Scanning Microscopy

Volume 1 | Number 2 Article 2

2-20-1987

On-Line Analysis of Electron Back Scatter Diffraction Patterns. I. Texture Analysis of Zone Refined Polysilicon

D. J. Dingley
University of Bristol

M. Longden
University of Bristol

J. Weinbren
University of Bristol

J. Alderman
Plessey Research (Caswell) Ltd.

Follow this and additional works at: https://digitalcommons.usu.edu/microscopy

Part of the Life Sciences Commons

Recommended Citation
Dingley, D. J.; Longden, M.; Weinbren, J.; and Alderman, J. (1987) "On-Line Analysis of Electron Back Scatter Diffraction Patterns. I. Texture Analysis of Zone Refined Polysilicon," *Scanning Microscopy*: Vol. 1 : No. 2 , Article 2.
Available at: https://digitalcommons.usu.edu/microscopy/vol1/iss2/2

This Article is brought to you for free and open access by the Western Dairy Center at DigitalCommons@USU. It has been accepted for inclusion in Scanning Microscopy by an authorized administrator of DigitalCommons@USU. For more information, please contact digitalcommons@usu.edu.

Scanning Microscopy, Vol. 1, No. 2, 1987 (Pages 451-456) 0891-7035/87$3.00+.00
Scanning Microscopy International, Chicago (AMF O'Hare), IL 60666 USA

ON-LINE ANALYSIS OF ELECTRON BACK SCATTER DIFFRACTION PATTERNS. I. TEXTURE ANALYSIS OF ZONE REFINED POLYSILICON

D J Dingley*, M Longden, J Weinbren and J Alderman+

H.H.Wills Physics Laboratory, University of Bristol
Bristol BS8 1TL, England

+Allen Clark Research Centre, Plessey Research (Caswell) Ltd.
Caswell, Northants, NN12 8EQ, England

(Received for publication March 22, 1985, and in revised form February 20, 1987)

Abstract

A technique has been developed for determining crystal orientations on-line from bulk polycrystalline materials using wide angle back scatter electron diffraction patterns. The patterns were imaged on a phosphor screen and viewed using a low light level television camera. A computer generated cursor superimposed on the diffraction pattern, permitted the coordinates of zone axes to be determined. These were interpreted by the computer to yield the crystal orientation. The accuracy of the technique for absolute orientation was shown to be of the order 1° and the precision for relative orientation better than 0.5°. The technique was used to investigate texture and nearest neighbour orientation relationships in polysilicon, recrystallised using a graphite strip heater technique. It was shown that the orientations become less random as the recrystallisation front proceeded along the specimen.

KEY WORDS: Electron diffraction, backscattering, diffraction patterns, imaging diffraction patterns, on-line analysis, crystal textures, grain orientations, orientation distribution, grain growth, polysilicon.

*Address for Correspondence:
D.J.Dingley, H.H.Wills Physics Laboratory,
University of Bristol, Tyndall Ave.
Bristol, BS8 1TL, England Tel.0272 303030

Introduction

The first applications of the technique developed by Venables and Harland (8) for obtaining electron back scatter diffraction patterns (EBSPs) in the scanning electron microscope, were for the determination of crystal orientation, Venables and co-workers, (7-11). The highest accuracy achieved was 0.5 degrees. These diffraction patterns, which are in effect wide angle back scatter Kikuchi patterns, were imaged on a phosphor screen placed in the vacuum of the microscope specimen chamber. They were observed by eye or using an image intensifying micro-channel plate. The orientation of a crystal was obtained by analysis of the diffraction pattern recorded by photographing the phosphor screen. This paper describes a method of determining the orientations on-line using a low light level television camera to image the pattern and a microcomputer to interrogate it.

Crystal texture determination is still carried out mostly using x-ray diffraction techniques. However this method does not provide complete information. It is based on determining the directions in which a particular reflection from the crystals is diffracted. The directions are recorded with respect to a fixed axis system and plotted in stereographic projection to form the texture map, where any clustering of the selected reflection indicates a preferred orientation in the sample. The method only allows for the distribution of one crystal reflection to be investigated at a time and crystals which direct the reflection close to the specimen surface are not detected. No information is provided as regards orientation relationships between neighbouring grains and data from two phase materials may be ambiguous. Orientation determination from EBSPs on the other hand, is based on measuring the absolute orientation of each grain from the positions of zone axes in the diffraction pattern. All grains in the specimen can thus, in principle, be sampled. Two or multiphase materials can be investigated and nearest neighbour orientation relationships can be determined. The smallest areas from which EBSPs have been obtained are less than 100nm, Harland et al. (7). The technique can thus be applied to the investigation of textures in finely grained material.

One such material that is receiving considerable attention at present is polysilicon. Polysilicon films are usually deposited on silicon oxide at various stages during the fabricaton of integrated circuits (4). The films may be grown by evaporation or by low pressure chemical vapour deposition (LPCVD). In both cases the films are fine grained and highly strained. Subsequent annealing of the films in a furnace is current industrial practice. However, several new techniques for the annealing process are being investigated including, laser annealing, electron beam annealing and graphite strip heating, Chen et al. (2), Geis et al. (5). The recrystallised silicon however, may remain fine grained and not completely free of strain. A partially satisfactory result is obtained if the polysilicon film becomes highly textured such that the preferred crystal direction normal to the surface is the same as the initial substrate and that the interfaces between the crystals are low angle boundaries Geis et al. (6).

This paper describes the experimental procedure and results obtained for determining textures and nearest neighbour orientation relationships in polysilicon recrystallised using the graphite strip heater technique.

Experimental

A description of the method for determining EBSPs has been given previously, (see refs. 3 and 8 for example) and will not be repeated here. The experimental arrangement adopted to obtain the patterns is shown in Fig.1 and was used here on a JEOL 840 SEM. The imaging screen was a 35mm diameter Yttrium Aluminium garnet single crystal (YAG:Ce). It was mounted in a brass tube fixed to the side port of the microscope normally used for attaching the optical microscope used in microanalysis. The screen was positioned 30 mm from the specimen giving a capture angle of approximately 60°. It was coated with 10nm of aluminium to prevent charging and could be used over an energy range of back scattered electrons from 40keV to 4keV at beam currents 0.1nA to 3nA. The low light level television camera used was a Mullard Intensified Silicon Intensifed Target system (ISIT camera). The camera was at intermediate gain for the range of current values quoted above. Below 0.1 nA and at the lowest voltages image enhancement was necessary to improve the signal to noise ratio. An Arlunya image store with temporal filtering was used to achieve this.

A BBC microcomputer was interfaced to the television camera so that the computer graphics could be displayed superimposed on the image of the diffraction pattern. A movable cursor was thus generated and positioned successively at different zone axes of the diffraction pattern so allowing their coordinates with respect to the screen coordinates to be determined. From this the crystal orientation could be computed. The computer program and data acquisition system is described below.

Polysilicon test specimens were provided by Plessey Research (Caswell) Ltd. They measured 10mm square and consisted of a 0.5 micron thick polysilicon film deposited on silicon oxide. They had been annealed using the graphite strip heater technique (5). In this method an electrically heated graphite rod is moved slowly and continuously above the surface of the silicon causing the silicon beneath it to melt. The silicon resolidifies when the rod moves on. The intention is that the previously melted and solidified silicon would act as a seed crystal and so force the solidifying silicon into a single orientation. An alternative employing seeding to the substrate or the use of entrainment can also be employed (5,6). The technique relies on correct power supply and velocity control of the graphite rod.

Previous attempts to measure the orientations of recrystallised grains utilised the shapes of pits etched into the surface using potassium hydroxide, and delineation of defects by Secco etch. For a silicon film orientated such that the surface normal is [100], the edges of the pits lie parallel to 110 directions so forming a square. Distortions of the shapes of the etch pits from square denotes deviation of the silicon film from the [100] orientation (1). Fig.2 is a scanning micrograph of the surface of one of the specimens examined. The etch pits are clearly distorted from square. The grain boundaries of the polysilicon are also clearly visible. It is seen that the grain size is 2-10 microns and that some of the grain boundaries appear to stop within a grain. Grain orientations in this investigation were determined on-line using the EBSP technique. They were determined close to the start of the recrystallisation point and at different distances along the recrystallisation path. Absolute grain orientations were determined, from which texture maps were drawn and nearest neighbour orientation relationships which revealed any trend towards single crystal orientation, were calculated. The results of the analyses are given in Table 1 and Fig.4.

Acquisition of data for orientation determination

As mentioned above the coordinates of zone axes in the diffraction pattern, imaged using the television camera, were determined with respect to the screen coordinate system through superposition on the image of a computer generated cursor. A calibration procedure was thus needed to relate the screen coordinates of zone axes so determined, to a coordinate system centred on the specimen at the point of incidence of the electron beam, and orientated such that one axis was parallel to the specimen surface normal and another parallel to a horizontal line drawn in the specimen surface.

Thus prior to viewing and interrogating the diffraction patterns from the polysilicon, a calibration specimen consisting of a polished single crystal wafer with surface normal parallel to [001], was mounted in the microscope on a chamfered specimen stub. A cleaved edge of the crystal, which intersected the crystal surface along [110], was made to lie parallel to the horizontal direction. The arrangement was such that with the stage controls set to zero tilt and rotate, the wafer faced the viewing screen and the [001] direction was inclined at an angle of 19.4°

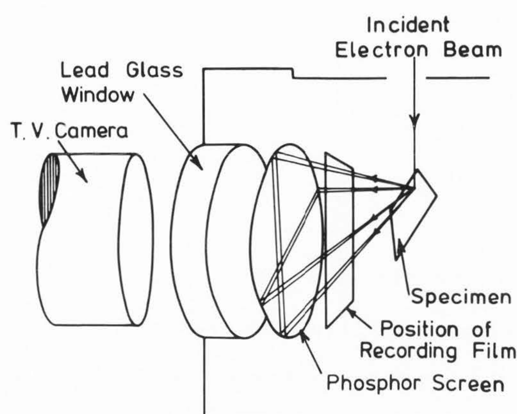

Fig 1 Schematic diagram illustrating experimental arrangement for imaging EBSPs.

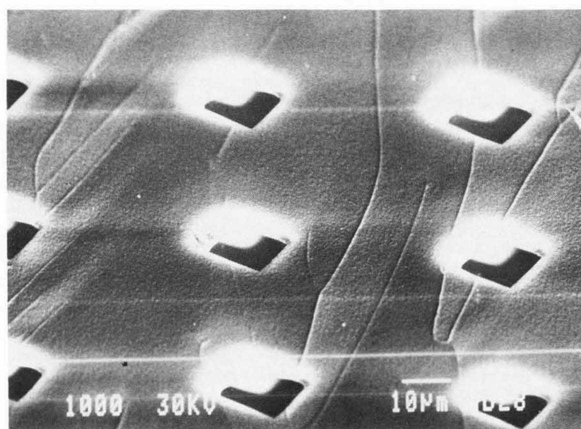

Fig 2 SEM micrograph for polysilicon taken 1 mm from origin of recrystallisation.

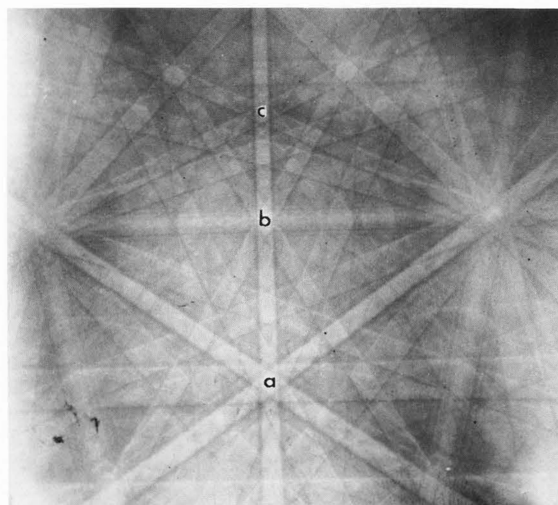

Fig 3 EBSP from silicon. Zone axes a,b,c [111], [112] and [114] respectively.

with respect to the incident beam direction. The calibration crystal was thus set at an orientation such that the [114] crystal direction was normal to the viewing screen and the [111] direction was directed 35° vertically below it. The position of the [114] zone axis thus defined the pattern centre on the viewing screen and the line at right angles to the line joining [114] to [111], defined the horizontal axis. The x,y coordinates of the coordinate system centred on the specimen were the same as those of the pattern centre on the viewing screen. The z coordinate was equal to the specimen to film distance. The x axis of the specimen coordinate system was parallel to the horizontal axis on the viewing screen and the z axis lay along the direction 19.4° vetically above the screen normal, i.e. parallel to [001]. The specimen to screen distance was determined by measuring the distance, L, between the [114] and [111] zone axes as viewed on the screen and using the formula:

$$z=L/(\tan(35)) \quad (1)$$

where 35° is the angle between [114] and [111] directions in silicon. This method gave the pattern centre and specimen to film distance to an accuracy of 0.5%. The coordinates of the [114] and [111] zone axes and value of the specimen to screen distance were stored in the computer memory. The polysilicon specimens to be examined were then mounted on similar stubs to that used for the calibration specimen. In order to locate a specimen so that the gain whose orientation was to be determined was at the origin of the specimen coordinate system, it was necessary to position the specimen at the same working height as used during calibration and to set the incident beam at the same lateral settings. Maintenance of the same lateral setting was achieved by working at high magnification and switching off the electromagnetic shift controls. Constancy in specimen height was achieved by maintaining the objective lens setting at a fixed value, and focusing the specimen using the specimen height shift control. The error introduced by these constraints was less than the 0.5% error in determining the pattern centre.

With a polysilicon specimen thus mounted the orientation of any crystal was determined by obtaining a diffraction pattern from it and locating the computer generated cursor, first on a [112] zone axis and next on either the adjacent [114] or [111] zone axes. These zones are quite distinct and recognisable(Fig.3) and were chosen for this reason and because as there are sixteen possible 112 zone axes it was certain that at least one of them would appear in the portion of the diffraction pattern viewed and thus also either a [111] or [114] zone axis. The coordinates for each point were transferred to the computer memory and the orientation of the crystal obtained without any additional information needed as input. The time required to position the electron probe, locate the zone axes using the computer generated cursor, and compute the crystal orientation was 20-30s.

Computer program procedure

The following description outlines the procedure by which the orientation was determined. The measured screen coordinates of the two selected zone axes, [112] and [114], or [112] and [111], were transformed to the specimen coordinate system, defined above. This permitted the vectors defining the positions of the zone axes to be obtained in terms of the specimen coordinate system. The scalar product of the two vectors was formed, the inverse cosine giving the angle between them. If the measured angle was equal to 19.4° the second zone axis chosen had been [111] as 19.4° is the angle between [112] and [111]. Conversely if the angle was 16.4°, the second zone axes was [114]. The first and second zone axes were so indexed. The vector product of these two vectors was then obtained so defining the vector normal to both of them. This vector is the [110] crystal direction. The sign chosen for the [110] direction depended on the relative positions of the [114] and [111] axes across the [112] zone axis. A right handed axes system was selected. The correspondence between the three crystal directions [112], [111] (or [114]) and [110] and their vectors as defined in the specimen coordinate system were then known. The crystal indices of any other vector in the specimen coordinate system could thus be determined. For example, the crystal direction parallel to the normal to the screen was found by first calculating the angles between the three known vectors and the normal. Let these be a,b c and let the crystal indices of the normal be hkl. Three simultaneous equations can be constructed out of the scalar products of the three crystal directions and hkl. They are:

$$1h+1k+2l/\mathrm{sqr}(6)=\cos(a) \quad (2)$$
$$1h+1k+1l/\mathrm{sqr}(3)=\cos(b) \quad (3)$$
$$1h-1k+0l/\mathrm{sqr}(2)=\cos(c) \quad (4)$$

from which the values of hkl can be found. In this way the crystal directions parallel to the three axes defining the specimen coordinate system can be found. This defines the crystal orientation.

These operations were carried out automatically after the second set of coordinates from each diffraction pattern were acquired by the computer. As a check the screen positions of eight additional major zone axes of the crystal were calculated using the just calculated orientation of the crystal as starting data. The positions were plotted on the video monitor allowing visual inspection to judge whether the plotted points matched the actual crystal zone axes. If there were any discrepancy the orientation was redetermined.

When required the data was transferred to a program which plotted the orientations in stereographic projection or alternatively, calculated nearest neighbour orientations. Nearest neighbour orientations are described by an axis angle pair relationship. If R is a rotation matrix which maps one set of orthogonal axes on to another, an infinite set of points lying on a straight line through the origin of both systems remain invariant. This is the axis of rotation. Regarding R as an operator, $\underset{\sim}{x}$ is an eigen vector of R such that $R\underset{\sim}{x}=\underset{\sim}{x}$.

If I is now the identity matrix

$$(R-I)\underset{\sim}{x}=0 \quad (5)$$

where 0 is the null vector. The three rows of the matrix (R-I) can be described by vectors ui, vi wi. Thus equation 5) is equivalent to three vector equations

$$ui.x=0 \quad vi.x=0 \quad wi.x=0 \quad (6)$$

Hence (R-I) consists of three vectors all lying in a plane perpendicular to the rotation axis. The rotation axis is thus found from the vector product of any two rows. The rotation matrix R was found from the orientation matrices S1, S2 of neighbouring grains through the matrix product

$$R=S1 \times S2 \quad (7)$$

The angle of rotation was obtained from the trace of the rotation matrix by the formula

$$tr(r)=1+\cos\theta \quad (8)$$

where θ is the angle of rotation.

Owing to the crystal symmetry of the cubic system there are twenty four alternative ways of describing the orientation relationship whilst retaining the same sense. The particular choice of axis is arbitrary. The computer was thus programmed to search through all twenty four variants and select that which gave the lowest angle of rotation.

Results

The results obtained from examination of the recrystallised polysilicon are listed in table 1 and displayed in Figs 4a and b. In table 1 are listed the nearest neighbour orientation relationships. The axes are written in terms of direction cosines as they do not in general correspond to rational indices. The absolute orientations are plotted in the form of a stereographic projection in Fig.4. Fig.4a shows the <100> poles of grains close to the start of recrystallisation whilst Fig.4b shows the same poles for grains 1mm distant from the start of recrystallisation.

Discussion

The accuracy of the above technique was tested by removing and replacing the calibration specimen in the microscope and determining its orientation, and also by determining the orientation relationship across an annealing twin boundary, the orientation of which was known. The results on repeat measurement of the orientation of the calibration specimen were always within the error of determining the pattern centre, i.e. better than 1.0°. The precision in relative orientation measurement was better than 0.5°. The method is thus better than the standard Laue x-ray

diffraction technique for orientation determination, but not quite as precise as when using Kikuchi patterns in transmission electron microscopy.

The speed of determination of orientations was not high compared with standard x-ray texture measurements, but satisfactory amounts of data could be accumulated in a day's work, 300-500 grains, to obtain reasonable statistics. An advantage in this respect however is that only one set of data had to be acquired to enable texture maps of any desired type, i.e. 100, 111, 110 etc. to be plotted, as is shown in Fig.4.

The use made of the technique in this paper for the determination of orientations in polysilicon, has shown that the additional information acquired, namely the distribution of absolute orientations of individual grains and the determination of nearest neighbour orientation relationships, is a significant advantage over texture determination alone. It can thus be seen that the recrystallisation process began with the formation of a large number of small grains which differed markedly in orientation from each other. As the recrystallisation front moved forward the number of grains decreased, the orientations present reduced in number and the orientations of nearest neighbours differed by only small angles. The significance of these results as regards the recrystallisation process will be discussed in a later paper. It should be noted that attempts to obtain electron channelling patterns, SACPs, from this material were only partly successful. Whereas SACPs could be obtained from the larger grains well removed from the origin of the recrystallisation process. They could not be obtained from the smaller grains.

Conclusions

It has been shown that using a microcomputer to determine on-line the coordinates of zone axes in electron backscatter diffraction patterns, provided a direct and efficient method for determining crystal orientations. It has been demonstrated that this information can be obtained from fine grained polycrystalline material such as recrystallised polysilicon and that the information obtained can be used to show spatial changes in recrystallisation behaviour as well as the overall texture of the material.

References

1.Bezjiam KA, Smith H, Carter J, Geiss M (1982) An etch pit technique for analyzing crystallographic orientation in silicon films. J. Electrochem.Soc. 129,1848-1850.

2.Chen CK, Geis MW, Tsaur B-Y, Chapman RL Fan JCC (1984) Topographic Imperfections in zone melting recrystallised Si Films on SiO_2. J. Electrochem. Soc. 131,1707-1711.

3.Dingley DJ (1984) Diffraction from sub-micron areas using electron backscattering in a scanning electron microscope. Scanning Electron Microsc. 1984; II: 569-575.

Table 1

Orientation between nearest neighbour grains expressed as axis angle pairs: a) for grains close to start of recrystallisation; b) for grains 1mm from start of recrystallisation.

Grain pair	Axes	Angle
	(a)	
1,2	-.015, .248, .967	< 0.5°
2,3	.070, -.969, .233	< 0.5°
3,4	.754, .544, .367	46.5°
4,5	-.094, -.605, -.790	4.8°
5,6	-.516, -.327, .792	44.7°
6,7	-.235, -.708, .665	37.8°
7,8	-.093, -.296, .950	39.4°
8,9	.250, -.014, .968	8.0°
9,10	-.757, -.537, -.372	49.3°
10,11	.793, .547, .392	45.2°
	(b)	
20.21	-.429, -.902, -.025	0.5°
21,22	-.915, -.331, -.229	0.5°
22,23	.445, .441, .778	0.5°
24,25	.543, -.088, .834	0.5°
25,26	.181, .769, .612	0.5°
26,27	.778, .465, .421	0.5°
27,28	.251, .885, .391	0.5°
28,29	.940, .196, .278	0.5°

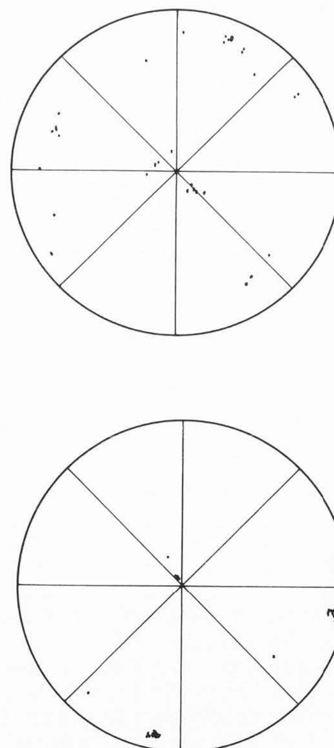

Fig 4 Stereographic projection of <100> axes of grains from polysilicon.
(a) for grains close to recrystallisation point.
(b) for grains 1 mm from recrystallisation point.

4. Gat A, Gemberg L, Gibbons JF (1978). Use of scanning cwKr laser to obtain diffusion-free annnealing of B-implanted silicon. Appl. Phys. Lett. 33, 389-391.
5.Geis MW, Smith Henry I,Tsaur B-Y, Fan JCC, Silversmith DJ, Mountain RW (1982) Zone melting Recrystallisation of Si Films with a moveable -strip heater oven. J. Electrochem. Soc. 129, 2812-2818.
6.Geis MW, Smith Henry I, Silversmith DJ, Mountain RW (1983) Solidification-front modulation to entrain subboundaries in Zone-melting recrystallisation of Si on SiO_2. J.Electrochem. Soc. 130, 1178-1183.
7.Harland CJ, Akhter P, Venables JA (1981) Accurate microcrystallography at high spatial resolution using EBSP in a field emission gun SEM, J.Phys. E. 14, 175-182.
8.Venables JA and Harland CJ (1973) Electron backscatter patterns- a new technique for obtaining crystallographic information in the SEM. Phil. Mag. 27, 1193-1200.
9.Venables JA, Harland CJ Akhter P (1976) Accurate microcrystallography in the SEM. In: Proc. 38th annual meeting EMSA, Claitors Publ. Div., Baton Rouge, LA 184-187.
10.Venables JA, Harland CJ, Bin-Jaya R (1976) Crystallographic orientation determination in the SEM using electron backscatter diffraction patterns and channel plates. In: Developments in electron microscopy and analysis, Academic Press London 101-104.
11.Venables JA, Bin-Jaya R (1977) Accurate microcrystallography using EBSPs. Phil. Mag. 35, 1317-1321.

Discussion with Reviewers

D.L.Davidson: Can you justify a little more the claim that the error in pattern orientation is 0.5%?

Authors: The error in determining the orientation arises from four causes: 1) the determination of the pattern centre and specimen to screen distance, 2) error in positioning the specimen in the specimen holder, 3) error in locating the zone axis in the diffraction pattern, 4) non linearity in the television image. The first two errors were checked by first inserting the calibration specimen in the microscope and determining the pattern centre and specimen to film distance. It was then withdrawn from the microscope, reinserted and its orientation determined as described. If there were no error in pattern centre, specimen to film distance or specimen location, then the experimentally determined orientation would be exactly the same as the input orientation of the calibration specimen. Repeated measurements of this kind gave an actual error between measured and known orientation of 1°. This is the absolute error in determining orientation. For relative orientations between adjacent grains the error must be smaller than this as the location error is not involved, the location of the specimen being the same for both grains. The errors that now have to be considered are those listed 3 and 4 above. The typical angle between two Kikuchi lines is 1 to 2 degrees. Hence there is no difficulty in locating the cursor to within 0.25°. The error in the non-linearity of the TV screen turned out to be negligible. Hence, as four points only are needed to determine the relative orientation of two grains, the error in relative orientation is of the order 0.5°.

M.Brunner: What is the smallest angular range of the EBSP to be recorded at any orientation to include all zone axes necessary for computation?

Authors: The algorithm used for computing the orientation needs only the location of a single [112] zone axis and any other point on the [110] Kikuchi band passing through it. Hence the method needs at least one [112] zone axis to appear on screen. If the angular range of the screen is 71° then at least one [112] must appear.

M.Brunner: Do you think that a fully automated orientation measurement could be developed by using some kind of pattern recognition in the computer?

Authors: We have attempted to make measurements along these lines using an Arlunya image store coupled to a VAX computer, but without success. However, it is not inconceivable that eventually such a system may be developed.

D.L.Davidson: Does there seem to be any physical explanation for the random orientations of Fig.4a to be regrouped as shown in Fig.4b?

Authors: What is happening as the recrystallisation front moves forward is the high angle grain boundaries become eliminated. The exact way in which this occurs is uncertain and the explanation of which is the long term objective of the research. The answer to this question must therefore be deferred until further experimentation is complete.