

3-5. Cross section of the volume of primary excitation illustrating zones from which signals may be detected.

nucleus of a specimen atom scatters electrons, the excess energy is released in the form of x-rays of widely varying wavelengths. This radiation is referred to as "white" or "Bremstrahlung radiation" or as the x-ray continuum. It is not characteristic of specimen composition, and therefore cannot be used to accumulate information. It becomes part of the background in the measurement of characteristic x-rays.

Thus far, the causes have been mentioned for the generation of six of the seven useful signals illustrated in Figure 3-1. The seventh signal, specimen current, is the result of the negative charge of electrons, both those incident to and emitted by the specimen. If the current present in the backscattered group is subtracted from that of the incident electrons, the result is called the "absorbed current." However, a specimen current meter would read a somewhat lower figure, due to the loss of current as secondary electrons and Auger electrons. In the case of thin specimens where beam electrons are transmitted through the sample, their magnitude would also be a debit to specimen current. Thus, it is clear that specimen current is dependent on the extent of beam-specimen interaction in producing backscattered, secondary, Auger and transmitted electrons, as well as on changes in current of the incident beam. A summary of events mentioned to this point is given in Table 3-1.

Of the seven signals described above, six will be covered in more detail in the following sections. The generation, range, and resolution of the secondary electron signal, discussed in Chapter 2, will be omitted here.

EVENT	BETWEEN	SIGNAL	RESOLUTION	USE
Elastic	Beam electrons / specimen nuclei or electrons	Backscattered electrons	> 1 micron 1,000 nm	Atomic number contrast Topographical contrast
Inelastic scattering	Beam electrons or backscattered electrons / specimen electrons	Secondary electrons	10 nm or better	Surface structure
Recombination	Free electrons generated / positive holes	Cathodoluminescence	< 80 nm	Impurity concentration
		Characteristic x-rays	Measured in eV's, an integral of peak width of a given line	Elemental composition
Transmission	Electrons through specimen	Auger electrons	Measured in eV's, an integral of peak width of a given line	Surface composition
		Transmitted electrons	5 nm or better	Internal ultrastructure
Energy transfer	Specimen / ground	Specimen current	poorer than 2° electron image	Atomic number contrast
Inelastic scattering	Beam electrons or back-scattered electrons / specimen nuclei	Continuum x-rays		Not a useful signal

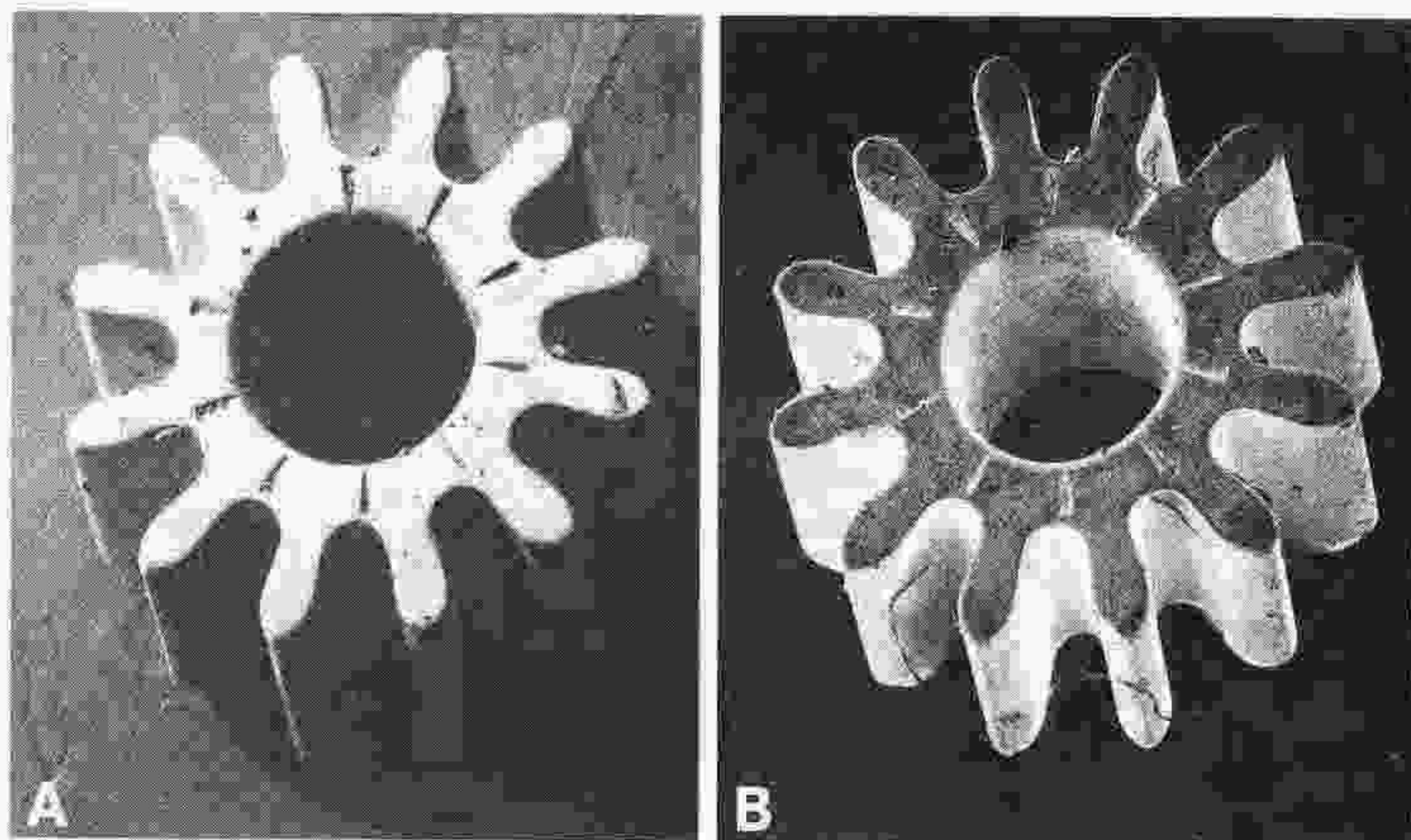
Table 3-1

THE BACKSCATTERED ELECTRON SIGNAL

Backscattered electrons are those electrons which have undergone single or multiple scattering events (36,37) and escape back through the surface of the specimen with energy greater than 50 eV. This definition corresponds with a widely accepted convention of bracketing those electrons escaping the surface with energies of 50 eV or less as secondary electrons. The backscattered group, in terms of this broad definition, will contain electrons with a wide range of energies; however, they are thought to possess on the average about 80 percent of their original energy (2). In other words, a primary electron beam of 25 KeV could generate a backscattered signal of 20 KeV.

Backscattered electrons possessing energies near those of the incident beam behave in a fashion similar to beam electrons. They may subsequently interact with atoms of the specimen to produce secondary electrons, x-rays, cathodoluminescence, and Auger electrons. Backscattered electrons travel in straight lines, being impervious to attraction by the low voltage secondary electron collector (Fig. 2-20 and 3-6). As depicted in Figure 3-2, backscattering increases as the atomic number of the specimen increases. In fact, backscattered electron production shows such a strong correlation with atomic number, that this relationship has become the basis for a contrast mode in scanning electron microscopy. If the atomic numbers of two phases in a specimen differ by more than three, those phases may be distinguished in the backscattered electron image. The element of higher atomic number will show stronger emission, resulting in lighter (brighter) areas on the screen (9). A graphical comparison of this relationship has been plotted (9) and can be seen in the generalized diagram in Figure 3-7. It should be noted that secondary electron generation also generally increases with higher specimen atomic number.

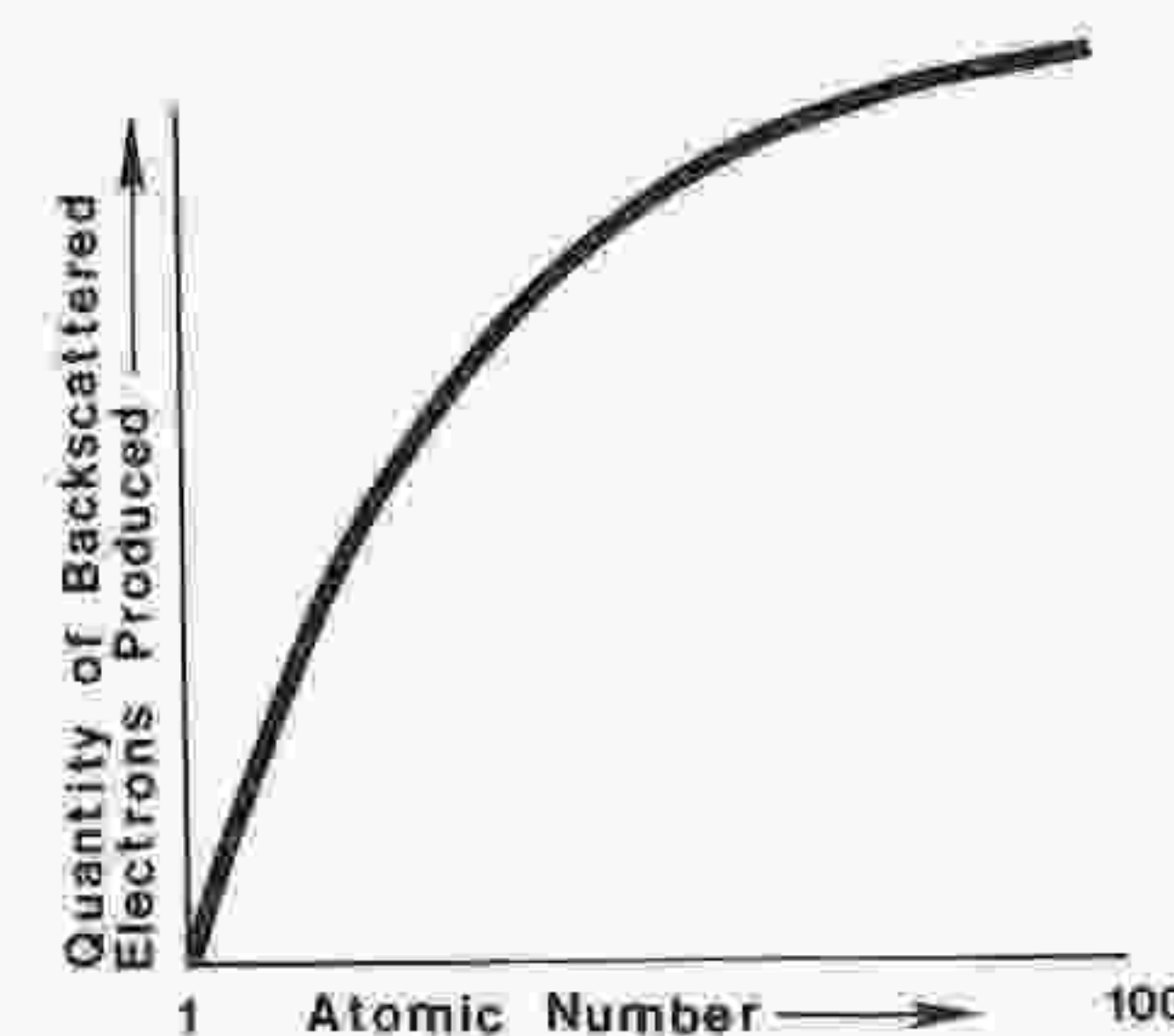
Due to their straight line paths of travel, the detected backscattered elec-



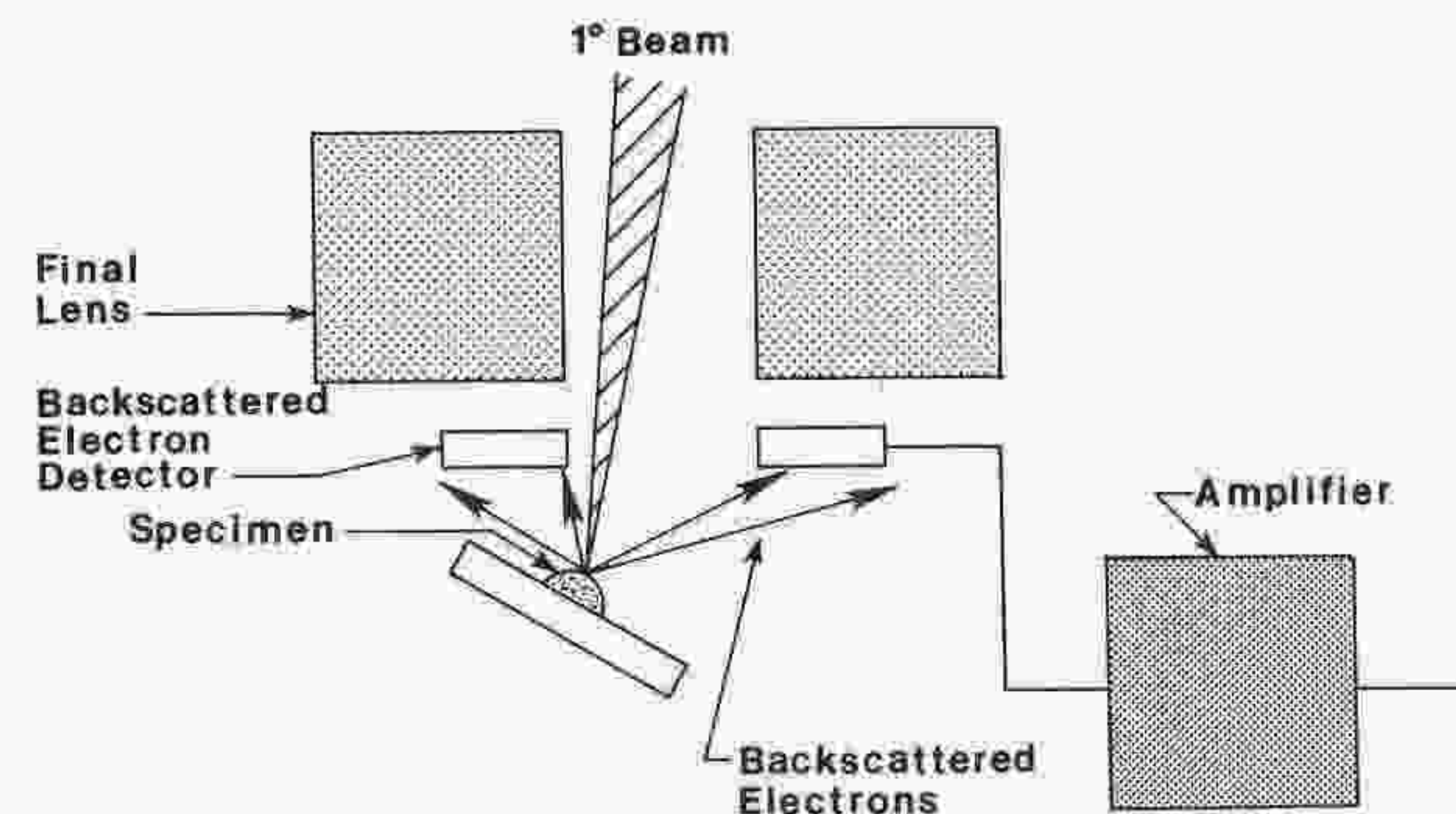
3-6. Comparison of (A) a backscattered electron image to (B) a secondary electron image.

tron signal is weaker than the secondary electron signal. This signal's weakness is demonstrated by the ability to collect a strong secondary electron signal in spite of the constant noise contribution of the backscattered electrons because only those backscattered electrons directly in line with the detector will be imaged. In order to use the backscattered signal in image formation, the secondary electron signal must be prevented from reaching the detector. To accomplish this objective, the positive detector bias is disconnected. Some microscopes have the ability to apply a slightly negative bias to the detector to repel secondary electrons. Other techniques employ modified scintillator detectors (23,32,38) or other accessories (26,30) as seen in Figure 3-8.

The area of excitation from which backscattered electrons are emitted is at least ten times larger than that of secondary electrons (Fig. 3-5). If beam current is increased for a stronger signal, resolution of backscattered electrons will differ from that of secondary electrons by more than one order of



3-7. Diagram demonstrating the yield of backscattered electrons produced in relation to the atomic number.

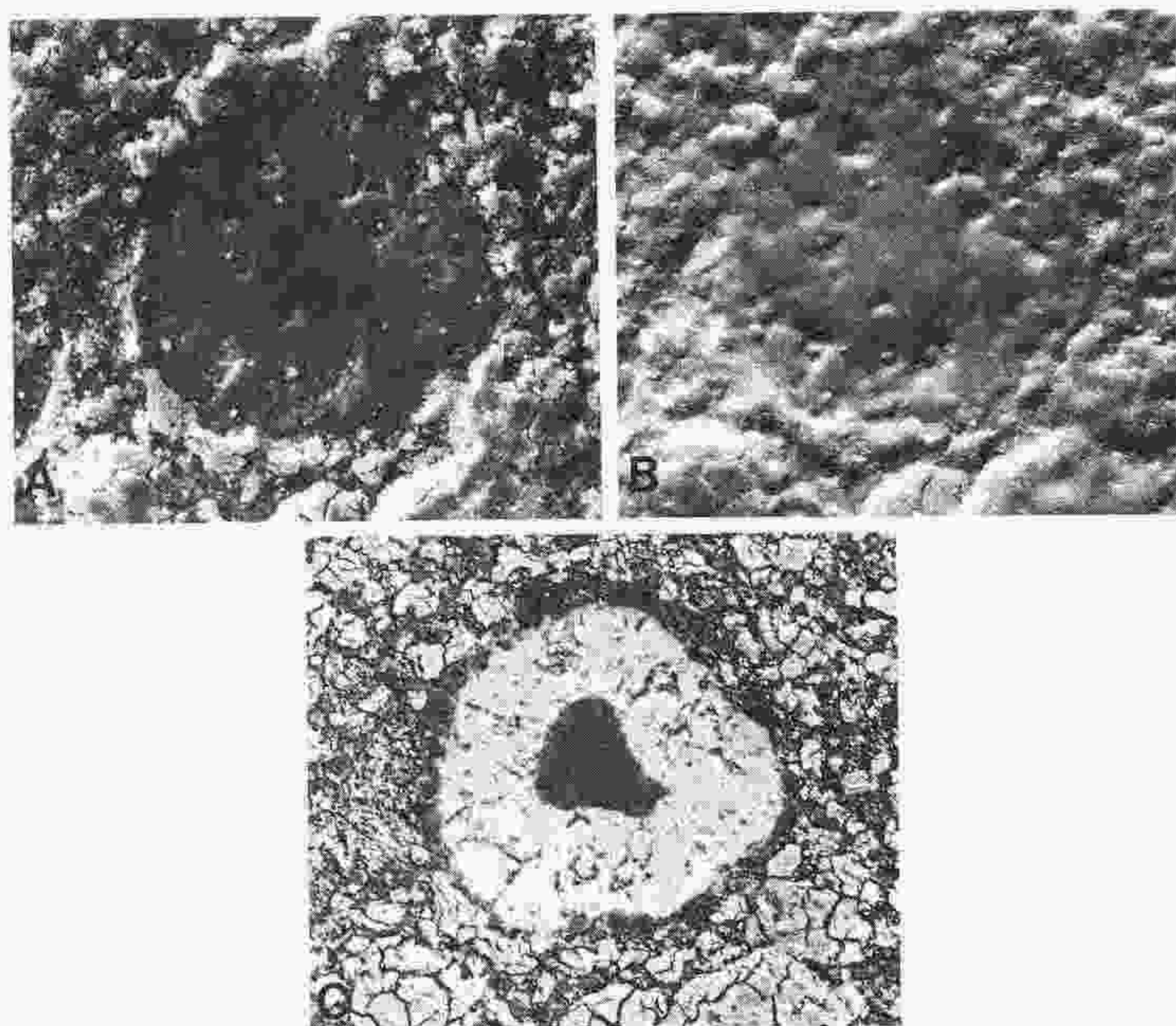


3-8. Block diagram of one type of backscattered electron detector system.

magnitude. Despite the lower spatial resolution, the backscattered mode has been useful for several applications. One is atomic number contrast. Another is topographical contrast, which maximizes the effect of the straight line paths of these electrons. Instead of imaging around corners as secondary electrons permit, the backscattered electrons may be obscured from the detector by a topographical irregularity (Fig. 3-6). This results in interesting shadowing effects. Relatively smooth specimens are particularly suited for this imaging mode, since slight topographical irregularities appear more distinct due to shadowing (Fig. 3-9).

CHARACTERISTIC X-RAYS

Characteristic x-ray emission is one process by which an atom may stabilize itself following ionization by the electron beam. When an electron from an inner atomic shell has been dislodged by the beam, an electron from an outer shell will fill the vacancy. The difference in energies between the initial and final states of the transitional electron may be emitted as x-radiation (22). The various shells of an atom have discrete amounts of energy. It follows that their

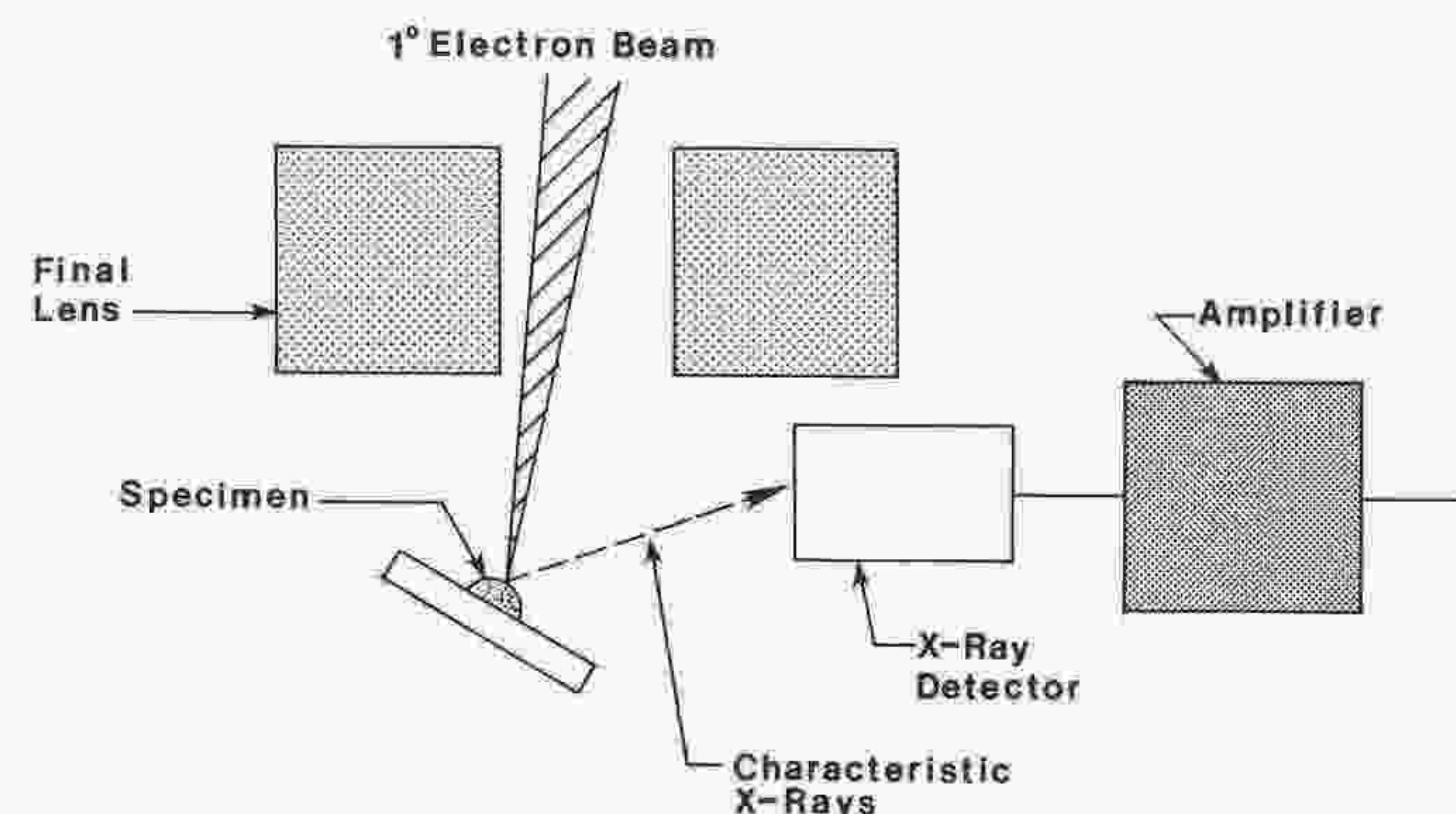


3-9. Backscattered electron detection of the polished surface of dolomite. (A) Secondary electron image. (B) Backscattered electron image (topography). (C) Backscattered electron image (composition) (Micrographs courtesy of JEOL.).

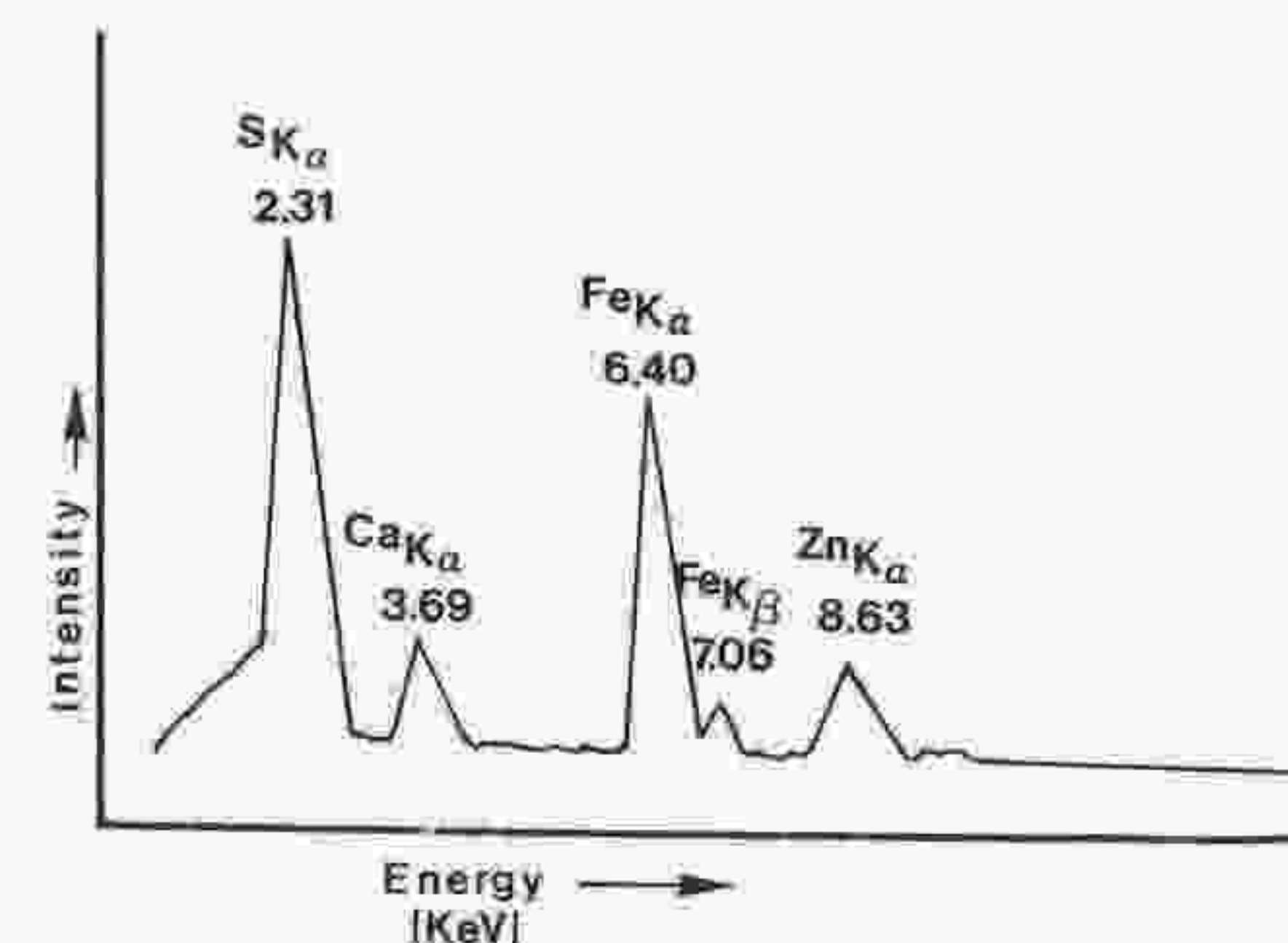
energy difference, emitted as x-radiation, is also a discrete quantity and is characteristic of the atom from which it was released (this mode of the scanning electron microscope is covered more completely in Chapter 4).

X-ray spectroscopy in the SEM involves the identification of radiation of specific wavelength or energy for elemental analysis of the specimen. This analysis requires a special detector which may be one of two types. A detector system which separates x-rays according to wavelength is a wavelength dispersive spectrometer. One which measures the energies of the x-radiation is termed an energy dispersive spectrometer (Fig. 3-10).

The set of peaks which rises above the Bremsstrahlung radiation (continuum) during measurement of x-ray energies or wavelengths of a given element is the spectrum of that element (Fig. 3-11). For example, an electron falling from the



3-10. Block diagram of the components of an x-ray analysis system in the scanning electron microscope.



3-11. Characteristic x-ray spectrum obtained with an energy dispersive spectrometer. The specimen analyzed contains sulphur, calcium, iron and zinc.

L to K shell generates $K\alpha$ radiation specific for a given element; the transition from M to K gives $K\beta$, and so on.

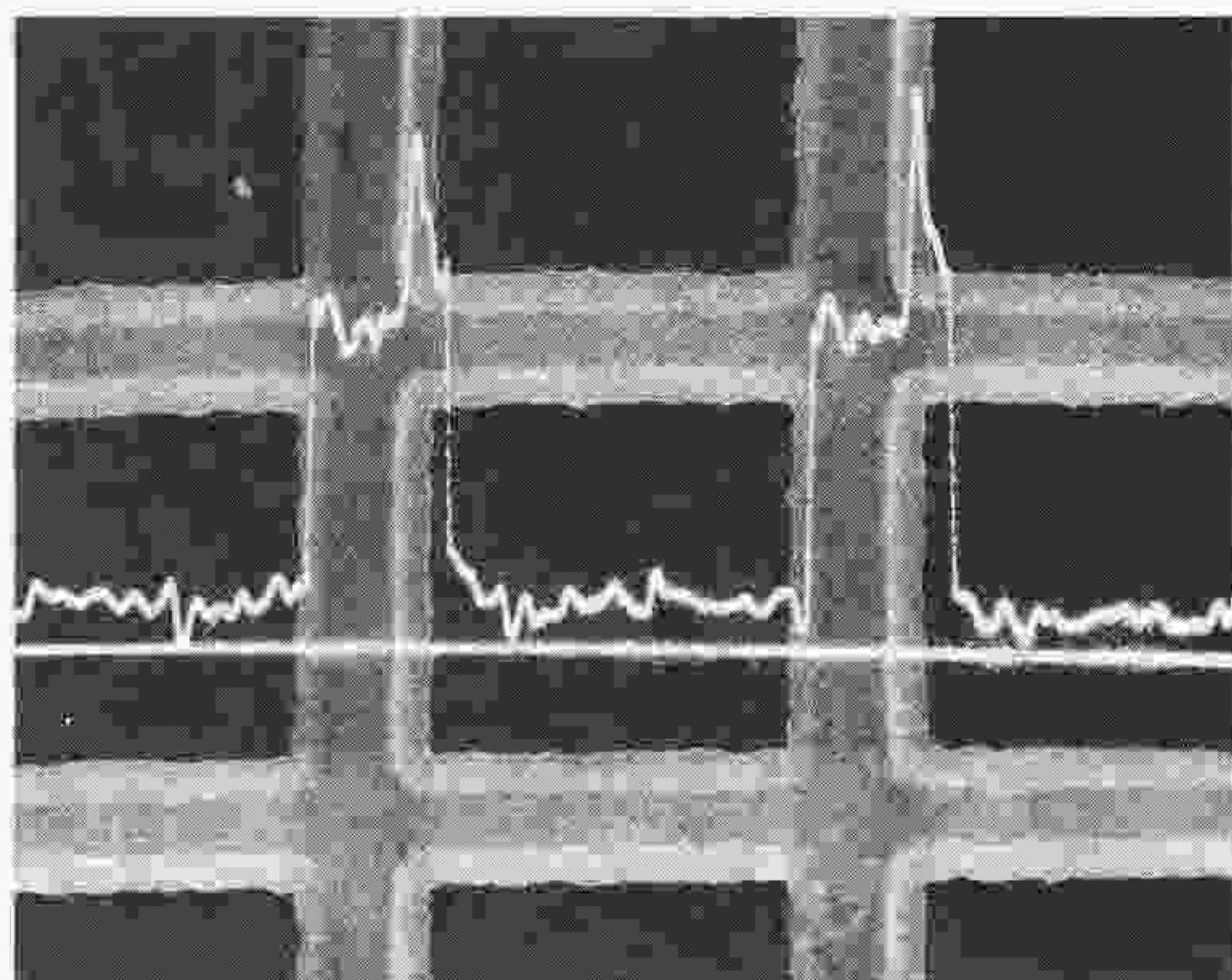
Information concerning the distribution of a given element may be obtained by allowing the x-ray signal from that element to modulate the Y axis of the cathode ray tube. Modulation of the Y axis as the beam makes a single sweep across the region of analysis in the X direction results in a line profile (Fig. 3-12). The vertical (Y) displacement corresponds to the intensity of x-rays from the selected element. Modulation of CRT brightness by the x-ray signal results in an x-ray map of the distribution of the chosen element as the beam travels through a raster pattern over the region of analysis (Fig. 3-13).

Spatial resolution of the x-ray signal from the specimen surface is determined by a number of factors. Characteristic x-rays are detectable from a greater specimen volume than that of the primary excitation, which greatly decreases spatial resolution of x-rays as compared to secondary electrons. The atomic numbers of elements in the specimen are also important in resolution, since a high atomic number element is more electron dense and yields more x-rays for a given voltage. The effect on resolution of these and other factors, such as accelerating potential and specimen topography, are discussed in Chapter 4.

Spectral resolution (measured in electron volts, eV) is the ability of the instrument to separate wavelengths or energies that are near each other in magnitude. This property is largely dependent on the detector itself, and is represented by the width of a specific peak generated in the SEM by a specimen of known composition at standard conditions.

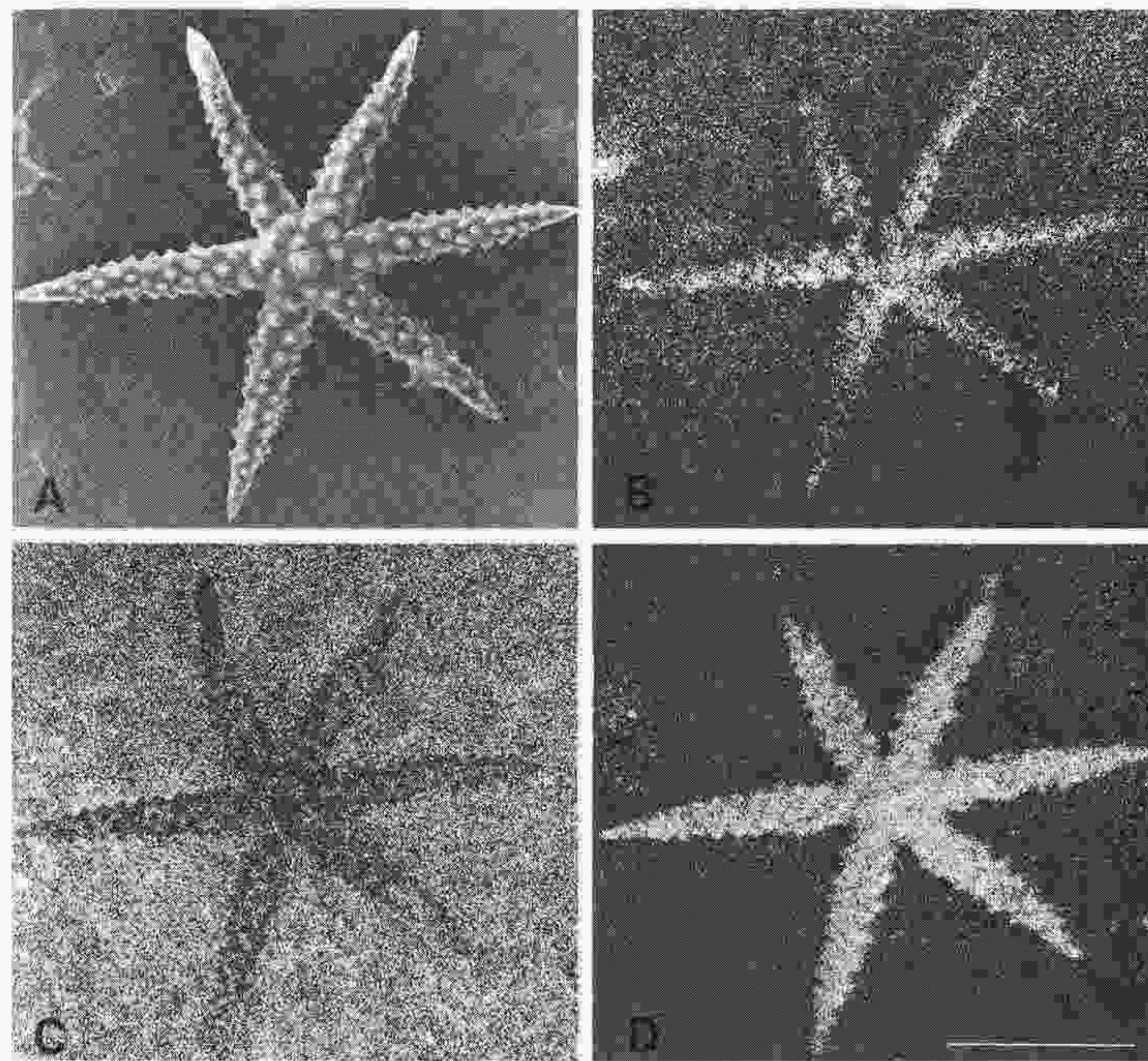
AUGER ELECTRONS

When an outer shell electron fills an inner shell hole caused by primary or backscattered electron excitation, small quantities of the excess energy



3-12. X-ray line profile of a gold grid on an aluminum stub demonstrating a line scan for gold.

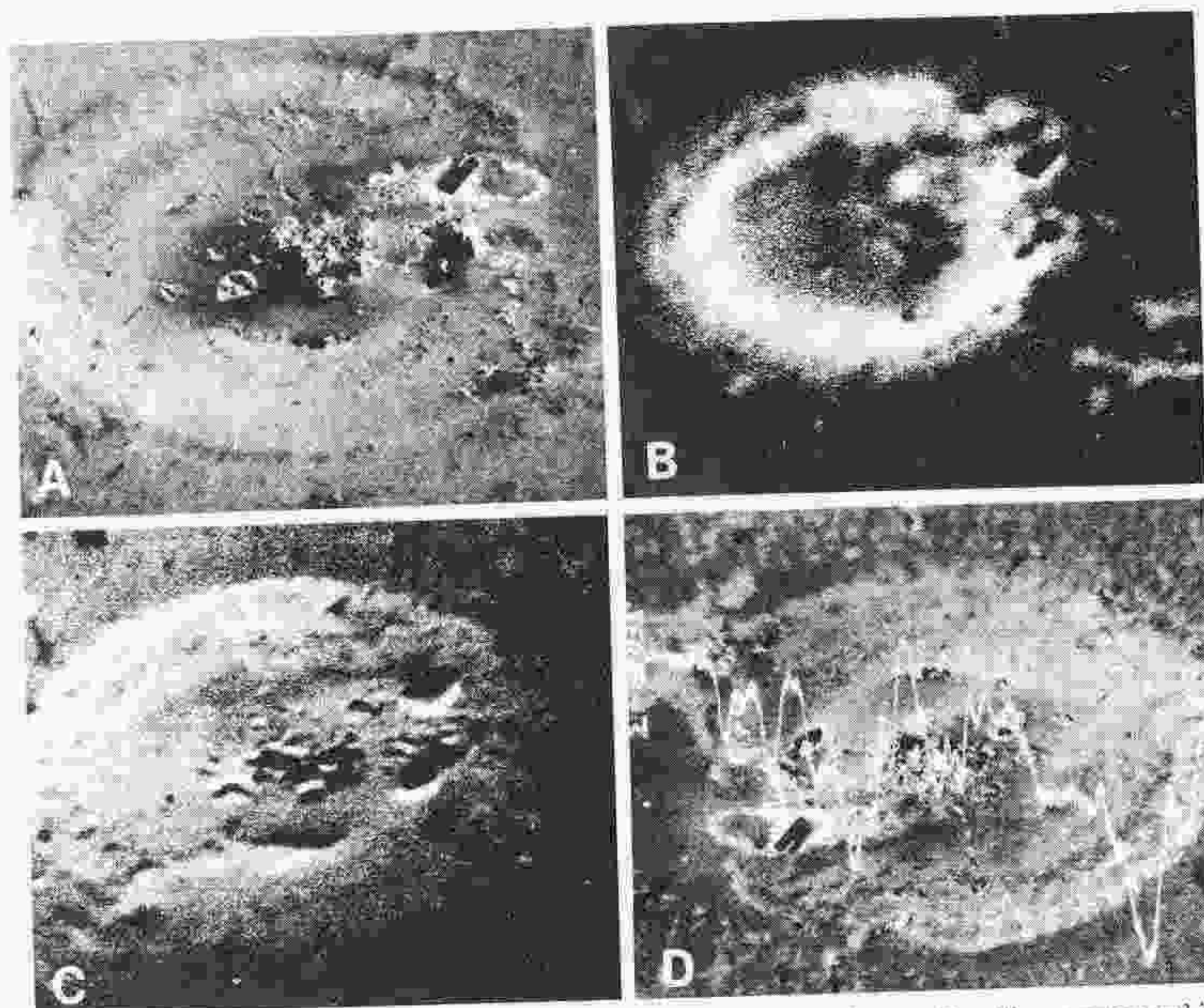
available may be carried away through the emission of a low energy Auger electron. The Auger electron has energy approximately equal to the difference between the two shells involved in the transition. This electron energy is characteristic of the atom from which it was released, and can be used in chemical analysis of the specimen surface. Two instances when Auger Electron Spectroscopy (AES) might be preferred over x-ray spectroscopy are in studies of specimens of low atomic number and in surface studies involving only the top few layers of the specimen. Due to the low energies of Auger electrons, they have an escape depth of about 0.5 to 2 nm (19) which accounts for their giving accurate information of monolayers near the surface. The Auger yield is higher in elements of low atomic number because the electrons are less tightly bound to the nucleus. These characteristics are quite different from those of x-rays, as x-rays are transmitted from a volume greater than that of secondary electrons, and their production increases with increasing atomic number. Further theoretical considerations on AES can be found in a review by Venables and Janssen, 1978 (34).



3-13. Characteristic x-ray detection of mineral deposits in a trichome on a *Deutzia* leaf. (A) Secondary electron image. (B) Magnesium $K\alpha$ image. (C) Potassium $K\alpha$ image. (D) Calcium $K\alpha$ image. Line scale is equal to 50 μm (Courtesy of JEOL, Ltd.).

Chemical analysis in this mode is based on analysis of secondary electron energies. Secondary electrons that have undergone Auger transitions produce peaks in the electron energy distribution curve (20). The resulting spectrum gives a series of peaks, or families, for each element present in detectable amounts at the specimen surface. Like x-rays, the signal of a selected element may be used to modulate the Y axis and brightness of a CRT to produce line profiles and maps (16,21,33, and Fig. 3-14).

The best use of Auger electron spectroscopy in surface studies is not accomplished in a scanning electron microscope equipped with an oil diffusion pump system. Contamination of most solid surfaces by residual chamber gasses occurs at a rate of 1 monolayer per second at 10^{-6} torr. At a vacuum of 10^{-10} torr, a few tenths of a monolayer per hour would be deposited at the surface (35). Since an oil diffusion pumping system does not generally provide a vacuum higher than 10^{-7} torr, an acceptable level of contamination for most AES surface studies must be obtained by ion pumping with the vacuum chamber and column being sealed with metal gaskets (17). In addition to the vacuum requirements, Auger electron spectroscopy requires cleaning or other means of exposing fresh surfaces of the specimen while inside the vacuum



3-14. Auger analysis of Al-14% Be alloy. (A) Secondary electron image. Beryllium segregated in the annular portion appears lighter. (B) Beryllium Auger image. (C) Oxygen Auger image. (D) Beryllium Auger line profile superimposed upon the secondary image (Courtesy of JEOL, Ltd.).

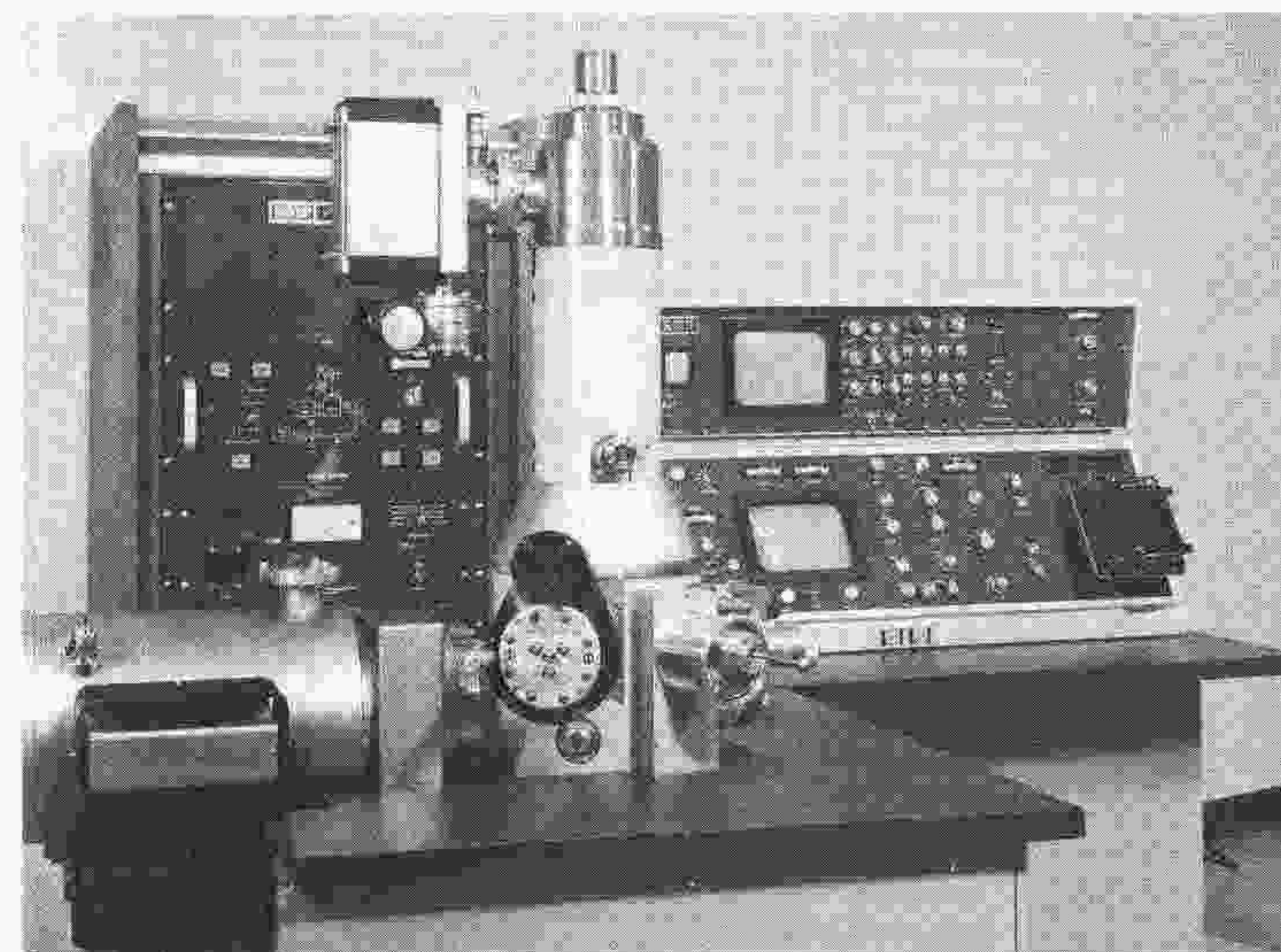
chamber. Since cleaning solutions leave residues, other methods such as ion bombardment (31), high temperature heating or cleaning in vacuum are used.

Since escape depth of Auger electrons is low, the emission volume is small, and spatial resolution is limited by beam diameter. High resolution chemical profiles are routinely obtained from thin films (19,20) and surface elements may be detected in concentrations of 100 ppm (16). Spectral resolution in AES, as with x-ray spectroscopy, is a function of analyzer efficiency and varies with each instrument (Fig. 3-15).

CATHODOLUMINESCENCE

Certain classes of materials radiate visible, ultraviolet, or infrared wavelengths when electrons recombine with holes formed by electron beam interactions. This phenomenon, termed cathodoluminescence, has been observed and employed as a scanning electron microscope signal in studies in the fields of biology, medicine, semiconductor technology, mineralogy, and others (4,5,6,7,10,11,13,14,18,28).

To observe cathodoluminescence from a suitable material in an SEM not previously adapted for this mode, one would remove the scintillator from the secondary electron light pipe. The secondary electrons will not be converted to photons after this modification is performed, and only those photons present due to cathodoluminescence will be detected (Fig. 3-16). Most newer microscope models may be obtained with a second light pipe and



3-15. A scanning electron microscope equipped with Auger electron analysis equipment (Courtesy of AMRAY).