

7.2. Detectors for electrons

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7.2.1. Introduction

It is convenient to divide instruments used to form electron-diffraction patterns and images into two categories. In the first, all electron beams are static in the sense that neither the beams incident on nor emergent from the specimen are varied in position or orientation during the detection and recording process. Such is the case, for example, in a conventional transmission electron microscope (CTEM) where diffraction patterns and images must be recorded using a parallel or flux-density detector. By contrast, instruments in the second category employ a scanning system to vary the position or orientation of the electron beams before or after the specimen. Thus, in a scanning transmission electron microscope (STEM), diffracton patterns may be formed by rocking a beam about a point on the specimen or by positioning a stationary probe at the selected point and deflecting the beams as they emerge from the specimen. Under these conditions, the electrons are detected by a small total flux detector located on the optic axis of the instrument and the diffraction pattern (or image) is built up in a time-sequential or serial manner.

Whilst many of the properties required of total flux and flux-density detectors differ significantly, all detectors must satisfy certain criteria. A means of characterizing detectors is given in Section 7.2.2 together with a brief description of which particular features are desirable in each of the two categories. Specific detectors suitable for parallel recording are considered in Section 7.2.3, where a description is given both of their operation and of the extent to which they fulfil the criteria outlined in the preceding section. Finally, in Section 7.2.4, similar details are given for detectors suitable for serial recording purposes.

7.2.2. Characterization of detectors

Electron-diffraction patterns and images are inherently noisy due to the random arrival of electrons at the detector plane. The number of electrons N arriving in a specified time interval at the detector (or a particular detector element in the case of a flux-density detector) fluctuates in such a way that the variance of the signal σ_N^2 is equal to $\langle N \rangle$ where $\langle N \rangle$ denotes the expectation value of N . This is in accord with Poisson statistics and it follows that the signal-to-noise ratio of the incident signal $(\text{SNR})_i$ may be increased indefinitely, in principle, by simply increasing the recording time until a sufficiently large number of electrons has arrived at the detector.

An ideal electron detector would be one in which the signal-to-noise ratio of the output signal was also equal to the limit imposed by Poisson statistics. Such a detector would impose no additional noise onto the signal and there would be no further loss of information. In practice, this is unattainable, and it is convenient to define a detective quantum efficiency (DQE) to provide a quantitative description of the signal degradation or information loss directly attributable to the detector. If an input signal S_i (variance σ_i^2) gives rise to an output signal S_o (variance σ_o^2), the DQE is defined as

$$\text{DQE} = (dS_o/dS_i)^2 \sigma_i^2 / \sigma_o^2, \quad (7.2.2.1)$$

where dS_o/dS_i is the gradient of the output/input characteristic. For detectors with a linear response, (7.2.2.1) may be simplified and it is convenient to express the DQE as

$$\text{DQE} = (\text{SNR})_o^2 / (\text{SNR})_i^2, \quad (7.2.2.2)$$

where the subscripts i and o again denote input and output. In all cases, the DQE is necessarily less than unity and factors that frequently limit the performance of detectors have been discussed in a general way as well as for specific cases by Herrmann (1984), Chapman & Morrison (1984), and Chapman, Craven & Scott (1989).

Although the DQE provides a useful quantitative figure of merit for a detector, alone it does not provide enough information to determine whether a particular detector will be suitable for a chosen application. To ascertain this, it is necessary to consider the following attributes of individual detectors:

- (a) dynamic range;
- (b) fraction of the dynamic range over which the detector response is (approximately) linear;
- (c) ease of access to the output signal;
- (d) suitability for use over a wide range of electron energies;
- (e) speed of response;
- (f) resolution;
- (g) information-storage capability;
- (h) single shot or repeated use;
- (i) susceptibility to radiation damage;
- (j) simplicity of construction;
- (k) ease of use;
- (l) cost.

For all detection and recording systems, a high DQE over a wide signal range is desirable. This is particularly so when diffraction patterns from single crystals are being studied as the intensity of diffraction spots in a single pattern can vary over many orders of magnitude. In addition, it is advantageous if an unvarying and simple relation exists between the output and input signals over the complete range of the latter. Thereafter, important attributes for parallel and serial systems can differ markedly.

Parallel detectors should have high spatial resolution and a large information-storage capacity. The latter requirement ensures that extensive and complex diffraction patterns and images may be studied at one time while the two requirements together ensure that the overall size of the detector is minimized. This is generally advantageous when optimizing overall instrumental performance. Broadly speaking, two options exist. The first is to employ a relatively complex system that may be used repeatedly and that allows easy access to a quantitative output signal, but that is inevitably expensive and complex in construction (see *e.g.* Subsection 7.2.3.3); the second is to use a simple, cheap system such as film (Subsection 7.2.3.2), which must be replaced each time a new image is to be recorded. The choice between the two options depends largely on the experiment to be undertaken and may involve such factors as whether the required information can be obtained from, for example, the symmetry or separation of spots or lines in a diffraction pattern or whether quantitative intensities across the entire field are needed. Also relevant is the delay that is acceptable between initiating a recording and obtaining the information in the form required. By contrast, the actual speed of response of the detector itself is rarely the limiting factor.

In serial electron detection systems, however, the speed of response of the detector can be crucial. The detector is generally a single undivided element with a fast enough response to ensure

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that recording times are sufficiently short to avoid problems arising from specimen drift or the build up of contamination during the recording. Clearly, a serial detector must also be well suited to repeated use and (reasonably) resistant to radiation damage. The output must be easily accessible and at least temporary storage must be available to allow an entire field to be examined. Although segmented detectors are sometimes used in serial detection systems (*e.g.* Burge & van Toorn, 1980), the number of individual elements is usually small and the spatial resolution of the detector is rarely a relevant consideration.

7.2.3. Parallel detectors

7.2.3.1. Fluorescent screens

The fluorescent screen offers the simplest means of rendering a spatially distributed electron signal visible to the eye. Screens are frequently made using ZnS powder to which small numbers of activator atoms have been added to make the wavelength at which maximum emission occurs match the maximum sensitivity of the eye. This occurs in the yellow-green region of the spectrum.

The light output from a fluorescent screen is proportional to electron current density over a wide range and, for a given current density, increases slowly with electron energy. For electrons of energy greater than ~ 40 keV (as are used in RHEED, CTEM, and HVEM), the output level is generally satisfactory under normal experimental conditions; however, when significantly lower electron energies are involved (as is the practice in LEED where energies are typically less than 1 keV), the electrons must be accelerated onto the screen to increase to a suitable level the number of photons emitted by each incident electron. In practice, an accelerating voltage of ~ 5 kV is used.

The resolution of a fluorescent screen is typically in the range 20–50 μm for powders, although significantly smaller values are achievable, particularly if single crystals are used instead. Powder phosphor screens can generally be made as large as required so that the field of view is limited by instrumental constraints rather than by any imposed by the detector itself. On removal of the electron signal, the light intensity decays in a two-stage process. The initial decrease is rapid with a time constant < 1 ms after which an afterglow lasting ~ 1 –5 s remains. Further details of commonly used fluorescent materials have been discussed by Garlick (1966) and Reimer (1984).

Fluorescent screens may be viewed in reflection or transmission, although the optimum thickness of material (for a given incident electron energy) differs significantly in the two cases. Reflection screens are widely used simply as viewing screens and are rarely used as a component in a recording system; by contrast, transmission screens are the first stage in many systems that combine detection and recording and will appear in this context in Subsections 7.2.3.3 and 7.2.3.4.

7.2.3.2. Photographic emulsions

Photographic emulsions provide the most frequently used means of recording spatially distributed electron signals. They are of little use alone in that the output signal is not available until the emulsion has been developed and fixed and so are normally used in conjunction with a viewing system such as that described above. A photographic emulsion is an example of an analogue storage medium and further equipment is required (see below) if quantitative electron intensity data are to be extracted from the developed emulsion.

In most instances, the electron image or diffraction pattern is allowed to impinge directly onto a desiccated photographic

emulsion stored inside the vacuum system. The probability that a silver halide grain will be rendered developable by an electron of energy ~ 100 keV is high and so, in practice, a single electron may release ~ 10 grains. This is in contrast to what is observed when photographic emulsions are exposed to light where several quanta must be absorbed by one grain to render it developable. For this reason, there is no illumination threshold when electrons are used and the law of reciprocity is applicable over a very wide electron intensity range. Fuller details of the theory of the interaction between electrons and photographic emulsions are given by Hamilton & Marchant (1967), Valentine (1966), Farnell & Flint (1975), and Zeitler (1992).

The alternative to directly exposing film within the vacuum system to the electron beam is to convert the electron signal into an equivalent photon signal, which is then recorded outside the vacuum system. Conversion may be achieved by use of a transmission fluorescent screen, and the photon signal may be led out of the vacuum system using a fibre-optic plate (Guetter & Menzel, 1978). In this way, the need to open the vacuum system every time new films are required is eliminated, but the noise properties of the overall system are generally inferior to those achievable using direct exposure.

The relation between the density D of the developed emulsion and the exposure q (expressed as a charge/unit area) has been widely studied theoretically and experimentally over a range of electron energies (Hamilton & Marchant, 1967; Valentine, 1966). To a good approximation, the characteristic takes the form

$$D = D_s[1 - \exp(-cq)] + D_o, \quad (7.2.3.1)$$

where D_o is the 'fog' level, D_s the saturation density, and c the speed of the emulsion (defined by the gradient of the characteristic dD/dq at $q = 0$). Given that saturation densities up to 6 are not uncommon and the fog can be kept small, it can be seen that the variation of D with q is approximately linear to densities of ~ 1 .

The DQE for a number of emulsions has been measured [for typical results see Herrmann (1984)] and, over a limited range of exposure, values between 0.7 and 0.8 may be achieved. Below and above the optimum exposure, the DQE falls. For low exposures, the effect of the background fog becomes important while saturation effects cause a fall in DQE at high exposures. These effects can be serious when, for example, diffraction patterns with a very high dynamic range are to be recorded and a number of different exposures must be used if maximum information is to be obtained.

Within bounds, the exposure at which the optimum DQE occurs can be varied by selecting different emulsions and also by varying development conditions. As faster emulsions tend to have larger grain sizes, the spatial resolution cannot be regarded as an independent or fixed parameter. For this reason, it is generally preferable when comparing different emulsions to plot the variation of DQE not with the number of electrons falling on unit area of emulsion but with the number of electrons falling on the pixel area. The latter quantity may be defined conveniently as the size of the point spread function of a single electron. Unfortunately, further complications ensue as the resolution of the emulsion depends not only on the grain size but also on the diameter of the electron diffusion cloud in the emulsion, a quantity that varies markedly with electron energy.

Using emulsions commonly employed for recording diffraction patterns and images with 100 keV electrons, a resolution of ~ 30 μm is typical. The film size used in electron microscopy has an area of ~ 50 cm^2 so that a single recording contains $\sim 5 \times 10^6$ pixels. This represents a very high storage capability

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and is particularly useful when recording fine detail over a wide field as occurs both in convergent-beam electron-diffraction patterns and in high-resolution images.

If quantitative electron intensity data are required, the density distribution on the emulsion must be digitized. High-precision TV cameras, flying-spot devices, drum scanners, or flat-bed scanners have all been used for this purpose. While the first named provides the data in the shortest time, flat-bed scanners offer superior precision in both intensity measurement and definition of the area from which the measurement is made, and can digitize a larger number of pixels at one time.

In summary, the extensively used photographic plate provides a cheap, convenient electron detector with a high storage capacity. It may be used under a wide range of experimental conditions and has an input/output characteristic typical of that produced when any ionizing radiation is incident on a photographic emulsion. As such, it closely resembles that obtained when X-rays are used instead of electrons. Against its advantages must be offset the inevitable delay in access to any information while the emulsion is being developed and the further delay incurred (if quantitative data are sought) while densitometry is undertaken.

7.2.3.3. Detector systems based on an electron-tube device

Detector and recording systems based on electron-tube devices have been reviewed in detail by Herrmann & Krahl (1984). The first stage is a transmission fluorescent screen (as described in Subsection 7.2.3.1), which converts the electron image to its photon counterpart. A fibre-optic plate may then be used to transfer the photon image to a low-light-level TV camera located outside the vacuum system. In some instances, an image intensifier is included before the TV camera to increase the intensity of the light signal being recorded. An alternative means of increasing the light signal, preferable when the energy of the incident electrons is low, is to employ a channel plate before the fluorescent screen.

Electronic systems are capable of detecting single electrons and, provided the electron current density incident on the fluorescent screen is sufficiently low, can have a DQE of ~ 0.9 . The restriction to low current densities arises from the need to ensure that in a single TV frame the number of electrons arriving at any pixel is either 0 or 1. Under these conditions, truly digital images may be obtained in which the number associated with each pixel is the number of electrons that arrived at the corresponding small area of the fluorescent screen during the exposure. To achieve such an image in practice requires the accumulation of many individual TV frames (the precise number depending on the statistical accuracy required) and this is normally achieved using an image memory or frame store.

At higher electron current densities, the number of electrons arriving at each point on the fluorescent screen in a TV frame interval can be considerably greater than one. The TV output signal then varies continuously and it is impossible to determine the exact number of electrons associated with each pixel. When used in this way (the analogue mode), the maximum value of DQE is ~ 0.8 .

The number of pixels in a frame is typically $\sim 3 \times 10^5$, a number appreciably lower than the storage capacity of the film commonly in use. A consequence of this is that diffraction patterns may have to be recorded at a range of camera lengths if fine detail and high-scattering-angle information are both to be observed.

The electronic detector system as described, particularly if it incorporates an intensifier, has a higher sensitivity than the naked

eye. This is particularly beneficial when focusing fine structures or when working with radiation-sensitive specimens where limitations are imposed on the exposure to which the specimen may be subjected. Perhaps of even greater value, however, is the fact that the system as a whole provides storage (thus removing the need to irradiate the specimen continuously during observation) and that the storage is in a digital form. As a result, it is straightforward to interface a computer to the system and on-line processing of the stored intensity values may be undertaken readily.

A major disadvantage of the system is the cost and susceptibility to damage (if subjected to excessive intensities) of high dynamic range, low-noise electron tubes, which should be used if the highest performance is to be achieved; a further drawback for many applications is the barely adequate number of pixels/frame.

7.2.3.4. Electronic detection systems based on solid-state devices

Electronic detection systems resembling those described in the preceding section but with the electron tube replaced by a semiconductor array detector have been the subject of intense development recently. Initially, both charge-coupled devices (CCD) and self-scanned photodiode arrays (PDA) were explored (Herrmann, 1984), but the former have now assumed the dominant position. Early devices suffered from a barely adequate number of pixels ($\sim 10^4$) but those currently in use have between 2×10^5 and 4×10^6 pixels. As such, they frequently allow larger image fields to be explored than do standard TV systems. Furthermore, CCD-based systems offer a wide dynamic range ($> 16000:1$), linearity better than 1% over the entire dynamic range, and extreme sensitivity. They display no lag or sensitivity to magnetic fields and they are not damaged by overlighting. Details of the performance of fully operational systems have been given by Krivanek, Ahn & Keeney (1987), Daberkow, Herrmann, Liu & Rau (1991), Kujawa & Krahl (1992), and Ishizuka (1993).

To achieve optimum performance, a number of precautions must be taken including modest cooling of the CCD to suppress thermally generated charge carriers. Readout speeds are long compared with TV rates so that any noise associated with this process is minimized. In practice, high-precision correlated double-sampling techniques are used for analogue-to-digital conversion to realise this end. There is also the need to compensate for fixed-pattern noise due to non-uniformities of dark current and due to the fibre plate. For these purposes, use is made of a reference image recorded when the device is simply flooded with uniform illumination. An alternative approach to reduce at least some of the fixed-pattern noise is to use a lens rather than a fibre-optic plate between the transmission fluorescent screen and the device (Fan & Ellisman, 1993). When all these precautions are taken, the resulting system has sufficient sensitivity to detect single 100 keV electrons.

From the above, it should be clear that the CCD-based system offers many advantages over the TV system if quantitative electron data (images or diffraction patterns) are required for subsequent computer analysis. However, its slower response speed means that its use is limited for alignment purposes and much *in situ* experimentation. It is advantageous, therefore, to have both systems available.

7.2.3.5. Imaging plates

A recent advance using a medium related to film involves the imaging plate (Mori, Katoh, Oikawa, Miyahara & Harada,

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1986), which relies on the phenomenon of photostimulated luminescence. Here the active area is a coating of a photo-stimulable phosphor that can store energy when excited by electrons. The energy absorbed is then emitted as photons when the medium is illuminated with visible or infrared radiation and this signal is detected using a photomultiplier. The read signal is provided by a scanning He-Ne laser so that, as with the detectors in Subsections 7.2.3.3 and 7.2.3.4, information is recorded in parallel but accessed serially. Initial experiments suggest that the imaging plate offers higher sensitivity and a wider dynamic range than most film, but currently suffers from inferior spatial resolution. Recent discussion of the performance and limitations of imaging plates has been supplied by Mori, Oikawa, Katoh, Miyahara & Harada (1988) and Isoda, Saitoh, Moriguchi & Kobayashi (1991).

7.2.4. Serial detectors

7.2.4.1. Faraday cage

The Faraday cage is the most convenient means of determining electron-beam currents. It consists of a small electrically isolated cage with a small hole in it through which electrons enter. Provided the hole subtends a sufficiently small solid angle and the inner surface of the cage is made of a material with a low back-scattering coefficient, the probability that any electrons will re-emerge is negligible. An electrometer is normally used to measure the charge that has entered the cage.

The main use of the Faraday cage is to calibrate other detectors when absolute electron intensities are required. It also serves a very important role when the total specimen exposure in an experiment must be kept below a critical value owing to the susceptibility of the specimen to radiation damage. As electron charge is being measured, the Faraday cage may be used with electrons of any energy.

7.2.4.2. Scintillation detectors

One of the most widely used total flux detectors is a scintillator, the output from which is coupled into a photomultiplier by a light-pipe. In the first stage, an incident electron deposits its energy in the scintillator, producing a number of light photons with an energy deficiency of up to $\sim 20\%$ (Herrmann, 1984). By careful design of the light-pipe, an appreciable fraction of the photons will reach the photomultiplier, which should have a photocathode whose quantum yield peaks around the wavelength of the photons from the scintillator. Even though the quantum yield of the photocathode is likely to be < 0.2 , the number of photoelectrons emerging from the photocathode for each electron incident on the scintillator should be considerably greater than unity, provided the incident electron energy exceeds ~ 10 keV. With lower-energy electrons, it is advantageous to provide an additional acceleration onto the scintillator to ensure that an adequate number of photons is generated.

Following the production of photoelectrons, considerable multiplication takes place down the dynode chain of the photomultiplier and a current pulse may easily be detected at the anode. Electron counting is therefore a possibility and to take advantage of this over as wide a current range as possible scintillators with very fast decay times (10^{-8} to 10^{-7} s) should be used. Such scintillators have the additional advantage that they may be used in systems where beam scanning is performed at TV rates.

When the rate of arrival of electrons at the detector appreciably exceeds 10^6 s^{-1} , it becomes increasingly difficult

to distinguish the output from individual electrons and the detector must be operated in an analogue mode. Despite this, the individual electron pulse-height distributions from good scintillators are sufficiently narrow that values of DQE greater than 0.8 may be obtained (Chapman & Morrison, 1984). Scintillators meeting both speed and pulse-height distribution requirements include plastics (e.g. Nuclear Enterprise NE102A) and Ce-doped YAG (Schauer & Autrata, 1979). It should be noted that the materials discussed in Subsection 7.2.3.1 are generally unsuitable for fast detector systems because of their relatively long time constants and the existence of an afterglow that persists for several seconds.

To handle the large dynamic range encountered in diffraction patterns, it is advantageous to use a detector system capable of both counting the arrival of individual electrons and making analogue current measurements. Such a system, which allows signals whose magnitudes differ by a factor of 10^8 to be recorded with a high DQE in a single scan, has been described by Craven & Buggy (1984). More general advantages of detector systems based on scintillation counters are their desirable input/output characteristics, which are essentially linear, and the fact that a quantitative measure of electron intensity is directly available in a form suitable for input to a computer. Analysis of the data may thus begin as soon as its collection is completed.

The susceptibility to radiation damage of many high-efficiency scintillators, resulting in a diminution of light output with increasing use, is probably the major disadvantage of these detectors. Of importance in some instances is the fact that the entire detection system is relatively bulky and it may not always be possible to position it satisfactorily within the apparatus.

7.2.4.3. Semiconductor detectors

The most commonly used semiconductor detector is a silicon photodiode whose $p-n$ junction is reverse biased. A fast electron incident directly on the device produces electron-hole ($e-h$) pairs and the two charge carriers are swept in opposite directions under the influence of the bias field. Thus, a charge pulse is produced and as, on average, an $e-h$ pair is produced for every 3.6 eV deposited in the silicon, a 100 keV incident electron produces a pulse equivalent to $\sim 3 \times 10^4$ electrons. In practice, many devices have a contact layer on the silicon surface (which itself may be less perfect than the bulk of the material) so that some of the energy of the incident electron is lost before the sensitive volume is reached. As the energy deposited in surface layers is rarely less than 5 keV, semiconductor detectors are unsuitable for direct use with low-energy electrons.

The magnitude of the signal produced by each incident electron in a semiconductor detector is typically $\sim 10^4$ times lower than that emerging from the anode of the photomultiplier in scintillation detector systems and it is difficult to measure such small signals without adding substantial noise. A further complicating factor arises from thermally generated carriers, which can give rise to a substantial dark current from detectors of area $> 1 \text{ mm}^2$. Thus, photodiode detectors cannot normally be operated in an electron-counting mode and suffer from a low DQE whenever incident electron currents are small. To optimize their performance in this range, the device should be cooled (to reduce the thermally generated signal) and the electron beam should be scanned relatively slowly so that high-gain low-noise amplifiers may be used for subsequent amplification of the signal from the photodiode. Above the low-signal threshold, the output from the photodiode varies linearly with incident electron intensity and is once again in a form suitable for direct display or for being digitized and stored.

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The main advantages of semiconductor over scintillation detection systems are their robustness, cheapness, and compactness. The latter is particularly valuable for certain applications in that it allows the detector to be sited very close to the specimen even when space is very confined. This occurs, for example, when the specimen is immersed in a magnetic lens and channelling patterns from back-scattered electrons are to be recorded. A further advantage arises if images are to be formed in scanning microscopes using signals from a number of closely positioned detectors whose shapes may be quite complex. Using lithographic techniques, several detectors may be fabricated on a single silicon substrate and, provided the gains of any succeeding amplifiers are well

matched, a detection system with a well defined response function results.

7.2.5. Conclusions

A wide variety of different means exists for detecting electrons. Many are almost perfect in that they add very little noise to that already present in the electron beam. However, no single detector meets all the requirements of different experiments and, before selecting a detector for a specific purpose, it is necessary to consider the relative importance of the attributes listed in Section 7.2.2. Once this is established, it should be straightforward to determine the optimum detector for the task in hand.

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