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RECOMMENDATIONS FOR INTERNATIONAL STANDARD DEVELOPMENT FOR GRAIN SIZE MEASUREMENT BY EBSD OF ADDITIVELY MANUFACTURED MATERIALS

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Recommendations for international standard development for grain size measurement by EBSD of additively manufactured materials

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ABSTRACT

This report extends analysis of an interlaboratory comparison of Electron backscatter diffraction (EBSD) analysis of average grain size and size distribution measurement of an additively manufactured Ni alloy. Based on the results, proposals are made for ways in which a new standard for grain size measurement of additively manufactured materials would differ from the existing ISO13067 grain size standard. In particular, the significant effects of choice of minimum grains size, edge grain inclusion and extrapolation of grain boundaries that are incomplete at the chosen threshold angle have been investigated and recommendations made for their inclusion in the standard. The importance of reporting area weighted averages has been illustrated and should form part of any new standard.

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Approved on behalf of NPLML by
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1 INTRODUCTION

Additively manufactured materials have heterogeneous microstructures with broad grain size distributions and highly anisotropic and/or non-convex grain shapes. Standard good practice for measuring and reporting grain size, including electron backscatter diffraction (EBSD)-based methods, have been optimised for equiaxed grains with a unimodal grain size distributions. Currently there is no EBSD grain size measurement standard suitable for typical additively manufactured material microstructures.

The above introductory paragraph is taken from NPL Report MAT130, which described the initial results of an interlaboratory comparison study aimed to find out what grain size metrics and grain size distribution summary statistics are used to describe an additively manufactured nickel alloy microstructure measured using EBSD. This new report follows on from that report with further analysis of the data, and provides more detailed examples which illustrate the issues highlighted by the data. To this analysis is added further examples of AM materials which present challenges to simple mean grain size analysis which are not adequately covered by the existing ISO 13067:2020 "Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size". The report concludes with an outline of the issues that will need to be covered in a new International Standard or Technical Specification and how the treatment of the issues will need to be different from the existing ISO13067 :2020.

2 ILC RESULTS SUMMARY

Table 1 summarises results from 5 participants from Report MAT130 with some additional columns of newly calculated values. It is again worth noting that the number of grains counted varied by 100%. The arithmetic mean values also vary by ≈100%, while the area weighted means vary only by ≈30%.

For Participant 2, the column P2 shows the original reported results and the three further columns have been added to show subsequent recalculations from the identical data using different grain boundary closure angles (P2(2) and (3)) and minimum grain size P2(4). These recalculations have been used to produce a clearer understanding of how these approaches change the appearance of the microstructure revealed in EBSD maps of various grain size parameters, and thus a qualitative understanding of how different mean values are arrived at.

Although the previous report noted that equivalent circle diameter is not the most appropriate parameter to measure for these elongated grain structures, it was the only parameter reported by all the participants, so it is difficult to avoid making observations without reference to it. The main conclusions were:

- Area weighted mean values are more consistent than the number weighted mean values.
- The degree of cleaning could have a significant effect on results by reducing the number of small grains analysed. However the effect was difficult to quantify because the areas analysed were not clearly reported and it was also impossible to separate the effect of cleaning from other changes in analysis methods.
- A 10° grain boundary threshold was used in all but one case.
- Incomplete boundary segments were treated in very different ways by the participants but no conclusions could be drawn because of its convolution with other changes in analysis.

Table 1 Summary Statistics

	Size distribution summary metric	**P1**	**P2**	**P2(2)**	**P2(3)**	**P2(4)**	P3[1]	**P4_1**	**P4_2**	**P4_3**	**P5**	**Variation**
Software/Noise Reduction level (see footnotes)		**A/3**	**A/4**	**A/1**	**A/2**	**A/1**	**M/3**	**A/1**	**A/2**	**A/2***	**O/2**	**Range as % of average)**
Grain size metric	**Number of grains**	**6439**	**7649**	**12432**	**11282**	**5716**	**7752**	**12778**	**6917**	**9401**	**9040**	
mean D(A)			32.5	33.4	36.6		-					
Equivalent circle diameter / µm	**Number weighted arithmetic mean**	**38.2**	**22.5**	**21.8**	**24.1**	**38.0**	17	**16.2**	**25.6**	**22.32**	**22.12**	**94**
	Area weighted arithmetic mean	**97.1**	**82**	**98.9**	**104.1**	**102.1**	111	**96.2**	**107.3**	**88.3**		**30**
	Minimum		4.5	4.5	4.5			4.5	4.5	4.5		
	Maximum		243	469	428			444.3	460.6	352.6		**57**
	[Size distribution width] Std deviation		23.5	25.3	27.6			21.8	28.7	24.2		**27**
Area / μm^2	Number weighted arithmetic mean		831	876	1051.9						861.72	24
Max Feret diameter / µm	Number weighted arithmetic mean	88	49.8	49.6	53.6	89.4	37				48.77	94
	Area weighted arithmetic mean	233.9	194.2	250.3	257.1	258.6	281					36
	Area weighted median		173.5				175±25					
Min Feret diameter / µm	Number weighted arithmetic mean						13				18.08	33
	Area weighted median						53 ± 8					
Ellipse major axis diameter / µm	Number weighted arithmetic mean	73.9	42.3	41.3	45.5	72.8					24.39	109
	Area weighted arithmetic mean	188.5	153.2	195.8	204.3	202.3						28
Ellipse minor axis diam / µm	Number weighted arithmetic mean		12.9	12.4	13.8						6	69
Aspect ratio	Unknown, > 1 convention;		3.6	3.7	3.5			3.52	3.53	3.6	(3.1)	6
	weighted >1		3.84									
	Arithmetic mean, < 1 convention										0.32	
Grain ellipticity											0.94	
Grain circularity											0.33	

Footnotes on software and noise reduction types in row 2: **A** – Aztec, **M** – MTEx, **O** – OIM. **1** - Raw data, **2** - noise reduction, no boundary closure (**2*** - 5° boundaries), **3** - Noise reduction, boundary close to 5°, **4** - Noise reduction, boundary close to 0°. All boundaries 10° except **2*** . Minimum grain size = 10 pixels (P2, P4), 1 pixel P3, 100 pixels (P1, P2(4)). P2 cropped, P2(2-4) uncropped

However, the additional data from reanalysis of the P2 participants data has enabled some further comparisons to be made and conclusions drawn from both the maximum feret diameter and the circle equivalent diameter. It should also be noted that, given the spread of orientations within most grains within the microstructure, it would be difficult to justify using a low 5° threshold of misorientation, so the effect of this threshold has not been examined further in this report.

Figure 1 provides an additional way of viewing the effect of area weighting on the distribution of sizes, and how this can show up differences between the equivalent circle diameter size and the Maximum Feret Diameter. The number weighted plots of the two size measurements appear to run approximately parallel with each other up to ≈ 100 µm, well above the mean values, before diverging slightly. In contrast, the area weighted plots show a greater divergence possibly from half the size of the number plots and from a long way short of the mean values. This latter description matches more closely to the visual observation from the maps that the majority of the structure is composed of large elongated grains, with the larger grains having a higher aspect ratio than the smaller ones.

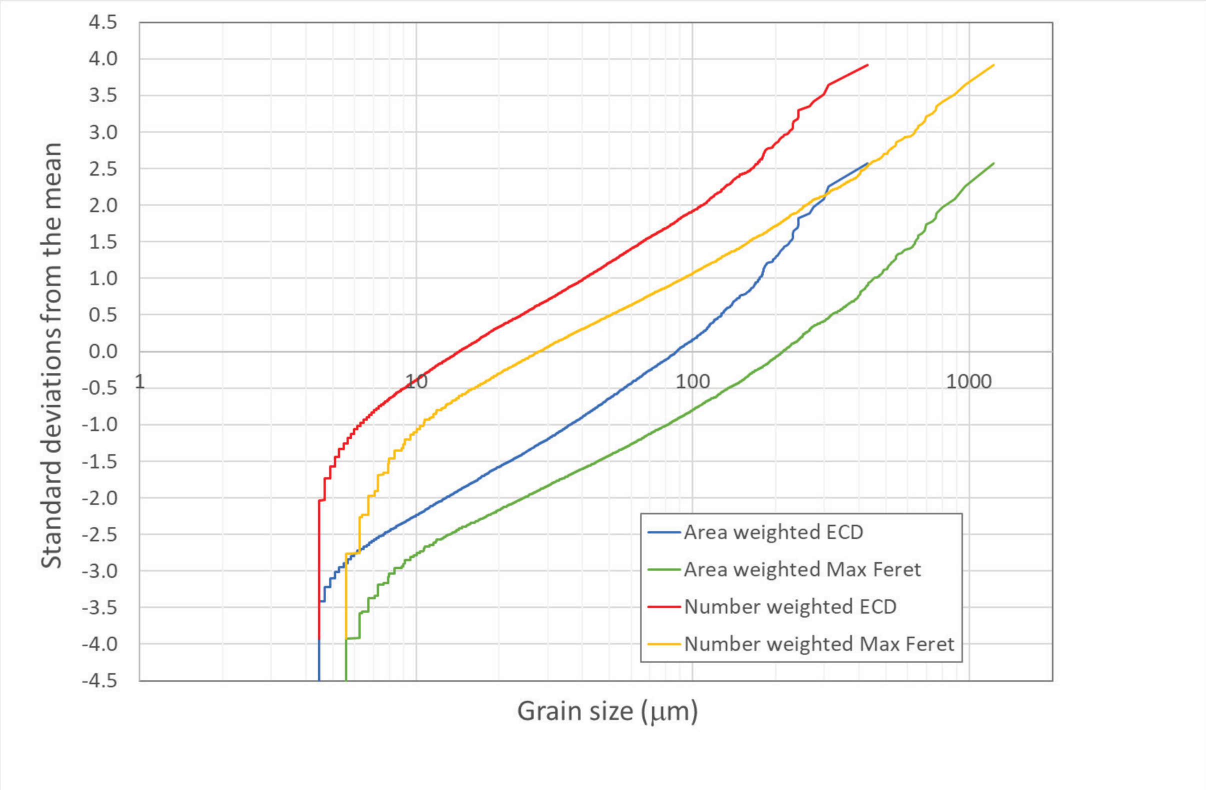

Figure 1.

3 ADDITIONAL ANALYSIS

3.1 MINIMUM GRAIN SIZE

It was concluded in the previous report that more aggressive data cleaning had a large effect on number weighted mean grain size, based partly on the large number mean ECD of P1. However, Table 1 shows in column P2(4), values from the same data as used for P2(2) but with a 100 pixel minimum size like P1, instead of 10 pixels like P2(2) . The similarity between P1 and P2(4) for both ECD and MFD clearly shows that the biggest effect on the number weighted mean arises from changing the minimum grain size from 10 to 100 and not data cleaning.

The differences between P2(2) and P2(4) show the effect of ignoring all sub-100 pixel grains increases the number weighted mean by a similar amount (75 -80%) for both ECD and MFD. However, for the area weighed mean the change is only 3% for both parameters.

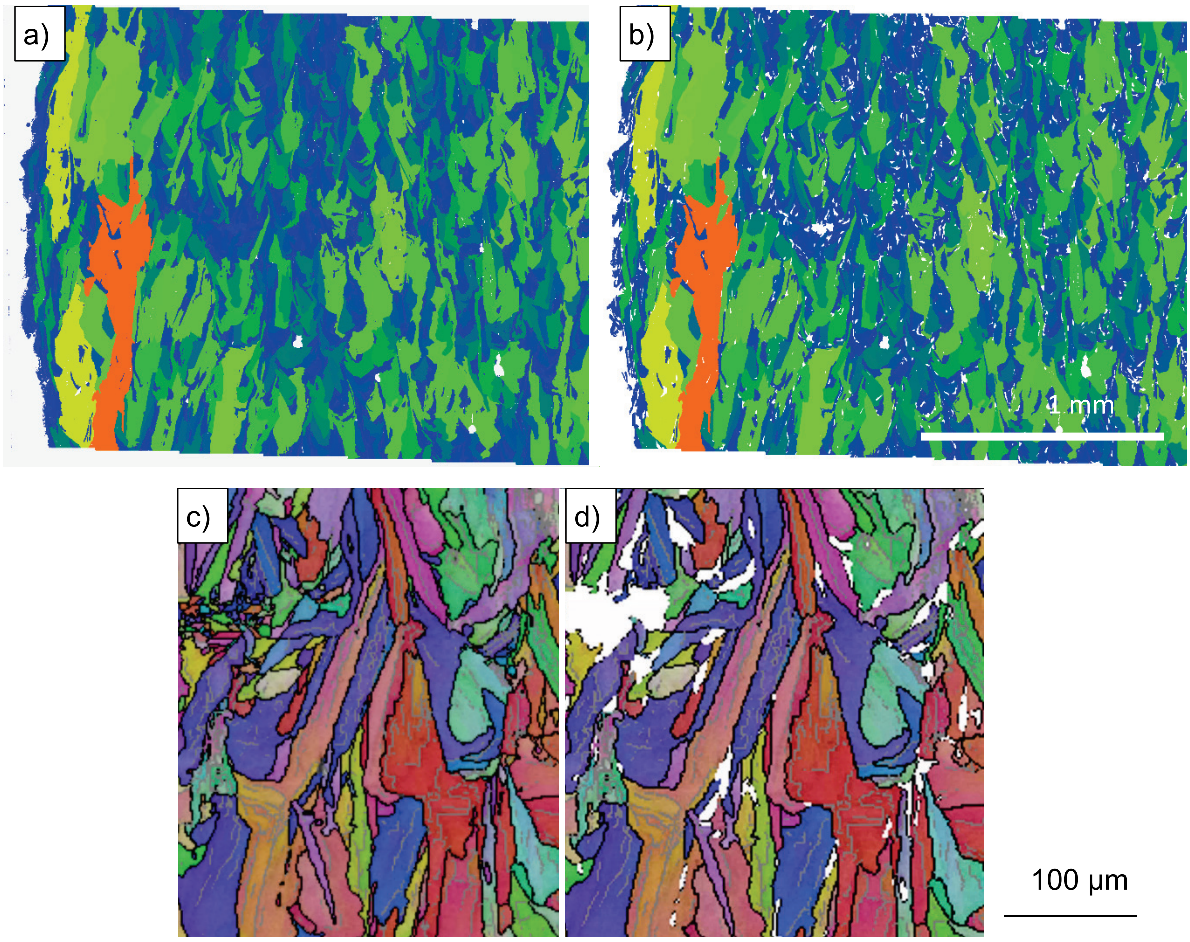

Figure 2. Visualisation of the exclusion of grains < 100 pixels in size using examples from map used in ILC. a) Low magnification map of CED grainsize showing all grains >10 pixels, and b) the same region with all <100 pixel grains removed (left showing as white). c, d) enlargement of central region of a) and b) with locations of <100 pixel grains in c) shown in white in d)

Comparing both the >100 pixel results (i.e. P2(4) with P1) shows the number weighted mean values are very close but the area weighted MFD is 10% larger for P2(4). The main differences between the two data sets are the degree of cleaning and the area analysed: it is difficult to see why less cleaning should produce larger grains, so the probable explanation is that it is the exclusion of the very largest grains at the left hand edge of the full area in P1 that resulted in a smaller area weighted value for this sample.

Figure 2 shows an example at low and high magnification of the exclusion of 100 pixel grains. These suggest it is difficult to justify their exclusion, particularly if they occur in clusters.

3.2 GRAIN BOUNDARY EXTRAPOLATION

It was noted in the previous report that it was difficult to compare number of grains counted because the areas analyses were not clearly documented. The data in Table 1 is also potentially confusing because the originally reported results for P2 show the results after cropping the area measured, which reduces the number of grains substantially, whereas extrapolation of grain boundaries will increase the number of grains breaking up larger grains into smaller ones.

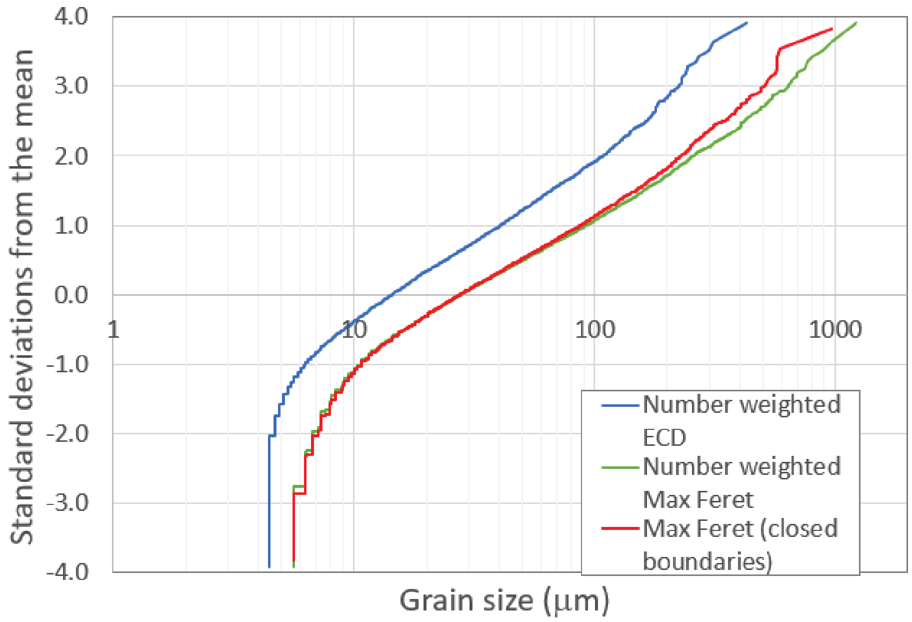

Figure 3. Number weighted size distribution plots of grain size (ECD and MFD) with no grain boundary extrapolation (data as Figure1) and with grain boundary extrapolation to 0°.

Figure 3 above demonstrates, from the deviation of the two max.feret diameter plots from each other, that the effect of grain boundary extrapolation is to break up large grains above about 100 µm in length, with minimal effect below this.

Figure 4 illustrates possible effects of both data cleaning (indexing un-indexed pixels) and extrapolation of grain boundaries. Figures 4a and b show just the effect of data cleaning from approximately 92% to 99% (for the full map the successful indexing increased from 90 to 98%). Most non indexed pixels appear to be on grain or sub-grain boundaries, so the effect of indexing them is not just to increase the area of very small grains, but to link large areas of similar orientation and produce much larger grains, as can be seen by comparing the grain outlined in Figure 4c (raw map) with that after reindexing in Figure 4d.

The break-up effect of extending boundaries to 0° is then clearly seen in Figure 4e, producing several grains smaller than in the raw data map. Comparing equal mapped areas before and after grain boundary extrapolation shows that this resulted in an increase in number of grains measured from 11282 ((P2(3)) to 13272, or about 17%.

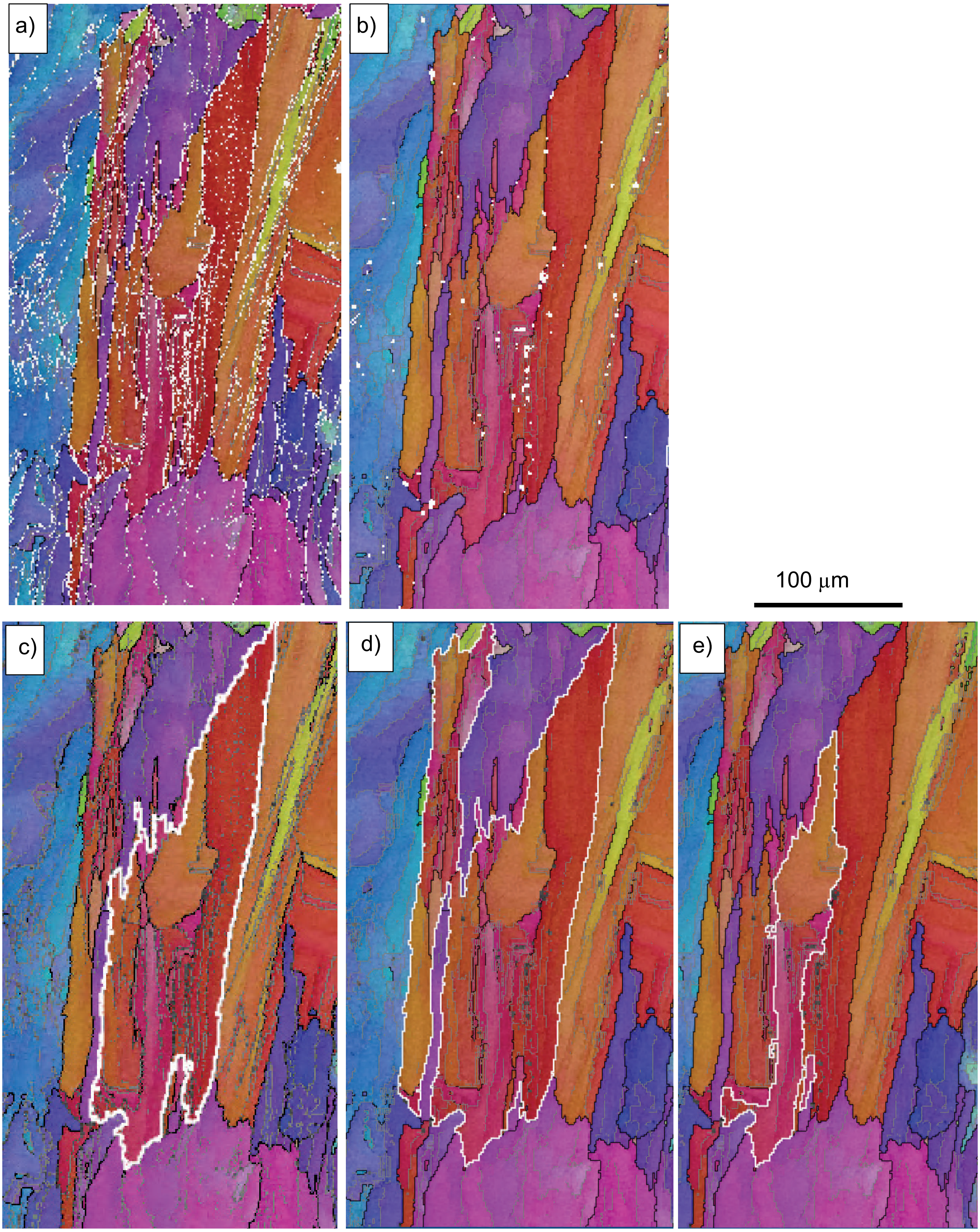

Figure 4. Example of grain and grain boundary extrapolation on grain identification. a) raw map, b) after cleaning (as P2) with 10° grain boundaries shown. c) raw map a) with a single grain identified (non indexed pixels now shown with band contrast rather than white). d) cleaned map b) showing how re-indexing can increase grain size. e) after extrapolating incomplete grain boundaries to 0°, resulting in much smaller grains.

Figure 5 illustrates the effects of data cleaning and grain boundary extrapolation over the whole map and how this can affect the visualisation of grain size. For both measures of size (CED and MFD), the main effect on visualisation is only to affect the very largest grains on the left hand outer sample edge using a single scale for all three maps in Figure 5. These maps show, just for CED, the change from raw data (top), through noise reduction (middle) to grain boundary extrapolation (bottom) with the only noticeable effect being on the very largest grain on the far left. The MFD maps are not shown because, despite having values significantly greater than for the CED, when plotted with the same continuous colour scale from 0 µm to the maximum value, there is very little difference in the map appearance. For this sample at least, the most elongated grains are also the largest in area.

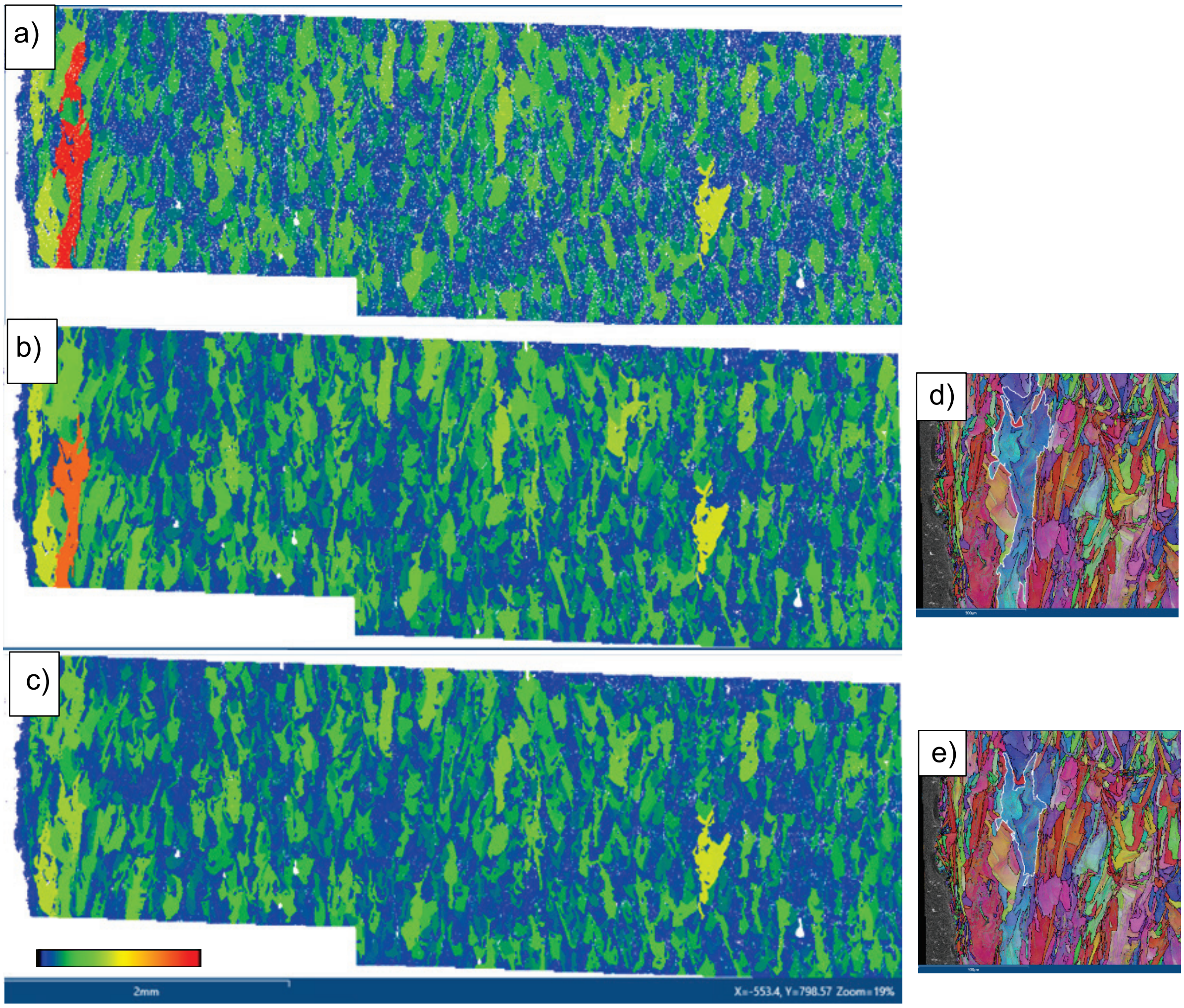

Figure 5 a), b) and c): maps of CED size using a rainbow scale of 0 - 500 µm for the full mapped area. a) raw data, b) after cleaning c) after extrapolating incomplete grain boundaries to 0°. d) and e) the same region from the far left of maps b) and c) shown with inverse pole figure orientation colouring with the largest grain highlighted with a white border before and after grain boundary extrapolation.

Alternatively, the maps can be plotted with a smaller upper limit so that all large grains are shown at the maximum of the scale, even where they exceed it. This reveals the grain size variation across the entire map more clearly, and should show the effects of grain boundary extrapolation can be seen more generally. Figure 6 maps the MFD for the raw data and the data cleaned+grain boundary extrapolated data: the visual changes are, however, relatively slight even though the mean value reduces by about 15% from the raw to the extrapolated map.

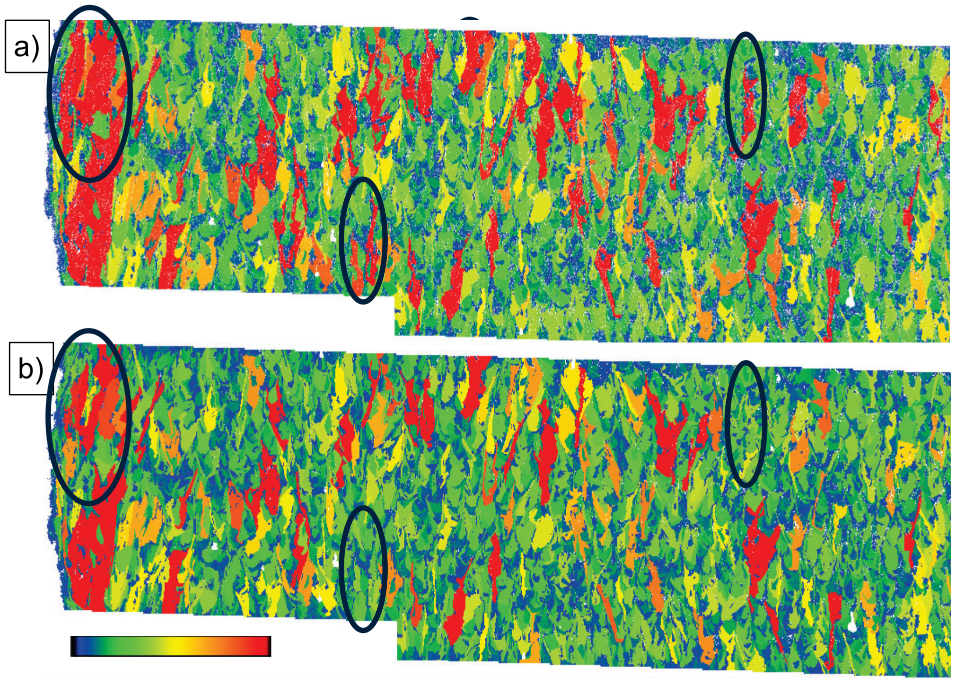

Figure 6. a), b) maps of MFD size using a rainbow scale of 0 - 500 µm for the full mapped area. a) raw data map and b) data cleaned and grain boundary extrapolated map. Three equivalent regions ringed as examples to facilitate comparison of where extrapolation has broken up larger grains.

3.3 EFFECT OF EDGE GRAIN EXCLUSION

It was noted that the use of area weighted averages reduced the spread of mean values. However, the values of area weighted CED and MFD for P2 do seem particularly low compared with all the other reported results.

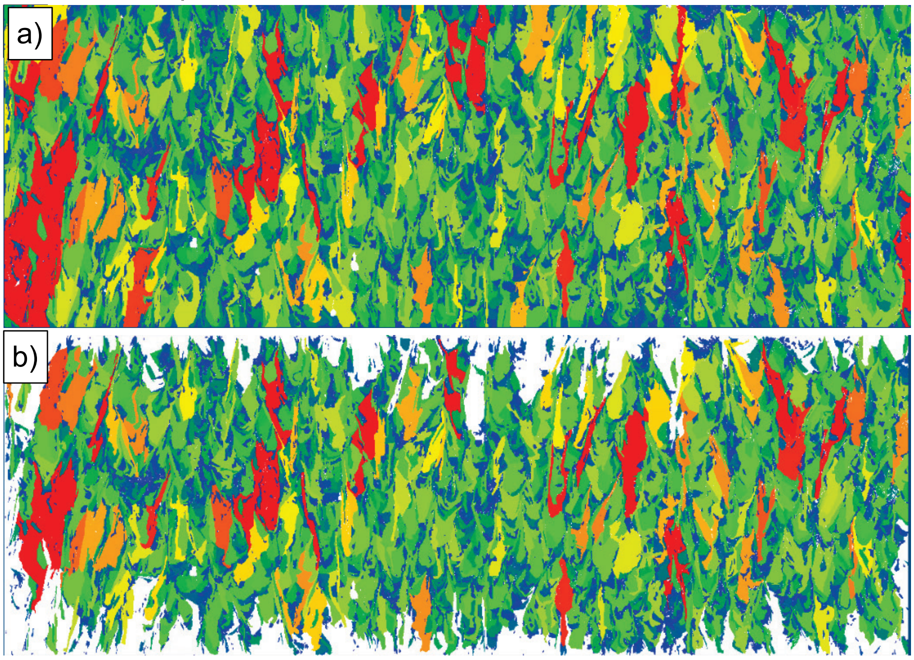

Figure 7. a), b) data cleaned and grain boundary extrapolated maps of MFD size using a rainbow scale of 0 - 500 µm, a) including edge grains and b) excluding edge grains.

Clearly grain boundary extrapolation must account for some of this but another factor that could also contribute to a low average was not documented. This factor was the exclusion of grains as a result of cropping the total area, which is then compounded by the exclusion from the cropped area of grains touching the edge of this area.

Figure 7 above shows the grains excluded from a rectangular area cropped from the full dataset. Clearly a significant number of grains with a MFD greater than the mean value are excluded because they touch the edge of the map. If all grains, even those cropped at the edge, were included in the area weighted calculation for the maps shown, the mean MFD would increase from 201.2 to 218.8 µm, or 18% while the number weighted mean would only increase by 2% from 51.5 to 53.2 µm.

However, map border grains can only be unambiguously defined for a rectangular map. Thus the need to crop a dataset such as the one provided in this comparison to a defined rectangular region is essential not just to be clear which regions of a sample are being assessed, but also so that it is clear what is being excluded from the calculations. Conversely, a non-rectangular region may include data from grains which have been truncated during mapping and thus produce an inaccurate indication of the size and number of large grains in the sample.

4 RECOMMENDATIONS FOR A NEW STANDARD

Based on the results described in this and preceding reports it is possible to summarise the potential ways in which a new standard for grain size measurement of additively manufactured materials would differ from the existing ISO13067 grain size standard. These recommendations are listed in Table 2 and encompass acquisition, data processing and data presentation aspects.

For the first item in Table 2, the current ILC dataset clearly illustrates what is possible with modern EBSD systems for acquiring large areas by stitching of multiple maps. It shows the advantages of mapping a large single area to illustrate variation with position and the applicability (or not) of determining a single average value for the whole area. It also illustrates the difficulties caused by not matching the size of the mapped area to the general grain shape and the need to exclude edge grains (item 2), but show what has been excluded. The requirements for choice of step size (item 3) remain broadly the same but can frequently be complicated by bimodal size distributions in AM materials. The arguments remain for retaining grains of < 100 pixels in size but excluding those < 10 pixels, but where there are large ranges of misorientation within grains, improved indexing to the 90% level should be aimed at. However, large ranges of misorientation still pose a challenge for deciding whether incomplete boundaries should be extrapolated down to lower misorientation.

Ultimately, the grain characteristics to be reported will be determined by the application and any previously established correlation of properties with characteristics. In the absence of such correlation it would seem sensible to use Feret diameters rather than equivalent circle diameters for non-equiaxed grains. In all cases though, an area weighted average of the characteristic should be used as this clearly reduces the sensitivity of the results to the acquisition and analysis parameters discussed above.

Table 2

Settings/ Parameters	ISO 13067:2020	Recommendation for New Standard
1. Area to map	Minimum 3 maps of >300 grains, avoid stitching	Large area, allow stitching, match area aspect ratio to grain shape.
2. Edge grains	No edge grains	Include elongated large grains as missing area? Or specify dimensions relative to mean size in different directions.
3. Step size to use	10% of mean size	10% of mean smaller dimension for elongated grains. If bimodal and variation with position, extra maps with smaller step size?
4. Minimum grain size	> 10 pixels	> 10 pixels, account for excluded area in cumulative size distribution.
5. Indexing and Data Cleaning	Minimum 80%. Up to 10% re-indexed.	Increase to 90%? Up to 10% re-indexed where majority are at boundaries.
6. Grain boundary threshold angle	10 - 15° for complicated structures, report extrap-olation to lower values	Determine suitable threshold angle as percentile of misorientation angle distribution?
7. Grain charac-teristics to report	Mean sectional size, Equiv. circle diameter, Grain areas	Use AREA WEIGHTED sectional size, report percentiles, use Feret diameters for non-equiaxed grains.
8. Data representation - distributions	Sectional grain size cumulative distribution	MUST PLOT Sectional grain size as CUMULATIVE distribution.
9. Maps		PLOT maps with grain size colour scale. Show regions where grains excluded by size or where touching edges.

5 SUMMARY

It has been shown that significant variation of the average calculated grain size of AM materials can result from changing the chosen minimum grains size, including or excluding grains touching the edge of the mapped area and deciding to extrapolate incomplete grain boundaries at the chosen threshold angle.

Based on these results, proposals have been made for ways in which a new standard for grain size measurement of additively manufactured materials would differ from the existing ISO13067 grain size standard. The importance of reporting area weighted averages has also been illustrated and should form part of any new standard.