THE TEACHING OF SCANNING ELECTRON MICROSCOPY AND ELECTRON PROBE MICROANALYSIS

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ABSTRACT

This paper discusses the objectives, syllabus, laboratories and teaching materials that are being used at Lehigh University for the teaching of scanning electron microscopy and electron probe microanalysis. Because of the similarities in the design and capabilities of the scanning electron microscope (SEM) and electron probe microanalyzer (EPMA) both instruments are discussed in the same course. The objectives of the Lehigh course in the SEM-EPMA are threefold: (a) to present fundamental material on such topics as electron optics, electron beam-sample interactions, and X-ray optics, (b) to present the practical material necessary for effective use of the instruments, and (c) to provide an opportunity to operate and use the instruments through laboratories and individual or group projects. Because of time limitations a heavy emphasis is placed on the discussion of fundamentals rather than on specific techniques or applications. The laboratories are used to illustrate many of the fundamental ideas developed in the lectures and in addition allow the students to operate the instrument(s). Specific projects allow the students a chance to tackle one problem in depth and to use the instrument(s) without specific directions.

KEY WORDS: Teaching, Scanning Electron Microscopy, Electron Probe Microanalyzer
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Introduction

A course on the scanning electron microscope (SEM) and electron probe microanalyzer (EPMA) has been taught to senior level undergraduates and graduate students at Lehigh University for the past seven years. This course is aimed primarily at students in geology and materials science and engineering but has often included interested biologists. Including laboratories, this course has been in a constant state of development over the past few years. With the subject matter rather well developed and a suitable textbook(s) becoming available, this course and others like it can be used as guides for courses to be developed at other universities. It is the purpose of this paper to outline the objectives, syllabus, laboratories and teaching materials that are being used at Lehigh University for the teaching of the SEM-EPMA.

Objectives and Purposes of a Course in the SEM-EPMA

It has been clear to this investigator for many years that the SEM and EPMA are, in reality, one and the same instrument and can be treated as such. Both instruments permit the characterization of heterogeneous materials and surfaces on a micrometer (µm) and submicrometer scale. The electron optics system and the signals produced during electron bombardment are very similar in the SEM and EPMA. The two instruments differ from each other primarily in the way in which they are utilized. Because of the similarities between the two instruments, the Lehigh course has been designed to discuss both the SEM and EPMA as one instrument except in specific cases.

The objectives and purpose of the Lehigh course in SEM-EPMA are threefold: (A) To present fundamental material on such topics as electron optics, electron beam sample interactions, and X-ray optics. This material is necessary for understanding the theory and practice of scanning electron microscopy-electron probe microanalysis. (B) To present the practical material needed to use the instrument effectively. Topics such as specimen preparation, quantitative X-ray analysis, and practical applications are presented. (C) To give "hands on" experience in using the two instruments through laboratories, and individual or group projects.

The presentation of fundamentals usually takes more than half of the lecture time available in the course. This material is very valuable not only for the effective use of the two instruments but also for the student's overall undergraduate or graduate education. In some cases, therefore, the coverage is broader than what would be necessary for SEM-EPMA applications alone. For example, the concepts of electron optics which are taught are particularly useful in a complementary course on the transmission electron microscope (TEM). In addition an attempt is made to provide the student with enough of the fundamentals that he can understand future developments in instrumentation, such as field emission guns, STEM, and Auger analysis.

Fundamental Topics

There are many difficult fundamental concepts which must be developed clearly and teaching these concepts is a challenge to the best instructors. One of these concepts is the relationship of the final electron probe diameter d to the incident beam current, I_B. As expressed by Pease and Mixon the maximum incident beam current is given by:

$$I_B = \frac{1.26}{(0.51)\frac{d^{6/3}}{C_6^{2/3}}\lambda^2 - 1}\times10^{-10}$$

where \(J_e\) = emission current density (A/cm²) of the filament, \(T\) = emission temperature of the filament (°K), \(C_6\) = spherical aberration coefficient of the final lens (cm), \(\lambda\) = wavelength of the electrons (cm), \(d\) = electron probe diameter (cm). The interrelational of the aperture angle, lens aberrations and the brightness of the electron source to \(I_B\) and \(d\) must also be thoroughly explained. This also provides a theoretical basis for inherent limitations in the instrument. With an understanding of this concept, the ideas of spatial resolution and X-ray resolution limits can be discussed and possible improvements in SEM-EPMA design can be outlined.

Another fundamental area of great importance is that of electron beam sample interactions. It is very important for the student to have an understanding of just how all the signals (secondary electrons, backscattered electrons, characteristic and continuum X-rays, cathodoluminescence, Auger electrons, etc.) are developed. In addition the spatial resolution of each signal can only be described by having an understanding of the nature of the signal itself (Figure 1). The student can then understand for example why X-ray spatial resolution is dependent mainly on incident beam voltage \(E_0\), X-ray line measured, and material characteristics, or why secondary electron spatial resolution is dependent on the final electron probe diameter \(d\) and contributions by backscattered electrons.

Probably the most difficult fundamental concept for the student to grasp is the relationship between contrast \(C\), the variation of signal from point to point in a scanning image, and the thre-
hold or minimum beam current, $i_{th}$, necessary to detect the level of contrast inherent in the image. Such a relationship has been discussed by Joy. It can be shown that for a high quality image with $10^5$ picture elements, the threshold current is given by:

$$i_{th} > 1.6 	imes 10^{-11} \text{Ampere}$$

(2)

The term $t_f$ is the frame or scan time for the image. This equation sets the fundamental limitation on the beam characteristics for successful operation. If the contrast in the signal is low, for example in electron channeling, $t_f$ must be high for the image to be visible. A high beam current will increase the beam diameter (Equation 1) and necessarily decrease the spatial resolution of the signal. Therefore what is most important for the student to understand is that our ability to resolve fine detail in a picture is often limited by the noise in the signal and not by the size of the beam diameter alone. In many cases this concept can be best illustrated by laboratory experiments, as discussed in a later section.

X-rays are used for chemical characterization in both the SEM and EPMA. Besides understanding the basic concepts of X-ray production and spatial resolution, the student must have a fundamental understanding of X-ray optics. The differences between the two methods of measuring X-rays, energy dispersive analysis and wavelength dispersive analysis should be clearly established. It is often convenient to contrast the performance of the two systems by discussing the process by which the proportional detector and the Si(Li) solid state detector operate. With this background it is possible to describe the use of the two X-ray optical systems on a basis of inherent or design strengths and weaknesses.

**Practical Topics**

The presentation of the practical material, needed to use the SEM-EPMA instrument effectively, takes up the balance of the lecture time. Specific applications are used to illustrate the various modes of instrument operation (X-ray-scanning and element identification, secondary electron topography and electron channeling, etc.). Specific discussions of specimen preparation techniques are of great importance. For biologists these discussions are absolutely necessary as specimen preparation critically affects the results obtained. The problems of specimen preparation for X-ray analysis must also be discussed since it is important to know the X-ray emergence angle as well as to have a suitably prepared area free of contamination and/or other polishing artifacts ready so that accurate analysis can be performed. Specimen coatings which prevent charging and enhance secondary electron production are also necessary discussion topics.

One of the most practical, yet sophisticated areas that should be discussed is that of quantitative X-ray analysis. Measurement of chemical composition in micron sized areas of specimens in the EPMA has been performed using wavelength spectrometers for some years. With the development of the energy dispersive detector, however, quantitative analyses can also be obtained using the SEM after suitable corrections are made for the relatively large continuum background and possible characteristic peak overlap. At this point the usual ZAF (Z-atomic number, A, absorption, F-fluorescence) or empirical techniques can be applied to the data to obtain chemical composition. The presentation of quantitative X-ray techniques should be considered as a necessary component of any course in scanning electron microscopy. Other practical X-ray techniques such as light element analysis, trace element determination, and thin film measurements may also be mentioned.

**Instructor Experience**

The third objective of a course in the SEM-EPMA is to give the student some actual experience in using the two instruments. This objective is accomplished in our course by having the students use our SEM and EPMA in the laboratory. The laboratories will be discussed in a later section.

**Syllabus of the Lehigh SEM-EPMA Course**

Experience has shown that a one semester, 13-14 week course with 2 lectures and 1 lab per week allows enough time to carry out the 3 major objectives of the course as outlined above. The list of lecture topics given in Table 1 indicates the material covered in each of the lectures in the Lehigh course entitled Electron Metallography (Net. 334). Several exams are also given during lecture times and are used to evaluate the student's progress. On several occasions we have been constrained to a 7 week time period so that a TEM course could be taught to the same students in the remaining 7 weeks. In this situation the amount of material covered in lecture...
is decreased, leaving out most of the lectures on applications, the discussion of quantitative analysis and also reducing the number of lectures on electron optics and electron beam specimen interactions. It is suggested that a half semester course in SEM-EPMA is too short for fully achieving the 3 objectives of the course.

Table I

LECTURE TOPICS IN SEM-EPMA
(Electron Metallography, Lehigh Met. 334)
1. Introduction to the SEM and EPMA
2. Electron Optics, Electron Guns (tungsten filament, LaB6, field emission)
3. Electron Optics, Electron Lenses (minimum spot size, aberrations)
4. Electron Optics, Relationship of Final Electron Probe Diameter d vs. Incident Beam Current I
5. High Resolution Scanning Electron Microscopy and Depth of Field
6. Electron Beam Specimen Interactions, Electron Scattering and Range
7. Electron Beam Specimen Interactions, Emitted Electrons (backscattered electrons, low energy electrons)
8. Electron Beam Specimen Interactions, X-ray Production, X-ray absorption
9. Electron Beam Specimen Interactions, Spatial Resolution of Emitted Signals
10. SEM Imaging Process and Signal Detectors
11. SEM Contrast Formation and Image Quality
12. Resolution Limitations in the SEM (signal, specimen)
13. Signal Processing of SEM Images
14. Special Contrast Mechanisms (electron channeling, magnetic contrast, etc.)
15. Specimen Preparation and Applications of the SEM
16. X-ray Crystal Spectrometers
17. X-ray Solid State (energy dispersive) Detectors
18. Comparison of Crystal Spectrometers and Solid State Detectors
19. X-ray Scanning
20. Quantitative X-ray Analysis, ZAF Technique
21. Quantitative X-ray Analysis, ZAF Technique
22. Quantitative X-ray Analysis, Empirical Technique
23. Quantitative X-ray Analysis, Computational Techniques
24. Specimen Preparation and Applications of the EPMA
25. Special X-ray Techniques (light element analysis, thin film measurements, etc.

It is evident from Table I that a heavy emphasis has been placed on the discussion of fundamentals. Experience has shown that our senior undergraduate and graduate students will master specific techniques suited to their research needs without direct instruction in class. Therefore in many cases major concepts have been emphasized rather than specific techniques or applications. This emphasis therefore required elimination of detailed lectures about several SEM and EPMA techniques (voltage contrast, low loss images, trace element analysis, cathodoluminescence, etc.). Specific reference material describing these techniques is available as noted in the next section.

Textbooks and Other Teaching Materials

Up to this year it has been very difficult to obtain a suitable textbook for our course on the SEM and EPMA. Different texts6,7 have been used as the major reference for the course. However, these texts covered less than 50% of the lecture material. For the Lehigh course we have taken material from various technical articles and have given reading assignments from over half a dozen texts or articles. Last year (spring semester, 1974) reading was assigned from Oatley,8 Everhart and BAY9s, Pease and Nixon,10 Heale et al.10 and Andersen11 for electron optics and SEM imaging, contrast formation and applications. A new book by Wills,12 which is an advanced treatise on the SEM will also complement these texts. Reading assignments on X-ray optics and microanalysis were taken from Andersen,7 Poole and Martin12 and Beaman and Zaeh13. Reading material on the topic of electron beam/specimen interactions was made available in a set of notes developed by the instructor and incorporated in a new text14.

Laboratory Experiments and Homework Assignments

The laboratory experiments have changed from year to year particularly due to the student registration which has varied from a low of 18 to a high of 42. Since it is impractical to run a laboratory on the SEM or EPMA with more than 6 students, the content and number of laboratories have had to be shifted from year to year. Last year 6 laboratories were run and the students completed a cooperative project. A list of the 6 laboratory topics and the titles of the cooperative projects are given in Table II.
Laboratory Topics

I. Introduction to the Lehigh SEM and EPMA
II. SEM operation, secondary and backscatter modes
III. Resolution, depth of field and minimum beam currents in the SEM
IV. EPMA operation, sample current and X-ray modes
V. X-ray detection and X-ray scanning modes
VI. Quantitative X-ray analysis

Projects

1. Characteristics of gold films deposited by pulsed wave D.C. current
2. Comparative study of sensitization in a stainless steel
3. Study of the fracture surfaces of various plastic materials
4. Metallographic studies of P-21 mold steel in the annealed, quenched and heat treated conditions
5. SEM and EPMA study of a Ni-Nb-Al directionally solidified ternary alloy
6. Distribution of elements in the region of a weldment in a cast iron valve

The purpose of Laboratory I was to demonstrate how the SEM and EPMA work and to introduce the student to the types of instrumentation under study. Laboratory II allowed the student to gain "hands on" experience operating our ETEC-SEM. Copies of the operation manual were passed out and the student, with individual assistance, was able to run the SEM and examine a typical fracture sample in secondary electron and backscatter electron modes. The third laboratory demonstrated the importance of resolution, minimum beam current and depth of field on SEM images. One experiment involved varying the beam current (10^{-9} to 5 \times 10^{-14} A) on a high resolution (high contrast) sample and noting the maximum magnification at which structural details could still be resolved. A comparison of these results to the calculated relationship of beam current to beam diameter (Equation 1) was also made. Another experiment involved viewing a high contrast sample at varying beam currents at T.V. scan rates. The student was asked to compare the variation of the quality of the T.V. picture with measured specimen current and magnification. The concept of threshold current, \( t_{th} \), as given in Equation 2, is well illustrated by this experiment. The variation of depth of field with divergence angle and magnification was also investigated by varying the working distance and then the magnification. The trade-off between depth of field and high resolution was illustrated by this experiment.

Laboratory IV allowed the student to have "hands on" the Lehigh ARL-EPMA. The student familiarized himself with the operation of the instrument, especially with techniques for beam saturation, focus and measurement of X-ray intensity using the wavelength dispersive spectrometer. Laboratory V illustrated the use of the wavelength and energy dispersive detectors. An unknown sample was given to the students and they were responsible for using the two X-ray techniques to determine the elements present in the phase(s) of the unknown. X-ray scanning displays of the sample were obtained by using both techniques and the results were compared for the two X-ray optical systems. The EPMA was used in Laboratory VI to obtain quantitative chemical analyses of micron sized areas on an unknown sample. A calibration curve of X-ray intensity ratio vs. composition was developed first using alloys of known composition. A ZAF correction for one of the standard alloys was calculated by hand to illustrate the use of the quantitative analysis technique. These 6 laboratories therefore illustrate many of the fundamental ideas developed in the lectures and in addition allow the students to become familiar with the operation of the SEM and EPMA.

The 6 projects chosen by the 18 students in the Spring 1974 class and listed in Table II, provided the students with an opportunity to tackle at least one problem in depth. Each laboratory group spent up to 10 hours on one or both of the instruments examining their material. In most cases the students ran the instruments themselves and were gently guided by the instructor through the usual difficulties experienced in doing a given project in a limited time period.

Most of the homework given to the students was either a specific reading assignment or a problem associated with or assigned to the laboratory write-ups. Homework problems on X-ray production, absorption and measurement as well as electron optics were assigned to insure that the student understood the concepts taught in lecture. The problem dealing with electron optics is illustrated in Figure 2 where the ray diagram of a two lens SEM-EPMA system is given. The student is asked to determine the final probe diameter, \( d \), and current in the final spot for various combinations of condenser lens focal lengths, \( f' \), objective aperture sizes and for given values of \( C_{v} \) for the final lens and brightness of the electron gun. This problem allows the student the opportunity to see how changes in various parameters in the operation of the SEM-EPMA affect resolution and emitted signal intensity.
Summary

A description of the objectives, syllabus, textbook, laboratory experiments and homework of the Lehigh University course on the SEM and EPMA has been given. The similarity of these two instruments allows for a discussion of both instruments in the same course. The Lehigh course has been developed over the last 7 years and can serve as a guide to setting up similar courses at other universities. With the development of a new textbook on the subject it should be much easier now to develop a senior undergraduate-graduate course dealing with the Scanning Electron Microscope and Electron Probe Microanalyzer.

References

DISCUSSION WITH REVIEWERS

Reviewer IV: What level of sophistication in math and physics do you assume the students have? What prerequisites do you list? Do you allow biologists, who in my experience are deficient in math and physics, to take your course?

Author: It is assumed that the student has a normal undergraduate background in math and physics, 1½ years of both. The SEM course is a 300-level course (upper undergraduate) and no prerequisites, other than permission of the instructor, are required. Biologists have taken our course and have done quite well.

Reviewer I: What do you expect the student to be capable of doing at the end of the course?

Author: With respect to the operation of the SEM instrument, I only expect the student to be able to run the instrument so that he can obtain secondary electron images from rather high contrast samples. More important than this accomplishment the student should have a good feel for the various modes of operation and how to vary instrumental parameters (operating voltage, tilt, signal mode, dark level, etc.) to obtain the best data possible.

Reviewer I: Please provide details of instrument time scheduling to provide students with adequate 'hands-on' experience.

Author: Each student is scheduled for at least 1 hour of 'hands-on' experience early in the course. A technician or the instructor is responsible for scheduling. Other 'hands-on' experience is obtained in other lab sessions and/or during the laboratory cooperative project.

Reviewers II, III & IV: Would you indicate the number of sections and the number of teaching assistants necessary? What was the actual number of hours per week spent by each student engaged in the laboratory topics? Do you find that this time conflicts with your research projects at Lehigh?

Author: Three to six sections of laboratories are required. Usually there are 5-6 students in each lab session. There are 2 technicians and 2 instructors available for instruction in the laboratories and lab project sessions. The students spend 3 hours in each of six laboratory periods. In addition up to 10 hours are spent on the cooperative project. The time used for laboratories does conflict with some of our research project work. However, this conflict is only particularly bad for 3 weeks on each instrument, SEM and EPMA, out of the year.

Reviewer I: What procedures and techniques do you test for in a comprehensive laboratory examination?

Author: No laboratory examination is given. The student is asked to write a short description of each lab and to answer a set of questions given on each lab handout sheet.

Reviewer V: What changes would you make in your approach if you had to teach biology students also?

Author: If biology students were present in our course, lectures on biological specimen preparation and interpretation would be included in the syllabus. Several practical laboratories would also have to be included. These additions to the course would be handled with the active participation of the pertinent biology faculty.

Reviewer IV: What do you suggest that the student can do with regard to the high costs of textbooks in areas such as the SEM-EPMA?

Author: I can suggest the following: 1) If the student is taking a course in electron metallography for general interest or to learn something about electron optical instruments he can avoid buying a text altogether. In our course we keep several copies of the text and required reading on reserve in the library. 2) If the student is going to use the instrument seriously in his research, he should be willing to part with his money; the textbook(s) will definitely be useful as a reference. In this case the investment is worth it. 3) The SEM-EPMA laboratory should have a small library associated with it. At Lehigh all the SEM-IITRI Proceedings and the International Electron Probe Conference Proceedings are available for the student to use in the library. Finally 4) the instructors in courses on the SEM should pressure the publishers to develop a paperback edition of the most useful textbooks so that the students can afford to buy the texts.

Reviewer III: Please comment on the more general chemical characterization relating to chemical bonds and molecular structure that might be available to SEM techniques?

Author: Soft X-ray emission spectra can be used to determine the structural role of elements in complex materials. These spectra can be obtained by using wavelength dispersive X-ray optics. Suitable X-ray optics can be installed in a SEM.

Reviewer III: Would you give the definition and the units for the term contrast C, as used in your paper?

Author: Contrast, C, is defined as: \( C = \Delta s/s \) where \( \Delta s \) is the change in the signal between any two points in the image, and s is the average signal. It is expressed as a fraction or percent (no units).
Additional discussion of paper on "Routine Use of SEM and EPMA in Forensic Science," by R.H. Keeley and M. Robeson, continued from p. 486.

Reviewer I: Can you provide examples of SEM use in forgeries?

Authors: See Fig. 9. During the investigation of a massive forgery of 10 p National Savings stamps, tins of colored printing inks were found on premises occupied by a suspect. It was necessary to quickly provide evidence linking the ink with the forged stamps, in order to prevent possible destruction of incriminating material by the suspect. Good matches between the four colors on the stamps and four of the suspect inks were rapidly obtained using microanalysis. In addition a small fragment of charred paper bearing perforations similar to both genuine and forged stamps was found. Analysis of the filler indicated that the sample was from a forged stamp. After questioning of the suspect the press, plates, perforator and paper supply were recovered.

Fig. 9. E.D. Spectra of green printing ink from forged stamp and ink found in suspect's possession.