doi:10.1017/S1431927616006954
Microsc. Microanal. 22 (Suppl 3), 2016

Instrumentation/Technique Developments in Gareth Thomas's Research Group

Ondrej L. Krivanek[1]

[1]Nion Co., 11511 NE 118th St., Kirkland, WA, USA

Gareth Thomas was a tireless advocate for progress in transmission electron microscopy (TEM). He infused his whole research group at UC Berkeley with the "can do" spirit, and this led to many pioneering advances. This short paper summarizes three such developments.

Among his key accomplishments, Gareth initiated the National Center for Electron Microscopy (NCEM) at Lawrence Berkeley National Laboratory (LBNL). The center has its origins in the mid 1970s, when it became clear that high resolution electron microscopes (HREMs) were capable of giving better than 2 Å resolution, and that this would allow many structures to be solved by direct imaging rather than indirect methods. Gareth organized a workshop on the future of HREM, with international experts giving talks and working groups analyzing the most promising future avenues. The conclusions of the workshop were not altogether unexpected: high voltage HREMs were thought to have the best chance of resolving a wide range of unknown structures, and were likely to be expensive. Quoting from the workshop report [1]: *"The conclusion of the workshop was that there is indeed a need for a national facility for high resolution electron microscopy. The facility should be a center of excellence, located at a site with established reputation and dedication to electron microscopy and having existing scientific … resources."* NCEM was the logical result.

NCEM's flagship instrument, a 1 MV HREM called JEOL ARM (atomic resolution microscope), cost 3M in 1980 dollars (plus a new building) and eventually reached 1.3 Å resolution. Its usage, however, turned out not to be simple, and radiation damage and instabilities were important obstacles. Alternate paths were fortunately found: through-focus reconstruction [2], followed by the one that has now been universally adopted - aberration correction. And two further developments took place in Gareth's group, with a long-term impact that history may judge as even more significant than the original ARM.

One development was imaging semiconductor cross-section samples by HREM [3, 4]. This was done at a time when the structure of interfaces such as Si-SiO_2 in working MOSFETs (metal-oxide-semiconductor field-effect transistors) was simply unknown, and it introduced HREM as an important technique for analyzing semiconductor devices. An early example, obtained in the Thomas lab using a 125 kV Siemens 102 is shown in Fig. 1. Combined with focused ion beam (FIB) sample preparation, HREM later became an indispensable analytical technique used by all major semiconductor manufacturers.

Another significant development that started in Gareth's group was an electron energy loss spectrometer [5]. The impetus came from a meeting too: the 1978 Cornell workshop on Analytical Electron Microscopy, which convinced me that electron energy loss spectroscopy (EELS) would become a versatile and powerful EM technique. There were no commercial spectrometers that seemed attractive back then, and I decided to build one. I needed Gareth's approval and support, so I emphasized that EELS would allow us to analyze the oxygen content of grain boundary films in high temperature nitrogen ceramics, a subject of major interest to the group. Gareth asked just one question: "How much will it cost?," I answered "about $10k," and I had Gareth's blessing. A couple of years later, Peter

Swann, Joe Lebiedzik and I designed a mark II spectrometer that became the Gatan 607 serial EELS. This instrument led to Gatan's parallel-detection spectrometers and imaging filters, and helped EELS become the powerful and user-friendly technique that it is today.

Further down the road and less directly, the experience I gained from building spectrometers and imaging filters convinced me to try to build a STEM aberration corrector. This resulted in the first direct (no reconstruction) sub-Å EM images [6], and later on in the Nion UltraSTEM. More recently still, we have reached 8 meV energy resolution by monochromated EELS, and recorded vibrational spectra in an aloof beam mode that essentially avoids radiation damage [7] (Fig. 2).

I believe that imparting a "can-do" spirit is the very best thing a professor can do for his students and post-docs. Many of us are deeply grateful to Prof. Gareth Thomas for doing exactly that.

References:

[1] Workshop on High Resolution Electron Microscopy – Final Report. G. Thomas, R.M. Glaeser, J. M. Cowley, R. Sinclair, JOM (Journal of The Minerals, Metals & Materials Soc.) **29** (1977) 16.
[2] M.A. O'Keefe, Phil Mag. B **81** (2001) 1861-1878.
[3] O.L. Krivanek et al. *in:* The Physics of SiO_2 and its interfaces (Pergamon Press, 1978) 356-361.
[4] O.L. Krivanek and J.M. Mazur, Appl. Phys. Letts. **37** (1980) 392-394.
[5] O.L. Krivanek, Proceedings 38th EMSA meet. (1980) 86-89.
[6] P.E. Batson et al., Nature **418** (2002) 617-620; P.D. Nellist et al., Science **305** (2004) 1741.
[7] P. Rez et al., Nature Communications, in print (2016).

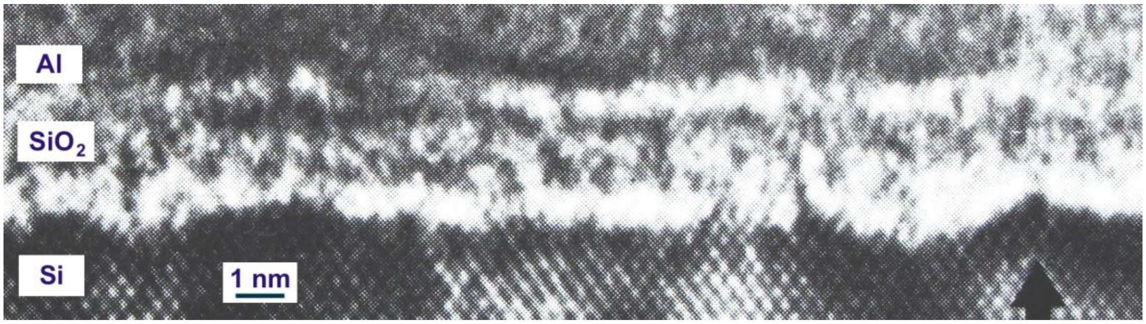

Figure 1. High resolution bright field image of ~3 nm thick SiO_2 on (100) Si. (ref. [4])

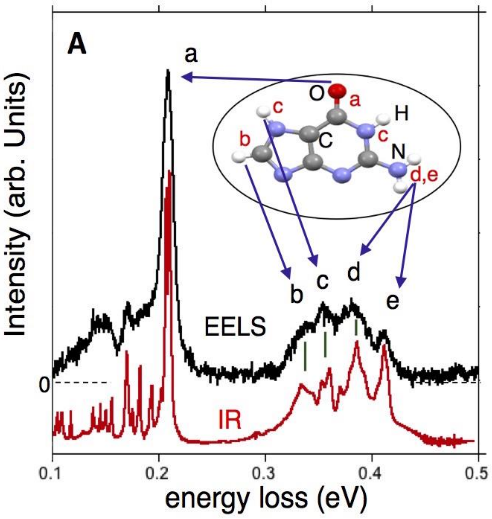

Figure 2. EELS of guanine acquired in aloof beam mode, with no visible radiation damage, compared to an IR spectrum acquired from ~10,000x larger sample area. Insert shows the guanine molecule, the blue arrows point to spectral features due to different bonds. (ref. [7])