EELS at high spatial resolution

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The combination of the analytical information available from electron energy loss spectroscopy (EELS) and the spatial resolution available from the transmission electron microscope provides one of the most powerful sources of information on atomic scale structures available today. Over the last few years, there have been major advances in aberration correction leading to electron probes that are smaller than interatomic spacings. In achieving this, there has been a major increase in column stability and a major reduction in the sources of "noise" that limit probe size.

In most cases, probe size is no longer the limiting factor in determining the spatial resolution in EELS. In reality, the limitations arise from other factors including the interaction between the fast electron and an atom, beam damage, the specimen (thickness and structure), signal-to-noise ratio, specimen drift and still, all too frequently, contamination.

The effect of these factors is interdependent in a way that depends on the particular specimen under study. For example, in an ultrathin specimen like a sheet of graphene, there is no effect of beam spreading through the sample but the dose required to get an adequate signal-to-noise from an energy loss with an interaction distance consistent with atomic resolution is high so that the actual limit is often knock-on damage. This can be eliminated by going to a lower accelerating voltage. This in turn requires an increased probe half angle, α, to maintain the same contribution to the probe size from diffraction. However, the effects of both geometric and chromatic aberrations increase and the probe size may once again become a limiting factor. The chromatic effects are more serious and can be tackled either by monochromation, at the expense of brightness, or by recent developments in chromatic aberration correction. The larger probe half angle will also require further developments of the post-specimen optics to ensure efficient collections of the EELS signal. Even if the knock-on damage in the main structure is eliminated in this way, single atoms may continue to move about in or on the specimen due to beam induced "diffusion".

However, true atomic resolution in EELS is not always necessary to provide key information. For example, from an atomic resolution image, it can often be difficult to tell whether "graphene" is truly a single graphene sheet or actually a multilayer. As shown in Figure 1, the shape of the low spectrum gives a definitive answer to this question [1].

The EELS signal is generated from all atoms irradiated by primary electrons and hence beam spreading and channeling start to play a key role as the specimen thickness increases. Even then, the ability of EELS to identify features that have little or no contrast when directly imaged offers great benefits even if atomic resolution is again not achieved e.g. identifying the locations of carbon nanotubes in a carbon matrix [2].

Analytical spatial resolution is typically defined so that a large fraction of the signal (typically 90%) comes from within it. Hence, the probe half angle needs to be chosen to balance the initial probe size against the beam spreading and this will depend on the sample thickness. To achieve the resolution offered by aberration correction, specimens a few nm

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thick are required and to derive any benefit from aberration correction, in terms of analytical rather than image resolution, specimens less than a few tens of nm are required.

Strong channelling conditions can "trap" primary electrons on atom columns, introducing a range of effects including atomic number dependent depth sensitivity and a contrast that is thickness and energy loss dependent as shown in Figure 2 [3]. Here the contrast reversal shows the maximum signal need not occur when the probe is on the column.

When real materials are involved, the situation is even more complicated. Here, there can be a wide range of atomic numbers, overlying phases, interfaces that are not atomically flat or sharp and considerable difficulty in making an ultrathin specimen without artifacts. On the other hand, it is possible to see relative displacements of the atomic distributions that are separated by less than the broadening from beam spreading or roughness. The key thing is achieving an adequate signal-to-noise ratio. Where damage is limiting, an average over equivalent or near equivalent positions becomes necessary.

In summary, there are many challenges in optimising the high spatial resolution analysis of real materials and these are posed by the instrumentation, the beam-specimen interaction, the specimen itself and the interpretation of the data.

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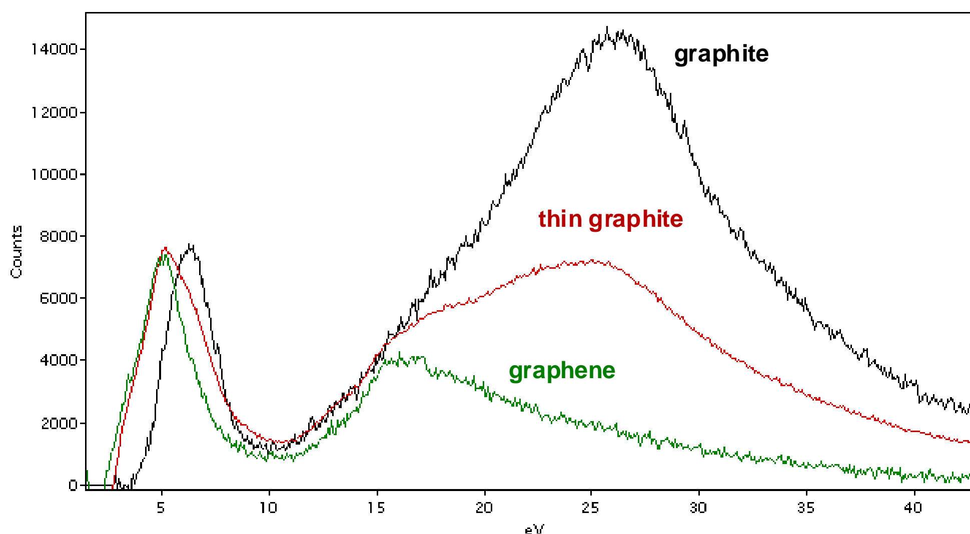

Figure 1. The differences between the low loss spectrum shapes in graphite and graphene [1].

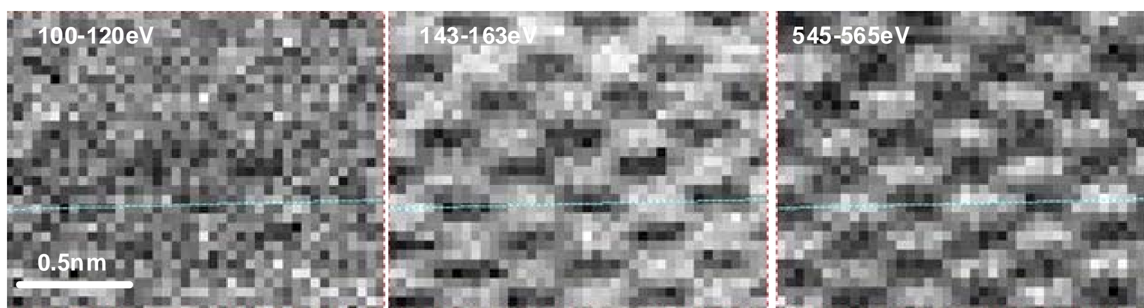

Figure 2. Energy filtered images from Si [110] in exact spatial registration, using different energy ranges of the background subtracted Si $L_{2,3}$-edge and showing contrast changes [3].

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