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ERDF Nitropep

Tribo-simulation of surface wiping

Compiled By
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1 Revision History

Revision	Prepared by	Checked by	Approved by	Comment
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2 Aims & Objectives

2.1 Aim

Nitropep have produced a novel surface modification technology which can provide a treatment to ensure metal surfaces can kill bacteria. Changes have been made to previous formulations removing peptides, and with it fluorescent markers. As a result, significant surface investigations on the latest formulations to establish its stability and robustness, as well as it durability needs to be carried out. Therefore a need to measure and compare the performance of a coating under dry and cleaning lubricating conditions, in a wiping motion with that of a non-coated substrate is conducted.

2.2 Objectives

Samples were provide from Nitropep, with and without the coating. This requires an initial investigation on the methodology to provide a closest form of cleaning wipe processes replication on the available equipment of the UMT. Various set-ups and contact between the counter faces were also investigated to provide a substantial data. The testing were conducted under dry and lubricated conditions with detergents to access the friction of the coated and uncoated samples, and surface characterisation conducted to address the effect of contact.

- Perform a literature review on the parameters and motion of wiping the surfaces, and the most appropriate test methodology.
- Establish a methodology and procedure to replicate the wiping of the surfaces with a cleaning cloth on the UMT Tribolab.
- Perform a surface analysis on the coated and uncoated samples to analyse the surface metrology before and after the experiment.
- Measure the friction coefficient of the interacting surfaces of the coated/uncoated substrate and the cleaning cloth under wiping motion, and possibly measure the wear as well.
- Present a comparative analysis of the results with a report.

3 Wiping the surface

Surface cleaning refers to the removal of dirt, dust or other debris from the surface of an object and the bona-fide method of cleaning the surface has always been with the use of a cloth to remove these from the surface. Recently, various cleaning agents and surface treatments are also used which cleans and prepare surfaces by interacting or reacting with contaminants and altering their properties according to their functions. Cleaning agents provides common functions of liquid cleaners, degreasers, etchants etc. and surface treatments provides protection to the surface including antibacterial properties. However, one thing remains in common between the process and the treatments is the act of rubbing or wiping.

In order to achieve the spread of various cleaning agents, and also to remove dust, dirt etc. from the surface, an act of wiping remains crucial in how the cleaning agents or the surface treatment functions. Therefore, it is crucial to understand how the interaction takes place between the surfaces and the motion of the wiping as well.

Tribology is the science and technology concerned with the ‘interacting surfaces in relative motion’ focusing on the fundamental investigations of friction, wear and lubrication. A typical tribology test overcomes the lack of understanding in friction and wear, with a common approach to measure them in a laboratory experiment. Therefore, in order to design a test around tribological approach, various factors needs to be considered.

Verkouteren et al [1] provides a bases for surface wipe sampling to trace narcotics and explosives collection and stated in general, no overwhelming consensus had been drawn from the current literature on how to collect a wipe sample. However, they list the common factors to be considered and derived as below –

- Wipe material
- Solvent
- Applied force
- Sampled area
- Surface characteristics
- Physical/chemical nature of the contaminant

Verkouteren et al [1-3] has also displayed various parameters of the testing conditions of wiping and the parameters discussed further in Section 6 (Experimental Procedure).The average surface wipe sampling force was listed in a range of 7 to 10 N [1-3] and similarly listed the rubbing speed between 50 mm/s to 400 mm/s [2 – 3].

4 Experimental Procedure

4.1 Pre-surface characterisation

Initially, before wear testing the surfaces of the coated and uncoated samples were characterised using White light interferometry, Raman and also by scratch testing on the samples. These are discussed and results presented in Section 7.1

4.1.1 White Light Interferometry (WLI)

White Light Interferometry is a non-contact measurement technique, which uses a broadband light source and combines vertical (z-axis) scanning techniques with optical interferometry techniques, to achieve a three-dimensional (3D) surface measurement. The WLI provides a range of data from 2D TO 3D surface measurement, providing quantified information on surface topography such as roughness and waviness along with surface profiles and imaging. The instrument used was a Bruker ContourGT (Figure 1).

Figure 1. The Bruker ContourGT-K series 3D Optical microscope.

This is a bench top device utilising an automated motorized XY sample stage. Images are taken through at magnifications of between 1.375 to 100 times the original and has a Z-axis resolution of 0.1nm.

4.2 Tribo-testing

4.2.1 UMT Tribolab

The UMT Tribolab platform is a versatile tribology and mechanical test system. It offers a range of versatile modularity which translate into linear and rotary motion to provide a full range of rotary, reciprocating, block-on-ring, and linear tribology test configurations, accommodating range of speeds, torque and force measurement, along with a wide range of temperature testing.

The system consists of precision positioning system in the X and Z axis, as well as various optional Y and axial motion systems axis to control the location and loading force of the upper or lower test specimen. The Z axis (Carriage), X axis (Slider) and Y and axial motion systems axis can be controlled and monitored through the UMT Operating software, **Error! Reference source not found.**.

Figure 2. The Bruker UMT Tribolab

4.2.2 Methodology development

Following on the factors listed by Verkouteren et al [1], parameters regarding the appropriate factors needed to be developed and factored into the design of the wear test. To simulate the motion of wiping, a reciprocating module was chosen to replicate the motion of cleaning the surface.

The reciprocating drive (Figure 3) has a built-in LVDT positioning sensor which moves in a back and forth motion to simulate an oscillating linear motion, with speeds up to 60Hz stroke from 0.1 mm to 25 mm and is designed to conduct ball-on-plate, pin-on-plate, and abrasive testing.

Coated and uncoated plate samples were placed on the reciprocating drive and moved in a linear oscillating motion. The upper assembly holds the wiping material and was static. The wiping material was chosen to be a general purpose cloth (Figure 4) which served for a general cleaning process.

Figure 3. The reciprocating module.

Figure 4. General Purpose cleaning cloth

Figure 5 shows the initial set-up of the interaction between the sample plate and the wiping cloth. Although this methodology covered a larger surface area, due to the method of screwing the cloth onto the flat levelling sample assembly resulted in loosening of the cloth threads and hence resulting in variation among the friction data.

To maintain constant repeatability between the contact surface and the wiping cloth, the wiping cloth was inserted into a ball holder with 4 layers of cloth pressed by a steel ball. The radius of the ball maintained constant surface area in contact between the coated/uncoated sample and the wiping cloth, with significant repeatability between the samples.

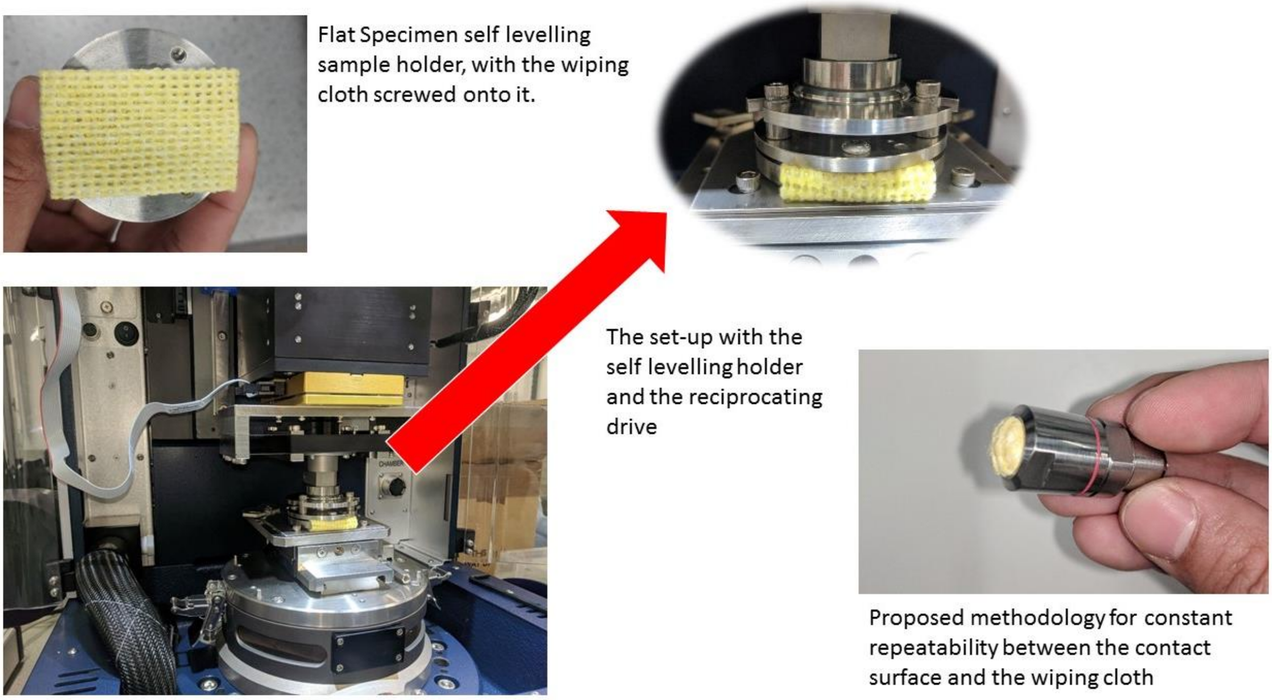


Figure 5. Initial set-up for the wear testing of the coated/uncoated samples with the cleaning cloth in a flat-specimen holder and the proposed methodology with the ball holder.

With the methodology development mentioned in section 5, wear testing were conducted in a reciprocating motion to replicate the wiping of surface with the parameters listed in Table 1 below. There were 8 variations of the testing conducted based on coated and uncoated samples under dry or lubricated conditions and with two speeds of 50 and 400 mm/s.

Table 1. Parameter and factors listed for various test conducted with the UMT Tribometer.

		Load (N)	Distance (mm)	Speed (mm/s)
Uncoated	Dry	10	78000	50
		10	78000	400
	Lubricated	10	78000	50
		10	78000	400
Coated	Dry	10	78000	50
		10	78000	400
	Lubricated	10	78000	50
		10	78000	400

According to Verkouteren et al [1 – 3] the average surface wipe sampling force was listed in a range of 7 to 10 N. Hence, a max averaged force of 10 N was adapted to conduct the tribo-test to replicate average rubbing force. Similarly, they also listed the rubbing speed between 50 mm/s to 400 mm/s [2 – 3] and hence the lowest speed of 50 mm/s and 400 mm/s was chosen. Apart from the load, the distance of rubbing was also kept at a constant length of 78000 mm. Each of the test were conducted with three repeats. The wear tracks were re-analysed with the White light interferometry.

5 Results and Discussion

5.1 Pre-characterisation of the sample surfaces

In order to chemical characterise the sample surfaces, the samples were analysed with Raman spectroscopy.

Raman Spectroscopy is a characterisation and analytical technique based on an inelastic scattering or Raman scattering of monochromatic light. This provides information about vibrational, rotational and other low frequency transitions in molecules, which are unique to a molecule and individual molecular structure. It is a technique that can be used for the analysis of solids, liquids and solutions and can provide information on the physical characteristics of the molecule.

The Raman spectra of various peptides have been reported in several literature, specifically identifying the amide bonds [4].

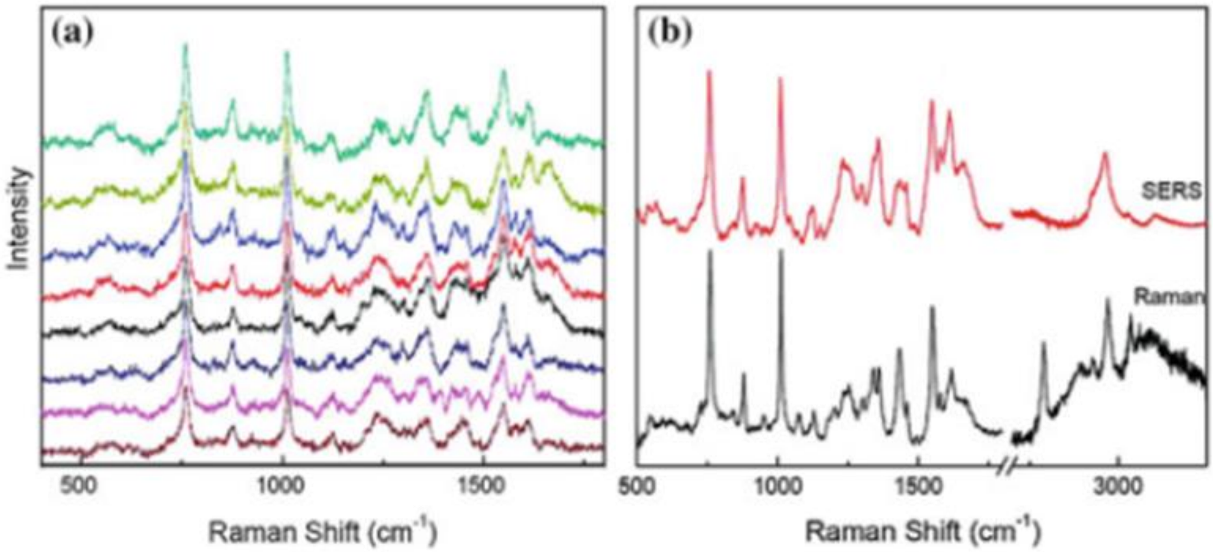


Figure 6 . Intrinsic SERS spectra of peptides (a) SERS spectra of Try-Cys (b) Comparision of SERS with NRS spectra (adapted from Wei et al. 2008) [4]

To indicate the presence of Nitropep coatings on the substrate, Raman analysis were undertaken with the Renishaw 'Qontor' Raman confocal microscope. Initial experiments were conducted to obtain a reasonable response of the material from the sample. Various combinations of the lens objective, laser wavelength (532nm and 633nm), accumulation and the exposure time were applied.

No immediate response of the peptide coatings were observed, unlike as mentioned in the literature. Responses from the substrate steel, as shown in the graph (Figure 7), were observed for both samples with and without the coating. No response of the peptide bonds were observed on the Raman analysis.

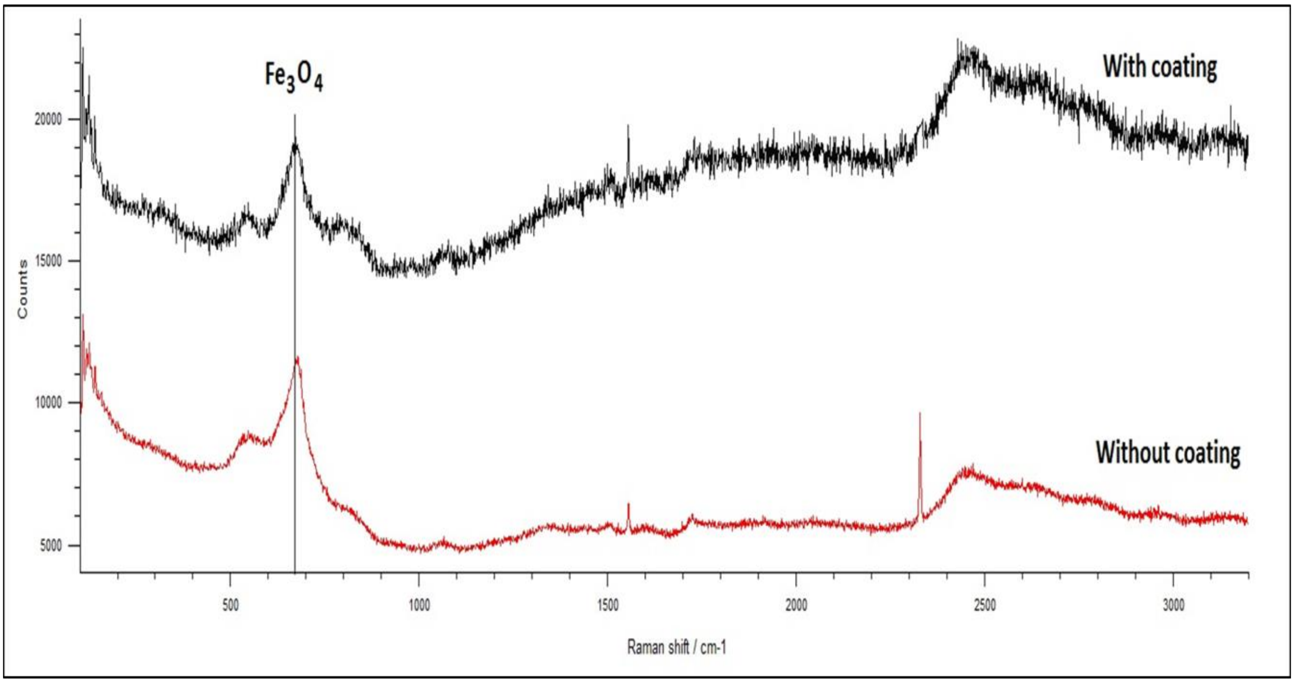
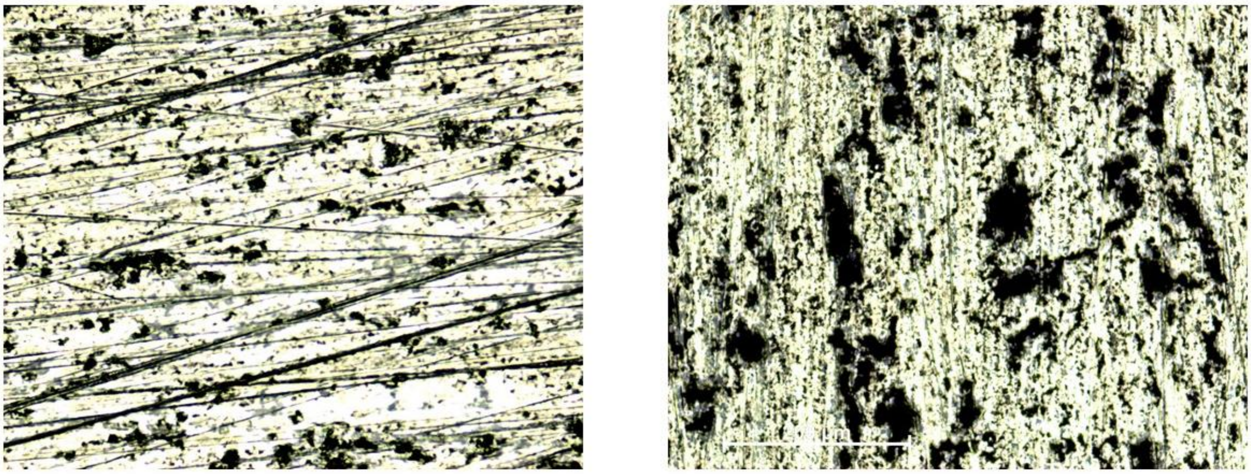


Figure 7. Raman spectra observed from the coated and uncoated samples.

Peptides Raman response, as indicated in the literature have been utilised with SERS (Surface enhanced Raman spectroscopy). Similarly, the penetration depth of the current Qontor Raman is listed as in the range of nm, whereas the peptide coatings is mentioned to be in the range of Angstrom (Å). Hence, a more sophisticated range of surface analysis which covers coating thickness in the range of Angstrom (Å) needs to be considered to analyse the coating chemically. Further investigation was deemed to be outside the scope of work.

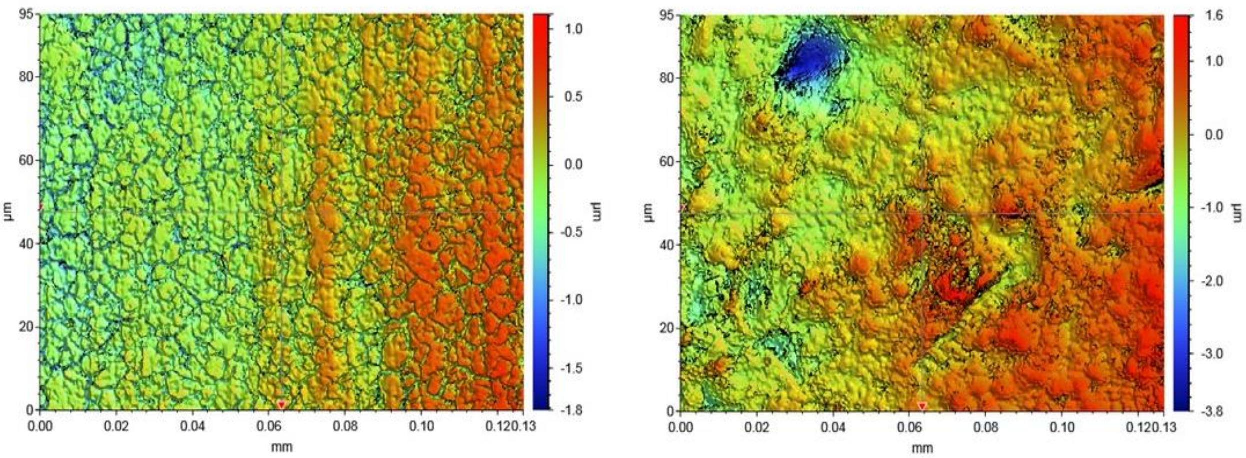
The samples were also observed under the Optical microscope to initially characterise the surface. Figure 8 presents the uncoated and coated sample with a magnification of 20x over an area of 500 x 500 µm. A subtle difference cannot be observed however, the coated samples seems to cover some darker areas compared to the uncoated samples.



(a) (b)

Figure 8. Optical Bright field images of the (a) uncoated samples and (b) uncoated samples.

Further analysis conducted with the White light interferometry shows a difference in the surface topography between the coated and the uncoated samples (Figure 9). The uncoated samples showed features appearing like small flat islands across the sample surface. The coated samples had a very different surface structure, the scaly, island like appearance was not present being replaced with a more continuous but undulating surface.



(a) (b)

Figure 9. White light interferometry images over an area of 100 x 100 µm of the (a) uncoated samples and (b) uncoated samples.

The average roughness value measured with the WLI showed the uncoated samples to be smoother in comparison to the coated samples (Figure 10). The higher average roughness nature of the coated samples could be attributed to the undulating structures on the coated surface. At higher magnification the coated samples also showed pit like structures across the area, which was attributed as to the cause of the higher average roughness value (Figure 11).

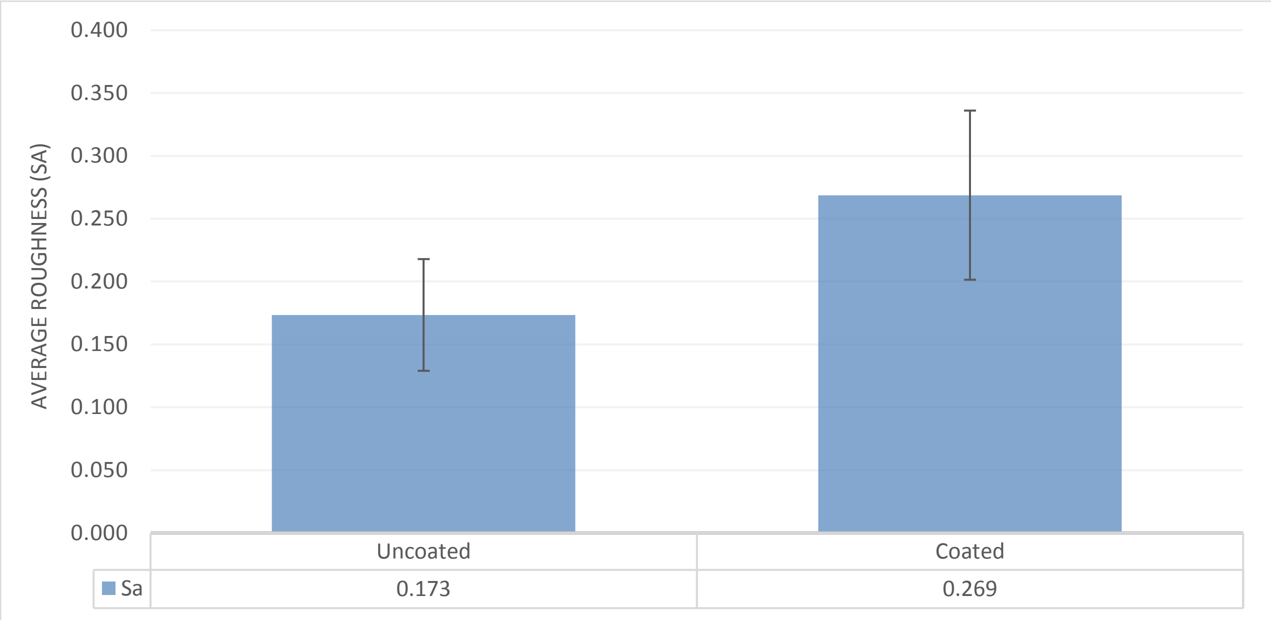


Figure 10. Average roughness value of the uncoated and coated samples.

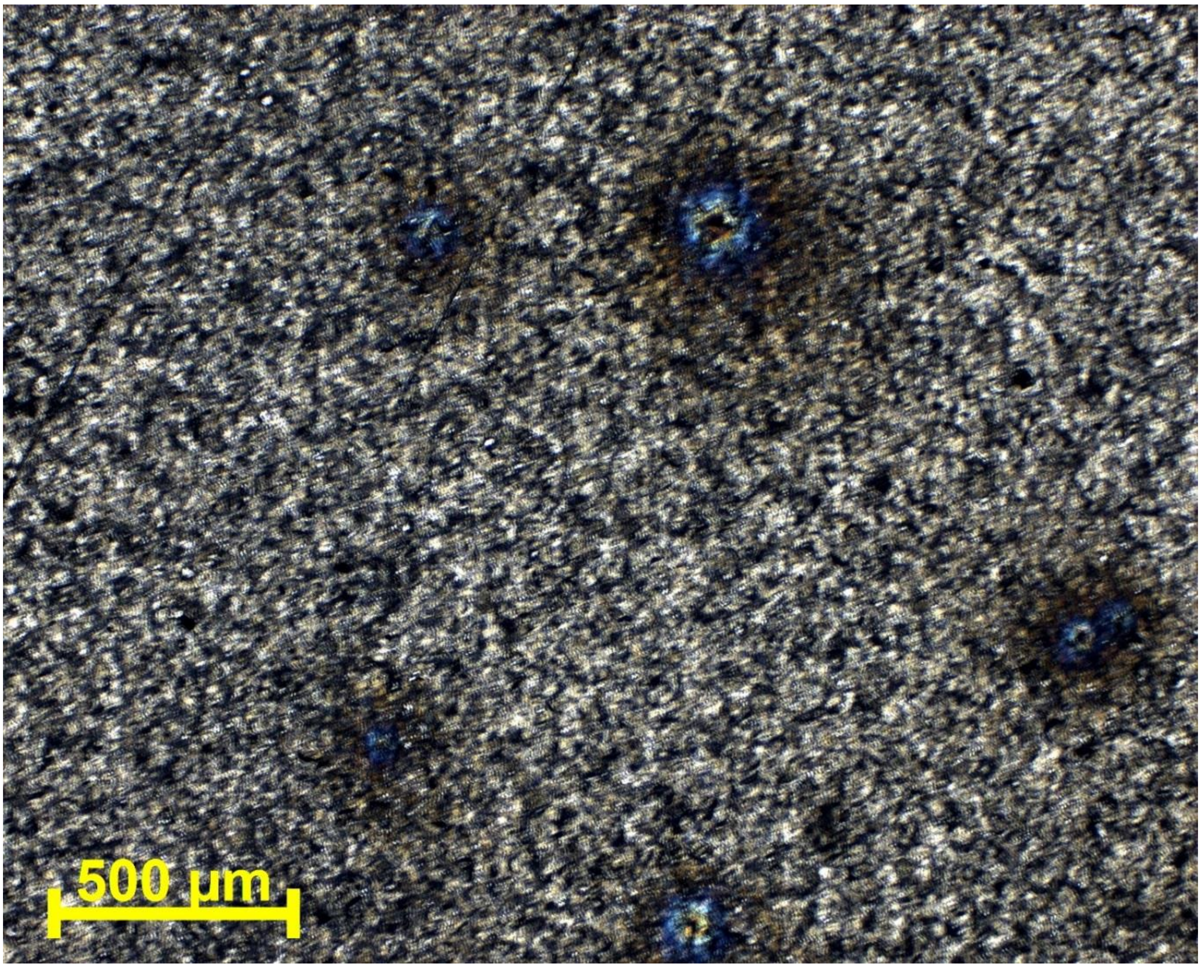


Figure 11. Image showing pit like structure around the coated samples.

5.2 Tribo-testing

Figure 12 shows the overall test result with three repeats for the coated and uncoated samples, under dry and lubricated conditions with varying speeds of 50 mm/s and 400 m/s. In terms of repeatability, all of the samples follow a similar trend except for the coated samples under dry conditions and speed of 50 mm/s.

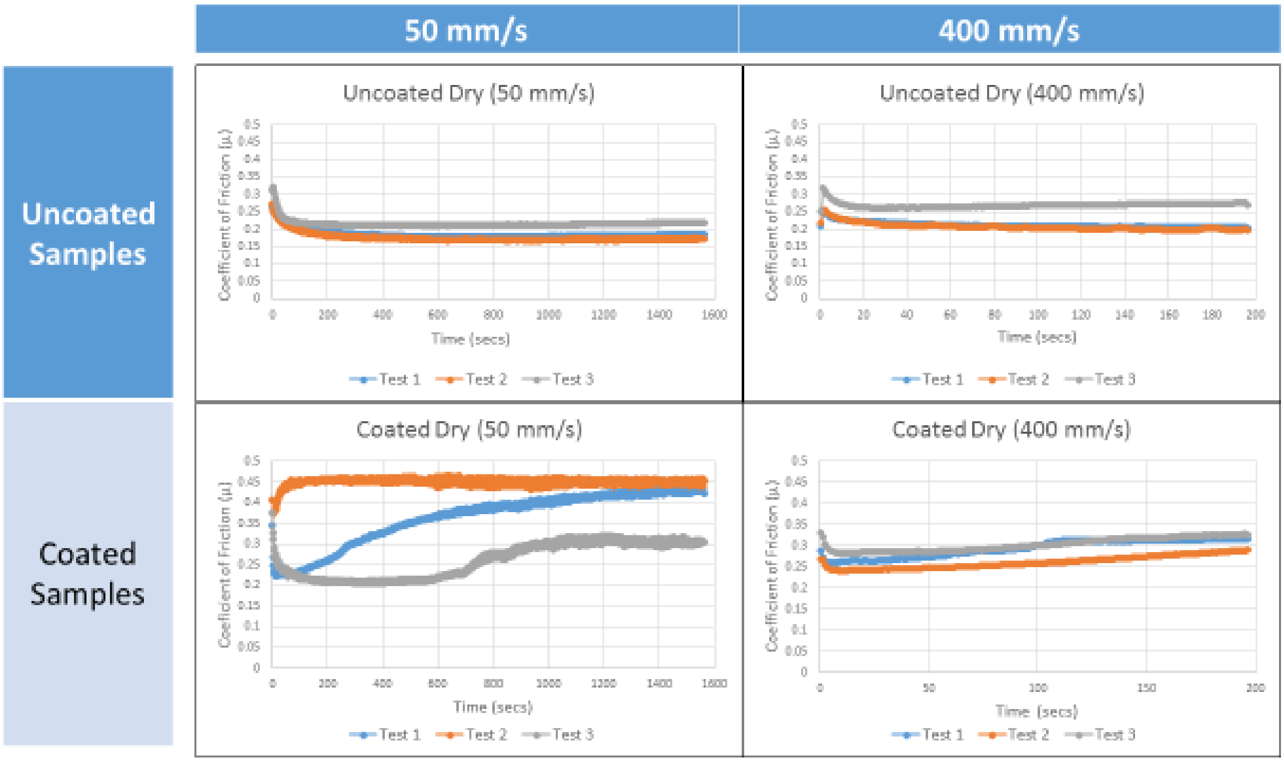
The varying factor of friction can be attributed to the rougher area of the coated samples as described in the previous section. Friction test for each repeats were conducted across different areas of the samples meaning that there could be variability across the samples. In addition, the slow speed of 50 mm/s exposes a longer contact duration between the interacting surfaces, which could then exacerbate any differences. The contact area exposed also will depend on the pits observed in the previous section. However, at the faster speed of 400 mm/s the contact duration was minimal and the energy required to overcome the friction was minimal as well.

In order to further analyse the friction behaviour of the samples under various condition, the last minute averaged friction data was produced and compared below in Figure 13. Under dry conditions for high and low speed, the uncoated samples showed the lowest friction overall. Due to the direct contact, the asperities in the uncoated surfaces were breaking down easily creating a smoother surface, developing high conformity between the interacting surfaces. For coated samples, high contact speeds seem to influence low friction under both dry and lubricated conditions.

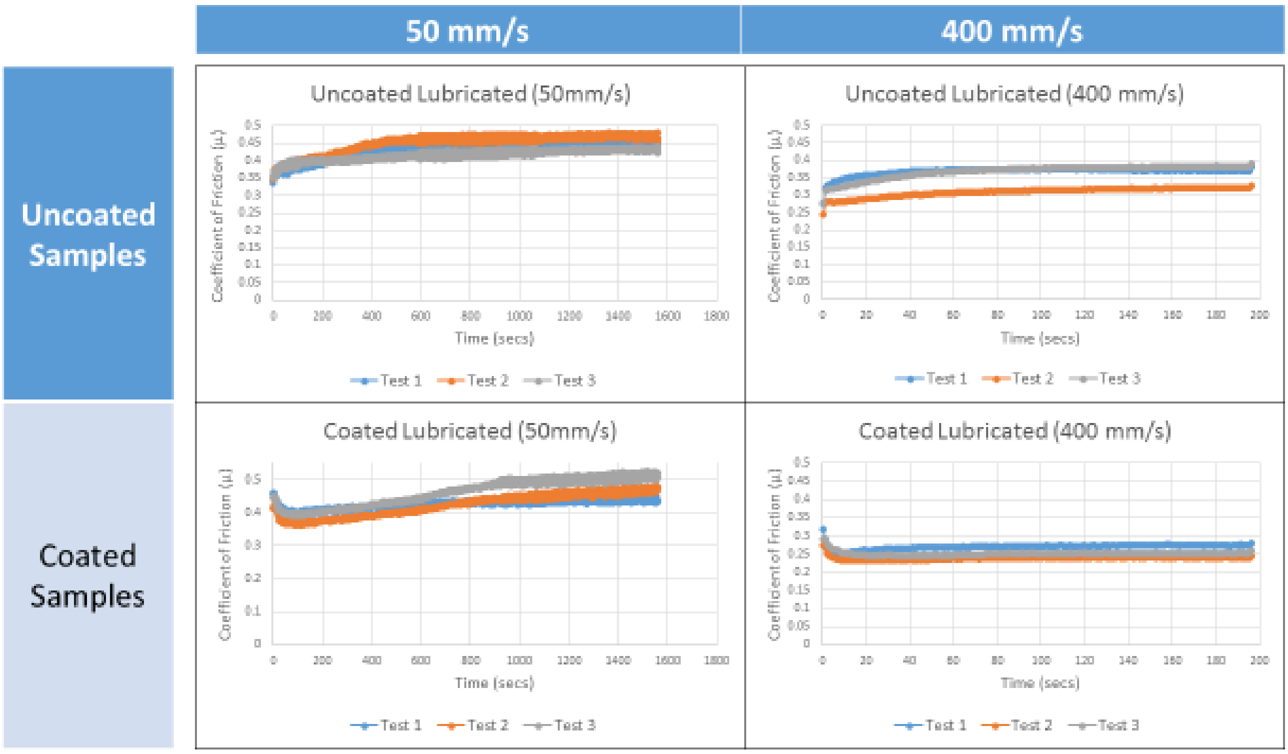
Lubrication was ineffective in most of the conditions for friction values, except for the coated sample at high speeds. The influence of lubricant was significant for the coated samples when high speed was applied, as it was the only condition among the tests, where the friction value for lubricated conditions was lower compared to that of the dry conditions. This showed that the lubricant was effectively superimposed on to the surface of the coated samples at high speeds.

Lubrication does not succeed in successfully separating the surfaces, as the surfaces will be undergoing boundary lubrication at the chosen parameter. Under boundary condition, even with the presence of lubricant the contact acts as if in the dry condition, since the loading between the contacts is high. The other reason could be the cleaning cloth under lubrication gets softer due to expose of the lubricant and soaking the liquid between the threads of the cloth, therefore requiring more energy to overcome the friction under motion.

Under Lubrication and low speeds, highest friction value was observed for both uncoated and coated samples. At low speeds apart from requiring more energy to overcome the friction under motion due to the cloth soaking the liquid, a stick and slip boundary condition could possible occur due to the longer duration of contact area exposure.



(a)



(b)

Figure 12. Friction coefficient of coated and uncoated tribo-testing (3x repeats) under (a) dry and (b) lubricated conditions, with varying speeds of 50 mm/s and 400 mm/s against time.

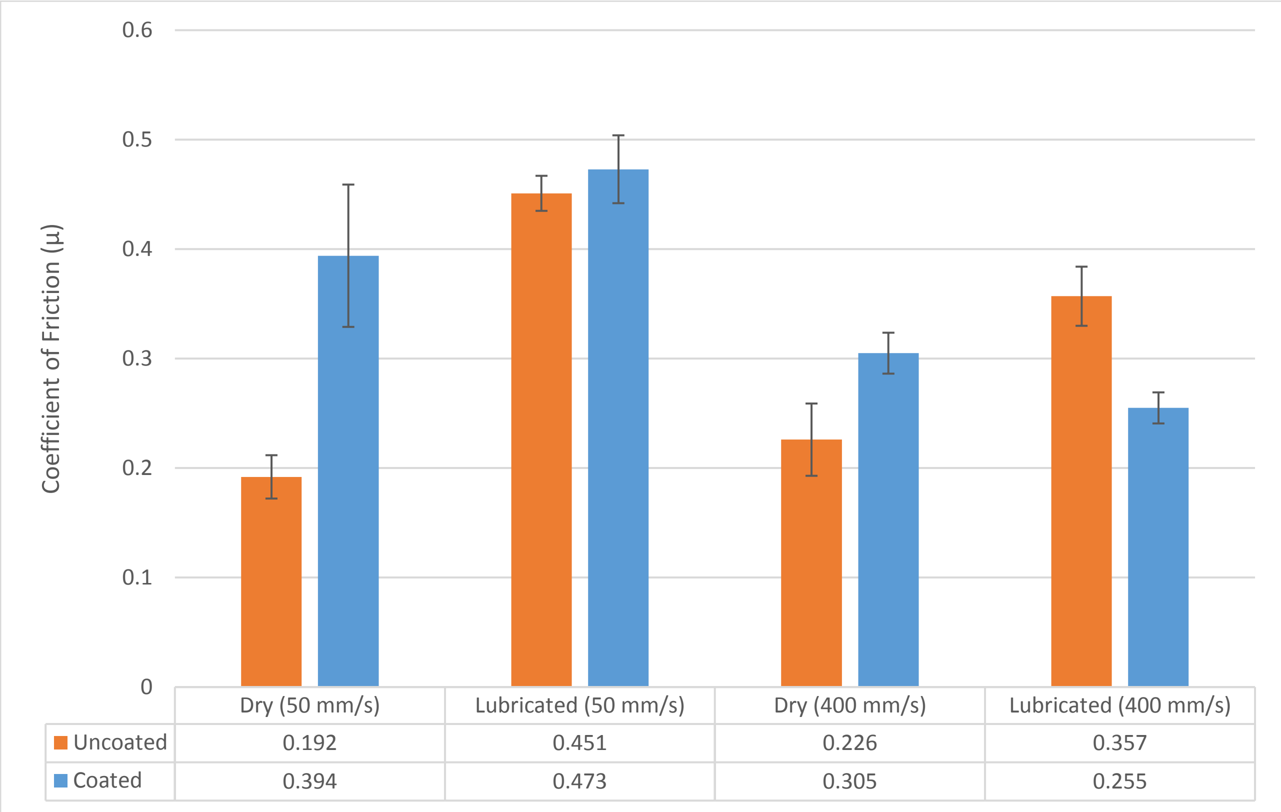
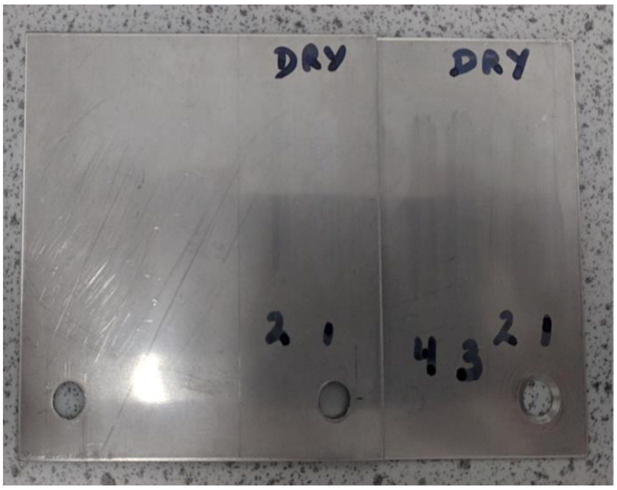


Figure 13. Last minute averaged friction of the coated and uncoated samples under dry and lubricated conditions and speeds of 50 mm/s and 400 mm/s.

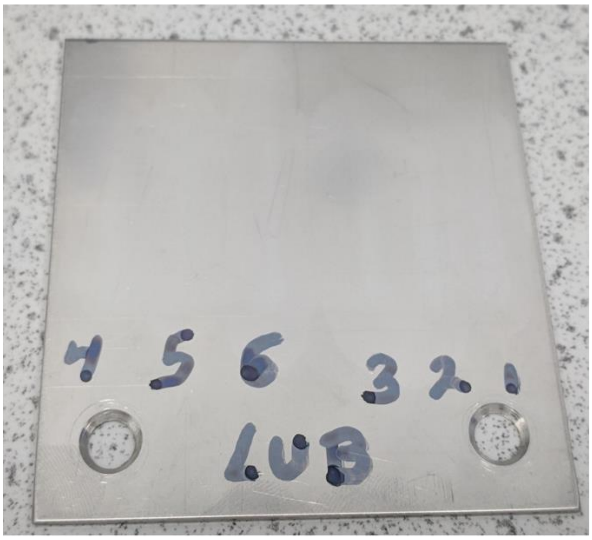
Optically observing the wear area, a minimal wear could be observed right away. Figure 14 shows an optical comparison of the various wear areas and although the wear area can be almost observed, with the data provided in Figure 15 from the white light interferometry analysis, the exposed area seems to have been smoothed out. However, a depth analysis of the wear area was almost absent.



Uncoated

Coated

(a)



Uncoated

Coated

(b)

Figure 14 (a) Wear area of the coated and uncoated samples under dry conditions with 1, 2 and 1 (L to R) under 50 mm/s and 2, 4 and 3 under 400 mm/s. (b) Wear area of the coated and uncoated samples under lubricated conditions with 3, 2 and 1 (L to R) under 50 mm/s and 4, 5 and 6 under 400 mm/s.

When looking at the images presented in Figure 14, the appearance of the coated and uncoated samples should be considered. Those that have gone through the coating process appear to have a mottled appearance. It is unclear as to the cause at this time.

Figure 15 shows the comparison for the averaged roughness value for the coated and uncoated samples, before and after the test under various conditions. In terms of the roughness value, after test the samples do not vary significantly from the pre-test data. A slight drop in the averaged roughness value can be observed, which signifies that the contact area smoothed out during the wear. No samples gave significant wear.

A longer duration of test could be conducted to observe higher wear rate values, however it is possible that the cleaning cloth might be going through a higher wear compared to the sample plates. The wear on the cleaning cloth was not measured due to the complexities involved with measuring it.

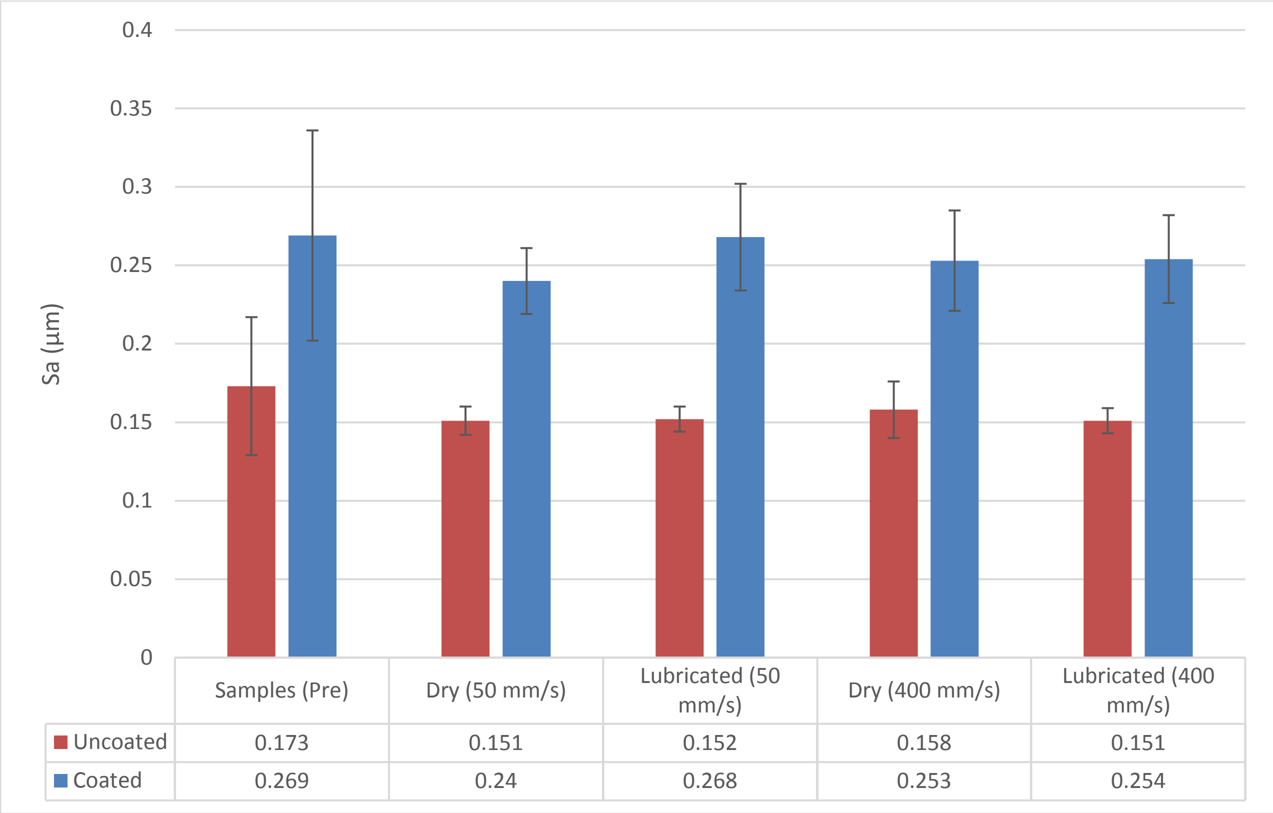


Figure 15. Averaged roughness (Sa) comparison of the uncoated and coated samples before and after test under various conditions.

6 Conclusion

- Raman analysis conducted on the coated and uncoated samples did not show any difference between the samples and could not identify the coating. The response from the coating substrate could be restricted by the thickness of the coating which in terms of Angstrom (Å). A more sophisticated surface technique such as SERS (Surface Enhanced Raman spectroscopy) could help to characterise the coatings, similar to the literature.
- Coated samples showed some darker areas optically in comparison to uncoated samples. A closer look into the uncoated samples showed region of pits spreading across the sample.
- With the aid of white light interferometry, the uncoated samples showed island like features over the sample surface, whereas the coated surfaces were more undulating, wavelike in appearance. The uncoated samples showed a higher value of average roughness value compared to the uncoated samples.
- Tribo-testing conducted on the samples under various conditions showed a high repeatability, except for the coated samples under dry conditions and speed of 50 mm/s. This was attributed to the slow speed of 50 mm/s which exposes a longer contact duration between the interacting surfaces and the presence of pit region across the sample surface.
- Under dry conditions for high and low speed, the uncoated sample shows the lowest averaged friction overall, due to direct contact the asperities of the uncoated surfaces are breaking down easily and getting smoother developing high conformity between the interacting surfaces.
- For coated samples, high contact speeds seem to influence low friction under both dry and lubricated conditions.
- Lubrication was ineffective in most of the conditions for friction values, except for the coated sample at high speeds where the lubricant was effectively superimposed on to the surface of the coated samples at high speeds.
- Lubrication does not succeed successfully in separating the surfaces as the surfaces will be undergoing boundary lubrication at the chosen parameter. The other reason could be the cleaning cloth under lubrication gets softer due to expose of the lubricant and soaking the liquid between the threads of the cloth, therefore requiring more energy to overcome the friction under motion.
- Under Lubrication and low speeds, highest friction value was observed for both uncoated and coated samples. At low speeds apart from requiring more energy to overcome the friction under motion due to the cloth soaking the liquid, a stick and slip boundary conditions could possible occur due to longer duration of contact area exposure.
- Wear on the after test samples showed an absent of the wear depth, similarly very minimal change in the average roughness. The slight drop in the average roughness, is attributed to the smoothing of the surface on the interacting surface.

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