edge, Fig. 2.3.5.3(*b*). The combination of discrimination and filter produces mainly the $K\alpha$ doublet, Fig. 2.3.5.3(*d*). Spectral analysis of the background of a non-fluorescence powder sample using this method with 50 kV Cu radiation and a scintillation counter shows it to be 50–90% characteristic radiation.

The linearity of the system is determined by the dead-time of the detector, and the resolving times of the pulse-height analyser and scaling circuit. The observed intensity n_{obs} is related to the effective dead-time of the system τ_{eff} by the relation

$$n_{true} = n_{obs}/(1 - \tau_{eff}\, n_{obs}). \qquad (7.1.4.2)$$

The value of τ_{eff} can be measured with an oscilloscope, or with the multiple-foil method in which a number of equal absorption foils (*e.g.* Al 0.025 mm or Ni 0.018 mm for Cu $K\alpha$) are inserted in the beam one or two at a time. To make certain monochromatic radiation is used, a single-crystal plate such as Si(111), which has no significant second order, and low X-ray tube voltage are employed. The linearity is determined from a regression calculation. A less accurate method is to plot n_{obs} on a log scale against the number of foils on a linear scale. Recent developments in high-speed scintillation counters have extended the linearity to the 10^5–10^6 counts s^{-1} range.

7.1.4.5. *Escape peaks*

The pulse-amplitude distribution may have two or more peaks, even when monochromatic X-rays are used (Parrish, 1966). Absorption of the incident X-rays by the counter-tube gas or scintillation crystal may cause X-ray fluorescence. If this is re-absorbed in the active volume of the counter only one pulse is produced of average amplitude A_1 proportional to the incident X-ray quantum energy e_1 (k = constant)

$$A_1 = ke_1. \qquad (7.1.4.3)$$

However, the gas or crystal has a low absorption coefficient for its own fluorescent radiation, hence, some quanta of the latter of energy e_2 may escape from the active volume of the counter, the amount depending on the geometry of the tube, gas, windows, *etc*. The average amplitude A_2 of the escape pulses is

$$A_2 = k(e_1 - e_2). \qquad (7.1.4.4)$$

Thus,

$$A_1 - A_2 = ke_2. \qquad (7.1.4.5)$$

The pulse-height analyser discriminates against pulses only on the basis of their amplitudes. When it is set to detect X-rays of energy e_0, it is also sensitive to X-rays of energy $e_0 + e_2$. For example, when using an NaI scintillation counter for Cu $K\alpha$, $e_0 = 8$ keV, and for the escape X-rays I $K\alpha$, $e_2 = 28.5$ keV. A pulse-height analyser set to detect X-rays of energy 8 keV is also sensitive to X-rays of energy 36.5 keV, because, from equations (7.1.4.3) and (7.1.4.4),

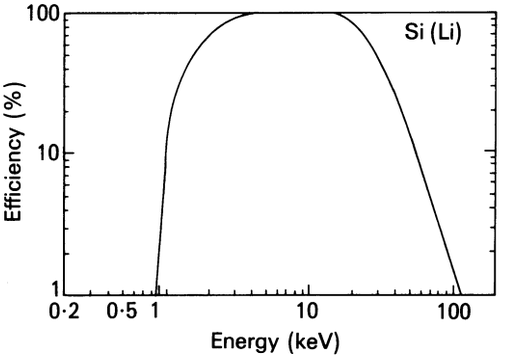
$$A_0 = k.8 = k(36.5 - 28.5) = A_2. \qquad (7.1.4.6)$$

In Figs. 2.3.5.3(*c*), (*d*) and 7.1.4.1(*d*), the escape peak E.P. shows clearly at 0.35 Å, the wavelength of 36.5 keV X-rays. There may be a number of weak escape peaks arising from the stronger powder reflections. In practice, the escape peak should not be confused with a small-angle reflection. It can be tested by reducing the X-ray tube voltage to below the absorption-edge energy of the element in the detector from which it arises.

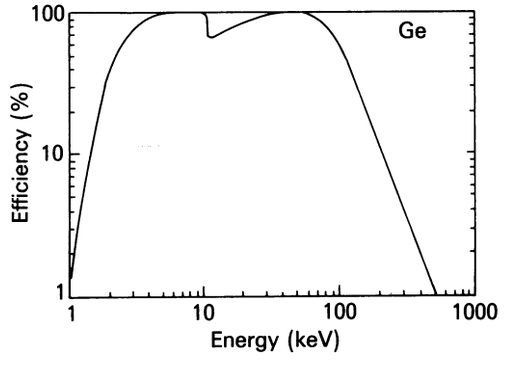
7.1.5. Energy-dispersive detectors (By B. Buras and L. Gerward)

In white-beam energy-dispersive X-ray diffraction, the spectral distribution of the diffracted beam is measured either with a semiconductor detector (low-momentum resolution) or with a scanning-crystal monochromator (high-momentum resolution) (see Subsection 2.5.1.3). Commercially available detectors are made of lithium-drifted silicon or germanium [denoted Si(Li) and Ge(Li), respectively], or high-purity germanium (HPGe). There are, however, other materials that are good candidates for making energy-dispersive detectors.

The semiconductor detector can be regarded as the solid-state analogue of the ionization chamber. Charge carriers of opposite sign (electrons and holes) are produced by the X-ray photons. They drift in the applied electric field of the electrodes and are converted to a voltage pulse by a charge-sensitive preamplifier. The energy required for creating an electron–hole pair is 3.9 eV in silicon and 3.0 eV in germanium. The number of electron–hole pairs is proportional to the energy of the absorbed photon (the intrinsic efficiency is discussed below). There is no intrinsic gain and one has to rely on external amplification. The preamplifier employs an input field-effect transistor (FET), cooled in an integral assembly with the detector crystal in order to reduce thermal noise. Usually the detector is operated at liquid-nitrogen temperature. However, Peltier-cooled silicon detectors are available, removing the maintenance concerns of cryostat cooling. The basic counting system consists further of an amplifier, producing a near-Gaussian pulse shape, and a multichannel pulse-height analyser. It is common to use an



(*a*)



(*b*)

Fig. 7.1.5.1. Intrinsic efficiency of semiconductor detectors. The dimensions are selected to give typical best values of the energy resolution. (*a*) Si(Li), detector thickness 3 mm, Be-window thickness 25 μm. (*b*) HPGe, detector thickness 5 mm, Be-window thickness 50 μm.

additional circuit to reject pileup pulses that can distort the spectrum at high count rates.

The intrinsic efficiency, defined as the ratio of the number of pulses produced to the number of photons striking the detector, is close to 100% in a large energy range. Because of the penetrating power of high-energy X-rays, the efficiency declines at high energies. The low-energy limit is set mainly by the absorption in the beryllium entrance window of the detector. Fig. 7.1.5.1 shows the intrinsic efficiency for an Si(Li) detector and HPGe detector with typical crystal size and window thickness. It is seen that the useful photo-energy range is about 1–40 keV for the Si(Li) detector and 2–150 keV for the HPGe detector. Some minor complications of the HPGe detector are a dip in efficiency around the germanium K absorption edge at 11 keV and the presence of Ge $K\alpha$ and $K\beta$ escape peaks in the measured spectrum.

The energy resolution is commonly expressed as the full width at half-maximum (FWHM) of a peak in an energy spectrum. For a spectral peak with Gaussian shape, ΔE(FWHM) corresponds to 2.355 times the root mean square of the energy spread. The energy resolution, including both the detector and the associated electronics, is given by

$$\Delta E_{\rm FWHM} = \{e_n^2 + [2.355(F\varepsilon E)^{1/2}]^2\}^{1/2}, \qquad (7.1.5.1)$$

where e_n is the electronic noise contribution, F the Fano factor (about 0.1 for both silicon and germanium), and ε the energy required for creating an electron–hole pair.

The energy resolution is generally specified at 5.9 keV (Mn $K\alpha$) as a reference energy. Typical best values for a detector with 25 mm^2 area are 145 eV (2.5%) for an HPGe detector and 165 eV (2.7%) for an Si(Li) detector. The resolution is degraded for larger detector areas.

Count-rate limitations are particularly obvious in synchrotron-radiation applications, where high photon fluxes are encountered (Worgen, 1982). The count rate is limited to below 10^5 counts s^{-1}, mainly by the pulse processing system.

Cadmium telluride, mercury iodide and other wide-band-gap semiconductors could be good candidates for energy-dispersive room-temperature X-ray detectors. Until now, the best energy resolution of the Hg_2I spectrometer with both the detector and the preamplifier operating at room temperature is 295 eV (FWHM) for the 5.9 keV Mn $K\alpha$ line, corresponding to a relative resolution of 5.0%. By lowering the noise level of the preamplifier FET with cryogenic techniques, a resolution of about 200 eV (3.4%) has been achieved (Warburton, Iwanczyk, Dabrowski, Hedman, Penner-Hahn, Roe & Hodgson, 1986).

7.1.6. Position-sensitive detectors (By U. W. Arndt)

Most X-ray diffraction or scattering problems require the quantitative evaluation of a linear or of a two-dimensional pattern. Recent years have seen the development of many different types of linear and area detectors for X-diffraction purposes, that is, of position-sensitive detectors (PSD's) that allow the recording of the positions of the arrival of X-ray photons (Hendricks, 1976; Hendrix, 1982; Arndt, 1986). In addition, imaging detectors have found increasing use in related fields, such as in X-ray astronomy (Allington-Smith & Schwarz, 1984), in X-ray microscopy, in X-ray absorption spectroscopy, and in topography. Here, the emphasis is on the production of an image for direct viewing rather than on the making of quantitative intensity measurements; these applications, in general, require an ultra-high spatial resolution over a relatively small field of view and the ability to cope with very low contrast images: Imaging detectors for topography are discussed in Section 7.1.7. Lessons can also be learnt, and component parts utilized, from quantitative imaging devices developed for visible light.

Progress in these fields has been covered in the *Symposia on Photoelectronic Image Devices* held every 3 years at Imperial College London (since 1960) and in the *Wire Chamber Conferences* (since 1978) and the *London Position-Sensitive Detector Conferences* (since 1987), both reported in full in *Nuclear Instruments and Methods*. Detectors are always one of the principal subjects considered at synchrotron-radiation conferences and workshops, the highlights usually being reported in *Synchrotron Radiation News*. Detectors feature prominently in the proceedings of the *IEEE Symposia on Nuclear Science*, which appear in the *IEEE Transactions*.

Other recent reviews of X-ray detectors are by Fraser (1989), Stanton (1993), Stanton, Phillips, O'Mara, Naday & Westbrook (1993) and Sareen (1994).

The detection of X-ray photons in the energy range of interest for diffraction studies (3 to 20 keV) always involves the interaction of the photon with an inner-shell electron and its complete absorption. The processes that are of interest for the construction of PSD's are of three kinds:

(1) Photography. The characteristics of X-ray film are discussed in Section 7.1.1.

(2) The use of storage phosphors, such as europium-activated barium halide (BaFX:Eu^{2+}, X = Cl or Br) (Sonada, Takano, Miyahara & Kato, 1983; Miyahara, Takahashi, Amemiya, Kamiya & Satow, 1986), which are exposed like photographic film and then scanned with a laser beam causing photon-stimulated light emission of an intensity proportional to the original exciting X-ray intensity; this is measured with a photomultiplier. The plate is re-useable when the X-ray image has been erased. These X-ray detectors have a low background, a large dynamic range, and an adequate spatial resolution. See Section 7.1.8.

(3) Processes that involve the production of electrons. These may be the result of the ionization of a gas; they may be due to the production of electron–hole pairs in a semiconductor; they may be produced in an X-ray photocathode; finally, a phosphor may be used to convert the X-rays into visible light that then produces photoelectrons from a conventional photocathode.

At the present time, X-ray-sensitive photographic emulsions are mainly of historical interest. Storage-phosphor image plates have not only largely replaced photographic film; they have also taken over many of the applications of electronic detectors.

In the following, we are concerned only with detectors that depend on the production of electrons by incident X-rays.

In all detectors, except in semiconductor detectors, the number of primary electrons is multiplied by gas amplification, or in some device such as a microchannel plate or by some other intermediate process. In a PSD, the electron multiplication must take place with a minimum of lateral spread.

Many methods are available for deriving the position of the amplified electron stream or of the cloud of electron–ion pairs (avalanche) in an ionized gas and some of these are discussed below. However, almost any combination of photon detection, electron multiplication, and localization procedure can be used in the construction of PSD's (Fig. 7.1.6.1).

7.1.6.1. *Choice of detector*

Detectors may either be true counters in which individual detected photons are counted or they may be integrating devices that generate a signal that is a function of the rate of arrival of

references