LUBRICANTS CHARACTERIZATION

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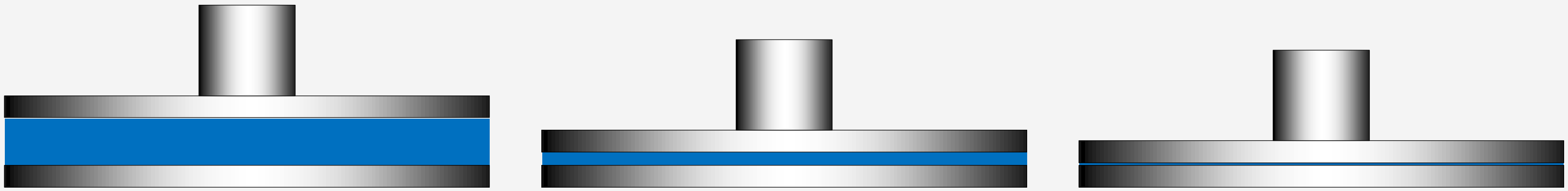
BASICS OF TRIBOLOGY

Rheology ↔ Tribology

Rheology **Tribology**



Bulk **material** properties

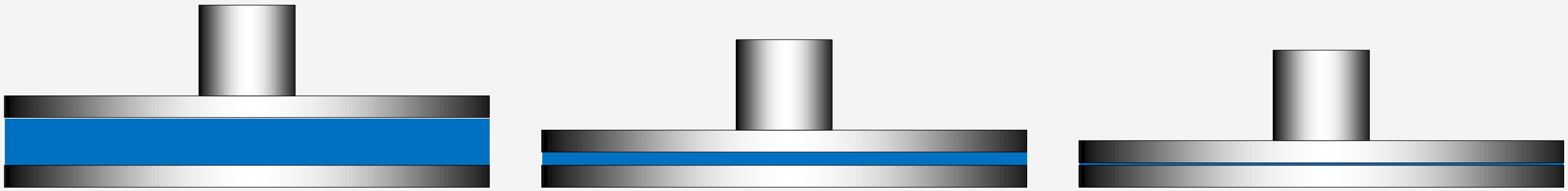
- *Inner friction*
- *Rheology characterizes material properties*

Properties of the **tribosystem**

- *Inter-surface friction*
- *Tribology characterizes system properties*

Rheology ↔ Tribology

Rheology **Tribology**



- The **Lubricant** is part of the **Tribosystem**.
- Hence, understanding of interdependency of rheological and tribological properties is crucial.

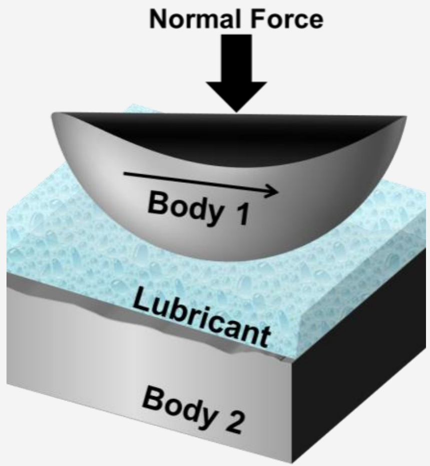
Terminology

Tribology

“Tribo(s)” + “logy”

(I rub) (Science)

… scientific study of friction, wear, and lubrication.



Tribosystem:

Two bodies (surfaces) in relative motion, with or without the presence of a medium separating them.

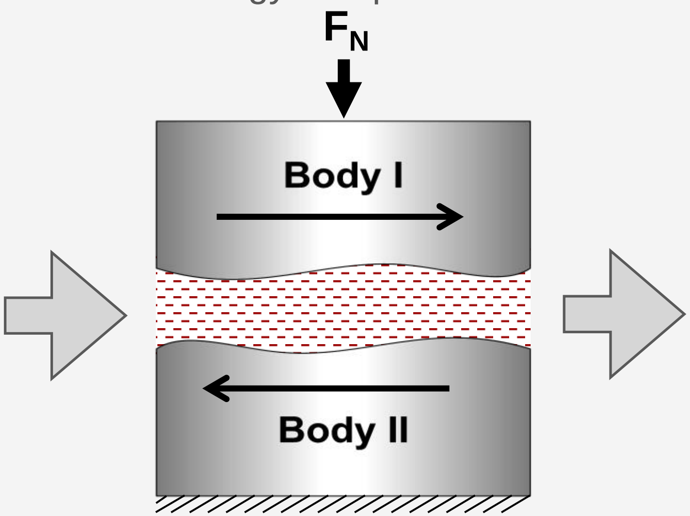
Influencing Factors

What happens…

- Frictional losses
- Surface alteration
- Tribochemical reactions
- Energy dissipation

What we set…

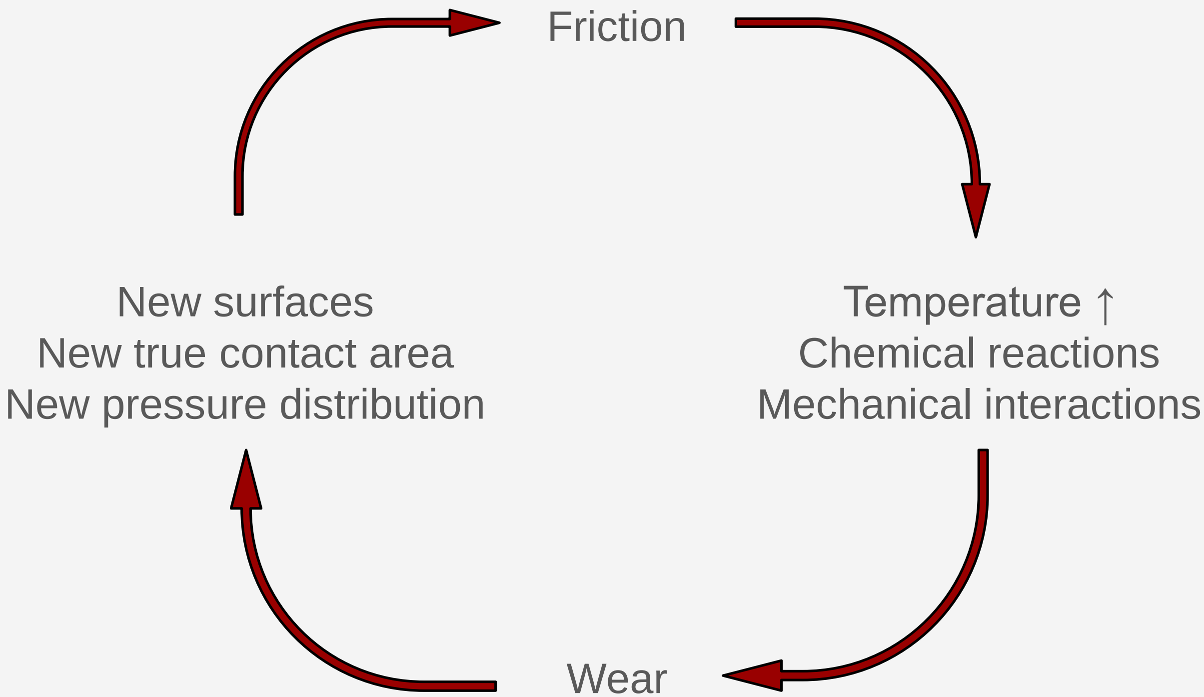
- Environmental conditions
  temperature, humidity, …
- Contact conditions
  pressure, type of contact, lubricated/dry, …
- Motion
  linear, rotation, oscillation, …



What we measure…

- Coefficient of friction
- Frictional torque
- Wear
  volume, height, rate, …
- Vibrations
- …

Cyclic Interactions



Dynamic nature of Tribology

Friction Factor

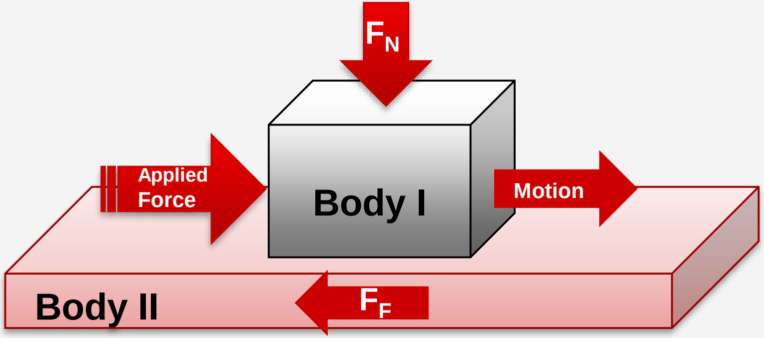
– The friction factor **μ** is the ratio of the force of friction between two bodies (Friction or Tangential Force $\mathbf{F_F}$) and the force pressing them together (Normal Force $\mathbf{F_N}$).

– μ is defined as

$$\mu = \frac{F_F}{F_N}$$

F_F → Friction Force

F_N → Normal Force

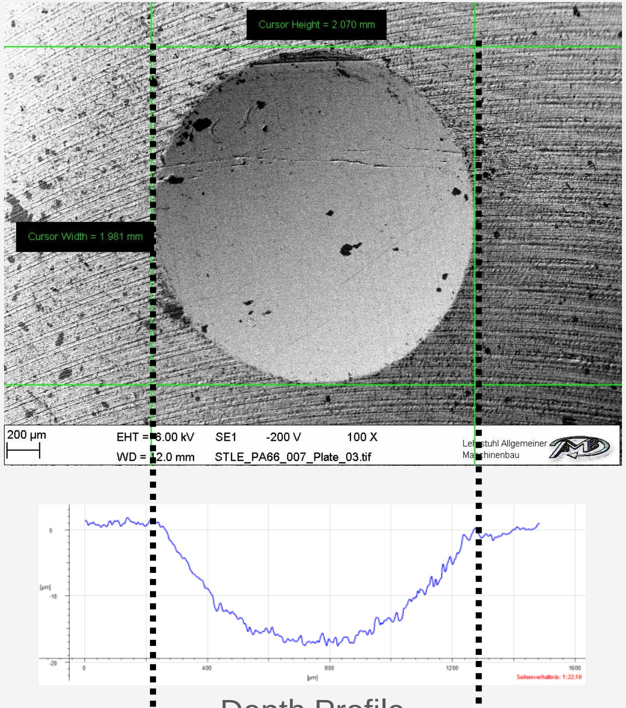


Wear

– Wear is progressive material removal or sideways displacement of material from its "derivative" and original position on a solid surface performed by the action of a solid, liquid or gaseous counter-body.

– Origins of wear:
  – Adhesion
  – Abrasion
  – Erosion
  – Fretting
  – Tribochemical reactions
  – …

Wear scar on a tested PA66 plate

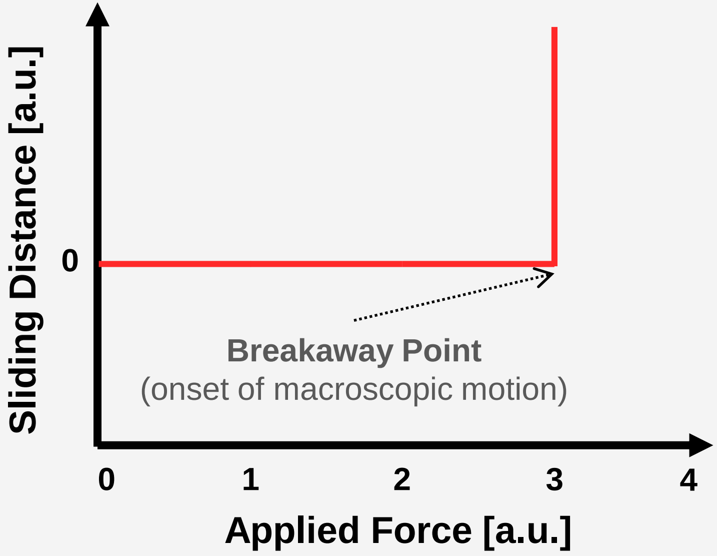


Depth Profile

Limiting Friction

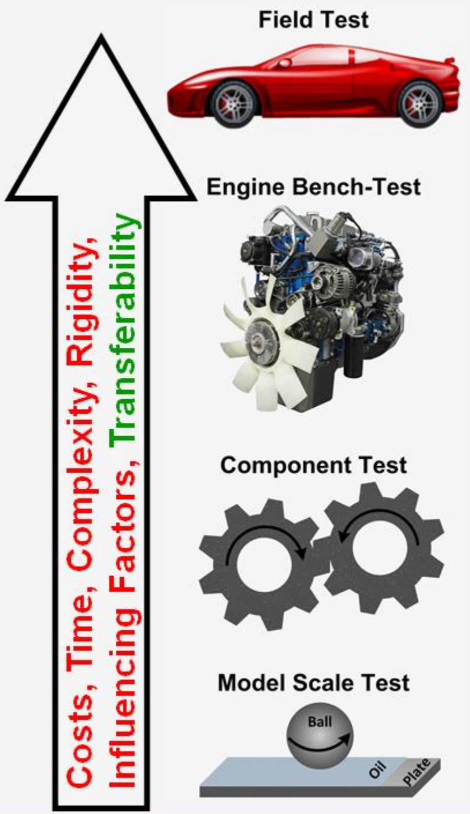
– The force inhibiting two surfaces at rest from sliding against each other is known as the limiting frictional force or breakaway force. In order to achieve macroscopic (sliding) motion between two surfaces, the force applied should overcome this limiting force.

– This limiting frictional force can be measured through a simple experiment, wherein the applied force is gradually increased and the corresponding sliding distance is measured. As long as the applied force is lower than the limiting force, there will be no macroscopic motion. The breakaway point indicates the onset of motion and applied force at this point is the breakaway force of the system. Likewise, the corresponding friction coefficient is the limiting friction of the system.



Model Scale Testing

- Most tests carried out on tribometers are at model scale.
- It is necessary to choose the appropriate geometry that closely represents the real-life application.
- This is in terms of the type of contact, speed, contact pressure, temperature, etc.
- If an existing system (geometry) does not suit the client's application, there is always a possibility to customize the holder or the specimen.



TYPICAL MEASUREMENTS

Stribeck Curve

Why do we need Stribeck curves?

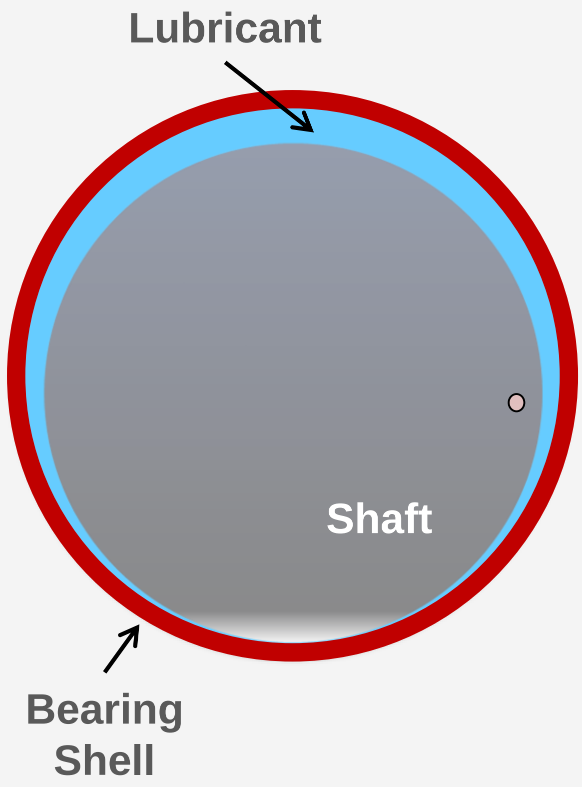
- Stribeck curves have long been used for understanding the lubricating behavior of oils and greases in journal bearings.
- In the recent years, the range of applications increased. Nowadays, Stribeck curves are also applied to ball point inks, food and beverages, cosmetics or synovial fluids.

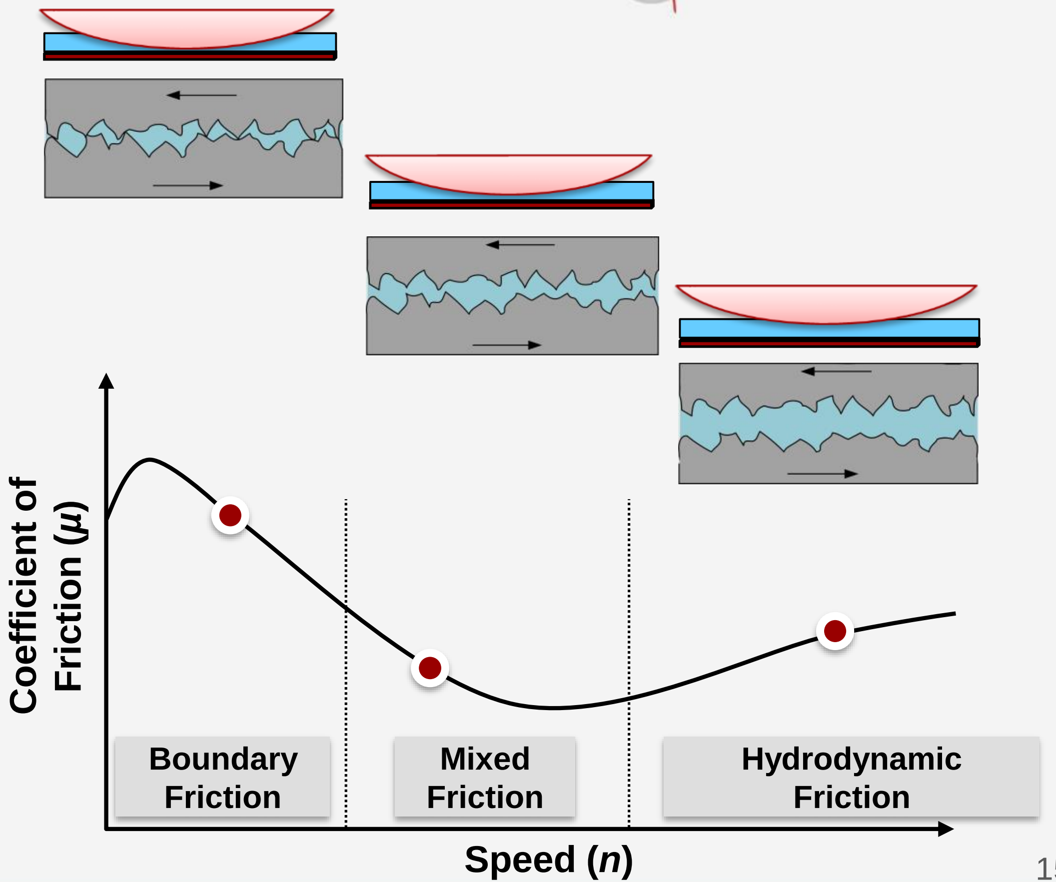
- Stribeck curves are being used to describe the frictional behavior of lubricated tribosystems. A typical Stribeck curve shows how the friction evolves as a function of speed. Roughly spoken, Stribeck curves can be divided in three regimes with their own characteristics:

  - Boundary friction → frictional behavior predominantly influenced by surface interactions
  - Mixed friction → transition from boundary to hydrodynamic friction with increasing speed
  - Hydrodynamic friction → frictional behavior predominantly influenced by lubricant viscosity

- An example Stribeck curve is shown on the following slide.

Stribeck Curve





Stribeck Curve: Friction/Lubrication Regimes

Boundary Friction

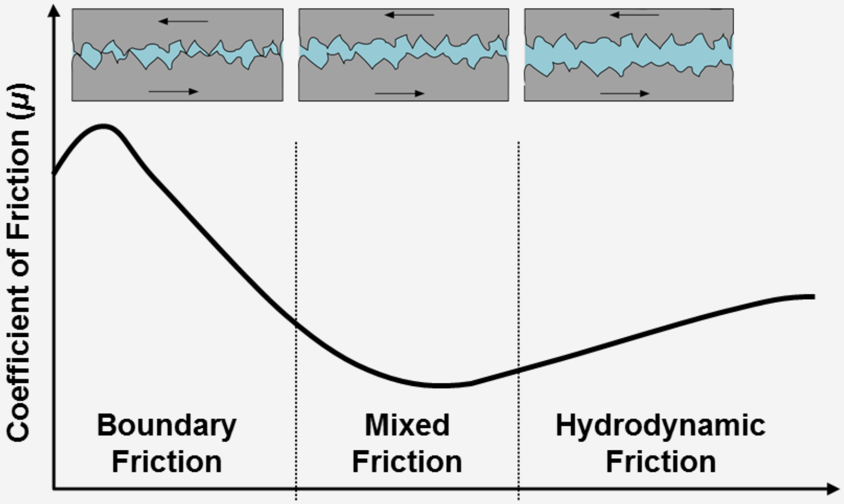
- No fluid/lubricant film
- Asperity (body/body) contact
- High friction and wear…

Mixed Friction

- Lubricant film just thick enough to separate the surfaces
- Asperities come in contact occasionally
- Low friction and wear …

Hydrodynamic Friction

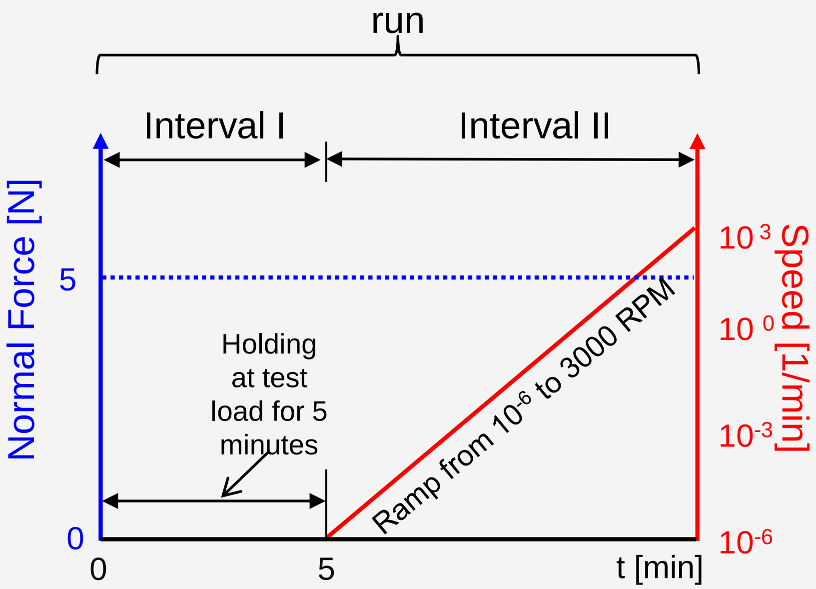
- Lubricant film totally separates the surfaces
- No asperity contact
- Friction only due to viscosity
- No wear*…



* while there is absence of wear due to asperity contact, other types of wear such as erosion, cavitation, etc. can still occur.

Extended Stribeck Curve

Test procedure



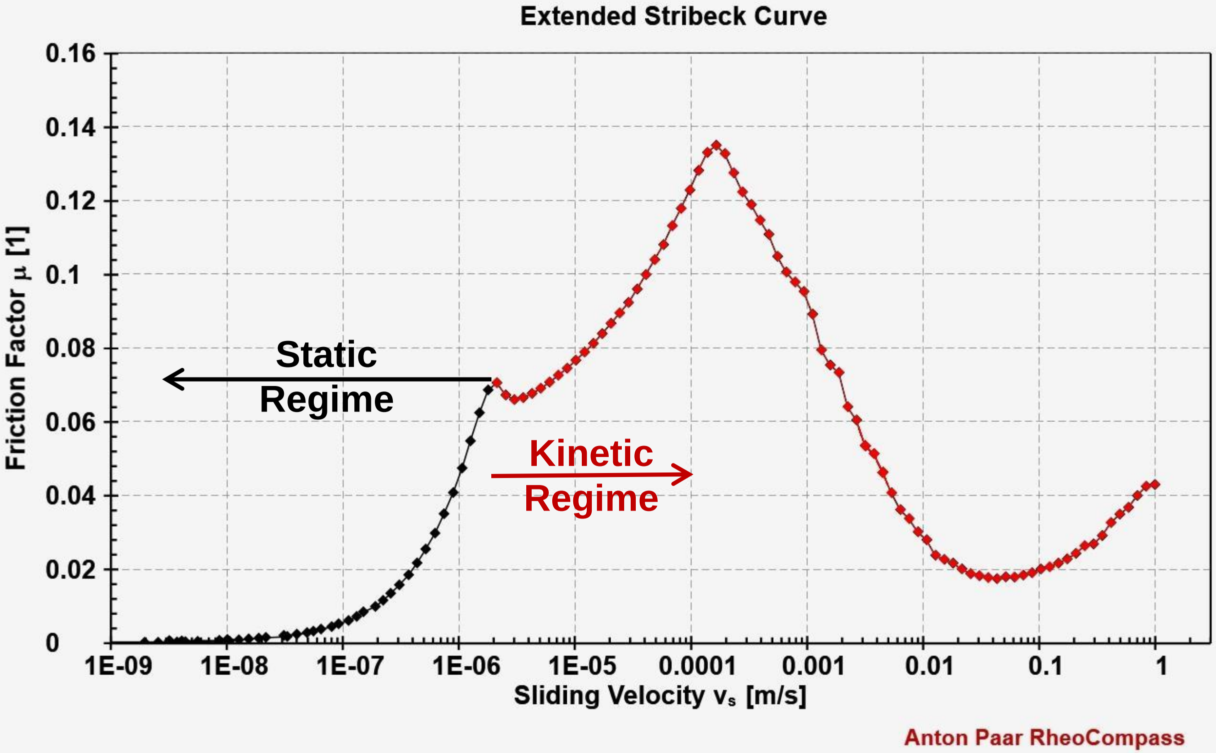
One test consists of two intervals:

- **Interval I**: The system is allowed to relax at the freshly applied load.
- **Interval II**: The sliding velocity respectively the speed is increased logarithmically.

The temperature and the normal fore are maintained constant. Speed, temperature and fore are chosen according to the respective application.

The values of test parameters indicated here are only notional and may change depending upon the application.

Extended Stribeck Curve



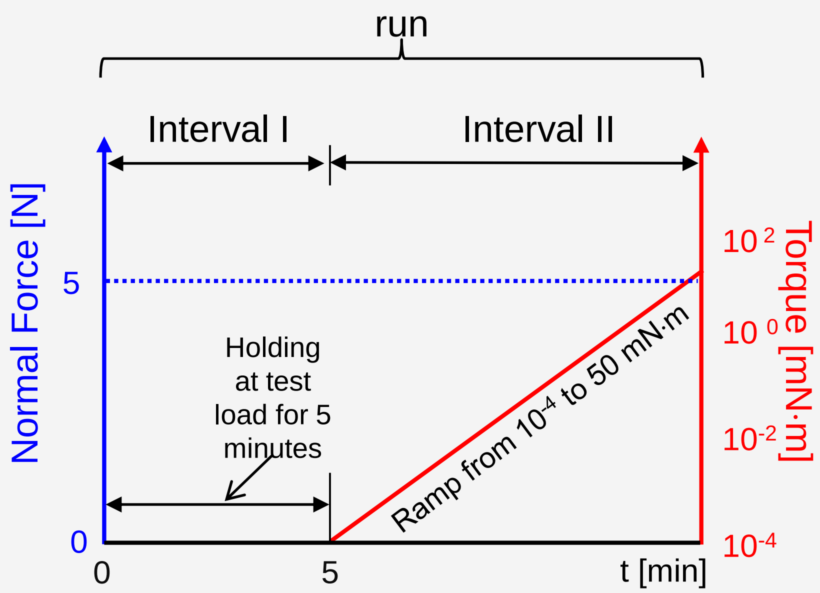
Extended Stribeck curves describe the frictional behavior of the system in:

- the **static** regime
- the **kinetic** regime

They also help in identifying the transition point between the **static** and the **kinetic** regime.

Breakaway Torque

Test procedure



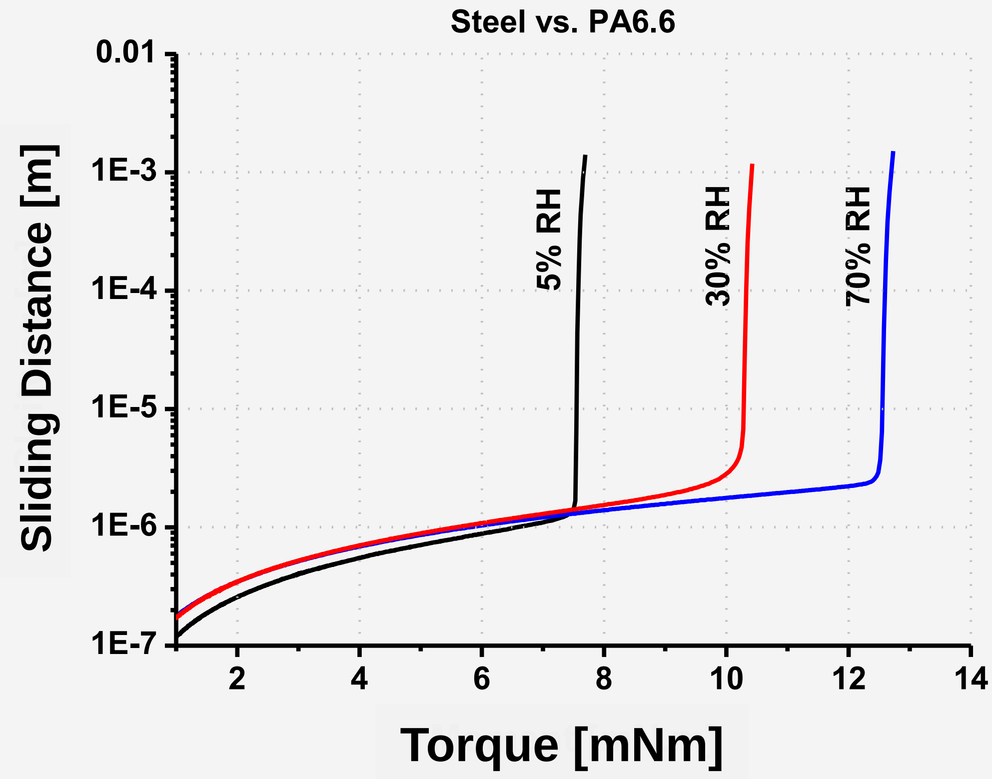
One test consists of two intervals:

– **Interval I**: The system is allowed to relax at the freshly applied load.
– **Interval II**: The torque is increased logarithmically.

The temperature and the normal fore are maintained constant. Torque, temperature and normal fore are chosen according to the respective application.

The values of test parameters indicated here are only notional and may change depending upon the application.

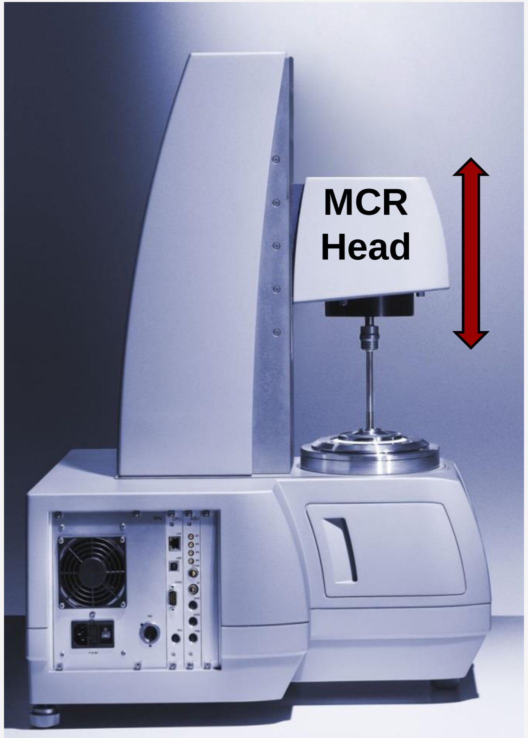
Breakaway Torque



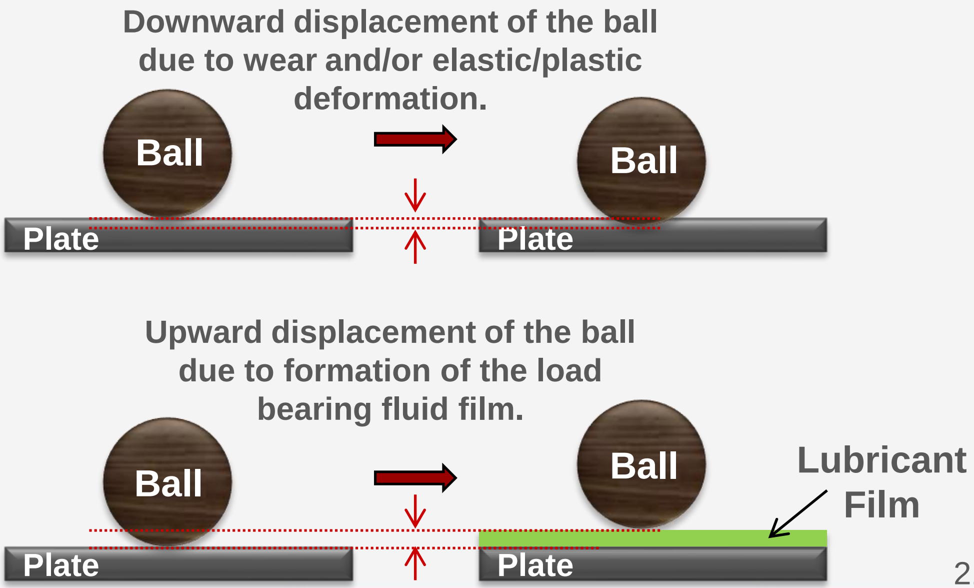
- Real life systems tend to deform elastically/plastically prior to the onset of motion.
- The measurement is carried out by logarithmically increasing the torque and measure the corresponding deflection.
- The breakaway point indicates the onset of macroscopic motion and applied force at this point is the **breakaway force/torque** of the system.
- Additionally, it is also possible to determine the influence of humidity on the breakaway force with the help of the Humidity Cell option.
- With increasing humidity (**5 %**, **30 %**, **70 %**), the breakaway torque increases. The hydrophilic nature of PA66 allows it to adsorb water and this has a significant influence on its tribological behavior.

Gap Measurement

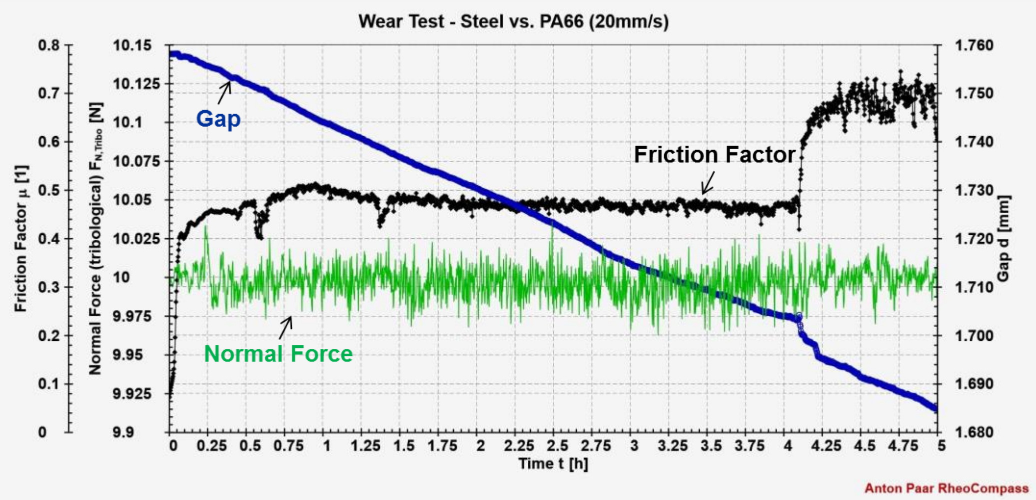
The movement of the head (upward or downward) is precisely controlled by the stepper motor with an accuracy of ~ 0.65 µm. This movement is responsible for the application of set normal force at the contact interface.



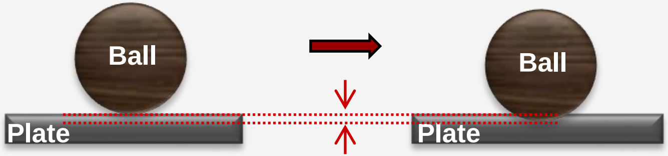
Head movement controlled with an accuracy of ~ 0.65 µm.

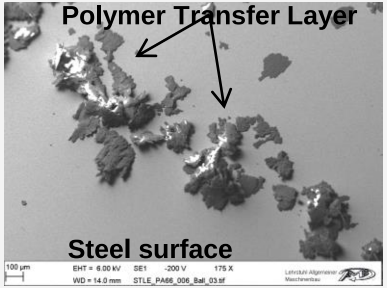


Wear Measurement and Gap Measurement



Downward displacement of the ball.





- Measurements at constant sliding velocity can be carried out to evaluate how the tribological behavior changes over time, e.g. due to wear.
- A downward displacement of the upper measuring system can be indicated by a decreasing “gap” value. This can allow for information on wear and/or plastic deformation of the specimen.
- Example measurement with steel/PA tribopair:
  - After 4 hours of testing, there was enough wear debris (polymer) at the contact to form a thin transfer layer on the surface of the steel ball.
  - At this point, the contact is between the transfer layer and the polymer, which resulted in higher frictional resistance (like vs. like).
  - Higher friction leads to greater wear (adhesive wear) and, hence, a more pronounced decrease in the gap values.

APPLICATIONS

Applications

Automotive

- Gears
- Clutches
- Bearings …

Polymer Tribology

- Coatings
- Composites …

Biotribology

- Dental tribology
- Skin
- Implants, cartilage …

Food Tribology

- Beverages
- Chocolate, cheese

Nanotribology

- Hard discs
- Biomimetics …








Anton Paar

Applications

AUTOMOTIVE

Applications: Automotive

- This section deals with some of the tribological needs in the transportation sector comprising of cars, trucks, ships, planes, etc.
- Like most other applications, the major concern in this sector is reduction in friction (in most cases) and wear.
- This is where lubricants and new materials and surface treatments become relevant.
- The following set of slides in this section show as to how well the MCR Tribometer can be used to characterize tribological properties of lubricants and greases.
- While the MCR Tribometers are not employed to test materials (solids) under dry conditions, friction and wear behavior of surface treatments or surface coatings under lubricated conditions can be tested using this instrument.

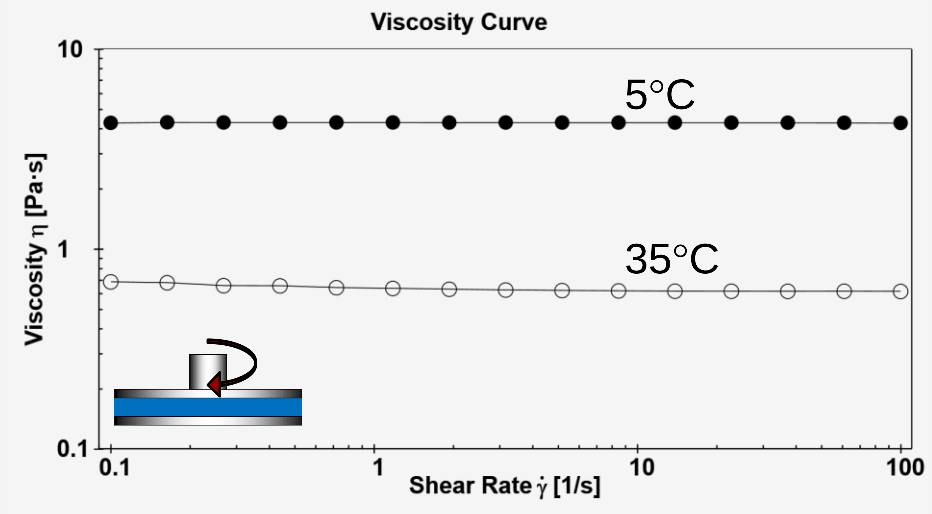
Engine Oil: Effect of Viscosity

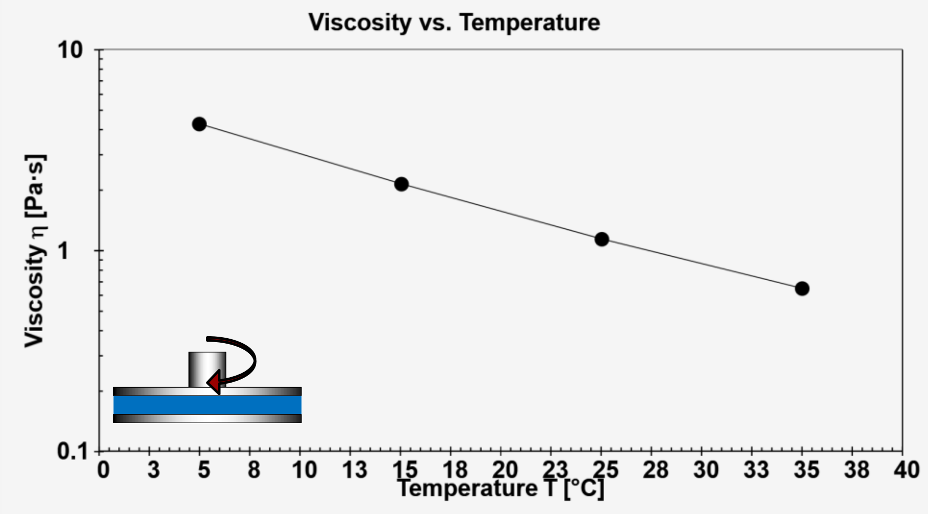
- Sample
  - lube oil at different temperatures
- Specimen
  - 100Cr6 ball
  - 100Cr6 pins

The aim here is to study the effect of viscosity on the friction behavior of lubricants.

100Cr6 or AISI52100 is standard bearing steel used for testing engine oils

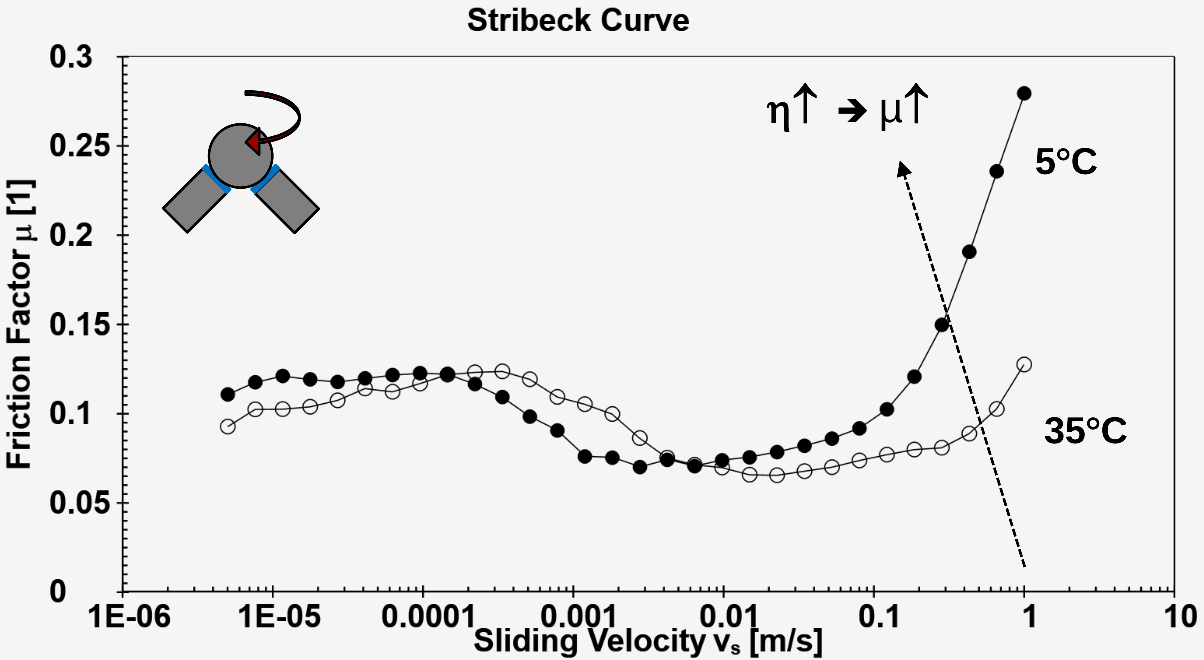
Oil Viscosity Curves

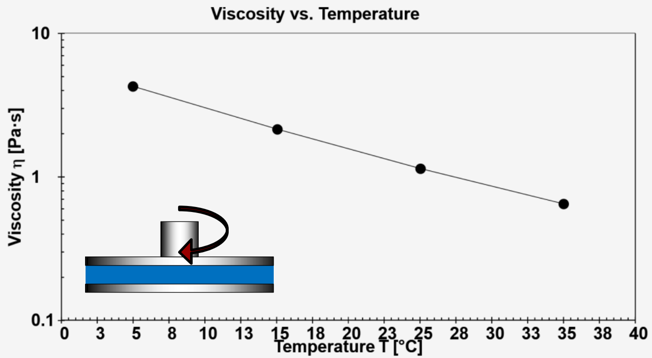




- The **Oil** shows Newtonian flow behavior (no effect of shear rate on the viscosity).
- The oil viscosity drops from around 4 Pas at 5 °C to about 0.6 Pas at 35 °C.

Stribeck Curves with Oil Samples





– Increase in viscosity leads to an increase in friction in the hydrodynamic regime.

– With increasing viscosity, transition into hydrodynamic regime occurs at a smaller sliding velocity.

Applications

AUTOMOTIVE (ENGINE OILS)

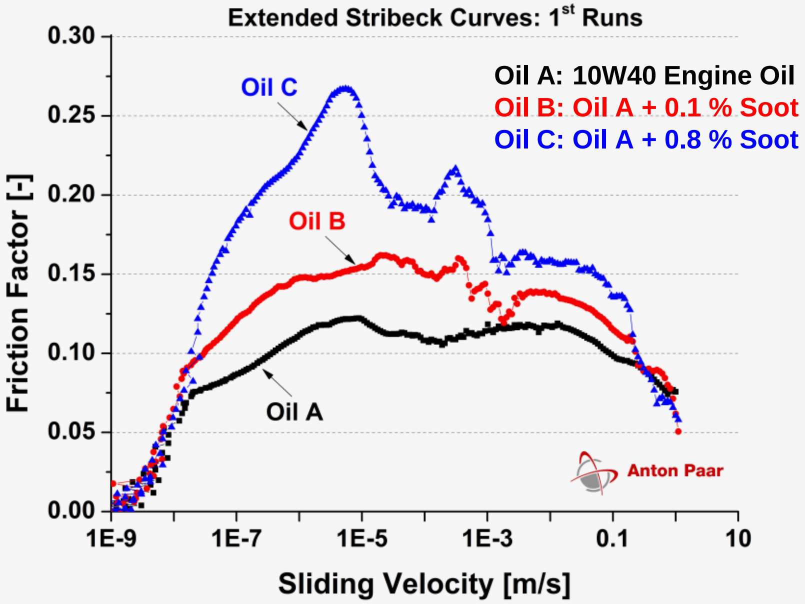
Effect of Soot on Engine Oil Performance

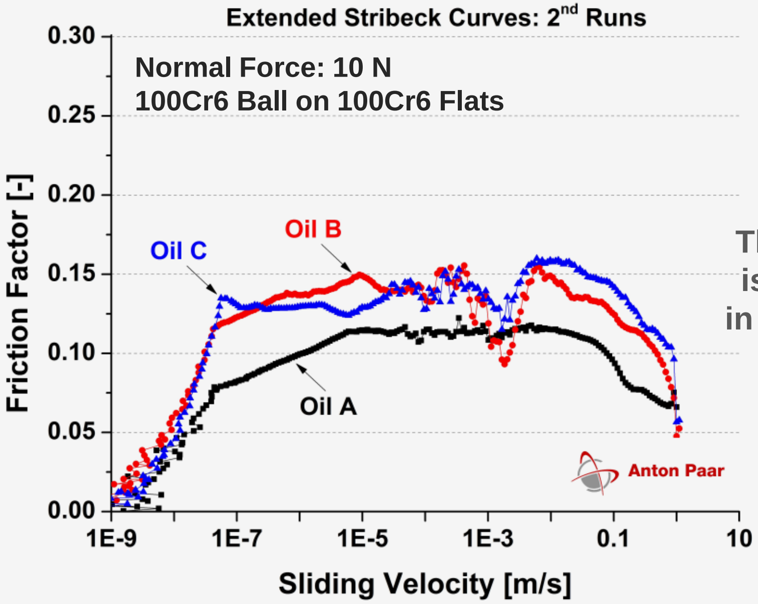
What happens when soot gets introduced into the engine oil during its service life?

– The efficiency of additives in the engine oil decreases.
– Lifespan of components is affected due to increased wear.
– Ultimately, it leads to wasted resources (time, money, and natural resources).

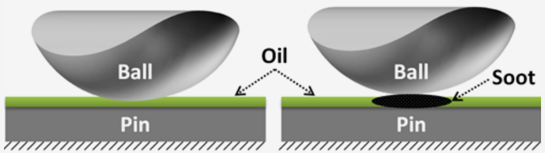
It is, therefore, important to understand the mechanism and the extent of damage soot can have on different engine components.

Soot (I)



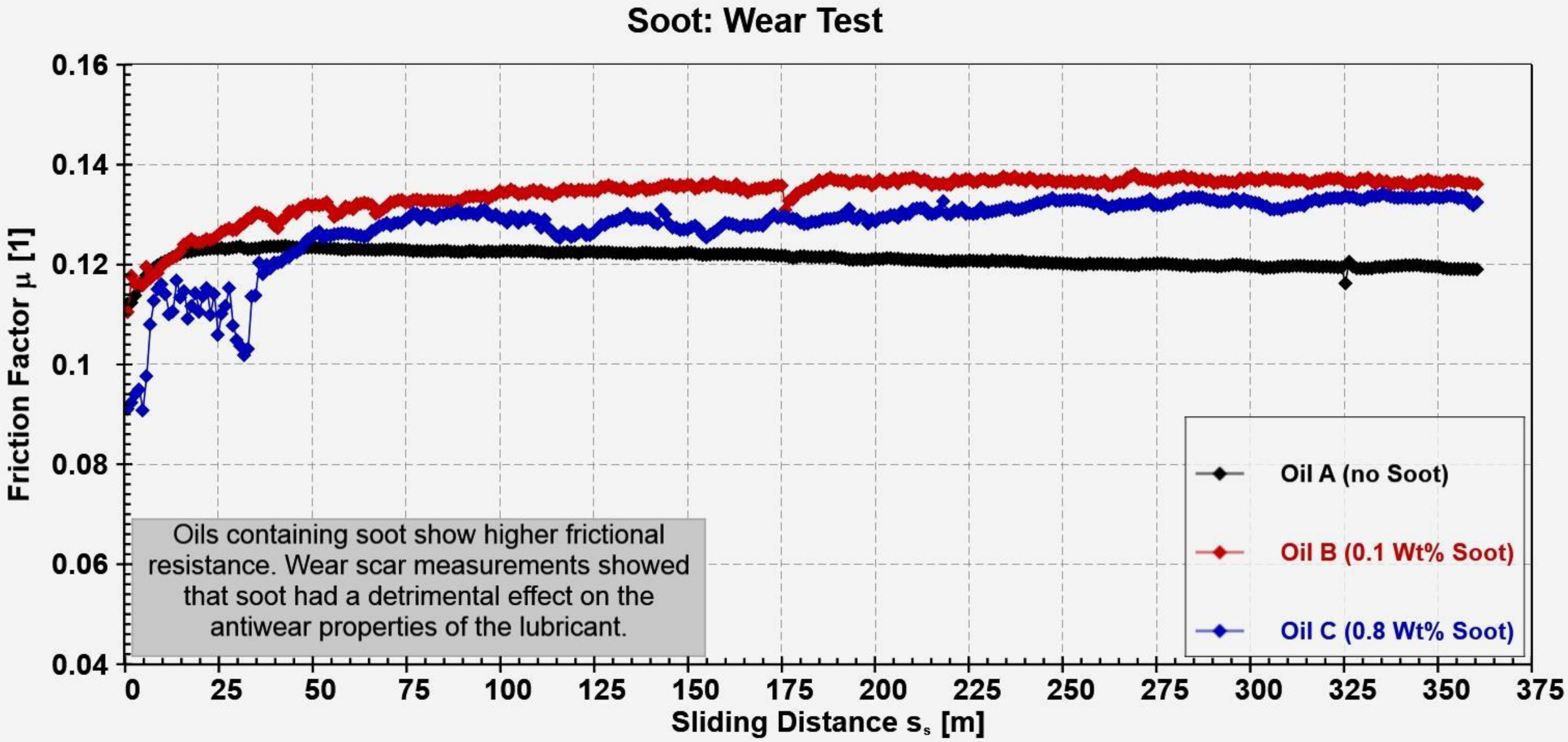


The effect of soot is already visible in the 1st run of the tests

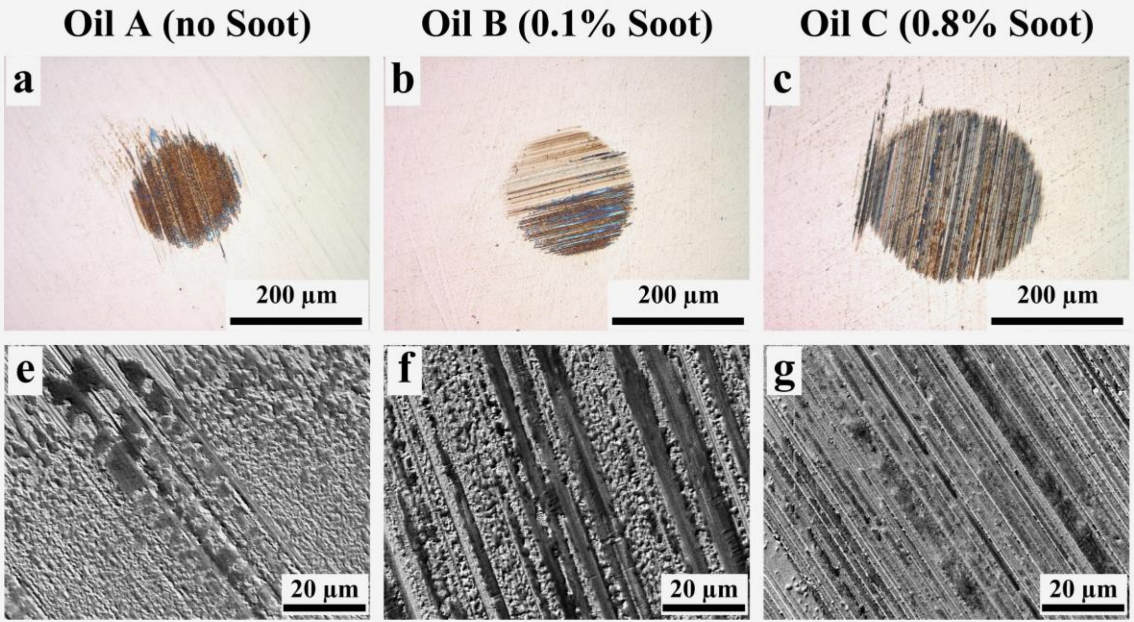


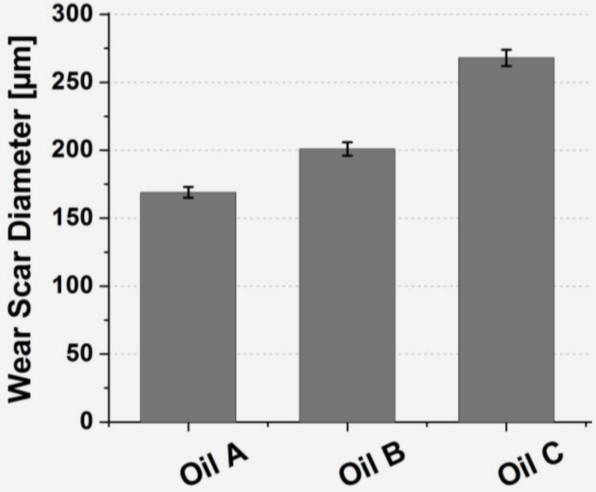
Soot particles at the contact interface are harder to shear, as compared to oil, which would explain the higher frictional resistance for oils with soot.

Soot (II)


Soot: Wear Test
Friction Factor μ [1]
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0
25
50
75
100
125
150
175
200
225
250
275
300
325
350
375
Sliding Distance s_s [m]
Oils containing soot show higher frictional resistance. Wear scar measurements showed that soot had a detrimental effect on the antiwear properties of the lubricant.
Oil A (no Soot)
Oil B (0.1 Wt% Soot)
Oil C (0.8 Wt% Soot)

Soot (III)



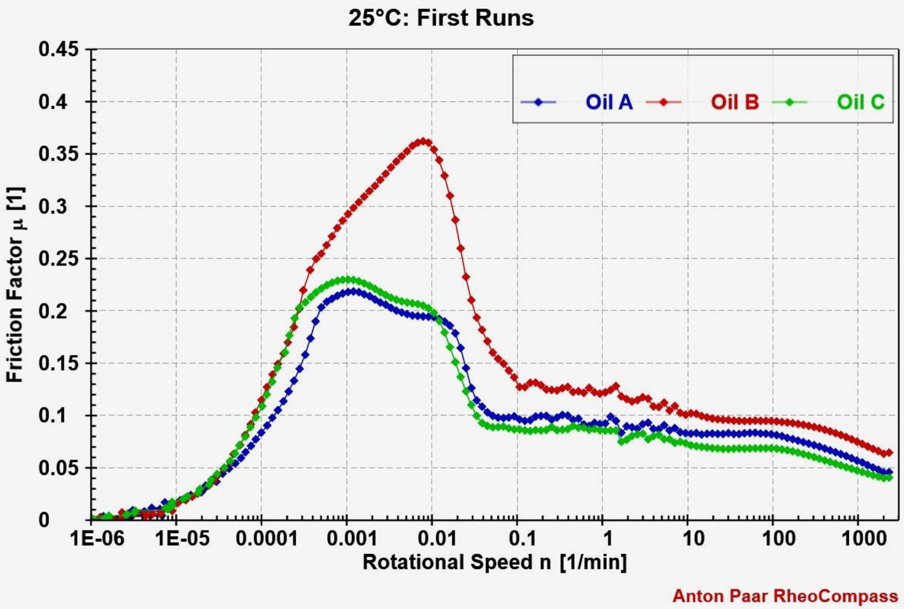


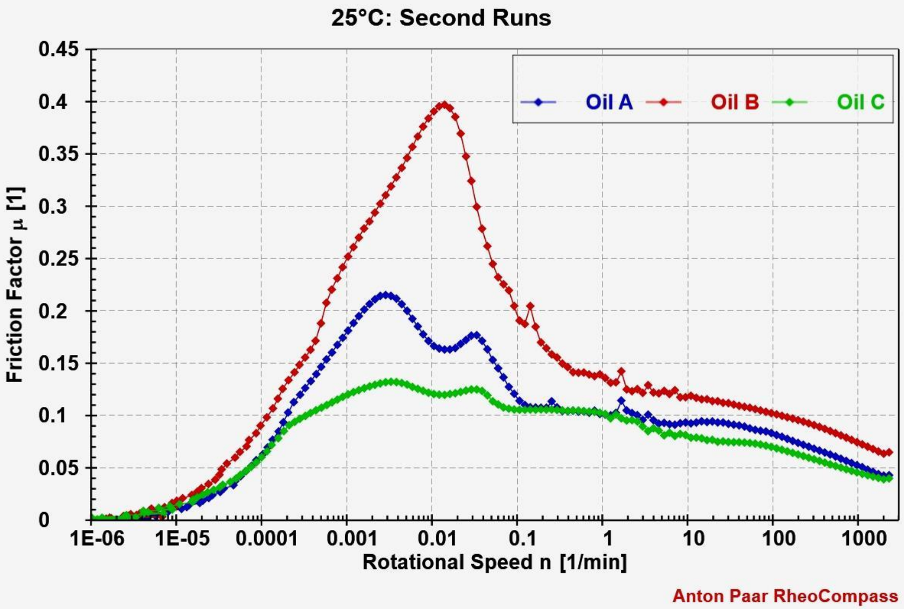
Tribofilms help increase wear resistance of the surfaces.

Testing Additive Performance

- Three oils with slightly difference formulations were tested for their friction behavior.
- Stribeck tests were carried out at two different temperatures, i.e. 25 °C and 100 °C.
- The applied normal force was 10 N (Hertzian pressure of around 700 MPa).
- 100Cr6 (AISI 52100) pins and ball were used in these tests.
- The test surfaces had an average roughness of around 0.02 µm.
- The choice of temperature was based on the fact that most EP/AW additives need a minimum activation temperature (> 80°C).

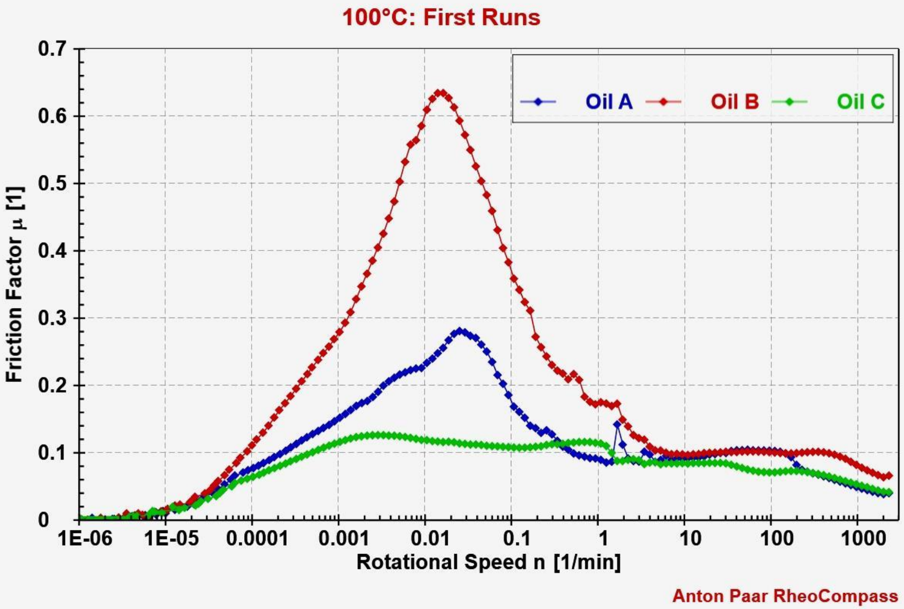
Lubricant Additives (25 °C)

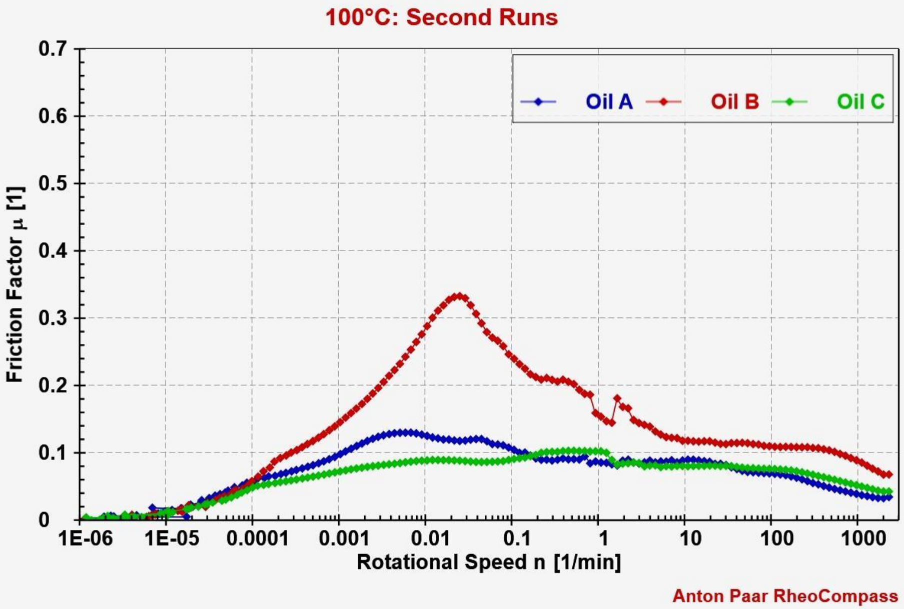




Oil B: Relatively higher friction over entire speed range

Lubricant Additives (100 °C)





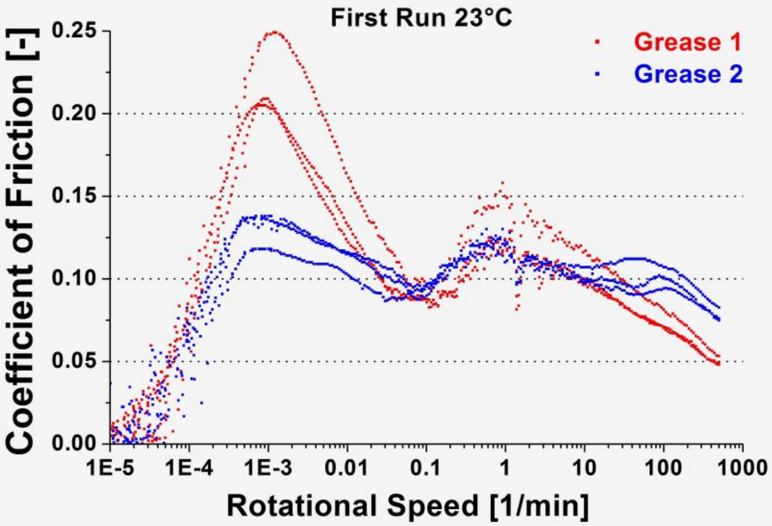
Oil B: Even at 100 °C, oil B shows higher frictional resistance

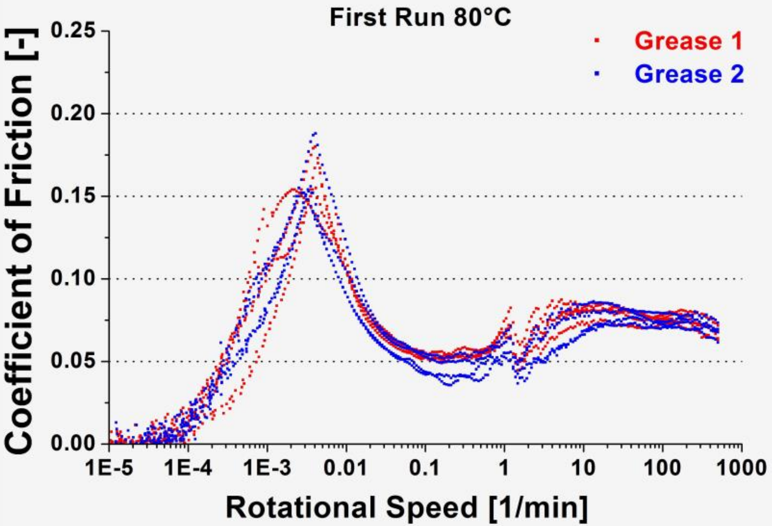
Applications

GREASES (I)

Please refer to our application reports on Greases for detailed information

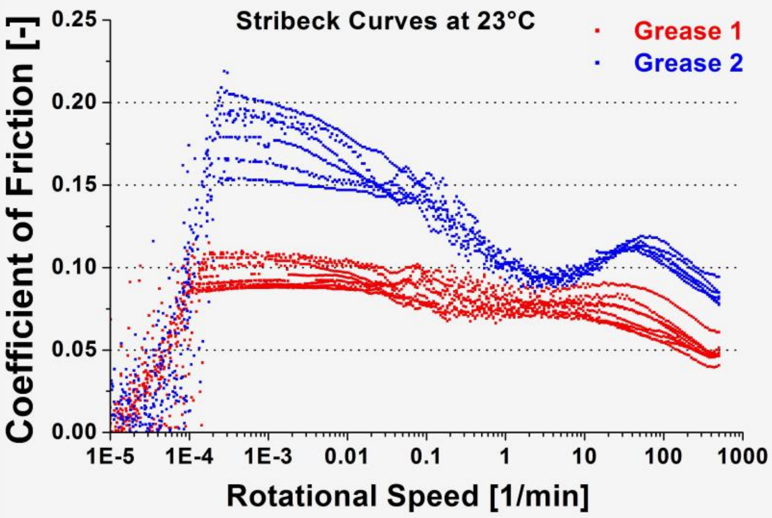
Grease: Stribeck Curves – 1st Runs

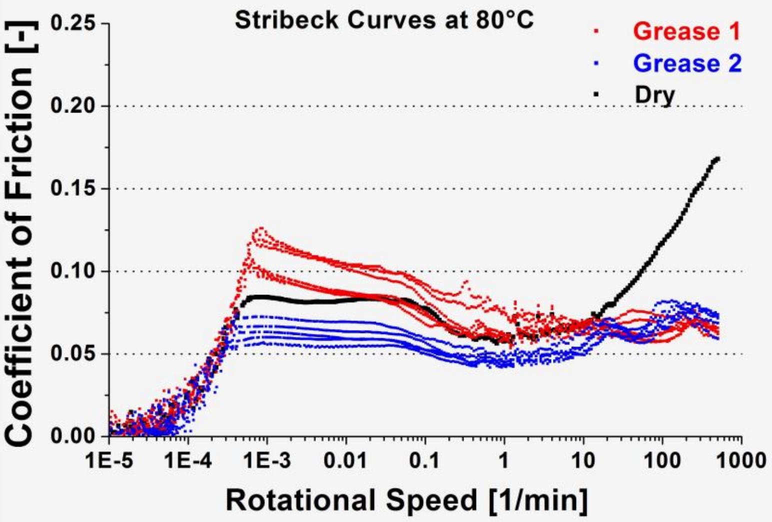




- At 23 °C, **grease 1** has higher limiting friction
- At 80 °C, there is no difference between **grease 1** and **grease 2**

Grease: Repeat Runs (2nd, 3rd, and 4th Runs)

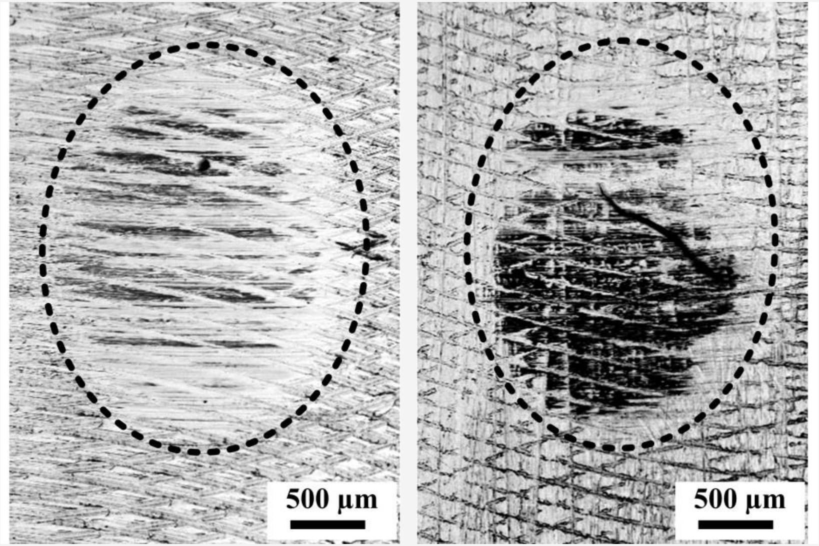




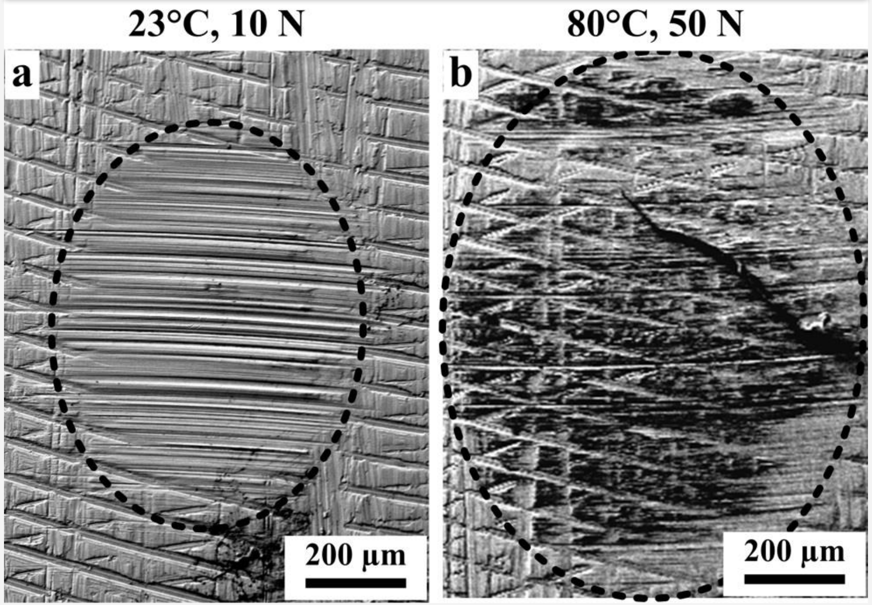
- **Grease 1** maintains its frictional resistance over the temperature range
- **Grease 2** is affected greatly by increase in temperature (formation of reactive films at the contact interface – see next slide)

Grease: Microscopic Analysis of Wear Scars

Grease 1	Grease 2



Grease 2



Friction with **Grease 2** is decreased due to formation of reaction films

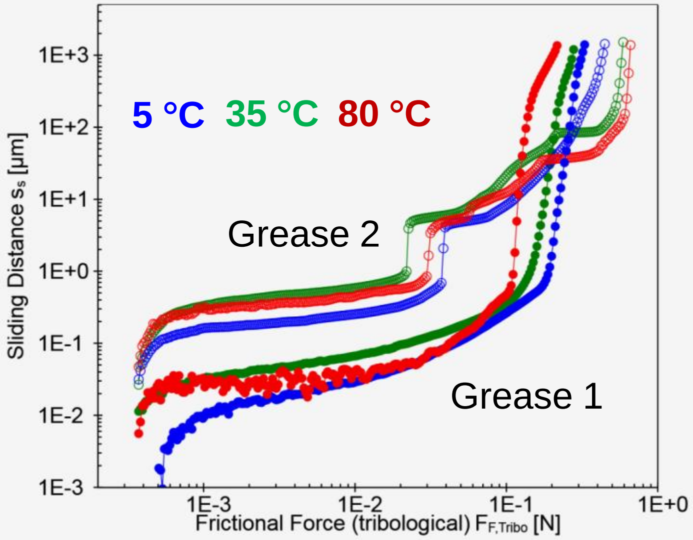
Such films only form at temperatures above 70 °C

Applications

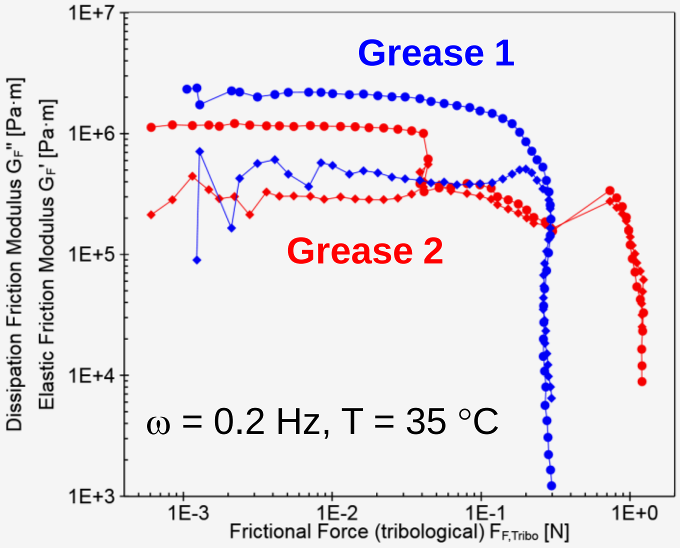
GREASES (II)

Breakaway Torque Measurements

Rotational torque ramp

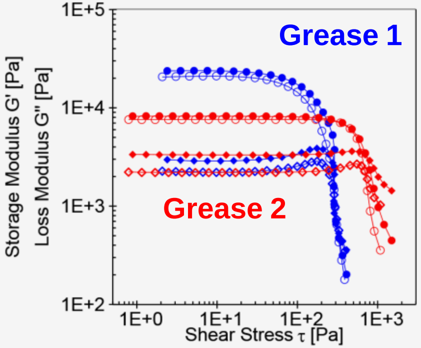


Oscillatory sliding distance sweep



Amplitude sweep

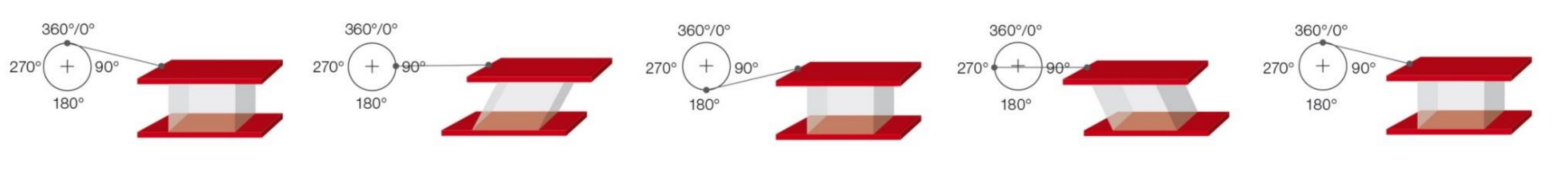
T_1 = 5 °C (closed symbols)
T_2 = 35 °C (open symbols)



- Grease 1: T dependency
- Grease 2: 2-step breakaway process

Correlation between rotational and oscillatory measurements.

Oscillatory Tests

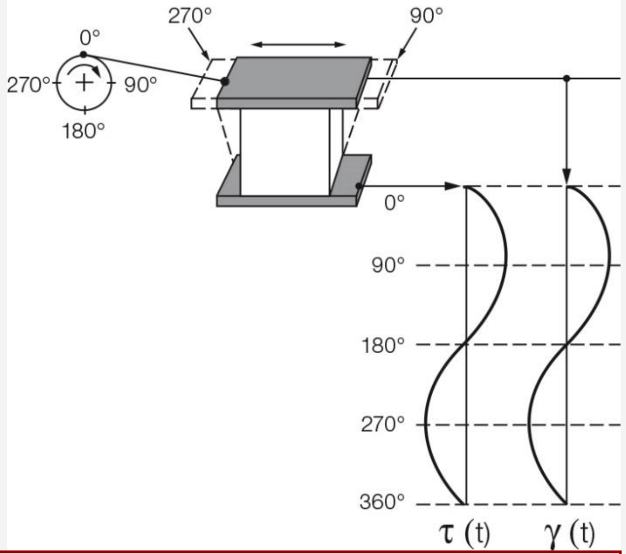


Two- plates model, equipped with two sensors,
top preset of deflection path (strain or deformation)
bottom measurement of resulting force (shear stress)

sinusoidal preset

ideal-elastic behavior
stiff sample (e.g. a stone or steel):
no time shift between the sine curves of
preset strain and resulting shear stress:

the curves of γ and τ are "in phase"



→ *movie (2-plates-model, ideal-elastic behavior)*

Oscillatory Tests

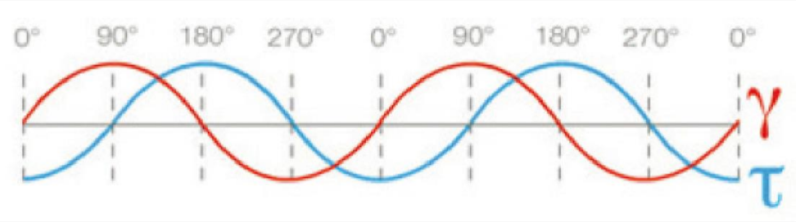
Anton Paar

ideal-viscous behavior
fluid, liquid: **90° ≥ δ > 45°**

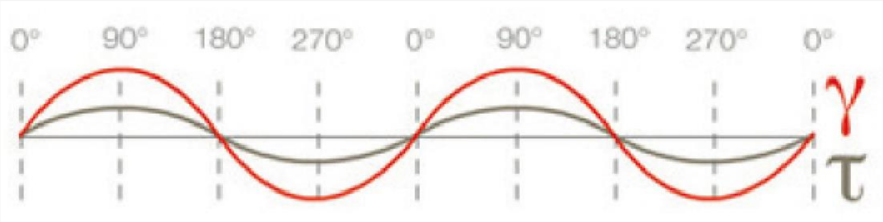
and

ideal-elastic behavior
solid, gel-like: **45° > δ ≥ 0°**

Illustrative concept: δ as the “street number in Rheology Road”

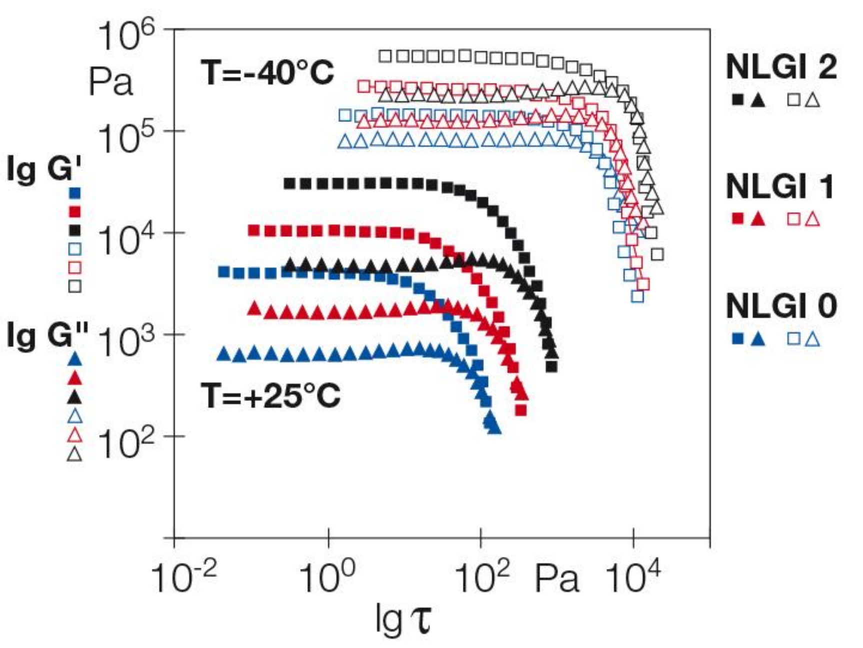


ideal-viscous: δ = 90°



ideal-elastic: δ = 0°

Amplitude Sweeps



Lubrication greases
flow point τ_f
acc. to DIN 51810-2
crossover point G' = G''

NLGI classification	T = +25 °C	T = -40 °C
NLGI 0	100 Pa	5 kPa
NLGI 1	200 Pa	7 kPa
NLGI 2	400 Pa	10 kPa

Consistency according to *NLGI*-classification (*National Lubrification Grease Institute*, USA) via pen-values, using a penetrometer

Applications

GREASES (III)

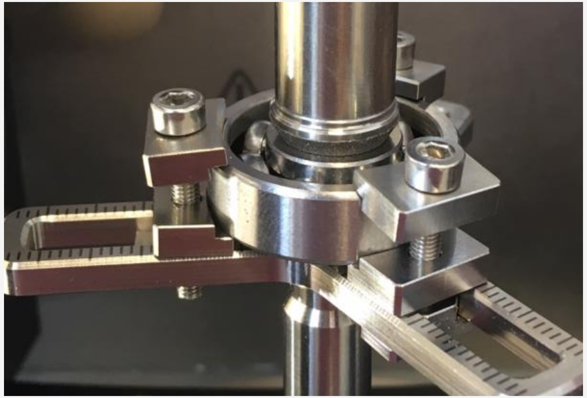
Greases: Different Levels of Abstraction

- Samples
  - Two different grease samples
- Specimen
  - 100Cr6 ball and 100Cr6 pins
  - Bearings

Greases: Bearing Adapter for CTD

MCR Tribometer

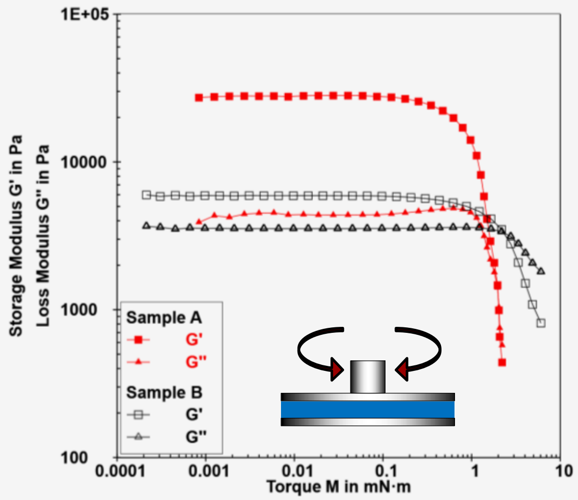
Bearing Adapter



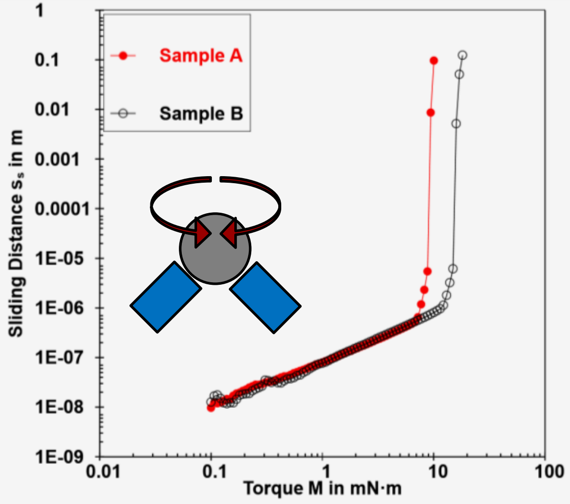
The Tribosystem

Level of Abstraction: Breakaway Torque

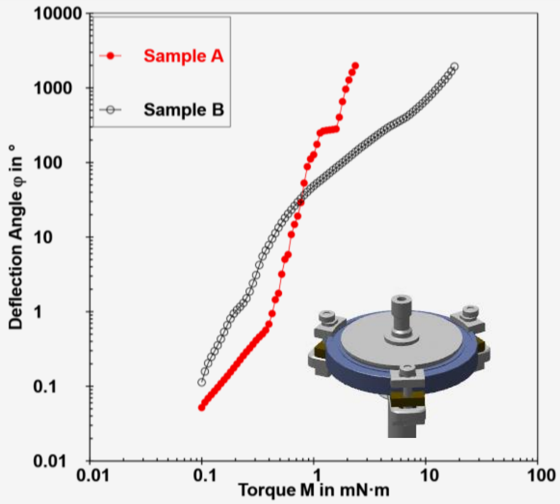
Amplitude sweep (Rheo)



round-on-flat (Tribo)



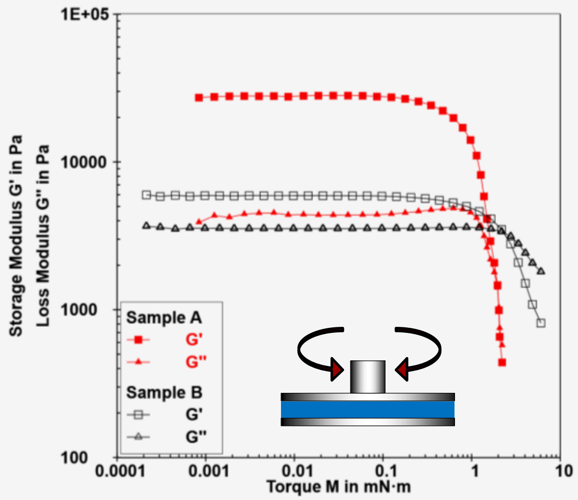
bearing (Tribo)



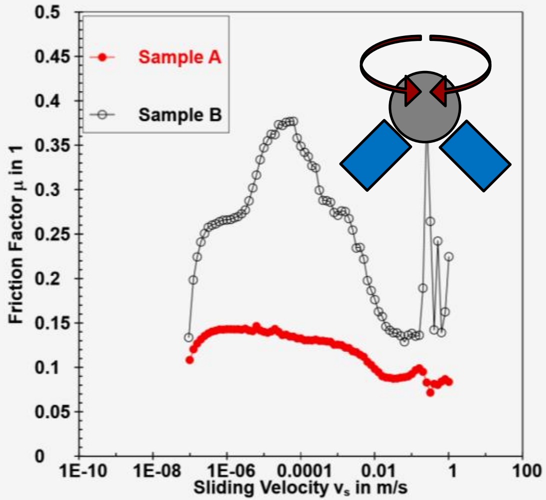
- Amplitude sweep: Crossover at higher torque for **Sample A**, lower G' and G'' for **Sample B**.
- Breakaway torque lower for **Sample A** in tribological tests, but obviously deformation with **Sample B** in bearing test before onset of macroscopic motion.

Level of Abstraction: Extended Stribeck Curve

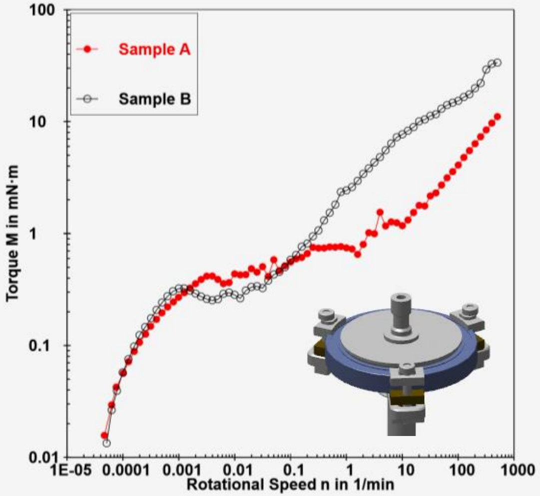
Amplitude sweep (Rheo)



round-on-flat (Tribo)



bearing (Tribo)



- Amplitude sweep: Crossover at higher torque for **Sample A**, lower G’ and G’’ for **Sample B**.
- Higher friction/torque in tribological tests for **Sample B**.

MCR Tribometers Overview

Lift motor

- Used to apply normal force
- Step width of ≈ 0.65 µm allows for smallest adaptions of vertical position

Normal force sensor

- High sensitivity and increased data sampling rate
- Large range with high resolution

→ individual normal force control

→ online wear measurements (qualitative)

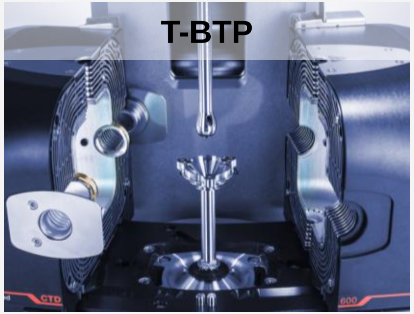
MCR Tribometers Overview

Peltier Temperature Control

- Ball-on-three-plates
- Ball-on-three-pins
- Bearing option
- 4-ball setup
- Isolated holders
- Foil holder

Peltier Temperature Control

- Pin-on-disc
- Balls-on-disc
- Ring-on-disc
- Foil holders



Peltier Temperature Control or

Electrical Temperature Control

- Ball-on-three-plates
- Ball-on-three-pins
- Bearing option

There is always scope for customizing solutions.
Please get in touch with your Anton Paar representative.

T-BTP Sample Holder and Rotating Shaft

T-BTP

Parameter	Min	Max
Torque [mNm]		300
Speed [rpm]	10^{-6}	3000
Normal Force [N]	0.1	50
Temp. [°C]	-140	600

T-PTD200 Sample Holder and Rotating Shaft

T-PTD200

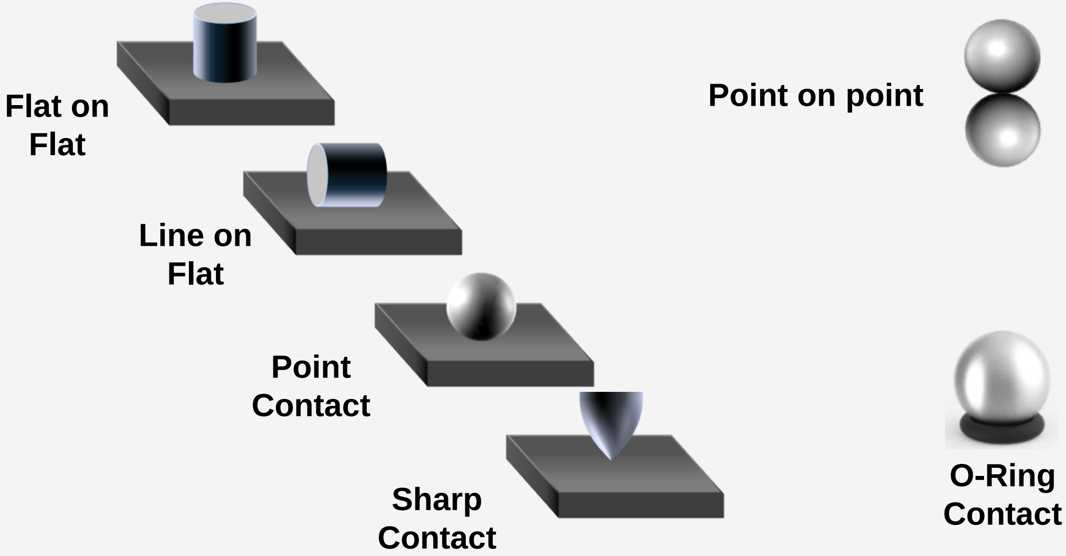
Parameter	Min	Max
Torque [mNm]		300
Speed [rpm]	10^{-6}	3000
Normal Force [N]	1	50
Temp. [°C]	-40	200

T-PID44 Sample Holder and Rotating Shaft

T-PID44

Parameter	Min	Max
Torque [mNm]		300
Speed [rpm]	10^{-6}	3000
Normal Force [N]	1	50
Temp. [°C]	-30	190

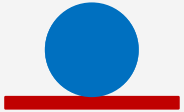
Contact Geometries (Examples)

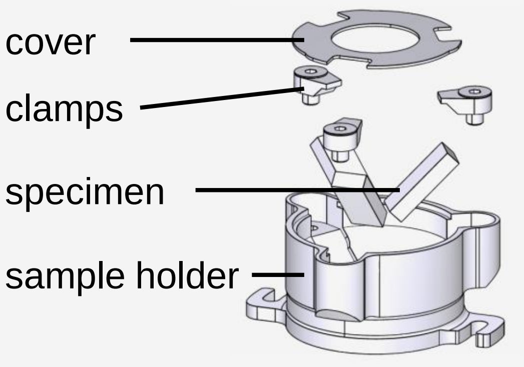


or: customized holder or specimen

T-PTD200 Sample Holders for Ø12.7 mm Balls

round-on-flat contact:





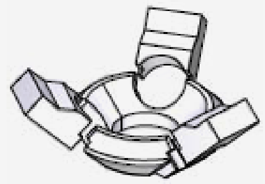
- Sample holders equipped with sample holder cover to avoid spurting out of fluid sample.
- Customized specimen available on request
- Low sample volume (1.5 ml to 2.5 ml)
- Specimen are either fixed with screws and clamps (plates) or just fit in the sample holder (pins, balls)
- Specimen dimensions
  - Plates: 15 mm x 6 mm x 3 mm
    or 10 mm x 10 mm x 3 mm (holder 45 °)
  - Pins: ∅ 6 mm x 6 mm
  - Balls: ∅ 12.7 mm

Plate holder *)

Pin holder *)

Plate holder 45°

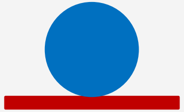
Cylinder option



*) standard scope

T-PTD200 Sample Holders for Ø30 mm Balls

round-on-flat contact:



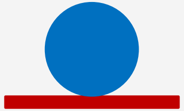
Pin holder

Square plate holder

Plate holder 45°

- Sample holders equipped with sample holder cover to avoid spurting out of fluid sample.
- Customized specimen available on request
- Customized sample holder on request
- Specimen are either fixed with screws and clamps (plates) or just fit in the sample holder (pins, balls)
- Specimen dimensions
  - Plates: 10 mm x 10 mm x 3 mm
  - Pins: ∅ 6 mm x 6 mm

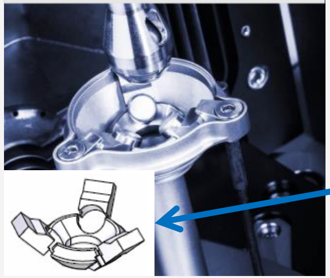
T-BTP Sample Holders

round-on-flat contact: 

Measuring Shaft BC12.7

Sample holder for plates

Cylinder option *)



These adapters are placed in the plate holder. This enables for fixation of pins.

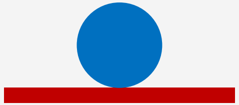
- Sample holder equipped with sample holder cover to avoid spurting out of fluid sample.
- Specimen are either fixed with screws and clamps (plates) or by aims of the cylinder option (pins).
- Specimen dimensions
  - Plates: 15 mm x 6 mm x 3 mm
  - Pins: ∅ 6 mm x 6 mm
  - Balls: ∅ 12.7 mm
- Specimen ball is pushed into the shaft and removed with the ejector

*) not within the standard scope

T-PID44 Sample Holders

flat-on-flat contact:

round-on-flat contact:

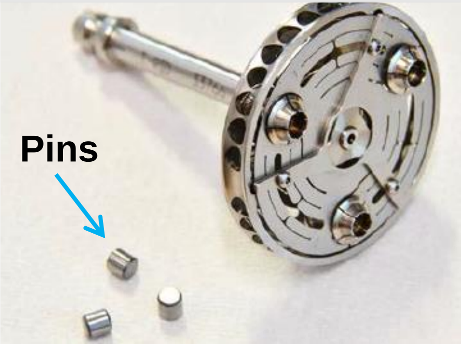


- T-PID44 measuring shaft can be used with
  - Pins: ∅ 6 mm x 6 mm
  - Balls: ∅ 6 mm
  - Films/foils: ∅ ≥ 5.6 mm x < ≈ 2 mm (height)
- Use torque wrench for defined fixation of specimen

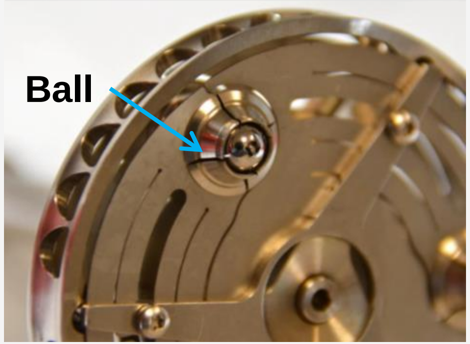
T-PID44 measuring shaft …

… used as pin holder	**… used as ball holder**	**Foil/film measuring system adapters *)**

… used as pin holder



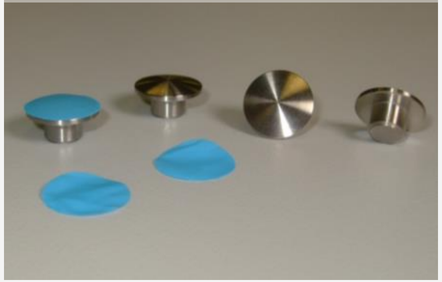
… used as ball holder



Foil/film measuring system adapters *)

clamping

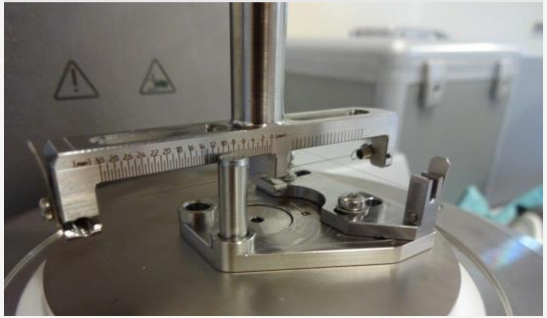
adhesive



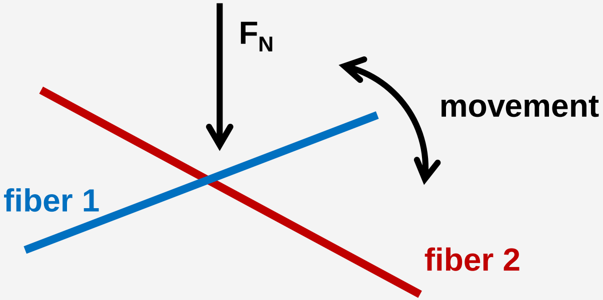
*) not within the standard scope

Exotic Solution: Fiber Setup

Fiber setup



- Horizontal fixation of two fibers
- Enables for measuring the friction between fibers



There is always scope for customizing solutions.
Please get in touch with your Anton Paar representative.

Specifications

Temperature Control Technology	Peltier			Electrical
Measuring Cell	T-PTD200	T-PID/44	T-BTP	
Environmental conditions				
Temperature Range	-40 °C to 200 °C	-30 °C to 190 °C	-20 °C to 180 °C	-160 °C to 600 °C
Relative Humidity Level	-	-	5 % to 95 %	-
Contact conditions				
Normal Force Range	1 N to 50 N	1 N to 50 N	0.1 N to 70 N	
Normal Force Resolution	0.005 N			
Contact Type	Point, Bearing	Point, Line, Flat	Point ***)	
Motion conditions				
Continuous rotation				
Speed Range	10^{-6} rpm to 3000 rpm	10^{-6} rpm to 1000 rpm	10^{-6} rpm to 3000 rpm	
Sliding Speed Range	10^{-8} m/s to 3.3 m/s **)	10^{-8} m/s to 2.3 m/s	10^{-8} m/s to 1.4 m/s	
Torque Range *)	1 nNm to 300 mNm			
Torque Resolution *)	0.1 nNm			
Oscillatory rotation				
Frequency	10^{-7} Hz to 100 Hz			
Angular Amplitude	1 µrad to ∞ µrad			
Angular Resolution	10 nrad			
Additional parameters				
Min. Online Wear Depth	0.65 µm			

*) Friction force range and friction force resolution depend on the measuring geometry.

**) 1.4 m/s for BC12.7 and 3.3 m/s for BC30.

***) bearing option for T-BTP on request

Please refer to the current brochure for latest information on specs.

Anton Paar

Tribometers (pin on disk test method)

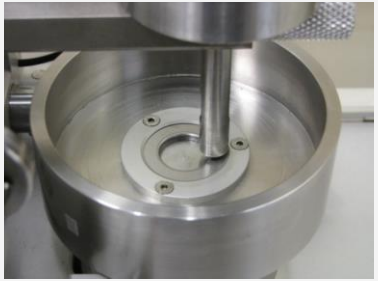
Tribometer (TRB^3)

- Proven dead weight load application
- Rotation and linear motion
- Many options: heating, linear reciprocating movement, etc.

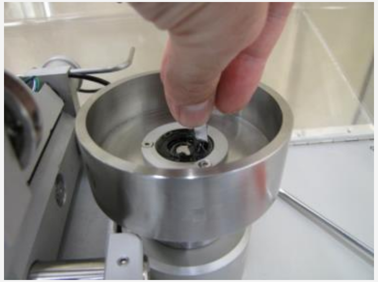
Friction properties of lubricants

Two oils and two greases

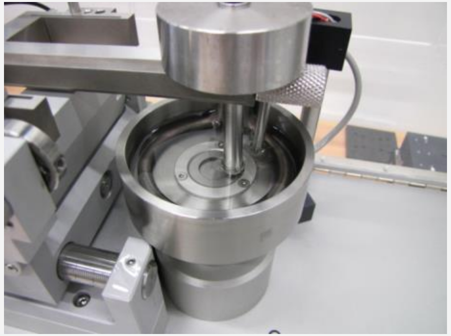
Dry test; RT



Application of the greases



Oil 1, 50°C

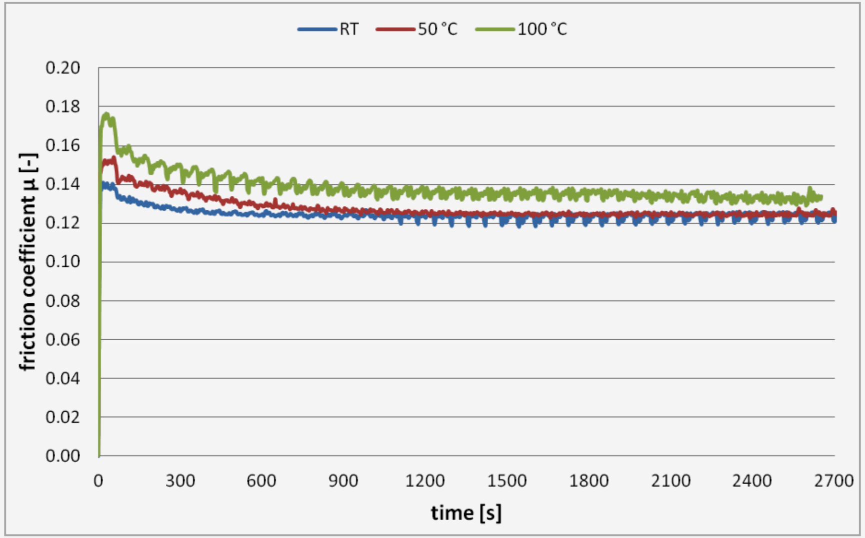


Oil 2; 100°C

Typical application – fluid lubricant

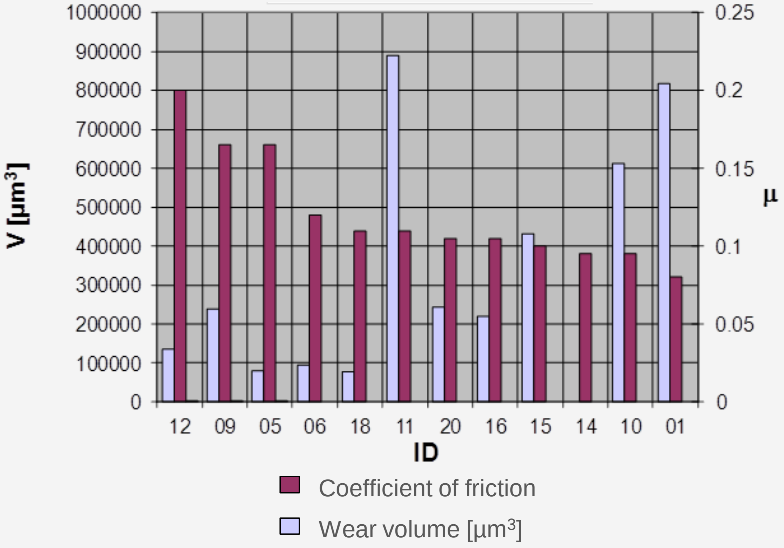
Study of engine oils lubrication behavior at different temperatures

Engine oil tribology testing



Typical application – fluid lubricant

Comparison of tribological properties of 18 motorcycle chain lubricants



ID	Designation
15	MR.FAST'NER CHAIN LUBE SPRAY
11	AISIN SYNTHETIC CHAIN LUBRICANT
10	CASTROL CHAIN WAX
01	FINISH LINE KRYTECH WAX LUBRICANT
16	SILKOLENE CHAIN LUBE
20	GP PRODUCTS SUPER CHAIN LUBE
16	SILKOLENE CHAIN LUBE
18	SILKOLENE MEDIUM GEAR OIL
14	MOBIL SYNTHETIC MOTORCYCLE CHAIN LUBE
12	MOTOREX MOTOLINE CHAINLUBE 62 STRONG
09	TECFLOW CERAMIC CHAIN SPRAY
06	YOSHIMOTO
05	KAL GARD RACING SPEC. CLEAR CHAIN KOTE WITH PTFE
19	MORRIS CHAIN LUBE FULLY-SYNTHETIC FORMULA
13	ROCK OIL PROFESSIONAL CHAIN LUBE
08	MOTRAX HIGH PERFORMANCE CHAIN LUBE FOR MOTOCYCLES
03	MOTUL CHAIN LUBE
17	BLUE LABEL PJ1 CHAIN LUBE
-	Without lubricant

Thanks