Questions for Professor Ray Egerton

1. *Could you please comment on possible paths to minimize irradiation damage when conducting EELS analysis of organic samples?*

(a) Try cooling the specimen using a liquid-nitrogen holder. This reduces the radiolysis rate typically by a factor of 3-10 but has negligible effect on knock-on damage and could make electrostatic-charging problems worse.

(b) Consider specimen coating or encapsulation (more details in response to Question 9).

(c) Ensure that the collection efficiency of the EELS signal is as high as possible.

(d) For STEM and INORGANIC (but non-metallic) specimens, try collecting images quickly and adding them, or even interleaved scanning (Velazco *et al*. Ultramicroscopy, in press). Threshold and healing effects can be controlled to some degree by using a scanning probe.

2. *Are EELS and EDX incompatible in one instrument?*

No, they are essentially compatible since they use entirely different signals and detectors. Recent AEM hardware and software allows simultaneous EDX/EELS collection and data analysis in the same computer system, making direct comparison easier. EELS is often preferable for low-Z analysis, EDX more realistic for higher-Z elements and thicker specimens.

3. *What do you think will be the next breakthrough in analytical techniques in S/TEMs?*

The development of EDX detectors with few-eV energy resolution would allow quantitative analysis of low elemental concentrations and fine structure of absorption edges, giving band-structure information. This is currently possible using WDX spectroscopy but with low collection efficiency, or using bolometric detectors that require cooling with liquid helium and are expensive.

Spectroscopy of secondary electrons and Auger electrons could also provide band-structure information. Together with low-Z analysis with high spatial resolution, but will only be reliable for specimens with a clean surface, meaning near-UHV conditions and/or in-situ specimen-cleaning facilities.

4. *Can you comment on the possibilities to perform soft Xray emission spectroscopy?*

X-ray detectors with large solid angle have made EDX analysis more feasible for light-element analysis and beam-sensitive specimens, but accurate quantitative analysis remains difficult due to the large absorption of soft x-rays within a TEM specimen, which is hard to model precisely.

The observation of X-ray fine structure would also be useful if the energy resolution can be improved, as noted in the reply to Question 3.

5. *Would you please comment on how cryo-STEM could help to probe lithium species (as people have been pursuing in battery community)?*

If the lithium is present as a compound, the beam sensitivity may be high due to the possibility of radiolysis. In this case, cooling to liquid-nitrogen temperature should help.

6. *For imaging Li metal, I originally thought knock-on damage would be the primary factor and cryo-T may not help that much. Do you have more insights on this?*

Metallic Li should not incur radiolysis, and knock-on damage is relatively slow and insensitive to specimen temperature. But even a small amount of beam heating (if thermal contact is poor) could allow Li to diffuse out of the electron beam. Such diffusion would be reduced by cooling.

7. *Can you comment on vortex beam or phase structured beams to perform Dichroism or magnetic scattering is EELS. Will it be competitive with synchrotron-based techniques?*

I think the main advantage of EELS is that it can be done in a TEM and achieve high resolution, although not atomic resolution because the magnetic objective lens has to be turned off to avoid saturating many magnetic materials. The signal tends to be low, so effort has been devoted to finding a technique that is efficient and not highly sensitive to specimen thickness. Vortex beams may be promising in that regard.

8. *Veering towards 3D analyses, any comments on atomic probe tomography? Do you think that this is an exciting application?*

Atom probe tomography usually means the use of a sharp-tip specimen and high-voltage or laser pulsing that removes atoms sequentially and determines their 3D coordinates using a position-sensitive detector and time-of-flight techniques. It is certainly a powerful technique, capable of atomic-scale resolution, but requires very careful specimen preparation (often using a FIB machine) and is inherently destructive to the specimen. So any additional TEM measurements (diffraction etc.) must be done prior to use of the atom probe or even before preparing the sharp-tip specimen, if information about the surrounding material is needed.

9. *Please, for protection of beam damages, can you give examples what "encapsulation of the specimen / have the specimen in a cage" in practice means?*

Occasional studies over the years have indicated that coating a specimen with carbon, metal or even a thin insulating layer can reduce both mass loss and structural damage. Coating with graphene sounds attractive since it is an electrical conductor and a diffusion barrier to all elements except hydrogen. Encapulation of small molecules within carbon nanotubes or fullerenes has shown promise but the reliability and range of application of these techniques are issues that require further study.