

NPL REPORT MAT 56

**Determination of grain size by EBSD – Report on a round robin
measurement of equiaxed Titanium**

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Approved on behalf of NPLML by Markys Cain, Knowledge Leader

ABSTRACT

An interlaboratory exercise was carried out across 12 laboratories to measure the average grain size of a titanium metal sample with an approximate average grain size of 30 μm using Electron Backscatter Diffraction (EBSD). Participants were asked to follow the proposed draft International Standard, ISO DIS13067 "Microbeam analysis-Electron Backscatter Diffraction-Measurement of Grain Size and Distribution".

Of the initial results submitted the equivalent circle diameter values from 4 of the 12 laboratories reported differed significantly from the overall mean value. Causes of these differences were identified in three cases and corrections made for two after which a full statistical treatment of the data was then used to remove remaining outliers.

The average grain size calculated from equivalent circle diameter measurements gave a value approximately 10% greater than calculated using linear intercept measurements. The results showed much greater variation of the average between laboratories (reproducibility) than between several measurements made by a single laboratory (repeatability). The reproducibility limit for equivalent circle diameter measurements was about 80% greater than that for linear intercept measurements, probably because additional errors from calibration drift and tilt correction perpendicular to the tilt axis contribute only to the former method.

Sources of differences in results are discussed and it is concluded that the choice of smallest grain size to be included in the calculation of the average has the greatest effect on the reported values. Choice of a relatively large cut off size is likely to give the best agreement because the smallest grains can have a significant effect (disproportionate to the area they occupy) and are also the grains most likely to vary in number and size with the step size chosen, the quality of the data and/or the treatment of poorly indexed points.

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1 INTRODUCTION

Electron Backscatter Diffraction (EBSD) is increasingly used to measure two dimensional grain sizes from sections through engineering materials. The application of the technique can be found in a wide range of applications and examples are readily found for aluminium alloys [1]; iron and steel [2]; hardmetals [3] and the effects of deformation on grain structure [4]. The technique is capable of measuring grain size (better than 100 nm) at and below the spatial resolution [5] of current methods such as optical microscopy. It also has advantages for providing additional information on the complex and refined structures that are increasingly required for advanced engineering applications. The metrological requirements for EBSD and in particular grain size measurement have been reviewed [6,7].

There have been several studies comparing the results of grain size measurements by optical and EBSD methods [1,6,8-12] and for thin films using FIB, AFM microscopies and EBSD [13]. In some of these works differences were observed in grain size between optical and EBSD and besides the greater spatial resolution of EBSD technique other factors were implicated, such as limitations of optical methods in detecting boundaries below a certain misorientation or grain boundary planes in optical methods and the variable effectiveness of etching in different samples affecting optical measurements.

EBSD is used for research and increasingly for quality control, but to fully exploit its potential, accurate knowledge is required of the issues affecting the reproducibility or repeatability of measurement to enable comparison not only of different EBSD data sets but also comparison with measurements by other methods.

Some previous comparative data exists on the reproducibility and repeatability of grain sizing by EBSD for establishing uncertainties associated with the method. An early intercomparison by Randle [14] *et al* in 2003 concluded that there was a wide disparity in results between laboratories. A more comprehensive Round Robin [15] looking at various aspects of EBSD was performed on 7 different materials, delivered by Nordic metal producing companies and investigated by up to 9 laboratories. Four of the laboratories acquitted themselves well. The author concluded that EBSD can do a very good job on very difficult materials, but the success depends on :

- Resolution of the SEM
- Type of EBSD-camera.
- Specimen preparation
- Operator skill

As a result a meeting was arranged to improve the quality of measurement in November 2004. The author of this report stated recently that in her opinion [16] the development of equipment and expertise since 2004 made a new round robin exercise appropriate.

This present report contains results from a preliminary interlaboratory comparison carried out to measure the grain size by EBSD in a titanium metal sample with an average grain size of approximately 30 μm . The sample is also amenable to optical microscopy methods of grain size measurement such as ASTM E112 [17]. This work is part supported by VAMAS TWA 37 Project 1 with the objective to validate the proposed International Standard, ISO/DIS ISO13067 “Microbeam analysis-Electron Backscatter Diffraction-Measurement of Grain Size and Distribution” prepared by ISO TC202/WG7.

To enable a round robin to be carried out in a reasonable timescale, multiple samples were prepared from a single source for distribution to participants rather than sending one sample on from laboratory to laboratory. While the latter route might be preferable, in theory enabling a better comparison between laboratories by eliminating sample to sample variability, in practice variable sample contamination from each EBSD mapping run accumulating with each analysis and handling damage

would require regular sample reparation, negating the benefit of a single sample as well as substantially slowing down the time to obtain results.

The laboratories participating in the round robin represented:

- 3 EBSD equipment manufacturers
- 5 National Measurement Institutes (NMIs)
- 1 university
- 2 industrial research laboratories

They also represented a wide geographical spread, coming from 8 countries: France; Germany; Sweden; UK; USA; South Africa; China and Brazil.

The main body of this report presents a summary of the results and discusses reasons for the observed variability of results in detail including:

- Magnification errors
- Orthogonality of scan
- Step size
- Specification of minimum grain size
- Clean up of map noise
- Tilt
- Stability of system

A complete list of the results and the statistical analysis are included in Appendix A.

2 ORGANISATION OF ROUND ROBIN

2.1 SAMPLE PREPARATION

Samples were cut using electrodischarge machining from a 15 mm diameter bar of commercial purity titanium to provide 2 mm thick slices. The slices were not kept in sequence and were randomly assigned an identifying number. To enable polishing of multiple samples the slices were hot-mounted in bakelite and then ground and polished by a conventional mechanical polishing route but finishing with a combined chemical-mechanical stage using colloidal silica diluted with 20% H₂O₂. The samples were marked with a hardness indent at one point on the edge and one at the approximate centre of the sample to assist with identification of positions where measurements were made.

Each sample was then imaged in a Zeiss Supra40 FEG-SEM and a minimum of one region mapped by EBSD adjacent to the central pyramidal hardness indent to determine an approximate grain size. While this only measured approximately 400 grains from a single region it was used to identify and reject samples with grain sizes outside a range of $\pm 5\%$ or approximately $\pm 1.5 \mu\text{m}$ of the overall mean size. Figure A1 in Appendix A shows the results of these measurements although it should be noted that a better indication of the homogeneity of the samples was subsequently obtained from the larger amounts of data produced by all the participants.

2.2 SAMPLE DISTRIBUTION AND COLLECTION OF RESULTS

Each laboratory was assigned a letter and it is not planned to reveal the identity of the laboratories to other laboratories. Pairs of samples were selected from the samples with grain sizes within the $\pm 5\%$ range and sent to the round robin participants. (Appendix B shows the full instruction and the return sheet). The principal points of the instructions given were to follow ISO/DIS13067 and to map 5 areas of the sample from close to the central indent. Some samples were returned from one participant and sent on to a second participant; this provided further information on the inter-laboratory variation in comparison with real sample to sample variation.

Information was requested about the SEM operating conditions and on the EBSD mapping parameters including the step size used and the method used to clean the data. Appendix B shows an example of

the information requested on the return sheet. Participants also returned the raw EBSD data so that data from different participants could be reanalysed in an identical manner (as far as different software/EBSD manufacturers allow) where this could help identify causes for differences.

After return of their results, participants were shown their data in relation to the values returned from the other laboratories and invited to comment and where appropriate reanalyse their measurements to see if discrepancies could be better understood.

3. RESULTS

3.1 SUMMARY OF LABORATORY REPORTED MEAN EQUIVALENT CIRCLE DIAMETER GRAIN SIZE - D_{circle}

Figure 1 summarises the data as returned from 11 laboratories, each denoted by a letter. Each laboratory measured two samples but in some cases the laboratories measured the two samples with two different microscopes and EBSD systems or in one case with two magnifications, in which case two sets of two values are shown. The basic operating parameters used by the laboratories varied quite widely and are summarised in Appendix A, Table A1 which also explains the differences for those laboratories that measured samples in two microscopes. It should also be noted that Laboratories A and F measured the same sets of samples; similarly the samples measured by C were then measured by H, and those measured by E were measured by L. The mean value of these raw results before removal of any outliers or corrections for errors (see section 3.4) was 33.7 μm .

The data shown for each sample in Figure 1 is the mean of the five (in one case eight for laboratory B) maps' mean equivalent circle diameter grain size D_{circle} acquired for each sample. The data is plotted as the difference from the grand mean value calculated from all the data after excluding a number of data points outlined in Appendix A. The actual values are also given in Table A4 in Appendix A. The error bars on Figure 1 show ± 1 standard deviation calculated from the 5 maps. The mean values are all simple number mean values i.e.

$$D = \frac{1}{N} \sum_{i=1}^N d_i \quad (1)$$

where D is the mean grain size (equivalent circle or linear intercept), N the total number of measurements and d_i the individual grain size measurements.

The instructions did not specify the type of mean to report but in all but one case the number mean was reported without comment on the type of mean value calculation. In a single case, the mean values were initially reported as area weighted means i.e.

$$D = \frac{\sum_{i=1}^N A_i d_i}{\sum_{i=1}^N A_i} \quad (2)$$

where A_i is the area of the grain with diameter d_i , but the results are reported here after recalculation as number means.

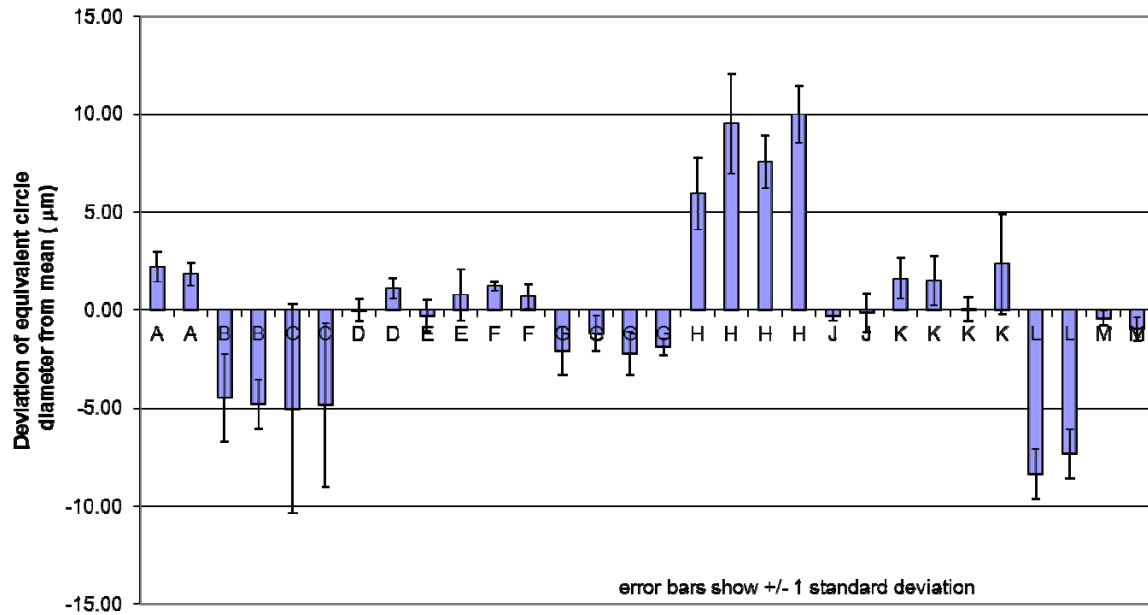


Figure 1 Mean values of D_{circle} for each sample as initially reported by the participants (before any corrections noted in section 4) Values are plotted as the difference from the mean of $33.7\mu\text{m}$. Error bars show ± 1 standard deviation from 5 maps contributing to the mean. Actual values are reported in Appendix A.

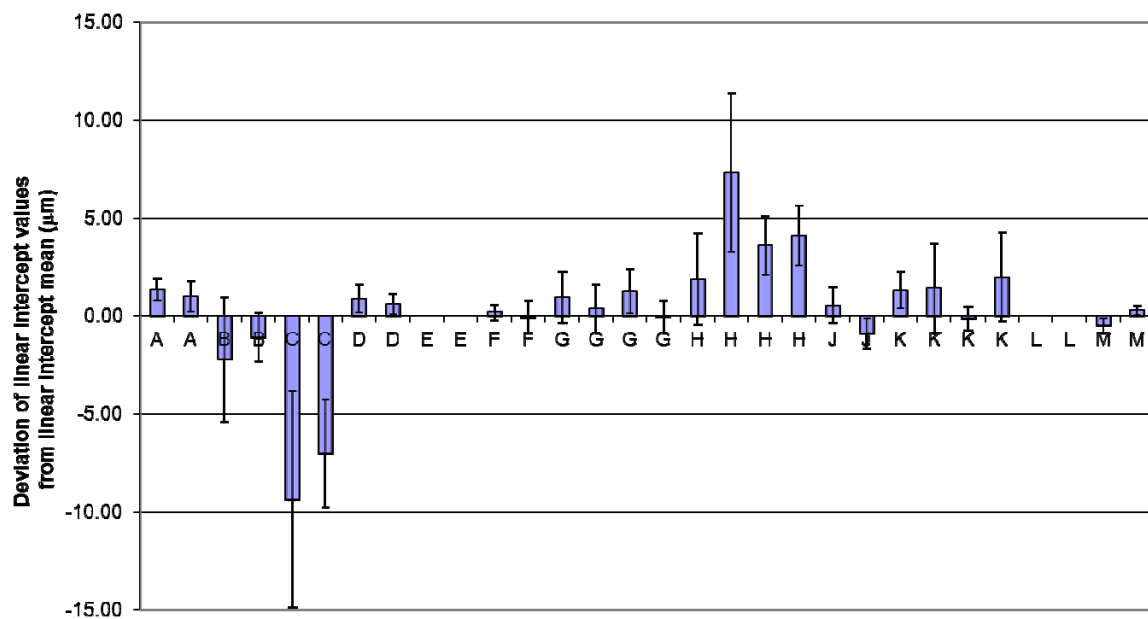


Figure 2. Mean values of D_{lin} for each sample as initially reported by the participants (before any corrections noted in section 4) Values are plotted as the difference from the mean of $30.0\mu\text{m}$ (calculated after exclusion of laboratory L and second set of H). Error bars show ± 1 standard deviation from 5 maps contributing to the mean. Actual values are reported in Appendix A.

The average difference across all the laboratories between samples measured by the same laboratory is 2.9%, or after excluding the results of laboratory H, 2.1%.

3.2 LINEAR INTERCEPT GRAIN SIZE

Figure 2 shows the results, equivalent to those in Figure 1, of using linear intercepts to measure the linear intercept mean grain size D_{lin} . Before exclusion of outliers, this mean value was 30.0 μm . Note that laboratories E and L did not report linear intercept values. Figure 3 shows the same results but with reference to the mean equivalent circle grain size used in Figure 1, from which it is clear that the linear intercept values were approximately 10% smaller than equivalent circle diameters. Interestingly the individual laboratory variability given by the standard deviation on each sample is very similar between D_{circle} and D_{lin} (1.4 and 1.6 μm respectively) but the overall spread of results between laboratories, given by the standard deviation of the laboratory mean values is reduced from 3.0 to 1.8 μm , i.e. 40%, if laboratory C is excluded.

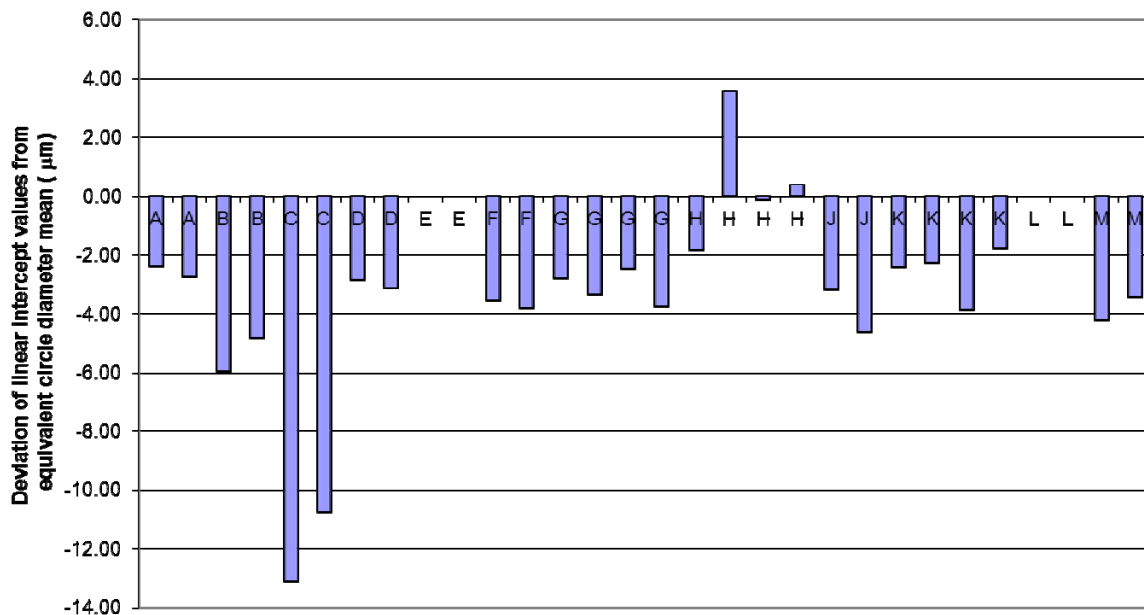


Figure 3. Mean values of D_{lin} for each sample as shown in Figure 2 but plotted as the difference from the mean D_{circle} value used for Figure 1.

3.3 GRAIN SIZE DISTRIBUTION

In measuring the mean grain size, EBSD calculates the size of all the grains observed and thus a full size distribution can be plotted. Figure 4 shows the distribution of equivalent circle diameter sizes on both log and linear scales for clarity of, respectively, the smaller and large diameter ends of the distribution. Data sets have been taken from one or more maps for a single sample for each lab with the exception of Laboratory C for which the wide range of results has already been noted. With the exceptions of Laboratories B and L (see section 4 for further discussion) the spread of results at the 50% fraction, d_{50} is about 5 μm , and this is maintained at d_{10} and d_{90} . However, the maximum size observed varied from <100 μm to >120 μm and the smallest size retained also changes (section 4.3).

3.4 SUMMARY OF RESULTS

In both sections 3.1 and 3.2 it was noted that some values appeared to lie outside the main range of results. The results were analysed statistically following the procedures outlined in ISO5725-2 and fuller details are given in Appendix A. This shows that a number of results could be classified statistically as outliers. Reasons for the occurrence of these outliers are discussed in the following sections and after excluding these outliers, the final results of the round robin may be summarised as follows:

Table 1. Summary of results after statistical evaluation in Appendix A (repeat of Table A10) including exclusion of outliers

		Equivalent Circle Diameter D_{circle}	Linear Intercept D_{lin}
Overall Mean (μm)		33.5	30.1
Repeatability standard deviation (μm)	S_r	0.88	0.74
Reproducibility standard deviation (μm)	S_R	1.98	1.15
Repeatability limit (μm)	r	2.46	2.07
Reproducibility limit (μm)	R	5.55	3.22
Number of Laboratories after removal of outliers		12	11

The repeatability and reproducibility limits are the values less than or equal to which the absolute difference between two test results obtained under repeatability/reproducibility conditions may be expected to be with a probability of 95%.

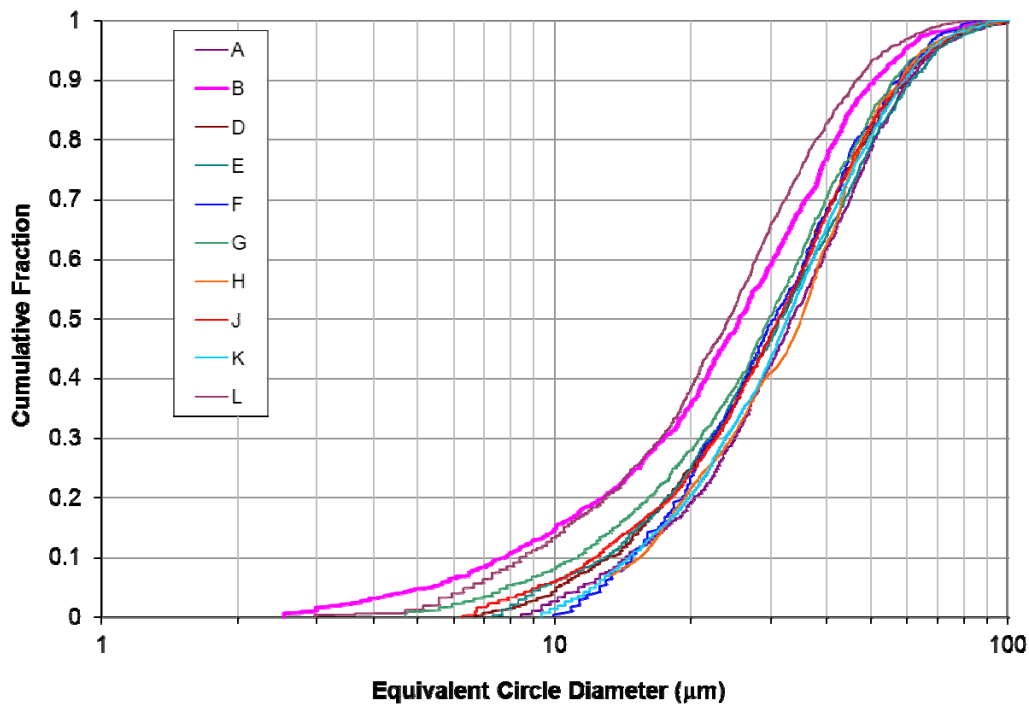


Figure 4a Cumulative distribution of equivalent circle diameter grain sizes plotted on a log scale.

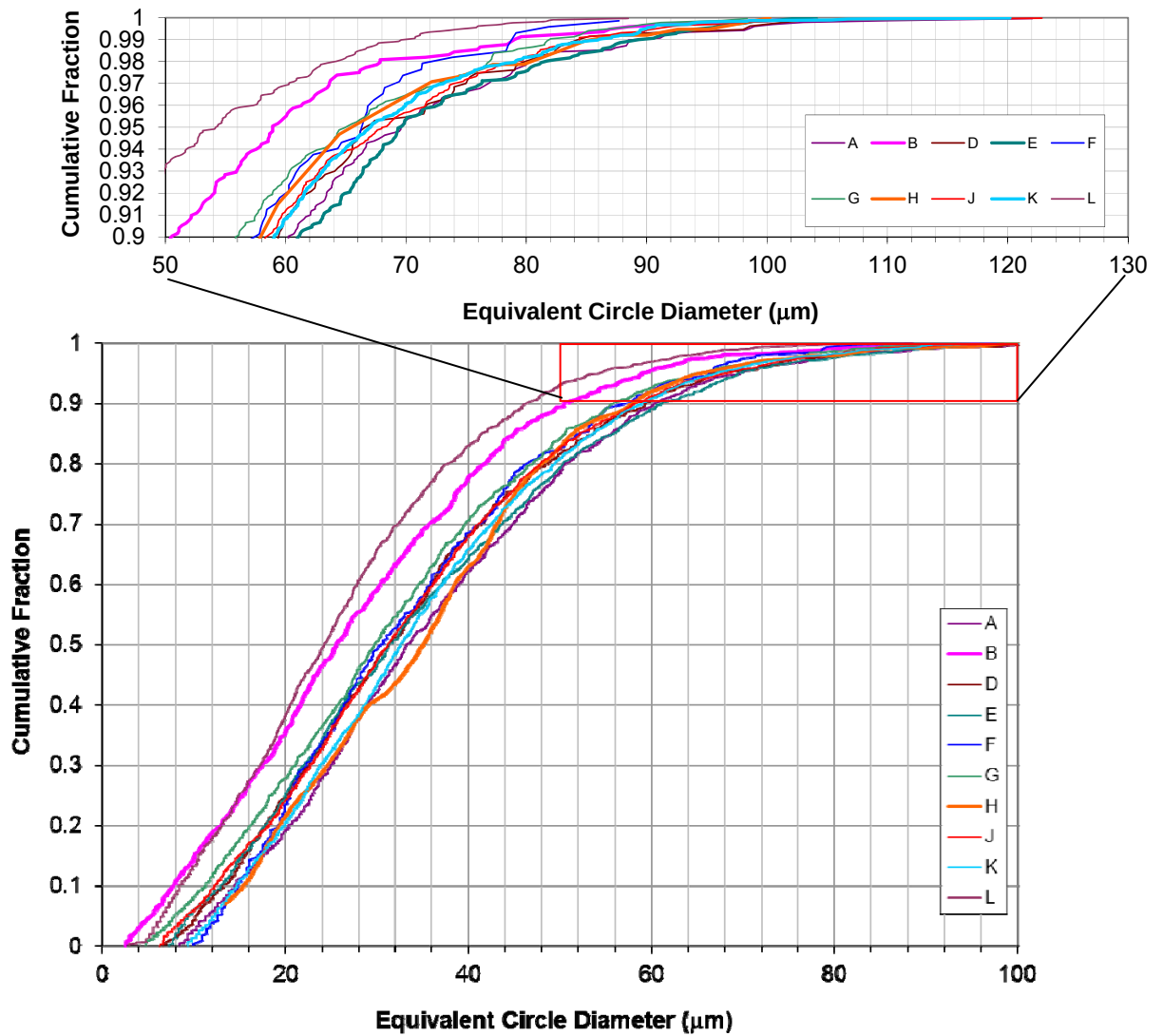


Figure 4b Cumulative distribution of grain sizes plotted on linear scale with enlargement showing the distribution of the largest 10% of grains

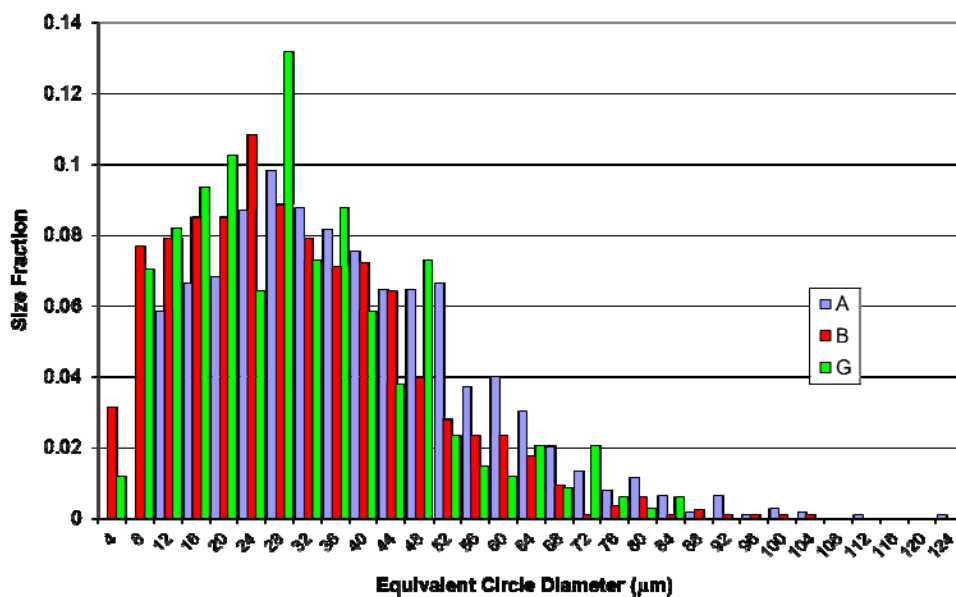


Figure 4c Histogram of the equivalent circle diameter grain sizes measured by three laboratories representing distributions at the lower, middle and top end of the ranges reported.

4. CAUSES OF INTERLABORATORY VARIATION IN MEASUREMENTS OF D_{CIRCLE}

Before exclusion of any outliers the mean value of D_{circle} was $33.7 \mu\text{m}$ but with a spread ($\pm 2\sigma$) of $\pm 8.6 \mu\text{m}$. This wide spread can be attributed largely to significantly different values reported by just 4 of the laboratories, B, C, H and L. If these were completely excluded the mean value changes little ($34.0 \mu\text{m}$), but the spread reduces to $\pm 2.9 \mu\text{m}$ ($\pm 2\sigma$). The causes of variation for the 4 laboratories B, C, H and L will therefore be discussed in more depth, using the data in Appendix A (Table A2 documents the primary factors reported in preparation and analysis of the EBSD maps and operating parameters in Table A1) and the maps supplied with the measurement data to identify some of the principle causes for much of the variation.

4.1 MAGNIFICATION ERRORS

Laboratory H was the only participant to report their calibration procedure for magnification in the initial returns. However, when shown that their results were significantly larger than those from all the other laboratories they re-examined their procedure. This showed that the initial calibration had not actually altered the magnification factors in the EBSD software so that the maps were not incorrect by a single scaling number but were distorted (stretched) in the Y direction by a further 13%. Applying the correction factors to the already measured mean sizes produced new values much closer to the grand mean, as shown in Figure 5.

Laboratory L rechecked their magnification calibration on seeing the complete set of results and although confirmed it to be correct, did find that problems with dynamic focus at low magnifications gave very poor image orthogonality and may have caused the low values measured. No recalculation of the results was performed.

It may be noted in passing that, in the authors' experience, some SEM/EBSD software combinations allow changes in the SEM recorded magnification value (e.g. from screen magnification to print size magnification), causing the EBSD software to use the new magnification value (print size) even though it would only have been set up with the previous (screen) magnification value. This results in the wrong sizes being measured by EBSD even though the system was originally calibrated correctly: comparison of scale marker bars on SEM acquired images and EBSD acquired images or maps will avoid propagation of this mistake.

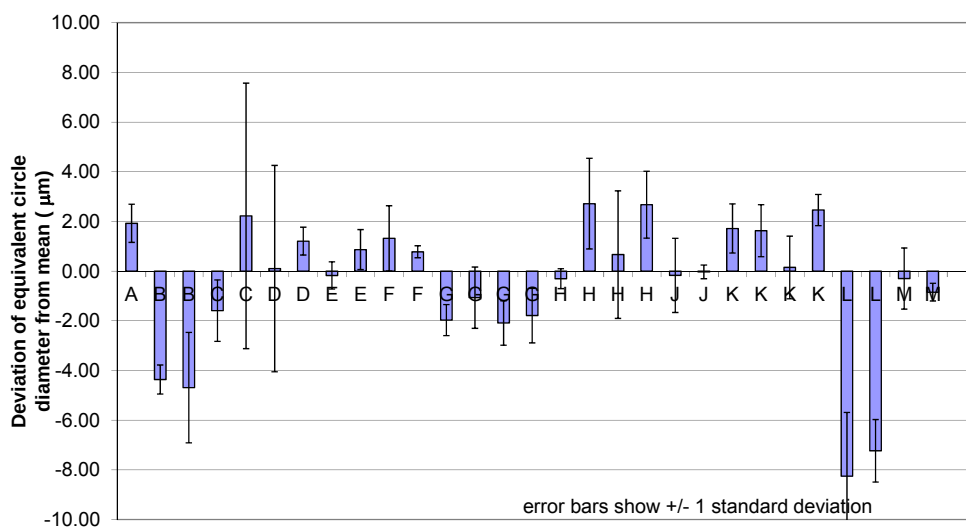


Figure 5 Mean values of D_{circle} for each sample as initially reported by the participants (as plotted in Figure 1) except for Laboratories C and H. For these two laboratories the values are shown after applying the correction where corrections described in Sections 4.4 and 4.1 respectively.

4.2 STEP SIZE AND SAMPLE SIZE

An obvious difference between Laboratory B and all the other laboratories is the use of a fine step size of 1 μm and the consequent relatively small areas mapped, containing only 70 grains on average over 8 maps (560 grains in total), compared with anywhere between 300 to over 1000 grains over 5 maps (i.e 1500 to 5000 in total) in other laboratories. The fine step size means that small grains will be mapped in detail which at coarser step sizes would not be detected or would only be mapped by a few pixels and therefore discarded as being inaccurately characterised. The following section discusses this in more detail, but as an example, Figure 6a shows a map from Laboratory B in which grains with equivalent circle diameters $< 10 \mu\text{m}$ are shown in red that are likely not to have been mapped or to have been discarded in data analysis by other laboratories. The small area mapped as a consequence of the finer step size increases the probability of large grains intersecting the edge of the map and therefore being discarded; Figure 6a also illustrates this with one large grain marked out by a yellow line.

It is also worth discussing at this point the minimum number of grains that need to be measured to obtain a stable average value. Figure 6b plots the running average grain size values from single maps for Laboratories A, B and K (D_{circle}) and J (D_{lin}) and from all the maps combined from Laboratory B. This shows that, at least for this material, a minimum of between 200 and 300 grains is sufficient to obtain a stable average that will change little as further grains are measured. This seems to apply even to the combined results of the incomplete sampling of the Laboratory B.

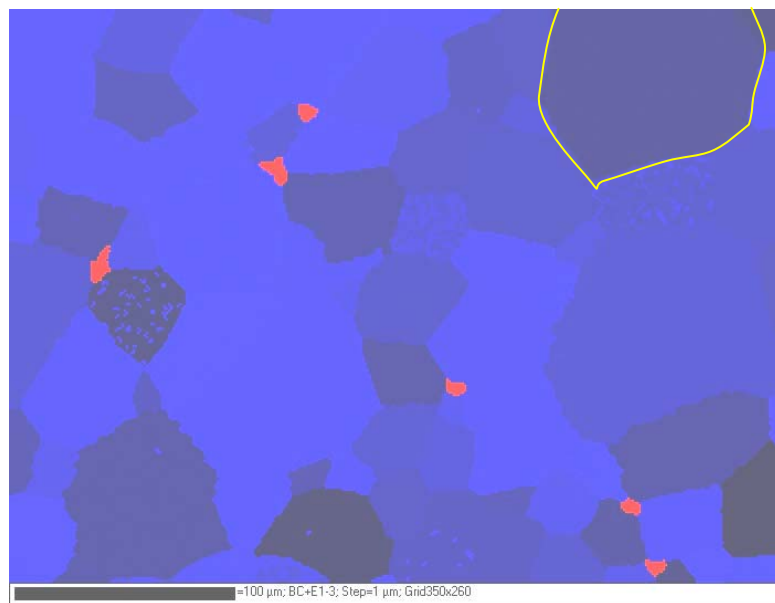


Figure 6a. EBSD map produced with a 1 μm step size, showing the general grain structure in a blue scale with grains with $D_{\text{circle}} < 10 \mu\text{m}$ highlighted in red. Also highlighted with a yellow boundary is the largest grain in the area mapped but which has been excluded from the calculation of mean grain size because it is touching a map edge and thus has not been fully mapped.

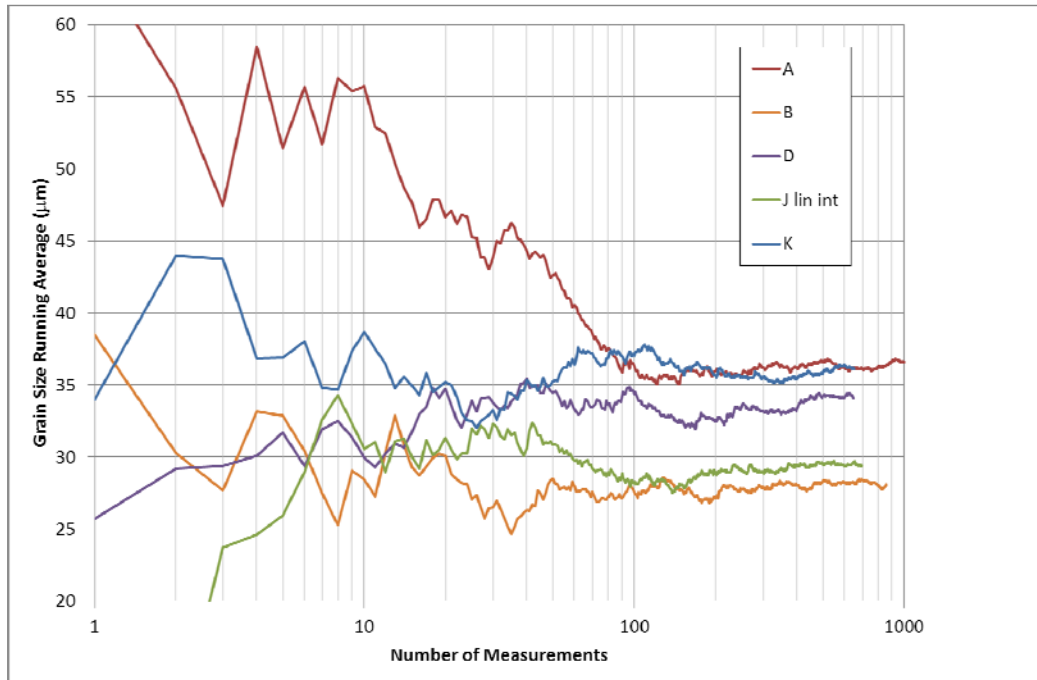


Figure 6b. Grain size running average plotted from the data from single maps for Laboratories A, B and K (D_{circle}) and J (D_{lin}) and from all the maps combined from Laboratory B

4.3 SPECIFICATION OF MINIMUM GRAIN SIZE INCLUDED IN CALCULATIONS

It recommended in the Standard ISO FDIS13067 that grains >10 pixels in area will give valid results since EBSD validates each pixel in a grain by measuring its orientation. Pixellation errors, which alter the true grain size at small sizes, are approximately 5% at the 10 pixel area level. This cut off limit also agrees approximately with conventional image analysis where the minimum size of an object is 9 (3×3) pixels for a square grid or 7 for a hexagonal grid to avoid erosion of step size 1 deleting an object completely.

In the round robin however a variety of cut off limits were used. Most were stated in terms of pixels although a couple were in terms of a minimum size in micrometres and laboratory C did not state what cut off was used. The cut offs selected by the participants included $> 1, 4, 5, 6$ or 8 pixels, but most chose 10 pixels, although there was variation in whether the cut offs were $>$, "greater than", or $\geq$, "equal to or greater than". Because of the variation in step sizes chosen, the actual sizes of the cut off limits varied from $2 \mu\text{m}$ to $10.75 \mu\text{m}$, equivalent to $3.46 \mu\text{m}^2$ to $91 \mu\text{m}^2$. Figure 7 plots the mean grain size against the cut off limit used (where this was known). The cut off is plotted in micrometres rather than pixels to take into account the step size used as well as the number of pixels and suggests that the mean size initially increases before beginning to plateau as the cut off is increased. (If plotted against pixel size a more linear relationship was observed).

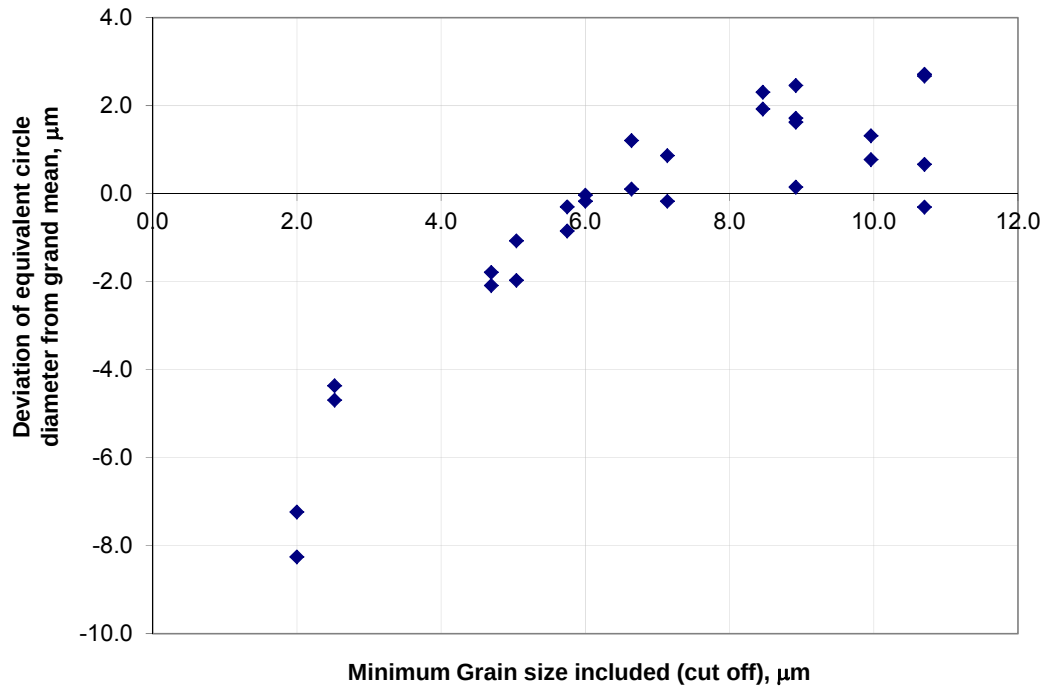


Figure 7. Mean values of D_{circle} for each sample (calculated as the difference from the overall mean value) plotted against the cut off minimum grain size (expressed in μm , not pixels) used by each participant in the calculation of their sample mean size.

The data supplied by individual laboratories has been re-examined to give a clearer idea of the effects of varying the cut off limit. Figure 8 plots a re-analysis of the average grain size against different cut off limits using the data files supplied by some of the laboratories for which the authors had access to the necessary EBSD software. To limit the reanalysis to a manageable amount, data from maps from only one sample to give at least 1000 grains was considered so the absolute values plotted do not coincide exactly with the average reported by each laboratory plotted in Figure 1. In some cases the raw data files supplied were analysed and then cleaned using the laboratory's own method before reanalysing; in other cases only the cleaned data was supplied and so only this data could be plotted with consequent limits on how low a cut off value could be used. The data plotted covers the range of step sizes used, from 1 μm to 3 μm .

For all but two of the laboratories (B and L) choosing a single cut off value above 8 μm i.e. specified in terms of micrometres rather than pixels would give agreement between all the results of about 2 μm . It should be noted that good agreement would be achieved at any given cut off above about 8 μm , as long as a single consistent cut off is applied to all the data.

For Laboratory B with the small 1 μm step size, at any given cut off, the mean value would still be 2 μm outside of the main group of data, although this value of 2 μm is closer than the discrepancy in the original results with a cut off quoted in pixels. It is possible that the difference of 2 μm even after applying a consistent cut off was caused by the exclusion of large grains discussed in Section 4.2. It should also be pointed out here that the samples mapped by Laboratory B were also mapped by Laboratory J (see Appendix A) which measured values very close to the overall mean. Laboratory L remained significantly lower than the trend indicated by the data in Figure 8, indicating that the data analysis was not the main cause of the difference and suggesting the problem of non-orthogonality in the size calibration of X and Y directions noted in Section 4.1 was the major cause.

It should also be remarked that choosing a suitable cut off minimises the risk of poor data cleaning by removing most or all of the poorly indexed pixels although if too large a cut off is chosen before data

cleaning, subsequent reindexing of the removed pixels may lead to overestimation of the remaining grains (see following sections).

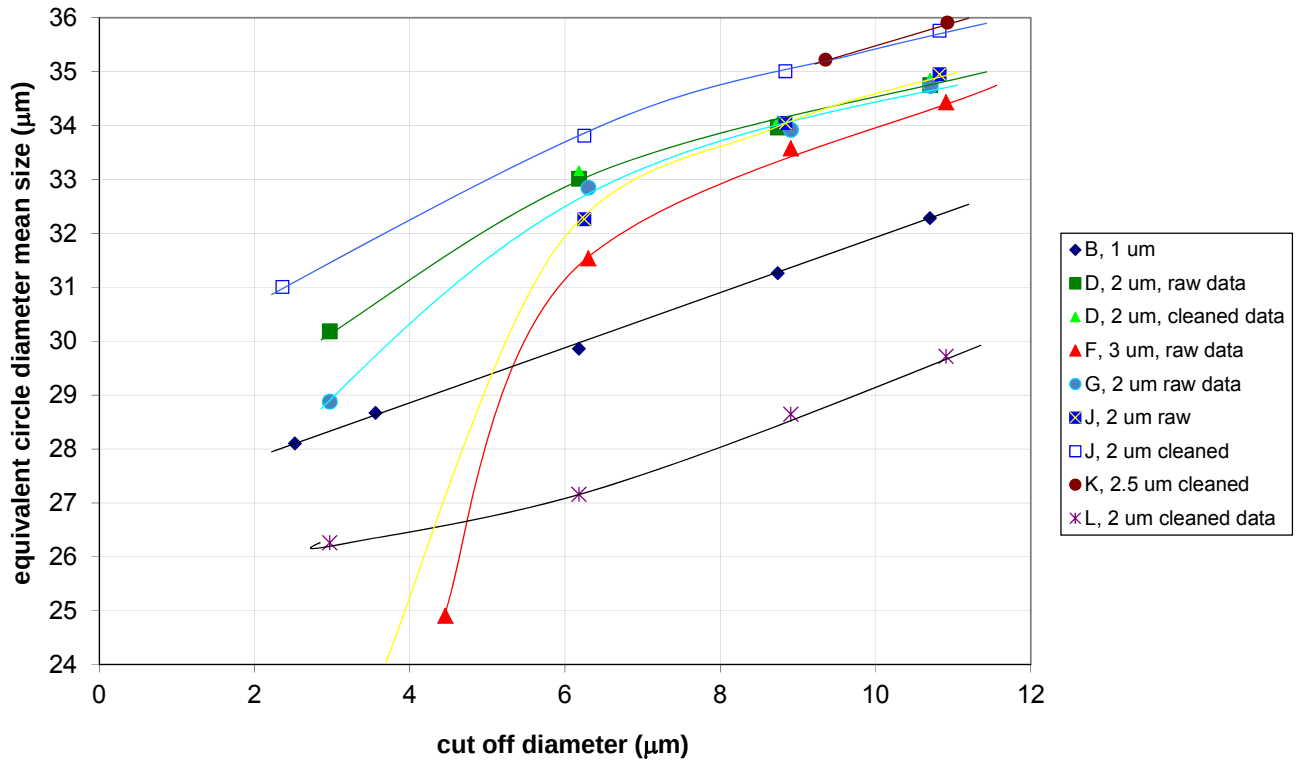


Figure 8. Data illustrating the variation of the mean value of D_{circle} that can be obtained as the cut off minimum grain size is varied.

4.4 CLEANUP OF MAP “NOISE”

Correction of mis- and non-indexed points will be considered in detail in Section 5, but in trying here to account for the specific outliers of Figure 1, Laboratory C presents a very obvious case. It is immediately noticeable from Figure 1a that the spread of results between all the maps for both Laboratory C samples was between 2 and 5 times greater than for the other laboratories and examination of the maps from which the data was taken showed large numbers of very small grains in some maps that had not been removed by correction or cleaning of the data (compare Figure 9a and 9b) and which therefore reduced the number mean grain size significantly. It was not clear from the information supplied by Laboratory C whether a cut off was applied and how noise reduction was applied. On reanalysis by Laboratory C a reduced number of small grains was obtained and the corrected values were much closer to the mean as shown in Figure 5.

Some orientations of the Ti hcp phase are likely to produce diffraction patterns exhibiting pseudosymmetry [18] which accounted for a misindexed pixels for a number of laboratories. One laboratory in particular reported this issue in detail and noted that corrections for several pseudosymmetric orientations enabled a significant reduction in noise.

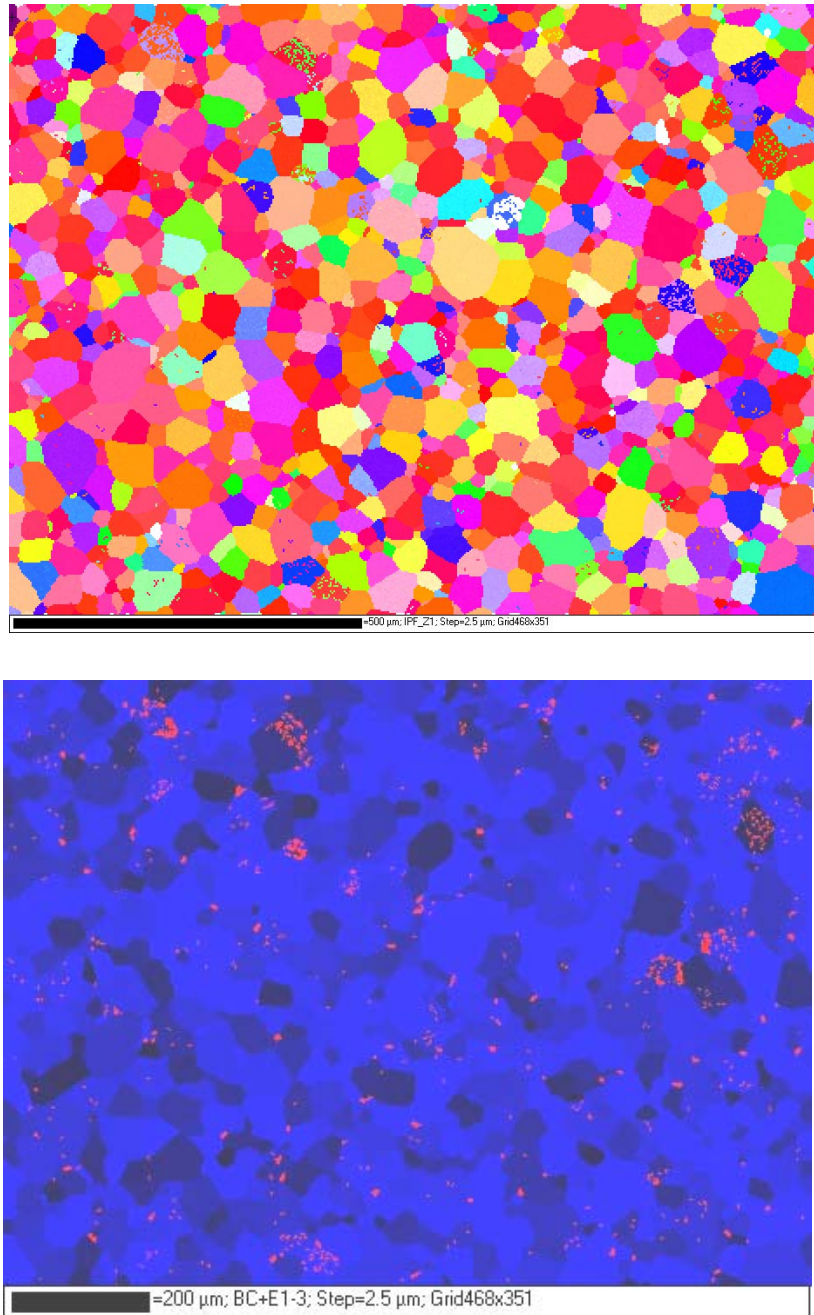
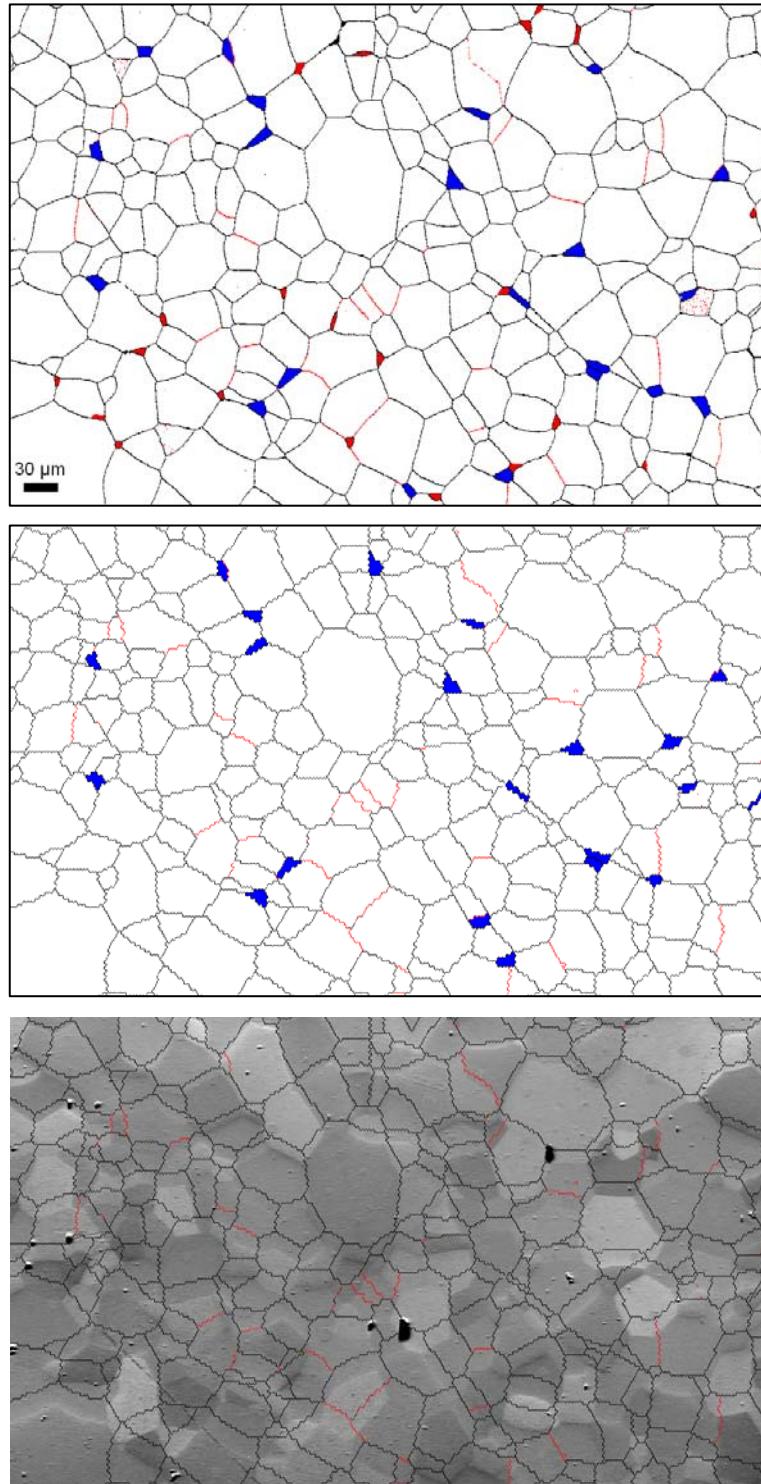


Figure 9 a (top) and 9b (bottom). EBSD maps from Laboratory C prior to clean up of map noise, showing: a) inverse pole figure orientation colour maps of the grain structure (note pseudosymmetry indexing problems in some grains), and b) in blue scale with grains with $D_{\text{circle}} < 10 \mu\text{m}$ highlighted in red.

4.5 INVESTIGATION OF OTHER POTENTIAL SOURCES OF ERROR

The above discussion does not include all the possible sources of error or reasons for differences in results. The following results were produced to illustrate some of these sources of error by mapping a single region from a round robin sample at the National Physical Laboratory. The maps in Figure 10 a and b show the overlap of two regions which were mapped at step sizes of $0.5 \mu\text{m}$ and $3 \mu\text{m}$ respectively; Figure 10c shows the boundaries of 10b overlayed on the forescatter SEM image acquired just before mapping. In addition the same area was mapped with the specimen tilted by a further 2° from the set value of 70° tilt.



Figures 10a (top), b (middle) and c (bottom), showing grain boundaries produced by EBSD mapping of a single region at step sizes of a) 0.5 μm and b) and c) 3 μm . Grains with $D_{\text{circle}} < 5 \mu\text{m}$ are shown in black (only just visible in top middle of a), grains with sizes in the range $5 \mu\text{m} < D_{\text{circle}} < 10 \mu\text{m}$ in red and in the range $10 \mu\text{m} < D_{\text{circle}} < 15 \mu\text{m}$ in blue. Boundaries with a misorientation difference of $>15^\circ$ are shown in black those with a misorientation difference between 5° and 15° in red.

Figure 10c) shows the boundaries of 10b overlayed on the forescatter SEM image acquired just before mapping, with drift during mapping visible. It should also be noted that although of the same width as Figure 10a, Figures 10b and 10c are 7% less in height.

In Figure 10 it is clear that at the 3 μm step size, after clean up of the data all of the grains with D_{circle} smaller than 10 μm are not mapped, and a few < 15 μm in size have also been missed. A significant number of boundaries between 5° and 15° can also be seen. The effect of these differences on the calculated mean grain size is shown in Table 2.

Table 2. Effect of step size, grain boundary definition and cut off on grain size measurement for maps shown in Figure10.

Imaging conditions	0.5 μm		3 μm	3 μm + 2° tilt
Cut off (μm)	2 μm	10 μm	10 μm	10 μm
5° grain boundary angle	30.5	35.9	34.9	33.6
15° grain boundary angle	31.8	37.5	36.9	35.4

Also visible from Figure 10 is the effect of specimen drift: firstly by comparison of Figures 10a and 10b, in which it can be seen that the largest grain in the centre of the map is elongated at the smaller step size, while in Figure 10c the superposition of the grain boundaries shows good co-incidence at the top of the image but an increasing discrepancy in the vertical direction towards the bottom of the image. The difference between the results in Table 2 for the two step sizes with the same cut off will largely result from the extra drift that would have occurred under the slower acquisition conditions of the smaller step size. It can also be seen that an extra 2 degrees of tilt of the specimen led to a smaller grain size, since during mapping the beam will move in the y-direction more than is actually required to step a given distance by a factor of $\cos(72)/\cos(70)$ and thus foreshorten the image in the y direction. Since the grain size is calculated from an area, the actual size reduction this produces will be:

$$\sqrt{x.y} = \sqrt{\cos(72)/\cos(70)}$$

giving a factor of 0.95.

A detailed examination of the effects of large tilt angles on distortions and errors can be found in Nolze [19].

In Table A2 it can be seen that most laboratories used a misorientation angle of 5° to define grain boundaries, but several also used 10°. All laboratories stated a 70° tilt, but this value is extremely difficult to measure accurately on an actual sample, and is a value generally not calibrated during regular use of an SEM. For the purposes of the round robin it has to be assumed that there was no drift during acquisition of the data submitted by the participants, and while most laboratories would normally check sample stability before carrying out mapping, this was not actually reported on any of the return sheets.

With the exception of step size which has been discussed earlier, of the possible sources of error above, it is thought that the accuracy of the tilt used during acquisition is the most likely to have caused additional errors in the sizes reported.

4.6 DIFFERENCES BETWEEN EQUIVALENT CIRCLE DIAMETER AND LINEAR INTERCEPT MEASUREMENTS

Several factors stand out when the results of measuring D_{circle} and D_{lin} are compared:

- i) Comparing the overall mean values, $\frac{D_{\text{circle}}}{D_{\text{lin}}} = 1.09$, which is close to the ratio suggested in the appendix to ISO13067 [A1]
- ii) The repeatability of the two measurements is very similar ($r_{\text{circle}} = 2.54$ is 20% greater than $r_{\text{lin}} = 2.13$), but the reproducibility of D_{lin} is much better than that of D_{circle} ($R_{\text{circle}} = 5.79$ is 70% greater than $R_{\text{lin}} = 3.36$).
- iii) Only one laboratory (A) reported linear intercept measurements in both X and Y directions (although the form did not state whether results should be derived from measurements made in one or both directions or an average of the two).
- iv) Only one laboratory (again, Laboratory A) explicitly reported using a cut off for minimum linear intercept size in the same way as a cut off value was reported for the equivalent circle diameter. *This point illustrates the difference between removing small numbers of pixels because of reducing noise/indexing errors and removing small numbers of pixels after data cleaning/noise reduction because they may be measured inaccurately.*

It has already been noted in the preceding section that errors in tilt and specimen drift can alter measurements in the vertical/Y direction without affecting the accuracy of measurements in the horizontal/X (tilt axis) direction so it seems reasonable to surmise that the reduced repeatability and particularly the reduced reproducibility in the measurement of D_{circle} results from the additional errors in measurement of dimensions in the Y direction which contribute to the measurement of area but not to the horizontal linear intercept measurement.

For isotropic materials measurement of the linear intercept in both X and Y directions would be useful to identify any tilt or drift errors from significant differences between the two values. To investigate this with the current data, the sets of maps from two samples from two participants were analysed. The results of subtracting the mean linear intercept measured in the Y direction from those in the X direction are plotted in Figure 11. The mean values were calculated from approximately 750 intercepts for each map in each direction after applying a cut off of 10 μm .

The variation between X and Y directions for the two samples measured by Laboratory A appears to be generally random, while for the samples measured by Laboratory J there is a consistent difference in one direction for one sample and in the other direction for the second. Without knowing if the samples were fixed in position for all five maps for each sample it is difficult to draw any firm conclusions, but there is some suggestion here of possible tilt errors in one case.

The same investigation could not be carried out on maps made with a hexagonal grid because intersections of the grid in the X direction are different from the intersections in the Y direction as seen in Figure 11b. It can be seen that a vertical line can result in many intersections of a vertical boundary where a horizontal line will intersect either one grain or another where there is a horizontal boundary. Definition of a cut off value for the linear intercept would reduce this effect.

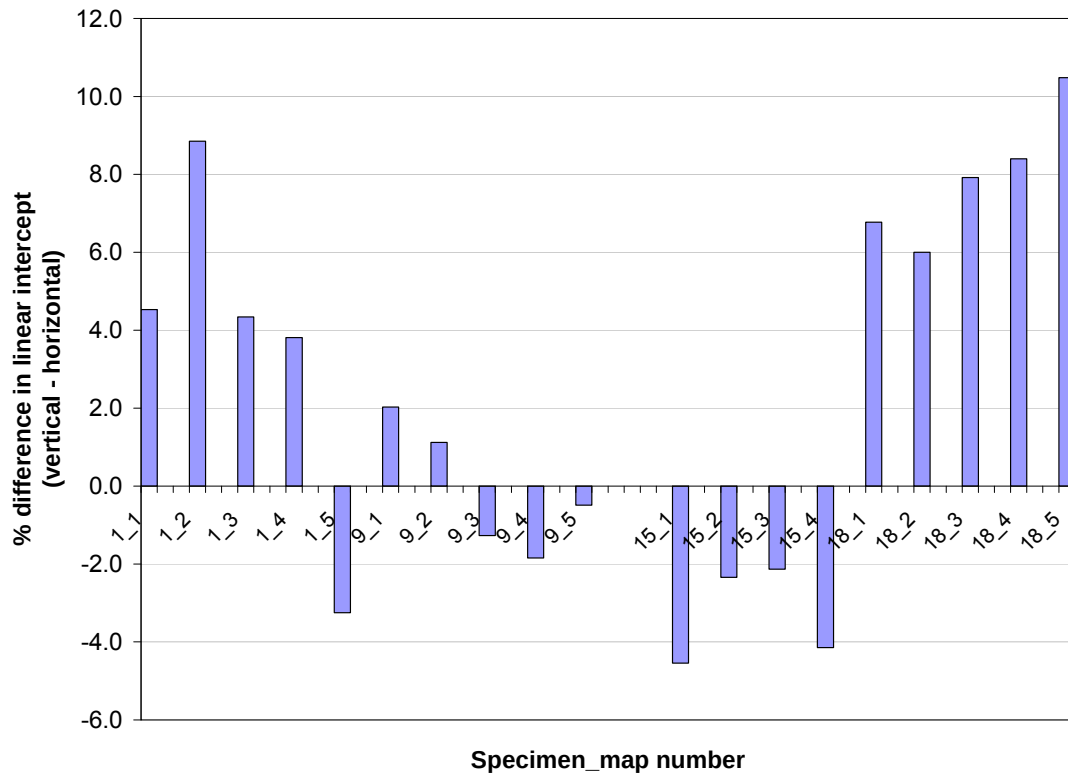


Figure 11a. Difference between measurement of mean linear intercepts in Y and X directions for individual maps taken from four samples (1 and 9, Lab A and 15 and 18, Lab J) from two laboratories.

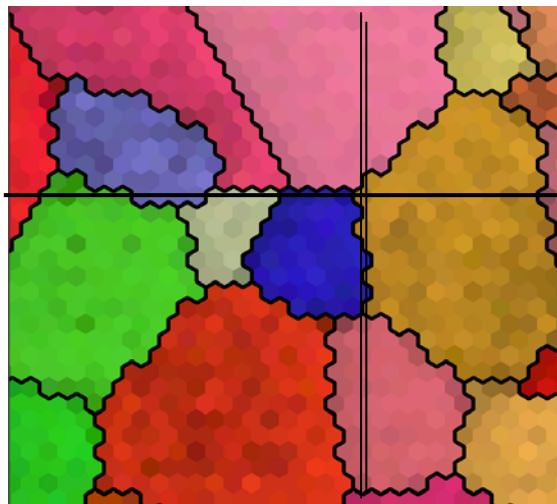


Figure 11b. EBSD map produced with a hexagonal grid showing the difficulty of measuring linear intercepts in the vertical direction because of the increased number of intercepts made with boundaries lying close to the vertical. compared with the horizontal direction.

5. EFFECTS OF MAP QUALITY AND DATA CLEANING ON MEAN GRAIN SIZE.

The results in section 4 are all based on the laboratory reported values after their individual choice of data cleaning procedures to produce a clear map of the grain structure. Participants also supplied maps before cleaning and/or files with the raw data so that the effect of the quality of the initial data and the cleaning procedures could be studied.

Figure 12 shows examples of the raw maps from each of the laboratories which used software to which the authors had access. The maps show the lower left quarter of a map and are presented at identical magnifications to illustrate the difference in areas examined. The maps shown in the left hand column are from one particular EBSD system which indexes/assigns an orientation to all pixels but with differing levels of confidence, while the maps in the right hand column are produced by an EBSD system that does not index pixels where the patterns cannot be fitted to possible orientations for the crystal structure(s) within a prescribed level of accuracy. In the latter case these non indexed pixels are shown in black, and the percentage of the indexed pixels are given in Table 3. For the former case, the raw map as displayed by the system is shown on the right and on the left the same map with pixels with a confidence level <0.1 shown in black; the percentage of the pixels left indexed after removal of those with confidence <0.1 are also given in Table 3.

The methods used to index the non-indexed pixels or improve the indexing of the black pixels by the laboratories are listed in Table A2. When these methods were applied by the authors to the raw data supplied with the results it was often the case that, the calculated mean size from the reanalysed maps did not reproduce precisely the values reported, with differences typically of between 0.1 and 0.5 μm . However, what should be clear from Figure 8 is that the precise method of cleaning up or reducing noise in a map has much less influence on the mean grain size, *when reported as a equivalent circle diameter D_{circle}* , than the selection of the cut off value for the minimum grain size. This is because D_{circle} is calculated from areas of grains, so slight variations in the number of pixels in larger grains (i.e. >10 pixels at least), or clusters of small poorly indexed regions resulting from poor clean up which are less than the cut off value, will have little or, in the latter case, no influence on the mean size.

For linear intercept measurements, however, poorly indexed regions may have a much larger influence by for instance breaking up a single large grain into two. As noted in section 4.6 only one laboratory reported a cut off limit for the linear intercept values reported, yet for this method the cut off may be the more important.

Table 3. Examples of the percentage of data points indexed before clean up by various laboratories, calculated from one (of ten) maps. Laboratories are given in the sequence shown in Figure 12 and the figures should be compared with the maps in these figures.

Laboratory	D	L	G	F	J	K	C	B	A
% indexed	98.8	96.6	93	75	97.2	94.5	87.2	81.8	80.9

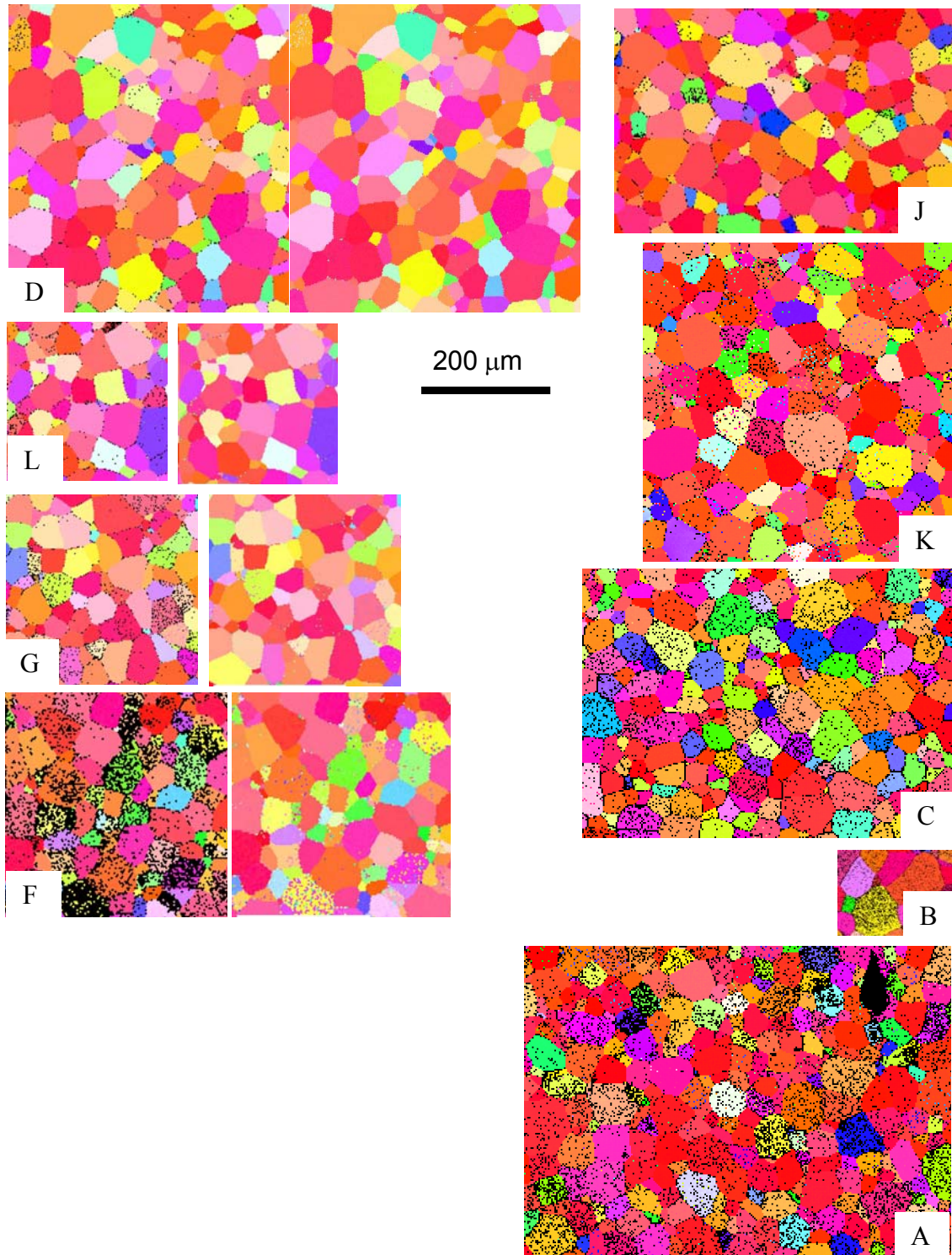


Figure 12. Raw EBSD maps (only one quarter of the area mapped is shown for clarity) before noise reduction, all shown at the same magnification. Maps from Laboratories D, L, G and F shown in the left hand column are shown as indexed on the right and after removal of all pixels with a low confidence index (<0.1) (shown black) on the left. Maps from Laboratories J, K, C, B and A in the right hand column are shown as indexed with pixels in black where pixels could not be indexed with sufficiently high angular accuracy.

6. COMPARISON WITH OPTICAL MEASUREMENTS

Optical images of Sample 7 in the as polished (and examined by EBSD) state were acquired at NPL on a Nikon MM60 optical microscope using a Nomarski filter to produce differential interference contrast (DIC) illumination and give sufficient contrast between grains. Images 595 x 445 μm in size and 1600x1200 pixels were captured and intercepts measured digitally using a Kontron Image Analyser and KS400v.3 software. Figure 13 shows an example. Typically between 9 and 20 intercepts were measured on each digitally drawn line and a total of 431 intercepts were measured from separate images taken from the centre of the sample. The mean grain size from these measurements was 34.6 μm .

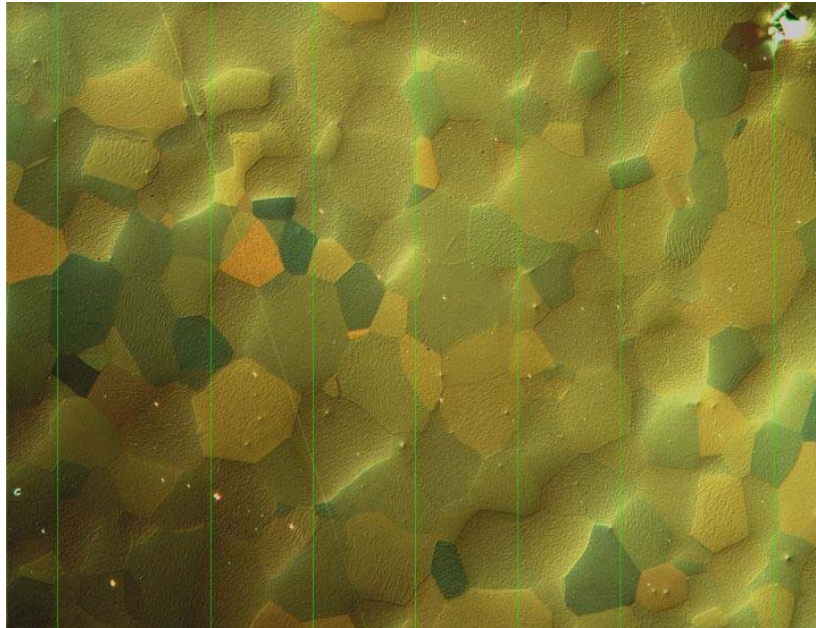


Figure 13. Optical differential image contrast image of Ti sample. Image width 595 μm

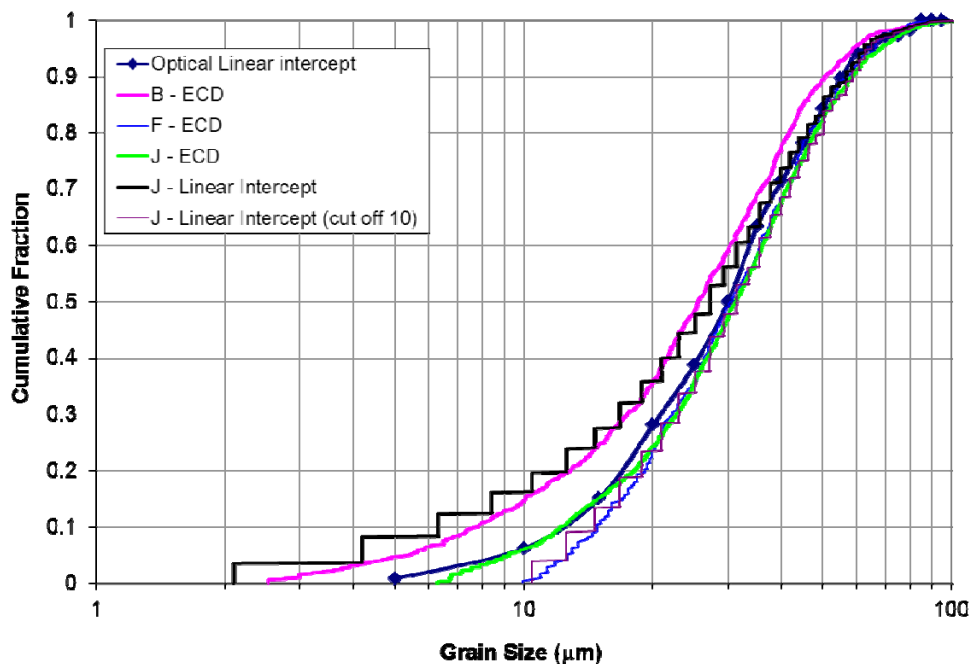


Figure 14. Distribution of optically measured linear intercepts compared with EBSD measurements by linear intercept and equivalent circle diameter.

Figure 14 plots the cumulative size distribution of the optically measured linear intercepts from one map. For comparison it also plots cumulative distributions for a map from the laboratory with results closest to the mean (J) both using equivalent circle diameter and linear intercept grain size measurements. It also plots measured equivalent circle diameter grain size measurements for the laboratories with the extreme step sizes used of 1 μm (Laboratory B) and 3 μm (Laboratory F). The graph clearly demonstrates the effect of cut off diameter on the distributions, with the optical measurement lying between the two EBSD linear intercept measurements produced with cut offs of 2 μm (the step size) and 10 μm .

7. DEFINITION OF MEAN GRAIN SIZE

In section 3.1 the use of an area weighted mean value was discussed. This could have the advantage of reducing the large effect on the mean size of many small grains even though they contribute only to a small fraction of the total area examined. Figure 15 shows area weighted mean sizes (equation (2) in section 3.1) for different cut off values produced using the same original data to that in Figure 8. It is immediately obvious from the mean sizes calculated with low cut off values that the effect of the small grains is reduced significantly and the choice of cut off now has little effect. However, use of the area weighted means has also increased the spread of results. Compared with the spread of about 2 μm (or about 6%) for most of the Laboratories (excluding B and L) in Figure 8 for cut offs greater than 7 μm , the same results give a spread of about 4 μm or 8% using area weighted values.

Further consideration is needed for the best way of describing the mean value. For example the Hall-Petch equation on which many calculations of strength are based uses effectively a linear value rather than an area weighted value. The method of weighting by area in the way defined by equation (2) is also effectively similar to the value used in particle sizing to produce a value that reflects the surface area of a distribution of particles.

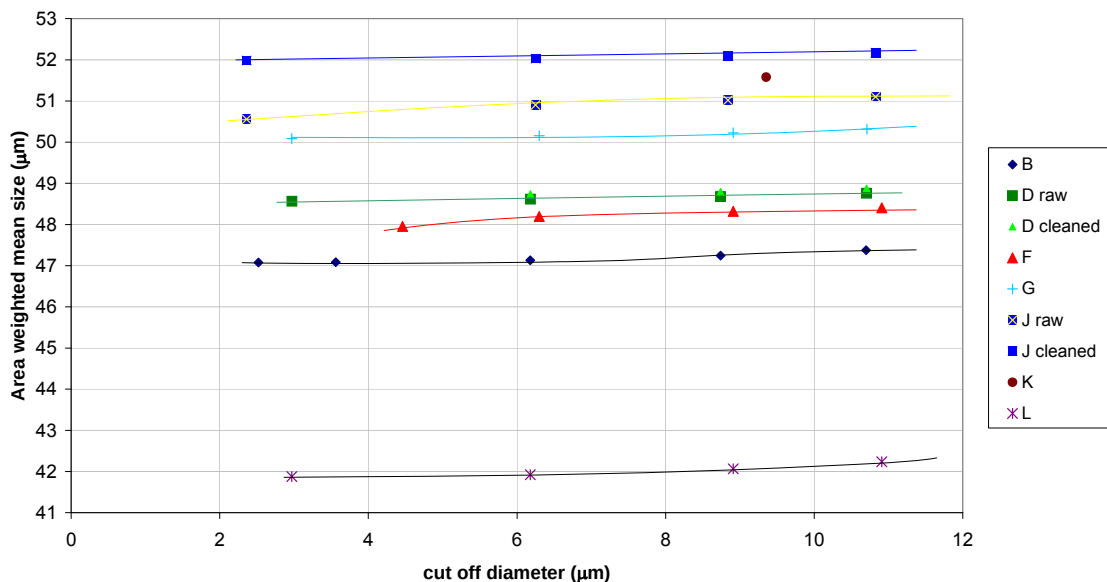


Figure 15. Area weighted mean values plotted against cut off diameter, using the same data as that used to produce Figure 8.

8. SUMMARY

An interlaboratory exercise was carried out across 12 laboratories to validate the proposed International Standard, ISO DIS13067 “Microbeam analysis-Electron Backscatter Diffraction-Measurement of Grain Size and Distribution” prepared by ISO TC202/WG7.

Participants were asked to follow the draft international standard and measure the average grain size of a titanium metal sample with an approximate average grain size of 30 μm .

Of the initial results submitted the equivalent circle diameter values from 4 of the 12 laboratories reported differed significantly from the overall mean value. Causes of these differences were identified in three cases as:

- i) magnification calibration error
 - ii) poor noise reduction/data clean up procedure
 - iii) very small step size and too small an area mapped so larger grains missed
- but the fourth case remained unresolved.

Corrections for the errors for the first two cases were made and a full statistical treatment of the data was then used to remove remaining outliers. After removing outliers the final results were as summarized in Table 1, repeated here as Table 4 for clarity.

Table 4. final results of interlaboratory exercise after removal of outliers and statistical treatment described in Appendix A (Values also repeated in table A10). Repeatability and reproducibility as defined in BS ISO 5727:2 [A2]

		Equivalent circle Diameter D_{circle}	Linear Intercept D_{lin}
Overall Mean (μm)	$\bar{d}$	33.5	30.1
Repeatability standard deviation (μm)	S_r	0.88	0.74
Reproducibility standard deviation (μm)	S_R	1.98	1.15
Repeatability limit (μm)	r	2.46	2.07
Reproducibility limit (μm)	R	5.55	3.22
Number of Laboratories after removal of outliers		12	11

The main source of differences in results would appear to be the choice of smallest grain size to be included in the calculation. Choice of a relatively large cut off size is likely to give the best agreement because the smallest grains can have a significant effect (disproportionate to the area they occupy) and are also the grains most likely to vary in number and size with the step size chosen, the quality of the data and/or the treatment of poorly indexed points.

If the mean grain size is calculated by area weighting, then the step size and cut off have little effect on the final value calculated. However, even after calculating an area weighted mean value a range of values was still obtained for the equivalent circle diameter. Some of this difference may be accounted for by errors perpendicular to the tilt axis (calibration and inaccurate tilt angle measurement).

Linear intercept values were reported by 10 of the 12 laboratories and were on average 10% lower than the equivalent circle diameters, but with a lower variation between laboratories. Linear intercept values were in almost all cases not reported with a cut off minimum grain size (presumably because this had been applied already to production of the map from which equivalent circle diameter

measurements were made) so in general included smaller grain size values than the equivalent circle diameter distribution and hence produced a smaller mean size.

The lower variation in linear intercept values may be because, with one exception, measurements were made only in the horizontal direction so there was no contribution by errors perpendicular to the tilt axis which contribute to measurements of area.

9. RECOMMENDATIONS

The results of the round robin, and comments by participants, have come too late to be incorporated into ISO13067, but it is recommended that any future revision of this standard should incorporate the following points:

1. More emphasis should be put on the acquisition of reporting of grain size distribution information as well as average grain size, since this data is readily available from EBSD and in the long term may lead to improved understanding by industry of sample variability.
2. An appendix to the standard should include the repeatability and reproducibility data determined by this round robin.
3. The comments in ISO13067 on estimation of uncertainty through measurements from multiple areas, currently in Section 5.8 should be re-emphasised in Section 7 on measurement uncertainty.
4. The currently stated target in Section 6.2 of 95% acceptably indexed pixels could be revised. Although the best maps were obtained in the current case with this level of indexing, acceptable results were also obtained with levels of $\approx 80\%$, and greater emphasis should be placed on choice of a cut-off value for small grains.
5. The cut off value chosen is currently recommended as 10 pixels in 6.3.4 of ISO 13067. The round robin results show that, at the very least, it should be reported clearly in micrometres as well as number of pixels. Consideration should also be given to plotting a graph of average grain size against cut off value.
6. Cut offs should also be applied to linear intercept measurements; in the current round robin this was done in only one case. This is a result of the way in which most data cleaning or noise reduction steps automatically apply a cut off to the equivalent circle diameter measurements, whereas the separate linear intercept measurement on the final cleaned maps will not do it automatically.
7. Linear intercept measurements in X and Y directions should be emphasised for use even with equiaxed materials since this can give indications of systematic errors.
8. An increased emphasis should be made on measuring a large enough area (5.8), especially if a small step size is chosen in relation to mean size. The current comment on a minimum of 50 grains could be removed or a new value chosen with a comment on the need to ensure that large grains are not preferentially excluded.
9. Review specific editorial comments sent in by a couple of the participants. These comments will be held by NPL until required by a revision process.

The following points should also be noted:

- Prior to revision, discussion with users of EBSD on the methods of calculation of average grain size could yield new more useful definitions that could be included in future standards.
- While it would be good to include a section on how data cleaning or noise reduction are likely to alter the uncertainty, at present it is unclear how the effects of these operations could be calculated for all common occurrences and further work is needed in this area.
- This study has been carried out on a relatively coarse grained material. Further round robin studies would be useful to determine the applicability of the current findings to other size ranges and characteristics, particularly for materials with mean sizes below $1\text{ }\mu\text{m}$, or for example in two phase materials or materials with less-equiaxed shapes.

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APPENDIX A

DATA RETURNED BY PARTICIPANTS AND STATISTICAL ANALYSIS.

A1. INTRODUCTION

The objective of this preliminary round robin was to test the results obtained using the draft standard ISO DIS13067 [A1]. A review of the original project, at the VAMAS meeting in March 2010, asked that this should be a preliminary exercise involving a few laboratories to test the methodology.

There are two major standards for conducting Interlaboratory Studies [A2, A3]. They differ in detail such as the use of additional tests: the Cochran test for within-laboratory variability, and Grubb's test for between-laboratory variability are used in ISO 5725-2 but are not employed in the ASTM E691. More detailed comparisons between the methodologies are given in reference A4.

A2. CHOICE OF PARTICIPANTS

The participants were chosen from the following three categories:

- Main equipment manufacturers (3)
- National Metrology Laboratories or equivalent which perform this test. (7)
- Selected industrial laboratories with a track record in making these measurements. (2)

Two laboratories (G and K) had two systems and agreed to perform two sets of tests. These were considered to be independent results. Many of the organisations were involved in writing the standard. These laboratories were considered to have:

- Proper laboratory facilities and testing equipment.
- Competent operators
- Familiarity with the test method
- A reputation for reliable testing work
- Sufficient time and interest to do a good job

The laboratories were invited to take part. They provided confirmation and a contact person. In view of the experience of the laboratories a training programme was not thought necessary.

Each participating laboratory was assigned a letter between A and M in the order in which the results were returned and anonymity has been strictly maintained by using only the letter in any dissemination of results to the participants or in this report.

A3. TEST MATERIALS

As this was a preliminary exercise only one type of material was chosen and the average grain size was chosen to be accessible by other methods such as optical microscopy.

The number of replicates chosen was 2 although the exercise could have been improved by the use of 3 or 4. The decision was taken that the material was expected to be reasonably homogeneous and that the reproducibility standard deviation would be significantly higher than the repeatability standard deviation; this was subsequently proved to be the case.

As described in section 2.1, each sample was then imaged in a Zeiss Supra40 FEG-SEM and a minimum of one region mapped by EBSD. Figure A1 in Appendix A shows the results of these measurements and identifies the samples chosen within a total range of 3 μm , although it should be noted that a better indication of the homogeneity of the samples was subsequently obtained from the larger amounts of data produced by all the participants.

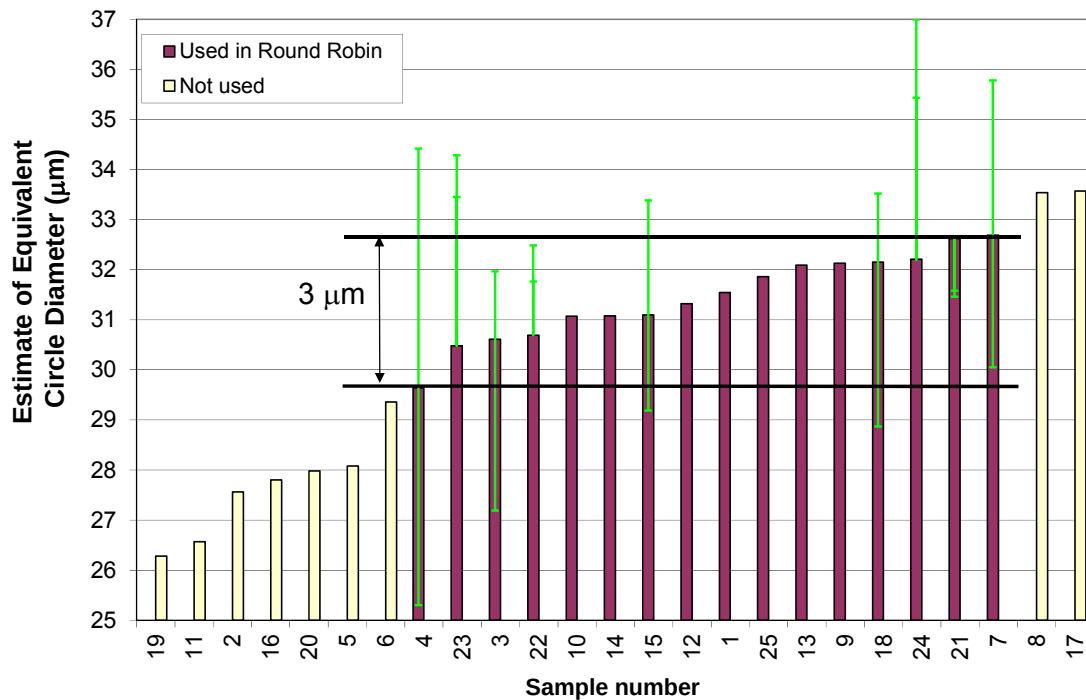


Figure A1. Equivalent circle diameter measurements made on each sample prior to dispatch of samples for the round robin. 'Error' bars show range of measurements made by other laboratories as part of the round robin where one sample was measured by more than one laboratory, so the mean may not fall either on the bar value above or midway between max. and min. values

A4. METHOD AND RETURN OF RESULTS

The principal points of the instructions given were to follow ISO DIS13067 and to map 5 areas of the sample from close to the central indent. (Appendix B shows the full instruction and return sheet). Some samples were returned from one participant and sent on to a second participant to provide further information on the interlaboratory variation in comparison with sample to sample variation. Details are given after the main results in sections 5 and 6.

Information was requested on the SEM operating conditions and on the EBSD mapping parameters including the step size used and the method used to clean the data. Appendix B shows an example of the information requested on the return sheet and Tables A1 to A3 summarise this information.

Table A1 Summary of instrumental conditions used by participants in this exercise

	A	B	C	D	E	F	G	G	H	H	J	K	K	L
Accelerating voltage (kV)		18	20	20	20	20	20	30	25	25	20	30	30	25
Working distance, (mm)		18	16 or 18	15	18.2	14	19	23 - 25	7	7	16	25-28	≈18	15
Step size (μm)	2.5	1	2.5	2	2	3	2	2	1.51	3.01	2	2.5	2.5	2
pixel area (μm ²)	6.25	1	6.25	3.46	4	7.79	4	3.46	2.27	9	4	6.25	6.25	3.46
Map size (number of steps in X and Y)	535x401	350 x 260	468x351	453 x 469	800x600		300 x 300	600 x 600	256	256	512x352	400x400	400 x 400	250
Magnification		1000	100		100	400	150	120	1000	500	120		90	
Image size (X by Y mm)	1338x1003	350 x 260	1170x875	906x 939	1600 x 1200	690 x 690	625 x 625	625 x 625	359 x 350	771x699		1500 x 1150	1400x 1050	500x500
Grid shape (square/hexagonal)	S	S	S	H	S	H	S	H	S	S	S	S	S	H
Notes		8 maps					FEG	W				LaB6	FEG	

Table A2 Summary of criteria used in the analysis of results

	A	B	C	D	E	F	G	G	H	H	J	K	K	L
Threshold for grain size (no. pixels or μm)	9 pixels #	> 4 pixels	?	10 pixels, CI>0.3	10 pixels	10	5 pixels	5 pixels	10 pixels	10 pixels	6 μm	10 pixels	10 pixels	2 μm
min grain size area (μm ²)	56.25	5	?	34.64	40	77.94	20	17.32	22.7	90.7	28.4	62.5	62.5	3.1
D _{circle} Min Grain size (from above) (μm)	8.5	2.5	?	6.6	7.1	10								2
calculated D _{circle} (μm)	8.46	2.52	?	6.64	7.14	9.96	5.05	4.70	5.38	10.75	6	8.92	8.92	2
Misorientation angle defining boundaries (°)	10 or 15?	3	?	5	5	5	5	5	5	5	10	5	5	10
Typical no. of grains/map	1100 *	70	1100			425						531-740		270

- (>8) / > 8.5mm (2 pixels/ 5 mm for lin int)

* - (1400 vert+horiz lin int).

Table A3 Data Cleaning operations used by participants

	Data Cleaning operations
A	Not known
B	Noise reduction, extrapolation, noise reduced till 0% zero solution, Kuwahara 3x3 + 3°, critical misorientation 3°, CEDs in subsets except grains <5µm ²
C	Noise reduction, 5 neighbours (or 4 for revised results). No border grains
D	Confidence Index Standardization (changes CI value but no measured orientations), Neighbour Orientation Correlation Level 2, Grain Dilation, 5° grain tolerance with 10 pixels min per grain, single iteration
E	Only isolated pixels (zero solutions or missindexed) and pixels with 7 or 6 neighbours sharing the same orientation have been cleaned.
F	CI std (5deg, min size 2), grain dilation 5 deg, 10 pixels, no iteration
G	1 st cleanup: Dilation (multiple iterations) 2nd cleanup - Grain CI Standardization
G	1 st cleanup: Dilation (multiple iterations) 2nd cleanup - Grain CI Standardization
H	Considered pixels with pattern quality number greater than the minimum pattern quality number. Fill with nearest neighbour if isolated by more than 5° or has no solution. Grains touching the edges of the image were removed
H	Considered pixels with pattern quality number greater than the minimum pattern quality number. Fill with nearest neighbour if isolated by more than 5° or has no solution. Grains touching the edges of the image were removed
J	Wild Spike extrapolate, plus extrapolation to 100% completion
K	Grains ≤ 4 pixels set to zero solution. Fill zero solutions
K	Grains ≤ 10 pixels set to zero solution. Fill zero solutions
L	Grain Dilation (5° Tolerance, 5 Pixel size, single pass)

A5. RESULTS

A5.1 INITIAL RESULTS

The results for equivalent circle diameter (D_{circle}) and linear intercept (D_{lin}) from the completed proformas were transferred to a spreadsheet. Two laboratories did not measure the linear intercept. (Labs E and L). The full set of results are shown in tables A4 and A5.

From inspection of the D_{circle} results, 4 laboratories were found to have results deviating significantly from the mean. Laboratories B, C and L had significantly smaller than average values and Laboratory H showed a higher result. From the return sheet proformas it was established that:

- Laboratory B had used smaller step length than other laboratories. (1 μm against normally 2-3 μm) This would have the effect of resolving more small grains and also the area chosen was such that some of the larger grains would not be included because they intersected the edge of the image.
- Laboratory C performed no cleaning operation, which gives a number of small spurious grains reducing the average.
- Laboratory L used a very small cut off so the greater number of small grains may have reduced the average grain size.
- Discussions with Laboratory H revealed a problem of magnification calibration and they subsequently resubmitted their results.

For D_{lin} results, laboratory C had a significantly lower result than the grand mean and H a significantly higher result. Similar explanations were ascribed to the results as for D_{circle} measurements.

A5.2 CORRECTED RESULTS

The following corrections were made:

- For laboratory B the results were corrected for step length by removing grains below a certain size (see Figure 7). This still gave a smaller average grain size and this was ascribed to exclusion of larger grains from the calculation because many of these touched the map edge.
- The results for laboratory C were cleaned and resubmitted.
- The application of a correction for small grains for laboratory L still left a significantly lower than average grain size. There are believed to be orthogonality problems with the scanning leading to problems in calibration. The results were not adjusted.
- The new results from laboratory H were used.

The corrected results from labs C and H are shown in Table A6 for D_{circle} and for laboratories H in Table A7 for D_{lin} .

Table A4 Results for D_{circle} for preliminary VAMAS Intercomparison Late 2010/early 2011. Based upon 2 results from each laboratory. All units in μm

Laboratory i	Samples	Laboratory i result 1	Laboratory i result 2	Mean, $\bar{d}_i$	Std. Deviation s_i	h statistic	k statistic
A	1,9	35.86	35.48	35.67	0.27	0.64	0.35
B	15,18	29.19	28.86	29.03	0.23	-1.07	0.31
C	3,7	28.64	28.82	28.73	0.13	-1.14	0.17
D	12,25	33.66	34.76	34.21	0.78	0.26	1.02
E	4,14	33.38	34.42	33.90	0.74	0.18	0.97
F	1,9	34.42	34.87	34.65	0.32	0.37	0.42
G1	21,22.	31.46	31.8	31.63	0.24	-0.40	0.32
G2	21,22	31.6	32.5	32.05	0.64	-0.29	0.84
H	3,7	41.23	43.65	42.44	1.71	2.37	2.25
J	15,18	33.38	33.52	33.45	0.10	0.07	0.13
K1	23,24	35.27	35.18	35.23	0.06	0.52	0.08
K2	23,24	33.7	36.01	34.86	1.63	0.43	2.15
L	4,14	25.3	26.3	25.80	0.71	-1.89	0.93
M	1,7	33.26	32.71	32.99	0.39	-0.05	0.51

Table A5 Results for D_{lin} for preliminary VAMAS Intercomparison Late 2010/early 2011. Based upon 2 results from each laboratory. All units in μm

Laboratory i	Samples	Laboratory i result 1	Laboratory i result 2	Mean, $\bar{d}_i$	Std. Deviation s_i	h statistic	k statistic
A	1,9	31.30	30.94	31.12	0.25	0.42	0.31
B	15,18	27.73	28.86	28.30	0.80	-0.56	0.98
C	3,7	20.58	22.92	21.75	1.65	-2.82	2.04
D	12,25	30.82	30.56	30.69	0.18	0.27	0.23
E	4,14						
F	1,9	30.13	29.88	30.01	0.18	0.03	0.22
G1	21,22.	29.90	30.9	30.40	0.71	0.17	0.87
G2	21,22	30.30	31.2	30.75	0.64	0.29	0.78
H	3,7	33.35	33.55	34.05	0.14	1.43	0.17
J	15,18	30.50	29.1	29.80	0.99	-0.04	1.22
K1	23,24	31.30	31.4	31.35	0.07	0.50	0.09
K2	23,24	29.80	31.9	30.85	1.48	0.32	1.83
L	4,14						
M	1,7	29.46	30.24	29.85	0.55	-0.02	0.68

Table A6 Results for D_{circle} for preliminary VAMAS Intercomparison Late 2010/early 2011. Based upon 2 results from each laboratory. . All units in μm

Laboratory i	Samples	Laboratory i result 1	Laboratory i result 2	Mean $\bar{d}_i$	Std. Deviation s_i	h statistic	k statistic
A	1,9	35.86	35.48	35.67	0.27	0.99	0.24
B	15,18	29.19	28.86	29.03	0.23	-1.47	0.21
C	3,7	31.97	35.78	33.88	2.69	0.32	2.45
D	12,25	33.66	34.76	34.21	0.78	0.45	0.71
E	4,14	33.38	34.42	33.90	0.74	0.33	0.67
F	1,9	34.42	34.87	34.65	0.32	0.61	0.29
G1	21,22.	31.46	31.8	31.63	0.24	-0.51	0.22
G2	21,22	31.6	32.5	32.05	0.64	-0.35	0.58
H	3,7	33.25	36.27	34.76	2.14	0.65	1.94
J	15,18	33.38	33.52	33.45	0.10	0.16	0.09
K1	23,24	35.27	35.18	35.23	0.06	0.82	0.06
K2	23,24	33.7	36.01	34.86	1.63	0.68	1.48
L	4,14	25.3	26.3	25.80	0.71	-2.67	0.64
M	1,7	33.26	32.71	32.99	0.39	-0.01	0.35

Table A7 Results for D_{lin} for preliminary VAMAS Intercomparison Late 2010/early 2011. Based upon 2 results from each laboratory. All units in μm

Laboratory i	Samples	Laboratory i result 1	Laboratory i result 2	Mean $\bar{d}_i$	Std. Deviation s_i	h statistic	k statistic
A	1,9	31.3	30.9	31.12	0.25	0.65	0.30
B	15,18	27.7	28.9	28.30	0.80	-0.44	0.94
C	3,7	20.6	22.9	21.75	1.65	-2.94	1.94
D	12,25	30.8	30.6	30.69	0.18	0.48	0.22
E	4,14						
F	1,9	30.1	29.9	30.01	0.18	0.22	0.21
G1	21,22.	29.9	30.9	30.40	0.71	0.37	0.83
G2	21,22	30.3	31.2	30.75	0.64	0.50	0.75
H	3,7	27.7	29.0	28.35	0.92	-0.42	1.08
J	15,18	30.5	29.1	29.80	0.99	0.14	1.16
K1	23,24	31.3	31.4	31.35	0.07	0.73	0.08
K2	23,24	29.8	31.9	30.85	1.48	0.54	1.74
L	4,14						
M	1,7	29.46	30.24	29.85	0.55	0.16	0.64

A6. ANALYSIS OF RESULTS.

Using the data in Tables A6 and A7, the following parameters were calculated for D_{circle} and D_{lin} .

Table A8

	Equivalent Circle Diameter (μm)	Linear Intercept (μm)
Overall Mean,	33.0	29.4
Standard deviation of cell averages	2.70	2.61
Repeatability standard deviation, S_r	1.10	0.85
Reproducibility standard deviation, S_R	2.81	2.68

Critical values of h and k statistics were calculated at 1% and 5% significance levels for the number of laboratories, $P = 14$ for D_{circle} and $P = 12$ for D_{lin} and $n=2$ (number of results per laboratory), giving the following values

Table A9

	Equivalent Circle Diameter Significance Levels		Linear Intercept Significance Level	
	1% (Outliers)	5% (stragglers)	1% (Outliers)	5% (stragglers)
critical h statistic	2.30	1.85	2.25	1.83
critical k statistic	2.40	1.92	2.37	1.92

Laboratories with h or k statistics greater than the 1% level were omitted from the calculations after which recalculation gave the following values

Table A10

	Equivalent Circle Diameter (μm)	Linear Intercept (μm)
Overall Mean,	33.5	30.1
Standard deviation of cell averages	1.88	1.02
Repeatability standard deviation, S_r	0.88	0.74
Reproducibility standard deviation, S_R	1.98	1.15
Repeatability limit r	2.46	2.07
Reproducibility limit R	5.55	3.22

A7 SUMMARY OF THE STATISTICAL ANALYSIS

Discussions were held with laboratories where outlying results were obtained and re-analysis of the data was possible which could lead to values closer to the average. Some of the outlying results could have been avoided by improvements to the standard and the main body of this report contains a section of recommendations for such improvements. These recommendations incorporate comments received from a couple of the participants but are open for further comment.

The precision established by these experiments justify the establishment of repeatability and reproducibility values for relatively large grain size materials which can be incorporated into

any future revisions of ISO13067. Further studies with smaller grain sizes would be advantageous.

REFERENCES

- [A1] " Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size", ISO DIS 13067
- [A2] "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method" ASTM International Standard E691-09
- [A3] "Accuracy (Trueness and Precision) of measurement methods and results-Part 2: Basic method for the determination of repeatability and reproducibility of the standard measurement method." BS ISO 5725-2:1994
- [A4] Luping, T and Scouenborg, B "Methodology of intercomparison Tests and Statistical Analysis of Test Results-Nordtest project No. 1483-99" SP Report 2000:35 SP Sveriges Provnings-och Forskninginstitut, Borås, Sweden

APPENDIX B
INSTRUCTIONS AND RETURN SHEET FOR RESULTS

Pilot Round Robin Exercise on the Measurement of Grain Size
Using Electron Back Scattered Diffraction
Organised by VAMAS TWA 37.

Measurement of Grain Size in Commercial Purity Titanium

Details of Organisation.

Principal Investigator	
Affiliation of Participant	
Address	
Post code	
Telephone	
Fax	
E mail	
Comments	

Details of Equipment

SEM	
Manufacturer	
Model	
Gun Type	Field emission/LaB6/W
Comments	

EBSD Hardware/software	
Manufacturer	
Model	
Comments	

Details of Experiments.

The specimens have been cut from a single bar of Commercial Purity Titanium. All of the specimens have been polished at NPL and preliminary EBSD maps acquired prior to distribution. There are small differences between specimens so each participant has been sent two specimens so that sensitivity to specimen variability can be taken into account when analysing the total data set.

To facilitate identification of the areas examined, each specimen has been marked with small pyramidal hardness indents, one at a point close to the centre and one at the edge, the latter also identified with red ink. Please measure the grain size from areas close to the central indent (within 5 mm).

We are suggesting that 5 areas on each specimen are mapped, but if this causes too many problems in terms of time required, please let us know and map what you can. In all other respects we are asking that you follow ISO DIS 13067 “Microbeam analysis-Electron Backscatter Diffraction-Measurement of average grain size”: please report any major difficulties in following this document so that it can be improved in future revisions.

Experimental Details

SEM	
Accelerating voltage (kV)	
Probe current (nA)	
Working distance, (mm)	
Specimen tilt angle (°)	
Step size (µm)	
Map size (number of steps in X and Y)	
Magnification and image size (X by Y µm)	
EBSD	
Grid shape (square/hexagonal)	
Phase / crystallographic structures used for EBSD indexing	
Data Display	
Data Cleaning operations	
Threshold for grain size (number of pixels or µm)	
Misorientation angle defining boundaries (°)	
Other issues (e.g. twin definition)	
Sample condition as received or re-prepared. (Please give details of conditions if sample reprepared.)	

Results

Please measure five different areas (see previous page), within the central 5 mm of the specimens. Please state if you used the data calculated directly by the EBSD software (using the data cleaning operations described on the previous page), or if you subjected the data to further analysis.

Sample 1.

Specimen Number	
-----------------	--

Area	1	2	3	4	5	Std. Deviation of the mean
Equivalent circle diameter mean grain size (μm)						
Mean linear intercept (μm)						
Comments / notes on data analysis methods						

Sample 2.

Specimen Number	
-----------------	--

Area	1	2	3	4	5	Std. Deviation of the mean
Equivalent circle diameter mean grain size (μm)						
Mean linear intercept (μm)						
Comments / notes on data analysis methods						

If you wish to measure more areas please list results together with any changes in operating conditions or operators.

Comments on ISO DIS 13067

Any Deviations from ISO DIS 13067	
Any suggestions to improve ISO DIS 13067	

Please return on a CD (or by some other electronic means) together with the supplied sample:

- 1) Maps of the raw data showing the grain structure and indexing.
- 2) Maps after data cleaning.
- 3) The file listing individual grain sizes.
- 4) Cumulative distribution of grain sizes from 3.
- 5) Values of the measured mean grain sizes and their standard deviation.
- 6) The raw EBSD data files in *.cpr + *.crc or *.osc or *.ang formats, if they are available.

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