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JRP-Coordinator	
Name, title, organisation	Dr Mark Gee, National Physical Laboratory, UK
Tel:	+44 (0) 20 8943 6374
Email:	mark.gee@npl.co.uk
JRP website address	http://projects.npl.co.uk/engineered_surfaces/
Other JRP-Partners	NPL, UK BAM Germany CNAM France INRIM Italy MIKES Finland PTB Germany ALICONA Austria FAU Germany VTT Finland

REG-Researcher
(associated Home Organisation)

Researcher name, title (Home organisation Short name, country)	Julian Le Rouzic, IC, UK	Start date: 01/12/2011 Duration:32 months
Researcher name, title (Home organisation Short name, country)	Terry Harvey, UoS, UK	Start date: 01/10/2012 Duration: 22 months

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Programme of EURAMET

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1 Executive Summary

Introduction

Techniques were developed to more accurately measure friction and wear of engineered surfaces (tribology), including approaches to measure nano-scale wear, the long-term performance of low-friction coatings, real-time temperature and chemical changes at surface contacts, and the mechanical degradation of tools. These techniques will promote the development and adoption of low-wear, low-friction surface in European industries, enhancing industrial competitiveness and reducing environmental impact.

The Problem

The engineering of surfaces is recognised as one of the ways that European industry can meet the challenge of improving competitiveness with the aim of leading world industry in sectors such as transport (air, sea and land), energy generation, manufacturing and mineral extraction. This transformation will take place not only through the improved efficiency and cost effectiveness of the European manufacturing industry, but also through products with a better functional profile such as world leading efficient air and road transport systems and consumer products.
However, all this can only be obtained through knowledge based design and manufacture of surfaces with enhanced wear resistance, and lower controlled friction. This will require better measurements of the properties and performance of surface engineering solutions such as hard wear resistant and low friction coatings so that improved understanding of how their performance depends on their make up and processing.

The Solution

This pan-European project explored a range of tools and techniques to more effectively measure the performance of engineered surfaces, to be used primarily by industrial users and industrial researchers.
Techniques that were developed included:

- The use of AFM, SEM, relocation profilometry, and confocal optical probes to measure small dimensions of wear damage
- The development of traceable, self-calibrating, stable measurement techniques for the determination of the long term friction performance of low friction coatings
- The development of measurement methods including infra-red sensing, thin film sensors, and fibre-optic probes for the measurement of the temperature of tribological contacts (i.e. at interacting surfaces)
- The development of methods for assessing chemical changes at tribological contacts (i.e. at interacting surfaces)
- The development of methods to monitor the degradation of surfaces due to wear

Impact

The project resulted in the development of a series of five good practice guides for industrial users. These guides were on the assessment of: chemical changes at the wear interface, small wear volumes, wear to tools, “zero wear”, and temperature.
The research consortium worked closely with standards organisations and it is anticipated that new procedures for assessing wear and friction will be developed and adopted by the relevant technical committees within CEN, ISO and ASTM. Partners are active in these committees, thus giving a straightforward route to standardisation. Already project results have been used to contribute to the development of a new standard for wear testing of diamond-like carbon coating in ISO standard *TC 107 Metallic and other inorganic coatings*, to a new ASTM standard *G211-14 Test Method for Conducting Erosion Tests by Solid Particle Impingement Using Gas Jets* published in 2014, and to a new standard on nano-scratch testing, initially within *CEN 352 Nanotechnologies* and eventually through *ISO 229 Nanotechnologies*.
A number of project outputs have already been adopted by industry, including the following. The new measurement technology developed in the MADES project is being used in core NPL project work including consultancy work for industry. Techniques developed in the project have inspired an SME instrument manufacturer to develop low-wear volume measurement devices. The development of these devices has been greatly enhanced by the knowledge they gained during the MADES project. The new measurement technology is being used in collaborative work within Annex IX of the Implementing Agreement for Advanced Materials for Transport of the International Energy Agency which is concerned with the development of better surface engineering for internal combustion engine components. Over the longer term it is expected that the techniques developed will be adopted by industry and used to develop further advances in low-friction, low-wear surfaces; leading to improvements in process efficiency, in the performance of products and components, and reductions in the environmental impact of European industry.

2 Project context, rationale and objectives

Context

Friction and wear in industrial processes wastes energy and degrades materials. The development and adoption of 'engineered surfaces' that reduce friction and wear are important to the development of high-performance products and improved process efficiency in key European sectors including transport, energy generation, manufacturing and mineral extraction. Advances in surface engineering will also allow industry to meet demanding sustainability requirements.

The development and adoption of surface engineering innovations by industry will:

- Improve the efficiency of production through minimising energy lost as friction, and through increasing the longevity of production machinery such as forges, presses and cutting tools; minimising maintenance and replacement costs.
- Support European industry to develop higher performance products. For instance, the downsizing of products, such as car engines, places increased mechanical and thermal stresses on components, demanding the use of lower-friction, lower-wear surfaces.
- Reductions in friction and wear will also help European industry to meet sustainability targets. Reducing friction improves the efficiency of energy use, reducing emissions. The requirement to achieve emissions of 95 g CO_2/km in all road vehicles by 2020 (2008/692/EC) will need significant reductions of friction within engines. Improvements such as dry materials processing may also lead to reductions in the use and disposal of contaminated lubricants and cutting fluids.

To facilitate the development and adoption of lower-friction, lower-wear engineered surfaces, increasingly sophisticated techniques are needed to more accurately assess their properties and performance, specifically to measure:

Nano-scale wear of new, durable engineered surfaces

The development of nano-scale surface coatings has produced durable engineered surfaces with very low-levels of wear but current measurement techniques are not sufficiently accurate to assess their performance.

Friction on low-friction surfaces over extended periods

Similarly, current measurement techniques are insufficient to assess the performance of innovative low-friction coatings, particularly over extended time periods due to issues with drift in the measurement instrumentation.

Temperature, structural and chemical changes at interacting surfaces

Temperature increases and chemical changes at interacting (contacting) surfaces can make them more vulnerable to wear. Real-time and in situ measurements are needed to provide a deeper understanding of the changes taking place, to aid the development of improved coatings and finishes.

Wear of cutting tool surfaces

The efficient use of cutting tool inserts by industry is currently hampered by an absence of models and techniques to monitor their wear in real-time use, and to predict their wear over time.

Objectives

The overarching project objective was the development of tools and techniques for accurate measurement of friction and wear of engineered surfaces. This included:

1. The development of improved techniques for the measurement of small volumes and material loss from wear (nano-scale), with sensitivity to achieve relative measurements of 0.1 µm or less over a period of several days.
2. The development of traceable, self-calibrating, stable measurement techniques for determination of the long-term friction performance of low-friction coatings.
3. The development of measurement methods for the measurement of the temperature of tribological contacts (i.e. at interacting surface).
4. The development of methods for assessing chemical changes at tribological contacts (i.e. at interacting surfaces).
5. The assessment of methods to monitor the degradation of surfaces due to wear.

3 Research results

Materials Supply and Baseline Evaluation

The materials needed for the project were carefully evaluated through a survey of partner requirements to ensure that all partners would have the necessary materials to carry out the work of the project. Many of the partners agreed to supply their own samples for specific aspects of the project, but a central stock of samples was also procured and held at NPL to ensure adequate materials supply.

These included:

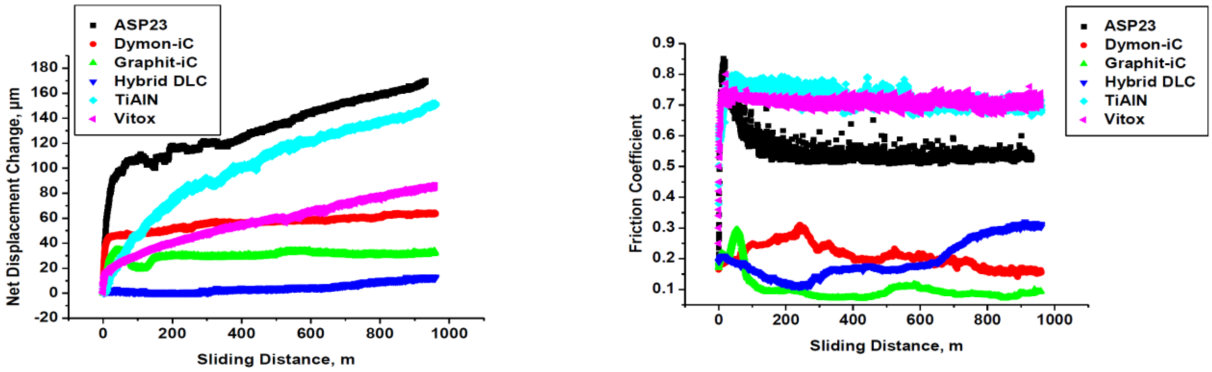
- ASP23 powder route processed hardened steel as a material in its own right, but also as a substrate for various coatings. The ASP23 had a nominal hardness of 65 HRC.
- Lapped 99.99 % pure fine grained alumina with a nominal hardness of 2300 HV1.0.
- Carbon based coatings including Graphit-ic, Dynom-ic, and hybrid DLC supplied as an in-kind contribution by Teer Coatings Ltd.
- TiAlN coatings supplied by Tecvac Ltd as an in kind contribution.

Samples of the six different materials were subjected to baseline pin-on-disc sliding wear tests by NPL and PTB with test conditions of 10 N applied load, a wear track diameter of 20 mm, a linear speed of 0.1 ms^{-1}, and a sliding distance of 1 km (i.e. test duration of 10000 s). The wear displacement and friction results with example micrographs of the worn surfaces are shown in Figure 1.

The wear displacement was relatively large for the TiAlN coatings and the ASP23 samples, and was low for the carbon based coatings. The wear of the Vitox alumina was intermediate. The friction was found to be high for the ASP23, the Vitox and the TiAlN, and was low for the carbon based coatings.

Objective 1: The development of improved techniques for the measurement of small volumes and material loss from wear (nano-scale), with sensitivity to achieve relative measurements of 0.1 µm or less over a period of several days.

A number of different methods were investigated for their potential for the accurate measurement of small wear volumes. These included AFM surface characterisation, optical measurements and relocation profilometry, 3D SEM reconstruction, confocal probe displacement measurement and laser scatterometry.



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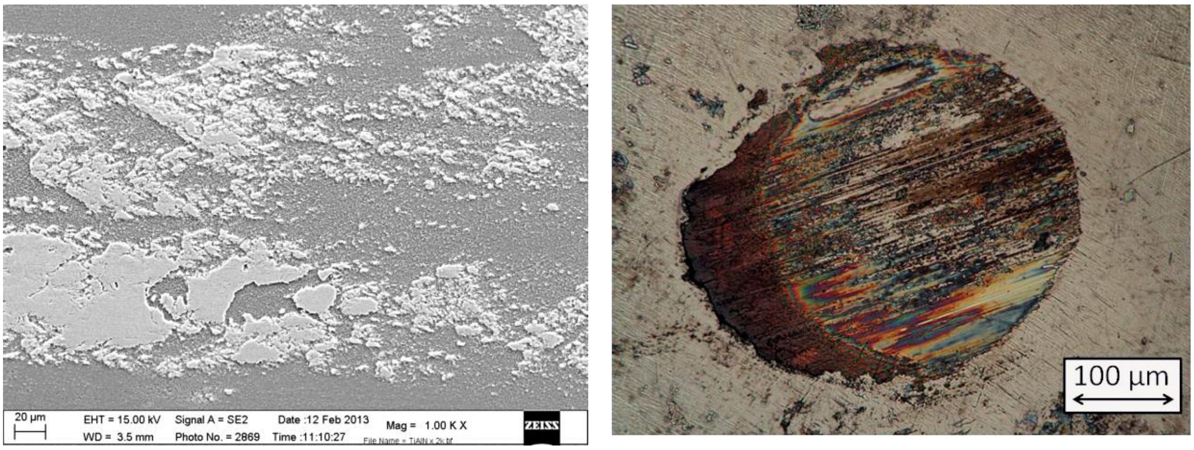


c) d)

Figure 1, a) wear displacement, b) friction, c) transfer film generated on wear track of TiAlN surface, d) wear scar on ball from hybrid DLC coating test.

AFM Surface characterisation

An atomic force microscope (AFM) ("Dimension Icon" (Bruker)) was used by PTB for the quantitative wear assessment of high-performance DLC-based thin film coatings, which had undergone tribological tests under rolling/slip-rolling conditions. The AFM is traceably calibrated by applying a set of step heights and lateral gratings calibrated by the metrological large range AFM at PTB. Figure 2 shows two AFM images of the DLC coated surface, one of the worn and the other of the unworn area. The general surface structure (morphology) seems to be similar, but the surface of the worn area (Figure 2b) has some flat areas (marked with circles) on the top of asperities that are not present on the unworn surface shown in Figure 2a. These flat areas are the result of interactions between the top of asperities on the two surfaces without significant destruction. It should be noted here that most of the asperities have not undergone such changes on their asperity tips during friction. It was also found in an analysis of different areal S-parameters that the skewness (Ssk) was the most sensitive and reliable parameter for assessing the nano wear, which indicated the loss of the peak structures of the asperities during the friction test. This agreed well with the physical understanding of the "zero-wear" process.

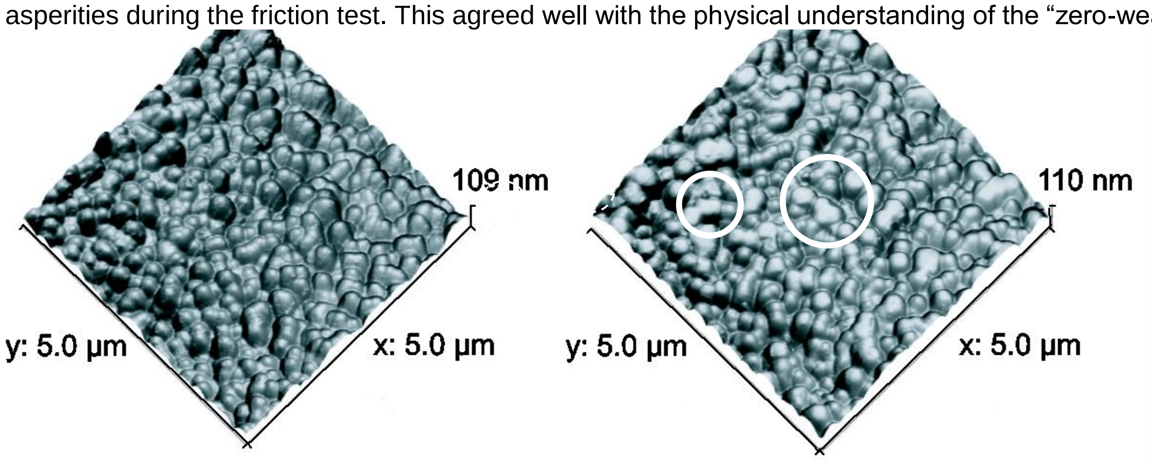


Figure 2, AFM images of an unworn (a) and a worn surface (b) of the tested DLC coating. One flat area on the top of asperities of the worn surface are marked.

Wear assessment using confocal microscopy

A commercial confocal laser scanning microscope type "LEXT OLS 4000" was used by PTB to examine a partly worn steel sample (Figure 3). The left image was taken in the BF mode, where a very weak intensity contrast between the worn and unworn area can be seen. The right image was taken in the confocal imaging mode with a 50x objective lens at the worn area. Some scratch tracks are clearly visible in the image. Several measurements were also taken across the border between the worn and unworn areas, however, no height contrast between the different areas is visible in the image due to the axial resolution limit of the microscope.

The size of the scratch tracks was measured using a 100x objective lens with 4x digital zoom (Figure 4); the scratch tracks were found to typically have a width of approx. 4 µm, a length of tens of micrometres and a depth of approximately 50 nm.

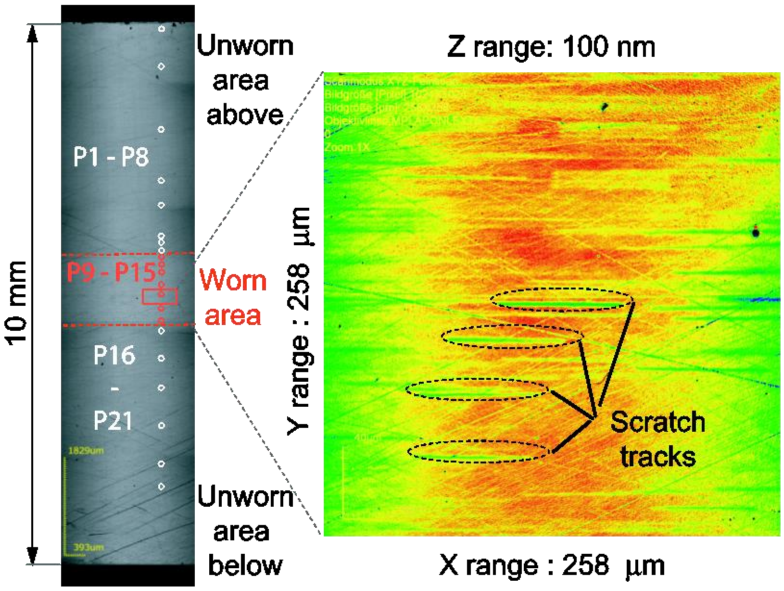


Figure 3, Overview image of the worn and unworn surfaces taken by the confocal microscope LEXT. Image clearly shows the scratch tracks. AFM measurement locations P1-P21 are marked.

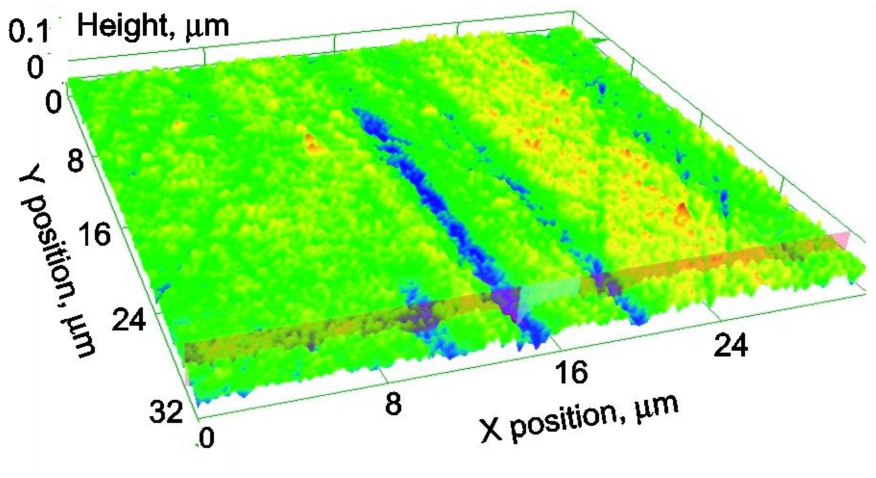


Figure 4, 3D view of scratch tracks measured by the confocal microscope LEXT with a 100x objective and 4x digital zoom.

Relocation Profilometry

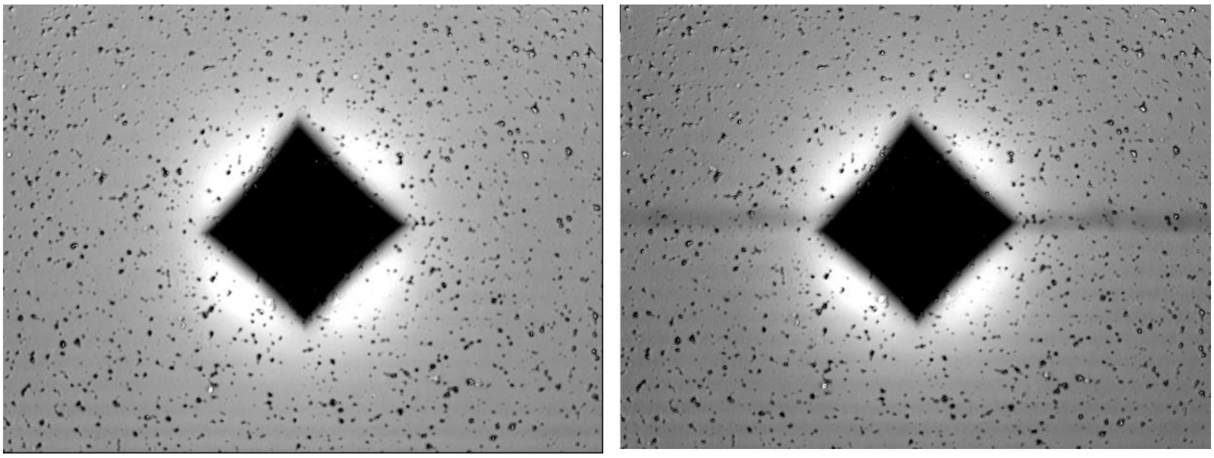
NPL carried out a series of relocation profilometry experiments using their microtribology test rig. An important addition to the test system was a kinetic mount arrangement to allow for rapid accurate relocation of the sample in the test system. Another key aspect was the development of a procedure to mark samples with Vickers indentations and then using these as signposts to aid relocation with the microtribometer. The overall process is then to measure the surface before the tribological test, carry out the tribological test, and measure the surface after the test. The comparison and analysis of the surface topography measured before and after the test then yields measurements of surface damage with much improved precision and accuracy compared with conventional post-test measurements.

In practice it was found that relocation could be carried out to an accuracy of about 3 µm. A number of different approaches to analysing and comparing the before and after height information were explored. Figure 5 shows an example of an analysis using the bunwarpj plug-in for ImageJ. This uses an elastic morphing algorithm to enable registration across the whole of the image. Good results were obtained as shown in the comparison of the profile made before the registration and subtraction and the one achieved after the application of the bunwarpj process.

Wear assessment using chromatic confocal sensor

Optical, non-contact chromatic confocal microscopy (CCM) exploits the chromatic dispersion of an objective lens that focuses different colours at varying distances from the lens. The distance between lens and surface can be retrieved by spectral analysis of the light reflected from the target surface, that is, there is no need for a mechanical scan of the depth profile.

At MIKES the feasibility of using a commercial CCM probe was explored in wear tests on steel samples with twin disc equipment. This work was carried out with VTT who gave access to MIKES to the tribological testing equipment. Although it was found that some data processing was necessary to remove artefacts in the test data good agreement was found in a calibration study on an unworn sample between CCM results and the results of profiles measured traditionally with a stylus (contact) instrument (Figure 6a). In a real wear test, the CCM was able to measure progressive wear to the test disc as the experiment was interuppted periodically (Figure 6b).



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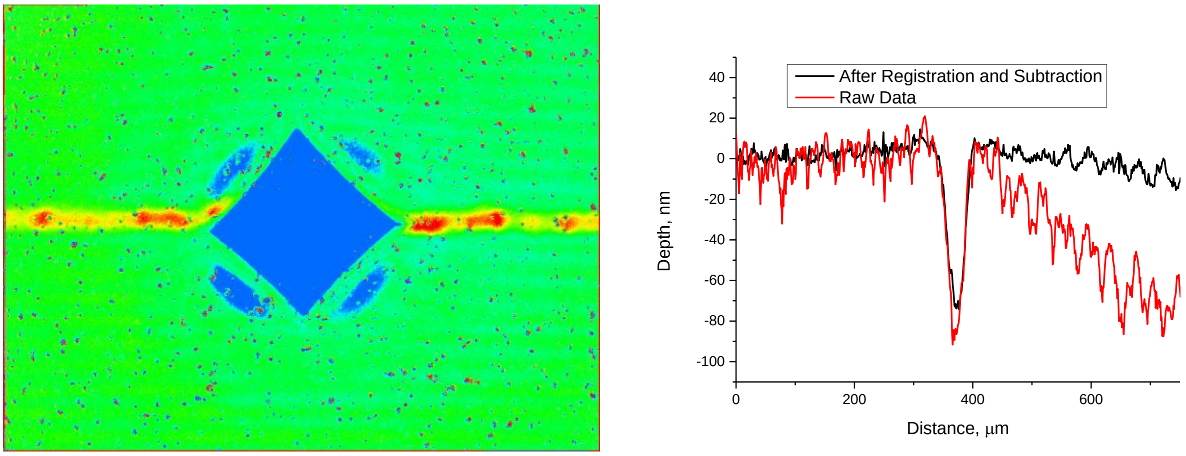
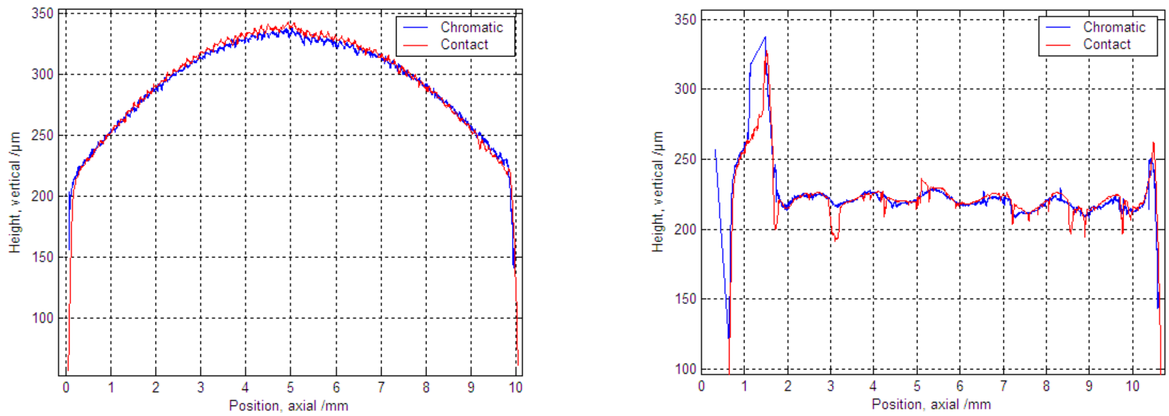


c) d)

Figure 5, Analysis of a scratch on a hybrid DLC sample with bunwarpj plug-in for ImageJ, a) height map before test, b) height map after test, c) registered and subtracted height map, d) profiles across scratch.



a) b)

Figure 6, Comparison of profiles measured with chromatic probe and stylus instrument,a) before the test, b) after the wear test.

Evaluation of Cutting Tool Wear

The University of Erlangen (FAU) carried out work to develop and validate methods for the optical characterisation of wear to cutting tools. These tools were used for metal cutting, and FAU showed that non-contact focus variation microscopy, and interference microscopy were effective traceable methods for the accurate evaluation of tool wear. FAU was supported in their work by Alicona who are a major supplier of focus variation microscopes.

Three-Dimensional Surface Reconstruction from SEM Images Using Photogrammetric Methods

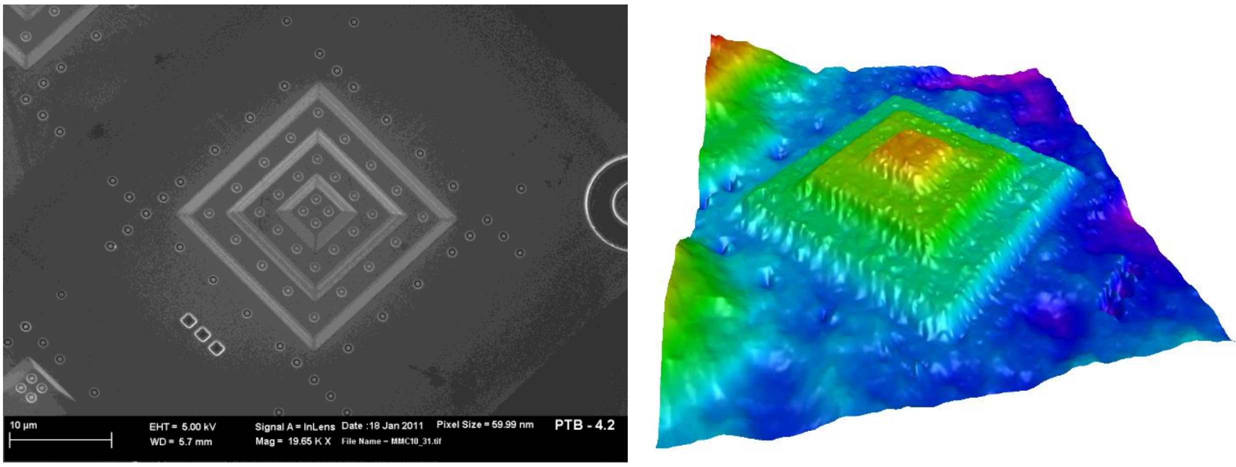
Photogrammetry is a method capable of reconstructing object shapes from multiple photographs. This technique may also be used in electron microscopy, particularly in SEM, providing the ability to reconstruct surface shapes of imaged micro-objects.

For small volume determination with SEM, the accuracy of the photogrammetric 3D surface reconstruction techniques has been evaluated. This is a requirement if the method is to be employed in metrology. Tests have been carried out with real, Mote-Carlo-based and Monte-Carlo-less images. For the real sample, the popular calibration pyramid sample MMC–10 was utilised. The PTB-developed MCSEM software was used for SEM image Monte-Carlo-based simulation and the new super-fast image Monte-Carlo-less simulator software also developed at PTB provided the fast images.

In order to determine the accuracy of the 3D-reconstruction method, it is necessary to compare the original sample with the result of the reconstruction. This is only possible with the simulated images because only then is the sample fully known and all of the artefacts present in the SEM images can either be taken out (producing a perfect image) or deterministically simulated enabling the sensitivity of the reconstruction technique to these effects to be evaluated.

The height map of the original sample and the reconstructed result have been compared in the pixel-by-pixel manner. This method is very simple and quick, but it is not perfect. A histogram of the height differences has to be calculated and the corresponding sigma-like values have to be worked out. However, for reasons that are not clear, the statistical basis of this distribution was not normally distributed.

The test with real images of the MMC–10 calibration sample demonstrated the ability of the photogrammetric technique to successfully reconstruct objects several micrometers wide and over one micrometre high. Thus Figure 7a shows an SEM image of the calibration sample, and Figure 7b shows a 3D reconstruction of sample height calculated from a set of tilted images.

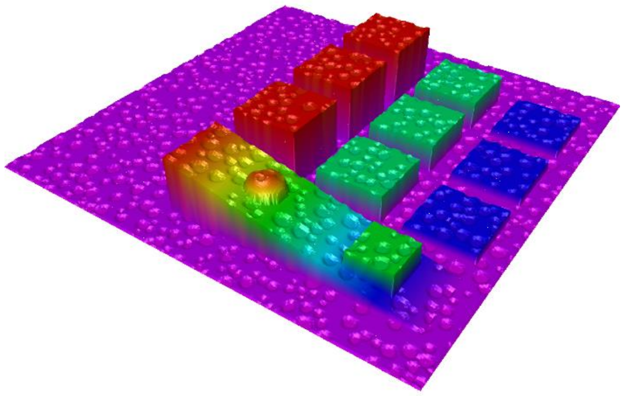


a) b)

Figure 7, a) MMC-10 calibration sample, b) 3D reconstruction of sample height calculated from a set of tilted images.

Tests with Simulated SEM Images

Multiple and various test structures have been applied to assess the photogrammetry technique with simulated images. The following figures show the simulated sample that provided the best results achieved. Figure 8a shows the 3D view of the model structure, Figure 8b shows a simulated SEM image, Figure 8c shows the results of a photogrammetry reconstruction of two SEM images, and Figure 8d shows an analysis of the deviations in height between the original height data (Figure 8a) and the reconstructed height data (Figure 8c). The results show that the best possible sigma value achieved has been 42 nm. Such a deviation is definitely too high if features around 100 nm need to be measured. According to the results with the MMC–10 calibration sample (real SEM images) the photogrammetric reconstruction works quite well with several-micrometre-wide structures. However, it does not appear to be very suitable for the processing of tribological samples within the scope of this project.



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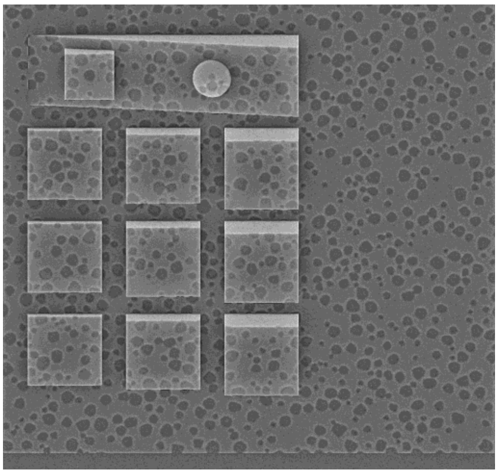

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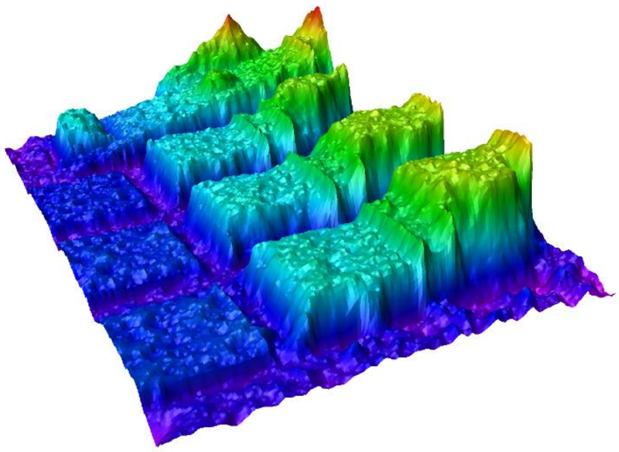

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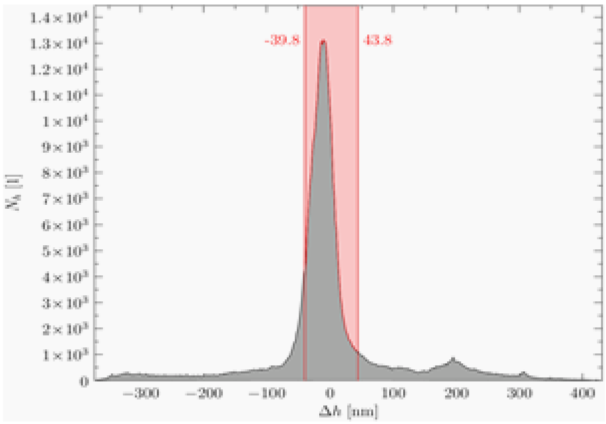

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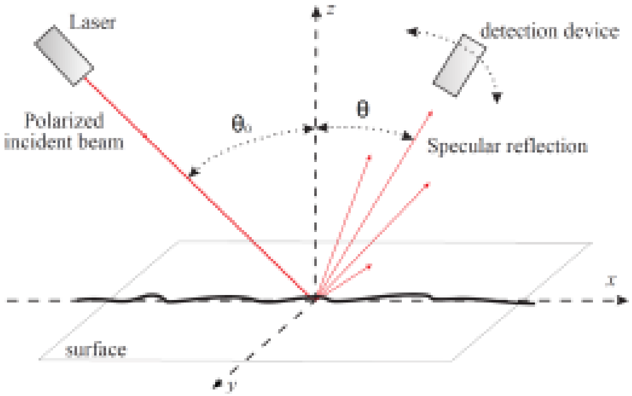
Figure 8, Simulation and analysis of test structure, a) 3D visualisation of the simulated test structure, b) simulated SEM image of the test structure using a Monte Carlo based method with a tilt of 10º, c) 3D visualisation of the recunstructed surface, d) Distribution of the height deviations. The pink area corresponds to 1σ.

Evaluation of worn surfaces using laser scatterometry

CNAM, supported by PTB, evaluated the use of laser scatterometry to evaluate wear surfaces. The system at CNAM, shown in Figure 9a, consists of a 635 nm laser source, an optical arrangement (collimation and filtering system), a motion-controlled sample holder, a detector on a movable arm and a photomultiplier tube. Measurements are made at a specific angle of incidence and the scattered light intensity is evaluated for each scanned position.

A major advantage of the technique is that compared to others methods, this measurement is relatively fast. Indeed, it takes about 3 min to measure the angular distribution of the scattered light for a given site on a sample and about 30 min for a scattered light map on 1 mm² with steps of 25 µm. 100Cr6 samples used in an SRV test were supplied by BAM. The sample had been subjected to a lubricated tribological test in a reciprocating sliding tribometer. Figure 9b shows the differences in the power spectral density between the centre of wear tracks (T1 and T2) and away from the wear tracks. Figure 9c shows a scattered light map for one of the wear tracks.



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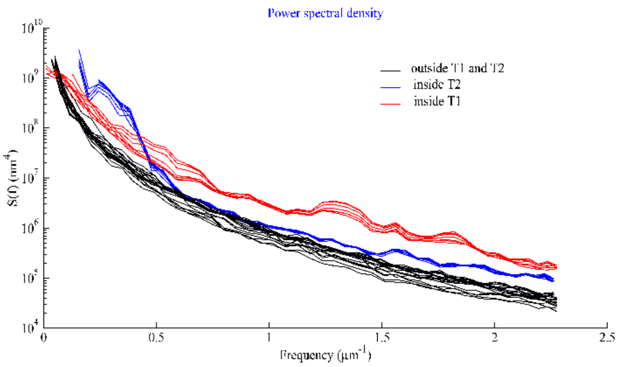

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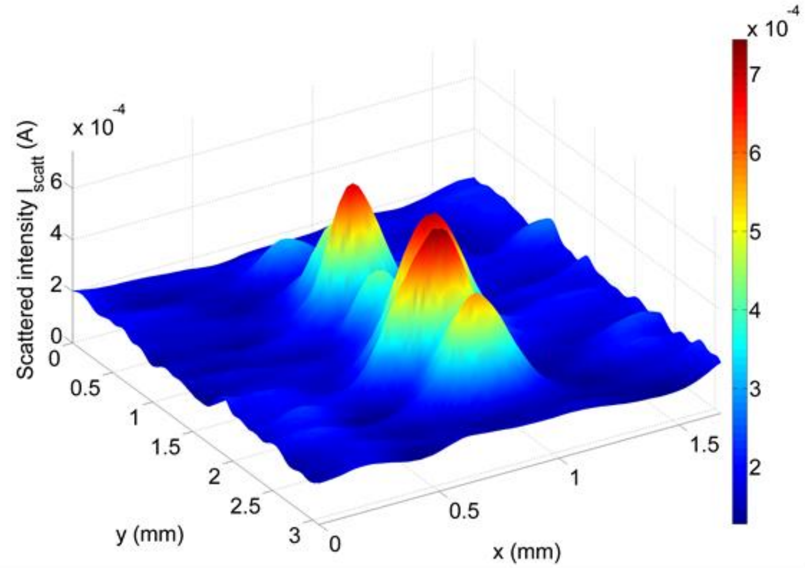

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Figure 9, a) schematic of the system, b) power spectral density (PSD) curves, also called "roughness spectra" from unworn (i.e. outside) and worn (i.e. inside T1 and T2 tracks) areas, c) scattered light map at an incident angle of 30º for wear tracks.

Objective 2: The development of traceable, self-calibrating, stable measurement techniques for determination of the long-term friction performance of low-friction coatings.

Accurate friction measurement is becoming more and more important as coatings are developed with low friction properties. Normal methods of friction measurement often prove to be inadequate because of their lack of stability. Thus conventional strain gauge load cells drift with time, so if there is a need to measure low forces (which is true for materials with low friction), errors can be generated.

Two approaches to improving the measurement of friction were evaluated in the project. These different approcahes were developed by BAM and NPL with advice from PTB.

BAM looked at the use of small, accurate interferometers combined with carefully designed stiff reaction elements. Figure 10 shows the design, construction and finite element (FE) modelling of the friction measurement element. Careful design and modelling of the device ensured that it has appropriate sensitivity.

Figure 11 shows the device mounted in the test system with the miniature interferometer to give the necessary measurement of displacement in the vertical and horizontal directions and to give measurement of force in these two dirctions.

The utility of the system was demonstrated in tests carried out with an alumina wear couple against a DLC coated steel. The modified test system was found to perform well.

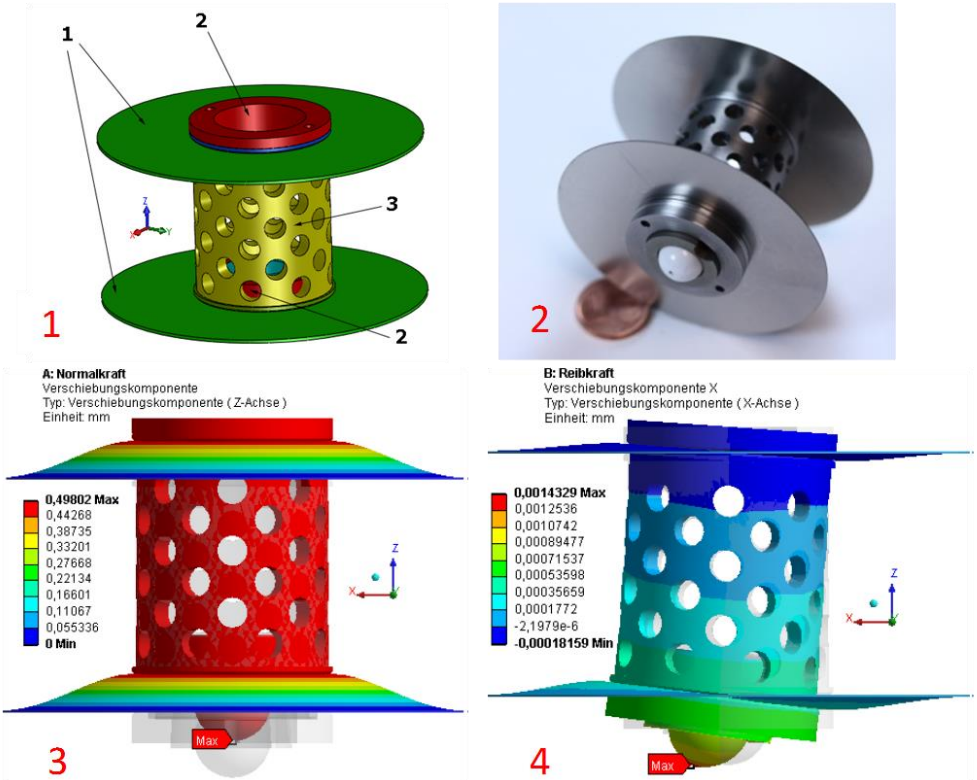
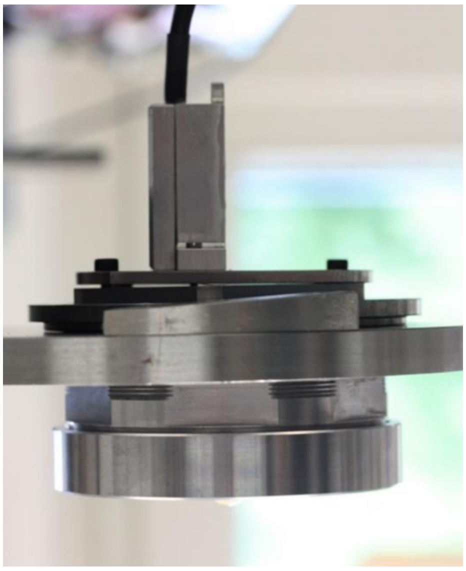


Figure 10, Central deformation element for force detection and sample holding, 1 CAD drawing, 2 Assembled steel membranes and cage, 3 Simulation of deformation upon normal force, 4 simulation of deformation upon friction force.



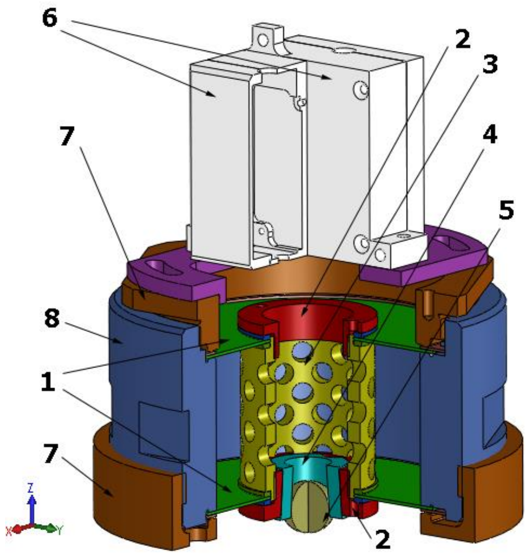


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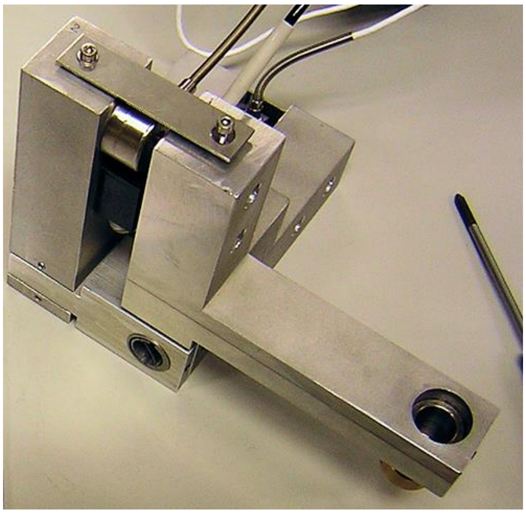
Figure 11, Combined friction measurement system with a deflection element and interferometers, a) image, b) schematic diagram.

NPL took a different approach which used conventional strain gauge load cell technology to develop a system which gives self zeroing friction measurement. This system uses a pneumatic cylinder to remove the load from the load cell at periodic intervals set by software. The removal of load enables the load cell to be zeroed at these intervals. It should be noted that the load is taken by the pneumatic cylinder so that the load acting between the pin and disc remains in place, thus the test is not interuppted during the periodic zeroing of the load cell. The normal applied load and frictional force are both measured so that an instantaneous measurement of the friction coefficient can be made. Figure 12a shows a photograph of the NPL device, and Figure 12b shows an example of the benefit of using the device which shows a 40 % reduction in the friction coefficient when the self zeroing device is used.



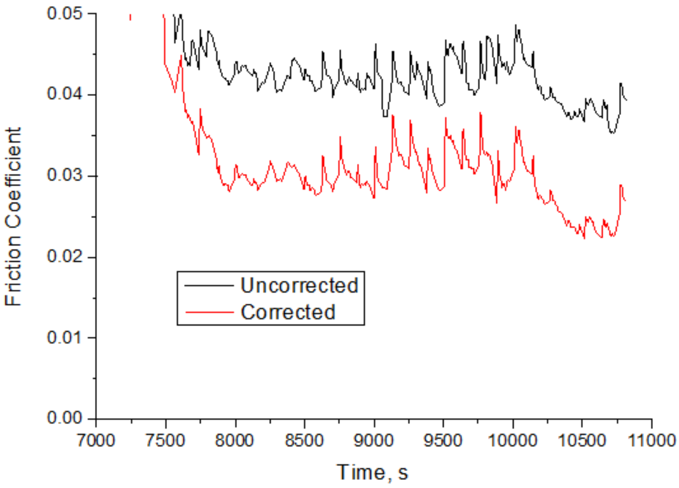


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Figure 12, NPL's self zeroing friction device, a) photograph showing the pin holder on the right, b) example results from using the self zeroing device.

Objective 3: The development of measurement methods for the measurement of the temperature of tribological contacts (i.e. at interacting surface).

Three different methods were investigated for the measurement of interface temperature. NPL investigated the use of thermal imaging cameras looking at both *ex situ* and *in situ* measurement of the surface of the wear track. INRIM developed a new fibre measurement system, and DTI developed a sub-surface platinum thin film measurement technique. The three techniques were compared through simultaneous measurement carried out at NPL towards the end of the project. It was found that the results for the three techniques were consistent bearing in mind the differences in the physics behind the three different techniques.

There was also some exploratory work at BAM looking at how Stokes-antiStokes Raman scattering could be used to measure temperature. This work was not successful due to limitations with the instrumentation that was used.

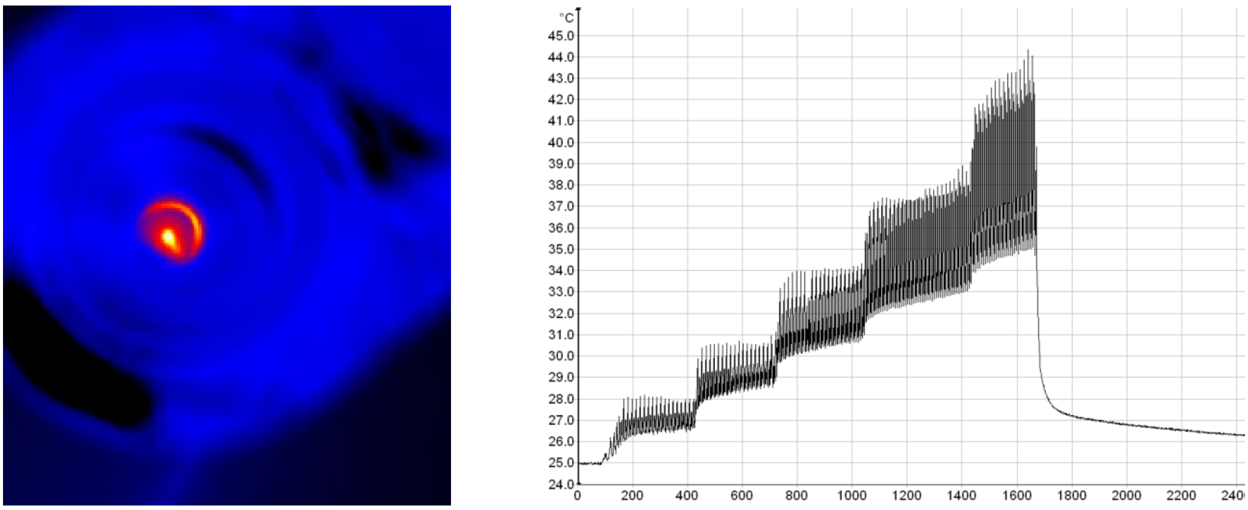
PTB gave support to the work of the project to address objective 3 through the provision of access to temperature measurement standards and accurate temperature measurement.

Thermal imaging

Initial results were obtained using a thermal imaging camera looking at the wear track on the disc from a pin-on-disc test in progress. This clearly showed the hot zone in the wear track as it emerged from the wear contact with gradual decay of the temperature as the distance from the contact increased.

Later tests were carried out using a sapphire hemisphere transparent to the IR radiation used by the thermal imaging camera. Figure 13a shows an image with the raised temperature at the contact shown as a bright spot. It should be noted that for these tests the imaging was not optimised as due to the size of the pin holder the thermal imager could not be brought close enough to the sample surface to bring it into good focus. It should also be noted that the intrinsic resolution of the thermal imaging camera is 25 µm. Figure 13b shows that the quantitative results showed a sudden rise in temperature of the wear contact on application of increased load with a subsequent slower increase in the temperature until the contact temperature equilibrated. The variability in the temperature measurements that is observed is due to slight differences in the contact as the disc rotates.

Figure 14 shows results from experiments where the sample holder was redesigned to allow the thermal imaging system to be focussed on the contact. It is clear that much more detail is shown in the contact zone showing that the contact is not a single point and varies with time.



a) b)

Figure 13, Thermal imaging results with a non-optimal position of the thermal imaging camera, a) thermal image viewing through a sapphire contact, b) numeric results showing the increase in the average temperature of the hotspot as the applied load is stepped up by 15 N increments to a maximum of 75 N *followed by the removal of the applied load with a decay of temperature. Much of the variability in the temperature is due to the rotation of the test disc.*

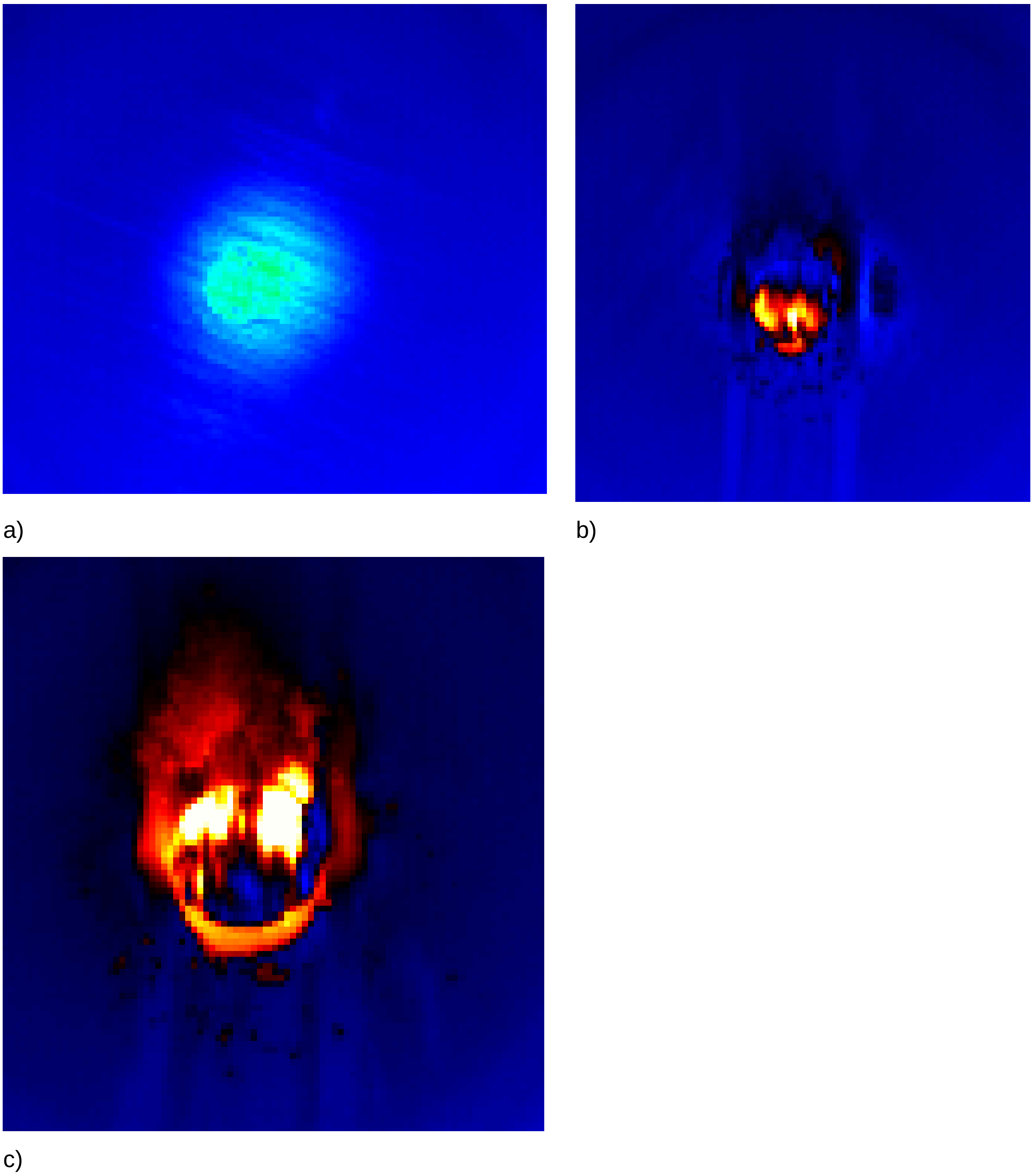
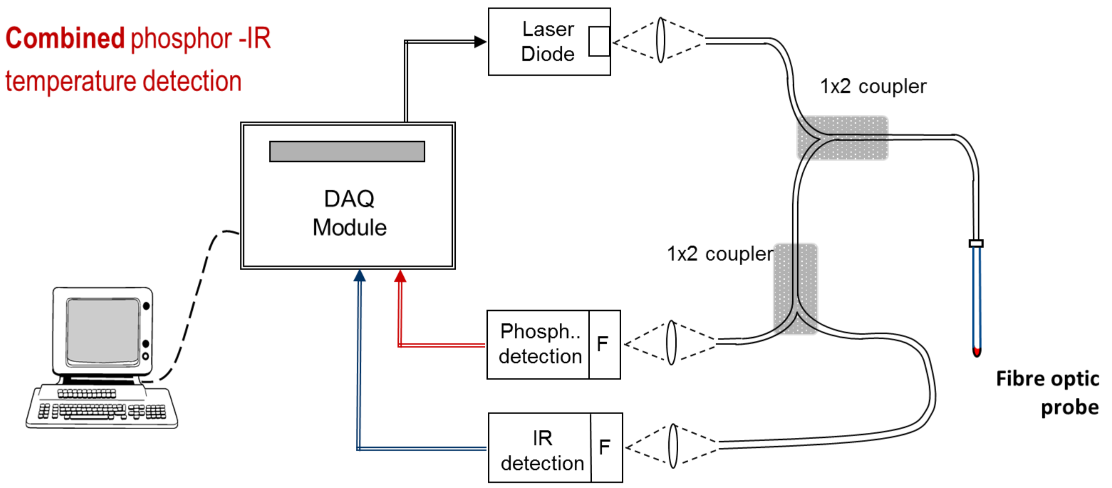


Figure 14, Thermal images taken from a video taken through a sapphire contact under an applied load of 30 N, speed of 20 rpm, a) start of test, b) after 1500 s, c) after 2500 s.

Fibre probe measurement

INRIM developed novel fibre based measurement technology for the measurement of temperature. The technique that was developed was a combined phosphor and IR temperature detection. The system relies on the heating of a phosphor and the detection of IR radiation to measure temperature. Initial trials proved the technology (Figure 15a) which was then installed into a sapphire in situ probe system (Figures 15b-15d). This was found to give good results in practice.



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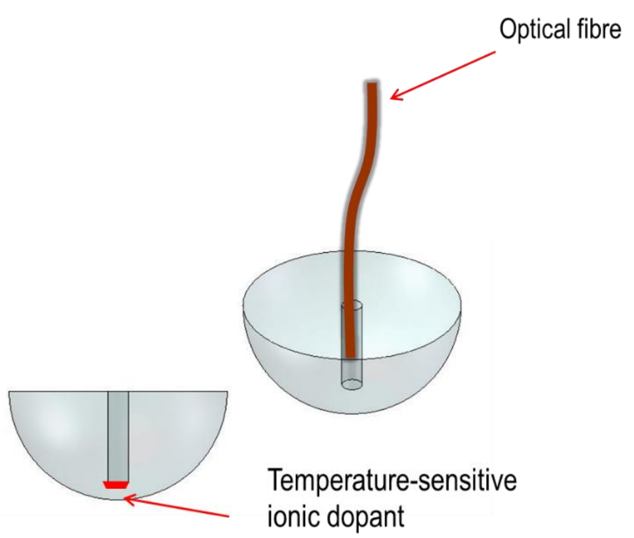

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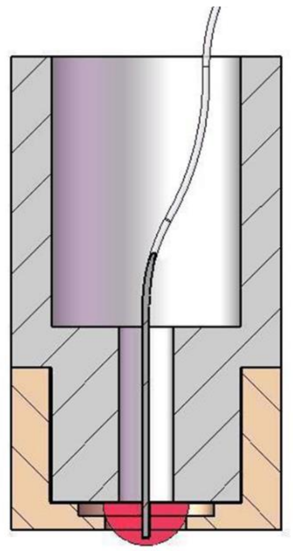

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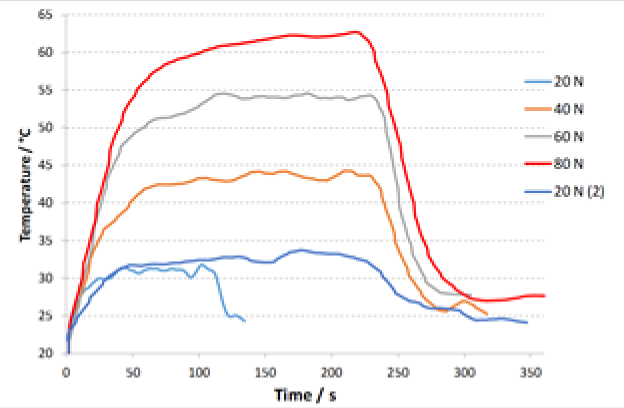
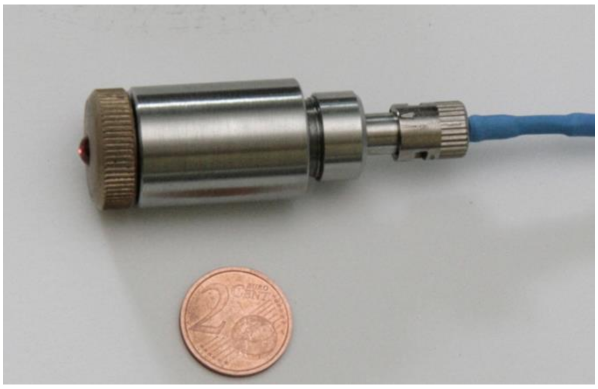




d) e)

Figure 15, In situ fibre optic measurement device, a) schematic visualisation of the measurement scheme, b) schematic modified hemisphrical sapphire probe, c) schematic of probe assembly, d) photograph of probe assembly, e) typical results showing the effect of increasing applied load.

Thin film platinum thermometer

DTI developed a thin film thermometer to measure temperature at wear contacts. The sensor was based on the deposition of a platinum layer on a substrate followed by deposition of the surface engineering coating that acts as the functional wear resistant layer. This technology is therefore appropriate for the incorporation of sensing into surface engineering systems as they were being fabricated. This gives the potential for integration of sensing into the improved protection of surfaces that comes from the application of surface engineering.

In comparative studies performed at NPL the thin film thermometer gave results which were consistent with the results obtained with the INRIM fibre probe and the NPL thermal imaging results. The temperatures measured with the thin film sensor were lower than the temperatures measured with the NPL thermal imager and the INRIM fibre probe because the thin film sensor was inevitably a small distance beneath the wear contact itself.

Objective 4: The development of methods for assessing chemical changes at tribological contacts (i.e. at interacting surfaces).

The work on the measurement of interface chemistry examined the feasibility of developing methods to characterise the chemical changes that have occurred at worn surfaces. Two methods were examined: Raman spectroscopy (BAM, NPL), and Thermal Desorption mass Spectrometry (TDS) by CNAM. Samples have been assessed using a TDS device on worn and unworn surfaces.

Thermal desorpsion

The basic principle is to induce desorption of physisorbed molecules on a surface by heating it under low pressure. By performing this thermodesorption under vacuum, it is simple to analyse desorbed molecules using a mass spectrometer. Temperature Programmed Desorption (TPD) is used because of its simple implementation: a regulated temperature ramp allowed us to follow the partial pressure (or intensity) of desorbed molecules and to determine kinetic and thermodynamic parameters during a desorption.

The test system is shown in Figure 16a. A sample of 100Cr6 cd-401 steel was supplied by BAM (Figure 16b) which had been run against a polished 100Cr6H ball. Several wear tests had been carried out on this sample giving the wear tracks shown in Figure 16c. A separate unworn sample was used as a reference.

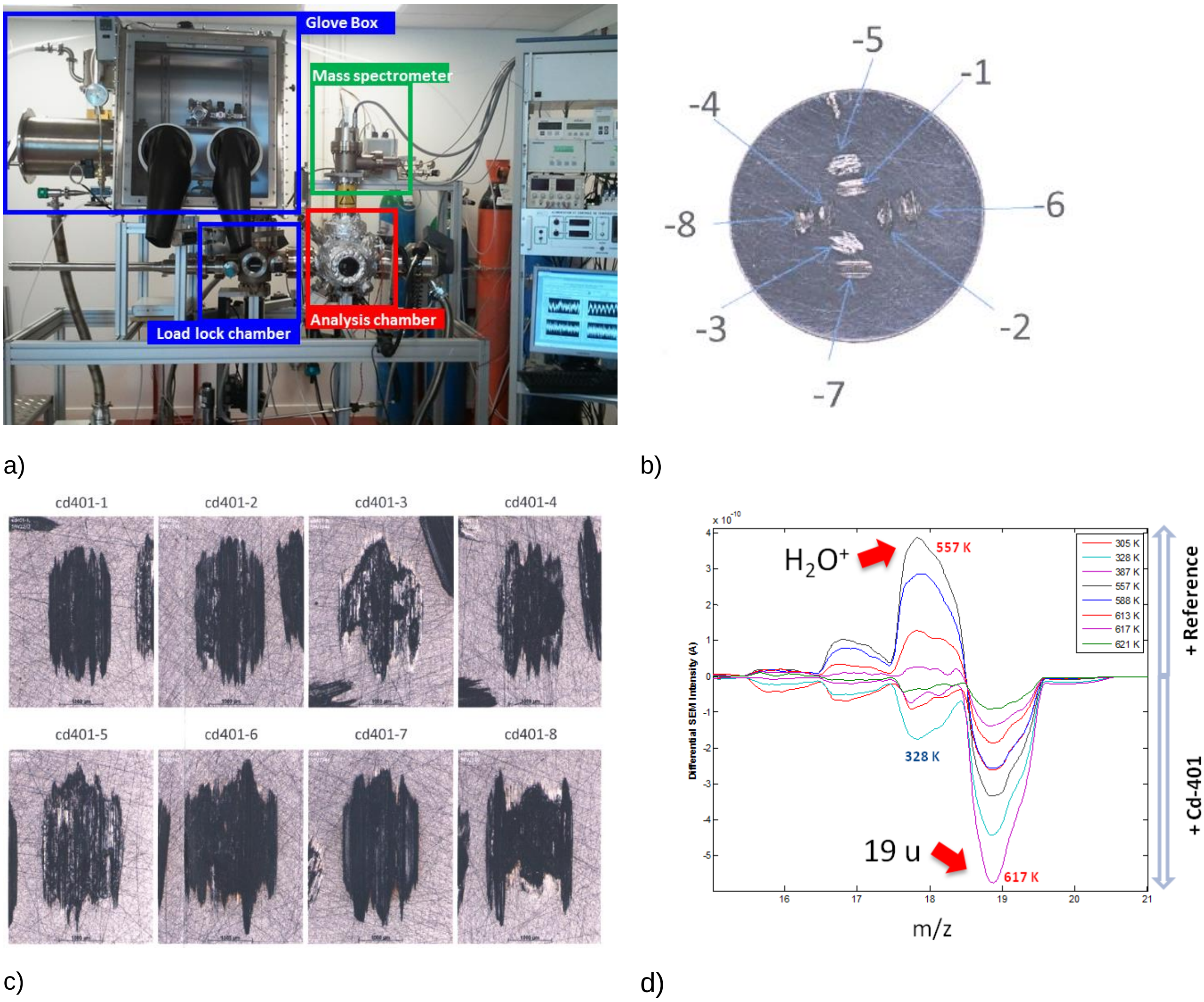


Figure 16, Thermal Desorption mass Spectrometry (TDS), a) measurement system, b) test sample supplied by BAM, c) wear scars on sample, d) results.

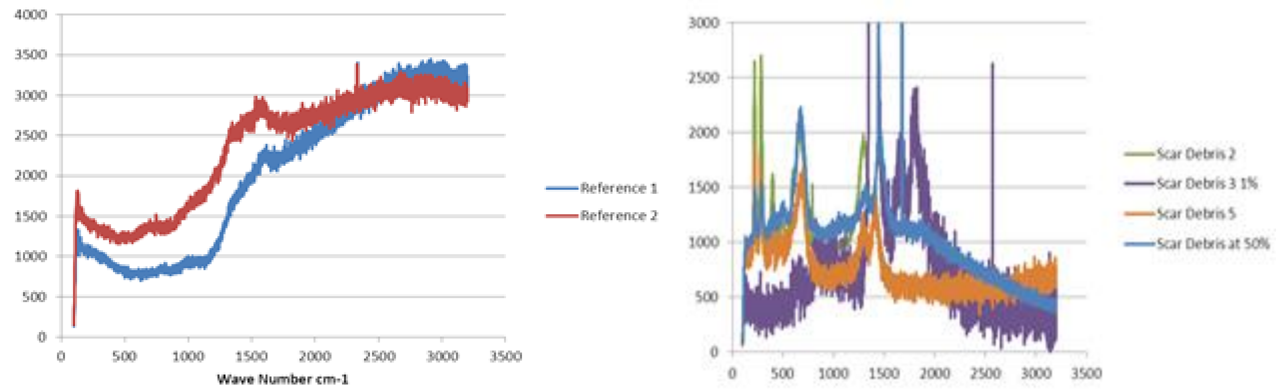
Before the study, samples were cleaned by thermal desorption in a vacuum at 600 °C for 2 hours to desorb the oil used during the wear tests. Then after a two-week exposure to lab air, reference and damaged samples have been successively analysed by thermal desorption. It was found that for temperatures between 300 and 450 K, there is higher desorption of H_2O and CO_2 in the damaged sample than in the reference sample (Figure 16d). Above 450 K it is the reference sample which desorbs more than the damaged sample. For temperatures between 300 and 630 K, masses 19 u, 69 u (possible fragments of CxHy+ or CxHyO+) and 119 u also desorb more on the damaged sample than on the reference sample. The meaning of these results is not clear, but it does show that differences in the desorption characteristics of a steel sample occur when the sample is worn.

Raman spectroscopy

The feasibility of using Raman spectroscopy to evaluate changes to the chemistry of the wear contact was explored by both NPL and BAM. Both ex-situ (examination of a wear track on a sample removed from a test) and in situ (wear contact on sample examined through a sapphire contact) experiments were carried out.
The ex-situ experiments were carried out on laboratory Raman microscopes. The wavelength of IR radiation used in these experiments was 514 nm. These instruments have a lateral resolution determined by the wavelength of the IR light used in the instrument (~1.5 µm). Typical results are shown in Figure 17 showing distinct differences in the spectra between the worn and unworn steel samples rubbed against an alumina ball.

These spectra are complex and show peaks characteristic of alumina as well as peaks from unknown species on the surface.

The in situ experiments were not successful. In these experiments portable Raman devices that operated at a wavelength of 732 nm were used to observe the wear contact. However, there were no differences in the spectra between worn and unworn samples.



a) b)

Figure 17, Raman spectra produced by 514 nm incident radiation, a) unworn steel sample, b) wear track on the steel sample worn by an alumina ball.

Objective 5: The assessment of methods to monitor the degradation of surfaces due to wear.

Evaluation of mechanical damage

Two techniques were evaluated for the measurement of the degradation of mechanical properties during wear. These were nanoindentation and laser surface wave spectroscopy where a fast laser beam is used to excite surface acoustic waves in the surface of a sample. Analysis of the shape of the resulting dispersion curve yields information on the mechanical properties of the surface. Both techniques were explored by both NPL and PTB. However, the laser surface acoustic waves technique was found to damage the carbon based coatings examined as part of the work, so these measurements were not considered further.

With respect to the nanoindentation, six samples (including the substrate) were tested to a full ISO14577-4 analysis (Table 1).

The plane strain modulus (E*) and the hardness (H) values were obtained. The results are also given in Table 1. It can be seen that as expected the TiAlN coating is the hardest of the materials examined at 16.1 GPa, with the steel having a lower hardness at 12.1 GPa and the three DLC coatings have similar lower hardness values of about 10.5 GPa. The materials with the highest reduced modulus was the TiAlN at 342 GPa, the steel had a reduced modulus of 285 GPa and the DLC coatings had a range of reduced moduli from 139 GPa for the Graphit-iC down to 84 GPa for the Hybrid DLC coating. PTB got similar results from their measurements.

The same samples were subjected to pin-on-disc wear tests, but it was found that the results of the nanoindentation experiments were the same as before wear testing, taking into account the measurement uncertainty of the measurements.

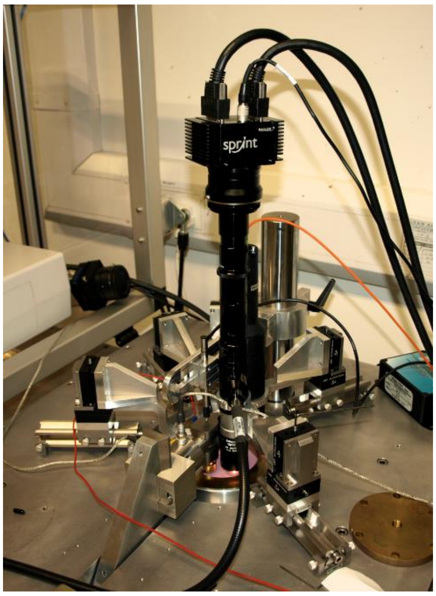
Table 1, Results of NPL nanoindentation measurements on MADES samples

Specimen description	E*, GPa	H, GPa
Steel	285	12.1
TiAlN Coating (t ≈ 4 µm)	342	16.1
Graphit-iC coating (t = 2.46 µm)	139	10.5
Dyon-iC coating (t = 2.6 µm)	95	10.5
Hybrid DLC coating (t = 2.3 µm)	84	10.4

Real-time observation of degradation from wear

NPL carried out experiments using its integrated pin-on-disc tribometer to make observations of the degradation of wear surfaces in real-time whilst wear was occurring. BAM helped NPL through advice and discussions on the work. The integrated tribometer uses a novel loading mechanism to ensure that the full surface of the test disc is accessible to sensors. The sensors that are fitted to the system include a Linear Voltage Displacement Transformer (LVDT) for the measurement of total system wear displacement, a chromatic aberration displacement probe for the measurement of the displacement of the wear track on the test disc, load cells to measure both the applied load and the friction force generated in the test, a linescan camera which enables real-time imaging of the wear track during the wear test, and an electrostatic sensor.

Figure 18 shows the integrated tribometer and some typical results. One of the issues that can be seen in Figure 18b is that the size of the sensors is quite large, limiting the applicability of this technology to test systems. However, it is known that the sensors are currently being redeveloped to make them smaller and more convenient to use. Figure 18d shows the displacement and friction results for a wear test where a bearing steel ball was worn against an alumina disc. The chromatic aberration probe enabled the measurement of the wear displacement or wear to both components of the wear couple at the same time. This has not been achieved previously. It can be seen that the pin wear (red) is much more than the wear to the disc (green). Little overall wear to the disc took place, but it was noticeable that a small amount of disc wear initially took place (loss of material from the surface), but after that a gradual incremental build-up of material on the surface of the disc took place (negative wear). When the images of the disc obtained with the line-scan camera are examined (Figure 18c) it can be seen that the early loss of material from the disc wear track is due to localised removal of material from the surface leaving the surface in a pitted state. Following this, small areas of transferred material are seen on the surface and these grow in size as the test proceeds. These transferred layers are not static but move around on the surface, appearing and disappearing. The growth of these areas is associated with an increase in the variability in the friction (blue). It is thought that this variability in friction is associated with the way these larger areas of transferred material break away from the disc surface.



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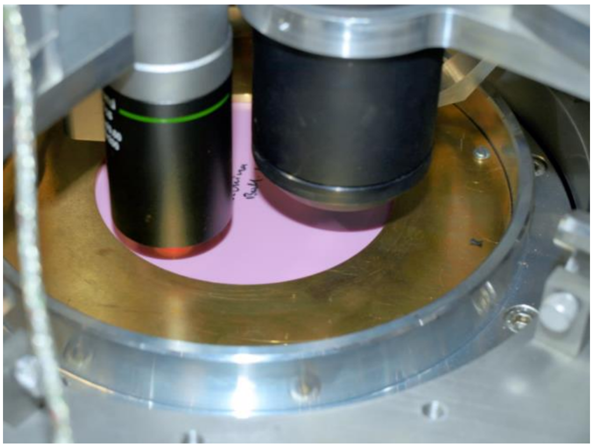

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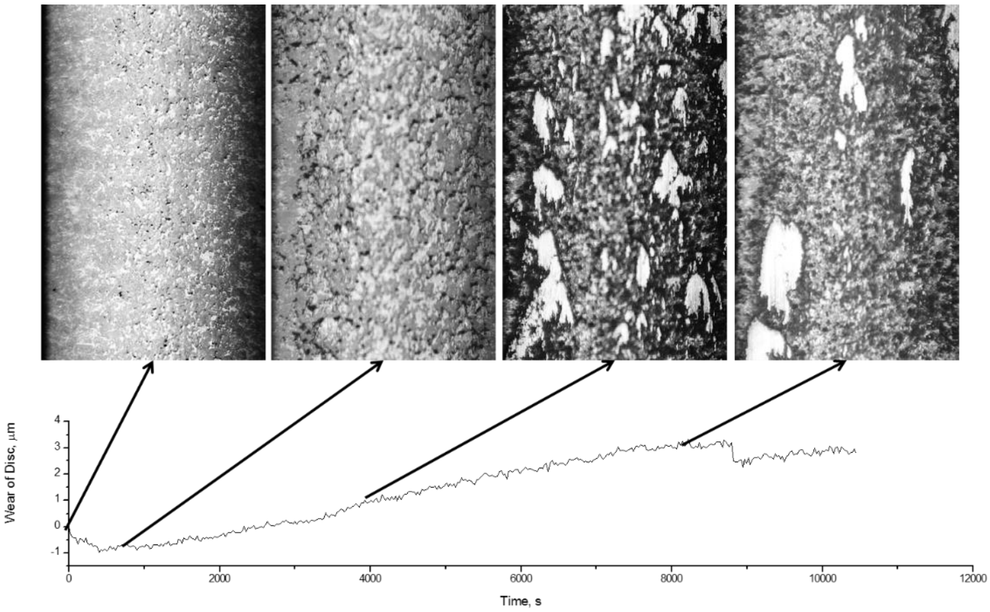

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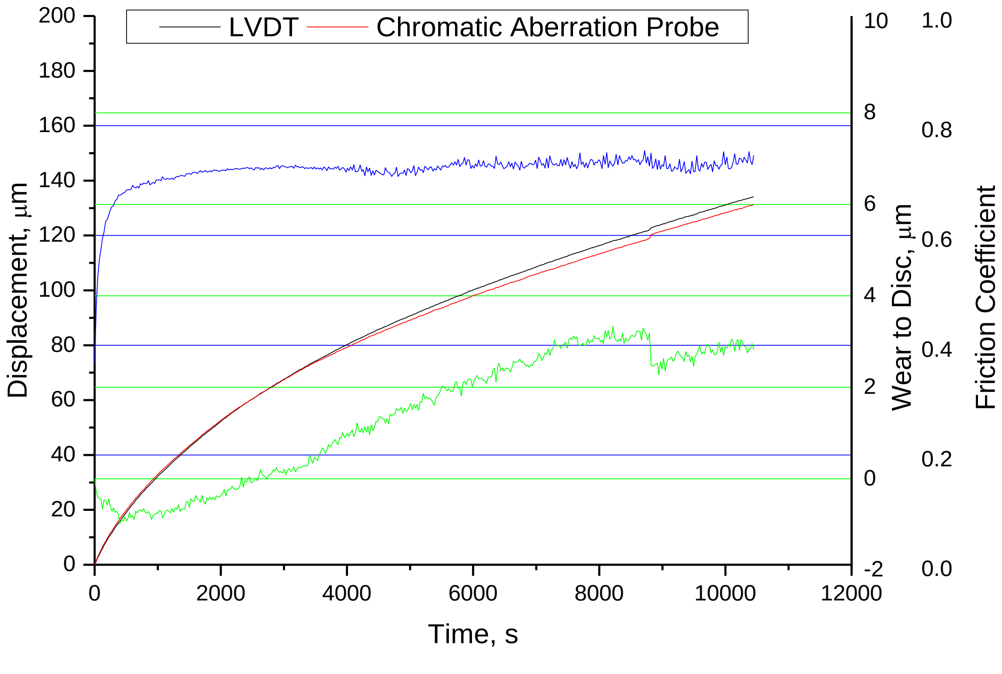

d)

Figure 18, Real time observation of surface damage, a) integrated pin on disc tribometer, b) close-up showing line-scan camera lens and chromatic aberration displacement probe, c) composite image showing images extracted from a real-time video of the wear surface indicating when image was captured on the trace of the disc wear, d) friction and displacement results.

Summary

The key results of the work have led to the development of new measurement capabilities and procedures in the following areas:

- The development of improved techniques for the measurement of small volumes and material loss from wear (nano-scale), with sensitivity to achieve relative measurements of 0.1 µm or less over a period of several days. The techniques included the application of AFM to the measurement of wear, the development of relocation profilometry, the analysis of electron interactions with materials as a basis for topographical measurement, and the application of chromatic aberration probes.
- The development of traceable, self-calibrating, stable measurement techniques for the determination of the long-term friction performance of low-friction coatings. Two different world leading technologies were developed which were the use of high precision miniaturised interferometry, and novel cost effective self-zeroing friction measurement.
- The development of measurement methods for the measurement of the temperature of tribological contacts (i.e. at the interacting surface). Three different techniques were proven to be feasible based on IR imaging, and the European leading development of thin film sensors and fibre-optic based probes.
- The assessment of methods to monitor the degradation of surfaces due to wear with world leading capability developed for the measurement and visualisation of damage to surfaces from wear.

4 Actual and potential impact

Dissemination of results

To ensure uptake of project results a variety of channels was used to disseminate the findings to the research and industrial communities. In addition to peer-reviewed scientific papers (listed below) presentations at international scientific meetings and conferences, the project held international workshops and webinars with the end-user community and developed a series of good practice guides for industrial users:

- Good practice guide on the assessment of chemical changes at the wear interface
- Good practice guide on assessment of small wear volumes
- Good practice guide on the assessment of wear to tools
- Good practice guide on the assessment of “zero wear”
- Good practice guide on on-line assessment of temperature (to be finalised)

These will be available shortly on the EURAMET website.

Impact on standardisation:

The research consortium worked closely with standards organisations and it is anticipated that new procedures for assessing wear and friction will be developed and adopted by the relevant technical committees within CEN, ISO and ASTM. Partners are active in these committees, thus giving a straightforward route to standardisation. Already project results have been used to contribute to:

- The development of a new standard for wear testing of diamond-like carbon coating in ISO standard *TC 107 Metallic and other inorganic coatings.*
- The development of a new ASTM standard *G211-14 Test Method for Conducting Erosion Tests by Solid Particle Impingement Using Gas Jets* published in 2014
- As the basis for work to develop a new standard on nano-scratch testing, initially within *CEN 352 Nanotechnologies* and eventually through *ISO 229 Nanotechnologies*. This work was approved and started in November 2015.

Early impact on industry:

A number of project outputs have already been adopted by industry including:

- Use of the new measurement technology developed in the MADES project in core NPL project work including consultancy work for industry.

- Techniques developed in the project have inspired an SME instrument manufacturer to develop low-wear volume measurement devices. The development of these devices has been greatly enhanced by the knowledge they gained during the MADES project.
- The new measurement technology is being used in collaborative work within Annex IX of the Implementing Agreement for Advanced Materials for Transport of the International Energy Agency which is concerned with the development of better surface engineering for internal combustion engine components.

Over the longer term it is expected that the techniques developed will be adopted by industry and used to develop further advances in low-friction, low-wear surfaces; leading to improvements in process efficiency, in the performance of products and components, and reductions in the environmental impact of European industry.

5 Website address and contact details

A public website was created where the main public deliverables have been made available for the end-users and to keep them informed about project meetings and events:

http://projects.npl.co.uk/engineered_surfaces/

The contact person for general questions about the project is Dr Mark Gee (mark.gee@npl.co.uk).

6 List of publications

1. A. Kovalev, M. Hartelt, D. Spaltmann, R. Wäsche, M. Woydt, *Zero Wear (Null-Verschleiß)*, Proceedings of 53. Tribologie-Fachtagung, ISBN 978-3-00-039201-6, 53. Tribologie Fachtagung Bd. II, 43/1-43/13
2. Julian Le Rouzic, Tom Reddyhoff, *Development of Infrared Microscopy for Measuring Asperity Contact Temperatures*, ASME Journal of Tribology, December 2012
3. D. García-Jurado, J.M.Mainé, M.Batista, L. Shaw, M.Marcos, T. Hausotte, *Metrological Evaluation of Secondary Adhesion Wear Effects in the Dry Turning of UNS-A92024-T3 Alloy through Focus-variation Microscopy (FVM)*, Procedia Engineering, Volume 63 (2013), pp. 804–811
4. A. Kovalev, M. Hartelt, D. Spaltmann, R. Wäsche, M. Woydt, *Zero wear (Null Verschleiß)*, Tribologie und Schmierungstechnik, Vol. 60 (2013) pp. 5-12
5. N. Myshkin, A. Kovalev, D. Spaltman , M. Woydt, *Contact mechanics and tribology of polymer composites*, Journal of Applied Polymer Science, Vol. 131 (2014), pp. 39870(1)-39870 (9)
6. G. Dai, F. Pohlenz, A. Felgner, H. Bosse, H. Kunzmann, *Quantitative analysis of nano-wear on DLC coatings by AFM*, CIRP Annals - Manufacturing Technology, Vol. 62 (2013) 543–546
7. Julian Le Rouzic, Tom Reddyhoff, *Spatially resolved triboemission imaging*, Tribology Letters, January 2014
8. R Leach, A Weckenmann, J Coupland, W Hartmann, *Interpreting the probe-surface interaction of surface measuring instruments, or what is a surface?*, Surface Topography: Metrology and Properties STMP, 2 (2014) 3, p. 035001 (10p)
9. W Hartmann, A Weckenmann, *Model-based testing for the verification of the functional ability of microstructured surfaces [Modellbasiertes Prüfen zur Verifikation der Funktionsfähigkeit von mikrostrukturierten Oberflächen]*, Technisches Messen, 81 (2014) 5, pp. 228-236
10. A Weckenmann, W Hartmann, *Verifying the Functional Ability of Microstructured Surfaces by Model-Based Testing*, Measurement Science and Technology, 25 (2014) 9, p. 094012
11. *B. Hemming, P. Andersson, The determination of wear volumes by chromatic confocal measurements during twin-disc tests with cast iron and steel, Wear 338 (2015) pp 95-104*
12. *K Holmberg, A Laukkanen, H Roikainen, R Waudby, G Stachowiak, M Wolski, P Podsiadlo, M Gee, J Nunn, C Gachot, L Li, Topographical orientation effects on friction and wear in sliding DLC and steel contacts, Part 1: Experimental, Wear 330-331(2015)3–22*
13. *A Weckenmann, W Hartmann, Verifying the Functional Ability of Microstructured Surfaces by Model-Based Testing, Measurement Science and Technology, 25 (2014)*
14. *W Hartmann, A Loderer, Automated Extraction and Assessment of Functional Features of Areal Measured Microstructures Using a Segmentation-Based Evaluation Method, Surface Topography: Metrology and Properties, 2 (2014)*
15. *P Cizmar, CG Frase, H Bosse Novel super-fast three-dimensional SEM image simulation Microscopy and Microanalysis, 19 (2013),pp 796 - 797*