photomultiplier are mounted in a light-tight cylinder surrounded by an antimagnetic foil. The high X-ray absorption of the crystal provides a high quantum-counting efficiency.

A Cu $K\alpha$ quantum produces about 500 visible photons of average wavelength 4100 Å in the scintillation crystal (which matches the maximum spectral sensitivity of the photomultiplier), but only about 25 will be effective in the photomultiplier operation. High-speed versions with special pulse-height analysers have recently become available; they are linear to about 1% at 10^5 counts s^{-1} and can be used at rates approaching 10^6 counts s^{-1} (see Rigaku Corporation, 1990).

The detector system is as described in Subsection 7.1.3.1.

7.1.4.2. *Solid-state detectors*

The following description applies primarily to the use of solid-state detectors in powder diffractometry. Further details of their operation and their use in energy-dispersive diffractometry are treated in Section 7.1.5.

The most common form of solid-state detector consists of a lithium-drifted silicon crystal Si(Li) and liquid-nitrogen Dewar. A perfect single crystal is used with very thin gold film on the front surface for electrical contact. The first amplifier stage is a field-effect transistor (FET). The unit must be kept at liquid-nitrogen temperature at all times (even when not in use) to prevent Li diffusion and to reduce the dark current when in use. The unit is large and heavy and, if not used in a stationary position, a robust detector arm is required, which is usually counter-balanced. The crystal is made with different-size sensitive areas and the resolution is somewhat dependent on the size of the area. In the detector process, the number of free charge carriers (the electron and electron–hole pairs) generated during the X-ray absorption changes the conductivity of the crystal and is proportional to the energy of the X-ray quantum. Details of the mechanism are given in several books [see, for example, Heinrich, Newbury, Myklebust & Fiori (1981) and Russ (1984)].

Intrinsic germanium detectors have higher absorption than silicon detectors, but they have lower energy resolution and there are more interferences from escape peaks. A mercuric iodide (HgI_2) detector can be operated at room temperature and has high absorption (Nissenbaum, Levi, Burger, Schieber & Burshtein, 1984). They have poorer resolution than Si or Ge detectors but can be improved to FWHM = 200 eV at 5.9 keV by cooling to 269 K (Ames, Drummond, Iwanczyk & Dabrowski, 1983).

A small (about 16.5×10 cm), lightweight (3.2 kg) silicon detector with Peltier thermoelectric cooling is available (*e.g.* Kevex Corporation, 1990). This development has supplanted a number of the methods of collecting powder data. The elimination of the liquid-nitrogen Dewar and the compact size makes it possible to replace conventional detectors and the diffracted-beam monochromator in scanning powder diffractometry. The spectrum is displayed on a small screen and the window of the analyser can be set closely on the energy distribution obtained from a powder reflection to transmit, say, only Cu $K\alpha$. The monochromator can be eliminated for a large gain of intensity without loss of pattern resolution. The energy resolution is FWHM $\approx$ 195 eV at 5.9 keV. Elemental analysis can be performed by energy-dispersive fluorescence, and the background can be restricted to the narrow energy window selected. Bish & Chipera (1989) used it to obtain a 3–4 times increase of intensity, the same pattern resolution, and lower tails than with a graphite monochromator and scintillation counter in conventional diffractometry. The major limitation at present is the limited input intensity that can be handled. The limiting (total) count rate is about 10^4 counts s^{-1} and the detector becomes markedly nonlinear at 2×10^4 counts s^{-1}. Internal dead-time corrections can extend the range by increasing the counting times.

7.1.4.3. *Energy resolution and pulse-amplitude discrimination*

The pulse amplitudes are proportional to the energy e of the absorbed X-ray quantum so that electronic methods can be used to reduce the background from other wavelengths and sources. The rejection range is limited by the energy resolution of the detector. As noted above, the pulse amplitudes have distributions that vary around the average value A, Figs. 7.1.4.1(*a*),(*b*). The FWHM of the distribution increases linearly with increasing e (eV) and is proportional to $e^{1/2}$, *i.e.* it improves inversely with $\lambda^{1/2}$. The ratio FWHM/A (expressed in %) is a measure of the energy resolution at a given wavelength; the smaller the ratio the better the resolution. For example, as e increases from 5 to 45 keV, the FWHM approximately doubles while FWHM/e decreases from 5 to 1%. The resolution of proportional counters is about 18% for Cu $K\alpha$ and somewhat better for high-pressure gas fillings; in scintillation counters, it is about 45%. The solid-state detectors have much better resolution. The best are about 2.4% (145 eV) at 5.9 keV (which is the energy of Mn K X-rays from a radioactive ^{55}Fe source used as a standard for calibration).

The electronics include a high-voltage power supply to about 1200 V for scintillation counters and 2000–3000 V for proportional counters, and a single-channel pulse-amplitude discriminator. The latter contains pulse-shaping circuits and the amplifier, and is designed to transmit pulses whose amplitudes lie within the selected range. The lower level rejects all pulses below the selected level and the upper level rejects the higher amplitudes (Figs. 7.1.4.1*a*, *b*). The range selected is called the window and determines the pulse amplitudes that will be counted by the scaling circuit.

The multichannel analyser is generally used with solid-state detectors. It may have up to 8000 channels and sorts the pulses from the amplifier into individual channels according to their amplitudes, which are proportional to the X-ray photon energies. The pattern can be stored and displayed on a CRT screen, but nowadays a personal computer with a suitable interface card is normally used in place of the analyser. Various programs are available for peak-energy identification, spectral stripping, intensity determination, and similar data-reduction requirements. The limiting count rate that can be handled by the electronics is determined by the total number of photons striking the detector. Pulse-pileup rejectors are used to stop counting momentarily when another pulse is too close in time to allow the original pulse to return to the baseline voltage. A live-time correction extends the counting period beyond the clock time to compensate for the time the analyser is gated off. About 50 000 counts s^{-1} is the maximum rate so that the individual powder reflections have a much smaller number of pulses. If good statistical accuracy is required, the count times are, therefore, much longer than in conventional diffractometry.

For a given e, the pulse amplitudes of scintillation and proportional counters increase with increasing voltage (internal gain) and amplifier setting (external gain). The detector must be operated in the plateau region for the wavelength used (Fig. 7.1.4.1*c*). The counts are measured as a function of the voltage and/or gain, and the plateau begins where there is no further significant increase of intensity. In selecting the operating