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BAND POSITIONS USED FOR ON-LINE CRYSTALLOGRAPHIC ORIENTATION  
DETERMINATION FROM ELECTRON BACK SCATTERING PATTERNS

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Abstract

A computer procedure for on-line analysis of electron back scattering patterns (EBSP) has been developed. An experimental EBSP is computer recorded and displayed on a computer monitor. The user identifies the positions of at least two bands in the EBSP with a cursor. Based on this input the computer calculates possible crystallographic orientations. The corresponding EBSPs are simulated and superimposed on the experimental EBSP. The correct crystallographic orientation is determined from a comparison between the experimental and simulated EBSPs. Typically, the analysis takes a 10-30 seconds per pattern. Advantages with the present procedure are that it can be applied for any crystal symmetry, that it requires no knowledge about electron diffraction maps, that it can be used for EBSPs with relatively low contrast, and that the indexing is very precise. For relative orientation measurements the accuracy is found to be within range  $0.05^{\circ}$ - $0.20^{\circ}$ , whereas, for repeated measurements of a given grain after complete remounting of sample and EBSP equipment, it was determined to be  $0.5^{\circ}$ . Furthermore, the procedure facilitates fully automatic pattern recognition.

KEY WORDS: Electron back scattering patterns, computer procedure, crystallographic orientation, on-line analysis, backscattered electrons, lattice plane directions, electron diffraction, texture pole figures, indexing procedure.

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Introduction

It is well established that electron back scattering patterns (EBSP) can be observed on a fluorescent screen placed in the specimen chamber of a scanning electron microscope (SEM) when the microscope is operated in the spot mode and the glancing incidence angle is large (typically  $70^{\circ}$ ) (Alam et al., 1954, Venables and Harland, 1973, Venables and bin-Jaya, 1977, Dingley 1981, 1984, 1988, Dingley and Baba-Kishi, 1990). The crystallographic orientation of selected grains or areas down to  $\sim 0.5 \mu\text{m}$  diameter can be determined in this way. Experimental investigations have already shown that the technique is very powerful for many types of investigations; for example, studies of texture-microstructure relationships, local texture variations and grain boundary geometries (e.g. reviewed by Juul Jensen and Randle (1989) and Dingley and Randle (1991)). An easy indexing procedure for determination of the crystallographic orientation is therefore important and several procedures have been proposed (Dingley et al., 1987, Venables et al., 1976 and Young and Lytton, 1972). The most widely used is the one developed by Dingley et al. (1987). In this method a camera is focussed on the fluorescent screen and the image is transferred to a microcomputer. A computer generated cursor is then superimposed on the image to determine coordinates of specific poles in the pattern. This procedure requires knowledge about the appearance of the actual electron diffraction map. In a recent paper by Juul Jensen and Schmidt (1990), the ideas behind a more user-friendly procedure were outlined. In the present paper the procedure is described in detail, the accuracy is tested and the performance is discussed with reference to the Dingley procedure.

Equipment

A CCD very-low-light TV camera (MERLIN LTC1162F40) is mounted in a JEOL 840 SEM on the rear port to detect EBSPs from a 50 mm diameter phosphor screen. The screen is mounted parallel to the sample surface at a distance of approximately 40 mm (see Fig. 1).

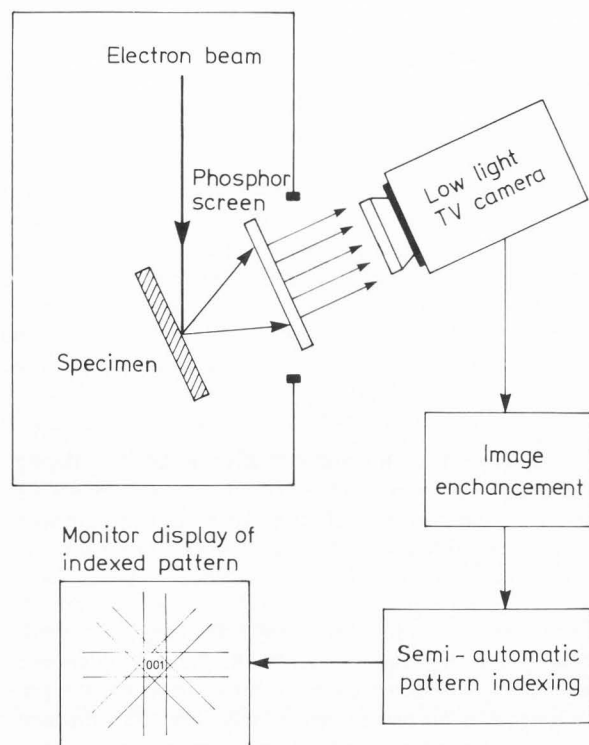


Fig. 1. Schematic diagram illustrating the experimental EBSP set-up.

This is the standard JEOL set-up. However, as can be seen in Fig. 1 the screen is placed rather high relative to the sample. The fraction of backscattered electrons in this direction is not very high and consequently the obtained EBSPs are weak. A lower screen position, for example using the side port in the specimen chamber, would improve the pattern quality significantly (Hjelen, 1990). Samples are mounted in a specially designed pre-tilted specimen holder (inclined at  $20^\circ$  to the incident beam) (see Fig. 2). Besides the sample, a silicon single crystal is mounted in the holder for calibration purposes. A spring arrangement ensures that the sample height and calibration sample height are always identical. From the TV camera the video signal is converted to a real time frame averaged digital image through a (Deben) frame grabber. The digitized EBSP image is accessed and processed by a 20 MHz based PC. A typical example of an EBSP from commercially pure aluminium (of the 1100 series) is shown in Fig. 3.

#### Pattern Interpretation

In the standard method (Dingley 1988) the incident electron beam in the SEM is focussed as a stationary probe on the specimen surface in the sampling point (SP), and the EBSP is a gnomonic projection of those lattice planes

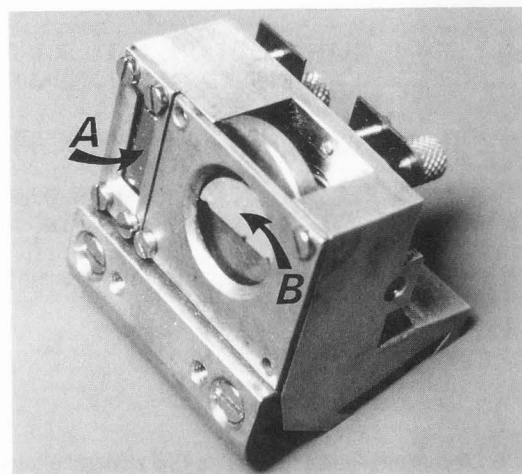


Fig. 2. Sample holder. A - calibration Si single crystal, B - Al sample.

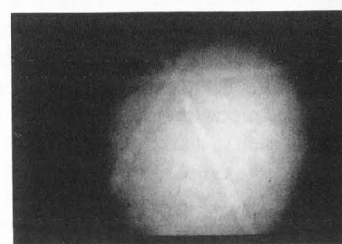


Fig. 3. EBSP from commercially pure Al (1100). The specimen was mechanically polished down to  $1 \mu\text{m}$  diamond paste and finally electropolished in STRUERS A2 electrolyte for 30 seconds at a current of 2A. The acceleration voltage was 20 kV and the beam currents 6 nA.

in SP which diffract the electrons. To determine the crystallographic orientation of the "SP-grain" the EBSP has to be transformed from the pattern coordinate system to the crystal coordinate system (see below). In the present procedure this is done by determining transformation matrices between 3 coordinate systems: f1: The specimen system, f2: The crystal system, f3: The pattern system.

All three systems are Cartesian coordinate systems. The unit axes (x,y,z) for f1 and f3 are defined in Fig. 4, and for f2 the unit axes are chosen along principal crystallographic directions for example  $\langle 100 \rangle$ ,  $\langle 010 \rangle$  and  $\langle 001 \rangle$  in cubic systems. The aim is to determine the orientation or transformation matrix T12 which expresses the crystal orientation in the specimen system. (T12 is often in the literature referred to as the orientation matrix g (e.g. Bunge (1969))).

T12 is determined as:

$$T12 = T32 * T13 \quad (1)$$

where T13 and T32 are the transformation matrices between f1-f3 and f3-f2, respectively.

In the present set-up, the specimen surface is parallel to the phosphor screen. In principle f1 and f3 should therefore be identical and T13 a unit matrix. However, the camera is mounted with the scanning axis not exactly along the x-axis in f1, but slightly rotated ( $\Theta \sim 1^\circ$ ) around the camera axis, i.e.

$$T13 = \begin{pmatrix} \cos \Theta & -\sin \Theta & 0 \\ \sin \Theta & \cos \Theta & 0 \\ 0 & 0 & 1 \end{pmatrix} \quad (2)$$

The transformation matrix T32 is determined by expressing 3 non-coplanar vectors (M) in f3 and in f2 ( $M_3$ ,  $M_2$  respectively)

$$T32 = M_2 * M_3^{-1} \quad (3)$$

To transfer lines in an EBSP into unit vectors ( $M_3$ ) requires determination of the specimen to screen distance (L) and position of the pattern center (PC) (see Fig. 4). This is done in a calibration procedure. The crystallographic identification of the corresponding EBSP lines ( $M_2$ ) is done by an indexing procedure. In the following these two procedures are described.

#### Calibration

PC and L (see Fig. 4) are calibrated in the standard way using a <001> cleaved silicon single crystal placed with the surface level identical to that of the sample. With the phosphor screen parallel to the specimen, the pattern centre coincides with the specimen normal and therefore with the (001) pole. The position on the screen of this pole is first identified. From a further identification of the position and crystallographic indices of another pole, the screen-specimen distance L is calculated as  $d/\tan(\alpha)$ , where d is the distance in the pattern between the two poles and  $\alpha$  is the angle between the corresponding two crystallographic directions. Depending on the surface quality of the silicon crystal the EBSP can be rather weak and diffuse. The identification of the (001) pole is, however, always rather precise, whereas the other poles are less clear. To assure a reasonably accurate determination of L, two poles — besides the central (001) pole — are used in the present calibration routine; i.e. 3 L values are calculated and the averaged value is stored.

Using the present non-eucentric sample stage a

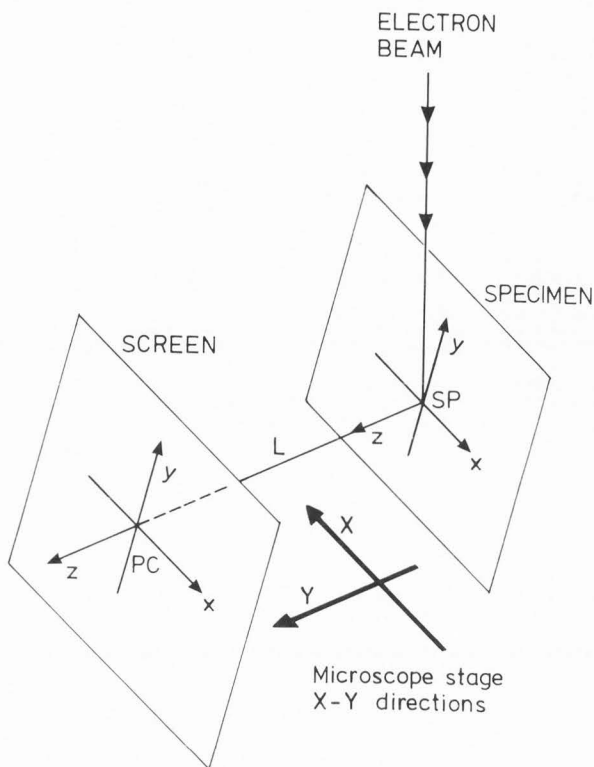


Fig. 4. Schematic diagram illustrating the orientations of the reference coordinate systems.

movement of the specimen in the Y-direction (see Fig. 4) to detect EBSPs from different "height-positions" of the specimen will result in a change in the position of PC and L. As can be seen from Fig. 4 the position of PC and L will vary linearly with Y. During the calibration, the procedure described above to find PC and L is repeated for a typical range of Y settings and the best fit straight lines through the experimental points are determined by linear regression. These lines are used as calibration curves; examples are shown in Fig. 5.

#### Indexing

The crystallographic identification of EBSP-bands is, in the present program, entirely based on a comparison of the interplanar angles as they are expressed in the crystal and pattern coordinate system (f2, f3).

The diffracted intensity of a (hkl) EBSPs bands is proportional to the square of the structure factor (S) of the corresponding (hkl) crystal plane. The structure factor is complex and given by:

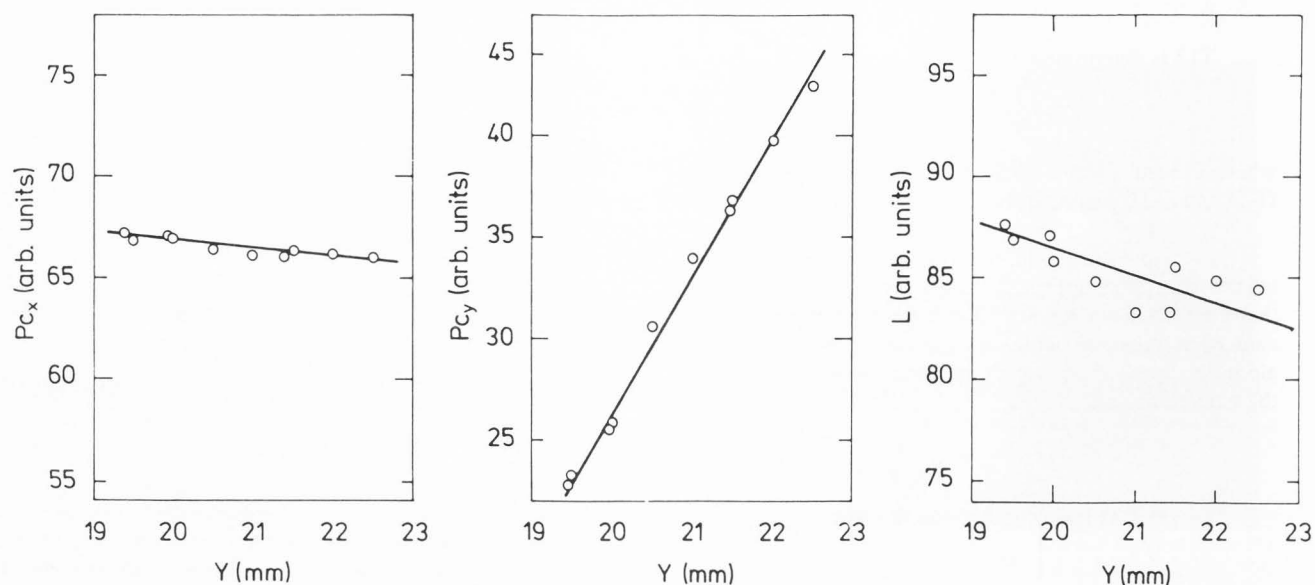


Fig. 5. Calibration for PC and L versus the microscope stage Y-position.

$$S(hkl) = \sum_{j=1}^n f_j(hkl) \exp\{-i 2\pi(hx_j + ky_j + lz_j)\} \quad (4)$$

where  $n$  is the number of atoms in the unit cell basis,  $(x_j, y_j, z_j)$  is the atom position within the unit cell and  $f_j(hkl)$  is the atomic form factor (e.g. Ashcroft and Mermin, 1976). For the actual sample material, the number of atoms  $n$ , their position in the lattice  $(x_j, y_j, z_j)$  and the corresponding form factors ( $f_j$ ) are required, and the computer calculates  $S$  for all low index  $(hkl)$  reflections. These are then sorted according to intensity and the strongest are used for further calculations. A total number of 30-60 of these reflectors is adequate for typical EBSPs. As a next step the interplanar angles between the reflectors are calculated (e.g. Young and Lytton, 1972), ordered according to magnitude and stored in the final look-up table characteristic for the given crystal symmetry. This table forms the basis for the indexing analysis of EBSPs from the present material.

#### Experimental Indexing Procedure

For each experimental EBSP the positions of two or more bands are identified by the user, these are converted by the computer into 3D unit vectors in the pattern system ( $f_3$ ) and the corresponding interplanar angle(s) are calculated. These are compared with the values in the look-up table for  $f_2$ -interplanar angles and the values, which give the best agreement with the experimental values, are determined. The corresponding  $(hkl)$  plane normals (stored in the look-up table) are then used to calculate the T32 matrix using equation [3]. When the positions of only two bands are used as input (the two

band situation) the third vector required to calculate T32 is created as the cross product of the first two plane normals. Consequently, a two band situation results in four possible T32 matrices if the interplanar angle between the two bands is different from  $90^\circ$ , whereas a two band situation with a perpendicular interplanar angle gives eight possible T32 matrices. A complete simulated EBSP is worked out and superimposed on the experimental EBSP. By comparing the experimental and simulated EBSPs the correct crystallographic orientation is determined. In the general case, crystal symmetry introduces equivalent EBSPs and correspondingly equivalent T32 matrices. For example, for aluminium (Laue group  $m\bar{3}m$ ) 24 equivalent matrices exist. The size of 'the unit triangle' is consequently  $1/24$  of the surface of a sphere. The position of the unit triangle for Laue group  $m\bar{3}m$  was chosen to be the triangle  $[001] - [111] - [\bar{1}\bar{1}\bar{1}]$  (Schmidt and Olesen, 1989). In order not to look at simulated patterns from equivalent T32 matrices it was chosen that the  $f_2$   $z$ -direction is confined within the defined unit triangle.

An "indexing sequence" is shown in Fig. 6. Fig. 6a shows an experimental EBSP with two bands identified (marked by thin lines), and in Fig. 6b the first simulated EBSP is superimposed. This solution is not correct, although the two bands identified experimentally agree with the simulation. This solution is rejected and another solution which has the same (or almost the same) value of interplanar angle is then proposed by the computer. As can be seen in Fig. 6c this gives a correct simulation of the experimental EBSP. The number of simulations depend on which and number of identified EBSP bands and on the precision of the identification. For the two band situation

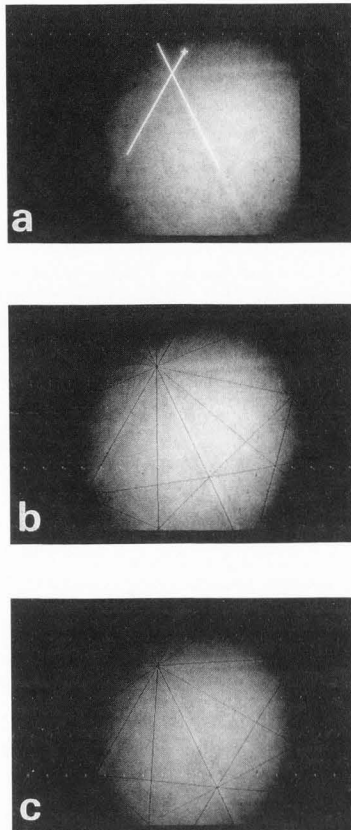


Fig. 6. Typical indexing procedure (for details see text). The specimen is commercially pure Al as in Fig. 3.

a typical number of simulations is in the range 1 to 4, on average 1.4, if the angle between the two bands is different from  $90^\circ$ . If the angle is equal to  $90^\circ$ , 9 to 15 simulations are typically needed (on average 10.2). This may take from 1 to 30 seconds. For the three band situation, the correct solution is simulated the first time for about 80% of the EBSPs, which requires  $\sim 15$  seconds computer time. For the remaining 20% of the EBSP up to 5 simulations may be necessary before the correct solution is found.

Experimentally the procedures are menu driven. Each band is identified using a mouse-controlled cursor. First the cursor is placed in the centre of a band, then a rubber stick appears on the screen and the direction of the bands is easily followed using the mouse. Besides accepting or rejecting a simulated EBSP it is possible to translate or rotate the simulated pattern. This is needed when the resulting T32 is not completely orthonormal, and indicates that the input precision is too poor or a new calibration is needed. For each simulated EBSP, the orientation matrix, the corresponding set of Euler angles and the ideal orientation  $\{hkl\}\langle uvw \rangle$  are shown on the screen. When a simulated EBSP is accepted the corresponding orientation

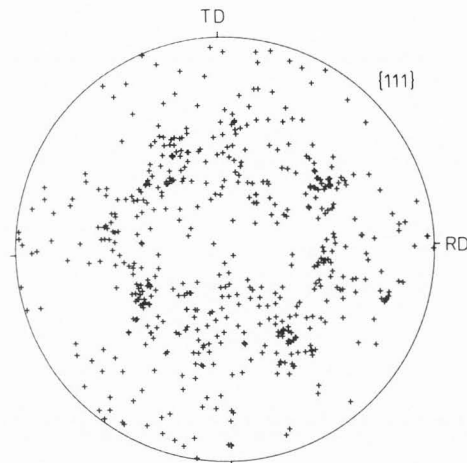


Fig. 7. Resulting  $\{111\}$  pole figure for Al-SiC<sub>w</sub>. The EBSPs for Al-SiC<sub>w</sub> are of a quality similar to that shown in Fig. 3, only very near the SiC whiskers (within a few microns) the patterns get too diffuse to index.

Table 1. Resulting Euler angles for repeated indexing of one EBSP from commercially pure Al.

$\varphi_1$	$\phi$	$\varphi_2$
69.2	37.5	80.0
68.8	37.3	79.7
69.5	37.5	79.7
69.5	37.4	79.7
69.0	37.5	79.7
68.8	37.4	79.8
69.0	37.6	80.1
69.1	37.5	79.9
69.4	37.4	79.9
68.8	37.4	80.0

matrix is stored together with an identification label for further processing.

Besides grabbing and indexing an EBSP it is possible to store the EBSP for later processing. Finally, EBSPs can be simulated from the input of  $\{hkl\}\langle uvw \rangle$ .

#### Data Representation

The measured data can be plotted as pole figures or inverse pole figures on the screen or on a HP-plotter. Further, a list of Euler angles can be typed. In the pole figure/inverse pole figure plots it is possible to see which poles belong to which crystallite, i.e. for a line scan, where the orientations of subgrains/ grains have been measured along a line, one can follow how the orientation changes from grain to grain in the pole figure. As an example of the data output, the  $\{111\}$  pole figure for Al-2 vol% SiC<sub>w</sub> cold rolled 90% and recrystallized at  $450^\circ\text{C}$  is shown in Fig. 7.

### Accuracy

To test the accuracy of the indexing, a series of EBSPs from 3 different Al-based materials were indexed 10 times each. The resulting Euler angles from this repeated indexing are listed for a typical EBSP in Table 1. The standard deviation ( $\sigma$ ) of the Euler angles (calculated as  $\sigma=1/3(\sigma_{\varphi_1}+\sigma_{\varphi_2}+\sigma_{\varphi_3})$ ) was found to be within the range  $0.05^\circ$ - $0.20^\circ$ ; the larger values are for fairly unsharp EBSPs of partly recrystallized Al-SiC<sub>w</sub>. This is the uncertainty for relative orientation measurements.

The uncertainty is larger, for absolute orientation measurements due to the difficulties in mounting samples, etc. This was tested by measuring the EBSP from a specific grain close to a fiducial mark in a commercially pure Al (1100) sample twice. In between the measurements the EBSP set-up was completely disassembled i.e. the phosphor screen was removed and the sample was taken off the holder. The experimental scatter in the Euler angle determination was in this case equal to  $0.5^\circ$ .

### Discussion

The basic aspects of a new computer procedure for analysis of EBSPs have been presented. A series of other methods are already available (Dingley et al., 1987, Vanables et al., 1976 and Young and Lutton, 1972). Among these the pole/zone axes (P/Z) technique developed by Dingley and coworkers (1987) is the most automated and therefore the most widely used. In the following the present and the P/Z procedures shall be compared.

The same principles are used for getting computer access to the EBSP, but the data acquisition is different. The new method is directly applicable to all crystal structures — metals, ceramic minerals etc. — and there is no need for electron diffraction maps. The only crystallographic information needed is that required to calculate the structure factor. Compared with the P/Z method where specific poles (e.g. (111), (112), (114)) have to be nominated, this means a real ease for the non-expert user and also for the expert when EBSPs from a new crystal structure have to be analyzed. Further, it is an advantage that at least two distinct bands can always be seen in an EBSP, whereas for some crystal orientations close to the four and two fold axes, the necessary poles for the P/Z procedure are not always present in the EBSP; and for deformed materials (weak diffuse EBSPs) the precise identification of the poles is difficult. The visual inspection of the correspondance between a simulated and an experimental EBSP is very precise with the new routine (see Fig. 6c). A mismatch of even tenths of a degree can easily be detected by eye and corrected. This is of importance for relative orientation measurements. The necessary data input and processing is slightly more time-consuming than with the P/Z technique: according to

Dingley et al. (1987) 300-500 orientations can be determined in a day's work. So far the maximum number obtained with our technique is 250. For most investigations this number (250 or 500) gives reasonable statistics, however, after such a day, measuring 250 orientations, one realizes the need for a fully automatic technique, and work is underway to automate the present procedures (Juil Jensen and Schmidt, 1990).

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#### Discussion with Reviewers

**D.C. Joy:** Is the internal calibration procedure using the silicon wafer actually required every time, or is the set-up stable enough to permit the calibration to be transformed?

**Authors:** The set-up is relatively stable. As a part of the EBSP start up procedure the calibration is every time checked by comparing the experimental and simulated EBSP for the reference silicon wafer. Only rarely, about twice a year, the full calibration procedure is needed.

**J. Hjelen:** Regarding the accuracy of the orientation measurements I assume that you get a measure of the reproducibility instead of the uncertainty of the absolute orientation. The accuracy of the absolute orientation measurement depends on specimen mounting, accuracy of the stage/pretilted specimen holder, phosphor screen mounting, camera alignment, and distortions in the camera/optical system. Do you have any idea how to measure the accuracy of absolute orientations where all these factors are included?

**D.E. Newbury:** In order to achieve an absolute accuracy of 0.5°, please describe the details of how the specimen is so accurately positioned. If the placement of the reference crystal is critical, how do you ensure that the reference crystal is attached to the surface of the unknown with such accuracy.

**Authors:** The spring arrangement which presses the silicon and the specimen towards a fixed top plate ensures that the surfaces of the two samples are identical (see Fig. 2). The plate on which the samples are mounted is made with a sharp edge, i.e. if the sample is prepared with an edge along a reference sample axis (e.g. the rolling direction) it is straight forward to mount the sample very precisely. In the paper we state, that the experimental scatter in the Euler angle determination for a given grain after a completely new mounting of the sample and EBSP equipment (sample holder and phosphor screen) was equal to 0.5°. The calibration procedure using the known silicon single crystal, corrects for camera misalignments (see "calibration"), i.e. assuming that the sharp edge of the sample is exactly along a reference axis, the ~0.5° is the precision with which an orientation can be determined.

**D.C. Joy:** Is the method applicable outside the cubic system? Do any other problems arise in this case?

**J. Hjelen:** Your applications seems mainly to be on aluminium, but your method is applicable to all crystal structures. Do you have any experience on EBSP-analysis of hexagonal materials like for instance magnesium and titanium?

**Authors:** The method is in principle applicable to all crystal structures and has been used successfully on e.g. titanium. In materials with structures that exhibit only small deviations from a higher symmetry space group, other crystallographic techniques (e.g. TEM diffraction) can distinguish smaller deviations than can be distinguished by the EBSP technique.

**D.C. Joy:** It appears from your text that the test of whether or not the computer generated fit is acceptable is a subjective one. Have you tried any two-dimensional statistical tests to quantify 'goodness of fit'?

**Authors:** No, only visual inspection has been used.

**D.C. Joy:** Is any detailed listing of the code available?

**Authors:** The programs are commercially available from one of the authors (NHS).

**D.J. Dingley:** Reference should be made to S. Vale, *Inst. Phys. Conf. Series No. 78, EMAG '85*, ed. G. Tatlock, p. 79 (1985), who has published virtually an identical procedure, though for SACP, and to D. Dingley, *Inst. Phys. Conf. Series No. 98, EMAG '89*, publishers Inst. of Phys. Bristol and New York, ed. P.J. Goodhew and H. Y. Elder, p. 473 (1989), as he describes there a new method to eliminate the need to recognise the pattern zone axis, and is hence equivalent to the author's contribution.

**Authors:** What is essential and new in the present method, is that i) it is based on calculations of the structure factor and ii) the input are center lines along EBSP bands. Structure factor calculations are not included in any of the two papers, and the input also differs: Vale (1985) uses the edges of the bands, a method which is applicable to SACPs but problematic for EBSPs since the edges are more blurred in the latter case. In the procedure by Dingley (1989) the position of three unknown zone axes are used as input. The present procedure can utilize bands crossing outside the screen.



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