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INTERLABORATORY COMPARISON OF EBSD GRAIN SIZE DISTRIBUTION ANALYSIS METHODS ON AN ADDITIVELY MANUFACTURED NI ALLOY

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Interlaboratory comparison of EBSD grain size distribution analysis methods on an additively manufactured Ni alloy

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Advanced Engineering Materials

ABSTRACT
This interlaboratory comparison study aimed to determine the current conventions and good practice for measuring, reporting, and visualising the grain size distribution in an additively manufactured nickel alloy microstructure measured using EBSD. Currently there is no suitable standard for EBSD-based grain size measurement of additively manufactured material microstructures, which typically have spatially heterogeneous, broad grain size distributions and anisotropic, non-convex grain shapes. Feret diameters and area-weighted average metrics were more suitable for describing the microstructure. Detailed reporting of the data processing method and grain size metric(s) chosen is crucial because measured value is very sensitive to the grain size metric and data processing methods, both of which are chosen by the operator.

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CONTENTS

1 INTRODUCTION

Additively manufactured materials have heterogeneous microstructures with broad grain size distributions and highly anisotropic and/or non-convex grain shapes. Currently there is no EBSD grain size measurement standard suitable for typical additively manufactured material microstructures. The ISO 13067:2020 [1] standard for measuring and reporting grain size, including electron backscatter diffraction (EBSD)-based methods, have been optimised for equiaxed grains with unimodal grain size distributions. The 2023 NPL Good Practice Guide on EBSD measurement of additively manufactured materials [2] contains pre-standardisation work and recommendations for optimising EBSD grain size measurement of additively manufactured Ni-superalloy microstructures.

This interlaboratory comparison study aimed to find out what grain size metrics and grain size distribution summary statistics are typically used by skilled operators to describe an additively manufactured nickel alloy microstructure measured using EBSD. Experimental details are provided in Appendix 1: Experimental method.

The same orientation map dataset was sent to all participants, to clean the data, reconstruct grains, measure and plot the grain size distribution, and report the grain size using suitable summary statistics. Exported images showing the expected EBSD map microstructure and a reporting template were provided (see Appendix 1: Experimental method for more details).

Five independent participants (P1 – P5) from four institutions in three countries participated in the comparison study. Participant P4 submitted three reports using different processing parameters (P4_1 – P4_3).

2 RESULTS AND DISCUSSION

The anonymised reports from all participants are provided in section ‘Appendix 3: Participants’ reports’.

2.1 ANALYSIS METHOD COMPARISON

All participants used qualitatively similar EBSD data cleaning methods but to very different degrees, ranging from no cleaning (P3, P4_1) to four steps of up to 25 iterations of removing small grains of no more than 4 pixels if they have indexed neighbours (P1), as well as somewhere in between (P2 – single iteration of removing un-/mis-indexed points with at least 3 indexed neighbours; P4_2, P4_3 – 10 iterations, at least 4 indexed neighbours). P4_2 and P4_3 used the ‘Auto Clean-up’ method provided by AztecCrystal 2.2.

Three analysis software types (AztecCrystal, OIM, MTEX) were used for data cleaning and grain reconstruction. AztecCrystal and OIM are supplied by EBSD system manufacturers (Oxford Instruments and EDAX respectively); Several software-specific phrases, are included in the reports, and the data cleaning steps are not directly comparable. Table 1 lists approximately equivalent data cleaning steps available in different analysis softwares. Software-specific phrases are documented only in the user manual which is not publicly available, This is a challenge for standardisation, especially in additively manufactured microstructures with broad grain size distributions, since the measured grain size is highly sensitive to data cleaning method. Future work could focus on developing a standard vocabulary for generic and unambiguous descriptions of the data cleaning methods.

MTEX is an open-source MATLAB toolbox for generic EBSD data analysis. P5 initially tried to use MTEX (versions 5.8.2 and 5.10.2 running in MATLAB R2023b) for grain size analysis, but grain reconstruction in MTEX produced around 90000 artefact grains within actual grains, often in interdendritic regions, which persisted even after applying several filtering steps. However, this behaviour was not observed by P3, who also used MTEX (version 6.0beta2, running in MATLAB R2023a).

This highlights a risk of using open-source scripting-based tools for data analysis: unintended changes to the source code (MTEX) and/or software environment (MATLAB) may produce unexpected results and may be difficult to trace. In contrast, compiled programs, such as those provided by the EBSD system manufacturers, cannot be easily modified by the user, but are less flexible than scripting-based tools, and the analysis method is not transparent as it is in open-source software.

Table 1 Comparison of data cleaning and grain reconstruction steps between available software types and ISO 13067:2020.

ISO 13067:2020	AztecCrystal	OIM	MTEX

Remove grains smaller than *n* pixels, where n is typically between 3 and 5		Define minimum grain size = *n*	`[grains,ebsd.grainId] =calcGrains(ebsd);`
			
			`ebsd(grains(grains.grainSize < n)) = [];`

ISO 13067:2020	AztecCrystal	OIM	MTEX

[none]	[none]	Define minimum anti-grain size = *m*	Only keep 'notIndexed' grains at least *m* pixels, treat everything else as unknown:
			
			`grains( grains('notIndexed').id( grains('notIndexed').grainSize < m) )=[];`
			
			or use the 'alphashape' flag during grain reconstruction
			
			`grains = calcGrains(ebsd, 'alpha', sqrt(m/pi));`

ISO 13067:2020	AztecCrystal	OIM	MTEX

	Remove Wild Spikes (reindex single pixels completely surrounded by one grain)		Remove single-pixel indexed grains
			
			`ebsd(grains(grains('indexed').id(grains('indexed').grainSize < 2)))=[];`
			
			Fill missing points within grains with nearest neighbour orientations
			
			`ebsd = fill(ebsd,grains)`

Reindex singly unindexed pixels surrounded by *x* or more pixels of similar orientation	(Level 9-*x*) – *x* Neighbors Zero Solution Removal	Neighbor Orientation Correlation – Level (6-*x*) cleanup (for a hexagonal measurement grid)	[no equivalent]

More aggressive data cleaning protocols decreased the total number of measured grains. Results between participants are not directly comparable because of differences in total grain measurement area due to image cropping, which ranged from crop to rectangle (P2, P4, P5) to no cropping at all (P3). The sample measurement area should also be included in the future to avoid this ambiguity.

The total numbers of grains measured by P4_3 and P5 were very similar (9401 and 9040 respectively); both used 5° grain boundary misorientation threshold angles and cropped the data to a rectangle.

Broadly similar grain reconstruction methods were used. P5 chose a grain boundary threshold angle of 5° by average grain diameter . P1, P2, P3 and P4 used a grain boundary threshold angle of 10°. P4 also compared threshold angles of 10° (P4_2) and 5° (P4_3) using otherwise identical processing methods. This increased the number of detected grains by 36 %.

Participants chose to deal differently with incomplete boundary segments (always extrapolate to close, close down to 5°, do not close). Additively manufactured materials with strong textures or subgrain structures may contain significant numbers of incomplete boundary segments as well as low-angle grain boundaries. The number of additional grains created by closure of these boundary segments could be explored in future work and reported for relevant microstructures.

Two out of five participants chose to exclude map border grains. In ISO 13067:2020, border grains should be excluded to avoid counting partial grain areas, but if the grain size is too large, the Miles-Lantuéjoul correction can be applied to compensate for the inclusion of partial grain areas. This appears to be an error: the Miles-Lantuéjoul correction [3] compensates for the increased likelihood of a large particle cutting the image border by assigning a weighting factor to each grain in the grain size distribution. Therefore, it should be applied to grain size distributions where edge grains have been excluded, to account for the fact that the bigger grains were more likely to have been excluded; not where they are included, because no correction is available to account for partial grain areas.

Additively manufactured microstructures typically have highly anisotropic microstructures where the grain size is large compared to the EBSD map area. Future work should explore the effect of excluding border grains, as well as statistical methods such as the Miles-Lantuéjoul correction, on the measured grain size distribution and the maximum measured grain size.

Table 2 EBSD data processing parameters chosen by study participants, and ISO 13067:2020 standard requirements and recommendations for comparison

Participant	P1	P2	P3[1]	P4_1	P4_2	P4_3	P5	ISO 13067:2020
Analysis software	**AztecCrystal** v.3.1.390	**AztecCrystal** v2.2	**MTEX** 6.0.beta2 / MATLAB R20 23a	**AztecCrystal** v2.2.302			**OIM** Version 9.0.0.208	
Data cleaning method	Remove wild spike pixels Remove grains up to 4 pixels, slowly decreasing the number of necessary neighbours: Eight neighbours (x2) 3. Seven neighbours (x25) 4. Six neighbours (x25) 5. Five neighbours (x25)	Remove wild spike pixels Unindexed points reassigned where 5 pixels surrounding are unindexed (no iteration) Kuwahara filter (quadrant, 3 pixel radius)	None	None	Auto clean up Wide peaks remove Zero solution removal 4 neighbourhood 10 iterations		1) Grain CI Standardization: 5 degree grain misorientation definition, 2 pixel minimum anti-grain size, no multi-row requirement 2) Grain Dilation - 5 degree grain misorientation definition, 2 pixel minimum anti-grain size, Single iteration 3) Neighbor CI Correlation – Minimum CI of 1 (to combat artifacts from creating .ang in MTEX), Single iteration 4) Neighbor Orientation Correlation – Level 2 cleanup, 5 degree grain misorientation definition, Minimum CI of 1 (to	Remove grains smaller than 3-5 pixels Absorb single unindexed pixels, but percentage indexed should not increase by > 5 – 10 %. Report the % increase in indexing rate.

[1] Method parameters in brackets (*) were included in the report, but qualified as unsuitable for describing the grain structure.

Participant	P1	P2	P3[1]	P4_1	P4_2	P4_3	P5	ISO 13067:2020
							combat artifacts from creating .ang in MTEX)	
Data cropping	Excluded ~200 µm from left hand edge	Exclude left hand edge Cropped to a rectangular region	None	<inferred from map plots, not explicitly mentioned> Cropped to a rectangular region			Cropped to remove edge noise (~85% of total area still surveyed)	
Grain reconstruction method	10° misorientation threshold Close incomplete boundaries extrapolated to 5°	10° misorientation threshold Close incomplete boundaries extrapolated to 0°	10° misorientation threshold Don’t close incomplete boundaries Draw grain boundaries between unindexed points	10° misorientation threshold All pixels in the subset	10° misorientation threshold All pixels in the subset	5° misorientation threshold All pixels in the subset	5° misorientation threshold	Between 10° and 15° Optional - close incomplete boundaries, record extrapolation angle and report effect on mean size
Map border grains	Excluded	Excluded	Included	Included			Included	Exclude Use Miles-Lantuéjoul correction
Min grain size	100 pixels (ASTM E2627-13)	10 pixels	1 pixel	10 pixels			10 pixels	10 pixels

Participant	P1	P2	P3[1]	P4_1	P4_2	P4_3	P5	ISO 13067:2020
Grain size metric(s)	Equiv. circle diameter Max Feret diam. Ellipse major axis diam. Ellipse minor axis diam.[2]	Equiv. circle diameter Max Feret diam.	(Equiv. circle diameter) Max Feret diam. Min Feret diam.	Equivalent circle diameter Aspect ratio			Diameter Aspect ratio of ellipse fit to grain Max Feret diam. Min Feret diam. Ellipse major axis diam. Ellipse minor axis diam. Grain ellipticity Grain circularity	Sectional grain size: Equiv. circle diameter Feret diameters Area 3D-grain size: Mean linear intercepts perpendicular and parallel to sample tilt axis, or along sample processing axes.
Size distribution summary metric(s)	[Number-weighted?] arithmetic mean Area weighted [arithmetic?] mean[3]	Number weighted arithmetic mean Area weighted arithmetic mean	(Number weighted arithmetic mean) (Area weighted arithmetic mean) Area weighted median	Arithmetic mean Minimum Maximum Standard deviation			Arithmetic mean	Mean Minimum Maximum Standard deviation (of distribution width) D10, D50, D90 Uncertainty from three mapped areas quoted with a 95 % confidence interval

[2] This was mentioned in the ‘Analysis Method’ section, but no results were included in ‘Summary Statistics’.

[3] The report states ‘Arithmetic’ and ‘Area weighted’ means. These refer to different parameters (arithmetic vs geometric mean, and area weighted vs number weighted mean). The words enclosed in square brackets [*?] are guessed or inferred from the size distribution plots later on in the report.

Participant	**P1**	**P2**	**P3**[1]	**P4_1**	**P4_2**	**P4_3**	**P5**	**ISO 13067:2020**
			Area weighted geometric mean (Area weighted histogram mode)					
Number of grains measured	6439	7649	32148 (7752 grains >= 10 pixels)	12778	6917	9401	9040	3 image fields 300 grains per field

2.2 SUMMARY STATISTICS COMPARISON

All participants produced very different numerical values for nominally similar grain size metrics. Number and area-weighted arithmetic means for the equivalent circle and max Feret diameters were compared as all participants reported these values.

The range of number-weighted mean values differed between participants by more than a factor of 2 for both diameter metrics. The area-weighted mean values were more consistent between participants.

ISO13067:2020 distinguishes between 3D grain size and sectional grain size, and both can be represented as equivalent circle diameters or grain areas. The number-weighted mean 3D grain size, expressed as an equivalent circle diameter, is directly comparable to the mean intercept lengths, but the sectional grain size is not. All five participants reported sectional grain size metrics only.

2.2.1 Effect of data cleaning

P4 also reported minimum and maximum grain sizes, and the standard deviation of the grain size distribution. The minimum grain size is an artefact of the data processing method, since it is always equivalent to the minimum grain area of 10 pixels considered in the grain size distribution calculation (Table 2). 4.5 μm is the equivalent circle diameter of a 10 pixel area (15.6 μm^2).

The maximum grain area measured using AztecCrystal is underestimated when no data cleaning is applied (P4_2), since unindexed points are absorbed into the grains around them during the ‘zero solutions removal’ step. This does not generally apply to grain sizes measured using MTEX (P3), because the grain segmentation method draws grain boundaries between unindexed points instead around clusters of indexed points.

2.2.2 Relationships between summary statistics and size distributions

P5 reported grain area as well as grain diameter (as-calculated by OIM software, presumably equivalent circle diameter). Note that the average equivalent circle diameter cannot be directly computed from the average grain area, even though the equivalent circle diameter of a single grain can be directly computed from its area.

The ‘aspect ratio’ metrics reported by P4 and P5 could not be compared. P5 used the ratio of ellipse axis diameters and summarised this using the arithmetic mean. The values provided by P4 were ambiguous because no method details were provided, and there are multiple ways of calculating aspect ratio. For example, aspect ratio can be calculated using the ratio between maximum and minimum Feret diameters, as an alternative to ellipse axis diameters (P5).

Aspect ratios are expressed as either > 1 or < 1, although the ISO 13607:2020 standard specifies < 1. Converting between these conventions is simple for single grains, but the mean aspect ratio of of a distribution cannot be directly converted between conventions by inverting the mean values. If the aspect ratio distribution is broad, the original elliptical grain size distributions cannot be trivially recovered from, for example, the major axis diameter distribution and the aspect ratio distribution.

The physical aspect ratios distribution is not close to normal or log-normal, since it is the ratio of two correlated (log-)normal distributions.

Table 3. Grain size and size distribution summary metrics chosen by study participants, and the mean and standard devation of the reported values.

Grain size metric	Size distribution summary metric	P1	P2	P3[4]	P4_1	P4_2	P4_3	P5	Participants avg (mean ± 1 σ)
Equivalent circle diameter / μm	Number weighted arithmetic mean	38.2	22.5	(17)	16.2	25.6	22.32	22.12	**23 ± 7.3 μm**
	Area weighted arithmetic mean	97.1	82.0	(111)	[96.2][5]	[107.3]	[88.3]		**97 ± 11 μm**
	Minimum				4.5	4.5	4.5		**4.5 ± 0 μm**
	Maximum				444.3	460.6	352.6		**419 ± 58 μm**
	[Size distribution width] Std deviation				21.8	28.7	24.2		**25 ± 3.5 μm**
Area / μm^2	Number weighted arithmetic mean							861.72	**817 μm^2**
Max Feret diameter / μm	Number weighted arithmetic mean	88.0	49.8	(37)				48.77	**58 ± 27 μm**
	Area weighted arithmetic mean	233.9	194.2	(281)					**236 ± 43 μm**
	Area weighted median			175 ± 25					**175 μm**
Min Feret diameter / μm	Number weighted arithmetic mean			(13)				18.08	**15 ± 3.6 μm**
	Area weighted median			53 ± 8					**53 μm**
Ellipse major axis diameter / μm	Number weighted arithmetic mean	73.9						24.39	**49 ± 35 μm**
	Area weighted arithmetic mean	188.5							**189 μm**
Ellipse minor axis diam / μm	Number weighted arithmetic mean							6	**6 μm**

[4] Numbers in brackets (*) were included in the report, but qualified as unsuitable for describing the grain structure.
[5] Numbers in square brackets [*] were not included by participant P4 in their 'Summary Statistics' table, but were exported with the histogram images.

Grain size metric	Size distribution summary metric	P1	P2	P3[4]	P4_1	P4_2	P4_3	P5	Participants avg (mean ± 1 σ)
Aspect ratio	Unknown, > 1 convention;				3.52	3.53	3.6		**3.4 ± 0.2**
	Arithmetic mean, < 1 convention							0.32	**0.32**
Grain ellipticity								0.94	**0.94**
Grain circularity								0.33	**0.33**

2.3 QUALITATIVE JUSTIFICATIONS FOR CHOICE OF GRAIN SIZE METRICS

Two participants (P1, P3) noted that equivalent circle diameter is not suitable for elongated grains typical of additively manufactured materials. P5 recommended reporting grain area as a metric for the grain domain size; grain area does not imply nor require any assumptions about the grain shape.

P1 recommended choosing a grain size metric with good correlation to mechanical properties would enable translation between additively manufactured and conventionally processed materials. P5 recommended the use of maximum Feret diameter in ‘Hall-Petch and similar calculations’, which link mechanical strength to the mean free path length of a dislocation gliding in the grain. However, additively manufactured alloys often contain non-convex grains, which could reduce the correlation between Feret diameters and mechanical properties. Feret diameters may not correspond to a physical line section across a non-convex grain, since they are defined using the convex hull of a polygon. The correlation between mechanical properties, physical grain section lengths and Feret (and ellipse-fit) lengths could be explored in further work.

P3 recommended choosing a grain size metric that is relatively insensitive to large numbers of small grains, instead of attempting to filter them out during EBSD data cleaning.

Table 4. Qualitative justifications of choice of grain size summary statistics provided by participants. (P2 and P4 did not complete this section.)

P1	P3	P5
Equivalent circle diameter is not suitable for elongated grains	Equivalent circle diameter is not suitable for elongated grains Max and min Feret diameters are more suitable, since the Max Feret angles are aligned close to the build direction	Recommend the use of area for measuring the domain size of each grain
Suggested choosing a metric to fit into the Hall-Petch relationship between mechanical properties (hardness / yield point) and grain size of equivalent conventionally processed material. Values obtained this way would make translation from additively manufactured materials grain size/properties to conventional one grain/size properties.		Recommend maximum Feret diameter for “grain size” in Hall-Petch and similar calculations
	Since the grain size distribution is very wide, small grains should not be filtered out using an arbitrary grain size threshold. Instead, choose a grain size metric that is relatively insensitive to large numbers of small grains, e.g. area-weighted median.	
	Geometric mean is more suitable than arithmetic mean when describing a log-normal grain size distribution	

2.4 SIZE DISTRIBUTION PLOTS COMPARISON

All participants chose different plots to visualise the grain size distribution.

Log-normal probability plots (P2) allow both the geometric mean grain size and log-normal distribution width (in units of standard deviations) to be read directly off the plots. The linearity of the plot lines shows how well the grain size distribution fits to a log-normal distribution. The area-weighted grain sizes fit better to a log-normal distribution than the corresponding number-weighted grain sizes, which are skewed towards small grain sizes.

Both the number-weighted (P1) and area-weighted (P3, P4) histogram modes are significantly below the corresponding mean values for all grain size metrics. Additionally, the position of the histogram mode is sensitive to bin width, which is an operator choice. Since the minimum, mode and maximum of a distribution are the main parameters that can be read directly from a histogram plot, histograms are not that useful.

Cumulative histograms (P3) are an improvement over conventional histograms as they are insensitive to bin width, and any percentile value, including the median, can be read directly from the plot. The area-weighted median shows good correspondence to the area-weighted geometric mean values.

Grain-average EBSD maps colour-coded by grain size (P3) allow the spatial heterogeneity of the grain size distribution to be qualitatively observed. For example, the band of small grains along the left-hand edge of the map, which was excluded from analysis by two participants (P1, P2), is obvious in the map but not the size distribution plots.

Maxima in the EBSD maps colour-coded by Euclidean distance to nearest grain boundary (P4) correspond to half the maximum Feret diameter. It is easier to see potential weak points caused by large grains from Euclidean distance maps, but the grain size is not as easy to read compared to grain-average EBSD maps.

Table 5. Plot types used by participants to visualise the EBSD grain size distribution.

P1	**P2**	**P3**	**P4**	**P5**	**ISO 13067:2020**
Number-weighted histogram				Number-weighted histograms (for ellipse fit diameters, Feret diameters, aspect ratio, ellipticity, circularity)	Cumulative distribution (mandatory) Binned histogram (optional) Can be number- or area-weighted
		Area-weighted histogram and cumulative histogram	Area-weighted histogram	Area-weighted histograms (for grain diameter, grain area)	

	Log-normal probability plots [standard deviations from mean vs log(grain size)]				
		Grain-average EBSD maps colour-coded by grain size	EBSD maps colour-coded by Euclidean distance to nearest grain boundary		

3 SUMMARY AND RECOMMENDATIONS

- There was no consensus in measured grain size even for nominally similar metrics, because the measured values were extremely sensitive to EBSD data cleaning method. Area-weighted values are less sensitive than number-weighted values because they are affected less by large numbers of small grains.
- A lack of standard methods and vocabulary related to EBSD data cleaning methods, combined with high sensitivity of EBSD grain size to the data cleaning method, leads to limited comparability between measurements produced by different software systems.
- Equivalent circle diameters were not a suitable grain size metric for this microstructure, but maximum (and minimum) Feret diameters could be used instead.
- Log-normal probability plots and cumulative histograms are useful for visualising the grain size distribution as well as showing summary statistics such as mean and median values. Grain size colour-coded maps are useful for qualitative inspection and visualising microstructural heterogeneity. However, conventional histograms are not as useful.
- The total measurement area after EBSD data cleaning should be reported in the future, to account for differences in map crop area.
- If aspect ratio is used, its computational method details should be reported as well.
- The number of additional grains created by closure of incomplete boundary segments, and the sensitivity of measured grain size to boundary threshold angle, could be explored in future work and reported for relevant microstructures.
- A suitable grain size metric should be sensitive to the relationship between grain size and mechanical properties to be practically useful in characterising additively manufactured materials. The correlations between Feret and ellipse-fit grain size metrics, grain section lengths, and mechanical properties could be explored in future work, especially for non-convex grain microstructures.

4 REFERENCES

[1] ISO, ISO 13067:2020 Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size, 2020.
[2] K.P. Mingard, The use of EBSD for analysis of additively manufactured materials, (2023). http://eprintspublications.npl.co.uk/id/eprint/9639.
[3] J. Escoda, D. Jeulin, F. Willot, J. Escoda, D. Jeulin, F. Willot, Simulation of 3D granular media by multiscale random polyhedra To cite this version : HAL Id : hal-00879260 SIMULATION OF 3D GRANULAR MEDIA BY MULTISCALE RANDOM POLYHEDRA, (2013).

5 APPENDIX 1: EXPERIMENTAL METHOD

The Ni alloy sample was manufactured using a laser melting powder bed additive manufacturing method. The surface was polished on the X-Z plane with the build direction pointing up in the EBSD map.

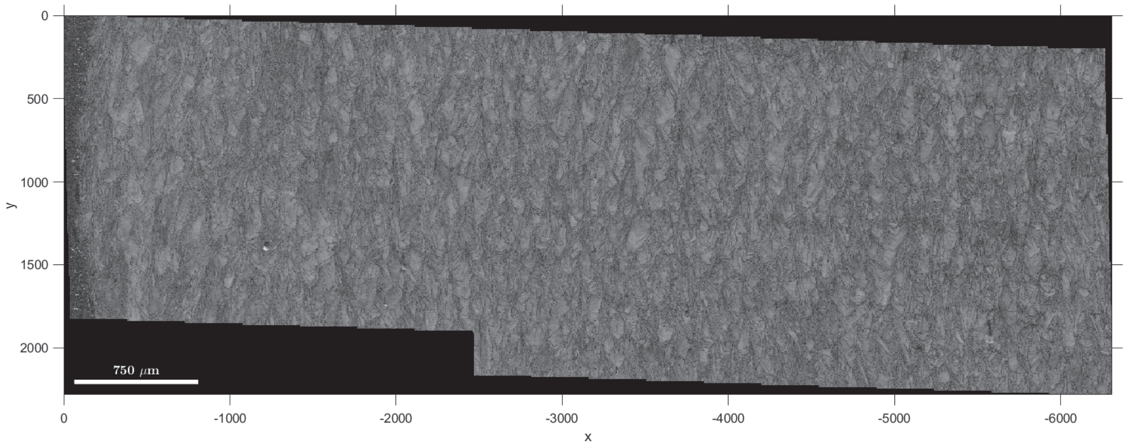
The scanning electron microscope (SEM) used was a Zeiss Auriga-60 field emission gun SEM operating at 30 kV accelerating voltage. The specimen tilt angle was 70° and the working distance was 15.5 mm.

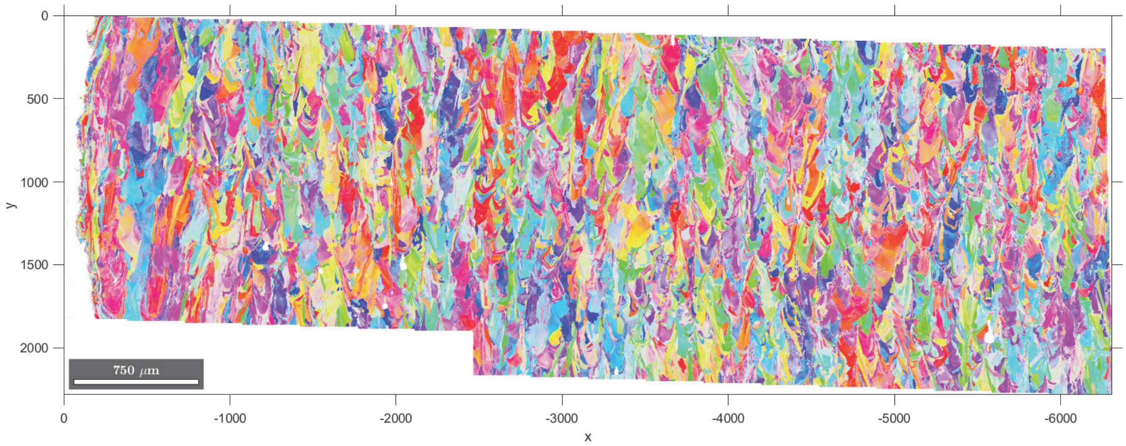
The EBSD map was acquired using an Oxford Instruments Symmetry2 EBSD camera running Aztec 2.2 software. The exposure time was 0.4 ms per pattern and the EBSD pattern resolution was 156 × 128 pixels (‘Speed 2’ mode). The candidate phase used was face-centred cubic nickel superalloy ($\langle a \rangle$ = 0.357 nm). The overall indexing success rate, after map tile stitching, was 89 %.

The total image field step size of 1.251 µm and the map dimensions were 5044 × 1825 pixels (6310 × 2283 µm^2) on a square pixel grid. The measured image field was divided into 137 tiles of 303 × 288 pixels each.

6 APPENDIX 2: FILES SENT TO PARTICIPANTS

Two EBSD map images and a reporting template were sent to participants alongside the orientation data, which was provided as an Oxford Instruments *.h5oina file directly exported from the EBSD map acquisition sofware, a MATLAB *.mat file with an MTEX loading script, or an EDAX *.ang text file created using the MTEX file export tool.





Grain size distribution report template

6.1 ANALYSIS METHOD

Please add additional rows to report any parameters that are not currently included.

		Details	Notes / References
1	**Analysis software**	[include version details e.g. MTEX # / MATLAB R#, OIM Analysis v#, AztecCrystal v#]	
	Data cleaning method	[e.g. remove wild spike pixels, orientation smoothing, how unindexed points are handled, any cropping or subsets extracted]	[e.g. reference to detailed method description]
2	**Grain reconstruction method**	[method details, e.g. boundary misorientation threshold angle[s], orientation interpolation / data cleaning, how were incomplete boundaries handled]	
3	**Map border grains**	[Included / excluded]	
4	**Minimum grain size**	[none / # pixels / other metric]	[e.g. reference or justification for selecting a certain threshold size]
5	**Grain size metric(s)**	[e.g. intercept lengths, circle equivalent diameter, ellipse major/minor axes]	
6	**Size distribution summary metric(s)**	[e.g. arithmetic / geometric mean, median, mode(s). Include details about how you calculated this, e.g. histogram bin width, CDF]	
7	**Number of grains measured**	[#]	

[if using a scripting software – optionally include source code here]

6.2 RESULTS

6.2.1 Summary statistics

	Grain size metric	Size distribution summary metric	Value	Unit	Additional notes
1	[e.g. Circle equivalent diameter]	[e.g. arithmetic mean]	[#]	[e.g. μm]	[e.g. excluded # outliers]
2					
3					

[some kind of qualitative note to which metric(s) are more or less appropriate for this dataset]

6.2.2 Size distribution plots

[e.g. histogram or cumulative histogram plots corresponding to each line in the Summary statistics table]

6.3 REFERENCES

This standard may be helpful: ISO. (2020). *ISO 13067:2020 Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size*.
[Add any more if required / used in the previous sections]

7 APPENDIX 3: PARTICIPANTS' REPORTS

The completed grain size reports from all participants are included below.

7.1 P1

7.1.1 Analysis method

Please add additional rows to report any parameters that are not currently included.

		Details	Notes / References
1	**Analysis software**	AztecCrystal v.3.1.390	
	Data cleaning method	1. Wild spike removal 2. Eight neighbours (x2) 3. Seven neighbours (x25) 4. Six neighbours (x25) 5. Five neighbours (x25)	Grains up to 4 pixels are removed. Slowly decreasing number of necessary neighbours. ~200 µm from the left side of the dataset were excluded due too low hit rate and because these looked like a surface layer and would not reflect the bulk properties.
2	**Grain reconstruction method**	Boundary misorientation threshold 10 degrees Close boundaries down to 5 degrees	
3	**Map border grains**	excluded	Some of the grains are very long, therefore I'm not convinced to include border grains.
4	**Minimum grain size**	100 pixels	ASTM E2627-13
5	**Grain size metric(s)**	Circle equivalent diameter Ellipse major axis diameter Ellipse minor axis diameter Max Feret diameter	
6	**Size distribution summary metric(s)**	Arithmetic Area weighted (both as delivered by)	
7	**Number of grains measured**	6439	

7.1.2 Results

7.1.2.1 Summary statistics

	Grain size metric	Size distribution summary metric	Value	Unit	Additional notes
1	Circle equivalent diameter	Arithmetic mean	38.2	µm	
2	Circle equivalent diameter	Area-weighted mean	97.1	µm	

3	Fitted ellipse major diameter	Arithmetic mean	73.9	µm	
4	Fitted ellipse major diameter	Area-weighted mean	188.5	µm	
5	Max Feret diameter	Arithmetic mean	88.0	µm	
6	Max Feret diameter	Area-weighted mean	233.9	µm	

It is more a question what do we want to achieve. If we want to use a metric that better describes these elongated grains most probably equivalent circle diameter is not the best. Typically we measure grains because grain size affects mechanical properties (Hall-Petch). If we would know the hardness/Yield point for this material we could choose a method that would fit into the Hall-Petch relationship of the conventional material. Values obtained this way would make translation from additively manufactured materials grain size/properties to conventional one grain/size properties.

7.1.2.2 Size distribution plots

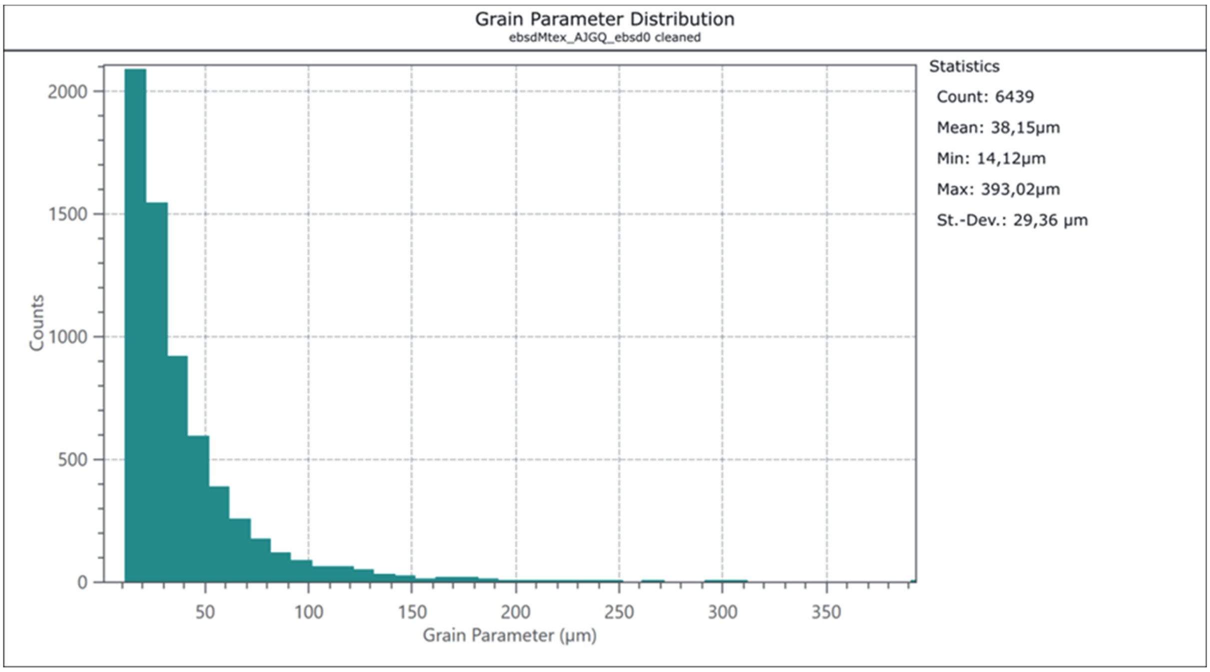


Figure 1 Equivalent circle diameter distribution

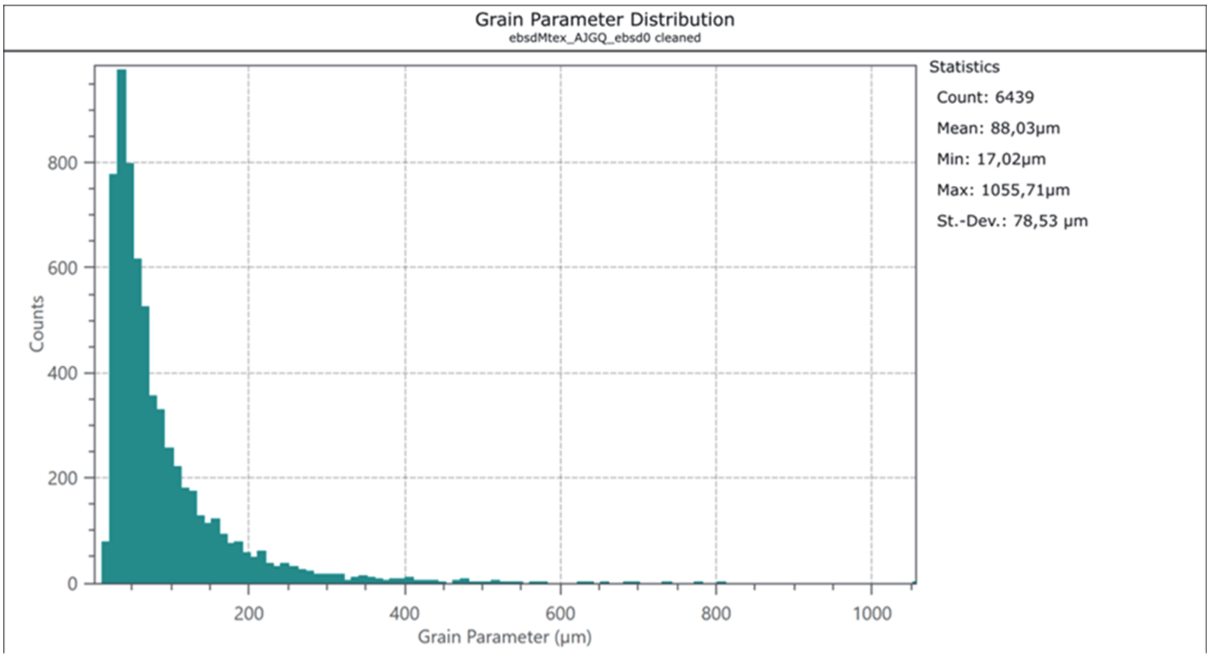


Figure 2 Max Feret diameter distribution

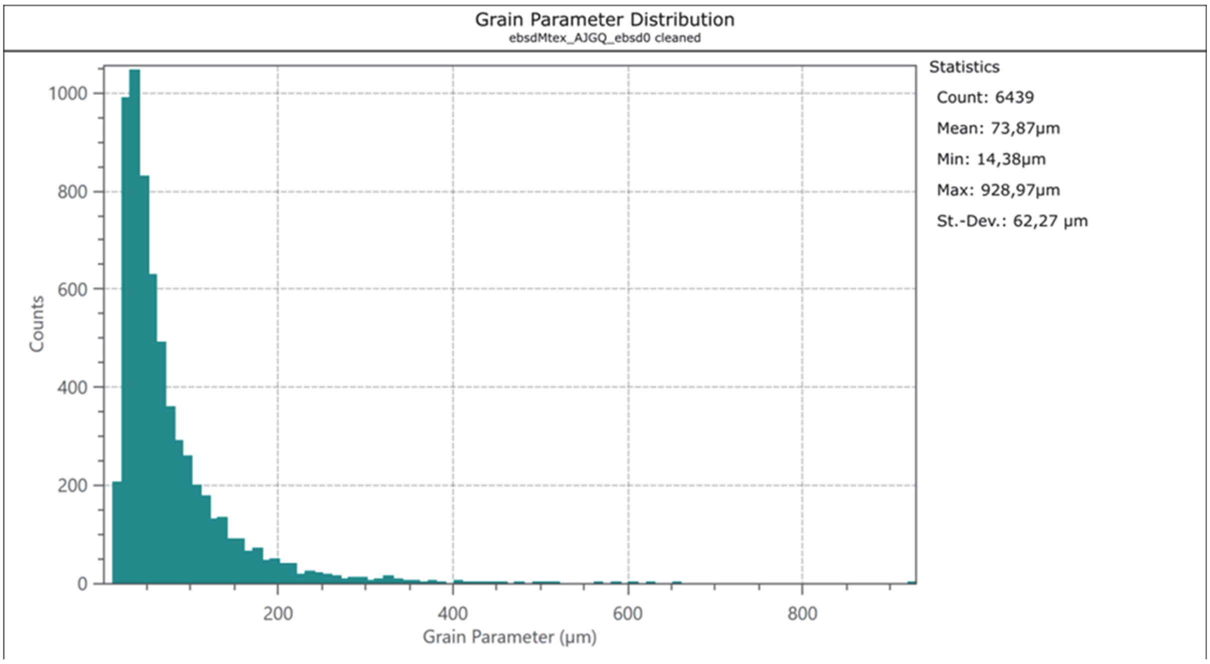


Figure 3 Fitted ellipse major diameter distribution

7.1.3 References

This standard may be helpful: ISO. (2020). *ISO 13067:2020 Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size*.
ASTM E2627-13(2019) Standard Practice for Determining Average Grain Size Using Electron Backscatter Diffraction (EBSD) in Fully Recrystallized Polycrystalline Materials
Short video on grain size measurements using Oxford Instruments system: https://www.youtube.com/watch?v=7WoI1XX6xhM&t=15s
AztecCrystal Help
[Add any more if required / used in the previous sections]

7.2 P2

7.2.1 Analysis method

Please add additional rows to report any parameters that are not currently included.

		Details	Notes / References
1	**Analysis software**	AztecCrystal v2.2	
	Data cleaning method	remove wild spike pixels, unindexed points reassigned where 5 pixels surrounding unindexed (no iteration) ,Kuwahara filter (quadrant, 3 pixel radius). Cropped to a rectangular region to exclude left hand edge	90.44% raw increased to 98.21% pixels indexed (7.77% “re”indexed) for cropped dataset
2	**Grain reconstruction method**	10 degree threshold angle, incomplete boundaries extrapolated to 0°	
3	**Map border grains**	excluded	
4	**Minimum grain size**	10 pixels	
5	**Grain size metric(s)**	Circle equivalent diameter and max feret	
6	**Size distribution summary metric(s)**	Number and area weighted arithmetic mean values	
7	**Number of grains measured**	7649	

[if using a scripting software – optionally include source code here]

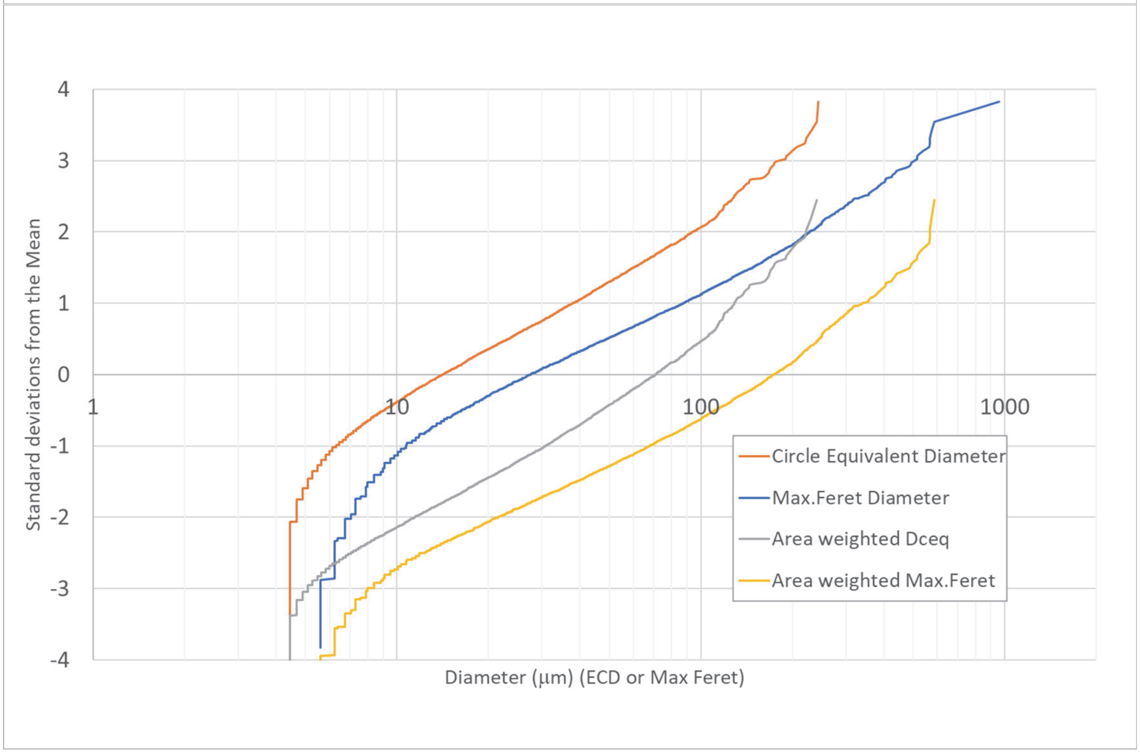
7.2.2 Results

7.2.2.1 Summary statistics – cropped to remove left hand edge

	Grain size metric	Size distribution summary metric	Value	Unit	Additional notes
1	Circle equivalent diameter	arithmetic mean	22.5	µm	
2	Circle equivalent diameter	Area weighted arithmetic	82.0	µm	
3	Max Feret diameter	arithmetic mean	49.8	µm	
	Max Feret diameter	Area weighted arithmetic	194.2	µm	

[some kind of qualitative note to which metric(s) are more or less appropriate for this dataset]

7.2.2.2 Size distribution plots



7.2.3 References

This standard may be helpful: ISO. (2020). *ISO 13067:2020 Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size*.

[Add any more if required / used in the previous sections]

7.3 P3_1

7.3.1 Analysis method

The method parameters finally selected for reporting the summary grain size statistics are marked in **bold**.

		Details	Notes / References
1	Analysis software	**MTEX 6.0.beta2 (release sept 2023)** **MATLAB R2023a**	
	Data cleaning method	**As-indexed grains** remove grains < 2 pixels and recalculate grains remove grains < 10 pixels and recalculate grains	[e.g. reference to detailed method description]
2	Grain reconstruction method	**Boundary misorientation threshold angle = 10°** **Draw grain boundaries between missing points** (including grains below threshold size) **Ignore incomplete boundaries**	
3	Map border grains	**Including border grains** (Border grains were nominally excluded in MTEX, but since the map area is not rectangular, the majority of grains on the edge of the map were not counted as intersecting the rectangular map border)	
4	Minimum grain size	**All grains** 2 pixels area 10 pixels area	[e.g. reference or justification for selecting a certain threshold size]
5	Grain size metric(s)	Circle equivalent diameter **Maximum Feret diameter** **Minimum Feret diameter**	
6	Size distribution summary metric(s)	Area-weighted histogram mode (using 20 bins between 0 and approx. max grain size) **Area-weighted median, computed as 50th percentile of cumulative histogram** Area-weighted geometric mean Area-weighted arithmetic mean Number-weighted arithmetic mean	
7	Number of grains measured	**Total: 32148 grains** Consisting of: • 14322 1-pixel grains • 10074 <10-pixel grains • 7752 >-=10-pixel grains	

[if using a scripting software – optionally include source code here]

7.3.2 Results

7.3.2.1 Summary statistics

The area-weighted medians of the maximum and minimum Feret diameters are 175 μm ± 25 and 53 μm ± 8 respectively.

	Grain size metric	**Size distribution summary metric**	**Value**	**Unit**	**Additional notes**
	Circle equivalent diameter	Area weighted histogram mode (bin width 20 μm)	50 ± 10	μm	All grain sizes
			50 ± 10		Excluded grains < 2 pixels
			50 ± 10		Excluded grains < 10 pixels
		Area weighted median (cumulative histogram, bin width 20 μm)	70 ± 10		All grain sizes
			70 ± 10		Excluded grains < 2 pixels
			90 ± 10		Excluded grains < 10 pixels
		Area weighted geometric mean	83		All grain sizes
			84		Excluded grains < 2 pixels
			111		Excluded grains < 10 pixels
		Area weighted arithmetic mean	111		All grain sizes
			111		Excluded grains < 2 pixels
			144		Excluded grains < 10 pixels
		Number weighted arithmetic mean	17		All grain sizes
			20		Excluded grains < 2 pixels
			29		Excluded grains < 10 pixels
	Max Feret diameter	Area weighted histogram mode (bin width 50 μm)	75 ± 25		All grain sizes
			75 ± 25		Excluded grains < 2 pixels
			75 ± 25		Excluded grains < 10 pixels
		Area weighted median (cumulative histogram, bin width 50 μm)	**175 ± 25**		**All grain sizes**
			175 ± 25		Excluded grains < 2 pixels
			175 ± 25		Excluded grains < 10 pixels
		Area weighted geometric mean	194		All grain sizes
			195		Excluded grains < 2 pixels
			237		Excluded grains < 10 pixels
		Area weighted arithmetic mean	281		All grain sizes
			282		Excluded grains < 2 pixels
			338		Excluded grains < 10 pixels
		Number weighted arithmetic mean	37		All grain sizes
			45		Excluded grains < 2 pixels
			57		Excluded grains < 10 pixels
	Min Feret diameter	Area weighted histogram mode (bin width 15 μm)	23 ± 8		All grain sizes
			23 ± 8		Excluded grains < 2 pixels
			23 ± 8		Excluded grains < 10 pixels
		Area weighted median (cumulative histogram, bin width 15 μm)	**53 ± 8**		**All grain sizes**
			53 ± 8		Excluded grains < 2 pixels
			68 ± 8		Excluded grains < 10 pixels
		Area weighted geometric mean	61		All grain sizes
			61		Excluded grains < 2 pixels
			84		Excluded grains < 10 pixels
		Area weighted arithmetic mean	82		All grain sizes
			82		Excluded grains < 2 pixels
			109		Excluded grains < 10 pixels
		Number weighted arithmetic mean	13		All grain sizes
			15		Excluded grains < 2 pixels
			23		Excluded grains < 10 pixels

Qualitative notes on which metrics were most appropriate for this dataset:

- Minimum and maximum Feret diameters are good for characterising the anisotropy of the build structure, since the max Feret angles are all aligned close to each other and along the sample build direction. In contrast, the circle equivalent diameters do not correspond to any physical lengths that can be measured from the EBSD map.

- Area-weighted metrics are less sensitive to minimum grain size threshold than the number-weighted arithmetic mean (also other number-weighted metrics that are not shown here). Number-weighted mean values are far smaller than the area-weighted values and do not match the qualitative 'average size' determined from looking at the EBSD maps.

- For area-weighted geometric and arithmetic means, excluding grains < 10 pixels skews the mean values, whereas excluding grains < 2 pixels does not. Since the grain size distribution is very wide, single-pixel grains cannot be assumed to be misindexed points and should be included if an alternative method of filtering out misindexed points is available.

- Geometric mean values are always smaller than arithmetic mean values. The geometric mean is more suitable for describing the grain size distribution here because the distribution tails are assymetric and qualitatively fit a log-normal distribution better than a normal distribution.

- The area-weighted histogram mode and median values are least sensitive to choice to exclude or include small grains, which also means these values will be least sensitive to EBSD map step size and EBSD pattern indexing quality. These parameters also come with a natural uncertainty bound of half the histogram bin width. However, histogram modes are sensitive to bin width, which is an arbitrary operator choice and not as robust as using the cumulative histogram.

- The area-weighted medians of the min and max Feret diameters are most suitable as summary metrics. The area-weighted median is insensitive to both bin width and minimum grain size, and close to the area-weighted geometric mean values. Its value can be read off the cumulative histogram plots and correspond well to the approximate 'average size' from looking at the EBSD map plots. Both min and max Feret diameters should be reported to describe the anisotropy along and perpendicular to the sample build direction.

7.3.2.2 Size distribution plots

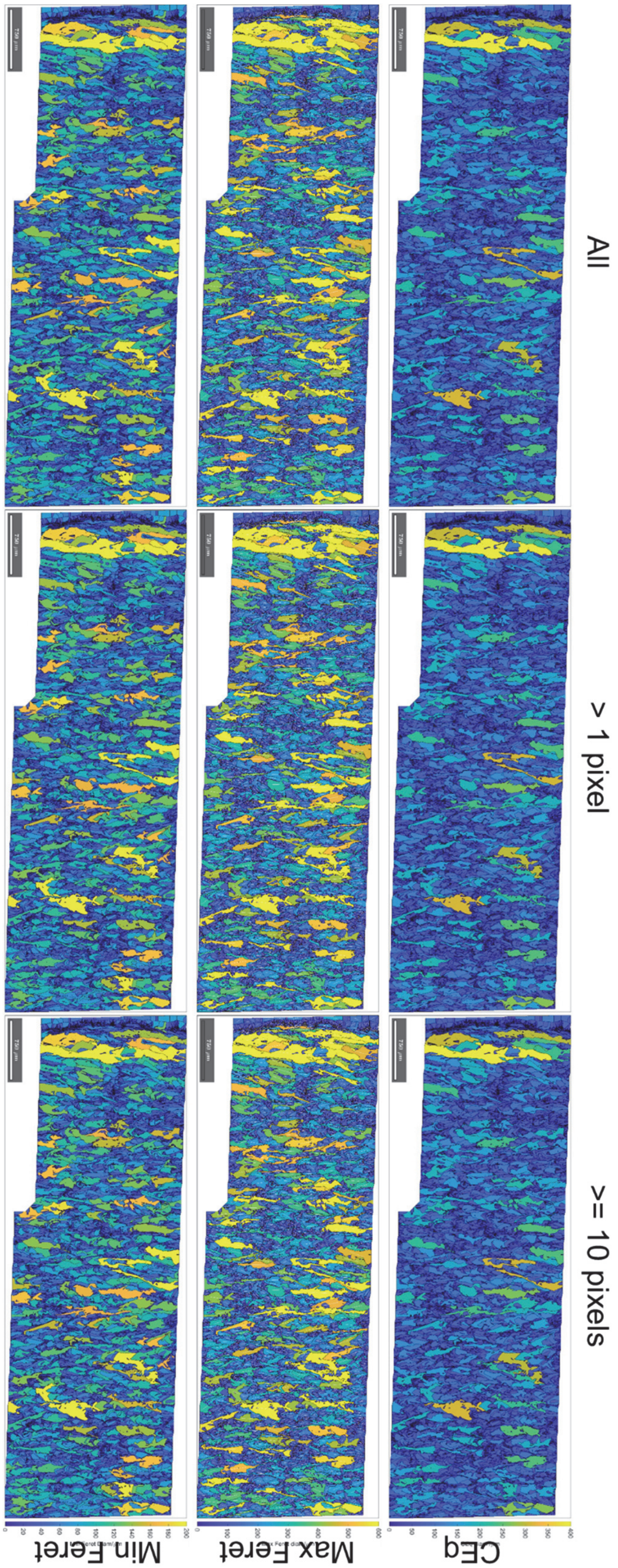


Figure 1: Grain size maps. White line annotations on the 'Max Feret' maps show the maximum Feret length direction.

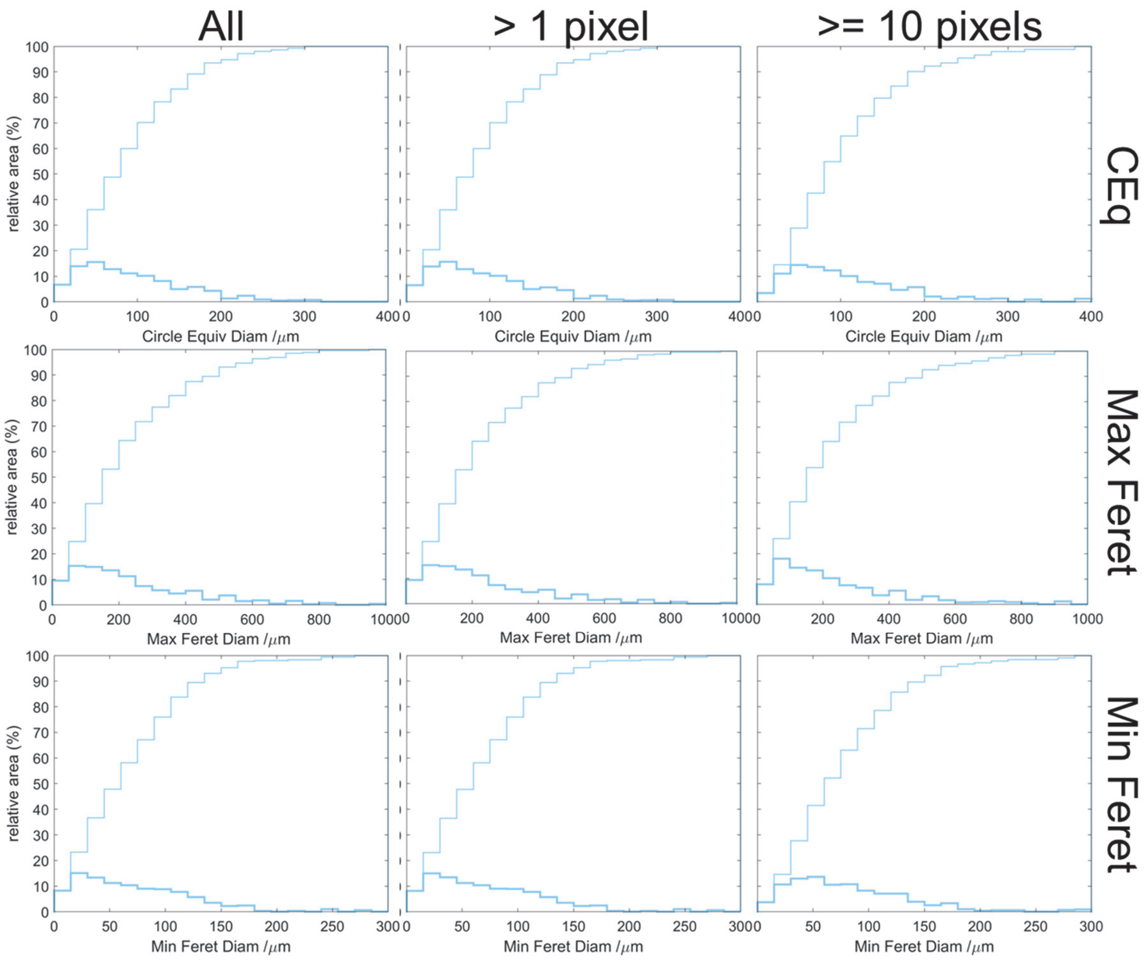


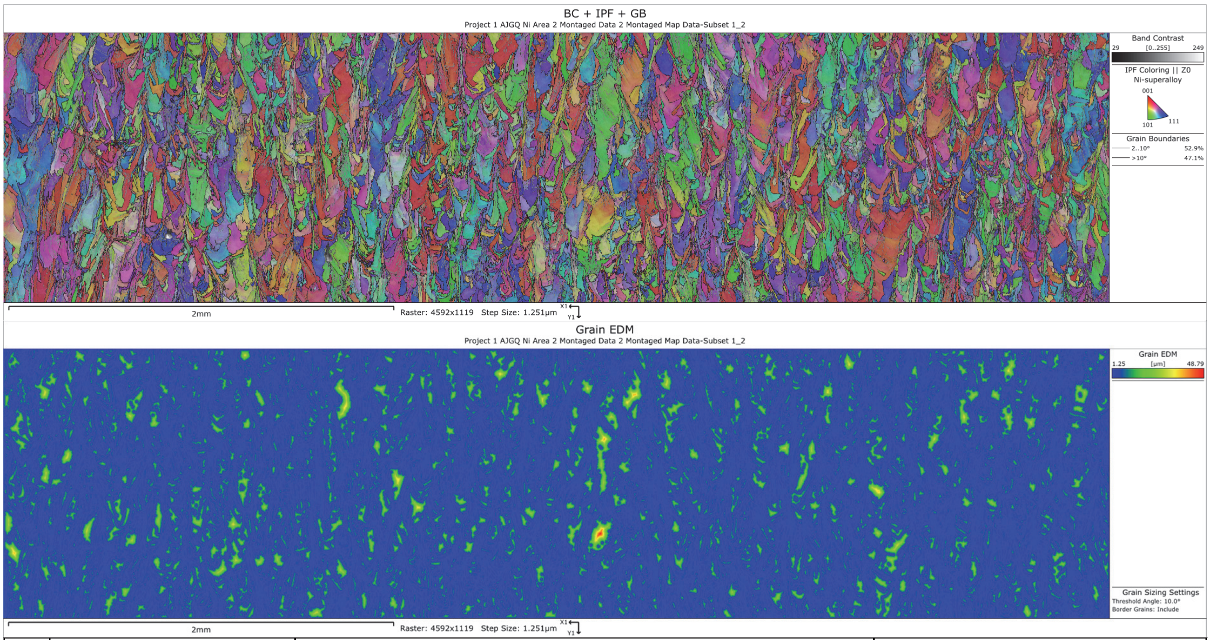
Figure 2: Grain size distribution plots

7.3.3 References

This standard may be helpful: ISO. (2020). *ISO 13067:2020 Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size*.
[Add any more if required / used in the previous sections]

7.4 P4_1

7.4.1 Analysis method raw data



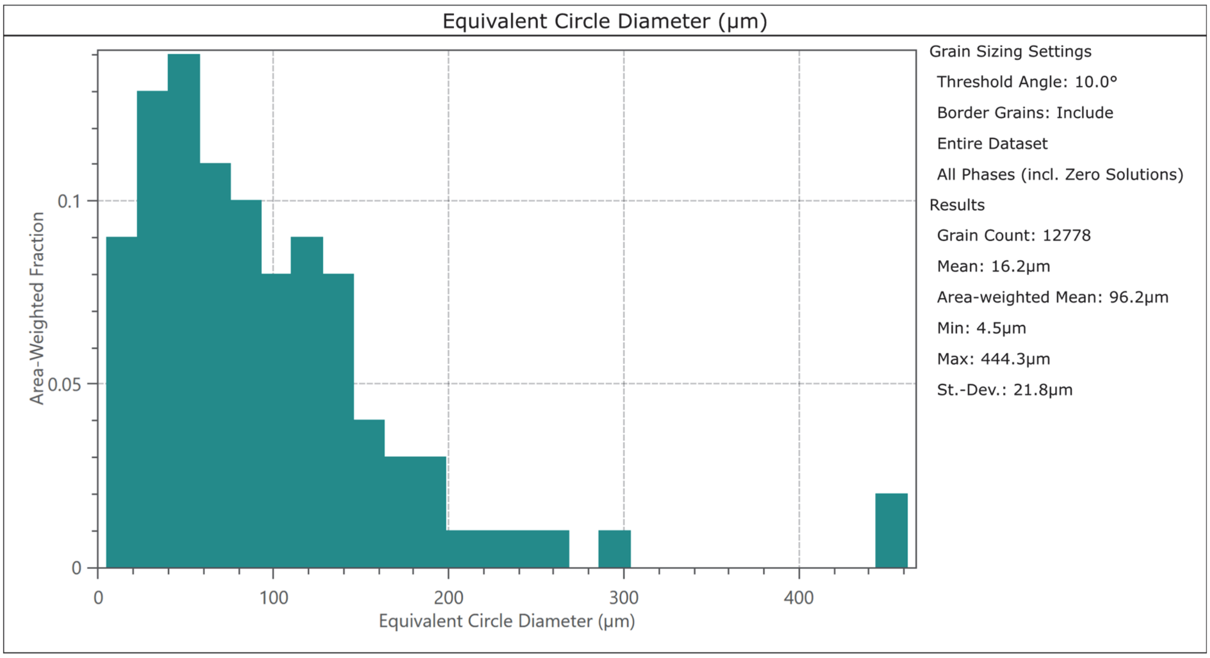
		Details	Notes / References
	Instrument	Zeiss Auriga 60 with an Oxford Symmetry 2 detector	
	Settings	Maps were acquired with 30 kV/ 10 nA, µm, individual maps 0.38x 0.29 mm in area, 2311 patterns/s with a step size of 1.25 µm	
1	**Analysis software**	[include version details e.g. MTEX # / MATLAB R#, OIM Analysis v#, AztecCrystal v#]	AztecCrystal 2.2.302
	Data cleaning method	[e.g. remove wild spike pixels, orientation smoothing, how unindexed points are handled, any cropping or subsets extracted]	
2	**Grain reconstruction method**	[method details, e.g. boundary misorientation threshold angle[s], orientation interpolation / data cleaning, how were incomplete boundaries handled]	All pixels in the subset Threshold 10°
3	**Map border grains**	[Included / excluded]	Included
4	**Minimum grain size**	[none / # pixels / other metric]	10 pixels
5	**Grain size metric(s)**	[e.g. intercept lengths, circle equivalent diameter, ellipse major/minor axes]	Circle equivalent diameter
6	**Size distribution summary metric(s)**	[e.g. arithmetic / geometric mean, median, mode(s). Include details about how you calculated this, e.g. histogram bin width, CDF]	Aritmetic mean

7	**Number of grains measured**	[#]	12778

7.4.2 Results Threshold 10°

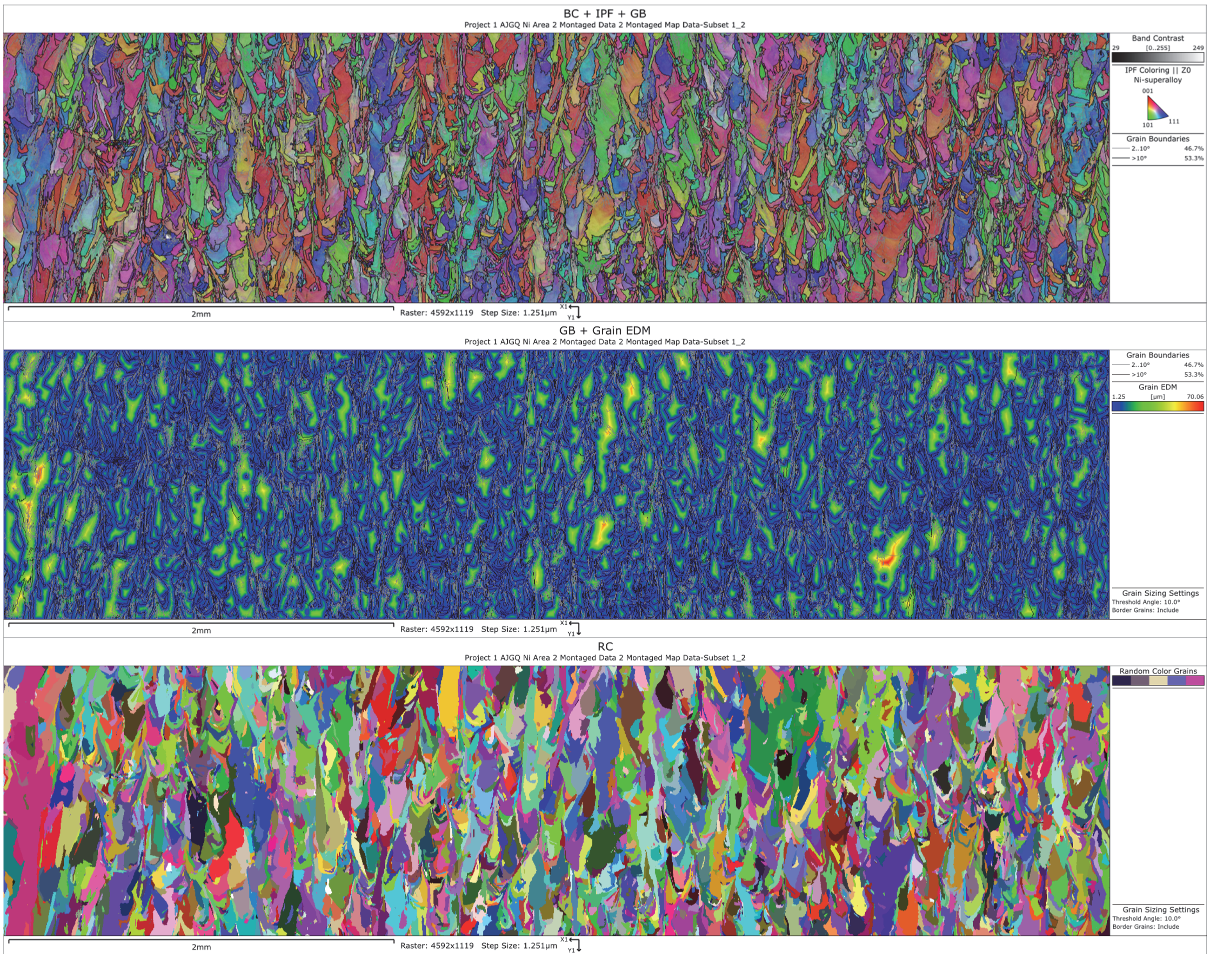
7.4.2.1 Summary statistics

	Grain size metric	Size distribution summary metric	Value	Unit	Additional notes
	Pixels count		5138488		
	Zero solution		469915		
1	Equivalent diameter	Arithmetic mean	16.2	µm	
2	Aspect ratio		3.52		
3	Minimum	diameter	4.5	µm	
4	Maximum	diameter	444.3	µm	
5	Standard deviation	Diameter	21.8	µm	



7.5 P4_2

7.5.1 Analysis method clean up



		Details	Notes / References
	Instrument	Zeiss Auriga 60 with an Oxford Symmetry 2 detector	
	Settings	Maps were acquired with 30 kV/ 10 nA, µm, individual maps 0.38x 0.29 mm in area, 2311 patterns/s with a step size of 1.25 µm	
1	**Analysis software**	[include version details e.g. MTEX # / MATLAB R#, OIM Analysis v#, AztecCrystal v#]	AztecCrystal 2.2.302
	Data cleaning method	[e.g. remove wild spike pixels, orientation smoothing, how unindexed points are handled, any cropping or subsets extracted]	• Auto clean up • Wide peaks remove • Zero solution removal 4 neighbourhood 10 iterations
2	**Grain reconstruction method**	[method details, e.g. boundary misorientation threshold angle[s], orientation interpolation / data cleaning, how were incomplete boundaries handled]	All pixels in the subset Threshold 10°

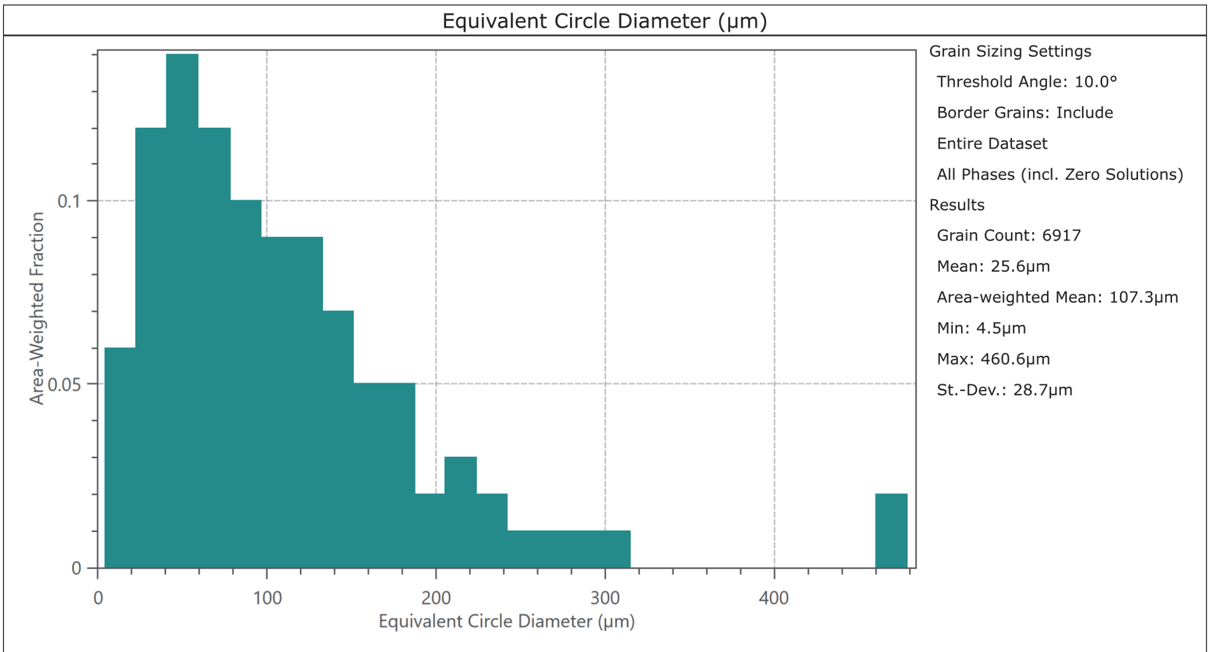
3	**Map border grains**	[Included / excluded]	Included
4	**Minimum grain size**	[none / # pixels / other metric]	10 pixels
5	**Grain size metric(s)**	[e.g. intercept lengths, circle equivalent diameter, ellipse major/minor axes]	Circle equivalent diameter
6	**Size distribution summary metric(s)**	[e.g. arithmetic / geometric mean, median, mode(s). Include details about how you calculated this, e.g. histogram bin width, CDF]	Aritmetic mean
7	**Number of grains measured**	[#]	6917

7.5.2 Results Threshold 10°

7.5.2.1 Summary statistics

	Grain size metric	**Size distribution summary metric**	**Value**	**Unit**	**Additional notes**
	Pixels count		5138488		
	Hit rate		99.45%		
1	Equivalent diameter	Arithmetic mean	25.6	µm	
2	Aspect ratio		3.53		
3	Minimum	diameter	4.5	µm	
4	Maximum	diameter	460.6	µm	
5	Standard deviation	Diameter	28.7	µm	

7.5.2.2 Size distribution plots



7.6 P4_3

7.6.1 Analysis method clean up

<same as Section 7.5.1>

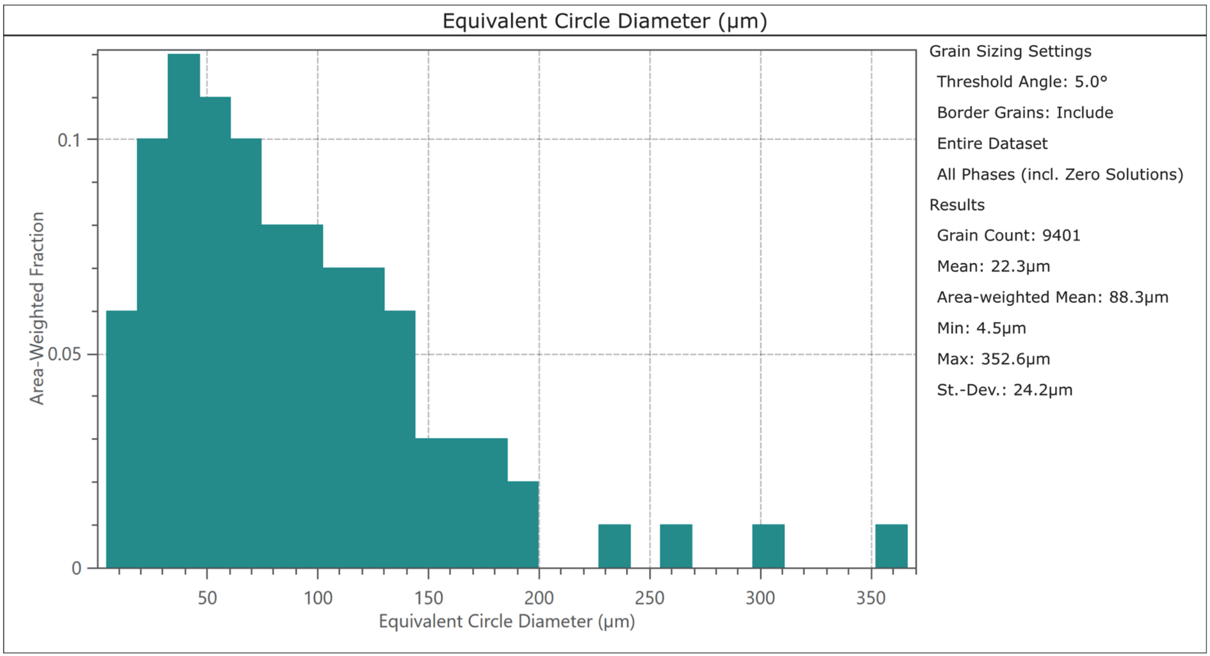
7.6.2 Results Threshold 5°

7.6.2.1 Summary statistics

	Grain size metric	Size distribution summary metric	Value	Unit	Additional notes
1	Equivalent diameter	Arithmetic mean	22.32	µm	
2	Aspect ratio		3.6		
3	Minimum	diameter	4.5	µm	
4	Maximum	diameter	352.6	µm	
5	Standard deviation	Diameter	24.2	µm	

[some kind of qualitative note to which metric(s) are more or less appropriate for this dataset]

7.6.2.2 Size distribution plots



[e.g. histogram or cumulative histogram plots corresponding to each line in the Summary statistics table]

7.6.3 References

This standard may be helpful: ISO. (2020). *ISO 13067:2020 Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size.*

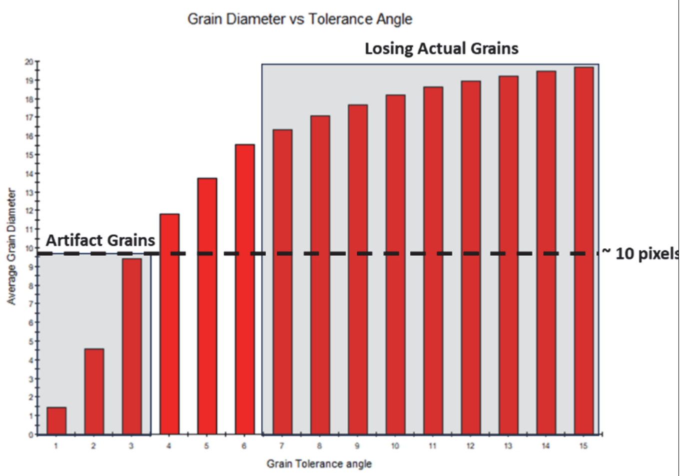
ASTM E2627-13(2019)

7.7 P5

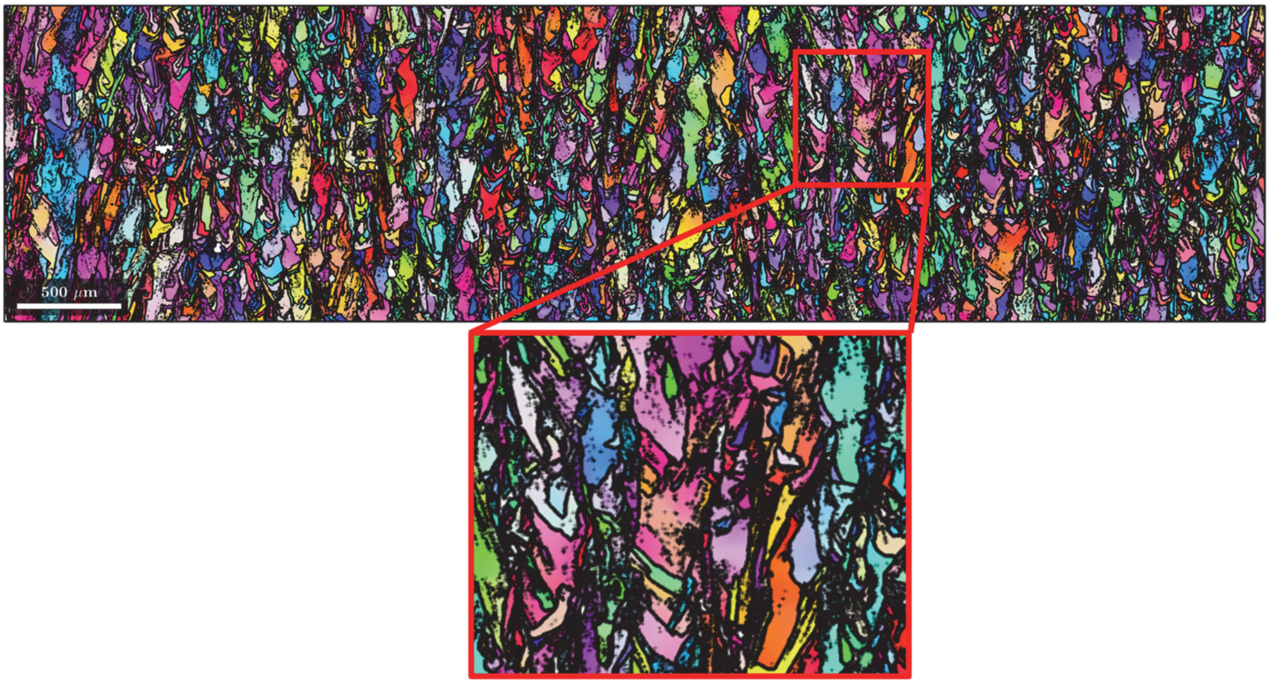
7.7.1 Analysis method

Please add additional rows to report any parameters that are not currently included.

		Details	Notes / References
1	**Analysis software**	OIM Version 9.0.0.208	Used .ang format provided in study (CI and IQ were not accurate from MTEX conversion)
	Data cleaning method	Removed any grains less than 10 pixels in size, cropped dataset to remove edge noise (~85% of total area still surveyed)	Standard processing route in OIM Grain reconstruction and data cleaning occurred together – see below for more
2	**Grain reconstruction method**	Cleanup: 1) Grain CI Standardization: 5 degree grain misorientation definition, 2 pixel minimum grain size, no multi-row requirement, 5 pixel anti-grain minimum size, multi-row requirement 2) Grain Dilation - 5 degree grain misorientation definition, 2 pixel minimum grain size, no multi-row requirement, 5 pixel anti-grain minimum size, multi-row requirement, Single iteration 3) Neighbor CI Correlation – Minimum CI of 1 (to combat artifacts from creating .ang in MTEX), Single iteration 4) Neighbor Orientation Correlation – Level 2 cleanup, 5 degree grain misorientation definition, Minimum CI of 1 (to combat artifacts from creating .ang in MTEX)	
3	**Map border grains**	Including map border grains	
4	**Minimum grain size**	10 pixels (see justification from data below)	Evaluated grain size versus grain definition angle to decide cut off

			
5	**Grain size metric(s)**	Diameter, aspect ratio, length of ellipse major axis, length of ellipse minor axis, grain ellipticity, grain circularity, maximum feret diameter, minimum feret diameter	
6	**Size distribution summary metric(s)**	Arithmetic mean for all calculated values once artifact grains have been removed	
7	**Number of grains measured**	9040	

Note: OIM was selected after using MTEX versions 5.8.2 and 5.10.2 for grain calculation in MATLAB R2023b. MTEX continued to create artifact grains within actual grains, often in interdendritic regions despite filtering out inclusions, smoothing, removal of small grains, etc. No obvious solution was found, hence the switch to OIM from MTEX. See the graphic below for an example where the actual grains are outlined, but so too are inclusion grains that add ~90000 grains to the dataset.



7.7.2 Results

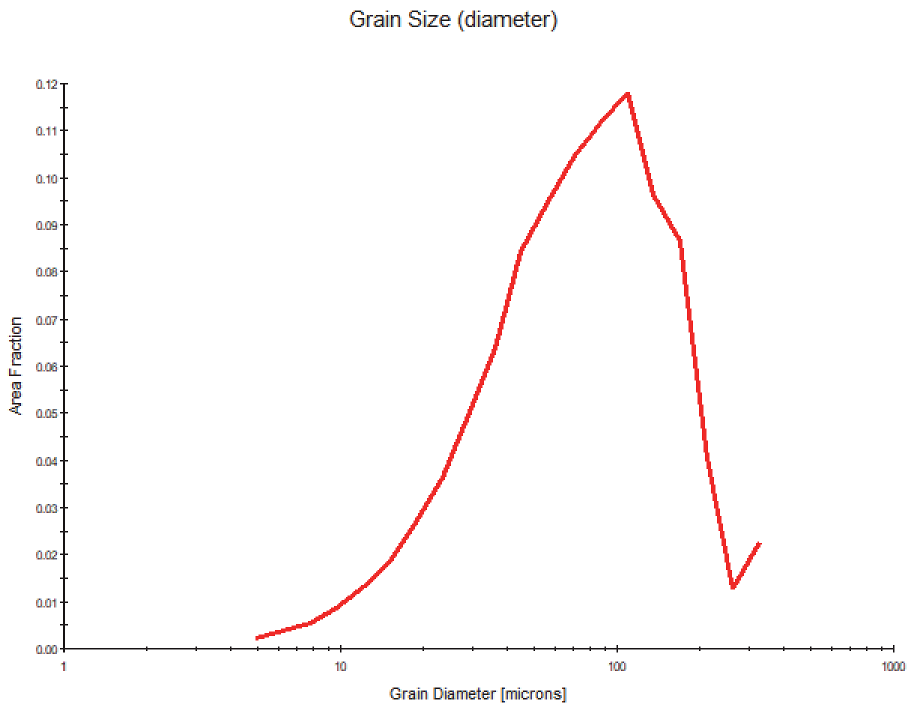
7.7.2.1 Summary statistics

	Grain size metric	**Size distribution summary metric**	**Value**	**Unit**	**Additional notes**
1	Diameter	Arithmetic mean	22.12	um	As-calculated by OIM
2	Area	Arithmetic mean	861.72	um^2	
3	Aspect ratio of ellipse fit to grain	Arithmetic mean	0.32	Unitless	Qualifier of if diameter is accurate
4	Length of major axis of ellipse fit to grains	Arithmetic mean	24.39	um	
5	Length of minor axis of ellipse fit to grains	Arithmetic mean	6	um	Describes elongation of grains when compared to longest axis
6	Grain ellipticity	Arithmetic mean	0.94	Unitless	Confirms elongation of most grains
7	Grain circularity	Arithmetic mean	0.33	Unitless	Confirms elongation of most grains
8	Maximum feret diameter	Arithmetic mean	48.77	um	More accurate representation of grain size with elongated character
9	Minimum feret diameter	Arithmetic mean	18.08	um	Confirms diameter is inaccurate, and that max feret diameter is more representative

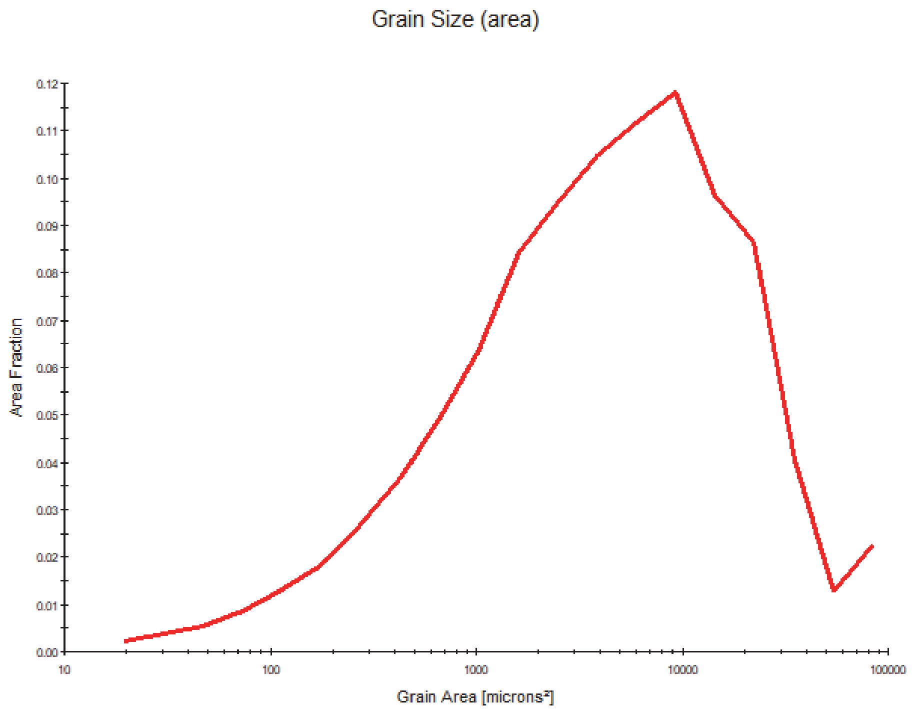
As most datapoints show an elliptic grain character for the dataset, I recommend the use of area for measuring the domain size of each grain, and the maximum feret diameter for “grain size” in Hall-Petch and similar calculations.

7.7.2.2 Size distribution plots

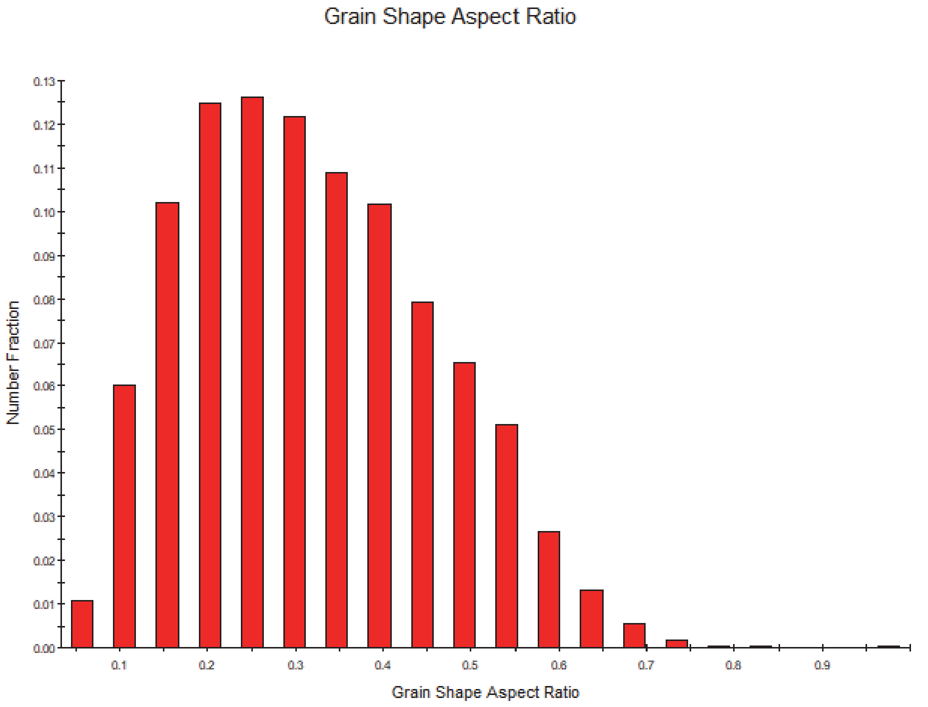
Diameter



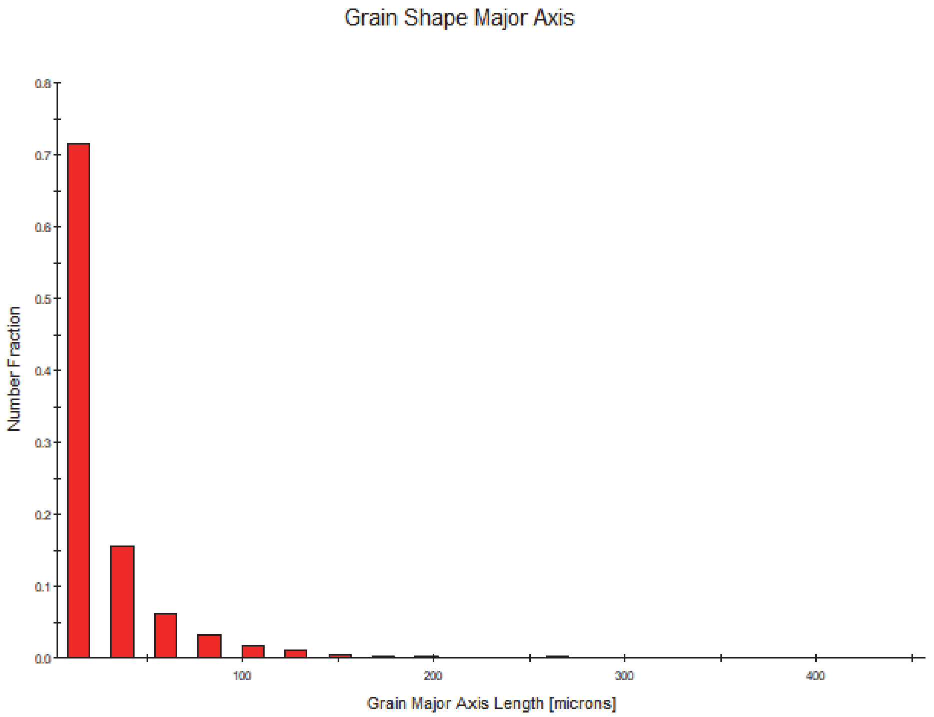
Area



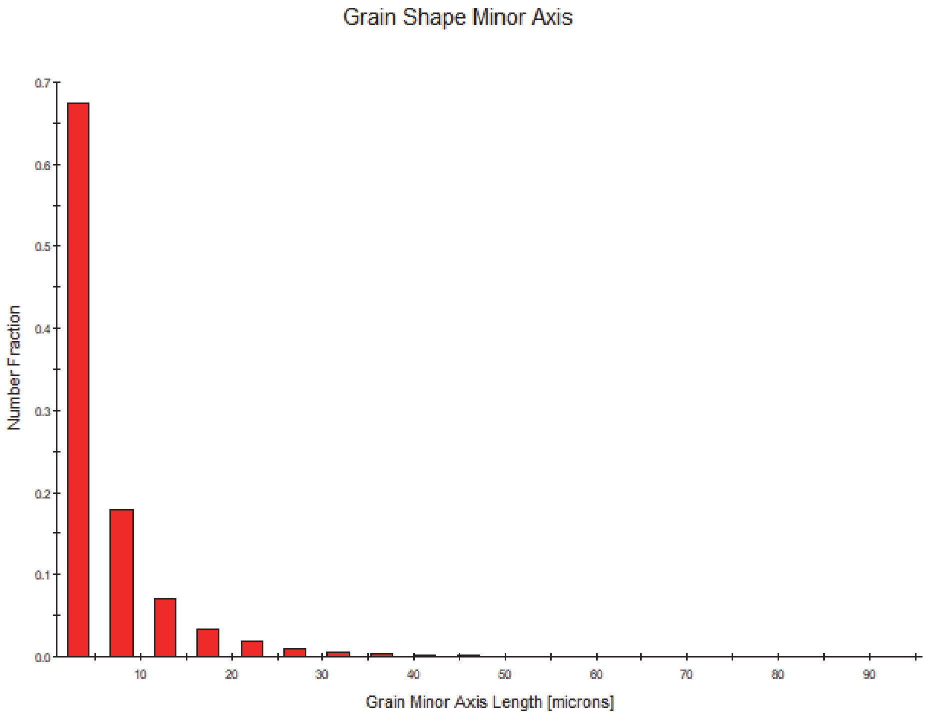
Aspect ratio of ellipse fit to grain



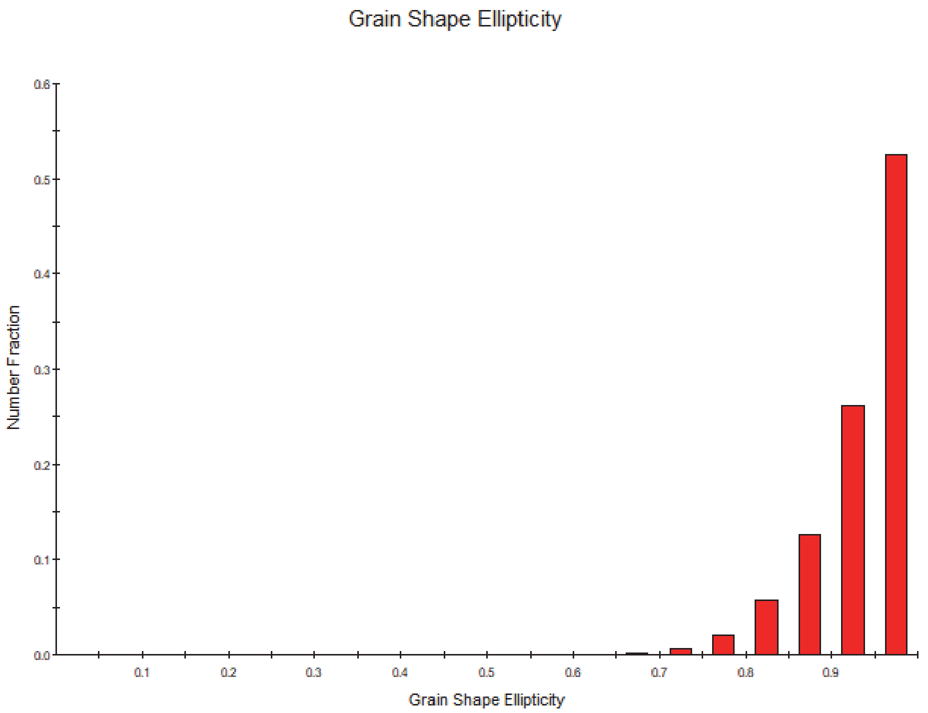
Length of major axis of ellipse fit to grains



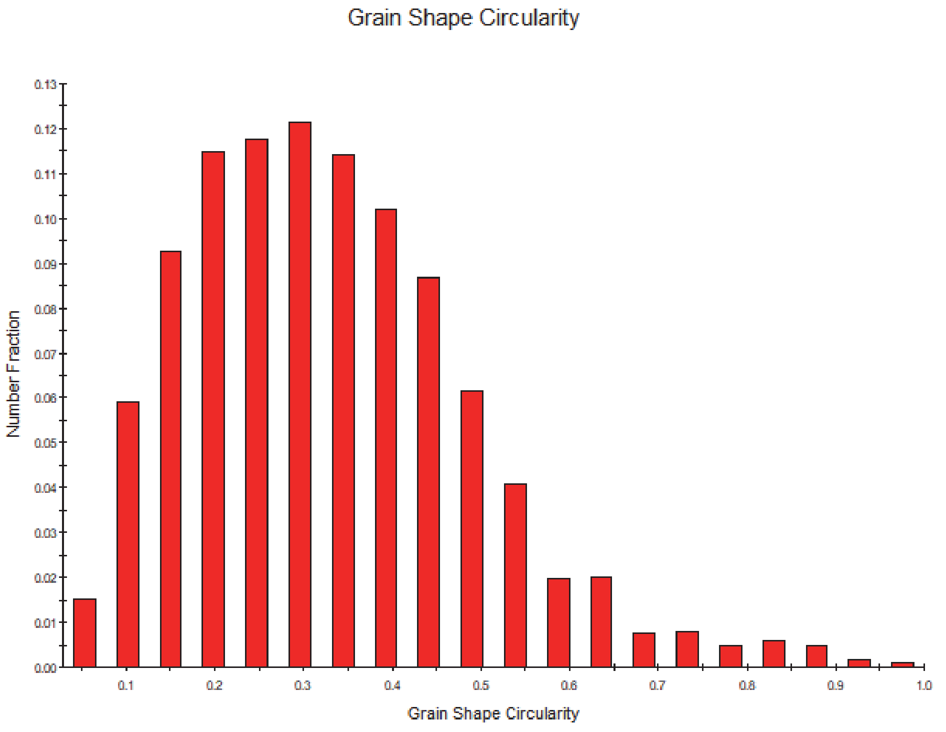
Length of minor axis of ellipse fit to grains



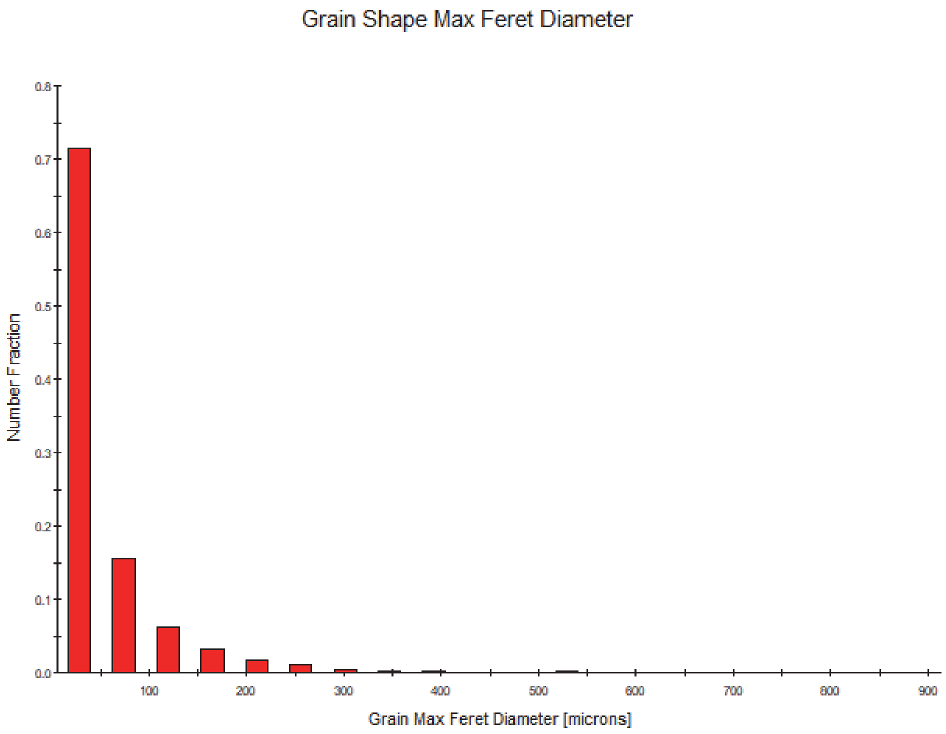
Grain ellipticity



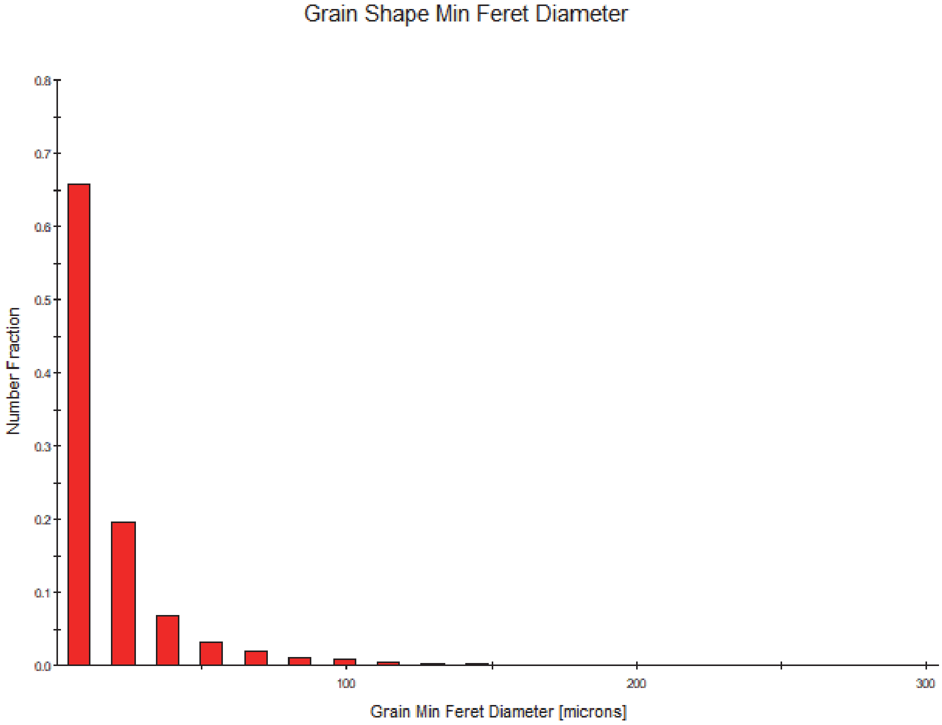
Grain circularity



Maximum feret diameter



Minimum feret diameter



7.7.3 References

This standard may be helpful: ISO. (2020). *ISO 13067:2020 Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size*.