33

X-ray Spectra and Images

CHAPTER PREVIEW

The X-ray spectrum generated within your specimen consists of element-specific characteristic peaks with well-defined energies superimposed on a non-characteristic background. While the XEDS system is a remarkable piece of technology, we've already described its limited resolution and we will see in this chapter that it is also prone to creating small artifact peaks in the spectrum. Furthermore, the unavoidable presence of scattered electrons and X-rays within the AEM conspire to degrade the quality of the generated spectrum and increase the number of false peaks in the displayed spectrum. The AEM illumination system and specimen stage are rich sources of powerful radiation, not all of it by any means coming from the area of interest in your specimen. So you have to take precautions to ensure that the X-ray spectrum you collect comes predominantly from the area of the specimen that you want to analyze and we describe several tests you should perform to ensure that the XEDS-TEM interface is optimized. Once you understand the desirable and undesirable contents of a spectrum, we'll show you the various ways to gather your spectra, display them, and also form X-ray images from them. In particular, digital spectrum imaging is the most powerful technique for optimizing the information that you can gather from the low number of counts generated in thin foils.

33.1 THE IDEAL SPECTRUM

Back in Chapter 4 we learned that electrons generate two kinds of X-rays. When electrons ionize an atom which returns to ground state, the emitted characteristic X-ray *energy* is unique to the ionized atom (Figure 4.2). When electrons are slowed by electrostatic interaction with the nucleus they produce a continuum of bremsstrahlung X-rays and together the characteristic peaks and the bremsstrahlung background comprise the X-ray spectra we detect and display via the XEDS (as shown schematically back in Figure 4.6 and experimentally in Figure 32.2 and many others).

33.1.A The Characteristic Peaks

Some more detailed revision: a beam electron ionizes an atom in your specimen by ejecting an inner or core-shell electron, leaving a hole in the shell. The probability of this event occurring is governed by the ionization cross section. Then a cascade of electron transitions occurs, with each transition filling the hole with an electron from a more weakly bound shell (leaving a hole in that shell, and so on) and ultimately the last electron falls into a core-shell from the conduction band. Depending on the fluorescence yield, each transition results in either a characteristic X-ray or an Auger electron. The characteristic X-rays have a well-defined energy and a natural 'line width' (the FWHM of the Gaussian distribution of X-ray energies) of typically 1–5 eV. But, as you already know from the previous chapter, the XEDS degrades this width to a Gaussian-shaped peak with a FWHM of about 135 ± 10 eV. We'll use the term 'line' to denote the actual X-ray energy at the peak of the Gaussian and we'll talk about K, L, and M families of lines (and indeed the XEDS computer display includes lines superimposed on the spectra at the K, L, M, etc., peak energies). The actual number of counts (intensities) of the characteristic peaks from a given element, and the relative differences between spectra from different elements, are really quite complex and we'll go into this more in the next chapter when we discuss details of spectra and qualitative analysis. For the time being, all you need to know is that, generally speaking, lower-energy X-ray peaks are more intense than higher-energy ones and the heavier the element, the more complex the characteristic spectrum. Also X-rays with energies $< \sim 1$ keV are absorbed both within your specimen and within the detector and ultimately, the combination of absorption and low fluorescence yield means it is not possible to detect X-rays with energies $< \sim 110$ eV (the Be K line).

33.1.B The Continuum Bremsstrahlung Background

The background in the X-ray spectrum is bremsstrahlung (braking) radiation arising as beam electrons are slowed down or stopped by electrostatic interactions with nuclei in the specimen. The intensity of the bremsstrahlung is zero at the beam energy (since we can't create more energy than the beam has) and rises until it is effectively infinite at zero energy. This distribution is well understood and described mathematically by variations on Kramers' law as we also noted back in Figure 4.6. Modifications to Kramers' law account well for the absorption of the X-rays and accurately describe the experimentally detected bremsstrahlung distribution, even from thin specimens. So the net result is that X-ray spectra consist, as we have seen, for example, in Figures 1.4A and 32.2, of characteristic, Gaussian-shaped peaks superimposed on a background of bremsstrahlung X-rays, most clearly visible in Figure 32.2F. Many more spectra will appear throughout this and subsequent chapters and, as already noted, we can create elemental images from specific characteristic peaks.

Unfortunately, what makes X-ray spectrometry challenging is that the spectrum generated within your thin specimen, the spectrum detected by your Si(Li) detector, and the spectrum displayed on your computer screen are all quite different. So understanding what controls the X-ray counts that you measure in the characteristic peaks in your spectra is absolutely essential. Otherwise it will be difficult to first identify (Chapter 34) and then quantify (Chapter 35) the peak intensities in terms of the presence and the amount, respectively, of the elements in the analysis volume of your specimen.

We'll first describe how the XEDS system generates artifact (escape, sum, and internal fluorescence) peaks and then we'll show how the AEM itself also contributes unwanted X-rays, all of which can confuse the unwary operator into thinking certain elements are in their specimen when, in fact, they are not. Misinterpretation of the presence or absence of certain elemental signals could cause real problems if, for example, you are making decisions on the suitability or otherwise of a specific material for a given application, or even more seriously, making a forensic or medical decision based on the chemistry of your specimen: more about this in Chapter 34.

33.2 ARTIFACTS COMMON TO Si(Li) XEDS SYSTEMS

The XEDS system introduces its own artifacts into the spectrum. Fortunately, we understand all these artifacts, but they still occasionally mislead the unwary operator; see the review by Newbury. We can separate the artifacts into two groups

- Signal-detection artifacts: examples are escape peaks and internal fluorescence peaks.
- Signal-processing artifacts: e.g., the sum peaks.

Escape peak: Because the detector is not a perfect sink for all the X-ray energy, it is possible that a small fraction of the energy is lost and not transformed into electron-hole pairs. The easiest way for this to happen is if the incoming photon of energy E fluoresces a Si K_α X-ray (energy 1.74 keV) which escapes from the intrinsic region of the detector. The detector then registers an apparent X-ray energy of (E – 1.74) keV, as shown in Figure 33.1.

ESCAPE PEAK

Si escape peaks appear in the spectrum 1.74 keV below the true characteristic peak position.

The magnitude of the escape peak depends on the design of the detector and the energy of the fluorescing X-ray. The most efficient X-ray to fluoresce Si K_α X-rays is the P K_α, but in a well-designed detector even the P escape peak will only amount to <2% of the P K_α intensity. This fact explains why you can only see escape peaks if there are major characteristic peaks in the spectrum. More escape peaks occur in IG spectra because we can fluoresce both Ge K_α (9.89 keV) and L_α (1.19 keV)

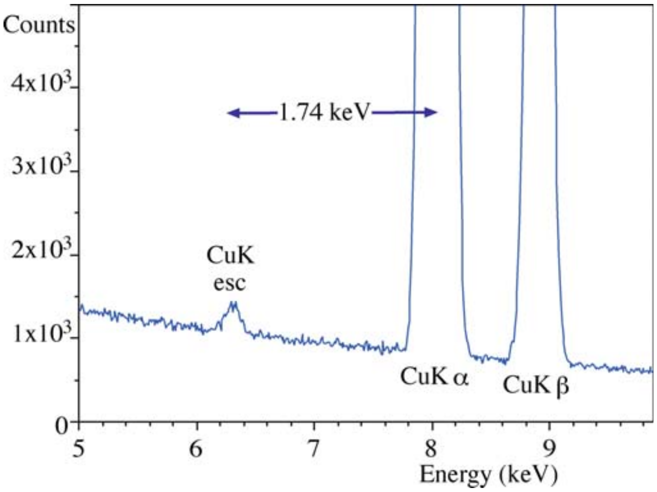

FIGURE 33.1. The escape peak in a spectrum from pure Cu, 1.74 keV below the Cu K_α peak. The intense K_α peak is truncated in the display because it is 50–100× more intense than the escape peak.

characteristic X-rays in the detector. Each of these will cause corresponding escape peaks. SDDs are thinner than typical Si(Li) crystals so there may be a slightly enhanced possibility for an escape peak. The analysis software should be able to recognize any escape peak, remove it, and add the intensity back into the characteristic peak where it belongs. Because the escape-peak intensity is so small it is rarely a problem unless you misinterpret it as coming from an element which is not present.

The internal fluorescence peak: This is a characteristic peak from the Si (or Ge) in the detector dead layer. Incoming photons can fluoresce atoms in the dead layer and the resulting Si K_α or Ge K/L X-rays enter the intrinsic region of the detector which cannot distinguish their source and, therefore, register a small peak in the spectrum. As semiconductor detector design has improved and dead layers have decreased in thickness, the internal fluorescence peak artifact has shrunk but it has not yet disappeared entirely. So beware.

Obviously if you are looking for small amounts of Si in your specimen you'll always find it in a Si(Li) spectrum after long enough counting time! Depending on the dead-layer thickness, the Si K peak intensity corresponds to $\sim$0.1–1% of the specimen composition (see Figure 33.2) so it is hardly a major problem if you are aware of it. Similar effects are observed in IG detectors also, but the very thin dead layer in SDDs should be an advantage.

LONG COUNTING TIMES

A small Si K_α peak will occur in ALL spectra from Si(Li) detectors after long counting times.

Sum peak: As we described earlier, the processing electronics are designed to switch off the detector while each pulse is analyzed and assigned to the correct energy channel. The sum peak arises when the count rate exceeds the electronics' ability to discriminate all the individual pulses and so-called 'pulse pile-up' occurs. This is likely to occur when

- The input count rate is high.
- The dead times are $>\sim$ 60%.
- There are major characteristic peaks in the spectrum.

The electronics simply cannot be perfect. Occasionally, two photons will enter the detector at exactly the same time. The analyzer then registers an energy corresponding to the sum of the two photons. Since this coincidence event is most likely for the X-ray giving the major peak, a sum peak first appears at twice the energy of the major peak, as shown in Figure 33.3.

If you are using an SDD because you have a C_s-corrected AEM and are seeking to maximize count rates, you can generate multiple sum peaks, causing serious interpretation problems in your analyses. There is evidence (see Newbury's paper) that digital processing is, in fact, worse than analog processing in terms of generating sum peaks in SDD systems, and hybrid analog/digital systems are being considered in the SEM field where this is a bigger problem.

THE SUM PEAK

The sum peak should be invisible if you maintain a reasonable input count rate, typically <10,000 cps, which should give a dead time of <60%.

Since you can't usually generate very high count rates, unless your specimen is really thick, there is little need to worry about sum peaks in AEM spectra but, as always,

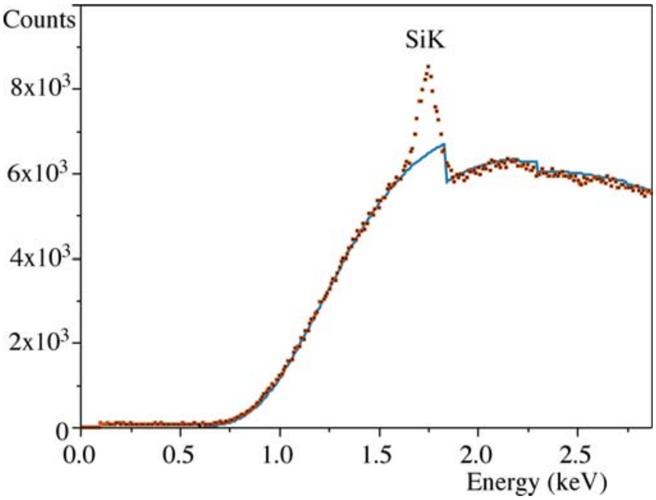

FIGURE 33.2. The Si internal fluorescence peak in a spectrum from pure carbon obtained with a Be-window Si(Li) detector. The ideal spectrum is fitted as a continuous line that only shows the Si absorption edge.

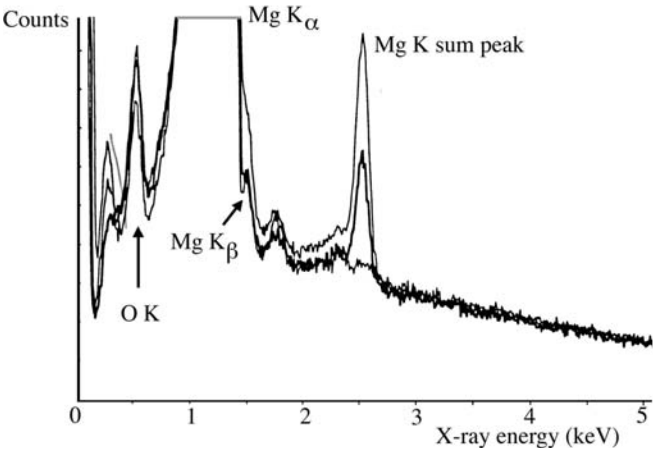

FIGURE 33.3. The Mg K sum (coincidence) peak occurs at twice the Mg K_α peak in this spectrum from a bulk specimen of (oxidized) pure Mg. The sum peak decreases change rapidly with decreasing dead times; upper trace 70%, middle trace 47%, lower trace 14% dead time. The sum-peak artifact is close to the background intensity at 14% dead time.

you should at least be aware of the danger. (For example, the Ar K_α energy is almost exactly twice the Al K_α energy and the sum peak has led some researchers to report Ar being present in Al specimens when it wasn't, and has caused others to ignore Ar, which actually was present in ion-milled specimens!) One exception to this lack of concern is if you have intense low-energy peaks where the residual noise in the electronic circuitry interferes with the pile-up rejection. So if you're analyzing elements lighter than Mg, take care to use low input count rates. Reducing the dead time to 10–20% should remove even the Mg K_α sum peak, as shown in Figure 33.3.

Much of what we have just discussed and most of what we'll cover in Chapters 34 and 35, can be observed experimentally on the AEM. But it is often just as instructive, and certainly easier, to simulate the spectra. To this end, we strongly advise you to download the public-domain simulation software 'Desktop Spectrum Analyzer' (DTSA) from NIST (Fiori and Swyt and URL #1); it is listed in the recommended software in Section 1.6 and its use is described in depth in the companion text. In fact, almost all the spectra in this and the subsequent chapter were generated by DTSA (in part, to ensure a uniform appearance). This software permits realistic simulation of XEDS data in TEMs (and SEMs) and introduces you to all the aspects of spectral processing, artifacts, modeling, etc., that are discussed in this and the next three chapters.

33.3 THE REAL SPECTRUM

In the perfect AEM, all spectra would be characteristic only of the chosen analysis region of your specimen with which the beam interacts. The analysis of bulk specimens in the SEM/EPMA approaches this ideal but, in the AEM, two factors combine to introduce false information which we call system and spurious X-rays. These X-rays introduce small errors into both qualitative and quantitative analysis, unless you are aware of the dangers and take appropriate precautions to identify and minimize the problems. The two factors that make the AEM different to the EPMA and that are responsible for these problems are

- The high-voltage electrons which generate intense doses of stray X-rays and scattered electrons in the illumination system.
- The thin specimen which scatters high-energy electrons and X-rays around the limited confines of the AEM stage.

Modern AEMs are designed to minimize some of these problems, but they have not disappeared entirely. It is important for you to understand that these problems are *not* major distortions of the X-ray spectrum, and only introduce small peaks or changes in intensity equivalent to composition changes at the 1% level or less. Nevertheless, identification and quantification of such small elemental amounts are often the *raison d'être* for an analysis (e.g., impurity segregation), so you have to be wary of these artifacts which we'll now discuss in some detail. Remember, these artifacts are in addition to the XEDS artifacts, described in the previous section.

33.3.A Pre-specimen Effects

The TEM illumination system produces high-energy bremsstrahlung X-rays and electrons scattered outside the main beam, both of which may strike the specimen, producing spurious X-rays.

In inhomogeneous specimens (which are usually just the kind that we want to analyze) the presence of significant amounts of spurious X-rays means that the quantification process could give the wrong answer. There are reviews (e.g., Williams and Goldstein or Allard and Blake), which describe in detail how to identify and minimize these artifacts from the illumination system, so we will just describe the precautions necessary to insure that the AEM is operating acceptably. Since these artifacts are primarily a result of the high-energy electrons interacting with column components, such as diaphragms and polepieces, you must take extra care when using intermediate-voltage instruments.

SPURIOUS X-RAYS

We define **spurious X-rays** as those that come from the specimen but are **not** generated by the electron probe in the chosen analysis region.

The standard way to detect stray radiation from the illumination system is to position the focused electron beam to pass through a hole in your specimen and see if you can detect an X-ray spectrum characteristic of the specimen.

If the major peak in the hole-count spectrum contains more than a few percent of the characteristic intensity in the same major peak obtained from a thin area of your specimen, under similar conditions, then the illumination system needs attention.

You can easily determine whether stray electrons or X-rays are the problem, as illustrated in Figure 33.4, which shows spectra obtained on a silver disk specimen (A) and down the hole (B). Ag has a high-energy K_α line at ~23 keV and a low-energy L_α line at ~ 3 keV. With the beam through the hole in the disk, stray X-rays will fluoresce the high-energy peak more efficiently and stray electrons will preferentially excite the low-energy peak. So changes in the K/L ratio from a heavy metal test specimen such as Ag are determinative experiments. The NiO specimen that we described in Chapter 32 is available on Mo support grids and the Mo K/L ratio is equally diagnostic.

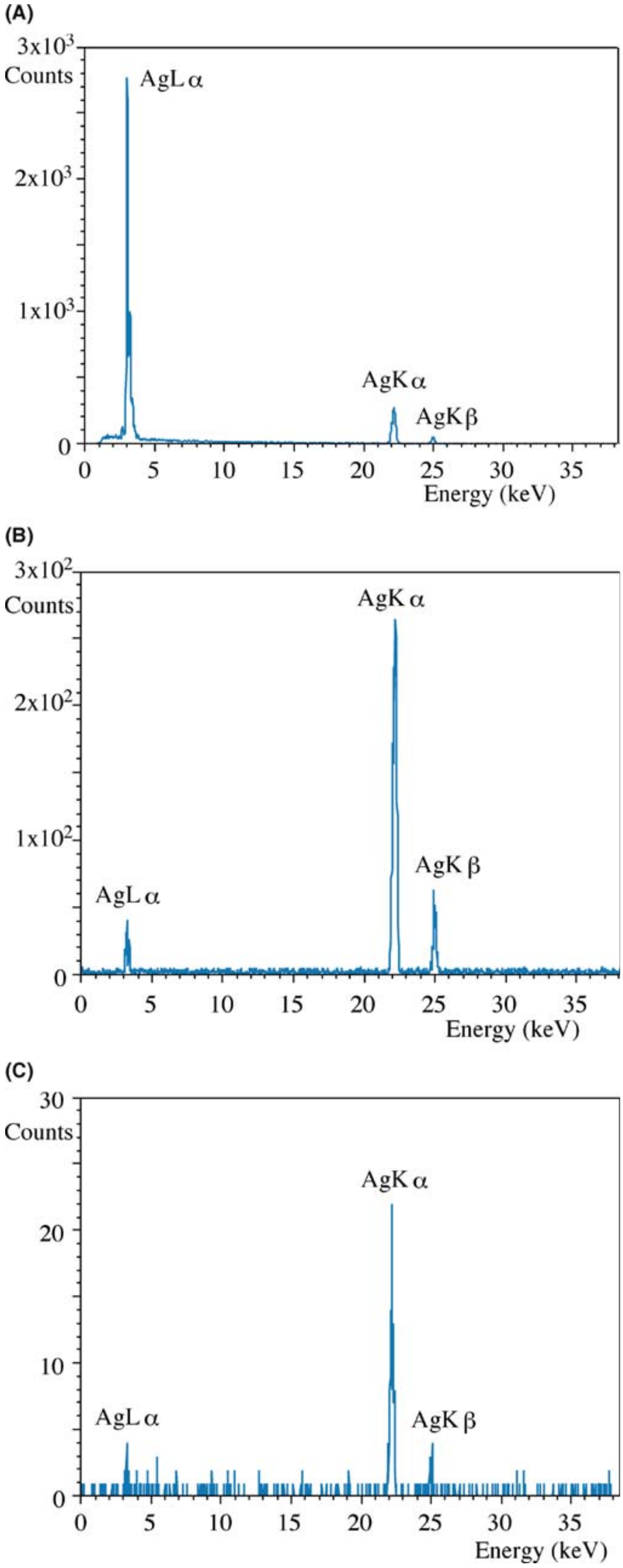

FIGURE 33.4. The 'hole count.' (A) A Ag self-supporting disk produces an electron-characteristic (high L/K ratio) spectrum when struck by the primary beam. (B) Without a thick C2 diaphragm, a reasonably intense Ag spectrum is also detected when the beam is placed down a hole in the specimen. This spectrum has a high K/L ratio which indicates high-energy

FIGURE 33.4 (Continued) bremsstrahlung fluorescence. Note that the (spurious) Ag K_α hole-count intensity (B) is ~ 50% of the (real) Ag K_α intensity (A) recorded from the specimen! (C) Use of a thick Pt C2 diaphragm reduces the intensity of the Ag K_α hole count substantially. The K_α intensity in (C) is about 30 times less than in (B). (Note the scale change.)

THE HOLE COUNT

We cannot reduce spurious radiation sources to zero, so a spectrum, sometimes termed a 'hole count,' is **invariably** obtained in all AEMs if you count for long enough.

Let's consider first the problem of high-energy bremsstrahlung X-rays generated in and penetrating through a standard Pt C2 diaphragm (go back and look at Figure 6.10 to remind yourself why diaphragms stop beam electrons). These X-rays flood your specimen and fluoresce characteristic X-rays from a large area around the analysis region. These stray X-rays would give a high Ag (or Mo) K/L ratio in the spectrum from your test specimen (see Figure 33.4B).

These diaphragms should be a standard fixture in your AEM (check with your lab manager) but they are expensive and they cannot be flame-cleaned in the usual way. When the thick diaphragms do contaminate, you have to discard them, otherwise the contamination itself will become a source of X-rays and will also deviate electrons out of the main beam by charging.

Pt TOP HAT

The solution to this problem is to use very thick (several mm) platinum diaphragms which have a top-hat shape, and a slightly tapered bore to maintain good electron collimation (see Figure 33.4C).

Alternatively, some AEMs incorporate a small diaphragm just above the upper objective lens to shadow the thicker outer regions of the specimen from stray X-rays.

Another way you can minimize the effects of this undesirable bremsstrahlung is to use an evaporated film or window-polished flake on a Be grid rather than a self-supporting disk, but you don't always have this choice. If your specimen is thinner than the path length for fluorescence (a few tens of nm in many cases), spurious X-rays will not be generated. Of course, it may not be possible to prepare such thin specimens, or it may take a great deal of effort, while self-supporting disks are relatively easy and quick to produce; so this isn't a popular suggestion with graduate students. However, with increasing interest in the properties of nano-scale thin films you may be fortunate enough to be studying such ideal specimens.

For a quantitative, reproducible measure of the hole count, you should use a uniform thin specimen, such as the NiO film we have described. This film should be supported on a bulk material that has a low-energy ($<\sim 3$ keV) L line and a high-energy ($>\sim 15$ keV) K line. A thick Mo or Au washer or support grid is ideal. Any high-energy bremsstrahlung X-rays penetrating through the C2 diaphragm will strongly fluoresce the Mo K or Au L line while stray electrons will excite the Mo L or Au M lines preferentially.

With thick diaphragms and thin foils, the remaining stray X-rays will not influence the accuracy of quantification or introduce detectable peaks from elements not in the analysis region. For more details on this test, see Lyman and Ackland. If you don't want to go to the trouble of this test, then the least you should do is measure the in-hole spectrum on your specimen and subtract it from your experimental spectrum if the hole spectrum contains peaks with >1% of the counts in the characteristic peaks that come from your specimen.

RULE OF THUMB

The ratio of Mo K_α or Au L_α intensity detected (when the beam is down the hole) to the Ni K_α intensity obtained with the beam on the specimen should be less than 1%.

Now that we've minimized stray X-rays, let's consider the possibility that all the electrons are not confined to the beam. Stray electrons will generate a hole spectrum with a low K/L ratio (the opposite of Figure 33.4B). If your microscope has a non-beam defining spray diaphragm below the C2 diaphragm it will eliminate such stray electrons without generating unwanted X-rays. Then the main source of poorly collimated electrons is usually the 'tail' of electrons around the non-Gaussian-shaped probe that arises from spherical aberration in the C2 lens, as shown in Figure 33.5 (Cliff and Kenway). The best way to minimize this effect is to image the beam on the TEM screen under the conditions that you will use for analysis, and select the best C2 aperture size to define the probe, as we discussed way back in Section 5.5. It is a simple test to move your probe closer and closer to the edge of your specimen and see when you start generating X-rays. Do this with different size, top-hat C2 diaphragms. If you are fortunate enough to have a C_s corrector in your probe-forming lens then the probe will have much smaller tails, but this is a very expensive solution, not available to all.

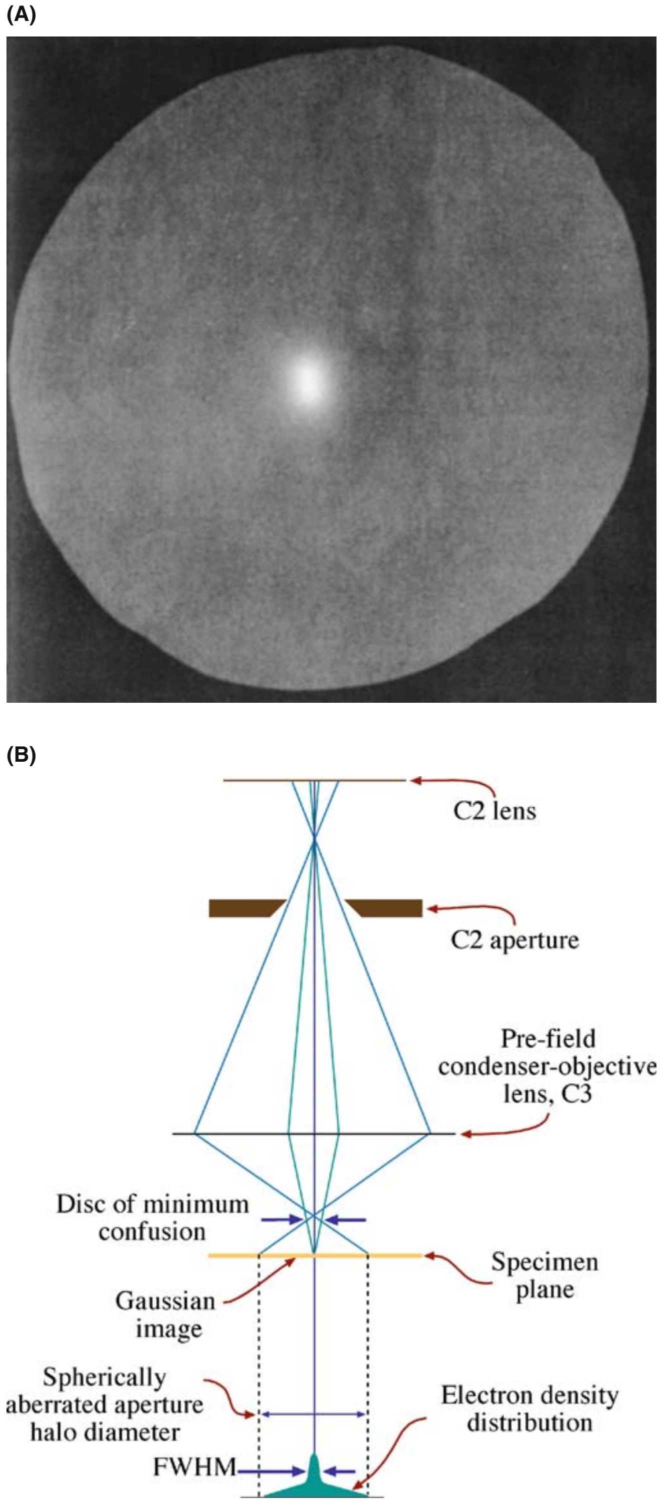

FIGURE 33.5. (A) The shadow of the C2 diaphragm defines the extent of the halo of electrons which excites X-rays well away from the chosen analysis region. (B) Ray diagram showing how the STEM probe formed with a large C2 aperture generates a broad halo around the intense Gaussian-shaped probe. Such beam tailing is the major source of uncollimated electrons and arises due to spherical aberration in the probe-forming lens. Choosing a smaller aperture will limit the tailing but cut down the probe current.

In summary

- Stray X-rays will give a high Ag (or Mo) K/L ratio.
- Stray electrons will give a low Ag (or Mo) K/L ratio.
- Always operate with clean, thick, top-hat C2 diaphragms.
- Use very thin flake specimens or uniform thin films, if possible.
- Always image the electron beam on the TEM screen prior to analysis, to insure that it is well collimated by the C2 aperture.
- Use a C_s-corrected AEM if you can find one.

Now the only significant X-ray source will be the region where you position the probe.

33.3.B Post-specimen Scatter

The electrons interact with the specimen and are scattered elastically or inelastically. It is fortunate for us that the intensity of such scatter from a thin specimen is greatest in the forward direction. So most of the scattered electrons are gathered by the field of the lower objective polepiece and proceed into the imaging system of the AEM away from the XEDS detector. Unfortunately, some electrons are scattered through high enough angles that they strike other parts of your specimen, the support grid, the holder, or the objective lens polepiece or other material in the AEM stage.

SYSTEM X-RAYS

X-rays that come from parts of the AEM other than the specimen.

It is instructive to insert the objective diaphragm during an analysis (just once!) to see the enormous increase in spurious and system X-rays that result. (Actually, you'll do this experiment by mistake anyway.) Usually the X-ray flux is so great that the pulse-processing electronics are saturated, the dead time reaches 100%, and the automatic shutter will activate. However, even when you remove the diaphragm, electrons scattered by the specimen may still create X-rays characteristic of the materials in the holder (brass), the polepiece (Fe and Cu), and the collimator (e.g., Al, W) and any of these X-rays could be picked up by the XEDS detector.

Cu IS EVERYWHERE

Remember the post-specimen scatter will still generate specimen-characteristic X-rays remote from the area of interest, even if a Cu peak is not present.

Furthermore, despite the strong field of the upper objective polepieces in probe-forming STEMs (another good reason to always operate in STEM mode) some back-scattered electrons may travel directly into the XEDS detector, generating electron-hole pairs. Other scattered electrons may hit your specimen at some point well away from the area of interest where they will still produce specimen-characteristic (and therefore) spurious, X-rays. All these possibilities are undesirable but unavoidable because, without the beam-specimen interactions that produce this scattering, we would get no information at all from the specimen. Figure 33.6 summarizes all the possible sources of spurious and system X-rays from post-specimen scatter.

In addition to electron scatter, there will be a flux of bremsstrahlung X-rays produced in the specimen. The intensity of these X-rays is also greatest in the forward direction (see gray shaded area in Figure 33.6). Since they possess a full spectrum of energy, the bremsstrahlung will fluoresce some characteristic X-rays from any material that they strike. The easiest way to discern the magnitude of this problem is to use a uniformly thin foil (such as our standard NiO film) on a Cu grid. When you position the probe on the film in the middle of a grid square, many micrometers from any grid bar, the collected spectrum will invariably show a Cu peak arising from the grid, as a

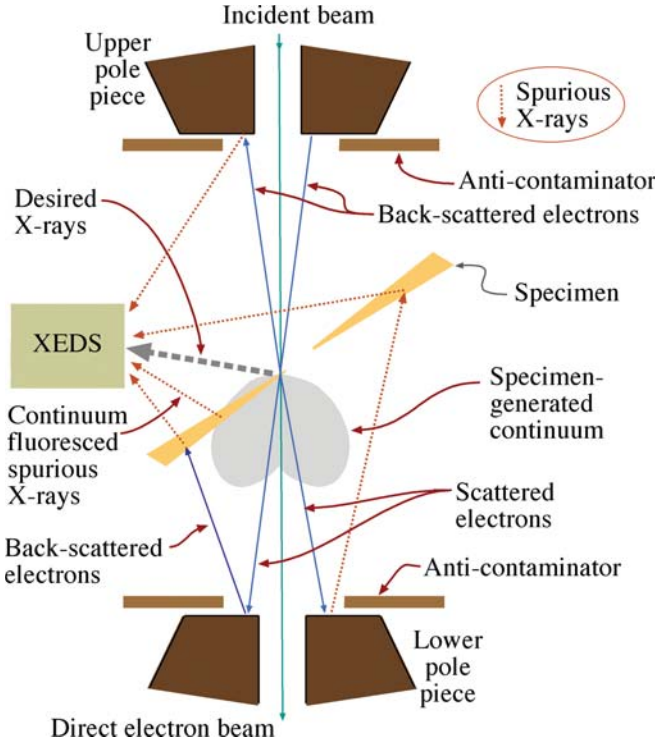

FIGURE 33.6. Sources of system and spurious X-rays generated when the incident beam (green) is scattered by the specimen. BSEs and forward-scattered electrons (blue) excite system X-rays in the stage and spurious X-rays (red dotted lines) elsewhere in the specimen. Bremsstrahlung (gray-shaded region) fluoresces the specimen away from the analysis region (also red dotted lines). The gray dotted line represents the desired X-rays from the analysis region.

result of interactions with electrons or X-rays scattered by the film. An example of this effect in a Cr film is shown in Figure 33.7. The presence of the Cu peak can be removed by using a Be grid, since Be K_α X-rays are much more difficult to detect. However, using Be grids merely removes the observable effect in the spectrum, not the cause.

To minimize the effects of the scattered radiation, you should keep your specimen close to zero tilt (i.e., normal to the beam). If you tilt $< \sim 10^\circ$ then the background intensity is not measurably increased. Under these conditions, your specimen will undergo minimum interaction with both the forward-directed X-rays and any backscattered electrons. Both of these phenomena have only a small horizontal component of intensity. The effects of your specimen interacting with self-generated X-rays will be further reduced by using specimens, such as evaporated films or window-polished flakes rather than self-supporting disks, just as we suggested in the previous section. In self-supporting disks, the bulk regions will interact more strongly with the bremsstrahlung. It is not known what fraction of the post-specimen scatter consists of electrons and what fraction is X-rays, because this will vary with both specimen and microscope conditions. However, there is no evidence to suggest that this X-ray fluorescence limits the accuracy of quantitative analyses (which is at best ±3–5%, as we'll see in Chapter 35).

In addition to keeping your specimen close to zero tilt, you can further reduce the effects of post-specimen scatter by surrounding the specimen with low-Z material. Use of these materials will also remove from the spectrum any characteristic peaks due to the microscope constituents. Be is the best material for this purpose and, as we said right at the beginning of this part of the book, Be specimen holders and Be support grids are essential for X-ray analysis. Ideally, all solid surfaces in the microscope stage region that could be struck by scattered radiation should also be shielded with Be. Unfortunately, such modifications are rarely available commercially.

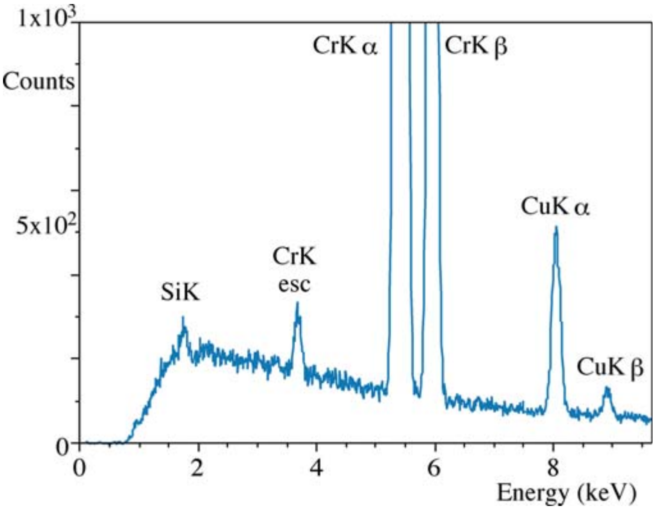

FIGURE 33.7. Cu peaks in a spectrum from a thin Cr film on a Cu grid. Although the beam was many micrometers from the grid, Cu X-rays are excited by electron scatter from within the specimen, and their intensity generally increases with specimen tilt. The Cr escape peak and the Si internal-fluorescence peaks are also visible.

Be

Be oxide is highly toxic if inhaled, so if you have to handle Be grids or other Be components, use gloves and tweezers and don't breathe!

The narrow polepiece gap, required to produce high probe currents, and the cold finger, used to reduce hydrocarbon contamination, both tend to increase the problems associated with post-specimen scatter. In the ideal AEM, the vacuum would be such that a cold finger would not be necessary and the polepiece gap would be chosen to optimize both the detected peak to background ratio and the probe current. When an AEM stage was substantially modified with low-Z material (e.g., by Lyman et al.) a large reduction in bremsstrahlung intensity was reported and X-ray peak to background ratios were produced that are still unmatched by most commercial AEMs. We'll discuss this more in Section 33.5.

You must note, however, that whatever precautions you take, the scattered electrons and X-rays, which are invariably present, result in a specific limitation to X-ray analysis. (See the below box on this.)

SMALL AND LARGE

If you are seeking small amounts (<2%) of element A in a specific region of your specimen, and that same element A is present in large amounts, either elsewhere in your specimen or in the microscope stage, then you ***cannot*** unequivocally identify the presence of that element A in the specific region of your specimen! If you count for long enough, a small peak from A will *invariably* be present in all spectra, just as surely as the Si internal-fluorescence peak from your detector will be present.

Obviously then, you must determine the contributions to the X-ray spectrum from your microscope, and this is best achieved by inserting a low-Z specimen in the beam that generates mainly a bremsstrahlung spectrum, such as an amorphous-carbon film, supported on a Be grid or a pure B foil. If a spectrum is accumulated for a substantial period of time (say 10–20 minutes, or even over lunch), then in addition to the C or B peak (if your XEDS can detect them) the various instrumental

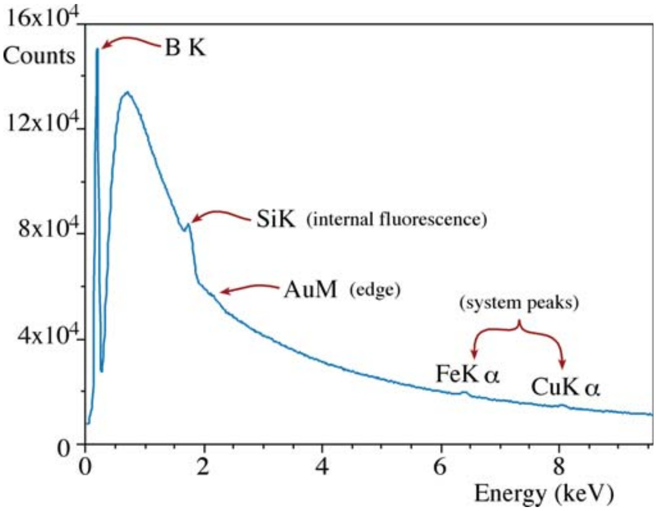

FIGURE 33.8. An XEDS spectrum from high purity boron, showing system peaks. The Si K_α peak and the Au M absorption edges are detector artifacts but the small peaks at 4.6 and 7.5 keV are system peaks from elements in the microscope stage.

contributions to the spectrum should become visible. Such an 'instrument spectrum' (see Figure 33.8) should only exhibit the internal-fluorescence peak and possibly the Au absorption edge from the detector. Any other peaks will be from the TEM itself, assuming the specimen is pure. These peaks will tell you which elements it is *not* possible to seek in small quantities in your specimen because of their presence in your AEM.

We can summarize the methods used to minimize the effects of post-specimen scattering quite simply

- Always remove the objective diaphragm.
- Operate as close to zero tilt as possible.
- Use a Be specimen holder and Be grids.
- Use thin foils, flakes, or films rather than self-supporting disks.

Remember, that even with these precautions, you will still have to look out for artifacts in the spectrum, particularly those from the XEDS system.

33.3.C Coherent Bremsstrahlung

As we noted earlier, the bremsstrahlung spectrum is sometimes referred to as the continuum because the intensity is assumed to be a smooth, slowly varying function of energy. This assumption is perfectly reasonable when the bremsstrahlung is generated in bulk polycrystalline materials by electrons with energies <~30 keV, such as in a SEM. However, in thin single-crystal specimens illuminated by high-energy electrons, it is possible to generate a bremsstrahlung X-ray spectrum that contains small, Gaussian-shaped peaks known as 'coherent bremsstrahlung' (CB). The phenomenon of CB is well known from high-energy physics experiments, but no one thought it would occur at AEM voltages until it was clearly demonstrated by Reese et al. Figure 33.9A shows a portion of an X-ray spectrum

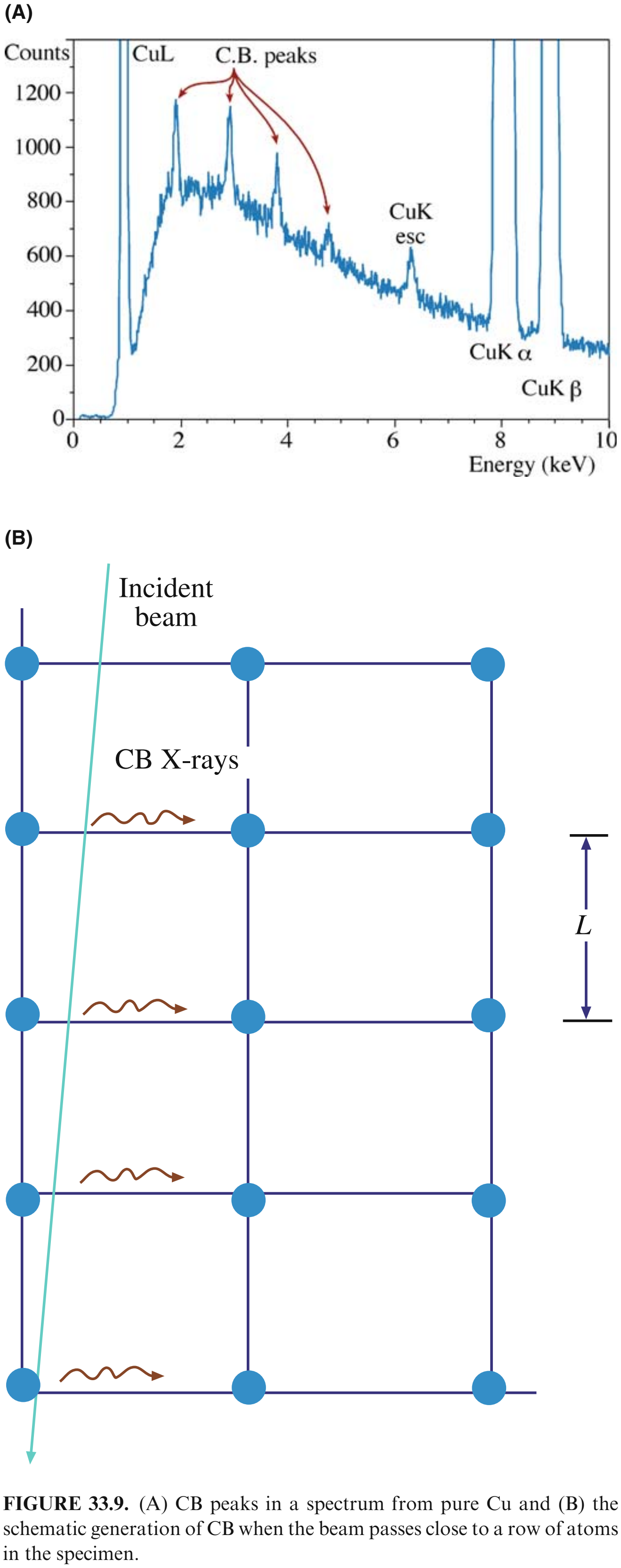

FIGURE 33.9. (A) CB peaks in a spectrum from pure Cu and (B) the schematic generation of CB when the beam passes close to a row of atoms in the specimen.

from a thin foil of pure copper taken at 120 keV. The primary peaks, as expected, are the Cu $K_{\alpha/\beta}$ and the L family of lines. In addition, the escape peak is identified. The other small peaks are the CB peaks. They arise, as shown in Figure 33.9B, by the nature of the coulomb interaction of the beam electrons with the regularly spaced nuclei in the crystal specimen. As the beam electron proceeds through the lattice, close to a row of atoms, each bremsstrahlung-producing event is similar in nature and so the resultant radiation tends to have the same energy. The regular interactions result in X-ray photons of energy E_{CB} given by

$$E_{CB} = \frac{12.4\beta}{L(1 - \beta\cos(90 + \alpha))} \qquad (33.2)$$

where β is the electron velocity (v) divided by the velocity of light (c), L is the real lattice spacing in the beam direction, equal to $1/H$ in a zone-axis orientation (go back and check Section 21.3.B), and α is the detector take-off angle. More than one CB peak arises because different Laue zones give different values of L. The CB peak intensity seems greatest when the beam is close to a low-index zone axis, and these conditions should be avoided if possible. Operating with a convergent beam reduces the intensity of the CB peaks and this will be helped with a C_s-corrected AEM since larger convergence angles can be used to give more probe current without degrading the probe size.

CB PEAKS

You may mistakenly identify these CB peaks as characteristic peaks from a small amount of some element in the specimen, but fortunately, you can easily distinguish CB peaks from characteristic peaks.

Unfortunately, you can't remove the CB effects entirely, even by operating far from a major zone axis, since some residual peaks are invariably detectable if you count for long enough.

As predicted by equation 33.2, the CB peaks will move depending on both the accelerating voltage (which will alter v and hence β) and the specimen orientation, which will change the value of L. Of course, characteristic peaks show no such behavior and are dependent only on the elements present in your specimen. While CB peaks are a problem, you should only be concerned if you are seeking to detect a small amount of specific element in your specimen. More about this problem in the next chapter.

33.4 MEASURING THE QUALITY OF THE XEDS-AEM INTERFACE

In the end, what we need is some measure of how well our XEDS system is working and to be able to compare it with values from other systems. There are two ways to do this, both of which use a thin film, such as our standard NiO (although in these examples, we'll use Cr since that was how the original experiments were done. The principles are the same).

33.4.A Peak-to-Background Ratio

The first test of how well your XEDS is interfaced to your TEM is to measure the peak to background (P/B) ratio in the film.

FIORI P/B

There are several definitions of P/B ratio, but the best one, termed the Fiori definition, is shown in Figure 33.10A.

For the Ni K_α peak, you should integrate the peak intensity from 7.1 to 7.8 keV and divide this by the average background intensity in a 10 eV window (i.e., one or two channels depending on the display resolution) under the peak. In the Cr thin-film example shown in Figure 33.10A, the Cr K_α peak is summed from 5.0 to 5.7 keV. In a well-constructed AEM, the P/B ratio will increase with keV. The P/B values shown in Figure 33.10B (measured again on a Cr film rather than a NiO film (Zemyan and Williams)) should be achievable in any modern AEM. This value is an important test of the XEDS-AEM interface and the design of the stage. Higher is better!

33.4.B Efficiency of the XEDS System

The relative detector efficiency is a measure of how many X-ray counts per second (cps) are collected, detected and processed by the XEDS system. This is very important because of the overwhelming need to gather the most X-ray counts possible, given all that we've described about the inherent inefficiencies of X-ray generation and detection in XEDS of thin films.

In a fixed live time the detector efficiency will be affected by the specimen thickness, the probe current, and the solid angle of collection of the detector. So you should use the standard NiO specimen again, to fix the thickness variable and measure the probe current with a Faraday cup, as we've mentioned several times before (this gives cps/nA). Last, you need to factor in the collection angle given by the XEDS manufacturer (which is in fact calculated, not measured) to give the best figure of merit in terms of cps/nA/sr as described by Zemyan and Williams. Typical values are shown (again for Cr) in Figure 33.10C and, as you can see, the efficiency decreases with increasing kV because of the decrease in ionization cross section. But at any given kV, a higher number indicates higher efficiency.

It is possible to determine both the P/B ratio and the efficiency from direct spectral measurement and software to do this is described in the companion text.

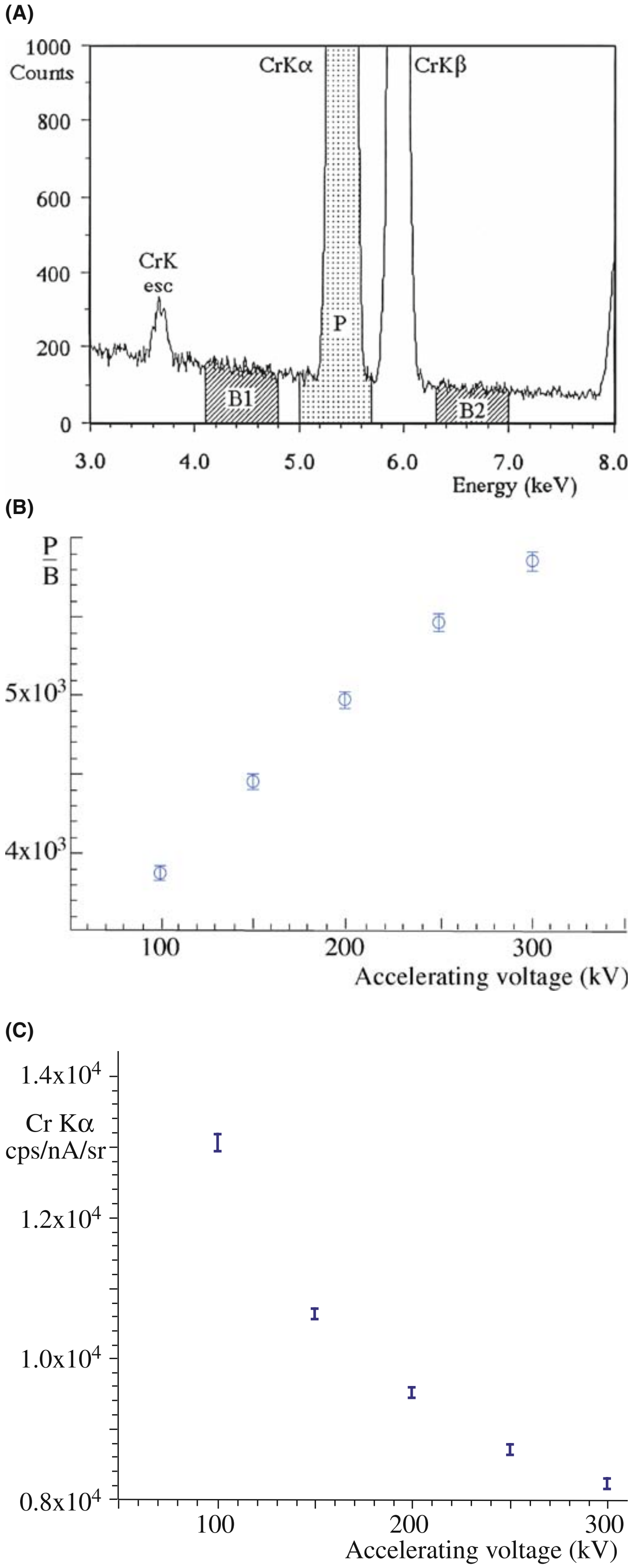

FIGURE 33.10. (A) Definition of the Fiori P/B ratio in a Cr thin film. (B) Change in the Fiori P/B as a function of kV for a high-performance 300-kV FEG AEM. (C) Decrease in the cps/nA/sr from a thin Cr film demonstrating decreasing detector collection and processing efficiency with increasing kV.

33.5 ACQUIRING X-RAY SPECTRA

There are many commercial systems for acquiring spectra and images and many of these differ in terms of the file formats used within the computer system. Consequently, you might find it difficult to exchange spectra between different XEDS systems (e.g., for comparison of data gathered on two different AEMs). There has been a concerted attempt by the various manufacturers and professional societies to rectify this by creating the so-called 'EMSA/MAS standard file format' (Egerton et al.). You should check to ensure that your XEDS system supports this format.

So from what we've told you so far, you now know what information is likely to be in your spectra; which peaks might be real, which are most likely artifacts, and which ones you can and can't interpret as coming from the analysis region. Now we can concentrate on how best to actually gather the spectra before proceeding with analyses.

33.5.A Spot Mode

The standard way of gathering spectra, from the earliest days of AEM (and preceded by SEM/EPMA) was simply to use the beam deflectors to position the spot on a feature in your image and switch on the XEDS. We call this 'spot mode.' You could do this in a TEM by condensing the beam down with the C2 lens and adjusting C1 iteratively until the beam is small enough to interact only with the feature you wanted to analyze, such as a precipitate. In STEM spot mode you simply stop the beam from scanning and move the probe onto the feature, hoping to get it in the right position before the image fades from the STEM screen. In either case you also have to hope that both the probe position and the feature in your image stay stationary for long enough to gather a spectrum with sufficient counts to give you the composition information you need. On vintage AEMs, this method ensures that any carbon contamination buildup precisely buries the feature of interest! However, that same contamination spot would also show you if the beam or specimen had drifted during the analysis.

So spot mode is time consuming, the statistical confidence is appalling, and the pre-selection of which feature(s) to analyze introduces serious operator bias (and if you're not careful, all that you might analyze is an artifact of the specimen preparation rather than a key microstructural feature). Despite all this, it is still common to see publications in which this method is precisely how the analysis was performed and, in terms of giving a gross indication of local variations in chemistry, say for a complex extraction replica containing multiple phases, it certainly has some benefit, as shown in Figure 33.11. So, be careful when you build on someone else's results.

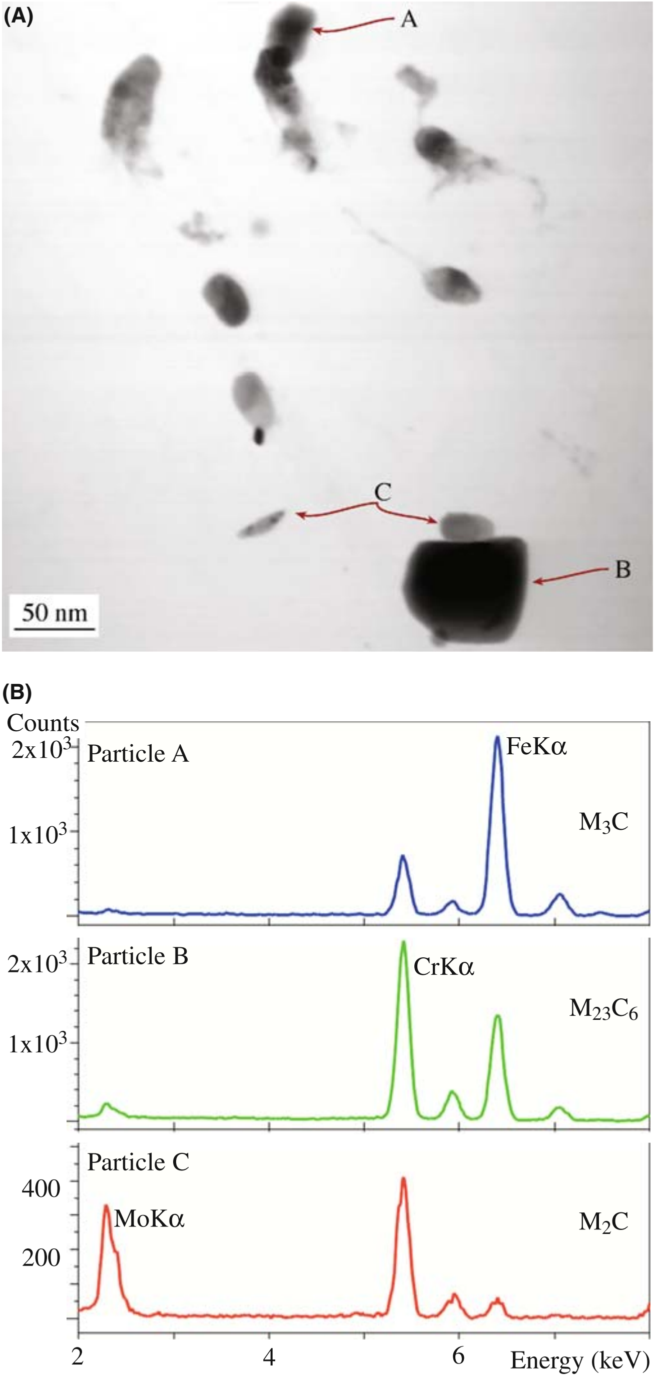

FIGURE 33.11. (A) STEM image of different carbide particles on an extraction replica from stainless steel specimen. (B) Multiple spectra taken from an array of points in the micrograph illustrating the variable chemistry of the different carbides.

33.5.B Spectrum-Line Profiles

A variation of the spot mode of analysis is to take a series of spot analyses across a linear feature of interest in your specimen, such as a grain boundary or interphase interface, as shown in Figure 33.12A, and build up a set of spectra which, when analyzed, will reveal the composition profile across the interface. The information can be displayed as a set of superimposed spectra, termed a spectrum-line profile, as shown in Figure 33.12B, and the changes in the characteristic-peak intensities reflect significant composition changes. Since any kind of interface is a major planar defect in an engineering material, this approach at least removes some of the operator bias of the single-spot mode. However, line profiles still only reveal the composition across a single point on the boundary and many such profiles are required if you are to determine composition variations along the defect. So this approach is also tedious.

The solution to the limitations of both spot and line profiles approaches is to gather parts of spectra or preferably full spectra at every pixel in the STEM image, producing compositional images or maps.

This method is by far the best way to gather X-ray information with some semblance of statistical significance and without operator bias, so we'll now spend the rest of the chapter describing this method.

33.6 ACQUIRING X-RAY IMAGES

Mapping, or compositional imaging, used to be rarely used for analysis in the AEM because of the low X-ray count rate due to the small probe currents and collection angles. The overall inefficiency of the process meant that to gather a map with sufficient X-ray counts to be able to draw any conclusions about the variations in chemistry in the thin foil, you would have to scan the area for many hours. During this time, specimen drift, contamination or damage would occur and the resultant information would be compromised. However, with the developments of intermediate-voltage FEG sources, detectors with higher collection efficiency, drift-correction software, cleaner stage vacuums, and, most recently, C_s correctors, it is now possible, on the best AEMs, to gather quantitative X-ray maps in a matter of minutes. So it's worth looking at the various imaging options which have been developed, along with these improvements in AEM design and computer technology. In all of these approaches it will take significant

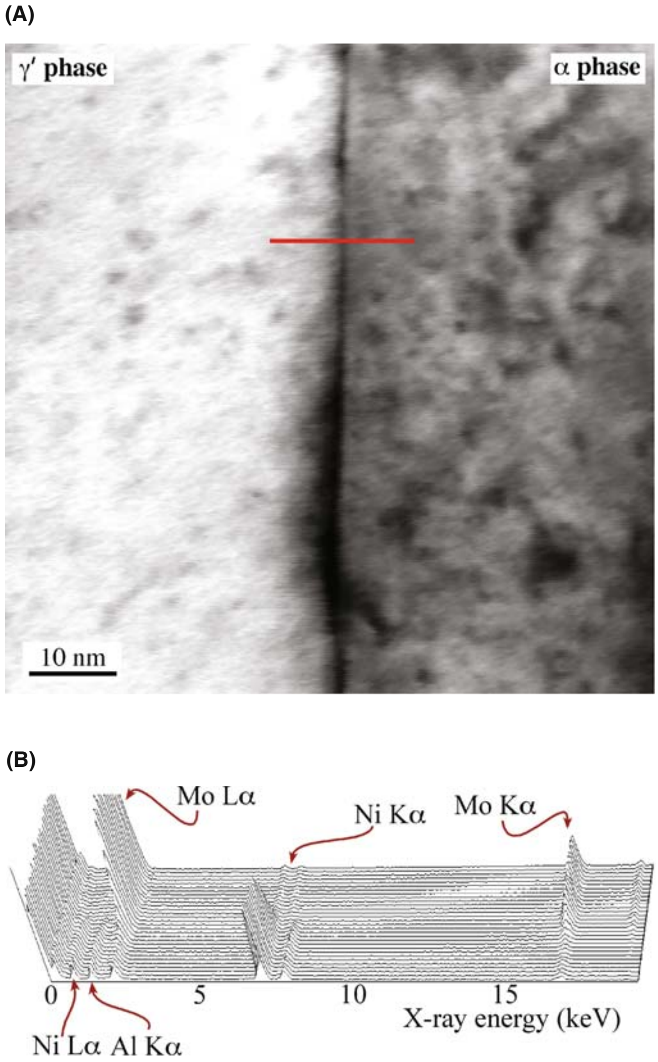

FIGURE 33.12. (A) STEM image of an α/γ' interphase interface in a Ni-base superalloy with a line showing from where X-ray spectra were obtained. (B) A spectrum-line profile taken along the line in (A) showing clearly the change in Ni and Mo composition across the interface.

time to acquire a map and the biggest danger is that the specimen will drift during that time or the beam current will change (particularly, if you have a cold-FEG source). So learn about the drift-correction software that should be available in any standard commercial XEDS software package. Unless you can measure the probe current *on the fly* in a cold-FEG AEM, a Schottky FEG is recommended for mapping.

Analysis of bulk specimens in the SEM or EPMA is not limited by X-ray counts so the X-ray imaging techniques we'll discuss were generally pioneered on the SEM/EPMA. Likewise, as we'll see in Chapter 37 and beyond, EELS measurements have many millions or even billions of counts and so EELS mapping of thin films was used long before thin-foil X-ray analysts were able to benefit. But in this book, we'll rewrite history a little and talk first about all these techniques in reference to X-ray mapping.

33.6.A Analog Dot Mapping

Dot maps are the original method of acquiring qualitative X-ray images and, somewhat surprisingly, were first created more than half a century ago by Cosslett and Duncumb and progress since then has been reviewed by Friel and Lyman. The approach is simple: you select a specific energy (or wavelength) channel (or a window (range) of channels) in your X-ray spectrum, scan the beam across the area of interest and when the X-ray detector registers an X-ray of the selected energy (range), record and display it. So as the beam scans, the display intensity builds up and the changes in intensity reflect the changes in the number of X-rays detected. For example, if you select the Pd L_α peak channel or a window covering the peak, then regions showing lots of dots on the screen are high in Pd, as shown in Figure 33.13 (we'll go into more detail about how to do this in Section 34.7). This qualitative, analog

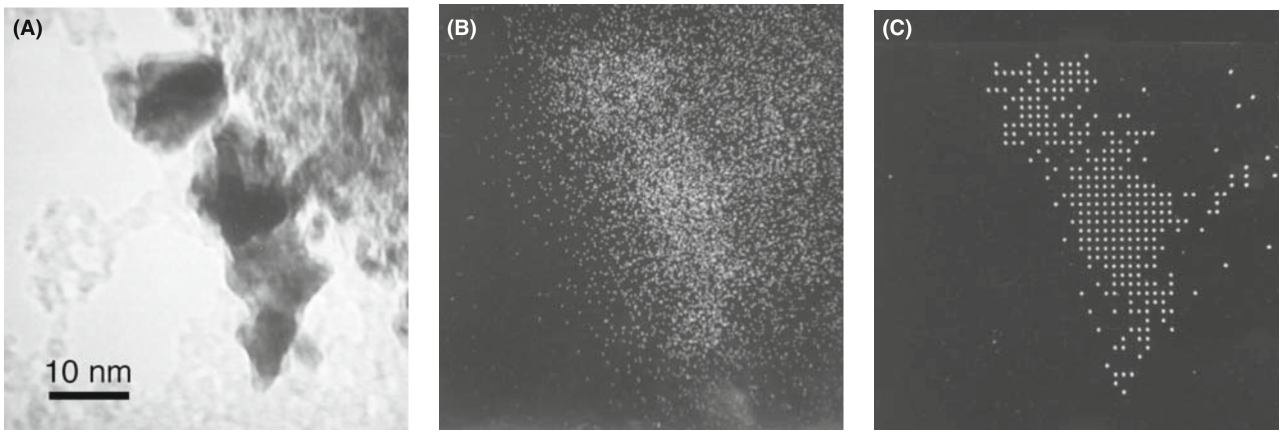

FIGURE 33.13. (A) STEM BF image of a Pd catalyst particle on a support film. (B) Analog dot map using the Pd L_α signal. Note the correlation between thicker (darker) regions of the specimen and increased Pd signal. (C) Early digital map taken with Pd L_α signal but subtracting the background signal.

approach is not directly quantifiable since the background can't be removed, unless you simultaneously gather a dot map from a bremsstrahlung window adjacent to the peak window and subtract the one from the other (more about this in Chapter 35). Furthermore, in thin foils, changes in thickness will also produce changes in intensity in both the peak and background maps and this is clear in Figure 33.13B. It is possible to refine this approach by gathering multiple X-ray maps, assigning colors to different X-rays, and overlaying the maps to give an indication of relative composition changes. Again, this process is *not* quantitative.

33.6.B Digital Mapping

As computers became more powerful, it was possible to collect X-rays from multiple channels or windows and thus acquire several maps simultaneously. If one or more of the maps was the bremsstrahlung intensity then you could produce quantitative maps, on the fly, as the data accumulates. The first success in digitizing thin-foil X-ray maps was carried out by Hunneyball et al. almost 30 years ago. It was only possible to build up 128×128 pixel images with 256 X-ray counts at each pixel using a 100-nm probe with 5 nA of probe current over 200s. But the maps were fully quantitative, removed the effects of foil-thickness variation, and revealed relatively small composition variations around GBs in aged Al alloys, as shown in Figure 33.14A. Because of the central role of the computer in the acquisition, this approach has the advantage of post-acquisition processing and comparison of different quantification routines.

Also after acquisition it was possible to use such processing techniques as false coloring, computerized image overlays, scatter diagrams, etc. (Bright and Newbury). All these advantages combine to make digital imaging a most attractive approach for displaying XEDS data. This process really came of age in the mid-1990s with the availability of faster computers, improved data storage, intermediate-voltage FEG sources, reasonable X-ray collection angles, and stable AEMs permitting long collection times without drift. An example from such an AEM is shown in Figure 33.14B which is 256×256 pixels and was acquired using a 1-nm probe with 0.9 nA of current for 5400 s. Comparing Figure 33.14A and B is instructive and

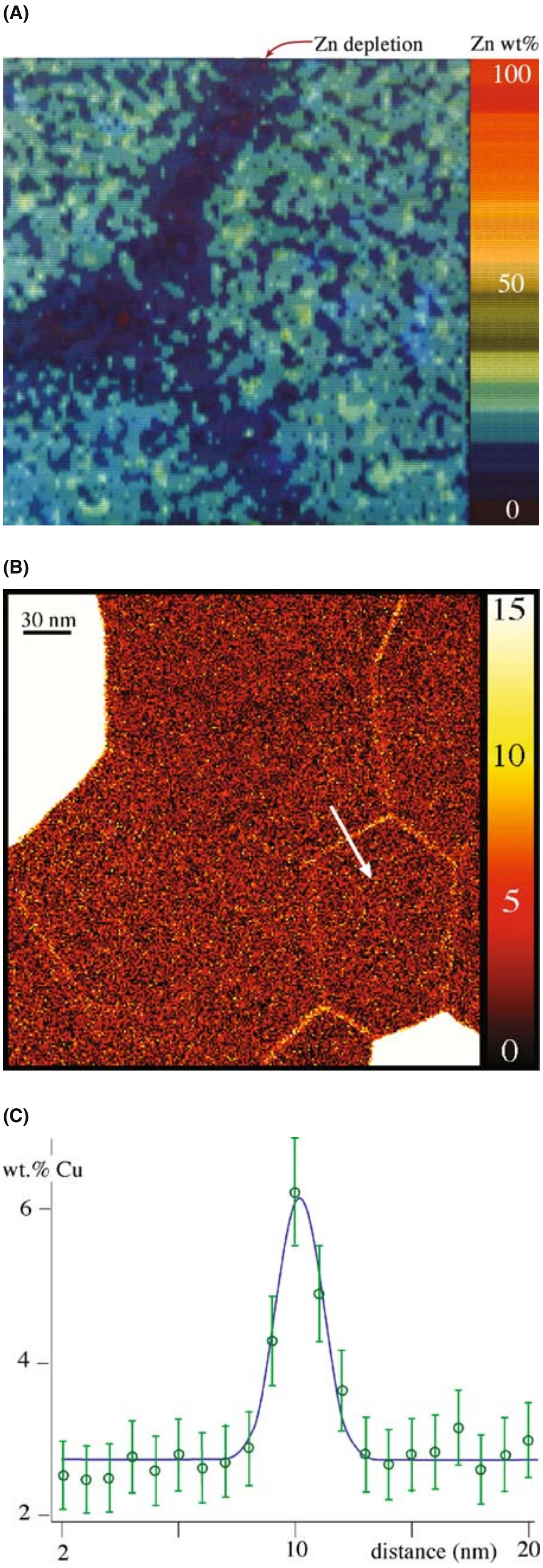

FIGURE 33.14. (A) The first quantitative digital map (128 × 128 pixels) obtained in an old thermionic-source AEM generating a low count rate. The image from a thin foil of Al-Zn shows depletion of Zn around a triple point (compare the colors in the map with the quantitative look-up 'table' on the right side of the image). (B) More recent digital map from a 300-kV FEG AEM showing enrichment of Al at GBs in an electro-migrated specimen of Al-4% Cu. The bright regions are $CuAl_2$ intermetallics. In both (A) and (B) quantification was achieved by subtracting the bremsstrahlung intensity at each pixel. (C) Quantitative Cu line profile taken across a GB indicated in (B), by arrow.

shows the enormous progress in both the quality of mapping and the spatial resolution. Once you have a digital map such as this you can go into the map and extract quantitative data from any region, e.g., the line profile taken across the GB in Figure 33.14C. However, you still have to select the window(s) or peak(s) in the spectrum that you map, thus introducing your own bias in terms of what you expect to be in the specimen. Furthermore, once the map is recorded and stored, you cannot return to re-check the data, or map another element from the same area, since all the other information in the spectrum was lost. Likewise, unless you store the image from the mapped area, it too cannot be re-examined. So you have to get everything right, collect all the X-ray images you need, as well as the background spectra to permit subsequent quantification. Gathering multiple maps was limited by the computer memory, but that is no longer a problem since memory is cheap. Now, as we discuss below, you don't even have to bother about pre-selecting which X-rays you want to map, you just gather them all and later on decide which you want to image. How do we do this? It's called 'spectrum imaging.'

SPECTRUM IMAGING

SI is *the* preferred method for X-ray (and EELS) mapping.

33.6.C Spectrum Imaging (SI)

As the term implies, SI collects a full spectrum at every pixel in the digital image (so you can only do this in STEM mode (although there are analog versions in energy-filtered TEM, as we'll see in Chapter 39)). The result of the SI process is a 3D data cube, as shown in Figure 33.15A, with the electron image constituting the *x*–*y* plane and all the XEDS spectra in the *z* direction. The SI term was first used for EELS in the late 1980s and we'll mention this topic again in Chapter 37. Only much later did SI became feasible for X-ray mapping although it is now common enough to be used in materials problem solving (e.g., Wittig et al.). On a historical note, you should be aware that SI methods have been practiced in other fields, such as radio astronomy for several decades and, indeed, Legge and Hammond took the output of their EDS and WDS spectrometers and synchronized the

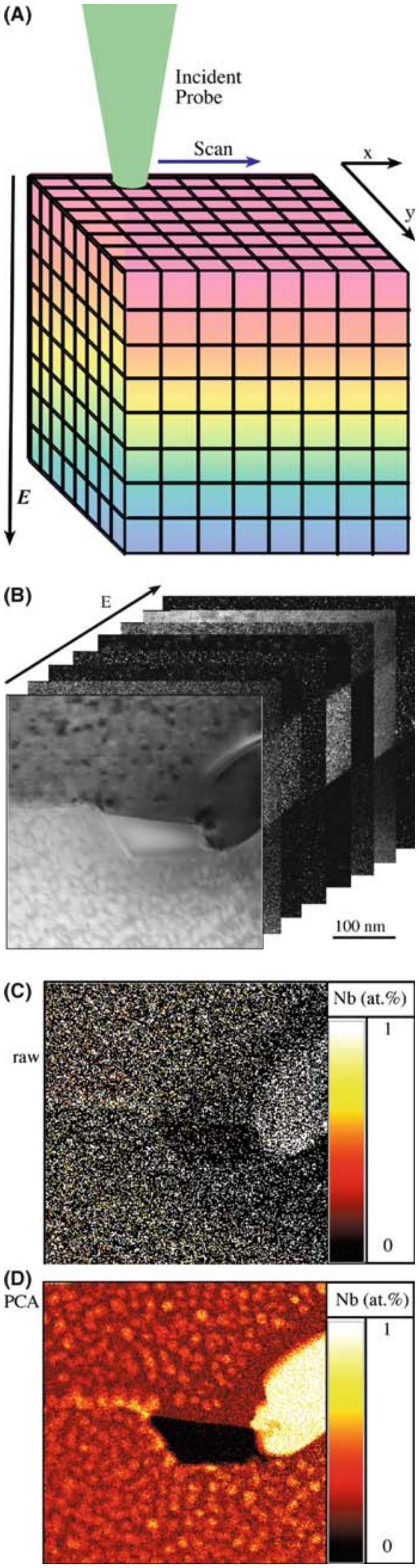

FIGURE 33.15. (A) Schematic spectrum-image data cube showing how as the beam stops at each pixel in the *x*–*y* plane as it is creating a STEM image, a full X-ray spectrum is gathered at each pixel. The different colors at different energy (*z*) values indicate different signals from different elements that appear at different energies. (B) A series of X-ray maps of a GB region in a Ni-base superalloy taken at specific energies from an SI data cube. (C) An example of a map from a single channel in the X-ray spectrum (i.e., a single image plane) in the SI in (B) coinciding with the Nb K_α peak energy. (D) The application of multivariate statistical analysis and principal-component analysis to remove noise and enhance the Nb signal.

detectors' output pulses with the position of the beam 30 years ago. They collected the data on magnetic tape, and later reconstructed the data into a 3D file, so it can be argued that this was the first demonstration of the SI concept, at least in electron-beam instruments, so we aren't really revising the historical record!

The beauty of the SI technique is that, once you have this data cube stored, you can go back to it at any time, recheck the data, and re-do any analysis, search for other spectral features that you might not originally have thought present or important, look at different images at different energies, and yet always have the original image and spectra at your disposal.

If you think about the diagram in Figure 33.15A, you'll see that there are many ways to slice and dice the data cube which reproduce all the other methods of analysis that we've described. If you select a single pixel in the x–y (image) plane, then there is a full spectrum attached to it and so, if you wish, you can select a set of individual pixels and do as many spot analyses as you wish. Likewise, you can select a line of pixels in the image, effectively slicing the cube along the x–z, y–z, or some combination of these directions and thus produce spectrum-line profiles. You can (and this is where it gets really useful) slice the cube at any plane in the z (labeled E for energy) direction and, at each plane, you'll get a different image consisting of X-rays of that particular energy. You can add planes, subtract planes, and sum image pixels only in certain features (e.g., a strangely shaped precipitate or along a boundary plane). You can also envisage gathering spectra at certain pixels as a function of time (chrono-spectrometry?) and there are surely other options which you can think of. Now if you do the math and consider a 1 k × 1 k pixel image with a 2048-channel spectrum at each pixel, you'll find that the data cube is about 2 GB in size. In fact, in gathering such an enormous amount of data, the bigger challenge is to find ways both to search this enormous database efficiently and extract meaningful data. Various advanced software methods, such as multivariate statistical analysis, principal-component analysis, and maximum-pixel spectrum analysis are available to remove noise and extract rare events from the data cube. Such processes are closely related to the parameterizing of HRTEM images discussed in Chapter 31, and are discussed in far more detail in the companion text.

Just to give you a hint of what can be done, Figure 33.15B shows a series of X-ray images cutting the x–y plane at different energy values, displayed 'behind' the original STEM ($E = 0$) image. If we select a single x–y plane such as that at the Nb K_α line energy (Figure 33.15C), that image is very noisy because of the limited signal captured in that one plane. However, we can use sophisticated software to remove all the noise components in the plane and also add in all the other Nb signals from other energy planes to produce the image showing the Nb distribution (Figure 33.15D). Before you think that such an extraordinary change is unreasonable and that information is being created where it did not exist, compare the processed image (D) with the original STEM electron image (the top slice in B). Clearly the small matrix precipitates that are imaged in (A) are also mapped in (D). Extracting information like this from the SI data cube tells you how much more powerful the SI approach is compared with spot or line-profile analyses. Now it is routine to gather both XES and EELS SI simultaneously, thus completely optimizing the acquisition of analytical data.

33.6.D Position-Tagged Spectrometry (PTS)

PTS is a specific commercial version of generic SI, developed by Mott and Friel at Princeton Gamma Tech (PGT, now Bruker AXS). PTS eliminates the conflict between your wishing to view full X-ray images quickly versus the analytical advantages of having complete spectra saved at each pixel (which even on the best AEMs still takes 30–45 minutes if you want to quantify the data). In PTS, the beam is scanned rapidly relative to traditional mapping, and the X-rays are counted in the analysis computer, preserving both spatial information from the image and spectral information. Sophisticated processing software can be used to interrogate the data *during* acquisition, which is not possible in conventional SI. Alternatively, this software can be used after the complete spectrum is stored, as with conventional SI, where you acquire the full spectrum at a single pixel and then move to the next pixel and gather another spectrum. PTS also permits relatively easy monitoring of such phenomena as specimen drift, contamination, or damage during the acquisition.

CHAPTER SUMMARY

XEDS in the AEM is a challenge because the detection/processing system creates artifact peaks in the spectrum and X-rays are generated and detected from sources in the AEM other than the region of your specimen where you put the beam. Nevertheless, there are well-defined precautions you can take so that you are sure that the artifacts, the spurious and system X-rays are minimized and that your subsequent interpretation and quantification are not compromised. There are also several standard tests you can carry out to compare your AEM system performance with other instruments.

In summary, to understand your AEM and to acquire meaningful spectra, you should

- Buy your own NiO standard thin film on a Mo grid.
- Test your XEDS to determine the artifact peaks that it produces.
- Gather a spectrum down a hole in the NiO film to see what spurious X-rays the AEM illumination system produces. Use top-hat C2 apertures at all times.
- Gather an XEDS spectrum from a light-element film to see what system peaks your AEM introduces into the spectrum. Use thin foils, flakes, or films rather than self-supporting disks if possible.
- Be aware of CB if you count for a long time to detect small peaks from trace elements.
- Image the electron beam on the TEM screen to ensure that it is Gaussian.
- Always remove the objective diaphragm.
- Operate with the specimen as close to zero tilt as possible.

Check that

- The hole count is <1% of your experimental spectrum under the same operating conditions.
- The *P*/*B* ratio and the detector efficiency data are acceptable and do not change with time.
- If you're looking for characteristic peaks from trace or minor elements in your specimen, understand that such peaks are much more likely to be confused with artifacts or system peaks (much more about this in the next chapter).

Once you are sure of what's in your spectrum, decide whether you want to do quick and dirty point analyses of some features in your image or, if you are confident of the importance of the area that you want to analyze completely, gather X-ray maps or, ideally, acquire a SI data cube which will really give you the full picture of your specimen composition.

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URLs

1) www.cstl.nist.gov/div837/Division/outputs/DTSA/DTSA.htm To download DTSA

SELF-ASSESSMENT QUESTIONS

Q33.1 What's the difference between spurious X-rays and system X-rays?
Q33.2 What are the best steps you can take to minimize spurious X-rays?
Q33.3 What causes incomplete charge collection (ICC) in an XEDS?
Q33.4 Name three common artifacts in the spectra generated by XEDS systems.
Q33.5 Which of the various artifacts should you be particularly aware of while operating your AEM and why?
Q33.6 Distinguish the sources of desired and undesired radiation impinging on the detector.
Q33.7 How can you find out what X-rays are generated by your AEM-EDS system rather than by your specimen?
Q33.8 Why is it important to know what your system X-rays are?
Q33.9 What's the danger of not minimizing any spurious X-rays?
Q33.10 Why would you not use a Cu grid to analyze diffusion profiles in brass using XEDS in the AEM?
Q33.11 Why is it challenging to detect trace (~0.1 wt%) amounts of Si unambiguously in an Fe alloy using XEDS?
Q33.12 Why is a top-hat C2 aperture/diaphragm so called and why is it essential to have such apertures in your AEM?
Q33.13 Ideally how many C2 apertures should you have in an AEM?
Q33.14 How can one minimize post-specimen scattering?
Q33.15 What causes coherent bremsstrahlung (CB)?
Q33.16 If the hole count is >>1%, what is likely causing this?
Q33.17 Is STEM well suited for AEM? If so, why?
Q33.18 From the extra information in this chapter, explain why it is so important to align your X-ray detector with any planar interface.
Q33.19 List four ways by which you can minimize post-specimen scattering.

Q33.20 Why might CB be a problem that could cause you to misinterpret your analysis?
Q33.21 Give a simple way to distinguish a CB peak from a characteristic peak.
Q33.22 Why is it better to form X-ray images rather than individual spectra or line profiles?
Q33.23 Why is it much more difficult to acquire good X-ray images than individual spectra?

TEXT-SPECIFIC QUESTIONS

T33.1 A P/B ratio is measured on the Ni K_α line at 300 kV using the Fiori method and is determined to be 1000. What should you probably do?
T33.2 Why don't microscope manufacturers just coat the insides of the AEM with Be on all surfaces?
T33.3 Why coat any of the insides of the microscope at all with a low-Z material?
T33.4 To decrease the X-ray background for a particular specimen, the kV should be increased. Why?
T33.5 In an experiment, some unidentified X-ray peaks are present. Name some possible causes.
T33.6 Upon measuring a hole count for a particular specimen, you discover that the illumination system is not 'clean.' Besides being floored by this discovery, you want to know what you can do to fix it. So what do you do?
T33.7 Having become quite the expert on XEDS in the SEM, your lab partner tilts the specimen in the TEM chamber to 45° 'just like we did in the SEM.' Before smacking your lab partner repeatedly, what should you remind him of?
T33.8 Upon analyzing the XEDS spectra of your material, you cannot conclude what several peaks correspond to. You have tried sum peaks, escape peaks, and the internal fluorescence peak with no luck. Have you discovered a new element, or is there another answer that is more likely?
T33.9 David and Stuart are working at the 1210 when we arrive one morning. They are using XEDS, and all their spectra have peaks below 1 keV. What in the world are they doing?
T33.10 How would you discern if your X-ray spectrum contains a significant amount of spurious or system X-rays? How can you distinguish the spurious contributions from stray X-rays and stray electrons?
T33.11 Calculate the energies of the principal coherent bremsstrahlung peaks generated from a thin foil of Cu in a <001> orientation by 120-kV electrons when the EDS detector has a 20° take-off angle. Compare your answer with Figure 33.9 and comment on any discrepancies. (Hint: you'll have to find the lattice parameter of pure Cu.)
T33.12 How wide do you think the probe in Figure 33.5 is? Explain your reasoning.
T33.13 If the electron probe that you use to do analysis is as shown in Figure 33.5, what effect would this have, for example, on a line-profile analysis across an interface?
T33.14 Which effect(s) in Figure 33.6 is (are) causing the extra peaks in Figure 33.7?
T33.15 Are any other peaks in Figure 33.7 not caused by effects in Figure 33.6 and if so what is causing them?
T33.16 Explain the reasons for each of the directives at the end of Section 33.3.B.
T33.17 Distinguish analog and digital mapping and explain why one is so much better than the other.
T33.18 Distinguish SI and PTS.
T33.19 Make a list of the different ways to cut up the SI data cube and explain the different information that you would get from each cut. See if you find one that has not been published and make a name for yourself.