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TRANSMISSION ELECTRON MICROSCOPY: MINERALOGY AT THE NANOSCALE

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Martin graduated from the University of Leicester in 1986, and received a PhD in Geology from the University of Newcastle upon Tyne in 1990. Following postdoctoral positions at the universities of Essex and Edinburgh, he joined the University of Glasgow in 2000. Martin became Professor of Earth and Planetary Science in 2011, and is currently Head of the School of Geographical and Earth Sciences. The focus of Martin's work is on using mineral microstructures and chemical/isotopic compositions to research topics including the optics of trilobite eyes, silicate mineral weathering, geological histories of igneous and sedimentary rocks, and shock metamorphism. He is currently working on the history of magmatism and water on Mars through analysis of shergottite and nakhlite meteorites, and on the evolution of C-type asteroids via studying carbonaceous chondrites. His research work principally utilises scanning and transmission electron microscopy, electron backscatter diffraction, cathodoluminescence spectroscopy, ion microprobe analysis and argon isotope geochronology.

ABSTRACT

The transmission electron microscope (TEM) and scanning transmission electron microscope (STEM) enable microstructural, crystallographic, compositional and electronic information to be obtained from micrometre to sub-nanometre sized regions of suitably prepared rock and mineral samples. They are very powerful tools for describing and understanding important processes in Earth and planetary science such as crystal growth, exsolution and twinning, dissolution and replacement, radiation damage, and tectonic/shock deformation. However, as with most techniques TEM and STEM do not provide all of the answers – they are most helpful to the geoscientist when used in combination with other imaging and analytical tools such as scanning electron microscopy, electron probe microanalysis, electron backscatter diffraction and atom probe tomography.

1. INTRODUCTION

The focus of this chapter is on TEM and the closely related technique of STEM; the notation "(S)TEM" is used where the two techniques are discussed together. (S)TEM represents the pinnacle in sample characterisation – whilst other techniques enable chemical analysis (and isotopic analysis) at higher spatial resolution, and with superior accuracy and precision, (S)TEM is unrivalled in the range of mineralogical, microstructural and compositional information that can be obtained from one sample, on a single instrument and in a single session. However, the ability of (S)TEM to comprehensively characterise materials down to the nanoscale is a double-edged sword, and the geoscientist must take great care to relate results from (S)TEM work back to the bulk sample. This point is emphasized by Williams and Carter [1], who estimated that between instruments first becoming commercially available (in the 1950s) and their time of writing, only 0.6 mm^3 of material had been studied by TEM! Another constraint to bear in mind is cost. In a typical UK university the preparation of a single sample using the focussed ion beam (FIB) technique (~10 × 5 × 0.1 μm in size), would be ~£ 100, and the cost to study that one sample would be ~£ 150, depending on the information required (or put another way, ~£ 50/μm^3).

This chapter first outlines the ways in which samples can be rendered sufficiently thin for (S)TEM work, concentrating on the FIB technique. Descriptions of the various modes of imaging, electron diffraction and X-ray and electron spectroscopy are then described, with an emphasis on technologies that are of particular relevance to geoscientists. These include Z-contrast imaging by high angle annular dark-field (HAADF) STEM, atomic resolution imaging, electron crystallography by precession electron diffraction, and compositional analysis by X-ray and electron spectroscopy.

For a comprehensive theoretical and practical account of the interaction of electrons with thin specimens see Williams and Carter [1, 2]. Descriptions of TEM and related techniques with a geoscience emphasis can also be found in Buseck [3], Wirth [4], and Nieto and Livi [5]. This chapter is based on a review paper in *Mineralogical Magazine* [6].

2. PREPARING ELECTRON TRANSPARENT SAMPLES

A sample suitable for (S)TEM is sufficiently thin that when irradiated with a ~100 - 300 kV electron beam the incident electrons can pass through it and without being scattered too highly or losing a significant proportion of their initial energy (i.e., " electron transparent"). A ~100 nm thick sample is acceptable for TEM imaging, but analysis by electron energy loss spectroscopy (EELS) requires samples to be <~50 nm, and atomic resolution TEM imaging ideally ≤~10 nm thick. As lenses are not used in STEM, thickness requirements for imaging are less stringent. Prior to 2000, electron-transparent samples of geological samples were prepared by mechanical/chemical comminution, ultramicrotome (slicing with a glass/diamond knife), or broad-beam argon ion milling [7]. Today almost all geological samples are prepared by FIB (e.g., Heaney *et al.* [8]; Lee *et al.* [9, 10]). Samples are most commonly cut from polished thin sections with the advantage that regions of interest can first be characterised by techniques including light microscopy, scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD). FIB samples can also be extracted from the rough surfaces of mineral grains or rock fragments (e.g., [10]; Fig. 1A).

Most FIBs are equipped with an electron gun for imaging, and a source of high energy heavy ions, typically 30 kV Ga^+, for milling – although the new generation of instruments that use Xe^+ ions are superior to Ga^+ FIBs in many respects. The milling process is largely automated, and electron-transparent samples are typically ~10 × 5 × 0.1 µm in size. The initial stage is deposition of a protective layer, typically platinum, over the area of interest (using the electron beam and/or ion beam). A pair of closely-spaced trenches is then cut into the sample surface to leave a thin slice remaining between them (Fig. 1A). This slice is then cut free, welded to a micromanipulator needle, then moved next to a TEM holder to which it is welded (Fig. 1B) before being cut free from the micromanipulator needle. High energy Ga^+ ions amorphise the outermost parts of milled surfaces [10], but these amorphous envelopes may be removed by final polishing using lower energy (e.g., 5 kV) Ga^+ ions, or the sample can be polished *ex-situ* using a low energy (<1 kV) Ar^+ ion mill. The presence of amorphous envelopes on sample surfaces can be a problem if attempting to make them sufficiently thin for high-resolution TEM imaging or EELS work.

3. TEM AND STEM INSTRUMENTS

For (S)TEM work the TEM grid (Fig. 1B) is mounted in a holder that allows tilting about one or two axes. The holder is inserted into the electron column *via* an airlock then illuminated using a ~100 - 300 kV electron beam (Fig. 2). TEM imaging and electron diffraction uses a beam that is broad and for most purposes parallel. Lenses beneath the sample form the image or diffraction pattern that is viewed directly on a fluorescent screen or via a digital camera (Fig. 2). Many TEMs can also be operated in STEM mode, whereby the electron beam is converged to a very small probe that is rastered over the thin sample and images are formed using electron detectors. Geoscientists mainly use TEM, but STEM has important applications, particularly for high spatial resolution

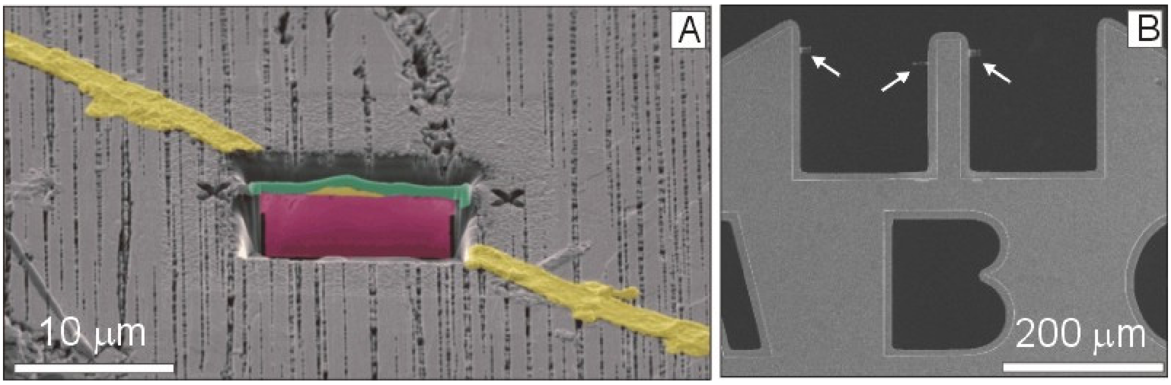

Figure 1. A) False coloured SEM image of the surface of a weathered grain (grey) on which is a fungal filament (yellow). A slice (pink) has been cut using the FIB and is about to be lifted out. Protective Pt is green. B) secondary electron image of three FIB slices (arrowed) welded to the tines of a copper TEM grid.

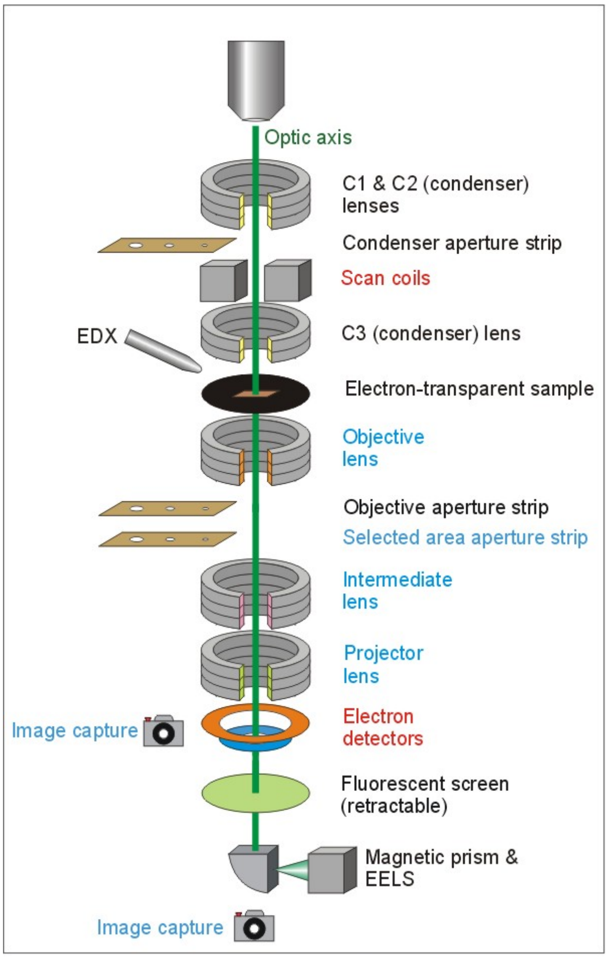

Figure 2. Configuration of a typical (S)TEM instrument.

atomic number (Z) contrast imaging, nanoscale electron diffraction, and analysis by energy-dispersive X-ray spectrometry (EDS) and electron energy-loss spectroscopy (EELS). For imaging, STEM has the advantage over conventional TEM of delivering a lower electron dose, which facilitates the study of beam-sensitive materials. STEM is also better than TEM for thick samples as image formation is less affected by chromatic or spherical aberration.

(S)TEM imaging and electron diffraction utilise the (predominantly) elastic forward scattering of incident electrons by atomic nuclei in the thin sample. The scattered electrons change direction (mainly by < ~3°) but lose very little energy; those electrons that pass close to the atomic nucleus can however be scattered at higher angles (up to 180°, i.e., backscattering) and lose some energy. Contrast in (S)TEM images is generated by two mechanisms, as described below: (i) amplitude contrast; (ii) phase contrast.

4. AMPLITUDE CONTRAST IMAGING

Amplitude contrast arises from variations in the intensity or angle of electron scattering throughout the volume of the thin sample. These images are made by isolating electrons that have been scattered over a certain angular range in one of two ways (termed 'bright-field' and 'dark-field'). Bright-field uses the 'direct beam', which contains unscattered and low angle forward scattered electrons. These electrons are isolated either using an objective aperture (in TEM) or an electron detector (in STEM). Dark-field images are formed solely from forward scattered electrons. In TEM mode the incident beam is tilted so that preselected scattered electrons are accepted by the objective aperture (for crystalline materials typically a single Bragg scattered beam), whereas STEM dark-field imaging uses annular detectors. These detectors intercept all those electrons that have been scattered over a certain angular range – for example, collection semi-angles of ~3 to 10° provide the commonly used HAADF images. Three properties of a thin sample will determine the intensity and angle of scattering of the incident beam and so its appearance in bright- and dark-field images: thickness (*t*) and atomic mass (Z) (together termed mass-thickness contrast), and Bragg diffraction (in crystalline materials).

4.1. Mass-thickness contrast

Mass-thickness contrast is an outcome of incoherent elastic scattering of incident electrons by the thin sample, the intensity of which increases with both Z and *t*. Thus, thicker and higher Z regions of a sample will be relatively dark in bright-field images because a greater proportion of incident electrons are scattered, thus lowering the intensity of the direct beam. Z contrast imaging utilises electrons that have undergone high angle (Rutherford) scattering. At scattering semi-angles of > ~3 - 5° Z contrast dominates over *t* contrast, and electrons that have been Bragg scattered by crystalline material are absent. Z contrast imaging of crystalline samples is most effective using HAADF. As the intensity of Rutherford scattering is proportional to $Z^{1.7-2}$, HAADF images are interpreted in the same way as those obtained from a SEM backscattered electron detector (Fig. 3).

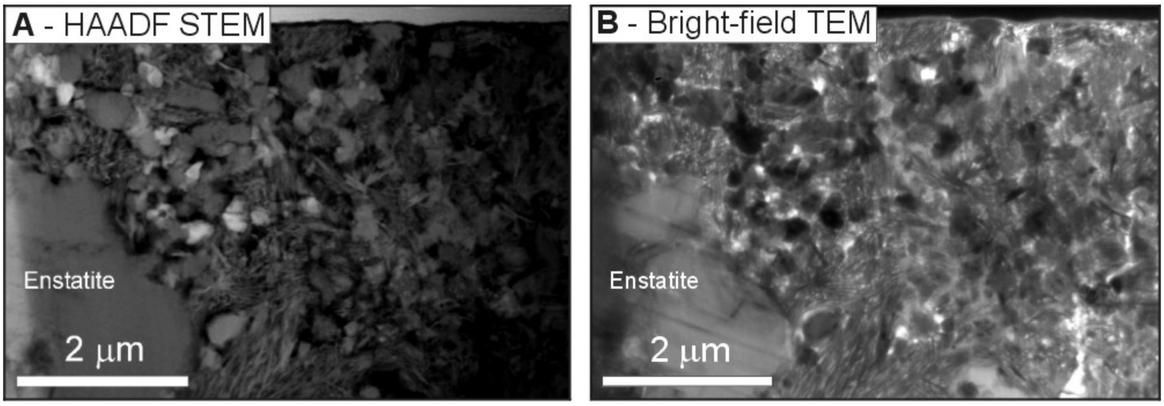

Figure 3. HAADF STEM and bright-field TEM images of a FIB-produced sample of a carbonaceous chondrite. The filed of view contains an enstatite crystal, next to which is an intergrowth os serpentine fibres (mid-grey in A), olivine (mid-grey rounded grains in A), and Fe-sulphide (white grains in A). pore space is black in the HAADF image and white in the TEM image. FIB-deposited platinum is along the top edge.

Atomic resolution HAADF images can be obtained by scanning a very fine probe over a crystal has been oriented so that atom columns are parallel to the microscope's optic axis. Owing to the Z dependence of high angle scattering, atoms columns with different atomic numbers can be recognised.

4.2. Diffraction contrast

Diffraction contrast is formed by coherent elastic scattering of incident electrons and is mainly utilized in TEM mode (Fig. 4). It enables: (i) discrimination of amorphous from crystalline regions; (ii) visualisation of intra- and inter-crystalline orientation differences (e.g., twins); (iii) identification of mineral inclusions (e.g., exsolution lamellae) by their contrasting diffraction properties to the host crystal. For incident electrons to be Bragg scattered (diffracted) by a crystal, one or more sets of its lattice planes must be oriented at the Bragg angle (typically < 1º) relative to the incident electron beam. As the Bragg angle is so small, diffraction-contrast imaging is highly sensitive to tiny (fractions of a degree) intracrystalline orientation differences. Such differences may arise due to elastic strain induced by exsolution lamellae (Fig. 4), vacancies and dislocations or the very-fine scale intergrowths of domains formed by processes such as spinoidal decomposition.

5. PHASE CONTRAST IMAGING

Phase contrast is present in all (S)TEM images and is formed by the interference of electron waves that have been scattered by the sample and are out of phase. By optimising phase over amplitude contrast, images can be obtained that contain information on the periodicity of one or more sets of

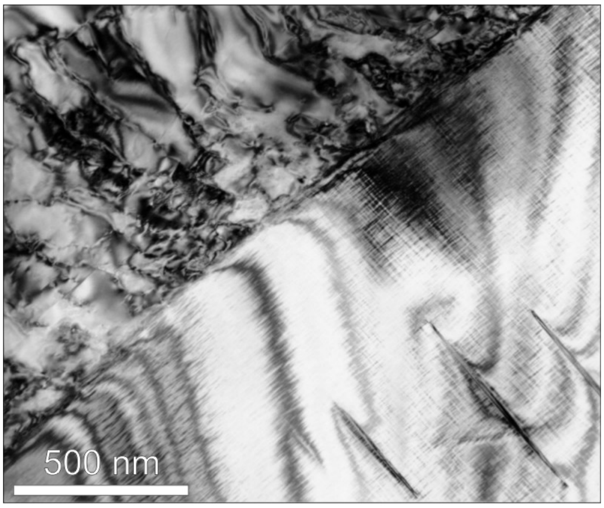

Figure 4. Bright-field TEM image of microcline (lower right) overgrown by adularia (upperleft). Contrast in the microcline is from cross-hatched twins, and exsolution lamellae. Contrast in the adularia comes from subgrains with dislocation-rich boundaries.

atomic planes (i.e., lattice images; Fig. 5), and that also can reveal the positions of atom columns. Lattice fringe images provide detailed information on local crystal structure and orientation, and are used extensively in geoscience, especially for identifying and studying minerals such as phyllosilicates (Fig. 5).

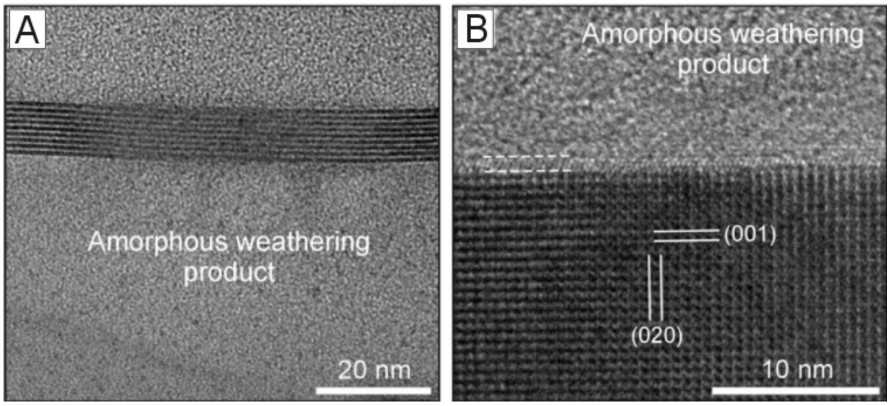

Figure 5. TEM lattice fringe images of the surface of a naturally weathered feldspar grain. A) A clay mineral, with a lattice fringe spacing of ~ 1 nm, within otherwise amorphous material. B) the outer surface of the feldspar grain. The (020) and (001) lattice spaces have spacings of 0.650 and 0.646 nm, respecyively.

In order to obtain high-resolution images that can be interpreted to reveal the locations of atom columns it is necessary to use a large objective aperture that accepts many diffracted beams. A series of images are obtained that contain a representation of the crystal structure, but in order to interpret features as atom columns it is necessary to use computer programmes to simulate the crystal structure expected when looking down the relevant zone axis and given the appropriate values for defocus and sample thickness. This technique is used much less frequently in geoscience than lattice fringe imaging due to a combination of reasons including the specialist skills and equipment required to acquire and correctly interpret the results, limitations on image acquisition times imposed by damage to the thin sample, and simply by the necessity of knowing the arrangement of atom columns.

6. ELECTRON DIFFRACTION

Diffraction patterns record the angular distribution of electrons that have been coherently elastically scattered and by < ~3°. Amorphous materials produce patterns with diffuse intensity variations whereas single crystals yield geometrical arrays of spots generated by Bragg scattering (Figs. 6 & 7). Spot patterns are effectively a two dimensional section through the reciprocal lattice of the crystal and the positions of spots are determined by the separation (d-spacing) of atomic planes and their orientation relative to each other. The TEM based technique of selected area electron diffraction (SAED) is used most widely in geoscience, but other methods including precession electron diffraction, convergent beam electron diffraction and electron nanodiffraction have specific applications.

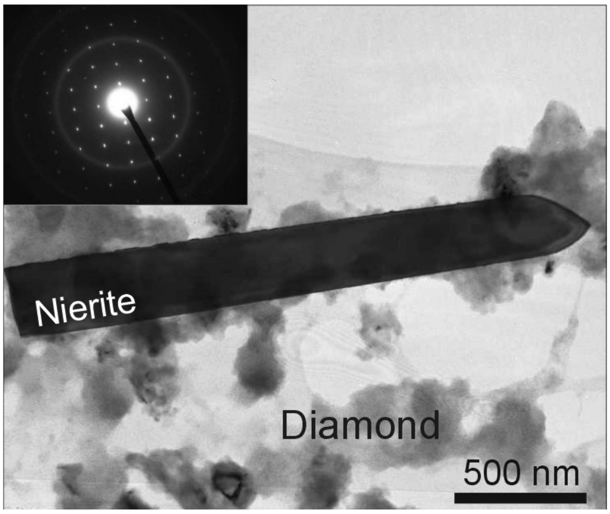

Figure 6. Bright-field TEM image of a single crystal of nierite (Si_3N_4) surrounded by clumps of ~ 4 nm size diamonds. The inset SAED pattern shows that the nierite forms a spot pattern whereas the diamonds generate a series of rings.

To obtain a SAED pattern the electron-transparent sample is illuminated using a broad and parallel electron beam. An area of interest is then isolated using the selected area aperture, whose diameter can be as small as a few hundred nanometres. If one crystal is present, and oriented with several

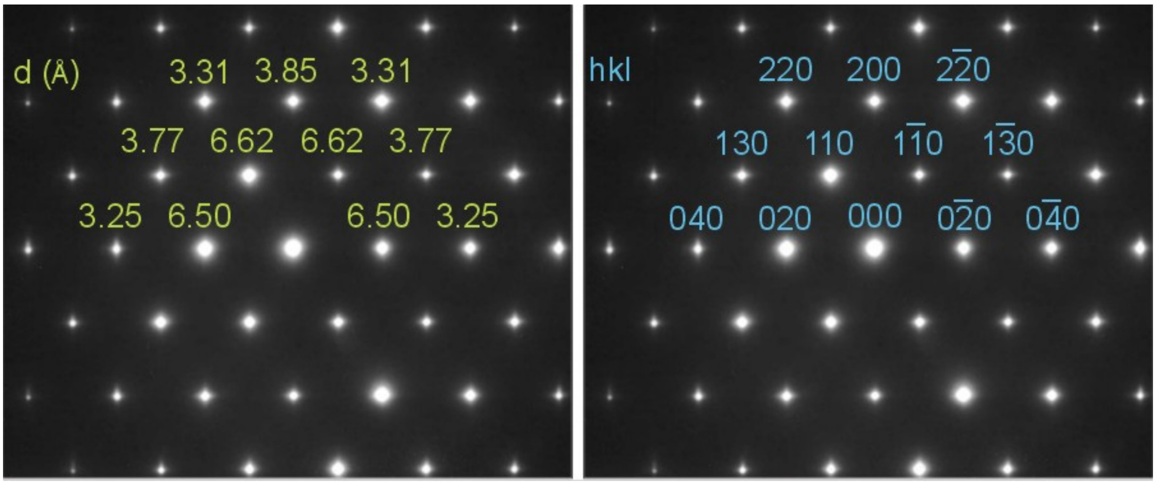

Figure 7. SAED patterns of orthoclase, with the electron beam parallel to [001]. The d-spacings of each spot are indicate”d in green on the left hand side, whereas their corresponding (hkl) lattice plane is indictaed in blue on the right hand side. Note also the asterism of each spot, which is produced by the very fine-scale ‘tweed’ microtexture that is characteristic of orthoclase. The undiffracted ‘direct beam’ is in the centre of the pattern.

sets of atomic planes (i.e., a zone axis) at the Bragg angle relative to the incident beam, a geometric array of spots is formed (Figs. 6 & 7). A SAED pattern from two or more crystals will yield superimposed patterns and a region containing many crystals in random orientations relative to each other will generate a large number of spots that merge into continuous rings (Fig. 6). In geoscience SAED is used most commonly to: (i) to identify minerals and (ii) determine the crystallographic orientations of features such as dislocations, twin composition planes, exsolution lamellae, intergranular boundaries and planar deformation features (PDFs) in shocked quartz.

Minerals are identified using the spacings of spots and angles between rows of spots (Fig. 7); pattern symmetry of can also provide a guide to the crystal system. SAED is inferior to X-ray diffraction (XRD) in the accuracy and precision of d-spacing measurements. To determine the crystallographic orientations of a feature, crystallographic directions/planes obtained from the SAED pattern are plotted on a corresponding image (after correction for any rotation between the two). However, in order to establish the absolute orientation ‘trace analysis’ using several diffraction pattern-image pairs is needed.

Precession electron diffraction (PED) enables a much more sophisticated interpretation of spot patterns derived from single crystals. PED is undertaken by precessing the electron beam around the optic axis of the microscope, and the resulting patterns contain spots whose intensities (in addition to d-spacings and angles) can be used to undertake sophisticated crystallographic analysis including crystal structure determinations and assessment of bonding. Convergent beam electron diffraction (CBED) patterns are formed using a focused electron probe (and a large condenser aperture) so that the size of the diffracting volume is essentially determined by the dimensions of the electron beam. Thus, CBED patterns can be obtained from ~10 nm sized regions. The patterns contain discs of intensity whose structure contains a wealth of crystallographic information unobtainable by conventional SAED.

Fourier transforms of high-resolution phase contrast TEM images can also be used to obtain information on orientations and spacings of atomic planes. This technique converts the periodicity of a high-resolution digital image into a diffractogram that can be indexed in the same way as a SAED pattern to: (i) help identify minerals, (ii) confirm orientation data derived from the source image, and (iii) to assess crystallinity. Diffractograms are useful because they greatly reduce the complexity of the high-resolution image and so can reveal features that are hard to identify visually, but in doing so information may be lost and so they are not a replacement for conventional SAED.

7. COMPOSITIONAL ANALYSIS BY (S)TEM

Incident electrons that interact with the electron clouds surrounding sample atoms are inelastically scattered and in the process change direction (by < ~1°) and lose up to ~5 % of their energy. Inelastic scattering provides most of the information in EELS spectra, and by exciting sample atoms also generates X-rays for EDS. Both EDS and EELS enable qualitative and quantitative determination of elemental compositions, but EELS can additionally yield information on bonding and valence states. Geoscientists with experience of EDS by SEM/EPMA will be able to interpret (S)TEM EDS data with little difficulty. EELS is a more specialist tool but very powerful for certain applications.

7.1. EDS analysis

X-rays produced during ionisation of specimen atoms can be collected and their energies analysed *via* an EDS detector located above the thin sample (Fig. 2). (S)TEM-EDS differs from microanalysis of bulk materials in two important respects: (i) 100 - 300 kV incident electrons will generate much higher energy X-rays than 20 - 30 kV electrons employed by SEM/EPMA, and (ii) the volume of a (S)TEM sample from which X-rays are generated is far smaller than from a thin section, which is a consequence of the < ~100 nm thickness of an electron-transparent sample, the small incident beam diameter and high electron energy.

Quantitative elemental analysis by (S)TEM-EDS is simpler than by SEM/EMPA because X-ray absorption and fluorescence can be essentially ignored (i.e., the ‘thin film criterion’; [11]). (S)TEM-EDS can achieve detection limits of ~0.1 wt% and yield concentrations within error of those acquired from the same materials using EPMA. Optimising instrumental conditions for quantitative (S)TEM-EDS requires a trade-off between competing variables. In the thinnest samples electron beam spreading will be at a minimum, giving the highest spatial resolutions for a given probe size, but X-ray count rates will be correspondingly low, possibly necessitating relatively long counting times so that specimen drift, electron beam damage and carbon contamination may cause significant problems. Beam damage can also be alleviated by analysing thicker regions of the sample (if available), which will have the additional advantage of giving greater count rates, although electron scattering will increase, thus limiting spatial resolution, and absorption and fluorescence corrections may have to be incorporated into quantification routines. Spot analyses can be obtained by TEM and STEM, whereas element mapping requires STEM.

7.2. EELS

EELS quantifies the energy lost by incident electrons during ionisation of sample atoms; losses range from ~5 eV to 2 keV. The EELS detector is positioned beneath the sample (Fig. 2). It collects unscattered and low angle (< 1º) scattered electrons and separates them using a magnetic prism according to energy loss. EELS can be done by TEM or STEM, and the spectra obtained provide qualitative and quantitative analysis of elemental abundances and determination of valence states and bonding of sample atoms.

An EELS spectrum contains three energy loss regions: (i) the zero loss peak (< 5 eV) formed from the direct beam and scattered electrons that have lost little energy, (ii) the low loss region (5 to 50 eV) representing ionisation of outer shell electrons of specimen atoms, and (iii) the high loss region (50 to ~2000 eV) produced by excitation of inner shell electrons. The high loss region comprises a small proportion (~5 %) of the net intensity of the spectrum but contains a series of sharp increases in energy loss called ‘edges’ (Fig. 8C). The energy loss of an edge is indicative of the element present (Fig. 8C), and information on bonding and valence states can be obtained from subtle changes in the energy loss and the fine structure of the 30 - 50 eV long tail to the edge. As the intensity of the core edge of a given element is a function of the number of atoms that are present in the analysed volume, EELS can be used to quantify elemental compositions. EELS has been used extensively for determining valence states of elements including manganese, iron (e.g., [12]), chromium and cerium.

Using STEM, EELS analyses can be acquired at spatial resolutions of <1 nm, although such fine-scale analyses can be obtained only from those materials that are sufficiently stable to withstand the high beam currents needed to generate a usable signal from such small volumes. By acquiring spectra from grids of closely spaced points, maps can be constructed to show spatial variations in elemental concentrations or valence states (Fig. 8B). It is important to note that a significant proportion of the volume of a sample sufficiently thin for good EELS work will be close to its surface so that if near-surface regions have been compositionally modified during ion milling of (S)TEM work, obtaining information on the elemental composition, valence and bonding that is a true reflection of the original mineral may be challenging.

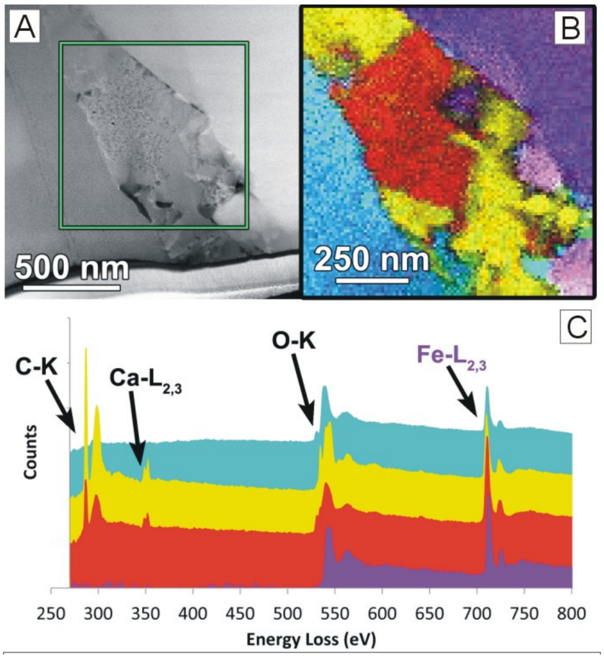

Figure 8. STEM and EELS results from the Nakhla Martian meteorite. A) HAADF STEM image of a vein of 'iddingsite' within olivine. B) False-coloured EELS map of (A) revealing four constituents: ferrihydrite (light blue), olivine (purple), siderite (yellow), and partially oxidised siderite (orange). C) EELS spectra used to make the map, with the same colour coding.

7.3. Energy-filtered TEM (EFTEM) imaging

EFTEM uses electrons from a specific region of the EELS spectrum to form an image, for example the zero loss peak or one or more core loss edges. Those images formed using the zero-loss peak are free of inelastically scattered electrons and so EFTEM is useful for studying thick samples. Using the core loss edges enables chemical imaging, and this method has the major advantage over point-by-point X-ray or EELS mapping that images can be acquired rapidly (seconds to a few minutes) and with sub-nanometre spatial resolutions. For example, Moore *et al.* [13] demonstrated how EFTEM could be used to differentiate augite from pigeonite *via* contrasts in intensity of images acquired using Mg, Ca and Fe core loss edges; a difference between the two minerals of 2 atom% Mg was readily resolvable. The downside of EFTEM is that electron beam damage to the very thin samples can be severe, especially as high beam currents are needed to obtain sufficient signal intensities from the narrow regions of the EELS spectra that contain core loss edges.

8. LIMITATIONS TO (S)TEM WORK BY ELECTRON BEAM DAMAGE

Even before (S)TEM work is undertaken, minerals may be modified by exposure to a vacuum during ion milling. For example, smectite typically dehydrates with a consequent change its (001) layer spacings from ~1.5 - 1.2 nm to ~1.0 nm (Fig. 9). Electron beam irradiation during (S)TEM can alter thin samples by three mechanisms: (i) radiolysis; (ii) heating; (iii) knock-on damage (Fig. 9). Beam damage is typically severe in hydrous minerals and those that are alkali-rich, e.g., feldspars. *Radiolysis* is the ionisation of sample atoms during inelastic scattering and leads to breaking of bonds, resulting in defect formation and amorphisation, sputtering of atoms and changes in chemistry. *Heating* also takes place as energy is transferred from incident electrons to sample atoms during inelastic scattering, and common effects are breaking of bonds, vapourisation and chemical reactions including recrystallisation. Silicate minerals are very prone to radiolysis and heating, but it may be minimised by cooling the sample using liquid nitrogen or increasing accelerating voltage in addition to using a broader beam (in TEM mode), or rastered beam (in STEM mode). *Knock-on* damage is due to momentum transfer from incident electrons to atomic nuclei during high-angle elastic scattering. It can lead to displacement of internal atoms, especially those of low Z, and sputtering of the more weakly bonded atoms from sample surfaces. Another electron beam-induced artifact is hydrocarbon contamination. Hydrocarbon molecules may be deposited during ion milling, or derived from vapour present in the microscope column, and are polymerized on the sample surface by the electron beam. Contamination can be mitigated by plasma cleaning the sample and holder prior to (S)TEM work and heating/cooling the sample.

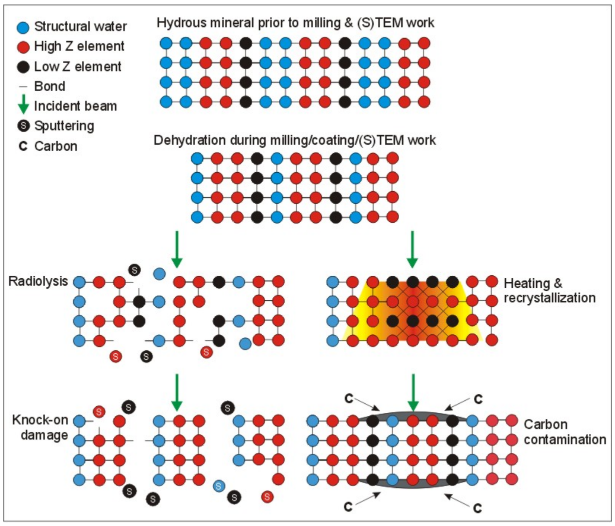

Figure 9. The main mechanisms of electron beam damage during (S)TEM work, using a hydrous mineral as an example.

9. SUMMARY AND PROSPECTS

(S)TEM is a very powerful technique for the mineralogical, microstructural and compositional characterization of a material, and FIB has made this technology readily accessible to the geoscientist. Nonetheless, questions that could once only be answered by (S)TEM can increasingly be tackled using by other techniques such as EBSD, and the related technique of transmission

Kikuchi diffraction, atom probe tomography and various synchrotron-based methods. Areas of geoscience where (S)TEM remains the technique of choice include:

(i) Characterisation of the density, crystallographic orientation and microstructural context of dislocations and other defects (vacancies, stacking faults, fine-scale twins);
(ii) Determination of the mineralogy of phyllosilcates, especially where different minerals are intergrown on a very fine-scale;
(iii) Identification of nanoscale inclusions, and exsolution lamellae;
(iv) Valence state determinations at the nanoscale;
(v) Characterization of amorphous and poorly crystalline materials (including organics), and amorphous materials within crystalline hosts (e.g., planar deformation features within shocked quartz, amorphous rims on irradiated mineral grains).

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ABBREVIATIONS

CBED = Convergent beam electron diffraction.
EBSD = Electron backscatter diffraction.
EDX = Energy dispersive X-ray.
EELS = Electron energy loss spectroscopy.
EFTEM = Energy filtered transmission electron microscopy.
EPMA = Electron probe microanalysis.
FIB = Focused ion beam.
HAADF = High angle annular dark-field.
PED = Precession electron diffraction.
SAED = Selected area electron diffraction.
SEM = Scanning electron microscopy.
STEM = Scanning transmission electron microscopy/microscope.
(S)TEM = STEM and TEM referred to together.
t = Thickness.
TEM = Transmission electron microscopy/microscope.
XRD = X-ray diffraction.
Z = Atomic number.