Institution:	**Imperial College London**
Unit of Assessment: 13A – Electrical & Electronic Engineering	
Title of case study:	**Case 2 - Device Applications of 3D Silicon Microstructures**

1. Summary of the impact

The Optical and Semiconductor Devices group led by Richard Syms has been a major innovator in fabrication methods for 3D silicon microstructures, and has developed a wide range of novel devices and techniques based on these innovations. The impact of their research has been to:

I1) bring the power of mass spectrometry to individual chemists' lab benches and fume hoods, raising their effectiveness and productivity through the launch in 2011 of the world's first commercial desk-top mass spectrometer by **Microsaic Systems plc**, a start-up company founded by members of the group;

I2) create a second start-up company, **Nexeon Ltd**, to manufacture nanostructured silicon anode materials, resulting in reduced battery size and weight for electric vehicles and portable electronics;

I3) add to mankind's journey of discovery in space with NASA's **Phoenix Mars Mission** in 2008, as part of the Atomic Force Microscope team, helping to investigate the presence of liquid water on the surface of Mars.

2. Underpinning research

The research activity at Imperial College in microsystems (also MEMS – micro-electro-mechanical systems), led by Professor Richard Syms, began in 1993. MEMS uses methods from integrated circuit technology to develop devices with optical, mechanical, fluidic and other functions, but is restricted by the largely 2-dimensional nature of IC processing. From the beginning, Syms' group focused on developing methods to make 3D microstructures. Their first major contribution was the surface tension self-assembly method (1995) [R1], which allows batch fabrication of 3D structures – such as optical scanners and inductors for high frequency circuits on silicon – by rotating lithographically defined 2D parts. Other devices developed by the group included the first microengineered circuit breaker (2004), and radio frequency switches (2006).

The group's emphasis on 3D microengineering extended to other techniques such as precision optical fibre alignment. In 1995 Syms began a project to miniaturise quadrupole mass spectrometers (QMS). The use of silicon alignment structures for the quadrupole rods, adapted from the fibre alignment methods, was a breakthrough that allowed the Imperial team to develop the world's first microengineered QMS (1996) [R2]. Other developments drawing heavily on 3D structuring included a self-aligned electrospray ion gun (2005) [R3], the basis of an electrospray mass spectrometer. Research was supported initially by two EPSRC projects and later by DTI/LINK in collaboration with the pharmaceutical company GSK. The latter project resulted in the realization in microengineered form of three major building blocks needed to construct a compact mass spectrometer for life science applications: a spray source, a vacuum interface, and a high-performance mass analyser.

Another technique pioneered by the group for 3D structures involves the use of natural resist, a method by which thin films "ripen" into sub-micron islands, which then act as masks for deep etch processes to form structures such as nanopillars (1999) [R4]. This work was led by Emeritus Professor Mino Green. It has the advantage compared to other sub-micron patterning techniques that it can be used to process large areas rapidly and at low cost, making it suitable for applications where electron-beam writing, for example, would be prohibitively expensive. A number of applications enabled by this technique were developed, including high surface area substrates for Raman spectroscopy. One application with particular commercial impact was opened up when the group demonstrated that such nano-structured surfaces could be used in silicon anodes for lithium ion batteries (2003) [R5]. The energy density in lithium batteries is limited by the anode's ability to dissolve lithium, which in silicon causes damage due to lattice strain. The ability to relax such strain using a high density nanopillar array allows significant energy density enhancement at low production cost.

Professor Tom Pike, who joined the group in 2001, further developed the field by using 3D structuring of silicon to create textured substrates for the microscopic examination of the soil of Mars. These substrates trapped and separated samples collected by a Mars surface lander for detailed imaging by an optical and atomic force microscope (2010) [R6]. The combination of Pike's space research background and the group's microfabrication capabilities, including precision through-wafer etching, provided the basis from which this innovation arose.

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

[R1] Green P.W., **Syms R.R.A., Yeatman E.M**. "Demonstration of three-dimensional microstructure self-assembly" IEEE/ASME J. Microelectromech. Syst. 4, 170-176 (1995) DOI: 10.1109/84.475543

[R2] **Syms R.R.A,** Tate T.J., Ahmad M.M., Taylor S. "Fabrication of a microengineered quadrupole electrostatic lens" Elect. Lett. 32, 2094-2095 (1996) DOI: 10.1049/el:19961362

[R3]* **Syms R.R.A.**, Zou H., Bardwell M., Schwab M.-A. "Microengineered alignment bench for a nanospray ionisation source" J. Micromech. Microeng. 17, 1567-1574 (2007) DOI:10.1088/0960-1317/17/8/020

[R4] **Green M.**, Tsuchiya T. "Mesoscopic hemisphere arrays for use as resist in structure fabrication" J. Vac. Sci. and Tech. B17, 2074-2083 (1999) DOI: 10.1116/1.590875

[R5]* **Green M.,** Fielder E., Scrosati B., Wachtler M., Serra Moreno J. "Structured silicon anodes for lithium battery applications" Electrochem. & Solid-State Letts. 6, A75-A79 (2003) DOI: 10.1149/1.1563094

[R6]* Goetz, W., **Pike, W.T.** et al., Microscopy analysis of soils at the Phoenix landing site, Mars: Classification of soil particles and description of their optical and magnetic properties", J. Geophys. Res. 115, 2156-2202 (2010) DOI: 10.1029/2009JE003437

4. Details of the impact

We now provide details of the 3 aforementioned impacts and how they are underpinned by research in section 2:

I1) **Desktop Quadrupole Mass Spectrometer** by **Microsaic plc** - The novel 3D micro-engineering methods pioneered by the group [R1], and applications arising from these [R2], provided the basis for the establishment by Syms, Holmes and Yeatman of Microsaic Systems Ltd in 2001. The company obtained a pipeline agreement to acquire intellectual property arising from the micro-engineering research of the founders, and the unique technologies arising from the group's research provided the basis for a series of contracts and awards which funded the growth and development of the company. In 2006 mass spectrometers were selected as the main business focus, and Microsaic did a private equity fundraising of £4M to begin a programme of product development in this field. Subsequently, Microsaic developed a set of micro-engineering innovations [e.g. E1] that enabled the launch of the world's first commercial compact mass spectrometer for liquid analysis, the MiD, in 2011. Microsaic has over 40 granted patents, of which the substantial majority are based wholly or in part on research carried out in the EEE department by the founders.

Mass spectrometry (MS) is the "gold standard" method for identifying the constituents in a sample, and is used in an enormous range of applications including drug development and production, forensics, food and drink analysis, security, water quality and many others. However, although MS is a $7B market, the size, cost and complexity of MS instruments greatly restrict their deployment, so MS is generally provided in analytical laboratories, to which users must bring their samples. The Microsaic system is the size of a desktop PC, has no external pumps or other peripherals, and thus can be deployed much more widely. For example, thousands of chemists work routinely with chromatographic separation methods such as HPLC (high performance liquid chromatography) in the development, synthesis, purification and production of drugs and other chemicals. Typically they have their own HPLC equipment, but to definitively assess their samples they use a central MS facility. Microsaic has made it practical for each HPLC station to include mass spectrometry. The MiD also benefits from much lower power, gas and solvent consumption than conventional MS instruments.

Quadrupole mass spectrometers work by ionising the molecules in the sample, and then "flying" the ions in a vacuum chamber through a mass filter, which uses electric fields to separate them by mass-to-charge ratio, directing the selected mass onto a detector. To make miniaturisation possible, Microsaic developed three micro-engineered components: the miniature quadrupole filter itself, an electrospray ionisation source, and a vacuum interface chip for bringing ions into the vacuum system. All are based on Imperial's research [R2, R3].

Microsaic's 3500 MiD was launched at a major US trade fair, Lab Automation, where it won a prestigious New Product Award. In 2012 the 3500 MiD won an R&D 100 award as one of the major technological innovations of the year. Initial systems were purchased by a number of major pharmaceutical companies and university labs in the first year of the product's release, and these quickly led to new methods being adopted in those labs. For example, Merck & Co Inc, in collaboration with Microsaic and Imperial College, published the first demonstration of the use of a miniature MS with HPLC [E2]. Subsequently, Cambridge University researchers applied Microsaic's compact MS to continuous reaction monitoring, and showed that the use of an on-line mass spectrometer "*enabled the flow conditions to be quickly tuned for safe operation and optimal generation of the desired product*", and "*paves the way to discovering untapped synthesis methods*" [E3].

In May 2012 Microsaic announced a contract with Biotage AB, a major supplier of flash chromatography equipment, to supply a minimum of 50 MS instruments per year, rebadged as the Isolera Dalton Mass Detector, as part of a combined MS-flash system. This system was launched by Biotage in April 2013. The system identifies compounds by mass in real time during flash separation, "*leading to greater confidence in purification and a significant saving in time and money. This combination of identification and purification removes complex off-line analytical steps…, vastly increasing throughput and putting the entire purification and analysis in the hands of the chemist*" [E4]. Microsaic was listed on the London Stock Exchange (AIM) in April 2011. It employs over 35 people, has a market capitalisation of £23.4M (on 21/10/13) and has raised a further £11M in equity funding.

I2) **Nanostructured silicon anode materials for lithium batteries** - The research group's other innovation in microstructured silicon that led to a new company was high capacity anodes for lithium batteries based on "natural" lithography [R4]. Initial proof of concept research by Prof. M Green gave a clear indication that this technology could provide substantial enhancement to battery capacity, while being scaleable to production volumes at reasonable cost [R5]. As a result the company Nexeon Ltd was established in 2006 [E5]. Between 2009 and 2011 Nexeon attracted over £50M in investment funds, which it has employed to develop and scale up the technology. It is one of the "top three portfolio companies" [E6] of Imperial Innovations plc, which itself has a market cap. of £256M (6/10/13). Nexeon has won a number of awards in the Clean Tech sector, including the Rushlight Award for Energy Efficiency in 2013 [E7], a Climate Week Award in the Best Technological Breakthrough category (2012), and has been named twice in the Global Cleantech 100 list (2011 and 2012). It has "*signed a development agreement with a major consumer electronics and battery OEM*" (2012) [E6] and "*completed a strategic deal with WACKER Chemie AG to provide access to engineering expertise for* *the design and construction of a 250 tonne per annum plant*" (2013) [E6].

I3) **NASA's Pheonix Mars Mission** - The external impact of the group's microengineering research has not only been through commercialisation of its technology. One example is the work of Prof. Tom Pike and colleagues on microstructured substrates for soil analysis [R6], which led to these substrates being included as part of the atomic force microscopy instrument on the 2008 NASA Phoenix mission to Mars [E8,E9]. The mission, and the microscopic analysis of the Martian soil, were successful, and added important evidence in the ongoing quest to investigate the possibility of life on Mars, by assessing the exposure of soil samples to liquid water [E8]. These results generated considerable public interest and media coverage [E9] and provided the context for the NASA's subsequent 2012 Mars Science Laboratory mission [Science articles citing E9]. A micro-seismometer based on the group's 3D silicon micromachining technology has also been chosen for inclusion in the forthcoming 2016 Insight Mission to Mars, where the Imperial group is the only team from the UK included in this multinational project led by NASA [E10].

5. Sources to corroborate the impact

[E1] US Patent 7,786,434 B2 : Syms R.R.A., Moseley R. "Microengineered vacuum interface for an ionisation system" granted on Aug 31, 2010 to Microsaic, describing an interface for a miniature mass spectrometer.

[E2] A. Malcolm et al, "A miniature mass spectrometer for liquid chromatography applications", Rapid Commun Mass Spectr. 25 (21), 2011, pp 3281–3288. DOI: 10.1002/rcm.5230.

[E3] D.L. Browne, "Miniature Mass Spectrometry and On-line Analysis of Flow Chemistry Research", The Column 9(6), 2013, pp 2-5. http://www.chromatographyonline.com/lcgc/Articles/Miniature-Mass-Spectrometry-and-On-line-Analysis-o/ArticleStandard/Article/detail/809098 Archived here on 23/10/13.

[E4] Biotage AB Web site: http://www.biotage.com/product-page/isolera-dalton ("Read More" tab). Archived on 23/10/2013 at https://www.imperial.ac.uk/ref/webarchive/y8f.

[E5] Nexeon web site: http://www.nexeon.co.uk/about/history/ Archived on 23/10/1013 at https://www.imperial.ac.uk/ref/webarchive/7zf

[E6] Imperial Innovations Interim Report HY2013, 22/3/2013 – page 1 http://www.imperialinnovations.co.uk/investor-relations/documents/ Archived here on 23/10/2013.

[E7] Nexeon Rushlight Award 2009: http://www.rsaaccreditation.org/index.php/rushlight-awards-cs/109-nexeon-clean-energy-award . Archived on 23/10/2013 https://www.imperial.ac.uk/ref/webarchive/8zf.

[E8] Paper announcing results of the Phoenix mission microscope station: Pike W. T. Staufer, U., Hecht M. H., Goetz W., Parrat D., Sykulska-Lawrence H., Vijendran S., Madsen M. B. "Quantification of the dry history of the Martian soil inferred from in situ microscopy" Geophys. Res. Lett., 38, L24201 (2011), DOI: 10.1029/2011GL049896.

[E9] Example of press coverage of Imperial's contribution to Phoenix mission: http://www.msnbc.msn.com/id/46282840/ns/technology_and_science-space/t/surface-mars-may-be-too-dry-any-alien-life-exist/ Archived on 23/10/2013 https://www.imperial.ac.uk/ref/webarchive/9zf.

[E10] UKSA press release on Imperial's microseismometer's inclusion on NASA's InSight mission to Mars: http://www.bis.gov.uk/ukspaceagency/news-and-events/2012/Aug/new-mars-mission-to-take-first-look-at-whats-going-on-deep-inside-the-red-planet. Archived on 23/10/2013 at https://www.imperial.ac.uk/ref/webarchive/c1f.