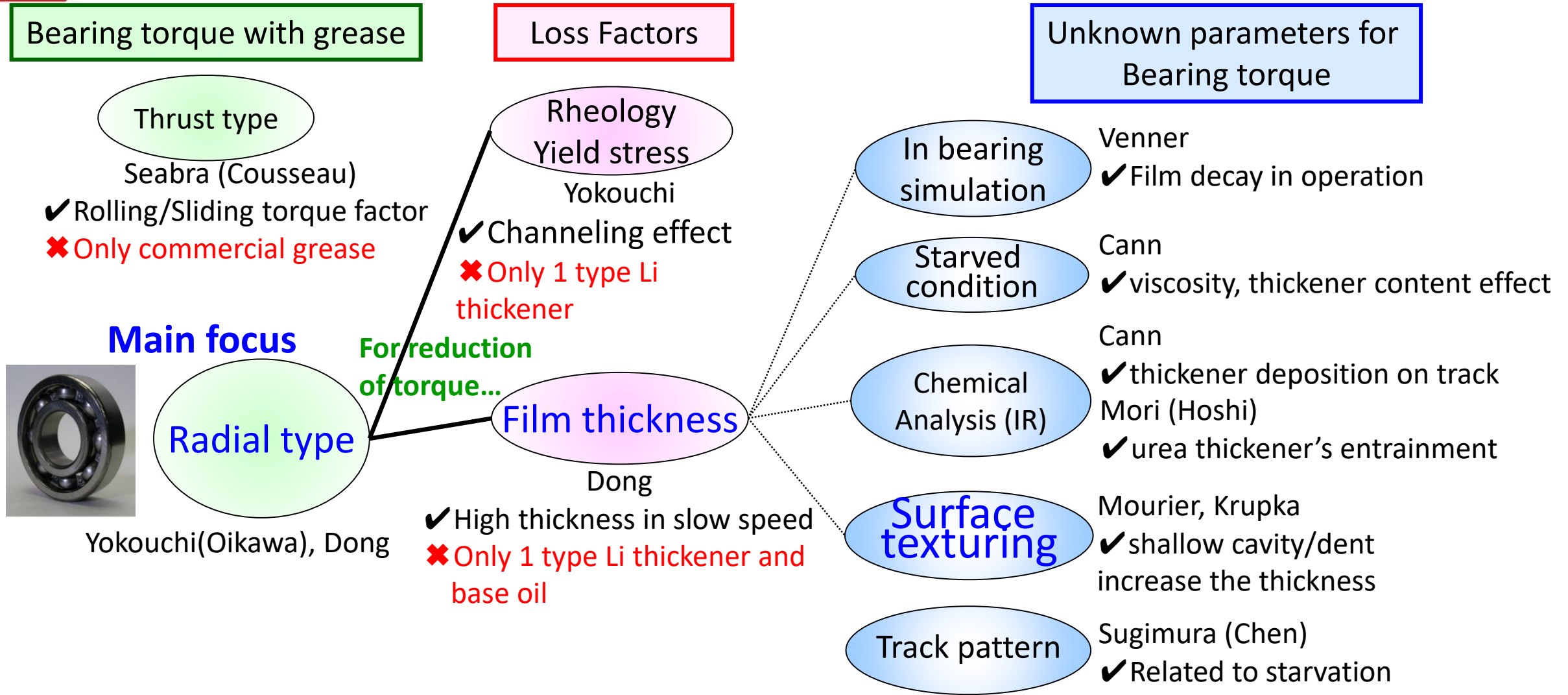
Limitation: no application to **grease lubrication**

Mourier, L., Mazuyer, D., Lubrecht, A. A., and Donnet, C., "Transient Increase of Film Thickness in Micro-Textured EHL Contacts," *Tribology International*, 39, 2006, 1745-1756.

Krupka, I. and Hartl, M., "The Effect of Surface Texturing on Thin EHD Lubrication Films," *Tribology International*, 40, 2007, 1100-1110.

Krupka, I. and Hartl, M., "The Effect of Surface Texturing on Very Thin Film EHD Lubricated Contacts," *Tribology Transactions*, 52, 2009, 21-28.

State of the art review and analysis



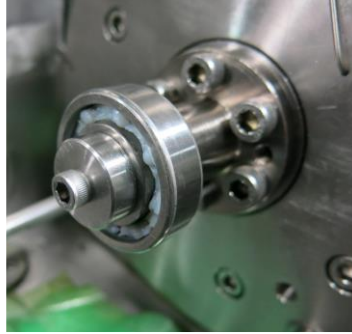
Essence and goals of the PhD thesis

- 1. Understanding of influence of grease formulation (focused on **Li thickener**) on radial ball bearing torque**
- 2. Analysis of grease properties for clarification of lubrication mechanism**
- 3. New approach as **grease EHL film** observation including **surface texturing****

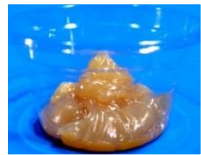
New findings for future grease product development

Essence and goals of the PhD thesis

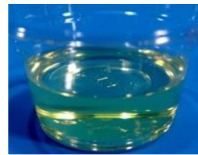
◎ Study of the relationship between grease formulations and bearing torque



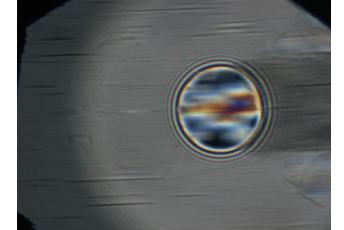
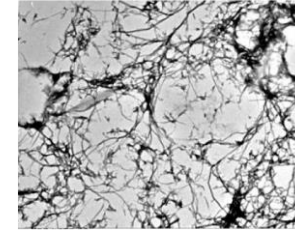
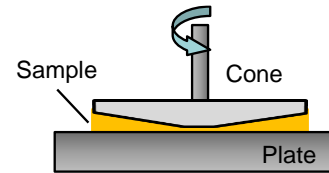
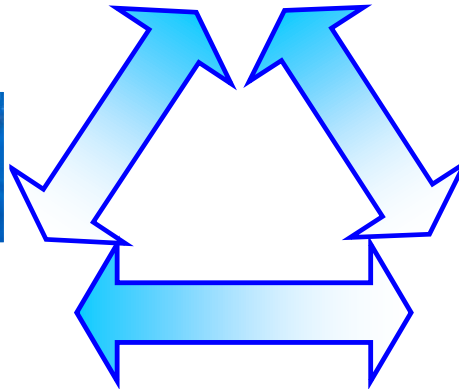
Bearing torque



- Base oil
- Thickener



Grease formulation



- Rheology
- Thickener fiber structure
- Film thickness including surface texturing

Factor evaluation

Scientific questions and working hypotheses

Questions

- How do properties of grease influence on the radial ball bearing torque?
- Are there relations among the grease properties?

Working hypotheses

1. Rheological factor \Rightarrow Yield stress and/or viscosity
2. Thickener structure \Rightarrow Thickener fiber shape and size
3. Ability of film thickness and adaptability to surface conditions \Rightarrow
Thicker film on both smooth and textured surfaces is better for torque reduction
4. Those factors correlate each other

Research method

1. Grease samples

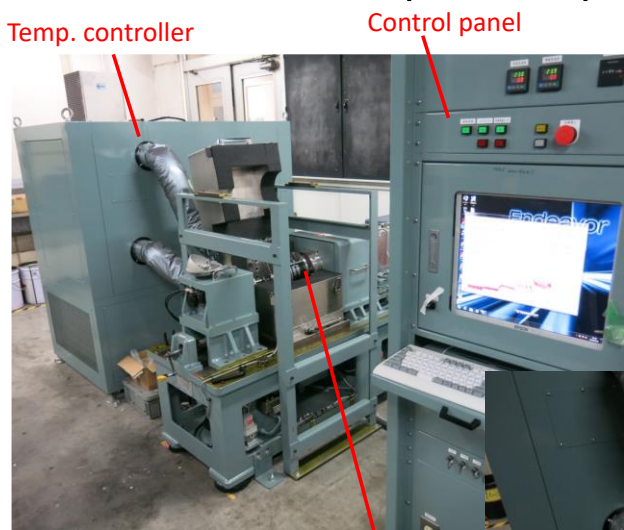
- Low viscosity grade oil
- Lithium type thickener : Considering recent trend of grease development

Samples	L- (Li-Complex)	S1- (Single Li)	S2- (Single Li)	Base oil
Base oil	OilA: Mineral oil (API Group-I), Kinetic viscosity(40°C): 33.2 mm ² /s, VI:107 OilB: Poly-α-olefin (API Group-IV), Kinetic viscosity(40°C): 30.6 mm ² /s, VI:135			
Thickener	12OH-stearic/ azelaic-Li	Stearic-Li	12OH- stearic-Li	-
Dropping point	250 °C	200 °C	200 °C	-

Research method

2. Bearing torque (JXTG)

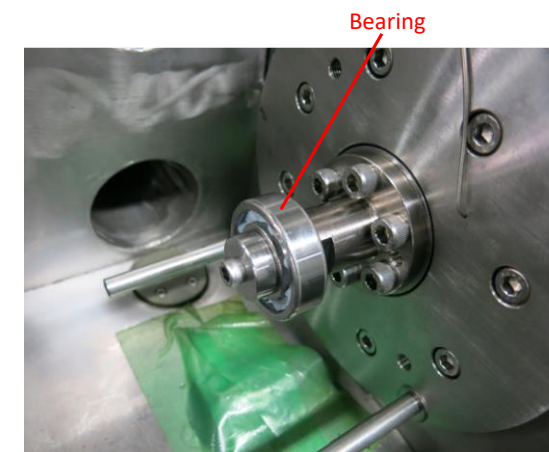
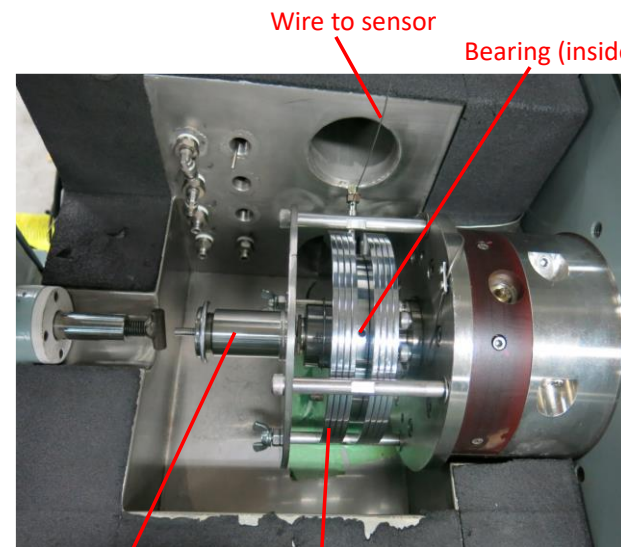
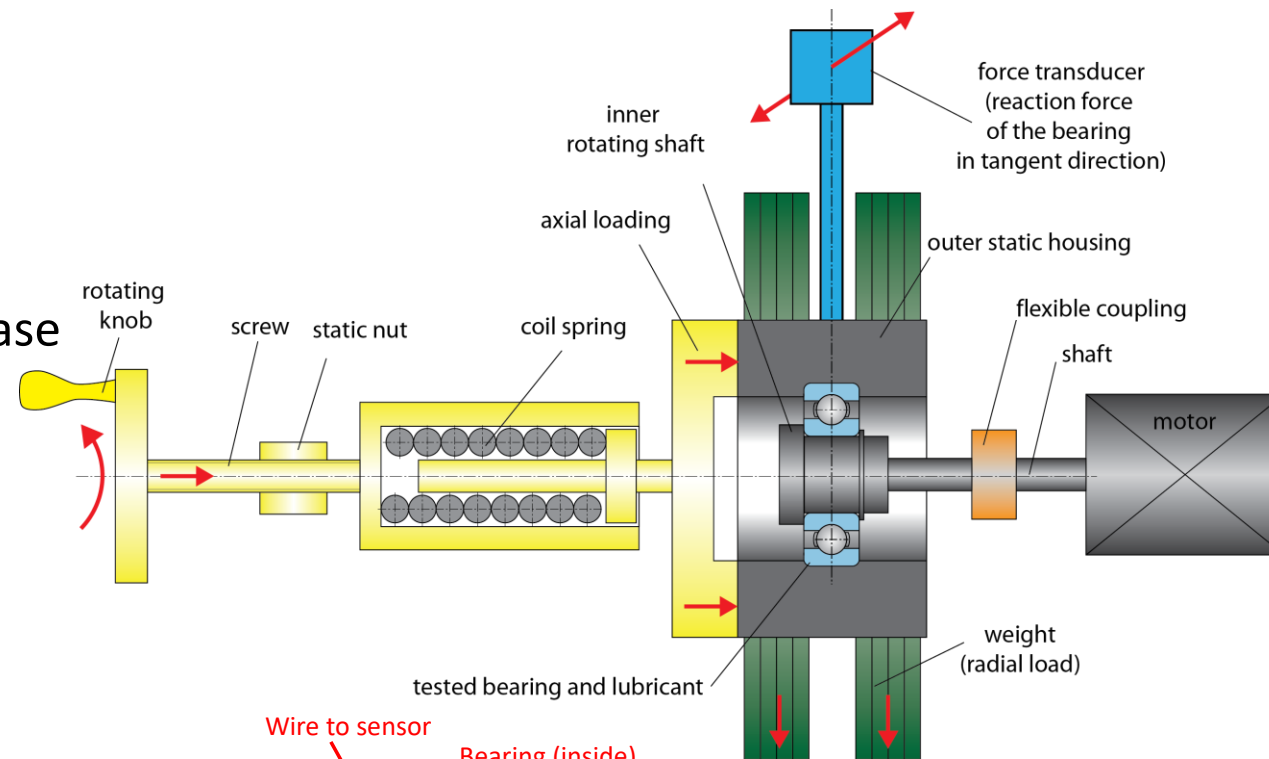
- Original frictional torque measuring rig for grease
- Rotation speed dependence for each grease



Bearing unit



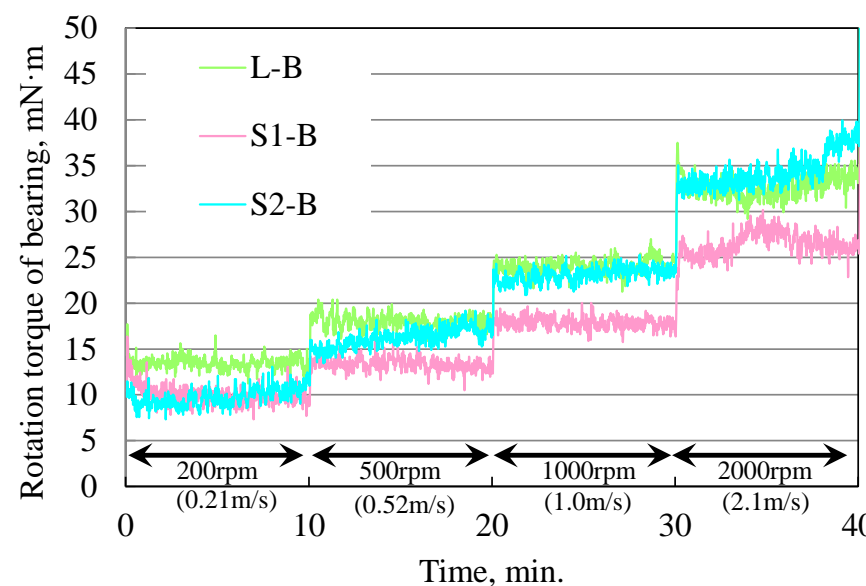
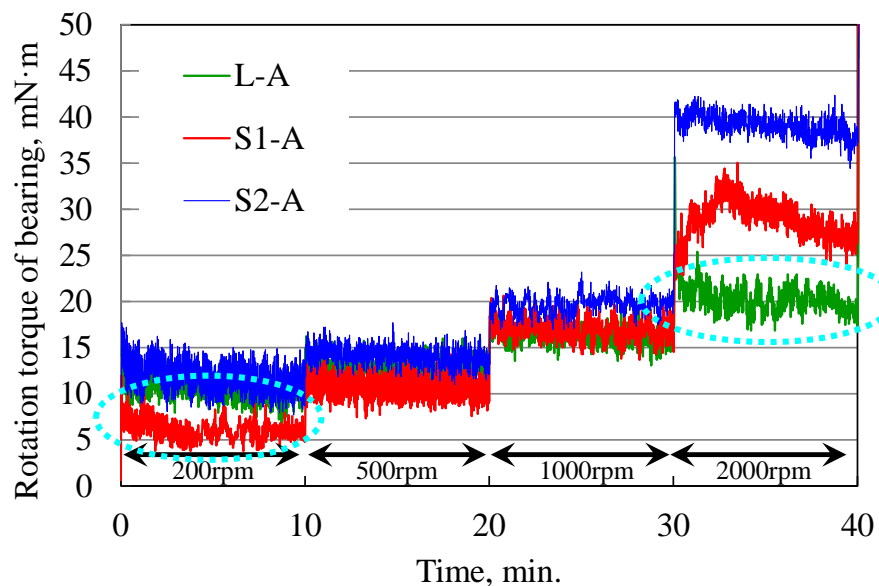
Bearing unit (inside)



Bearing torque -screening-

Grease		L-A	S1-A	S2-A	L-B	S1-B	S2-B
Base oil	Mineral oil (G-I), %	88	92	92	-	-	-
	Poly- α -olefin, %	-	-	-	88	92	92
Thickener	Li complex, %	12	-	-	12	-	-
	Li stearate, %	-	8	-	-	8	-
	Li-12OH-stearate, %	-	-	8	-	-	8
Penetration (60W)		265	293	359	311	305	341

	Test condition
Bearing	6204 without seal
Lubricant content	2g
Rotation speed	200-500-1000-2000rpm
Duration	10 min. each
Temperature	25 °C
Thrust load	50 N
Radial load	50 N
Evaluation	Friction torque

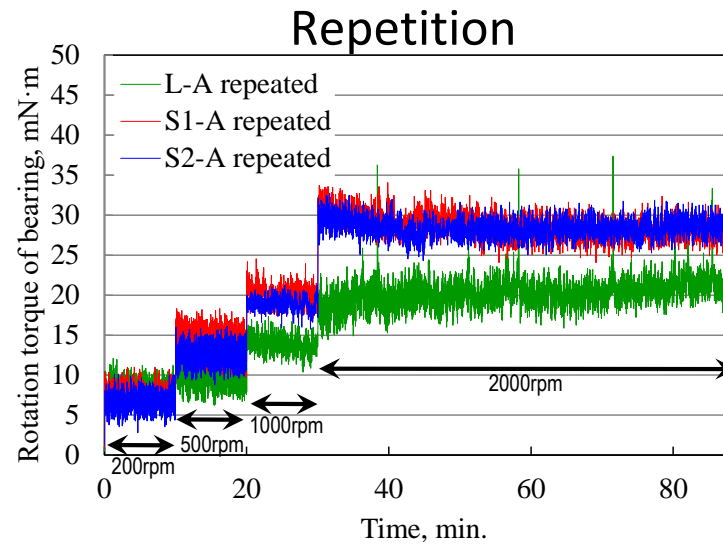
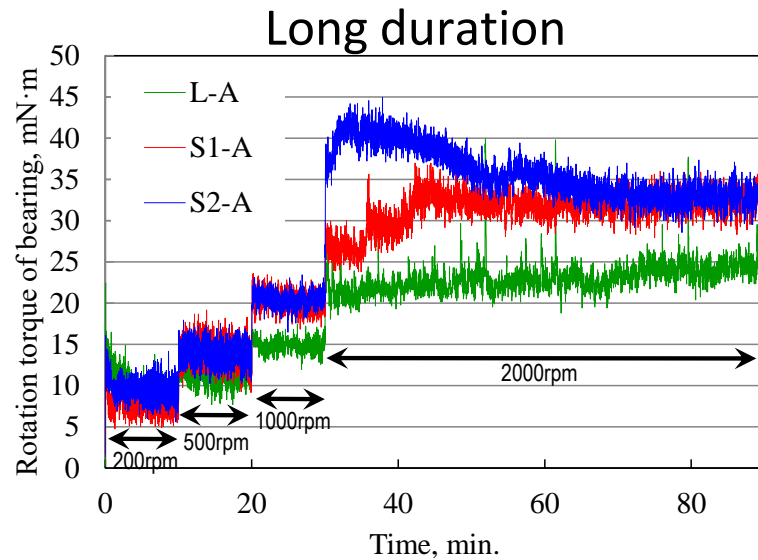


L-A: the lowest torque
high (2000 rpm) speed

S1-A: the lowest torque
in low (200 rpm) speed

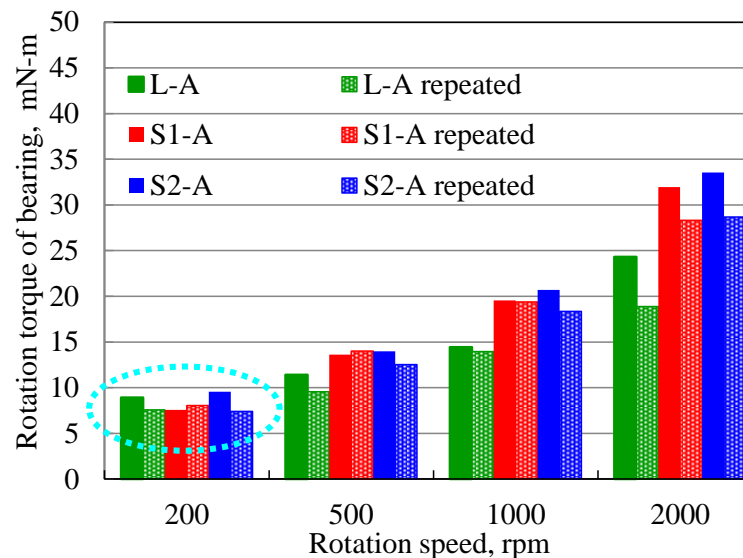
Bearing torque -long duration and repetition-

L-A: Li-complex
 S1-A: Single Li-Stearate
 S2-A: Single Li-12OH-Stearate



L-A shows lower torque in high speed in repeated condition

	Test condition
Bearing	6204 without seal
Lubricant content	2g
Rotation speed, Duration	200(10min.) -500(10min.) -1000(10min.) -2000rpm(60min.)
Temperature	25 °C

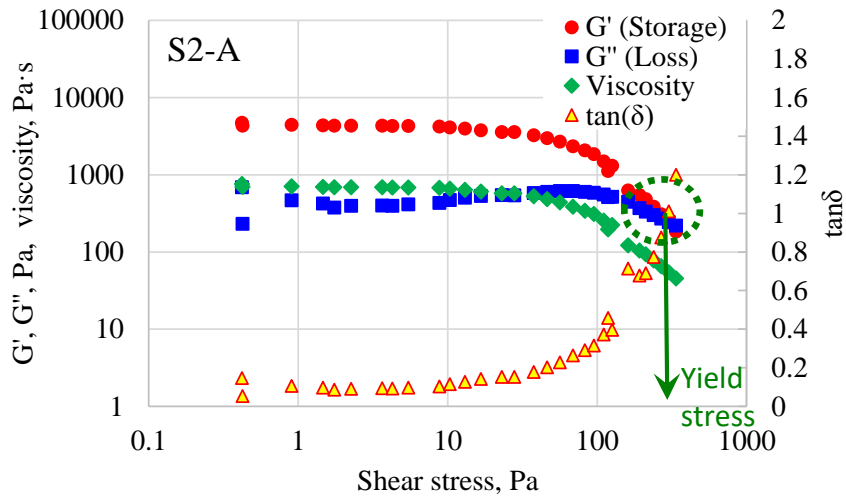


The lowest torque of S1-A in low speed disappeared in repeated condition

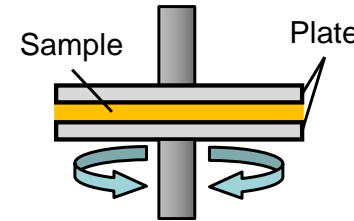
→ Grease property investigation

Rheology -Viscoelasticity-

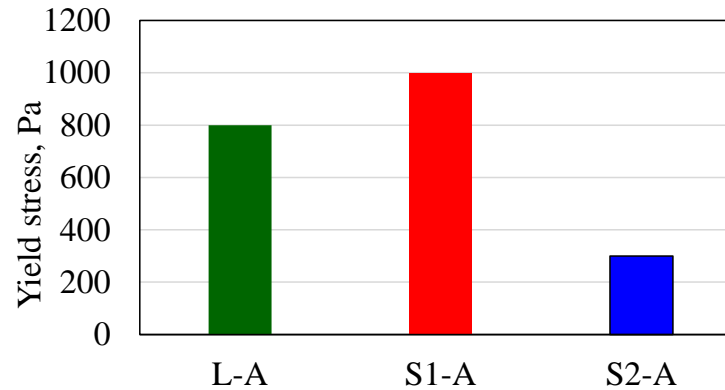
L-A: Li-complex
 S1-A: Single Li-Stearate
 S2-A: Single Li-12OH-Stearate



G': Solid parameter
 G'': Liquid parameter
 $\tan\delta = G'/G'' \geq 1$
 → Yield

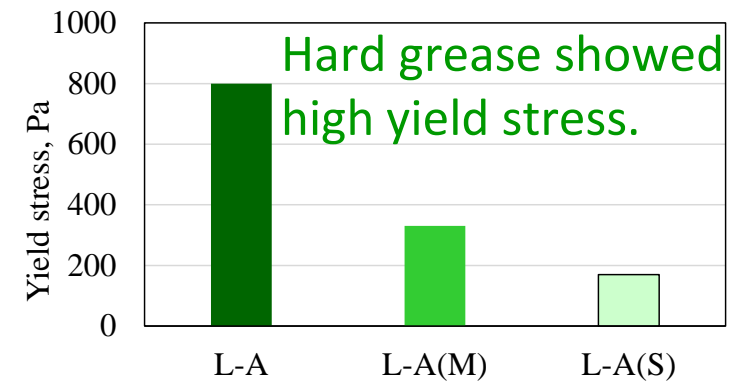


	Test condition
Plate diameter	25 mm
Frequency	1Hz
Deformation	0.001-100%
Temperature	25 °C



S1-A showed the highest yield stress.
 (Contrary to past reports)

Grease		L-A	S1-A	S2-A	L-A(M)	L-A(S)
Base oil	Mineral oil (G-I), %	88	92	92	90	92
Thickener	Li complex, %	12	-	-	10	8
	Li stearate, %	-	8	-	-	-
	Li-12OH-stearate, %	-	-	8	-	-
Penetration (60W)		265	293	359	289	360



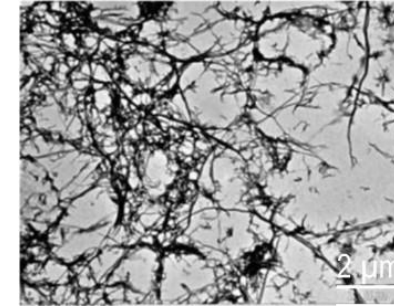
Yield stress does not predict torque behavior
 for different thickener types

Thickener structures of thickeners observed by TEM

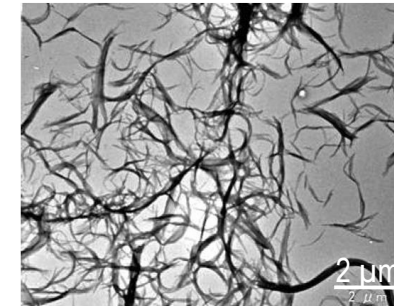
TEM
(Transmission Electron Microscope)

Oil Extraction	Hexane
Acceleration voltage	20kV
Amplitude	× 10000

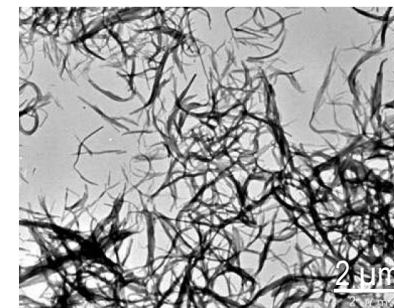
L-A:
Li-complex



S1-A:
Single Li-Stearate



S2-A:
Single Li-12OH-Stearate



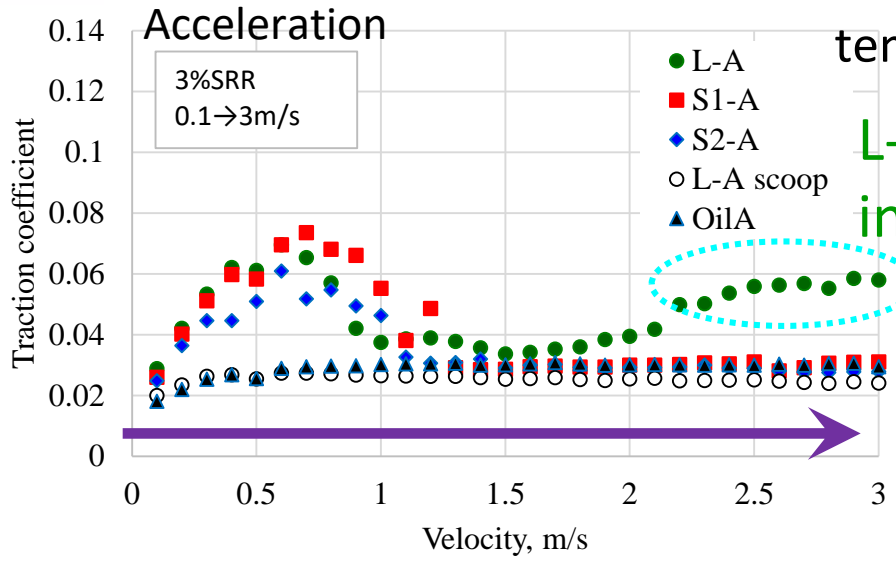
L-A: thin and long fiber networks

S1-A and **S2-A:** thick and short fiber networks

Relationship with bearing torque is not confirmed.

Traction coefficient -velocity dependence-

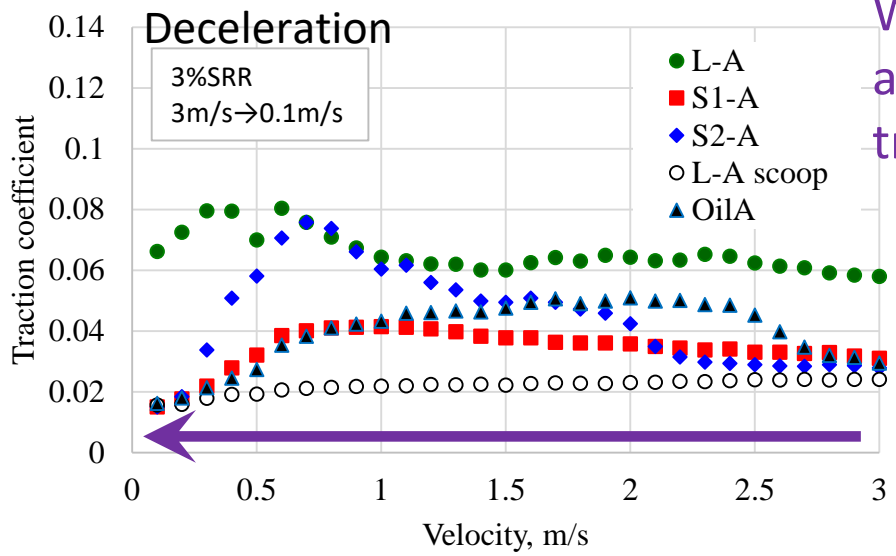
L-A: Li-complex
 S1-A: Single Li-Stearate
 S2-A: Single Li-12OH-Stearate



Without scoop,
 temporary traction increase

L-A showed traction increase at high speed.

With scoop, the traction is the same to base oil.



Without scoop,
 all samples showed traction increase.

MTM		Test condition
Specimen	Ball	Steel ϕ 19.05 mm
	Disk	Steel
Mean Hertzian pressure		430 MPa (10N)
Velocity		0.1-3-0.1 m/s
Slide roll ratio		3 %
Temperature		25 °C
Grease supply		V-shaped scoop

Traction increase of L-A could be due to lack of lubricants (channeling in bearing)

Sample	L-A	S1-A	S2-A	OilA	
Base oil	Mineral oil (G-I), %	88	92	92	100
Thickener	Li complex, %	12	-	-	-
	Li stearate, %	-	8	-	-
	Li-12OH-stearate, %	-	-	8	-
Penetration (60W)	265	293	359	-	

Film thickness and indentation

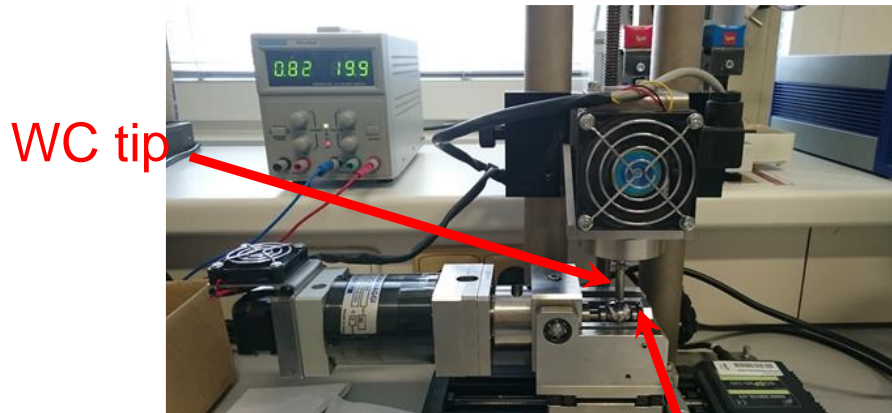
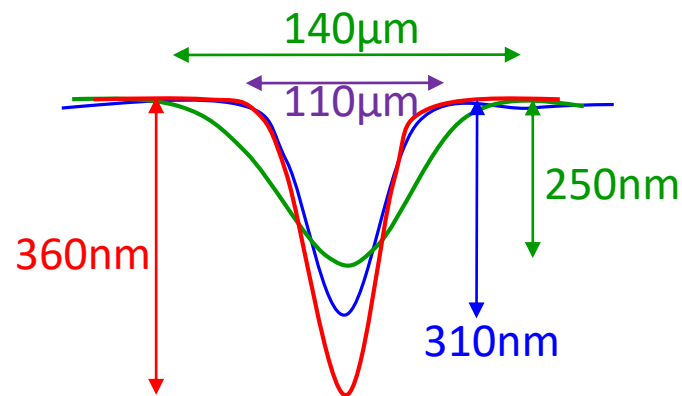
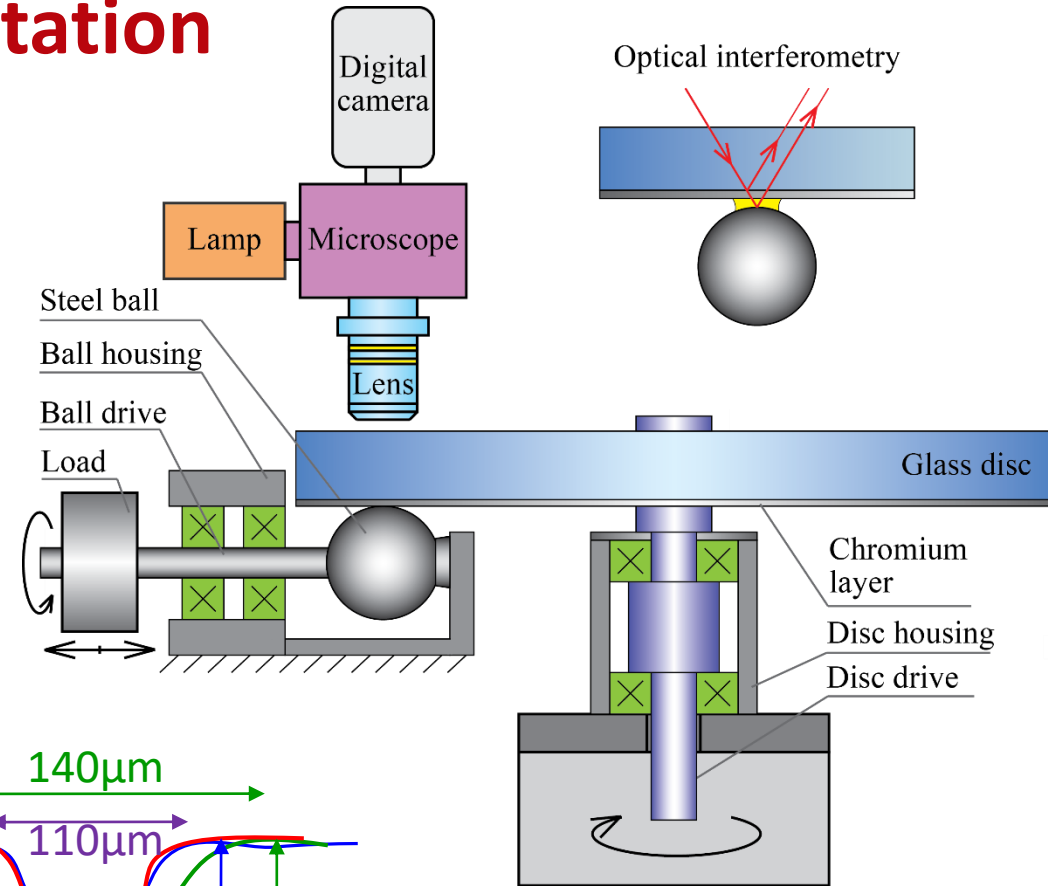
Film thickness

- Colorimetric interferometry technique
- Smooth / dented steel ball on glass disk
- Fully-flooded / starved condition

Target depth: 200-400nm depth

→ Tungsten carbide (WC) ball indentation

Diameter: 2.5mm, 1.6mm, 1.27mm

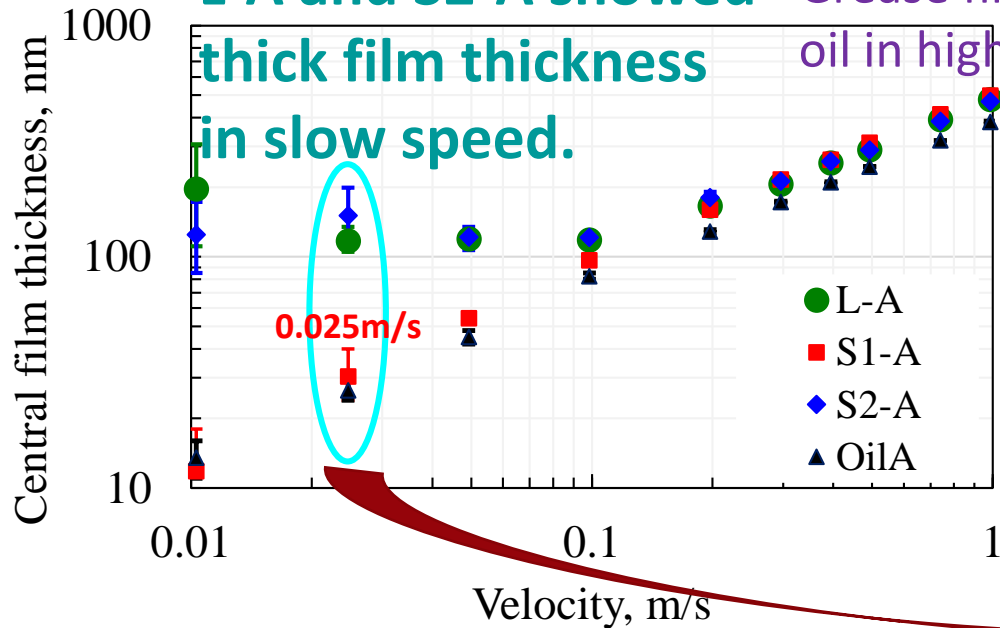


Steel ball

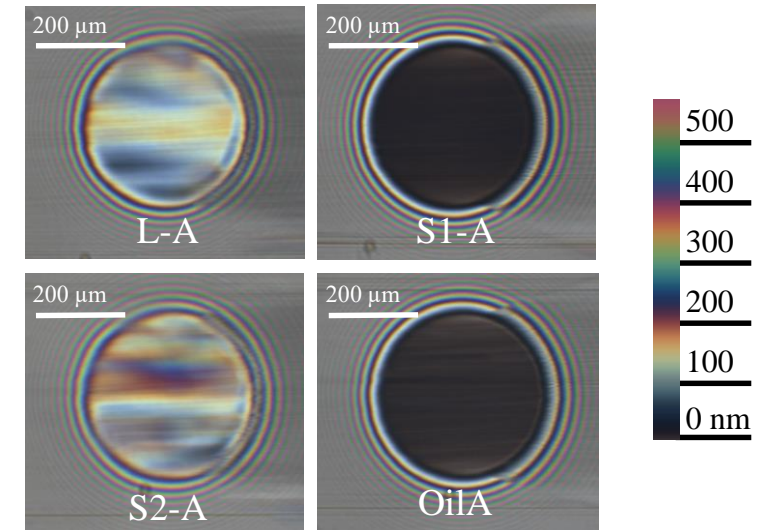
Fully flooded condition -smooth, acceleration-

L-A: Li-complex
 S1-A: Single Li-Stearate
 S2-A: Single Li-12OH-Stearate

L-A and S2-A showed thick film thickness in slow speed. Grease film thicknesses close to base oil in high speed.



Interferograms



Rolling direction

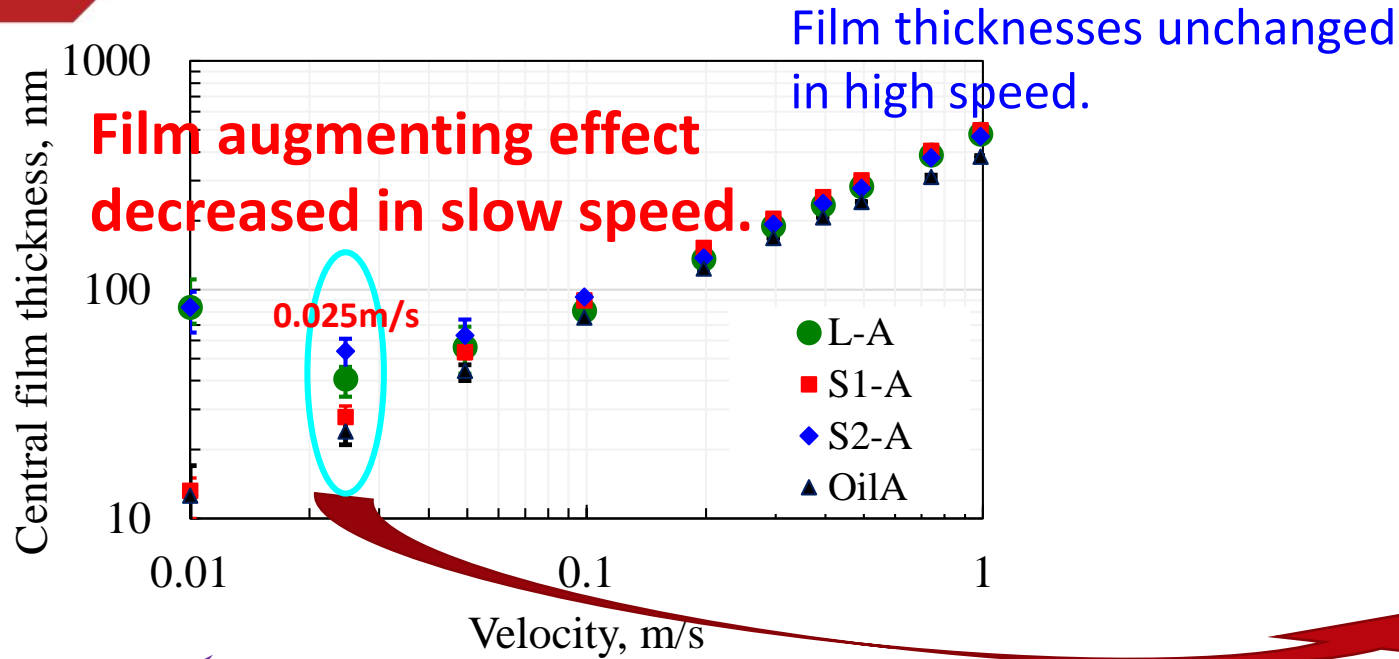
**Thickener particles entrainment
 increased film thickness**

Thickener type effect is larger in slow speed.

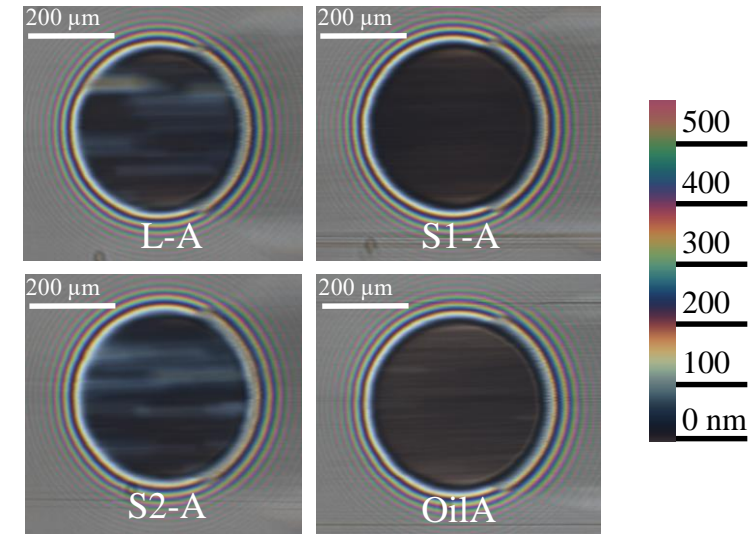
		Test condition
Specimen	Ball	Steel ϕ 25.4 mm
	Disk	Cr-coated glass
Mean Hertzian pressure		290 MPa
Hertzian contact diameter		348 μ m
Velocity		0.01-1 m/s
Slide roll ratio		0 % (nominally)
Temperature		23 $^{\circ}$ C
Grease supply		V-shaped scoop

Fully flooded condition -smooth, deceleration-

L-A: Li-complex
 S1-A: Single Li-Stearate
 S2-A: Single Li-12OH-Stearate



Interferograms

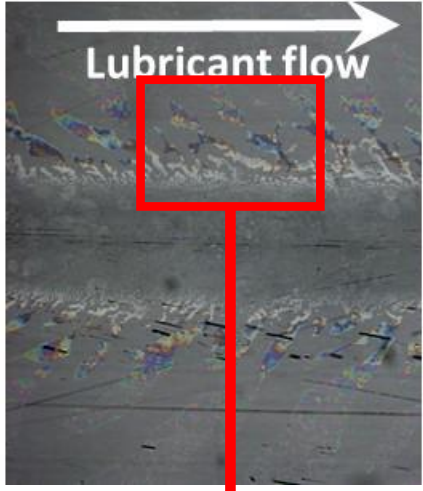
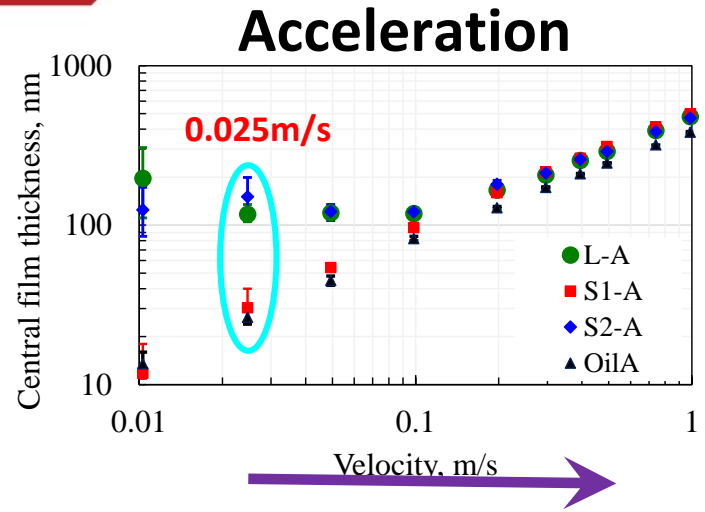
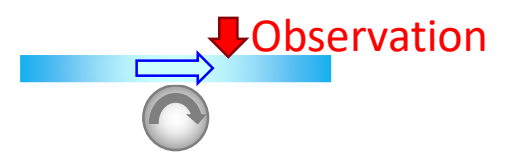


Thickener particles effect decreased

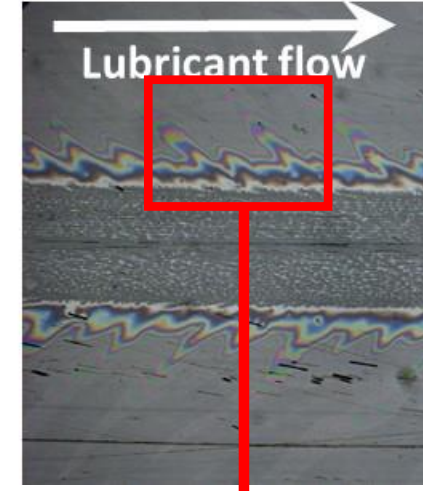
Thickener particles could be influenced by high speed condition.

		Test condition
Specimen	Ball	Steel φ25.4 mm
	Disk	Cr-coated glass
Mean Hertzian pressure		290 MPa
Hertzian contact diameter		348 μm
Velocity		0.01-1 m/s
Slide roll ratio		0 % (nominally)
Temperature		23 °C
Grease supply		V-shaped scoop

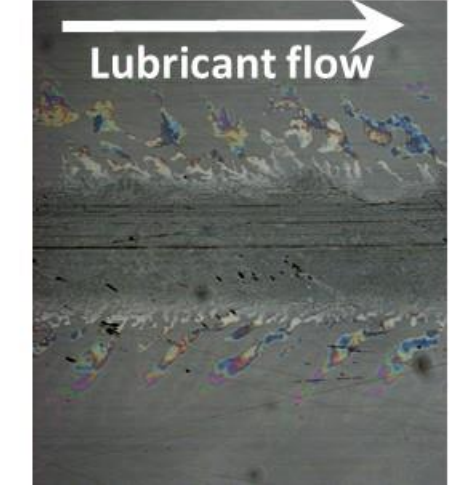
Track patterns of downstream (0.025m/s)



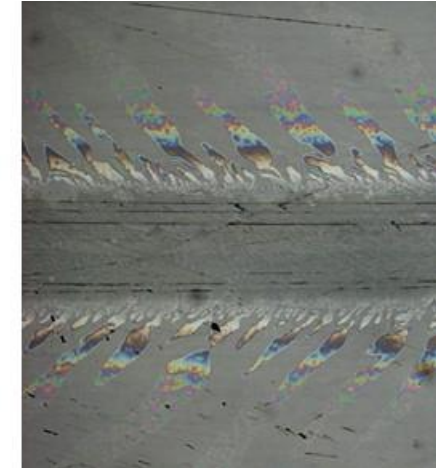
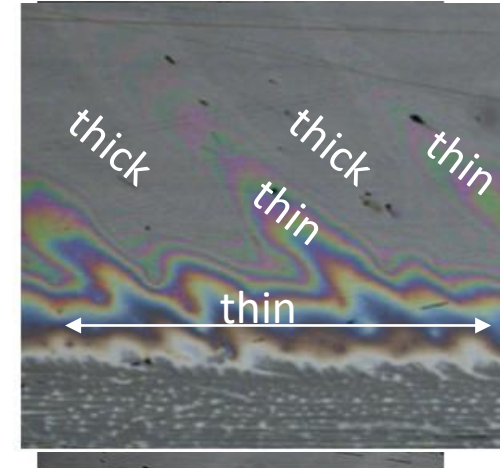
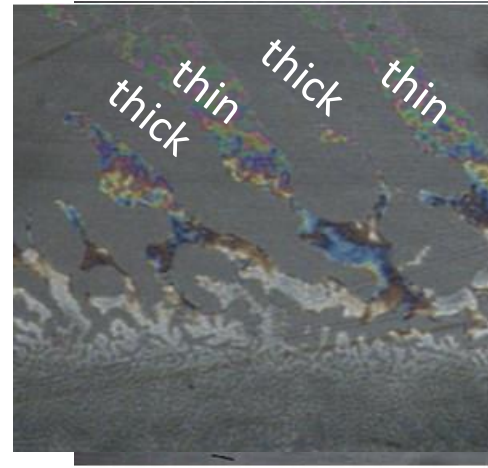
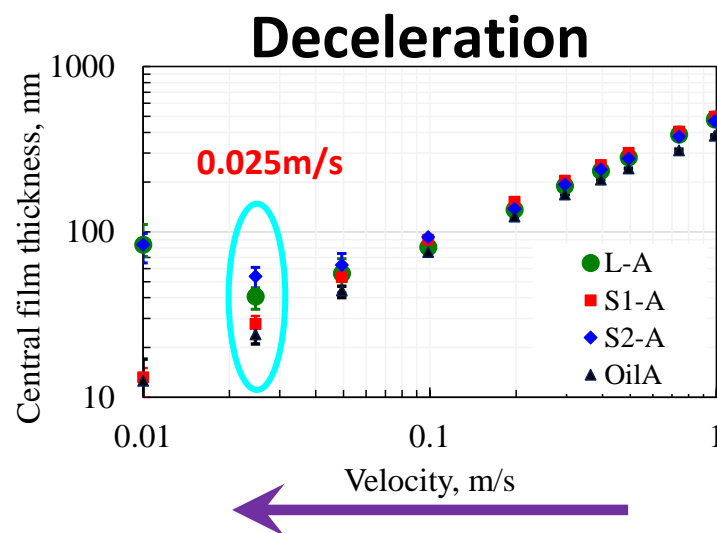
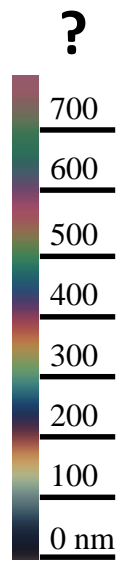
L-A: Li-complex



S1-A: Single 11-Stearate



S2-A: Single Li-12OH-Stearate

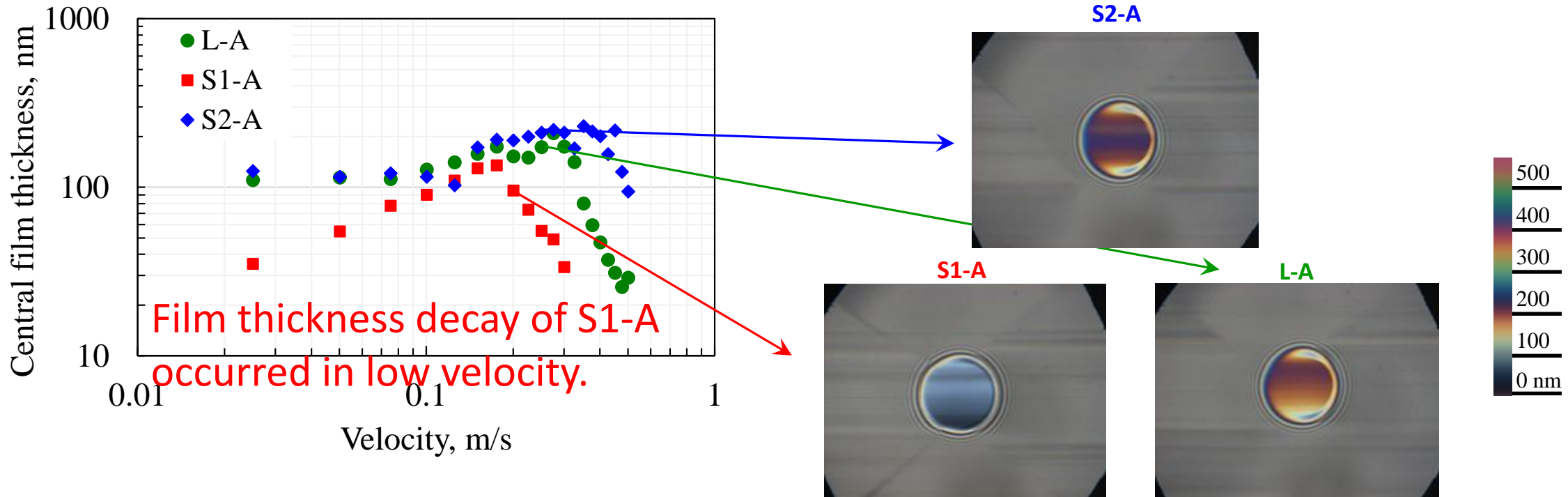


Side "fingers" sharpened after high velocity conditions for L-A, S2-A
 The fingers of S1-A were not clear

Starved conditions

L-A: Li-complex
S1-A: Single Li-Stearate
S2-A: Single Li-12OH-Stearate

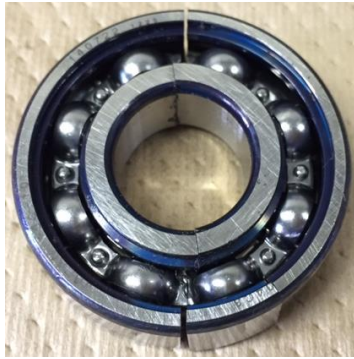
Velocity increased by 0.024 m/s every 30 sec. without grease supply



Interferograms under 0.2-0.3 m/s

The tendency that thickener particles of S1-A are not dragged into the contact area might cause early starvation without grease supply.

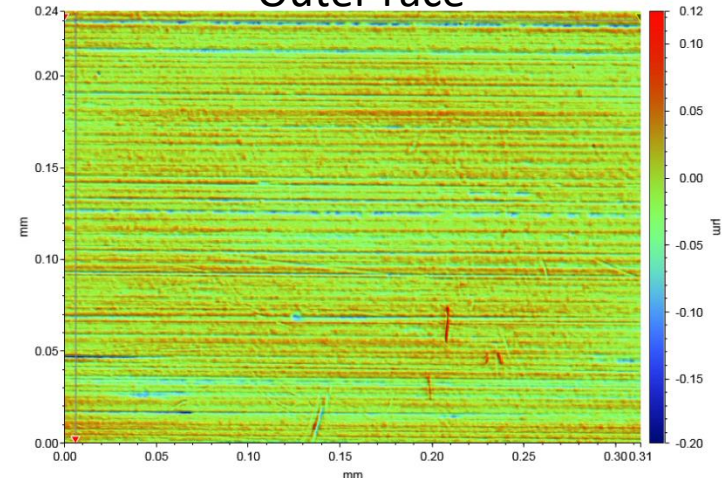
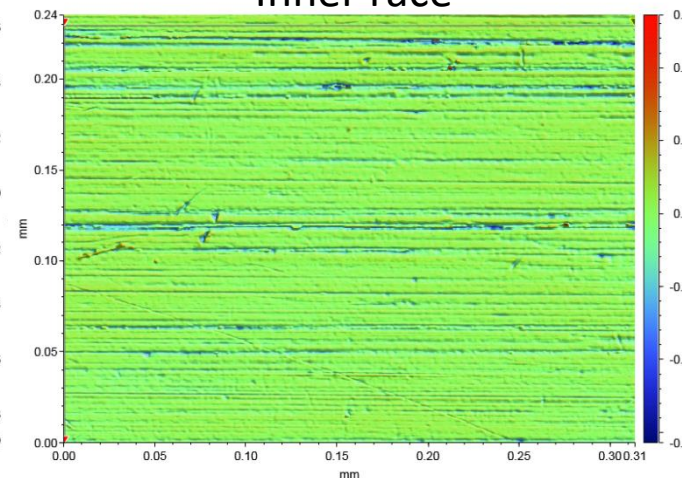
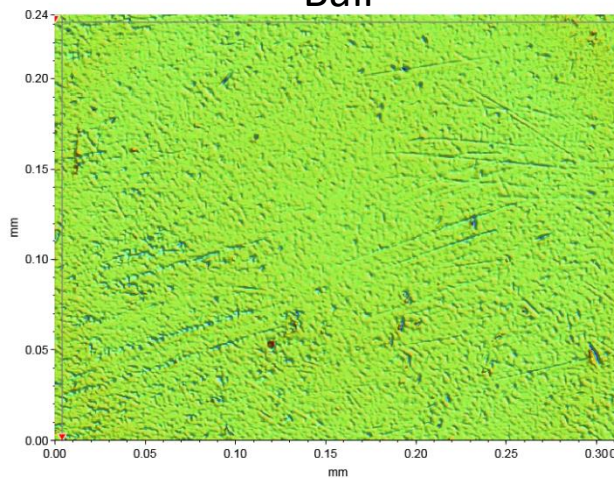
Bearing surface profile



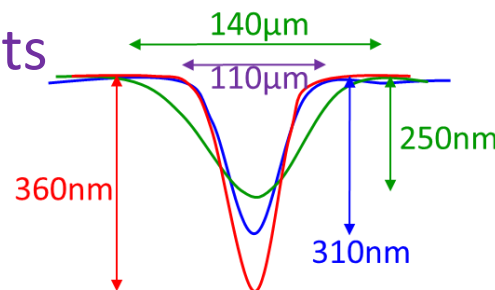
Ball

Inner race

Outer race



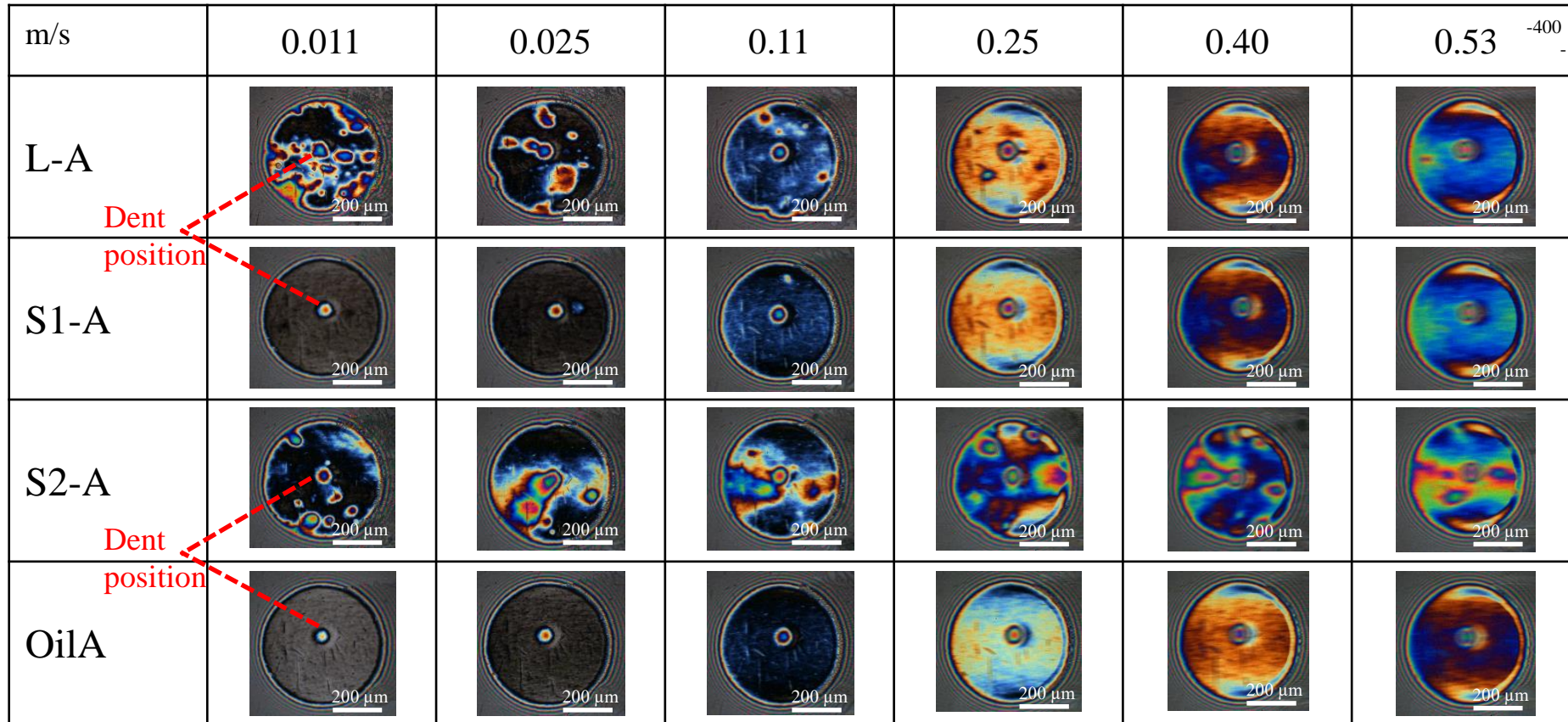
Asperity of hundreds of nanometer exists on the real bearing surfaces.



Influence of 3 types of dents on film thickness were investigated.

Fully flooded conditions -dented, sharp-

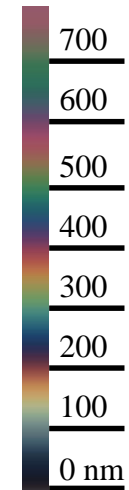
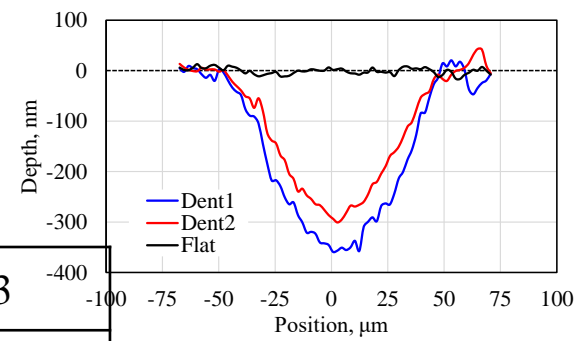
Acceleration dent1



Dent position

Dent position

Rolling direction →



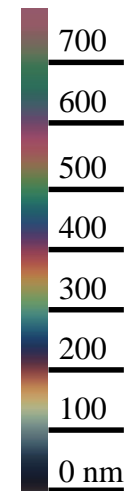
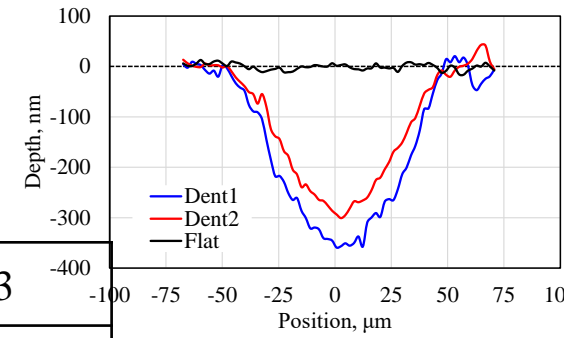
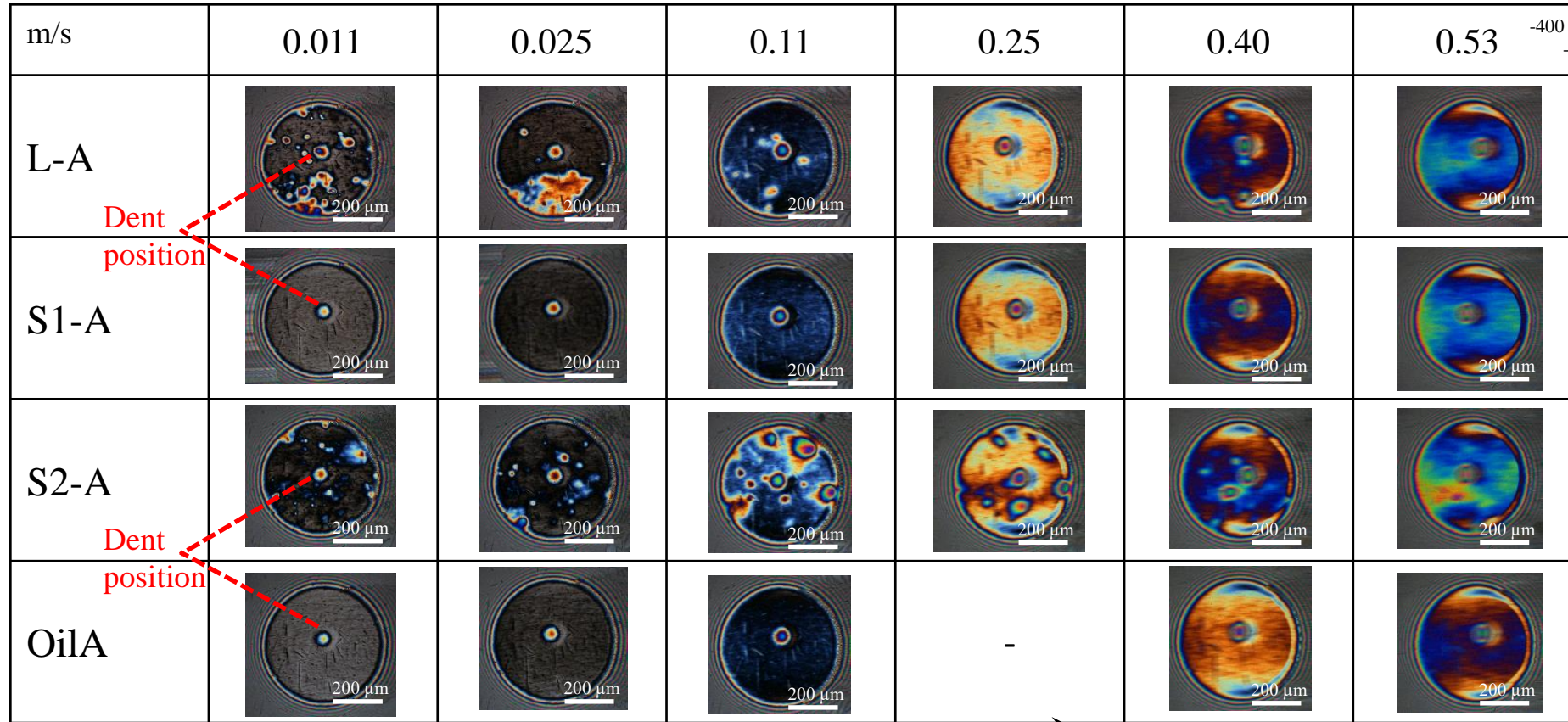
L-A: Thickener particles were entrained.

S1-A: Thickener particles were not entrained.

S2-A: Thickener particles were entrained and some particles accompanied with dents.

Fully flooded conditions -dented, sharp-

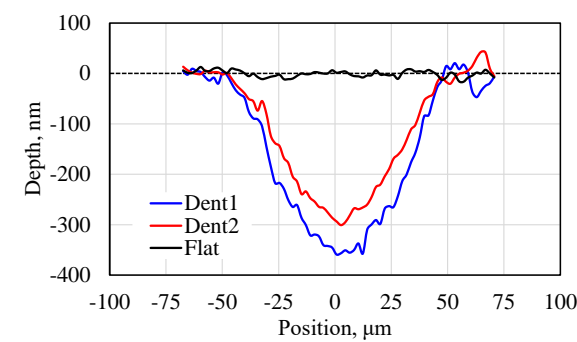
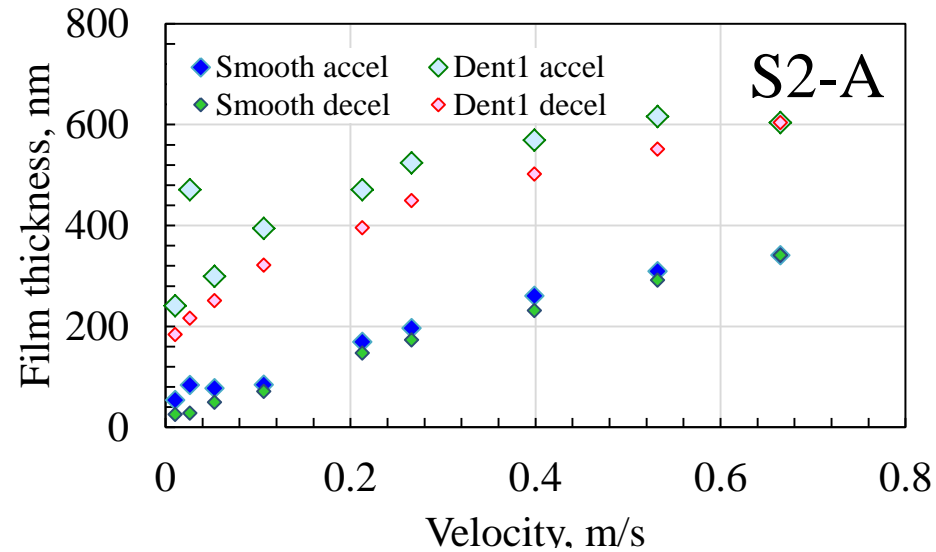
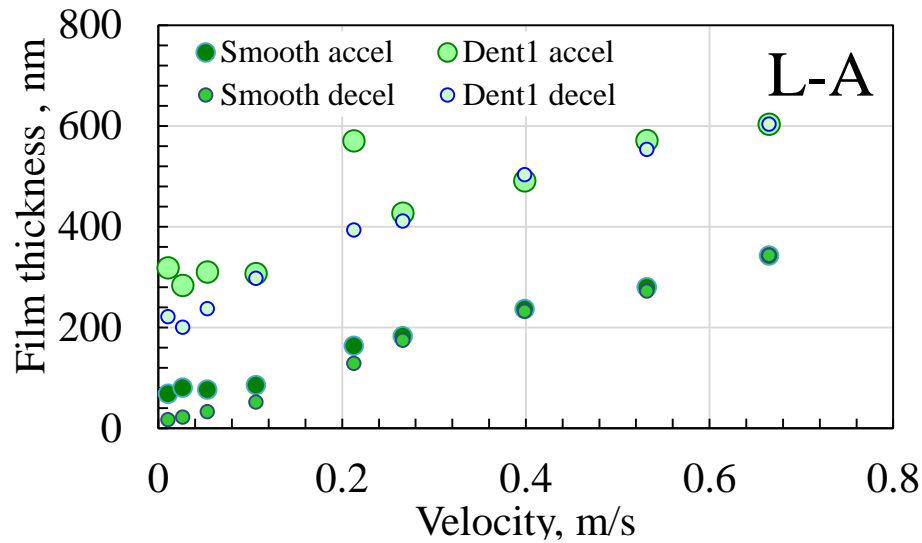
Deceleration dent1



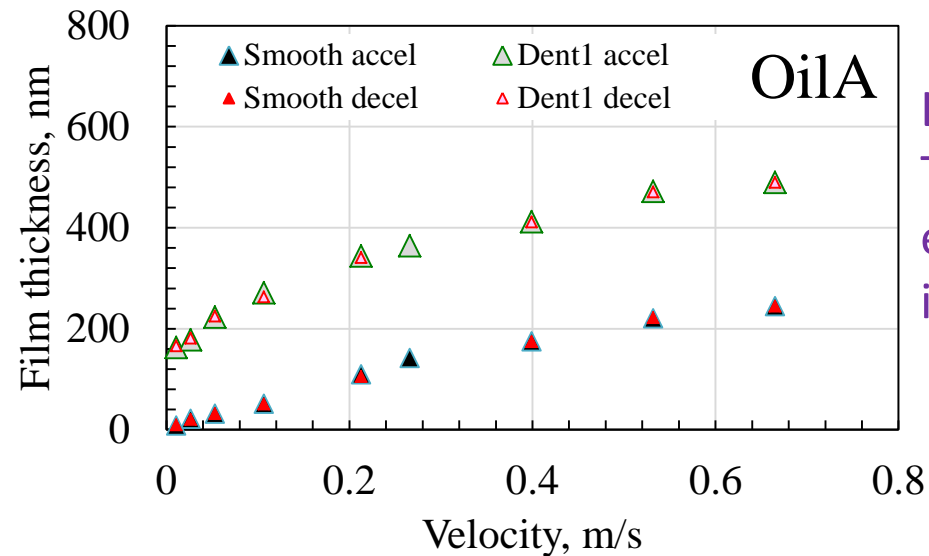
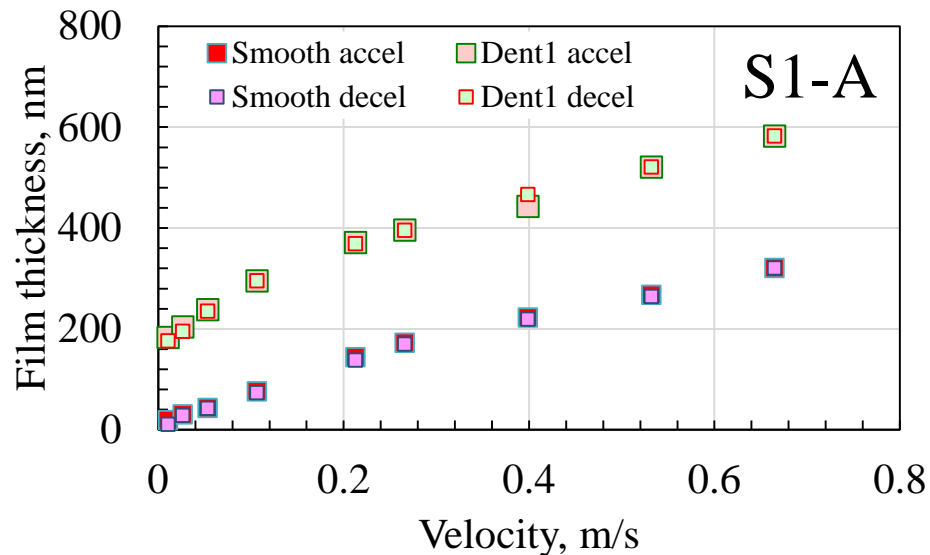
Rolling direction →

L-A, S2-A: Particle entrainments diminished.

Fully flooded conditions -dented, sharp-



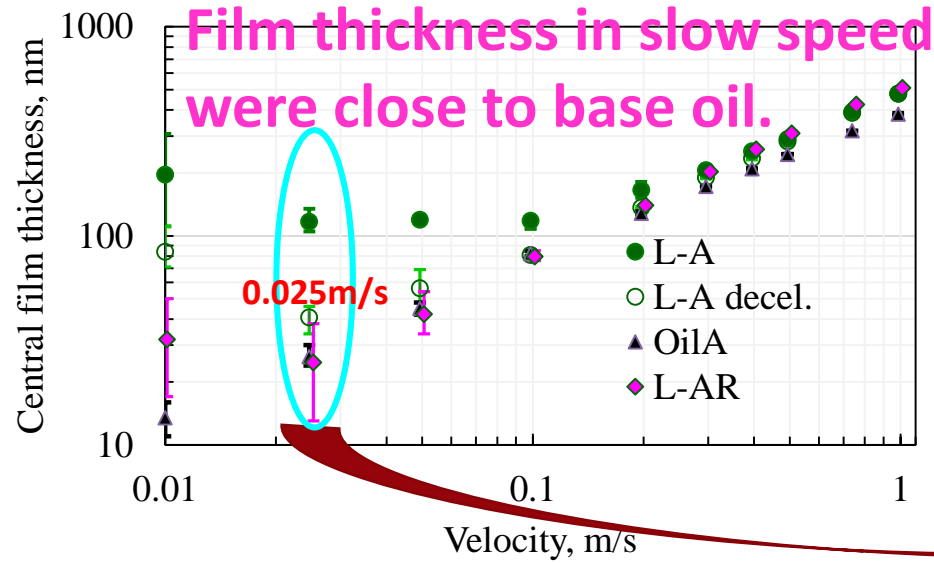
S2-A:
Thickener particle
entrainment was
more frequent.



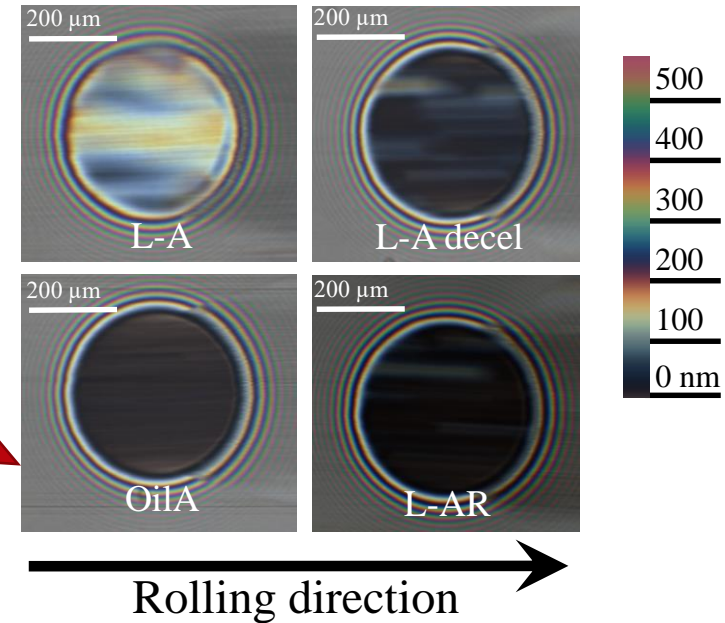
L-A, S2-A:
Thickener particle
entrainments diminished
in deceleration condition.

Fully flooded condition -Rolled grease, smooth-

L-AR: modified L-A rolled by ceramic roller in order to make finer particles



Interferograms



Sample		L-A	L-AR	OilA
Base oil	Mineral oil (G-I), %	88	88	100
Thickener	Li complex, %	12	12	-
	Li stearate, %	-	-	-
	Li-12OH-stearate, %	-	-	-
Penetration (60W)		265	250	-

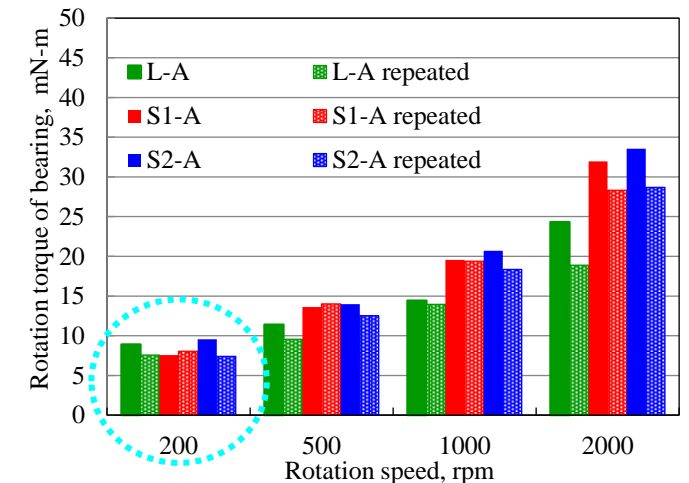
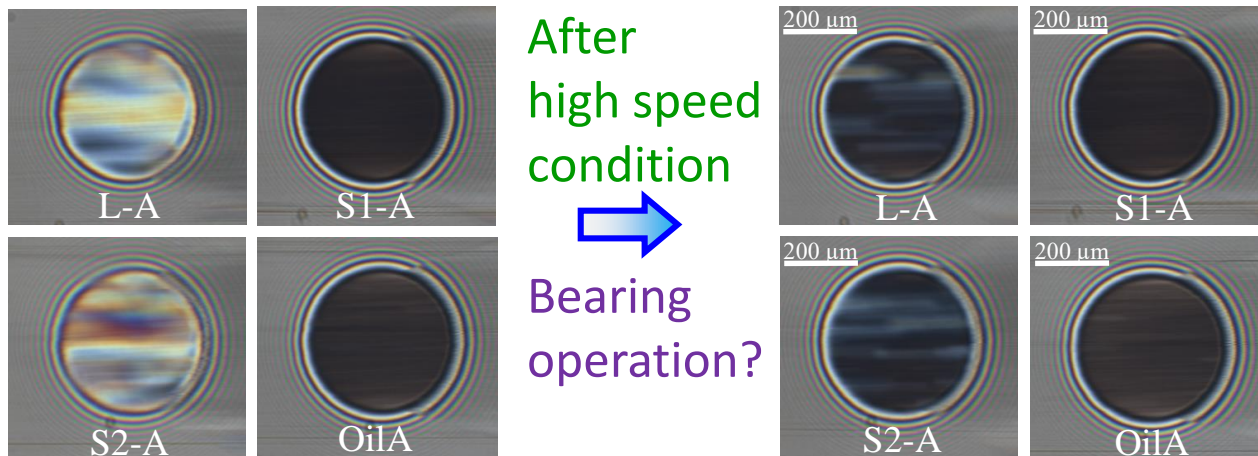
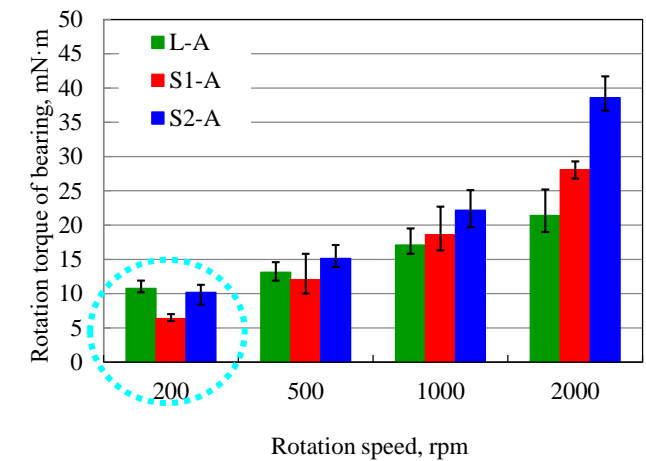
High film thickness of grease in slow speed was due to big thickener particles.

Discussion

L-A: Li-complex
S1-A: Single Li-Stearate
S2-A: Single Li-12OH-Stearate

Relation with bearing torque in low speed (200rpm)

- Film thickness in slow speed
 - **NOT** particle entrainments caused smooth rotation?
- 3 greases showed the same bearing torque in slow speed on repeated bearing tests
 - Suggestion of thickener particles breakdown



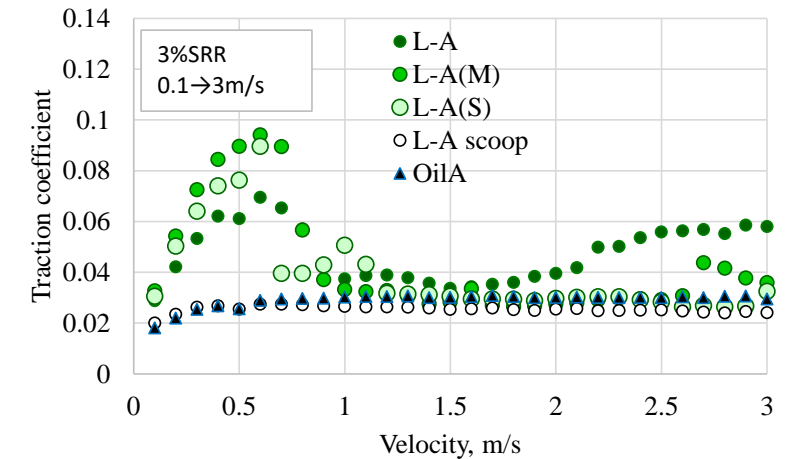
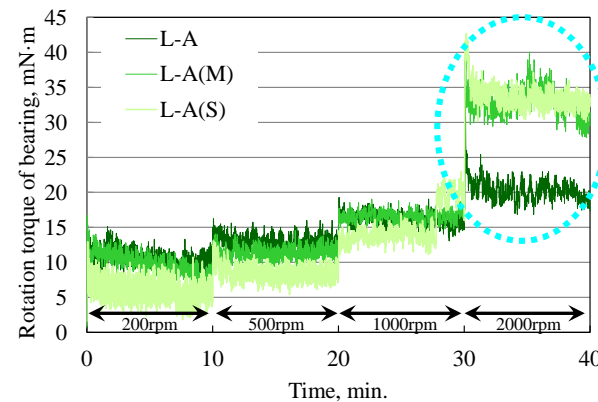
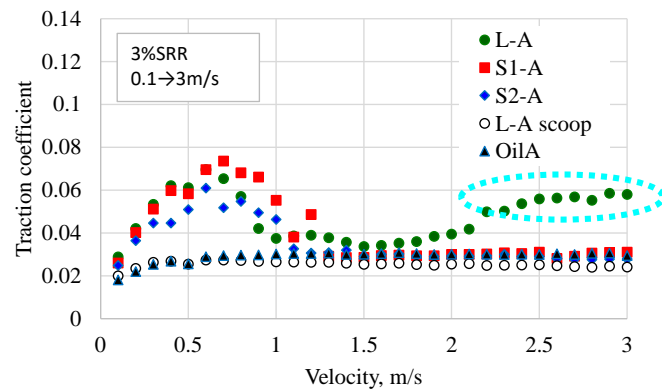
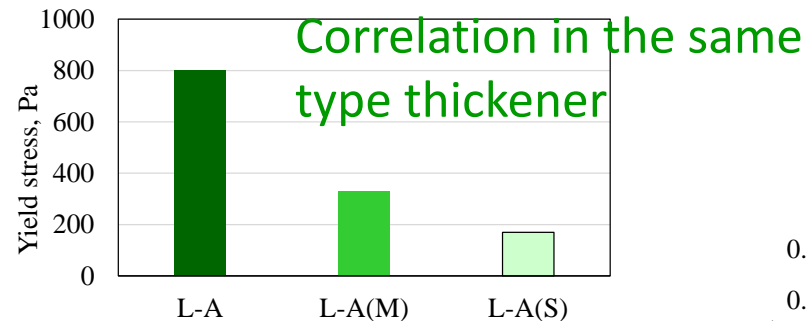
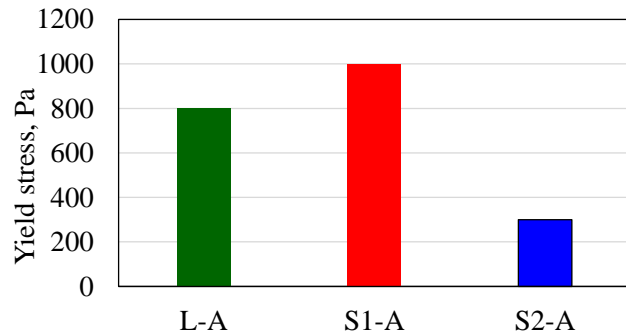
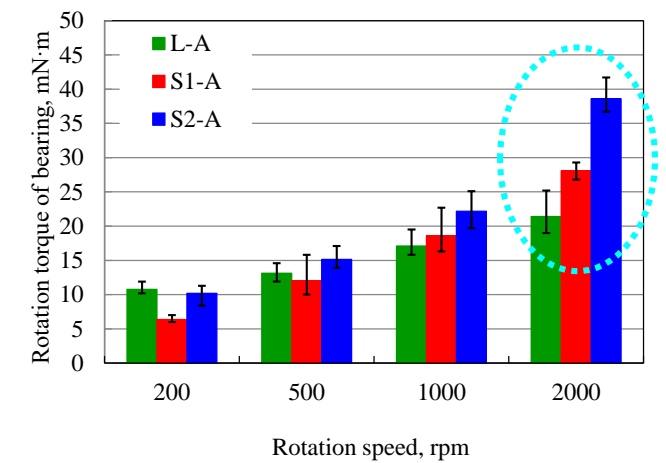
Discussion

L-A: Li-complex
S1-A: Single Li-Stearate
S2-A: Single Li-12OH-Stearate

Relation with bearing torque in high speed (2000rpm)

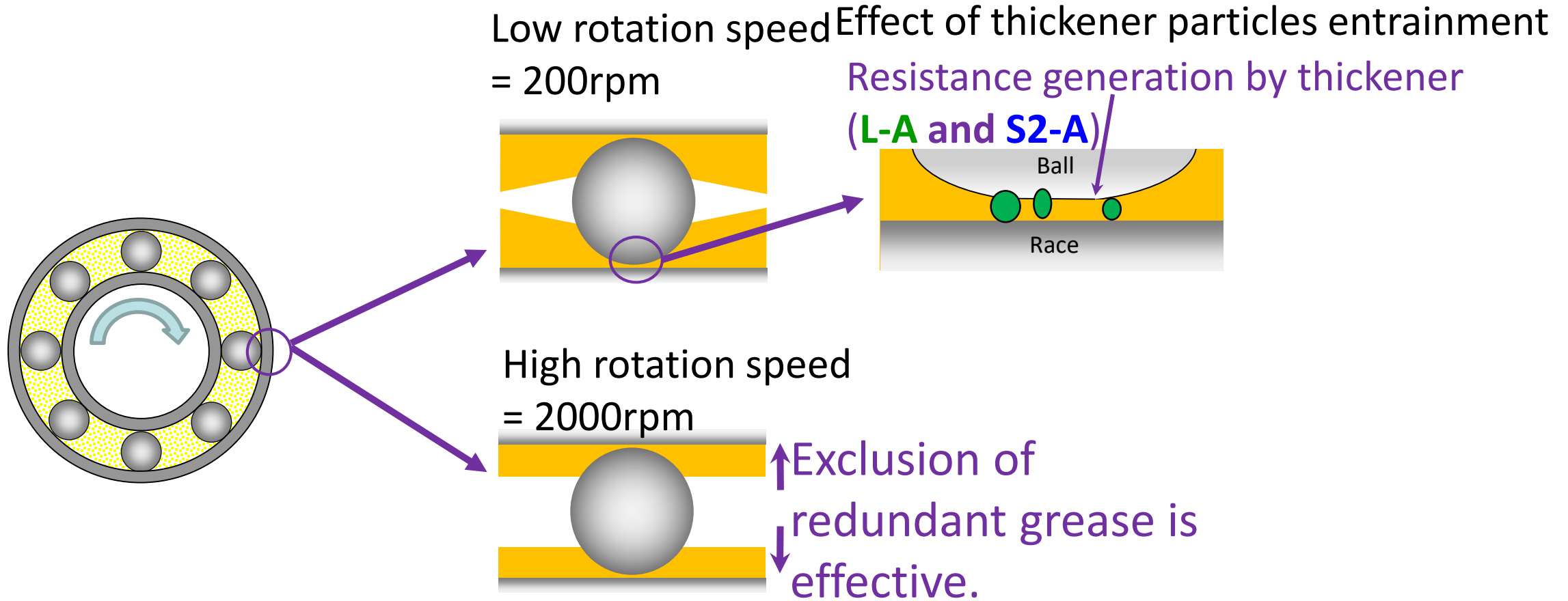
☉ Grease channeling should be related.

- **Not always** related to yield stress of greases, different from past report
- Traction coefficient behaviors without grease supply indicates grease channeling of Li-complex grease.



Discussion -mechanisms

L-A: Li-complex
S1-A: Single Li-Stearate
S2-A: Single Li-12OH-Stearate



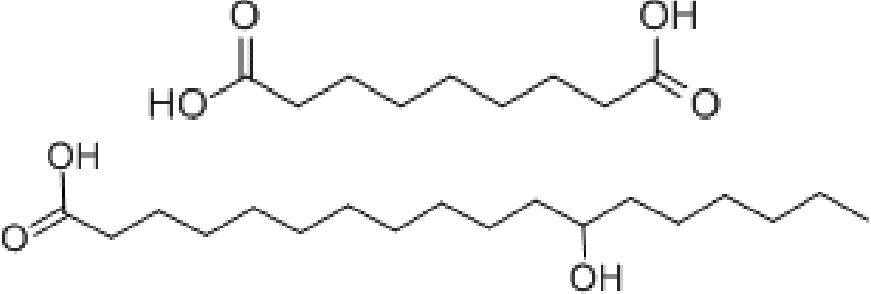
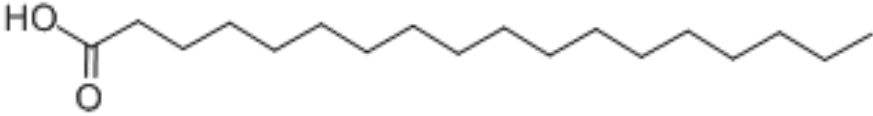
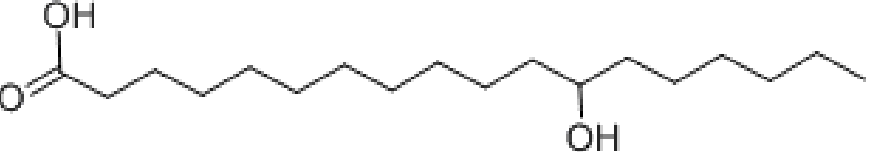
Discussion - Tendency of grease thickener type

L-A: Li-complex
 S1-A: Single Li-Stearate
 S2-A: Single Li-12OH-Stearate

Supply	Surface	Property	Li-complex	Li-12OH-stearate	Li stearate
Flooded	Smooth	Thickness	Thick in slow speed Influenced by high speed history		Close to base oil No change after high speed
		Flow pattern	Finger patterns on side track Sharpened by high speed history		No fingers in high speed
Starved		Film decay	Late starvation		Quick starvation
Flooded	Dented	Particle entrainment	Occurred	Frequently occurred	Not occurred

The cause of difference between L-A/S2-A and S1-A is on whether the thickener particles are dragged into contacts??

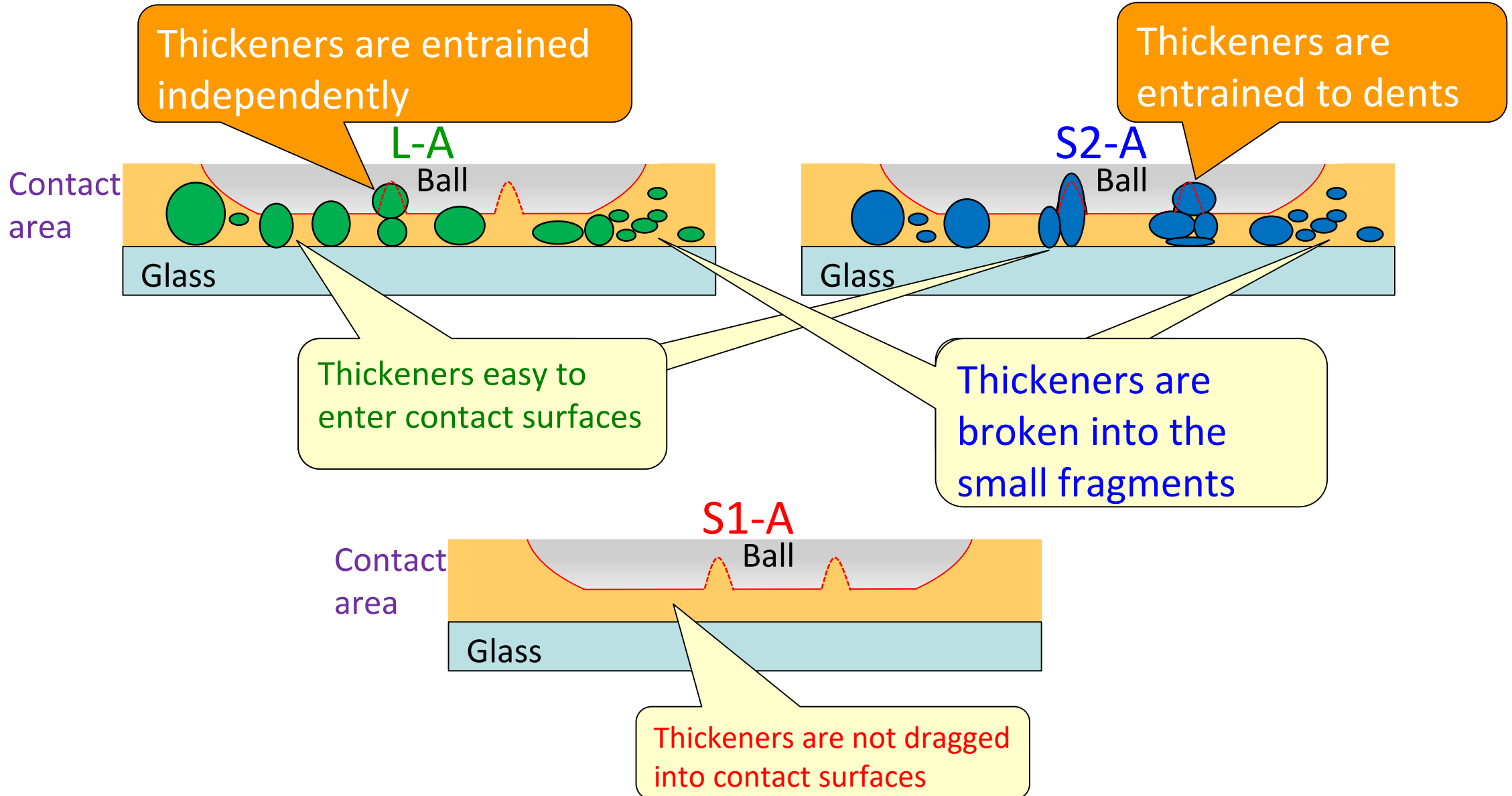
Chemical structures of thickeners

L-A: Li-complex	 <p>+ Li-OH</p>
S1-A: Single Li-Stearate	 <p>+ Li-OH</p>
S2-A: Single Li-12OH-Stearate	 <p>+ Li-OH</p>

(12-)Hydroxyl groups bonded to carbon chains could influence on particle entrainments

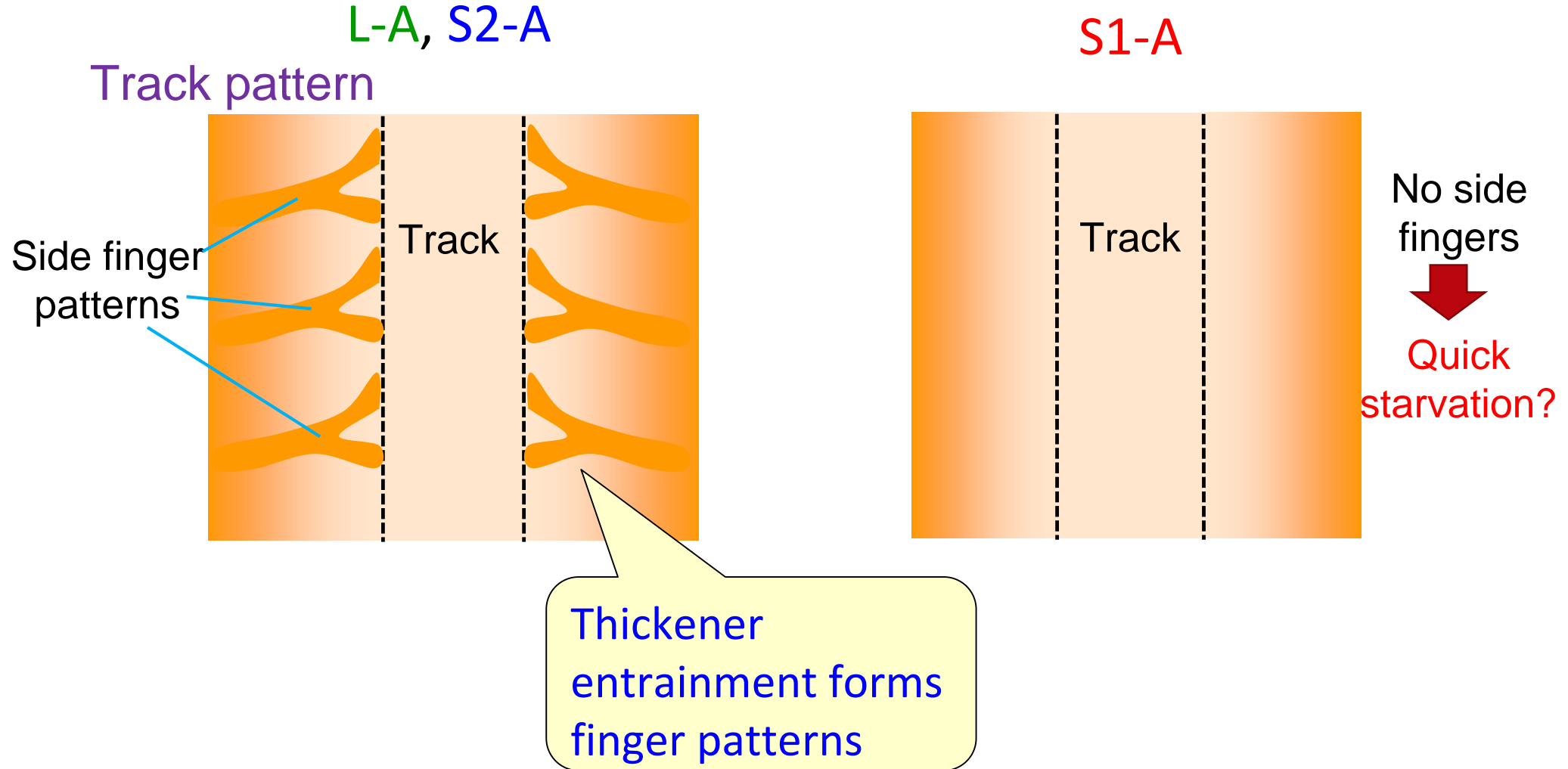
Discussion -mechanisms

L-A: Li-complex
S1-A: Single Li-Stearate
S2-A: Single Li-12OH-Stearate



Discussion -mechanisms

L-A: Li-complex
S1-A: Single Li-Stearate
S2-A: Single Li-12OH-Stearate



Conclusions

1. Relation with bearing torque at **low speed**

⇒ Thin **film thickness** without particle entrainment is effective in torque reduction

2. Relation with bearing torque at **high speed**

⇒ Grease channeling represented by **yield stress** and **traction** behaviors is effective

3. Thickener **entrainments** to the contacts

⇒ Film **thickness increase**, **track pattern** formation, **starvation delay**

4. Thickener entrainments to the **dents**

⇒ Promotion by thickener with **higher polarity**

List of publications

- SAKAI, K., D. KOSTAL, Y. SHITARA, M. KANETA, I. KRUPKA and M. HARTL. Influence of Li Grease Thickener Types on Film Thicknesses Formed between Smooth and Dented Surfaces. *Tribology Online*. 2017, **12**(5), 262-273.
- NITTA, M., T. TSUDA, H. ARAI, K. SAKAMOTO and K. SAKAI. Effects of Transition Point of Viscoelasticity of Diurea Grease and Molecular Structure of Thickener on Running Torque of the Ball Bearing -Effects of the Alkyl Chain Length of Aliphatic Diurea-. *Journal of Japanese Society of Tribologists*. 2016, **61**(10), 699-708 (in Japanese)
- SAKAI, K., Y. TOKUMO, Y. AYAME, Y. SHITARA, H. TANAKA and J. SUGIMURA. Effect of Formulation of Li Greases on Their Flow and Ball Bearing Torque. *Tribology Online*, 2016, **11**(2), 168-173. ⇒ITC Tokyo 2015 Young Researcher Paper Award
- SAKAI, K. and Y. SHITARA. Influence of Physical States of Amide Type Gel-Lubricants on their Tribological and Rheological Properties. *Tribologia: Finnish Journal of Tribology*. 2014, **32**(2), 20-28.
- SAKAI, K., Y. SHITARA, et al. Tribological Properties of Thermo-Reversible Gel-Lubricants Containing Solid Lubricants. *Tribology Online*, 2011, **6**(1), 26-31.

List of publications -conference abstracts-

- SAKAI, K., D. KOSTAL, Y. SHITARA, M. KANETA, I. KRUPKA and M. HARTL. Film Thickness Behaviors of Lithium Type Greases between Smooth and Dented Surfaces. The 18th Nordic Symposium on Tribology - NORDTRIB 2018. 2018, Uppsala, Sweden.
- SAKAI, K., D. KOSTAL, Y. SHITARA, M. KANETA, I. KRUPKA and M. HARTL. An Experimental Study on the Film Thickness and Grease Flows of Lithium Type Greases. 2nd Czech-Japan Tribology Workshop. 2017, Takamatsu, Japan.
- SAKAI, K., D. KOSTAL, Y. SHITARA, M. KANETA, I. KRUPKA and M. HARTL. The Experimental Study on the Relationship between Grease Film Thickness and Grease Flows. 6th European Conference on Tribology - ECOTRIB 2017. 2017, Ljubljana, Slovenia.
- SAKAI, K., D. KOSTAL, Y. SHITARA, M. KANETA, I. KRUPKA and M. HARTL. Effects of Li Grease Components on Radial Ball Bearing Torque and the Grease Properties. 72nd STLE Annual Meeting & Exhibition. 2017, Atlanta, USA.
- SAKAI, K., Y. TOKUMO, Y. AYAME, Y. SHITARA, H. TANAKA and J. SUGIMURA. Effect of Formulation of Li Greases on Their Flow and Ball Bearing Torque. 7th International Tribology Conference Tokyo 2015 -ITC Tokyo 2015. 2015, Tokyo, Japan.
- SAKAI, K. and Y. SHITARA. Influence of Physical States of Amide Type Gel-Lubricants on their Tribological and Rheological Properties. The 16th Nordic Symposium on Tribology - NORDTRIB 2014. 2014, Aarhus, Denmark.
- SAKAI, K., Y. SHITARA, et al. Tribological Properties of Thermo-Reversible Gel-Lubricants Containing Solid Lubricants. World Tribology Congress 2009 -WTC 2009. 2009, Kyoto,

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- Appreciation to prof. Krupka for supervisor of my PhD study and providing superior experimental test rigs
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- Special thanks to Dr. Kostal for arrangement of test rigs, discussion on results, assistance for submitting thesis.
- Many thanks to my colleagues at Institute of Machine and Industrial Design for friendly and positive atmosphere.
- Thousand thanks to prof. Kaneta for advice on overseas study, research discussion, daily life assistance, and many many things.
- Thanks to my colleagues in our company, JXTG Nippon Oil & Energy Corporation for giving chance for 2 years overseas study and covering my work for 2 years

Thank you for attention

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Institute of Machine
and Industrial Design