Using other methods in conjunction with electron diffraction

Electron diffraction is a powerful technique - but other techniques must be used with it to put the results in context. This is a brief synopsis of how other methods can be used to help.

X-ray Diffraction

The majority of novel / unknown crystalline materials are indexed by single crystal X-ray diffraction methods. Originally, photographs were taken along crystallographic axes and indexing performed manually. Nowadays, data are collected with a single point or two-dimensional electronic detector and the data are indexed with automatic computer programs.

When single crystals cannot be grown, data from a polycrystalline sample may be used to determine unit cells of crystal structures. Simple structures, such as cubic crystal structures, can be indexed manually by looking for integer relationships between interplanar spacings. For more complex structures, such as orthorhombic, monoclinic and triclinic, there are several different types of computer program. However, as there is a substantial loss of information in going from single crystal diffraction data to powder diffraction data, indexing powder diffraction data is demanding. Single crystal X-ray diffraction data are the first choice.

Information from both electron and X-ray diffraction are sometimes combined to tackle difficult crystal structures. Hints from other methods maybe also be useful. Indexing X-ray reflections from both single crystals and powders also gives information about the space group of the material under investigation from considerations of symmetry and systematic absences. Measurement of accurate unit cell lattice parameters can also be undertaken - this requires high quality, high angle X-ray diffraction data.

Optical imaging

This is a very important way of analysing a specimen. Using the naked eye and optical microscopes we can determine down to a point-to-point resolution limited by the wavelength of

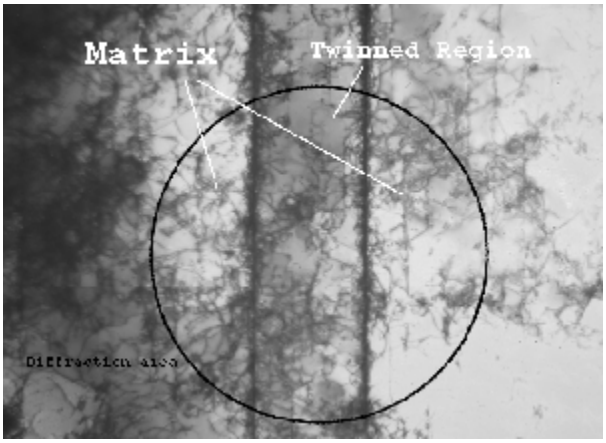
light how many phases there are and how they relate to one another. We can also infer what type of material they are likely to be and how they may have been processed.

Chemical analysis

A wide range of chemical techniques can be used to find out what components are present in the different phases and in what proportions. This will narrow the field of possible elements that we need to consider when analysing our diffraction results. These techniques range from simple chemical tests, through infrared spectroscopy of organic samples, to a wide variety of chemical characterisation techniques that can often be performed within the transmission electron microscope.

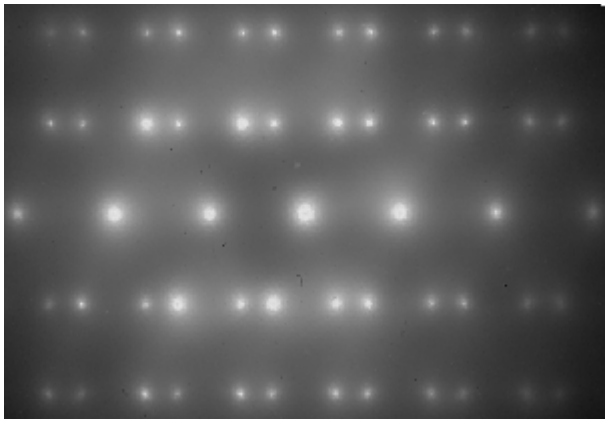
TEM imaging

Using the TEM to image the same area of sample that is being used to produce the diffraction pattern is an invaluable technique:



Nitrided surface layer of austenitic stainless steel

(Click on image to view larger version)



Diffraction pattern from nitrided surface layer of austenitic stainless steel

(Click on image to view larger version)

Using the image to verify that the double dots in the diffraction pattern are being caused by the two crystal structures either side of the twin boundary, we can index the pattern and determine the twin plane and the crystal structures either side of it.

Source: http://www.doitpoms.ac.uk/tlplib/diffraction-patterns/other.php