

Measurement Note

DEPC (MN) 037

Microstructure Mapping in High Temperature Compression Testpieces - *Grain Size Metrology by Electron Back Scatter Diffraction*

Summary

Electron Back Scatter Diffraction, EBSD, is increasingly being used to characterise the microstructure of many engineering materials by mapping the crystallographic orientation in testpiece sections. This crystallographic information traditionally is used to reveal detailed phase and textural information, but it can also provide a wealth of information on grain size and related parameters. These latter parameters are sometimes regarded as simple measurements for straightforward optical techniques. However, the automated nature of EBSD means it can potentially provide much more information less subject to the skill and subjectivity of individual operators, e.g. in the setting of sample illumination for automated image analysis.

Although EBSD can automate the process of grain size measurement there is still a need for care in specimen preparation, choice of operating conditions and use of post-acquisition noise reduction. Practical examples of these effects on the measured grain size are reported and EBSD results compared with those obtained optically, highlighting the effects of the greater resolution of EBSD in detecting smaller grains and in detecting twin boundaries. It discusses ways of reporting results and compares the results with a theoretical prediction of grain size distributions. This work has been performed within the wider context of needing to quantify micro-structural heterogeneity in order to validate deformation models of hot deformation of engineering alloys, produced as part of a joint project with the Universities of Sheffield and Wales (Swansea).

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1 Introduction

To study the effects of specimen preparation and system operating conditions, sections through test samples of uniaxially compressed cylinder were cut and polished (using colloidal silica for 30 minutes as the final polishing stage) and examined both optically and by SEM. This Measurement Note reports on test samples of a 316L stainless steel and a Ni superalloy, Waspaloy which had both been compressed under dry non-lubricated conditions at a strain rate of 1 s^{-1} to a total strain of 0.5 at temperatures of 950 °C and 1040 °C for the 316L and Waspaloy respectively. Regions from the centres of these samples, comprising roughly equiaxed recrystallised grains, were mapped by EBSD in a Cambridge 360 W-filament SEM operating at 15kV with a 1-3 nA probe current producing a spot size of approximately 0.15-0.25 μm . The EBSD patterns were acquired on a Nordlys detector using HKL software. After EBSD the 316L sample was electrolytically etched with 10% oxalic acid to reveal grain boundaries for optical examination and linear intercept measurement of grain size.

2 Data acquisition and grain diameter measurement

Figure 1 shows raw (no noise reduction applied) EBSD maps of an area of the 316L specimen examined in the SEM. The colour scale shows the crystallographic orientations of the grains, with grain boundaries and twin boundaries identified by thin and broad lines respectively. The central $300 \times 200 \mu\text{m}$ area of each map is common to all three maps; the different size maps result from allowing regions not previously scanned to be included in each successive map to check that contamination build up in the region already mapped did not affect the results by degrading EBSD pattern quality. No effects of contamination were seen. The three maps were acquired with step sizes of 0.5, 1 and 2 μm ; in other words the HKL software steps the beam by 0.5, 1 or 2 μm and assigns the crystallographic information at each step to an area (one pixel on the map) of 0.25, 1 and 4 μm^2 . The total number of grains in each map before any noise reduction varied from approximately 750 to 1000, ignoring all

incomplete grains touching the boundaries of the map.

Individual grains can be defined as areas enclosed by boundaries between pixels with a misorientation between them of more than a particular value, commonly taken as 10 or 15° [3, 4]. The area A of each grain can be converted to a circle equivalent diameter, $d_{\text{ceq}} = 2\sqrt{(A/\pi)}$. The software automatically determines the position of grain boundaries by comparing the misorientation between neighbouring pixels and defines areas enclosed by these boundaries as constituting single grains, hence determining A and d_{ceq} of all the grains within the mapped area. Alternatively a measurement of grain diameter d_{lin} by linear intercept can be made automatically using the same defined grain boundaries and their intersection with a line or a number of evenly spaced lines, the number being chosen to maximise the number of data points without any one grain being cut by more than a single line. Because twin boundaries can be identified from the crystallographic information, these boundaries can be ignored in grain size determination. Measurements of A , d_{ceq} and d_{lin} were made for each map; in this report a 10° misorientation was used to define grain boundaries unless stated otherwise.

3 Measurement of d_{ceq} and the effect of EBSD step size

Figure 2a shows grain size distributions derived from the three maps in Figure 1, plotted as normalised frequency against $\ln(d_{\text{ceq}})$, and Figure 2b the same data as cumulative number frequency distributions. The graphs clearly show the effect of altering the step size on the lower end of the size distribution below about 5 μm . This divergence of the distributions results in a difference in calculated number average mean grain size of approximately 40% in the worst case, depending on the EBSD conditions and the method of calculation used. Table 1 shows the range of different possible values which could be used to characterise the grain size.

Figure 1

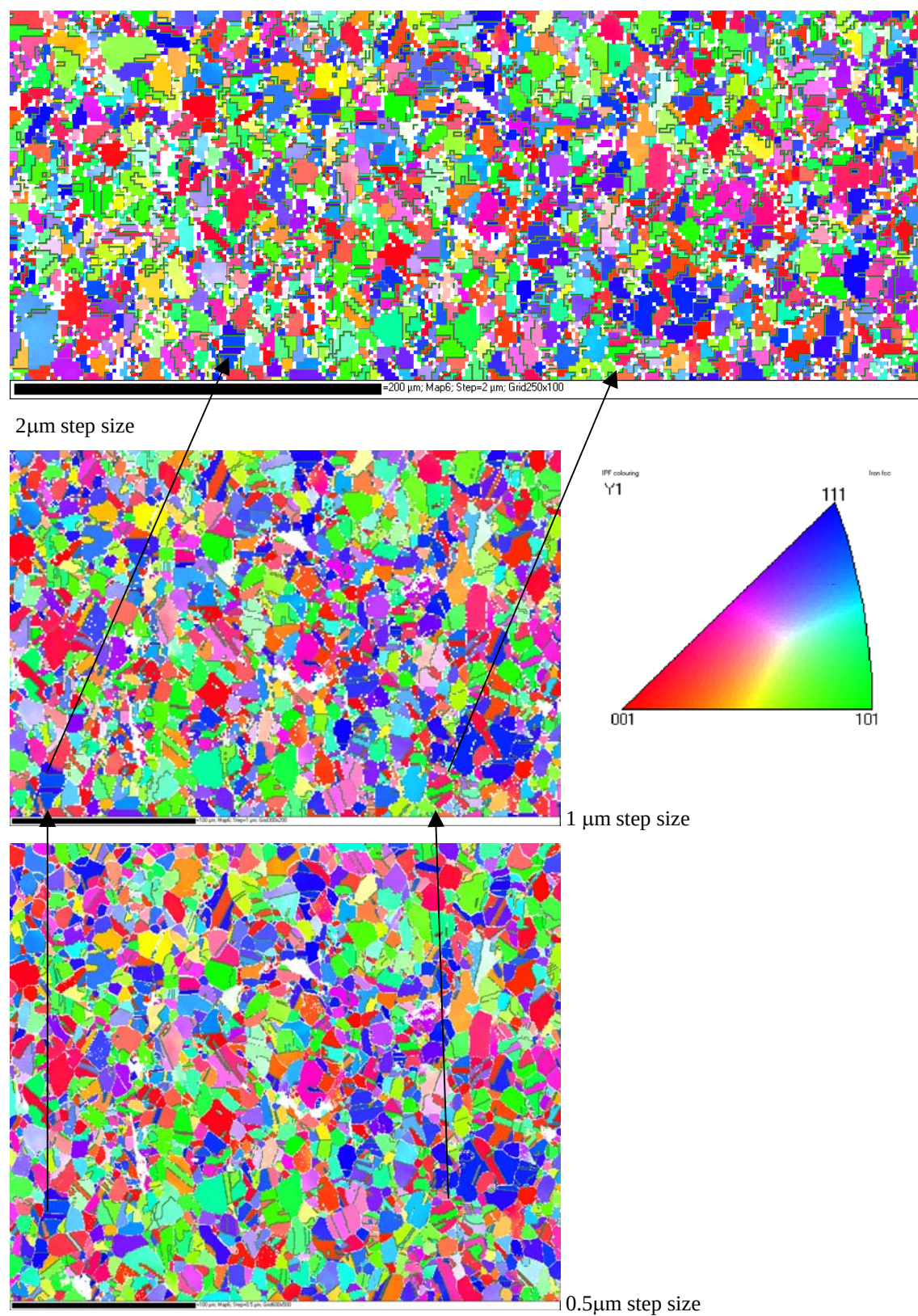


Figure 1: Raw EBSD maps (no noise reduction) of overlapping areas, made at the three different step sizes indicated. Grains coloured according to the Inverse Pole Figure colouring shown. Identical grains shown arrowed.

Table 1: Variation with EBSD step size of d_{ceq} number average grain sizes for Figure 1 maps.

	Circle Equivalent Diameter d_{ceq} (μm)		
Map step size (μm)	0.5	1.0	2.0
Arithmetic Mean (μm)	7.2	7.8	8.4
Geometric Mean (μm)	4.6	5.8	6.8
Median (μm)	5.9	6.6	7.0
% of map area indexed	90.3	89.6	85.9

The relative error in the calculated mean at the 95% confidence level can be estimated [5] as $\pm 4.5\%$ from the number of grains analysed (on average 850). However, dividing each map into 9 sections and comparing the means of each section suggests a larger 95% confidence range of $\pm 8\%$. Thus for the 1 μm step size the arithmetic mean is estimated as $7.8 \pm 0.6 \mu\text{m}$. With this range and the fact that the areas for the different step sizes overlap, the differences between the three mean values in table 1 are felt to be significant. The difference between the largest and smallest arithmetic mean is about 15% which is approximately consistent with the findings of Humphreys [1] if it is assumed the smallest step size is more accurate.

However, it should be noted that between geometric means the difference is greater than 35% and consideration needs to be given to which mean is more appropriate since for each mean there is standard deviation σ associated with the assumed distribution of results which will give an indication of the spread of grain size in a given region.

Grain size distributions have frequently been reported to be lognormal [6, 7] or distributions that closely approximate to this [8]. It was shown [5] that if a lognormal distribution is followed then the mean of $\ln(d)$ (also the geometric mean of d) equals the natural log of the median value i.e.

$$\langle \ln(d) \rangle = \ln(d_{50}),$$

and the distribution of $\ln(d)$ follows a normal distribution so the mean $\langle \ln(d) \rangle$ should also equal the mode.

Whilst plotting the distribution number frequency against $\ln(d)$ produces a peak (as opposed to a plot of number frequency against d , which does not) the distribution is clearly not symmetrical, with a long tail towards lower values if the peaks at the lowest values

measured are ignored. Thus the discrepancy between the geometric mean and the median value is not surprising and suggests a log normal distribution is not followed by the measurements. The high occurrence of measurements at the very smallest sizes (relative to the step size chosen) clearly has a large effect on the above results; the following three sections examine whether these measurements are real or artefacts of the EBSD technique and how they may be handled depending on whether they are real or artefacts.

4 Data analysis – handling measurement of very small grains

4.1 Removal of Wildspikes

A frequent technique for editing an EBSD map is to remove ‘wildspikes’, often 1 but up to 5 pixels with an orientation not matching that of any surrounding pixels. The criteria for this removal should be clearly established: the number of pixels removed should be related to the average grain size but a blanket removal of all such pixel areas could lead to errors. Ideally further criteria such as position (see for example figure 12 in section 7) and pattern quality/angular deviation should also be used.

The danger of blanket removal of all grain below a certain number of pixels, particularly if the wrong step size is chosen may be illustrated by considering the distribution of raw data shown in Figure 2a. The number of grains represented by the “wildspike” peak ($d_{\text{ceq}} = 2.25 \mu\text{m}$) of the 2 μm distribution is 15% of the total; for the 1 μm and 0.5 μm step sizes, the number of grains of this size down to but not including their respective “wildspike” peaks are 14% and 11%. This implies that a substantial fraction of the “wildspike” peaks of the 2 μm distribution are likely to be real grains somewhat less than 2.25 μm diameter, comprised of multiple pixels when mapped with the smaller step size. This can be confirmed by comparing maps of the different step sizes in which the smallest grain sizes have been highlighted. Figure 3 shows a common area selected from the centre of the 2 μm and 0.5 μm maps in which grains $< 2.25 \mu\text{m}$ (d_{ceq}) have been highlighted in red. Pixels that were not indexed are shown in green, and the remaining pixels follow a grey scale showing the quality of the EBSD pattern at that location where white = high quality,

darker = poorer quality). Some of the individual pixels at the 2 μm step size corresponding to a collection of pixels at the 0.5 μm step size are numbered; these pixels could therefore be considered as real grains, $\leq 2.25 \mu\text{m} d_{\text{ceq}}$. There are, however, some grains for which there is no correspondence, some of which may still be real if they correspond to a grain slightly larger than 2.25 μm but not oriented such that it was mapped by 2 pixels at the 2 μm step size. Others may be misindexed noise, but equally, the higher resolution 0.5 μm step size identifies a number of real grains missed at the coarser step size. If the 2 μm wildspikes include real grains, then this argument could be extended to smaller and smaller step sizes; for example, the 1 μm step size wildspike peak could be simply the accumulation of grains smaller than the step size of 1 μm . Certainly the rejection of wildspikes should not be carried out without first verifying the likelihood of the sites being misindexed (e.g. where they occur surrounded by one single larger grain, as opposed to at a triple point where the intersection of the plane with the tip of a grain is more likely).

4.2 Comparison with theoretical distributions

To examine whether the number of very small grains identified by EBSD are likely to be noise (in particular at the 0.5 μm step size), or whether they should in fact be expected, use was made of an NPL program [9] to simulate 2D measurements from 3D shapes. This program generates statistical data on 2D measurements of grain area and linear intercept to be expected from a collection of grains with a 3D tetrakaidecahedron shape, lognormally distributed in size. By rotating the grains and continuously varying the plane of intersection either side of the grain centre, there is an equal random probability of intersecting a grain at a particular orientation and position. The number cumulative frequency plots in Figure 2b shows, together with the measured data, curves resulting from simulating distributions from 10,000 grains lognormally distributed in size in which the spread is defined by standard deviations of $\sigma = 0.3 \mu\text{m}$ and $0.6 \mu\text{m}$ for the dimension shown in Figure 2c set with a mean of 1 μm . Figure 4 is a histogram of the same data in which the bins

are truncated for $\sigma = 0.3 \mu\text{m}$ at the same point as the measured data, but for $\sigma = 0.6 \mu\text{m}$ the breakdown of the data into bins below 1 μm is shown.

The theoretical distributions show long tails of very small sizes consistent with at least some of the EBSD 0.5 μm step size wildspikes being real. The distribution with $\sigma = 0.6 \mu\text{m}$ fits most closely with the EBSD measurements, which compares with previously reported good fits of linear intercept data to a theoretical spread of $\sigma = 0.6 \mu\text{m}$ for Waspaloy and $\sigma = 0.3 \mu\text{m}$ for an Al alloy [10]. If the measured and/or theoretical data for d_{ceq} is lognormally distributed, a probability plot of the data should yield a straight line. Figure 5 is such a probability plot, showing clearly that a straight line does not result from the data. Thus, while the initial 3D size distribution of grains may be lognormal, the process of sectioning at random depths through these 3D grains produces a non lognormal distribution for the 2D size distribution. This type of divergence at lower values for planar sections has been observed simultaneously with the measurement of the 3D grain size even though theory shows the two measurement types should yield very similar values [6].

This theoretical view, combined with the analysis in the preceding section 4.1 suggests that removing all “wildspikes” cannot be justified, at least for the case of the stainless steel, on the simple grounds that it is likely to be noise. It is probable that such smaller grain sizes have not previously been considered because they are approaching or below the resolution of optical microscopes with which most previous microstructural image quantification has been carried out

4.3 Plotting data as a function of area

The large number of small grains clearly dominate the number probability distributions but the total area of the section examined for which they account is actually small: grains less than 5 μm in diameter only account for 5% of the total area. It may therefore be more appropriate to plot the results as an area (size probability) plot, where the probability P_i on the y-axis is defined as

$$P_i = \sum_{i=1}^i (A_i) / \sum_{i=1}^N (A_i)$$

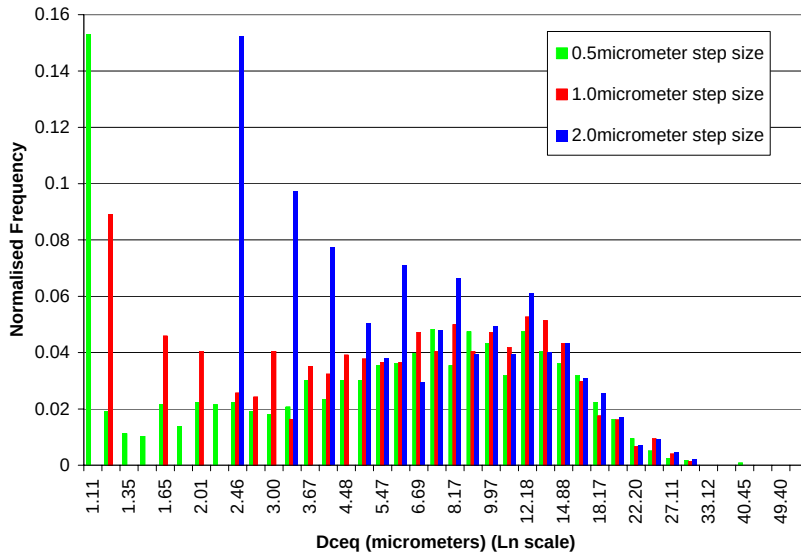


Figure 2a. Histograms of grain size distribution for the three maps shown in Figure 1

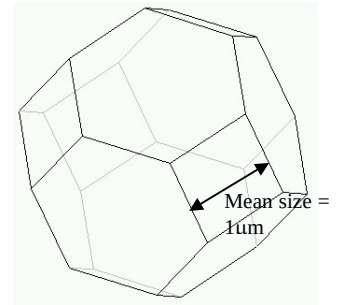
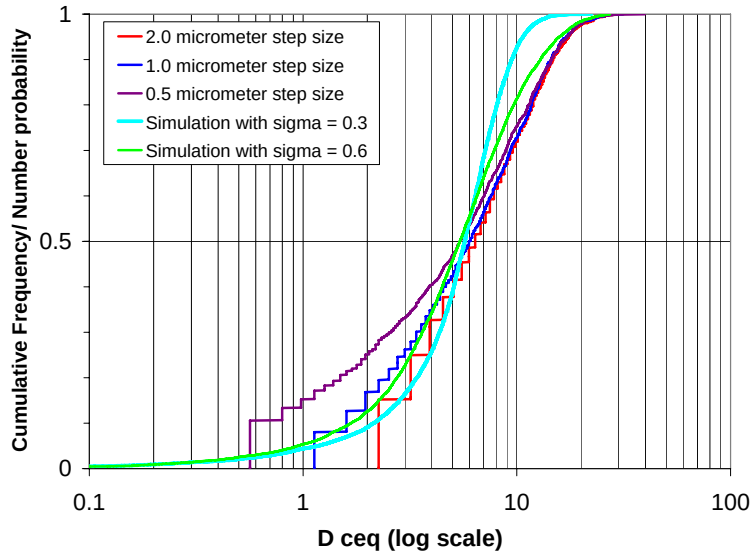


Figure 2c. Tetrakaidecahedron used in simulation showing dimension set to a mean of $1\mu\text{m}$ and varied about this log normally with $\sigma = 0.3$ or $0.6\mu\text{m}$

Figure 2b. Cumulative frequency plot of the grain size data shown in figure 2a (Figure 3 overleaf)

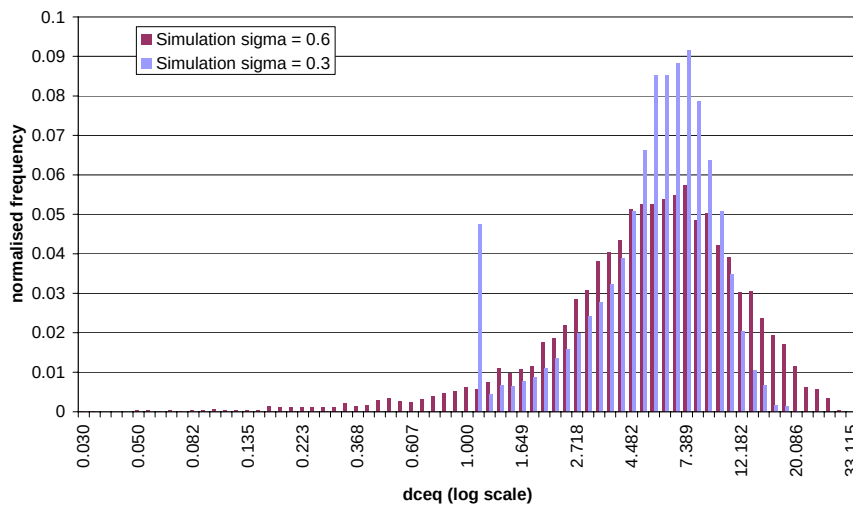


Figure 4. Histograms of simulated grain size measurements from theoretical spacefilling tetrakaidecahedron model. Full histogram shown for simulation with $\sigma = 0.6\mu\text{m}$, histogram for simulation with $\sigma = 0.3\mu\text{m}$ truncated at $d_{\text{ceq}} = 1\mu\text{m}$ with all data below this added to $1\mu\text{m}$ bin.

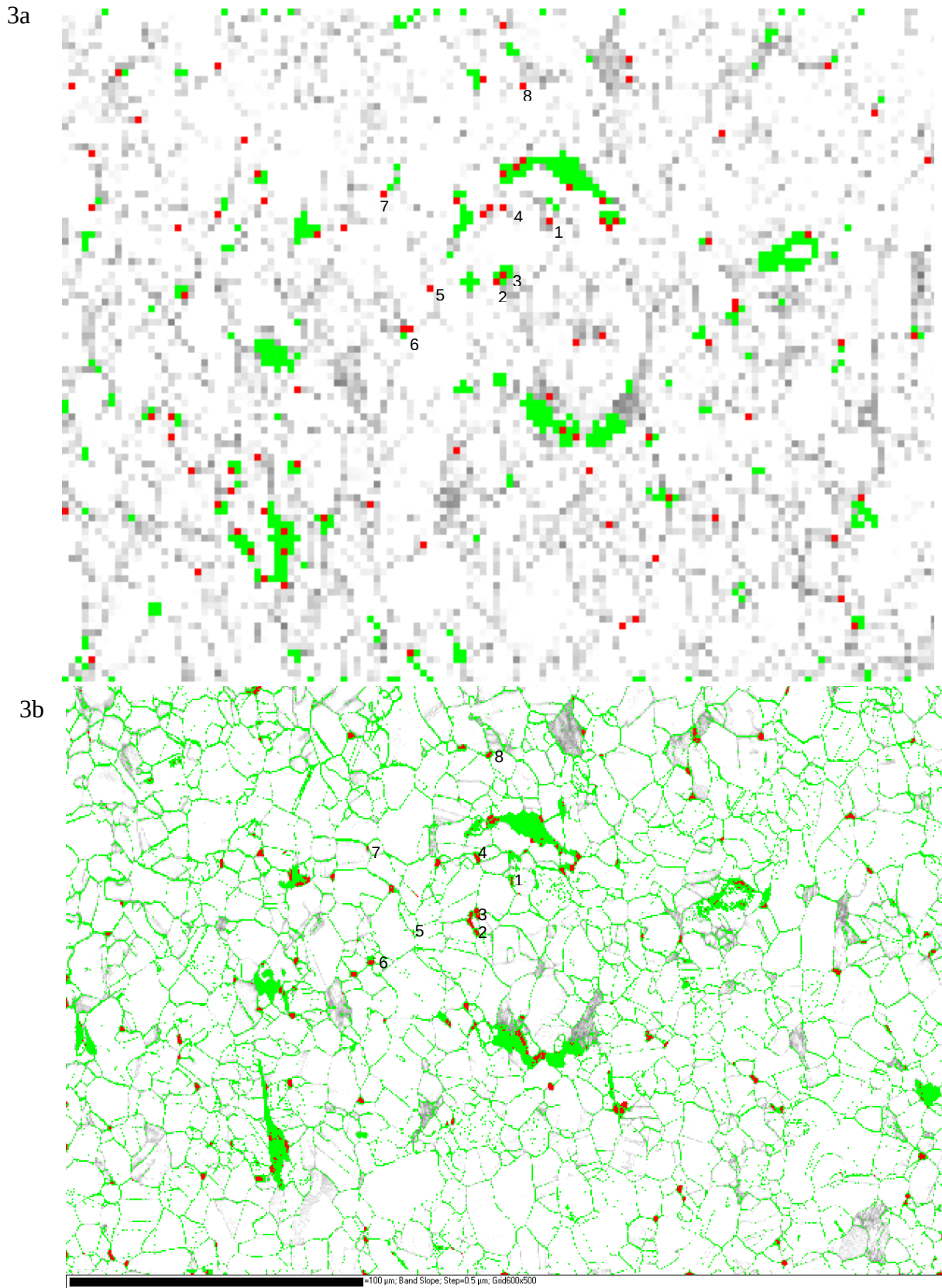


Figure 3. EBSD pattern quality maps produced at 2µm (Fig 3a) and 0.5µm (Fig 3b) step sizes, with areas less than 2.25 µm (d_{ceq}) coloured red and some example equivalent areas numbered. Non-indexed areas coloured green, grey = poor pattern quality.

Figure 6 shows that when this is used the probability distributions produced by the 3 different step sizes appear very similar, but the distribution remains non lognormal. Thus a similar plot is obtained for both but the median value is now substantially higher than previously measured, at 10.3 μm .

5 Grain size from measurement of linear intercepts

Linear intercept measurements were made with the EBSD software, inserting measurement lines approximately 20 μm apart, horizontally across the images shown in Figure 1. The software determines the positions of the intercepts in the same way that it defines grain boundaries. It was found essential that the software should be programmed to ignore zero solutions (non-indexed points) to prevent large grains being measured as 2 grains where the line intercepts a zero solution. Typically 350 values were obtained by this method, compared with an average of 850 using the same dataset for grain size area.

Table 2. Variation with EBSD step size of d_{lin} number average grain sizes for Figure 1 maps.

	Linear intercept length, d_{lin}		
	0.5	1.0	2.0
Map step size (μm)	7.8	8.1	8.7
Arithmetic mean (μm)	6.1	6.6	7.3
Geometric mean (μm)	6.9	7.1	7.6

Results are shown in Table 2 and Figure 7 shows the cumulative frequency curves for the three step sizes linear intercept d_{lin} measurements, plotted with the data for circle equivalent diameter d_{ceq} from Figure 2b and also the data for d_{lin} from the theoretical model. The different step sizes produce some divergence in linear intercept data at small values, but the reduced number of these small values means the results for d_{lin} are less sensitive to step size and 95% confidence intervals are very slightly smaller at $\pm 7\%$. Furthermore, the ratio $d_{\text{ceq}}:d_{\text{lin}}$ is < 1 , in contrast with an expected value of 1.22 [1]. In contrast to the reasonable correspondence of the theoretical and EBSD measurements based on area, the theoretical linear intercept measurement is very different from the EBSD measurements because, firstly, the model assumes a linear intercept is measured for every

single grain and thus the theoretical curve has a high frequency of very small measurements, whereas with a limited number of lines, the chances of intersecting a very small grain area is reduced significantly. Secondly the vast majority of linear intercept values will be less than and in some cases much less than the calculated circle equivalent diameter.

5.1 Comparison between EBSD and optical measurements of d_{lin}

Linear intercept measurements were made optically in the same region of the sample, after etching. They were also compared with measurements by Swansea University [11] on a separate but identically processed sample (Table 3). Digital images of the etched samples (Figure 8) were acquired with a 20x objective lens and two operators then measured intercepts on sets of lines 25 μm apart, giving approximately 250 measurements. Twin boundaries were ignored. Operator variability is clearly seen in the results shown in Table 3 but with 95% confidence intervals of $> \pm 10\%$ they are approximately equivalent. However there is a 25% increase over the EBSD measurements in the same orientation. Possible reasons for this include failure of the etch to reveal all grain boundaries, and a failure to resolve all the small grains identified by EBSD. These effects would both tend to cause overestimates of grain size by the optical method, although they would to some extent be counteracted by the mistaken inclusion of some twin boundaries.

Table 3. Optical measurements of d_{lin} (note - orientation same as vertical measurements shown in Table 4)

	Optical Linear intercept length, d_{lin}		
	1	2	Swansea
Operator			
Arithmetic mean	12.7	11.8	13.8
Geometric mean	10.0	9.3	
Median	10.9	10.5	

Figure 9 is a probability plot showing that, relative to the EBSD results, a) the optical linear intercept data follows a very similar distribution but offset by the difference in mean values, and b) that the linear intercept data show a slightly closer correspondence to a lognormal distribution, but there is still a deviation away from the line at larger values.

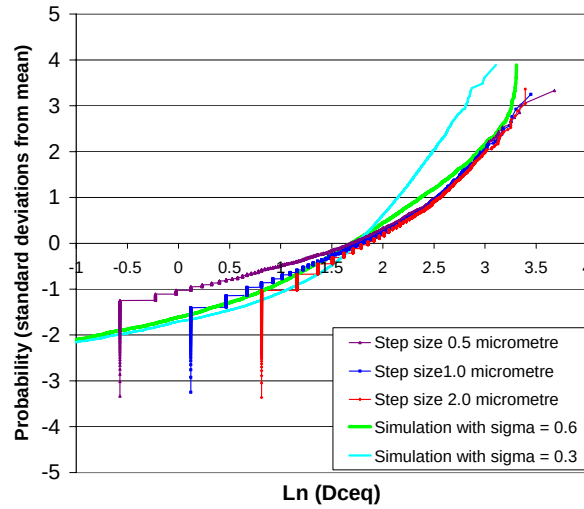


Figure 5. Probability plot of grain size distribution measured with step sizes of 0.5, 1 and 2 μm and theoretical distribution predicted for tetrakaidecahedron with standard deviations σ of 0.3 and 0.6 μm

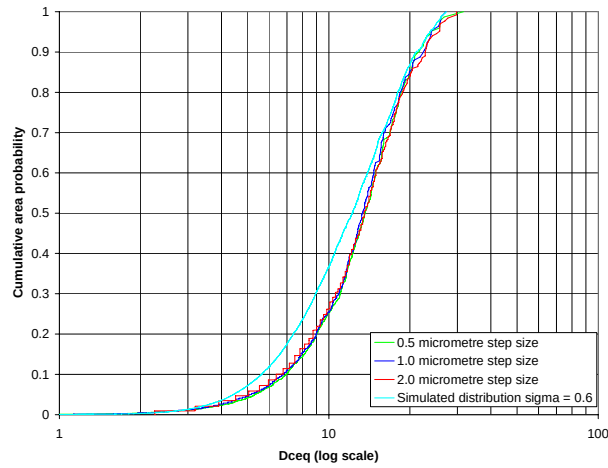


Figure 6. Cumulative area plot of grain size distribution for the data in figure 5 showing closer matching of the three step size measurements resulting from the reduced effect of the high numbers of small grains when expressed as a function of area

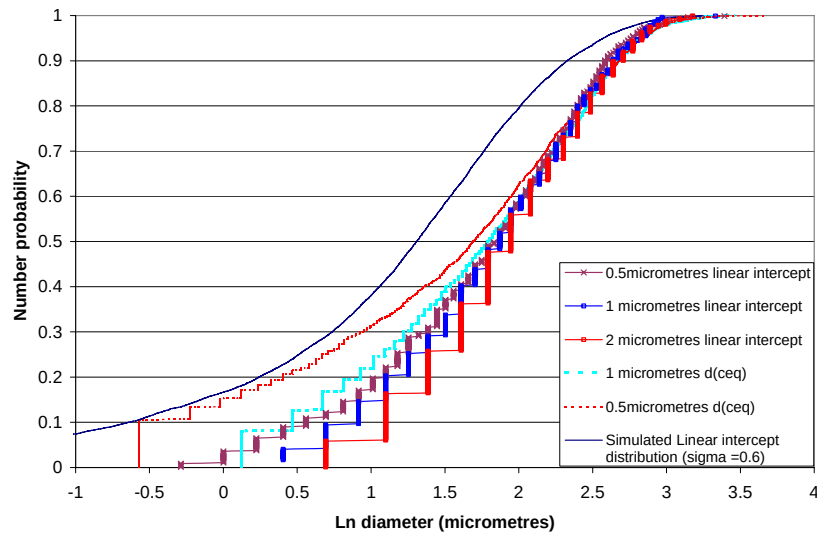


Figure 7. Cumulative number frequency plot of grain size distribution measured by linear intercept, d_{lin} , from the maps with step sizes of 0.5, 1 and 2 μm , compared with circle equivalent diameter size d_{ceq} data (figure 5) and theoretical linear intercept size for the tetrakaidecahedron model.

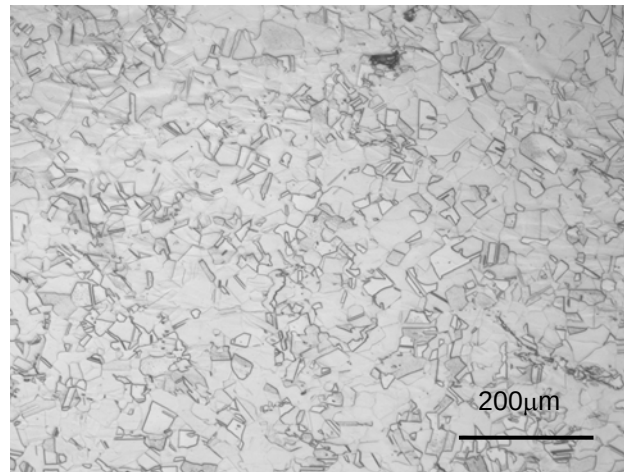


Figure 8. Optical micrograph of stainless steel sample in same region as that used for EBSD maps, and from which optical linear intercept measurements taken.

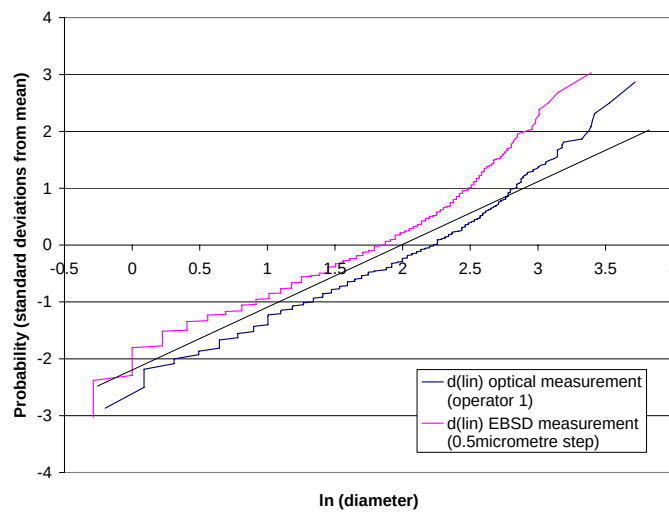


Figure 9. Probability plot of d_{lin} grain size distribution measured by linear intercept optically (from figure 8) and by EBSD (from figure 1).

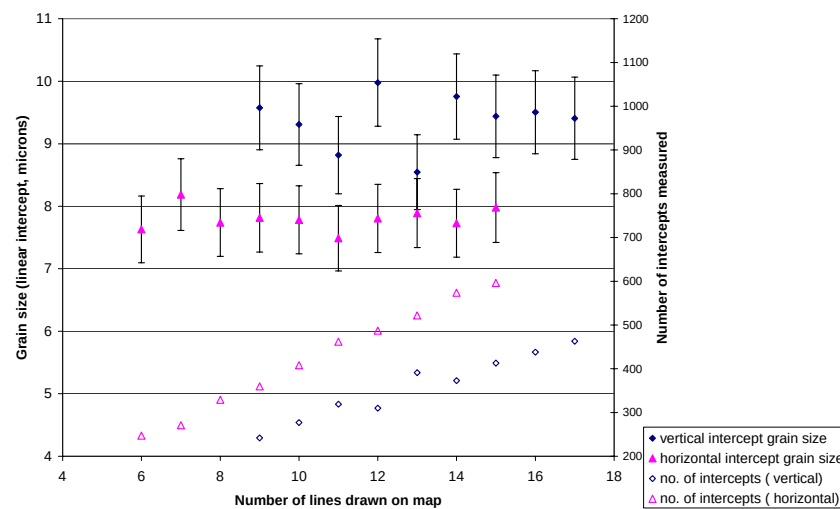


Figure 10. Graph showing variability of linear intercept measurements with number of lines used for measurement, using the 0.5 μm step size EBSD map in figure 1.

6 Directionality of grain size measurements

By drawing lines at right angles direct measurement of distortion from an equiaxed structure is possible by the linear intercept method. The data in Table 2 were derived from lines drawn horizontally across the maps in Figure 1 (in the axial direction of the specimen) and in Table 3 from lines normal to those of Table 2. Table 4 shows EBSD measurements of linear intercept sizes from vertical (radial relative to the forging direction) lines drawn across the same map. Clearly a small degree of elongation of grains is measured in the vertical direction; on average the aspect ratio is 1.1, which is less than a value of 1.3 obtained by optical measurements [11].

Table 4. d_{lin} measurements in orthogonal directions at different step sizes.

	Horizontal linear intercept length, d_{lin} (μm)			Vertical linear intercept length, d_{lin} (μm)		
	0.5	1.0	2.0	0.5	1.0	2.0
Map step size (μm)						
Arithmetic mean	7.8	8.1	8.7	8.8	9.0	9.4
Geometric mean	6.1	6.6	7.3	6.7	6.8	7.7
Median	6.9	7.1	7.6	6.8	7.1	7.6

It is also possible to derive directional measurements from the EBSD based area measurements by calculating aspect ratios for each grain (major:minor axes of an ellipse) and the angle the max. diameter makes to the horizontal. This enables a variety of ways of presenting directional information; one possible method is to separate the grains elongated in the horizontal direction from those in the vertical direction and compare the aspect ratio of the 2 classes. Applying this to the current data (and eliminating grains of <4 pixels) gives mean aspect ratios horizontally and vertically of 1.73 and 1.88 respectively, a ratio of 1.09, while the ratio of numbers of grains oriented vertically to those horizontally is 2.25.

An advantage of EBSD is that multiple linear intercept measurements can be made quickly once a map has been acquired. By altering the number of lines measured an estimate of the variability of the data can be obtained. Figure 10 plots the arithmetic mean of d_{lin} for the

0.5 μm step size against the number of equispaced lines 'drawn' on the map. On the second y-axis the number of intercepts measured on these lines are plotted. From the number of intercepts an estimate at the 95% confidence level of the relative error of the mean can be made and this error (an average for each of the vertical and horizontal data) is shown as error bars. A variation in mean size greater than the relative error gives an indication of the degree and scale of inhomogeneity in the microstructure.

7 Application of map noise reduction techniques on measurement analysis.

As noted in Section 4. a frequent technique for editing EBSD maps is to remove wildspikes, isolated single pixels. Also commonly employed is the technique of filling in non-indexed zero solution pixels by growing neighbouring regions into the zero solutions, with growth limited to non indexed regions having a minimum number of neighbours of the same orientation.

To explore these factors, the 0.5 μm step size data was edited using various combinations of the above noise reduction routines. The effect on the results of mean d_{ceq} grain size measurements is shown in Figure 11 and given in Table 5.

Table 5. Effect of noise reduction methods on number average d_{ceq} grain size measurements for the 0.5 μm step size map.

	Raw data	Remove wildspikes	Grow into non indexed areas where 6 neighbours	Remove wildspikes and grow where 6 neighbours	Remove wildspikes and grow where 4 neighbours
Arithmetic mean (d_{ceq} , μm)	7.2	7.9	7.9	8.3	8.5
Geometric mean (d_{ceq} , μm)	4.6	5.8	5.3	6.1	6.4
Median (d_{ceq} , μm)	5.9	6.8	6.6	7.1	7.3
% map area indexed	90.3	90.3	95.9	95.9	98.4

Clearly even though 90% of the original map was indexed it is possible to alter the d_{ceq} grain size by a significant percentage by filling in

Figure 11. Graph showing effect of various noise reduction methods on measured grain size.

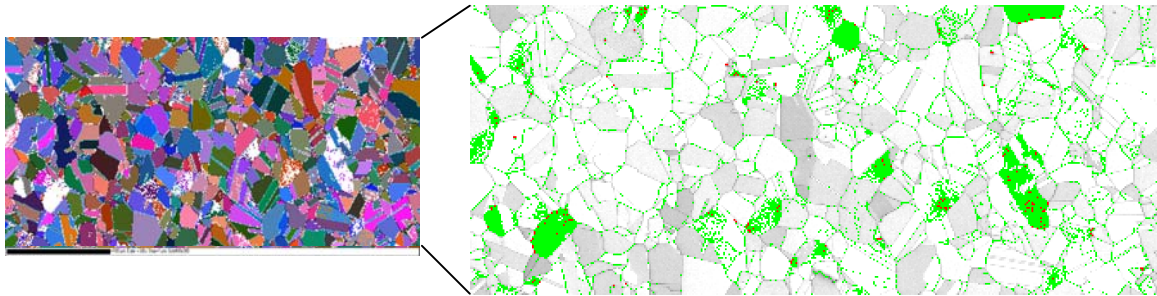
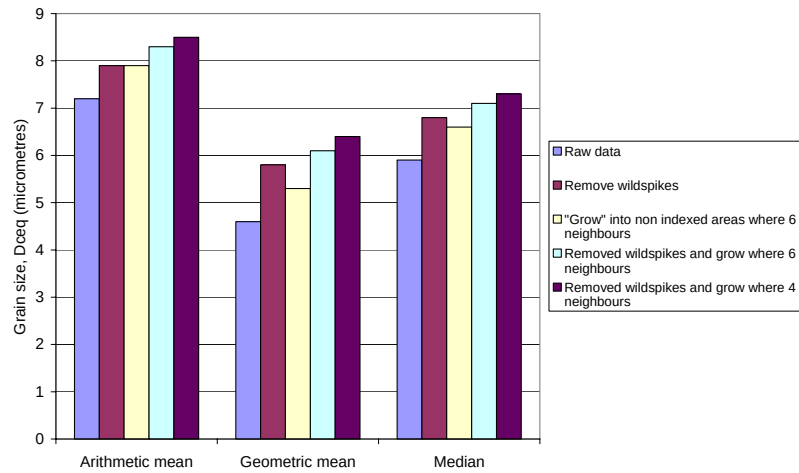


Figure 12. Map of Waspaloy showing position of virtually all small grains (in red) located next to non-indexed pixels (in green). Scale bar on left side = 100 μm .

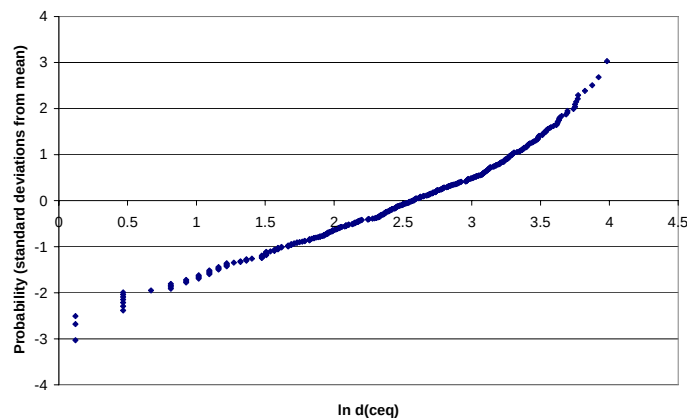
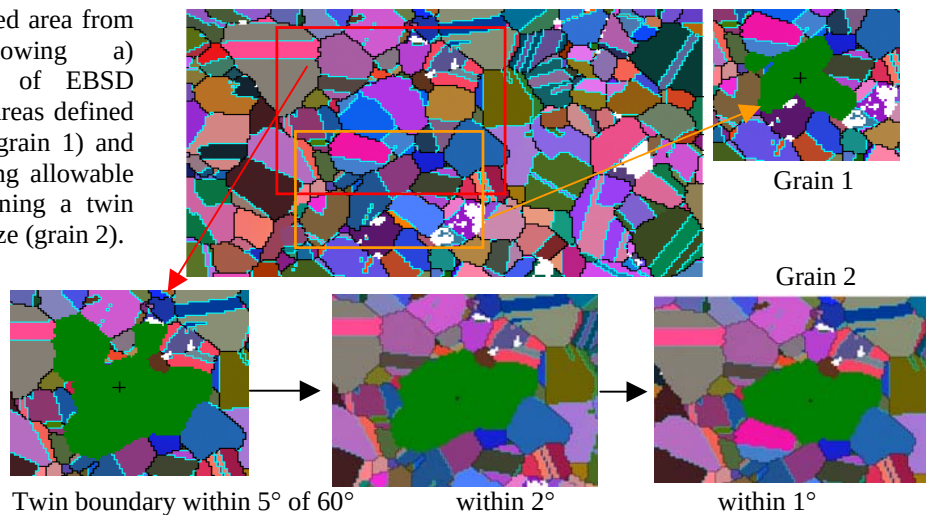


Figure 13. Probability plot of grain size distribution measured from Waspaloy map shown in Figure 12.

Figure 14. Enlarged area from Figure 12 showing a) unexpected effect of EBSD defining twins on areas defined as a single grain (grain 1) and b) effect of reducing allowable misorientation defining a twin on possible grain size (grain 2).



the non indexed zero solutions, so care must be taken in deciding if the noise reduction/infilling is a valid procedure. These results contrast with a simulation [1] where little error in grain size was predicted with variable indexing levels. It should be noted that the linear intercept d_{lin} measurements are much less sensitive to noise reduction since infilling of grain boundaries has little effect on intercept position.

The following example, from the Ni superalloy specimen Waspaloy, shows a case in which removal of wildspikes and very small grains is an essential part of obtaining a suitable grain size measurement.

Figure 12 shows a map of the microstructure produced with a 1 μm step size. The percentage indexed is 90.6%, very similar to that of Figure 1 for the 316 stainless steel, but in this case 24% of the grains identified in the raw map are wildspike 1 μm grains and a further 8% between 1 μm and 2 μm (compare with 9% and 4% for the same size ranges in the equivalent step size in Figure 2b). Inset is part of the map plotted as pattern quality (grey scale) to facilitate identification of grains 1 or 2 pixels in size (red). It can be seen that these almost all arise from larger grains in which the majority of the grain was unindexed (green pixels) because of poor pattern quality. In such cases it is valid to remove these wildspikes. The effect of doing this is to change the average arithmetic grain size from 11.2 to 16.5 μm , reducing the number of “grains” from 517 to 336. Growing the remaining grains where 4 neighbours of a non indexed cell increases the grain size to 17.8 μm .

Figure 13 is a probability plot of the grain size data after the noise reduction employed above. The removal of the wildspikes in this case does lead to a closer fit to a straight line; however, the deviation at large values appears to follow the same pattern seen in the case of the stainless steel.

8 Influence of grain boundary definition.

Section 2 explained how grain boundaries are defined by the misorientation between pixels. The effect on measured grain size of altering the limit to this misorientation has been explored for both the Waspaloy and stainless steel samples and also extended to the limits set on the precision with which twin boundaries

are defined, since this can also strongly influence what is defined as a grain.

Using the data for the 1 μm step size stainless steel map, mean d_{ceq} grain sizes were calculated for cases where grain boundaries were defined by minimum misorientation angles of 2°, 5°, 10° (used in the data previously discussed in this report) and 15°. Twin boundaries were defined as boundaries within 5° of theoretical.

Table 6. Effect of grain boundary definition on number average d_{ceq} grain size measurement

Misorientation angle defining a grain (degrees)	2	5	10	10 with twins $\pm 1^\circ$	15
Arithmetic mean (d_{ceq} , μm)	7.0	7.7	7.8	7.7	8.2
Geometric mean (d_{ceq} , μm)					
Median (d_{ceq} , μm)	5.5	6.5	6.6	6.3	6.8
Number of grains counted	886	778	724	768	648

Misorientations of 2° and 5° are normally taken to indicate sub-grain boundaries, but even if these are ignored, the results shown in Table 6 show that a difference of calculated mean grain size of about 5% can result from altering the definition of a grain boundary.

Figure 14 shows an enlargement of an area of Figure 12 together with examples of three grains as automatically identified by the software. Grain boundaries (where misorientation is $>10^\circ$) are shown in black, and twin boundaries (defined as 60° misorientations about the 111 axis, to within 5°). All the grains appear to be formed of several smaller grains which would not normally, if seen optically in an etched sample, be counted as one grain. By tightening the definition of the twin boundaries to only having a maximum deviation from the theoretical of 2° and 1° (which is close to the limitations on angular accuracy of the EBSD here) it was observed that grain 2, originally measured with a d_{ceq} of 53.7 μm , appears to be separated into several smaller grains.

15a.

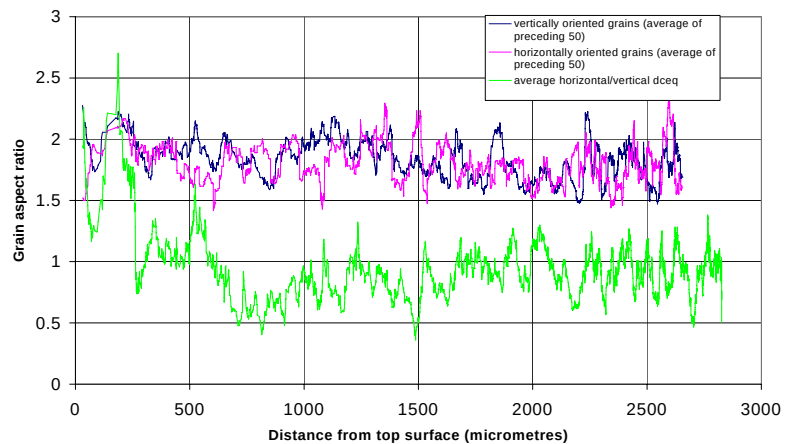
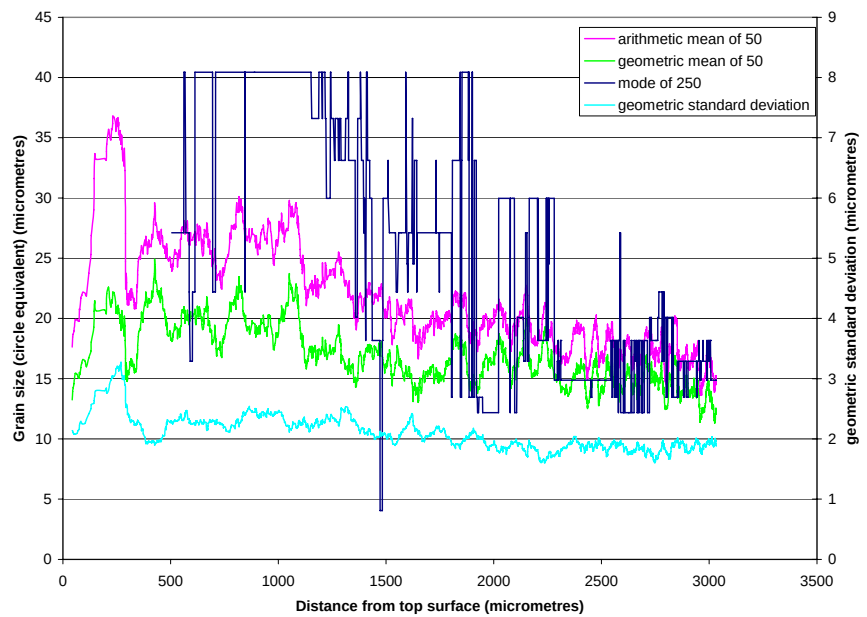
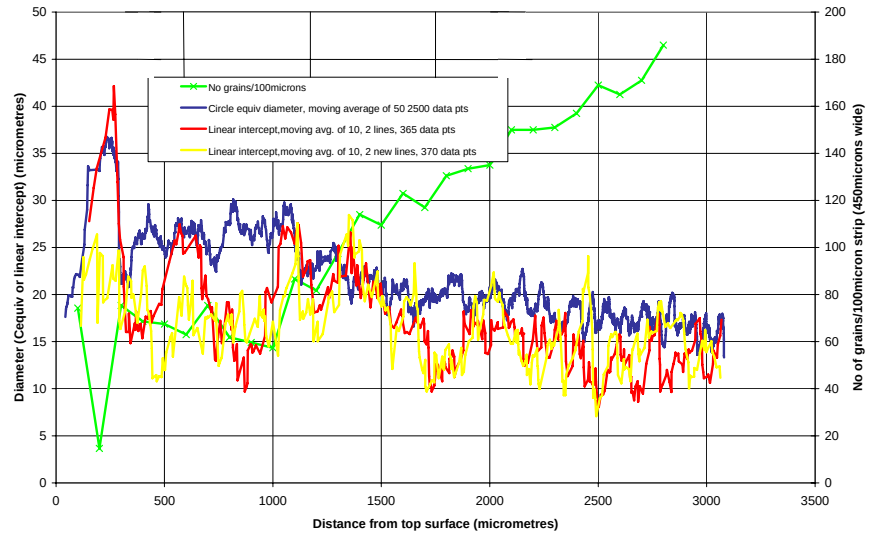
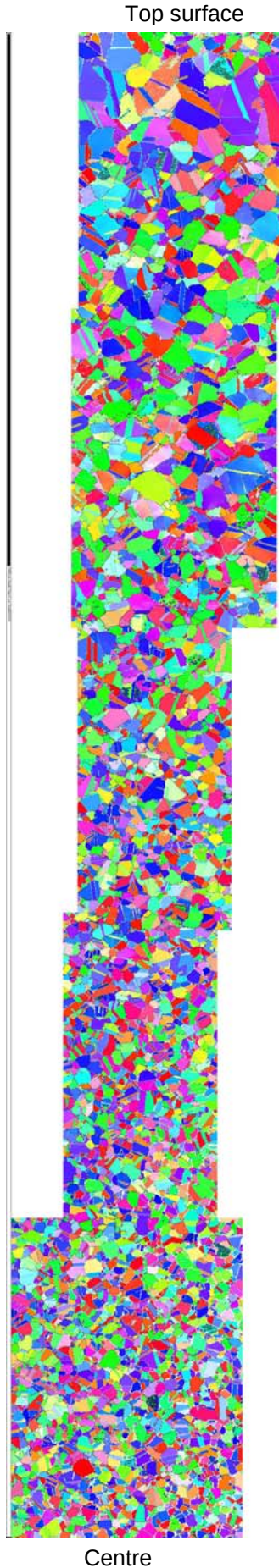


Figure 15 a) (left) map of hot deformed specimen
b) (top) graph of d_{lin} and d_{ceq} and grain number density
c) (middle) graph of arithmetic mean, geometric mean, geometric standard deviation and mode
d) (bottom) graph showing aspect ratios and mean grain size ratios in horizontal and vertical orientations

9 Application to assessment of heterogeneity in a hot deformed sample.

Figure 15 is an EBSD map (produced by stitching several smaller maps) through the hot deformed Waspaloy sample, axially from the top surface towards the centre of the sample. Wildspikes were removed and the grain areas grown outward (where 4 neighbours bordering) to fill in nonindexed boundaries. Figure 15b compares different measurements of grain size by linear intercept and circle equivalent diameter and grain density. The grain sizes are effectively arithmetic averages; because of the significantly reduced number of linear intercept values these are averaged over 10 grains whereas the d_{ceq} measurements average over 50.

Figure 15c plots arithmetic and geometric means as well as standard deviation and mode derived from the distribution of logarithmic values. The former gives an indication of the spread of results whereas the latter gives a representation of the peak of the distribution. Figure 15d shows the aspect ratios of grains oriented vertically and horizontally, and the ratio of the moving averages (50 grains) for grains oriented horizontally to those oriented vertically, suggesting little preferred orientation.

10 Summary

EBSD enables the rapid quantification of grain size parameters for planar sections from a large number of grains and in principle should thus give reproducible measurements from a representative sample of a microstructure. However even if the data are generated accurately (issues of calibration and instrumental setup have not been discussed in this note for reasons of space), automated software techniques can give a wide range of results, depending on a) the scale of the EBSD scan, b) how the raw data are analysed and manipulated to reduce noise with reference to the scale of the scan, c) the size parameter used (arithmetic or geometric mean or median

value), and d) the method of measurement (e.g. linear intercept or grain area) and e) how a grain boundary is defined. Differences with optical techniques will also result because of both calibration and resolution.

Because the EBSD method can map microstructures with high resolution it can detect small features which may produce very different results from optical measurements if present in high numbers. This probably explains why linear intercept measurements by EBSD yield smaller values than optical measurements and in turn why EBSD grain size area measurements yield smaller values than EBSD linear intercepts. EBSD linear intercept measurements have some advantages over grain size area methods, including better correspondence with conventional optical techniques, lower sensitivity to noise (because the chances of intercepting individual pixels is small) and lower sensitivity to step size and grain boundary width effects where, at small scan step sizes, EBSD does not index all pixels on or either side of a boundary. However, results in this note show the variability induced by positioning of the measurement lines can be significant even in an apparently uniform microstructure.

Determining grain size from measurement of grain area by EBSD can sample many more grains for a given area than the linear intercept method and should therefore give more representative results. Finer step sizes should give more accurate results, but may also reveal very small grains (or rather, small intersections with grains) which will skew the results if not handled correctly. For the results shown in this paper, the structure revealed at 0.5 μm steps size looks to be more accurate, which agrees with estimates [1] of step sizes needing to be a factor between 0.07 to 0.1 times the grain size for good accuracy. Grain size area measurement is, however, very sensitive to noise and/or methods used to reduce noise and ideally any noise reduction should only be applied after examining the location of wildspikes and taking into account their size relative to the rest of the size distribution.

For accurate and representative results a better function than the lognormal distribution needs to be derived which fits the typical distribution seen in Figure 2 (0.5 μm step size) and simulated by the theoretical model using an idealised tetrakaidecahedron grain shape. This function should take into account the long tail of very small values which will only be measured at high resolution and hence is unlikely to be seen by optical techniques. This tail means that the resulting measured distribution is not lognormal although possibly based on a distribution for which the 3D volume is actually lognormally distributed.

The following EBSD parameters should be stated in any measurement of grain size by this technique:

- step size;
- % points indexed and measured grain size before and after noise reduction;
- the noise reduction methods used;
- number of grains measured and an estimate of the 95% confidence interval.

Since little extra effort is usually required to generate linear intercept and circle equivalent diameter, results of both methods should be quoted where possible.

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