

7. MEASUREMENT OF INTENSITIES

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,