Institution: University of York

Unit of Assessment: 13, Electrical and Electronic Engineering, Metallurgy and Materials

Title of case study: Developments toward low and ultra low voltage electron microscopy

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

Since the 1950s, when they were first developed, scanning electron microscopes (SEMs) have revolutionised science. However, the large physical size of these machines and their aggressive treatment of samples has limited their use. Now research carried out by Professor Mohamed El-Gomati has led to the development of products for global companies including Agilent, Carl Zeiss and Shimadzu. These products include the world's first low-voltage desktop SEM, capable of resolving features smaller than 5 nanometres, and handling radiation sensitive samples such as biological and medical materials, novel photoresists, nanotubes and nanorods. The smaller size has also improved accessibility of such instruments leading to significant efficiency gains for companies and academia worldwide.

2. Underpinning research (indicative maximum 500 words)

Conventional SEMs are large in physical size and often require their own room, along with dedicated operator and facilities. Having to rely on a centralised SEM for imaging hampers the innovation process and can delay the time to market. Researchers and developers are forced to plan their research and product development programs around the limited availability of a centralised SEM. Furthermore, conventional SEMs are limited to solid, inorganic samples small enough to fit inside the vacuum chamber that can handle moderate vacuum pressure.

In order to overcome some of these limitations, Professor El-Gomati and his team from the Department of Electronics at the University of York, have been developing and carrying out research on the electron source for electron microscopy. There are two electron source types used in electron microscopes: thermionic and field emission. Thermionic sources only require high vacuum for their operation but suffer from a large source size (>10μm), short lifetime (~ 100s hrs), emit electrons with a spread in their initial energies (>1.5eV), and added together cause large final beam sizes, which are not suitable to resolve nanostructures. Field emission sources have smaller source size (~30nm), a small energy spread (<0.6eV), a longer life time (>10,000hrs) and are therefore becoming increasingly popular – especially for nano-scale technologies. However, conventional field emission sources are limited in voltage used (3,000-5,000V) and the corresponding current density (300-500μA/str). Whilst this suited the existing microscope technology, operating outside these parameters to suit new developments proved inaccessible, leading to beam current instability (for low current density operation) or the source's total destruction, when operated for high current applications. The York team have improved the electron source in three major ways and has helped improve the understanding of low energy SEM imaging.

Miniaturisation of field emission technologies. This research in electron optics and field emission cathodes has led to the development of several small-sized electron columns, some measure <10cm in height, typically 10 times smaller than conventional column sizes. This development, which is based on using electrostatic electrodes instead of the normally used magnetic coils that require water cooling, is vitally important in reducing the size of SEMs. In addition, SEM miniaturisation requires low voltage cathodes, and this trend of miniaturisation led to the development of an integrated cathode-extractor electrode as part of a pre-aligned column [1, 2].

High angular intensity Schottky electron source. Operating conventional Schottky emitters beyond 0.5mA/str leads to their destruction, which has limited their use in high current applications. El-Gomati and his group have been researching alternative source configurations where the electron optical characteristics of the electron gun part are (focal properties) taken into consideration when realising the electron emitter. In collaboration with Shimadzu Corp. of Japan, El-Gomati and co-workers have developed a new Schottky emitter capable of operating well beyond 1mA/str. [3]

Operating a LaB_6 cathode in 'Virtual source Mode' Operating ultra-high vacuum (UHV), as is required for field emitters, is both expensive and time consuming. Lanthanum hexaboride (LaB_6) electron guns depend on thermionic emission of electrons from a heated LaB_6 crystal, which are ~10x brighter than tungsten and have significantly longer lifetimes, but require higher vacuum levels, often increasing the SEM's cost. The emitting area of LaB_6 is large thus reducing its brightness and consequently its beam current capability. El-Gomati and co-workers demonstrated in 2010 [3] the advantages of operating the LaB_6 cathode in the field emission mode. In doing so, they were able to exploit smaller energy spread and source size than in the thermionic mode, which led to a higher brightness source that is essential for high resolution microscopy [4].

Low Voltage Microscopy Research in low voltage microscopy mode, led to a better understanding of the signals obtained from the SEM. A new model describing the mechanism of image contrast was proposed in 2001 [5] and a set of guiding rules to assist users was published in 2005 [6]. Further research in low voltage SEM led to a systematic study of backscattered and secondary electron emission signals forming SEM images. El-Gomati's research led to an overhaul of the accepted theory (60 years old), explaining secondary electron emission (Cosslett award, MSA, USA; 2008).

This research was carried out at the University of York, led by Professor El-Gomati (at York as Professor since 1998). Others involved in this research at York are: Roberts, Kudjoe, Bakush, and Zaggout (PhD students) and Prutton,Tear, and Wilson (Research Associates).

3. References to the research (indicative maximum of six references)

[1] A miniature all-electrostatic field emission electron column for surface analytical microscopy. R. Roberts, **M. El_Gomati**, J. Kudjoe, M. Prutton and *S. Bean.* Meas. Sci. Tech., Vol 8, 536-545, 1997. doi:10.1088/0957-0233/8/5/012. Scopus citations (12/11/13): 8

[2] A Manufacturable Miniature Electron Beam Column – *J. P. Spallas, C. S. Silver, L. P. Murray*, *T. Wells*, **M. El-Gomati, Microelectronic** Engineering 83, pp984–989, 2006. Source: http://www.sciencedirect.com/science/article/pii/S0167931706001869 Scopus citations (12/11/13): 12

[3] Enhanced Angular Current Intensity from Schottky Emitters, *S. Fujita*, *T. R. Wells*, *W. Ushio*, *H. Sato*& **M. M. EL-GOMATI** Journal of Microscopy, Vol. 239, Pt 3 2010, pp. 215–222. Source: http://www.ncbi.nlm.nih.gov/pubmed/20701659. Scopus citations (12/11/13): 5

[4] The use of a LaB_6 cathode for high angular current intensity. S. Bakush*, T Wells* and **M. El-Gomati**. Charged Particle Optics VIII, July 2010 Singapore 2010. This conference is the world leading conference series on charged particle optics. *(Available on request)*

[5] Very-low-energy electron microscopy of doped semiconductors. **M. El-Gomati** and *T. Wells*, Appl. Phys. Lett, **79 No.18**, 2931-2933, 2001. DOI: 10.1063/1.1415045 Scopus citations (12/11/13): 31 Work in this paper contributed to the Cosslett Award in 2008.

[6] Why is it possible to detect doped regions of semiconductors in low voltage SEM: a review and update, **M. El-Gomati**, F. Zaggout, H. Jayacody, S. Tear, and K. Wilson., Surf. & Interface Anal. Vol.37, 901-911, 2005. DOI: 10.1002/sia.2108 Scopus citations (12/11/13): 31. 31 Work in this paper contributed to the Cosslett Award in 2008.

All the above references are for research that was carried out at the University of York. All contributors were employed by the university of York at the time of publication except for those in italics, who were commercial collaborators.

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

In 1997 Professor El-Gomati formed York Probes Sources Ltd (YPS Ltd) to exploit his expertise to

collaborate with commercial companies to develop new SEM devices, pushing them beyond their conventional limits. YPS won a SMART award (1997) to develop novel field emitters that incorporated an electron cathode with other electrodes in a single unit to be used in miniaturised and conventional SEMs based on El-Gomati's research at York. Today, this company employs five members of staff and averages an annual turn-over of about £300k. El-Gomati's research group have worked with some of the largest surface science and microscopy companies including Shimadzu, Cameca, FEI Inc., Novelx (Agilent) and Carl Zeiss.
The group has delivered impact in three main areas, as follows:

Miniaturisation of Field Emission Technologies.

The research lead to the development by York Probe Sources Ltd of small-sized electron columns and a novel Field Emission source/cathode (operating at <1000V and delivering current intensity ~ 50-100μmA/str). A US-based start-up, Novelx Inc. successfully trialled and used the source to realise the first and still the only commercially available desktop, field emission Scanning Electron Microscope (2008), approximately the same size as a laser printer. Their desktop SEM product, *MySEM,* won the USA Prestigious R&D 100 Award for Innovative Scanning Electron Microscope (2009). *MySEM* is now in use in several top US academic and research laboratories including MIT, Berkeley and the Naval Research Labs for research into nanotechnology, MEMS and polymers as well as process development, verification and failure analysis in laboratories.

In 2010, Agilent Technologies acquired Novelx for an undisclosed sum [2]. The desktop SEM (8500 FE-SEM [3, 4) retails for **[text removed for publication]** (less than half the price of a conventional SEM), bringing it within reach of a far wider market. Furthermore, it occupies a footprint of less than 3 cubic feet; significantly smaller than conventional room-sized SEMs, again allowing it to be used in a wider range of applications and locations such as clean rooms and confined areas. Since the Agilent acquisition, it is estimated that more than **[text removed for publication]** have been sold world-wide [1]. Lawrence Murray, Director of R&D at FESEM for Agilent Technologies says "This product particularly stands out from others in this market because it provides customers the only desktop tool with enough resolution and surface fidelity to image features at nanoscale dimensions." and "Without the novel TFE work of Professor El-Gomati … this type of tool would not have been possible." [1]

High angular intensity Schottky electron source.

As a result of the collaboration with Shimadzu they have, to date, invested **[text removed for publication]** in the evaluation and refinement of the new cathode. The work has now been transferred to their Business Production Division, and they are making significant investment through a team of a dozen engineers with the aim of launching "the most powerful electron beam instrument to the world" [8].

LaB_6 in 'Virtual Source Mode'

Together with project partner, Carl Zeiss (UK), a conventional thermionic electron cathode, using a lanthanum hexaboride crystal (LaB_6) as the electron source, was configured to be used in the virtual source mode [5]. This configuration provides three benefits in comparison to operation in the thermionic mode 1) a smaller source size by a factor of more than 10 times, 2) a reduced initial energy spread of the emitted electrons by approximately 3-4 times and 3) increased source brightness of approximately 10 times on its value when operated in the conventional mode. These factors lead to a smaller beam size for a given beam current and allow one to use a lower beam voltage than previously necessary.
The advantages of these features are that they allow users of this instrument to investigate radiation sensitive and insulating materials at much higher resolution than previously possible. In addition, operating this class of cathodes in the virtual source mode, whilst similar to a field emitter, does not require altering the shape of the commercially available cathode to produce a sharp needle as is required for the more expensive field emitters. Further, the vacuum requirements of the LaB_6 electron cathode in this mode are only high vacuum which is much simpler and less expensive to achieve than UHV.

This virtual source mode of operation was used by Carl Zeiss SMT (Cambridge, UK) in developing a new and unprecedented generation of SEM – the 'EVO HD' ([6],[7],[8]) launched in 2011. This instrument, which is less expensive (approx. half the price) than an FE-SEM, has high imaging capabilities at lower beam energies (<2keV) – an essential feature, especially for radiation sensitive materials, such as biological and medical materials, novel photoresists, nanotubes and nanorods. Dr. S. Beam, Physics manager at Zeiss said [5]: "This instrument has the best performance of any conventional scanning electron microscope whilst maintaining a price that is both acceptable for a conventional microscope and lower than any high performance, field emission based, microscope. EVO® HD instruments have been sold within Europe, North America and the UK with other regions expected to follow in due course."

Low Voltage Microscopy

The semiconductor industry remains the single largest beneficiary of the advances and developments made in electron microscopy. SEMs occupy a key role in quality control assessment, where for example Critical Dimension SEMs (or CD-SEMs) are a major tool for critical dimension measurement, as well as for defect review analysis.

Research in this low voltage microscopy mode, has led to a better understanding of the signals obtained from the SEM. The understanding coming from this research underpins the growing use of low voltage SEM with its superior surface contrast, thus allowing nanoscale features to be observed on a wide variety of nanostructured materials, including polymers, thin films, biomaterials, and other energy-sensitive samples on any substrate, even glass. For example, using low voltage SEMs (<5keV), it is now possible to differentiate between differently doped semiconductors (i.e. p- and n-types; the fundamental building blocks in semiconductor devices).

Low and variable voltage operation of the SEM also negates the requirement for coating insulating materials that would charge up under a given electron beam energy, leading to both efficiency savings (as the materials do not need to be coated) and the removal of interference from the coatings in the images themselves.

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

[1] Letter from Director, R&D FESEM, Agilent Technologies.
This validates that the technology underpinning the Agilent 8500 FE-SEM is based on the underpinning research and associated claims.

[2] Agilent acquires Novelx Inc, (2010) with key interest in the MySEM and Desktop SEM
http://www.photonics.com/Article.aspx?AID=40849

[3] Agilent marketing the MySEM as a new product 8500 FE-SEM and selling it world-wide.
http://www.home.agilent.com/en/pd-1878801-pn-U9320A/8500-field-emission-scanning-electron-microscope-fe-sem?&cc=GB&lc=eng

[4] Marketing literature for SEM – includes the spec and the applications of the Agilent 8500 FE-SEM. http://cp.literature.agilent.com/litweb/pdf/5990-6183EN.pdf

[5] Letter from Physics Group Manager, Carl Zeiss - Confirms where sales of EVO HD instruments have been, and applications of the product.

[6] Carl Zeiss SMT marketing the new SEM model, EVO HD – the product spec and applications are available for download
http://microscopy.zeiss.com/microscopy/en_gb/products/electron-microscopy/evo-materials.html#inpagetabs-3

[7] PCT Patent jointly submitted with Zeiss.
Hayn Armin, El Gomati Mohamed: Electron gun used in a particle beam device. Carl Zeiss Smt Dec, 22 2010: EP 2264738-A1
https://register.epo.org/application?number=EP09163176
https://data.epo.org/publication-server/pdf-document?pn=2264738&ki=A1&cc=EP

[8] Letter from Analytical and Measurements Division, Shimadzu. (Confirms relationship with Shimadzu and their commitment to the research developments).