USING A MICROCOMPUTER TO AID IN THE TEACHING OF BEAM-SPECIMEN INTERACTIONS IN SCANNING AND TRANSMISSION ELECTRON MICROSCOPY

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Abstract

A microcomputer can be usefully incorporated into the classroom and the laboratory to aid in the teaching of electron microscopy concepts. Two examples are described showing a) how the microcomputer can be used as a low resolution multichannel analyzer to process X-ray spectra in a variety of ways and b) how Monte Carlo simulations of electron trajectories in electron microscope samples can be used to illustrate beam-specimen interaction phenomena.

KEY WORDS: Microcomputer, multichannel analyzer, Monte Carlo simulation, beam-specimen interaction

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Introduction

Recent advances in both scanning and transmission electron microscopy (SEM and TEM) have used the computer to aid in their development. When attempting to describe these advances in the classroom, it is not usually convenient to enlist the aid of a mainframe computer. However, it is often possible to obtain satisfactory results using a microcomputer which is portable and therefore easily used in the classroom or in the laboratory adjacent to the microscope itself.

Traditionally the role of the computer in the SEM field has developed in conjunction with developments in X-ray microanalysis and the creation of more sophisticated correction routines (such as ZAF) for quantifying X-ray spectra. Similar developments have taken place in the TEM area with the evolution of analytical electron microscopy (AEM). There are now several alternative methods for manipulation of X-ray spectra, even at the initial stage of extracting characteristic intensity information from the bremsstrahlung background. These necessary procedures are often incomprehensible to the initiated X-ray microanalyst since they are now customarily carried out without the direct control of the operator. Thus the multichannel analyzer (MCA) system becomes a 'black-box' in which unknown calculations are performed when the operator selects the 'analyze' control on the MCA computer. The various ways in which characteristic X-ray intensities are measured can be described simply on a microcomputer such as an Apple II. In this paper we will describe three different ways in which commercial MCA systems extract X-ray intensity information from an X-ray spectrum prior to quantification. The three operations can be demonstrated in a matter of minutes in the classroom, and thus the microcomputer can be used as a portable MCA. As a final step to quantification we will not carry out a full ZAF correction (although this is possible) but rather we will use the more simple thin film analysis procedure typical of an AEM system.

The second and more recent area where computers have begun to play an increasing role is in the simulation of beam-specimen interactions using Monte Carlo techniques. This is rapidly transforming the SEM into a far more quantitative...
In this section we will discuss various quantification procedures using the data from an energy dispersive x-ray spectrum (EDS) obtained from a binary alloy system in an AEM at 120 kV. An Apple IIe microcomputer is used as a low resolution MCA with a video display. A typical EDS spectrum will be analyzed in alternative, different ways, typical of commercial MCAs. The particular spectrum, shown in Figure 1, is from a specimen of nominal composition Fe-35 wt% Ni. (All figures in this paper are photographed directly from the screen of the Apple.) The spectrum contains characteristic peaks from Fe (Kα and Kβ) and Ni (Kα and Kβ) on a slowly decreasing background. The first step to quantify this spectrum is to determine characteristic intensities (I_Fe and I_Ni) above background.

The simplest method of quantification involves determining the Fe and Ni Kα peak intensities using 'windows' or 'regions of interest' on the MCA display to select the region of the peak over which integration is required to give I_A and I_B etc. for quantification. To do this we use the cursor or equivalent selecting tool to pick out the desired peak, such as the Fe Kα peak, as shown in Figure 1. From the readout at the base of Figure 1 we see that the peak channel contains 2221 counts. We then move the cursor and select channels on either side of the peak which contain just less than half 2221 counts. This defines the 'window' in the spectrum just greater than the full width at half maximum (FWHM) of the characteristic peak. Theoretically the best width is 1.2 FWHM since it can be shown that this width optimizes the peak to background ratio (P/B) and the counting statistics, both of which are important in X-ray microanalysis.

Similar procedures can be carried out for each characteristic peak, and this is shown in Figure 2. In order to obtain the net counts necessary for quantification it is necessary to subtract the background. The simplest way to do this is to select windows of the same channel width (~1.2 FWHM) in the background above and below the characteristic peak, and this has also been carried out in Figure 2. This background intensity either side of each peak is then averaged and this average value is subtracted from the gross number of X-ray counts in the characteristic peak.

The 'window' method for estimating the background is simple and is as accurate as any other method so long as the characteristic peaks of interest are isolated (i.e., no spectral overlaps occur) and the bremsstrahlung background intensity is a slowly varying function of the X-ray energy (i.e., at energies >2 keV). If these conditions do not apply we have to use one of the next two methods to estimate and subtract the background.

The second quantification method involves mathematically modelling the spectrum. The background intensity distribution can be modelled using a Kramers' Law type approach for bulk samples. This gives an expression of the form:

\[ I_E^B = KZE_0 - E \]

where \( I_E^B \) is the generated X-ray intensity per unit energy interval per incident electron, \( E_0 \) is the electron beam voltage, \( E \) is the photon energy, \( Z \) is the specimen atomic number, and \( K \) is a constant, often called Kramers' constant.

Over the small energy range we are looking at in the Fe-Ni specimen (#6-8 keV) the model describes a slow, almost linear decrease in background intensity with increasing X-ray energy. When we apply this Kramers' Law type of fit to the background in our spectrum we get the dotted line visible along the base of Figure 3. In this case the low resolution of the microcomputer display limits the detail that can be shown, and for all practical purposes the background intensity is a straight line variation with energy. Substantial deviation from a straight line only occurs at energies <2 keV when absorption of low energy X-rays in the sample and in the EDS detector itself causes a rapid decrease in the detected intensity.

Having modelled the background we now have to model the characteristic peaks and the simplest method is to approximate the peak shape to a Gaussian. This is not an exact description but accurate enough for teaching purposes. (In practice, the detected experimental peak shape may not be actually Gaussian due to such phenomena as incomplete charge collection, but this can be accounted for in more complex modelling programs.) Careful inspection of Figure 3 shows the good fit (white dots) to the Fe Kα, Kβ and Ni Kα peaks obtained using a simple Gaussian model. The Ni Kβ peak however is not well fitted, because of the poor counting statistics (total Ni Kβ intensity is ~500 counts) and this illustrates well the need to obtain good statistics prior to quantification. In combination with the background fit we can again obtain the intensities above background in the Fe Kα and Ni Kα peaks.

The third method for quantification involves removing the background by a process known as 'digital filtering' and then determining the peak intensities by comparing them with previously
Energy dispersive spectrum obtained from a thin foil of Fe-35% Ni at 120 kV in the AEM. The Fe and Ni Kα peaks are identified and a cursor is visible at the top of the Fe Kα peak. The alphanumeric display shows that the cursor is at 6.41 keV (Fe Kα peak energy) and that the single channel at 6.41 keV contains 2221 counts.

Fig. 2. The spectrum in Fig. 1 with suitable 'windows' or 'regions of interest' inserted. The windows are 1.2 FWHM in width. The background intensity is obtained from windows B1, B2, and B3. By averaging B1 and B2, the average background below the Fe Kα peak can be estimated. A similar procedure using B2 and B3 permits the Ni Kα background to be determined.

Fig. 3. The Fe-Ni spectrum showing the background intensity modelled according to Kramers' Law and the characteristic peaks modelled using Gaussian peaks. The Kramers' Law fit is essentially a straight line over this limited energy range, and the Gaussian fit is reasonable except in the case of the Ni Kβ peak, which has a low intensity and therefore poor counting statistics.

obtained standard spectra. This process simply involves taking our experimental spectrum and approximating it to a combination linear + Gaussian plot, then convoluting it with a function that, because of its shape, is often termed a 'top-hat' filter. The original spectrum and the resultant convolution are shown in Figures 4a and 4b respectively. The background has been converted to a straight line and the characteristic peaks have been smoothed. Although there are now apparently 'negative counts' below the straight line background it can be shown that relative peak intensities, as used in quantification procedures remain unchanged.

Fig. 4. The Fe-Ni spectrum (a) prior to and (b) after digital filtering. In (b), the background intensity distribution is transformed into a straight line of value zero, and the major characteristic peaks are shown still retaining their Gaussian shape. This process also results in a smoothing of the spectrum.
To obtain the peak intensities from a digitally-filtered spectrum it is usual to compare the filtered peaks with the intensity in previously obtained 'library standard peaks.' Figure 5 shows such standard peaks for Fe (Kα and Kβ). These library standards are also digitally filtered and knowing accurately the counts in the library standard peaks the counts in the experimental peaks can be obtained by directly scaling the height of an experimental peak to its standard peak. Using this process we can again obtain intensities in the Fe Kα and Ni Kα peaks.

The most important step is to ensure that, whatever method is used to extract intensities, indeed the answer to the quantification procedure is correct, and independent of the method used. In this example from a thin foil sample, quantification is simple, using the Cliff-Lorimer equation for two elements A, B:

\[
\frac{C_A}{C_B} = \frac{k_{AB} I_A}{I_B}
\]

Knowing that \(k_{FeNi} = 0.88\) and the values for \(I_{Fe}\) and \(I_{Ni}\) we find the results from our three methods as given in Table 1. From this we can see that, within the errors (which are ± 3 \(\sigma\) using the counting statistics only) the answers are identical. The specimen used was Fe-35 wt% Ni so the answers are also precise. From a teaching standpoint it is worth noting therefore, that in the case of a simple binary system the 'window' method is as good a quantification procedure as the more sophisticated, computerized modelling and filtering procedures. However in 'real' multi-component specimens, particularly containing light elements where the bremsstrahlung background changes quite rapidly (below 2 keV), the simple 'window' method is difficult, if not impossible, to use with acceptable (<± 10%) levels of accuracy.

Use of Monte Carlo simulations on a microcomputer

The Monte Carlo simulation in SEM is commonly used to model the interaction of an electron probe with a bulk specimen. Most Monte Carlo programs that have been described in the literature or in textbooks require large mainframe computers and expensive amounts of computer time. However we have recently developed a Monte Carlo program which runs on an Apple microcomputer and can be used to illustrate beam-specimen interactions in the classroom.

Because the microcomputer-based Monte Carlo takes many hours to run, particularly when simulating interactions in bulk SEM specimens, it is best used to illustrate the principles of electron beam-specimen interactions or to consider thin foil applications only. We will show examples of both these approaches which are suitable for instruction in the classroom and also in the research laboratory. Again the advantage of the microcomputer approach is that the instrument is available to a wide range of users at their convenience (i.e., students may experiment with the program in their own time, and simulate interactions of interest to themselves).

First we will illustrate some of the simulations that can be used in the classroom simply to describe bulk specimen interactions with the electron beam. Figure 6a shows the electron trajectory simulation for 25 kV electrons interacting with an 'ideal' specimen consisting of a precipitate of Au surrounded by a matrix of Mg. The difference in penetration, electron path length and total excitation volume are obvious. Knowing the path length for X-ray excitation in each

### Table 1

<table>
<thead>
<tr>
<th>Method</th>
<th>Background</th>
<th>Gaussian Peak Fitting with Kramers' Law Background Fitting</th>
<th>Peak Fitting Using Library Standards, with Digital Filtering of Background</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt% Fe</td>
<td>65.9 ± 1.1</td>
<td>66.1 ± 1.1</td>
<td>63.9 ± 1.1</td>
</tr>
<tr>
<td>wt% Ni</td>
<td>34.1 ± 1.1</td>
<td>33.9 ± 1.0</td>
<td>36.1 ± 1.0</td>
</tr>
</tbody>
</table>

ERRORS ARE ± 3 \(\sigma\) (99% confidence limits using counting statistics only)

SPECIMEN is Fe-35% Ni (using electron microprobe bulk analysis)
Microcomputers--the teaching of electron microscopy

Fig. 6. a) Monte Carlo simulation of electron trajectories from three points of incidence (arrowed) in an 'ideal' specimen consisting of a 'precipitate' of Au (central region) surrounded by a 'matrix' of Mg. The difference in electron trajectories as a function of atomic number (Z) is obvious. Fifty trajectories (histories) were simulated at each point.

Fig. 6. b) X-ray generation distribution for the electron trajectories in Fig. 6a. The differences in excited volume (and therefore spatial resolution of microanalysis) are clear.

It is a simple matter to ask the computer to simulate the beam-specimen interactions that give rise to X-ray generation, and the resultant X-ray generation plot is shown in Figure 6b. From the printout that accompanies such plots (see Figure 7 for example) it is also obvious that such factors as the backscatter coefficient are readily calculated. The depth distribution of X-ray production (\( \phi(\rho) \)) can be deduced, and the matrix and precipitate dimensions and chemical characteristics can also be changed at the whim of the instructor running the program.

To run a full simulation such as that in Figures 6 and 7 may take 50 hours and is therefore not useful on-line in the classroom. These simulations are best run over a weekend and the results collected, stored and used as static displays to illustrate certain points such as effects of atomic number, density, beam KV, etc. on beam-specimen interactions in the SEM. For on-line instruction, a single trajectory simulation is best, since it can be clearly seen on the video display (Figure 8). Alternatively a thin foil specimen can be used as shown in Figures 9a and 9b. In this case, the specimen is Cu with a thin layer of Bi segregated to the boundary, typical of equilibrium segregation phenomena. By placing the beam on the boundary, it is possible to calculate total electron path lengths through the Bi and through the Cu as shown in the printout in Figure 10. From this printout direct concentrations can be calculated. Such a simulation can easily be run throughout the duration of a class and the results compared with experimental data. Furthermore, in a laboratory class it is possible to simulate specific experimental variables that are difficult to carry out on the microscope such as

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Sample parameters:

- **Backscatter Fraction**: 0.3
- **Beam Angle (deg)**: 0
- **Beam Energy (KV)**: 25
- **Spec. Type**: Bulk
- **HOR. SCALE (NM)**: 9000
- **VERT. SCALE (NM)**: 6000
- **Total Path Length (NM)**: 194696.487
- **Backscattered**: 15
- **Histories**: 50
as the analysis of a totally buried precipitate in a thin foil such as shown in Figure 11. This is very difficult to do in a microscope but using the Monte Carlo simulation the precipitate size, chemistry and position in the foil can be varied at will and the results of the X-ray analysis will show the effects of these variables on any quantification procedure.

It should be noted that Monte Carlo simulations are no more than simulations. That is, they do not replace experiments and are only as good as the beam-specimen interaction information incorporated in the program. From a teaching standpoint however, they are most useful as dynamic displays of what is happening in the microscope. From the research standpoint, Monte Carlo simulations permit the student to simulate possible experiments, determine the major variables and sources of error, and most importantly can indicate whether a particular phenomenon is detectable and/or quantifiable, given the current level of instrumentation that is available.

Acknowledgements

The authors wish to acknowledge Graham Cliff of UMIST/Manchester University, Department of Metallurgy for his role in developing the Monte Carlo program while on leave at Lehigh University.

Discussion with Reviewers

K. Murata: Have you investigated systematically which can give us the best result among three quantification methods in an application to various mixed targets?
Authors: Experience with the three techniques of window, background modelling with Gaussian fitting has shown that in general the most flexible technique is the filter-fit. The window technique is tedious and of little value in the low energy range when the background is changing rapidly. The modelled background with Gaussian fitting works well, but has difficulty when severe peak overlap occurs. The digital filter with multiple least squares fitting is able to handle the low energy region and can deconvolute spectra which contain severely overlapped peaks, i.e., BaL and TiK.

K. Murata: You can save computational time to some extent with a fixed step length model, especially in an application to thick samples. Namely, with the model the step length and the energy at each step can be tabulated if the memory size is allowed. Is your model based on a fixed step length or a variable step length?
Authors: The Monte Carlo simulation is based on a variable step length in which the distance between each interaction is randomly distributed about the MFP (mean free path). The technique the reviewer suggests may only be useful if homogeneous samples are modelled. Any inhomogeneities (i.e., precipitate, boundary layer, etc.) would destroy any correlation between the step, step length and energy.

K. Murata: In trajectory plotting or X-ray generation plotting, it is hard to see how dense trajectories or dots are. With a colored display, it is much nicer to have a different color, depending on the density. Have you done this?
Authors: Color trajectory plotting is possible on the Apple, however it is difficult in practice to achieve the resolution that would be required.

K. Murata: Please give us a brief description about your Monte Carlo model.
Authors: For a fuller description of the particular Monte Carlo program we use as well as more details on the technique and its applications see the paper by J.R. Michael, G. Cliff and D.B. Williams in this volume.

J.C. Russ: We are using some rather similar programs (at North Carolina State University) as part of our teaching program, also running on an Apple computer. Some of the specific additional routines which we have found particularly useful are: a) To plot the effect of sample composition, as evidenced through the matrix mass absorption coefficient, on the spectrum. (This changes both the peak heights and the shape of the background.) b) To show the effect of surface orientation on relative elemental intensities, by entering a composition and plotting the intensities as a function of tilt or takeoff angle (taking into account the variations due to backscattering (R factor) and absorption (F(x))). c) Calculation of Phi(rho-z) curves using the Robinson & Brown model, to very rapidly illustrate the depth of analysis as a function of composition.

In addition, we find that a multiple scattering Monte-Carlo model, while admittedly less precise than the one the authors use, and inadequate for thin film analysis, is quite acceptable for bulk specimens and much faster than the quoted speed. In compiled Basic, we can generate about one thousand trajectories, and draw them on the screen, in 12-15 minutes. This gives adequate statistics for most purposes, and permits direct comparison to experiments using the SEM.

Authors: We agree that there are many more micro-computer routines that can be used in the teaching of electron microscopy and the two described in our paper were meant to be illustrative examples rather than a comprehensive review. Since we do a lot of thin specimen work at Lehigh we find that a multiple scattering Monte Carlo simulation is not ideal for teaching. However we note that D.C. Joy at Bell Laboratories, Murray Hill has developed a multiple scattering Monte Carlo program for thin specimens that runs on an Apple.

R.C. Farrow: How were the X-ray spectra obtained using the Apple IIe?
Authors: The X-ray spectra were first collected on a TN2000 MCA. The spectra were then transferred to the Apple using a communications interface. The data are transferred channel by channel in ASCI code which is then converted to numerical values and stored in an array.
Fig. 9a. Simulation of 2000 electron trajectories through a thin foil sample. The number of histories is required for good statistics, but a reasonable simulation using ~50-100 trajectories can be completed in a thin foil sample in ~10 mins., depending on the specimen (light elements are faster).

9b. The x-rays generated by the trajectories in Fig. 9a.

Fig. 10. Alphanumeric display showing the details from the simulation in Fig. 9. The specimen is Cu containing a grain boundary with a thin layer of Bi as the '2nd phase' at the boundary. Again the specimen and probe characteristics can easily be adjusted to simulate different experimental situations. It is also worth noting the very low backscatter fraction ($1.5 \times 10^{-3}$) from a thin foil sample compared with that from a bulk sample ($\sim 0.3$) in Figure 7.

Fig. 11a. Electron beam interactions with a rectangular Cu precipitate arbitrarily positioned near the bottom of a thin Al foil sample.

11b. The x-rays generated in each phase. From this type of simulation the number of x-rays generated in each phase can be determined and used directly for quantification.