JOURNAL OF MATERIALS SCIENCE 6 (1971) 213-217

The Generation and Identification of SEM Channelling Patterns from 10 μm Selected Areas

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In a previous note [1] we have briefly described a new method of generating electron channelling patterns (ECPs) from selected areas as small as 10 μm across, on the surface of solid specimens. The ECP, which is the reciprocal analogue of the reflection Kikuchi pattern, can be used to orient, and sometimes to identify, the region selected. The details of the method are now described, and ways of reducing the selected area size still further are proposed. Identification of ECPs is facilitated with a channelling map and the preparation of such maps is described.

1. Introduction

The electron channelling pattern (ECP) obtained in the scanning electron microscope (SEM) is the reciprocal analogue of the reflection Kikuchi pattern. The information in the ECP can be used to determine the orientation of the specimen relative to the optic axis of the SEM. In addition, in certain cases the crystal type and lattice parameter of the specimen may be determined and thus the material identified. The ECP is displayed on the SEM screen and the specimen may easily be tilted to a given orientation, or the pattern photographed for a given specimen tilt. At any time the SEM may be used for examination in other micrographic modes.

In order to obtain the ECP of a small region
(i) the probe conditions of divergence and current must be suitable [2]
(ii) the probe must "rock" about the area of interest. By this we mean that the probe must always be incident on the region of interest, while varying its direction of incidence.

An undistorted ECP is displayed if the directions of incidence of the probe, back projected to a plane normal to the optic axis, form an array exactly similar to that of the corresponding points on the display screen.

The beam must necessarily suffer at least two time-varying deflections in order to rock about a point. Errors are easy to accumulate, and the residual lateral movement of the probe, rather than the probe diameter, sets the limit to the smallest size of selected area. The method which we have described [1] shows the smallest errors of any proposed system.

2. Outline of the Method

Fig. 1 shows the beam suffering two deflections to make it rock about the point of intersection of the optic axis and the specimen. The basis of the method (the "deflection focusing" technique) is to make the second (lower) deflection not by scan coils, but by means of an electron lens. Conjugate points of this lens are:
(*a*) The intersection of the optic axis and the upper scan deflection.
(*b*) The intersection of the optic axis and the specimen surface.
Thus all electrons from point (a) passing through the lens will strike point (b). If point (a) is made the image point of the previous lens, then the beam pencil, as well as the scan, is focused on to the specimen surface. The aperture of the final lens does not define the beam pencil but, depending on the focal length of that lens, limits the total angle of scan. The probe divergence is set by the defining aperture of the system, since the probe is focused. In the case of the "Stereoscan" SEM the defining aperture is just above the plane of lens 2 (numbering from the gun) and referring to

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fig. 1, the total angular divergence is

$$\phi = \frac{d_1 h_2}{h_1} \cdot \frac{1}{S}$$

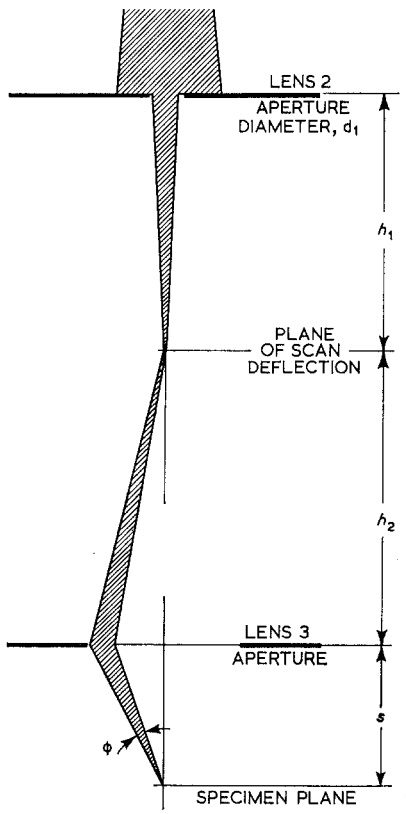


Figure 1 Ray diagram for the deflection-focusing method of selected area scanning.

Experimentally, it is found that patterns are difficult to identify unless the total scan angle exceeds 0.15 radian and the divergence is less than 5×10^{-3} radian. This is achieved by
(i) increasing the size of the final aperture from 100 μm to approximately 2 mm diameter (with the existing aperture changer)
(ii) decreasing the size of the lens 2 aperture from 1 mm to approximately 100 μm diameter (using a new aperture changer).

A probe current of approximately 10^{-9} A is necessary to display good pattern contrast on the screen at reasonable scan rates. The brightness of the tungsten hairpin filament gun is approximately 10^2 A/mm^2/sr (at 20 kV) and with a divergence of 3 m radians, a probe diameter of 1 μm can be achieved in theory. The detail resolved on test specimens is of this order of size. The desired combination of probe diameter and current is achieved by adjusting lens 1.

3. Summary of Instrument Modifications

(*a*) Final aperture increased from 100 μm to 2 mm diameter.
(*b*) Single scan deflection. The lower scan deflection of the usual double-deflection system is bypassed.
(*c*) Lens 2 aperture decreased from 1 mm to 100 μm diameter.
(*d*) Lens 2 image point to be in the plane of the scan deflection. In the "Stereoscan" this requires extending to the range of lens control to lower currents, or removal of the lens pole-piece.

4. Use of the Method

The apertures and lenses are set as described. The specimen is examined at a magnification setting of 500 $\times$ or 1000 $\times$ and with double-deflection scan. The region of interest is brought to a predetermined spot on the screen. On switching to single-deflection scan and a magnification setting of 20$\times$, an image of the specimen again appears on the screen. (In general the rocking point will not be exactly on the specimen surface and a raster will still be traced.) Lens 3 is then adjusted to bring the rocking point into coincidence with the region. The image of the region expands to fill the screen and its channelling pattern appears. The exact setting for lens 2 is determined in the following way: having brought the rocking point and specimen into coincidence as described above, the specimen is re-examined at high magnification with double-deflection scan. Leaving the lens 3 setting and specimen position alone, the image is focused with lens 2. This puts the lens 2 image point in the plane of the scan deflection.

5. Errors in the System

In spite of a probe diameter of 1 μm, it is difficult to obtain ECPs from grains smaller than 10 μm across. This is due to the spherical aberration of the final lens. Fig. 2 shows how a specimen placed at the (near-axis ray) conjugate point is scanned to a width of $2C_s\alpha^3$ as the beam rocks an angle $\pm\alpha$. This can be reduced to $\frac{1}{2}C_s\alpha^3$ if the specimen is placed at the position of the circle of confusion which would be formed by a wide angle beam focused by the lens with semi-angular aperture α. This is approximately $\frac{3}{4}C_s\alpha^2$ above the conjugate point.

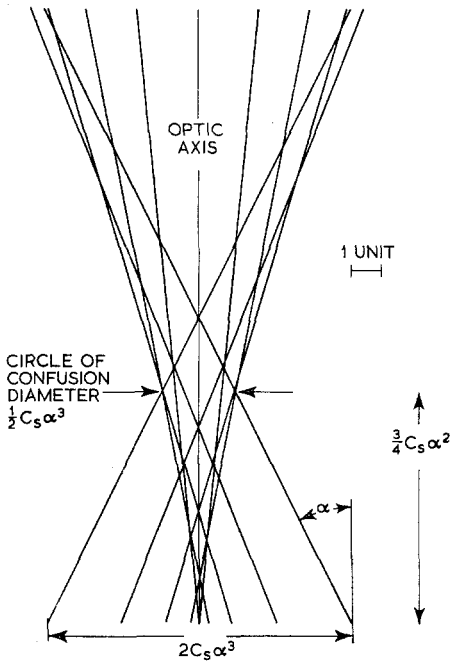


Figure 2 The effect of spherical aberration. The diagram is drawn for $\alpha = \frac{1}{2}$ radian and $C_s = 40$ units.

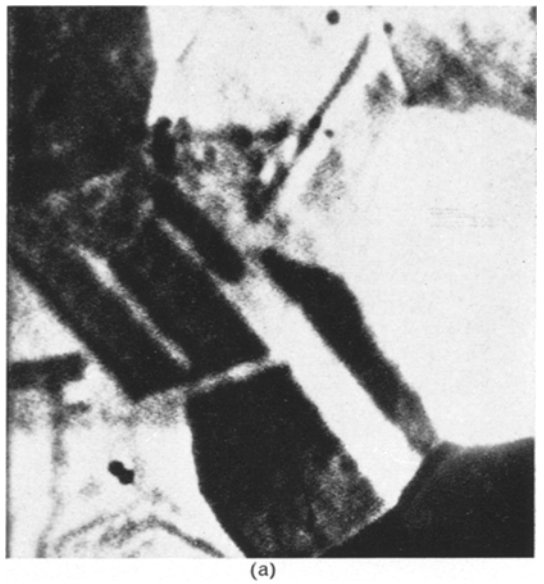
It should be possible to correct for the spherical aberration by one of two methods:
(i) lateral shift coils placed in any plane other than that of the scan deflection, and fed with a suitable waveform.
(ii) variation of the focal length of lens 3 with the off-axis angle (dynamic focusing)
These are being investigated.

6. Results

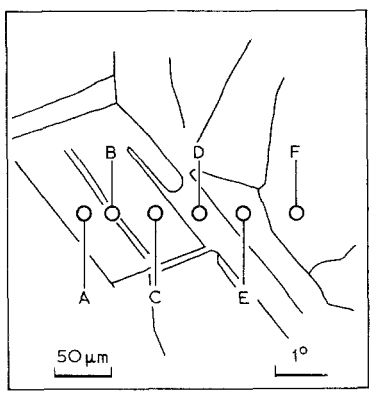
Fig. 3 shows a micrograph of grains and twins on the surface of an annealed copper specimen. At this magnification (1000×) the probe is essentially at normal incidence over the whole picture and each grain shows the shade of the centre of its channelling pattern. The pattern from twin "B", which is 10 μm wide, is also shown.

7. Channelling Maps

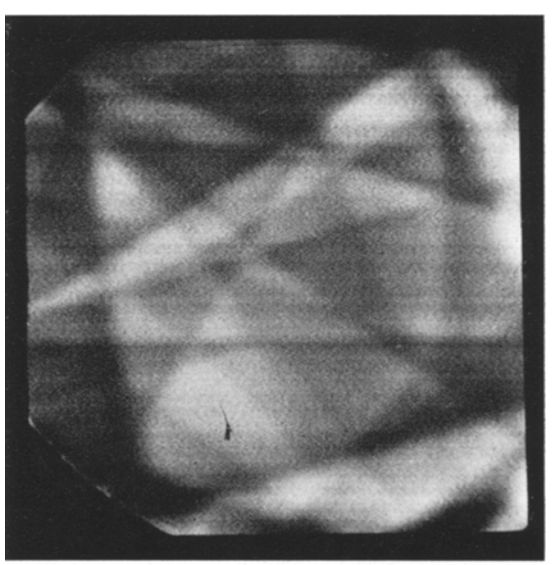
The patterns obtained, unless they happen to contain low-index poles, are most easily identified by visual comparison with a map of patterns for all possible orientations (i.e. the stereographic triangle for cubic materials). The preparation of such a map is rendered less tedious if large total angles of scan are available. The deflection focusing technique can be used



(a)

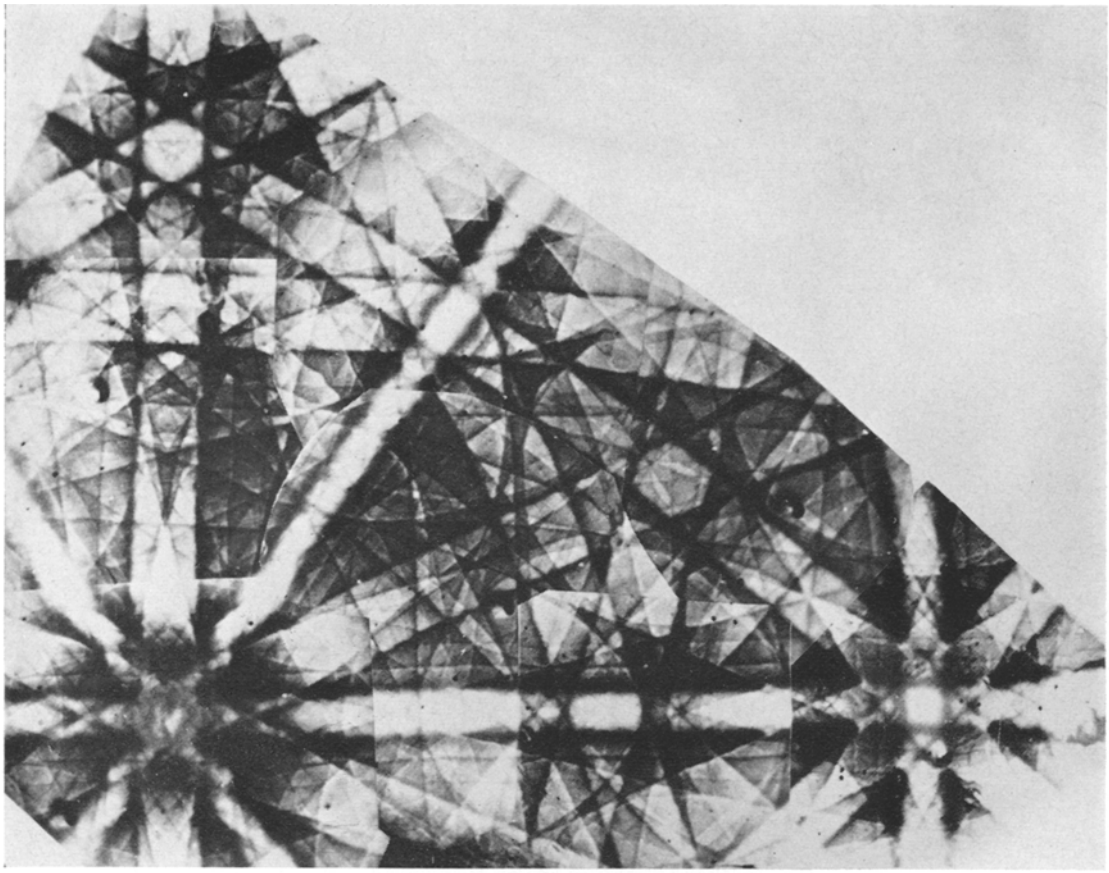


(b)

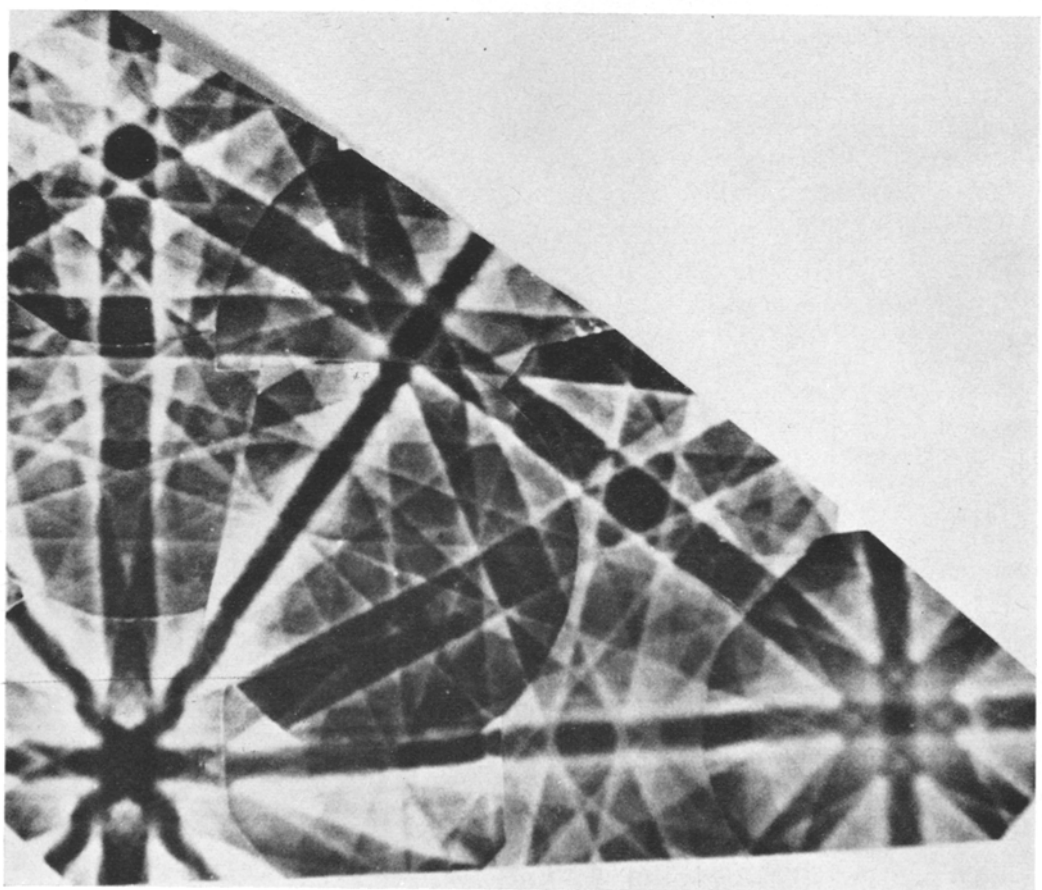


(c)

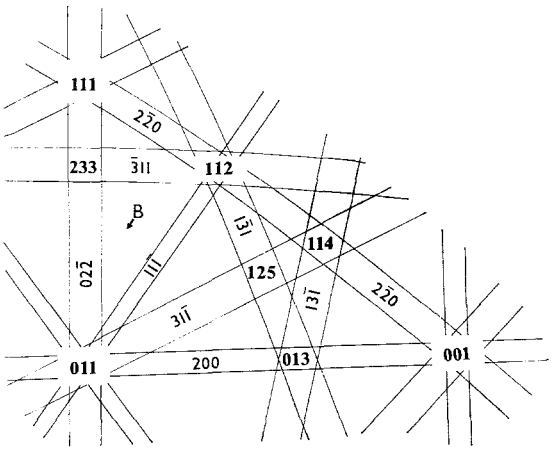
Figure 3 (a) Grains and twins in an annealed copper polycrystal. (b) Identification of the grains. (c) Channelling pattern from twin B. Taken at normal incidence with backscattered electrons, 20 kV.



4(a)



4(c)



4(b)

Figure 4 (a) Channelling map for Copper, back-scattered electrons, 20 kV. (b) Indices of the major poles and bands of the maps. B indicates the orientation of the normal to twin B, and the arrow shows the direction of the top of the channelling pattern. (c) Channelling map for Copper, absorbed electrons (specimen current), 30 kV.

for this purpose. In double-deflection, the maximum total scan angle is about 20°, but this can be increased to over 30° by using a single deflection and running the final lens at maximum excitation. In order to keep the probe divergence low, lens 2 is adjusted so that its image point lies above lens 3, a distance equal to the focal length of lens 3. The probe is thus collimated and a divergence of 2×10^{-3} radians can be achieved with a total scan angle of $\sim 30°$.

The specimens used to prepare maps are preferably hemispherical, single crystals. Any desired orientation can then be obtained at normal mean incidence and the greatest deviation from normal incidence over the entire map will be rather more than half the total scan angle or 15°. Channelling contrast changes with tilt and identification is most reliable if specimens are examined at normal mean incidence and compared with maps taken in the same way.

The spacing between the pairs of channelling lines is twice the Bragg angle for the planes concerned. This changes with electron wavelength and with lattice parameter. The details of the map will therefore differ for all materials and accelerating potentials used. A large number of maps must therefore be prepared. The contrast is also reversed between the images from emitted electrons and from specimen current, but as the reversal is exact for specimens at normal incidence, photographic methods can be used to prepare the complementary map.

Two maps for copper are illustrated in fig. 4, together with the indices of the major poles and bands. A montage of only nine photographs was necessary to cover the entire stereographic triangle. The position of the pattern in fig. 3 is indicated. For orienting materials for which no map is available, it is either necessary to tilt to a recognisable low-index pole, or to attempt recognition of the bands visible. This might be achieved by measurement of intersection angles or by noting the movement of lines for an incremental change in accelerating potential [3].

8. Conclusions

A novel method of scanning has been developed to enable SEM channelling patterns to be generated from 10 μm diameter regions on solid specimens. The limit of size is set by spherical aberration. The patterns obtained are identified by channelling maps, which are most easily prepared by using a variation of the same scanning method.

Acknowledgements

We thank Dr G. R. Booker and Dr D. C. Joy for helpful discussions, and Messrs D. Kynaston and A. D. G. Stewart of Cambridge Scientific Instruments Ltd for their co-operation. Thework was supported by the Science Research Council, the National Research Council of Canada and the English Electric Co Ltd.

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Received 16 November 1970 and accepted 6 January 1971.