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Review: transmission scanning electron microscopy

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The availability of scanning attachments for transmission microscopes and the advent of very high resolution scanning microscopes now enables materials to be studied in both the back scattered and transmission scanning modes. It is the purpose of this review to present in outline the subject of transmission scanning microscopy, the advocated advantages in comparison with conventional transmission microscopy and some of the achieved and potential applications.

1. Introduction

Transmission scanning electron microscopy (TSEM) is the mode of microscope operation in which a small focused probe of electrons is scanned over the surface of a specimen and the electrons transmitted are used to form an image. The essential requirements of a microscope operating in the TSEM mode are a scanning system and a probe forming lens before, and an electron detector after the specimen. These requirements may be met either by a scanning microscope with a collector for transmitted electrons or by a conventional transmission electron microscope (CEM) fitted with a detector and a device for scanning a focused beam over the specimen.

Although transmission scanning microscopy was demonstrated a considerable time ago – in the original design for a scanning microscope by von Ardenne [1] the transmitted electrons were used to form the image – it has only been the recent work of Crewe and his group that has shown the capabilities of the technique. In a quite remarkable piece of work they built a scanning microscope, with a field emission electron source, having a resolution equal to a conventional electron microscope. With this instrument they have recently demonstrated what is believed to be resolution of uranium atoms. [2]. That a similar picture of thorium atoms was later obtained in a conventional electron microscope illustrates the reappraisal of the capabilities of the electron microscope, caused by the advent of a high resolution scanning microscope. (Hitherto it was thought impossible to obtain atomic resolution with existing microscopes.)

2. Principle of reciprocity

To introduce a transmission microscopist to the operation of a scanning microscope it is useful to employ the concept of reciprocity. When it is used to compare the CEM and the TSEM, it just says that the scanning microscope is equivalent to a conventional microscope except that the electron directions are all reversed. The two microscopes are illustrated schematically in Fig. 1; the CEM with the electron direction from top to bottom and the TSEM from bottom to top. From the top, in the CEM, a divergent beam of electrons from a heated filament is accelerated through an apertured set of condenser lenses and down the optical axis of the system and used to illuminate a large area of a specimen. A diffracted or scattered beam of electrons from the specimen is selected by an objective aperture and passes through a system of projecting lenses to be imaged on a fluorescent or photographic screen. The objective aperture not only selects a particular scattered or diffracted beam, but also controls the aberrations present in the final image. In the scanning microscope some of the electrons leaving the small source of the microscope pass through an aperture and are then imaged by the condenser lens system to form a demagnified image of the aperture on the specimen. The scanning system merely has the effect of moving the source in the source plane. Those electrons diffracted or scattered by the specimen are detected on the axis and a signal propor-

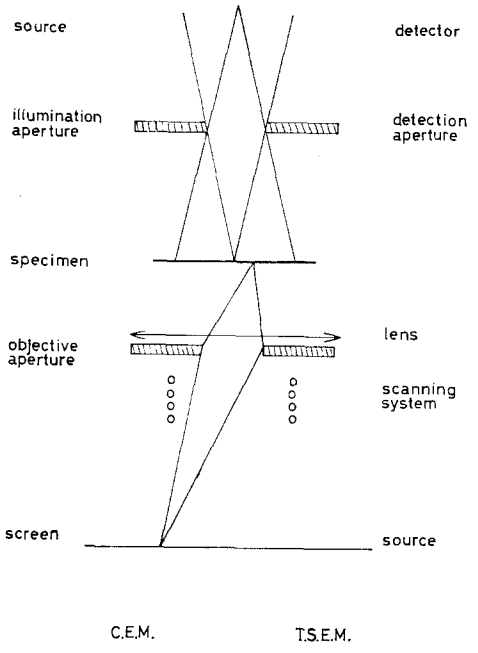

Figure 1 Schematic illustration of conventional (CEM) and transmission scanning (TSEM) electron microscopes.

tional to the number of electrons detected is subsequently displayed.

Two important points can be understood from an examination of Fig. 1; firstly, in the conventional electron microscope the contrast seen is due to the removal of electrons from the beam by physical aperturing in the back focal plane. In the transmission scanning microscope the contrast arises by virtue of the finite angular acceptance of the detector. Secondly, the major aberrations in the CEM are caused by the post-specimen lenses and can be restricted only by the size and position of the objective aperture. By comparison the aberrations in the TSEM lie in the illuminating system. The resolution in the transmission scanning microscope is of course determined by the spot size used, which in turn is affected by the aberrations of probe forming lenses present. It is therefore possible to consider a scanning microscope as being equivalent to a conventional electron microscope. This is particularly useful in analysing the contrast in TSEM images since the formation of identical contrast features in the CEM, especially diffraction contrast, is very well understood. Such an exercise has been made by Crewe and Wall [3].

3. Advantages of the scanning system

As the principle of reciprocity shows that there is no essential difference between the image formation in the CEM and the TSEM, provided that the probe size used is smaller than the resolution required, why is the TSEM being developed? The reason is that the scanning system promises to do many things better because of the simpler construction, the absence of lens induced aberrations after the specimen, the much smaller size of the probe in comparison with the area examined in the selected area diffraction mode of the conventional microscope and finally, by virtue of the enhanced possibilities of signal processing.

The basic scanning microscope is more simple in construction than the conventional microscope since there are no lenses after the specimen and hence no accompanying lens distortions in the imaging system. A related benefit is that any aberrations that do exist, other than the spherical aberration are independent of the magnification. This is not the case with the conventional microscope where they dominate at very high magnifications. The most important aberration in a scanning microscope is the spherical one which in fact decreases with increasing magnification. This may be seen by considering what is meant by the magnification of a scanning microscope. It is equal to the ratio of the areas of the displayed image to that being scanned on the specimen, and is changed by altering the excitation of the scanning coils. The larger the area being scanned, and hence the lower the magnification, the further the scanning coils must deflect the electron beam from the axis of the illuminating lens system and so the larger the spherical aberration becomes. The absence of any lenses after the specimen also means that very small movements of the specimen may be replaced by a relative deflection of the illuminating beam so the mechanical design of the specimen stage movements may be made less exacting.

In addition to the advantages of the TSEM over the CEM arising from the relative instrumental simplicity there are a further set that are due to the use of a time dependent, or scanning, imaging system as distinct from a time independent one. As a consequence of this the only part of the specimen being irradiated is that corresponding to the picture point being recorded. Also all of the transmitted beam may, if required, be collected and used. In fact, if a sufficiently noise-free detection and display system is used, each electron transmitted by the specimen gives rise to a point on the final screen. Compare this

with the situation in conventional electron microscopy where not only is the area illuminated on the specimen many times larger than that corresponding to the image but also a highly inefficient display system is used (fluorescent screen) and only part of the transmitted beam selected. The combination of illuminating only the area of interest and the use of an efficient detector is particularly important in the examination of material liable to irradiation damage, as was pointed out by Cowley in 1966 [4].

Further consequences of the time dependent imaging system are that the spatial resolution in the final image can be modified before the signal is displayed. The action of the transmission scanning microscope system can be summarized by saying that it transforms the spatial variation of the specimen transmission into a time varying electronic signal. It is then in a suitable form for processing using established electronic techniques prior to subsequent inverse transformation and display as a spatial function.

The ability to process the signal is particularly useful when the images are of the type that are traditionally difficult to record in conventional electron microscopy, for instance, weak contrast on a high background level. In the scanning microscope it is possible to bias the signal so as to remove most of the d.c. component. In practice, the amount of bias is adjusted until the area of specimen giving lowest signal appears dark, and then the signal is amplified until the area with the highest signal appears bright. Where the signal contrast varies strongly over the field of view it is possible to use non-linear amplification of the detected signal so that signals of small contrast are amplified more than those of large contrast. A related technique is that of signal differentiation in which short range contrast can be enhanced at the expense of long range contrast. This is relevant to the situation where there is detail in both the dark regions and the light areas of an image and under normal circumstances it is impossible to display both simultaneously.

The uses of signal processing are not merely limited to changing the relative contrast of areas in an image but can be extended to obtaining specific information correlated to spatial occurrences. Recent exploratory work in a number of laboratories has shown that it is feasible to feed the picture signals straight into a computer for the determination of information such as distribution analyses, shape classifications and also direct comparison with computed micrographs, which can be subsequently displayed.

4. Applications of transmission scanning microscopy

Although the advantages of the scanning mode of transmission are generally known, few applications have been reported because of the very small number of instruments that exist at present. However, three main areas of application may be recognized for which the instrument is potentially superior to the conventional electron miroscope. These are the ease of energy analysis, the ability to image thicker materials than the conventional microscope, and thirdly the ability to obtain a diffraction pattern from a very small volume of material. The addition of an energy loss facility to a microscope enables an image to be formed with electrons elastically scattered or with those that have specified energy loss. One of the major difficulties of energy loss analysis of specimens in a conventional electron microscope is that it is not possible to analyse with any accuracy all the electrons that make up an image simultaneously, since the analyser must transmit both spatial information and energy information. The difficulty in transmitting spatial information is that the electrons from the specimen are distributed over a very large area in comparison with the input area of any accurate analyser. In the transmission scanning microscope the spatial information for the final image is provided by the scanning system. Also the area of even a small detector is many times greater than the projected area of the region of the specimen scanned by the illuminating beam, so that an energy selecting or analysing device, such as a spectrometer, may be fitted before the detector to intercept the chosen transmitted beam. By selecting the energy that the spectrometer will pass to the detector, pictures of the specimen may be displayed corresponding to particular energy losses. Such a method was used by Crewe *et al* [5] to image evaporated aluminium islands on a carbon film using electrons having a characteristic energy loss due to plasmon excitations in the aluminium metal, so as to display the aluminium only. In a way similar to that adopted by Cundy *et al* [6] it will be possible to carry out simple elemental analysis. However, a more sophisticated application would be to make use of the very fine probe size to carry out elemental analyses and concentration determinations from both point to point and

complete image energy loss measurements. Here the association with a simple computing system would be most useful for the de-convolution of the energy loss curves into the constituent absorption peaks, for the assimilation of the information and possibly also for concentration contour mapping.

It has been proposed many times that the absence of chromatic aberrations after the specimen in the transmission scanning microscope means that for a given desired resolution thicker materials can be examined than in the conventional electron microscope, because all the electrons irrespective of their energies can be used to form a signal. Whether this is true for materials of very high atomic number is in doubt (Howie, private communication). This is not the case in the conventional transmission microscope

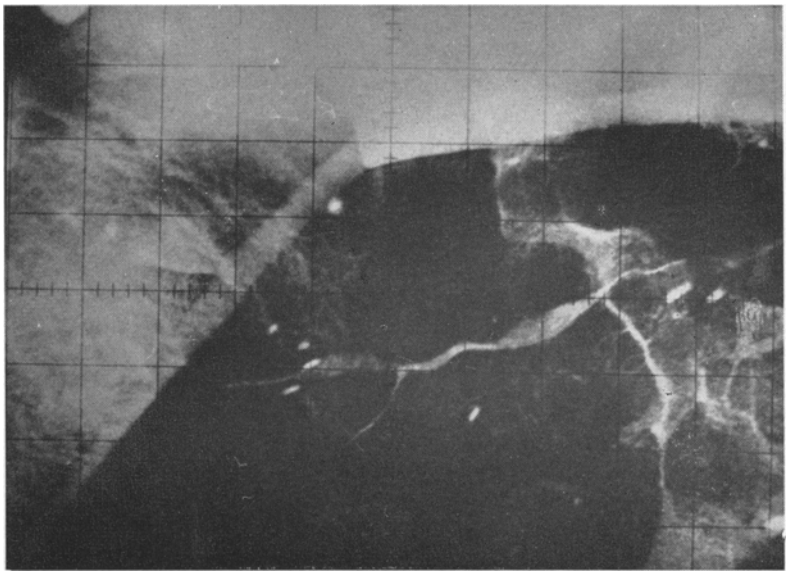

Figure 2 Scanning transmission micrograph of a very thick region of heavily deformed aluminium. Magnification × 3300.

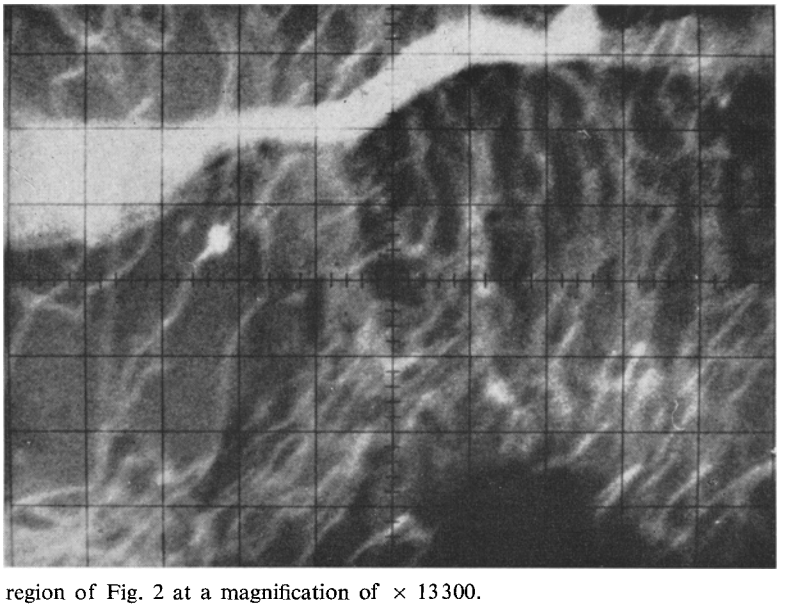

Figure 3 Central region of Fig. 2 at a magnification of × 13300.

where electrons of differing energy cannot be brought to focus in the same place and thus cause a blurring; a loss of image information in the final pictures. As was pointed out by Cosslett in 1968 [7], this resolution limitation is particularly severe for thick specimens as they introduce multiple energy losses and hence lead to a broad energy spectrum in the transmitted beam.

As yet there has been no really convincing experimental proof of whether there is a thickness advantage based on observations under identical diffraction conditions. However, as an example of what is thought to be increased penetration the aluminium crystal in Fig. 2 was too thick to see through at 100 kV in a normal transmission electron microscope. It was imaged at 80 kV in the transmission scanning mode merely by subtracting the d.c. level. The central region of Fig. 2 is shown at higher magnification in Fig. 3. Both pictures are slightly blurred because of the relatively large probe size and the difficulty in focusing with the instrument used.

It is not obvious why there should be a thickness advantage of the TSEM over the CEM, as the principle of reciprocity would suggest that the chromatic aberrations in the illuminating system of the TSEM are equivalent to those of the imaging system of the CEM. The explanation probably lies in the relation between the position in the system chain at which the chromatic aberrations are introduced and that at which the energy losses are suffered. In both instruments the electron beam suffers aberrations in the illuminating system prior to reaching the specimen which then introduces further achromatism to the beam because of randomly suffered energy losses. In the CEM the system of imaging lenses causes yet more achromatism, whereas in the TSEM no more are introduced, and so for the same limit of resolution set by the aberrations and their effect on the multiple energy lost electrons, thicker specimens may be examined.

If it could be proved that it is possible to examine thicker materials in the transmission scanning mode than in the conventional microscope for a given voltage, many of the advantages of a high voltage microscope would be available on existing 100 kV machines.

The third group of applications make use of the very fine probe size that is obtainable in the scanning system. As yet very little has been done in this field but a number of extensions of existing work can be envisaged. In the selected area diffraction mode of a CEM the volume of crystal that contributes to the diffraction pattern may be typically 2.5×10^6 nm^3 – about 10^7 atoms – for a 100Å thick carbon specimen. Using a spot size of 50Å (within the capabilities of scanning attachments to existing 100 kV machines) and a foil of 100Å in thickness, the diffraction pattern is obtained by averaging over only about 10^3 unit cells. This is of some importance in crystal

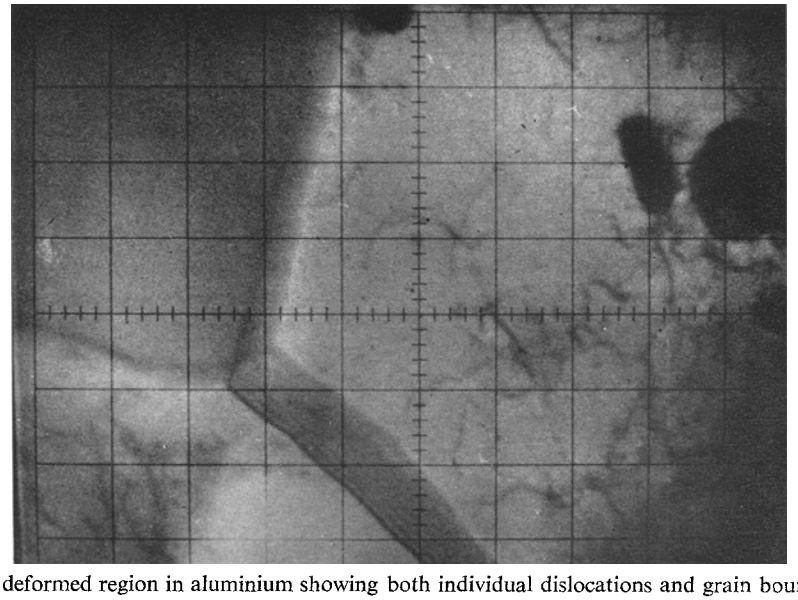

Figure 4 Heavily deformed region in aluminium showing both individual dislocations and grain boundary fringes. Magnification × 13200.

structure research as the radial distribution of nearest neighbours can be calculated from the intensities in the diffraction pattern. It would then be possible to move the probe about the specimen and determine local variations in structure and packing densities over a very large region. A particularly inviting application would be the determination of radial distribution functions from very small areas of amorphous materials, for which no other technique can foreseeably be used.

An associated application of probably more direct usefulness is the measurement of lattice rotations caused by local strains, in the neighbourhood of very small particles or interfaces. Such a study was made by Chapman and Stobbs [8] using a Le Poole lens to form a spot of 2500Å diameter in a conventional 100 kV

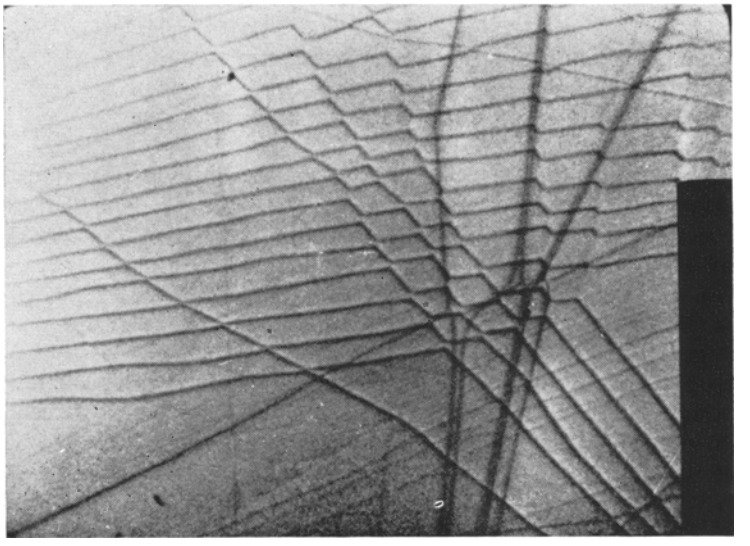

Figure 5 Dislocation network in molybdenite. Magnification × 13300 (Courtesy of R. M. Stern.)

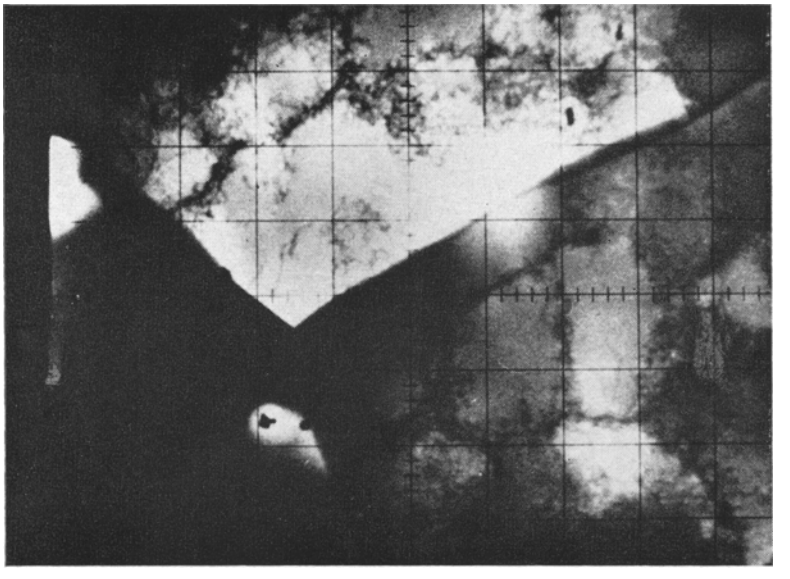

Figure 6 Triple point boundary in aluminium with two of the grains in contrast. Within the two grains can be seen a set of dislocation cells. Magnification × 3300.

machine, in measuring lattice rotations in copper around silica particles 3000Å in diameter. The availability of much smaller probes would extend their technique to a wide range of metallurgical problems involving precipitation, dispersion strengthening, deformation and micro-twinning.

In addition to those uses to which the transmission scanning microscope might be put by virtue of its advantages over a conventional microscope, the machine may be used for straightforward transmission work. As examples of such transmission scanning a number of pictures of different structures have been included. (Figs. 4, 5 and 6.)

As much of the latter part of this article has been written citing possible rather than achieved applications it will be seen that there is plenty of scope for transmission scanning microscopy even for those people having relatively simple pieces of equipment.

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