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//// T-09-113 –Wear and Friction Analysis of Thin Coatings

An in-depth study of the tribological properties of thin coatings on stainless steel substrates using a CSM Instruments Tribometer

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Report Reviewed By: Drew Griffin

//// OBJECTIVE

The purpose of this testing was to provide Silcotek with the tribological wear rates and friction properties of three steel coupons coated with thin layers of Si-based coatings.

//// EXPERIMENTAL PROCEDURE

Sample Description

CSM Instruments received three testing coupons. Both sides of each sample were coate, one side was polished to a mirrored finish and the other side was not finely polished. All wear tracks were created on the mirrored finish side.

Sample	**1**	**2**	**3**
Label	2000	08J04D	09G11D
Thickness/Roughness (Ra)	175 nm, <1u"	640 nm, <1u"	200 nm, <1u"
Material	Si	Si, N	Si, C
Substrate Material	316 SS	316 SS	316 SS
Coating Deposition Method	CVD	CVD	CVD

Instrument Overview

A CSM Instruments Tribometer (S/N 18-343) was used to perform the wear studies.

A CSM Instruments NanoScratch Tester (S/N) with a 1 µm radius spheroconical diamond indenter (S/N SE-A16) was used to perform the track profilometry.

Experimental Parameters

Tribometer Parameters

A 6mm 100Cr6 steel ball bearing was used as the static contact partner.

Sample	**1**	**2**	**3**
Label	2000	08J04D	09G11D
Track Radius (mm):	14.03	14.02	14.03
Linear Speed (cm/s):	3.00	3.00	3.00
Normal Load (N):	0.50	0.50	0.50
Stop Condition (laps)	1000	4000	1002
Acquisition rate (Hz):	2.0	2.0	2.0
Static Partner:	6 mm 100Cr6 Ball	6 mm 100Cr6 Ball	6 mm 100Cr6 Ball
Temperature (^{O}C):	23.6	23.6	23
Humidity (%RH):	44	44	46

Sample Preparation

The top of the samples were tested. All samples were cleaned gently with a non-abrasive wipe to remove any surface debris. Each sample was attached to a steel mounting sample with double sided tape, which was in turn attached to the motor using the round sample chuck.

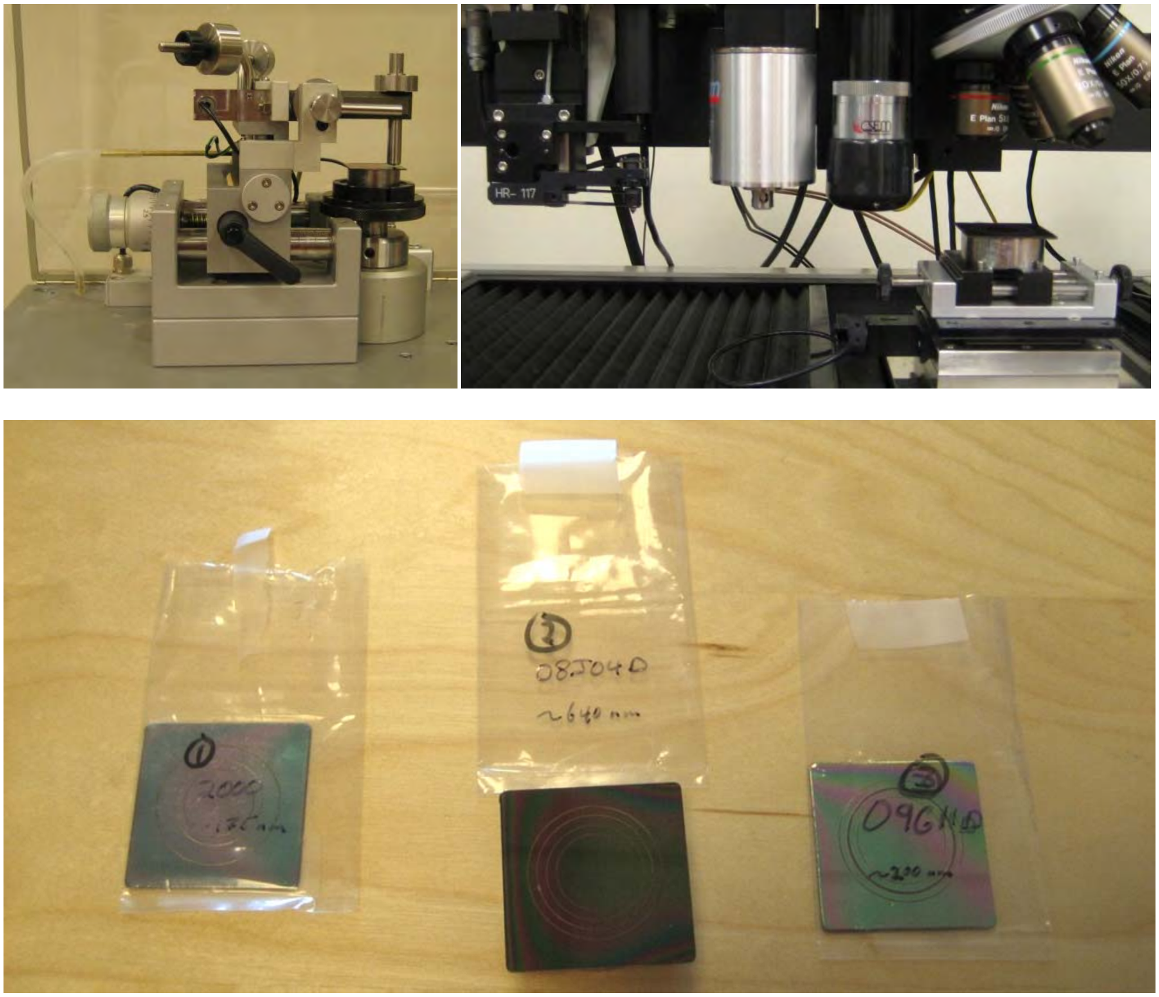


Figure 1: (Top Left) A sample during testing; (Top Right) a sample during optical track analysis after contact profilometry; (Bottom) the samples after testing.

//// RESULTS SUMMARY

Tribometer Results

Tribology is the study of the interactions between two surfaces in relative motion and includes the study of the components of friction, wear and lubricity. CSM Instruments studies tribological relationships between two surfaces using the Pin-on-Disk method. This method consists of a stationary pin, or static partner, which is held in contact with a rotating disk by some fixed normal load (F_N). The deflection of the static partner is measured for load and this is recorded as a tangential force (F_T). The ratio of F_T to F_N is calculated as the coefficient of friction (COF). The coefficient of friction is not only materials dependant but can be affected by changes in temperature and relative humidity. Wear rate is calculated by measuring the volume of material removed and normalizing that to the load and the distance travelled during the test. In order to measure the volume of track material removed, track profiles were taken after testing. Please refer to the appendix for a more detailed explanation.

It should be noted that Sample 2 was run to 4000 laps while Samples 1 and 3 were run to roughly 1000 laps; this was because of different failure modes observed. Two graphs were generated in order to compare the COF data. In the first graph (see Figure 2), the COF data over the entire test period for each wear track is compared. In the second graph (see Figure 3), only the COF data over the first 1000 laps is compared in order to show more detail in this region.

COF Comparisons

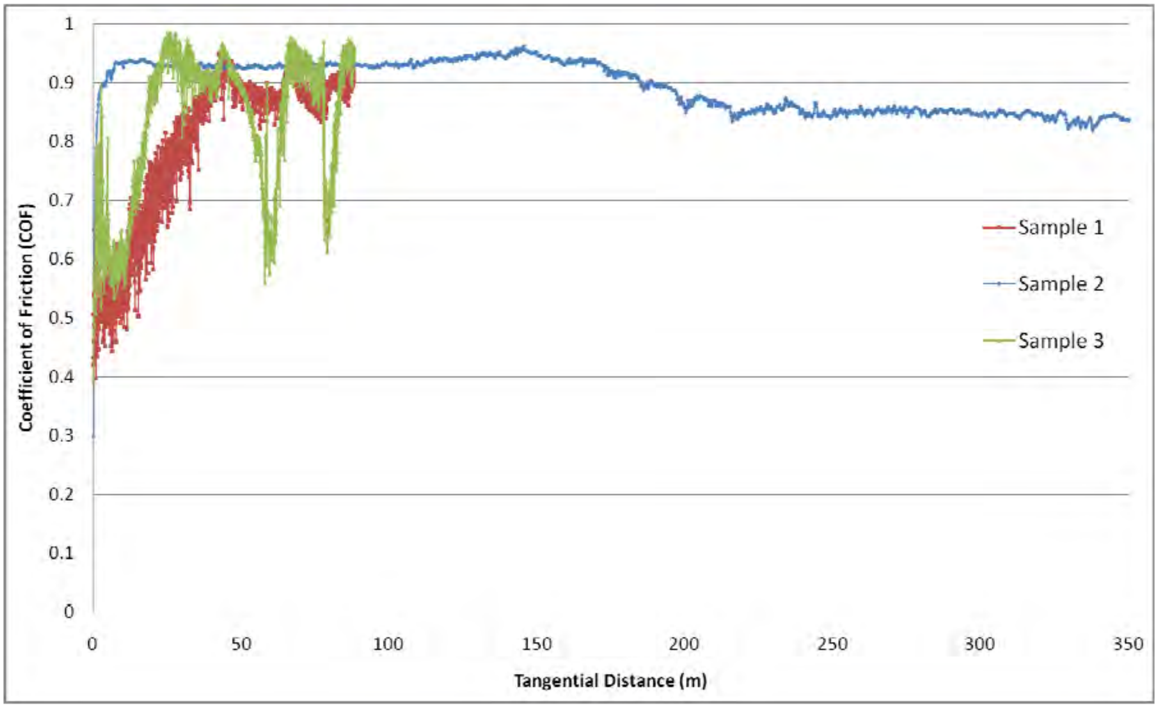


Figure 2: COF comparison over the total distance run for each sample

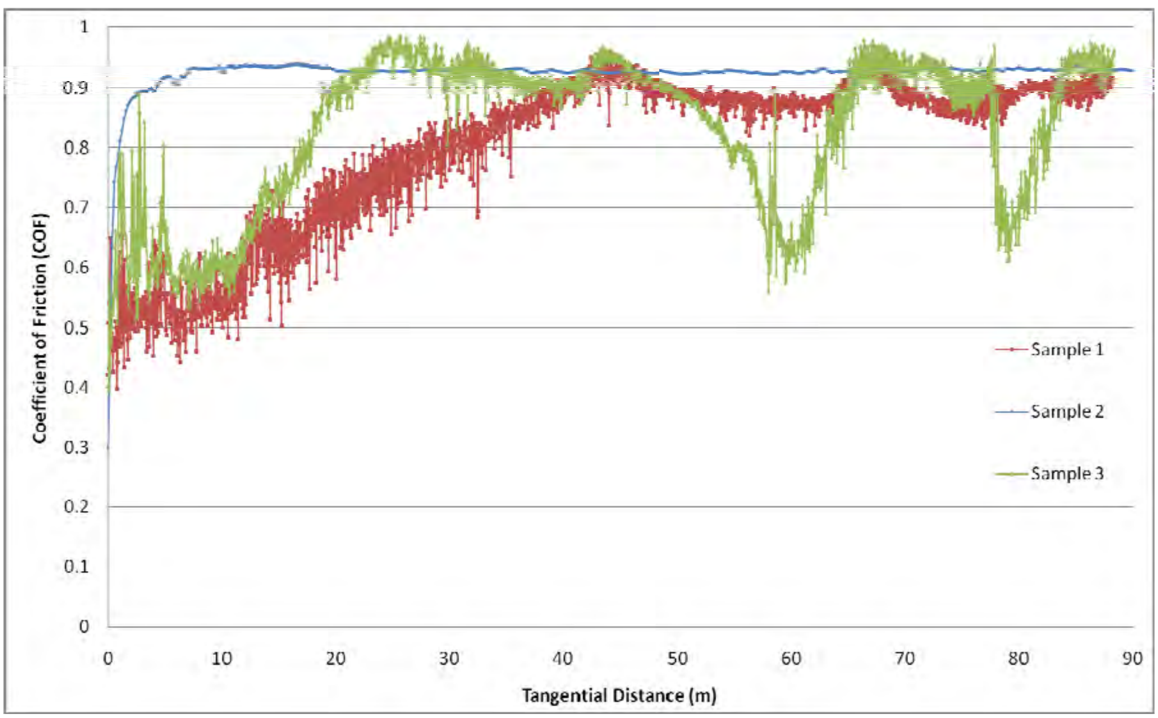
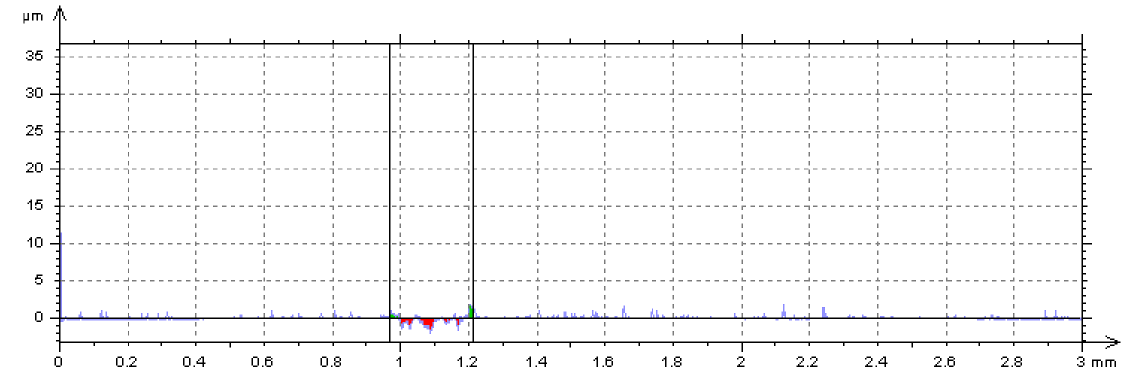


Figure 3: COF Comparison over distance of 1000 laps

Wear Rate Calculations

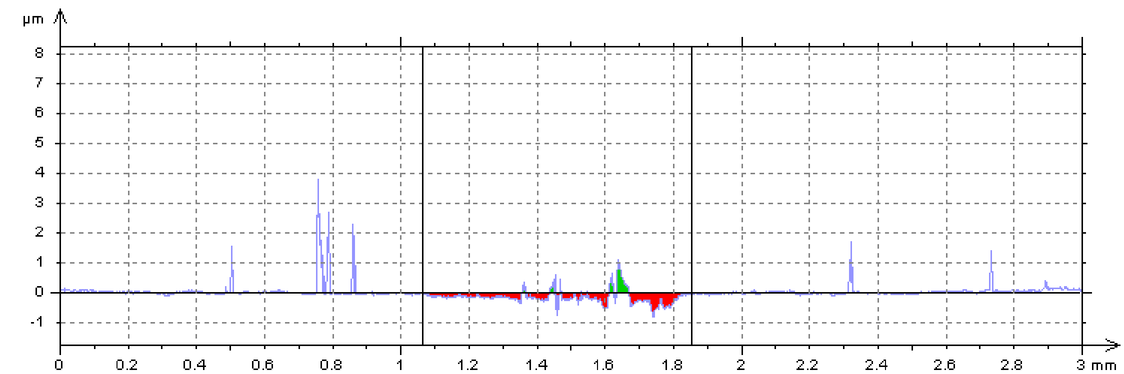
Sample 1 (2000)



Maximum depth : 2.04 µm Area of the hole : 107 µm2

Maximum height : 1.76 µm Area outside : 48.5 µm2

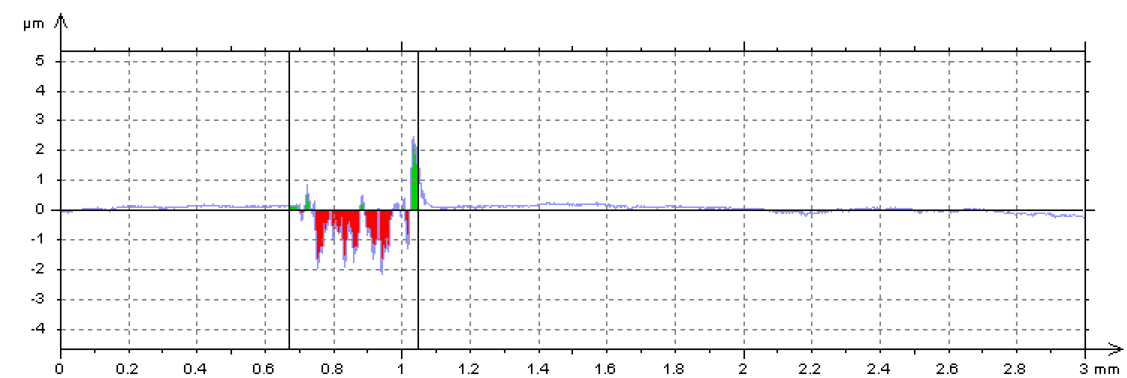
Sample 2 (08J04D)



Maximum depth : 0.866 µm Area of the hole : 187 µm2

Maximum height : 1.04 µm Area outside : 28.4 µm2

Sample 3 (09G11D)

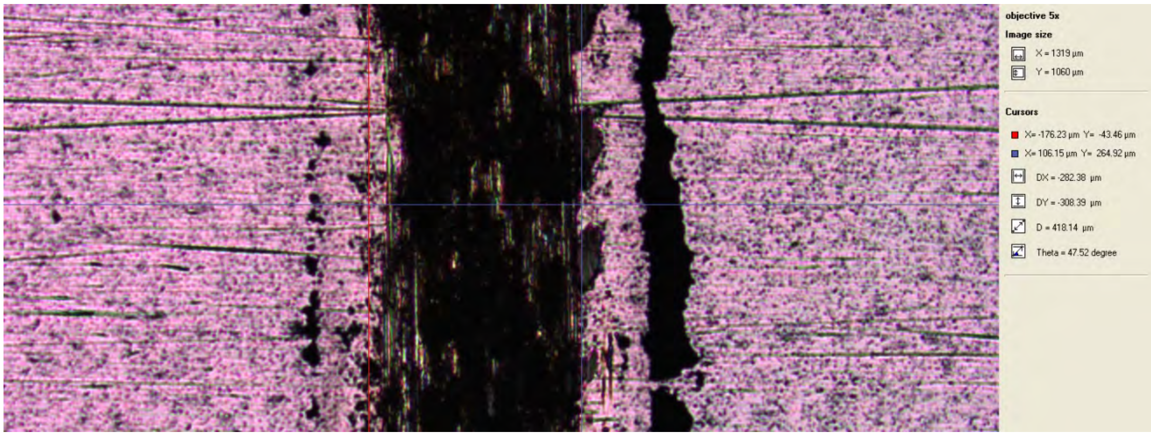


Maximum depth : 2.19 µm Area of the hole : 237 µm2

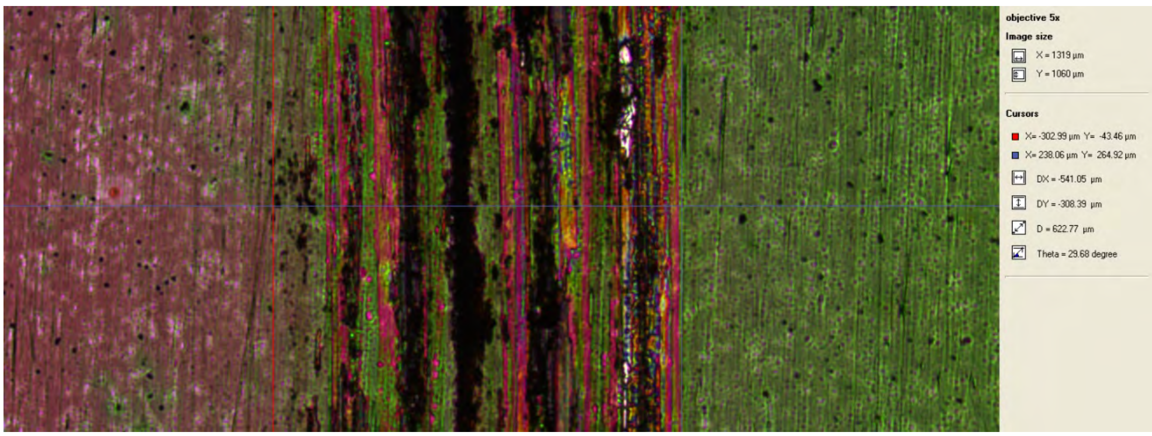
Maximum height : 2.38 µm Area outside : 59.8 µm2

Figure 4: Wear track profiles as determined by contact profilometry

Sample 1 (2000)



Sample 2 (08J04D)



Sample 3 (09G11D)

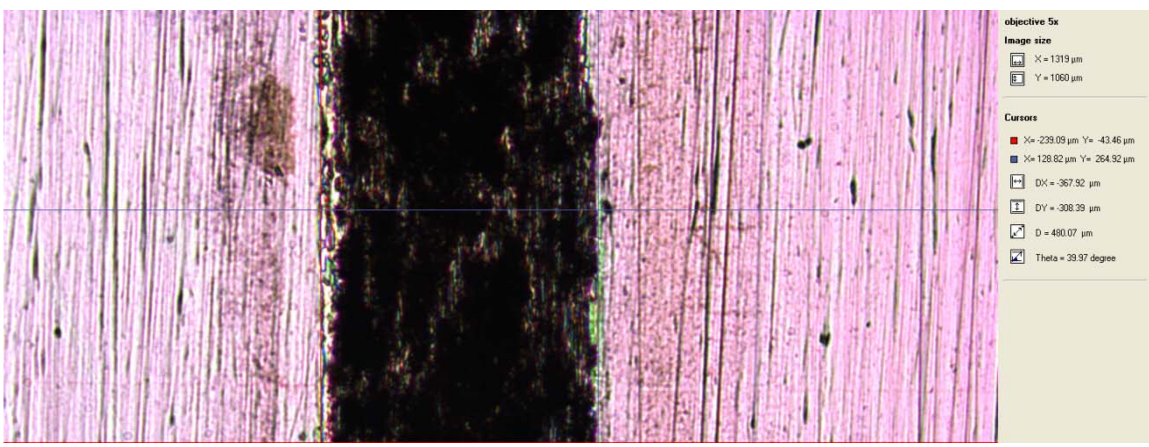


Figure 5: Figure 6: Photomicrographs of the wear track profile areas

Sample		1	2	3
Label		2000	08J04D	09G11D
Radius	mm	14.03	14.02	14.03
Laps		1000	4000	1002
Track Area	µm^2	107	187	237
Track Wear Rate	mm^3/N/m	2.14E-04	9.35E-05	4.73E-04
Ball Worn Diameter	µm	370	720	393
Ball Wear Rate	mm^3/N/m	6.97E-06	2.51E-05	8.85E-06

Figure 7: Table of Wear Rate Calculations

//// *OBSERVATIONS*

- Samples 1 (2000) and 3 (09G11D) completely delaminated after 1000 laps, as evidenced by the substrate visible in the wear tracks. The wear rate of sample 3 was highest, while that of sample 1 was roughly one half of that of sample 3. The ball wear rates were lowest for samples 1 and 3.

- Sample 2 (08J04D) did not show complete delamination even after 4000 laps. The sample had the lowest wear rate at less than one half that of sample 1, and had the highest ball wear rate. Because this sample was run to so many more laps, the ball wore to a larger flat area; correspondingly, the wear track was much wider and shallower for this sample than for the others.

- The COF behavior of the samples differed significantly. The COF of Sample 1 (2000) gradually increased until a plateau at just under 0.9; this plateau was unsteady. The COF of Sample 2 (08J04D) increased very quickly to a plateau at just over 0.9 and remained steady. The COF of Sample 3 (09G11D) increased at a moderate pace to a plateau, and then dipped several times only to recover quickly. This may be interpreted as the slow accumulation of debris, which clears out quickly and then begins to accumulate again.

//// *APPENDIX – RAW DATA*

CSM Instruments
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T-09-113 Tribology Testing (Silcotek)

Sample 1 (2000)

Tribo measurement3

Tribo parameters

Acquisition
- Radius : 14.03 [mm]
- Lin. Speed : 3.00 [cm/s]
- Normal load : 0.50 [N]
- Stop condit. : 1000 [lap]
- Effective Stop : Laps
- Acquisition rate : 2.0 [hz]

Sample
- Coating : Silicon Based
- Substrate : Stainless Steel
- Cleaning : Dry Soft Cloth
- Supplier : SilkoTek

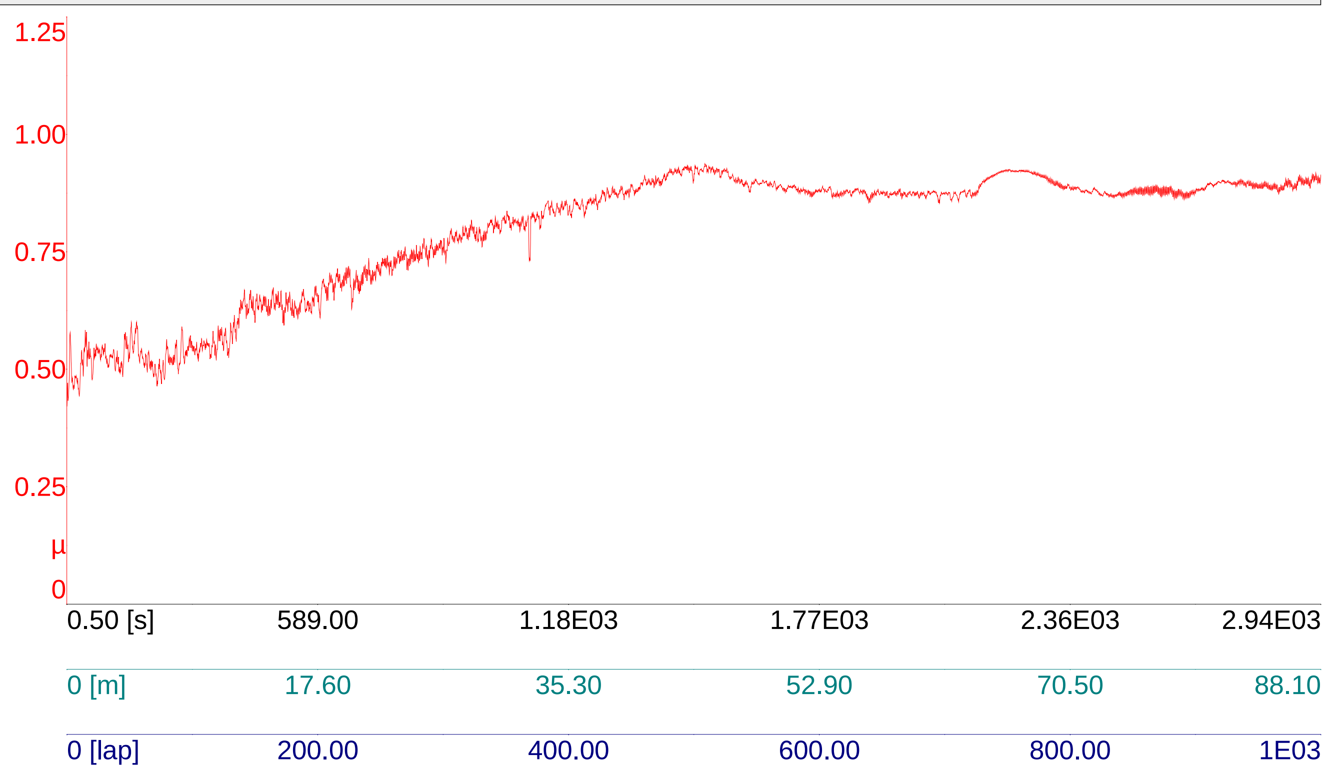
Static partner
- Substrate : 100Cr6
- Cleaning : IPA
- Dimension : 6.00 [mm]
- Geometry : Ball

Environment
- Temperature : 23.60 [<deg>C]
- Humidity : 44.00 [%]

Sample	Static partner	Calculations
Worn track section : 107.0 µm2	Worn cap diameter : 370.0 µm	Sample wear rate : 0.000214 mm3/n/m
Young's modulus : 0.0 gpa	Young's modulus : 0.0 gpa	Partner wear rate : 6.967E-006 mm3/n/m
Poisson ratio : 0.000	Poisson ratio : 0.000	Max Herzian stress : 0 gpa

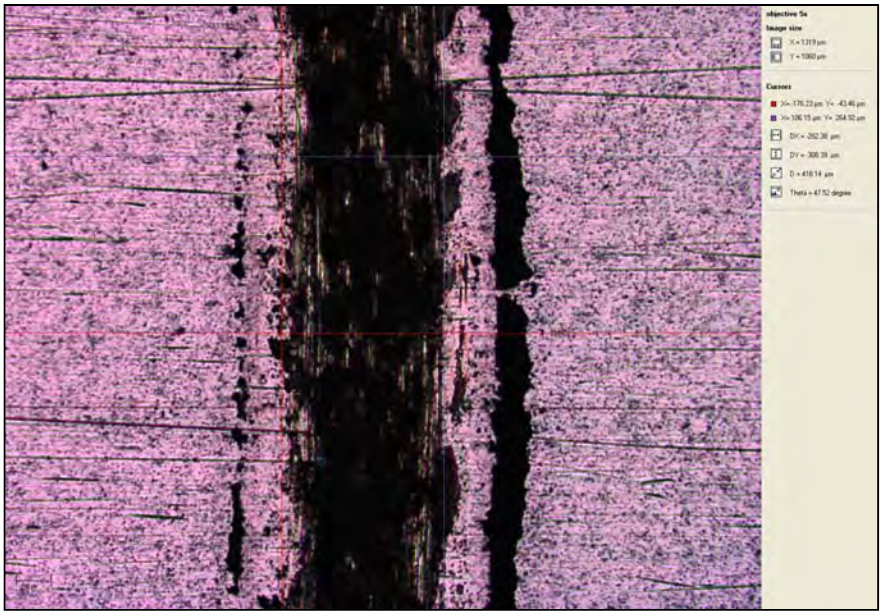
Curve

Start : 0.421 min : 0.421 max : 0.937 mean : 0.798 std. dev. : 0.132



Friction coef.

Sample 1 R=14 mm Wear Track 5x



Sample 2 (08J04D)

Tribo measurement4

Tribo parameters

Acquisition
- Radius : 14.02 [mm]
- Lin. Speed : 3.00 [cm/s]
- Normal load : 0.50 [N]
- Stop condit. : 4000 [lap]
- Effective Stop : Laps
- Acquisition rate : 2.0 [hz]

Sample
- Coating : Silicon Based
- Substrate : Stainless Steel
- Cleaning : Dry Soft Cloth
- Supplier : SilkoTek

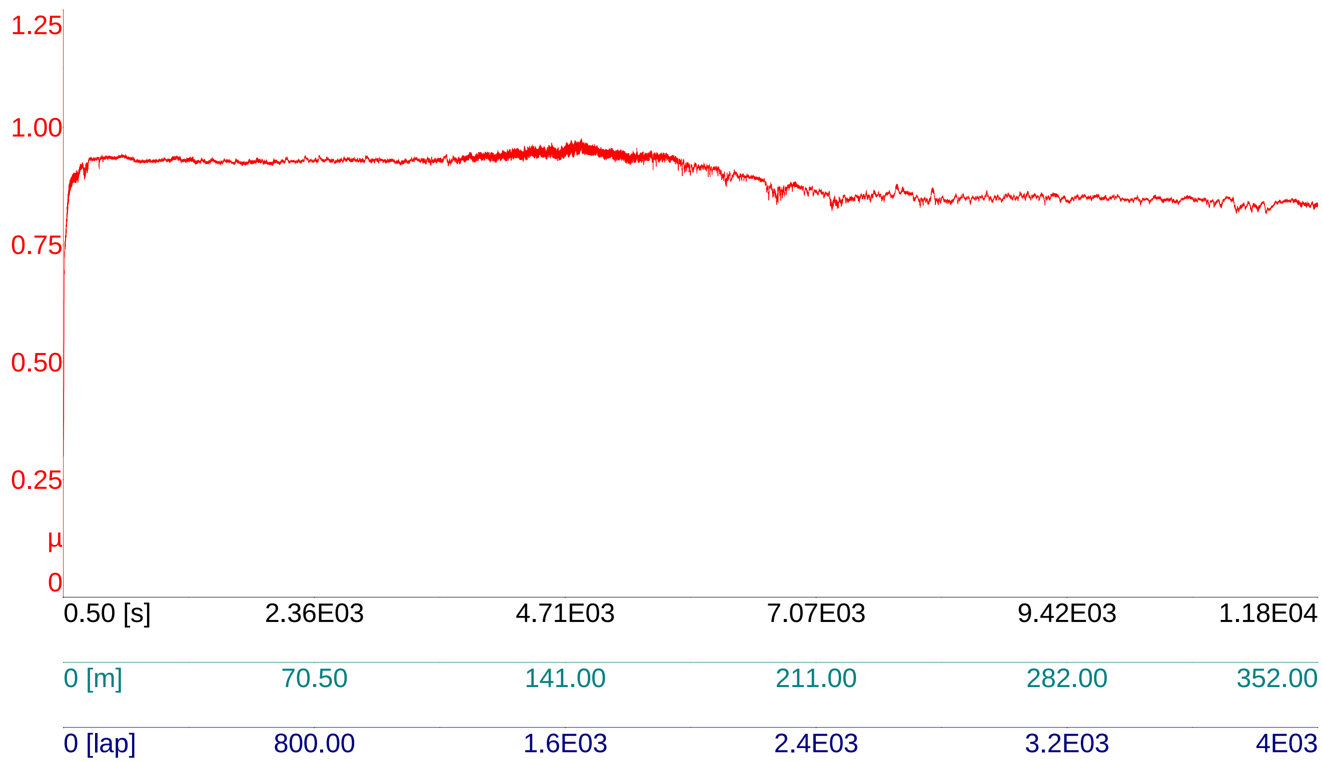
Static partner
- Substrate : 100Cr6
- Cleaning : IPA
- Dimension : 6.00 [mm]
- Geometry : Ball

Environment
- Temperature : 23.60 [<deg>C]
- Humidity : 44.00 [%]

Sample	Static partner	Calculations
Worn track section : 187.0 µm2	Worn cap diameter : 720.0 µm	Sample wear rate : 9.35E-005 mm3/n/m
Young's modulus : 0.0 gpa	Young's modulus : 0.0 gpa	Partner wear rate : 2.509E-005 mm3/n/m
Poisson ratio : 0.000	Poisson ratio : 0.000	Max Herzian stress : 0 gpa

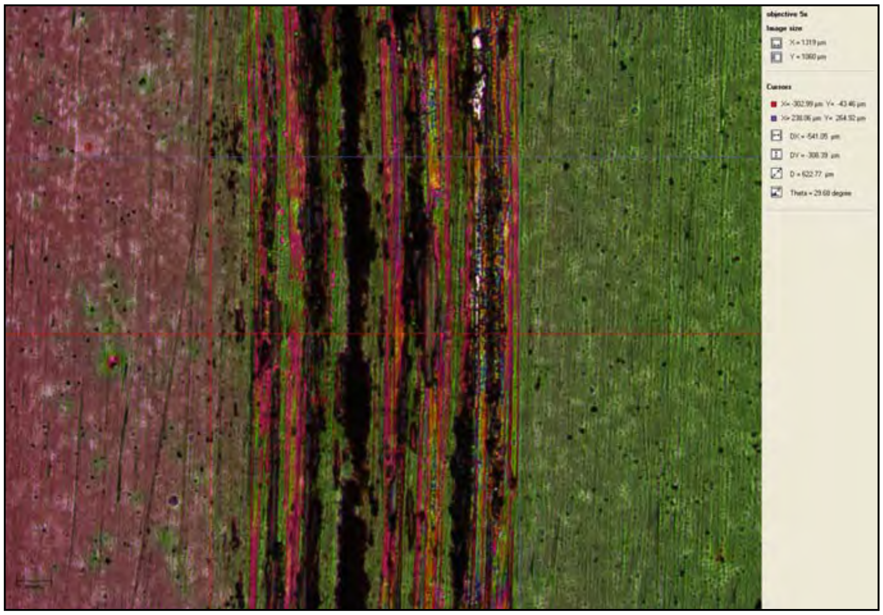
Curve

Start : 0.300 min : 0.300 max : 0.976 mean : 0.893 std. dev. : 0.044



Friction coef.

Sample 2 R=14 mm Wear Track 5x



Sample 3 (09G11D)

Tribo measurement3

Tribo parameters

Acquisition
- Radius : 14.03 [mm]
- Lin. Speed : 3.00 [cm/s]
- Normal load : 0.50 [N]
- Stop condit. : 1002 [lap]
- Effective Stop : Laps
- Acquisition rate : 2.0 [hz]

Sample
- Coating : Silicon Based
- Substrate : Stainless Steel
- Cleaning : Dry Soft Cloth
- Supplier : SilkoTek

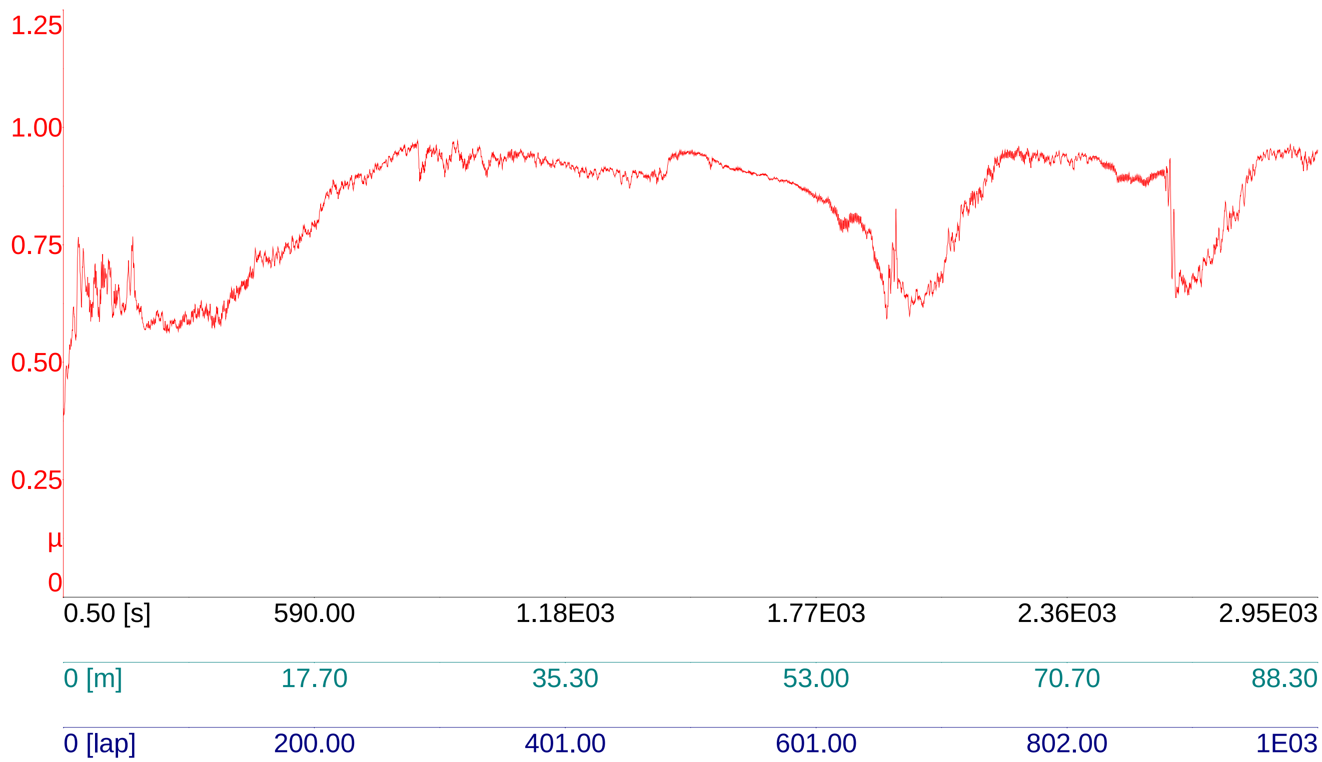
Static partner
- Substrate : 100Cr6
- Cleaning : IPA
- Dimension : 6.00 [mm]
- Geometry : Ball

Environment
- Temperature : 23.00 [<deg>C]
- Humidity : 46.00 [%]

Sample	Static partner	Calculations
Worn track section : 237.0 µm2	Worn cap diameter : 393.0 µm	Sample wear rate : 0.0004731 mm3/n/m
Young's modulus : 0.0 gpa	Young's modulus : 0.0 gpa	Partner wear rate : 8.851E-006 mm3/n/m
Poisson ratio : 0.000	Poisson ratio : 0.000	Max Herzian stress : 0 gpa

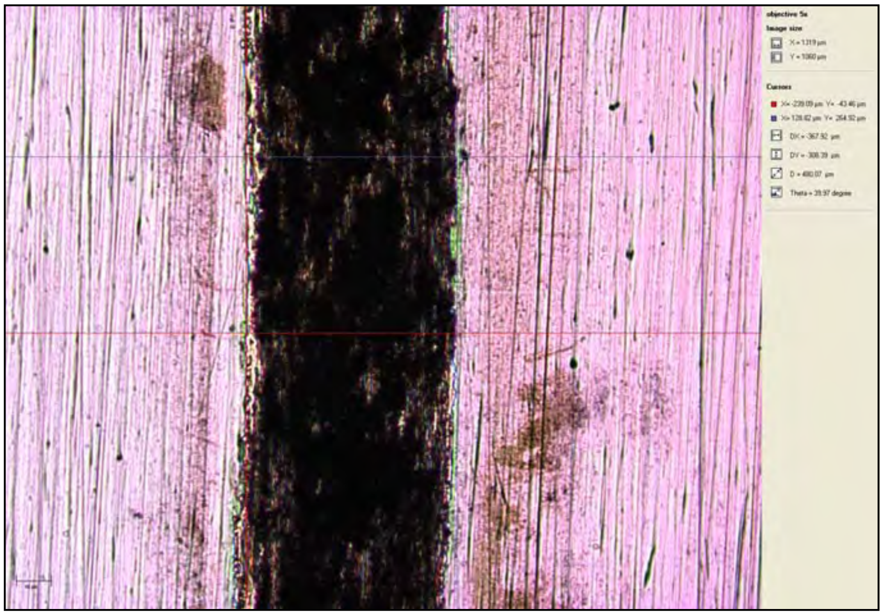
Curve

Start : 0.402 min : 0.388 max : 0.971 mean : 0.830 std. dev. : 0.123



Friction coef.

Sample 3 R=14 mm Wear Track 5x



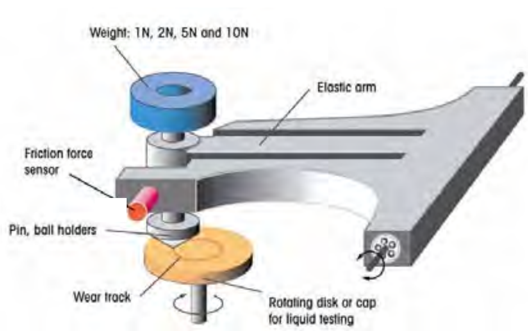
APPENDIX

//// Tribometer Testing

In tribometry, a flat, a pin or a sphere is loaded onto the test sample with a precisely known force. The pin is mounted on a stiff lever, designed as a frictionless force transducer. The friction coefficient is determined during the test by measuring the deflection of the elastic arm. Wear coefficient for the pin and disk materials are calculated from the volume of material lost during the test. This simple method facilitates the study of friction and wear behavior of almost every solid state material combination with or without lubricant. Furthermore, the control of the test parameters such as speed, frequency, contact pressure, time and the environmental parameters (temperature, humidity and lubricant) allows simulation of the real life conditions of a practical wear situation.

//// *Principle of Pin-on-Disk Tribometer*

The principle of the pin-on-disk Tribometer is shown below:



The static partner is usually a pin or a ball of defined geometry, in contact with the sample which is often a disk. The applied load can be varied by changing the weights mounted above the contact. Load range is 0.25 – 60 N. For most standard testing (to ASTM G99 or G133 specifications) the static partner is a steel ball of diameter 6 mm. The circular geometry of the ball is preferable to a pin because it is a perfect sphere and is therefore highly reproducible.

The basic equation on which tribology testing is based, is known as Archard's wear equation:

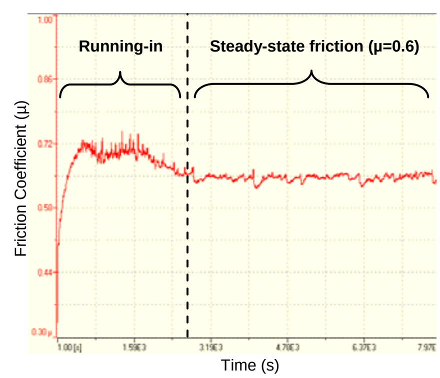
$$V = \frac{KW}{H}$$

where V is the volume of wear debris produced, K is the dimensionless wear coefficient, W is the applied load and H is the hardness of the material. It is often useful to use *k* which is the ratio K/H, or the dimensional wear coefficient. *k* is the volume removed (in mm^3) per unit distance slid (in m), per unit normal load on the contact (in N). *k* has the units $mm^3\ (Nm)^{-1}$

The friction coefficient, μ, is defined as the ratio of the tangential force, F_T, and the applied normal force, F_N:

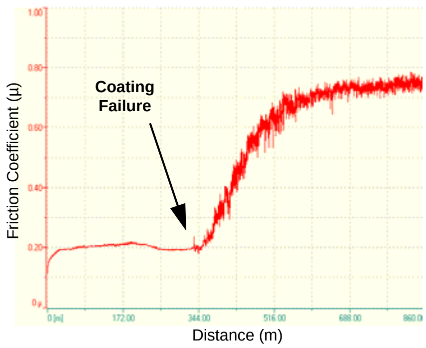
$$\mu = \frac{F_T}{F_N}$$

Once the static and rotating partners are in contact under load, the test consists of rotating the sample for a predefined number of cycles and recording the measured frictional force versus the number of cycles (or time, or distance covered). The software can also be configured so that the test is automatically stopped when a predefined friction coefficient value is reached. In this way, it is possible to monitor the evolution of friction over various phases of the test. For example, when the steady-state friction coefficient between 2 materials is of interest, one might run a test with the following result:

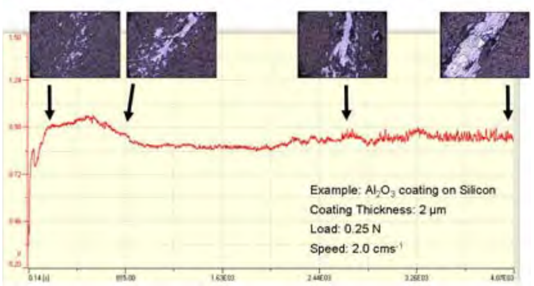


The initial portion of the data plot can be discarded as this represents the "running-in" period during which the 2 surfaces are becoming bedded to each other. The second portion represents the "steady-state" where the 2 surfaces are running smoothly over each other with a steady friction coefficient value (in this case, μ = 0.6)

In many practical applications of tribology, it is interesting to investigate the "lifetime" of the material pair, i.e., the amount of time that steady-state friction is maintained before the surfaces break down. This is particularly applicable to surface coatings, whose integrity can be qualified by a tribological test which simulates true in-service conditions of contact pressure, speed, lubrication and/or temperature. For example, a coating designed to give a low coefficient of friction over the lifetime of a component can be tested by running a simple test and monitoring when the friction starts to increase from steady-state conditions. This increase corresponds to failure of the coating:



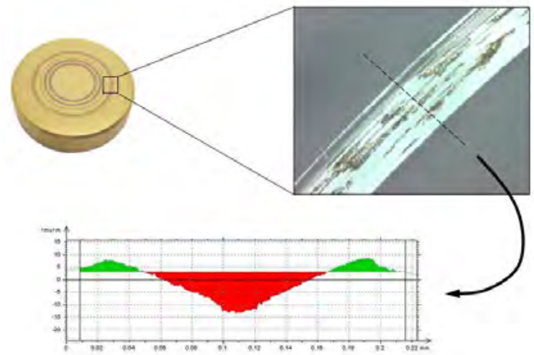
It can sometimes be beneficial to stop a test at various intervals and examine the wear track under an optical microscope. This can yield valuable information about the amount of wear debris generated and the mode of failure of the coating:



//// *Calculation of Tribological Parameters*

Once a test has been completed, the sample and static partner can be analyzed to calculate their respective wear rates. The simplest way to measure the material removed during testing is to weigh the sample and partner before and after testing. However, the volumes of material are often very small and even a high resolution balance may not be able to assess the mass lost with sufficient accuracy. Therefore, the method usually used is to measure the volume lost by a direct method, usually profilometry. This has the added advantage of measuring the actual volume of material lost, whereas the mass loss method may be inaccurate if some debris has been transferred to the static partner.

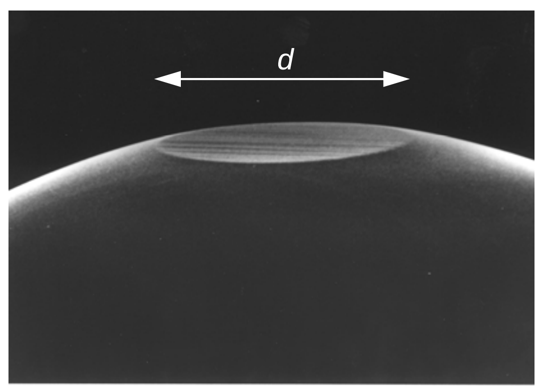
Once a test is completed, the sample is placed under a profilometer in order to make a line scan across the worn track. The area inside the profile can then be multiplied by the circumference of the wear track to determine the volume of material removed. In certain cases, the volume of material piled-up along the edges of the wear track may also be of interest.



The sample wear rate (in mm^3/N/m) is calculated by dividing the worn track section, S, by the measurement distance, L, and the applied load, F_N:

$$WearRate_{Sample} = \frac{S}{L.Fn}$$

The wear rate of the static partner may also be calculated. If a ball has been used as the static partner then the diameter of the worn cap is measured using an optical microscope as shown below:



The height, h, of the worn cap is given by:

$$h = R - \sqrt{R^2 - \frac{d^2}{4}}$$

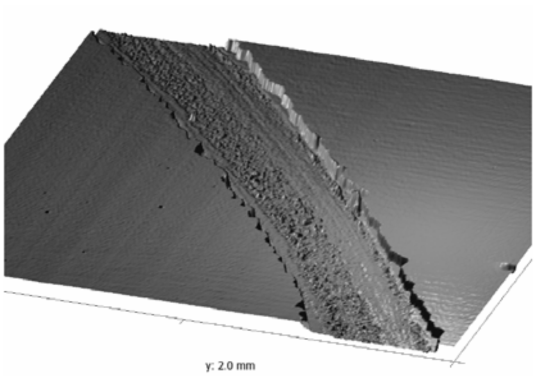
The volume, V, of the worn cap is then:

$$V = \frac{1}{3}\pi h^2 (3R - h)$$

And the wear rate of the static partner is thus:

$$WearRate_{Partner} = \frac{V}{L.Fn}$$

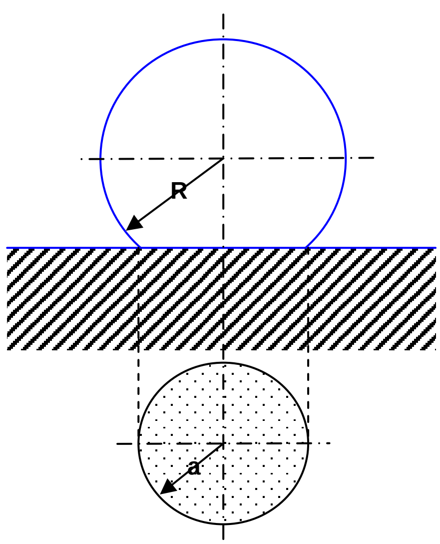
It may also be of interest to image the worn track with a high resolution technique such as confocal microscopy:



//// *Hertzian Contact Stress*

The maximum contact stress between the static partner and the sample can be calculated from the test parameters. If a ball of radius, R, is in contact with a sample under an applied load, F_N, then the contact area radius, a, is given by:

$$a = \sqrt[3]{\frac{3F_N R}{4E^*}}$$

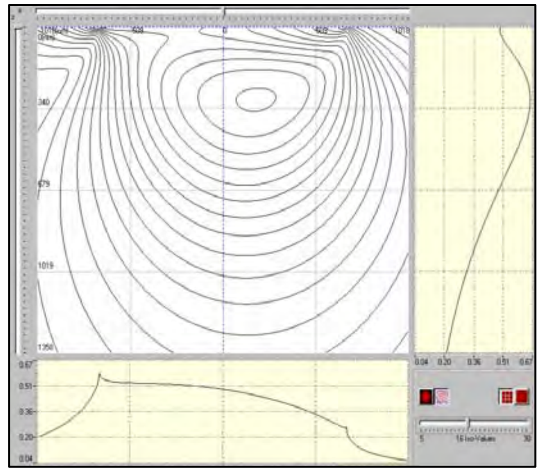


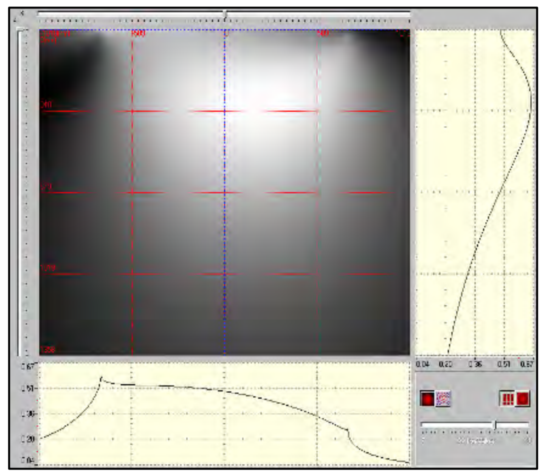
The equivalent Young's modulus, E* is given by:

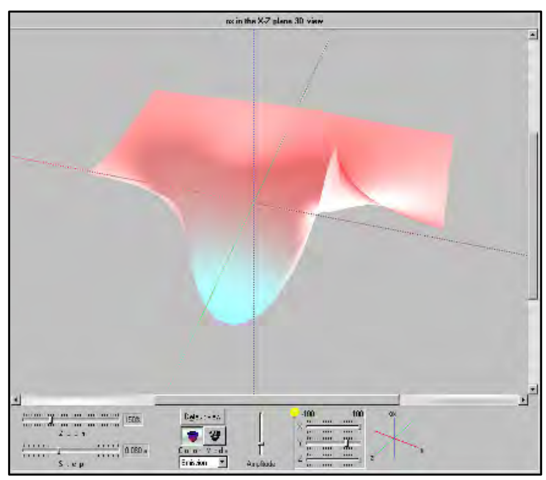
$$E^* = \frac{1}{\frac{1-\nu_{Sample}^2}{E_{Sample}} + \frac{1-\nu_{Partner}^2}{E_{Partner}}}$$

Where v is the Poisson's ratio and E the Young's modulus of the sample and static partner.

The distribution of stress at the contact can be modelled by plugging the relevant parameters into the TriboX software:



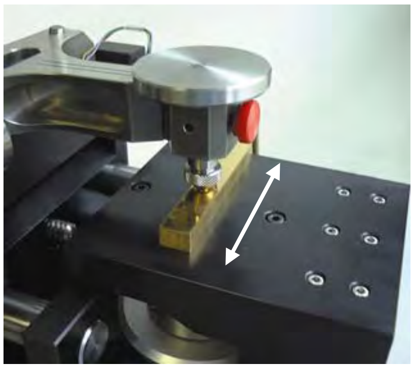




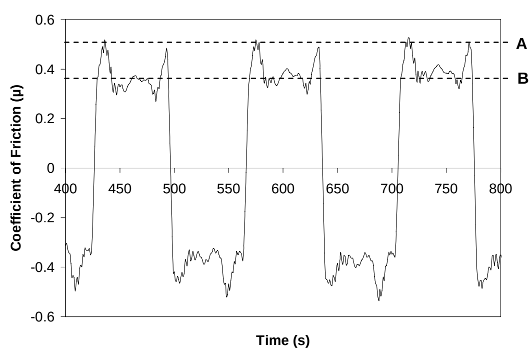
Such modelling allows the user to tailor the contact conditions of the test using a judicious choice of parameters such as applied load, radius of static partner, speed, etc.

//// *Linear Reciprocating Method*

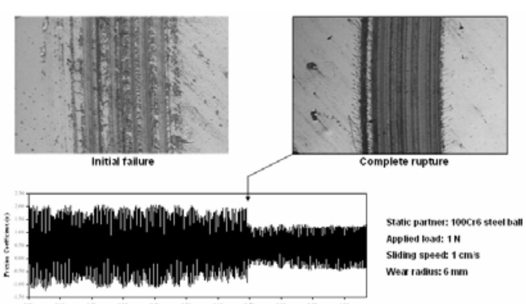
The linear reciprocating module reproduces the reciprocating motion typical of many real world mechanisms, by translating rotational motion into linear motion. This allows a friction coefficient to be measured for both the forward and backward displacement of the stroke. The reciprocating technique is also very useful for studying the variation over time of the static coefficient of friction.



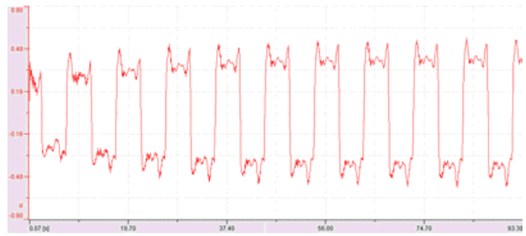
The data from a linear reciprocating test can be displayed in several different ways. The example below shows a test where the transition between the static coefficient of friction (A) and the sliding friction (B) is being investigated over a number of reciprocating passes:



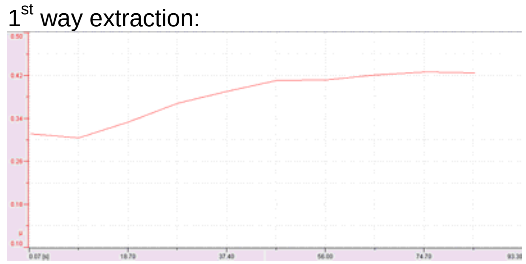
Coating integrity may also be evaluated by performing repetitive linear reciprocating passes over the same track until coating failure occurs:



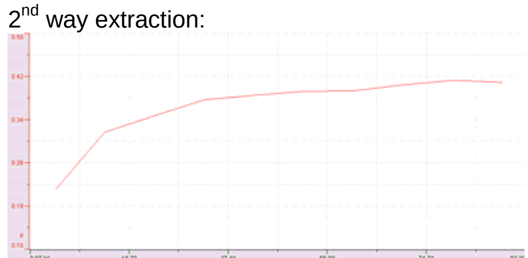
Data can be extracted from a linear reciprocating plot in several ways. Any position can be chosen along the wear track and the coefficient of friction extracted at that point in the forward direction (1st way extraction), the backward direction (2nd way extraction) or the average of both (2 way extraction):



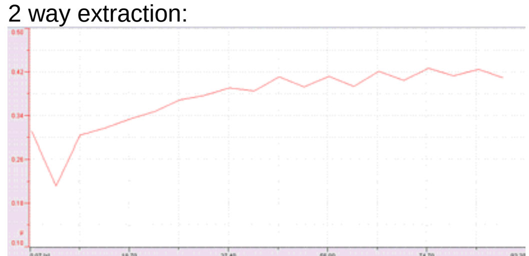
1st way extraction:



2nd way extraction:



2 way extraction:



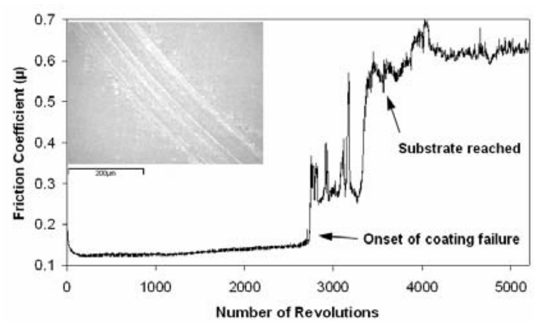
This technique can be very useful to determine the friction coefficient at various points along the forward/backward cycle. It also allows a correlation between friction coefficient and speed as the sliding speed will vary from zero at the test extremities to a maximum value in the middle of the wear track.

The sample wear rate and the static partner wear rate can be calculated from a linear reciprocating test in exactly the same way as for a rotating test. The principle standard for linear reciprocating is ASTM G133.

//// *Liquid Heating Option*

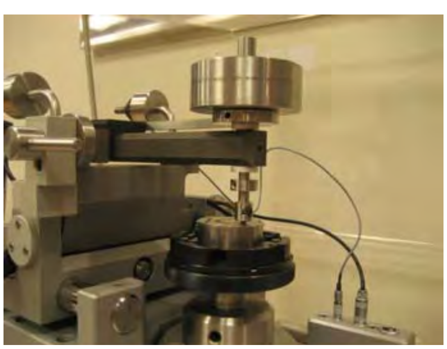
The liquid heating option allows the sample to be completely submerged in a liquid and a heating element allows the liquid to be maintained at a predefined temperature.

A good example of such testing might be the simulation of human body conditions: the example below shows the result of a test between a Tin oxide coating on a prosthetic joint (sample) mated against a polymer pin (static partner). The materials were submerged in Body Mimicking Fluid (BMF) maintained at 37°C.

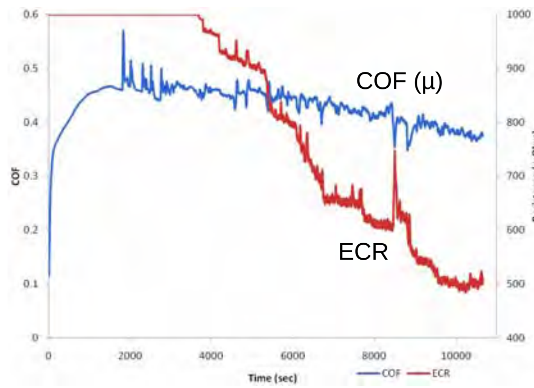


//// *Electrical Contact Resistance (ECR)*

The electrical contact measurement is based on an electrical connection between the grounded sample and a 6 mm ball holder insulated from the rest of the Tribometer arm. The system measurement range is 0 - 1000 Ohm



This module could be used to evaluate the breakdown in an insulating coating deposited on a conducting substrate. The example below shows results for a polypropylene (PP) coating on a steel substrate. The goal of this test was to detect the failure of the PP layer and see if there was a corresponding change in the coefficient of friction:



The above graph represents a comparison of the coefficient of friction (COF) and the electrical contact resistance (ECR) as measured in mOhm plotted versus time. Note that the change in contact resistance does not correlate to a change in the COF.

//// *Continuous Wear Depth Measurement*

This option allows the depth of the pin or ball in contact with the sample to be continuously monitored during a Tribometer test. The wear depth measurement records the vertical displacement of the arm during the test.

The RVDT sensor provides a resolution of 1 µm over a maximum displacement range of +/- 2 mm

Some care must be taken when interpreting the wear depth data plot as three different wear situations might occur for a ball-on-disk contact situation, for example:

a) only the ball wears;
b) only the disk wears;
c) both ball and disk wear

In the cases a) or b), the estimation of the wear rate according to the depth measurement is straightforward. For case c), the estimated wear rate must take into account the wear of both ball and disk. A stylus profilometer may be additionally used to estimate the worn volume of the sample.