14.2.1.1. Instrumentation for ESCA/XPS

A commercial ESCA/XPS instrument consists of four major components housed in a UHV system with magnetic shielding: (1) the radiation source, consisting of an X-ray source and a means of providing highly monochromatic X-rays; (2) the sample holder; (3) the energy analyzer, which resolves the electrons generated from the sample by energy; and (4) an electron detector. Modern instruments have computerized data recording and processing systems. The pressure required for ESCA must be very low, often less than 10^{-9} Pa in order to prevent adsorbed residual gas from interfering with the surface analysis. This requires a UHV system. As will be seen, many of the instrument components are also used for Auger spectroscopy instruments. The sample holder will be discussed in Section 14.2.3.

Radiation Source. The radiation source used in ESCA is a standard X-ray anode tube, as described in Chapter 8. Soft X-rays are used, with Al and Mg being the most common anodes. Many commercial systems offer a dual anode X-ray tube so that the analyst can switch between excitation wavelengths. It is very important that the X-ray source be monochromatic, with a linewidth extending over as narrow an energy range as possible. Al and Mg have narrow K emission lines. The Mg K_α line has an energy of 1253.6 eV and a linewidth of 0.7 eV, while the Al K_α line has an energy of 1486.6 eV and a linewidth of 0.85 eV. Linewidths for other elements are generally about 1–4 eV. It can be seen from Eq. (14.1) that any variation in the energy of the impinging X-rays will produce a similar variation in the energy of the ejected ESCA electron. There are several ways to reduce the bandwidth of the source, especially if anodes other than Al are used. One of the most accurate is to use a crystal monochromator, such as the Rowland circle system shown in Fig. 14.3. (If necessary, the student should review diffraction of X-rays by crystals in Chapter 8.) Although this instrument gives very monochromatic radiation and narrow linewidths, which increase the spatial resolution of the technique, the intensity of the radiation is reduced in the process. Resolution achievable by using a crystal monochromator is on the order of 100 μm. It should be noted that Mg cannot be used as the source with an X-ray monochromator; there are no crystals available with the proper spacing.

If an aluminum target is used, an aluminum window placed after the target removes much of the K_β and background radiation (**Bremsstrahlung**) from the aluminum source. A magnesium filter can be used in a similar fashion with a magnesium source.

One problem with using X-ray beams as an energy source is that they cannot be focused; therefore, relatively large surface areas are examined, which limits the

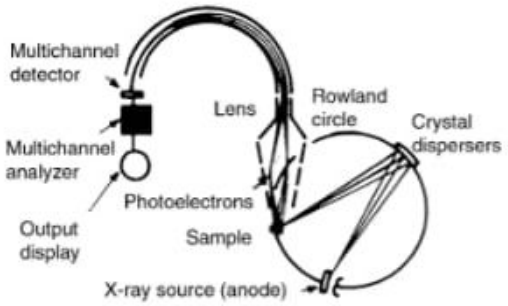

Figure 14.3 Schematic diagram of an ESCA instrument using an X-ray monochromator and a multichannel detector.

spatial resolution of XPS. This problem can be overcome by attaching or depositing the sample on one side of a thin aluminum foil. The Al foil is then bombarded with an electron beam. The electron beam can be focused to a small point and excites the Al foil at that point. The excited Al emits Al K_α lines with a narrow wavelength range (narrow energy range). Only the sample in the immediate vicinity is exposed to this radiation. The net result is that samples in a small region, on the order of a few mm, can be excited with narrow band radiation, thus increasing the spatial resolution of the method. The disadvantage to this approach is that samples must be extremely thin, <1 μm, for the Al X-rays to reach the sample surface.

An alternative method for improving resolution, shown schematically in Fig. 14.4, selects the photoelectrons from a given area of the sample surface using an aperture and electron-focusing lens. Only the photoelectrons from a given small area on the sample surface are passed into the energy analyzer. The spatial resolution of this approach is on the order of 25 μm. The sample surface must be uniformly irradiated with a conven-