Characterization of average grain size and grain size distribution

Summary

The importance of grain size to material properties is highlighted, in particular the influence of grain size distribution for heterogeneous microstructures. At present there are no established methods for the characterization of heterogeneous microstructures, and thus the suitability of existing methods is investigated. It is shown that existing methods can be applied when the measurement data is analyzed in a meaningful way. Furthermore, new methods for grain size characterization are shown to have linear correlation with existing methods, thus allowing for ‘backwards compatibility’. In addition, the functionality of the Matlab codes is explained and recommendations are given for use of different grain size parameters.

Theoretical background

In engineering the mechanical properties of a material are one of the key features to be. Furthermore, understanding the relationships between microstructural quantities and material properties is needed in order to utilize the materials in the best way possible.

A fundamental characteristic measure for microstructures of metallic materials is the average grain size. It is known based on the work of Hall [2] and Petch [3] that various material properties, such as hardness, stress-strain properties and fatigue [4–10] scale with the average grain size. This information can also be used to avoid fatigue failures of a component during its lifetime [11,12]. The microstructural understanding is also important in the development of new materials [13].

The relationship between average grain size and yield strength is formulated as:

\[ \sigma = \sigma_0 + k d^{-1/2} \]

where \( \sigma_0 \) is the lattice friction stress required to move individual dislocations, \( k \) is a material-dependent constant known as the Hall-Petch slope, and \( d \) is the average grain size [14]. As the Hall-Petch relationship is related to the measure of grain size, the correct definition is crucial. Typically the average grain size is used to describe the microstructure [15], but this is not a suitable measure for heterogeneous microstructures. Several investigations [14–20] have shown that the grain size distribution has an effect on the mechanical properties. For example, Berbenni et al. [19] showed that for a given average grain size, broadening of the grain size dispersion reduces the strength of the material.

Based on the abovementioned observations it can be concluded that the average grain size does not adequately represent the physical response of heterogeneous microstructures. In a microstructure, the largest grains can be associated with low strength due to the length of the slip bands, causing them to yield first; see e.g. [21,22]. Furthermore, even a low number of large grains can occupy a significant material volume. To capture the influence of grain volume, a rule of mixtures approach is proposed for heterogeneous microstructures. The contribution of each grain to the strength of the material is considered to be proportional to the volume of the grain; see e.g. [16,23]. Thus, the volume-weighted average grain size is defined as:
where $V_r$ is the total volume of material and $V_i$ the volume of grains corresponding to the grain size $d_i$. Due to the definition of the volume-weighted average grain size, it is always larger than the average grain size. The results presented in [1] indicate that the volume-weighted average grain size is able to capture the influence of grain size distribution.

Grain size measurement (average grain size)

The average grain size, as used in Eq. (1), is often measured using the linear intercept method. As per the recommendation of ASTM E1382 [24], four evenly spaced directions (0°, 45°, 90°, 135°) should be used. The MATLAB code measures these four directions, and considers measurements smaller than three pixels as noise and thus excludes them; see Figure 2. Results from each measurement direction are saved into their own variables, named `grain size_hor, _ver, _45_1, _45_2`. All measurement directions are combined into a single dataset in variable `grainsize`.

\[
\Delta d = \frac{d_{\text{max}} - d_{\text{min}}}{d} = \frac{P_{99.5\%} - P_{0.5\%}}{d}, \tag{3}
\]

where the maximum and minimum grain sizes are taken as the 99% and 1% probability level grain sizes. This is done to minimise measurement uncertainty, which is inherently at its largest at the extremities of the distribution due to the finite number of measurements. The relative grain size dispersion characterises the spread of grain size, and is typically within the range of 2.5…6. Small values are seen for annealed base materials, while multiphase and welded microstructures are in the upper range. The grain size at probability levels 1-99.9% is saved into the variable `grainsize_probability_1`.

In addition to outputting the average grain size, a relative grain size dispersion value, $\Delta d/d$, is determined from the measured distribution. This value is defined as, being modified from the work of Berbenni et al. [19]:

\[
\frac{\Delta d}{d} = \frac{d_{\text{max}} - d_{\text{min}}}{d} = \frac{P_{99.5\%} - P_{0.5\%}}{d}, \tag{3}
\]

For more detailed analysis of the results, the use of distribution fitting tool (`dfittool`) of Matlab is advised. Example density and probability plots for the provided `example_EBSD_1.png` microstructure are shown in Figure 3.
Grain size measurement (volume-weighted average grain size)

Obtaining true three-dimensional information of a microstructure is very labour-intensive and has traditionally been done by means of serial sectioning. For this reason, it is common practice to perform three-dimensional estimations from two-dimensional sections. Stereology is the field concerned with indirect methods for estimating three-dimensional features from two-dimensional sections.

The volume-weighted average grain size is obtained by weighting each measurement with the corresponding grain volume, as defined in Eq. (2). This presents the problem of defining the grain volume for each measurement. Although general formulations have been derived for the correlation between intercept length and grain volume [25], it is difficult to determine the accuracy and reliability of such an approach. Particularly so as several different intercept lengths can be measured within a single grain.

The MATLAB code for measurement of the volume-weighted average grain size is based on the point-sampled intercept length method defined by Gundersen and Jensen [25,26]. Similar to the ASTM E1382 procedure, intercepts are measured in four evenly spaced directions. However, the measurements are carried out at random points, and the direction of the intercept chosen randomly. Consequently, different grain sizes are measured proportionally to their surface area fraction. Based on relationships of stereology [27,28] the surface area fraction can be used to estimate the volume fraction; see Appendix B in Ref. [1] for further details. Thus, the average value of the distribution can considered as the volume-weighted average grain size, \( d_v \), as defined in Eq. (2). An example of the measurement is shown in Figure 4. The output of the Matlab code is similar to the ASTM E1382 method, and grainsize variables for each measurement direction are named identically.
Correlation of grain size parameters

Comparison of the data between the two methods reveals that a correlation can be found between average grain size \( (d) \), volume-weighted average grain size \( (d_v) \), and the relative grain size dispersion \( (d/d) \): see Figure 5. The first and last parameter are obtained with the traditional ASTM E1382 linear intercept method, while the volume-weighted average grain size is measured with the point-sampled intercept length method. Thus, existing data can be re-analysed by determining the relative grain size dispersion, for example by utilising Matlab commands:

\[
\begin{align*}
  d_{\text{min}} &= \text{quantile(grainsize,0.01)} \\
  d_{\text{max}} &= \text{quantile(grainsize,0.99)}
\end{align*}
\]

This enables the volume-weighted average grain size to be estimated with the relationship:

\[
\frac{d_v^{-1/2}}{d^{-1/2}} = -0.0635 \frac{\Delta d}{d} + 1.0059
\]

The constant of the linear fit is rounded to value 1.0, and thus

\[
\frac{d_v^{-1/2}}{d^{-1/2}} = d^{-1/2} \left(1 - 0.0635 \frac{\Delta d}{d}\right).
\]

The expression is mathematically consistent, as the two grain size parameters are the same in the theoretical case that all grains (measurements) are the same, i.e. \( d = d_v \).
Use of Matlab implementation

The matlab codes of the two methods are available as an attachment, the current version is 1b: Public_matlab_code_v1b.zip. The compressed zip-file includes the following files:

- example_EBSD_1.png: EBSD grain boundary map that is suitable for use with the code, scale of the image is 10 pixels / µm
- example_optical_1.png: A processed optical micrograph that is suitable for use with the code, scale of the image is 2.72 pixels /µm
- example_optical_original.jpg: The original of the file mentioned above
- linesampled_intercept_length_v1a.m: The implementation of the ASTM E1382 linear intercept method, from now onwards referred to as the line-sampled method
- pointsampled_intercept_length_v1a.m: The implementation of the point-sampled linear intercept method, from now onwards referred to as the point-sampled method.

The Matlab code is provided under the GNU General Public License v3 (http://www.gnu.org/licenses/). The code is provided as is and can be freely modified. The version represents the code used in Ref. [1]. The code requires the Matlab 'Image Processing Toolbox' and 'Statistics Toolbox' to run. The code is thoroughly commented to explain the functionality and different parameters.

Should you find the code as a worthwhile tool and use it for research to be published, please kindly cite the original article [1] and/or this website (https://wiki.i.aalto.fi/display/GSMUM) so others could benefit from it as well, thank you!

If you have questions about the code, you can leave a comment on this page or email me at Pauli (dot) Lehto (at) aalto (dot) fi.

Input data

As shown by the example figures, the expected image data is in black (grain interiors) and white (grain boundaries). The image can be in binary or RGB format, a binary conversion is always carried out in the code when the images are saved into variables. For example the EBSD grain boundary maps generated by Channel 5 are suitable as is, after they have been inverted in color. Note: Do not use image processing software to thin the grain boundaries (skeletonize), i.e. make them a single pixel in thickness. This results in non-continuous grain boundaries, which will result in bleeding measurements in the 45 degree measurement directions. Always check the output figures to make sure the grain boundaries have been detected correctly.

The files are to be named using the logic filename_1.png, filename_2.png, etc. Should you want to use a different file type, you can change it in the code: imdata = imread(fullfile(num2str(o) '.png')). The number of images to be analysed is set in n_fields. Set the scale according to image magnification /EBSD parameters. For example if the EBSD scan was done with a step size of 0.1 µm or 0.2 µm, then the scale is 10 (pixels/µm) or 5 (pixels/µm), respectively.
Optical image pre-processing

For optical images pre-processing is required to generate the grain boundary maps. In many cases this requires manually tracing/enhancing the grain boundaries. The freely available ImageJ software is very useful for carrying out binary image operations. An example of the image processing steps is shown in Figure 6. Note that the colors in the analysis figures have been inverted to match the original micrograph.

Figure 6. Image processing steps for the measurement of the volume-weighted average grain size.

Linear intercept parameters

The parameters that need to be set are the spacing of test-lines for the line-sampled method, or the number of measurement points for the point-sampled method. Aim to have at least 5 000 measurements per image, preferably over 20 000 for accurate distribution analysis. For the line-sampled method the spacing is set separately for horizontal/vertical & 45 degree directions. 45 degree line spacing should be vertical/0.7, so that equal line length is measured for all directions (sin 45°=0.707 defines the perpendicular distance between measurement lines). Analysis about number of measurements and convergence is presented in Appendix C of Ref. [1].

The code also offers low- and high pass filtering. By default measurements smaller than 3 pixels are eliminated (noise), but this provides an additional level of filtering to, for example, remove bleeding large measurements from the distributions. Usually these are seen as a discontinuity in the large grain sizes in the probability plot. However, it should be noted that a few erroneous measurements have a very small influence on the average grain size or the relative grain size dispersion. Default is not to use the filtering, and the better measure for bleeding grain boundaries is to improve grain boundary delineation manually (especially optical micrographs).

Recommended use of grain size parameters

It is recommended that the measurement results of both methods are reported in scientific research. The average grain size, $d$, provides comparability to previously published research. When the relative grain size dispersion, $d \sigma$, is reported in conjunction with the average grain size, it gives a good representation of the grain size distribution with just two values. The volume-weighted average grain size, $d_v$, accounts for the differences in grain size dispersion and thus just gives an ‘effective’ grain size value that correlates more accurately with the mechanical properties.

The values of $d \sigma$ and $d_v$ correlate well with how the grain size and the grain size distribution look with visual observation. Figure 7 has an example that compares the grain size distributions and the measured parameters for two weld metals. As measured by the line-sampled method they both have the same average grain size ($d$), although visually WM2 seems larger and has a wider spread in grain size. This is reflected by the relative grain size dispersion value ($d \sigma$) and volume-weighted average grain size $d_v$, which are noticeably larger for WM2.

It is noted that the $d_{\text{min}}=1\%$ grain size is slightly resolution limited in these cases, however, its influence on the relative grain size dispersion is very small, approximately 0.1 µm. In an EBSD scan a step size equal to or smaller than 1/10th of the average grain size ($d$) is usually sufficient. For more information about the choice of step size refer to Mingard et al. [29,30].
Figure 7. Comparison of grain size parameters for two weld metals that have the same average grain size \( \bar{d} \). The log-normal probability plot shows data measured with the line-sampled method. (click to enlarge)

References


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