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Quantitative Characterization of Boundary Roughness in Metals

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DTU Wind Energy PhD-0066
August 2016
Abstract:

The boundary migration during recrystallization is by nature a heterogeneous process and local structural variations form on recrystallization boundaries, as revealed from modern techniques such as synchrotron X-rays and advanced electron microscopy. The local structural variations, in the form of protrusions and retrusions, can provide a dragging/driving force due to the local boundary curvature and affect the further migration of recrystallization boundaries through the deformed matrix. In order to develop new understandings and models for boundary migration that take the heterogeneous local structural aspects into account, a detailed characterization is essential of partly recrystallized microstructures focusing on the local shapes of the boundaries, in particular on whether protrusions and retrusions are formed or not. Quantification of the “amount” of boundary roughness in the form of protrusions and retrusions is of importance for statistical investigations into the factors that potentially influence the recrystallization boundary roughening.

A method is developed for quantitative characterization of 2-D line features. The area integral invariant (AII) is employed as a morphological variable to obtain information of local structural variations such as protrusions and retrusions formed on recrystallization boundaries. The AII value is direction-independent allowing unbiased characterization of morphological irregularities with both closed and non-closed boundary profiles. The length scale at which the rough features are characterized is determined by a parameter termed sampling radius used to measure the AII values. A number of roughness parameters are developed based on the AII dataset for a boundary or boundary segment, whose local morphological characteristics are represented by individual AII value acquired along the boundary or boundary segment.

With the quantified boundary roughness at two length scales: 1 µm and 3 µm, the roughening behaviors of a large number of recrystallization boundaries are statistically analyzed and the effects of several parameters: materials purity, deformation strain, annealing temperature and boundary alignment direction, are evaluated. It is revealed that recrystallization boundaries in general are rough and the roughening behaviors of recrystallization boundaries are affected by the investigated parameters, more significantly at the length scale of 1 µm. It is found that the higher roughness is often associated with the higher migrating rates of recrystallization boundaries.

A new method is presented to quantitatively characterize the morphology of graphite nodules in cast iron, as an extended application of the AII method to characterize the 2-D line features. This method develops a morphological variable “dispersion” to obtain information about local morphological characteristics that is subsequently merged into a parameter termed dispersion index, to represent the nodule’s morphology as a whole. The potential of the method is validated by quantifying the morphology of graphite nodules with complicated shape and by measuring the nodularity of an image with many graphite nodules.
Quantitative Characterization of Boundary Roughness in Metals

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August 2016
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Preface

This thesis is submitted in partial fulfillment of the requirements for the PhD degree at the Technical University of Denmark. The project was carried out within the Danish-Chinese Center for Nanometals, under the supervision of Drs. Dorte Juul Jensen and Yubin Zhang. The study was conducted during the period from September 2013 to August 2016.

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Publications


Chapter 1
Introduction

The mechanical properties of metals, including hardness, strength, formability, fracture toughness etc. are strongly dependent on the thermomechanical processing. During recrystallization of deformed metals, new almost perfect nuclei form in the deformed matrix and grow by boundary migration until the deformed matrix is replaced by recrystallized grains. Traditionally, the boundary migration is considered to be a homogeneous process with the boundaries between recrystallized grains and deformed matrix moving at a constant rate and being smooth with only relative small boundary curvatures. Modern techniques including synchrotron X-rays and advanced electron microscopy have however revealed the heterogeneous nature of boundary migration and the importance of local structural variation formed on recrystallization boundaries.

In order to develop new understandings and models for boundary migration that take the heterogeneous local structural aspects into account, a detailed characterization is essential of partly recrystallized microstructures focusing on the local shapes of the boundaries, in particular on whether protrusions and retrusions are formed or not. Quantification of the “amount” of boundary roughness in the form of protrusions and retrusions is of importance for statistical
investigations into the factors that potentially influence the recrystallization boundary roughening.

One of the main objectives of this study is to develop methods for quantitative characterization of irregular recrystallization boundaries, providing information on both local structural variations and global boundary roughness. The second objective is to statistically investigate the roughening behaviors of recrystallization boundaries using the quantified boundary roughness, and to analyze the effects of various parameters on recrystallization boundary roughness.

The thesis is structured as following: Chapter 2 provides the necessary background knowledge for the present work; Chapter 3 describes the methods developed to quantitatively characterize the rough morphologies of 2D line features; Chapter 4 gives the experimental details for the materials science part of this study; Chapter 5 reports the investigation of the effects of parameters such as materials purity, annealing temperatures and deformation strain on recrystallization boundary roughness; Chapter 6 presents the quantitative characterization of irregular graphite nodules in cast iron, to illustrate the wider potentials of the developed roughness quantification method. Finally, Chapter 7 gives conclusions and outlook.
Chapter 2
Background

This chapter provides the background for this thesis. First the concepts of recrystallization and related microstructural evolution processes are introduced. The migration of grain boundaries and their roughening behaviors during recrystallization are explained in detail. Potential factors causing the recrystallization boundary roughness are reviewed. Such background information is necessary since it provides the motivation and problems to be addressed for the present research.

2.1 Recrystallization

As substantial amount of stored energy is introduced into metals during plastic deformation, deformed metals are therefore in a thermodynamically unstable state. If the deformed metals are subsequently annealed at elevated temperatures, recrystallization typically occurs, leading to significant changes in both the metals’ microstructures and mechanical properties including hardness, strength, ductility, fracture toughness, etc. The conventional definition of recrystallization is the formation of new grains in a deformed material by nucleation and migration of high angle boundaries, driven by the stored energy within the deformation microstructure [1]. Recrystallization is generally separated
into two stages: nucleation and growth. A schematic illustration of the recrystallization process is shown in Figure 2.1. During the nucleation stage, nearly defect-free nuclei form in the deformed matrix (Figure 2.1b). The nuclei subsequently grow into the deformed matrix (Figure 2.1c) and the recrystallization is completed when the entire deformed matrix is replaced by recrystallized grains (Figure 2.1d).

Figure 2.1 - Schematic illustration of recrystallization process in lightly deformed metals: (a) a sketch of a simplified deformed matrix; (b) defect-free nuclei form in the deformed microstructure; (c) partly recrystallized microstructure: nuclei grow by grain boundary migration and (d) fully recrystallized microstructure—the entire deformed matrix is replaced by recrystallized grains.
Recrystallization was considered to be a homogeneous process in early studies. For example, in the classic Johnson-Mehl-Avrami-Kolmogorov (JMAK) model [2,3,4,5,6], recrystallization kinetics was described using the JMAK equation:

\[ V_v = 1 - \exp(-Bt^n) \] \hspace{2cm} \text{Equation 2.1}

where \( V_v \) is the recrystallized volume fraction, \( t \) is annealing time, \( B \) is a coefficient depending on the nucleation rate and growth rate of recrystallized grains and the exponent \( n \) is related with the time-dependence of nucleation rate, growth rate and the growth dimensionality. The idealized JMAK model assumes that the nuclei are randomly distributed and that all recrystallizing grains grow at constant rate. However, most experimental measurements of recrystallization kinetics cannot be described by the JMAK model [7]. One of the reasons is the non-random distribution of nucleation sites. During recrystallization, the nuclei may form in clusters at preferential sites such as triple junctions, original grain boundaries and second-phase particles. Another reason is that the assumption of constant growth rate for all recrystallizing grains is not fulfilled. Usually the measured growth rates of recrystallizing grains decrease dramatically with time [8,9], which may be related with heterogeneous distribution of stored energy, as recrystallization occurs firstly at regions with high stored energy [10]. Recrystallizing grains with different crystallographic orientations may also have different growth rates, e.g., recrystallizing grains of cube orientation grow faster than those with other orientations [8,11]. It is now generally recognized that recrystallization is not a homogeneous process but varies locally, affected by many factors such as heterogeneous deformation microstructures, second phase
particles and the properties of the migrating grain boundaries. Hence, detailed investigations of local recrystallization behaviors are of significant importance to further the understandings of recrystallization.

2.2 Boundary migration during recrystallization

During recrystallization, the nuclei grow by boundary migration through the deformed matrix. The boundary migration rate is usually expressed as:

\[ v = M \cdot F \]  

Equation 2.2

where \( M \) is the boundary mobility and \( F \) is the driving force for migration. For recrystallization, \( F \) is typically considered to be the stored energy in the deformed matrix. In traditional studies on recrystallization, it is generally assumed that the recrystallization boundaries move smoothly following Equation 2.2. Most of the experimental evidences in traditional studies quantifying the migration rate of recrystallizing boundaries are, however, based on static, 2D stereological characterizations and therefore only revealing the average behaviors of recrystallizing grains on global scales [9,11,12].

In the last two decades, advanced experimental techniques have been developed, such as in-situ measurements using electron backscattered diffraction (EBSD) techniques and 3-dimensional X-ray diffraction (3DXRD). These modern characterization techniques allow detailed investigations into the growth of individual recrystallizing grain [13,14,15] as well as local boundary migration behaviors [16,17,18]. For example, using synchrotron X-ray radiation to illuminate the recrystallizing grain in the bulk of deformed samples, in-situ
observation of the grain’s growth in 3D is possible. Figure 2.2 shows four snapshots of a recrystallizing grain growing into a slightly deformed (30% thickness reduction by cold rolling) aluminum single crystal [14]. It can be seen that at various growth stages, the recrystallizing grain has an irregular morphology, with local structural variations as well as planar facets on the boundary. The experimental evidences in 4D (x, y, z and time) have thus revealed that, in contrary to the classical studies, the boundary migration during recrystallization is non-homogeneous both spatially and temporally, with boundary segments moving forward for a period of time, then stopping, i.e. stop-go type of motion, and often with large protrusions and retrusions forming locally on the migrating boundaries [19,20]. The terms protrusions and retrusions refer to boundary segments that are bulged outwards and dented inwards with respect to the neighboring boundary segments, respectively [18].

Figure 2.2 - Snapshots a recrystallizing aluminum grain at 4 different time steps during its growth into a slight deformed single crystal. The colored dots and arrows show the formation and migration of a boundary facet. The length scale is given by the tripod showing in the frame, with the legs 42 µm in length. The figure is reproduced from reference [14].
When the recrystallization boundaries are examined on 2D sections statically by e.g. EBSD, electron channeling contrast (ECC) imaging or optical microscopy, they are often observed to be rough \([17,21,22]\). An example of a rough recrystallization boundary in a partly recrystallized microstructure is shown in Figure 2.3: protrusions and retrusions (as marked by the write arrows in Figure 2.3) form on the boundary with varying sizes and shapes.

Examination of partly recrystallized microstructure from 2D images has shown that, small protrusions/retrusions sized about 1 µm are observed as ripples on the boundary and larger protrusions/retrusions with sizes in the order of tens of micrometers can also form. Generally, the shapes of larger protrusions may be classified into two types: saw-tooth shaped and rounded, as schematically shown in Figure 2.4 [23]. In the case of saw-tooth shaped protrusion, one of the straight sides is along an extended dislocation boundary in the deformed matrix, indicating a possible relation between the recrystallization boundary and the extended dislocation boundary. More rounded protrusions (Figure 2.4b) are also observed and in many samples they appear more frequently than the saw-tooth shaped ones. Retrusions are often narrower and sharper than protrusions, and experimentally larger local curvatures are often observed at the tip of retrusions than protrusions [17,23].
Figure 2.3 - EBSD image showing the presence of protrusions/retrusions (one of each is marked by white arrows) in a partly recrystallized pure Al specimen (annealed at 250 °C for 10 min). R: recrystallized grain; D: deformed matrix. Different colors represent different orientations and the thin and thick black lines represent boundaries with misorientation larger than 2° and 15°, respectively. The figure is reproduced from reference [24].

Figure 2.4 - Sketches of protrusions and retrusions on recrystallization boundaries in a high purity Al sample (50% reduction in thickness by cold rolling and annealed at 250 °C for 10 min). (a) Sketch based on an ECC image showing saw-tooth shaped protrusions. (b) Sketch based on a TEM image showing rounded protrusions. R represents the recrystallizing grain. The fat lines in the deformed matrix represent extended dislocation boundaries. The arrows indicate the expected direction of boundary migration. The figure is reproduced from reference [23].
Roughening of recrystallization boundary can affect its further migration through the deformed matrix, as revealed from both experiments and theoretical simulations. When a protrusion/retrusion forms on the recrystallization boundaries, it will provide a dragging/driving force due to the local boundary curvature. Measurements of the curvatures of protrusions and retrusions along recrystallization boundaries have shown that the magnitude of the local curvature-based driving force can be comparable to that of the stored energy within the deformed microstructures [17,18]. The presence of protrusions and retrusions on the recrystallization boundaries can thus be considered to provide locally an extra driving or dragging force. The migration rate of recrystallization boundary at local scale shall then be described by modifying Equation 2.2 to:

\[ v = M \cdot (F_d + F_\sigma) \]  \hspace{1cm} \text{Equation 2.3}

where the contribution from curvature driving force \( F_\sigma \) is added to the stored energy in the deformed matrix (\( F_d \)). Results from phase-field simulations of recrystallization boundary migration have also revealed that the formed protrusions/retrusions can contribute an additional dragging/driving force of the same order of magnitude as to the stored energy [24]. Additionally, phase-field modeling has shown that the highly asymmetrical protrusions and retrusions on migrating recrystallization boundaries can result in an overall increased migration velocity [25]. Molecular dynamics (MD) simulations of boundary migration under artificial driving forces have shown that the roughening of grain boundaries is associated with the variation of mobilities, and the boundaries with rough
morphologies correspond to higher mobility while smooth boundaries can lead to stagnation of boundary migration [26,27].

2.3 Possible factors affecting recrystallization boundary roughening

It is now generally accepted that the migration of recrystallization boundaries is a complex process, and there are many factors contributing to the roughening of the boundaries [28,29]. The growth of recrystallizing grain is directly related to the deformed matrix in front of the recrystallization boundary. During plastic deformation, the structure evolution in both polycrystals and single crystals can be described as a structure subdivision by dislocation boundaries, forming hierarchical structures on a finer and finer scale as strain increases [30]. This subdivision leads to local variations of crystallographic orientations and stored energies. Furthermore, the spatial arrangement of the dislocation boundaries may be important for the recrystallization boundary migration [23].

The stored energy on the local scale can vary significantly even over short distances along a recrystallization boundary. The stored energy in the deformed matrix is mostly in the form of dislocation structures. An example is shown in Figure 2.5, where the local variations within the deformed microstructures of three aluminum single crystals with different orientations are shown by the sketches [31,32]. For the \{112\}<111> and \{123\}<634> orientations, the microstructure consists of localized glide bands (LGBs) and matrix regions, while for the \{110\}<112> single crystal, the microstructure is relatively homogeneous.
Transmission electron microscopy (TEM) based analysis of the stored energies in the three samples shows that the LGBs regions in the \{112\}<111> orientation have the highest stored energy, followed by the LGB region in the \{123\}<634> orientation. Next are the matrix regions of both \{112\}<111> and \{123\}<634> orientations. The matrix in \{110\}<112> has the lowest stored energy [31]. It is further revealed from this example that the local stored energy also depends on orientation beside the dislocation structures. A few models simulating recrystallization boundary migration have incorporated the variation of stored energy in deformed matrix and in that case, the formation of local protrusions/retrusions is reproduced [24,25,33]. From experimental data, it is however found the correlation between boundary migrating rate and local stored energy has large variations [18], which indicates the influences of other factors on boundary migration.

Figure 2.5 - Sketches illustrating the arrangements of dislocation boundaries for three channel-die deformed aluminum single crystals with different orientations: \{112\}<111>, \{123\}<634> and \{110\}<112>. LGB refers to localized glide band. The figure is reproduced from reference [32].
A recrystallizing grain will typically experience large variations in misorientation across its length to the subdivided deformation microstructure and the misorientation relationships will change both temporally and spatially as the grain grows [34,35]. The various misorientation relationships may further lead to the difference in mobility of boundary segments. For example, boundaries with angle/axis pair of $40^\circ<111>$ misorientation have been observed to have the highest mobility [36,37,38]. A recrystallization grain having relatively low-mobility boundaries to its surrounding deformed matrix may experience an abrupt increase in mobility if some boundary segments migrate into a deformed region with new orientations forming high-mobility boundaries, and vice versa [35]. Retardation of boundary migration might also occur, as illustrated by the sketch in Figure 2.6: if a recrystallizing grain grows into areas of nearly its own orientation and forms low-angle boundaries (LABs) with the deformed matrix. Because the LABs are generally known to have low mobility [39], this “orientation pinning” effect can also lead to local variations in boundary migration behaviors.

When the recrystallization boundary migrates through the deformed matrix, the adoption of dislocation boundaries into the new grain may be a factor that influences the migration of the recrystallization boundaries. It has been shown from MD simulations that protrusions, a few atoms in size, may form when the recrystallization boundary interact with individual dislocations. Also the simulations have revealed that different roughening behaviors occur when the boundaries interact with edge dislocations and screw dislocations [40,41]. In the case shown in Figure 2.4(a), the recrystallization boundary appears to preferentially migrate along the extended dislocation boundary instead of
migrating across it, indicating that the recrystallization boundary may interact differently with different types of dislocation boundaries in the deformed matrix.

Figure 2.6 - Schematic illustration of orientation pinning: imaginary recrystallizing grain is shown in typical deformed microstructure. When the grain grows into areas of nearly its own orientation, as those shown in grey, motion of corresponding boundary segment will be retarded by formation of low-angle boundaries. The figure is reproduced from reference [28].

Other factors that may affect the roughening of recrystallization boundary include the impurities in materials and annealing temperature. Second phase particles formed from impurities can have significant effects on recrystallization. Normally large particles accelerate recrystallization by stimulating nucleation and small particles inhibit boundary migration by Smith-Zener pinning [42,43,44,45]: the pinning of local boundary segments can result in a rough morphology of the recrystallization boundaries. Solutes may interact with the grain boundaries, reducing their mobilities and hence affect the migration rate [46] as well as the local morphology. Another factor that may affect the roughening of recrystallization boundaries is the annealing temperature. As revealed from MD
simulation, the boundary structures transit from smooth/low-mobility to rough/high-mobility at a characteristic temperature, which is referred to as roughening temperature [27]. The roughening temperature can vary by hundreds of degrees from boundary to boundary [26], meaning that at different annealing temperatures, the mobility of recrystallization boundaries may differ, in accord with the roughening transition.
Chapter 3
Methods to Characterize the Roughness of Boundaries

This chapter describes various methods that have been developed during this thesis work and tested to enable quantification of the roughness of 2D line features such as recrystallization boundaries. The potentials of the methods are illustrated by examples and their limitations are evaluated.

3.1 Surface roughness characterization

When considering roughness as a parameter, surface roughness is an important one for many manufacturing processes for which a specific surface finish is required. Meanwhile, it is also important for many fundamental aspects of other issues related to the surface, such as friction, wear resistance, corrosion susceptibility, tightness of contact joints, etc. [47]. For these reasons, quantification of surface roughness is a necessity to provide guidelines and references for industrial surface treatments processing.

The surface roughness can be quantitatively characterized from the surface profile, which is normally measured using a profilometer, either a contact one with diamond stylus or an optical one. This is schematically shown in Figure 3.1. A
mean line is assigned to represent the ideally flat surface, and a number of equally spaced points are placed along the profile. In this way, a sample of vertical distances from the mean line to the profile \((Y_1, Y_2, Y_3, \ldots Y_n)\) is obtained and a parameter representing roughness of the profile can be calculated as:

\[
R = \frac{1}{n} \sum_{i=1}^{n} |Y_i|
\]

Equation 3.1

The arithmetic mean deviation as calculated in Equation 3.1 is often used to represent the roughness [47]. With the assigned mean line, samples of other quantities from the profile can be obtained such as the amplitudes of the peaks/valleys, distances between adjacent peaks/valleys, etc., and roughness parameters indicating other characteristics of the surface profile can be derived based on specific requirement.

Figure 3.1 - A sketch showing surface profile measurement using a profilometer. \(Y_i\) is the amplitude at sampling position \(X_i\). The surface roughness can be quantified deriving roughness parameters from the sampling quantities.
This approach to quantify the roughness of a profile is only applicable when a mean line can be determined. However, for the profiles of most microstructural features, assignment of mean line is not possible.

### 3.2 Boundary fractal analysis

For most natural objects, the perimeter is often a problematic measurement, since the value of perimeter is an artifact of image resolution and magnification. In fact, the concept of perimeter can be fundamentally flawed when it is applied with many physical objects. “How long is the coast of Britain?”, asked by Mandelbrot who established the principles of fractal geometry [48]. The answer is that its length is uncertain but depends on the length of the ruler used for the measurement, as shown from the illustration in Figure 3.2 [49]. Essentially the coastline is irregular and features of smaller scale than the ruler length is neglected from the measurement, leading to an inaccurate length. “Fractal”, as proposed by Mandelbrot, refers to the objects whose complex geometry cannot be characterized by an integral dimension (Euclidean dimension) [50]. Objects that have fractal geometry seem to be the norm rather than the exception in nature [48,51]. Euclidean geometry, with its well-defined and mathematically descriptive lines and planes, is usually only found as an approximation in limited situations.
Figure 3.2 - “Measuring” the length of the British coast. (a) ruler length = 200 km, coast length = 2400 km (approx.). (b) ruler length = 100 km, coast length = 2800 km (approx.). (c) ruler length = 50 km, coast length = 3400 km (approx.). This figure is reproduced from Wikipedia [49].

The application of fractal geometry provides an effective tool in the study of highly irregular profiles and surfaces. The concept of “fractal dimension” is employed to characterize the space-filling capacity of a profile or pattern [51] and it can be used as a quantity describing the deviations of irregular features from Euclidean lines and planes. The ideal and theoretical fractal geometries have the invariance property of self-similarity under scale transformation. However, a physical object is fractal only within a limited range of scales and may not strictly confine the property of self-similarity. Moreover, in practice the magnification is restricted to a particular range of interest or experimental feasibility, so the fractal dimension is confined to a limited range.

Since Mandelbrot firstly made the correlation between the fractal dimensions of fracture surface roughness and the impact energy absorbed in fracture specimens [52], fractal analysis has been used extensively in characterizing the rough morphology of fracture surfaces [e.g.53,54,55,56], as well as other
morphological features in materials microstructures, such as boundary morphology [e.g. 57,58] and shapes of second-phase particles [e.g. 59,60]. In most of these studies, the fractal dimension serves as a roughness parameter representing the irregularity of the morphological features. An example is shown in Figure 3.3 of the fractal dimension corresponding to various morphologies of dust [61]. Trials have been made to investigate the correlation between the fractal dimension of a feature and any physical parameters but no solid correlation has been established. While fractal analysis could be an efficient tool to make a comparative study dealing with the roughness of morphological features, including fractal dimension in the description of any physical process of materials remains unfeasible, since most of the microstructure evolutions in materials involve more than one physical mechanisms and may not be self-similar, not even over a limited range of scales [53].

![Figure 3.3 - Fractal dimensions of dust particles with various morphologies. This figure is reproduced from reference [61].](image)

Numerous methods have been proposed and validated in obtaining the fractal dimension, such as the divider method, the box counting method, Euclidean distance method, etc. [62,63,64]. These methods basically follow the same algorithm: (1) measure the quantities (perimeter, area, etc.) of the objects using
various step sizes; (2) plot the measured quantities versus step sized on the logarithm scale and fit a least-square regression line through the data points; (3) estimate the fractal dimension as the slope of the regression line.

When using fractal analysis to quantify the roughness of irregular features, there are limitations that should be respected to ensure valid results. The accuracy, reliability and the associated error margins of the various methods calculating fractal dimension have been evaluated [64,65,66,67]. Problems involved include the generation of insufficient data points to calculate the fractal dimension, different fractal dimension yielded from different measurement methods or the errors caused by digitalization of the images. These problems have lowered the reliability of early works on fractal analysis of irregular features and the most suitable method to provide reliable results is still a matter of discussion.

3.2.1 Fractal analysis of two boundaries in partly recrystallized copper

To evaluate the potential of fractal analysis method to quantify the roughness of recrystallization boundaries, the grain boundaries of two recrystallized grains have been analyzed [68].

Two cube orientated recrystallized grains (Figure 3.4 a and b) in partly recrystallized microstructures were investigated. Both are from oxygen free high conductivity copper (99.9% purity), 90% cold rolled and annealed at 150 ºC. The interior twin boundaries are ignored in the analysis and the boundaries between the recrystallized and the deformed regions are extracted, as shown in Figure 3.4 (c) and (d). The boundary shown in Figure 3.4(c) is designated as B1 and the boundary shown in Figure 3.4(d) is designated as B2. Both B1 and B2
are rough with protrusions/retrusions formed on certain length scales, but visually B1 appears more irregular as it has more long, narrow branch-like features.

Figure 3.4 - EBSD images measured with step size of 0.1 µm of two copper grains. The boundaries surrounding the two grains are shown in (c) and (d), with a pixel bar indicating the length scales of the digital images. Interior twin boundaries are ignored. This figure is reproduced from reference [68].

The method used to measure fractal dimension is the Minkowski “sausage” method, which follows the algorithm mentioned in the previous section. The initial outline of the feature is coarsened by using circles of finite diameters drawn around each point of the grain outline to form a ribbon, which is usually called covering or “sausage” [67]. In practice, the sausage method is implemented using an image processing algorithm named dilation that adds a background pixel for every pixel in contact with the feature. Successive dilation operation on the outline of the feature produce a ribbon of a finite width, as illustrated in Figure 3.5 using the coastline of Britain as an example. The “perimeter” of the dilated outline is obtained by dividing the resultant area by the corresponding diameter of the dilation circle. The log of that perimeter is then plotted against the log of the diameter and the fractal dimension can be obtained from the slope based on the following equation:
\[
\log(\text{perimeter}) = (1 - F_d) \cdot \log(\text{diameter}) + C \quad \text{Equation 3.2}
\]

where \( F_d \) is the fractal dimension and \( C \) is a constant.

Figure 3.5 - Illustration of dilation operation used for “sausage method”. (a) dilated with circle of radius 2 pixel, (b) dilated with circle of radius 5 pixel and (c) dilated with circle of radius 10 pixel. The dilation operation is performed with MATLAB\textsuperscript{®} Image Processing Toolbox.

An artificially constructed fractal curve, the Koch curve [69], has been used to validate the method and as a reference for the fractal analysis of the two grain boundaries. The Koch curve is constructed by iteration, and the geometrical structures are constructed following the same scaling law, whereby it obtains a fractal dimension of around 1.26. Koch curves with 2, 3 and 4 iterations are shown in Figure 3.6 and the characteristic edge length of the finest repeating motif is indicated in Figure 3.6(d). This length is 675, 225, 75 and 25 pixels for Koch curves constructed with 2, 3, 4 and 5 iterations, respectively. The plot on logarithm scale of perimeter versus diameter is shown in Figure 3.7. For the ideal Koch curve with infinite iterations, the log(perimeter) versus log(diameter) plot shall be a straight line with slope of about -0.26. However, with limited iterations,
the Koch curves in the current example are not ideal fractal geometry and the whole shape is constructed with non-continuous motifs. This result is reflected from the inflections of slopes in Figure 3.7(a). With increasing iterations added to the Koch curve, the fractal geometry extended to the smaller length scales. The fractal dimension obtained from the part exhibiting fractal geometry is about 1.26 calculated from the slope of regression line, so the sausage method is validated to provide reliable results of fractal dimension.

Figure 3.6 - Koch curves with (a) two, (b) three and (c) four iterations. The one with five iterations is not shown here due to limited resolution. (d) The red arrow indicates the characteristic edge length of the finest motif.

Figure 3.7 - Log(perimeter) versus log(diameter) plots for (a) Koch curves and (b) grain boundaries B1 and B2.
Using the same sausage method, the fractal behavior of B1 and B2 were analyzed and the results are shown in Figure 3.7(b). The slope varies in a complicated manner for both B1 and B2 and thus a single fractal dimension value corresponding to the entire boundaries cannot be found. Two or more fractal dimensions can be obtained from each plot in Figure 3.7(b) and such behavior is termed multi-fractal, implicating that the morphological features on the boundaries are not alike and exist on different length scales.

To obtain more information from the data, a new parameter, “2-point fractal dimension”, is calculated based on the following equation:

\[
2 - \text{Point Fractal Dimension} = 1 - \frac{\log(\text{perimeter}_1) - \log(\text{perimeter}_2)}{\log(\text{diameter}_1) - \log(\text{diameter}_2)}
\]

Equation 3.3

where \(\text{perimeter}_1\) and \(\text{perimeter}_2\) are adjacent data points corresponding to dilation \(\text{diameter}_1\) and \(\text{diameter}_2\), respectively. This parameter indicates the variation of fractal behavior of the analyzed curve and it is expected that information of local morphological features could be revealed. The 2-point fractal dimension have been calculated and plotted versus dilation diameter for both the Koch curves and the two grain boundaries.

The saw-tooth shapes of the plots as shown in Figure 3.8(a) are due to the limited iterations of Koch curves; since with the ideal Koch curve with continuous motifs, the 2-point fractal dimension calculated from any 2 adjacent data points would return the theoretical fractal dimension of 1.26. The transitive points as indicated by the green arrows ①-④ reveal the positions where the finest motif is resolved by the measurement with increasing dilation diameter. When a smaller
motif is constructed (i.e. more iteration), the transitive point moves towards the smaller diameter that corresponds to the characteristic edge length of the finest motif. This correspondence provides a correlation between the dimension of the features, e.g. finest edge length and the transitive points in the plots. As shown in Figure 3.8(b), the major transitive points for B1 and B2 are marked by the green arrows ⑤-⑪. For B1, there is an almost steady increase of the 2-point fractal dimension values between arrow ⑤ and ⑥. Unlike the sharp increase or saw-tooth behavior in Koch curves, the increase seen in B1 indicates that there are protrusions/retrusions distributed over the entire length scale between ⑤ and ⑥, which agrees with the visual impression of the morphology of B1 (see Figure 3.4c). The transitive point ⑥, at the diameter of about 300 pixels, corresponds to the approximate width of the narrow branches in B1. For B2, two distinct plateaus (between ⑤ and ⑧, ⑨ and ⑩) are seen and they indicate that the protrusions/retrusions are mostly on two scales. Referring to the morphology of B2 (see Figure 3.4d), relative small protrusions/retrusions are mostly on the lower boundary (bottom right) and their dimensions are about 50 pixels corresponding to transitive points marked by ⑤, while the larger protrusions/retrusions are more frequently observed on the upper boundary and their dimensions are about 250 pixels corresponding to ⑨. The 2-point fractal dimension values of B1 and B2 show an almost linear drop at positions ⑦ (diameter at about 660 pixels) and ⑪ (diameter at about 500 pixels) respectively, and both diameters correspond to the approximate overall geometrical width along the horizontal direction of B1 and B2, respectively.
3.2.2 Summary of fractal analysis

The fractal dimension can be used as a roughness parameter to indicate the irregularity of a morphological feature, as exemplified in Figure 3.3. However, the fractal geometry requires similitude in the structural variation at different length scales, which is often not the case for many natural objects, on which the rough features typically are only on limited length scales. As revealed from the fractal analysis of the two grain boundaries, multi-fractal behavior is manifested and roughness of entire boundary profile cannot be represented by fractal dimension. The variation of 2-point fractal dimension plot can provide some information about the length scale of the morphological features, however, the information is not straightforward to analyze and characterization of local features, such as single protrusion/retrusion is not possible.

Despite the fact that fractal analysis provides insufficient quantitative information in the case of grain boundary morphologies, the understandings regarding the characterization of irregular features have been furthered through
the applied fractal analysis process, and the major points of interest include the following:

(1) examples of fractal geometry show that morphological features can exist on multiple length scales and fractal analysis provides information of the features on different length scales through variation of measuring unit. A clear specification of the length scale is the prerequisite for quantitative characterization of irregular features;

(2) in the plots for extrapolating fractal dimensions (e.g. Figure 3.7), the decrease of measured quantity with increasing magnitude of measuring unit is a collective behavior incorporating the “interactions” between local morphological characteristics and the measuring unit. A similar approach can be applied by locally quantifying the morphological characteristics and assembling them in an appropriate manner to represent the overall roughness of a feature. Essentially, any features that appears irregular attributes to local structural variations.

3.3 A new method for quantification of roughness

Based on the cases and results discussed above, it is found that a proper quantitative characterization of irregular features on recrystallization boundaries should apply with the following criteria:

1) The features are on either closed or non-closed boundaries;
2) Mean line or other types of reference curves cannot be easily defined;
3) The length scale of characterization is controllable;
4) Proper sampling of local morphological characteristics can be obtained to calculate representative roughness parameters.
To fulfill these requirements, a new method is developed for better characterization of the irregular features, with a special focus on 2-D line morphology.

The area integral invariant (AII) is one of the integral invariants used in digital image computing for applications such as shape matching, and geometry processing [70, 71]. It is adopted here as a morphological variable. As schematically illustrated in Figure 3.9, the AII is generally obtained in the following way: a circle with a specified radius that is termed sampling radius, is drawn with the center of the circle positioned on the boundary of the object. The AII is then determined as the area of the circle on the object side (the regions marked in red in Figure 3.9), $A_{object}$, divided by the area of the entire circle, $A_{circle}$:

$$AII = \frac{A_{object}}{A_{circle}}$$  \hspace{1cm} \text{Equation 3.4}

In this way, a value ranging between 0 and 1 is obtained. As illustrated in Figure 3.9, if the circle encloses a protrusion (circle a), the AII value will be smaller than 0.5 as $A_{object}$ will be less than half of $A_{circle}$ and conversely for a retraction (circle c). If the boundary within the circle is planar (circle b), the AII value will be exactly 0.5 as the $A_{circle}$ is equally distributed between the two sides of the boundary. If the structural variations on the boundaries are on a scale finer than the sampling radius (circle d), the AII value may also be close to 0.5 if the roughness is distributed so that $A_{object}$ is about half of $A_{circle}$. In this case, $AII = 0.5$ refers to a ‘quasi-planar’ boundary segment covered within the sampling circle, with structural variations on a finer scale than the sampling radius. A smaller sampling radius may pick up such finer scale structural variations (circle e).
Similarly, finer scale structural variations may exist when $AII > 0.5$ or $AII < 0.5$ at a specified sampling radius, as an irregular boundary in reality normally has structural variations on various length scales. Therefore, defining the length scale is necessary when using the $AII$ value to analyze the morphological features. On the other hand, the sampling radius used for obtaining the $AII$ value can also be used to specify the length scale. For clarity in the following discussion, $AII = 0.5$ at a specified sampling radius is considered to represent a planar boundary segment relative to the sampling circle, ignoring any structural variation at finer scales. The same applies for $AII < 0.5$ and $AII > 0.5$, representing a protrusion and retrusion, respectively. This assumption is reasonable as it reflects the characteristics of the structural variation with its relevant length scale.

Figure 3.9 - Sketch showing the area integral invariant ($AII$). With the center of the sampling circle positioned on the boundary, each circle area is separated by the boundary into two regions and the $AII$ value is calculated as the area marked in red (which is the part of the circle on the object side of the boundary) divided by the entire circle area.
For validation of the method and testing its potential, two recrystallization boundaries are used, as shown in Figure 3.10. The two boundaries are from a partly recrystallization aluminum (99.996% purity) specimen, which was annealed at 550 °C for 24 h to obtain a grain size of several millimeters and subsequently cold rolled to 50% reduction in thickness. The specimen was then annealed in a tin bath at 250 °C for 7 min to obtain a partly recrystallized microstructure. The initially large grain size before cold rolling was chosen so that relatively long recrystallization boundaries were obtained after the annealing.

Figure 3.10 - (a) EBSD image showing a small part of the partly recrystallized microstructure in a cold rolled pure Al specimen. Two recrystallization boundaries are selected for testing purposes. Bs: smooth boundary; Br: rough boundary. Different color in the deformed matrix show the misorientation angle of the dislocation boundaries. (b) Post-processed image of the recrystallization boundaries. The red arrow marks out a retrusion.
3.3.1 Characterization of specific rough feature (local quantification)

In the following it is described how the length scale for roughness characterization is determined by the sampling radius. As shown in Figure 3.11, the $AII$ value of the retrusion (which is marked out by the red arrow in Figure 3.10b) is calculated with sampling radii ranging from 20 to 220 pixels. With varying sampling radius, the $AII$ value is not constant but increases with increasing sampling radius, reaching the maximum at a sampling radius of about 60 pixels and then decrease with increasing sampling radius. This variation can be understood from the illustration in Figure 3.12: when the sampling radius is 20 or 40 pixels, the $AII$ sampling circle does not cover the entire retrusion. When the sampling radius is 80 or 100 pixels, the $AII$ sampling circle embraces segments of the neighboring relatively planar boundaries, and the $AII$ value decreases. At the sampling radius of 60 pixels, the $AII$ sampling circle just covers the entire retrusion and returns the maximum $AII$ value. The $AII$ value of a rough feature depends not only on its morphology, but also on “how much” of the feature is included, which is determined by the radius of the sampling circle.

Based on the $AII$ value’s dependence on the sampling radius, two aspects of a rough feature’s morphology can be quantitatively characterized: the sampling radius at which the $AII$ value reaches its minimum (for a protrusion) or maximum (for a retrusion) can be considered as the size and the corresponding $AII$ value can be considered as a quantity representing the sharpness of the rough feature. For example, for the exemplified retrusion, its size is about 60 pixels and it has the $AII$ value of 0.75. In this way, the structural variation of a 2-D line feature can be measured.
Figure 3.11 - Variation of the \( A_l \) value as a function of the sampling radius for one retrusion on \( B_r \) (marked by the red arrow in Figure 3.10).

![Graph showing variation of \( A_l \) value with sampling radius](image)

Figure 3.12 - Schematic illustration of \( A_l \) variation for one retrusion using sampling radius of 20, 40, 60, 80 and 100 pixels.

![Schematic illustration of \( A_l \) variation with different sampling radii](image)

Using this approach to quantify the morphology of specific rough features, the protrusions and retrusions on the boundary \( B_r \) are measured, using sampling radii in the range between 20 pixels and 200 pixels in steps of 2 pixels. With the calculated \( A_l \) values at each position along the boundary, the local protrusions/retrusions on \( B_r \) were determined in the following steps (below only protrusions are referred to but retrusions are treated in the same manner):
1) Locate the protrusion position by searching for the minimum $AII$ values from the sampling radii at every position of the boundary;

2) Record the size of a protrusion as equal to the sampling radius corresponding to the minimum $AII$ value;

3) Filter the data to check the detected protrusions for overlap. This is to avoid repetitive representations of an individual protrusion. The filtering criteria cover two cases: (i) where the sampling circle of a smaller protrusion is entirely enclosed within the sampling circle of a larger protrusion and (ii) where the sampling circles of two protrusions partially overlap. For case (i), it is tested if the center of the sampling circle of the larger protrusion is within the sampling circle of the smaller protrusion, in which case the protrusion with larger $AII$ value is ignored, otherwise both protrusions are kept. For case (ii), it is tested if the center of the sampling circle of either one of the two protrusions is within the sampling circle of the other, in which case the protrusion with larger $AII$ value is ignored, otherwise both protrusions are kept.

The protrusions and retrusions identified by this procedure are shown in Figure 3.13 (a) and (b), respectively. It can be seen that protrusions and retrusions of various sizes have been measured. Visual inspection shows that the $AII$ sampling circles properly covered the protrusions and retrusions, and there are small protrusions/retrusions positioned inside larger ones, which is reasonable as the structural variations exist on different length scales. This suggests that the current approach to quantify specific rough features is applicable.
The distributions of the size and \textit{All} values for protrusions and retrusions are shown in Figure 3.14. In general, the protrusions tend to be larger than the retrusions: there are several large protrusions with sizes over 100 pixels, while the retrusions are mostly with small sizes, with the largest one being around 80 pixels. There exist several very sharp retrusions with \textit{All} value larger than 0.7: while the protrusions are relatively round and smooth without sharp tips (\textit{All} value > 0.3). The quantified results of the rough features’ morphology have revealed the significant difference between protrusions and retrusions even on the same recrystallization boundary.

Figure 3.13 - The measured protrusions (a) and retrusions (b) on the B$_r$ boundary. The circles of certain sizes represent the sampling circles with minimum (a) or maximum (b) \textit{All} values. The protrusions/retrusions close to the grain boundary junction are ignored intentionally.
Figure 3.14 - Histograms of (a) size (sampling radius) and (b) $AII$ value for the protrusions and retrusions as measured in Figure 3.13.

### 3.3.2 Development of roughness parameters

If the $AII$ value is measured at every position consecutively along the boundary, with a constant sampling radius to ensure unbiased measurement of all structural variations on the boundary, a quantification of the morphology of the entire boundary at a specified length scale can be obtained. In this manner, the $AII$ resembles a profilometer (Figure 3.1) that measures the boundary “profile”.

Figure 3.15 shows the consecutive $AII$ values along the boundary of $B_r$, measured using sampling radius of 100 pixels. The peaks and valleys on the plot reflect the structural variations on $B_r$. As mentioned above, $AII$ equals to 0.5 represents planar boundary at the specified length scale, so a “mean line” can be assigned at $AII$ value of 0.5, as illustrated by the red dash line in Figure 3.15. In this way, roughness parameters summarizing the roughness of the boundary can be derived from the deviation of $AII$ values from 0.5. One example roughness parameter for the overall roughness of a boundary is boundary roughness, $R_a$, which is calculated as:
\[
R_a = \left\{ \frac{\sum_{i=1}^{N} (All_i - 0.5)^2}{N} \right\}^{1/2} / 0.5
\]

Equation 3.5

where \( N \) is the number of pixels along the boundary (i.e. sampling circle centers) and \( All_i \) is the \( All \) value at each boundary position. The way \( R_a \) is calculated is similar to the descriptive statistical parameter “coefficient of variation”, but instead of using the arithmetic mean, the reference \( All \) value 0.5 is used as the basis. This roughness parameter has a value ranging from 0 to 1. For a straight line \( R_a \) is 0 while larger values of \( R_a \) indicate that the boundary is more irregular. There are many other parameters that can be developed describing various aspects of the boundary morphology. Several of them are listed in Table 3.1.

Figure 3.15 - Plot of consecutive \( All \) values along the boundary \( B_r \) at sampling radius of 100 pixels. The plot resembles a measured “profile” of the boundary. The red dashed line represents the “mean line” which can be considered as the profile of a planar boundary.
Table 3.1 Roughness parameters globally quantifying a boundary based on a series of $All$ values measured along the boundary

<table>
<thead>
<tr>
<th>Roughness parameter</th>
<th>Calculation equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Protruding boundary roughness</td>
<td>$R_p = \left( \frac{\sum_{i=1}^{N_p} (All_{i} - 0.5)^2}{N_p} \right)^{1/2} / 0.5$</td>
</tr>
<tr>
<td>Retruding boundary roughness</td>
<td>$R_r = \left( \frac{\sum_{i=1}^{N_r} (All_{i} - 0.5)^2}{N_r} \right)^{1/2} / 0.5$</td>
</tr>
<tr>
<td>Fraction of protrusion/retrusion</td>
<td>$R_f = \frac{\text{Number} \ (All &lt; 0.4 \text{ or } All &gt; 0.6)}{N}$</td>
</tr>
</tbody>
</table>

The roughness of protruding and retruding boundary segments, $R_p$ and $R_r$ respectively, can be determined separately in a similar manner as $R_a$, but including only the $All$ values smaller than 0.5 for protruding and $All$ values larger than 0.5 for retruding boundary segments (see Table 3.1). $N_p$ and $N_r$ represent the number of $All$ values where $All < 0.5$ and $All > 0.5$, respectively. Also listed in Table 3.1 is the fraction of protrusions and retrusions ($R_f$) which is calculated as the number of $All$ values with $All < 0.4$ and $All > 0.6$, divided by the total number the $All$ dataset. $R_f$ provides an estimation of the fraction of rough features with respect to the entire boundary.

The boundary roughness, the protruding and retruding boundary roughness and fractions of protrusion/retrusion are calculated for both $B_r$ and $B_s$ with $All$ sampling radius ranging from 20 pixels to 200 pixels, and shown in in Figure 3.16.

Figure 3.16(a) shows that for all the length scales investigated, the boundary roughness parameter $R_a$ of $B_r$ is much larger than that of $B_s$, corresponding to the clear visual difference in roughness of the two boundaries. For $B_r$, the variation...
of \( R_a \) reaches a maximum value at sampling radius of 60 pixels. A similar pattern of variation is exhibited for the retruding boundary roughness \( R_r \) of boundary \( B_r \), as shown in Figure 3.16(b). Referring to the measured size of retrusions on \( B_r \), the largest size of retrusions is about 80 pixels, which explains well the decrease of \( R_r \) when the sampling radius is above that value. \( R_p \) continues to increase until the length scale exceeds the size of small protrusions (~120 pixels), and then begins to decrease, see Figure 3.16(b). When the fraction of protrusions and retrusions, \( R_f \) is plotted as a function of sampling radius, as expected, a maximum value between 60 and 120 pixels is seen (Figure 3.16c).

For the smoother boundary \( B_s \), the variation in roughness parameters as a function of sampling radii is rather simple. With increasing sampling radius, \( R_a \), \( R_p \) and \( R_r \) all decrease continuously. The fraction of protrusions/retrusions, \( R_f \), decreases to 0 at sampling radius of 120 pixels (Figure 3.16c). As observed in Figure 3.10(b), on boundary \( B_s \) there are some small variations, which contribute to the roughness parameters only at small sampling radii.

![Image](image_url)

Figure 3.16 - Calculated roughness parameters (a) \( R_a \), (b) \( R_p \) and \( R_r \) and (c) \( R_f \) for the two boundaries \( B_r \) and \( B_s \). The blue markers are for \( B_r \) and the red markers are for \( B_s \).

In summary, the developed roughness parameters based on the boundary profile through consecutive \textit{All} measurement along the boundary are shown to
quantitatively characterize how irregular of the boundaries’ morphology is. If a large number of boundaries are investigated of their roughening behaviors, the roughness parameters can be used for a statistical investigation of potential important factors.

### 3.3.3 Analysis of All value distributions

Essentially the All values measured using different sampling size from a specific boundary compose a dataset containing the morphological information of the boundary. Analysis of the All datasets at one or more sampling radius can provide further information regarding the boundary’s morphology.

Figure 3.17 shows the histograms of All datasets measured at sampling radius of 20, 40, 80, 160 and 320 pixels for both B_r and B_s. Additionally, the correlation scatter plots are shown to highlight the difference between the distributions at different sampling radius.

As an All value reflects the structural variation at a specific boundary position, the distribution of an All dataset reflects the morphology of the boundary. For example, the left and right tails reflect the protrusions and retrusions on the boundary, respectively. As shown in Figure 3.17(a), for B_r the All distributions at five sampling radii appear similar: widely spread distributions with the center at approximately 0.5. With increasing sampling radius, there is a slight increase in the width of the distributions, as the rough features at larger length scales are revealed with increasing sampling radius. The distributions are asymmetric exhibiting longer and higher tail on the right side, indicating that the B_r has more retruding than protruding boundary segments. While for B_s, it can be observed that with increasing sampling radius, the distributions become narrower and
exhibit a sharp peak at All equaling to 0.5. As the sampling radius increases, the small structural variations on Bs become less prominent in the All measurement. All the five distributions appear symmetric. The exhibited differences in distribution at the same sampling radius as well as in distribution variation with sampling radii between Br and Bs match the visual impression of the two boundaries, that Br is a rough boundary with more structural variations than Bs, which is a rather smooth boundary.

Further information on the length scale of the rough features on one boundary can be obtained from a correlation analysis between two All datasets at different sampling radii. The extent of correlation between two distributions measured with different sampling radii indicates how much the morphology of the features differs across the length scale. For example, as shown in Figure 3.17(a), the distributions at sampling radii of 20 and 320 pixels for Br appear similar, but the correlation plot between the two shows that they are poorly correlated with each other. This is because the rough features on larger length scales are quantified using increased sampling radius, and the All dataset measured using a sampling radius of 320 pixels is thus much different from the one measured using 20 pixels. For Bs, the distributions of different sampling radii appear differently while in the correlation plots the scatter points are significantly more densely distributed compared with those of Br. As Bs is very smooth, the All datasets measured using varying sampling radii are thus similar since either sampling radius would generate All values that are mostly close to 0.5.
Figure 3.17 - Histograms and correlation plots of the All values measured with selected sampling radii: 20, 40, 80, 160 and 320 pixels. The plots in blue are from B_r and the red ones are from B_s. The Y-axis for the histograms is the probability density and the X-axis is the All value, all on the same scale (0.2 - 0.8). Each of the scatter plots shows the correlation between datasets from two sampling radii, e.g. 40 to 20 pixels as indicated from the plot axis title. The X-axis and Y-axis of scatter plots are on the same scale (0.2 – 0.8).
3.3.4 Estimation of boundary curvature from All

An estimation of the local curvature can also be obtained from the All value [70], and the approach is schematically illustrated in Figure 3.18.

![Figure 3.18 - Sketch of estimation of curvature from All. (a) Computation of the angle $\theta$. (b) Approximation of All. The sampling circle with radius $r$ is in blue and the osculating circle with radius $R$ fitting the boundary segment is marked in black. In (b), the shaded sector represents the area that gives a good approximation of All.](image)

An idealized boundary is sketched in blue and the All is calculated with the sampling radius $r$. If the intersected part of the boundary within the circle is a smooth curve, the curvature can be approximated locally with an osculating circle with radius $R$ as shown by the black circle in Figure 3.18(a). According to the law of cosine:

$$\cos(\theta) = r/(2R)$$  \hspace{1cm} \text{Equation 3.6}

The area enclosed by the boundary and the circle of sampling radius $r$ is approximated by the area of the sector (shaded area in Figure 3.18b), and hence the All value is calculated as:
\[ AII \approx \frac{1}{2} \cdot (2\theta) \cdot r^2 = \frac{\theta}{\pi} \]

Equation 3.7

therefore the curvature can be derived as:

\[ k = \frac{1}{R} = \frac{2}{r} \cdot \cos(AII \cdot \pi) \]

Equation 3.8

Equation 3.8 reveals that the measured curvature is determined by both the \( All \) value and the sampling radius. The direction of curvature can be estimated by connecting the center of the circle and the centroid of the shaded area, as shown in Figure 3.18(b).

The method to estimate the curvature from \( All \) value is based on an assumption that needs to be discussed when applying the method with real boundaries. Equation 3.6 and Equation 3.7 are based on that the intersected boundary is a smooth curve so the osculating circle can be approximated, and for a smooth curve, every point on the curve has the same curvature, so the estimated curvature based on \( All \) value is accurate. However, for the real boundaries, most of the segments cannot be approximated by smooth curves and normally only the peak position of the protrusions/retrusions will contribute to the curvature. For example, the retrusions numbered 3, 9 and 13 as shown in Figure 3.13(b), have valley-type shapes with sharp tips and relatively flat sides. In this case, only the tip part of the retrusion will possess curvature, and if the \( All \) values are calculated by the sampling circle covering the entire retrusion as shown in Figure 3.13(b), the curvature can be regarded as “averaged” over the entire boundary segment covered by the sampling circle.
In the model where curvature driving force is included in the local boundary migration kinetics, normally only the curvature contribution from the tip part of a protrusion/retrusion is considered [72]. Therefore, to estimate the curvature of protrusions and retrusions on the real boundaries, a smaller sampling radius instead of the measured size of protrusion/retrusion is used to calculate the curvature.

The curvature energy can be calculated using the following equation [72]:

$$ F_\sigma = 2 \cdot \sigma \cdot k $$

Equation 3.9

where $\sigma$ is the grain boundary energy ($\sigma = 0.324$ J·m$^{-3}$ is used for the present calculation) and $k$ is the local curvature. The curvature energy of retrusions is defined to be positive as they will provide an additional driving force and the curvature energy of protrusions is defined as negative as the curvature provides a dragging force for the boundary migration. Using sampling radius of 20 pixels (equaling to 10 $\mu$m), the curvature energy for protrusions and retrusions as measured in Figure 3.13 are calculated and the histograms are shown in Figure 3.19.

The curvature energies of protrusions are relatively small with absolute values smaller than 0.13 MJ·m$^{-3}$, while retrusions have higher curvature values: seven out of the identified 20 retrusions have absolute curvature energy value equal to or larger than 0.13 MJ·m$^{-3}$. The curvature energies calculated based on the All values are on the same order of magnitude as the reported values and are comparable with the stored energy in a deformed microstructure very similar to the present one (0.4 - 0.6 MJ·m$^{-3}$) [72].
Figure 3.19 - Histogram of calculated curvature energies (absolute value) of the protrusions and retrusions shown in Figure 3.13.

3.3.5 Summary of area integral invariant

The area integral invariant is employed as a morphological variable in the development of the method for quantitative characterization of 2D line features that have irregular morphologies. The potentials of the method and the derived roughness parameters are demonstrated by characterizing the roughness of two boundaries with different morphologies in a partly recrystallized aluminum specimen. The key properties of $AII$ as a morphological variable are summarized as following:

1) As a circle is used as the basis for the morphological variable, the $AII$ is direction independent and this allows unbiased characterization of morphological irregularities;

2) The $AII$ is measured directly at a position on a boundary, regardless of whether the boundary is closed or non-closed.
3) The length scale of the rough features to be characterized is controllable by specifying the sampling radius.

4) Essentially, the $AII$ values for a boundary calculated with one specified sampling radius is a dataset with each data point containing the local morphological information at a boundary position. Global parameters representing the roughness of the entire boundary, can thus be obtained from the of $AII$ dataset.

In the case of studying the roughening behavior of recrystallization boundaries, a proper characterization of local structural variation is important when locally effects are investigated. The morphological variable, as an independent variable, can be employed in a multivariate analysis with other microstructural variables such as local distribution of stored energy and misorientation relationships, in order to evaluate their effects on the local boundary morphology during recrystallization. Meanwhile, global parameters representing the roughness of an entire boundary are necessary when a large number of boundaries from different specimens are to be comparatively analyzed. With the capability for both local and global characterization of the rough features, $AII$ and the derived roughness parameters from $AII$ values are applicable to provide an in-depth analysis of the recrystallization boundary roughness.
Chapter 4
Experimental Design and Procedure

This chapter describes the experimental scenarios. Explanations for the selected materials and related thermomechanical processing parameters are discussed. An introduction to the microstructure characterization techniques and image processing issues are also given.

4.1 Experimental purpose

Previous works on boundary migration during recrystallization as well as investigations of structural variations formed on the recrystallization boundaries, as introduced in Chapter 2, were conducted focusing on single recrystallization grain or a limited number of boundaries. A statistical characterization of the roughening behaviors of recrystallization boundaries as a function of different thermomechanical processing parameters or in different materials has not been performed. The lack of a proper method to quantify boundary roughness was a main limitation, as a qualitative description of the boundaries’ morphology is not enough for comparative analysis.

The morphological variable, area integral invariant, and the derived roughness parameters, as introduced in Chapter 3, quantify the roughness of boundaries and hence allow a statistical investigation of their roughening behaviors, so that
the roughness of recrystallization boundaries in different samples can be compared objectively. Chapter 2 introduces many factors and parameters that can affect the roughening behavior of recrystallization boundaries. In the present study, the parameters of materials purity, deformation strain, annealing temperature and boundary alignment direction are evaluated of their effects on boundary roughness, and the samples are prepared accordingly.

4.2 Materials and deformation

Aluminum of two grades, high purity (99.996 wt pct) and commercial purity (AA1050), are used in the present study. Chemical composition of the commercial purity aluminum AA1050 was measured by optical emission spectroscopy and the result is shown in Table 4.1 [73]. The initial microstructures of the two materials, as characterized by light optical microscopy using polarized light on the anodized surface, are shown in Figure 4.1. The initial grain sizes of high purity aluminum are on the millimeter scale and the average grain size of commercial purity aluminum is about 100 µm.

Table 4.1 Chemical composition (wt pct) of commercial purity aluminum AA1050.

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>V</th>
<th>Zn</th>
<th>Ti</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>99.5</td>
<td>0.16</td>
<td>0.24</td>
<td>0.004</td>
<td>0.005</td>
<td>0.003</td>
<td>0.007</td>
<td>0.013</td>
<td>0.023</td>
<td>0.004</td>
</tr>
</tbody>
</table>

Cold rolling is chosen as the deformation mode in the present study and two laboratory rolling mills with different roll diameters (340 mm and 75 mm) were used considering the initial thickness of the specimens. The rolling speed was between 80 to 90 mm/s for both. After pre-treatment by annealing at 550 °C for
24 h, the high purity aluminum specimens were rolled at room temperature to 50% reduction in thickness, corresponding to true strain of 1.5 and the as-deformed sample is designated as HPAI50 in the following text. The commercial purity aluminum specimens were rolled at room temperature to 50% and 90% reduction in thickness, corresponding to true strain of 1.5 and 2.6, respectively. The specimens were rolled unidirectionally by alternating the top and bottom sides between passes. Oil lubrication was used to reduce surface friction. To ensure a homogeneous rolling condition, the roll gap geometry, \( l/h \) ratio, where \( l \) is the chordal length of the contact between the rolls and the specimen and \( h \) is the mean sample thickness, was maintained within the range 0.5 to 5 [74,75].

Driven by the stored energy in the deformed materials, recovery and recrystallization might occur at ambient temperature. Therefore, immediately after cold rolling, the specimens were stored in a freezer (\( \sim -20^\circ \text{C} \)) to retard recovery and recrystallization.

![Images showing the initial microstructures of (a) high purity aluminum and (b) commercial purity aluminum AA1050. Images are taken using light optical microscope with polarized light.](image)

Figure 4.1 - Images showing the initial microstructures of (a) high purity aluminum and (b) commercial purity aluminum AA1050. Images are taken using light optical microscope with polarized light.
4.3 Annealing treatment

The deformed material will soften during recrystallization, with a gradual decrease in the hardness. Figure 4.2(a) schematically shows how the hardness value varies with annealing temperature and deformation strain (deformation strain: S1 > S2 > S3) for a constant annealing time. Figure 4.2(b) shows the measured hardness variations of specimens after annealing for 60 min at different temperatures, and the specimens are from commercial purity Al, cold rolled with 50% and 90% reduction. Annealing temperatures were chosen from the mid-point on the hardness versus temperature curves.

Figure 4.2 - Hardness values as a function of annealing temperature at a constant annealing time, shown for different deformation amounts. (a) Schematic illustration (deformation amount S1 > S2 > S3. (b) Measured hardness values of annealed specimens from commercial purity Al, cold rolled to 50% and 90% reduction.

An air furnace is used for most of the annealing treatments. The furnace can be pre-heated to the specified temperature and held stable within ±2 °C. The samples were placed in the middle of the furnace tube, where the temperature is
monitored by thermocouple. The sizes of the samples are small so that they can reach the desired temperature in a short time.

The annealing parameters for various samples are listed in Table 4.2. Three annealing temperatures were applied for the samples of commercial purity Al with 50% cold rolling reduction. Proper annealing time was chosen coupled with the annealing temperatures, aiming was to achieve partly recrystallized microstructures with around 50% recrystallization fraction by the annealing treatments.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Thickness reduction</th>
<th>Annealing temperature</th>
<th>Annealing time</th>
<th>Designation</th>
</tr>
</thead>
<tbody>
<tr>
<td>High purity Al</td>
<td>50%</td>
<td>200°C</td>
<td>60 min</td>
<td>Al50-200</td>
</tr>
<tr>
<td>Al AA1050</td>
<td>50%</td>
<td>325°C</td>
<td>60 min</td>
<td>Al50-325</td>
</tr>
<tr>
<td>Al AA1050</td>
<td>50%</td>
<td>350°C</td>
<td>30 min</td>
<td>Al50-350</td>
</tr>
<tr>
<td>Al AA1050</td>
<td>50%</td>
<td>375°C</td>
<td>10 min</td>
<td>Al50-375</td>
</tr>
<tr>
<td>Al AA1050</td>
<td>90%</td>
<td>300°C</td>
<td>60 min</td>
<td>Al90-300</td>
</tr>
</tbody>
</table>

**4.4 Microstructure acquisition**

Electron backscattered diffraction (EBSD) was employed as the main technique for characterization of microstructures in the present study. EBSD is an accessory system attached to a scanning electron microscope (SEM) and can provide quantitative microstructural information about the crystallographic nature of most inorganic crystalline materials [76]. The working principles of EBSD are illustrated in Figure 4.3 [77]. The incident electron beam hits the surface of the specimen that is tilted 70° with respect to the beam. The interference of
channeled backscattered electrons generates a Kikuchi diffraction pattern. With a phosphor screen placed close to the specimen, the diffraction pattern can be seen and captured by a CCD camera. The diffraction pattern is then analyzed automatically by a computer to obtain the crystallographic orientation of the scanned point. The spatial resolution of EBSD is primarily determined by the SEM: with a field-emission gun (FEG), the spatial resolution is about 20 nm. The angular resolution is determined by the resolution of EBSD detector and its position with respect to the sample, and is currently limited to 0.5° [76].

In the present study, a Zeiss Supra-35 FEG SEM equipped with an HKL Channel 5 EBSD system was used for microstructure characterization. Samples to be examined by EBSD were mechanically polished followed by electropolishing to prepare a flat surface as well as to remove the residual stress brought by mechanical polishing. For electropolishing, A2 electrolyte (70 wt-% ethanol, 12 wt-% water, 10 wt-% 2-butoxy-ethanol and 8 wt-% perchloric acid) was used and the samples were immersed in the electrolyte at 2~5 °C for 45 s with a potential difference of 13 V.

The partly recrystallized microstructures were characterized in the plane defined by the rolling direction (RD) and normal direction (ND) of the samples. In reality the recrystallization boundaries are rough surfaces, and 3-dimensional characterization of the recrystallization boundaries in partly recrystallized aluminum have revealed that the protrusions and retrusions appear as ridges prolonged along transverse direction (TD) of the sample [29], so the 2-dimensional microstructural characterization from the RD-ND plane should well indicate the morphological characteristics of recrystallization boundaries. In the present study,
all of the partly recrystallized microstructures were characterized from the RD-ND plane.

Figure 4.3 - Schematic illustration of (a) typical EBSD installation and (b) electron interaction with crystalline materials [77].

The recrystallized grains were identified from the acquired EBSD data using an in-house MATLAB program named DRG. This program was developed with the purpose to detect recrystallized grains in partly recrystallized microstructures and the algorithm can be found in reference [78]. The recrystallized grains are selected based on the following parameters:

1) The misorientation inside a recrystallized grain should be smaller than 1°;
2) The minimum grain size, calculated as the equivalent circle diameter, should be no smaller than 5 µm;
3) At least part of the boundaries surrounding recrystallized grain should be high angle boundaries (misorientation angle > 15°).

An example of the partly recrystallization microstructure processed by the DRG program is shown in Figure 4.4.
4.5 Image processing and computation

Calculation of the \( AII \) values for a recrystallization boundary involve digital image processing and computation, and the related issues are illustrated in this section.

A digital image described in a 2D discrete space is derived from an analog image in a 2D continuous space through a sampling process, which is frequently referred to as digitization. The digital image is composed of many basic elements that are often called pixels and arrangement of pixels in a digital image is determined by the sampling processes, which typically include rectangular sampling and hexagonal sampling. In the present study, square pixels from rectangular sampling are used for all the digital images.

In the case of rectangular sampling, two types of neighborhoods or connectivity relations are present between pixels: 4-connected neighborhood or
8-connected neighborhood. As illustrated in Figure 4.5, the retrusion as marked out in Figure 3.10(b) is enlarged to show the exact pixels that form it: constructed with 4-connectivity (Figure 4.5a) and 8-connectivity (Figure 4.5b). The inserts in the figure show the neighbors (in red) of a pixel (in blue) for 4-connectivity and 8-connectivity: 4-connected pixels are neighbors to every pixel that touches one of their edges and these pixels are connected horizontally and vertically; 8-connected pixels are neighbors to every pixel that touches one of their edges or corners and these pixels are connected horizontally, vertically, and diagonally. So the same boundary in a digital image stored with 4-connectivity or 8-connectivity can have different morphologies and thus the computation concerning the boundary will have different results. In the present work, all the boundaries are constructed of 1 pixel in width with 8-connectivity, for consistency.

Figure 4.5 - Enlarged illustration of the retrusion marked out in Figure 3.10 to show the exact pixels that form it: constructed with (a) 4-connectivity and (b) 8-connectivity. The inserts show the neighbors (in red) of one pixel (in blue) for each type of connectivity.

The circles for calculation of the Adl value are constructed using the midpoint circle algorithm [79]. The exact pixels that form the circles with radius of 5, 10 and
20 pixels are shown in the enlarged illustration in Figure 4.6(a). It can be seen that the constructed circle of smaller radius is more affected by digitization and further away from the ideal shape of the circle. The deviation of the constructed circle from the ideal circle can affect the calculation of All values, especially for line features with variations in the alignment direction. To evaluate this effect, the All values of a circle with radius of 500 pixels are calculated with increasing sampling radius ranging from 5 pixels to 50 pixels. The standard deviation as a function of sampling radius is shown in Figure 4.6(b): with increasing sampling radius, the standard deviation decreases. To reduce the effects of the bias from digitization, sampling radius smaller than 20 pixels will be not used for All calculation in the present study.

![Figure 4.6](image.png)

Figure 4.6 - (a) Enlarged view of the pixels forming circles of radius 5, 10 and 20 pixels. (b) Standard deviation of All values as a function of sampling radius. The All values were measured from a circle of 500 pixels radius.

The step size used to acquire the EBSD data determines the resolution of the reconstructed image from the EBSD data, i.e. one pixel in the reconstructed
image corresponds to a unit step size in the microstructure. As discussed in Chapter 3, the length scale should be defined before quantification of roughness, and the fixed physical length to pixel ratio in the reconstructed image of EBSD data makes it feasible to control the length scale.
Chapter 5
Roughness of Recrystallization Boundaries

In this chapter, the results of the quantitative characterization of boundary morphologies and boundary roughness are presented. A statistical analysis of the effects of materials purity, deformation strain, boundary alignment direction and annealing temperatures on the roughening behaviors of recrystallization boundaries is the main focus.

5.1 Recrystallization boundaries in partly recrystallized microstructures

After the annealing treatments described in Chapter 4, the samples were partly recrystallized. EBSD observations were carried out to characterize the partly recrystallized microstructures. Examples of typical microstructures including recrystallized grains and deformed matrix separated by recrystallization boundaries are given in this section.

In the partly recrystallized high-purity Al samples, the size of recrystallized grains can extend up to several millimeters along the RD direction. Therefore, most of the orientation maps only showed incomplete recrystallized grains.
Figure 5.1 shows the partly recrystallized microstructure of HPAI50-200 sample. Two recrystallized grains, RG1 and RG2 are marked. The recrystallization boundaries of RG1 are heterogeneous: the bottom and right boundary segments of RG1 are smooth with tiny structural variations that are hardly identifiable with the current step size of 1 µm of EBSD measurement, while the top boundary segment has a rough morphology with protrusions and retrusions of large wavelength. Meanwhile, the recrystallization boundary of RG2 shows a quite different morphology, forming protrusions/retrusions on a relatively smaller length scale than the top part of RG1. It is thus clear that the roughening behaviors of recrystallization boundaries vary, for different recrystallized grains in the same sample, and even for different segments surrounding the same recrystallized grain.

![Figure 5.1 - Orientation map showing the partly recrystallized microstructures of the HPAI50-200 sample. The EBSD data was acquired with step size of 1 µm. The black lines show dislocation boundaries with misorientation angle larger than 15°. RG1 and RG2 mark two recrystallized grains.](image-url)
The structural variations can also form on different length scale, as shown in Figure 5.2 of the partly recrystallized microstructures of the HPAI50-200 sample. Figure 5.2(a) shows the orientation map obtained using a step size of 1 µm. The large recrystallized grain has a recrystallization boundary of wavy morphology, having structural variations on the length scale about 100 µm. The area of a large protrusion, as marked by the white rectangle, was scanned with a finer step size of 0.1 µm, as shown in Figure 5.2(b). It can be seen that the two sides of the large protrusion, which appears to be relatively smooth in Figure 5.2(a), both have protrusions/retrusions on length scale below 10 µm when examined with the finer step size. It is thus revealed that rough features commonly exist on various length scales, and a proper sampling radius on the relevant length scale should be specified for quantification of roughness for recrystallization boundaries.

Figure 5.2 - Orientation maps showing the partly recrystallized microstructure of HPAI50-200 sample. (a) EBSD data was acquired using a step size of 1 µm. (b) The area marked by the white rectangle in (a), and ESBD data was acquired using a step size of 0.1 µm.
Examples of the partly recrystallized microstructures in the Al50-325 and Al90-300 samples are shown in Figure 5.3. In the Al50-325 sample (Figure 5.3a), the recrystallized grains are well distributed in the deformed matrix. Some of the recrystallized grains are equiaxed, but the majority of the grains are elongated along RD. In contrast to the boundaries between recrystallized grains, which are smoothly curved, most of the recrystallization boundaries separating a recrystallized grain from the deformed matrix have rough morphologies with structural variations of different shapes and sizes. It can also be observed that some of the recrystallization boundary segments, e.g. recrystallization boundary segments surrounding RG3 and RG4, are parallel to the extended dislocation boundaries in the deformed matrix. In the Al90-300 sample (Figure 5.3b), the recrystallized grains tend to appear in bands aligned along RD and the grains are mostly elongated and impinged upon each other within the bands. The boundary segments aligned along ND are thus mostly between recrystallized grains. In the deformed matrix, the dislocation boundaries can be seen and the recrystallization boundary segments that are parallel to the dislocation boundaries, e.g. recrystallization boundary segments surrounding RG7 and RG8, are relatively smooth with small structural variations formed. Other recrystallization boundaries, such as those from RG5 and RG6, appear to be more irregular with rough features at different length scales.

From the above examples, it can be concluded that the rough morphology of recrystallized boundaries is a common feature in partly recrystallized microstructures. The roughening behavior of recrystallization boundaries vary between different samples, different grains and different segments of individual
grains, indicating that the boundary migration is a complicated process, affected by many factors or parameters.

![Orientation maps showing the partly recrystallized microstructures of the (a) Al50-325 and (b) Al90-300 samples. EBSD data was acquired using a step size of 0.1 μm for both maps. RG3-RG8 mark six recrystallized grains.](image)

5.2 Quantification of boundary roughness

The recrystallized grains in the partly recrystallized microstructures were detected using the DRG program as introduced in Section 4.4. The recrystallization boundaries separating the recrystallized grains from the deformed matrix can be subsequently extracted. In the current study, only the recrystallization boundaries were considered, because only these boundaries will migrate further during recrystallization while the boundaries between recrystallized grains are impinged and typically only move during grain growth at higher temperatures.

For analysis purposes, the selected recrystallization boundaries to be characterized are limited to extended boundary segments that distinctly align along RD or ND (within 20° deviation). The grain boundary junctions and grain
boundary “corners” are excluded as these structural variations will affect the calculated roughness parameters but they are not in the “regime” of recrystallization boundary roughening. As an example, two boundary segments, RB1 and RB2, along RD matching the selection criteria are marked by the white lines in Figure 5.4(a). The selected boundary segments are then extracted and processed as one-pixel wide line profile with 8 connectivity, as shown in Figure 5.4(b).

The sampling radius for calculation of $AII$ value determines the length scale of the quantified roughness, as discussed in Chapter 3. In the present study, sampling radii of 1 µm and 3 µm were chosen as they largely cover the length scales of the rough feature and correspond to the length scale of banded dislocation structures in the deformed matrix. With the specified sampling radius, the $AII$ value at every position of the selected boundary segments was calculated. Then the roughness of the boundary segment at that length scale was quantified by $R_a$, calculated using Equation 3.5.

Several groups of $R_a$ are calculated, with each group corresponding to one sample listed in Table 4.2, except that there are two groups of $R_a$ from the Al50-325 sample, of boundary segments aligned along RD and ND, respectively. The boundary segments in other samples are all aligned along RD. Each group consists of $R_a$ dataset with more than 25 boundary segments. The Welch t-test is used to compare the $R_a$ datasets from different groups to evaluate the effects of parameters of materials purity, deformation strain, annealing temperature and boundary alignment direction on the roughening behaviors of recrystallization boundaries. The Welch t-test is unpaired, two-sample t-test, assuming that both
groups of data are sampled from normal distribution but not assuming the two populations have the same variance. This assumption of Welch t-test is applied in the present analysis, because whether the $R_a$ datasets from different groups have the same variance is unknown. The null hypothesis is that the tested two $R_a$ datasets have the same mean value but possibly different variance. The result is 1 if the test rejects the null hypothesis with 5% confidence level, and 0 otherwise.

![Diagram of boundary segments](image)

Figure 5.4 - (a) Two boundary segments, RB1 and RB2, matching the selection criteria are marked by the white lines intersecting the boundary. R represents the recrystallized grains. (b) Extracted boundary segments marked in (a).

### 5.3 Effects of material purity on boundary roughening

The AA1050 Al material used in the present study contains 0.5% volume fraction of FeAl$_3$ and FeAlSi particles with average size of 1.7 $\mu$m [13]. The effects
of impurities, e.g. particle pinning, on the roughening of recrystallization boundaries can be investigated through comparative analysis of boundary roughness between the commercial-purity and high-purity Al samples. The Al50-325 and HPAI50-200 samples were chosen for the analysis, as the two samples were deformed to the same strain. Only the boundaries segments aligned along RD were included in the analysis, as the grain boundaries aligned along ND in HPAI50-200 sample are mostly between recrystallized grains.

The $R_a$ of 53 and 25 boundary segments aligned along RD were calculated for the Al50-325 and HPAI50-200 samples, respectively. The distributions of the two $R_a$ datasets are shown in Figure 5.5. At the sampling radius of 1 $\mu$m (Figure 5.5a), wide distributions of $R_a$ for both samples are seen, and apparently there are more boundary segments with higher roughness in the HPAI50-200 sample. At the sampling radius of 3 $\mu$m, except for a few boundary segments that exhibit much higher roughness in the HPAI50-200 sample, the two samples have almost identical distributions of $R_a$ (Figure 5.5b), i.e. have similar roughening behaviors for most of the boundary segments. The result from t-test is shown in Table 5.1: at sampling radius of 1 $\mu$m, the null hypothesis is rejected, meaning that the $R_a$ datasets from the two samples are different with 95% statistical significance; while at sampling radius of 3 $\mu$m, the null hypothesis is accepted, indicating that the difference between the two $R_a$ datasets are not so significant at this larger length scale.

As introduced in Section 2.3, second phase particles can exert dragging forces on a moving boundary, which may retard local boundary migration and lead to boundary roughness. However, the comparison of $R_a$ datasets as shown in
Figure 5.5 reveals that the recrystallization boundary roughening is more pronounced in the high-purity Al sample than in the commercial purity one. This suggests that the pinning effects from particles are not the main reason accounting for the roughening of recrystallization boundaries in the present samples. The Zener drags force caused by the particles, as estimated in a previous study of a sample similar to the present commercially pure Al [13], was about a factor of 10 smaller than the stored energy in the deformation matrix. The driving force for recrystallization boundary migration is therefore much larger than the particle dragging force, which may explain the present lack of effect from particles on the roughening behavior. Also, in the present Al50-325 sample, the average spacing between particles is larger than the length scale of the measured roughness, supporting the conclusion that the observed roughness is not due to particle pinning. The retarding forces from second phase particles on boundary migration might be considerable when the deformation strain is smaller or in materials with smaller particle sizes and inter-particle distances.

The solute atoms (e.g. Fe, Si, Mn) in the commercial-purity Al sample, on the other hand, may affect the boundary migration behavior during recrystallization. Previous experimental evidences have shown that a certain amount of impurity has a larger influence leading to a decreasing recrystallization rate when it is dissolved than when it is precipitated [42]. According to the solute drag theory [80,81], the solutes can interact with the recrystallization boundaries, and due to this interaction, the solute atoms concentrate near the boundaries forming an “atmosphere”, which must be dragged by the migrating boundary. Hence, the mobility of the boundary is reduced and so is the migrating rate. Figure 5.6 shows
Experimental results revealing that during recrystallization the grain growth rate decreases and the activation energy for boundary migration increases with increasing concentration of solute atoms [44]. Boundaries with low mobility may be less rough as suggested by MD simulation [26,27]. In the present experiment, the solute atoms in the commercial-purity Al sample can lead to a reduced boundary mobility and thus lower roughness compared with those in the high-purity Al sample. This difference in roughness is predominantly on a finer scale, as the t-test results shows the difference between $R_a$ datasets is statistically significant at a scale of 1 $\mu$m but not at 3 $\mu$m.

![Histogram of $R_a$ for recrystallization boundary segments aligned along RD in the Al50-325 and HPAI50-200 samples. $R_a$ was calculated using sampling radii of (a) 1 $\mu$m and (b) 3 $\mu$m.](image)

**Figure 5.5**

<table>
<thead>
<tr>
<th>Sampling radius</th>
<th>1 $\mu$m</th>
<th>3 $\mu$m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hypothesis test result (h)</td>
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<td>0 (accepted)</td>
</tr>
<tr>
<td>p-value</td>
<td>0.0015</td>
<td>0.2786</td>
</tr>
</tbody>
</table>

**Table 5.1** Results from two-sample t-test evaluating the difference of $R_a$ dataset between the Al50-325 and HPAI50-200 samples (confidence level for rejection of null hypothesis: 0.05)
Figure 5.6 - (a) Growth rate of recrystallizing grains in a cold-rolled aluminum specimen with addition of Cu or Mg annealed at 132°C. (b) Activation energy for boundary migration during recrystallization of an Al sample with addition of Ag. The figure is reproduced from reference [44].

5.4 Effects of deformation strain on boundary roughening

The Al50 and Al90 samples are from the same material but with different deformation amounts. The effects of deformation strain and thus the deformation microstructures on boundary roughening behaviors can be evaluated by compare the $R_a$ datasets of the two samples. The boundary segments aligned along RD in Al50-325 and Al90-300 samples were chosen for comparative analysis. The above mentioned $R_a$ dataset of 53 boundary segments from the Al50-325 sample was used in the analysis, and the $R_a$ dataset of the Al90-300 sample includes 48 boundary segments.
Figure 5.7 shows the distributions of $R_a$ for boundary segments aligned along RD in the Al50-325 and Al90-300 samples. It is seen that at the sampling radius of 1 $\mu$m, both distributions have peak values at $R_a$ equal to 0.10, while the distribution of $R_a$ for the Al90-300 sample evidently has a longer tail towards high values, i.e. there are more boundary segments of high roughness in the sample with higher deformation amount. At the sampling radius of 3 $\mu$m, the difference between the two distributions becomes less prominent, but the distribution of $R_a$ for the Al90-300 sample still exhibits a shift towards high values. This observation suggests that larger strain may induce more rough boundaries. The result of t-test is shown in Table 5.2: at sampling radius of 1 $\mu$m, the null hypothesis is rejected, meaning that the $R_a$ datasets from the two samples are different with 95% statistical significance; while at sampling radius of 3 $\mu$m, the differences are not so significant, i.e. the null hypothesis is accepted.

Figure 5.7 - Histogram of $R_a$ for recrystallization boundary segments aligned along RD in the Al50-325 and Al90-300 samples. $R_a$ was calculated using sampling radii of (a) 1 $\mu$m and (b) 3 $\mu$m.
Table 5.2 Results from two-sample t-test evaluating the difference of $R_a$ dataset between the Al50-325 and Al90-300 samples (confidence level for rejection of null hypothesis: 0.05)

<table>
<thead>
<tr>
<th>Sampling radius</th>
<th>1 µm</th>
<th>3 µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hypothesis test result (h)</td>
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<td>0 (accepted)</td>
</tr>
<tr>
<td>p-value</td>
<td>0.0030</td>
<td>0.1441</td>
</tr>
</tbody>
</table>

During cold rolling deformation, the energy stored in the materials is mostly in the form of dislocations and dislocation boundaries. The deformation microstructures are subdivided by various types of dislocation boundaries, forming hierarchical structures on a finer and finer length scales as the strain is increased [19,82,83]. At low strains (strain $< 0.8$), as shown in Figure 5.8(a) [83], the deformed microstructure is typically composed of cell blocks or bands, which are bounded by dense dislocation walls (DDWs) and microbands (MBs). The DDWs/MBs are commonly observed to align at an inclined angle with respect to the rolling direction and have large misorientation angles across them. At 50% cold rolling reduction, as the example in Figure 5.8(a), the average spacing of DDWs/MBs have average spacing of about 1~2 µm and align at an angle of about $40^\circ$ to RD. The cell block structure is further divided into cells, which are volume elements almost free of dislocations, delineated by cell boundaries. The structural morphology changes with increasing deformation strain. At higher strains (strain $> 0.8$), as shown in Figure 5.8(b) [84], the deformation microstructure is typically characterized by extended lamellae separated by lamellar boundaries (LBs), which are more or less parallel to the rolling plane. Between the LBs, interconnecting boundaries are formed. The DDWs/MBs at low strains and LBs at high strains are geometrically necessary boundaries (GNBs) since they are assumed to delineate regions with different slip activities. Cell boundaries at low
strains and interconnecting boundaries at high strains are both incidental dislocation boundaries (IDBs) since they are assumed to form by mutual trapping of glide dislocations [19].

When the strain is increased, for both GNBs and IDBs, the average boundary spacing decreases and the average misorientation angle across the boundary increases, as shown in Figure 5.9 (a) and (b) [85]. Due to the higher misorientation and reduced boundary spacing, the stored energy in the deformed matrix also increases with increasing strain. As shown in Figure 5.9(c) [86], the estimated stored energy increases monotonically with increasing strain independent of the measuring technique and deformation mode.

Figure 5.8 - (a) TEM image showing a cell block structure in a 50% cold rolled high-purity Al specimen from the longitudinal plane of view. (b) TEM image showing the lamellar structures in a AA1200 Al specimen cold rolled to a strain of 2.0. The figure is reproduced from reference [83] and [84].

The deformation microstructures of the present samples, HPAI50, Al50 and Al90, were characterized using EBSD. Example orientation maps of the deformation microstructure of HPAI50 sample are shown in Figure 5.10.
Figure 5.10(a) was measured with a step size of 1 µm and shows a relatively large area of the deformed matrix. The original grain was subdivided, and some banded structures can be seen on the top part of the image. Due to the large step size used for data acquisition, the DDWs/MBs and IDBs were not clearly resolved as those examined by TEM as exemplified in Figure 5.8. When using a smaller step size of 0.1 µm, as shown in Figure 5.10(b), the extended dislocation structures can be observed, aligning at an angle of about 30°~40° with respect to the rolling direction. The spacing between the extended dislocation boundaries can be roughly estimated to be around 2~3 µm, which agrees with the reported value in literature of 1~2 µm, considering that some of the dislocation boundary might not be clearly resolved due to the limited resolution of EBSD compared with TEM.

Figure 5.9 - Variation of the average boundary spacing and the average misorientation angle as a function of strain, measured from TEM in 99.999%-purity nickel for (a) GNBs and (b) IDBs. (c) Stored energy measured by differential scanning calorimetry (DSC) and TEM for 99.996%-purity nickel deformed by cold rolling (CR) and accumulative rolling bonding (ARB). The figure is reproduced from reference [86].

Figure 5.11 shows the examples of orientation maps of the deformation microstructures for Al50 and Al90 samples. After 50% reduction (Figure 5.11a),
the prior grain boundaries are still visible and regular array of parallel bands can be observed, which are typical for the deformed matrix of Al with 50% cold rolling reduction. In some of the deformed grains the banded structures are difficult to identify while in other grains they can clearly be seen. This may be due to the orientation dependence of dislocation structures during deformation [87]. The observed bands align at an angle of about 40° with respect to the rolling direction. After 90% reduction (Figure 5.11b), the prior grains are extensively flattened and the lamellar boundaries predominate in most areas. There are also areas where DDWs/MBs are still visible delineating cell blocks and other areas with cell or subgrains. The transition from low strain cell block structure to high strain is aided by coarse slip in S-bands, which can be seen to coexist with the LBs [88]. After larger amount of deformation, the misorientation across extended dislocation boundaries has developed to such an extent that the DDWs/MBs become high angle boundaries.

Figure 5.10 - Orientation maps showing the deformation microstructure of the high-purity Al sample with 50% cold rolling reduction. (a) EBSD data measured with step size of 1 µm and (b) EBSD data measured with step size of 0.1 µm.
Figure 5.11 - Orientation maps showing the deformation microstructures of AA1050 Al samples with (a) 50% and (b) 90% cold rolling reduction. The black lines show the dislocation boundaries with misorientation angle larger than 15°. The white area is unindexed points. Both EBSD maps were scanned with step size of 0.1 µm.

During recrystallization, the recrystallization boundary segments aligned along RD in the Al90-300 sample, in an overall sense, migrate in the direction perpendicular to the lamellar boundaries, whereas in the Al50-325 sample, the migrating boundary segments aligned along RD lie about 40° to the GNBs. This
means that the local dislocations arrangement and thus the local distribution of stored energy in front of the recrystallization boundaries are different between the Al50-325 and Al90-300 samples. The more heavily deformed sample (Al90-300) has higher stored energy and smaller boundary spacing for both GNBs and IDBs, as illustrated in Figure 5.9. Since the stored energy is mostly in the form of dislocation boundaries, it is expected that the recrystallization boundary in Al90-300 would experience more heterogeneous distribution of stored energy locally on the length scale of about 1 µm, which corresponds to the length scale of boundary spacing in the deformed matrix. This may explain the significant difference in boundary roughness between Al50-325 and Al90-300 samples at sampling radius of 1 µm. When a sampling radius of 3 µm is used, it exceeds the length scale of boundary spacing in the Al50-325 sample. Therefore, the recrystallization boundaries in both samples would experience similar heterogeneity in the local distribution of stored energy and the difference in boundary roughness between the two samples becomes less prominent at this larger length scale.

5.5 Effects of boundary alignment direction on boundary roughening

To evaluate the effects of grain boundary direction relative to the processing direction on the boundary roughening behavior, $R_a$ of 25 boundary segments aligned along ND and 53 boundary segments aligned along RD in the Al50-325 sample are analyzed. Since the boundaries aligned along ND are mostly between...
recrystallized grains, a smaller number of those are collected compared with those aligned along RD. The distributions of $R_a$ are plotted in Figure 5.12 for comparative analysis. As shown in Figure 5.12(a), at the sampling radius of 1 µm, the two distributions both have the largest fraction of $R_a$ around 0.10, while the distribution of the boundary segments aligned along ND shifts towards high values, i.e., there are more boundary segments of high roughness aligned along ND. At sampling radius of 3 µm, the difference between the two distributions becomes even more pronounced. The result from the t-test is shown in Table 5.3: at both sampling radii, the null hypothesis is rejected, meaning that the $R_a$ datasets from boundary segments aligned along different directions are significantly different.

As described in Section 5.4, for the Al50 sample, the DDWs/MBs are aligned at an angle about 40° to RD. Therefore, the deformation microstructures in front of the recrystallization boundary segments aligned along RD and ND are largely similar. The higher roughness of the recrystallization boundary segments aligned along ND than those along RD is therefore not expected to be a direct consequence of the surrounding microstructures. The different migrating rate may be a reason for the difference of roughness. Most of the recrystallized grains in the Al50-325 sample are elongated along RD with an average aspect ratio of 1.9 ± 0.5, as measured from the orientation maps. It is thus clear that the recrystallization boundary segments aligned along ND migrate faster than those along RD. MD simulations have shown that grain boundaries with rough morphology correspond to higher mobility/migrating rate [26,27]. However, whether the higher roughness of boundary segments aligned along ND relates to
this faster migration or to other factors related to other more complex issues of
the morphology of the deformed microstructure cannot be evaluated based on
the present data.

![Figure 5.12 - Histogram of $R_a$ for recrystallization boundary segments aligned
along RD and ND in the Al50-325 sample. $R_a$ was calculated using sampling
radii of (a) 1 $\mu$m and (b) 3 $\mu$m.]

<table>
<thead>
<tr>
<th>Hypothesis test result (h)</th>
<th>1 (rejected)</th>
<th>1 (rejected)</th>
</tr>
</thead>
<tbody>
<tr>
<td>p-value</td>
<td>0.0057</td>
<td>0.0004</td>
</tr>
</tbody>
</table>

### 5.6 Effects of annealing temperature on boundary roughening

Thermal roughening of grain boundaries at typical recrystallization
temperatures has been reported in previous studies [27] and the fraction of rough
boundary associated with higher mobility increases when the temperature is
increased. Therefore, the annealing temperature may also affect the
recrystallization boundary roughness. The Al50 samples was annealed at three different temperatures, with suitable annealing time to obtain a recrystallization fraction of about 50%. The effects of annealing temperature on the roughening behaviors of recrystallization boundaries can be investigated by comparative analysis of the $R_a$ datasets of those samples.

Figure 5.13 (a) and (b) show the distributions of $R_a$ for the boundary segments aligned along RD the Al50-325, Al50-350 and Al50-375 samples and the $R_a$ datasets for each sample include 53, 34 and 40 boundary segments, respectively. At sampling radius of 1 µm, the distribution of $R_a$ shifts towards the higher roughness values with increasing annealing temperature. A similar trend can be observed at sampling radius of 3 µm, while the difference of the $R_a$ datasets between Al50-350 and Al50-375 samples are not as prominent as for the sampling radius of 1 µm, but both samples apparently have more boundary segments with higher roughness than the Al50-325 sample. The t-test confirms the visual results, as listed in Table 5.4. At sampling radius of 1 µm, the differences of $R_a$ datasets between Al50-325 and Al50-350 samples as well as between Al50-325 and Al50-375 samples are significant although the null hypothesis is accepted for the t-test of Al50-350 and Al50-375, the p-value is low, close to the significance level. At sampling radius of 3 µm, the t-test result shows that the differences of boundary roughness between Al50-350 and Al50-375 samples are not significant. The $R_a$ datasets are further analyzed from the boxplots in Figure 5.13 (c) and (d), showing the mean value and standard deviation. It can be seen that the mean values of $R_a$ (marked by the dash lines) slightly increases when the annealing temperature is increased, at both sampling
radii. However, the standard deviations of $R_a$ (the range of the box) cover a wide range so the seen correlation between annealing temperature and $R_a$ is not solid.

Figure 5.13 - Plots showing the $R_a$ datasets for recrystallization boundary segments aligned along RD in the Al50 samples annealed at temperatures of 325°C, 350°C and 375°C. (a) and (b) are histograms of $R_a$ datasets calculated using sampling radii of 1 $\mu$m and 3 $\mu$m, respectively. (c) and (d) are boxplots of the $R_a$ datasets calculated using sampling radii of 1 $\mu$m and 3 $\mu$m, respectively. The boxes incorporate $R_a$ data within the range of standard deviation and the dash lines show the mean value of each dataset.

The increasing roughness of boundary segments observed in the Al50 samples with increasing annealing temperature is unlikely to associate with the thermal roughening as revealed from the studies of MD simulation. The grain boundaries undergo an abrupt thermal roughening at an elevated temperature.
and are separated into smooth (low-mobility) and rough (high-mobility) ones [26,27]. However, the $R_a$ datasets from samples with different annealing temperature are scattered and without any sharp transition. It should also be noted that the rough-boundary structures revealed from the MD simulation are on atomic scale, while the roughness parameter measured for the boundary segments in Al50 samples quantify the roughness on micrometer scale.

Table 5.4 Results from two-sample t-test evaluating the difference of $R_a$ datasets for different annealing temperatures of the Al50 samples (confidence level for rejection of null hypothesis: 0.05)

<table>
<thead>
<tr>
<th>Sampling radius</th>
<th>$1 \mu\text{m}$</th>
<th>$3 \mu\text{m}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R_a$ datasets</td>
<td>325-350</td>
<td>350-375</td>
</tr>
<tr>
<td>Hypothesis test result (h)</td>
<td>1 (rejected)</td>
<td>0 (accepted)</td>
</tr>
<tr>
<td>p-value</td>
<td>0.0161</td>
<td>0.0514</td>
</tr>
</tbody>
</table>

Annealing temperature has a profound influence on the recrystallization kinetics, affecting many aspects in the recrystallization process. It is well known that higher annealing temperature can accelerate the recrystallization process. If taking the time for 50% recrystallization as a measure of the rate of recrystallization, the rate of recrystallization will increase when the annealing temperature is increased, following approximately the Arrhenius relationship, as the example shown in Figure 5.14 [89]. Higher recrystallization rate means that the recrystallized grains growth faster and hence the recrystallization boundaries migrate faster. The larger fraction of boundary segments with higher roughness in the Al50-375 sample may be associated with the larger movement speed of
the recrystallization boundaries. The specific factors that affect the boundary roughening behavior under different annealing temperature are however difficult to investigate based on the present data.

![Graph of Fraction recrystallized vs Time for different annealing temperatures](image)

Figure 5.14 - (a) The effects of annealing temperature on the annealing of Fe-3.5%Si deformed 60%, (b) Arrhenius plot of the time for 50% recrystallization as a function of annealing temperature. This figure is reproduced from [89].

5.7 Summary

The roughening of recrystallization boundaries has been statistically investigated at two length scales, 1 µm and 3 µm. The effects of sample purity, deformation strain, boundary alignment direction and annealing temperatures were analyzed. The major findings are:

1) Recrystallization boundary segments with higher roughness were observed in the high-purity Al than in the commercial-purity Al samples, indicating that the particle pinning effects are not the main reason for recrystallization boundary roughness in the present samples, while the solute atoms in the commercial-purity Al sample may reduce the mobility
of recrystallization boundaries and hence affect their roughening behaviors.

2) The roughening of recrystallization boundary is found to be related to the deformation microstructures. The dislocation boundary structures in front of the recrystallization boundary segments are different for different deformation strains and the boundary segments with higher roughness were observed in the heavily deformed sample. It is thus indicated that the interactions between migrating boundaries and various types of dislocation boundaries may affect the migration of recrystallization boundaries.

3) The boundary segments aligned along ND were found to have higher roughness than those aligned along RD. It is suggested that the rough morphology of recrystallization boundaries might be associated with higher migrating rates.

4) The annealing temperature was found to affect the roughening behavior of recrystallization boundaries in the Al50 sample, in the sense that the boundary roughness increase when the annealing temperature is increased. Although MD simulation have shown a temperature effect leading to grain boundary roughening transition, the present observation does not seem to be explained by this phenomenon.

5) From the parameters analyzed of their effects on boundary roughening, it is found that higher roughness is often associated with higher rate of boundary migration, such as that the boundary segments aligned along ND have higher roughness, and that the boundary segments from sample
with higher annealing temperatures have higher roughness. However, the cause and effect between the migration rate and boundary roughness remain unclear. Further investigations may require experimental evidence from in-situ observation of boundary migration during recrystallization using synchrotron X-ray radiation.
Chapter 6
Characterization of Graphite Nodules in Cast Irons

This chapter presents a new method to quantitatively characterize the morphology of graphite nodules in cast iron. This method uses a morphological variable to obtain the information of local morphological characteristics and then aggregates the local information into a parameter representing the nodule’s morphology as a whole. The method follows similar principles as the All method for characterization of 2-D line features and hence can be considered as an extended application of the method developed and described in Chapter 3. Graphite nodules with different morphologies are characterized to validate the potentials of the new method.

6.1 Introduction

Cast iron is widely used in industries because of several manufacturing and engineering advantages such as low manufacturing cost, good wear resistance and easy fabrication of components with complicated geometries [90]. The term “cast iron” refers not to a single material, but to a family of materials in which the major constituent is iron, with carbon (C) ranging from 1.8-4 wt% and silicon (Si)
1-3 wt% as the main alloying elements. The cast irons are classified into several types based on their microstructures and mechanical properties: grey cast iron has flake-like type of graphite and appears grey on the fractures surfaces; white cast iron is characterized by carbide precipitation and displays white fractured surfaces; ductile cast iron has its graphite in the form of nodules that can stop the crack from further progressing and thus has the best mechanical properties [91].

The ductile cast iron is used here as an example, for a brief introduction of the microstructure evolution process. During solidification of ductile cast iron, inclusions in the melt act as potential nucleation sites for graphite nodules, which are encapsulated by an austenitic shell and grow by solid state diffusion of carbon from the melt to the particle [92]. The final microstructure of ductile cast iron is normally composed of graphite nodules surrounded by ferrite grains.

As an essential phase in cast iron, the graphite plays an important role on the mechanical properties of cast iron. Many aspects of graphite nodules such as the number, size distribution as well as their morphology can affect the properties of ductile cast iron. A sufficient number of graphite nodules are required in order to avoid the formation of carbides during solidification, and the presence of carbides in the final microstructure has detrimental effects on the mechanical properties. The number of graphite nodules influences the ferrite/pearlite content of the matrix and therefore affects the mechanical properties [93, 94]. The size distribution of graphite nodules is also an important parameter when characterizing the microstructure of cast iron. Small graphite nodules, for example, are favorable to the fatigue strength [94,95]. Lots of studies have been carried out to characterize the number and size distribution of graphite nodules...
in cast iron and evaluate the effects of thermomechanical processing parameters on the evolution of graphite nodules [e.g. 93,96,97,98].

Besides the number and size distribution, the shape of graphite nodules also affects the mechanical properties of cast iron. Figure 6.1 shows a typical microstructure of ductile cast iron with presence of graphite nodules of different morphologies. It can be seen that most of the graphite nodules have spherical or nearly spherical shapes whereas nodules with irregular morphologies also exist, as the two examples marked with red rectangles in Figure 6.1. The fracture toughness and ductility of cast iron depend strongly on the shape of the graphite nodules. Nodules with spherical shapes improve these properties whereas those with irregular contours can lead to significant degradation of mechanical properties due to stress concentration points [94]. A parameter termed nodularity is normally used to quantify the fraction of “acceptable” graphite nodules and is calculated by counting the number of graphite nodules that are spherical or nearly spherical divided by the total number of the graphite nodules. Figure 6.2 shows the variation of yield strength, tensile strength and fracture toughness as a function of nodularity [99]: higher nodularity can significantly improve the mechanical properties of cast iron.

The number, size distribution and shape of graphite nodules are all important parameters affecting the mechanical properties of cast iron. In this chapter, a method that quantitatively characterizes the shape of graphite nodule is illustrated.
Figure 6.1 - Typical microstructure of ductile cast iron. Graphite nodules with various shapes can be seen in the matrix. The rectangles mark graphite nodules with irregular morphologies.

Figure 6.2 - (a) Yield strength and (b) tensile strength as a function of nodularity and carbide content. The figure is reproduced from reference [99].
6.2 Shape characterization of graphite nodules

The shape of graphite nodules is not straightforward to measure, compared with the number and size distribution. The main difficulty in shape quantification is the lack of a precise, universal definition of the shape of an object. Intuitively, the shape of an object is described by comparison with another one or through the characteristics of its contour [61, 100]. The characterization of the graphite nodules' shape is normally done through visual comparison of sample cross section of cast iron with standard images or by digital image analysis of the microstructures. The ISO-945 standards include six reference types for qualitatively characterization of the graphite nodules in cast iron based on their different shapes, as seen in Figure 6.3 [101].

Figure 6.3 - Reference images (ISO-945) for the six classes of graphite nodules. The figure is reproduced from reference [101].

The visual comparison with standard images can be subjective and inefficient for industrial applications. In the fields of image analysis and pattern recognition,
parameters termed shape factors, which are sensitive to the change in shape, are normally used for quantitative characterization of objects’ shape. Shape factors are dimensionless having the same value for objects with the same shape but different size. The quantitative description is generated from how far a given shape deviates from a reference one. Most of the shape factors are calculated based on the basic geometrical parameters, such as area, perimeter, diameter, etc. Table 6.1 lists several representative shape factors, showing their definitions and the characteristics of the shapes to which their values are more sensitive [61,102,103]. It should be mentioned that the definitions of these shape factors may be different in different studies and the definitions from reference [61] are adopted in the present study. An example illustrating the form factor is given in Figure 6.4, where five objects with the same area but different shapes are shown. The form factor is 1 for the shape close to a perfect circle and decreases when the contour becomes irregular and the perimeter increases against the area.

The shapes of graphite nodules in cast iron can be characterized using shape factors, as the ideal shape of the graphite nodule is a sphere. Previous studies have however shown that generally more than one shape factors are required to characterize the shape of graphite nodules, because their shapes may simultaneously involve branching, elongation and contour irregularities [98,104].
Table 6.1 Representative examples of Shape Descriptors [61]

<table>
<thead>
<tr>
<th>Name</th>
<th>Definition</th>
<th>Sensitivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Form Factor</td>
<td>$\frac{4 \cdot \pi \cdot A}{p^2}$</td>
<td>Circular shape and contour irregularities</td>
</tr>
<tr>
<td>Roundness</td>
<td>$\frac{4 \cdot A}{\pi \cdot F_{\text{max}}^2}$</td>
<td>Circular shape and elongation</td>
</tr>
<tr>
<td>Aspect Ratio</td>
<td>$\frac{F_{\text{min}}}{F_{\text{max}}}$</td>
<td>Elongation</td>
</tr>
<tr>
<td>Convexity</td>
<td>$\frac{P_c}{P}$</td>
<td>Convex shape and contour irregularity</td>
</tr>
<tr>
<td>Solidity</td>
<td>$\frac{A}{A_c}$</td>
<td>Convex shape, thin and long ramifications</td>
</tr>
</tbody>
</table>

$A$: area, $A_c$: convex area, $P$: perimeter, $P_c$: convex perimeter, $F_{\text{max}}$: maxium Feret, $F_{\text{min}}$: minimum Feret.

Figure 6.4 - Form factor: the shapes have the same area, but the increasing perimeter changes the measured form factor as labeled on the shapes. This figure is reproduced from reference [61].

An example of using shape factors to characterize graphite nodules is shown in Figure 6.5. The shapes for the six types of graphite nodules in the ISO-945 standard (Figure 6.3) are characterized using both the form factor and the aspect ratio. The equations listed in Table 6.1 are used to calculated the two parameters. It can be observed that different types of graphite nodules possess varying combinations of aspect ratio and form factor, but the distribution of each type is rather scattered and substantial overlap exists between several types, e.g. class 2 and class 3, or class 4 and class 5. One reason for the overlap is that both the
form factor and aspect ratio are sensitive to elongation of the shape, especially for class 2 in which the graphite nodules have elongated shape with prominent branches. Moreover, although in each types as classified differently in ISO-945 the graphite nodules have similar shape, this classification is not quantitatively well-established based on the measured results from shape factors. The “outliers” in each type based on the combination of form factor and aspect ratio are marked and shown in Figure 6.6 [105]. It can be seen except the regular type (Class 6), the other five types as classified in the ISO-945 all incorporate graphite nodules from other types, showing disagreed classification from the shape factors.

Figure 6.5 - Aspect ratio against form factor for the six classes (C1-C6) of graphite nodules shown in Figure 6.3. The aspect ratio and form factor are calculated based on the equations listed in Table 6.1.
Figure 6.6 - The unfitted graphite nodules in classification of the ISO-945, examined from the measured values of form factor and aspect ratio. The figure is reproduced from [105].

As discussed above, the shape factors are capable to quantitatively characterize the shape of graphite nodules. However, the shape factors use the sphere as the reference shape and hence are most suitable for graphite nodules with relatively simple shapes. When graphite nodules with complicated local features such as branches, cleavages and voids, as the examples shown in Figure 6.7, are to be characterized, the feasibility of shape factors may be limited. Many shape descriptors are calculated using the perimeter of the contour, which, as mentioned in Section 3.2, often is problematic to measure. Moreover, most of the shape factors are calculated based on the basic geometrical parameters, such as area, perimeter and diameter, which are global parameters generated from the integrated object. In this way, information of local features such as the branches, cleavages and voids is limited.
6.3 New method to characterize the irregularity of graphite nodules

A morphological variable, *dispersion* \((d)\), is developed in the present work to obtain information about the local morphological characteristics, and the local information can subsequently be integrated into a parameter representing the particle’s morphology as a whole. The method is introduced below and applied to the six types of graphite nodules in the ISO-945 standards as well as to images of graphite nodules with different nodularity to validate its potentials.

6.3.1 Calculation of dispersion

First two concepts from the field of image computation used in the calculation of the *dispersion* are introduced.

1) Kernel. In image processing, a kernel is a small matrix of numbers that is used for image blurring, sharpening, edge detection etc. The processing
is accomplished by means of convolution between a kernel and an image. Examples of three commonly used kernels are shown in Figure 6.8 [61].

2) Image convolution. Convolution is the process of multiplying each element of the image with its local neighbors, weighted by the kernel [106]. Figure 6.9 schematically illustrates the image convolution process [107]. Generally, the convolution returns a new matrix with every pixel value containing the information of the original pixel and its neighbors in the original image.

\[
\begin{bmatrix}
-1 & -1 & -1 \\
-1 & 8 & -1 \\
-1 & -1 & -1
\end{bmatrix} \times
\begin{bmatrix}
0 & -1 & 0 \\
-1 & 5 & -1 \\
0 & -1 & 0
\end{bmatrix} =
\begin{bmatrix}
1 & 1 & 1 \\
1 & 1 & 1
\end{bmatrix}
\]

Figure 6.8 - Kernel used for (from left to right): edge detection, image sharpening and image blurring.

Figure 6.9 - Illustration of the convolution process. The image is reproduced from reference [107].
The dispersion value is computed in the following steps, using the graphite nodule in Figure 6.7(a) as the example:

1) The graphite nodule is segmented from the original image and processed to a binary image by setting a threshold to grayscale values of the pixels, as shown in Figure 6.10(a). In the binary image, the pixels representing the graphite nodule have values of 1 while those representing the cast iron matrix have values of 0. The binary image is denoted as \(M_o\).

2) A disk-shape kernel with all pixel values of 1 is used, with a specified radius that is termed “sampling radius” for the disk. Similar with the sampling circle used in the AII method, the disk-shape is employed for direction-independent characterization. The kernel is denoted as \(k_d\) and the total pixel number of the kernel is termed sampling range.

3) Convolution of \(k_d\) and \(M_o\) is computed, returning a new matrix in which each pixel has a value containing the information of the corresponding pixel and its neighbors in the original image of the graphite nodule. The number of neighbors is determined by the sampling radius. The returned matrix is denoted as \(M_c\).

4) The value in every pixel of \(M_c\) is then normalized by a reference number \(C\), which is equal to the total number of pixels in \(k_d\). \(C\) represents the pixel value after convolution of \(k_d\) with all its neighboring pixels within the sampling range belonging to the graphite nodule.

5) After the normalization, the pixels in \(M_c\) have values ranging from 0 to 1. The normalized pixel value at any position in \(M_c\) represents the extent of
dispersion of the graphite particle at that location and is thus termed \textit{dispersion}.

The \textit{dispersion} values at a sampling radius of 50 pixels for the graphite nodule shown in Figure 6.7(a) are plotted as a heat map, see Figure 6.10(b). It can be seen that the regions with voids and cleavages have lower values of \textit{dispersion} whereas the regions where the graphite nodules are more compacted have higher values of \textit{dispersion}. In this way, the extent of “how much” the graphite particle is dispersed at the local scale is quantified. The \textit{dispersion} value of 1 represents that within the sampling range is entirely filled with the graphite nodule, whereas low values are for regions with lots of voids or cleavage or close to the boundary in the nodule.

![Figure 6.10 - (a) Binary image of the segmented graphite nodule shown in Figure 6.7(a). (b) The \textit{dispersion} values calculated with a sampling radius of 50 pixels for the graphite particle in (a).](image)

One may also quantify the global morphology of a graphite nodule based on how the nodule is dispersed locally. The lower the \textit{dispersion} value, the more irregular morphology the graphite particle has. The morphology of graphite nodules can thus be expressed by a \textit{dispersion index} using the following equation:
\[ Dispersion \text{ Index} = \sqrt{\frac{\sum_{i=1}^{n} (d_i - 1)^2}{n}} \]  

Equation 6.1

where \( d_i \) is the dispersion value at every pixel in the graphite nodule and \( n \) is the total number of pixels within the graphite nodule. Larger values of *dispersion index* (DI) are for graphite nodules with irregular morphologies.

### 6.3.2 Effects of sampling range on dispersion index

As the *dispersion* value at a pixel is determined by all of its surrounding pixels within the sampling range, the calculated *dispersion index* not only depends on the local dispersion of the graphite nodules, but also depends on the size of \( k_d \). A parameter characterizing the shape of an object should be independent of the changes of the size when the shape is maintained, so it is expected that the DI values should be similar for the graphite nodules with similar morphology. The DI variation as a function of the size of \( k_d \) has been investigated by calculating the DI values for the graphite nodules of “Class 6- regular nodules”, which has similar shapes close to a sphere, and the results are shown in Figure 6.3.

Two methods were used to decide a proper sampling range: using sampling radius for the disk-kernel and using the ratio of the sampling range with respect to the area of the graphite nodule. As shown in Figure 6.11(a), when using the same sampling radius to calculate the DI values, the DI values exhibit large variation among the graphite nodules. When using a fixed ratio of the sampling range with respect to the area of graphite nodules as the sampling range, as shown in Figure 6.11(b), the DI values of all graphite nodules exhibit similar values. If the sampling radius is used to calculate the DI values, the nodules with smaller areas will return higher values of DI, as revealed from Figure 6.12, which
shows the variation of $DI$ values as a function of the graphite nodules’ areas. For graphite nodules with different areas, the same sampling range will result in a larger fraction of *dispersion* measurements across the boundary in the graphite nodule of smaller area than those of larger area, as the boundary regions are more dominating for smaller graphite nodules. This disparity increases when the sampling range becomes larger, as revealed from Figure 6.11(a). When the $DI$ values are used to quantify the morphology of graphite nodule, the size of $k_d$ will be decided by the ratio of the sampling range with respect to the area of the graphite nodule.

Figure 6.11 - The $DI$ values calculated for the “Class 6-regular nodules” using two methods to determine the sampling range: (a) by sampling radius and (b) by the ratio of sampling range to the area of graphite nodules.
6.3.3 Using DI to characterize the morphology of graphite nodules

The potential of using DI to quantitatively characterize the morphology of graphite nodules was further investigated by calculating the DI values for the six types of graphite nodules in ISO-945 standard classification (Figure 6.3). The sampling range used was set to be 20% of the graphite nodule’s area. Figure 6.13 shows the histograms and data points of DI values for the six types of graphite nodules. Except the regular types of graphite nodules, the other five types all have scattered distributions of DI values. This is a valid result, not affected by the method to calculate DI, and is an effect of shape variations within the individual types. The lamellar type of graphite nodules has the widest distribution of DI values, meaning that graphite nodules with qualitatively similar shapes do not have similar extent of dispersion: those with larger aspect ratio have larger
fraction of boundary regions and thus larger dispersion extent. Inspections of the lamellar graphite nodules show that the smaller ones are of similar morphology with the vermicular graphite nodules, corresponding to those marked in Figure 6.6.

To investigate the relationship between the extent of dispersion and the shape of the graphite nodule, the variations of DI values are further analyzed as a function of form factor for the irregular and lamellar types of graphite nodules, as shown in Figure 6.14. It can be seen that DI and form factor are closely correlated. The DI values decrease monotonically with increasing form factor values. With similar area, the graphite nodules with larger perimeter yield higher values of form factor, whilst larger length of the graphite nodule’s boundary also indicate its larger extent of dispersion in the cast iron matrix.

The regular type of graphite nodules exhibits low DI values. Some of the graphite nodules belonging to the irregular and uncertain also have low DI values, which are from those nodules without prominent branches. The shape of the graphite nodules with low DI values are actually spherical or nearly spherical.

From the calculated DI values, it is also revealed that each type as classified in the ISO-945 is actually a composition of graphite nodules with different shapes, which corresponds to Figure 6.6. The DI values for the graphite nodules with spherical or nearly spherical shape stand distinctively from those with irregular contour. Therefore, if a threshold value of DI is set, this method can be applied to measure the nodularity of an image with many graphite nodules, which, as introduced in Section 6.1, is an important parameter to characterize the quality of cast iron.
Figure 6.13 - The calculated $DI$ values for the six types of graphite nodules listed in ISO-945 standard classification. The sampling range used is determined as 20% of the graphite nodule’s area.

Figure 6.14 - $DI$ values as a function of form factor for the lamellar and irregular types of graphite nodules.

Five images of graphite nodules, as shown in Figure 6.15 [108], were used to test the potentials of $DI$ to measure nodularity. Each of the five images has 100 graphite nodules but different nodularity: the nominal nodularity of M1-M5 is 60%,
70%, 80%, 90% and 100%, respectively, as counted based on the shapes of graphite nodules.

The $DI$ values for the graphite nodules in each map were calculated using a sampling range of 20% of the graphite nodule’s area and the results are shown in Figure 6.15. Apparently, the $DI$ distribution becomes more concentrated in the smaller value range when moving from M1 to M5. The threshold value to separate spherical or nearly spherical nodules from other ones was set at 0.3, and the nodularity is calculated as the graphite nodules with $DI$ value smaller than the threshold value divided by the total number of graphite nodules in each map. The result is shown in Table 6.2. It can be seen that the measured nodularity from $DI$ values coincide well with the nominal one, so the applicability of using $DI$ values to measure the nodularity is validated.

Figure 6.15 - Maps of graphite nodules with different nodularity. Each map has 100 graphite nodules and the nominal nodularity is 60%, 70%, 80%, 90% and 100%, respectively. The figure is reproduced from reference [105].
Figure 6.16 - The measured DI values for the graphite nodules in the five maps (Figure 6.17) using sampling range as 20% of the graphite particle’s area.

<table>
<thead>
<tr>
<th>Table 6.2 Measured Nodularity using DI values.</th>
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<tbody>
<tr>
<td>Map</td>
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<tr>
<td>Nominal Nodularity</td>
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<tr>
<td>Measured Nodularity</td>
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</table>

6.4 Summary

A new method is developed to quantitatively characterize the morphology of graphite nodules in cast iron. The method employs a morphological variable, dispersion, to obtain the information of local morphological characteristics. Then the local information can be merged into a parameter “dispersion index”, to represent the graphite nodule’s morphology as a whole. Compared with the commonly used shape factors for characterization of graphite nodules, the dispersion method has several advantages:

1) Dispersion is a local morphological variable that does not require the measurement of geometric parameters such as perimeter and diameter, which may depend on the spatial resolution of the image. Graphite
nODULES WITH COMPLICATED SHAPES CONTAINING BRANCHES, VOIDS AND CLEAVAGES CAN BE EASILY CHARACTERIZED.

2) THE CALCULATION OF DISPERSION IS BASED ON IMAGE CONVOLUTION, WHICH IS BASED ON THE PIXELS COMPOSING THE KERNEL AND THE GRAPHITE NODULE ITSELF, SO DIGITALIZATION EFFECTS SUCH AS DISCRETE PIXEL REPRESENTATION IS MINIMIZED. BESIDES, THERE IS NO STRICT DEMAND ON THE MAGNIFICATION OR PIXEL SIZE OF THE IMAGED GRAPHITE NODULES.

EFFECTS OF SAMPLING RANGE IN CALCULATING DISPERSION HAVE BEEN DISCUSSED AND THE PRINCIPLE TO SELECT A PROPER SAMPLING RANGE HAS BEEN SUGGESTED. THE METHOD HAS ALSO BEEN VALIDATED FOR DIFFERENT TYPES OF GRAPHITE NODULES AS WELL AS BY MEASUREMENT OF THE NODULARITY OF A GIVEN IMAGE WITH MANY GRAPHITE NODULES.
Chapter 7
Conclusions and Outlook

Microstructures may contain various rough features. This thesis has focused on the roughening of recrystallization boundaries in the partly recrystallized states. Many factors may be important for the roughening of the recrystallization boundaries. Also the roughening has been shown to be important for the boundary migration rate. Yet no method to quantitatively characterize the roughness was available prior to the current study. A new method is presented in the thesis for quantitative characterization of 2-D line features. The area integral invariant (AII) is employed as a morphological variable to obtain information of local structural variations such as protrusions and retrusions formed on recrystallization boundaries. The AII value is direction-independent allowing unbiased characterization of the irregular line features with both closed and non-closed boundary profiles. The length scale at which the rough features are characterized is determined by a parameter termed sampling radius used to measure the AII values. It is shown that the sampling radius has to be chosen with care to quantify the roughness at relevant length scales. A number of roughness parameters are developed based on the AII dataset for a whole boundary or boundary segment. Thereby the local morphological characteristics
can be converted to a global parameter describing the roughness of the boundary or boundary segment.

With the boundary roughness quantified, the roughening behaviors of a large number of recrystallization boundaries are statistically analyzed and the effects of several parameters: materials purity, deformation strain, annealing temperature and boundary alignment direction are evaluated.

The effects of these parameters on boundary roughening are investigated at two length scales: 1 µm and 3 µm. The boundary segments in the high-purity Al are revealed to have higher roughness than those in the commercial-purity Al samples, showing that particle pinning effects are not the main reason for recrystallization boundary roughness in the present samples, while the solute atoms in the commercial-purity Al sample may reduce the mobility of recrystallization boundaries and hence affect their roughening behaviors. The roughening of recrystallization boundary is found to be related to the deformation microstructure. The dislocation boundary structures in front of the recrystallization boundary segments are different as from different deformations and the boundary segments with higher roughness are observed in the heavily deformed sample. The boundary segments aligned along ND are found to have higher roughness than those aligned along RD. The boundary segment aligned along ND move faster during recrystallization and it is suggested that the rough morphology of the recrystallization boundaries may be associated with the higher migrating rates. The annealing temperature is also found to affect the roughening behavior, in the sense that the boundary roughness increases when the annealing temperature is increased. Higher annealing temperature accelerates recrystallization process
and thus the recrystallization boundaries migrate faster at higher annealing temperature. The roughening behavior revealed in the present study cannot be explained by the thermal roughening transition reported from MD simulations, as no abrupt roughening transition is observed and the boundary roughness exceeds the length scale of several atoms. From the results in the present work, it is found that a higher migration rate often is associated with higher roughness of recrystallization boundaries, and the roughening behavior is more predominant at smaller length scale (1 µm) than larger length scale (3 µm). The latter match well with the scale of deformation induced subdivision in the matrix surrounding the recrystallizing grains.

The results of present study have documented that recrystallization boundaries in general are rough and therefore the roughness has to be taken into account when analyzing recrystallization boundary migration. Further work has led to a better understanding of the boundary roughening behavior during recrystallization, by the statistical investigation of the parameters affecting recrystallization boundary roughness. Future experimental work and theoretical modeling can be designed to selectively take one or more parameters into consideration and investigate the specific factors that influence the roughening of recrystallization boundary. For example, the roughness of recrystallization boundary segments in samples with increasing amount of solutes can be compared to determine the effect of solute atoms. The deformation matrix in front of recrystallization boundary can be analyzed in detail to investigate the interactions between recrystallization boundaries with different types of dislocation boundaries. Further investigation into the boundary migration during
recrystallization shall include in-situ observation of the boundary migration process with good spatial and temporal resolution, so the roughening transition of boundaries can be observed with more details. Since the grain boundary is a 3D feature and the boundary migration is a dynamic process, the roughening behaviors characterized from 2D static images are not sufficient to cover all the aspects. High resolution synchrotron X-ray measurements would be an appealing supplement. Furthermore, the experiments should be combined with theoretical modeling. Phase-field modeling can incorporate grain boundaries with varying mobility to investigate the response of boundaries with different mobilities to a heterogeneous stored energy, as it has been revealed from the present work that the roughening of recrystallization boundary is often associated with faster boundary migration.

The ideas of AII method, in particular the possibility to combine local and global information, are applied to quantitatively characterize the morphology of graphite nodules in ductile cast iron. Another new method is developed whereby a morphological variable “dispersion” is suggested to obtain information about local morphological characteristics that is subsequently merged into a parameter termed dispersion index, to represent the nodule’s morphology as a whole. The method follows similar principles as the AII method for characterization of 2D line features and hence can be considered as an extended part of the method development for characterization of irregular features in microstructures. The dispersion index can be used to quantitatively characterize the graphite nodules with complicated morphology as well as measure the nodularity of an image with many graphite nodules. In future work, it is suggested that the method can be
further developed for quantitative characterization of other irregular morphological features often seen in microstructure, such as the fracture surfaces or the clustering of particles.
Reference


[108] The images are acquired from Dr. Karl Martine Pederson through personal communication. Dr. Pederson is a metallurgist in Siemens Wind Power, Denmark.
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Vi har mere end 240 ansatte og heraf er ca. 60 ph.d. studerende. Forskningen tager udgangspunkt i ni forskningsprogrammer, der er organiseret i tre hovedgrupper: vindenergisystemer, vindmølleteknologi og grundlag for vindenergi.