# Project IND1705 Ultrafast Microprobes

## Deliverable D7

## Good Practice Guide – In-situ wear damage measurement using fast microprobes with integrated feed-unit

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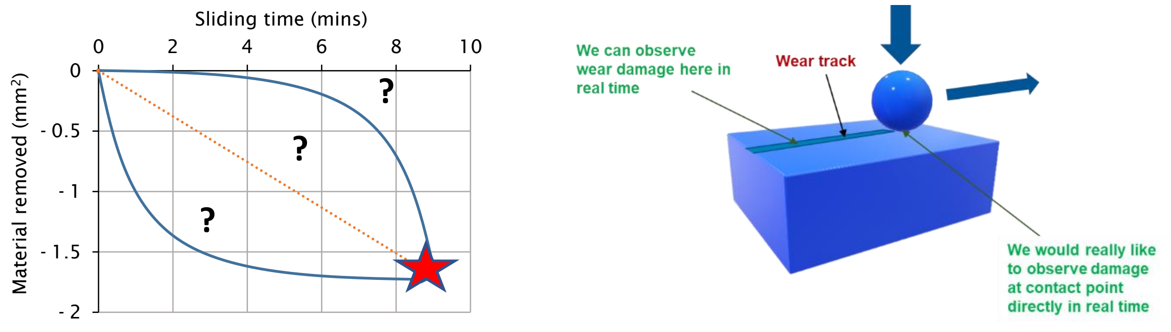
### Summary

This good practice guide describes how fast topographical microprobes can be used to measure the developing topography that takes place during tribological experiments. Fast microprobes were fitted to four different tribological test systems which were a scratch test system, a pin-on-disc wear testing system, a reciprocating tribometer and a microtribometer. Results of experiments carried out with the four different test systems are described and when appropriate are backed up by results from other analysis techniques. The development and use of a cost effective overload protector designed to protect the microprobes during use is also described.


### Introduction

Tribology is the study of the interaction of surfaces that interact whilst in relative motion. It encompasses the study of wear, friction and lubrication. The focus of this good practice guide is the introduction of fast microprobe technology for the in-situ measurement of wear. The reason that this is important is illustrated by Figure 1. Normally in a tribology test the volume of wear can only be evaluated at the end of a test. This gives an overall value for the material that is lost from the two contacting samples during the test, and is normally carried by mass loss measurements, change in dimensions or using profilometry. The issue with this approach is that no knowledge is available from the post test evaluation about the history of wear during the test. It is not known whether the wear rate changes during the test due to changes in the wear mechanisms that are occurring (illustrated by three different possible wear trajectories in Figure 1). This is key information that is needed to develop an understanding of the materials tribological behaviour.




a) b)

*Figure 1, Aspects of sliding wear testing, a) materials removal against time in wear test. The two blue and the orange paths could all be possible but cannot be determined from post-tests measurements made at end of test (red star), b) schematic showing contact between moving ball and flat in a wear test.*

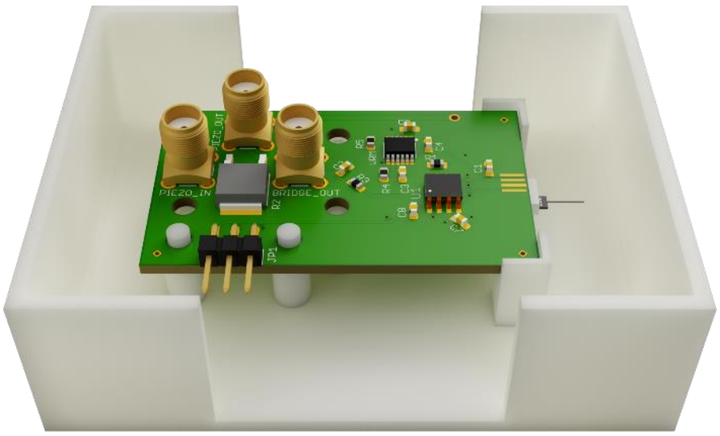
The concept introduced in this good practice guide is to show how fast contact microprobes can be introduced into tribological test systems to give in situ measurements giving information on the development of wear and any changes in wear rate during tests. Other non-contact alternatives do exist based on optical measurement systems. Digital holographic microscopy (DHM) can give excellent results capable of giving simultaneous images and height maps of the surface [1-3]. However, the DHM equipment is expensive, not that widely available and typically operates at frame rates less at 15 fps [1]. Chromatic aberration probes [4, 5] are also available that can give real time in situ information on the height of a surface relative to the probe. Single point probes only give measurements at a single point but can do so with sub micrometre resolution and at rates of about 180 kHz. Multipoint probes are also available which give complete profiles at rates of 18 kHz. These can be used to give complete real time in situ topographic maps of wear surfaces [5].

Although the tribological contact itself can only be observed if one of the contacting bodies is transparent, the wear to the surface that is not in contact all of the time can be observed away from the wear contact (Figure 1b).

The fast microprobes that are the subject of this good practice guide are based on cantilevers similar to those used in AFMs where the sensing element is a piezoresistive bridge that detects the bending of the probe when it contacts a surface. The application of these probes to four different tribological testing systems is described in the rest of this good practice guide.

## Microprobe Systems

The two different microprobe systems that were used are shown in Figure 2. Most experiments were carried out with the microprobe design from Technical Universiteit of Braunschweig (TUBS) and PTB (Figure 2a). For the experiments with the NPL microtribometer, a second microprobe design was used obtained from SCI Sensortech (Figure 2b).

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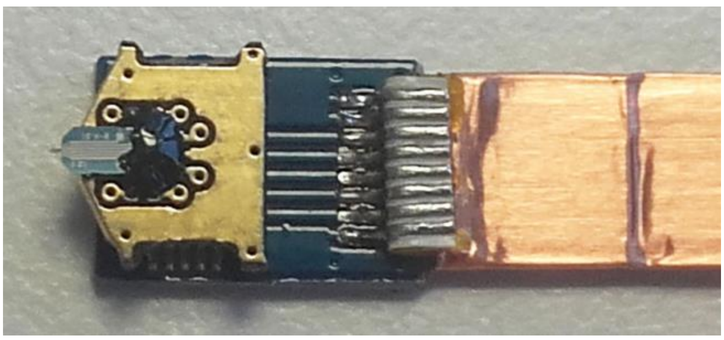

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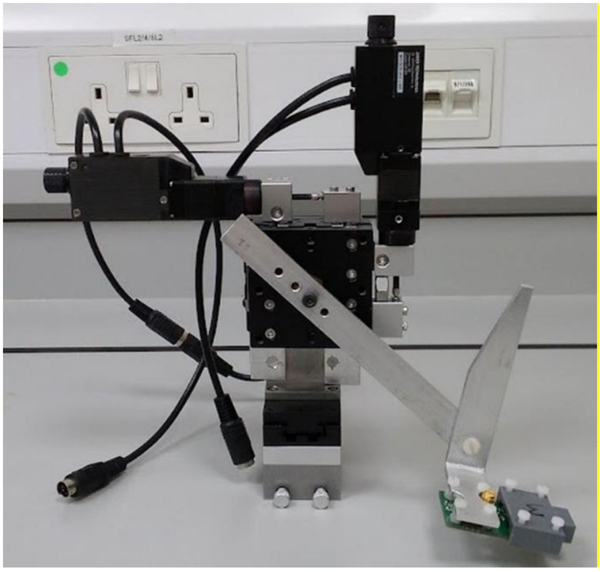
*Figure 2, microprobes used, a) TUBS / PTB microprobe mounted on pcb in carrier case. Note cantlilever projecting from edge of pcb, b) SCI Sensortech microprobe*

In both cases, the necessary electronics for operation of the microprobe systems was established so that an analogue voltage was obtained proportional to the displacement signal from the microprobe. This signal was fed into a National Instruments DAQ card to enable computer acquisition of the data from the microprobe.

The microprobe sensor systems were mounted into two different actuator systems for the two different probes (Figure3). Figure 3a shows the TUBS / PTB microprobe mounted in a two axis actuator system that was designed to give a vertical movement of 10 mm, and a horizontal movement of 10 mm when a profilometric scan was being made. The microprobe is mounted at an angle of 15°. Figure 3b shows the arrangement for the SCI Sensortech microprobe which is functionally similar, but was designed to fit with the NPL in situ microtribometer [6].



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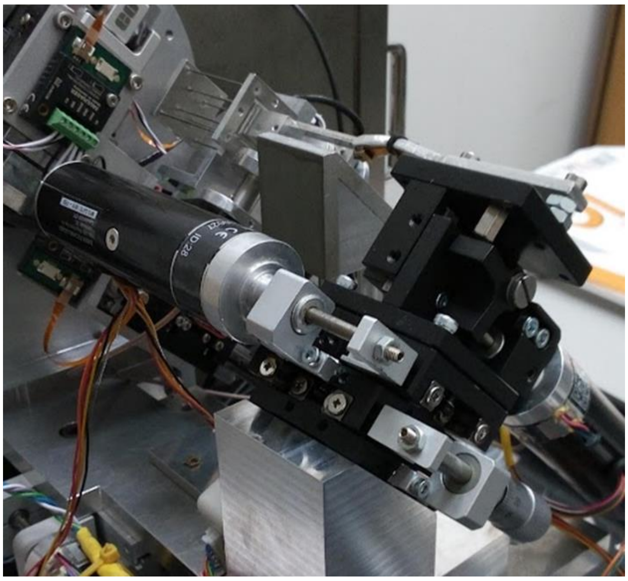

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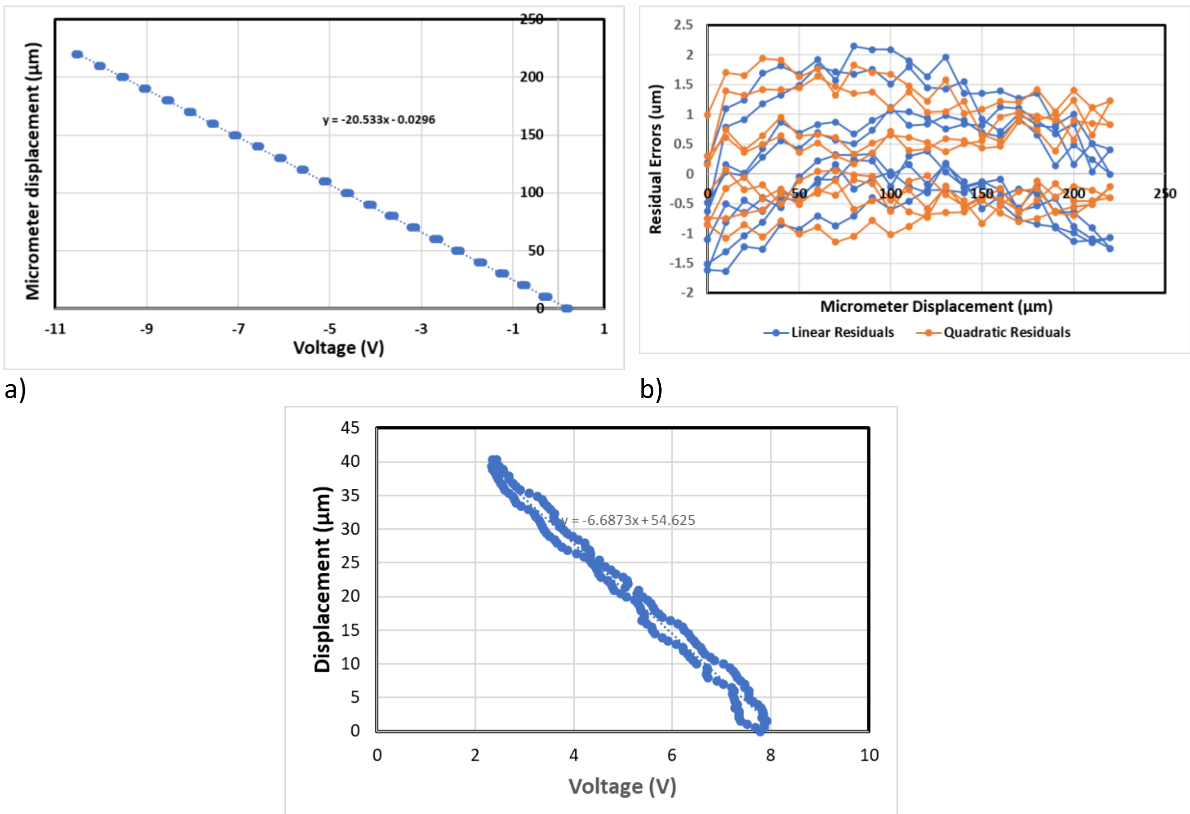
*Figure 3, Actuator systems for microprobes, a) TUBS/PTB microprobe, b) SCI sensortech microprobe.*

The TUBS / PTB microprobe was calibrated against a barrel micrometer that had previously been traceably calibrated. The cantilever was inclined at 15 degrees to the horizontal and a glass microscope slide was used as the contacting surface for the stylus. A typical calibration plot is shown in figure 4a. Repeated calibration runs were carried out and the data was fitted both linearly and quadratically. The residuals are plotted in figure 4b. It is acknowledged that the 'noise' in the residuals is most likely due to human error when reading the barrel micrometer (resolution of 1 µm), but the residuals of the linear fit make it clear that there is a small quadratic component in the voltage-displacement relationship of the microprobe cantilever.

The shorter length of the SCI Sensortech cantilevers makes their deflection range smaller, and hence makes them more susceptible to breaking when their small deflection range is exceeded. Since the calibration process for these shorter cantilevers is more delicate (and needed to be repeated more frequent through accidental damage), an automatic calibration routine was developed comparing the cantilever voltage output against the steps of the previously calibrated motorised actuator stage used to position it.

It is acknowledged that the yaw, pitch and roll of the positioning stage compounded by the length of the arm holding the microprobe against the sample surface produces an undesired "Abbe" error in the calibration (seen in the slight waviness of the plot), but this was accepted as it mitigates the risk of breaking the short cantilevers while attempting to calibrate them manually. The plot also shows the effect of backlash as the direction of travel is reversed, but there is sufficient data to obtain a reasonable calibration constant. There is no point plotting the residuals on a separate graph as they can be seen clearly already. These residuals are entirely attributable to the 'Abbe' error




a) b) c)

*Figure 4, Calibration curves a) TUBS/ PTB microprobe, b) residual plot from calibration of TUBS / PTB microprobe, c) SCIsensortech microprobe, d )residual plot from calibration of SCIsensortech microprobe.*

## Application to Tribological Test Systems

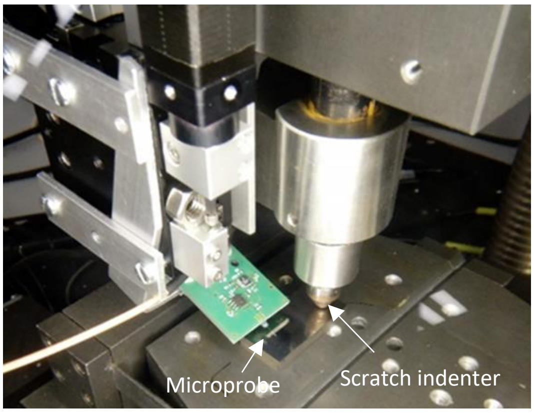
The microprobe systems were trialled on four different tribological testing systems. These were a scratch test system, a pin on disc test system, a reciprocating test system and the NPL microtribometer test system which can be used on the laboratory bench or within an SEM. The pin-on disc test system was also fitted with a multipoint chromatic aberration profilometry test system which enabled realtime profiles of the wear surface to be obtained during a test, and a linescan camera which gave realtime images of the wear surface during a test [7].

In most tests, the evaluation of damage was made by interrupting the test periodically through the test, making a profile measurement when all motion was stopped, restarting the test so that further damage occurred and repeating the cycle.

In one case with the pin-on disc test system a live test was carried out where the profilometric measurements with the microprobe were made while the system was still running.

### *Scratch Test*

The arrangement of the microprobe in the scratch testing system is shown in Figure 5a. In the test, the scratch indenter is lowered onto the sample (in this case aground 95 % alumina ceramic) and the sample traversed under the scratch indenter (Figure 5b). After the scratch has been carried out, the scratch indenter is removed from the surface and the sample is moved to the position where the microprobe scans a profile across the scratch. This cycle is repeated until the necessary number of repeats has been carried out. Figure 5c shows a higher magnification view of the cantilever of the microprobe close to the scratch on the pink sample.

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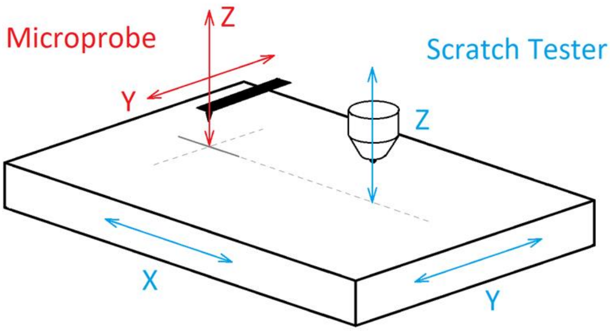


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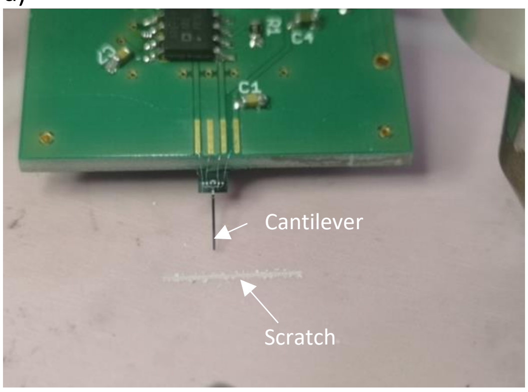


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*Figure 5, Microtribometer with scratch test system, a) test setup, b) schematic of testing arrangement, c) detail of microprobe and scratch to be measured*

Figure 6 shows quantitative results from the microprobe profile measurements. Figure 6a shows the raw profiles taken across the scratch. It should be noted that the surface of the alumina test sample was quite rough, as shown by the profile over the surface away from the scratch itself. The profiles were registered with one another by correlating the profiles in these regions away from the scratch. The progressive increase in the depth of the scratch can be seen clearly.

The profiles over the sample surface away from the scratch vary with the number of repeats. This is because there is an uncertinty in positioning of the microprobe for these scans which means that the different profiles are taken at slightly different positions.

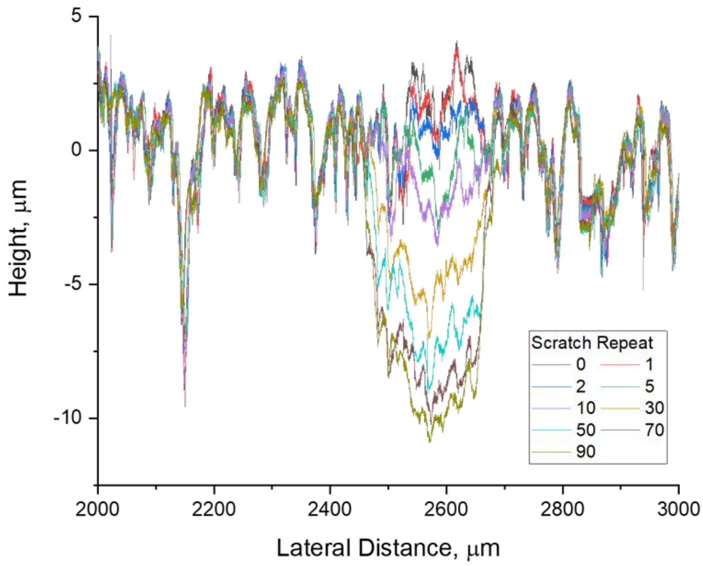
Figure 6b shows profiles across the scratch where the zero repeat scratch profile (the profile of the surface before any scratches) was used as a reference profile and was subtracted from the other profiles. There is some remnant variation in the profiles outside the scratched area due to the uncertainty in the position of the microprobe scan from one profile to another. The profiles across the scratch shown in Figure 6b were also zeroed by setting the average height of the profile away from the scratch to zero.

The area of the scratch can be found by integrating the area of the profiles. Figure 6c shows how the calculated area of the scratch increases with the number of scratch repeats. This provides good quantitative information on the degree of damage and its relationship to the number of scratches. This compares with the conventional method of monitoring how the damage increases with the number of scratch repeats which is to carry out a sequence of different scratches at different repeats which are individually measured after the scratching to determine the area of the scratch for a given number of repeats.

The surface of the indenter and the scratch itself were examined using an Alicona InfiniteFocus digital microscope. Figures 7a-c show the images of the scratch indenter tip and the scratch. The scratch indenter is quite rough and damaged. This resulted in the rough base of the scratch that was seen in the profiles and the images of the scratch. Figure 7d shows a comparison between profiles of the scratch taken with the microprobe at the end of the experiment, the profile of the scratch extracted from the Alicona height map, and the Alicona height map of the scratch indenter tip. The general form of all of these profiles is very similar.

### *Pin-on-Disc*

A schematic of the pin-on disc tribometer that was used to explore the utility of the TUBS / PTB microprobe for in situ measurement is shown in Figure 8. The system is fitted with an optical linescan camera which can be used to image the wear track in situ and in real time as it is formed, together with a multipoint chromatic aberration probe that forms in situ real-time non-contact profiles across the wear track [5]. The TUBS/PTB microprobe was mounted on the test system and orientated so that profiliometric scans could be made perpendicular to the wear track. The main experiment that was carried out was a rotating scratch test where a Rockwell 200 µm radius diamond indenter was pushed into a rotating disc of steel [6].

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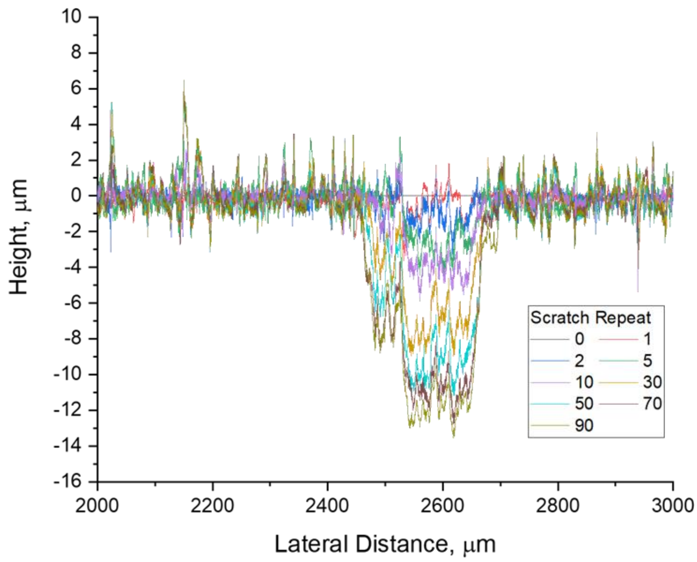


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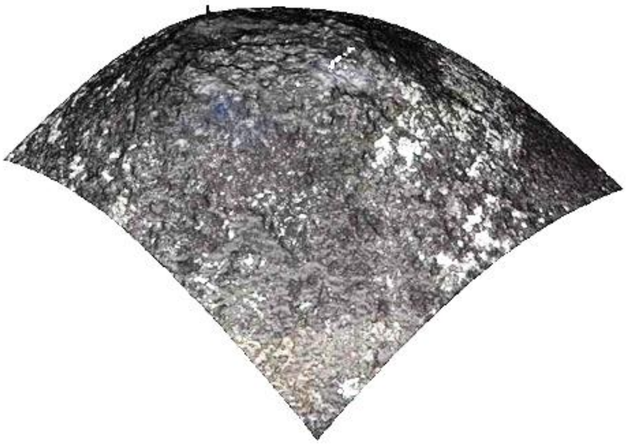
*Figure 6, Results from measurements with scratch testing system, a) raw profiles taken across scratch at different number of scratch repeats, b) profiles across scratch referenced to first profile, c) variation of calculated scratch area with scratch repeat.*



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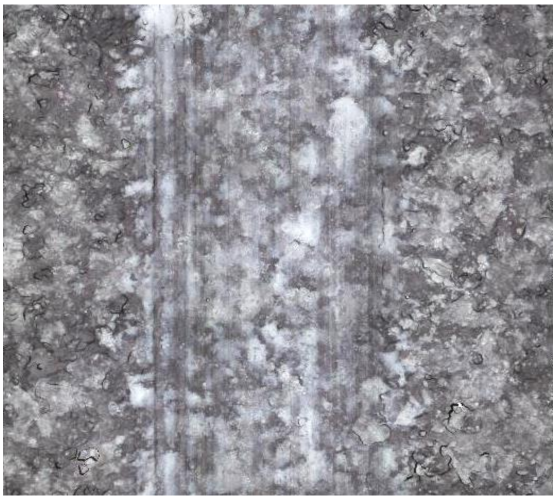

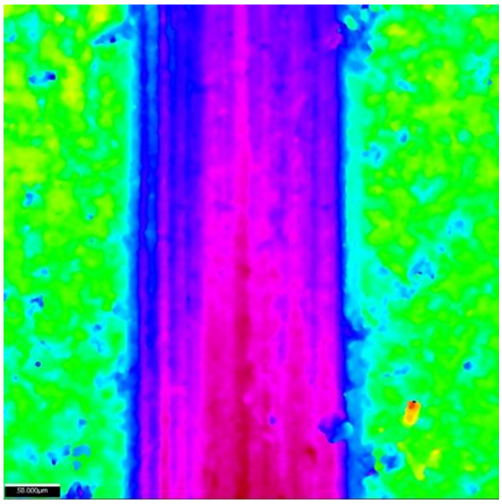

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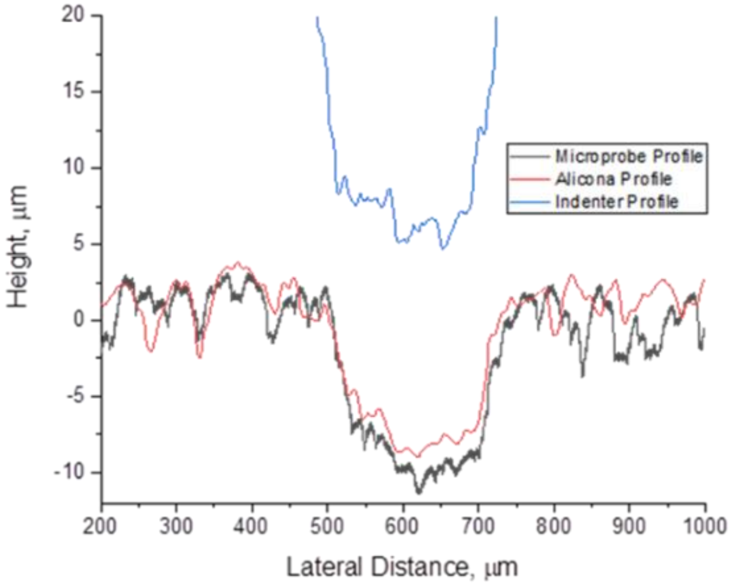


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*Figure 7, Results from measurements with scratch testing system, a) 3D optical image of scratch stylus, b) image of scratch, c) height map of scratch, d)) comparison between microprobe profile across scratch, Alicona optical height profile, and Alicona profile of scratch indenter.*

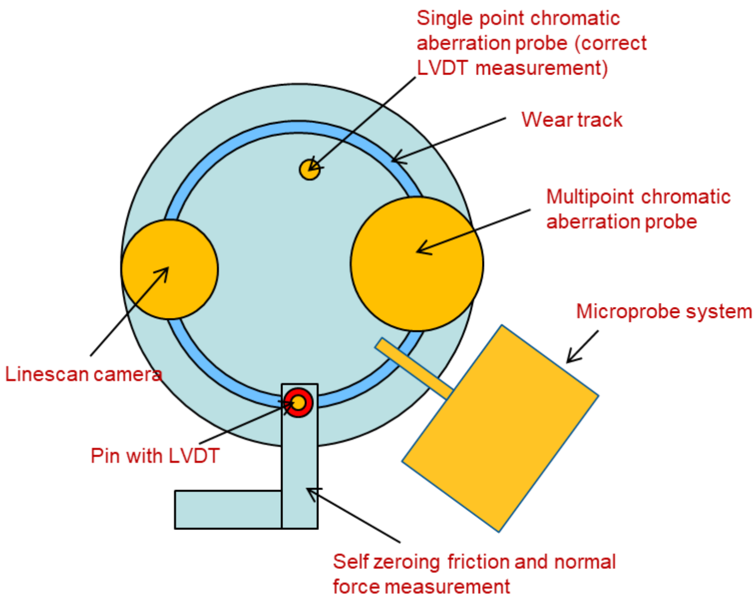



*Figure 8, Schematic of NPL integrated pin on disc tribometer*

Figure 9 shows the quantitative results from the experiment. Figure 9a shows a waterfall plot of the measurements with the microprobe which clearly show how the depth and width of the groove develops with increasing number of sample rotations. These results are compared with the simultaneous output from the multipoint chromatic aberration probe in Figure 9b. The overall form of the groove is similar, but there is less detail in the chromatic probe output. This is also shown in Figure 9c which shows a direct comparison of the profiles for three rotations of the disc. Again, the form of the profiles is very similar with much more detail in the profiles from the microprobe. This is due to the increased lateral resolution of the microprobe compared to the chromatic aberration probe which is physically limited to 180 measurement points. By integrating the profiles the area of pile up (material raised above the level of the surface outside the scratch), area of the groove (cross-section of area below surface), and area of material lost from surface (groove area – pile-up area). It can be seen that the results obtained from both systems are very similar.

Figure 10 shows the results of a post-test confocal microscopy examination of the groove produced and the indenter used. The reason that the profiles of the groove had a ridge at the base of the groove is seen to be due to the imperfections (grooves) at the tip of the scratch indenter. The profiles obtained in the confocal analysis show good agreement between the form of the groove and the form of the groove indenter.

One experiment was also carried out where the microprobe was orientated so that the microprobe measured the displacement of the wear track in a circumferential direction. A test was carried out where a $Si_3N_4$ ball was rubbed against a steel surface. The microprobe measured the position of the surface in the wear track continuously as the test was conducted. Figure 11 shows a sequence of profiles measured during the test with the first profile at the top of the stack and later profiles below this (Figure 11a). The general form of the profile is constant and is due to the unevenness of the movement of the surface of the disc with respect to angular position. As the test proceeds the measured roughness of the surface increases as wear processes roughen the surface of the wear track. Taking the average value of one of these profiles as a measure of the overall wear, the progression of these average values gives a quantitative measure of how the wear develops with disc rotations.

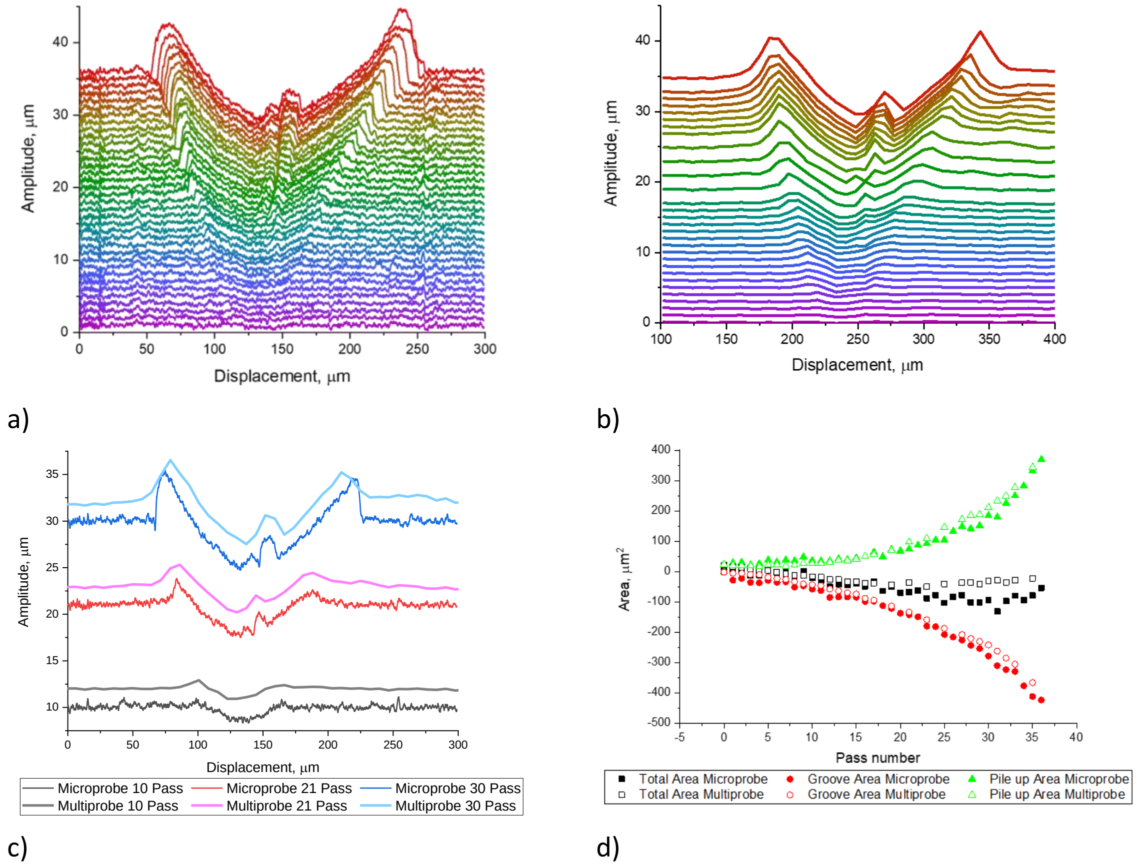



*Figure 9, Results from rotating scratch test on steel sample, a) sequence of profiles measured with the microprobe with respect to rotations of disc, b) sequence of profiles measured with chromatic aberration multiprobe with respect to rotations of disc, c) details of comparison between profiles for 3 different number of rotations, d) comparison of quantitative values of damage and their variation with number of rotations for the two different microprobes.*

### *Reciprocating*

Three in situ profilometry systems were fitted in turn to TE77 reciprocating tribometers. These were a system designed, manufactured and tested with the University of Southampton, the TUBS / PTB microprobe has been described in an earlier section, and a microprobe system designed, manufactured and now sold by Phoenix Tribology [9].

The University of Southampton (UoS) system [10] used a pressurised air bearing to ensure good low friction guidance of the probe as it moves in response to the topography of the surface under measurement (Figure 12). Before use the contact force in the UoS system was calibrated and optimised by measuring the contact force with an electronic balance (Figure 12c).

In the experiments to compare and contrast the functionality of the three different systems, both the UoS and the TUBS / PTB microprobe sub system were mounted on a simple two axis servo actuator system that was not optimised for the cost of the system but used components that were available within NPL. This two-axis actuator was controlled by a PC which also acquired the profile data from the profilometers. Both of these tests were carried out at the TE77 test system at the University of Southampton.

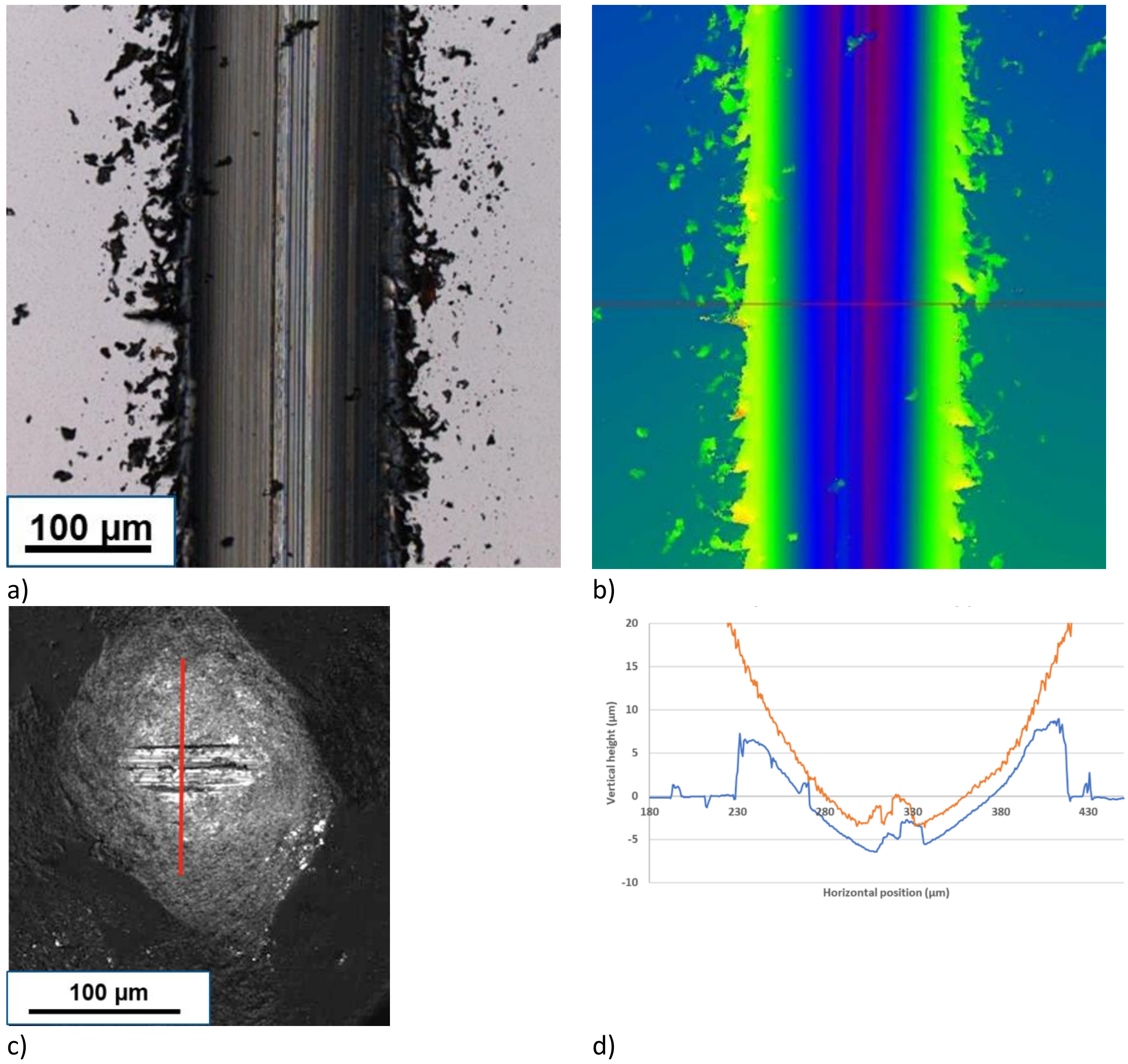



*Figure 10, Confocal microscopy of samples from rotating scratch test, a) optical micrograph of scratch, b) height map of scratch, c)optical image of scratch indenter, d) comparison between confocal profiles taken of scratch indenter |(orange) and scratch (blue).*

The original proof of concept design for the Phoenix Tribology microprobe used a two-axis servo-controlled positioning system [8]. The cost and complexity of this system was considered to render any resulting product not commercially viable. The chosen solution was to use a low-cost stepper driven lead-screw actuator driving a cam actuated lifting, positioning and lowering mechanism.

In the final design of the system that is already sold commercially [9], the stylus is fixed to one arm of a bell-crank cantilever, which is mounted on a flexural bearing. The opposite arm of the cantilever has a pin which engages with the cam track. The flexural bearing is adjusted to give the required normal force on the stylus, when in contact, and the required bias on the pin against the cam track. A vertical arm on the bell-crank provide the means for detecting the motion of the stylus (Figure 13). Originally the displacement of the vertical arm was sensed by a combination of Hall effect sensor and magnets, in “single sensor push-push” configuration. Although this arrangement worked, it was very sensitive to positional setting, did not provide a very large output signal (so has the potential for noise and interference) and was not very linear. The design was revised to incorporate two hall sensors, mounted side-by-side, so that they mirror each other, with a magnet traversed along the centre line, in slide-by mode (Figure 13e). In the work reported here the Phoenix Tribology Profilometer was tested with the TE77 system at Phoenix Tribology.

All the measurements were made under identical conditions using a dry sliding wear test using a 10 mm long, 6 mm diameter cylinder made from 52100 reciprocated over 25mm at 2Hz against a flat plate of cast iron. The depth of the cast iron surface was measured after each minute of sliding in the longitudinal direction along the wear track, ensuring that some of the profile extended over the unworn surface at the end of the wear scar to give an internal reference for the profile measurements. Profile measurements were made every minute of the test by stopping the test, carrying out a profile measurement and then restarting the test again until a total of 10 minutes sliding had been completed. A picture of the TUBS / PTB microprobe in operation making a profilometric measurement over the end of a wear track is shown in Figure 14

Results from the experiment are shown in Figure 15. All three profile measurement systems gave successful sets of profiles. The TUBS / PTB microprobe and the UoS system gave very similar results with a clear progression in wear as the test progressed. The worn area in these graphs is the left hand of the scan up to a displacement of about 4 mm. The Phoenix Tribology results looks different with no clear progression in profile in the wear scar as the test continues. The appearance of the profiles is different for the Phoenix Tribology profile with much less detail in the profile (The profile seems to be much less "noisy"). This is likely to be due to the differing signal conditioning systems for the different profilometers with the UoS and the TUBS/PTB systems using the same National Instruments hardware and software for acquisition and processing, and the Phoenix Tribology system using proprietary hardware and software. The other difference is that the edge of the wear scar in the Phoenix Tribology profile scans seems to move in towards the centre of the wear scar as the test takes place. This is possibly due to the build up of debris at the end of the wear scar.

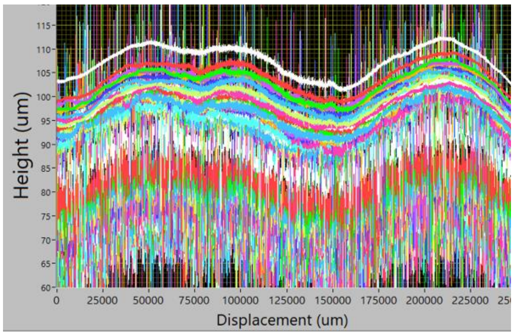
### *Microtribometer*

A SCIsensortech microprobe system was used with a two-axis actuator system with the NPL microtribometer. This microtribometer is designed to be used in situ within an SEM [6] but can additionally be used ex-situ on the laboratory bench. The microprobe was used in an ex-situ experiment where a 1 μm radius diamond indenter was reciprocated across a steel sample. Figure 16 shows profiles that were acquired during this experiment. Figure 14a shows how a scratch profile gets deeper and broader as repeated scratches are carried out along the same scratch path with increasing applied load from 10 mN to 100 mN (the first profile has been acquired before any scratching has taken place). Figure 16b shows multiple profiles acquired repeatedly across the final scratch, showing that the profiles are reasonably consistent and repeatable. The difference in the profile on the left-hand shoulder comes most likely from a slight variation in position for the microprobe scan.

An experiment was also tried with the microtribometer in situ in the SEM, but although the profile system worked, useable profiles were not obtained because the signal from the microprobe proved to be too noisy. The electronics for the microprobe and possibly for the microtribometer would need to be optimised to reduce this noise before a useable system was obtained.

### *Overload protector*

Initial experiments with the microprobes indicated that they were very fragile and that some protection was necessary to prevent overloading the microprobe cantilever beyond its deflection limit in the event of an unplanned event. Figure 17 shows a simple overload protector that was fitted to the TUBS / PTB microprobe pcb to protect the cantilever. The overload protector was designed and manufactured so that in the event of a crash where the microprobe moved suddenly against the surface to be measured, the overload protector took up the load and prevented undue deformation to the cantilever, preventing damage.



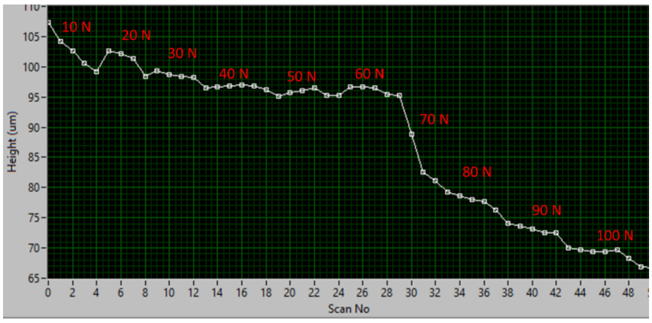




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*Figure 11, Results from in situ experiment where the microprobe was in contact with the rotating disc throughout the test, a) series of single profiles around wear scar, b) Progression in average depth of wear scar with number of disc rotations.*

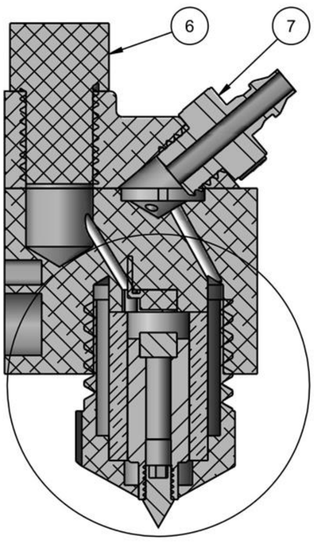


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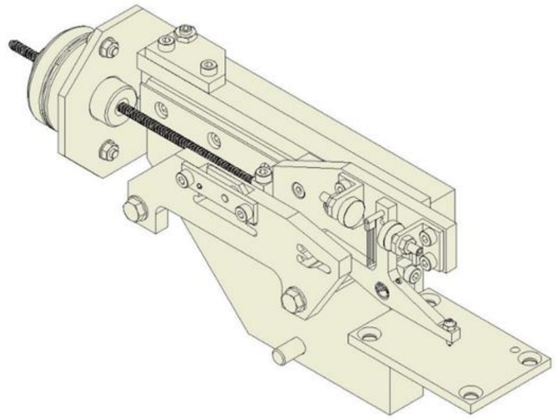
*Figure 12, University of Southampton profilometer system, a) schematic diagram, b) photograph, c) calibration and optimisation of probe force with an electronic balance*



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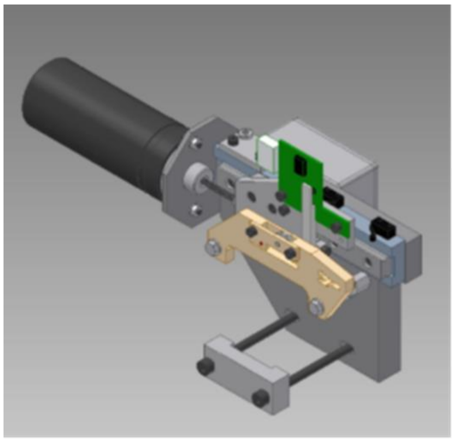

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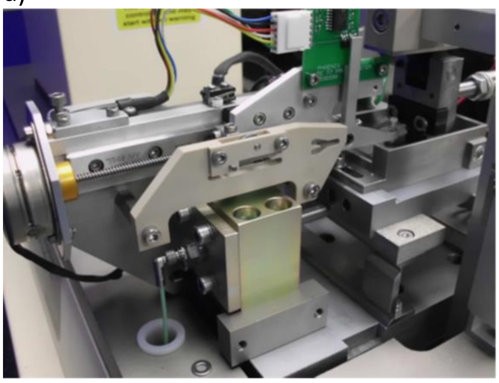

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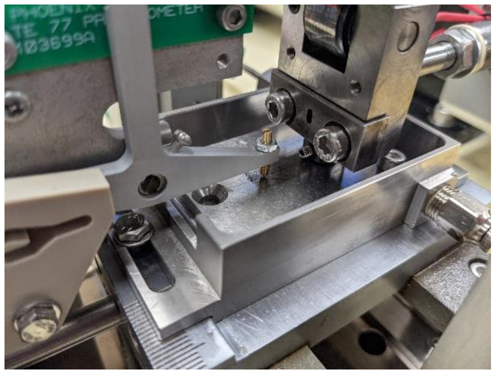

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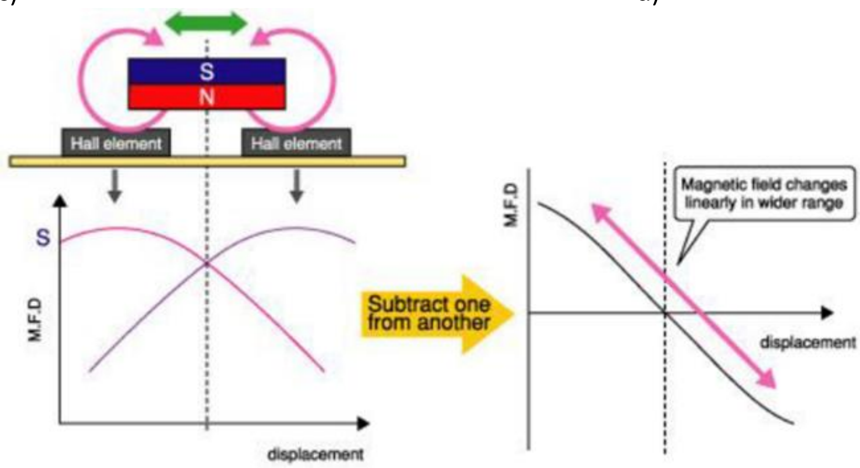



e)

*Figure 13, Phoenix Tribology in situ profilometer test system, a) schematic overview, b) 3D model, c) overall image of system, d) close up showing probe making a profile measurement across end of wear track, e) principle of magnetic sensor*

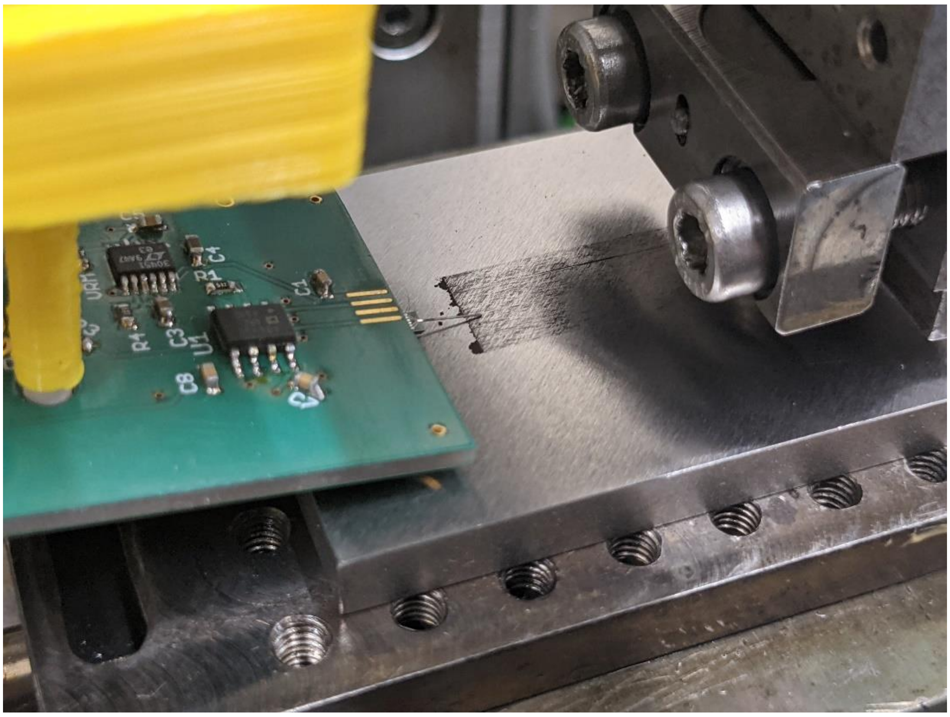


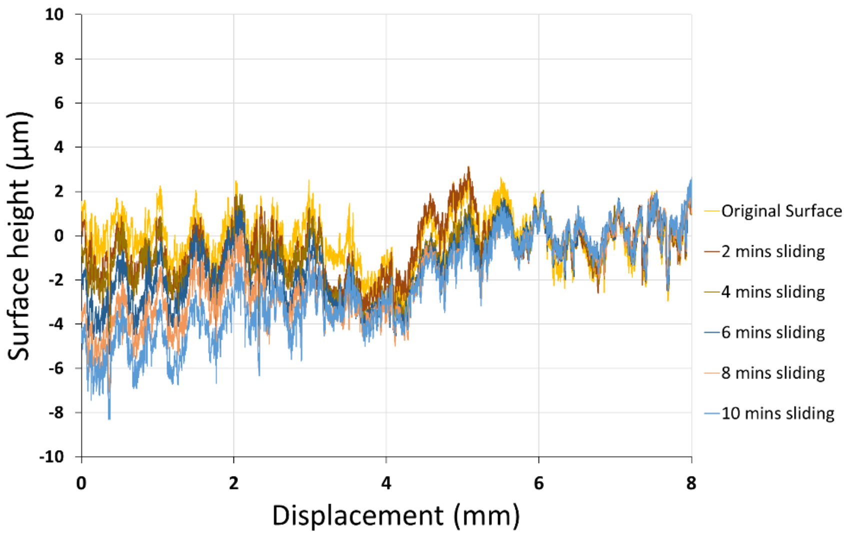
*Figure 14, TUBS / PTB microprobe making scan across end of wear track in reciprocating test.*

### *Conclusions*

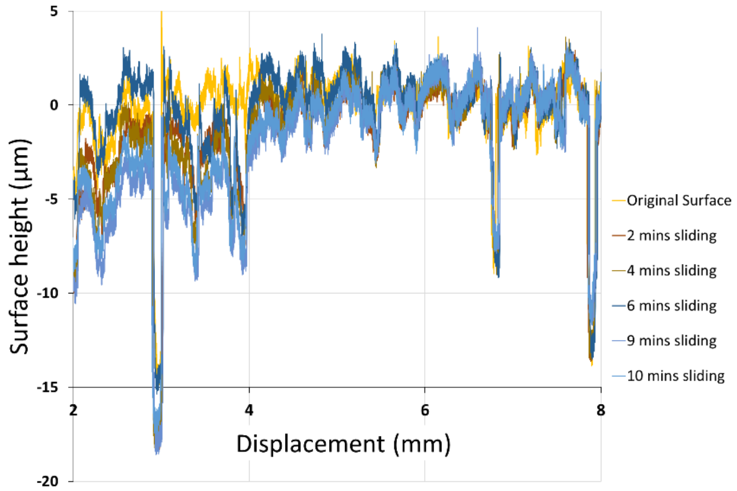
The introduction of fast microprobes into tribological testing systems has been demonstrated through their introduction into four different test systems. These were a scratch testing system, a pin-on disc test system, a reciprocating tribometer and a microtribometer that could either be used ex situ on the laboratory bench, or in situ inside an SEM. It was found that excellent results were obtained from the in situ measurements giving high resolution profiles of the wear damage that had been developed during tribological tests. The quality of the measurements and their sensitivity was good and compared well quantitatively with conventional post-test measurements.

Most of the tests that were carried out in an interrupted testing mode where the test was run for a period, sample movement stopped, a profile measurement made and then the cycle repeated until the end of the test. However, one test was also successfully carried out where the test sample was moving throughout the test and it was found that good measurements of wear damage were also obtained in this case.

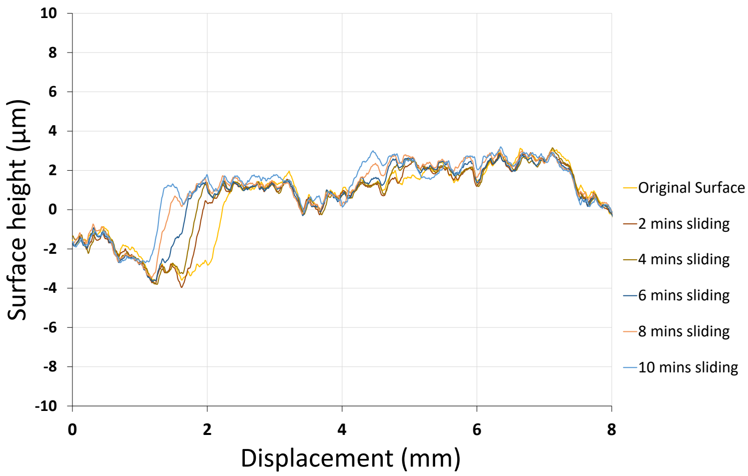
One drawback of the microprobes is that they are very fragile, so great care is needed to ensure that they are handled carefully. To help to protect the microprobes a simple overload protector was designed and fabricated using polymeric additive manufacturing. This overload protector was practically tested when a sample slipped in a tribological test bringing the microprobe head down sharply onto the opposing sample. The overload protector did its job preventing damage to the microprobe.




a)




b)




c)

*Figure 15, Comparison between profiles obtained on tests carried out under identical conditions for the a) TUBS / PTB microprobe, b) UoS air bearing profilometer, and c) Phoenix Tribology in situ profilometer system*

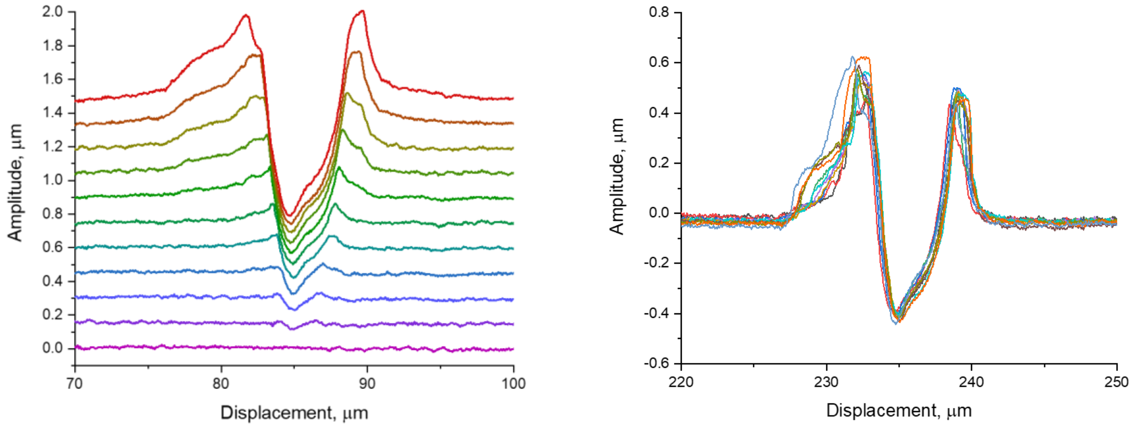



*Figure 16, Results of profile measurements made on ex situ microtribometer, a) series of profiles (offset from one another) starting before scratch test has been made and then progressing in 10 MN steps until 100 mN, b) repeated profiles made over final scratch.*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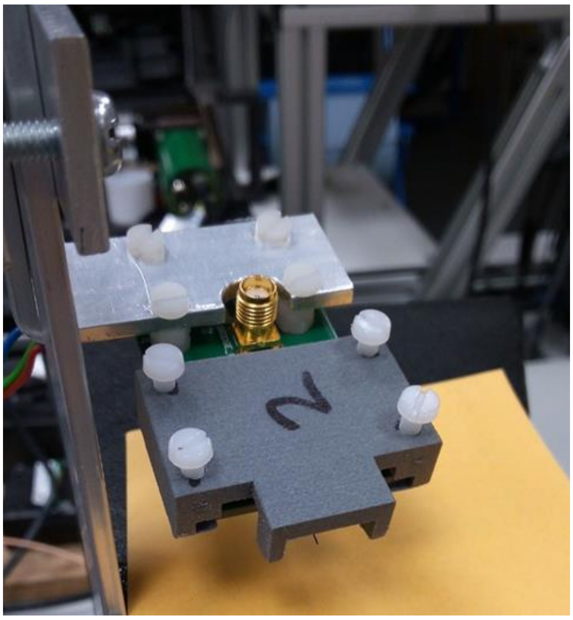
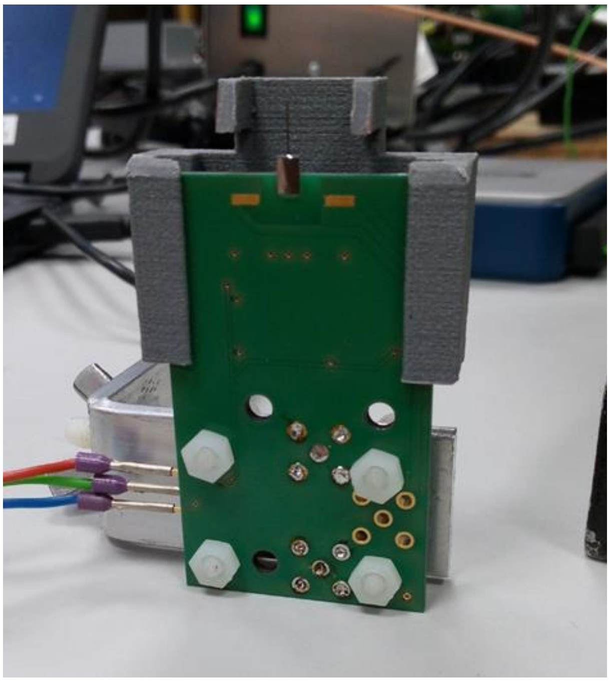
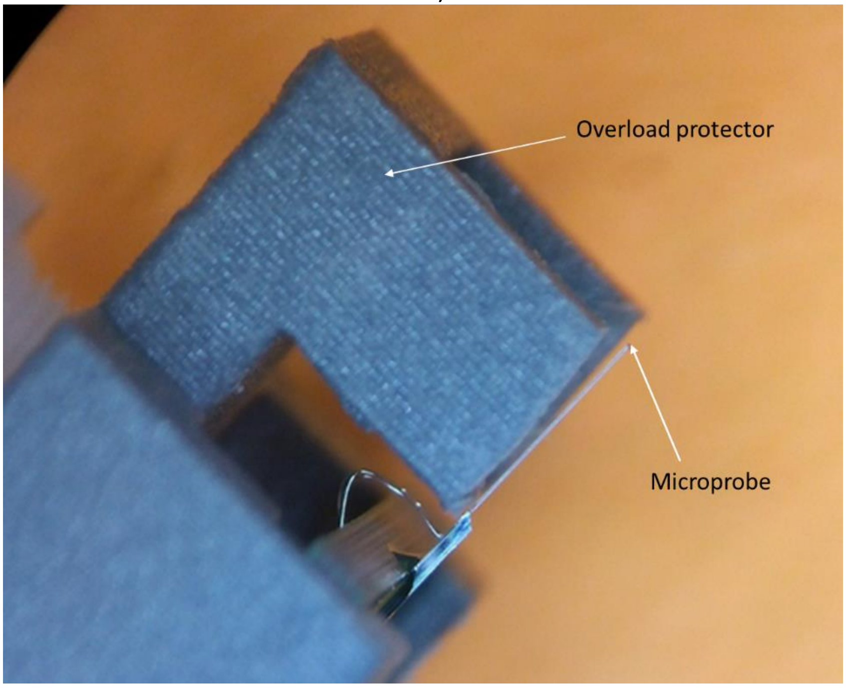




a) b)




c)

*Figure 17, Overload protector fitted to TUBS/ PTB microprobe, a) overall view, b) showing how protector fits around microprobe PCB, c) detail from side.*

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