Institution: Imperial College London

Unit of Assessment: 12 Aeronautical, Mechanical, Chemical and Manufacturing Engineering

Title of case study: 3. Commercialisation of Instrumentation for Testing and Development of Lubricants

1. Summary of the impact (indicative maximum 100 words)

Research in the Mechanical Engineering Department has led to a series of experimental techniques for measuring the fundamental properties of lubricants in a scientific manner. These include measurement of the thickness, structure and properties of fluid and solid lubricating films, as well as the friction, fatigue and wear behaviour of lubricated surfaces.

Measuring instruments based upon these techniques have been commercialised by a spin-out company, PCS Instruments, which has now become the largest supplier of Tribology test equipment in the world. Over the period 2008-13 it had a turnover of £39.8M (£7.63M in 2012-13). The research has changed the way in which lubricants and lubricant additives are developed, with PCS test rigs in use in all the major lubricant and additive companies, as well as many University tribology research and National Standards laboratories. The techniques have enabled the development by industry of a new generation of high-efficiency lubricants for automotive applications.

2. Underpinning research (indicative maximum 500 words)

Professor Hugh Spikes FREng, Professor Andy Olver and Dr Philippa Cann (all in post 1993-present) have developed a series of novel ways to study boundary lubricating films. These are solid or fluid layers, of thickness in the range 1 to 100 nm, formed on rubbing surfaces in lubricated contacts. The ability to create and measure such films, under controlled conditions, has led to a significant advance in the understanding of lubrication and, in particular, of the mechanisms by which lubricant additives operate. The techniques form the basis of modern experimental Tribology and are used extensively both for the development of practical lubricants and for fundamental research. A series of instruments based on the techniques developed at the College has been commercialised by a spinout company, PCS Instruments Ltd.

The approach is based upon measuring *in situ*, within rubbing lubricated contacts, the thickness (typically less than 100 nanometres), chemical composition and temperature of lubricant films between rubbing surfaces whilst controlling the conditions that generate and destroy such films, e.g. by independently varying the sliding speed and entrainment speed [1-4]. These techniques have greatly advanced the understanding of the nature and properties of boundary lubricating films.

A method for accurately measuring the thickness of boundary films was developed (the optical, ultra-thin film thickness technique, "Ultra") and this was used to demonstrate the effectiveness of polymer absorption as a boundary lubrication mechanism [2]. A variant of this, the "spacer layer interferometry method, SLIM", was devised to map the thickness of the film over the whole area of contact. These techniques exploited recent optical technology to measure and to image, for the first time, in-situ lubricant films that had a thickness as low as 1/500th of the wavelength of light.

Another technique was devised to simultaneously measure friction and film formation over a range of controlled conditions (the "mini-traction machine, MTM" and interferometric attachment, "MTM/SLIM" [3]). The MTM utilises precision numerical control to generate repeatable surface speeds and loads so that friction forces under known shear rates were accessible for the first time. The techniques were used, for example, to explore the kinetics of anti-wear film formation and to show the impact of such films on friction [3]. These two techniques were used together to show that molecules of different polarity within a lubricant blend, fractionate close to polar surfaces. The fractionation process can be exploited to control film generation and friction [4].

The techniques enabled the thickness and properties of friction-reducing additive films within rubbing contacts to be directly revealed for the first time [5]. The research has subsequently been employed by industry in the design of low friction, energy efficient lubricants, as described in section 4.

In parallel to *in-situ* lubricant measurement methods, the team developed techniques for the study of surface damage. One technique was the high-frequency reciprocating “HFRR” wear test, in which a test ball is vibrated electrically at high speed to produce sliding wear in the boundary regime. The method specifically generates a high repetition rate with a low surface speed, ensuring the contact remains in the boundary regime and at a controlled temperature.

Another is the “MPR” [6] technique for studying rolling contact fatigue phenomena using a triple-contact roller configuration. This rig is uniquely able to generate surface stress cycles rapidly (typically 10^6 per hour) without using high surface speeds, so that fatigue behaviour over a wide range of film thicknesses can be studied within a reasonable time-scale. HFRR and MPR rigs have been used to demonstrate and exploit the relationship between lubricant composition, film formation and component durability. An example is the detrimental effect of some anti-wear formulations on micropitting resistance, shown to be due to surface topography effects, and their mitigation by friction modifiers [6].

The combination of these techniques has enabled an improved linkage between measurements of the performance of lubricants in simple laboratory tests and prediction of behaviour in real equipment such as engines and mechanical transmission systems. This has led to their widespread use in industry for the development of lubricant formulations as described in section 4.

3. References to the research * References that best indicate quality of underpinning research.

[1] P.M. Cann, J. Hutchinson, H.A. Spikes, “The development of a Spacer Layer Imaging Method (SLIM) for mapping elastohydrodynamic contacts”, Tribology Transactions, Vol 39, pp. 915-921, (1996) DOI: 10.1080/10402009608983612

* [2] J. Fan, T. Stohr, M. Muller, H.A. Spikes, “Reduction of friction by functionalized viscosity index improvers”, Tribology Letters, Vol 28, pp. 287-298, (2007) DOI: 10.1007/s11249-007-9272-3

[3] L. Taylor, H.A. Spikes, “Friction-enhancing properties of ZDDP antiwear additive. Part I. Friction and morphology of ZDDP reaction films.” Tribology Transactions, Vol 46, pp. 303-309, (2003). DOI: 10.1080/10402000308982630

* [4] G. Guangteng, H.A. Spikes, “The control of friction by molecular fractionation of base fluid mixtures at metal surfaces”, Tribology Transactions, Vol 40, pp. 461-469, (1997) DOI: 10.1080/10402009708983681 **(best paper award)**.

[5] V. Anghel, C. Bovington, H.A. Spikes “Thick-boundary-film formation by friction modifier additives”, Lubrication Science, Vol 11, pp. 313-335, (1999) DOI: 10.1002/ls.3010110402

* [6] E. Lainé, A.V. Olver, M.F. Lekstrom, B.A. Shollock, T.A. Beveridge, D.Y. Hua, “The Effect of a Friction Modifier Additive on Micropitting”, Tribology Transactions, Vol 52, pp. 526-533. (2009) DOI: 10.1080/10402000902745507

4. Details of the impact (indicative maximum 750 words)

As well as supporting the Imperial research and attracting substantial funding from industry, the new test methods have also been commercialised by a spin-out company, now known as PCS Instruments Ltd, (www.pcs-instruments.com). Over the period 2008-13 (6 yr) it had a turnover of £39.8M (£7.63M in 2012-13), with 80% overseas sales, and now employs five Imperial Tribology PhD graduates, one of whom was recruited since 2008. There are a total of 11 permanent staff in the company. All the manufacturing added value associated with the company is in the UK; thus the company supports a range of subcontractors.

The main test rigs sold by PCS that were developed as a result of Imperial research are:

- Elasto-hydrodynamic lubrication ("EHL" or "Ultra") rig, (Ultra-thin lubricant film thickness measurement to 1 nm [2])
- High frequency reciprocating rig, HFRR (friction and wear tester, additive development, international standard for fuel lubricity [8, 9])
- EHL Spacer layer interferometry rig, SLIM (Film thickness distribution to 5 nm) [1]
- Mini-traction machine, MTM (Friction in rolling-sliding contact, basis of low efficiency oil formulation [4])
- Micro-pitting rig, MPR (Micro-pitting, fatigue, durability and friction at high pressure) [6]

The total number of rigs sold is 1300, of which 764 were sold between Jan 2008 and July 2013, as confirmed by the CEO of PCS Instruments Ltd [C].

The development and commercialisation of these test methods has made an impact in two significant areas: (a) their almost universal application in the global lubricants industry where they have become essential tools for the development of lubricant formulations and where they form the basis of a number of international standards [7-9] and (b) the widespread adoption of the techniques in the research community.

The most significant beneficiary has been the global lubricants industry and its customers. This includes *lubricant manufacturers* (major oil companies such as BP, ExxonMobil [10, 11], Shell, Petronas, Sinopec; and specialist suppliers such as Valvoline [12] and Syntech, together with global *additive suppliers* such as Lubrizol, Evonik [13], Infineum [14], Afton and Chevron Oronite. Around 38.5M (2006) tons of lubricants are manufactured annually and the global market has been estimated to have a value of nearly $50bn in 2012 [17]. Vice President Global Commercial Technology [A] for Shell says: "… *lubricant test machines from PCS-Instruments have become the tools of choice for lubricant development in almost all oil and additive companies around the world, including my own.*"

Senior Vice-President of Research and Development [B] , Valvoline, confirms this view: "*In the last decade, the new generation of lubricant test equipment developed by PCS Instruments (MTM, HFRR, EHL film thickness etc.) has led to a striking change in the way that we design and test lubricants. We now depend on PCS test equipment to develop formulations for our automotive engine and drivetrain lubricants at Valvoline and we consider them to be an essential tool for early assessment of lubricant parameters governing efficiency and durability. We currently have four PCS test rigs (one EHL, one HFRR, and two MTMs) in our New Product Development Centre in Lexington, Kentuck*y."

In the period 2008-2012, MTM and HFRR test rigs were cited in 380 US patent applications and in 113 granted US patents, highlighting its importance in specifying these new materials. PCS test rigs have also been sold to 41 University research groups world-wide, making them one of the most widely-used categories of equipment in tribology research laboratories. The technical literature in experimental tribology is dominated by work on PCS rigs, especially the MTM [14-16].

The value of the techniques developed and their impact has been greatly enhanced by the growing concern by the public and by governments worldwide to protect the environment. This has led to changes in lubricant design, primarily to reduce friction and thus reduce fuel consumption and hence CO_2 emissions, but also to provide engine lubricants that maintain durability and are compatible with vehicle exhaust after-treatment systems.

Examples include [10], which patents the addition of high viscosity esters to hydrocarbon oils to increase low speed film thickness and reduce friction. It used the "Ultra" technique and is based directly on [2]. Another is [13], one of many such patents by Evonik concerning the use of functionalised polymers to produce low friction engine/transmission oils. This work is based on the MTM test method and is a consequence of the research described in [3].

The best passenger car engine lubricants are currently about 4-5% more efficient than their early

1990s counterparts as a result of lubricant development based to a large degree on Imperial/PCS test methods. This increase in efficiency corresponds to an annual reduction of nearly 4×10^{10} kg (about 0.1%) of global man-made CO_2 emissions. (600M cars, 10k km/yr, 150 g/km CO_2, 4% reduction, 2010 total 30.6 Gtonnes.)

5. Sources to corroborate the impact (indicative maximum of 10 references.)

Examples of Standards based on the PCS test rigs and product developed and specified using it:

[7] DEXTRON® VI *Automatic Transmission Fluid Specification* – EHDPROC-11 Film Thickness Requirement. GMN10060, General Motors, April 2005 . Specification details available on request.

[8] The Coordinating European Council "Measurement of Diesel Fuel Lubricity" http://www.cectests.org/ (Archived at https://www.imperial.ac.uk/ref/webarchive/4rf . 2013 CEC F-06-96 http://www.cectests.org/tabtest1.asp?search=CEC+F-06-96+&searchBtn . Also available here

[9] ISO 12156-1 "1997 Diesel Fuel - Assessment of Lubricity Using the High Frequency Reciprocating Rig (HFRR). Part 1 Test Method" https://www.iso.org/obp/ui/#home

Examples of patents and other industrial publications based on PCS test rigs derived from Imperial research. [*Rig used*, organisation, country]

[10] Bovington, CH, Fuel economy additive and lubricant composition containing same, US patent 5962381 (1999) [*Ultra*, Exxon Chemicals, USA].

[11] Sullivan, WT, Webster, MN, Oumar-Mahamat, H and Brandes, EB, "Lubricating fluids with low traction characteristics", US patent 7732389, 2010 [*MTM*, Exxon-Mobil, USA]

[12] Saini, M, Lockwood, FE, Wang, JC and Mustoff, CF, Contribution of Oil Traction to Diesel Engine Cam Galling, SAE Paper number: 2001-01-3552 (2001) 1-5 DOI:10.4271/2001-01-3552. [*MTM*, Valvoline and Cummins, USA]

[13] Eisenberg, B, Jansen, D, Suchert, E, Sitihulka, MK and Voigt, L "Polyalkyl(Meth)Acrylate for Improving Lubricating Oil Properties", US patent 2013/0079265 ((2013) [*MTM*, Evonik, Germany]

Tribology literature which refers to the use of the PCS rig

[14] Castle, A, and Arrowsmith, S, Modelling lubricant related fuel economy in heavy duty diesel engines, Tribology and Interface Engineering Series 41 (2003) 491-500 DOI 10.1016/S0167-8922(03)80163-3 [*Ultra and MTM*, Infineum, UK]

[15] Costello, G, Effects of basestock and additive chemistry on traction testing, Tribology Letters, Vol. 18, No. 1, January 2005. DOI: 10.1007/s11249-004-1761-z [*MTM*, Chemtura, USA]

[16] Yakubov, GE, James McColl, J, Bongaerts, JHH, Ramsden, JJ, Viscous Boundary Lubrication of Hydrophobic Surfaces by Mucin, *Langmuir* 25 (2009) 2313-2321 DOI:10.1021/la8018666. [*MTM*, Unilever and SKF, Netherlands]

Other sources for corroboration of the industrial impact:

[17] www.thestreet.com/story/11064815/1/global-lubricant-demand-is-forecast-to-reach-488-billion-in-2012.html (Archived on 09 October 2013: https://www.imperial.ac.uk/ref/webarchive/3wf)

[A] Vice President Global Commercial Technology, Shell to confirm that PCS-Instruments have become the tools of choice for lubricant development.

[B] Senior Vice-President of Research and Development, Valvoline to confirm changes in how they design and test lubricants.

[C] CEO, PCS Instruments Ltd. to Confirm PCS activity and how it is underpinned by Imperial research