Grain interaction in local plastic deformation of welded structural steel

Influence of length scale on sub-grain deformation behaviour for polycrystalline BCC material

Pauli Lehto
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Abstract

Polycrystalline body-centred cubic (BCC) steel is the most commonly used structural material in the transportation industry. In order to optimize sustainable use of steel materials in welded structures such as ships, better fundamental understanding of the factors affecting material behaviour is required in different length scales. This thesis studies the length scale interface between microstructural features and continuum scale deformation in BCC steel materials. Special attention is paid on characterization of microstructure and local plastic deformation in heterogeneous steel weld metals.

In this work, microstructural characteristics are correlated with plastic deformation response, and the fundamental deformation mechanisms are resolved using electron backscatter diffraction. The local deformation process is characterized for welded steel using instrumented indentation testing in different length scales, ranging from a fraction of one grain to tens of grains. A serial sectioning procedure is developed in order to consider the stochastics of the plastic deformation process in heterogeneous microstructures. Furthermore, advanced orientation data post-processing and misorientation analysis are utilized to identify the plastic deformation zone and formation of dislocation cells beneath hardness indentations.

The results of the thesis reveal the importance of grain size dispersion to mechanical properties, and the significance of grain interactions for the plastic deformation process in polycrystalline BCC steel. Comparison between base and weld metal revealed that the size of the plastic deformation zone is proportional to the average grain size. A transition region was defined between continuum and single crystal material behaviour, where the interaction of grains of different size controls the local plastic deformation of polycrystalline steel. The strongest influence grain interaction on hardness variation was found to take place at indentation diagonal lengths $0.1 - 2d_v$, when slip transmission primarily occurs between two grains.

When the size of the plastic deformation zone is considerably larger than the average grain size, spatial hardness variation decreases significantly. In this regime, hardness and strength are affected by average grain size and grain size dispersion. To consider these aspects, a modified Hall-Petch relationship is introduced utilizing the volume-weighted average grain size based on the rule of mixtures. The modified Hall-Petch relationship is validated with literature data, showing its applicability to a wide range of materials that show grain size dependent mechanical properties.

Keywords  grain size, local plastic deformation, Hall-Petch relationship, heterogeneous polycrystalline material, EBSD
Tiivistelmä


Työn tulokset osoittavat, että kidekokojakauma vaikuttaa huomattavasti hitsatun teräksen lujuteen ja plastisen deformaatioalueen lujauksen osoitetaan riippuvaiseksi teräksen keskimääräisestä kidekoosta. Lisäksi eri kokoisten kiteiden vuorovaikutuksen vaikutus potkuleikkausten, jotta deformaatioprosessin tilastollinen vaihtelu pystytään määrittämään. Kideorientaatioanalyysiä varten kehitetään jälkikäsittely- ja analysointimenetelmiä, joilla paikalliset deformaatioalueet voidaan määrittää kiteeseen muodostuvien alirakenteiden perusteella.


Avainsanat kidekoko, paikallinen plastinen deformaatio, Hall-Petch yhtälö, heterogeeneinen monikiteinen materiaali, EBSD
Preface

The research presented in this thesis was carried out in the Marine Technology group of Aalto University’s Department of Mechanical Engineering. The research work was funded by multiple parties: DIMECC BSA (Breakthrough Steels and Applications), DIMECC Breakthrough Materials Doctoral School, Finnish Academy of Science (FASA 261286, mFAT 298762), Aalto University School of Engineering, and the Finnish Maritime Foundation. All the financial support is gratefully acknowledged.

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Due to the cross-disciplinary nature of the thesis, collaboration was fundamental to the successful completion of the thesis. I want to thank Professor emeritus Hannu Hänninen from engineering materials for his support to the initial research work carried out in 2012-2014. I also want to thank Professor Sven Bossuyt for many discussions and suggestions that shaped the direction of the thesis. I am grateful to D.Sc. Teemu Sarikka for carrying out EBSD acquisition, and always having time to discuss EBSD analysis and challenges in sample preparation. Thanks are also in order to D.Sc. Roman Mougnot, who carried out part of the scanning electron microscopy. I also want to thank staff members Kim Widell and Laura Tiainen for their continued support in practical matters.

I want to thank my family for the encouragement and seemingly unlimited support to study what I find interesting. Finally, my partner Annica, you give the meaning to my life.

Espoo, 6th of June 2019 (8th of October 2019)
Pauli Lehto
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List of Abbreviations and Symbols

( ) Miller indices for a single plane, e.g. (1 1 0)
[ ] Miller indices for a single direction, e.g. [1 1 1]
{ } Miller indices for a set of planes, e.g. {1 1 0}
< > Miller Indices for a set of directions, e.g. <1 1 1>
Δd/d Relative grain size dispersion
A₀(h) Indenter contact surface area as a function of indentation depth
b Burgers vector
d Average grain size
dᵥ Volume-weighted average grain size
h Displacement in indentation test
Tᵥ Critical temperature for dislocation motion

AF Acicular ferrite
ASTM American Society of Testing and Materials
BCC Body-centred cubic crystal lattice
BM Base metal
BSD Backscatter detector
CG Coarse grain
CT Computed tomography
CV Conventional arc welding
CV Co-efficient of variation in statistical analysis
DBTT Ductile-to-brittle transition temperature
DC Dislocation cell
DDW Dense dislocation wall
DT Dislocation tangle
EBSD Electron backscatter diffraction
EBSP Electron backscatter diffraction pattern
FC Ferrite carbide aggregate
FCC Face-centred cubic crystal lattice
FG Fine grained
FIB Focused ion beam
FSD Forescatter detector
<table>
<thead>
<tr>
<th>Acronym</th>
<th>Term</th>
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<tbody>
<tr>
<td>GB</td>
<td>Grain boundary</td>
</tr>
<tr>
<td>GRZ</td>
<td>Grain refined zone</td>
</tr>
<tr>
<td>HAGB</td>
<td>High angle grain boundary</td>
</tr>
<tr>
<td>HM</td>
<td>Martens hardness</td>
</tr>
<tr>
<td>HR-EBSD</td>
<td>High resolution electron backscatter diffraction</td>
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<tr>
<td>HV1</td>
<td>Vickers hardness, number shows test load in kg</td>
</tr>
<tr>
<td>HY</td>
<td>Laser-hybrid welded joint</td>
</tr>
<tr>
<td>IIW</td>
<td>International institute of welding</td>
</tr>
<tr>
<td>IPF</td>
<td>Inverse pole figure</td>
</tr>
<tr>
<td>KAM</td>
<td>Kernel average misorientation</td>
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<td>KMM</td>
<td>Kernel median misorientation</td>
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<tr>
<td>LA</td>
<td>Laser welded joint</td>
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<tr>
<td>LAGB</td>
<td>Low angle grain boundary</td>
</tr>
<tr>
<td>M</td>
<td>Martensite</td>
</tr>
<tr>
<td>MRSSP</td>
<td>Maximum resolved shear stress plane</td>
</tr>
<tr>
<td>nn</td>
<td>Nearest neighbours in misorientation analysis</td>
</tr>
<tr>
<td>P</td>
<td>Pearlite</td>
</tr>
<tr>
<td>PDZ</td>
<td>Plastic deformation zone</td>
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<tr>
<td>PF</td>
<td>Pole figure</td>
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<tr>
<td>PF</td>
<td>Primary ferrite</td>
</tr>
<tr>
<td>RVE</td>
<td>Representative volume element</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscope</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron microscope</td>
</tr>
<tr>
<td>WM</td>
<td>Weld metal</td>
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Welding is the most commonly used manufacturing method of steel structures in various applications, for example cars, ships and bridges. In order to guarantee structural safety, the influence of the welding process on material properties has to be understood. In particular, the metallurgy of the steel can transform during the welding process, leading to the degradation of the mechanical properties. Such metallurgical factors have caused serious accidents and loss of life in the 20th century. This thesis studies the relationships between the material microstructure and mechanical properties. Particular attention is given to the characterisation of the grain size dispersion and the local plastic deformation process for polycrystalline structural steel. The following features of this thesis are believed to be original:

1. Microstructural characterisation methods suitable for heterogeneous granular microstructures are developed and applied to welded steel on micro and macroscopic scales. The significance of grain size dispersion for macroscopic mechanical properties is shown and characteristic metrics for the description of grain size variation defined. The volume-weighted average grain size is found to capture the influence of grain size dispersion on macroscopic properties and a modified Hall-Petch relationship is formulated for the prediction of grain size dependent mechanical properties.

2. The deformation behaviour of welded steel with varying grain size dispersion is investigated experimentally at different plastic deformation zone sizes relative to grain size. A robust measurement methodology suitable for the detailed characterisation of local plastic deformation in polycrystalline material is developed, utilising cross-sectioned hardness indentations. Deformation mechanisms are revealed for complex heterogeneous microstructures, including the formation of sub-grain boundaries and dislocations cells. The size of the plastic deformation zone is determined experimentally on the basis of the formation of grain sub-structures, and found to be proportional to the volume-weighted average grain size.
3. A grain interaction regime is defined for welded structural steel, in which grain interaction has the largest influence on the local plastic deformation. The length scale of grain interaction is dependent on grain size dispersion, with the radius of the plastic deformation zone 0.1-2 times the volume-weighted average grain size in the interaction regime. When deformation happens above this length scale, spatial grain size variation and overall grain size dispersion are the key factors affecting the strength of the material.

4. The deformation of steel through dislocation-mediated plasticity is captured using electron backscatter diffraction. Active slip direction and dislocation motion directions are revealed using an innovative misorientation analysis. The results give insight into the material flow induced by screw dislocations and show the large number of slip systems that are activated. In addition, the dislocation cell size variation inside a grain is linked to the theoretical stress distribution of a screw dislocation.
Author’s contribution

The Author has carried out the research presented in this doctoral dissertation independently. The thesis supervisor and advisor have contributed by providing constructive feedback and helped define the scope and extent of research. The materials for the experiments have been defined in co-operation with the thesis supervisor. The Author has carried out all sample preparation and developed the hardness indentation cross-sectioning methodology. Acquisition of crystallographic orientation data has been carried out in co-operation with laboratory engineers and other staff members. All data analysis as well as development of characterization methods have been carried out solely by the Author. The grain size measurement methods, presented in Chapters 3.1 – 3.3, have been published in two co-authored journal articles, with the doctoral candidate as the first author. The doctoral dissertation extends these publications, explaining the observed correlations through extensive microstructural analysis of the plastic deformation process in welded structural steel.

As the supervisor of this dissertation, I hereby confirm that the author's contribution is valid and as presented in this chapter.

Professor Heikki Remes
1. Introduction

1.1 Background

Our current knowledge of material behaviour has been shaped by unexpected structural failures in various engineering applications. For example, the tragic events that befell the RMS Titanic on April 14th 1912 have been thoroughly investigated to understand the reasons that caused the “unsinkable ship” to lose its structural integrity and sink. As the hull of the ship sideswiped an iceberg, an approximately 100 metre section of the hull (Figure 1-1) was damaged, allowing six watertight compartments to flood [1]. Metallurgical investigations [2–4] indicate that the mechanical properties of the steel contributed to the extent of the damage to the hull and the consequent flooding. As a result, the entire ship submerged beneath the surface only two hours and forty minutes after the collision [2], a fraction of the time that it was claimed she would remain afloat (2-3 days) according to the designers [1].

Analysis of the mechanical properties of the steel hull showed good plastic deformation capability at room temperature [2], having 29% elongation to fracture and a tensile strength of 417 MPa. However, the fracture toughness of the steel at low temperatures was very poor, as shown in Figure 1-2A. While for a
ASTM A36 shipbuilding steel the ductile to brittle fracture transition temperature was approximately -15°C (at 27 J), the hull plates from the Titanic have transition temperatures of 40 °C and 70 °C, for the longitudinal and transverse specimens, respectively [3]. At the time of the collision the temperature of the seawater was -2 °C, and thus it is apparent that the steel plate used for the hull was not suitable for service at these low temperatures [2,3]. The plates recovered from the wreckage demonstrated a brittle fracture appearance, with very little plastic deformation having taken place [1,6]. The sister ship RMS Olympic displayed similar damage characteristics one year earlier when she collided with the Royal Navy cruiser HMS Hawke [3].

![Figure 1-2](image-url) Measured toughness properties of the steel used in the Titanic in comparison to an A36 shipbuilding steel, showing A) the energy absorbed by the fracture as a function of temperature, B) the proportion of ductile shear fracture, representing plastic deformation. Reproduced after [2].

Analysis of the metallurgy of the steel revealed several aspects contributing to the observed brittle behaviour in cold temperatures. As shown in Figure 1-3, the steel used in the Titanic has a banded microstructure (A, B), with an average grain diameter of 60 µm and 42 µm for the longitudinal and transverse sections, respectively [2]. The grain size is coarse in comparison to the tested A36 steel (C), which has a mean grain diameter of 26 µm. Note that the magnification is five times larger for the A36 steel. Furthermore, current generation D36 grade steels have an even smaller mean grain diameter of 4 – 10 µm [7]. In addition, the steel was found to have a relatively high amount of phosphorous, oxygen and sulphur in relation to manganese, which causes embrittlement at low temperatures [2]. The large grain size, and presence of large lenticular MnS particles (up to 20 x 5 µm in size) contributed to the poor toughness at cold temperatures [2], limiting the amount of plastic deformation, as shown in Figure 1-2B.
The metallurgical reason for the tragic accident that befell the Titanic is that the ductile-to-brittle transition behaviour of steel at low temperatures was not known. During that time the evaluation of mechanical properties was based on tensile test results, providing yield and ultimate strengths and elongation to fracture [4]. The testing methodology for investigating material fracture had been introduced in the late 1890’s by S.B Russell [8] and further developed by Georges Charpy in 1901 [9,10]. Charpy’s work on steel was carried out at room temperature and above, and thus the brittleness of steel at cold temperatures was not yet discovered. The American Society for Testing and Materials (ASTM) issued Charpy’s method as a standard test method in 1933. Since then the Charpy test has been used to the quantify ductile-to-brittle transition behaviour of steel in multiple fields of industry.

In the ship building industry a large amount of material joining is required in the construction of the large structures. Welding has been the primary joining method for steel structures since World War II, thanks to its versatility and relative cheapness [11]. However, welding can have a detrimental effect on the mechanical properties of steel, as was demonstrated by the World War II ships that suffered catastrophic brittle fractures; see Figure 1-4. Therefore, the weldability of the material is of key importance to structural safety.
The investigations of the Ship Structure Committee (SSC) revealed that most of the World War II failures happened in a cold wintertime environment [14]. A poor ductile-to-brittle transition behaviour of welded steel was found to be the main reason; high ductile-to-brittle transition temperatures (DBTT) were measured for the joints of the fractured ships [15,16]. Similar steels had been used successfully before in riveted constructions, however, the detrimental influence of welding was not known in this early adoption phase of welding. The geometrical and metallurgical discontinuities imposed by poor quality welds acted as stress concentrators [17], initiating brittle failures.

These failures of the World War II Liberty ships were analysed from a metallurgical point of view by Barr and Tipper in 1947 [18]. They showed that the DBTT of structural steel increased when the grain size was increased. The importance of grain size to the brittle fracture (cleavage) of steel was also investigated by Petch during 1946-1949, founded on a theory where yielding and cleavage are dependent on the stress concentration in front of a blocked slip band [19]. The work of Petch showing the relationship between grain size and cleavage strength was published in 1953 [19]. At the same time Hall was independently carrying out research focused on the yield strength of mild steel, formulating the relationship between grain size and yield strength in 1951 [20,21] on the basis of the Eshelby-Frank-Nabarro dislocation pile-up model [22]. Because of their contributions the grain size dependence of mechanical properties has been named the Hall-Petch relationship. On the basis of these investigations, a large amount of experimental work has been carried out during the six decades since then in order to optimise the mechanical properties of steels and other metals by controlling the grain size, see e.g. [23–28].

For shipbuilding applications it is imperative to use materials with high toughness and low DBTT, both in the as-received and welded conditions [29]. To date, fusion welding remains the primary method used to join ship structures. While conventional arc welding techniques are still widely used in shipbuilding, new joining technologies such as laser and laser-hybrid welding techniques have been introduced during the last 20 years in order better to utilise the properties of a base metal in a final product [30]. The shipbuilding industry was a pioneer in adopting these new welding techniques for the manufacturing of large steel structures [30]. The first application was a laser-stake welded sandwich panel in 1995 by Meyer Werft (Germany), with an application to passenger decks in 1998 [30]. To date, laser or laser-hybrid welding capabilities have been installed at most major European shipyards [30]. The utilisation of modern welding technology, together with stronger materials, has a significant weight-saving potential [31,32], improving the energy efficiency of ships and thus reducing CO₂ emissions. To ensure structural safety, new welding techniques and stronger materials need to be tested experimentally and the factors influencing strength understood; see Figure 1-5.
Figure 1-5. Influence of high-quality production on the fatigue strength of steel, showing the fatigue life of A) laser-welded 355 MPa steel [33], B) different (non-welded) steel grades after the shipyard production process [32]. The design curves for steel butt welds and base metal (FAT90, FAT160) represent the current design guidelines for conventional production methods.

The mechanical properties of steel, especially fatigue and fracture toughness, are susceptible to significant deterioration in the manufacturing process. Therefore the manufacturing processes used, including e.g. cutting and welding, have to be optimised for the steel grade used. Fatigue strength of steel is known to be reduced by small notches, cracks and imperfections; see e.g. Kitagawa and Takahashi [34]. The influence of notches and manufacturing imperfections on fatigue strength becomes more severe as steel strength increases [35–38]. As fatigue and fracture are known to be affected by stress and strain gradients, the geometrical discontinuities at e.g. welded joints have a detrimental effect on these properties. Furthermore, as the stresses and thus plastic deformations are highly localised, the local properties of the material at these locations play a significant role in the strength of the entire structure.

The structural design of complex structures requires a holistic strength and response analysis. In large thin-walled structures such as the ship hull-girder, homogenization of structural elements is required to enable assessment of the load-carrying mechanism. Homogenization can be based on a geometrical ‘representative volume element’ (RVE) [39] or a ‘statistical volume element’ (SVE) [40,41]. The geometrical RVE is defined as the smallest possible volume that is able to describe the microstructural effects on the macroscopic scale correctly. For example, the web-spacing (s) in a welded steel sandwich panel defines the geometrical RVE [42,43], as shown in Figure 1-6. Mathematically, the assumption is valid only asymptotically, i.e. when consecutive length-scales are far apart [39]. For statistical homogenization, the process has converged when e.g. mechanical properties no longer change as RVE size is increased [40].
Figure 1-6. The primary, secondary and tertiary length scales associated with ship design as defined by [44], and examples of the characteristic geometric length parameters adapted from [45–49].

The engineering tools for strength analysis are based on continuum mechanics, in which an assumption of material homogeneity within the computational volume is made [50]. In ship design the homogenisation of material properties is dependent on the length scale being investigated and the definition of stress and strain. For ship design these definitions are nominal stress, structural stress, and notch stress and strain, from the largest to the smallest length scale shown in Figure 1-6. Corresponding strength values are based on the statistics related to the investigated volume, representing e.g. the average value and standard deviation. The notch stress and strain analysis can consider the influence of e.g. weld geometry or the surface profile of the steel on structural design. However, in order to consider microstructural effects in continuum modelling, knowledge of material gradients within the calculation volume (RVE) is required. This is particularly important for welded joints with large geometrical variation and significant material gradients. In order to consider these aspects a better description of material heterogeneity is needed [51,52].

In welded ship structures the base metals are typically homogenous on the plate thickness scale. Welds can be either homo- or heterogeneous on the length scale of weld dimensions, and heterogeneous within different regions of the weld. Typically, the macroscopic material properties vary smoothly across the welded joint, from the base metal through the heat-affected zone to the pure weld metal [53]. Inside smaller regions of the weld the material properties fluctuate as a
function of the averaging length scale used \([35, 46, 51, 52, 54]\). When the gradient of mechanical properties from the base metal to the weld metal is large, the average properties and especially the peak values will become strongly dependent on the averaging length. Current ship design methods do not account for these microstructural effects. For instance, fatigue strength is assumed to be independent of microstructure and material grade in several design codes and guidelines \([55]\). While neglecting the material effects makes the design process easier, the full potential of stronger materials cannot be utilised in engineering applications.

In this thesis the aim is to extend the response analysis (see Figure 1-6) with the microstructural length scale. The aim of this work is to investigate the influence of the microstructure on the localised plastic deformation commonly observed for fatigue and fracture phenomena. The focus is on the geometrical and statistical homogenisation of welds on the microstructural length scale, and the work investigates how the selection of the RVE influences the characterisation of microstructural heterogeneity and mechanical properties. Geometrical effects are investigated by characterising the deformation of a welded detail at the level of individual grains, between grains (interaction), and over several grains. Statistical properties are derived on these length scales and linked to physical deformation phenomena. Macroscopic, continuum-scale strength properties are related to the geometric characteristics of the microstructure and their statistically converged values.

1.2 State of the art

The classical approach to the fatigue and fracture assessment of steel is based on the work of Griffith \([56]\), published in 1921 for brittle fractures and extended to plastic deformation by Orowan \([57]\) in 1950. The quantitative description of plastic deformation at the tip of a crack was formulated by Irwin \([58]\) in 1961, determining the approximate size of the plastic zone ahead of the crack tip on the basis of the stress intensity factor. Toughness is a manifestation of the ability of the plastic zone to absorb energy, highlighting the importance of localised deformation to macroscopic material behaviour \([59]\).

The classical theories for the fatigue and fracture assessment of steel structures are based on homogeneous isotropic material properties without explicit consideration of microstructural effects. The effect of microstructure is often introduced through size effect relationships, such as the Hall-Petch relationship between the strength of the material and grain size. In heterogeneous material systems such as welded joints these two assumptions become questionable. The small-scale yielding relevant for fatigue means that plasticity is focused onto a small material volume, the mechanical properties of which can show significant variation. Grain size statistics do not converge in this small material volume, making the applicability of the Hall-Petch relationship inaccurate. This is particularly important when the size of the flaw, i.e. notch or crack, is of the same order of magnitude as the grain size. Several investigations \([60–66]\) have
highlighted the fact that the microstructural characteristics need to be considered in order to provide better predictive capability of experimental results. Accurate description of the microstructural heterogeneity is also required for the mesoscale modelling of material behaviour [67]. Grain size is one of the fundamental geometric microstructural characteristics that need to be considered. Because of its stochastic nature the use of single-valued homogenisation can be problematic, especially for heterogeneous materials in which the spread of grain size is very broad.

It has been shown that the single-valued average grain size (arithmetic mean), as used by Hall and Petch, does not describe the plastic deformation process for a heterogeneous microstructure with different relative grain size dispersions [65,68]. Relative grain size dispersion is defined as the spread of grain size divided by the average grain size. The yield strength of a material has been found to decrease as the relative grain size dispersion broadens for a fixed average grain size [65,68]. Berbenni [68] studied the mechanisms of deformation for heterogeneous materials, showing that stresses and strains distribute unevenly for broad grain size dispersions. The localisation of plastic strain takes place in large grains (Figure 1-7A), with increasing strain heterogeneity for small average grain sizes and broad dispersions (Figure 1-7B). While large grains carry the highest plastic strains, the stresses are higher for small grains, especially when the macroscopic strain increases; see Figure 1-7C. As the localisation of the deformation takes place for the broad grain size dispersions, elastic energy is stored in the crystal lattice [68].

![Figure 1-7](image-url)

**Figure 1-7.** Evolution of plastic strain of grains for A) coarse grain size and narrow dispersion ($ΔD/D=1$) and B) fine grain size and broad dispersion ($ΔD/D=5$). C) Grain average stress as a function of grain size for macroscopic strains $Ε$ of 1% and 10% for the microstructure shown in B. Adapted from [68].

To consider the grain size dispersion more accurately, Kurzydlowski [69] investigated the distribution of log-normal grain volumes analytically. He introduced the concept of considering the volume-weighted grain size distribution in order to predict the strength of heterogeneous polycrystalline materials. A similar approach was utilised by Masumura [62], also incorporating a coble creep deformation mechanism for nanometre-sized grains to consider the applicability of the Hall-Petch relationship only at grain sizes larger than ~100 nm. Good agreement was found for the strength of multiple polycrystalline materials with fine grain size.
Raeisinia [70] expanded on the work of Kurzydlowski, proposing a geometric grain size parameter for lognormal grain size distributions. The representative grain size ($D_R$) was effective in capturing the influence of grain size dispersion for the simulated strength of heterogeneous polycrystalline materials. The fundamental assumptions of this approach are that all grains have the same shape and that the grain size distribution is log-normal. The same assumptions have also been used in various numerical simulations of fictitious grain size distributions [66,71–74]. In addition, the previous studies [60–66,71,74] are focused on single phase base metals and do not cover heterogeneous weld metals. Therefore experimental observations are required for heterogeneous materials in general, on length scales ranging from dislocation substructures to macroscopic response in order to link the plastic deformation behaviour with the geometric characteristics of the microstructure.

1.2.1 Dislocation-mediated plasticity

Plastic deformation of material creates dislocation motion so that the crystal lattice can accommodate the plastic strain. As a result of the individual dislocations the lattice microstrain increases and the lattice dislocations start to re-arrange to reduce the strain, eventually forming a refined grain structure. This deformation process can be divided into six steps according to the work by Tao et al. [75] on pure iron; see Figure 1-8. In the first deformation step dense dislocation walls (DDWs) and dislocation tangles (DTs) are formed inside the grains. As a result of the interaction of multiple slip systems [75], the DDWs and DTs transform into sub-grain boundaries with small misorientation angles. Individual dislocation cells are formed, the size of which is proportional to the shear stress that is acting on the slip plane [76]. With continued deformation in step 2, annihilation and rearrangement of the dislocations occurs, creating new sub-grain boundaries with a higher misorientation angle compared to the DDWs. In the third step the sub-grain boundaries become highly misoriented grain boundaries, forming new grains. This process is enabled by further accumulation and annihilation of dislocations in the grain boundaries. In steps 4 and 5, the previous process i.e. steps 1-3 is repeated inside the newly formed grains, further refining the microstructure. The level of refinement is proportional strain that is applied; however, the sub-grain size will stop to refine as dislocation annihilation rate equals the dislocation multiplication rate. In cases where the deformation is limited to a small material volume, the formation of new high angle boundaries should be minimal, making it easier to distinguish the original and deformation induced boundaries.
Figure 1-8. Grain subdivision process, where lattice dislocations rearrange to form dislocation tangles (DT) and dense dislocation walls (DDW). As deformation continues, dislocation density increases, and new sub-grain and grain boundaries are formed. With sufficient plastic deformation the process is repeated in the newly formed grains, further refining the grain size. Figure adapted from [77], reproduced from data originally published by Tao et al. [75].

The formation of grain sub-structures is a result of dislocation motion in the atomic lattice. The motion of dislocations is governed by the dislocation type, categorised as edge, screw or mixed dislocations [78]. The direction of dislocation motion is illustrated in Figure 1-9A&B for edge and screw dislocations. The Burgers vector represents the magnitude and direction of lattice distortion induced by the dislocation. In an edge dislocation an extra half-plane of atoms is moving through the crystal lattice. While edge dislocations move parallel to the Burgers vector (and the stress that is applied), screw dislocations move in the perpendicular direction. This causes a fundamental difference in dislocation motion as the slip plane must contain the Burgers vector. For edge dislocations this is satisfied for a single slip plane, while for screw dislocations the Burgers vector is parallel to multiple slip planes. Motion of screw dislocations on multiple slip planes is called cross-slip, allowing the dislocations to go around obstacles with relative ease; see Figure 1-9C. For this reason screw dislocations control the deformation behaviour of steel, as shown by the experimental investigations of Caillard [79–83]. A characteristic feature of the body-centred cubic (BCC) lattice of steel is that the motion of dislocations is affected by temperature; temperature increases the mobility of screw dislocations, enhancing plasticity. The slow moving screw dislocations control the plastic deformation behaviour of steel up to a critical temperature of 67 °C (340 K) [84], with their motion becoming increasingly difficult at cold temperatures below 0 °C. The critical temperature $T_c$ is defined as the point above which flow stress becomes insensitive to temperature, i.e. screw and edge dislocations have equal mobility [85].
As mentioned above, screw dislocations can cross-slip in multiple slip-planes to overcome obstacles in the crystal lattice. Generally dislocation motion takes place in the planes with the highest atomic density, in the direction of highest linear density [87]. The deformation in BCC steel is commonly attributed as 'pencil glide' [88], where dislocations can slip in any plane containing the <1 1 −1> slip direction. The particular <1 1 −1> slip direction is also a zone-axis, which defines the possible slip planes [89]; see Figure 1-10. The activation of a slip plane is dependent on the direction of the load that is applied, i.e. the maximum resolved shear stress plane (MRSSP) [90]. Experimental observations have shown that the presence of slip in particular slip planes is dependent on material and temperature [89,91], with dislocation slip reported in {1 1 0}, {2 1 1} and {3 2 1} planes. The motion of dislocations in the specific slip planes causes the crystal lattice to rotate, as shown in Figure 1-11. Rotation is expected around the [1 1 −1] slip direction, as well as in a direction perpendicular to the direction of the dislocation motion. This rotation axis is the slip plane normal, and lies in the (1 1 −1) plane defined by the slip direction.
Figure 1-10. Slip planes of a screw dislocation that are defined by the [1 1 1] slip direction. The slip direction is a zone axis, which intersects three (110) planes and three (211) planes [90].

Figure 1-11. Schematic of screw dislocation and directions of dislocation motion for (1 0 1) slip plane. Lattice rotation is expected around the slip direction and the slip plane normal on all active slip systems. Planes and direction vectors added to the figure of Callister [92].

While stress projection factors such as the ‘Schmid factor’ have good predictive capability for dislocation slip in face-centred cubic (FCC) materials, their applicability to BCC materials is limited [93]. This is due to the fact that in FCC materials the strain field is confined to a {1 1 1} plane and the resistance of the lattice to dislocation motion is low, which is why computational models of these materials have seen significant advances in recent decades [93]. On the contrary for BCC materials, the modelling of dislocation motion is much more challenging, affected by factors such as the complexity of the stress field of a screw dislocation, temperature and strain rate dependency, as well as higher lattice resistance to dislocation motion. [93,94]. For these reasons the Schmid factor has limited applicability to polycrystalline BCC materials [87,95]. The prediction of the active slip plane and amount of plastic deformation is difficult especially for polycrystalline BCC materials, as interaction of the grains creates complex stress fields in the microstructure [96]. A study by Carroll et al. [91] showed that the amount of plastic strain in a specific grain correlated well with the Schmid factor for a BCC tantalum oligocrystal, i.e. a specimen with 12 (typically 3 – 20) through-
thickness grains in the gage section. The complexity of plastic deformation in BCC material was apparent with a polycrystalline specimen, as the interaction of the grains reduced the Spearman rank correlation coefficient between the Schmid factor and the average plastic strain of a grain from approximately 80% to 10%.

Plastic deformation can cause grain interaction, which is related to the dislocation motions at the grain boundary. In polycrystalline materials dislocations interact with grain boundaries, with the impeding of their motion causing the dislocations to form pile-ups [59]. The fundamental theory by Frank, Eshelby and Nabarro [22] describes the elastic stress concentration in-front of a pile-up, shown schematically in Figure 1-12. The magnitude of the stress concentration is in proportion to the number of dislocations in the pile-up. As multiple dislocations of the same sign are moving in the same direction, equilibrium is established between the applied shear stress and mutual repulsion of the dislocations [21]. The distance between the first and last dislocation can be approximated as $2A/n \sigma_0$, where $A$ is a constant, $n$ the number of dislocations and $\sigma_0$ the applied shear stress [21]. Thus, for a given applied shear stress, the distance between first and last dislocations are known. Therefore, the maximum number of dislocations in a pile-up is dependent on the length of the slip plane, which is determined by the size of the grain. This is why grain size plays a fundamental role in the mechanical properties of many materials, i.e. smaller is stronger, exemplified by the wide range of applicability of the Hall-Petch relationship. As a critical stress level is reached, dislocations are able to transmit through the grain boundary, or a dislocation source to be activated in the adjacent grain, both enabling further plastic deformation to take place.

Figure 1-12. Schematic representation of dislocation motion impeded by a grain boundary, and the stress concentration caused by the dislocation pile-up. [59]
The interaction of neighbouring grains defines the resistance of the grain boundary to the transmission of dislocations. The main factors affecting slip transmission are 1) the orientation of the grains, defining the co-planarity of slip systems (Figure 1-13) [97], 2) the resolved shear stress on different slip systems, determining the energy required for different incoming-outgoing slip system combinations [98], and 3) the local stress state and amount of stored energy, which makes the activation of slip system combinations other than the most favourable one possible [96]. Furthermore, different dislocation-grain boundary interactions can take place, including direct transmission, indirect transmission, and activation of a dislocation source in the neighbouring grain, see Figure 1-14.

The disruption of dislocation motion at grain boundaries is shown in Figure 1-15. Thus the interaction of grains is expected to make a significant contribution to the plastic deformation response, especially at length scales where the size of the plastic deformation zone is of the same order of magnitude as the grain size.

**Figure 1-13.** Geometrical description of dislocation motion across a grain boundary. Note that the Burgers vector directions are shown for an edge dislocation (parallel to dislocation motion). [97]

**Figure 1-14.** Dislocation motion across a grain boundary showing: A) direct transmission with the same Burgers vector on each side (no strengthening), B) direct transmission with different Burgers vectors, resulting in a residual dislocation at the GB, C) the same as B, but transmission is indirect, D) no transmission with accumulation of dislocation along the grain boundary. [99]
1.2.2 Measurement of microstructural deformation

In order to measure deformation on the microstructural scale, the methods used need to be able to distinguish the state of the microstructure before and after deformation. This can be carried out three-dimensionally only by using non-destructive testing methods, such as X-ray computed tomography (CT), which also provides temporal information on the deformation process [101,102]. The limitation of the CT methods is that in order for the X-rays to pass through the test sample, there is a significant trade-off between the spatial resolution that is achieved and the maximum sample size; see Figure 1-16. In order to measure deformation inside individual grains, the spatial resolution has to be higher than the grain size by a factor of at least 10–20 [67,103], which imposes a spatial resolution requirement of at least 0.1–0.05 µm for fine-grained steels. At this resolution the sample size with NanoCT is limited to tens of micrometres, which is not sufficient for covering the stochastic variation of grain size or the deformation process itself.

In materials science surface deformation is most commonly measured using scanning electron microscopy (SEM and TEM). While in situ electron microscopy allows the deformation process to be observed in incremental time steps for small two-dimensional sections [104–106], the deformed and undeformed states cannot usually be analysed from the same location. This limitation can be overcome by analysis of the deformed microstructure, determining which features were present prior to loading (grain structure), and which are the result of plastic deformation (deformation structure).

While scanning electron microscopy (SEM) is a two-dimensional method, three-dimensional information can be acquired with consecutive material sections prepared with focused ion beam (FIB) milling [107,108]. This improves the resolution trade-off from NanoCT, but it is still impractical for regions of interest larger than 100 µm [107]; see Figure 1-16. FIB allows accurate placement of the cross-section and specimens can be measured using EBSD directly after milling because of their minimal sample deformation. However, FIB milling is time-consuming, imposing limits on the number of the experiments to be carried
out. While this is not an issue for single crystalline materials, the stochastics of deformation cannot be captured for polycrystalline materials without a large number of indentation cross-sections.

Electron backscatter diffraction (EBSD) in a SEM is able to map large areas with high resolution in a relatively short time [109], and it is the preferred engineering tool for deformation analysis. The limitation to two-dimensional observation can be partly overcome by utilising suitable sample preparation techniques, such as ion milling or serial sectioning, to reveal several cross-sections of the deformation field with reasonable work effort. For this reason EBSD methodology is used in this thesis to provide sufficient resolution and three-dimensional spatial information on the stochastic deformation process, covering length scales from grain substructures (<1 µm) to macroscopic length scale (>1 mm).

Figure 1-16. Methods for mapping deformation on the microstructural scale, showing the spatial resolution and typical sample sizes. Three-dimensional characterisation methods are shown with solid outlines and two-dimensional methods with dashed outlines. Adapted from [110], inset length-scale figures reproduced from [111].

Electron backscatter diffraction (EBSD) analysis resolves the crystal orientation by detecting lattice planes from a diffraction pattern. The surface of a sample tilted by 70° is struck by an electron beam and diffracted to a detector after interacting with a small material volume; see Figure 1-17. The size of the interaction volume depends on the electron beam settings and material used. Typically, the spot size of the electron beam is 30 nm and the interaction volume has dimensions 2-3x larger than the spot size; see Figure 1-17. Thus a spatial resolution in the range <0.05 µm without significant interaction volume overlap is achievable [109]. As electrons are diffracted from the sample, a distinct electron backscatter diffraction pattern is captured by the detector, with the orientation of the ‘Kikuchi bands’ in the figure being dependent on crystal orientation; see Figure 1-18A. Using a Hough transformation process (B), the location of the bands is detected automatically (C), enabling the crystal orientation to be determined by comparing it to a list of known solutions (D).
Figure 1-17. A) Schematic arrangement of electron backscatter diffraction [112]. B) Electron beam size and interaction volume characteristics, modified after [109].

Figure 1-18. In EBSD the sample surface is struck with an electron beam, and the diffraction pattern is recorded [113]. The location of crystallographic planes is determined from the diffraction pattern: (A) via Hough transformation; (B) where lines are converted into points. The planes that are determined (C) are matched with the best-fitting simulation of crystal lattice orientation (D). [114]

The definition of misorientation between two crystal lattices is fundamental for microstructural analysis. Many engineering materials are polycrystalline, i.e. they consist of multiple grains on the microstructural level. On an atomic scale these materials are constructed of atoms that are aligned in a crystal lattice. Regions where the crystal lattices have a similar orientation are called grains and the change in orientation between two adjacent grains is called the grain boundary; see Figure 1-19A. Misorientation is the rotation that brings two crystal lattices into co-alignment, as shown in Figure 1-19B. Misorientation of the grain boundary is defined by a rotation consisting of a rotation axis and a rotational angle. Several axis/angle pairs are possible, with the common convention being to select the one with the smallest rotational angle.
Figure 1-19. A) A two dimensional crystal lattice with three different crystal orientations. Grain boundary is located at the interface of different orientations, characterised by the misorientation angle. Modified after [115]. B) Characterisation of the grain boundary with a rotation axis and angle [116].

The standard method for defining grain boundaries using EBSD is to define a threshold misorientation angle value above which adjacent points in the data are classified as belonging in different grains. Grain boundaries can be divided into high-angle grain boundaries (HAGB), with a misorientation larger than $10^\circ$, and low-angle grain boundaries (LAGB), with a misorientation between $2^\circ$ and $10^\circ$ [117]. In the literature the threshold for HAGBs varies between $10^\circ$ and $15^\circ$ [118]. The analysis of misorientation angles smaller than $2^\circ$ is restricted by the angular resolution of EBSD, which is typically $\sim0.5^\circ$ for misorientation and $\sim2^\circ$ for orientation [117]. Therefore, without orientation data post-processing the identification of deformation-induced grain sub-structures is often not possible [119]. Humphreys et al. [120] proposed the improvement of angular resolution by using an edge-preserving Kuwahara filter for orientation averaging in order to resolve sub-grains misoriented by less than $0.5^\circ$ (point-to-point).

The analysis of orientation data provided by EBSD measurements can be used to map plastic deformation in the microstructure of e.g. steel [121]. Plastic deformation causes strain gradients, which the microstructural lattice has to accommodate. As a result, gradients will be induced into the orientation of the crystal lattice inside the grains. The level of deformation can be estimated with misorientation analysis, where the orientation of two or more data points are compared. This deformation analysis can be performed on the grain level or within calculation domains of a pre-specified size. In grain-based misorientation analysis a reference orientation has to be determined for each grain. Subsequently data points inside the grain are compared to the reference orientation. The accuracy of the grain based analysis is dependent on an appropriate definition of the reference orientation. Various approaches have been applied for the determination of the reference orientation for deformed polycrystalline materials,
The most common being the average orientation of the grain. When each measurement point inside the grain is compared to the reference orientation, the plastic strain localization as well as the deformation gradients inside the grains become visible; see Figure 1-20. Accurate determination of the reference orientation is important for accurate analysis of highly deformed polycrystalline materials, as shown in Figure 1-20A&B for average orientation and point with lowest local misorientation as the reference orientation, correspondingly.

Figure 1-20. Grain reference orientation deviation for partially recrystallized steel sample, with the reference defined as A) the average orientation of the grain, B) the point inside the grain with the lowest local misorientation. [121]

To avoid issues related to grain detection and the proper selection of reference orientation, local deformation analysis can also be carried out within a calculational domain, also known as kernel-based misorientation analysis [121]. The size of the kernel is defined as the number of nearest neighbours of a central point; see Figure 1-21A. The benefit of kernel-based analysis is that it enables local analysis without a reference orientation, while still using metrics similar to grain-based analysis. Most commonly the kernel average misorientation (KAM) is used metric in materials science, describing the average misorientation from a central point to all neighbours covered inside the kernel: see Figure 1-21B. The results obtained with KAM are sensitive to several factors [121]: 1) the step-size of the data, 2) the size of the kernel, and 3) the upper threshold angle for excluding data-points originating from neighbouring grains, often in the range 2–5°. In order to reveal the grain substructure effectively, the parameters need to be optimized on the basis of the characteristics of the boundaries. For reviews of EBSD strain mapping methods see Wright et al. [121] and Wilkinson and Britton [122].
1.3 Scope of research, objectives and limitations

Fundamental research on the dislocation-mediated plastic deformation process has mostly been carried out on single crystalline materials. These materials allow precise experimental design and enable individual factors to be separated and measured more accurately. For polycrystalline materials in engineering applications complex interactions take place at the level of neighbouring grains, and are fundamental for the deformation response. This presents major challenges for both experimental design and result analysis. Thus experimental and analytical methods need to be developed in order to study the microstructural deformation state of polycrystalline materials, with particular attention to studying how material heterogeneity affects dislocation motion between adjacent grains. The length scales relevant for this thesis are shown in Figure 1-22. The central role of grain structure is justified by the influence of grain boundaries on dislocation motion, affecting the physical deformation processes on both neighbouring length scales (macroscopic, dislocations). This enables statistics on grain size and strength to be linked, with insight provided by the active deformation mechanisms in dislocation scale. The scope of the thesis is focused on the length-scale interface between fundamental material research and continuum-scale material strength.
The experimental setup is defined so that it is suitable for the materials and manufacturing methods used in engineering applications, e.g. in the ship building industry. Thus, welded structural steel with a base metal yield strength of 355 MPa forms the basis of the test matrix. Because of the severe material gradient in welded joints, experiments are carried out for weld metal locations where a large enough area can be identified with relatively constant material phase composition and similar grain size dispersion. Thus, the thesis is focused on investigating the variation of material properties in microscopic scale within an area where macroscopic properties remain quite constant, spanning a few millimetres in size.

Welding is carried out with traditional arc welding, as well as laser and laser-hybrid welding, to determine how the heat-input of the welding process affects the strength of the material and the microstructure. The structural steel base metal is used for the study of deformation processes on the scale of dislocation motion, as the relatively large grain size (>10 µm) enables high spatial resolution inside the grains. When compared to weld metal, significant differences in grain size characteristics can be linked to plastic deformation behaviour. Since the thesis is focused on a polycrystalline materials with broad grain size variation, the grain structure of the material is chosen as the single most important length scale; see Figure 1-23. The analyses are extended both to grain deformation behaviour and macroscopic strength in different length scales. As the length scales range from 0.1 µm to over 100 µm, the only feasible method for microstructural analysis is EBSD. Macroscopic material behaviour is measured with hardness testing as it enables the length scale of plastic deformation to be varied. The local variation of toughness properties is not considered in this thesis. Furthermore, the experimental investigations are carried out at room temperature, with (cold) temperature effects left for future research.

Figure 1-22. Length scales of research in this work, and characteristic lengths associated with them. Length-scale figures reproduced/modified from [79,115,123–125].
This thesis is composed of six chapters. After the introduction the second chapter explains the experimental setup, materials used, sample preparation methods and material characterisation techniques. A serial sectioning technique is developed for plastic deformation analysis to capture the stochastics of the deformation process for polycrystalline material. Chapter 3 focuses on the microstructural characterisation methods for polycrystalline steel. The grain size distribution is measured, and an approach developed to define a characteristic geometric length parameter for grain size variation. The relationships between grain size and material hardness is then investigated on different length scales, showing the predictive capability of grain size for macroscopic hardness. In addition, a method is introduced for revealing the deformation structure, with a dislocation cell structure resolved inside the deformed grains.

Chapters 4 and 5 are dedicated to understanding the plastic deformation process, and its relationships to the strength of the material on different length scales. The deformation structure is used to define a plastic deformation zone for heterogeneous polycrystalline steel. The size of the plastic deformation zone is measured for a large number of hardness indentations, revealing the relationships between grain size and the size of the plastic deformation zone. In Chapter 5, the plastic deformation mechanisms enabling the formation of the deformation structure are investigated in different length scales. The interactions of dislocations and grain boundaries are investigated from the temporal analysis of instrumented indentation data, revealing the length-scale interaction between the
size of the plastic deformation zone and grain size. Finally, dislocation motion is linked with the local variation in the size of the deformation structure. In Chapter 6, the results are discussed and compared to the literature. Special emphasis is put on the microstructural characterisation of complex weld metal microstructures, deformation mechanisms, and the length scale interaction. In addition, a modified Hall-Petch relationship is formulated for the prediction of the strength of varying grain size dispersion and applied to data from the literature. This chapter is followed by conclusions and themes for future research.
2. Experimental setup

2.1 Materials and sample preparation

To investigate the influence of material heterogeneity on mechanical properties and local plastic deformation, various steel microstructures are examined. In addition to structural steel base metal, the weld metals (WM) of conventional arc (CV), laser (LA), and laser-hybrid (HY) welded joints are included in the test series. Table 2-1 lists the specimen nomenclature, together with the corresponding joint type and welding method. Transverse cuts in relation to the welding direction were used for the test specimens; see Figure 2-1. The arc, laser-hybrid, and laser welded joints are shown in Figure 2-2. In welds with distinct microstructural differences both the top (toe) and bottom (root) weld metal regions are characterised. The weld metals represent complex microstructures with different phase compositions, enabling the influence of grain size and grain size dispersion on macroscopic mechanical properties to be investigated. Two base metals with different grain sizes are included for comparison.

Table 2-1. Test specimen nomenclature and the corresponding joint types and welding methods.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Joint type</th>
<th>Welding method</th>
<th>Measurement location</th>
</tr>
</thead>
<tbody>
<tr>
<td>BM.1</td>
<td>Plate, 6 mm</td>
<td>-</td>
<td>1.0-1.9 mm (^a)</td>
</tr>
<tr>
<td>BM.2</td>
<td>Plate, 5 mm</td>
<td>-</td>
<td>0.75-1.35 mm (^a)</td>
</tr>
<tr>
<td>CV.1</td>
<td>Butt joint, 3 mm</td>
<td>Arc</td>
<td>Toe, root</td>
</tr>
<tr>
<td>CV.2</td>
<td>Block joint, 3 mm</td>
<td>Arc</td>
<td>Toe, root</td>
</tr>
<tr>
<td>CV.3</td>
<td>T-joint, 3/5 mm</td>
<td>Arc</td>
<td>Toe</td>
</tr>
<tr>
<td>HY.1</td>
<td>Butt joint, 3 mm</td>
<td>Laser-hybrid</td>
<td>Toe, root</td>
</tr>
<tr>
<td>LA.1</td>
<td>Butt joint, 3 mm</td>
<td>Laser</td>
<td>Toe</td>
</tr>
<tr>
<td>LA.2</td>
<td>Butt joint, 3/5 mm</td>
<td>Laser</td>
<td>Toe</td>
</tr>
</tbody>
</table>

\(^a\) Distance from the surface of the plate.
Figure 2-1. Example weld sample and the location of the transversal cut. A macrograph is shown for the polished and etched section A-A.

Figure 2-2. Micrographs of the weld cross-sections that were investigated, as well as the locations of the HV1 hardness measurements: A) Arc welded CV.1, B) Arc welded CV.2, C) Arc welded CV.3, D) Laser-hybrid welded HY.1, E) Laser welded LA.1, F) Laser welded LA.2.

The base metal (BM) for the welded joints is a shipbuilding structural steel with a minimum nominal yield strength of 355 MPa. The base metal steel grades and their properties are listed in Table 2-2.
Table 2-2. Mechanical properties and chemical composition of base metals.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Grade</th>
<th>Mechanical properties</th>
<th>Chemical composition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$R_{p0.2}$ (MPa)</td>
<td>$R_m$ (MPa)</td>
</tr>
<tr>
<td>BM.1</td>
<td>GL D36</td>
<td>343</td>
<td>472</td>
</tr>
<tr>
<td>BM.2</td>
<td>GL D36</td>
<td>400</td>
<td>533</td>
</tr>
<tr>
<td>CV.1</td>
<td>S355J2</td>
<td>466</td>
<td>564</td>
</tr>
<tr>
<td>CV.2</td>
<td>S355J2</td>
<td>466</td>
<td>564</td>
</tr>
<tr>
<td>CV.3, 3mm</td>
<td>S355J2</td>
<td>466</td>
<td>564</td>
</tr>
<tr>
<td>CV.3, 5mm</td>
<td>S355JO</td>
<td>432</td>
<td>521</td>
</tr>
<tr>
<td>HY.1</td>
<td>GL D36</td>
<td>399</td>
<td>531</td>
</tr>
<tr>
<td>LA.1</td>
<td>GL D36</td>
<td>414</td>
<td>567</td>
</tr>
<tr>
<td>LA.2, 3mm</td>
<td>S355J2</td>
<td>466</td>
<td>564</td>
</tr>
<tr>
<td>LA.2, 5mm</td>
<td>S355JO</td>
<td>432</td>
<td>521</td>
</tr>
</tbody>
</table>

For the microstructural analysis the cut sections of the specimens were mounted in an electrically conductive resin and ground using P180-P4000 grit abrasive papers, followed by polishing with 3-µm and 1-µm diamond paste. For the optical microscopy and hardness measurements (HV1), the specimens were etched with a 2% Nital solution. For the scanning electron microscopy, the samples were fine-polished with 0.25-µm diamond paste, followed by colloidal silica polishing in a vibratory polisher to minimise the deformation induced by the sample preparation. For details of the sample preparation steps see Appendix A.

To characterise the mechanical properties of the base and weld metals, instrumented indentation testing is carried out. For the analysis of the material microstructure and plastic deformation, hardness indentations are cross-sectioned for scanning electron microscopy. To enable the acquisition of large data sets, an adaptation of the traditional serial sectioning technique [refs] is utilised. In serial sectioning material is successively removed from the sample surface to reveal the underlying microstructure. To find the cross-sectional plane perpendicular to the hardness indentations, a depth gauge is implemented in the sample surface for the measurement of material removal, and is used in conjunction with knowledge of the material removal rates in all the sample preparation steps. In addition, to minimise the plastic deformation induced by the sample preparation a systematic and careful sample preparation procedure was applied. A nickel plating deposited on the hardness indentations is utilised to avoid deformation at the sample edge.

The general outline of the cross-sectioning procedure for a welded joint is shown in Figure 2-3. First, the location of interest is determined in the transverse cross-section of the welded joint, in this case the toe side (top) weld metal (Step 1 in Figure 2-3). Two orthogonal cuts are performed to create flat surfaces on the side and the top of the weld. Both of these surfaces are used for the alignment and measurement of indentation locations. Before hardness measurements are carried out, the toe side surface (A) is mounted in an epoxy resin and ground up
to P4000 grit abrasive paper and briefly diamond polished to ensure flatness (Step 2). The mount is broken and the specimen is remounted with cross-section surface B facing up (Step 3). A rectangular section of structural steel is placed adjacent to surface A. This reduces the sample preparation deformation at the edge of the specimen, since it equalizes the material removal rate on both sides. Before the hardness measurement of surface B, the surface is fine grinded up to P2000 grit abrasive paper, followed by 3 µm, 1 µm and 0.25 µm diamond suspension polishing. Final polishing is carried out for one hour in a vibratory polisher with colloidal silica before hardness measurements. After that the sample is ready for the depth gauge implementation needed for the measurement of material removal.

To implement a depth gauge, the hardness measurements are carried out with the sample corner as a reference point (Step 4). A series of indentations with their distance increasing in steps of 2-15 µm from the surface (A) are used as the depth gauge for serial sectioning. Indentations of varying sizes are placed between the markers. Two identical sets of markers and indentations are created below the first one. After hardness measurements the epoxy mount is carefully broken, and an approximately 50-µm-thick nickel layer is deposited on the indentations (Step 5).
5). The nickel-plated surface A is also mounted with a steel support adjacent to the surface. As material is removed from surface (A) using serial sectioning, the underlying indentations are revealed (Step 6). Progressively finer abrasive particles need to be used as target cross-section approaches to minimise deformation on the sample surface. During serial section the location of an indentation is determined by measuring the length of the flat part at the bottom of the indentations (Step 7). The Vickers indenter is a four-sided pyramid, and thus the flat part represents the distance between the diagonals of an indentation. As the location of multiple indentations is measured, the location of the cross-section can be determined with an absolute accuracy of 3-5 pixels from optical images, corresponding to 0.4–0.7 µm with a magnification of 500x and image size of 1600x1200 pixels. Comparative analysis between closely spaced adjacent sections is accurate to 1-2 pixels, corresponding to 0.1–0.2 µm. Further sections are revealed by repeating the grinding and polishing steps (Step 8). For details of the serial sectioning procedure see Appendix A.

### 2.2 Microscopic analysis

The microstructures were characterised using scanning electron microscopy (SEM). A Zeiss Ultra 55 field emission scanning electron microscope equipped with a Nordlys F+ camera and Channel 5 software from Oxford Instruments was used for the electron backscatter diffraction (EBSD) analyses. The EBSD analyses were performed with varying step sizes between 0.06 µm and 0.2 µm. The acceleration voltage was 20 kV and the indexing rate of the EBSD maps was 90% or higher depending on the specimen. In addition, the Bruker e-Flash HR EBSD–detector, mounted in a Merlin VP Compact SEM, was used to capture forescatter detector images using the 3-diode detector, providing superior orientation contrast [126] compared to the Nordlys F+ camera. The Bruker system was also used for the cross-correlation analysis of diffraction patterns, i.e. HR-EBSD, using the Cross-Court 3 software. The EBSD data was post-processed and analysed using the open source Matlab toolbox MTEX (versions 4.5.2 and 5.0.1) [127,128]. The orientation data was post-processed with a half-quadratic filter to reduce measurement noise, and assign orientations to the non-indexed points.

The grain size was analysed from EBSD grain boundary maps and optical micrographs. The misorientation angle threshold for EBSD grain boundary detection is varied between 2° and 10°. In grain size analysis the criterion of 10° is used, while for plastic deformation analysis it is varied to ensure accurate grain detection. For the cases where e.g. a small segment of the grain boundary was missing, the reduced misorientation angle thresholds were used to make the definition of the grain boundary accurate. When suitable, optical micrographs and digital image processing are also utilised in the grain size analysis; see [31] for more details.

All the plastic deformation analysis is performed on the basis of EBSD data and the analysis carried out utilising MTEX [127,128]. This analysis includes orientation maps, misorientation angle and axis maps, kernel misorientation,
deformation analysis. As a result of the computational cost of large kernels in misorientation analysis, parallel computing was used because of the significant speed gains it offered. The following third party open source Matlab codes were utilised in the misorientation analysis: dbscan clustering [129], bi-linear knee-point fitting [130], and plane fitting to a point cloud [131].

Since local plastic deformation in heterogeneous microstructures is also affected by the material phase, the microstructural constituents were identified according to the IIW guideline for ferritic microstructure [132]. The volume fractions of the microstructural constituents were determined from optical micrographs or EBSD image quality maps using the systematic manual point counting method according to ASTM E562-02 [133]. For this analysis, a randomly placed measurement grid of 100 points was used over four to six micrographs to determine the material phase volume fractions.

2.3 Instrumented indentation testing

Instrumented indentation testing was carried out to measure the mechanical properties of the materials and to investigate the microstructural deformation. A CSM Instruments indenter was used, utilising both a micro-indentation tester and a nano-indentation tester. The micro-indentation tester was equipped with a four-sided Vickers pyramid tip, while the nano-indenter had a three-sided Berkovich pyramid tip. The micro-indenter was generally used for measuring hardness measurement load greater than 100 mN up to 16,000 mN, while the range 1...500 mN was covered with the nano-indenter. The contact points were manually checked for all tests to ensure reliable results. The results were analysed using Martens hardness, HM, which is equivalent in definition to the traditional Vickers hardness:

\[
HM = \frac{F}{A_s(h)} = \frac{F}{26.43h^2}
\]  

(1)

The calibrated indenter area function \( A_s(h) \) was used for all the analyses. A comparison of the Martens hardness values and the standard diagonal length measurement has been carried out, and proved to have good correlation with the HV1 measurement load [134]. For hardness testing linear 30 s loading ramps were used, with a hold time of 10 s at the maximum load before unloading.

To quantify the influence of the grain size dispersion on macroscopic hardness, the size of the indentations has to be large enough relative to the grain size. Large HV1 indentations are utilised, as the ratio between the indentation diagonal length and average grain size is approximately 10 for the coarse-grained base metal BM.1, and significantly higher for the more heterogeneous weld metals, being in the range of 30–80. Thus, a large population of grains is sampled by each indentation, and the average value of 10–12 indentations can be considered as a good representation of the macroscopic hardness of the material, mitigating the influence of grain interaction and spatial grain size dispersion. Example micrographs of indentation size are shown in Figure 2-4, where the indentation
diagonal lengths are approximately 115 µm, 95 µm, 85 µm, and 70 µm for A–D, correspondingly.

Figure 2-4. HV1 indentation size in relation to the grain size for (A) the base metal BM.1 and the weld metals (B) CV.2 root-side, (C) HY.1 toe-side, and (D) LA.1. [135]

Because of the spatial distribution of grain size and phase volume fraction, the mechanical response is dependent on the length scale of deformation. In a hardness test, the size of the indentation defines the plastic deformation zone. The size of the deformation zone interacts with the size of the microstructural features; with small indentations the response of individual grains or phases can be captured, while a large indentation represents the macroscopic response of the material. To investigate the interaction of length scales, hardness is measured covering a broad test load range between 1 mN and 16182 mN. The measurements are carried out for the base metal BM.1 and the weld metal CV.2 toe. These samples have contrasting grain size characteristics, with the base metal having large grains and a narrow distribution, while the weld metal has a small grain size with a broad dispersion. The measurement parameters are shown in Table 2-3. A single indentation can cover up to 10–20 grains, while the smallest indentations are only 0.05–0.15 times the volume-weighted average grain size.
Table 2-3. Hardness measurement matrices used for characterizing length scale interaction. The volume-weighted average grain sizes are 15.2 µm for base metal (BM.1) and 4.8 µm for weld metal (CV.2 toe).

<table>
<thead>
<tr>
<th>Test force</th>
<th>Indenter Number of Indentation diagonal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test force</td>
<td>Test area</td>
</tr>
<tr>
<td>(mN)</td>
<td>(µm x µm)</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>HV1.65</td>
<td>1618.6</td>
</tr>
<tr>
<td>HV1</td>
<td>9807.0</td>
</tr>
<tr>
<td>HV0.3</td>
<td>2942.1</td>
</tr>
<tr>
<td>HV0.1</td>
<td>980.7</td>
</tr>
<tr>
<td>HV0.05</td>
<td>490.4</td>
</tr>
<tr>
<td>HV0.025</td>
<td>245.2</td>
</tr>
<tr>
<td>HV0.01</td>
<td>98.1</td>
</tr>
<tr>
<td>25mN</td>
<td>24.5</td>
</tr>
<tr>
<td>5 mN</td>
<td>4.9</td>
</tr>
<tr>
<td>1 mN</td>
<td>1.0</td>
</tr>
<tr>
<td>Base metal</td>
<td>Indentation diagonal</td>
</tr>
<tr>
<td></td>
<td>(µm x µm)</td>
</tr>
<tr>
<td>HV1</td>
<td>9807.0</td>
</tr>
<tr>
<td>HV0.3</td>
<td>2942.1</td>
</tr>
<tr>
<td>HV0.1</td>
<td>980.7</td>
</tr>
<tr>
<td>HV0.05</td>
<td>490.4</td>
</tr>
<tr>
<td>HV0.025</td>
<td>245.2</td>
</tr>
<tr>
<td>HV0.01</td>
<td>98.1</td>
</tr>
<tr>
<td>25mN</td>
<td>24.5</td>
</tr>
<tr>
<td>5 mN</td>
<td>4.9</td>
</tr>
<tr>
<td>1 mN</td>
<td>1.0</td>
</tr>
</tbody>
</table>

In addition to hardness values, the temporal histories of the indentation data are analysed, providing hardness values as a function of the indentation depth and the gradient of the indenter movement speed. The dislocation pile-up and slip transmission events are captured by detecting a significant increase in the displacement speed in the load controlled test. The influence of dislocation pile-up and slip transmission through grain boundaries is expected to be most prominent on hardness measurements when the plastic deformation zone grows from the first grain to be affected to the neighbouring grains. Changes in indenter movement speed are used to detect the strain bursts resulting from slip transmission. The average displacement speed during the test is approximately 10 nm/s. In order to eliminate measurement noise as an error source, the detection bound is taken as 3 nm/s above the local moving average of the displacement speed. Furthermore, at least two data points are required above the threshold for detection.
3. Weld metal characterisation

3.1 Grain size distribution

Welds are an extreme case of heterogeneity since variation of the microstructure is present both on a macroscopic scale across the joint and on a microscopic scale within a single zone; see Figure 3-1. Thus, grain size measurement methods suitable for the characterisation of broad grain size dispersions are introduced. The methods presented in this chapter have been published by the author in references [134] and [135], and are available online [136]. This chapter explains the measurement methods, while detailed information of the measurement methods and analytical derivations are presented in the references and Appendix B.

![Figure 3-1. Macro section of an arc welded joint (CV.1) and an example of weld metal grain size variation showing fine-grained (FG) and coarse-grained (CG) areas. [134]](image)

The most commonly applied measure of grain size is the average grain size. A wide variety of grain size measurement methods [137–140] are available for this purpose. Furthermore, orientation imaging microscopy give the operator a greater degree of freedom when defining the measurement methodology with
parameters such as step size, grain boundary misorientation criteria and filtering of the data [67,141,142]. In this research focused on heterogeneous microstructures, the average grain size is measured using the ASTM E1382 [139] linear intercept length method in four evenly spaced directions (0°, 45°, 90°, 135°); see Figure 3-2A. All four measurement directions were considered as a single distribution for the analysis. Measurements smaller than three pixels, e.g. 0.3 µm for EBSD grain boundary maps with a step size of 0.1 µm, were considered as noise and removed from the distributions. This method was chosen for its robustness for cases where there is no clear granular structure such as a weld. The method is insensitive to EBSD indexing errors [143].

Although the average grain size is commonly used, the average grain size does not adequately represent the physical response of heterogeneous microstructures with broad grain size dispersion [65,68]. To provide a metric for the variation of grain size, the relative grain size dispersion is used in this work, modified from Berbenni et al. [64] to be suitable for experimental data, defined as:

$$\frac{\Delta d}{d} = \frac{d_{\text{max}} - d_{\text{min}}}{d} = \frac{P_{99\%} - P_{1\%}}{d},$$

where $d$ is the average grain size, and the maximum ($d_{\text{max}}$) and minimum ($d_{\text{min}}$) grain sizes are replaced by the 99% and 1% probability level grain sizes, respectively. This is done to minimise measurement uncertainty, which is inherently at its greatest at the extremities of the distribution because of the finite number of measurements. The relative grain size dispersion captures the degree of grain size variation on the macroscopic level effectively. Consideration of the grain size dispersion is crucial for heterogeneous material since the largest grains can be associated with low strength as a result of the length of the slip bands, causing them to yield first; see e.g. [144,145]. Furthermore, even a small number of large grains can occupy a significant material volume. Therefore, in this work a rule-of-mixtures approach is introduced for heterogeneous microstructures to capture the influence of grain volume. The contribution of each grain to the strength of the material is considered to be proportional to the volume of the grain; see e.g. [60,69].

The measurement of the volume-weighted grain size is based on the point-sampled linear intercept length method defined by Gundersen and Jensen [146,147], utilising stereological relationships for measuring the volume-weighted distribution of particle size. A set of randomly positioned points is placed on the image and an intercept length is measured through each point that strikes a location of interest [148], in this case the interior of the grain. The direction of the intercept is chosen randomly, in this case from the four measurement directions used for the ASTM linear intercept method (0°, 45°, 90°, 135°) for comparability of the results; see Figure 3-2B for a graphical illustration of the measurement procedure. Through the use of this sampling strategy the frequency of measurements in a specific grain is in proportion to its surface area, which approximates the volume fraction of the grain. Thus, the measured distribution is volume-weighted, and its mean value is the volume-weighted average grain size.
In addition to the characteristic grain size values, a visualisation of the grain size dispersion is required to analyse the spatial variation. The visualisation of the grain size requires grain identification, and is based on displaying the surface area of the grain, or a derivative of the surface area such as the equivalent circle diameter [67]. The point-sampled grain size measurement method enables the visualisation of the grain size for heterogeneous microstructures even when the grain boundaries are discontinuous; see Figure 3-3B. As the visualisation is spatially informed, the grain size can vary within a single grain, representing the variation in the length of the slip planes. No assumptions are made regarding the shape of the grains. The visualisation method that was developed for the point-sampled grain size measurement method also allows the utilisation of the Hall-Petch grain size parameter, $d^{-0.5}$, to enhance the resolution of the visualisation in the small grain size regime (0.1 – 10 µm), relevant for mechanical properties. Figure 3-3A also shows a moving average of the grain size with horizontal and vertical rectangular probes. However, it should be noted that the Hall-Petch relationship is not a valid measure of mechanical properties for a small material volume within a single grain.

The spatial variation of the grain size is visualised using kernel-based grain size averaging, which reflects local average grain size values. This enables the visualisation of grain size-dependent mechanical properties by utilising a kernel-based averaging approach for the grain size measurement. The average grain size is calculated inside a kernel, and the size of the square kernel is defined as a multiple of the volume-weighted average size, usually ranging from 0.5 – 4 times the grain size. This enables better presentation of uniform regions consisting of fine grains or coarse grains. An example of kernel-based averaging is shown in Figure 3-3C&D. The distinction between coarse- and fine-grained areas becomes clearer with $1^*d_v$ kernel (C), still showing clear local variations. A larger kernel size of $3^*d_v$ (D) shows continuous grain size contours, with diminishing detail of the local features. Numerical values of grain size and grain size variation can be extracted from all grain size maps using arbitrarily shaped kernels, which are typically rectangular in shape.
Figure 3-3. A) Moving average of grain size in horizontal direction based on B) measured spatial grain size variation. C, D) Kernel-based grain size averaging with kernel sizes of C) 1*dv (side length: 7.6 µm), D) 3*dv (side length: 22 µm).
3.2 Microstructure and macroscopic strength

To investigate the influence of grain size and grain size dispersion on macroscopic mechanical properties, a variety of welded joints are investigated, in addition to two base metals. This chapter explains the main findings, while the detailed information has been published by the author in reference [135]. Representative micrographs of grain structures are shown in Figure 3-4. The base metals (A, B) have the largest average grain sizes with narrow grain size dispersions ($\Delta d/d$). The weld metals show a wide variety of average grain sizes, while still significantly smaller than the base metals. A single weld metal (C) exhibits a narrow grain size dispersion similar to the base metal, while especially the samples with irregularly-shaped grains (C, I, J, K) have broad grain size dispersions. As a consequence, these samples show a proportionally larger increase from the average grain size to the volume-weighted average grain size. The measured grain size parameters are shown in Table 3-1.

![Figure 3-4. Example optical micrographs and orientation maps (IPF-Z) showing the grain structure for: A) BM.1, B) BM.2, C) CV.1 toe, D) CV.1 root, E) CV.2 toe, F) CV.2 root, G) CV.3, H) HY.1 toe, I) HY.1 root, J) LA.1, K) LA.2.](image-url)
Table 3-1. Measured grain sizes and hardness values and their respective 95% confidence intervals. Abbreviations used: BM: Base metal, CV: Conventional arc welding, HY: Arc & laser hybrid welding, LA: Laser welding. [135]

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Images analysed</th>
<th>$d$ (µm)</th>
<th>$\Delta d/d$</th>
<th>$d_v$ (µm)</th>
<th>Increase $d \rightarrow d_v$</th>
<th>Hardness HM (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BM.1</td>
<td>2 (Optical)</td>
<td>9.99 ± 1.62</td>
<td>3.20</td>
<td>15.29 ± 1.96</td>
<td>53%</td>
<td>1412 ± 29</td>
</tr>
<tr>
<td>BM.2</td>
<td>3 (Optical)</td>
<td>4.29 ± 0.20</td>
<td>3.10</td>
<td>6.53 ± 0.39</td>
<td>52%</td>
<td>1781 ± 53</td>
</tr>
<tr>
<td>CV.1 toe</td>
<td>6 (EBSD)</td>
<td>2.90 ± 0.15</td>
<td>5.95</td>
<td>7.28 ± 0.70</td>
<td>151%</td>
<td>1945 ± 33</td>
</tr>
<tr>
<td>CV.1 root</td>
<td>2 (Optical)</td>
<td>3.53 ± 0.46</td>
<td>3.54</td>
<td>5.95 ± 0.20</td>
<td>69%</td>
<td>1756 ± 17</td>
</tr>
<tr>
<td>CV.2 toe</td>
<td>6 (EBSD)</td>
<td>2.04 ± 0.13</td>
<td>4.66</td>
<td>4.00 ± 0.45</td>
<td>96%</td>
<td>1984 ± 34</td>
</tr>
<tr>
<td>CV.2 root</td>
<td>6 (EBSD)</td>
<td>1.89 ± 0.14</td>
<td>5.38</td>
<td>4.18 ± 0.49</td>
<td>121%</td>
<td>1931 ± 39</td>
</tr>
<tr>
<td>CV.3</td>
<td>4 (EBSD)</td>
<td>1.37 ± 0.08</td>
<td>4.54</td>
<td>2.58 ± 0.21</td>
<td>88%</td>
<td>2156 ± 29</td>
</tr>
<tr>
<td>HY.1 toe</td>
<td>6 (EBSD)</td>
<td>1.09 ± 0.06</td>
<td>4.34</td>
<td>2.06 ± 0.27</td>
<td>89%</td>
<td>2405 ± 38</td>
</tr>
<tr>
<td>HY.1 root</td>
<td>4 (EBSD)</td>
<td>2.30 ± 0.34</td>
<td>5.75</td>
<td>5.64 ± 1.06</td>
<td>145%</td>
<td>2617 ± 51</td>
</tr>
<tr>
<td>LA.1</td>
<td>6 (EBSD)</td>
<td>1.36 ± 0.06</td>
<td>5.42</td>
<td>3.17 ± 0.34</td>
<td>133%</td>
<td>3339 ± 101</td>
</tr>
<tr>
<td>LA.2</td>
<td>6 (EBSD)</td>
<td>1.32 ± 0.08</td>
<td>5.00</td>
<td>2.88 ± 0.24</td>
<td>118%</td>
<td>3801 ± 46</td>
</tr>
</tbody>
</table>

The microstructural constituent volume fractions and the corresponding 95% confidence intervals for the materials that were tested are listed in Table 3-2. Most microstructures have primary ferrite (PF) or acicular ferrite (AF) as their main constituent. The base metal and CV.1 root-side weld metal also have pearlite (P) in their microstructure, while the other weld metals have a small amount of ferrite-carbide aggregate (FC) (which includes pearlite). Laser welds have a martensitic microstructure (M) with a varying amount of ferrite with second phase (FS). The HY.1 root-side weld metal is a mixture of martensite and ferrite with second phase. According to their microstructural constituents, the specimens are divided into ferritic and martensitic microstructures.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Images analysed</th>
<th>Constituent volume fraction (%)</th>
<th>Constituent volume fraction (%)</th>
<th>Constituent volume fraction (%)</th>
<th>Constituent volume fraction (%)</th>
<th>M</th>
</tr>
</thead>
<tbody>
<tr>
<td>BM.1</td>
<td>6 (Optical)</td>
<td>AF 78.6 ± 5.3</td>
<td>PF 68.8 ± 5.3</td>
<td>FS 18.8 ± 7.2</td>
<td>FC/P 4.5 ± 1.2</td>
<td>-</td>
</tr>
<tr>
<td>BM.2</td>
<td>4 (Optical)</td>
<td>AF 34.2 ± 6.1</td>
<td>PF 78.7 ± 4.7</td>
<td>FS 46.0 ± 3.9</td>
<td>FC/P 4.5 ± 1.0</td>
<td>-</td>
</tr>
<tr>
<td>CV.1 toe</td>
<td>6 (Optical)</td>
<td>AF 3.4 ± 1.4</td>
<td>PF 50.3 ± 6.2</td>
<td>FS 45.7 ± 6.6</td>
<td>FC/P 4.0 ± 1.1</td>
<td>-</td>
</tr>
<tr>
<td>CV.1 root</td>
<td>4 (Optical)</td>
<td>AF 61.2 ± 6.3</td>
<td>PF 32.5 ± 6.6</td>
<td>FS 54.3 ± 5.6</td>
<td>FC/P 3.0 ± 0.9</td>
<td>42.7 ± 5.0</td>
</tr>
<tr>
<td>CV.2 toe</td>
<td>6 (EBSD)</td>
<td>AF 50.3 ± 6.2</td>
<td>PF 16.7 ± 2.1</td>
<td>FS 24.7 ± 5.0</td>
<td>FC/P 2.0 ± 0.9</td>
<td>81.3 ± 2.3</td>
</tr>
<tr>
<td>CV.2 root</td>
<td>6 (EBSD)</td>
<td>AF 45.7 ± 6.6</td>
<td>PF 54.3 ± 5.6</td>
<td>FS 32.5 ± 6.6</td>
<td>FC/P 3.0 ± 0.9</td>
<td>42.7 ± 5.0</td>
</tr>
<tr>
<td>CV.3</td>
<td>4 (EBSD)</td>
<td>AF 16.7 ± 2.1</td>
<td>PF 54.3 ± 5.6</td>
<td>FS 24.7 ± 5.0</td>
<td>FC/P 2.0 ± 0.9</td>
<td>81.3 ± 2.3</td>
</tr>
<tr>
<td>HY.1 toe</td>
<td>6 (EBSD)</td>
<td>AF 32.5 ± 6.6</td>
<td>PF 54.3 ± 5.6</td>
<td>FS 45.7 ± 6.6</td>
<td>FC/P 4.0 ± 1.1</td>
<td>-</td>
</tr>
<tr>
<td>HY.1 root</td>
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<td>AF 45.7 ± 6.6</td>
<td>PF 54.3 ± 5.6</td>
<td>FS 32.5 ± 6.6</td>
<td>FC/P 3.0 ± 0.9</td>
<td>42.7 ± 5.0</td>
</tr>
<tr>
<td>LA.1</td>
<td>6 (Optical)</td>
<td>AF 50.3 ± 6.2</td>
<td>PF 16.7 ± 2.1</td>
<td>FS 24.7 ± 5.0</td>
<td>FC/P 2.0 ± 0.9</td>
<td>81.3 ± 2.3</td>
</tr>
<tr>
<td>LA.2</td>
<td>5 (Optical)</td>
<td>AF 16.7 ± 2.1</td>
<td>PF 54.3 ± 5.6</td>
<td>FS 24.7 ± 5.0</td>
<td>FC/P 2.0 ± 0.9</td>
<td>81.3 ± 2.3</td>
</tr>
</tbody>
</table>
On the basis of a detailed analysis of grain size dispersions, the difference between the average grain size and the volume-weighted average grain size is found to be proportional to the relative grain size dispersion $\Delta d/d$. The ratio of the Hall-Petch grain size parameters, i.e. $d^{-1/2}$ and $d_v^{-1/2}$, is plotted as a function of $\Delta d/d$ in Figure 3-5. The regression analysis is performed for individual micrographs, with the legend sorted in descending order of phase volume fraction. One exception is the specimens with acicular ferrite and primary ferrite (AF, PF), which cover a wide range of phase volume fractions and which are shown as a single dataset. The regression has narrow 95% confidence and prediction bounds, shown by the dashed and dotted lines, respectively. The correlation between the two grain size parameters is:

$$d_v^{-1/2} = d^{-1/2} (c + f \frac{\Delta d}{d})$$

where $f$ and $c$ are the slope and constant term of the linear regression, respectively. The parameters of the linear regression and their respective 95% confidence intervals are $f = -0.0635 (-0.0662, -0.0608)$ and $c = 1.0059 (0.9926, 1.0191)$. Consequently, the constant term is taken to have the value $c \approx 1.0$ in the further analysis of the present study.

On the basis of the results in Figure 3-5, the relative grain size dispersions for the base metal and weld metal are significantly different. For the base metal with a primary ferrite-pearlite (PF, P) microstructure, the relative grain size dispersion is approximately 3.1, while the range for the weld metals is 3.5-6.4. Predominantly single-phase weld metals, such as martensite (M), are grouped within a small range of dispersion. As the volume fraction of ferrite with second phase (FS) increases in the martensitic microstructure, the relative grain size dispersion range expands from 4.8-5.3 up to a maximum of 6.0. Likewise, when primary ferrite-pearlite (PF, P) has a small addition of acicular ferrite (AF), the dispersion increases from 3.1 to 3.6. Most noticeably, the specimens with acicular ferrite and primary ferrite (AF, PF) cover a large range of dispersion ranging from 3.6 to 6.1.
To investigate the large range of dispersion for mixtures of acicular ferrite and primary ferrite, an analysis is carried out to determine the volume fractions of the microstructural constituents from individual micrographs. The analysis in Figure 3-6 shows that the relative grain size dispersion has a linear correlation with the volume fraction of acicular ferrite. With a high volume fraction of acicular ferrite the relative grain size dispersion is narrow, while with a decreasing amount of acicular ferrite the dispersion broadens, as shown in Figure 3-7.

**Figure 3-6.** Volume fraction of acicular ferrite as a function of the relative grain size dispersion $\Delta d / d$ for specimens with a mixture of acicular ferrite and primary ferrite. [135]

**Figure 3-7.** EBSD image quality maps and the microstructural constituents for three different relative grain size dispersions corresponding to Figure 3-6: A) $\Delta d / d = 4.0$, B) $\Delta d / d = 4.7$, C) $\Delta d / d = 5.7$. [135]
The influence of grain size dispersion on mechanical properties is quantified with HV1 indentations, representing macroscopic strength. The measured hardness values for the 11 samples are shown in Table 3-1. The samples cover a wide range of hardness values, with the base metal BM.1 being the softest (HM=1412 MPa) and the laser weld LA.2 being the hardest (HM=3801 MPa). The hardness of ferritic samples with a well-defined granular microstructure, excluding the CV.1 toe-side weld metal because of the irregular grain shape (planar solidification), is shown as a function of the average grain size in Figure 3-8A. Even though the coefficient of determination for the linear regression is high, the 95% confidence and prediction bounds shown by the dashed and dotted lines, respectively, are broad. The bounds are significantly narrower when the volume-weighted average grain size is used in Figure 3-8B. It should be noted that the sample size of seven influences the confidence and prediction bounds. Furthermore, the coefficient of determination is improved and the data follows the regression within the 95% confidence intervals of the data. These results indicate that the volume-weighted average grain size captures the influence of grain size dispersion effectively, providing a more representative microstructural metric for the prediction of macroscopic strength in heterogeneous welded microstructures. In order to understand the physical mechanisms behind the observed correlation and the influence of spatial grain size variation, local plastic deformation behaviour is investigated later, in Chapters 4 and 5.

![Figure 3-8](image)

Figure 3-8. Hardness of ferritic microstructures as a function of: A) average grain size \( d \), and B) volume-weighted average grain size \( d_v \). The error bars represent the 95% confidence intervals of measured hardness and average grain size values. [135]
3.3 Spatial grain size variation

Differences in local grain size dispersion and phase volume fractions can affect the mechanical response and localised plastic deformation. For the base metal under study, pearlite is present as a second phase, while for the weld metal the mixture of acicular ferrite and primary ferrite creates distinct regions of fine and coarse grains; see Figure 3-9. In terms of grain size variation, the phase mixture of the weld metal yields a large range of grain sizes, as shown in Figure 3-10B. The size of the ferrite grains is much more homogeneous for the base metal in Figure 3-10A. The differences in spatial grain size and phase distribution are expected to have a significant effect on material deformation behaviour in a hardness test, particularly when the deformed material volume contains a limited number of grains.

![Figure 3-9. The grain boundary maps used for grain size analysis of a) the base metal, b) the heterogeneous weld metal. [134]](image)

![Figure 3-10. Comparison of local grain size variation for a) base metal, b) the heterogeneous weld metal. The colour contours range between 0.18 and 1.05 \( \mu m^{-0.5} \), representing the largest (99%) and smallest (1%) probability level grain sizes for the two specimens. [134]](image)

The grain size map and moving averages of grain size are shown for the base metal in Figure 3-11. The moving averages show only little fluctuation as a result of the uniform, equiaxed shape of the ferrite grains. The weld metal, on the other hand, shows significant variation as a result of the primary ferrite/acicular ferrite phase mixture in Figure 3-12. In addition to the horizontal gradient, a vertical gradient is present, with the grain size increasing towards the bottom of the micrograph.
To investigate the influence of spatial grain size variation on mechanical properties, indentations of varying size are measured for the two microstructures. The aim was to define an indentation size at which the local variation in the grain size has an influence on the mechanical properties. Figure 3-13 shows indentation matrices and hardness contours with the load levels HVo.3, HVo.1, and HVo.01. The load ranges yield indentations with approximate diagonal lengths of 60 µm, 30 µm, and 10 µm, respectively. For both materials, the HVo.3 indentations are too large to capture the influence of local grain size variation. With HVo.1 some
local variations appear for both materials, although it seems that low hardness values do not correspond to large grains. With the smallest HV0.01 indentations local variations start to become apparent. In general hardness is quite constant for the base metal, with low values being recorded inside large grains, and high values when the indentations have hit the hard second phase. For the weld metal the majority of the low hardness values are associated with coarse-grained areas. Because of the indentation size and spacing, only a few indentations have struck the centres of large grains. In many cases the indentations are at the edge of a large grain, facing the adjacent fine-grained areas, thus increasing the measured hardness values.

To compare how well the observed hardness variation correlates with local microstructural features, an HV0.01 hardness measurement is carried out for the weld metal in conjunction with the EBSD grain size measurement. The grain size map, averaged with a kernel equalling the volume-weighted average grain size, is shown in Figure 3-14C. This reveals ‘soft’ and ‘hard’ regions of the microstructure, as expected according to the local grain size based on the Hall-Petch relationship. The measured hardness values are overlaid on the grain size map as rectangles with the secondary colour scale. In general, low hardness values are observed in coarse-grained areas, and medium-high values in fine-grained areas. The agreement of the local grain size and hardness values is shown in Figure 3-14B with three colours: 1) green (49/96, 51%): good correlation, 2) orange (21/96, 22%): correlation when surrounding grain size is considered, 3) red (26/96, 27%): weak correlation. In general hardness reflects local grain size variation, with fine
grained areas showing medium-high hardness and coarse grained areas showing low hardness values. Although approximately half of the hardness measurements agree with the surface grain size, other factors clearly need to be considered. Typically the weak correlation is observed at the edges of coarse-grained and fine-grained regions. The deviations may be caused, for example, by the grain size gradient below the indentation, and in particular by the interaction of neighbouring grains when only a few grains are in the plastic deformation zone. Therefore the microstructural changes taking place during plastic deformation need to be characterised to determine the role of local microstructural variation in the plastic deformation process.

Figure 3-14. a) Optical micrograph of the hardness measurement area for the heterogeneous weld metal area A2, b) grain boundary map and visual estimation of the correlation between local grain size and hardness; see the text for the explanation, and c) the hardness value for each indentation overlaid on the grain size contour, ranging from the 99% to 1% probability level grain size. Kernel-averaged grain size (kernel size is 6.4x6.4 µm). Revised after [134], see footnote 1.

1. Accuracy of contact point detection has been improved, slightly changing measured hardness values. Furthermore, the averaged local grain size is used instead of raw measurement data for visualization in (C). Consequently, the agreement between hardness and grain size has been updated in (B).
3.4 Misorientation analysis

The plastic deformation of various heterogeneous grain structures is quantified by analysing the orientation data gathered with EBSD, as the rotation of the crystal lattice provides a metric for the severity of the deformation. Grain-based misorientation analysis was used to study the localised material rotation behaviour caused by the indentation. In this analysis the rotation of a single measurement point inside a grain is compared to the reference orientation of the grain. An accurate definition of the reference orientation is crucial for the robust analysis of deformed polycrystalline materials. Commonly, the average orientation of the grain is taken as the reference; see Figure 3-15A. For deformed grains this can be inaccurate, showing up as deformation throughout the entire grain far away from the indentation.

In this work a clustering based method was developed and applied to determine a better estimate of the reference orientation. Using this method similar orientations are grouped together, and the cluster with lowest deformation is used to define the reference orientation; see Figure 3-15B. As a result the highly deformed areas under the indentation are excluded from the definition of reference orientation. Using this approach the misorientation below the indenter is higher and misorientation decreases continuously from the indented surface to the first grain boundary. Contrary to other commonly used methods, the clustering-based method is not sensitive to outliers in the orientation data. Furthermore, the reference cluster can be selected manually, increasing the versatility of the method, e.g. in cases where a grain boundary is missing between two grains. A detailed description of the clustering-based reference orientation methodology, as well as its validation and comparison to commonly used approaches, is presented in Appendix C.

Figure 3-15 shows example results from the clustering-based misorientation analysis. The misorientation in Figure 3-16A is highest under the indenter, with severe deformation extending to the first grain boundary. While a misorientation represents the smallest rotation to bring a point inside the grain into co-alignment with the reference orientation, it can also be decomposed into rotations around the specimen coordinate axes; see Figure 3-16 B-D. Rotation around the Z-axis...
takes place at the two flanks of the indentation, and around the Y-axis at the
corners of the indentation. The highest rotation is under the flat part of
the indentation, rotating around the X-axis. These rotations are a direct
consequence of the indenter geometry. The rotations around the X-, Y-, and Z-axes have been
validated using HR-EBSD, where the cross-correlation of diffraction patterns
provided very good agreement with the clustering-based analysis.

![Figure 3-16. Misorientation (A) decomposed into principal rotations around the x-, y-, and z-
axes (B-D). Axes and positive rotation directions are shown in the inset.](image)

While the misorientation angle (Figure 3-16A) can be used to interpret the
magnitude of deformation, the misorientation axis can reveal the deformation
mechanics, i.e. the areas that are rotating in the same direction. An example of
misorientation axes is shown in Figure 3-17A. The zones that share the same
misorientation axis have the same colour, indicating the formation of sub-grains
during the plastic deformation process. This analysis is carried out in the
specimen coordinate system to reveal the continuity of deformation between
adjacent grains; see the inset of Figure 3-16D for the coordinate system. In this
analysis the colouring is based on the pole figure (B), where the upper and lower
halves of a sphere are projected as two-dimensional surfaces.

![Figure 3-17. A) An example of misorientation analysis in the specimen coordinate system and
B) the pole figure colour key including coordinate system directions and selected data points
marked with corresponding numbers.](image)
The accuracy of the sub-grain deformation analysis is dependent on the quality of the misorientation data [149]. Therefore, to improve the angular resolution of the measurement, post-processing is applied to reduce the measurement noise. Firstly, half-quadratic filtering developed by Bergmann et al. [150], is utilised to reduce measurement noise and to assign an orientation to non-indexed locations. Noise reduction is based on the assumption that noise is spatially independent and can be separated from ‘true’ orientations [151]. Half-quadratic filtering was successfully applied to misorientation analysis by Hielscher et al. [151] and Kilian and Heilbronner [152]. Contrary to other commonly used filters, half-quadratic filtering is edge-preserving, and thus suitable for the analysis of sub-grain features. The half-quadratic filter is applied twice, with both passes having the same smoothing parameter, $\alpha=0.15$. The third step of the post-possessing is to apply the Kuwahara filter [153] to minimise the blurring of features in the misorientation analysis. This filter is an adaptive smoothing filter that preserves edges, i.e. continuous features such as the sub-grain boundaries, while removing noise inside the sub-grains. Thus, contrast is enhanced for the local misorientation analysis.

The influence of EBSD post-processing is demonstrated in Figure 3-18. The kernel misorientation is calculated with first nearest neighbours (3x3 kernel), using raw measurement data and two filtering options. With the raw data some dense dislocation walls (DDWs) are apparent beneath the indenter in the highly deformed zone (A, D); however, the measurement noise masks the details inside the grain. With one pass of the half-quadratic filter, the noise floor is reduced significantly, and the deformed and non-deformed grains can be identified. In addition, the blurred outlines of the DDWs are seen throughout the deformed area, including the area beneath the indenter (B, E). The second pass of the half-quadratic filter was found to reduce the characteristic blocky features often induced by Kuwahara filtering, providing optimal separation of the DDWs. With this filter combination a well-defined network of DDWs is revealed (C, F), and it is used for the characterisation of the sub-grain deformation structures. A more thorough analysis of the post-processing of the orientation data and its influence on the kernel average misorientation is presented in Appendix B.
In order to characterise the misorientation of DDWs under localised plastic deformation, the kernel size for the misorientation analysis is increased beyond conventional values, typically 1 – 3 nearest neighbours. The definition of the kernel size in this work is based on the physical size of the deformation structures; the kernel covers an entire sub-grain. As shown in Figure 3-19, an intermediate-size kernel (nn=5) results in the blurring of the deformation structure. With a further increase in the kernel size the average misorientation of the deformation structure starts to become uniform, forming a well-defined network of sub-grain boundaries at a kernel size of 60 nearest neighbours. A further increase in the kernel size does not significantly change the results. Kernel sizes of 60 nearest neighbours (side length 12.1 µm) are used for the base metal and 20 nearest neighbours (side length 2.5 µm) for the weld metal in the detailed analysis of the deformation structure. These values are approximately in proportion to the volume-weighted average grain sizes, being 15.2 µm and 4.8 µm respectively. As a result of the broad grain size dispersion and finer sub-structures the kernel size is relatively smaller for the weld metal.
Figure 3-19. Kernel average misorientation with kernel sizes ranging between 5…100 nearest neighbours. The physical size of the kernels is shown in the bottom right corner. Note: the data extends beyond the image limits to cover the dimensions of the largest kernel.

The combination of the misorientation axis and kernel misorientation analysis allows the grain interaction and slip transmission between grains to be characterised. To convey the approximate size of the plastic deformation zone, the misorientation axis map (A) and kernel misorientation analysis (B) are combined in Figure 3-20C. The black colour is adjusted in such a way that undeformed material is assumed below 0.2° of kernel misorientation. When the visualisations in A and B are combined, the rotation patterns remain only in the deformed grains (C).

Figure 3-20. Combination of misorientation axis (A) and kernel misorientation (B) to convey extent of plastic deformation with misorientation axis directions (C).

While the differently coloured regions in Figure 3-20C represent sub-granular features, their borders are not very distinct, especially in the grain under the indenter. To make distinction easier, sub-grain boundaries can be overlaid on the misorientation axis map, shown in Figure 3-21C. This highlights only those grains with severe deformation. Insights into the plastic deformation process are possible by analysing the continuity of the misorientation axes across grain boundaries and the size of the sub-grain structures in adjacent grains. The images
are combined numerically by utilising a colour space conversion and a smooth transfer function to adjust colour lightness, represented by the greyscale colour maps in Figure 3-20B and Figure 3-21B. Details of the procedure are presented in Appendix B. The same approach will be utilised later to analyse grain interaction by combining the dislocation cell structure and misorientation axis directions.

**Figure 3-21.** Combination of misorientation axis (A) and kernel misorientation (B) to convey grain sub-structure with misorientation axis directions (C).
4. Grain refinement

4.1 Grain sub-structure analysis

Plastic deformation is quantified by analysing the misorientation data, as the rotation of the crystal lattice correlates with the amount of plastic deformation. The rotation of the crystal lattice requires the motion of dislocations. During plastic deformation dislocations re-arrange in the crystal lattice, forming grain sub-structures and thus refining the original grain structure, as shown in Figure 4-1A. To reveal the degree of plastic deformation for welded steel, the sub-grain boundaries, dislocation cells (DC), and dense dislocation walls (DDWs) that are formed are characterised.

![Figure 4-1. A) Evolution of steel’s grain structure during plastic deformation, showing the formation of dislocation cells and sub-grain boundaries. B) Schematic kernel misorientation distributions corresponding to locations 1-4 shown in sub-figure A.](image)

The deformation structure is characterised with kernel-based misorientation analysis, in which the orientation of a central point is compared to its nearest neighbours, shown in the inset of Figure 4-1B. When plastic deformation takes place, orientation gradients are generated inside the grains, thus increasing the kernel average misorientation (KAM). Depending on the location and size of the kernel, the misorientation distribution can be used to reveal the deformation structure. As the steel deforms and dislocation cells form, the KAM increases inside the newly formed dislocation cells (2) in comparison to the undeformed grains (1). The orientation is relatively uniform inside a single DC and at the DDW separating adjacent DCs. When a kernel is in close proximity of the DDW, a bi-modal distribution with some large misorientations is expected. When the central
point is at a DDW, misorientation is low with regard to the other points on the DDW, and high regard to the points in the neighbouring DCs. This reverses the proportions of the bi-modal misorientation distribution (3); see Figure 4-1B and the inset schematic. As deformation progresses, the DCs transform into higher angle sub-grain boundaries (4), with a significantly higher average misorientation and a more pronounced peak in the distribution. To focus the analysis on specific deformation structures, e.g. DDWs or sub-grain boundaries, the range of misorientations that is analysed has to be limited.

Figure 4-2 illustrates the typical sub-grain boundary analysis using a large kernel size. The area inside the dashed line is the data within 2° of the misorientation, showing the effectiveness of the 2° threshold criteria to cover entire sub-grains. The misorientation distributions for three data points inside a sub-grain are shown in Figure 4-2D-F for central and near-boundary locations. While an undeformed location shows a kernel average misorientation of approximately 0.15°, the values inside the deformed grain are significantly higher. At the centre of a sub-grain (A) a bi-modal misorientation distribution is observed (D), with small misorientations corresponding to the ‘core’ area, and large values corresponding to the near-boundary regions. As the measurement point moves near the sub-grain boundary (B), the peak in the misorientation shifts towards larger angles (E). When the central point of the kernel is on the sub-grain boundary (C), the effective kernel extends on both sides of the boundary with sections of the adjacent sub-grains excluded. Furthermore, there is a strong peak at large misorientation angles (F), and a broad distribution of values extending over the entire measurement range. Because of the characteristic shape of the distribution, the median misorientation is significantly higher than the average misorientation for sub-grain boundaries (F). On the basis of these findings, the median value is a better measure for large kernels, as it increases the misorientation for boundary (C) and near-boundary locations (B), and reduces it for locations inside the sub-grain (A). For further instances this is termed kernel median misorientation (KMM).
4.2 Deformation structure in localised plastic deformation

The extent of the plastic deformation can be characterised by varying the misorientation range that is analysed. This enables the differentiation of sub-grain boundaries from dense dislocation walls and dislocation cells. Examples of analysis with the different thresholds, as well as average (KAM) and median (KMM) values, are shown in Figure 4-3. With the conventional upper threshold of 2° KMM enhances the contrast of sub-grain boundaries and the distinction between deformed and undeformed areas. The reduction of the threshold to 1° reveals a much finer sub-structure with DDWs between the sub-grain boundaries. Further reduction of the threshold to 0.5° reveals the finest DCs. The average misorientation (KAM) is more appropriate for the 1° and 0.5° thresholds, showing more consistent values especially for the smallest DCs.

**Figure 4-2.** A-C) Kernel average misorientation for different locations. The dashed black line showing the region inside the kernel that is within 2° misorientation threshold of the central point, i.e. the effective kernel size. D-F) Corresponding misorientation histograms.
Figure 4-3. Comparison of substructures revealed with kernel misorientation (NN=60) analysis using misorientation thresholds of 2°, 1° and 0.5°.

On the basis of the deformation process of steel [75], the misorientation of sub-grain boundaries continues to increase during the deformation process, revealing which boundaries have formed first and have then transformed into higher angle sub-grain boundaries. To study the character of different sub-grain boundaries, the misorientation between the central points of neighbouring sub-grains and dislocation cells is analysed for an example case. The analysis for the conventional 2° threshold KMM in Figure 4-4 shows a misorientation range of 2.3° – 5.6° between adjacent sub-grains, with an average of 3.5°. KAM with the upper threshold of 0.5° in Figure 4-5 shows that dislocation cells of varying sizes have a misorientation of 0.5° – 2.1°. The average misorientation between dislocation cells is 1.0°. The measured values are in good agreement with the values from the literature obtained by direct measurement using transmission electron microscopy [75], where dislocation cells were measured to have a misorientation of 0.5° – 0.8° and sub-grains a misorientation of 2.0° – 5.0°; see Appendix E for misorientation values and dislocation cell size measurement. The dislocation cells add another level of information, with the size of the dislocation cell being indicative of plastic deformation; locations with small dislocation cells are severely deformed, experiencing large local strains [76,154].
Figure 4-4. A) Band contrast map showing locations of pearlite phase. B) Kernel median misorientation (step size 0.06µm, nn=100, 2° threshold) showing sub-grains. The misorientation between adjacent sub-grains is overlaid for selected boundaries.
On the basis of the above findings, sub-grain structures are defined for the plastic deformation analysis. The smallest sub-grain deformation unit is the dislocation cell, bounded by dense dislocation walls. The dense dislocation walls are captured with a kernel misorientation upper threshold of $0.5^\circ$. The evolution of the DDWs between $0.5^\circ$ and $1^\circ$ is ignored, and the sub-grain boundaries obtained with a $2^\circ$ threshold are defined as the measure of severe deformation.

### 4.3 Plastic deformation zone size

The characterisation of the plastic deformation zone is based on the formation of deformation structures. The highly deformed volume under the indenter has dislocation cells and sub-grain boundaries, termed the grain refined zone (GRZ); see Figure 4-6. The extent of (measurable) plastic deformation is defined as the area with dense dislocation walls, termed the plastic deformation zone (PDZ). In many cases the dense dislocation walls form dislocation cells as a result of multiple active slip systems [75].
Figure 4-6. Schematic of the deformation zone separated into two regions: 1) grain refined zone: highly deformed volume with sub-grain boundaries; 2) plastic deformation zone: extent of plastic deformation defined by presence of dense dislocation walls.

An example of the deformation zones is shown in Figure 4-7 for a base metal HV0.05 indentation. In (A) the grain refined zone is defined from KMM (0-2°), and in (B) the plastic deformation zone from KAM (0-0.5°). In (C) the forescatter detector image shows that the GRZ contour closely matches with the area showing large orientation changes, while minor changes are visible in the PDZ. The surface area of the deformation zones is measured up to the sample surface prior to deformation, thus including the shape of the indentation in the measurement.

Figure 4-7. Deformation zones for an example case showing A) the grain refined zone and B) the plastic deformation zone for a base metal HV0.05 indentation. The two zones are overlaid on the forescatter detector image in (C).
To study the evolution of plastic deformation, the size of the deformation zone is measured for the base metal (BM.1) and weld metal (CV.2 toe); see Table 2-3 and Figure 3-9. The base metal has a uniform coarse grain size, while the weld metal has a broad dispersion with a mixture of fine and coarse grains.

Three indentation load levels (HV0.0025, HV0.01 and HV0.05) are used to vary the size of the plastic deformation zone. The size of the grain refined zone (GRZ) is shown in Figure 4-8 for all the cross-sections that were measured. At the lowest load level of HV0.0025, shown in Figure 4-8C, the GRZ is similar for the base metal and weld metal. Single outliers have been marked with an asterisk (*) in the figures. Because of uncertainties associated with cross-sectioning and the three-dimensionality of the deformation zones (e.g. grain boundary effects), some outliers are expected and they have been excluded from the fitting of the first degree polynomials. As the load level increases to HV0.01, the base metal shows a significantly larger deformation area; see Figure 4-8. Furthermore, the gradient in the size of the deformation zone is steeper when moving towards the edge of the indenter. A similar trend continues at HV0.05, with significantly more material deformed in the case of the base metal.

In Figure 4-8 the statistical scatter is characterised by fitting a first degree polynomial to the data. The uncertainty of the fits is shown by the dark shaded regions, representing the 95% confidence bounds (95% CB) of the fit parameters. The 95% prediction bounds (95% CB), shown by the light shaded regions, represent the bounds in which new observations are predicted to lie within. These two parameters indicate how consistent the trend in the data is, and how large is the spread in the observations. The absolute broadness of the prediction bounds increases while indentation load increases. In relation to the average values the prediction bounds become smaller as indentation load increases, being mostly similar for the two materials. At the largest HV0.05 indentation load the base metal shows broader dispersion than weld metal. In general the prediction bounds are broader for the plastic deformation zone than the grain refined zone.
Figure 4-8. Deformation zone sizes shown as a function of cross-section distance from indenter centreline, showing: A, C) grain refined zone and B,D) plastic deformation zone for different indentation load levels. Shaded areas represent the 95% confidence bounds (CB) and 95% prediction bounds (PB). The approximate location of the indentation edge is marked with the vertical dashed lines.

The size of the plastic deformation zone is shown in Figure 4-8B&D. Because of the sensitivity to residual deformation, values could not be reliably determined for all the cross-sections shown in Figure 4-8A&C. However, general trends can be established for all load levels. At the load level HV0.0025 the plastic deformation zones are similar for both materials, being approximately two times the GRZ. At the intermediate HV0.01 load level the plastic deformation zones are approximately 1.9 and 2.8 times the GRZ for the base metal and weld metal, respectively. Thus, the difference between the two materials is reduced compared to the GRZ. This trend is reversed at the HV0.05 load level, as the plastic deformation zone grows much larger for the base metal. The multipliers between the GRZ and PDZ are 2.6 and 2.0 for base metal and weld metal, respectively. The
scatter of the PDZ is significantly larger than for the GRZ. While the broader confidence and prediction bounds are partially caused by the smaller number of observations, the spread of data is significantly larger. Especially the base metal HV0.05 indentations show significantly broader prediction bounds for PDZ compared to GRZ.

The plastic deformation trends are compared by defining the average size of the deformation zones. To compare the material behaviour at different indentation load levels, the average values are estimated at the indenter centreline based on all observations; see Table 4-1. The coefficient of determination ($R^2$) varies between 0.03 and 0.67. The low values indicate that the deformation zone size does not change significantly when moving further from the indentation centreline. The scatter of the data can be estimated the root mean square error (RMSE), which is the standard deviation of the residuals, i.e. difference between fitted equation and observations. The RMSE values range between 7% and 30% of the fitted constant b, being similar for both materials.

### Table 4-1. Linear fitting parameters including prediction and confidence bounds for the grain refined and plastic deformation zones, corresponding to Figure 4-8.

<table>
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<tr>
<th>F</th>
<th>BM HV0.0025</th>
<th>BM HV0.01</th>
<th>BM HV0.05</th>
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The estimated average deformation zone sizes are shown in Figure 4-9A. The deformation zone sizes are significantly larger for the base metal, especially the PDZ as the indentation depth increases. In general the confidence and prediction bounds are small, with the exception of base metal HV0.05 PDZ. To compare the deformation zones with microstructural dimensions, the surface area is converted
to an equivalent radius of a hemisphere in Figure 4-9B. These values give an approximation of the depth reached by the deformation zones. For example at HV0.01, the weld metal deformation zones extend to 4.8 µm and 8.0 µm, while for the base metal the values are 7.6 µm and 10.4 µm. The plastic deformation zone scatter is considerably larger for weld metal HV0.01 indentations compared to base metal, shown by the broad confidence and prediction bounds. This is partially caused by the residual plastic deformation present in the weld metal, which masks the edge of the plastic deformation zone in many cases. In cases where the shape of a deformation zone is elliptical, the derived values can either over- or underestimate the maximum depth of the deformation reached by the zones. The variations in the shape and size of the deformation zones are analysed next.

![Figure 4-9](image)

**Figure 4-9.** Estimated average damage zone size at indenter centreline as a function of indentation depth for base metal and weld metal: A) damage zone area, B) equivalent damage zone radius. The error bars represent the 95% confidence bounds (CB) and shaded areas the 95% prediction bounds (PB).

### 4.4 Influence of grain boundaries on plastic deformation

It is expected that grain size and grain boundaries significantly affect plastic flow, and thus the size and shape of the deformation zones are compared to the local grain size characteristics. The influence of the grain size on the deformation structures and the size of the deformation zones is visible for the base metal HV0.05 indentations in Figure 4-10. While the grain refined zone containing sub-grain boundaries is similar in size for the two indentations, the density of the sub-grain boundaries is significantly higher for the grain containing most of the deformation (A). The deformation has been effectively blocked by the grain boundaries, limiting the size of the plastic deformation zone (C). For the second indentation (B) the long vertical grains have allowed an unobstructed expansion of the plastic deformation zone beneath the indenter (D). The grain boundaries clearly affect the growth direction of the plastic deformation and as a result the shape of the deformation zones varies.
The weld metal exhibits a significantly smaller variation for the deformation zone sizes at an HV0.05 indentation load compared to the base metal. As shown in Figure 4-11, two cross-sections with a major difference in grain size have approximately the same grain refined zone. However, the shape is clearly affected by the grain size. Compared to the base metal in Figure 4-10A&B, the grain refined zone extends more horizontally for the weld metal in Figure 4-11A. These observations highlight the complexity of the local plastic deformation process and the variability of the grain interactions.

The role of grain size in controlling the size of the deformation zones becomes more apparent for the weld metal with HV0.01 indentations as the number of deformed grains decreases. The contribution of the grain boundaries to the shape of the grain refined zone is visible in Figure 4-12. The shape of the grain refined zone is altered by the grain boundaries for the indentations shown in sub-figures...
A, B, and C. The grain refined zone in sub-figure C is mostly confined to the single large grain, limiting the extent of the deformation along the horizontal direction.

![Grain refined zone for the weld metal HV0.01 indentations (A: 33 µm², B: 31 µm², C: 34 µm²).](image)

Figure 4-12. Grain refined zone for the weld metal HV0.01 indentations (A: 33 µm², B: 31 µm², C: 34 µm²).

To analyse the evolution of the size and shape of the deformation zone, all the measured grain refined zones are presented in Figure 4-13. The grain refined zones are used for the comparison as they are less sensitive to experimental errors caused by e.g. sample preparation deformation. At an HV0.0025 indentation load the GRZ shapes are similar for both materials, being approximately hemispherical. With HV0.01 and HV0.05 indentation loads the base metal deformation extends significantly deeper into the material. The depth reached by the GRZ also shows more variation for base metal.

![Measured grain refined zones for HV0.0025, HV0.01 and HV0.05 indentations for base metal (A,C,E) and weld metal (B,D,F). The location of the cross-section relative to the indentation centre line is shown in colour.](image)

Figure 4-13. Measured grain refined zones for HV0.0025, HV0.01 and HV0.05 indentations for base metal (A,C,E) and weld metal (B,D,F). The location of the cross-section relative to the indentation centre line is shown in colour.
To compare the deformation zones of the two materials, the average shape of the GRZ is estimated at the indenter centreline from several cross-sections to remove the stochastic nature of local deformation; see Figure 4-14 A&B. The growth of the grain refined zone has different characteristics for the two materials. While initially the GRZ is similar at HV0.0025, the growth takes place primarily in the depth direction for the base metal at HV0.01, continuing in all directions at HV0.05. On the contrary, for the weld metal, the growth is nearly homogeneous in all directions as the indentation load increases. The horizontal extension is determined by the size of the indentation, being approximately equivalent at the same indentation load for both materials. This difference in behaviour is apparent when the GRZ shapes are normalised in Figure 4-14 C&D, which shows the shape of the GRZ as a multiple of the maximum width. The base metal shows a pronounced elliptical shape at HV0.01, shifting closer back to hemispheric at HV0.05. For the weld metal the normalised GRZ shape stays quite constant as the indentation load is increased. This behaviour is expected to be caused by the grain size characteristics of the materials.

Figure 4-14. Average shape of the grain refined zone at the indenter centreline for A) the base metal, B) the weld metal. The shape coordinates are normalised with the maximum x-coordinate of each indentation load for the base metal and weld metal in (C) and (D), correspondingly. The number in brackets indicates the maximum normalised depth.

The average shape of the grain refined zone is normalised with the local volume-weighted average grain size in Figure 4-15. The growth of the grain refined zone is similar for both materials, with some key differences in shape. Most notably the base metal grain refined zone at HV0.01 has grown significantly deeper into the material than that of the weld metal at HV0.0025, which matches the horizontal extension. The shape of the GRZ starts to have a similar shape at HV0.05/HV0.01 for the base metal and weld metal, respectively. Even though the
equivalent radius is larger for the weld metal, the deformation of the base metal extends slightly deeper into the material. This may be a consequence of the physical difference in grain size, affecting the strain hardening and grain interaction behaviour.

<table>
<thead>
<tr>
<th>F</th>
<th>BM</th>
<th>BM</th>
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<th>WM</th>
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</tr>
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<tbody>
<tr>
<td>d_v (µm)</td>
<td>HV0.0025</td>
<td>9.15</td>
<td>11.47</td>
<td>13.38</td>
<td>4.72</td>
<td>3.75</td>
</tr>
</tbody>
</table>

**Figure 4-15.** Average grain refined zone shape normalised with the local volume-weighted average grain size.

Comparison of the indentation and deformation zone sizes relative to the average grain size is shown in Figure 4-16. The deformation zone radii are shown relative to the local volume-weighted average grain size ($d_v$) in Figure 4-16A. This shows that for a given physical indentation size the deformation zones are significantly larger for the weld metal in relation to the grain size. Compared to the local average grain sizes, the plastic deformation zone radii range approximately between 0.4...1.8*$d_v$ for the base metal and 0.7...2.8*$d_v$ for the weld metal.

The indentation diagonal lengths are normalized in a similar manner in Figure 4-16B, such that both axes represent a multiple of grain size. With this approach both materials are shifted approximately to the same trend lines. The grain refined zone radius is approximately half of the relative indentation diagonal, showing a nearly linear trend. The plastic deformation zone radius has a non-linear behaviour for the largest indentations, being approximately equal to the relative indentation diagonal up to 2*$d_v$. At larger indentation sizes the GRZ and PDZ increase at a similar rate between HV0.01 and HV0.05 for the weld metal. This shows that the growth of the deformation zones is mainly controlled by grain size and grain boundaries when the average characteristics of multiple indentations are considered. There is a degree of uncertainty in the average size of the plastic deformation zone for weld metal, shown by the broader 95% confidence bounds (error bars). However, the interpretation of the results would remain largely unchanged even in the case that the true average value was at the 95% confidence bounds. The stochastic nature of the local plastic deformation process is visible in the broad 95% prediction bounds, shown by the shaded areas. The observations in this chapter confirm that macroscopic plastic flow is mainly controlled by grain size, represented by $d_v$ for the investigated materials. The influence of grain interaction on the plastic deformation process is analysed next.
Figure 4-16. A) Radii of the deformation zones as a function of indentation diagonal, normalised with the local volume-weighted average grain size. B) Radii of deformation zones normalised in a similar manner. The error bars represent the 95% confidence bounds (CB) and shaded areas the 95% prediction bounds (PB).
5. Plastic deformation mechanisms

5.1 Grain interaction analysis

The observed variation in the size of plastic deformation zones and their preferred growth directions is primarily caused by the interaction of grains. During a hardness test the size of the plastic deformation zone increases as the indenter tip penetrates into the material, as shown in Figure 5-1A. For the crystal lattice to accommodate the shape change, dislocation motion is required. As plastic deformation accumulates, the motion of the dislocations can be significantly impeded by grain boundaries, limiting the growth of the plastic deformation zone; see (1 & 2) in Figure 5-1. The dislocation pile-up creates an elastic stress concentration behind the grain boundary, opposing the shear stress acting on the dislocations. As the load acting on the indenter and thus the shear stress acting on the dislocations continues to increase, the dislocations are able to transmit through the grain boundary, further expanding the plastic deformation zone. Hardness measurements are expected to reflect the occurrence of slip transmission events, and significant pile-ups in particular. A break-through event is visible as a distinct strain burst ($\Delta h$) in the indentation load-displacement curve, which allows grain interactions to be detected and characterised; see (2 & 3) in Figure 5-1B.

![Figure 5-1](image)

**Figure 5-1.** A) Growth of the plastic deformation zone impeded by the grain boundary, causing an elastic stress concentration. B) Instrumented indentation load-displacement curve showing the influence of the grain boundary and a characteristic strain burst.
To study grain interactions, the temporal histories of instrumented indentation load displacement data are analysed for strain bursts. A statistical analysis of grain interaction is carried out for a 225 indentation matrix, where a maximum load of 500 mN is applied in 5 minutes with a quadratic loading curve. With quadratic loading displacement increases approximately linearly with time. Examples of indentation displacement speed histories with and without strain burst are shown in Figure 5-2. The strain burst events represent dislocation pile-up and slip transmission events taking place between neighbouring grains as the size of the plastic deformation zone increases. These strain burst events were observed for both the base metal and weld metal. The average speed of displacement throughout the test is approximately 10 nm/s, compared to the average strain burst (peak) speed of 35 nm/s for the base metal and 23 nm/s for the weld metal. The fastest individual strain burst speeds captured are 339 nm/s for the base metal and 202 nm/s for the weld. The average displacement of a strain burst is 16 nm (1.5...62 nm) for the base metal and 8.5 nm (1.3...30 nm) for the weld metal. The weld metal shows more subtle variations in displacement throughout the test compared to the base metal.

The statistical analysis of strain bursts is carried out for both microstructures from the 225 nano-indentation tests; see Figure 5-3. The weld metal with a broad grain size variation exhibits a significantly larger number of strain bursts compared to the base metal. For the base metal the average number of strain bursts stays below one until an indentation depth of approximately 1400 nm, and shows a steady increase afterwards. For the weld metal, the number of strain bursts already starts to increase at the beginning of the indentation test. In addition, the rate of additional strain bursts starts to decrease as the indentation depth increases. This may be a result of the plastic deformation zone being significantly larger than the grain size, and thus the long-range stresses allow easy transmission of dislocations through grain boundaries without distinct pile-up and slip-transmission events.
The number of strain bursts is further analysed in Figure 5-4, which shows the fraction of tests with at least the specified number of strain bursts as a function of indentation depth. For the base metal 20% of the indentations do not exhibit a single strain burst at the maximum load, associated with individual large grains. The fraction of tests with at least one strain burst increases linearly up to a displacement of ~1000 nm, and continues with a slightly shallower slope up to 2300 nm. For the weld metal 90% of the indentations have at least one strain burst at an indentation depth of approximately 600 nm. In general, the number of strain bursts is significantly higher for the weld metal, with 65% of the tests having at least 12 strain bursts at the maximum test load, while for the base metal the value is at least three strain bursts.

Figure 5-4. Statistical analysis of the number of strain bursts for A) base metal, and B) weld metal. The fraction of tests with at least a specified number of strain bursts are shown as a function of indentation depth. The measured load range is 0…500 mN, and the displacement represents an average value of 225 indentations at a specific load level.

5.2 Influence of grain structure on interaction

The influence of grain structure can be illustrated by plotting hardness as a function of indentation depth; see Figure 5-5 for examples. The strain bursts are visible as sudden drops in hardness. Depending on the microstructure of the material under the indenter and the resistance of the grain boundaries, the number of strain bursts varies. For the base metal (Figure 5-5A) the strain bursts are clearly distinguishable from the otherwise smooth curves. In particular, indentation (a) has a very large drop in hardness at a displacement of ~1000 nm.
For indentation (b) significant hardening is observed before a strain burst occurs at ~1100 nm. The hardness drops generally become smaller as the indentation depth increases (e), and they can be so small as to be indistinguishable from the hardness curve (c). The material gradient can also be detected as a hardening behaviour in indentation (d) at a displacement >1500 nm. The weld metal (Figure 5-5B) shows similar features, although the hardness profiles are more tortuous (g, h, i). Indentation (f) demonstrates two-part behaviour, associated with a material gradient, while (j) shows a very even low hardness throughout the test.

**Figure 5-5.** Hardness as a function of indentation displacement for A) base metal and B) weld metal examples. The insets show enlarged views of the tail ends of two indentations.

To study the drops in hardness in relation to material microstructure systematically, cross-sectioned base metal HV0.05 indentations are analysed in Figure 5-6A. Three different kinds of samples are selected, showing distinctly different hardness profiles as a function of displacement. For indentations 1 and 7 the response is similar except for the hardening and consequent strain burst observed between 1000nm and 1500nm for indentation 7. This is indicative of a dislocation pile-up at a grain boundary, followed by slip transmission to the neighbouring grain. Multiple strain bursts of varying magnitude are visible for indentation 6; they are marked with black arrows in both sub-figures. The displacement speed history for indentation 6 is shown in Figure 5-6B.

**Figure 5-6.** A) Hardness as a function of indentation displacement for cross-sectioned base metal HV0.05 indentations. B) Displacement speed for indentation 6.

To examine the link between strain bursts and microstructure, slip transmission is analysed using EBSD from indentation cross-sections. The first comparison of the grain interaction is carried out between indentations 1 and 7,
shown in Figure 5-6, which share the same hardness profile except for the strain burst at a displacement of ~1500nm. The orientation maps and cross-sectional plane locations are shown in Figure 5-7. Both indentations have struck approximately in the centre of a single grain. These grains have a similar orientation, as both have the side of the cubic unit cell approximately parallel to the specimen surface, and similar slip directions. However, the grain under indentation 1 is significantly larger. For both indentations there is a single large grain beneath the grain, extending through the different cross-sections.

Figure 5-7. Orientation maps (IPF-Z) with reference orientation unit cells overlaid for each grain. Three cross-sectional planes are shown for indentations 1 and 7.
Figure 5-8 shows the dislocation cell structure and misorientation axis directions for indentation 1. The plastic deformation zone covers the majority of the grain underneath the indenter (orange in orientation map), but no transmission has taken place to the next grain underneath (light grey) in plane 1 or plane 2. The edge of the (orange) grain is located near plane 2, as only the edges of that grain are visible in plane 3. Likewise in plane 3, the grain below the indenter (light green) is probably connected to the grain segment of the same orientation located towards the lower left corner, with no transmission taking place.

For indentation 7, the plastic deformation zone has propagated to the next grain (blue in the orientation map). Transmission has probably taken place near plane 1, as the rotation axes show continuity across the grain boundary and the deformation is more severe in that section indicated by small dislocation cells. Furthermore, the grain boundary has a sharp corner, which acts as a geometric stress concentrator.

**Figure 5-8.** Deformation analysis for indentations 1 and 7, showing the plastic deformation zone, misorientation axis (specimen coordinate system) and dense dislocation walls. Plane 1 has residual sample preparation deformation, showing up as streaks throughout the images.
As a second case, indentation 6 with several strain bursts is analysed; see Figure 5-9 for orientation maps. The area consists of many smaller grains, and most of the grains are visible in cross-sectional planes 1 and 2 near the centreline of the indentation. As in the previous case, the indenter tip is located approximately in the centre of a single grain.

![Figure 5-9](image)

**Figure 5-9.** Orientation maps (IPF-Z) with unit reference orientation cells overlaid for each grain. Three cross-sectional planes are shown for indentation 6.

The deformation analysis of the three cross-sectional planes is shown in Figure 5-10. The extent of the plastic deformation, shown by the presence of dense dislocation walls, extends to several grains. At least five significant slip transmission events across different grain boundaries are visible, indicated by the arrows in the figure. This is in good agreement with the multiple strain bursts in the indentation data (Figure 5-6). The first two strain bursts are large, as the deformation transmits from the first grain under the indenter to the adjacent grains. The following bursts are smaller in terms of hardness, as the plastic deformation zone is starting to cover multiple grains, and thus individual slip transmission events will have a smaller influence on the response. These results show that the strain bursts during a hardness test represent dislocation pile-up and transmission events through the grain boundary.

![Figure 5-10](image)

**Figure 5-10.** Deformation analysis for indentation 6, showing the plastic deformation zone, misorientation axis (specimen coordinate system), and dense dislocation walls. Plane 1 has residual sample preparation deformation, showing up as streaks throughout the image.
5.3 Length-scale interaction

The interaction between grains is inherently affected by the size of the plastic deformation zone. The growth of the plastic deformation zone is required to reach the first microstructural barrier, i.e. a grain boundary. Because of to the stochastics of grain size, the size of the plastic deformation zone required for slip transmission will also have a statistical distribution. As shown previously, hardness reflects the local dislocation slip transmission characteristics of the grain structure. To further elaborate the influence of grain interaction on plastic deformation behaviour, hardness measurements are carried out for the two example materials at indentation depths ranging between 100...21 000 nm; see Figure 5-11. This range covers the growth of the plastic deformation zone from a fraction of one grain to tens of grains for both materials. The measured average hardness values, as well as the normalised values, are presented in Figure 5-11. The mean hardness shows a steady increase as the indentation depth decreases, exhibiting the traditional indentation size effect. When compared to the hardness values at 9807 mN test load (HV1), the hardness increases more for the base metal at small indentation depths.

![Figure 5-11. Indentation size effect for hardness measurement load ranging from 1 mN to 16182 mN. A) Absolute values. B) Values normalized with the 9807 mN (HV1) test load results.](image)

The measured hardness distributions are shown for the base and weld metal in Figure 5-12. At large loads the distributions are narrow, and normally distributed. As the load decreases the distributions start to deviate from normal distribution, eventually showing a two-part behaviour, fitting two different distributions. For the base metal this is attributed to the two-phase composition of the material. At an indentation load of 1 mN the probability level of the transition point corresponds with the phase volume fractions (P~0.75, 21±5% pearlite). The weld metal shows similar bi-linearity at an indentation load of 1 mN with a similar transition point at P~0.75. The weld metal has 6.3±1.6 % carbides, with variation in local grain size and dislocation density possibly contributing to the behaviour.
The smallest indentation load at which the distributions follow a single normal distribution is HV0.025 for the base metal and 25 mN for the weld metal. The indentation diagonals at these measurement loads are 1.1 and 0.9 times the volume-weighted average grain size, respectively. At these load levels the plastic deformation zone is large enough to represent the combined response of all material phases. At the lowest load levels of 5 mN and 1 mN the plastic deformation zone is small enough to sample individual phases e.g. only the second-phase pearlite colonies for the base metal.

![Figure 5-12](image)

**Figure 5-12.** Hardness distribution using different indentation loads presented in normal probability plots, including the fitted normal distributions to the linear segments of the distributions. Distributions shown for A) base metal, B) weld metal.

The measurements are characterised by fitting a normal distribution to the lower linear part of the distributions, representing the response of the soft ferrite phase. The coefficient of variation (CV) is used as the characteristic value representing length-scale interaction, defined as the standard deviation divided by the mean value of the fit. The coefficient of variation is shown in Figure 5-13, with a distinct peak identifiable for base metal at HV0.01 test load. A similar peak is not visible for the weld metal, which has a plateau with relatively constant hardness variation below the HV0.01 test load. As the indentation depth increases, the coefficient of variation decreases, and starts to stabilise to a low value. A knee-point can be identified at the HV1 and HV0.3 load levels for the base metal and weld metal, respectively. Hardness variation decreases for the base metal at 25 mN test load as the plastic deformation zone becomes significantly smaller than the grain size in most cases; see Figure 5-14.
Figure 5-13. Coefficient of variation (CV) for normal distributions fitted to the linear sections of the hardness distributions (Figure 5-12).

Figure 5-14. Base metal 25 mN test load indentation cross-sections, showing the misorientation axis and dense dislocation walls for A, B) grains large enough to contain the deformation field, C) a small and large grain under the indenter.

The influence of grain size on hardness variation is investigated in Figure 5-15A, in which the physical indentation size is normalized with the volume-weighted average grain size. In conjunction, the number of strain bursts, i.e. grain interactions, is shown in sub-figure B. With intermediate-sized indentations \((1 - 10^*d_v)\) the spatial grain size and phase dispersion have the largest influence on the hardness variation. The weld metal is more heterogeneous than the base metal, and has, because of to its broader relative grain size dispersion \((\Delta d/d)\) it has a greater hardness variation at the length scales \(1 - 10^*d_v\). The spatial grain size dispersion is reflected in the number of strain bursts, which shows a broad range between 6 – 20 bursts as the indentation size is \(4.4^*d_v\) for the weld metal. This reflects the indentations in the coarse-grained and fine-grained areas of the microstructure, respectively. The spatial variation in the grain size at this indentation size and the corresponding plastic deformation zones are shown in Figure 5-16. The base metal with a narrower grain size dispersion shows a smaller amount of strain bursts on average compared to the weld metal. However, above the indentation diagonal of \(1^*d_v\) the 95% value is similar, and the 5% value (of zero) is smaller.
Figure 5-15. A) Covariance of hardness as a function of indentation diagonal relative to the volume-weighted average grain size for base metal (BM) and weld metal (WM). Percentages indicate fraction of indentations with at least one strain burst for base metal. B) Number of strain bursts and average location of the first and third strain bursts (Figure 5-3), with range 0.1 – 2*dv enlarged in the inset.

Figure 5-16. Weld metal HV0.05 indentation cross-sections, showing the misorientation axis and dense dislocation walls for A) fine-grained region, B) coarse-grained region, C) region with a significant grain size gradient under the indenter.

As the hardness variation stabilizes at a low value for large indentations, the hardness will be mostly sensitive to macroscale material gradients, such as those present over a welded joint, or in the through-thickness direction of a rolled steel plate. Relatively larger indentations are required for weld metal due to its’ broader relative grain size dispersion. The knee-points are located at approximately 7.5*dv for the base metal and 10.6*dv for the weld metal. At these indentation sizes the plastic deformation zone is large enough to cover a large number of grains, and thus the behaviour of the material above this length scale can be assumed to
correlate with the macroscopic response of the material. If the indentation size is increased further, the measurement area will become so large that it will sample the macroscale material gradients, and thus the variation can start to increase again.

The base metal hardness variation peak at the HV0.01 indentation load corresponds to a relative indentation diagonal of 0.63*d. This is in good correspondence with a test that has one strain burst on average (B), shown by the black arrow (1). This indicates that the plastic deformation zone has grown such that the deformation can be transmitted to the next grain, and this has taken place for 51% of the indentations (Figure 5-4A). Thus, a large variation in hardness is expected, as the compatibility for slip transmission between only two neighbouring grains has a large variation, and for half the indentations slip transmission has not yet taken place. The latter is caused either by a strong grain boundary blocking slip transmission, or a large grain size that is able to accommodate the plastic deformation zone. Hardness indentation cross-sections showing the above mentioned cases are shown in Figure 5-17.

For the base metal the fraction of tests with one strain bursts increases from 6% to 51% to 80% as indentation load increases from 25 mN to HV0.01 to HV0.05; see Figure 5-15A. This indicates that the strongest influence of grain interaction on hardness takes place approximately on the length scale 0.3 – 1.5*d for the base metal. Correspondingly, the average number of strain bursts has a slow linear increase between 0.2 – 0.7*d; see Figure 5-15B. Beyond the HV0.01 peak in the hardness variation, there is a significant increase in the strain burst slope above approximately 0.7*d, corresponding to the average location of the first strain burst; see the inset in Figure 5-15B. This indicates that as the dislocations are transmitted through the first grain boundary, subsequent grain boundaries are easier to overcome as the plastic deformation zone becomes larger.
The weld metal exhibits a similar increase in the strain burst rate after the first burst; however, this change is less pronounced. To explain the hardness variation plateau for weld metal, strain burst histograms for the 225 indentations (Figure 5-3) are shown in Figure 5-18. The strain bursts are divided into low- and high-speed categories, based on the bi-linear nature of the measured distributions. The threshold for high speed bursts is 22.9 nm/s for base metal and 13.9 nm/s for weld metal, which is approximately 20% of all strain bursts for both cases. Analysis is focused on the high speed strain bursts as they are related to the large drops in hardness during a test. Figure 5-19A shows that for the base metal the number of high-speed strain bursts starts to increase at 0.1*d_v , reaching its peak at approximately 0.4*d_v . A decline in strain bursts is visible until 0.67*d_v , coinciding with the peak in hardness variation, see Figure 5-19C. The decrease in rate of strain bursts is indicative of strain hardening, i.e. grain interaction. Beyond this point the number of strain bursts increases rapidly as the first barrier to dislocation motion has been overcome.

For the weld metal the low-speed bursts are observed with a slightly decreasing trend throughout the test, while high-speed bursts are concentrated to indentation depths below 1000 nm; see Figure 5-18B. As shown in Figure 5-19B&D, the depth range of the high-speed strain bursts (0.1-2*d_v ) coincides with the plateau in the hardness variation. This indicates that in this region it is possible to have significant individual slip transmission events, while at greater indentation depths the plastic deformation is not as strongly affected by individual grain interactions.

![Figure 5-18. Histograms of low- and high-speed strain bursts for A) base metal, and B) weld metal. Bin width for the histograms is 100nm.](image-url)
Figure 5-19. Histogram of high-speed strain bursts for A) base metal, and B) weld metal. The correlation between strain burst histograms and hardness covariance is shown in (C) and (D), correspondingly.
5.4 Rotation of the crystal lattice

In order to have a better understanding of the influence of grain size dispersion on localised microstructural deformation, a lattice rotation analysis is carried out for the base and weld metal. Figure 5-20 shows the results of the misorientation analysis for a base metal HV0.05 indentation. The misorientation axes in the specimen coordinate system (A) show that different colours join at a central point, coinciding with the misorientation minima. The misorientation axis distribution is shown in a pole figure in (B), revealing strong symmetry in the misorientation axis directions. Most misorientation axes with a misorientation angle higher than $2^\circ$ follow a single path, and a plane can be fitted through the data points. To give this plane crystallographic meaning, its normal vector is compared to the possible $<-1 1 1>$ slip directions. A close match is found with the $[1 1 -1]$ slip direction, indicating dislocation slip along this plane. The misorientation axes in the crystal coordinate system are shown in (C). Traces corresponding to the $\{110\}$ and $\{211\}$ slip planes radiate outward from a central point. This indicates a screw dislocation, with the motion of dislocations outward from the central point. There seems to be slip activity in all the $\{110\}$ and $\{211\}$ slip planes of the $[1 1 -1]$ zone axis, with the traces following the predicted directions of dislocation motion; see Appendix D for further details. The distribution of the misorientation axes in the crystal coordinate system is shown in the inverse pole-figure (D), with the highest density in the proximity of the $[0 1 1]$ direction.

![Image](image.png)

**Figure 5-20.** Misorientation axis plot in A) specimen coordination system, and C) crystal coordinate system. B, D) Misorientation axis distribution for the central grain in corresponding coordinate systems.

The dislocation motion has generated a grain sub-structure, evidenced by the formation of sub-grain boundaries and dislocation cells in Figure 5-21A&B. On the basis of the analysis, the dislocation cells are smallest at the screw dislocation
core (500 nm), increasing to 1800 nm in the outer region of the grain. The smallest dislocation cells coincide with the largest kernel misorientation angles (C). On the basis of the similitude principle [76,154], the size of the dislocation cells is proportional to stress that is applied. There are large stresses in the screw dislocation core region, decreasing as a function of 1/r when moving outward from the node location [155]; see Figure 5-22A. As shown in Figure 5-22B, the dislocation cell size follows the theoretical stress with the exception of the surface region affected by the indenter strain gradient, which shows large misorientation angles. The misorientation to the reference orientation has a local minimum approaching 0° at the core. This is consistent with the fact that atoms are displaced in the core region [156], rather than being rotated. Thus, misorientation to the reference orientation cannot capture the high stresses of the displacive motion, and kernel misorientation is a better measure of the local strain.

![Figure 5-21. Evolution of dislocation cell size inside a single grain. The average cell size is measured inside the averaging intervals (B). The size is compared to the kernel misorientation (A) and misorientation angle (D) in (C) and (F), respectively. The three red lines represent minimum, mean and maximum values. The misorientation axis in the crystal coordinates is shown in (E).](image-url)
Figure 5-22. A) Dislocation cell size and theoretical stress distribution around the screw dislocation. B) Dislocation cell size as a function of normalised theoretical stress.

In the experiments misorientation axes emanating from a node, similar to that shown in Figure 5-21, have been observed to occur frequently. In Figure 5-23A multiple features can be identified in the single grain, coinciding with locally fine dislocation cells in Figure 5-23C. Depending on the orientation of the grain and the active slip direction, alignment of the features relative to the cross-sectional plane is changed. When the slip direction is in-plane, contrary to pointing out-of-plane in previous examples, loops radiating outward from a single line are visible in Figure 5-23B. The contours provided by the kernel misorientation analysis show good agreement with these features in Figure 5-23D. In general, the misorientation axis and kernel misorientation contours show good agreement, confirming that clustering has yielded a good estimate for the reference orientation.

Figure 5-23. A, B) Analysis showing misorientation axis in the crystal coordinate system for two base metal HV0.05 indentations and C, D) Corresponding kernel misorientation analysis showing dislocation cell structure and dense dislocation walls.

Similar features are visible in the weld metal as well, shown by the arrows in Figure 5-24A. The contours and node locations show good agreement with the sub-grain boundaries in Figure 5-24B. The size of the dislocation cells is generally
extremely small, with the 0.06 µm step size not resolving the finest features reliably, particularly at the locations indicated by the two arrows in the centre in Figure 5-24C. Otherwise, the locations correspond with locally fine dislocation cell structures, and the misorientation axis contours (A) show fair agreement with either the sub-grain boundaries (B) or dense dislocation walls (C), shown in the enlarged sections in the right-hand column of the figure. Thus the deformation behaviour of the structural steel base metal and weld metal that were investigated share many of the same characteristics, spanning length scales ranging from 0.1 µm to over 100 µm.

**Figure 5-24.** Weld metal HV0.05 indentation showing A) misorientation axis in the crystal coordinate system, B) sub-grain boundaries, and C) dislocation cell structure and dense dislocation walls. An enlarged section from the middle is shown in the right-hand column.
6. Discussion

6.1 Statistical homogenisation of welded steel

The homogenisation of structural elements, as well as material properties, is required for computational efficiency in advanced structural design. At present, homogenisation is utilised in structural response analysis to define stresses for strength analysis, while strength properties are determined experimentally. Thus, the influence of microstructural variation on the strength of the material is only implicitly considered. The consideration of microstructural effects is becoming increasingly important as modern manufacturing techniques and new materials are utilised in the production of ships and other large steel structures. This presents a challenge to the strength assessment, particularly in fatigue analysis where local plastic deformation controls the failure process. Traditional design methods for fatigue are based on the assumption that macroscopic cracks are induced by the manufacturing process and thus crack propagation determines the fatigue life of a structure. In these cases the fatigue strength of a structure is insensitive to its microstructure, as macroscopic crack propagation rates show only minor differences between different structural steel grades [157]. By utilising modern manufacturing methods [31,32,158], the size of imperfections is reduced to such levels that the time to crack initiation determines the fatigue life [51,52,159]. The initiation and growth of microstructurally small cracks are highly sensitive to the local material properties and microstructural barriers [160]. In order to consider these aspects in continuum-based models, statistical homogenisation of the microstructure and material properties is required.

This thesis investigated how the geometric characteristics of the microstructure affect the local plastic deformation of polycrystalline BCC steel, and how they should be statistically homogenised for welded steel at different length scales. On the basis of the investigations, the role of grain interaction was established as a function of indentation size, shown schematically in Figure 6-1. The intermediate range in which grains interact, ranges in diagonal length of the indentation from approximately 0.1 to 10 times the volume-weighted average grain size. Thus, the influence of the microstructure on the local plastic deformation will be prominent, shown in recent full-field strain measurements of short fatigue cracks [160]. The influence of grain interactions on hardness diminishes as the plastic deformation zone becomes large, with a knee-point observed at $7d_v$ and $10d_v$ for the base.
metal and weld metal that were investigated, respectively. The larger knee-point for the weld metal is a consequence of the broader grain size dispersion ($\Delta d/d$). This point of ‘homogeneous response’ corresponds to a physical indentation size approximately 105 µm for the base metal and 50 µm for the weld metal. The defined length scales can be compared to the micro-support theory developed by Neuber [35], where a material dependent microstructural length $\rho^*$ is used for stress concentration analysis. The parameter $\rho^*$ is defined based on the fatigue tests and it is material dependent, also affected by yield strength. For the investigated base metal and weld metal, Neuber’s approach gives $\rho^*$ values of 80 – 100 µm and 40 – 60 µm, respectively. Similar values have been found suitable for the fatigue analysis of laser-hybrid steel welds by Liinalampi et al. [46]. Thus, the results of the present study are in good agreement with the results obtained from fatigue testing. However, these observations contradict the commonly used assumption that mild cast steel properties ($\rho^*=400$ µm) should be used for the notch stress fatigue analysis of welded joints [161,162].

On the basis of the above findings, the assumption of homogeneous material properties in continuum modelling is not valid for welded steel, when the length scale is smaller than 50– 100 µm, depending on the microstructure. Below this limit microstructural factors should be considered in e.g. fatigue assessment. This is an important finding since some local fatigue approaches use a continuum theory for very small computational units, e.g. a fictitious rounding radius of 50 µm; see e.g. Radaj et al. [163]. Thus, in order to further develop existing local fatigue approaches for welded joints, appropriate characterisation of microstructural features and an understanding of the factors affecting local plastic deformation are required. These issues are discussed in the following section, which identify the main factors that should be considered in statistical homogenisation. Particular attention is given to the interaction of length scales, and revealing the underlying deformation mechanisms.

**Figure 6-1.** Contribution of grain interaction to plastic deformation response as a function of length scale.
6.2 Modified Hall-Petch relationship

The Hall-Petch relationship is the most commonly used engineering approach for the characterisation of microstructural effects on strength. As the Hall-Petch relationship is related to the measure of grain size, the correct definition of the effective grain size is crucial. Typically the average grain size is used to describe the microstructure [66], but its suitability for heterogeneous microstructures is questionable. Several investigations of single-phase base metals have shown that the grain size distribution has an effect on the mechanical properties [60–66]. The present study of multi-phase weld metals confirms this tendency, with broader grain size dispersions showing lower hardness values.

To consider the variation in the grain size dispersion, the volume-weighted average grain size \(d_v\) is introduced in this work to replace the commonly used average grain size \(d\). The observed correlation between the Hall-Petch grain size parameters \(d^{-1/2}\) and \(d_v^{-1/2}\) provides the means for accurate estimation of the volume-weighted average grain size based on the broadness of the grain size dispersion \((\Delta d/d)\). The observed relationship is validated by measuring the grain size dispersion of several microstructures from the literature [164–167]. As shown in Figure 6-2, the data covers a wide range of grain size dispersion and conforms well to the experimental relationship determined in this thesis.

![Figure 6-2. Relationship between the average \((d)\) and volume-weighted average \((d_v)\) grain size as a function of the relative grain size dispersion \((\Delta d/d)\). Comparison of data in this thesis to analysis of literature data for various metallic materials and microstructures. The linear fit and related confidence and prediction bounds are based on the data of this thesis only [135]. Microstructural images of Dahlberg, Janssen, Mikkola and Lehtimäki are from references [164–167].](image)

To consider the influence of grain size distribution on strength, the volume-weighted average grain size is implemented into the Hall-Petch relationship. The volume-weighted average grain size can be determined experimentally or through linear regression with Equation (3). The modified Hall-Petch relationship for yield strength is then correspondingly:

\[
\sigma = \sigma_0 + k d_v^{-1/2} = \sigma_0 + k d^{-1/2} \left(1 + f \frac{\Delta d}{d}\right),
\]

(4)
A corrective term $f \Delta d/d$ is added to the Hall-Petch relationship ($f = -0.0635$; see Figure 6-2). For varying grain size dispersions it is able to improve the Hall-Petch relationship’s predictive accuracy significantly for both base and weld metal.

The modified Hall-Petch relationship is applied to the simulated yield strength of steel from Berbenni et al. [64] in order to validate the relationship with additional data. The distinct dependence of strength on the grain size distribution is shown when the average grain size is used in Figure 6-3A, with broad grain size dispersions reducing the strength significantly, especially for small average grain sizes. As the modified Hall-Petch relationship is applied to the data in Figure 6-3B, the volume-weighted average grain size yields a very good estimate of the yield strength for all grain size dispersions; all grain size dispersions are shifted close to the $\Delta d/d = 0$ slope. It should be noted that when engineered correctly, a broad grain size dispersion can also augment the material properties, as is the case for functionally graded materials [168]. A functionally graded microstructure has mechanical incompatibility because of its grain size gradient. A combination of strain gradients and back-stresses caused by the dislocation pile-ups enhances the yield strength and ductility of the material [168]. This highlights the need for spatial grain size characterisation methods.

![Figure 6-3. Mechanical response of steel as a function of average $d$ and volume-weighted average $d_v$ grain size: A) simulated results of Berbenni et al. [64], B) Eq. (9) applied to the same data to determine the volume-weighted average grain size.](image)

To consider the influence of grain size distribution, Kurzydlowski [69] has proposed an alternative approach, in which the strength of different grain sizes was estimated by applying a weighting factor equal to the volume of the grains. This approach was further developed by Raeisinia and Sinclair [70]. They proposed an analytically derived geometric grain size parameter, the representative grain size ($d_R$), which eliminates the influence of grain size distribution on the Hall-Petch relationship. The representative grain size is compared to the volume-weighted average grain size in Figure 6-4A. Both approaches yield similar results, despite the assumed shape similarity of all the grains (spherical) and a log-normal grain size dispersion for $d_R$. The experimental relationship for $d_v$ based on the rule of mixtures does not make these
assumptions, and yields a larger grain size for broad grain size dispersions, as shown in Figure 6-4B.

Figure 6-4. A) Comparison of the volume-weighted average grain size \( d_v \) against the representative grain size \( d_R \) defined by Raeisinia and Sinclair [70] for the simulated yield strength of steel by Berbenni et al. [64], also shown in Figure 6-2. B) Comparison of the ratio between the two parameters as a function of the relative grain size dispersion.

Grain size heterogeneity is often an issue when producing nanocrystalline materials. In this case, the perception of a lower-than-expected strength may be an artefact caused by the average grain size, in addition to the deformation mechanism changing from dislocation slip to grain boundary sliding with grain sizes smaller than 100 nm [169]. The modified Hall-Petch relationship developed in this thesis is applied to the simulated yield strength of nanocrystalline copper in Figure 6-5. Despite copper having a relatively weak grain size-yield strength dependence, the reduction in strength is apparent for broad dispersions in sub-figure (A). With the modified Hall-Petch relationship, shown in sub-figure (B), the grain size dispersion dependence is reduced significantly. Only the broad dispersions (\( \Delta d/d = 6 \)) are slightly above trend, which may be caused by the truncated distribution missing the largest grains in a finite simulation volume. This shows the versatility of the approach, with applicability to a wide range of materials that show grain size-dependent mechanical properties.

Figure 6-5. Mechanical response of nanocrystalline copper: A) as a function of average grain size for the simulated results of Ramtani et al.[66], B) Eq. (9) applied to the same data to determine the volume-weighted average grain size.
6.3 Length-scale interaction

The Hall-Petch relationship holds when the plastic deformation zone is large in comparison to the average grain size. For large deformation zones, the dependence of different macroscopic strength parameters on the grain size is widely reported in the literature for a large variety of materials [59,170,171]. The macroscopic hardness in this work for structural steel weld metals was also shown to follow a grain size dependence. However, as the size of the deformation zone decreases, a correlation between grain size and hardness can no longer be found. The interaction of the size of the plastic deformation zone with grain size was found to play a key role in the local deformation of polycrystalline steel, when the plastic deformation zone radius is smaller than approximately two times the volume-weighted average grain size ($d_v$).

On the basis of Chapter 4.3 there is generally a trend of large local grain size yielding large deformation zone size for both base metal and weld metal. This holds true at the largest indentation load of HV0.05. As load is reduced (HV0.01), the base metal deformation zone sizes show little correlation with grain size. This is related to the interaction of the grains, which is strongest for the base metal at this test load. Therefore, depending on the local grain size and compatibility of grains for slip transmission, the size of the plastic zone can vary significantly. Figure 6-6 shows an example of the evolution of deformation zone sizes. As the pile-up starts affecting the measured response at an indentation depth of 1210 nm, the plastic deformation zone approaches the grain boundary. As slip transmission takes place at an indentation depth of 1580 nm, the grain refined zone reaches the grain boundary, and the plastic deformation zone extends beyond the grain boundary. After slip transmission the plastic deformation zone mostly grows in the next large grain (shown in blue), following the path of least resistance, reaching its final size at an indentation depth of 3420 nm. Despite the variation observed in deformation zone sizes the average values correlate with the volume-weighted average grain size (Figure 4-16), agreeing with the Eshelby-Frank-Nabarro model [22] which the Hall-Petch relationship is based on.

![Image](image_url)

**Figure 6-6.** Estimated deformation zone sizes for pile-up and transmission, and the measured final deformation zone sizes overlaid on the orientation map (IPF-Z) for the base metal HV0.05 indentation #7 shown in Figure 5-7.
6.4 Microstructural characterisation methods

The characterisation of heterogeneous polycrystalline microstructures imposes many challenges, starting from the definition of grains. In the case of welded joints continuous grain boundaries can be difficult to identify because of irregular grain shapes (anisotropy) and orientation gradients inside the grains caused by the welding heat cycle. In this work the volume-weighted grain size measurement method was developed by adding capabilities for the measurement and visualisation of the spatial variation in the grain size, including kernel-based averaging to represent grain size dependent mechanical properties better. Since the relationship between average and volume-weighted average grain sizes is established on the basis of the relative grain size dispersion, the volume-weighted average grain size can also be estimated with the ASTM E1382 [139] linear intercept method. Because of the general nature of the grain size measurement methods that were developed, they are applicable to research on a wide range of material research. The grain size measurement methods developed in this thesis are suitable for the characterisation of various granular materials, with the open-source codes [136] having been applied to e.g. high-aluminium steel [172], aerospace titanium alloy forgings [173], polycrystalline ice [174], ferroelectric ceramic materials [175], and eolianite (rock formed by the lithification of sediment deposited by the wind) [176].

The characterisation of plastic deformation in heterogeneous polycrystalline microstructures is an additional challenge. Plastic deformation has traditionally been measured by cross-sectioning deformed material. In many cases hardness indentations of varying sizes are used to create localised deformation and study plastic flow [177–183]. Using traditional serial sectioning methods, the hardness indentations have to be very large for the characterisation. Srikant et al. [184] cross-sectioned Vickers indentations with a diagonal length of 800 µm, and mapped the plastic deformation with smaller, 22 µm diagonal indentations. This limits the spatial resolution of the method, as the small indentations are spaced at 80 µm intervals. A focused ion beam (FIB) has also been used in conjunction with TEM and SEM to reveal detailed microstructural information on plastic deformation, see e.g. [177,185,186]. These methods enable very small indentations, in the order of 1 µm, to be characterised effectively. However, FIB sample preparation is time-consuming, limiting the size/number of indentations that can be mapped, and in TEM it is commonly applied only to single crystal materials [187]. The custom cross-sectioning methodology developed in this thesis allows small indentations less than 1.5 µm in indentation depth to be characterised accurately with EBSD. The key advantage over FIB is that multiple indentations and large surface areas of interest are mapped efficiently, allowing the stochastics of deformation to be captured for heterogeneous polycrystalline materials. On the other hand, removal of residual sample preparation deformation can be challenging for the serial sectioning methodology. As shown in Figure A-9 of Appendix A, significant plastic deformation can be caused by sample preparation, masking the edges of the deformation zones. This deformation is not revealed by EBSD indexing rates or evaluation of surface
flatness. Forescatter detector images, as shown in sub-figures A-C, are sensitive to orientation changes, revealing continuous streaks and patches of deformation far from the indentations. While the grain refined zone can be defined quite accurately (D-F) even with moderate levels of sample preparation deformation, the plastic deformation zone (G-I) requires well-prepared sample surfaces. Thus, there is an uncertainty associated with the individual plastic deformation zones. This is particularly the case for weld metal, where the heat cycle of welding also induces plastic deformation. Despite these uncertainties, the trends and estimated average values in Chapters 4.3 and 4.4 give a good representation of the differences between the two examined materials, with differences far greater than the measurement uncertainty. For the above-mentioned reasons the grain refined zone was also used for the comparison of plastic deformation zone size and shape.

Plastic deformation can be characterised in several ways using EBSD [121]. Most commonly plastic deformation is measured by comparing the orientation of crystal lattice in different locations. Misorientation analysis is usually carried out relative to 1) the average grain orientation, or 2) to the nearest neighbours in a square-shaped calculational domain (kernel). Both methods have several aspects that need to be addressed prior to their application to steel welds. The present work shows that in order to reveal the sub-structure and deformation mechanisms effectively, the orientation data has to be post-processed. The traditional Hough transformation used in EBSD has an angular resolution of approximately $0.5\ldots1.0^\circ$, making the analysis of a smaller misorientation difficult. For a small misorientation of $1^\circ$, an orientation measurement error of $1^\circ$ causes an error of $45^\circ$ in the misorientation axis direction, meaning that it is practically unknown [188]. This can be overcome in traditional EBSD by using the pattern-matching algorithm by Nolze et al. [189,190] to improve the angular resolution from $0.5^\circ$ to approximately $0.05^\circ$. The post-processing of orientation data using filters is more commonly applied, with smoothing and edge enhancement [120,151,152] often being carried out. In this work the combination of a half-quadratic filter [150] that preserves that sharp transitions in combination with an edge-enhancing Kuwahara filter [153] were found to reduce the measurement noise optimally without detrimental effects on sub-grain orientation gradients, i.e. dislocation sub-structures.

The analysis of grain sub-structures is usually carried out with kernel misorientation analysis, with a kernel size between $1\ldots3$ nearest neighbours irrespective of its physical dimension, with the large kernels of 10 nearest neighbours smearing the grain sub-structures [121]. However, on the basis of the current research, details of the grain sub-structure are revealed in more detail as the kernel size is increased further. This is due to the fact that the large kernel sizes of 30 – 100 nearest neighbours cover entire sub-grains, providing more robust statistical analysis with the number of data points expanding up to $3720 – 40400$ (when within the misorientation threshold). On the basis of the above results, the kernel size should be in proportion to the physical size of grains or sub-grains. In this study, a physical kernel size slightly smaller than the volume-
weighted average grain size was found to be optimal for both the base metal and weld metal.

While the kernel misorientation methodology that was developed captures the grain sub-structure and the extent of the plastic deformation effectively, grain-based analyses were used to provide an in-depth understanding of the material behaviour caused by the motion of dislocations. While the average orientation of a grain is easily attained and commonly applied to sub-grain deformation analysis [55], it is only applicable when the general level of deformation inside a grain is low. The clustering-based reference orientation implemented in this work considers 50% of the lowest local misorientations, and groups them according to the similarity of their orientation. This removes single outliers effectively, and the cluster with the highest orientation density provides a good estimate of the least deformed orientation cluster in the cross-section containing the deformed grain. The accuracy of the reference orientation is particularly important for the analysis of material rotation axis directions in order to predict e.g. the directions of dislocation motion.

The effective analysis of localised plastic deformation requires several misorientation analyses to be assessed in conjunction. By combining the two misorientation analysis methods that were developed, the misorientation minima associated with screw dislocations were revealed, as was shown in Chapter 5.4. Similar features were observed for the base metal and weld metal. Depending on the orientation of the <11-1> slip direction in relation to the cross-sectional plane, the minima can be circular, rectangular or wedge shaped. The direction of the deformation was found to be consistent with the <11-1> slip direction (Figure 5-17). These observations indicate a cylindrical zone with low lattice misorientation angles in the core region of a screw dislocation. This is confirmed by analysing the work of Kiener et al. [177] for Vickers indentations in single crystalline tungsten (BCC). On the basis of the given crystal orientation, the most favourable <11-1> slip directions are overlaid on the original results in Figure 6-7. Because of the experimental setup, the [-1-11] and [-111] slip directions are aligned symmetrically in relation to the indentation direction, facing 28° out-of-plane. Misorientation fields progress in the [-111] direction, and a wedge shaped misorientation minimum appears near the indenter centre line (D) and starts to disappear in sub-figure (C). In cases where the slip direction is (nearly) in the plane of observation, the wedge shape is minimised, approaching a rectangular shape for the base metal HV0.01 indentation in Figure 6-8A. Otherwise the misorientation field is very similar to the single crystalline tungsten shown in Figure 6-7D. While the misorientation angle provides the approximate extent of the deformation [177], the kernel misorientation (threshold <0.5°) in sub-figure D reveals that the entire grain has been deformed. This stands in contrast to the low level of deformation in the neighbouring grain on the right-hand side.
Figure 6-7. Misorientation angle beneath a Vickers indentation in single crystalline tungsten [177]. Four cross-sections are shown, with distances to the indenter centre line of: A) 7.57 µm, B) 7.28 µm, C) 4.25 µm, D) 2.14 µm. The <1 1 -1> directions are added on the basis of the given crystal orientation.

Figure 6-8. Analysis of base metal HV0.01 indentation in close proximity to the indentation centreline (0.12 µm offset). A) Misorientation angle, B) misorientation axis in crystal coordinate system, C) sub-grain boundaries (KMM), D) dislocation cells and dense dislocation walls (KAM).
7. Conclusions

In this work, the influence of the material microstructure on the mechanical properties of welded steel was studied using instrumented indentation testing. To measure the strength of the material on different length scales, the size of the indentations was varied from a fraction of one grain to tens of grains. To link the deformation characteristics to a microstructural length scale, an innovative serial sectioning procedure and kernel misorientation analysis method were developed. This enabled an extensive statistical analysis of plastic deformation for the heterogeneous polycrystalline materials with ~150 cross-sections measured. The plastic deformation zone was divided into two regions: 1) a grain refined zone containing sub-grain boundaries, i.e. the highly deformed volume, and 2) a plastic deformation zone that covers the extent of the dislocation cells and dense dislocation walls. In order to obtain a proper analysis of the localised plastic deformation zone, careful sample preparation and advanced post-processing of the EBSD data were required. This is especially important for heterogeneous polycrystalline materials such as structural steel weld metals.

The analysis of the material response on different length scales revealed the importance of the grain size dispersion to the strength properties. When macroscopic properties are of interest, the indentation size has to be significantly larger than the average grain size (diagonal >7–10*\(d_v\)), with broad grain size dispersions requiring larger indentations than narrow dispersions. For the investigated base metal and weld metal statistical homogeneity of achieved with approximately 105 µm and 50 µm indentations. In this regime, the average grain size correlated with the measured hardness values when the grain size dispersion was considered. By extending the original Hall-Petch relationship with a relative grain size dispersion (\(\Delta d/d\)) parameter, the consideration of different grain size dispersions is possible and good agreement is observed for different materials and grain size dispersions. Alternatively, the volume-weighted average grain size (\(d_v\)) based on the rule of mixtures, can be used to describe the grain size dispersion.

For the structural steel materials that were studied, the influence of the material grain structure on plastic deformation becomes more significant as the indentation size decreases (diagonal 1–10*\(d_v\)), reflecting the spatial variation of mechanical properties. The spatial variation is related to the relative grain size dispersion (\(\Delta d/d\)), with heterogeneous weld metals showing more variation than base metal. A transition region was defined between the continuum and single-crystal material behaviour, where the interaction of differently-sized grains...
controls the local plastic deformation of polycrystalline steel. The strongest grain interaction effect was found to take place at indentation diagonal lengths $0.1 - 2^*d_v$, when slip transmission primarily occurs between two grains. For broad grain size dispersions grain interaction takes place over a wider range without a distinct peak. On the basis of the extensive experimental database gathered by cross-sectioning hardness indentations, the interaction grains of different size was found to control the local plastic deformation in the case of polycrystalline BCC steel. Furthermore, a comparison between the base metal and weld metal revealed that the size of the plastic deformation zone is proportional to the volume-weighted average grain size, verifying the theoretical assumption behind the Hall-Petch relationship of grain boundary strengthening being proportional to grain size.

This thesis investigated the influence of the microstructure on the localised plastic deformation commonly observed for fatigue and fracture phenomena. The mechanical properties of welded steel were characterised using instrumented indentation testing and systematic microstructural analysis of local plastic deformation. This was obtained by focusing the study onto a limited sample size and limited material volume. This enabled a fundamental understanding of deformation mechanisms of steel on different length scales, providing a foundation for investigating deformation under realistic loading conditions. Since the local plastic deformation process is a stochastic phenomenon and a large structure can have up to a thousand kilometre of weld seams, there is statistical uncertainty associated with the current measurements. Therefore, it is recommended as a future work that the established theoretical framework is applied to a large variety of different steel grades and welded joints to have higher statistical reliability and more robust conclusions in term of structural analysis. Current investigations have been carried out in room temperature, and should be extended with cold temperature testing where ductility is limited. Furthermore, the capabilities of instrumented indentation testing should be further utilised for the characterisation of material under cyclic loading to replicate the reversible motion of dislocations observed in fatigue. For further validation of the method, the dislocation sub-structures resolved with EBSD need to be compared with direct measurements using TEM, as well as with pattern matching for an improved angular resolution in EBSD experiments. To establish local microstructure-strength relationships, three-dimensional analysis of grain size and deformation fields is also recommended as a future work.
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Appendix A. Hardness indentation cross-sectioning

Details of the hardness indentation cross-sectioning procedure, shown schematically in Figure 2-3, are presented in this appendix. A transverse cross-section with the hardness measurement matrix is shown in Figure A-1. The top and right-hand-side surfaces are used as references for measuring the location of the indentations in serial sectioning. On each side of the indentations a series of 13 markers are placed, as shown in Figure A-1B. The distance from the surface is varied between 25 µm and 81 µm, with the finest resolution of 2 µm near the indentation centreline at 72 µm. The markers have a fixed indentation depth of 2.8 µm. This provides an accurate gauge to measure material removal using the method shown in step 7 of Figure 2-3. The distance from the right-hand-side reference surface is used to identify the markers in serial sectioning. The indentations to be cross-sectioned are placed between the markers, using the test loads HV0.0025, HV0.01, and HV0.05 (10x, 11x, 10x). The approximate indentation depths (and diagonals) are 0.7 µm (5 µm), 1.4 µm (10 µm), and 3.4 µm (24 µm), respectively.

Figure A-1. A) Low magnification micrograph of the hardness measurement matrix in the base metal. B) Enlarged view of the markers used, with numbers corresponding to their distance from the top surface in µm. C) Enlarged view of the indentations for cross-sectioning, and their Vickers load ratings.
The alignment of the specimen in the sample preparation is crucial for maintaining a deformation-free material edge. As shown in Figure A-2, the leading edge relative to the direction of the abrasives has a severe deformation layer of 40 µm. The steel support reduces the rounded and deformed edge layer to 25 µm for the leading edge, with the trailing edge having only an 8 µm deformation layer. Thus all the grinding and polishing of the samples is carried out manually, with the rotation direction of the abrasives being towards the steel support, as shown in Figure A-1A. The alignment of the specimen is only altered 10…20° from this direction between different grinding and polishing steps in order to distinguish the scratch marks from each other.

![Figure A-2. Influence of a steel support and grinding direction on the deformation of the sample edge.](image)

After the hardness measurements have been carried out, the sample is carefully broken out of the mount for nickel plating. Without the nickel plating the near-edge region could not be reliably indexed with EBSD and would contain a high level of deformation. The nickel plating enhances edge retention significantly and eliminates the influence of sample edge deformation on EBSD measurements.

The electroless plating bath composition by Cheong et al. [191] was used. The chemical composition and plating procedure are shown in Table A-1. To avoid sudden bath decomposition, using thiourea as a stabiliser was critical. With an addition of 2 mg/l, Cheong et al. [191] measured the bath decomposition time as increasing from approximately 1 minute to over 264 minutes. Furthermore, the deposition rate increases significantly for concentrations between 1…3 mg/l, while a concentration of 5 mg/l totally prevents the nickel plating reaction from happening. A concentration of 2.5 mg/l was used, yielding an approximately 55-µm-thick nickel layer with a plating time of 2 h 30 min (22 µm/h).
The most critical part of the preparation of the sample is serial sectioning, with the nickel-plated surface (A) mounted in an epoxy resin with a steel support. The steel support is offset to one side to indicate the corner where the hardness indentations are located. The initial stage is to remove the nickel layer and locate the first indentation markers, with their centres located at a depth of 25 µm, extending 5 µm on each side. Short successive material grinding steps are performed, followed by optical observation until the first indentations are visible. This allows approximate determination of the cross-sectional plane, guiding further material removal.

Before any serial sectioning can be performed, a pre-study is required to measure the approximate material removal rate of all the sample preparation steps. This was carried out for the S355 structural steel by indenting the sample surface with several Vickers indentations (HV10 and HV0.1). The initial diagonal lengths of the indentations are measured, and as the material is ground or polished, the changes in indentation size can be used for estimating the layer of material removed through the geometry of the indentation. Each grinding step was performed for 5 – 10 seconds at the outer radius (r≈ 100 mm, 300 rpm) for the measurement of material removal, and repeated 2..3 times for a more accurate average value. Diamond polishing and colloidal silica removal rates were measured by utilising the marker indentations in the cross-section. The measured values are presented in Table A-2. It should be noted that the material removal rate of abrasive SiC discs decreases rather quickly, up to 50% within 30 seconds. In the experiments a single disc was typically used up to 1-2 minutes.

### Table A-1. Chemical composition of the bath and sample preparation for nickel plating.

<table>
<thead>
<tr>
<th>Chemical composition</th>
<th>Concentration</th>
<th>Notes</th>
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<tbody>
<tr>
<td>Distilled water</td>
<td>H₂O 500 ml</td>
<td></td>
</tr>
<tr>
<td>Nickel Sulphate</td>
<td>NiSO₄ 30 g / l</td>
<td></td>
</tr>
<tr>
<td>Sodium hypophosphate</td>
<td>NaH₂PO₂ 20 g / l</td>
<td></td>
</tr>
<tr>
<td>Sodium acetate</td>
<td>NaC₂H₃O₂ 20 g / l</td>
<td></td>
</tr>
<tr>
<td>Thiourea</td>
<td>CH₄N₂S 2.5 mg / l</td>
<td></td>
</tr>
<tr>
<td>Sulphuric acid</td>
<td>H₂SO₄ 6 drops</td>
<td>pH controlled to 5.8</td>
</tr>
</tbody>
</table>

Plating temperature 83 ± 2°C, no agitation

### Table A-2. Sample preparation.

<table>
<thead>
<tr>
<th>Sample preparation</th>
<th>Time</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultrasonic cleaning with ethanol</td>
<td>5 min</td>
<td></td>
</tr>
<tr>
<td>Rinse with distilled water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cleaning, immersion in 10% NaOH</td>
<td>2 min</td>
<td>Directly from rinsing</td>
</tr>
<tr>
<td>Rinse with distilled water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Surface activation with Buehler pre-clean (&lt;1% HCl)</td>
<td>15s</td>
<td>Directly from rinsing</td>
</tr>
<tr>
<td>Rinse with distilled water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nickel plating</td>
<td>2h30min</td>
<td>Directly from rinsing</td>
</tr>
</tbody>
</table>
Table A-2. Measured approximate material removal rates for all sample preparation steps (base metal)

<table>
<thead>
<tr>
<th>Preparation step</th>
<th>Total time</th>
<th>Total removal</th>
<th>Removal rate</th>
<th>Particle size</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 P320*</td>
<td>5 s</td>
<td>13.2 µm</td>
<td>2.6 µm/s</td>
<td>40.5 µm</td>
</tr>
<tr>
<td>2 P800*</td>
<td>10 s</td>
<td>18.0 µm</td>
<td>1.8 µm/s</td>
<td>25.8 µm</td>
</tr>
<tr>
<td>3 P1200*</td>
<td>20 s</td>
<td>12.9 µm</td>
<td>0.64 µm/s</td>
<td>15.3 µm</td>
</tr>
<tr>
<td>4 P2000*</td>
<td>20 s</td>
<td>4.8 µm</td>
<td>0.24 µm/s</td>
<td>6.5 µm</td>
</tr>
<tr>
<td>5 P4000*</td>
<td>30 s</td>
<td>2.1 µm</td>
<td>0.07 µm/s</td>
<td>2.5 µm</td>
</tr>
<tr>
<td>6 3-µm diamond**</td>
<td>6 min</td>
<td>2.1 µm</td>
<td>350 nm/min</td>
<td>3 µm</td>
</tr>
<tr>
<td>7 1-µm diamond***</td>
<td>6 min</td>
<td>0.88 µm</td>
<td>147 nm/min</td>
<td>1 µm</td>
</tr>
<tr>
<td>8 0.25-µm diamond****</td>
<td>6 min</td>
<td>0.44 µm</td>
<td>74 nm/min</td>
<td>250 nm</td>
</tr>
<tr>
<td>9 Colloidal silica*****</td>
<td>6 h</td>
<td>0.4 µm</td>
<td>67 nm/h</td>
<td>40 nm</td>
</tr>
</tbody>
</table>

*) New grinding discs were used for each repetition of grinding
**) 275 rpm, Diamond added in 1-min intervals
***) 275 rpm, Diamond added in 2-min intervals
****) Old bath increased removal rate to 220 nm/h

The material removal rates are used for creating a sample preparation plan based on two factors: 1) the thickness of the material to be removed before the target cross-section is reached, 2) how much material needs to be removed to eliminate the deformation layer of the previous step. The latter is of the utmost importance, as any residual deformation from the sample preparation will obscure the deformation fields under the hardness indentations. If a grinding step is carried out only until the scratches of the previous step have been removed, the deformation field below the scratches will not be removed. As the particle size decreases in the sample preparation procedure, the preparation time should be increased proportionally. As a rule of thumb, the time allowed to remove visible scratches should be multiplied by at least a factor of 5 to remove the deformation field completely. Only light downward pressure should be applied to the specimen, applied from multiple points with two hands to avoid creating tilted surfaces. Furthermore, the use of coarse grits, especially P320 and P800, should be avoided, as they create thick deformation layers and remove material at a high rate.

Table A-3 shows the minimum material removal required to eliminate the deformation layer from the previous step. The estimates are conservative, particularly for the diamond polishing. In addition, the amount of material required to finalise the remaining steps at the start of each step is given. In practice the procedure is iterative by nature, as frequent measurement of material removal is required in order to avoid overshooting the target cross-section. This is particularly the case when finding the first indentations. The safe sample preparation method for a particular location can be defined on the basis of the distance to the target plane, and the smallest controllable material removal increment. Particularly for P800 and P1200 grinding, the removal increments should be kept to a maximum of 5 seconds and 10 seconds, respectively. The time required for removing the same amount of material increases exponentially as the distance to the target cross-section decreases.
Table A-3. Minimum sample preparation times for the complete removal of the deformation layer induced by the previous step. In addition, the material required for finishing sample preparation is given. Safe removal increments and distances to the target cross-section are given for all steps.

<table>
<thead>
<tr>
<th>Preparation step</th>
<th>Minimum time (µm)</th>
<th>Total removal (µm)</th>
<th>Material required (µm)</th>
<th>Removal increment (µm)</th>
<th>Distance to target range (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>After nickel plating</td>
<td>127 µm</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Only for nickel</td>
</tr>
<tr>
<td>1 P800</td>
<td>45 s</td>
<td>28.8</td>
<td>49.6</td>
<td>6-12 (10-20 s)</td>
<td>50-75</td>
</tr>
<tr>
<td>2 P1200</td>
<td>60 s</td>
<td>14.4</td>
<td>20.8</td>
<td>2-14 (10-60 s)</td>
<td>10-55</td>
</tr>
<tr>
<td>3 P2000</td>
<td>60 s</td>
<td>14.4</td>
<td>20.8</td>
<td>2-14 (10-60 s)</td>
<td>10-55</td>
</tr>
<tr>
<td>4 3-µm diamond</td>
<td>10 min</td>
<td>3.5</td>
<td>6.4</td>
<td>0.35-3.5 (1-10 min)</td>
<td>3.5-10</td>
</tr>
<tr>
<td>5 1-µm diamond</td>
<td>10 min</td>
<td>1.5</td>
<td>2.9</td>
<td>0.15-1.5 (1-10 min)</td>
<td>1.5-4.5</td>
</tr>
<tr>
<td>6 0.25-µm diamond</td>
<td>12 min</td>
<td>0.9</td>
<td>1.4</td>
<td>0.07-0.74 (1-10 min)</td>
<td>0.5-2.5</td>
</tr>
<tr>
<td>7 Colloidal silica</td>
<td>10 - 24 h*</td>
<td>0.5</td>
<td>0.5+</td>
<td>0.1-0.5 (2h-10h+)</td>
<td>0.1-1.0</td>
</tr>
</tbody>
</table>

* ) Quality of diffraction patterns, indexing rate and level of deformation checked with EBSD

An example of the workflow for reaching the target cross-section is shown next. The initial step in Figure A-3 needs to be cautious, as material removal with P800 and P1200 grit abrasives is quick. Short successive grinding steps with P800 are used only to remove the nickel coating, and after that only 10s with P1200 to remove the scratches. This does not remove the P800 deformation layer, but it will be removed in the next steps as the target cross-section is still far away. P2000 is the preferred grit for material removal as it has reasonable material removal capability, but only requires ca. 6.5 µm to be removed afterwards to produce a deformation-free sample. After P2000 grinding, 3-µm and 1-µm diamond polishing always has to be carried out for one minute each to remove severe scratches, and thus improve the edge visibility in the micrographs. If indentations are not found using optical microscopy (long working distance objectives preferred), the indicated P2000/3-µm/1-µm sample preparation procedure is repeated at 5-µm intervals until one of the markers is found.

Figure A-3. First step of serial sectioning to remove the nickel layer and find a marker indentation.
After an indentation has been found (Figure A-4), the distance to the target cross-section can be measured. An example of the optical micrographs used for location measurement is shown in Figure A-5. The cross-sections were typically inclined less than 0.5° relative to the centreline of the indentation, and, considering the manual sample preparation, are very close to parallel. To gain a better understanding of the material removal rates, less than half of the remaining material is removed in the next step. This gives important feedback for adjusting the sample preparation times in the next step.

Figure A-4. Second step of serial sectioning for approaching the target plane.

Figure A-5. Example optical micrographs (500x magnification) used for determination of the location of the cross-section. The flat part at the bottom of the indentations is compared to the distance between indentation diagonals. The base metal sample has been polished with colloidal silica.

As the target cross-section is approached (Figure A-6), the optical inspection intervals need to be more frequent. The removal of material is still effortless, and only two minutes of diamond polishing is required for optical observation. Conservative 5 – 10 µm steps are preferable, and a significant amount of reserve should be added to the 6.4 µm needed for diamond polishing and colloidal silica.
Figure A-6. Third step of serial sectioning for a slow approach to the diamond polishing region.

In the diamond polishing stage material removal becomes significantly slower. The ranges for different polishing steps are shown in Figure A-7. Briefly using 0.25 µm diamond (3-6 min) and silica (1-2 h) improves the cross-section location measurement accuracy; see Figure A-8. The last step with 0.25 µm diamond and colloidal silica is iterative, with the sample quality being verified in EBSD after the colloidal silica. The residual silica particles and other contaminants were cleaned from the samples using a high-velocity dry ice spray (a trigger-controlled nozzle connected to a compressed CO₂ container).

Figure A-7. Final step of serial sectioning with diamond and colloidal silica polishing.

Figure A-8. Visibility of indentation edges from optical micrographs (long working distance objective) after A) 1-µm diamond, B) 0.25-µm diamond (6 min), and C) 0.25-µm diamond (6 min) + silica (2 h).

Figure A-9 shows forescatter detector images for the same indentation in different cross-sections that have different amounts of sample preparation deformation. Sub-figure (A) shows moderate deformation from the 1-µm diamond polishing not removed by the 0.25-µm diamond polishing (6 min) and colloidal silica (19h). In (B) the next section of the material is polished with...
0.25-µm diamond (12 minutes), followed by 12h+ of silica, producing a low-deformation sample. The section in (C) shows surface relief and is deformation free after 12 minutes of 0.25-µm diamond polishing followed by 1 µm of material removal using colloidal silica. This amount of relief was not detrimental to the EBSD analysis. The size of the grain refined zone can be determined for all cases, as the grain sub-structures are clearly defined (D-F), despite the deformation in the moderately deformed sample (D). However, this deformation masks the extent of the plastic deformation zone (G). The dislocation structures are clearly distinguishable for (H), despite some deformation in the surrounding material. In the deformation-free sample (I), the extent of the deformation could also be characterised by setting a threshold value for KAM. However, in order to make the comparative analysis of the indentations robust, this approach is not utilised.

**Figure A-9.** FSD images showing the level of deformation from the sample preparation: A) moderate deformation, B) low deformation, C) deformation free with surface relief. Corresponding kernel misorientation analyses are shown in: D-F) KMM, 2° misorientation threshold, including the locations of the FSD, and G-I) KAM, 0.5° misorientation threshold.
Appendix B. Grain size measurement details

Grain size measurement

On the basis of the role of grain boundaries as an effective barrier to the movement of dislocations, the grain size dependence of yield strength can be explained by the pile-up of dislocations at grain boundaries [21]. The pile-up causes an additional stress, which allows the deformation to be transmitted to the next grain. The additional stress is in relation to the number of dislocations in a pile-up, which is limited by the length of the slip band that can be identified with the average grain diameter [21]. Other theories have also been proposed for the grain size dependence, such as the dislocation density model [192–194] and the geometrically necessary dislocation (GND) model [195–197]. A review of the models is given by Zhu et al. [198] and Evers et al. [199]. Each model implies a different Hall-Petch slope \( k \), but the mechanical properties are always scaled with the average grain diameter [198]. The average grain diameter, or in most cases the average grain size, is obtained from the experimental measurements [66,71], defined as:

\[
d = \frac{1}{n} \sum_{i=1}^{n} n_i d_i , \tag{B-1}
\]

where \( n \) is the total number of measurements and \( n_i \) the number of measurements corresponding to the grain size \( d_i \).

For heterogeneous microstructures it can be argued that the average grain size does not represent the physical response of the material adequately as a result of the broad grain size dispersion; see e.g. [64,65]. In a microstructure, the largest grains can be associated with low strength because of the length of the slip bands, causing them to yield first; see e.g. [144,145]. Furthermore, even a small number of large grains can occupy a significant material volume. In this work a rule-of-mixtures approach is proposed for heterogeneous microstructures to capture the influence of grain volume [69]. The contribution of each grain to the strength of the material is considered to be proportional to the volume of the grain; see e.g. [60,69]. Thus, the volume-weighted average grain size is defined as:
\[
\bar{d}_v = \frac{1}{V_T} \sum_{i=1}^{n} V_i d_i,
\]

where \(V_T\) is the total volume of material and \(V_i\) the volume of grains corresponding to the grain size \(d_i\). Because of the different definitions, the volume-weighted average grain size is always larger than the average grain size. The two parameters are equal only when all the grains are the same size.

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

The volume-weighted average grain size is obtained by weighting each measurement with the corresponding grain volume, as defined in Eq. (3). This presents the problem of defining the grain volume for each measurement. Although general formulations have been derived for the correlation between intercept length and grain volume [146], it is difficult to determine the accuracy and reliability of such an approach. This would also present the challenge that each linear intercept length measurement would have to be associated to a specific grain; however, the choice of the linear intercept length method was based on not having to identify grains before the measurement of the grain size.

The alternative approach is to modify the measurement procedure in such a way that the measured distribution of grain sizes is weighted by grain volume. The point-sampled linear intercept length method defined by Gundersen and Jensen [146,147] utilises stereological relationships for measuring the volume-weighted distribution of particle size. As described by Kurdykalowski and Ralph [148], a set of randomly positioned points are placed on the image and an intercept length is measured through each point that strikes a location of interest, in this case the interior of the grain. The direction of the intercept is chosen randomly, in this case from the four measurement directions used for the ASTM linear intercept method (0°, 45°, 90°, 135°) for comparability of the results; see Figure 3-2 for a graphical illustration of the measurement procedure.

As the measurement is carried out, the probability of a random point striking a grain of size \(i\) is proportional to the surface area fraction of the grain:

\[
P_i = \frac{A_i}{A_T},
\]

where \(A_i\) is the surface area of a grain of size \(i\), and \(A_T\) the total surface area. Based on relationships of stereology [200,201], the surface area fraction provides a statistical estimator for the volume fraction:
\[ V_v = \frac{V_i}{V_T} \approx \frac{A_i}{A_T}. \]  

Equation (A-4)

Equality exists between Eq. (4) and Eq. (5), and thus the probability of a grain being measured is approximately proportional to its volume fraction if the assumption of isotropy is made. When \( n \) measurements are taken with the point-sampled method, a distribution is generated in which the number of occurrences of a grain of size \( i \) is determined by the probability of being measured:

\[ n_i = nP_i = n \frac{V_i}{V_T}. \]  

Thus, the measured grain size distribution using the point-sampled method can be considered as the volume-weighted grain size distribution. The arithmetic mean grain size for the point-sampled distribution is:

\[ \bar{d}_{ps} = \frac{1}{n} \sum_{i=1}^{n} n_i d_i. \]  

Equation (A-6)

By substituting Eq. (5) into Eq. (6), the arithmetic mean grain size for the point-sampled distribution is:

\[ \bar{d}_{ps} = \frac{1}{n} \sum_{i=1}^{n} V_i \frac{V_T}{V_i} d_i = \frac{1}{V_T} \sum_{i=1}^{n} V_i d_i, \]  

Equation (A-7)

which is equal to the volume-weighted average grain size, \( d_v \), as defined in Eq. (2). Thus the average value of the distribution can be considered as the volume-weighted average grain size, \( d_v \).

**Visualisation of grain size measurement**

The procedure for the visualisation of the grain size measurement is shown in Figure 8-1 using a fictional single-phase microstructure. First, the grain size is measured for individual grains at random points that strike the interior of the grain (I a). The procedure is repeated a large number of times for one measurement direction, resulting in a densely measured grain size for the individual grain (I b).

To speed up the measurement only a fraction, e.g. 25\%, of all the points can be measured. For an improved visual representation, the non-measured points are filled with a value from the nearest neighbouring point. As shown in (I c), the interpolation results in a good visual representation of the grain size for the individual grain. The grain size is presented using the Hall-Petch grain size parameter \( (d^{-0.5}) \) in order to have a linear scale for the mechanical properties affected by the grain size. This approach is taken in order to improve the resolution of the visual representation in the grain size regime below 10 \( \mu m \). On the basis of the Hall-Petch relationship, a small change in grain size in this regime has a significant effect on the mechanical properties, e.g. the strength doubles as the grain size decreases from 4 \( \mu m \) to 1 \( \mu m \); see Figure 8-2. It is noted that the
The classical Hall-Petch relationship is applicable at grain sizes larger than 0.1 µm ($d^{-0.5} < 3.16 \mu m^{-0.5}$) [202,203].

**Figure B-1.** Flowchart of the grain size measurement and analysis procedure. [134]
The procedures (I a...c) are repeated for all the grains in the fictitious microstructure using four measurement directions (0°, 45°, 90°, 135°) as shown in (II a...d) of Figure 8-1. Typically at least 25% of the image points should be measured in each direction for accurate results.

To verify that the nearest neighbour interpolation does not introduce any bias or error to the data, the grain size distributions of the measured and interpolated data are compared in Figure B-3a. As the two counterparts overlap for each individual measurement direction, the interpolated data shown in (II a...d) can be used for further data analysis.

In order to compare the grain size with the mechanical properties, e.g. hardness, the four measurement directions need to be combined into a single visualisation. The three alternatives used are to take the minimum, mean, or maximum value of the four measurement directions at each point of all the grains; see (III a...c) in Figure B-1. As shown in Figure B-3b, the grain size distributions of minimum and maximum cases are the lower and upper bounds for the measurement data, respectively. The mean of the interpolated data is nearly identical to the measured volume-weighted average grain size ($d_v$), even though
the shapes of the distributions are different. The agreement of volume-weighted average grain size has been verified for various heterogeneous microstructures, with the error typically being smaller than 1%. Since the volume-weighted average grain size has been shown to correlate with hardness according to the Hall-Petch relationship [135], the mean grain size plot is used for further result analysis of hardness and grain size.

In addition to the above-mentioned visual options, the moving averages of the minimum, mean, and maximum grain size contours (Figure B-1, III a-c) are calculated across the micrograph using horizontal and vertical line probes. The line probes used in this study are ten pixels wide. In addition, the border regions of the micrographs were excluded from the averaging to eliminate large grains that extend beyond the micrograph. Grain size at a 90% probability level was found to be a suitable margin for exclusion at the edges of the image. The difference between the three moving averages represents the local variation in the grain size in different measurement directions.
Appendix C. Clustering based reference orientation

In order to quantify the amount of plastic deformation accurately from the orientation data, a reference orientation for the grain-based misorientation analysis needs to be defined. In the case of local plasticity, the level of deformation inside a single grain can be severe, and it limits the applicability of existing methods to finding the reference orientation. The commonly used methods are the average orientation of the grain, the point with the lowest local misorientation, and the point with the highest diffraction pattern quality. The average orientation of a grain is applicable if the level of deformation is low or a large area of the grain is undeformed. The point with the lowest local misorientation can also be assumed to have the lowest plastic deformation. However, the orientation of the single point can differ significantly from other points with a low local misorientation, especially for highly deformed small grains. Diffraction pattern quality is also sensitive to several factors, making it an unreliable metric for deformation [121].

Instead of a single point being used for reference orientation, the local misorientation methodology is enhanced with orientation clustering. The methodology is based on the assumption that plastic deformation creates local lattice rotation as new sub-grains are formed [75,77]. As a result of this, some segments of the grain can remain undeformed, or have a low level of deformation. In these areas the local misorientation is low, leading to a high orientation density in the Euler orientation space. On the basis of the orientation density, the data is grouped into clusters, allowing the detection of the least deformed orientation cluster.

The clustering process used is a thresholding procedure for estimating the least deformed area in the grain. Using local misorientation values, the highly deformed orientations can be excluded from the clustering. The thresholded group of orientations are clustered using a density-based algorithm, DBSCAN [204], implemented in Matlab by S. Mostapha Kalami Heris and available online at [129]. DBSCAN is suitable for discovering arbitrarily-shaped clusters from large spatial databases with noise. It is founded on a density-based notion, grouping data points that are closely packed together in a specific space. The algorithm requires two input parameters: 1) Epsilon, defining a neighbourhood
around a point in space, 2) MinPts, defining how many points are required in the Epsilon neighbourhood of the data point.

The clustering principle is visualized in Figure C-1A. Clusters are detected on the basis of the proximity of data points. If a data point has at least the number of neighbours defined by MinPts in the Epsilon neighbourhood, it is defined as a core point of a cluster. All the core points have at least three points in their vicinity, thus fulfilling the clustering criteria. Border points do not meet the criteria, but are reachable by the Epsilon neighbourhood of a core point, and are thus included in the cluster. All the points without a sufficient number of data points in their neighbourhood, and not reachable by the core points of a cluster, are considered as noise.

With predefined clustering parameters, the size of the cluster is dependent on the orientation density. In highly deformed areas the orientation density is low, as illustrated in Figure C-1A. In an undeformed region the orientations of the data points are closer to each other, forming high-density clusters; see Figure C-1B. Thus, the cluster with the highest density is usually the largest cluster. This is selected as the reference cluster, and the reference orientation is defined as the median orientation of the cluster. The two examples highlight the need for appropriate parameter selection: to detect only the high-density clusters, the Epsilon neighbourhood has to be small enough. Likewise, the number of points required in the neighbourhood has to be selected correctly. Clustering and parameter selection will be demonstrated with an example case below.

![Figure C-1. A two-dimensional schematic of the DBSCAN clustering principle. Using the same clustering criteria, clusters with high density, i.e. low deformation, form larger clusters.](image-url)

To demonstrate the clustering process, a reference orientation is determined for a large grain that has moderate deformation. The orientation map of the grain is shown in Figure C-2A. Figure C-2B shows the orientation relative to the average orientation for an enhanced view of the deformation level in different regions of the grain. Deformation has been caused by an indentation directly above the highest point of the grain; see Figure 5-20.
Figure C-2. A) Orientation map (IPF-Z) of the grain that was studied, and B) orientation map showing lattice rotation relative to the average orientation within 7.5° of misorientation. Areas with different levels of deformation are shown by dashed ellipses.

First, the local misorientation is used to measure the level of deformation, as shown in Figure C-3A. The aim is to eliminate the high and moderate deformation areas shown in Figure C-2B, leaving the low deformation areas for clustering. The size of the kernel used contains fifth nearest neighbours, creating an 11x11 kernel around the central point. The kernel size is increased from the conventional first nearest neighbours to reduce the influence of measurement noise. The 50% of the orientations with the lowest local misorientation are considered as candidates for the reference orientation, shown in red in Figure C-3C. It is evident that the right-hand side of the grain has the lowest amount of deformation. Furthermore, the points with the highest diffraction pattern quality (IQ) and lowest local misorientation (KAM) are located in this region.

In general, low misorientation values are located in the same region of the orientation space, shown in Figure C-3B. The majority of the lowest 50% are very similar in orientation, located in the dense area shown by the green circle in Figure C-3D. This indicates that orientation clustering can be carried out using orientation density metrics.
Figure C-3. A) Grain of interest coloured with kernel average misorientation (fifth nearest neighbours). B) Euler orientation angles (Bunge convention) coloured with a restricted KAM colouring. C) Lowest 50% KAM values inside the grain coloured red, and D) the same orientations coloured red in the Euler orientation space.

To use the DBSCAN algorithm, the input parameters Epsilon and MinPts need to be defined. As deformed areas have larger orientation gradients, the misorientation to a nearest neighbour is also larger. Suitable clustering parameters are dependent on e.g. grain size, the extent of the deformation, and the orientation gradients, and thus an approach for defining the clustering parameters for each grain is required.

To define the Epsilon value, the misorientation is calculated from a point to every other data point, disregarding spatial information. The misorientation to the fifth nearest neighbour inside the grains is used to characterise the intra-grain deformation, as shown in Figure C-4A. A subset of the right-hand side of the grain has the lowest level of deformation using this metric. A threshold value needs to be determined for clustering to separate deformed and non-deformed orientations. For this purpose, the number of orientations with a specific misorientation to the nearest neighbour can be used to determine a knee-point [204]; see Figure C-4B. This threshold is typically 0.03°-0.2°, being 1-2 orders of magnitude smaller than the grain boundary tolerance angle. The threshold is dependent on the size of the grain and extent of the deformation. A least squares bi-linear fit is used for the knee-point detection [130]. The knee-point is defined as the data point that minimises the root mean square error of the two linear fits meeting at that specific point.
The second clustering parameter, MinPts, is also dependent on the grain deformation characteristics. The general approach, as defined by the authors of the method [204], is to use the same value as was used for the definition of Epsilon, in our case five nearest neighbours. As the clustering is performed with the grain-specific parameters, the orientation data is divided into several clusters. Spatial information is disregarded in clustering, as the lattice rotation is continuous, resulting in spatially continuous clusters without information about location. Clustering results for the example grain are shown in Figure C-5, with five nearest neighbours (MinPts=5) required. A total of 20 clusters are formed, with many small clusters and a significantly large one covering most of the data to be clustered. The largest cluster is also located in the centre of the densest part of the orientation space, shown in Figure C-5B&C. The reference orientation is defined as the median orientation of the largest cluster, as the median is less sensitive to possible outliers compared to the arithmetic mean. In most cases the largest cluster is the best guess for a reference cluster, located in the part of the orientation space with the highest density.

The above-mentioned parameters are suitable for most cases. However, if the level of deformation is high or extends to the majority of the grain, the parameters need to be adjusted. For a stricter clustering criterion, the knee-point approach can also be utilised for the determination of MinPts. This is done by evaluating
how many neighbours each point has within the Epsilon neighbourhood [online ref]. Figure C-6A shows how many neighbours each data point has within the 0.089° misorientation neighbourhood (Epsilon), and Figure C-6B the determined knee-point for MinPts.

![Figure C-6](image)

**Figure C-6.** A) Number of nearest neighbours within a misorientation threshold of 0.089°.  
B) Number of orientations with a specific number of nearest neighbours within the misorientation threshold, and the knee-point that was determined. Data points (21805) are condensed into bins for robustness.

With the use of a higher number of required nearest neighbours, fewer clusters are formed and they are smaller. With this approach only three clusters are detected, with the right-hand cluster being split into three smaller section; see Figure C-7. On the basis of supplementary information from other analyses (local misorientation, FSD images), the cyan coloured cluster #1 is determined as a candidate for the reference orientation; see Figure 4-7 for supplementary images.

The stricter clustering criteria are sensitive to the filtering of orientation data; from the raw data the orientation noise can mask the subtle cluster boundaries and with over-filtering the boundaries are too smooth to be detected. Half-quadratic filtering ($\alpha=0.15$) was found to be a good compromise between noise reduction and retaining orientation gradients for reference orientation clustering.

![Figure C-7](image)

**Figure C-7.** DBSCAN clustering results using the parameters Epsilon=0.089° and MinPts=358.  
A) Cluster map, B) Euler orientation angle space with clusters coloured, C) enlarged view of the high orientation density region.
Validation is carried out by comparing the reference orientation determined with conventional methods and clustering. Reference orientations are presented in Table C-1 together with the average orientation, point with the highest diffraction pattern quality, and point with lowest local misorientation. The locations of highest pattern quality (IQ) and lowest local misorientation (KAM) are shown in Figure C-3C. All the reference orientations are within 0.7° of misorientation to strict clustering (#5), which is expected as a result of a large portion of the grain having low deformation. The orientation determined by strict clustering is nearly identical to the point with the lowest local misorientation, as that point is located inside the reference cluster. Despite only minor differences in the determined reference orientation, it can have a significant influence on the analysis of deformation mechanism.

**Table C-1.** Comparison of reference orientation Euler angles determined with clustering method and conventional methods. Data for the low-deformation section of the grain.

<table>
<thead>
<tr>
<th>Reference orientation</th>
<th>phi1</th>
<th>Phi</th>
<th>phi2</th>
<th>Misorientation to #5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average</td>
<td>37.85°</td>
<td>85.30°</td>
<td>70.79°</td>
<td>0.72°</td>
</tr>
<tr>
<td>Highest IQ</td>
<td>37.64</td>
<td>85.72°</td>
<td>71.15°</td>
<td>0.63°</td>
</tr>
<tr>
<td>Lowest KAM (nn=5)</td>
<td>38.34°</td>
<td>85.70°</td>
<td>71.20°</td>
<td>0.07°</td>
</tr>
<tr>
<td>Clustering</td>
<td>37.79°</td>
<td>85.71°</td>
<td>70.87°</td>
<td>0.60°</td>
</tr>
<tr>
<td>Strict Clustering</td>
<td>38.28°</td>
<td>85.70°</td>
<td>71.18°</td>
<td></td>
</tr>
</tbody>
</table>

A highly deformed section of the same grain is shown in Figure C-8. The overall level of deformation is much higher than for the section shown in Figure C-2, and extends to a larger area of the grain. Despite there being a region of low local misorientation on the right-hand side of the grain (Figure C-8C), the point of the lowest local misorientation is at the top of the grain in the highly deformed region.

**Figure C-8.** A) Orientation map (IPF-Z) of the grain that was studied, and B) orientation map showing lattice rotation relative to the reference orientation within 7.5° of misorientation. Areas with different levels of deformation are shown by dashed ellipses. C) Local misorientation map showing deformation (nearest neighbours = 5).
The reference orientations for the grain section shown in Figure C-8 are presented in Table C-2. The lowest local misorientation (#3) is highly misoriented from all the other references as a result of its location at the top of the grain. Because of the higher level of deformation, the average orientation starts to deviate significantly from the strict clustering (#5) reference. The effect of this deviation on the misorientation analysis is shown in Figure C-9, which compares average (#1) and strict clustering (#5) as the reference orientation. With average orientation the misorientation at the top of the grain is significantly lower than when clustering is used. Furthermore, moderate levels of misorientation are shown on the right-hand side of the grain, contradicting the local misorientation results in Figure C-8. In general the clustered result shows a logical contour of decreasing deformation towards the bottom of the grain, with the low deformation on the right-hand side agreeing with the local misorientation analysis.

Table C-2. Comparison of reference orientation Euler angles determined with clustering method and conventional methods. Data for the high deformation section of the grain.

<table>
<thead>
<tr>
<th>Reference orientation</th>
<th>phi1</th>
<th>Phi</th>
<th>phi2</th>
<th>Misorientation to #5</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Average</td>
<td>38.47°</td>
<td>82.12°</td>
<td>68.31°</td>
<td>2.82°</td>
</tr>
<tr>
<td>2 Highest IQ</td>
<td>37.71</td>
<td>84.23°</td>
<td>70.61°</td>
<td>1.02°</td>
</tr>
<tr>
<td>3 Lowest KAM (nn=5)</td>
<td>52.51°</td>
<td>66.93°</td>
<td>55.48°</td>
<td>24.94°</td>
</tr>
<tr>
<td>4 Clustering</td>
<td>38.06°</td>
<td>83.90°</td>
<td>69.63°</td>
<td>0.74°</td>
</tr>
<tr>
<td>5 Strict Clustering</td>
<td>37.64°</td>
<td>84.51°</td>
<td>69.64°</td>
<td></td>
</tr>
</tbody>
</table>

Figure C-9. Misorientation angle determined using A) the average orientation, and B) strict clustering as the reference orientation.
Appendix D. EBSD data post-processing using MTEX

**EBSD data post-processing for misorientation analysis**

Figure D-1 shows kernel misorientation calculated with first, second, and third nearest neighbours, using raw measurement data and two filtering options. With the raw data some DDWs are apparent beneath the indenter in the highly deformed zone. After one pass of the half-quadratic filter ($\alpha=0.15$), the noise floor is reduced significantly and the slightly blurred outlines of the DDWs are seen throughout the deformed area. The effectiveness of the noise reduction is shown in comparison to the raw data in Figure D-2, with a relatively uniform reduction throughout and no influence on the DDWs. After the application of a second pass of the half-quadratic filter ($\alpha=0.15$), followed by a first nearest neighbour Kuwahara filter to enhance the orientation gradients, a network of DDWs is revealed. The edge enhancing properties of the Kuwahara filter are shown in Figure D-3, with the DDWs being more prominent compared to first pass the half-quadratic filtering. This reveals that the deformation has extended beyond the grain boundary into the next grain.

To ensure that the post-processing of the orientation data does not introduce artificial features into the deformation analysis, the raw data is compared to several filtering options. Kernel median misorientation with 60 nearest neighbours is shown in Figure D-4, revealing the sub-grain boundaries using a misorientation threshold of $2^\circ$. The location and size of the sub-grains remains unchanged between the raw data and the used second-pass half-quadratic filtering followed by the Kuwahara filter. The same holds true for the kernel average misorientation in Figure D-5, which shows the dislocation cell structure using a misorientation threshold of $0.5^\circ$. While the measurement noise in the raw data masks many of the features, the distinct outlines of the dense dislocation walls (DDWs) are apparent. With the use of the second-pass half-quadratic and Kuwahara filter combination the DDWs become more distinct and the degree of misorientation within the dislocation cells remains similar; see the enlarged region for comparison.
Figure D-1. Kernel average misorientation for raw and post-processed data. Results obtained using three different kernel sizes (nearest neighbours 1-3) are shown.

Figure D-2. Difference in kernel average misorientation between first pass half-quadratic filtering and raw data from Figure D-1.

Figure D-3. Difference in kernel average misorientation between second-pass half-quadratic filtering combined with a Kuwahara filter, and first-pass half-quadratic filtering from Figure D-1.
Figure D-4. Comparison of filtering options using kernel size of 60 nearest neighbours, and an upper misorientation threshold of 2° (KMM).
Figure D-5. Comparison of filtering options using kernel size of 60 nearest neighbours, and an upper misorientation threshold of 0.5° (KAM).
Combination of grain-based and kernel misorientation results

To analyse plastic deformation and grain interaction, several misorientation analysis results need to be assessed in conjunction. For example, the misorientation axis directions, dislocation substructures, and amount of deformation provide further insight into the interaction of the grains. The basis for this analysis is the misorientation axis colour map, shown in Figure D-7A for two base metal indentations. The two have contrasting grain sizes, with the misorientation axis patterns having distinctly different characteristics. The extension of the zones rotating in a particular direction is clearly defined, but the visualisation has the limitation of not containing any information regarding the magnitude of the rotation or deformation.

To add a deformation quantity to the colour maps, the colour space is converted from RGB (red, green, blue) to HSL (hue, saturation, lightness). The colour spaces are shown in Figure D-6. Hue (0–360°) represents the shade of colour, and saturation (0–1) the intensity of colours with pure colours having a value of 1. The pure colours have a lightness value of 0.5. By adjusting the lightness value, the colour is changed from fully saturated (0.5) to black (0) or white (1).

![Figure D-6](image)

The kernel misorientation analysis developed in Chapter 4 captured the extent of the plastic deformation effectively. Therefore it is used for adjusting the lightness value of the misorientation axis map with a smooth transfer function, such that visual output reflects the amount of deformation. The lightness value of the original colour map (A) is multiplied by the transfer function value, resulting in a black colour map for undeformed regions below a defined threshold. For example, in Figure D-7B, a smooth transfer function between 1° and 1.7° of KMM creates a colour map reflecting the grain refined zone, showing the sub-grain boundaries. This shows that the sub-grain structure is significantly more refined for the indentation on the left, as its grain size and thus free path for dislocation slip are much smaller.

![Figure D-6](image)

By shifting the transfer function to 0.2–1°, the plastic deformation zone is captured effectively for the base metal, shown in Figure D-7C. Compared to the original misorientation axis map in (A), the identification of the deformed grains and their deformation patterns becomes very clear. Regions of residual deformation, and second-phase areas in particular, are visible further away from the indentations. When the plastic deformation zone adjustment is combined with KAM (<0.5deg) adjustment in the range 0.21–0.42°, the dense dislocation
walls are also incorporated into the colour map in Figure D-7D. This output maintains the deformation patterns of (C) quite well, incorporating further information about the local deformation state with the dislocation cells structure. In particular, the size of the dislocation cells is in relation to the applied stress, reflecting the severity of the deformation in a particular grain, and even the deformation state locally inside a single grain. The highly deformed grain under the indenter shows extremely small dislocation cells for the indentation on the left, while the dislocation cell size is significantly larger in the large grains for the indentation on the right. Transfer functions need to be adjusted depending on material microstructure, kernel size, and the level of deformation, and thus the values for the weld metal will need some adjustment because of the smaller kernel size and residual plastic strains.

Figure D-7. Misorientation axes in specimen coordinate system (A) and different outputs achieved with colour lightness adjustment. B) Grain refined zone (KMM 0-2°), C) plastic deformation zone (KMM 0-2°), D) output of (C) with an additional adjustment by KAM (0-0.5°) to convey DDWs and dislocation cells with the plastic deformation zone.
Dislocation motion analysis for screw dislocation

This analysis is an extension of Chapter 5.4. The misorientation axis directions are shown for a trace around the central point in Figure D-8A&B. The misorientation axis varies continuously around the central point, with local fluctuations in the gradient of the Miller indices. The estimated directions of dislocation motion for the slip planes of the [1 1 -1] zone axis are overlaid on the misorientation axes in Figure D-8C. There seems to be slip activity in all the (110) and (211) slip planes of the [1 1 -1] zone axis, with the traces following the predicted directions quite well. Two adjacent traces can be identified for (110) slip systems between (211) slip planes, which may be caused by the rotation of the slip plane away from the compression axis. There is three-fold symmetry with several traces having a wavy profile moving outwards from the central location. The individual steps correspond to the crystallographic directions; for example, the (2 -1 1) trace in D is composed of segments also moving in a direction consistent with the (1 -1 0) and (1 0 1) slip planes. Likewise, for the (0 -1 -1) trace (red) in E, the segments are consistent with the (-1 -1 -2) and (1 -2 -1) slip planes. This indicates cross-slip in two neighbouring {2 1 1} or {1 1 0} slip planes, resulting in macroscopic slip direction consistent with a {1 1 0} and {2 1 1} slip plane, correspondingly.

![Figure D-8](image)

**Figure D-8.** A, B) Measured misorientation axis directions for a trace around the central point, C) Comparison of misorientation axis traces with estimated directions of dislocation motion for slip planes of the [1 1 -1] zone axis. D, E) Segments forming the (2 -1 1) and (0 -1 -1) traces.
The directions of the misorientation axes are shown for circular traces around the central point in Figure D-9. It is apparent that the misorientation axes are radially aligned around the central point. For a screw dislocation, as shown in Figure 1-11, a misorientation axis in a particular slip plane would be perpendicular to the direction of dislocation motion. For example, if the dislocation motion was horizontal, the misorientation axis would be vertical, which is consistent with the experimental observation. The alignment of the misorientation axis relative to the slip direction and X-axis is shown in Figure D-10. At the centre the misorientation axis is inclined approximately 47° from the slip direction, while it is approximately perpendicular elsewhere. This indicates that the rotation is caused by the motion of dislocations along the slip planes.

**Figure D-9.** A) Circular traces centred at the intersection of colours on the misorientation axis map, coloured by the misorientation angle. B) Visualisation of misorientation axes around the central point.

**Figure D-10.** Angle of the misorientation axis relative to A) $X_s$ direction (in-plane), B) slip direction [1 1 -1].
Appendix E. Sub-grain boundary reconstruction

Sub-grain boundary reconstruction is demonstrated with a base metal HV0.05 indentation shown previously in Figure 4-4. The reconstruction is shown in Figure E-1. The procedure used for the reconstruction is the following:

1. Kernel misorientation analysis is carried out to identify sub-grain boundaries at various misorientation threshold levels (0.5°, 1°, 2°);

2. On the basis of the network of sub-structures, seed points are hand-selected at the centre of sub-structures, e.g. sub-grains and dislocation cells;

3. Next, the sub-grains are displayed using a random colour by colouring the area around the central point that is within the particular misorientation threshold. This is also the effective kernel size at each of the selected points;

4. The boundaries of the sub-grains are traced for the boundaries that have different degrees of misorientation. Several boundaries resolved using the 0.5° and 1° thresholds overlap;

5. The reconstruction is the overlay of the three different boundary types: grey indicates dense dislocation walls, black dense dislocation walls with higher misorientation, and red the highly misoriented sub-grain boundaries.

The misorientation of adjacent dislocation cells is measured in Figure E-2 using the threshold 0.5°. EBSD mapping has been carried out at a finer 0.06 µm step size in order to resolve the fine dislocation cell structure. The misorientation of a dense dislocation wall is defined as the misorientation between the central points of the neighbouring dislocation cells. Misorientation varies in the range 0.5°...2.1°, with an average of 1°.
Figure E-1. Reconstruction of sub-grain structure using kernel misorientation analysis with varying misorientation thresholds.

Figure E-2. Enlargement of measured dislocation cell structure and the misorientation angles between the central points of neighbouring dislocation cells. (0.06 µm step size)
The size of the deformation structure is measured in Figure E-3, with the dislocation cells and dense dislocation walls (black) replicated from Figure 4.5. On the basis of the measurement in sub-figure A, the smallest dislocation cells are approximately 400 nm in comparison to the parent grain size of 5...25 µm. The grain shown in Figure E-2 has the finest dislocation cell size in the middle of the upper half of the grain. The grain size is averaged with a 5.5×5.5 µm kernel in sub-figure B, showing the grain refinement contours. In general, the dislocation cell size is smallest in the grains directly in contact with the indenter. The deformation structure size for the weld metal has similar characteristics, shown in Figure E-4. This indicates that the deformation process has similar characteristics for both materials.

![Figure E-3](image1.png)  
**Figure E-3.** Grain size measurement for the deformation structure under a base metal HV0.05 hardness indentation: A) measurement data, B) grain size averaging with a 5.5×5.5 µm kernel.

![Figure E-4](image2.png)  
**Figure E-4.** Grain size measurement for the deformation structure under a weld metal HV0.05 hardness indentation: A) measurement data, B) grain size averaging with 2.6×2.6 µm kernel.
Welding is the most commonly used manufacturing method of steel structures in various applications, for example cars, ships and bridges. In order to guarantee structural safety, the influence of the welding process on material properties has to be understood. In particular, the metallurgy of the steel can transform during the welding process, leading to the degradation of the mechanical properties. Such metallurgical factors have caused serious accidents and loss of life in the 20th century. This thesis studies the relationships between material microstructure and mechanical properties across several length scales for welded structural steel. Relationships are formulated for the prediction of material strength and extent of plastic deformation based on the microstructural characteristics of the steel.