

## EXPERIMENTAL ASPECTS OF THE USE OF ELECTRON BACK SCATTERING FOR CHARACTERISATION OF MICROTEXTURE AND MESOTEXTURES

V. Randle\* and J. Furley†

\* H.H. Wills Physics Laboratory, University of Bristol,  
Bristol, BS8 1TL, UK

† Dept. of Materials Technology, Brunel University of  
West London, Uxbridge, UB8 3PH, UK

### ABSTRACT

The aim of this paper is to explain the experimental techniques required for the operation of the EBS system. Also, by use of suitable examples, its potential as a tool for the rapid analysis of the microtexture and mesotexture of materials is demonstrated.

### INTRODUCTION

The EBS technique was developed by Venables and coworkers<sup>1,2</sup> from work by Alam et al.<sup>3</sup>. The present system was developed by Dingley<sup>4</sup>. A microcomputer was used to enable very rapid analysis of the patterns viewed on the monitor. A special low-light video camera viewed a phosphor screen placed inside an SEM chamber. This enabled on-line orientation determination of grains leading to a very rapid technique for acquiring the microtexture (local texture) and mesotexture (or Grain Misorientation Texture) of materials.

### EXPERIMENTAL DETAILS

The computerised EBS system can be used in conjunction with any SEM providing there is room inside the chamber for a phosphor screen to detect the electrons. For computerised pattern indexing a low-light TV camera, an image processing unit, a video monitor and a computer whose graphics can be superimposed on the monitor are required.

The maximum specimen size which can be viewed is only limited by the space available within the microscope chamber. The current arrangement at Brunel University allows specimens up to 10mm thick and about 20mm wide (it is restricted by the proximity of other detectors within the microscope chamber).

To obtain the best results specimen surface should be as free from deformation as possible. The diffraction pattern information arises from the top 10-30nm of the material, and if this region contains significant deformation (ie. dislocations, lattice imperfections, etc.) the diffraction pattern will become confused. The electrons will be

backscattered from areas of different crystalline orientation and therefore there will be a diffuse pattern visible on the screen, making accurate indexing of the pattern very difficult. Specimens should therefore be ground and polished down to  $1\mu\text{m}$  and then etched to remove the final surface damage. For clearer patterns the specimen could also be electropolished and etched. It has been found by various workers<sup>5,6,7</sup> that it can be difficult to obtain a clear diffraction pattern from specimens deformed by more than 40%<sup>8</sup>.

Once the specimen has been prepared to an adequate standard the crystallographic orientation of individual grains can be acquired very rapidly. Given a reasonable quality diffraction pattern, the orientation of a grain can be determined within 30secs, 100 or more grains being analysed within an hour.

Figure 1 shows the arrangement of the system. The position of the camera, lead glass window and phosphor screen can be either parallel to the surface of the specimen (which is  $70^\circ$  to the horizontal to obtain the best pattern) or parallel to the electron beam, in which case the computer must allow for distortion of the pattern due to the angular difference between the diffracting surface and the screen.

A sample of  $\langle 100 \rangle$  cleaved silicon single crystal is used for calibration of the system. By indexing the pattern in two positions the spatial and angular positioning of the screen, camera and specimen holder are accurately determined by the computer and recorded. The system need only be re-calibrated after an alteration to any of its components.

For microtexture determination a  $3 \times 3$  matrix defining the crystallographic orientation of each grain is calculated and recorded. Once a suitable number of grains have been analysed, their raw orientations can be automatically plotted as a pole figure or inverse pole figure around various axes or, if an electron micrograph is taken of the specimen and the grains numbered, the raw orientations can be plotted on the micrograph to give an indication of microtexture.

The usefulness of the system is further increased by an automatic grain misorientation determination program. As each grain, whose orientation is measured, has a number the program calculates the misorientation axis/angle pair between consecutively numbered grains (eg. 3,4,5, etc) or between selected grains input by the operator (eg. between 5 and 21, etc). Therefore the misorientation axis/angle pair across every grain boundary in the sample can be rapidly found with no expertise or calculation required by the operator. The system displays all 24 possible axis angle pairs on the screen and outputs the lowest axis/angle pair.

A separate program is then used to find the coincident site lattice (CSL) value of each boundary. The program prints those CSL values below  $\Sigma=35^9$ , if the ratio  $V/V_m < 1$ , where  $V$  is the volume of the superstructure cell and  $V_m$  the volume of the lattice cell. This covers the range of deviations from exact CSL axis/angle pairs.

Once this data has been obtained it can also be superimposed on an electron micrograph of the microstructure

in a grain-specific manner (ie. each grain is numbered on the micrograph) so that the spatial arrangement of CSL boundaries can be obtained (see Fig. 2).

#### APPLICATIONS AND RESULTS

The system has been used to examine various materials, leading to a significant increase in the understanding of grain boundary structure/property relationships.

Dingley and coworkers<sup>10,11</sup> have used the technique on geological specimens, examining non-cubic and deformed structures, using the number and position of symmetry axes and information on the a:b and a:c ratios to identify rock fragments, thereby increasing the range of applications to cover materials characterisation from crystallographic parameters.

However, the chief application of the system has been to determine the effect of thermomechanical processing on various metals.

Randle and Ralph<sup>12,13,14</sup> examined Nimonic PE16, precipitation strengthened by coherent gamma prime, to study the effect on the mechanism energy reduction by grain growth whilst the boundaries are strongly pinned. The EBS technique was most useful due to the rapid orientation determination possible. Over 200 grains were analysed, representing a large database at the time. The material had a bi-modal grain size distribution with bands of large grains surrounded by smaller grains. It was found that the percentage of CSL boundaries increased as the boundary/gamma prime interactions became more significant. Given that the average energy of an exact CSL boundary is 15% lower than that of a random high angle boundary<sup>15</sup>, it was concluded that the total grain boundary energy was decreasing. Grain growth was significantly suppressed by the coherent gamma prime and therefore Randle and Ralph<sup>12,13</sup> suggested that another mechanism, that of lattice rotation, was leading to a reduction in the overall energy normally attributed to grain growth.

Further work by Randle and Ralph<sup>16</sup> on Nimonic PE16 applied the technique to the small and large grains in a specimen. It was found that different local textures existed for the two distributions. It was also found that boundaries between small grains were more likely to be "special" CSL boundaries than those between large and small grains. It was therefore concluded that anomalous growth in this case will stagnate because the boundaries of small grains surrounding the large grains were, on average, lower energy than those between large and small grains.

Randle and Brown<sup>17</sup> used the EBS technique to establish a link between the onset of secondary recrystallisation and the evolution of a GMT in terms of the distribution of CSLs. It was found by examining a specimen after various heat treatments that the properties of CSLs and their spatial arrangements improved during a grain growth incubation period. The CSL boundaries were seen to "cluster" together and it was postulated that this was instrumental to the incubation of

grain growth in solid solution alloys (also supported by Makita and coworkers<sup>18</sup>).

An investigation of the effects of thermomechanical treatments on pure nickel<sup>9</sup> supports the previous work. The samples were 40% worked and the effects of progressive heat treatments were followed. In this part of the investigation a total of almost 1000 grain orientations were determined, leading to nearly 2400 grain boundary axis/angle pairs. The "clustering" of "special" boundaries was evident throughout the samples. Area A in Fig. 2 seems to contain a high density of CSL boundaries whereas area B, containing equal area of material and boundary length, has a very low density. It was also found that, as the time of annealing increased the number of coincident sites along a boundary increased (see Fig. 3a, b and c) confirming that, even in a system where grain growth can freely occur, energy reduction by changes in grain boundary geometry still occurs (generally the lower the  $\Sigma$  value, the lower the boundary energy). There was also a preference of low  $\Sigma$  boundaries, especially those related to  $\Sigma=3$ , (ie.  $\Sigma=9$ , 27a, 27b) this is shown in Fig. 3 a, b and c for consecutive heat treatments.

Finally the deviation of CSL boundaries from exact matching can be seen from Fig.4<sup>19</sup>.

#### SUMMARY

It is apparent that the EBS technique allows rapid acquisition of large databases of grain orientation/misorientation information, especially when compared to conventional TEM microtexture techniques.

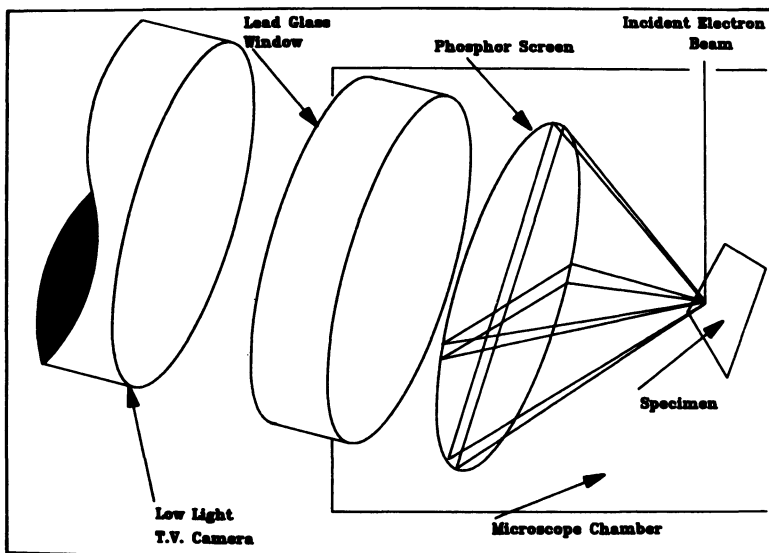


Figure 1: Schematic diagram of the EBS system in the microscope chamber.

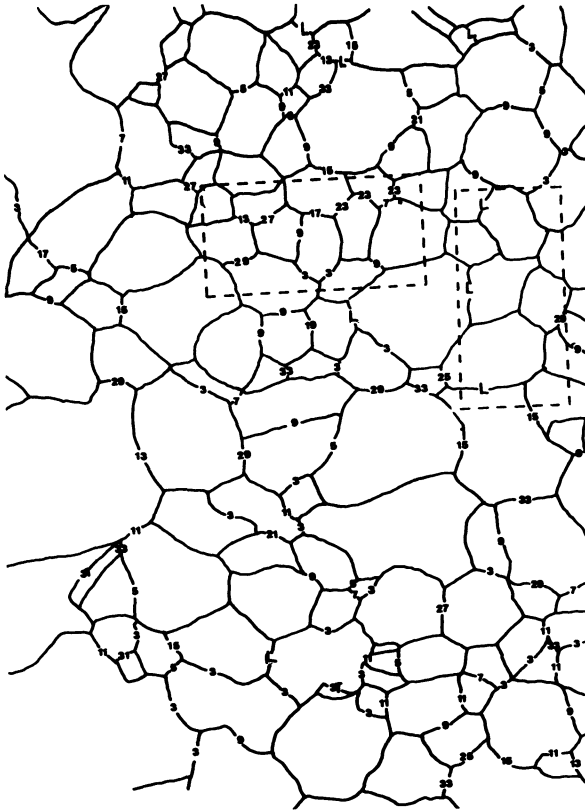


Figure 2: Grain map showing CSL boundaries.

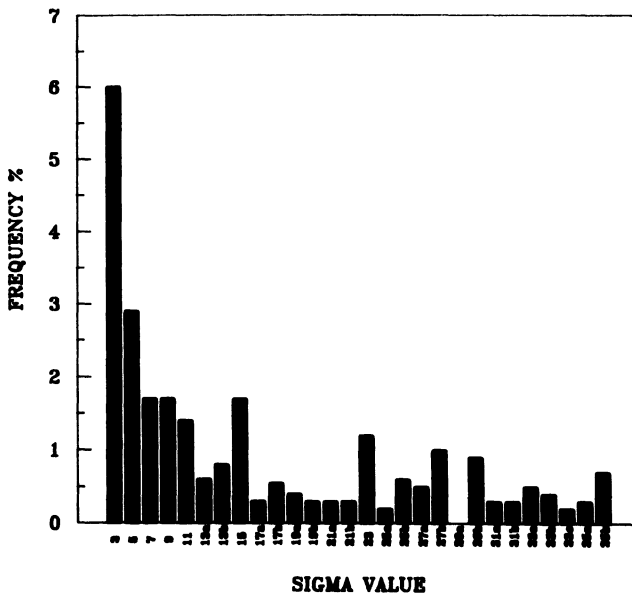


Figure 3a: Frequency distribution for CSL boundaries for pure nickel after 30 mins at 600°C.

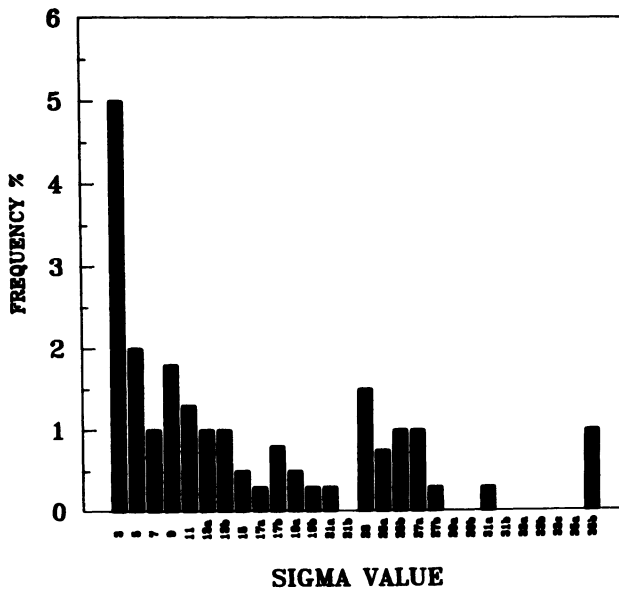


Figure 3b: Frequency distribution for CSL boundaries for pure nickel after 30 mins at 600°C followed by 30 mins at 900°C.

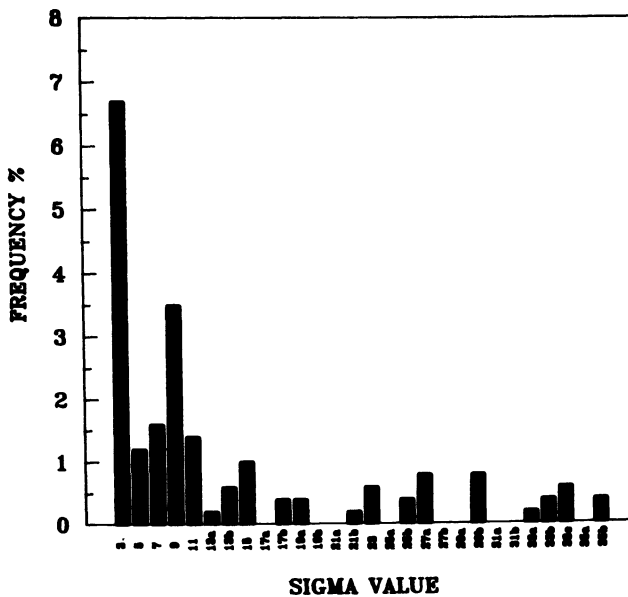


Figure 3c: Frequency distribution for CSL boundaries for pure nickel after 30 mins at 600°C followed by 50 mins at 900°C.

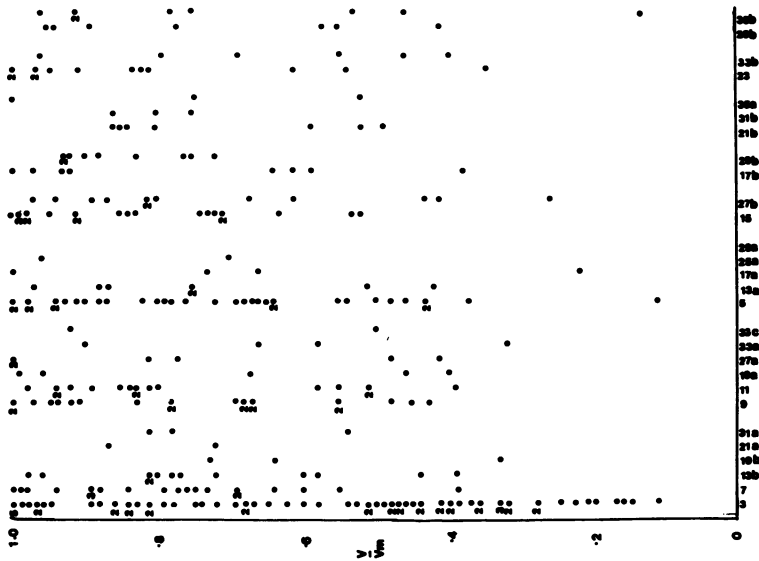


Figure 4: Plot showing the deviation of CSL boundaries from exact matching.

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