Micromechanisms of Failure under Static Loading in Sheet Metals for Automotive Applications

Ph.D. Thesis

Orlando León García
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Micromechanisms of Failure under Static Loading in Sheet Metals for Automotive Applications

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This thesis is dedicated to Valdi, my beloved wife
I, Nezahualcoyotl, ask this:
Is it true one really lives on the earth?
Not forever on earth,
only a little while here.
Though it be jade it falls apart,
Though it be gold it wears away,
Though it be quetzal plumage it is torn asunder.
Not forever on earth,
Only a little while here.

Niquitoa ni Nezahualcoyotl:
¿Cuix oc nelli nemohua in tlalticpac?
An nochipa tlalticpac:
zan achica ya nican.
Tel ca chalchihuitl no xamani,
no teocuitlatl in tlapani,
no quetzalli poztequi.
An nochipa tlalticpac:
zan achica ye nican.

Yo, Nezahualcóyotl, lo pregunto:
¿Acaso de veras se vive con raíz en la tierra?
Nada es para siempre en la tierra:
Sólo un poco aquí.
Aunque sea de jade se quiebra,
Aunque sea de oro se rompe,
Aunque sea plumaje de quetzal se desgarra.
No para siempre en la tierra:
Sólo un poco aquí.

Nezahualcóyotl, texcocan poet
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<th>Description</th>
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<tr>
<td>IF steel</td>
<td>Interstitial free steel</td>
</tr>
<tr>
<td>DP steel</td>
<td>Dual phase steel</td>
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<tr>
<td>Ref</td>
<td>Reference DP steel material</td>
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<tr>
<td>SH</td>
<td>Slow Heated DP steel material</td>
</tr>
<tr>
<td>SC</td>
<td>Slow Cooled DP steel material</td>
</tr>
<tr>
<td>HT</td>
<td>High Temperature DP steel material</td>
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<tr>
<td>EBSD</td>
<td>Electron backscatter diffraction</td>
</tr>
<tr>
<td>FIB-EBSD</td>
<td>Focussed ion beam – electron backscatter diffraction</td>
</tr>
<tr>
<td>$\varphi_1, \phi, \varphi_2$</td>
<td>Euler angles (Bunge notation)</td>
</tr>
<tr>
<td>$\nabla$</td>
<td>Nabla operator</td>
</tr>
<tr>
<td>$\varepsilon_{ijk}$</td>
<td>Permutation or Levi-Civita tensor</td>
</tr>
<tr>
<td>$\varepsilon$</td>
<td>True or logarithmic strain</td>
</tr>
<tr>
<td>$e$</td>
<td>Engineering or tensile strain</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>True or logarithmic stress</td>
</tr>
<tr>
<td>$S$</td>
<td>Engineering or tensile stress</td>
</tr>
<tr>
<td>$UTS$</td>
<td>Ultimate tensile strength</td>
</tr>
<tr>
<td>$RA$</td>
<td>Reduction of Area at fracture</td>
</tr>
<tr>
<td>$S_0$</td>
<td>Offset yield strength, offset strain = 0.2%</td>
</tr>
<tr>
<td>$R$</td>
<td>R value or Lankford parameter</td>
</tr>
<tr>
<td>GNB</td>
<td>Geometrically Necessary Boundary</td>
</tr>
<tr>
<td>DDW</td>
<td>Dense Dislocation Walls</td>
</tr>
<tr>
<td>GND</td>
<td>Geometrically Necessary Dislocations</td>
</tr>
<tr>
<td>SOS</td>
<td>Scalar Orientation Spread</td>
</tr>
<tr>
<td>KAM</td>
<td>Kernel Average Misorientation</td>
</tr>
<tr>
<td>GROD</td>
<td>Grain Reference Orientation Deviation</td>
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Chapter 1

Introduction

1.1 Metals in the contemporary automotive industry

The two oil crises of the 70’s, the current concern about global warming and the constantly changing social demands, have served as a strong motivation for the continuous improvement of automotive vehicles in terms of fuel consumption efficiency, passenger safety and comfort requirements. The case of the fuel consumption efficiency has received enormous and increasing attention during the last four decades. Encouraged at the beginning by the limited fuel supplies, the rapid growth in developing countries and the supply disruptions caused by political situations, the fuel consumption reduction is nowadays driven by the urgency to reduce emissions due to its negative effect on the global climate. This issue has had such an impact that the governments and industries all around the world have started taking actions. For instance, the Japanese automobile industry aimed to improve the fuel efficiency by 22.8% in 2008 compared with the level of 1995 in order to reduce CO₂ emissions [1]. The main approaches to tackle this international challenge were focussed on \( i \) improvement of engine efficiency, \( ii \) enhancement of the aerodynamic design of automobiles and \( iii \) vehicle weight reduction [2]. The last topic is one of the points of interest of materials manufacturers, because a reduction of 10% in the total vehicle weight could produce 8 to 10 % of the fuel economy [3]. Nevertheless, the main challenge of weight reduction relies on preserving, or even improving, the integrity of the vehicle at the same time; i.e. complying with the safety requirements and customers’ demands. Many governments are demanding an increase in the already rigorous passenger safety requirements with the main attention focussed on increasing the energy absorption from crash events referred to as crashworthiness. For example, the National Highway Traffic and Safety Administration in the USA is looking to improve side impact intrusion performance as well as to increase the standard velocity for frontal crash tests [4]. Therefore, inasmuch as a simultaneous improvement of weight reduction and safety requirements is required, the strategy of material suppliers has been directed towards the enhancement of the
mechanical properties of steels and/or the implementation of lighter weight materials such as aluminium or magnesium alloys.

Despite the emergence of light weight materials, steel continues to be the preferred material for most body and chassis structural components and the main reason for this is the technological development made on this material. Major improvements of the mechanical properties of steel have been achieved during the last century and have been accompanied by a deep knowledge and understanding of its structural properties. As a consequence, new steel grades with improved strength and ductility have been developed and these advances have allowed the production of thinner gauge materials for the automotive industry. The continuous improvements of the mechanical properties of steels have been made possible due to the optimization of chemical composition and an appropriate microstructural design. The latter includes grain refinement and microstructure strengthening via solid solution, precipitation and, in the case of advanced high strength steels, second phase hardening and strengthening mechanisms occurring during plastic deformation such as the austenite-to-martensite transformation or twinning.

A typical manner to qualitatively compare the mechanical properties of engineering materials is via the well-known diagram representing the elongation at fracture versus tensile strength. The relevance of such kind of diagram relies on the representation and comparative assessment of the formability and strength for different materials where the simultaneous improvement of both mechanical properties is desired but difficult to achieve. Figure 1.1 displays the mechanical properties of current high strength steels grades with application in automobile parts.

Conventional high strength steels (HSS) represent one of the first steel groups with an important impact on the automobile industry due to their high formability levels. They are characterized by their single phase microstructure and the optimization of solid solution and precipitation hardening for increasing the strength without significantly deteriorating the formability. One representative example of the HSS group are the High Strength Interstitial Free (IF-HS) steels, which have found their application in cases where deep-drawability characteristics are needed, such as high plastic strain ratio values and in-plane isotropy [7].

The first generation of the Advanced High Strength steels (AHS) was initiated with the intensive development of Dual Phase (DP) steels in the 1970’s [8] and further complemented with the introduction of Complex Phase (CP), Martensitic (MS) and Transformation Induced Plasticity (TRIP) assisted steels. The AHS steel grades possess up to five times the strength of mild steels combined with sufficient elongation
Introduction

for formability, adequate weldability and improved energy absorption. The principal strengthening mechanisms implemented in these steels are based on second phase hardening and/or the transformation induced plasticity (TRIP) effect although the matrix is still predominantly ferrite.

Figure 1.1: Elongation at fracture versus tensile strength diagram for several types of steels classified as High Strength (HS) steels, Advanced High Strength (AHS) steels and High Manganese (HMS) steels. Diagram based on [5] and [6].

In recent years, a second generation of AHS steels has emerged which is characterized by a combination of unusual formability and high strength. Examples of this group of steels are Twining Induced Plasticity (TWIP) steels, Austenitic Stainless Steels (Aust. SS) and the Al-added Lightweight steels with Induced Plasticity (L-IP). The microstructure of these steels is mainly based on an austenitic matrix which is stabilized due to the addition of higher amounts of alloying elements, especially manganese and nickel. Due to the high content of alloying elements, these steels are also known as High Manganese Strength (HMS) steels. However, the main drawback that has limited their widespread production is the higher costs involved in the high amount of alloying elements as well as other particular issues associated with the complexity of their microstructure. One example is the case of delayed fracture in TWIP steels where cracks occur after the formability process is finished [9].
A third generation of AHSS is currently in development with the aim to fill the gap of strength-ductility combination between the first and second generations but without the level of expensive alloy additions required in the second generation of AHSS [10].

1.2 Failure prediction: A challenge on formability of automobile parts

The concern of material suppliers for the automobile industry is not only focused on the material properties of final products but also on the processability of the material during manufacturing of the automotive parts, which is often specified in terms of formability. Springback control, issues with tool material selection and failure prediction are currently the main challenges for automotive metal manufacturing engineers [11]. The margins of formability are mainly dictated by the failure behaviour of materials as well as its precursor: strain localization. In fact, in many sheet forming operations, it is the formation of necking which effectively limits the process rather than fracture.

The formability limits of steels and other metals are usually related to critical levels of strain in the material and in industrial practice its prediction is commonly assessed through the use of *Forming Limit Diagrams*, FLDs. These diagrams, originally developed by Keeler et al. [12], delimit the zones of safe formability for different strain paths as a function of the maximum and the minimum principal strains. Nowadays two different limit strains are mainly used in FLD’s, one based on localized necking and another one based on fracture. In the first case, the boundary between safe deformation and strain localization in the diagrams is marked by the *forming limit curve* (FLC) which represents the point of necking for different combinations of major and minor strains. The FLD’s were later also applied for fracture prediction and in this case the delimiting region is named the *fracture limit curve* (FrLC).

FLD’s have been successfully applied in stamping of mild and conventional high strength steels, where the typical failure mode occurs by localized necking. However, the predictions of the formability limits for stretch-bending and edge cracking based on FLD and other methods based on localized necking has only offered partial results for AHS steels [11]. This may be attributed to the fact that fracture of these steels occurs by limited or even in complete absence of strain localization. The fracture is not associated to a brittle behaviour of the material because typical features of ductile failure micromechanisms are still observed, i.e. the fracture surface usually exhibits a dimpled topography rather than a cleavage one. The type of failure observed in AHSS is typically referred to as shear fracture [13] and although it is not yet fully understood, the characteristics of the microstructure are considered among the most important factors affecting its occurrence.
In order to overcome the formability limitations of AHS steels and the future generations of automobile materials, a better understanding of the failure mechanisms at a microstructural level is required where the concept of damage is fundamental.

From a mechanical point of view, the macroscopic failure of materials during deformation is the final consequence of a series of events initiated with the internal damage in the microstructure. The concept of damage is usually defined as degradation of the mechanical properties of the material, sometimes referred to as softening, due to the nucleation and growth of defects, such as microvoids or microcracks, and their coalescence into macrocracks [14]. The reason of the degradation of the material integrity is exclusively attributed to the null load-carrying capacity of the voids. However, the materials response to further damage development leads to a second important concept in fracture, referred to as damage sensitivity. A material is said to be damage sensitive when the first existing mesocrack of the process becomes unstable under the action of applied homogeneous stress [15]. These concepts in fracture damage are usually confined to a continuum medium and the only incorporation of microstructural information into this approach is made through the geometry of voids and phenomenological criteria of void initiation [16, 17].

Nevertheless, the evolution of damage in the microstructure of metals and alloys is more complex and is strongly linked to the deformation process of the microstructure which is highly heterogeneous due to the presence of (i) different phases and (ii) the polycrystalline character of the matrix.

The incorporation of the above variables in the improvement and prediction of the formability of materials with automotive application is a current challenge, which has been addressed by different approaches, from computational to metallurgical [18-21] ones, but still many aspects remain not well understood.

1.3 Objective and outline of the thesis

The principal objective of this thesis is focussed on the identification and characterization of the damage micromechanisms occurring during plastic deformation of three different materials IF steels, DP steels and AA6016 aluminium alloys that have found very wide application in the automotive industry. The methodology followed to achieve this goal consists of both an experimental and a modelling approach.

Although the three materials studied in this thesis are totally different in chemical composition, microstructure and mechanical properties, they present the same type of problem from a micromechanical point of view: a ductile matrix containing inclusions where both matrix and inclusion possess different mechanical properties. This similarity
allows for a generic approach aimed to obtain a better understanding of the ductile failure micromechanisms in modern alloys by comparing and studying three different cases (i) small rigid inclusions with cubical shape strongly embedded in a ductile matrix (IF steels); (ii) a dense population of hard “inclusions” in a ductile matrix (DP steel); and (iii) brittle inclusions weakly embedded in a ductile matrix of which the strength is controlled by nano-sized precipitates (AA6016 aluminium alloy).

The thesis is organized in eight chapters. The state-of-the-art on ductile failure mechanisms together with the microstructural features involved during plastic deformation of metals is given in Chapter 2. Although the introduction of this chapter is intended to give an overview of the failure mechanisms at different scales, the rest of the chapter as well as the thesis are limited to the micro- and mesoscale. This chapter also includes a review and assessment of the different EBSD-based methods developed for characterization of the deformed microstructure.

The materials and the techniques employed for their characterization are described in Chapter 3 with a special emphasis on EBSD technique. The latter was used as a tool for qualitative and quantitative characterization of the materials during deformation in order to understand better the crystallography of the deformed metal matrix and its association to damage initiation. The mathematical development and description of the tools used for post-processing the EBSD data are described in detail in Appendix A. Appendix B is dedicated to the methodology of acquiring a quantitative estimation of the geometrically necessary density of dislocations from the orientation gradient derived from crystal orientation data.

The following three chapters are devoted to the experimental results from the studied metals. The analysis of the failure mechanisms of the IF steel subjected to tensile deformation is presented in Chapter 4. Because of the low inclusion content in this steel, the evolution of the plastic deformation in the ferrite matrix is also analysed in detail to the purpose of employing this knowledge in a comparative study with DP steels. Chapter 5 is dedicated to DP steels with an extensive study on void creation within the microstructure. Finally, Chapter 6 reports a study on the different failure micromechanisms of two AA6016 aluminium alloys varying mainly in the Mg content.

Based on the experimental results from DP steels, the stresses generated from the strain partitioning between the ferrite and martensite is considered of importance for void initiation. A mathematical model is proposed in Chapter 7 in order to first quantify the critical conditions for void initiation and second to predict the optimum microstructure for damage resistance.
Finally, the general conclusions are presented in Chapter 8 together with recommendations for future research.

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Chapter 1

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Chapter 2

Failure micromechanisms and plastic deformation: A literature review

The mechanisms acting during ductile fracture involve different scales. A short survey related to the fracture micromechanisms occurring during plastic deformation of metals is given in this chapter. Firstly, a description of the overall mechanisms of failure occurring at different scales is provided together with the state–of-the-art on ductile fracture modelling. A further review is given on the critical aspects contributing to the stages of failure from a microstructural point of view. Because the plastic deformation is a fundamental part on the study of ductile failure a review of the plastic deformation processes occurring at the microstructural level is presented together with a state-of-the-art report on the use of Electron Backscatter Diffraction (EBSD) for characterization of local deformation.

2.1 Ductile fracture mechanisms in metals: from nano- to macroscale

The occurrence of ductile fracture in metals is a process involving a broad range of length scales, from the atomic to the macroscopic scale, where the characteristic mechanisms acting on each scale differ but are interconnected. Figure 2.1 shows examples of the failure mechanisms acting at different scales.

The principal nano-mechanisms occurring during failure of metals are attributed to vacancy movement, dislocation interactions, dislocations coalescence and atomic layers separation. In the first case, vacancies migrate and conglomerate into clusters and at certain point lead to void formation. As this process is diffusion controlled, it is exclusively associated with high temperature deformation and/or low strain rates and therefore ascribed to creep failure [1]. Experimental evidence also supports an alternative nano-mechanism based on a model of dislocation climb [2, 3]. In the case of
dislocation coalescence, this process involves the pile-up of dislocations at an obstacle, such as a grain boundary, until a critical stress is reached where the dislocations coalesce together to form a cavity. This mechanism was proposed by Zener [4].

Figure 2.1: Examples of different scales involved in the fracture of metals. (a) Molecular dynamics simulation showing the dislocation interaction with a void [5]. (b) 3D EBSD map from IF steel where two voids nucleate on the interface between a TiN particle and the ferrite matrix (Chapter 4 of this thesis). (c) Cross-section of a tensile sample prior to fracture where voids coalescence is taking place [6] and (d) Broken tensile samples of aluminium with two different fracture modes [7].

Finally, the separation of atoms from their neighbouring atoms by breaking atomic bonds is the main process attributed to void initiation during plastic deformation. This separation occurs preferentially in brittle particles or at sites where the bond is weaker, such as the bonding between atoms of different elements or regions where the atoms are less ordered. This mechanism is further facilitated under the influence of high stress concentration, such as observed at dislocation pile-ups. After voids are initiated, the
subsequent void growth is produced by further breaking of atomic bonds together with complex dislocations interaction with the cavities [8, 9], cf. Figure 2.1a.

By considering the microstructure in the micro- to mesoscale, void initiation occurs at sites where strain compatibility is difficult to achieve. This incompatibility eventually leads to stress concentrations in these sites. Therefore, possible features prone to void nucleation are the heterogeneities found in the microstructure such as second phase particles, inclusions, different phase constituents and grain boundaries. In the first case, voids may initiate by decohesion of the interface delimiting the particle and the matrix or by the internal cracking of the particles. Figure 2.1b illustrates the case of void initiation in a TiN particle by interface decohesion where intra-grain substructures are created around the particle. In the absence of particles in polycrystalline metals, voids nucleate along grain boundaries where the deformation is heterogeneous [10]. The subsequent growth of the void is controlled by the matrix deformation. The individual voids will continue growing until certain conditions are reached and as a consequence coalescence of voids occurs to form macrocracks. One example is given in Figure 2.1c where a macrocrack is already formed and its propagation starts. The void coalescence occurring in the microstructure marks the initiation of the fracture process on the macroscale.

The growth of macrocracks is initially stable. The stability of a macrocrack is related to its propagation speed and the boundary conditions, such as structure geometry or loading conditions. During this process, the macrocracks require considerably smaller average plastic strain as a result of the strain localization in the region in front of the crack, known as the fracture process zone (FPZ). The further propagation of the crack takes place in this zone and therefore it is confined within this region. However, the interaction between the boundary conditions and the crack growth reaches a critical point when the propagation of the crack occurs in an unstable way, i.e. growing with an increasing speed, and finally producing the global fracture of the structure, cf. Figure 2.1d.

The extent of knowledge generated on the failure process in each of the scales is proportional to the dimension of the scale. Most of the research has been done on the macroscale since the 50’s, whereas the micro- to mesoscale starts receiving nowadays more attention and there are only a few reports available on the nanoscale, largely based on atomistic simulations.
2.2 Ductile failure modelling: Some approaches with a historical perspective

The role of modelling in ductile failure is not an end in itself, but rather pursues a practical objective which could fit in one or more of the following categories:

- The prediction and prevention of failure in structural materials; e.g. the use of Finite Element Analysis for assessing metal forming processes [11].
- Modelling to the purpose of providing insight in the relevance of ductile fracture mechanisms as well as in the available quantitative data; one example is the study of Shabrov et al. [12] where Finite Element Analysis was used to determine the void nucleation stress of TiN particles or the work of McVeigh et al. [13] where a better understanding of the void coalescence mechanisms is pursued.
- Materials design aiming optimization of failure resistance.

As Brocks [14] pointed out, the different models pertaining to ductile failure have followed the historical evolution of fracture analysis with the particular characteristic of evolving from the macroscale to progressively smaller scales. Elastic-Plastic Fracture Mechanics and the related macroscopic parameters, like the $J$-integral, were one of the first attempts to predict ductile failure of structures [15]. However, this phenomenological approach found some limitations on predicting geometries or conditions different than the ones originally tested. Afterwards, the continuous improvement of computers together with numerical techniques, such as FEM, motivated the use of cohesive zone models [16] where traction-separation laws control the opening behaviour of cohesive zone elements in order to simulate cracks. Fracture mechanics and cohesive zone elements have the common characteristic of being able to explicitly model the crack geometry on the macroscale.

For this reason both modelling techniques are considered to use a discontinuous approach [17]. All models using the discontinuous approach depend on the mechanical behaviour of the FPZ of which the dimensions are usually considered in the mesoscale. In the quest of micro-mechanical and physically motivated models, Gurson [18] proposed a model, which together with the modifications made by Tvergaard and Needleman [19], gave place to the porosity-based model known as the GTN model. The GTN model intends to capture the softening effect on the mechanical properties produced by voids in a region of the microstructure, namely the Representative Volume Element (RVE), and incorporates it in a strain-damage constitutive equation. Rosselvier [20] afterwards proposed a similar model, based on continuum damage mechanics and thermodynamics, which basically describes void growth. This type of models gave origin to the concept of damage where the strength decline of the material is related to
the existence of voids. However these constitutive equations still depend on the void initiation and growth laws controlled by the microstructure whereby these laws are usually described by statistical and phenomenological models. The employed void coalescence laws are still primitive and one of the weak points in this type of models. Nowadays there is more emphasis on improving the mechanical response of the RVE by better describing the damage mechanisms in the microstructure; i.e. the nucleation, growth and coalescence of voids within the microstructure.

The present thesis is dedicated to the study of failure mechanisms in the micro- and mesoscale where dislocation patterning and grain assembles are considered. Thus hereafter all experimental work and model formulations are limited to this range of length scale.

2.3 Microstructural ductile failure mechanisms

The failure events taking place during ductile fracture of metals at microstructural level are usually classified in three consecutive stages, namely, void initiation, void growth, and void coalescence. Despite this classification is conceptually convenient and straightforward, these different stages are complex to define and experimentally difficult to separate. The importance and predominance of each failure stage differs, depending on the material as well as on the deformation mode.

In the context of fracture analysis, the term particle is used as a general and indiscriminate denomination to designate entities embedded in the material matrix. Thus, this term applies to non-metallic inclusions, second phases (e.g. martensite or austenite) or other microstructural constituents such as pearlite and bainite.

2.3.1 Void initiation

Many studies have been carried out to identify the sites and conditions for void nucleation [21-24]. In the case of plastic failure of metals at room temperature conditions, the most common sites for void initiation are second phase particles. Voids have been reported to initiate either by interfacial separation between the matrix and the particle or by fragmentation of the particle.

The principal factors controlling the occurrence of void initiation in particles include the interfacial bond strength between the particles and the matrix [25], the internal fracture strength, stiffness, size [26], aspect ratio, shape and spatial orientation [27] of the particles, the difference between the mechanical properties of particles and matrix [28], the particles volume fraction and the local strain-stress conditions. A typical example of these factors is the particle size dependency of the fracture, according to
which larger particles are more prone to rupture. Although this behaviour has been experimentally well documented, the responsible mechanism is still a point of debate [21, 26].

These factors are also of crucial importance for the type of the void initiation mechanism acting in the particle-matrix system. For instance, it has generally been observed that while inclusions with large aspect ratio are prone to multiple fragmentations, equiaxed inclusions most commonly nucleate voids by interface decohesion. The large number of factors controlling void initiation together with the heterogeneous nature of the microstructure has as a consequence that the void initiation occurs gradually with increasing strain, rather than taking place simultaneously at a large group of particles.

Several theoretical studies have been made on the conditions leading to void initiation and one common assumption in these studies is that a void initiates when the conditions inside or over the boundary of a particle satisfy one or a combination of the following three criteria namely: (i) an energy criterion, (ii) a local strain criterion and (iii) a local stress criterion. The energy criterion, which was initially proposed for particle fracture by Gurland et al. [29], is an extension of the Griffith energy balance [30]. This criterion establishes that a void is formed at the interface when the locally concentrated elastic strain energy ($U_{el}$) which could be released upon decohesion becomes comparable to the energy of the new stress-free surfaces, $U_{surf}$.

Because of the introduction of volume and surface in the analysis, the energy criterion exhibits a size-dependency factor in such a way that the larger particles satisfy this criterion with lower elastic stored energy. In this regard, Tanaka et al. [31] calculated that even in the elastic region before plastic yielding, the energy criterion was already achieved for particles larger than 25 nm. This was contrary to their experimental observations where some larger particles still remained attached to the matrix at large values of plastic strains. They concluded that the energy criterion is a necessary but not a sufficient condition for void nucleation and that the actual separation requires reaching the interfacial strength.

The stress criterion establishes that separation of the particle-matrix interface or fragmentation of the particle will occur if a critical local stress ($\sigma_N$) is attained, namely the void nucleation stress. However, different types of stress components and their combinations have been considered to be representative in this criterion. In the case of particle-matrix decohesion, the stress component normal to the interface between the particle and the matrix ($\sigma_n$) is usually considered in the criterion controlling void initiation. Additional modifications have been made in order to also incorporate the
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influence of the local shear stress \((\tau)\) on the interface. For example, Bonfoh et al. [32] proposed the following stress criterion:

\[
\sigma_N = \sigma_n + \beta \tau
\]  
(2.1)

The inconvenience of the previous criterion is the appearance of the material parameter \(\beta\), which represents an additional adjustable term that should be determined.

In a similar approach, Argon et al. [21] proposed the introduction of a term, which accounts for stress triaxiality. This criterion can be expressed as

\[
\sigma_N = \sigma_e + \sigma_h
\]  
(2.2)

Where \(\sigma_e\) is the average local stress in the region of the particle and \(\sigma_h\) is the hydrostatic component of the stress tensor, equal to one third of the stress tensor trace. An additional material parameter, \(c\), has been inserted in the hydrostatic term of the above equation in order to account for the effectiveness of the far-field hydrostatic field.

Then, the stress criterion is rewritten as

\[
\sigma_N = \sigma_e + c\sigma_h
\]  
(2.3)

Shabrov et al. [12] made a comparative study of some of the above stress criteria in order to characterize the particle fragmentation in titanium modified 4330 steel. It was found that the criterion of equation (2.3) displayed a better correlation with experiments although they recognize the convenience of the fitting parameter \(c\). Furthermore, it is important to mention that the relevance of the hydrostatic pressure on void initiation is still an issue. Cox and Low [26] observed that samples with different levels of stress triaxiality shared the same evolution of void nucleation sites when plotted against the applied stress. Contrary to these observations, Pyshmintsev et al. [33] reported the reduction of void nucleation sites under the influence of high hydrostatic pressures. Both observations are contradictive and one possible explanation is based on the difference between hydrostatic tension and compression. Another interpretation of the results obtained from the high hydrostatic pressure experiments [33] involves the fact that a void is only detectable when a certain amount of growth has occurred. Negative pressures are known to inhibit or reduce void growth and, under certain conditions, voids may become undetectable by microscopic techniques. Therefore, it is difficult to assert whether or not void nucleation is inhibited under high hydrostatic pressures because of limitations on detecting voids with little growth. However, this incapability to detect voids with reduced growth is related to one of the main experimental difficulties in fracture characterization, the separation of void initiation from its growth.

One common limitation to most of the stress criterion models is the lack of sensitivity to the particle size. As a consequence, the size dependency is usually
incorporated with the addition of other criteria, e.g. the energy criterion [32], or from supplementary considerations, such as particle interactions [21].

The strain criterion is characterized by a critical strain at which void initiation occurs. Although a nucleation strain is usually employed to quantify void nucleation, this criterion does not appear to be an absolute quantity for a given type of particles.

In the majority of cases, void initiation in metals preferentially arises in particles although it is not exclusively restricted to these sites. Alternative void nucleation sites have also been reported during plastic deformation. This is the case of the $\gamma$ phase in TiAl alloys where the interaction of deformation twins with grain boundaries produces voids along certain grain boundaries [34]. Even in the absence of second phases or grain boundaries, the intersection of shear bands in ductile single crystals can lead to the formation of voids [35]. Therefore, in general all regions with strain incompatibility are potential nucleation sites for voids.

2.3.2 Void growth

Once a void has been nucleated within the microstructure, it will start growing at a certain rate and along specific directions, which are determined by the material properties and test conditions. The void growth is mainly affected by the stress triaxiality, the size, spatial distribution and shape of the particles, the strain rate and the temperature.

On the fracture surface of broken structures, void walls usually exhibit stairs-like marks. The marks are the result of wavy or serpentine glide of slip planes which intersect the growing void. These experimental observations indicate that void growth is mainly controlled by dislocation interactions. Stevens et al. [36] proposed that void growth is an absorption process of dislocations into the void which occurs only when the dislocations move on planes that intersect the void edges. However this idea was refuted by Meyers et al. [37] because of the high probability of dislocations blocking the planes of the void edges and stopping void growth. An alternative mechanism proposed by Wolfer et al. [38] suggests that the emission of prismatic loops is responsible for void growth. This mechanism was adopted by Lubarda et al. [8] and they added that besides the prismatic loops, the cooperative expansion of shear loops originating from the void surface also contributes to the void growth. The molecular dynamics simulations were in agreement with this proposed mechanism [9].

Despite the importance of dislocations for void growth, most of the models describing void enlargement are based on continuum mechanics. One of the more frequently used models for void growth was formulated by Rice and Tracey [39]. This
model calculates the growth of a spherical void embedded in a rigid perfectly plastic matrix of infinite size subjected to a uniform remote strain field. The differential equation is given by

$$\frac{dR}{R} = \alpha \exp \left( \frac{3\sigma_h}{2\sigma_{eq}} \right) d\varepsilon^p_{eq}$$  (2.4)

Where $R$ is the radius of the void at the equivalent plastic strain $\varepsilon^p_{eq}$; $\sigma_h$ is the hydrostatic stress; $\sigma_{eq}$ is the Von Mises equivalent stress and $\alpha$ is an integration constant determined by Rice and Tracey [39] which value is $\alpha=0.283$.

One of the main drawbacks of the previous model is the neglected interactions between neighbouring voids which can be of importance on metals with large content of particles [40]. Although equation (2.4) includes several assumptions and drawbacks, it reflects the important influence of stress triaxiality (defined by the ratio $\sigma_h/\sigma_{eq}$), the initial void volume (incorporated by the integration constant of the void radius) and strain rate (indirectly given by $d\varepsilon^p_{eq}$).

As Cow and Low [26] proved in their experiments, the growth direction of a void is controlled by the degree of stress triaxiality in the surroundings. This means that voids located in regions with high stress triaxiality will experience growth along all directions, while in the case of uniaxial tensile conditions; voids will exclusively enlarge along the tensile direction. As a consequence of the void enlargement geometry, the void growth rate is higher under triaxial stress conditions. The situation is different when the stress triaxiality is low, such as under pure shear or compression. Most of the models describing void growth, e.g. equation (2.4), predict no void expansion for pure shear conditions. However, FEM simulations of voids subjected to shear fields [41, 42] showed that voids may grow under shear conditions into elongated tails along the principal shear plane. Xu et al. [43] experimentally observed this type of void growth geometry on a three-dimensional basis. The singularity of this void geometry is not in total contradiction with equation (2.4) because although the void surface shows an appreciable increment, the void volume expansion is kept low [42].

Because of the progressive increase of the void initiation process, voids represent a broad size distribution at a given level of strain 26. In the case of particle fragmentation, the void size heterogeneity is also affected by the particle size distribution because of two reasons. One reason is due to the earlier void nucleation in the larger particles and secondly because of the initial void size which is proportional to the particle size.

In summary, the void growth is highly dependent on the local stress-strain conditions and the initial void size. Therefore the control of any of these conditions has a direct influence on the void volume fraction evolution during deformation.
2.3.3 Void coalescence

The coalescence of voids represents the last stage of ductile failure at the microstructural level and the beginning of fracture at the macroscopic scale. Void coalescence is defined in general terms as the unification of growing voids [22]. However, this fusion process may occur by diverse mechanisms where different factors play a role; for instance the stress triaxiality conditions, the interaction between the neighbouring voids, the heterogeneous nature of the microstructure and the localization of the deformation. As the strain localization factor is relevant for the fracture of most of the industrial alloys, Pardoen and Brechet [44] proposed a more precise definition of void coalescence: “void enlargement evolution occurring after the transition to a localized mode of plastic deformation within the ligament between the voids”. The importance of the previous definition is the incorporation of the concept of strain localization produced by the interaction between neighbouring voids.

![Example images of void coalescence mechanisms](image)

*Figure 2.2: Examples of the principal void coalescence mechanisms observed in ductile metals. (a) Coalescence by void impingement in nickel [37]. (b) Coalescence by void sheet formation in AISI 4340 steel [26].*

Depending on the type of strain localization, different mechanisms of void coalescence are usually classified into void impingement coalescence and void sheet coalescence (cf. Figure 2.2). The first mechanism, also known as tensile void coalescence [44], is characterized by an internal necking down of ligaments between voids that have enlarged their sizes significantly until impinging on each other; e.g. Figure 2.2a shows an example of this mechanism observed in nickel. One characteristic of voids impingement coalescence is that the regions close to the fracture surface usually exhibit a high void volume fraction, (e.g. approximately 0.15 for HY-100 steels.
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[45]) which could be attributed to the extensive strain-induced void growth prior to catastrophic void linking and fracture.

Alternatively to void impingement, the void sheet coalescence mechanism is distinguished by the localization of the deformation between neighbouring primary voids by shear; reason by which this coalescence mechanism is also referred to as shear coalescence. If the material contains particles with a bimodal size distribution, a secondary population of voids starts nucleating at the smaller particles situated within the shear localization region where the growth of these microvoids eventually leads to the failure of this zone. The region of the localization is usually orientated along the plane of maximum shear and is dimensionally narrow as a sheet. A typical example is presented in the work of Cox and Low [26] in AISI 4340 steels where voids in the sulphide inclusions (primary voids) were coalesced by the void sheet mechanism at carbides (secondary voids) (cf. Figure 2.2b). Because of the limited volume expansion of voids in a shear field and the high localization of deformation, the resultant void volume fraction at final failure by this coalescence mechanism is lower in comparison to the void impingement mechanism. For example, Bandstra et al. [45] reported for HY-100 steels a value of void volume fraction of about 0.02 at failure where the MnS stringers acted as primary voids.

The type of acting void coalescence mechanism also depends on the stress triaxiality although contradictory results are reported in the literature. On the one hand, a series of publications by Bandstra et al. [45-48] examined the effect of stress triaxiality on the void coalescence mechanisms by experimental procedures on HY-100 steel completed with finite element analyses. They observed that void sheet coalescence occur at high stress triaxiality conditions while void impingement coalescence prevails at low triaxiality loading. It is important to mention that the stress triaxiality was experimentally varied by different notch sizes in the tensile samples. On the other hand, Bao and Wierzbicki [49] reported an opposite behaviour on a 2024 aluminium alloy, where void sheet coalescence was observed to occur at low stress triaxiality conditions and void impingement was prone at higher stress triaxiality conditions. The principal difference with the experimental work of Bandstra et al. [45], besides the material, was the consideration of compression and shear tests in addition to the notched tensile specimens. In fact, the void sheet coalescence was exclusively observed on the compression and shear samples, which involve different stress and strain conditions than in the notched tensile samples. The main conclusion from this discrepancy is that the transition between the void coalescence mechanisms cannot be described by a single parameter of the loading conditions.
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In summary, void impingement coalescence and void sheet coalescence mechanisms may occur in the same material depending on the deformation conditions. However, as Cox and Low [26] pointed out, the high strain localization associated with void sheet coalescence reduces the global deformation at fracture of the material.

2.4 Microstructural aspects of plastic deformation

It is well known that ductile fracture totally depends on the evolution of plastic deformation of the material. Characterization of microstructural damage in ductile metals is challenging due to the complex internal interaction of the heterogeneous deformation features preceding the void nucleation. Thus the microstructure evolution during deformation is a key point in the study of ductile fracture.

2.4.1 Experimental observation of microstructural deformation

Dislocation flow is one of the principal mechanisms to accommodate plastic deformation in metals. During this imposed flux, some of the dislocations are trapped in their journey due to the complex interaction with other dislocations or microstructural features such as grain boundaries or precipitates. The storage of these residual dislocations generates substructures inside the grains which are frequently accompanied by crystal orientation gradients. The characteristics of the grain substructures, e.g. geometrical arrangement or sizes, are mainly determined by the material and the deformation conditions. In the special case of metals with cubic crystal structure, an important parameter is the stacking fault energy because its value is associated to the occurrence of additional slip systems or twinning.

At low strain levels (ε < 0.1), the residual dislocation storage is firstly arranged in cell structures. The cells, whose boundaries are formed by tangles of dislocations and designated as cell boundaries, are the result of a statistical trapping of dislocations. As a consequence of this stochastic process, the misorientation angle between cells is small (~1°). Additionally, as Kuhlmann-Wilsdorf stated [50], tangled dislocations are the relaxed configurations arising from the uniform dislocation microstructure that is the direct result of homogeneous deformation. In general, the cells have an equiaxed shape and are relatively small (0.5 - 1 μm) [51]. Dislocation tangling can be triggered by dislocation interaction, grain boundaries or even particles. Figure 2.3a displays an example of cell structure formation in IF steels, where some cell boundaries are created by dislocation trapping at titanium carbide precipitates [52].

At higher deformations, dislocations are preferentially accumulated at certain cell boundaries, giving place to the cell blocks. The cell blocks, also known as cell bands,
enclose groups of previous cells and are delimited by longer and almost straight boundaries with high dislocation density. The cell block boundaries are also known as dense dislocation walls (DDW) or Geometrically Necessary Boundaries (GNB) and, as opposed to cell boundaries, these boundaries are able to accommodate higher misorientation angles. In some cases, the DDW begin to split along their length and the result is the creation of lenticular shaped features called microbands, cf. Figure 2.3b. One of the characteristics of microbands is that initially the misorientation between the interior of the microband and the neighbouring matrix is considerable (~2°) [52]. In the case of BCC metals, the microbands form on slip planes, which have maximum Schmid factor and share the same activated slip system [52].

Figure 2.3: TEM micrographs from IF steel samples deformed by rolling at different strains. (a) Tangled dislocations (ε = 4.2%) where cell structures were originated from the interaction between particles (arrowed) and dislocations [52]. (b) Microband growing from a precipitate at ε = 9.8% [52]. (c) Shearing process of microbands by ‘S’ bands (ε = 46%) [54]. (d) Shear bands crossing over several microbands [54].
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The increase of strain typically enhances the generation of microbands until another type of dislocation structure, known as shear band, appears. Shear bands are characterized by their intense shear localization into narrow regions, propagating independently of the grain structure and crystallography [53]. In other words, when these bands are in an advanced stage of evolution, the shear bands cross over several grains and their propagation orientation is controlled by the macroscopic conditions rather than by the crystal orientation of the grains. However, the origin of shear bands has indeed a microstructural and crystallographic nature: they are always parallel to one set of microbands and commence within individual grains [54]. At earlier stages of shear band formation, the microbands start being sheared at different positions as shown in Figure 2.3c. These microstructural features which resemble the ‘S’ shape (segments AA’A’’, BB’B’’ or CC’C’’ in Figure 2.2c) are known as ‘S’ bands and are the precursor of shear bands. The formation of these bands is caused by dislocation glide. After the formation of ‘S’ bands, the continuous shear process generates thin shear bands which have a misorientation angle of approximately 10º [54], cf. Figure 2.3d. Finally, the shear bands become thicker as a result of a relative shear displacement of two thin shear bands [55].

2.4.2 Characterization of plastic deformation by EBSD

Crystal orientation mapping by Electron Backscatter Diffraction (EBSD) has nowadays become a wide spread alternative method for plastic strain analysis at microstructural level. The advantages of EBSD over typical TEM-based methods are the larger analyzed areas together with their punctual crystal orientation data, which enormously improve the statistics of deformation analyses. Nevertheless, spatial and especially angular resolutions are the principal issues limiting the application of EBSD technique. One of the consequences of these limitations is the inability of the technique to detect substructures with a dislocation density lower than a certain value. For instance, the wide dislocation walls associated to substructures inside α-fibre ferritic grains in IF steels were detected by TEM but difficult to visualize by EBSD [56].

Several characterization methods have been proposed for EBSD-based plastic strain analysis and some of the principal approaches used in metals are given below.

The dislocations accumulated in the grain substructures during deformation produce a local elastic distortion of the crystal lattice. The volume affected by this disturbance has a detrimental effect on the quality of the acquired EBSD pattern. Wilkinson et al. [57] reported one of the pioneering works exploring and quantifying the influence of plastic deformation on the EBSD patterns. In this work, the sharpness and
contrast of the Kikuchi bands were quantified by different parameters and afterwards their sensitivity to plastic strain was assessed. Nowadays, similar parameters to measure the EBSD pattern quality are employed by different commercial software. Some examples are the image quality \[58\] (IQ) or the band contrast \[59\] (BC) parameters. Both of them are derived from the Hough transformation used for the detection of the Kikuchi bands. On the one hand, the IQ parameter is defined as the intensity sum of the detected peaks in the Hough transformation for each EBSP while the BC parameter is calculated as the average intensity of the Kikuchi bands normalized by the overall intensity within the EBSP. However, one of the main drawbacks of this type of parameters is their sensitivity to other defects or variables, such as the sample surface cleanliness (polishing effects, oxidation), equipment settings (electron beam current and energy, inclination of the sample) or even to the crystal orientation. As a consequence, the IQ and BC parameters cannot be used as absolute values to quantify the deformation and their numerical comparison between different scans or even between different grains of the same scan is not recommended. Therefore the parameters based on pattern quality are typically used for visualization of grain substructures \[60, 61\] although in some cases are not able to capture the deformation features of low strained materials \[62\].

Alternative methods to visualize the deformation of the microstructure are based on the crystal rotations created within grains. Some basic examples are the crystal orientation maps such as the Inverse Pole Figure (IPF) or the Euler Angle maps. In the IPF map a colour is associated to each pixel according to the crystal axis that is parallel to a specific sample direction (e.g. RD, TD or ND of a rolled sheet) whilst the Euler Angle map colours each pixel in accordance to its Euler angles. Although it is possible to visualize the colour gradients within large grains in samples with relatively large strains \[56, 61, 63\], in general these maps are not as sensitive to deformation as the pattern quality maps \[62\]. The reason for this inability is the fact that deformation in the microstructure is reflected by orientation gradients rather than just crystal orientations.

Other more efficient methods to visualize and characterize the in-grain crystal rotations of strained microstructures are based on misorientations. Within this category, a further classification can be made to separate short- from long-range orientation gradient methods. The short range orientation gradient methods are characterized by using the orientation data from the nearest neighbours of each data point. The low angle grain boundary (LAGB) maps are a simple example in this category, where the misorientation angle is calculated for each neighbour separately and afterwards lines are drawn along the boundary between pairs of orientation satisfying certain misorientation
angle ranges. Some studies have used the LAGB maps in order to measure the grain substructure morphologies [64, 65]. Although this type of data is important for characterization of plastic deformation, the angular and spatial resolutions obtained in EBSD data acquisition are still too restrictive to allow for detailed plastic analysis on the nanoscale, despite the efforts made to overcome these limitations by post-processing algorithms [66].

An alternative solution to visualize short-range orientation gradients is by averaging the misorientation angle between each pixel and its neighbours. The resultant value is known as Kernel Average Misorientation (KAM) although other names as pixel cluster misorientation [62] or average local misorientation [67] are also found in the literature. In order to avoid the influence of pixels from different grains, the KAM parameter only considers the neighbours with a misorientation angle lower than an upper limit threshold value. The KAM method is not only used for mapping but is also employed as a parameter to characterize the short-range orientation gradients within individual grains by averaging the KAM over all pixels corresponding to the same grain. This parameter is employed on the characterization of the grain substructures in Chapter 4 and the details of its calculation are given in Appendix A. The exclusion of data points with a KAM lower than a limiting threshold angle has been proposed to the purpose of eliminating the influence of instrumental noise or orientation gradient free regions [68, 69]. The KAM mapping has a better correlation with the distribution of dislocation densities within the microstructure than other methods. However, the absolute value of the parameter depends on the step size employed in the scan and, as a consequence, on the number of neighbours utilized. In general, the main limitation of the short-range orientation gradient methods is its high sensitivity to the EBSD angular resolution due to the fact that both magnitudes, i.e. the angular resolution of the equipment and the physical misorientation angle, are usually in the same range at low local strains.

In comparison to the aforementioned method, the approaches based on long-range orientation gradients are less sensitive to the EBSD angular resolution. The reason is that these methods usually deal with misorientation angles much larger than the angular accuracy. The main idea of this approach consists on calculating the misorientation angle of each crystal orientation within a grain with respect to a pre-selected reference orientation. Then, the crucial feature in this approach is the choice of the reference orientation. Brewer et al [62] suggested to employ the minimum distortion orientation as a reference orientation; i.e. the point with the minimum KAM. However, this approach leads in many instances to an ambiguous selection of the orientation reference due to the
existence of several regions within the grain with the same minimum KAM but different crystal orientations.

The use of crystal average orientation as reference orientation overcomes the ambiguity of using the minimum KAM and can be interpreted as the centre of an orientation cloud composed by the data points of a grain. The advantage of this kind of maps is the additional visualization of long-range orientation gradients [68], which is not possible with the local misorientation maps. Furthermore a parameter can be defined to account for the orientation spread around the reference crystal orientation which is referred to as the Scalar Orientation Spread (SOS) [70]. Further details on the calculation of the SOS parameter are given in Appendix A.

A more meaningful choice for the reference orientation is the original grain crystal orientation prior to deformation. The advantage for this preference is the direct link to the local rotations associated to the plastic deformation [71]. The difficulty of this method is to obtain the original crystal orientation.

Additional information of the grain substructures can be gained by taking into account the misorientation axis. This consideration allows for a better recognition of both short and long range orientation gradients [72] although works better for the large orientation gradients. The reason is that the instrumental error of the crystal orientation is incremented on the calculation of the misorientation axis.

A general drawback of all aforementioned methods based on misorientations is their intrinsic lack of connection to more quantitative measures of deformation such as strain, strain gradient or dislocation density. In the last case, the misorientations can be related to the local non-redundant content of dislocations, better known in literature as the Geometrically Necessary Dislocations (GND) [73]. However, the previous term is considered to be semantically incorrect†, whereby a more descriptive term should be Geometrically Necessary Density of Dislocations. One of the simple solutions used to overcome this issue is based on the formula by Ashby [73]:

\[
\rho_{GND} = \frac{\kappa}{b}
\]

In the above equation, the local GND density content, \(\rho_{GND}\), is estimated from the crystal curvature, \(\kappa\), and the burgers vector, \(b\), of the material investigated. This equation

† As W. Pantleon mentioned in a Work-Hardening course given in Leuven, Belgium (2009): “Every single dislocation is geometrically necessary because it is able to create an elastic distortion in the crystal structure. The misorientation observed on a larger scale is the product of a geometrically necessary density of dislocations and not the result of a density of geometrically necessary dislocations“.
has been used for example by Dillen et al. [74] to estimate the local non-redundant dislocation density of selected regions in DP steel ferrite grains. As this approach only considers the crystal curvature as a single value instead of all the components of the curvature tensor, the dislocation content obtained by this method represents a lower-bound limit.

A more elaborated approach to obtain a better description of the GND distribution is still being explored and is based on the crystal geometrical relations established by Nye [75]. In this method, Nye’s dislocation tensor which is used for the calculation of the GND is directly related to the lattice curvature tensor, where the curvature tensor can be directly estimated from the EBSD maps. A further development on the calculation of the GND content is given in Appendix B. In addition to the limitations imposed by the angular resolution of the EBSD technique, another drawback of this method is the difficulty to obtain all the components of the lattice curvature tensor. The reason is the two-dimensional nature of the EBSD maps and therefore the lack of information in the third-dimension. In special conditions, the orientation gradient in the third dimension can be assumed to be null and therefore the nine components of the curvature tensor are known. This is the case for instance of wedge indentation into single crystals [76] or compression of a columnar grain structure [77]. However, in most cases, any assumption for the components of the curvature tensor is not recommended. Another solution of this problem is the use of 3D techniques, such as the FIB-EBSD, in order to acquire the missing dimension. Demir et al. [78] were the first to employ this novel technique to determine the GND amount below indents of different sizes. This recent study showed the potential capability of FIB-EBSD on the characterization of local deformation, although nowadays this technique is still in development. There have also been improvements regarding the approach employed for the GND calculation. Initially, it was thought that only three components of nine of the Nye’s dislocation tensor could be obtained from 2D-EBSD maps [77] but Pantleon [79] demonstrated that six components of the curvature tensor can be directly used for the GND calculation. The results of Pantleon showed that the method based on Nye’s dislocation tensor represents a lower-bound limit of the real non-redundant dislocation density and this limit is improved as more components are used.

An alternative EBSD-based method for calculation of the GND density has been proposed by Wilkinson et al. [80]. This method is based on the direct analysis of the EBSD patterns instead of using the curvature tensor calculated from the crystal orientation maps. In this approach, small shifts in EBSD patterns are detected from image analysis and afterwards the associated elastic strain and lattice rotation tensors
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can be determined with high accuracy. The advantage of this method is that the all components of the elastic distortion tensor can be acquired (cf. Appendix B) and used to calculate the GND density without neglecting the elastic strains, which is a common assumption from all methods based on the curvature tensor. However, an important requirement for this method is a reference pattern which is used to compare the EBSP shifts. This is one of the reasons why this method is more suitable for small deformation studies where a reference EBSD pattern is easier to select. Some examples of the applicability of this method are the deformation zone of a fatigue crack [81] or the strain accommodation in the matrix from martensite transformation [82].

2.5 Conclusions

The ductile failure of metals incorporates several processes occurring at different scales and interacting among each other. On the micro-mesoscale, the failure process is considered to be composed of three subsequent stages — void initiation, void growth and void coalescence. Each of the three stages is controlled by different factors and the distinction between the stages is not easily made. In the case of void initiation, the identification of the void nucleation mechanisms and sites as well as the identification of the criteria controlling the initiation of voids are of primary importance.

The influence of plastic deformation on the microstructure is extremely strong and it is linked to the failure process although the incorporation of both of them in failure modelling is difficult and less explored. The emergence and development of crystal orientation maps by EBSD has brought the possibility to study the deformation at high strains and on the microstructural level. However this research area is still in development and critically depends on instrumental developments aimed to improve the resolution limits.

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Chapter 3

Materials and Experimental Tools

The descriptions of the materials and the experimental techniques used to study the micromechanisms of failure are given in this chapter. The materials used on this study are IF steel, DP steel and AA6016 aluminium alloys sheets (section 3.1). Tensile tests were used to study the failure mechanisms of all materials (section 3.2). The main techniques used to characterize the materials in the initial and deformed conditions were quantitative metallography (section 3.3), dilatometry (section 3.4), X-ray diffraction (section 3.5) and EBSD (section 3.6).

3.1 Materials

Three different industrial sheet materials, which are used for car-body structural applications, are considered in the present study. The materials are an interstitial free (IF) steel, four grades of dual phase DP600 steel and two AA6016 aluminium alloys.

3.1.1 Interstitial Free steel

Interstitial Free (IF) steels are widely used in the automotive industry due to their high ductility, deep drawability and good formability [1]. For example, this steel is used in the automobile industry for the rear floor pan, the spare wheel well, and the front and rear door inners. The superior formability performance of these steels is largely owed to the extremely low level of interstitial elements (i.e. carbon and nitrogen) in the solid solution. The non-aging property together with the low yield strength of the IF steels are also a consequence of the low level of interstitials in these steels.

The low amount of interstitial elements in the IF steels is firstly controlled by a strict steelmaking process in which vacuum degassing plays an important role. However, these processes are not able to completely eliminate all C and N from solid solution in the BCC crystal lattice, thus the remaining interstitial elements are removed by
stabilizing them through intentional precipitation formation. The use of the microalloying elements titanium and niobium is common to this purpose and, based on the relative amount of both elements, the IF steels can be classified as single stabilized titanium- or niobium-bearing or as dual stabilized (Ti-Nb) IF steel.

In the case of Ti-IF steels, the stabilization process occurs through the formation of precipitates mainly based on carbon, nitrogen and titanium, Ti(C,N). Moreover, the presence of sulphur and phosphorus can also produce another type of precipitates such as TiS, Ti₄C₂S₂ or FeTiP [2]. The scheme displayed in Figure 3.1 represents a thermodynamic stability diagram for the different precipitates formed in Ti-IF steels. The most stable reaction is the TiN precipitation which starts at the beginning of the liquid metal solidification stage during the casting process at around 1523°C [2], although this temperature depends on the stoichiometric ratio between Ti and N [3].

In addition to providing an interstitial free ductile ferrite matrix, the presence of Ti(C,N) precipitates plays an important role during thermomechanical processing of steel sheets in two ways: (i) by providing preferential nucleation sites for the recrystallization when they exceed a critical size of approximately 1 μm and (ii) by acting as pinning particles during grain growth if they are much smaller [1, 4]. However, the latter mechanism is still a point of debate as pointed out by Shi and Liu [5]. The precipitation of Ti-based particles usually gives rise to two types of precipitates, distinguished by their length-scale in nano- and micron-sized particles [4]. The characteristic features of the micron–sized particles are their cubical shape and chemical composition which is mainly titanium and nitrogen. These coarse precipitates are usually referred to as TiN particles and their cubic geometry is formed because the {100} cubic facets minimize the total surface energy of the particles [6].

Besides the importance of precipitation reactions for obtaining an interstitial free ferrite microstructure, the crystallographic texture is another critical factor controlling the formability of IF steels. The desired texture for these steels is a strong γ-fibre texture [7] (cf. Figure 3.2a), which is ideal to diminish the planar anisotropy, responsible for
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earing, and to maximize the normal anisotropy, which allows increasing the thinning resistance in deep-drawing applications [8, 9]. A typical recrystallization texture of an IF steel is shown in Figure 3.2b. The desired texture of IF steels is obtained through the control of the thermo-mechanical processes where the precipitates may play an important role, especially in the recrystallization process during the continuous annealing treatment [1, 4]. The precipitates exert a pinning force on the ferrite grain boundary mobility during their growth in the annealing process.

Figure 3.2: a) $\varphi_2=45^\circ$ section of the Euler space with the location of the important components and fibres for BCC metals and b) similar Euler space section showing the ODF of a recrystallized IF steel [7]. Intensity levels $2, 3, 4, 5, 6, 7, 8, 9, 10, 11$.

The effectiveness of this pinning effect mainly depends on the size and distribution of the precipitates. In the case of a dense dispersion of fine precipitates, the desired growth of the nucleated grains with $\gamma$-fibre orientations is not inhibited but rather retarded, which gives the opportunity that grains with unfavourable texture components start nucleating and growing [1]. Therefore, the size and distribution of the precipitates need to be optimized in order to increase the volume fraction of $\gamma$-fibre orientations while keeping the size of the grains small and without compromising other material properties. However, the influence of these precipitates on texture is still a point of controversy and according to Shi and Liu [5] there is little effect of the Ti(C, N) particles on the orientation selection during recrystallization. On the other hand, there is strong experimental evidence that it is essentially the absence of interstitials, which is of
crucial importance for the development of the $\gamma$-fibre. Then, it was suggested that the development of beneficial texture components in IF steels is not due to the size and distribution of precipitates but rather to the removal of interstitial elements from the ferrite matrix [1, 10, 11].

The first material used in the study of failure micromechanisms is a Ti-stabilized IF steel sheet with a thickness of 0.7 mm. The microstructural characteristics of interest are its ductile ferrite matrix and the few and well separated micron-sized TiN particles. The chemical composition is displayed in Table 3.1.

Table 3.1: Chemical composition of the IF steel used in this work (in weight %). The composition was measured by atomic absorption spectroscopy (AAS) technique.

<table>
<thead>
<tr>
<th>IF steel</th>
<th>C</th>
<th>Mn</th>
<th>Ti</th>
<th>Nb</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>Si</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.0015</td>
<td>0.16</td>
<td>0.0545</td>
<td>&lt;0.001</td>
<td>0.0165</td>
<td>0.0045</td>
<td>0.033</td>
<td>0.0065</td>
<td>0.014</td>
</tr>
</tbody>
</table>

3.1.2 Dual Phase steel

The wide deployment of Dual Phase (DP) steels in automobile components has been highly motivated by their well-balanced combination of high strength and good formability in comparison to other HSLA steels [12]. In particular, the principal mechanical characteristics of these steels include continuous yielding behaviour (i.e. absence of yield point), a low ratio of yield to strength stresses, good bake hardening performance, high work-hardening rate and high values of uniform and total elongation [13].

The unique mechanical properties of the DP steels are mainly attributed to their microstructure which consists of hard martensite islands (or in some cases aligned in bands) which are embedded in a ductile ferrite matrix. Despite the generic name “dual phase”, additional low temperature transformation constituents like bainite, pearlite and/or retained austenite [13, 14] may also be present in these steels. The amount of each of these constituents depends among other factors on the heat treatment parameters and the chemical composition.

The chemical composition of DP steels is of major importance in determining the final microstructure and the ensuing mechanical properties. Carbon and manganese are the principal alloying elements, although additions of silicon, chromium, molybdenum and titanium may also be incorporated [15]. Carbon is a crucial element for the resultant mechanical properties due to the fact that this element controls the amount and properties of the martensite phase including its morphological features and ensuing
micro-mechanical behaviour. However, amounts larger than 0.2% C have a detrimental
effect on the spot weldability of the material [16].

The solid solution strengthening is improved by additions of silicon [17, 19],
phosphorous [17], chromium [18], aluminium [19], molybdenum [18] and mainly
manganese [20]. The silicon inhibits cementite formation at the ferrite-martensite
interfaces or in the bainite phase, which increases the toughness and ductility of the steel.
The inconvenience of Si is that this element promotes ferrite formation and therefore
increases the Ar$_1$ temperature allowing easier formation of bainite during cooling [16].
Moreover, the high Si content produces segregation of this element to the steel surface
where subsequently oxides are created. The product of this oxidation is typically the
fayalite ($\text{Fe}_2\text{SiO}_4$) islands which are known to cause problems for the galvanizing
process [21]. As a solution to decrease the galvanizing limitations, aluminium can be
used to partially substitute silicon albeit it is less effective in both suppressing carbide
formation and strengthening the ferrite phase [19]. Aluminium increases the Ar$_1$ and M$_s$
temperature, which can promote the formation of bainite instead of martensite [22]. In
contrast to silicon and aluminium, manganese stabilizes austenite and thus decreases the
Ar$_1$ temperature [16, 23] which allows the formation of martensite at lower cooling rates.
Nevertheless, Mn has the drawback that in combination with sulphur, it produces MnS
inclusions, which are detrimental for the formability of final products [24].

Vanadium, titanium and niobium interact strongly with boron, carbon and nitrogen
to form fine precipitates and, similar to the case of IF steels, reduce the amount of
interstitial elements from the ferrite matrix. Due to their high temperature stability, they
also promote the formation of a finer microstructure by pinning the movement of
austenite-ferrite phase boundaries at intercritical temperatures [14, 16]. In addition,
these precipitates contribute to the ferrite strengthening although this is accompanied by
a reduction of ductility [25, 26].

The DP microstructure is produced by heating the material to the two phase
(ferrite-austenite) intercritical region (between A$_1$ and A$_3$ temperatures), and after
appropriate isothermal soaking time, the material is quenched in order to transform the
intercritical austenite to martensite. The content of the strengthening phase is controlled
by the intercritical temperature and is usually between 5 to 20% [17, 27]. In the steel
industry, the annealing treatment of DP steels is applied either in batch annealing or
continuous annealing [15].

In the case of the batch annealing process, the material is heated up to the
intercritical temperature range, maintained during a long time period (~3 h) and finally
slowly cooled (~20°C/h) [13]. Due to the slow cooling rates, higher amounts of
austenite-stabilizing elements such as Mn are required in order to achieve the martensitic transformation as well as other elements (Si, Cr, Al) in order to achieve the desired strength. The use of these alloying elements increases the cost of the material and, in the case of manganese, segregation problems occur across the thickness of the steel [23]. A different situation occurs in the case of the continuous-annealing technique where both shorter periods of time (~2 min) in the intercritical temperature region and higher cooling rates (~10 °C/s) are employed. These differences have as main consequences the increase in the production rates and the fact that leaner alloys can be produced. Furthermore, the steel sheets exhibit a better uniformity, i.e. a reduction of scatter in properties in comparison to the material produced by batch annealing [13].

From a mechanical point of view, the main variation of the DP steel with respect to the IF steel is the much higher amount of second phase particles, which results in the interaction between the constituent entities during plastic deformation. The differences in the mechanical properties between the ferrite matrix and the constituents can be modified by the application of different heat treatments. For this reason, the second material analyzed is a DP600 steel grade which was received in as-cold rolled conditions with a thickness of 1.5 mm. The chemical composition of DP600 is given in Table 3.2.

Different intercritical annealing treatments were applied to the as-cold rolled sheets using a Continuous Annealing SIMulator (CASIM) at the Tata Steel R&D Laboratories of IJmuiden, Netherlands. The purpose of the heat treatments was to vary the microstructure of the steels with respect to a reference material (Ref) and afterwards correlate these modifications to the failure performance of the material. The modifications applied to the heat treatments are the reduction of the heating rate (SH), the increase of the intercritical temperature (HT) and the decrease of the cooling rate (SC). The parameters, the scheme of the heat treatments and the identification of the DP steels are listed in Table 3.3 and Figure 3.3. The cooling system of the CASIM equipment is controlled by air flow where the maximum applied cooling rate is set to 45°C/s.
Table 3.2: Chemical composition of the DP600 steel (in weight %).

<table>
<thead>
<tr>
<th>DP steel</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Al</th>
<th>P</th>
<th>S</th>
<th>Nb</th>
<th>Ti</th>
<th>V</th>
<th>Cr</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.092</td>
<td>1.68</td>
<td>0.241</td>
<td>0.036</td>
<td>0.016</td>
<td>0.004</td>
<td>0.002</td>
<td>0.002</td>
<td>0.007</td>
<td>0.576</td>
<td>0.022</td>
</tr>
</tbody>
</table>

Table 3.3: Values of the parameters employed for the intercritical annealing of the DP steels. The meaning of the heat treatment parameters are illustrated in Figure 3.3.

<table>
<thead>
<tr>
<th></th>
<th>Ref</th>
<th>SH</th>
<th>SC</th>
<th>HT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heating rate (Q_H), °C/s</td>
<td>20</td>
<td>2</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>Intercritical temperature (T_1), °C</td>
<td>780</td>
<td>780</td>
<td>780</td>
<td>820</td>
</tr>
<tr>
<td>Soaking time (t_1), s</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
</tr>
<tr>
<td>Cooling rate (Q_C), °C/s</td>
<td>45</td>
<td>45</td>
<td>2</td>
<td>45</td>
</tr>
</tbody>
</table>

3.1.3 AA6016 aluminium alloys

The 6XXX aluminium sheet alloys are introduced in the automotive industry as a potential solution for weight reduction of the vehicle’s body-in-white [28-30]. The principal requirements for these aluminium alloy series to be used in the automotive panels are good formability, adequate final strength including dent resistance, and high surface quality. Among the 6XXX series, the aluminium alloys more commonly employed are the AA6111 (North America) and the AA6016 (Europe) [28].

The 6XXX series belong to the group of heat-treatable wrought aluminium alloys, which means that their final strength is mainly achieved by precipitation strengthening mechanisms. The principal alloying elements of this aluminium alloy series are magnesium and silicon which form coherent or semi-coherent Mg-Si precipitates. For this reason the 6XXX series are also known as Al-Mg-Si alloys. The precipitation process is believed to occur in five stages [31], as described in (3.1). In the first stage, atomic clusters of Mg and Si are formed from the aluminium supersaturated solid solution regions (Al SSS) and they act as precursors for the development of the Guinier-Preston (GP) zones. With the progress of the aging process, the GP zones give sequentially place to the primary (β′′) and the secondary (β′ and β) strengthening precipitates.

\[
\text{Al SSS} \rightarrow \text{Mg/Si clusters} \rightarrow \text{GP zones} \rightarrow \beta'' \rightarrow \beta' \rightarrow \beta
\]  

In addition to Mg and Si, other elements such as Cu, Mn and Fe are usually present. The addition of copper increases the strength and ductility of the AlMgSi alloys [32] although it also increases the susceptibility to intergranular corrosion [33]. It has been reported that the addition of manganese improves the failure resistance by reducing the slip band spacing [34]. In contrast to Mn and Cu, iron has in general a detrimental effect
on the formability of the AA6XXX aluminium alloys [35]. As Fe has low solubility in aluminium, most of the Fe is bound with Si, Al and Mn to create Fe-containing intermetallics. These particles are formed during solidification and are known as constituents, since they almost do not dissolve during the thermo-mechanical processing. The size of the constituents typically ranges from 1 to 10 μm and the most common types are the plate-like β-AlFeSi phase and the rounded α-Al(FeMn)Si phase [36].

As already mentioned, the formability is limited by the plastic anisotropy, which in the case of the AA6XXX aluminium alloys, is largely controlled by the crystallographic texture [28]. Figure 3.4 displays the typical texture of a recrystallized AA6XXX alloy together with the location of the principal orientations, indicated by arrows. After recrystallization, aluminium develops a characteristic cube texture, of which the intensity depends on the amount of previous deformation among other factors such as annealing temperature or second phase particle size [8]. It is known that the particle stimulation nucleation (PSN) mechanism results in the randomization of the texture [37] which improves the formability. Dao and Lie [38] conducted crystal plasticity FEM simulations where they showed that random texture produce lower strain localization during bending of aluminium. A weak cube texture is also considered to improve the formability while a strong Goss texture (cf. Figure 3.4a) is known to be detrimental [28]. In addition to the effect on the formability, the surface quality is also affected by the texture. This is the case of roping, a surface defect manifested as paint-brush lines oriented along the rolling direction. Bennett et al. [39] experimentally confirmed that the occurrence of roping phenomena is attributed to banded arrangements along the rolling direction of grains with cube orientation.

The texture and the precipitation hardening of the AA6xxx aluminium alloys are mainly controlled through the thermo-mechanical processing route. The process sequence is schematically shown in Figure 3.4b. The alloys are first DC-casted (i) as large ingots whose surfaces are afterwards scalped in order to remove surface blemishes. Before hot rolling, the ingots are preheated (ii) to increase the plasticity and to reduce the short-range intercellular segregation. The reheating also causes dissolution of soluble phases in the Al matrix. The hot ingots are rolled typically in a reversible breakdown mill (iii) and later in a high-speed multi stand tandem mill (iv). The resulting strips (thickness between 3 and 6 mm) are coiled and cooled to room temperature before the cold rolling (v) to their final gauge between 0.8 and 1.2 mm. The last step is the continuous annealing (vi) of the aluminium sheets where the intention is the achievement of the adequate microstructural state of the matrix, which should keep it
suitable for transportation, with good formability and potential for age hardening in the manufacturing process [28].

![Figure 3.4: a) Recrystallization texture of a thermo-mechanically treated AA6xxx aluminum alloy [28]. The principal recrystallization texture components are the Cube {001}<100>, the Goss {011}<100>, the R {124}<211> and the P{011}<122> orientations. b) Schematic representation of the typical steps of thermo-mechanical processing of AA6xxx aluminum sheet alloys [28].]

The improvement of the texture obtained by conventional processes has been explored using alternative methods. This is the case of asymmetrical rolling, where Sidor et al. [40] have found a significant improvement on the texture of an AA6016 aluminium alloy in comparison to the conventional cold rolling process. The improved texture exhibits a more random character and avoids the detrimental cube recrystallization component, eventually producing an average Lankford value $\bar{r} \approx 0.9$, exceeding the value of $\bar{r} \approx 0.6$ of conventionally rolled aluminium grades [41].

In summary, the AA6XXX aluminium alloys could be considered as a composite material with micron-sized particles (constituents) embedded in an aluminium matrix of which the strength is controlled by nano-sized precipitates. In the present study, two AA6016 aluminium alloys were studied. The principal difference among them is the magnesium content, as can be seen in Table 3.4. Both alloys were received as sheets of
1 mm thickness, in the case of the 4Mg type, and 0.9 mm for the 6Mg type. The analysis and tests performed on both alloys were performed on the overaged conditions.

Table 3.4: Chemical compositions of the AA6016 aluminium alloys (in weight %).

<table>
<thead>
<tr>
<th>Type</th>
<th>Mg</th>
<th>Si</th>
<th>Cu</th>
<th>Mn</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>4Mg</td>
<td>0.4</td>
<td>1</td>
<td>0.2</td>
<td>0.1</td>
<td>0.22</td>
</tr>
<tr>
<td>6Mg</td>
<td>0.6</td>
<td>1</td>
<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
</tr>
</tbody>
</table>

3.2 Tensile test

The assessment of the mechanical properties as well as the analysis of deformation and failure evolution in this study has been made based on uniaxial tensile tests. This type of test is considered by industry as a basic method to determine the stress-strain behaviour, also referred to as the flow curve of the material, and some sheet metal forming characteristics; e.g. the $R$ value. The strong points of the tensile test are its simplicity, its well-defined boundary conditions, i.e. the uniaxial stress in the sample gauge, and the absence of friction between the tool and the region of interest of the sample.

Figure 3.5: Schematic tensile stress-strain curve representing the three stages during tensile test.
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The flow curve of a metal obtained from the tensile test can be separated in three different stages (cf. Figure 3.5): i) elastic deformation, ii) uniform plastic deformation and iii) non-uniform plastic deformation (necking). The yield strength marks the boundary between the elastic and plastic deformations while the limit between uniform deformation and the onset of necking is attained when the macroscopic strain hardening rate along the tensile direction $\Theta_x$ becomes equal to the flow stress $\sigma_x$, i.e.

$$\Theta_x \equiv \frac{d\sigma_x}{d\varepsilon_x} = \sigma_x$$

Equation (3.2) is known as Conisidère’s criterion [42].

The sample geometry and sample dimensions used in this study are based on the ASTM E-8M standard [43] (Figure 3.6). Standard and subsize specimens were used for the tensile tests, depending on the available material. The standard geometry was used in the case of the IF steel, whereas the subsize geometry was preferred for the DP steel and the AA6016 aluminium alloys. All samples were machined by electro-discharge equipment in sets of three different directions ($0^\circ$, $45^\circ$ and $90^\circ$) of the tensile axis with respect to the rolling direction (RD) of the sheet. For DP steels, only the direction with the tensile axis parallel to the RD was tested. The tests were performed at room temperature using an Instron tensile test equipment with a load capacity of 2 tons. A minimum of four samples were tested for each condition for all materials; i.e. samples at different directions of the tensile axis or different heat treatments.

![Figure 3.6: Geometry of the tensile samples together with the dimensions of the standard and subsize specimens.](image)

The ductility of the different materials was evaluated by parameters directly acquired from the tensile tests as well as further post-processing, especially those parameters related to post-necking deformations and final failure.
As none of the materials displayed a sharp yield point, the offset method was employed [43]. The yield strength $S_{0.2}$, i.e. the stress required to produce a small specified amount of 0.2% plastic deformation was determined, as given by equation (3.3)

$$S_{0.2} = \frac{P_{\text{offset } 0.2\%}}{A_0}$$

where $A_0$ is the initial cross-section area calculated from the product of the initial thickness, $t_0$, and width, $w_0$.

The ultimate tensile strength, $UTS$, was calculated using the maximum load value, $P_{\text{max}}$, of the force-displacement curve divided by the initial cross-section area, $A_0$, of the sample.

$$UTS = \frac{P_{\text{max}}}{A_0}$$

The reduction of area at fracture, $RA$, was calculated using the following formula

$$RA = \frac{A_0 - A_f}{A_0} \cdot 100\%$$

where $A_f$ is the cross-section area at fracture of the sample which was calculated by measuring the fracture surface area with an image analysis software. Based on these parameters, the true fracture strain and stress was also calculated by applying equations (3.6) and (3.7)

$$\varepsilon_F = \ln \frac{A_0}{A_f}$$

$$\sigma_F = \frac{P_f}{A_f}$$

The load at fracture, $P_f$, is the last load value registered from the tensile test.

One of the parameters used to characterize the formability of sheet materials is the $R$ value. The $R$ value, or also known as Lankford coefficient [44], particularly measures the resistance of the material to thinning during plastic deformation. This parameter is usually calculated from tensile tests using the width strain ($\varepsilon_w$) and the thickness strain ($\varepsilon_t$) of the specimen and applying the following equation.

$$R = \frac{\varepsilon_w}{\varepsilon_t} = \frac{\ln(w_0/w_f)}{\ln(t_0/t_f)}$$

The normal anisotropy of the sheet materials, i.e. the difference of the plastic deformation along the in-plane and through-thickness directions, is evaluated from the $R$ value by measuring this parameter from samples orientated at different directions within
the sheet plane. For instance, customary practice is to test tensile specimens with the axis aligned along 0, 45 and 90° with respect to the rolling direction. Based on these values, the average $R$ value is determined by equation (3.9)

$$
\bar{R} = \frac{R_{0\circ} + 2R_{45\circ} + R_{90\circ}}{4} \quad (3.9)
$$

The average $R$ value is used to characterize the resistance to thinning while the parameter $\Delta R$ (equation (3.10)) serves as an indication of the in-plane anisotropy.

$$
\Delta R = \frac{R_{0\circ} - 2R_{45\circ} + R_{90\circ}}{2} \quad (3.10)
$$

The methodology to calculate the strain hardening rate $\Theta_x$ consisted in first fitting a spline curve to the true stress-strain data and secondly to calculate the first derivative from the fitting curve. This methodology was applied in order to reduce the noise of the measurements on the calculation of the derivative.

![Figure 3.7: Scheme depicting the methodology to calculate the local stress-strain information from a necked sample. The macrographs display two examples of the acquired measurements where the thickness ($t_1$, $t_2$) and width ($w_1$, $w_2$) of the sample are correlated to the longitudinal distance ($l_1$, $l_2$) measured from the minimum cross section area.](image)

One of the main drawbacks of the tensile test is the plastic strain localization occurring during necking, which restricts the calculation of the different local strains achieved along the samples. In the case of DP steels, an attempt to extract local stress strain data was performed on broken samples from tensile tests and tensile samples deformed up to one step previous to failure, which are referred to as necked samples. Macrographs of the samples profile were made on the RD-TD and RD-ND planes (cf. Figure 3.7) and processed by image analysis software to obtain the exact geometry of the profile. Finally the data from both sections were merged and used to obtain the local stress-strain information along the tensile direction. The main assumption of this
Chapter 3

3.3 Quantitative Metallography and Vickers microhardness

The metallographic examination of the microstructure of all materials in their initial conditions and after deformation was made by optical microscopy (OM) and scanning electron microscopy (SEM). In the case of SEM, two different equipments were used; the FEI XL30 environmental scanning electron microscope with a LaB₆ filament equipped with an EDAX system for energy dispersive X-ray spectroscopy (EDS) and a FEI NOVA600 Dual Beam scanning electron microscopy equipped with a field-emission gun and a Focussed Ion Beam.

The polishing procedures applied for all samples consisted of progressive grinding followed by mechanical polishing from 9 up to 1 µm using diamond suspension products. The last step of polishing was usually performed with OPS in order to remove the deformation layer produced from the previous polishing step. OPS (Oxide Polishing Suspensions) consists of silicon dioxide (SiO₂) particles with an average size of 0.05 µm suspended as a colloidal aqueous liquid with a pH of 9.8. The alkaline medium together with the small particles of the suspension produces a chemo-mechanical action during this final polishing step, which in combination with long polishing times and reduced contact force is an effective method to remove the deformation layer from the previous polishing steps.

The error from all measurements of the quantitative metallography was evaluated with the standard deviation. Furthermore, the relative accuracy (%RA) was calculated for all data sets and this parameter was kept below 5% by increasing the number of measurements when necessary. The relative confidence is calculated from the confidence interval (CI) by equation (3.11)

\[
\%RA = \frac{CI_{95\%}}{\bar{x}} \cdot 100\% \tag{3.11}
\]

In the previous equation, \(\bar{x}\) represents the arithmetic average from the dataset and the confidence interval with a significance level of 95% \((CI_{95\%})\) (assuming a normal distribution of the data) is given by

\[
CI_{95\%} = 1.95 \frac{\sigma}{\sqrt{n}} \tag{3.12}
\]

where \(\sigma\) is the standard deviation and \(n\) is the number of elements from the dataset.

The characterization of the initial microstructure was based on the grain size of the matrix and features of the particle or constituent populations.
3.3.1 **Grain size determination**

The assessment of the size and geometry of the matrix grains was made following the Heyn linear intercept method [45] applied along the principal directions of the material (RD, TD, ND). The grain boundaries of the different specimens of DP steels were revealed by etching with Nital 2% whereas the grain boundaries of the IF steel, were better etched with the Marshal reagent [46]. The grain boundaries of the aluminium matrix of the AA6016 aluminium alloys were revealed with the Dix-Keller reagent [47].

3.3.2 **Particle or constituent characterization**

The chemical composition and statistical information of the particles population in the IF steel prior to deformation were obtained by EDS and OM, respectively. In the first case, the EDS spectra were acquired at a working distance of 10 mm, an acceleration voltage of 15 kV and a beam current corresponding to ~1500 counts, yielding an EDS life time of 100 s. The statistical information of the particles was based on 450 optical micrographs at 500 times magnification. The particle-type fraction, the number of particles per area, the area fraction, the average edge-length and its standard deviation, the percentage of fractured particles and the percentage of particles in the grain boundaries were calculated from these images. The edge-length of the cubic TiN particles was calculated as the square root of the product between the shortest and longest visible length of the particles.

The void initiation mechanisms caused by the TiN particles were investigated by OM after different tensile elongations imposed by tensile tests. In this case, the fraction of voids initiated by particle fragmentation or particle-matrix debonding was quantified together with the edge-length of the particle and the area of the voids. A particle was considered to be fragmented if a void was located between two parts of the TiN particle. Conversely, two particles close to each other but without a void in-between are considered as different particles. At least 100 particles were analyzed for each deformation state using a magnification of 1000X.

The identification of the different constituents formed in DP steels as well as their quantification was made by OM, SEM and EBSD. The volume fraction of the phases was measured by image analysis from optical micrographs of the samples etched with LePera etching [48].

A similar methodology was used for characterization of the particles in the aluminium alloys. EDS was employed to determine the chemical composition of the constituent particles whereas the statistical information of the particle population was
obtained from SEM micrographs in backscatter mode in order to enhance the contrast between the constituents and the matrix.

Figure 3.8: (a) SEM micrograph of the AA6016 aluminium alloy type 4Mg using the backscatter detector. (b) Gray value distribution of the pixels from the SEM micrograph. Note the sharp contrast between the aluminium matrix (higher peak on the left of the plot) and the constituent particles (lower peak on the right of the plot).

Sets of 50 micrographs were acquired for each aluminium alloy using an acceleration voltage of 15 kV and a magnification of 500X on the normal plane of the sheets, i.e. the plane containing the RD and TD. Figure 3.8 shows an example of such micrograph of one of the aluminium alloys together with its grey value distribution. Further post-processing was made using image analysis software and employing a minimum size threshold of the particles equal to 1 $\mu$m$^2$.

3.3.3 Void Mapping

In this approach, an area of approximately 1.5 x 1.5 mm$^2$ from the broken and necked samples was assembled from contiguous SEM micrographs made at a magnification of 2000X with high resolution FEG-SEM. The magnification used has a pixel size of 0.1667 x 0.1667 $\mu$m$^2$ which gives a better resolution than from optical microscopy. The microscope used for the reconstruction of void maps was the FEG SEM previously described. The collection of contiguous micrographs was made possible by employing the stage movement control of the microscope, which allowed taking micrographs at predetermined distances from each other. The stage control of the microscope has a resolution in the order of hundreds of nanometre. The images were afterwards merged and post-processed in order to quantify the voids with a common reference system. A size criterion was used where voids smaller than 0.555 $\mu$m$^2$ (20
pixels) were not included in the data. Finally, the processing and visualization of the data was made using Matlab® software.

3.3.4 Vickers Microhardness

The Vickers microhardness of all materials was measured in order to obtain the initial hardness of the material as part of their characterization and comparison. The measurements were performed using the Buehler automatic microhardness testing system OmniMent. The applied load was different between the materials but similar among the specimens of each material in order to make the results comparable in the groups. At least 20 measurements were made for each material.

3.4 Dilatometry

The study of the formation of the different constituent phases in the various grades of DP steels was carried out with dilatometry. This technique allows detecting the different phase transformations occurring during a heat treatment based on the volumetric change of the samples. Therefore, the identification of the microstructural constituents in DP steels is based on the temperature at which the transformation starts. The dilatometry analysis was made in a Bähr 805 dilatometer on specimens with a rectangular cross section with 10 mm length, 5 mm width and the thickness of the sheets with the long axis parallel to the rolling direction of the sheet.

3.5 Electron Probe Micro Analysis

The distribution of the alloying elements (carbon, silicon, chromium and manganese) in the constituents of the DP steel was studied by the electron probe microanalysis. The measurements were performed with a JEOL JXA 8900R microprobe using an electron beam with energy of 10 keV and a beam current of 100 nA, employing Wavelength Dispersive Spectrometry (WDS). The composition at each analysis location of the sample was determined using the X-ray intensities of the constituent elements after background correction relative to the corresponding intensities of reference materials. The points of analysis were located along a line with increments of 0.5 μm and involved the elements C, Si, Cr, Mn and Fe. The energy of the spectral lines, detection limit and counting error are given in Table 3.5.

Data reduction was performed for the C signal. The background measured on Fe at the C-Kα spectrometer position was subtracted from the measured signal on the sample and the obtained intensity was used to correct the carbon content.
Table 3.5: EPMA parameters used for the chemical distribution within the microstructure of the DP steels.

<table>
<thead>
<tr>
<th>Element, X-ray line</th>
<th>Energy, keV</th>
<th>Detection limit, ppm</th>
<th>Counting error, wt %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon, C Kα</td>
<td>0.282</td>
<td>800</td>
<td>0.01</td>
</tr>
<tr>
<td>Silicon, Si Kα</td>
<td>1.74</td>
<td>20</td>
<td>0.1</td>
</tr>
<tr>
<td>Chromium, Cr Kα</td>
<td>5.414</td>
<td>170</td>
<td>0.06</td>
</tr>
<tr>
<td>Manganese, Mn Kα</td>
<td>5.898</td>
<td>180</td>
<td>0.18</td>
</tr>
<tr>
<td>Iron, Fe Kα</td>
<td>6.403</td>
<td>210</td>
<td>0.6</td>
</tr>
</tbody>
</table>

3.6 X-ray diffraction

The bulk texture of the IF steel and the AA6016 aluminium alloys prior to deformation was measured by X-ray diffraction (XRD) on a Siemens D5000® diffractometer with Mo Kα radiation (\(\lambda=0.070926\) nm) generated in a tube operated at 50kV and 50 mA. The orientation distribution function (ODF) was calculated from four incomplete pole figures (cf. Table 3.6) using the MTM-FHM software developed by Van Houtte [49]. The program calculates the ODF using the series expansion method. The maximum rank of the spherical harmonics used was \(L_{\text{Max}}=22\).

Table 3.6: Pole figures used for texture calculation together with the weight assigned.

<table>
<thead>
<tr>
<th>(\alpha)-Iron</th>
<th>{hkl}</th>
<th>{110}</th>
<th>{100}</th>
<th>{112}</th>
<th>{310}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight</td>
<td>1</td>
<td>1</td>
<td>0.5</td>
<td>0.5</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Aluminium</th>
<th>{hkl}</th>
<th>{111}</th>
<th>{100}</th>
<th>{110}</th>
<th>{113}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight</td>
<td>1</td>
<td>1</td>
<td>0.5</td>
<td>0.5</td>
<td></td>
</tr>
</tbody>
</table>

3.7 Crystal orientation mapping by Electron Backscatter Diffraction

As already mentioned in Chapter 2, the strain incompatibilities created during plastic deformation of metals are crucial for the ductile failure micromechanisms. The analysis and characterization of the local deformation occurring within the microstructure is possible using the crystal orientation maps by EBSD.

The term electron backscatter diffraction (EBSD) originally refers to the pattern formed from specific interactions among the electron beam of a scanning electron microscope and the incident region of a sample with crystal structure. This pattern is known as a backscatter Kikuchi pattern (BKP) or electron backscatter diffraction pattern (EBSP), and contains information of the crystal orientation of the diffracted volume [50]. Nowadays, EBSD frequently refers to the automated acquisition of local crystallographic orientations along a grid on the surface of a sample placed in a scanning
electron microscope. Other more suitable used names for this technique are orientation contrast microscopy, orientation imaging microscopy OIM [51], or automatic crystal orientation mapping ACOM [52]. The main characteristic of this technique is the coupling of the morphological aspects of the microstructure with its local crystal orientation, which is known as microtexture.

3.7.1 The physics of EBSD pattern formation

The EBSD technique is applied on a specimen tilted to 70° from the horizontal plane, which is hit by the incident electron beam with an accelerated voltage typically between 15 and 30 kV. The diffracted backscattered electrons are captured by a phosphorous screen where an electron backscatter pattern (EBSP) is formed. The acquired pattern is digitized and solved by identifying the directions of several Kikuchi bands in the pattern using a band detection algorithm. The Hough transform is usually employed by these algorithms.

Crystallographic orientation information is quantitatively obtained based on the geometry of the EBSD pattern. The mechanisms by which the diffraction patterns are formed have been reported by several researchers and a brief description is given below.

When the electron beam of the SEM hits the surface of the tilted sample, part of the electrons are incoherently spread beneath the surface by elastic interactions and scattered back out of the sample. These electrons are known as elastic backscattered electrons.

The trajectories of the backscattered electrons produce two cones on either side of the diffracting set of planes with an apex half-angle of 90° - \( \theta_B \) around the plane normal \(<hkl>\). These are known as Kossel cones. When the backscattered electrons are recorded on a phosphor-coated glass screen, the interception of a pair of Kossel cones with the screen is seen as a pair of hyperbolas, which approximate straight lines because \( \theta_B \) is relatively small (\( \sim 1.4° \)). The set of straight lines or bands formed on the phosphor screen are referred to as electron backscattered patterns (EBSP), also known as Kikuchi line patterns due to their resemblance to the diffraction patterns observed in transmission electron microscopy (TEM). The bands in the EBSP represent planes in the crystal where the widths of the bands is reversely proportional to the d-spacing of the diffracted crystallographic planes and the angles between bands in the patterns are directly related to the interplanar angles of the crystal lattice.

Basically, an EBSD system consists of two main elements: the EBSD acquisition hardware and the EBSD software. The EBSD acquisition hardware generally comprises a sensitive camera (CCD in the first generation systems, nowadays digital), and an
image processing system for pattern averaging and background subtraction while the function of the EBSD software is to control the data acquisition, solve the diffraction patterns and store the data.

3.7.2 **Capabilities of the system: angular and spatial resolution**

The angular resolution of the EBSD technique, i.e. the average angle deviation between the experimental and the real crystal orientation, is mainly limited by the pattern detected from the camera and the accuracy of computer analysis of the digitized patterns [53]. The range of values of such resolution lies usually between 0.5 and 1°. However, the resolution can be improved up to 0.1° by changing for example the system configuration by using less binning of the Charge Coupled Device (CCD) camera during patterning and smaller bin sizes in the Hough transform accumulator array. However, these actions lead to a decrease of the data acquisition speed. Other proposed methods are based on post-processing data averaging [54] or on the detection of the relative shift between features of two EBSD patterns for the case of misorientation angles [55].

Although an angular resolution of 1° is acceptable for some analyses, such as grain size or ODF calculation, this relatively low resolution can enormously affect other parameters such as in the case of the misorientation axis calculation at low misorientation angles. Bate et al. [55] demonstrated that the expected angular error \( \phi \) of the misorientation axis is a function of the orientation measurement error \( \delta \) (the angular resolution in this case) and the calculated misorientation angle \( \omega \), as depicted in equation (3.13)

\[
\phi = \tan^{-1} \left( \frac{\sin \frac{\delta}{2}}{\sin \frac{\omega}{2}} \right) \approx \tan^{-1} \frac{\delta}{\omega} 
\]

(3.13)

Therefore, the errors of misorientation axis calculation increase as misorientation angles decrease in such a way that the highest possible error angle is given by equation (3.13). For example, the calculated misorientation of 1° in conditions of 1° of angular resolution may bring an error as high as 45° in the misorientation axis.

Another important aspect of the EBSD technique is the spatial resolution. The spatial resolution in EBSD is divided in lateral and depth spatial resolution. The minimum step size possible in an orientation map is dictated by the lateral spatial resolution. The definition of the EBSD lateral resolution can be formulated following two different definitions, the physical and the effective lateral spatial resolution [56]. In the first case, the physical spatial resolution is related to the electron interaction volume
whereas the effective spatial resolution indicates how accurately an orientation can be resolved when a second orientation is partially within the electron interaction volume of the first data pixel. Due to the geometrical settings of the sample tilted to 70°, the lateral resolution differs between the directions parallel and perpendicular to the sample tilt axis. Zaefferer [56] found that the resolution parallel to the sample tilt axis for iron is $35 \pm 5$ nm while the resolution perpendicular to the sample tilt axis is $90 \pm 15$ nm, almost a factor of three larger.

The depth spatial resolution is related to the distance below the surface from where the backscattered electrons originate. The depth spatial resolution is more difficult to determine from experiments, although the values are lower than the lateral spatial resolution. Zaefferer [56] defined a parameter to characterize the depth spatial resolution based on the degradation of the EBSP quality when an amorphous layer is deposited at the surface of the material with a known thickness. In the case of iron, the quality is degraded 50% with an amorphous layer of 2 nm.

The principal factors determining the spatial resolution are the atomic number of the material, the accelerating voltage of the electron beam, the sample tilt angle and the software used for pattern analysis [56]. Modifications of these parameters could yield to an improved spatial resolution, although other parameters are also involved, such as the exposure time. One example on the improvements of the spatial resolution is the experiments by Steinmetz and Zaefferer [57] where the (physical) lateral spatial resolution was reduced from 30 nm to 10 nm by decreasing the acceleration voltage from 15 kV to 7.5 kV in a TWIP steel sample.

3.7.3 FIB-EBSD: an extension of EBSD to 3D

The recent synergy of the EBSD technique with the milling capabilities of a focussed ion beam (FIB) has enabled the reconstruction of the three-dimensional morphology of microstructures coupled together with the local crystal orientation. This new technique has been referred in literature as FIB-EBSD [58-60], 3D-EBSD [61-63] or FIB-OIM [64]. The acquisition and reconstruction of the materials microstructure on three dimensions is not a recent issue and has been accomplished by techniques like serial sectioning [65, 66] or 3D X-ray diffraction microscopy [67]. The innovative aspects and advantages that FIB-EBSD offers above the other 3D reconstruction techniques is mainly in the microtexture-morphology coupling, the submicron spatial resolution, the full automation of the acquisition process and the easier accessibility to the equipment in comparison to synchrotron-based techniques.
The FIB-EBSD follows a serial sectioning procedure, and as a consequence, has a destructive and ex-situ character. The methodology of the technique mainly consists in the removal of successive thin slices of material by FIB milling (milling configuration, cf. Figure 3.9a) and the acquisition of the microtexture data by the EBSD technique for each newly revealed surface (acquisition configuration, cf. Figure 3.9b). As the milling and the orientation mapping are performed under different geometrical configurations, a positioning procedure is needed to carry the sample from one setting to the other. This movement is achieved by tilting or rotating the sample holder; depending on the configuration of the beams within the microscope chamber [63]. In the case of a rotation setup (cf. Figure 3.9), the sample is positioned in such a manner that a rotation of 180° around the axis of a pre-tilted sample holder takes the sample from the milling to the acquisition stage and vice versa. If the observation area is not located on the rotation centre, then additional translation movements of the stage are required to place the region of interest in the milling or acquisition position. Following the stage movement, a final accurate positioning of the observation area is executed based on an iterative process of image recognition and alignment of two fiducial marks. Therefore, the accuracy of the sample alignment is not determined by the accuracy of the stage positioning but rather by the resolution of the SEM-image used for the iterative process.

As previously mentioned, the milling is performed using a Focussed Ion Beam. The removal of the material is made by collisions of accelerated heavy ions, usually Ga⁺, operating at grazing incidence. Liquid gallium is constantly supplied to a tungsten needle which has a strong electric field at the end of the tip. Then, the Ga⁺ ions are accelerated in the electric field towards the sample in order to remove thin slices of
material by sputtering. In the case of the FIB-EBSD, different geometries have been tested for milling. In the settings used for this equipment, a trapezoid is milled on the edge of the sample and this geometry is kept during the entire scanning process. The side walls of the trapezoid are milled at an angle higher than 60º in order to avoid shadowing effects during EBSD collection. The depth of the milling is limited by the curtaining effect produced by the channelling of the ion beam. The accuracy of cutting is established by the FIB image resolution and the accuracy of the automatic stage alignment via beam shift.

The spatial resolution of the FIB-EBSD is controlled mainly by three factors: the resolution of the EBSD, the resolution of the FIB-milling and finally the resolution of the positioning. The EBSD resolution was already discussed in the previous section. The FIB microscope is able to focus the ion beam to spot sizes smaller than 20 nm and control the position of the beam with an accuracy of nanometres, which together with the resolution of the EBSD system allows obtaining voxels with a dimension of 20x20x20 nm³. However, due to alignments problems a more realistic resolution has been set to 100x100x100 nm³ [63]. Another aspect linked to the spatial resolution which also limits the range of applications of this technique is the size of the investigated volume. Zaefferer et al. [63] mentioned that the largest investigated volume is on the order of 50x50x50 μm³ due to the stability time of the FIB. Therefore, this volume nowadays restricts the 3D reconstruction to fine grain metals or to local details of the microstructure.

Due to the lack of specialized computational software in this field, one common drawback of all 3D imaging techniques is the tediousness of post-processing and visualization of the obtained data, which limits the capabilities to study the microstructure topology together with their related crystallographic orientations.

3.7.4 Settings of the EBSD measurements

The system used for 3D-EBSD reconstruction is a Nova 600 Dual Beam FIB-SEM microscope which is equipped with an EBSD system. The SEM works with a Field Emission Gun (FEG) and electron optics that provide excellent spatial resolution. Both the FIB-SEM and the EBSD systems are controlled by the Autoreveal® software (β-version first generation (G1)) developed by FEI on a platform of Oxford-HKL Channel 6® software for collection and analysis of the EBSD data. This system allows, after initial setting of the measurement characteristics, recording a 3D data set in a fully automated collection mode. Before starting the data acquisition, a rough milling with the FIB is applied with the intention to create a clear surface on the sample edge on which
the measurements will be carried out. Two fiducial marks were produced from a platinum deposition layer, which was later milled in order to create the geometry shown in Figure 3.9c. The parameters used for the FIB milling and EBSD data collection are displayed in Table 3.1 although the dimensions of the analyzed volumes differ from sample to sample. In the case of the IF steel, the FIB-ESBD was used for two purposes: (i) to reveal morphological features and position of the voids created by particle-matrix decohesion, and (ii) to analyse the orientation gradients created around the TiN particles.

Table 3.7: Parameters used for the FIB-EBSD analysis.

<table>
<thead>
<tr>
<th>FIB</th>
<th>EBSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acceleration voltage:</td>
<td>30 kV</td>
</tr>
<tr>
<td>Rough milling current:</td>
<td>5 nA</td>
</tr>
<tr>
<td>Slice milling current:</td>
<td>1 nA</td>
</tr>
<tr>
<td>Imaging current:</td>
<td>10 pA</td>
</tr>
<tr>
<td>Slice removal thickness:</td>
<td>0.1 µm</td>
</tr>
<tr>
<td>Acceleration voltage:</td>
<td>20 kV</td>
</tr>
<tr>
<td>Current:</td>
<td>2.4 nA</td>
</tr>
<tr>
<td>Binning:</td>
<td>4x4</td>
</tr>
<tr>
<td>Detect:</td>
<td>Band centres</td>
</tr>
<tr>
<td>Step size:</td>
<td>0.1 µm</td>
</tr>
</tbody>
</table>

With the above settings the time for measuring a volume of ~20x20x20µm³, in the case of steel samples, is ~100h which is an additional restricting parameter for using the 3D EBSD system.

The equipment employed for EBSD analysis is a FEI XL30 type environmental SEM with a LaB₆ filament equipped with an EDAX/TSL OIM system. The microscope was operated at 25 kV. The crystal orientation maps were acquired in most of the cases using a step size of 100 nm. In the case of higher resolution EBSD scans, i.e. with step sizes from 50 nm to 100 nm, the Dual Beam FIB-SEM employed for the 3D EBSD was also used for 2D orientation mapping. The acceleration voltage and intensity current were the same as the ones employed for 3D-EBSD. All scans were made following a square grid.

The preparation of the samples was made similar as for quantitative metallography, i.e. grinding followed by mechanical polishing. The final polishing step was only with colloidal silica suspension (OPS) for times varying from 20 to 40 min with very low pressure.

As opposed to the IF steel and the aluminium alloys, the bulk texture of the DP steels was obtained from EBSD measurements. By applying EBSD for regular texture measurements in the case of DP steels, the orientation data from ferrite grains could be isolated, whilst excluding information from bainite or martensite. This separation was made by selecting the orientations of grains above a certain size. The orientation maps
contain more than 1000 grains and therefore an area of 700x700 μm² with a step size of 1 μm was scanned.

References
<table>
<thead>
<tr>
<th>Chapter 3</th>
</tr>
</thead>
</table>


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Materials and Experimental Tools


Chapter 4

Void initiation mechanisms in IF steels

This chapter is dedicated to the study of the micromechanisms inducing void formation during tensile deformation of an IF steel and the associated evolution of grain substructures. First a description of the initial microstructure is given, which includes the particles characterization followed by the mechanical properties acquired from tensile tests. Afterwards the failure micromechanisms were studied by observation of the fracture surface and examination of the cross-section of tensile samples. The grain substructures developed at different tensile deformation stages within the ferrite grains were analyzed and a special emphasis was given on the region surrounding the particles. This analysis was complemented with data from the novel FIB-EBSD technique. Although the IF steel has a relatively high purity, the primary void initiation preferentially occurs on the TiN particles embedded in the microstructure.

4.1 Introduction

The detrimental effect of inclusions on the mechanical properties of steels has led to the development of cleaner steels [1]. Nowadays, the inclusions in clean steels usually consist of a few micron-sized particles and a large amount of nano-size particles. This is the particular case of titanium-stabilized Interstitial Free (IF) steels where the large particles are mainly the cubic TiN micron-sized precipitates and the population of finer particles are titanium-based nano-precipitates. In addition, the TiN particles are also present in other steels, such as maraging or microalloyed steels, where they have a strong impact on the deformation and fracture properties.
Chapter 4

It is well documented that TiN particles play a role in the fracture mechanisms of steels by acting as void or crack nucleation sites. At temperatures below the ductile-brittle transition temperature, these particles behave as crack nucleation sites for brittle fracture [2-5]. In this case, the proposed micromechanism consists of (i) cleaving of the particle and, (ii) crack propagation into the ferrite matrix by cleavage as the inclusion-matrix interface is strongly bonded [3].

In the case of ductile failure, TiN particles have also been observed to act as preferential sites for void nucleation [6-8]. Cox and Low [7] and Shabrov et al. [8] experimentally found in maraging steels that void initiation, which occurs at low plastic strain, was produced by fragmentation of TiN inclusions. An important point of their study was the relevance of particle size. It was concluded that the size of the particle is the dominant factor for void nucleation following the rule that the stress required to fragment the TiN particle decreases with increasing particle size. With regard to the effect of stress triaxiality, no measurable difference was detected between the stresses generated on notched or smooth tensile samples [7]. However, numerical simulations about clustering of cubic particles [9] suggest that the void nucleation is more influenced by the particle size than by the spatial distribution at high stress triaxiality states. In addition, the presence of a secondary population of smaller particles, such as the TiC precipitates, may accelerate the ductile failure process by localizing the deformation. The hierarchical model simulations performed by McVeigh et al. [10] demonstrated that shear localization between TiN particles is worsened by the micro-void formation on the smaller TiC precipitates even if the stress triaxiality condition is zero. The particle morphology is another critical parameter, which was analyzed by Gänser et al. [11]. They studied the influence of the spatial orientation of a cubic particle on the void nucleation by FEM. The numerical analysis predicted a critical state for different damage parameters when the cube body diagonal was roughly parallel to the applied load. The best particle orientation was predicted to occur when the particle edge is aligned with the direction of the applied load.

In all previous cases, the development of effects produced by the local microstructure (crystallographic texture and creation of grain substructures) is not taken into account despite the high stress concentration and strain localization which can be generated at this scale. Humphreys [12] conducted dedicated transmission electron microscopy (TEM) observations in order to study the plastic relaxation mechanisms during deformation of non-deformable particles embedded in aluminium and copper matrices. It was concluded that the stresses generated are relaxed by crystal rotations in the surroundings of the particle. This region was referred to as the particle deformation.
Void initiation mechanisms in IF steels

However, there are no studies related to particle deformation zones on steels, and particularly, on the cube shaped TiN particles.

The present chapter is intended to analyze the void initiation mechanisms occurring in TiN particles during tensile deformation. This is done with a special emphasis on the relation between void initiation mechanisms and plastic deformation at the mesoscale. This relation is considered to be crucial on systems exhibiting strong interface cohesion between the rigid particles and the ductile matrix. To this purpose, crystal orientation mapping based on EBSD is used to characterize the plastic deformation produced around the TiN particles as well as the overall ferrite matrix. These experimental observations are made based on tensile test specimens of IF steel sheets elongated to various extensions. Later, these results are employed to a better understanding of the void initiation mechanisms in DP steels (cf. Chapter 5).

4.2 Initial microstructure

The ferrite matrix of the current IF steel consists of slightly pancaked and recrystallized grains in which the mean intercept lengths with respect to the rolling (RD), transverse (TD) and normal (ND) directions are 16.0, 14.3 and 10.7 μm, respectively. An inverse pole figure map made on the RD-TD plane of this steel is shown in Figure 4.1a. The crystallographic texture of the ferrite matrix is a strong \{111\}{(uvw)} texture (or γ-fibre texture) with a maximum intensity of around 18 times random in the vicinity of the \{111\}{(112)} texture component (cf. Figure 4.1b), which is typical for recrystallized low carbon steels of deep drawing quality [13].

Two main particle populations were found homogeneously distributed in the ferrite matrix and, based on EDS analysis, the inclusions were identified as titanium nitrides and aluminium oxides, Al₂O₃. The TiN particles exhibit a square or rectangular cross section whereas the aluminium oxide inclusions are with elliptical shape. In order to obtain statistically reliable quantitative data for the particle populations, a field of 1850 mm² was investigated by OM. In total 236 particles from both types were included in the measurement. The summarized characteristics of both particle populations are shown in Table 4.1. The titanium nitride particles are the most frequent with around 77% of the total number of particles. They have a characteristic amber colour when observed by optical microscope which makes them easily recognizable. The average edge-length of these inclusions is 2.99±1.1 μm while Figure 4.2a displays their size distribution. A total of 15.8% of these cubic inclusions were found to be fractured prior to deformation as shown in Figure 4.2b. All fractured particles are arranged in such a way that the fragments are aligned parallel to the rolling direction. This observation gives evidence to
the fact that these particles were fractured during the rolling process instead of being an artefact of sample preparation.

![Figure 4.1](image)

**Figure 4.1:** (a) IPF map of the IF steel recorded with a step size of 0.5 μm. The ND is used as a reference for the IPF. (b) ODF contour plot of the IF steel in a φ2 = 45° section of the Euler Space. Units are in times random.

| Table 4.1: Quantitative TiN particles characterization by optical microscopy. |
|-----------------|----------------|--|
|                  | TiN            | Al₂O₃ |
| Particle type fraction, % | 77            | 23    |
| Number of particles per area, inclusions/μm² | 12 × 10⁻⁶ | 3.5 × 10⁻⁶ |
| Particles area fraction, %     | 0.012 | 0.012 |
| Average edge-length, μm         | 2.99 ± 1.1 | 5.1 ± 3 |
| Fraction of fractured particles, % | 15.8    | 0     |
| Fraction of particles lying on grain boundaries, % | 68       | 87     |
| Minimum neighbour distance, μm | 101      | 303    |

Another specific feature of some of the cubic particles is a dark zone located in the core when observed by optical microscopy; e.g. the TiN particle of Figure 4.2c. The origin of this black core is supposed to be related to the nucleation of the TiN particles on other inclusions with different chemical composition during the liquid state, as was proposed by Fairchild et al. [5].
The Al₂O₃ inclusions rather exhibit an ellipsoidal shape and they have a larger equivalent diameter \( d_{eq.} = 5.1 \, \mu m \) than the TiN precipitates. In contrast to the TiN particles, no Al₂O₃ inclusions were found fragmented in the as-produced conditions (before tensile tests). Although the effect of this particle population on the fracture process is not neglected, the present study is limited only to Ti-based particles. The justification of this choice is based on the strong bonding forces between the TiN particles and the ferrite matrix which are similar to the martensite-ferrite interface cohesion in the DP steels.

### 4.3 Mechanical properties and macroscopic failure mechanisms

The engineering stress-strain curve obtained from the tensile tests is shown in Figure 4.3 while the tensile properties and microhardness are summarized in Table 4.2. The discontinuous jump in the curve occurring at \( \sim 6\% \) is due to the sudden change of the cross-head speed of the tensile equipment from 2.7 to 27 mm/min as it is recommended by the standard ASTM E-8M [14]. In general, the IF steel displays a relatively large ductility accompanied by a good drawability. The last property is based on the high average \( R \) value and its low in-plane variation \( \Delta R \).

---

**Figure 4.2:** (a) Edge-length distribution of the TiN particle population. (b) SEM micrograph of a fractured TiN particle prior to deformation and (c) a TiN particle observed by optical microscopy.
During the tensile tests, the area within the gage length of the sample deforms uniformly up to $e=\sim26\%$, after which, the reduction of the thickness and width becomes more discernible in the central zone of the sample. Such localization of the deformation is usually referred to as diffuse necking [15]. The diffuse necking continues up to the appearance of a narrow band(s) (*macroband*) located in the centre of the sample at approximately $e=38\%$ which is oriented at approximately $54^\circ$ with respect to the tensile axis direction (localized necking). In most of the cases, the localized necking is composed of two macrobands, mirroring each other, which intersect one another near the middle width. The final fracture of the sample occurs by thinning and later failure at one of the macrobands at $e=46\%$.

**Table 4.2: Mechanical properties of the IF steel (average value and the standard deviation). The Vickers microhardness is based on the average of 30 measurements using a load of 500 g.**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultimate Tensile Strength, MPa (<em>UTS</em>)</td>
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</tr>
<tr>
<td>Deformation at <em>UTS</em>, %</td>
<td>25.6 ± 0.4</td>
</tr>
<tr>
<td>Deformation at Fracture, %</td>
<td>46.2 ± 0.6</td>
</tr>
<tr>
<td>Vickers microhardness, HV$_{500}$</td>
<td>82 ± 2.3</td>
</tr>
<tr>
<td>$R$ value along RD (<em>$R_{0^\circ}$</em>)</td>
<td>2.63 ± 0.66</td>
</tr>
<tr>
<td>Average $R$ value ( $\bar{R}$ )</td>
<td>3.1</td>
</tr>
<tr>
<td>$\Delta R$</td>
<td>0.7</td>
</tr>
</tbody>
</table>

### 4.4 Failure micromechanisms

#### 4.4.1 Fractography

The fracture surface of the samples consists of a fibrous zone and a shear-slip zone as can be seen in Figure 4.4a. The fibrous zone is located in the centre of the fracture surface and it includes approximately 18% of the total fracture zone, which corresponds...
to approximately 73 μm of the sheet thickness. The surface of the fibrous zone contains two dimple populations of different size whereby the larger and deeper dimples are separated from each other and surrounded by smaller and shallower dimples. In the interior walls of the large dimples usually a stairs-like structure can be found which is associated to the slip traces of deformation and referred to as serpentine glide [16]. Some rare particles are observed in the interior of either the large or small dimples as can be seen in Figure 4.4b. From the particles located inside the dimples, EDS analysis showed that they were either TiN or Al₂O₃ with a predominance of the former particles by 70%. This observation confirms that both types of particles contribute to the fracture process; however, it is unknown and beyond the scope of this chapter whether the TiN particles or the Al₂O₃ inclusions play a leading role in the mechanisms of the IF steel failure. In the following sections, the focus is limited to the role of TiN particles in the void initiation mechanisms and the deformation process of the ferrite matrix.

4.4.2 Void initiation and growth at TiN particles

The examination on the cross-section of the tensile samples deformed at different strains allowed identifying two different micromechanisms by which voids were created in the vicinity of TiN particles. One of the micromechanisms observed is the fragmentation of the particle in two or more parts (cf. Figure 4.5a). In most of the cases,
the fracture plane of the TiN particle is roughly oriented perpendicular to the loading direction. The other type of void initiation detected in the TiN particles occurred by decohesion along the interphase between the ferrite and the particle. The interphase separation always occurs on the side of the particle which is nearest to the normal plane on the tensile axis and preferentially on one of the corners of the cuboidal particle, as can be observed on the micrographs of Figure 4.5a and b. It is important to mention that one void initiation mechanism does not exclude the other and thus both types of void initiation may take place in the same particle; e.g. the TiN particle in Figure 4.5a.

Figure 4.5: The OM and SEM micrographs shown in (a) and (b), respectively, are some examples of void initiation observed on the sample deformed to 40% elongation. Both mechanisms, fragmentation and debonding, may occur on the same particle as observed in (a). (c) Plot displaying the fraction features of TiN particles which have either been fragmented or debonded (solid lines) together with the generated void area associated with both mechanisms (dashed lines) at different tensile engineering strains.

The development of both void nucleation micromechanisms in the TiN particles through plastic straining is summarized in the graph of Figure 4.5c. The sequence of these mechanisms during tensile deformation can be divided into three stages. In the first stage, i.e. between 0% and 15% of deformation, the fraction of fragmented TiN particles displays an increasing trend. With regard to the fraction of debonded particles, this mechanism does not show a clear trend during the first stage. In the second stage, between \( e=15\% \) and \( e=35\% \), the fraction of broken particles remains almost constant
Void initiation mechanisms in IF steels

while the fraction of debonded particles increases with a factor of two. An increase of the debonded particle fraction is still observed between $e=25\%$ and $e=35\%$ although the rate of formation of debonded particles with respect to strain has decreased. In the third stage of deformation between $e=35\%$ and $e=40\%$, the fraction of both fragmented and debonded particles remains relatively stable.

As void initiation is a continuous process occurring during deformation rather than at a specific strain, the range of the void sizes is broad; in other words, while some voids are in the growth stage, others are just initiating. This fact is reflected in the scatter of the void area measurements for both initiation mechanisms in Figure 4.5c where the standard deviation (omitted in the graph) has comparable value to the average void area for all deformations. For instance, the average void area by particle fragmentation and its standard deviation at $e=40\%$ deformation are respectively $11.9 \, \mu m^2$ and $9 \, \mu m^2$. However, it is important to mention that the maximum void area is $32.2 \, \mu m^2$ for the same conditions while the corresponding minimum is $0.1 \, \mu m^2$. On the one hand, this value of the minimum void area is similar for all deformations and for both void initiation mechanisms, which provides evidence to the continuous creation of voids during all the stages of deformation and the different void growth rate for each void. On the other hand, the maximum void area is several times larger than the average value although both magnitudes have in common that they increase proportionally with the deformation.

In general, the growth direction of the voids is observed to be parallel to the tensile direction. In the first two stages of Figure 4.5c, the growth of the voids has a relatively slow rate for both particle fragmentation and decohesion. However, the situation drastically changes in the third stage, where the void growth of both mechanisms shows a noticeable increment in the rate of the average void area. The average void area values achieved at $e=40\%$ for the case of particle debonding and fragmentation are $4.8$ and $11.9 \, \mu m^2$, respectively. In addition, it should be mentioned that necking of the tensile specimens at large deformations represents another contribution to the large scatter on the void area measurements. As the plastic deformation starts localizing within the tensile sample, the local strain conditions are different for the nucleated voids depending on their position as well as the ensuing void growth. In other words, voids located within the localized necking area are expected to have grown more than the voids nucleated outside this region.

A noticeable fact is that the average void area produced by particle fragmentation is superior to the voids nucleated from debonding at all strain values. This relation is also valid for the respective maximum void area values. This difference between both
mechanisms is due to the size of the void initiation site; i.e. while the voids nucleated by particles fragmentation usually inherit the size of the particles as initial width (Figure 4.5a), in the case of debonding, the voids usually initiate on smaller interphase areas, such as the particle corners (Figure 4.5b).

Similar to the findings of Cox and Low [7] and Shabrov et al. [8], the average edge-length of the fragmented particles is larger with respect to the total particle population average and this is true for all deformations. The ratio of the fragmented edge-length to the population average edge-length is approximately $1.2 \pm 0.1$. This fact enhances the idea that larger particles are more prone to be fragmented. In the case of the particles with voids nucleated by decohesion, an opposite behaviour is observed, i.e. the edge length of these particles is almost equal or slightly inferior to the population average. The ratio of the edge length of the debonded particles with respect to the population average is approximately equal to $0.94 \pm 0.05$.

The influence of grain boundaries on void initiation was another factor analysed. In the initial conditions, 68% of the TiN particles are intercepted by ferrite grain boundaries (cf. Table 4.1). If only the TiN particles are considered at which voids are nucleated, then it is found that in average 66% of those particles in the deformed samples are located on grain boundaries and the rest in the interior of the ferrite grains. The percentages from the initial conditions and the deformed samples are similar. Particularly noteworthy is the fact that this similarity is still kept when considering separately the percentage of the TiN particles situated on grain boundaries which are separated from the matrix (68%) or fragmented (69%). These results suggest that there is no a noticeable preference for voids to initiate on TiN particles located in grain boundaries by any of the initiation mechanisms.

The examination on the cross-section of the interrupted tensile test samples gave an insight on the void initiation mechanisms at the TiN particles and their subsequent void growth in a statistical analysis. However, the characterization was made on a two-dimensional basis and some morphological aspects about the voids remain unknown. The following section is intended to clarify some of these issues based on the use of FIB-EBSD technique, which allows carrying out a metallographic characterization in 3D.

### 4.4.3 Particle-matrix decohesion at TiN particles by FIB-EBSD

In order to reveal the morphology of the voids and their location over the surface of the TiN particle, some cases of particle-matrix decohesion were analysed by FIB-EBSD. The 3D-EBSD scans were conducted in the tensile samples deformed up to $e=35\%$ and
$e=40\%$. A total of four cases were successfully acquired and the corresponding inverse pole figure maps are displayed in Figure 4.6. In most of the cases the TiN particle was indexed as ferrite; this misindexation allows the reconstruction of the particle geometry (coloured amber in the 3D maps of Figure 4.6). The only case where the particle was not indexed by the EBSD system corresponds to the scan in Figure 4.6a.

Similar to the observations made with the optical microscope, the growth of the voids is aligned along the tensile direction, parallel to the RD, and voids were observed to occur either along grain boundaries or within grains. A particular case is the scan shown in Figure 4.6c where two voids are observed on the TiN particle. One of the voids is located on the surface prior to the first milling and another one is located underneath the initial layers of the scan. The SEM micrographs presented in Figure 4.7a and b correspond to different slides of the scan shown in Figure 4.6c where sections of both voids are displayed separately. Although the sizes of the voids are similar, both of them have different shape: void 1 (cf. Figure 4.7a) has a rounded shape while void 2 has a planar geometry (cf. Figure 4.7b). An additional characteristic of void 2 is its spatial...
orientation which is diagonally aligned with respect to the tensile direction (cf. Figure 4.7b) rather than parallel to the tensile axis.

Figure 4.7c and d correspond to a 3D map showing the TiN particle, the voids and the grain boundaries from two different perspectives. As can be observed, void 1 is initiated close to the corner of the particle where no grain boundaries intersect the particle. The situation of void 2 is different. Based on Figure 4.7c and d, it is observed that the void was initiated in the intersection between the TiN particle and a grain boundary. The growth of void 2 follows the grain boundary which explains the spatial orientation independency from the tensile direction and the planar geometry of the void.

Therefore, although no preference is observed for void initiation to occur in the intersection of grain boundaries with TiN particles (in comparison to other void initiation sites of the particle), the growth trajectory of a void nucleated in this

![Figure 4.7: SEM images of the reconstructed volumes from the sample deformed to 40% (a, b). The micrographs were taken after positioning at the milling stage and include a tilt angle around TD. The micrograph in (b) corresponds to several slices below (a) and displays another void nucleated in the opposite corner of the particle. Curtaining is observed in all the surfaces as grooves with orientation parallel to RD which corresponds to the milling direction. (c) Different perspectives of the 3D reconstruction of the grain boundary of one of the grains (red colour) together with the voids (black colour) and the TiN particle (amber colour). The location of the voids with respect to the TiN particle is distinguished in (c) while the view in (d) allows discerning that void 1 is located out of grain boundaries and void 2 is located onto the grain boundary plane.]
intersection has a preference to follow the grain boundary. The consequences are the penny-shape geometry of void 2 (cf. Figure 4.7) and its spatial orientation, which is parallel to the grain boundary.

4.5 The relation between plastic deformation and void initiation

The heterogeneous plastic deformation occurring within the microstructure has an influence on the local stress and strain conditions around the TiN particles and consequently on the void initiation mechanisms. Therefore, the characterization of the particle deformation zone, PDZ, around the TiN particles was performed by EBSD at the samples elongated at different strains. In addition, the technique is also applied to the ferrite matrix with the intention of analyzing the evolution of the grain substructures during tensile deformation as well as assessing the different EBSD parameters for deformation characterization.

The automatic crystal orientation mapping was performed on tensile samples deformed up to 5%, 15%, 25%, 35% and 40%; cf. Figure 4.3 for reference to these deformation states in the tensile test curve of the IF steel. Additional orientation maps were collected from samples deformed until fracture and at the state previous to failure where localized necking is observed in the form of macrobands. All EBSD maps were cleaned up using a single iteration of the “Grain Dilation” algorithm defined by the TSL-OIM® software. The grain tolerance angle was set to 5° and orientation clusters with more than 50 points were considered grains.

The EBSD-based maps used for deformation assessment were the image quality (IQ) or band contrast (BC) maps, the grain reference orientation deviation (GROD) map, the Kernel Average Misorientation (KAM) map and the Geometrical Necessary Density of Dislocations (GND) map. The orientation reference used for each grain of the GROD maps is the average crystal orientation, which is determined by the calculation algorithm described in Appendix A. In the case of the KAM map, only the first neighbours are used for its calculation; this means that a maximum of eight crystal orientations are employed since the EBSD maps were acquired on a square grid. In the case of the deformation of the ferrite matrix, each of the grains is characterized by the Scalar Orientation Spread (SOS) and the KAM parameters which are respectively based on the GROD and KAM maps (Appendix A). A lower limit threshold of 2° was included in the calculation of both parameters where individual crystal orientations within a grain lower than this limit are not considered.

Misorientation profile charts are also used in some cases in order to give an idea about the size of the deformation zone produced by the particle. These charts plot the
misorientation angle formed between each orientation lying on a line traced with respect to (i) the previous adjacent point, (point-to-point -P2P), or (ii) the starting point of the line, (point-to origin -P2O). In the second case, all traced lines had their origin in close proximity to the particle.

4.5.1 Evolution of the grain substructures

As an initial step to analyse the grain substructures produced during deformation, the initial conditions of the material were studied with the intention of having a point of comparison and in addition analyzing the effects of sample preparation. An EBSD scan on the rolling plane of the IF steel in initial (undeformed) conditions is displayed in Figure 4.8 with different types of crystal orientation maps. A grey scale IQ map (cf. Figure 4.8a) shows some variations within the grains without any specific pattern. This map also delineates clearly the grain boundaries with a much lower intensity (dark grey to black) than the features located within the grains. The Inverse Pole Figure,(IPF), map (cf. Figure 4.8b) shows some colour variations in the grains which means certain variations of the crystal orientation within the grains, although this map is not the most appropriate to visualize orientation gradients [17]. The KAM (Figure 4.8c) and the grey scale GROD (Figure 4.8d) maps show the existence of short- and long-range orientation gradients, respectively. The existence of orientation gradients within the grains in the initial conditions of the IF steel is attributed to the initial conditions of the material as well as to artefacts from the sample preparation.

Another important point is the presence of sub-grain boundaries in the initial conditions. Although these boundaries show no geometrical distinction from the grain boundaries when observed in the IQ map, the misorientation angle can reach values lower than 15° or even 5°, as pointed out by arrows in Figure 4.8b. As the subsequent analysis is based on orientation gradients created during deformation, it is important to eliminate or diminish the influence of misorientations of these sub-grain boundaries on the results. The impact of the sub-grain boundaries on the deformation characterization is mainly reflected on the GROD maps and the associated SOS parameter, as it is mentioned later in this section. The reason is that the different crystal orientations of the subgrains which constitute a grain form separated and compacted orientation “clouds”. Then, the average orientation of the grain is located among the orientation clouds of the sub-grains. As a consequence of this situation, the GROD map is representing the misorientation between the subgrains instead of the orientation gradients within the grains. The way to control the subgrain influence on the deformation characterization is by selecting an appropriate misorientation angle threshold for grain definition: low
enough to separate for sub-grains but high enough to avoid the partitioning of deformed grains. Although the misorientation threshold for grain definition in Figure 4.8 was defined for illustration purposes as 2°, this limit is set to 10° on the following calculations and maps.

The deformation substructures in the grains can already be recognized after 5% of deformation. The interior of some grains displays wavy and ill-defined patterns which appears as low image quality lines in the IQ map of Figure 4.9a. These features are accompanied by a misorientation between the regions on both sides (as pointed out by an arrow in Figure 4.9b) and usually do not exceed 4°. One example is illustrated in the misorientation profile of the line AB, which crosses one of these curved patterns (cf. Figure 4.9).

As can be observed in the chart of Figure 4.9c, the pattern carries both a short- and relatively long-range orientation gradients based respectively on the peak of the P2P misorientation profile and the smooth transition of the P2O curve after crossing the pattern. The patterns visualized by the IQ map of Figure 4.9a are attributed to dense dislocations networks arranged in a curved alignment. The presence of these features produces a misorientation between both sides due to the net effect of the non-redundant dislocations.
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Figure 4.9: (a) IQ and (b) GROD maps from the IF steel sample deformed up to 5%. The grain substructures observed within the grains produce a misorientation, as it is pointed out in (b). The misorientation profile plot in (c) corresponds to the segment AB shown in (a). The P2P profile refers to point-to-point misorientation and the P2O refers to point-to-origin misorientation where the origin is the point A. The step size is 0.1 μm.

As deformation increases, the dislocation patterns intensify and start rearranging into straight lines. The alignment of the patterns is usually oriented approximately 40° with respect to the tensile direction. The IQ map of Figure 4.10a shows examples of such substructures within the grains which are aligned along two different directions, and in some instances crossing each other. In some cases, parts of the pattern still exhibit curved segments. The observed grain substructures are usually referred to as Geometrically Necessary Boundaries (GNBs) or Dense Dislocation Walls (DDWs) due to the high amount of dislocations arranged in a planar geometry and the misorientation formed between the separated crystal regions [18]. It is important to mention that the misorientation angle differs not only for each DDW but even along their length. This can be observed on the broad colour range of the DDW’s in the KAM map of Figure 4.10c. In addition to the misorientation produced by the DDW, long-range orientation gradients are accumulated within the entire grain from one edge to the other. This effect can be perceived by the smooth colour gradient in the IPF map of Figure 4.10b and better appreciated in the GROD map of Figure 4.10d where the regions close to the boundaries of the upper and lower grains show a large orientation deviation (close to 20°) from the crystal orientations in the centre of the grain.

In order to investigate the deformation of the microstructure one step before the fracture, two samples were deformed up to similar strains close to failure (e=45%). Both samples exhibited pairs of macrobands, which are the result of intense strain localization previous to fracture. The location and orientation of the macrobands on the tensile samples are shown in Figure 4.11c. Sample preparation was made on different planes of observation for each sample. In one case, the sample was cut in the middle width along
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the tensile direction and the observation was carried out in the RD-ND plane. In the case of the second sample, the plane of observation was chosen to be the plane of one of the macrobands; in other words, the sample was cut along the macroband and the newly revealed surface was prepared for observation. The EBSD maps shown in Figure 4.11 correspond to scans of both samples. The first observation shows an extension of the grains along RD which is accompanied by a reduction of the grain size in ND (cf. Figure 4.11a). The interior of the grains shows a further development of the substructures in comparison to the sample deformed to 25% (cf. Figure 4.10). Most of the substructure features are well-defined DDW mainly aligned at approximately 45°, as can clearly be observed in the KAM map of Figure 4.11b. The effect of the long-range orientation gradients is well visualized in the IPF map of Figure 4.11a where different ranges of colours are observed in the entire grain.

If the microstructure is observed over the macroband, as in the case of the IPF map of Figure 4.11d, then the grains show a lower aspect ratio and no preference in spatial directionality. The long-range orientation gradients in this observation plane are similar to the microstructure on the RD-ND plane, i.e. the long-range orientation gradients have an accumulative character within the grains from one edge to the other. In the case of the short-range orientation gradients, the DDWs in the interior of some grains show low misorientation angles which are characterized by the low KAM values, cf Figure 4.11e. It is suggested that the plane of the macroband is almost parallel to the plane of some of the DDWs in the grain interiors and this is the reason for the apparent absence of short-range orientation gradients when the observation plane is parallel to the plane of a macroband. Why the DDWs

\[ \text{Figure 4.10: Different EBSD maps from the same region of the sample deformed up to 25%.} \]

(a) IQ map. (b) IPF map (c) KAM (d) GROD. The step size used is 0.1 \( \mu \text{m.} \)
in the interior of several grains do coincide with the macroband? The possible explanation is that the grain interiors are predominantly controlled by the external boundary conditions of the tensile deformation and therefore the DDWs have a spatial orientation parallel to the macroband. In contrast to the interior of the grains, the regions closer to the grain boundaries show different types of substructures: the DDWs exhibit higher misorientation angles (cf. Figure 4.11e). This is due to the fact that the strain conditions in the proximities to the grain boundaries are mainly controlled by the interactions between the neighbouring grains rather than by the external conditions; as a consequence, the spatial orientation of the DDWs differs from the macroband inclination and are controlled by the local conditions.

Figure 4.11: EBSD maps made at the sample deformed at the point of localized necking. The (a) IPF and (b) KAM maps were taken in the middle width of the tensile sample on the intersection of the macrobands. (c) Scheme of the planes of observation made at the sample. The (d) IPF and (e) KAM maps were collected over the macroband plane.

Before reaching the fracture point, a large amount of deformation is imposed on the microstructure and the distinction of the original grains becomes complicated for two reasons. First, the crossing of high misorientation DDWs over a grain produces the partitioning of the grain due to the misorientation threshold specified for grain definition of the EBSD software. This partitioning means that one grain may fragment in several grains. The other complication appears due to the crystal rotation of the grain borders towards an orientation close to the border of the neighbouring grain. Then, the grain is
annexed to its neighbour and considered as the same grain. The last case is occasionally found and is identified by the low image quality of the grain boundaries and the geometry of the two grains.

The ultimate condition of the microstructure is shown in Figure 4.12, which represents the fractured edge of the tensile sample. The EBSD scan was acquired close to the fracture surface and in the middle thickness of the sample (cf. Figure 4.12c), which corresponds to the fibrous zone of the fracture surface (cf. Figure 4.4a). As can be observed in the IPF map of Figure 4.12a, the grains show an elongated shape and contain a lot of DDWs in their interior. The main difference between the broken sample and its previous step (localized necking) is that part of the grains display severe grain fragmentation where some of the fragments can be as small as $1 \, \mu m^2$.

![Figure 4.12: (a) IPF map of the region close to the fracture surface corresponding to the SEM micrograph in (b). (c) SEM micrograph with lower magnification enclosing the zone where the EBSD scan was acquired. The step size of the EBSD scan is 50 nm.](image-url)
Several voids are present in this region of the fractured sample and can easily be distinguished in the SEM micrograph of Figure 4.12b. Their size varies from sub-micron dimensions up to several square micrometers. The voids are surrounded by regions of fragmented grains and no apparent particle is associated to them.

In order to quantify the evolution of the grain substructures created as a result of the plastic deformation, the SOS and KAM parameters were calculated for the deformed grains of the samples. The average value of both parameters as a function of the tensile engineering strain is shown in Figure 4.13. The data show that both the short- and long-range orientation gradients, respectively represented by the KAM and SOS parameters, increase with the increase of the tensile engineering strain. The SOS curve exceeds the KAM curve for all deformation states and additionally exhibits a larger positive slope, which is a consequence of the definition of these parameters according to which the SOS corresponds to higher misorientation angles than the KAM.

The magnitudes of the SOS and KAM prior to deformation (\(e=0\%\)) are close to 2°, which corresponds to the threshold angle for both parameters calculation. The reason for these initial relatively high values is attributed to two factors, which are explained at the beginning of this section. In the first case, some grains contain sub-grain boundaries, which resemble the features observed in Figure 4.8 and, although the threshold to define a grain was set to 10°, some initial sub-grain boundaries have values below that limit. The second factor corresponds to a slight initial orientation gradient within the material that can be attributed to material processing, polishing procedure and angular resolution of the equipment. Therefore the down level of 2° was accepted as a noise level and only the grains which contain at least one component with a SOS or KAM magnitude higher than 2° are incorporated into the calculation of the average SOS and KAM for each engineering strain. The fraction of grains, which meet these conditions, is given as percentages in Figure 4.13. In the analysis of the average KAM prior to
deformation, solely 15% of the grains satisfy these conditions while the other grains contain elements with a KAM lower than 2°. The percentage increases as the applied strain is higher until reaching 100% for $\varepsilon=40\%$ and $\varepsilon=45\%$. The average SOS, in contrast, has 83% of the grains satisfying the misorientation threshold of 2° in the initial conditions and this fraction increases for the deformed conditions. Therefore, the magnitude of both parameters is largely affected at lower deformation conditions by sample preparation effects, the misorientation threshold chosen and the grain assembling. These effects are reduced at larger tensile engineering strains where the majority of the grains are above the limit of the misorientation threshold for the definition of the SOS and KAM parameters and the crystal rotations produced by the grain substructures surpassed the value of the parameters at the initial conditions.

A last point to emphasize is the increase of the standard deviation of the SOS parameter at higher strains. In this case, the standard deviation indicates the existence of grains with a broad range of SOS magnitudes at the same imposed tensile engineering strain. This SOS heterogeneity is the result of different effects, such as the grain size dependency of the SOS value [19], strain heterogeneity along the tensile sample (localized necking), different response based on the crystal orientations and the neighbouring grains, among other factors. While the grain size dependency effect also includes the two-dimensional aspect of EBSD measurements and is common and statistically similar at all strain conditions, the variation of the standard deviation among the different tensile engineering strain conditions is mainly attributed to the heterogeneous character of the deformed microstructure. Although the standard deviation is lower for the KAM parameter, a similar interpretation is formulated.

In summary, the tensile deformation imposed to the IF steel produces changes in its microstructure and these alterations can be characterized by the EBSD technique. The evolution of the grain substructures morphology with deformation as well as the associated crystallographic rotations can be visualized by different EBSD-based maps and characterized by the SOS and KAM parameters. In the following section, the particle deformation zone created by the interaction of the ferrite with the rigid TiN particles is analyzed using EBSD.

4.5.2 Particle deformation zone

The interaction between the rigid TiN particles and the surrounding ferrite grains during plastic deformation gives place to the creation and evolution of dislocation structures in the matrix. This region is commonly denominated as the particle deformation zone, PDZ [12]. The evolution of the PDZ during the tensile deformation is
associated with remarkable orientation changes, which can be characterized and analysed by EBSD.

Figure 4.14 displays an EBSD scan of the sample deformed to 5% where a TiN particle is embedded in one ferrite grain. As can be seen in the IQ map (Figure 4.14a), there are only slight variations in the grey scale which is an indication that there is almost no distortion of the crystal structure in the region close to the particle. A similar situation is observed in the GROD map (Figure 4.14b) where it is difficult to observe deviations of the crystal orientation within the grain. It is also important to mention that the short-range orientation gradients are also minimum and comparable to the noise generated by the angular resolution of the EBSD (~1°). This influence is observed in the point-to-point (P2P) misorientation profile charts of Figure 4.14c and d where the possible orientation changes produced by deformation are obscured by the angular resolution of the equipment. However, although the misorientation angle between neighbouring points is mostly lower than 1° in the vicinity of the TiN particles, the orientation gradient has a cumulative and long-range incremental character (cf. the point-to-origin, P2O curve of Figure 4.14c). This slight but consistent gradient is attributed to the initial stages of plastic deformation.

![Figure 4.14: (a) IQ and (b) GROD maps of an EBSD scan area made from 5% deformed sample. The TiN particle is located within the grain. Plots in (c) and (d) are misorientation profiles corresponding to the lines c and d traced in (b). The origin of each line is located close to the particle.](image)

In the 15% deformed sample, the presence of dense dislocation walls, DDW, is already observed both in the surroundings of the TiN particle and the entire grain (cf. Figure 4.15a). These grain substructures are responsible for the orientation changes (~2°) along the P2P misorientation profile (Figure 4.15c and d), slightly higher than the orientation noise of the technique. Similar to the particle deformation zone of the sample elongated to 5%, the misorientation profiles of Figure 4.15c and d also display low P2P
misorientation values accompanied by an accumulative character of the misorientation angle along the profile lines, which in this case exceeds 10° based on the P2O misorientation profiles.

![Figure 4.15: BC (a) and GROD (b) maps of an EBSD scan area made from the sample deformed to 15% elongation. The TiN particle is located in the grain boundary. Plots in (c) and (d) are misorientation profiles corresponding to the lines c and d traced in (b). The origin of each line is located close to the particle. The step size of the scan is 50 nm.](image)

As can be observed in Figure 4.16a, the microstructure of the sample deformed to 25% exhibits a higher amount of DDWs distributed over the grains. These DDWs give rise to a subgrain structure, which is more pronounced near grain boundaries or close to the TiN particles. In the vicinity of the particles one frequently observes a gradual and increasing rotation of the matrix with respect to the reference orientation of the grain where the higher orientation gradient is located close to the particle-matrix interface. One example of this smooth rotation can be observed in the darker regions close to the particle in Figure 4.16b and the misorientation profile of Figure 4.16c where the P2P misorientation exhibits small values (orientation gradient) but contributes the gradual and consistent increase in the P2O misorientation profile. Inside the particle deformation zone, the maximum misorientation angle with respect to the associated grain average orientation is in the order of 10°.
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The grain substructures become increasingly more complex as the deformation accumulates, and some dense dislocation walls in the 35% elongated sample show a curved pattern in the proximities of the TiN particle (Figure 4.17a). It is commonly observed that the gradually rotating crystal orientation in the particle deformation zone is disturbed by an abrupt misorientation peak (cf. the misorientation profiles of Figure 4.17c and d). This discontinuity can be of the order of 4 to 15° and is usually observed in the extension of the particle edges that are perpendicular to the tensile direction. In addition, dense dislocation walls originating from the corner of the particle (Figure 4.17a) and accompanied by an orientation deviation from the reference orientation are commonly observed (zone indicated by black arrow in Figure 4.17b).

Figure 4.16: IQ (a) and GROD (b) maps of an EBSD scan area made from the sample deformed to 25% elongation. The TiN particle is located in the grain boundary. The plot in (c) is a misorientation profile corresponding to the lines traced in (b). The origin of each line is located close to the particle.

Figure 4.17: IQ (a) and GROD (b) maps of an EBSD scan area made from the sample deformed to 35% elongation. The TiN particle is located in the grain boundary. Plots in (c) and (d) are misorientation profiles corresponding to the lines traced in (b). The origin of each line is located close to the particle.

Inside the diffuse necking zone of the sample deformed to 40% elongation an intense rotation of the matrix is observed in the regions close to the particle corner.
These particle deformation zones are often extended to both sides of the particle and are aligned along the tensile direction. One example is displayed in Figure 4.18 where both regions show a deviation of the reference orientation higher than 40°. These orientation deviations are a combination of gradual and abrupt rotations as can be observed in the misorientation profiles of Figure 4.18c and d. The particle displayed on Figure 4.18 has on one of its corners a void, nucleated by interface separation. The void is located along one segment of the grain boundary where a highly rotated region is adjacent to the void.

Based on the GROD maps and the misorientation profiles made in the PDZ for the different stages of deformation, it can be observed that the PDZ embraces a region which is in the order of the particle length. This a common point for all the cases analyzed and is better appreciated by the P2O misorientation profiles where a type of saturation is reached in the accumulative character of the misorientation angle at a distance similar to the TiN particle size. This information is used for the characterization of the PDZ described below.

The orientation gradient produced in the vicinities of the TiN particles has a long-range component in addition to a short-range component. This combined character makes it possible to visualize of the particle deformation zone by GROD maps because of the departure from the grain average orientation by local crystal rotations in the

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*Figure 4.18: BC (a) and GROD (b) maps of an EBSD scan area observed on the sample deformed to 40% tensile engineering strain. The step size of the scan was 50 nm. The TiN particle is located in the grain boundary and a void is nucleated in one of its corners. Plots in (c) and (d) are misorientation profiles corresponding to the lines traced in (a).*
vicinity of the TiN particles. However, the pixel GROD values have no direct correlation with the local orientation gradients, particularly if only a region of the grain is intended to be analyzed, as in the case of the PDZ.

The situation is different if the pixel values of the KAM are considered. The calculation of this parameter is based on a local approach due to the employment of the immediate neighbours instead of a reference orientation situated outside the considered location, as is the case of the GROD parameter. The KAM represents a more suitable parameter for characterizing the orientation gradient occurring within the PDZ.

The methodology used for this characterization consisted basically in averaging the KAM angles of the points located within the PDZ. Nevertheless, in order to minimize the impact of low deformed regions within the PDZ as well as the influence of the angular resolution of the technique, the PDZ was first partitioned into regions based on the radial distance from the TiN particles and afterwards the maximum KAM angle within each of these segments was averaged. This procedure was applied for all the EBSD scans of the TiN particle deformation zones observed at different tensile engineering strains. The resulting data are displayed in Figure 4.19. The average maximum KAM angle for the unstrained samples (0% deformation) has an initial value larger than 0° (cf. Figure 4.19). This value is attributed to several factors such as the angular resolution of the EBSD, imperfections due to the sample preparation and even the initial distortion created by the TiN particle during the thermo-mechanical treatment. Despite this initial value, the average maximum KAM angle for each PDZ shows an obvious increasing trend as the tensile engineering strain increases. In similarity to the SOS and KAM parameters used in Figure 4.13, the parameter employed for characterizing the particle deformation zone has variations for the same value of tensile engineering strain. The reason is attributed to similar causes; namely the orientation gradients within grains, the different deformation history for each PDZ or the strain heterogeneity along the tensile sample.

The highest maximum KAM angle observed in Figure 4.13 was 25° and occurs in the sample deformed up to 40%. The EBSD scan of such extreme conditions

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![Figure 4.19: Evolution of the maximum KAM angles within the particle deformation zone at different tensile engineering strain values.](image-url)
Void initiation mechanisms in IF steels

corresponds to the orientation map of Figure 4.18 and the KAM map of that scan is shown in Figure 4.20a. Most of the dislocation structures located within the particle deformation zone are associated to either the corners or edges of the TiN particle. The dislocation structures are arranged as closed loops around the TiN particle where misorientations vary along the length of these loops. The orientation gradients represented by the KAM angles could be used for description of the distribution of the dislocation structures formed in the PDZ, and in addition it is possible to calculate a lower bound of the geometrically necessary density of dislocations of those substructures.

Figure 4.20: (a) KAM map of the scan of Figure 4.18 which corresponds to the region with a particle of a sample deformed up to 40%. (b) GND map of the same region where the colour-code is given in logarithmic scale. The red circle contains a dislocation structure which is not totally revealed in the KAM map. (c) Graph showing the distribution of the maximum GND and KAM values as a function of the radial distance from the TiN particle.

The geometrically necessary density of dislocations, GND, were calculated based on the orientation gradients of that scan (Appendix B) and displayed in Figure 4.20b. The geometrical arrangement of the GND content of the grain substructures is similar
between the KAM and the GND maps, although some differences are found. One example is highlighted in Figure 4.20b, where the GND substructure within the red circle is not totally revealed by the KAM map. It is less probable that this substructure is a consequence of the angular resolution of the technique due to the coherency and continuity of the loop. In a similar manner as the average maximum KAM was calculated, the maximum GND density was obtained as a function of the radial distance from the TiN particle and plotted in Figure 4.20c. The maximum KAM was also included in the graph. Both parameters show variations along the distance but the higher values are located in the proximity to the particle. The average value of the maximum geometrically necessary dislocation density along the PDZ is $2.2\times10^{17} \text{ m}^{-2}$, whereas in the grain interior, far away from the particle, the average value is only $2.7\times10^{16} \text{ m}^{-2}$.

![Figure 4.21: (a) Kernel average misorientation map of 40% deformed region with TiN particle; (b) Zones with KAM >4° which form substructure around the particle; (c) bottom view of the same region displaying void formation in the volume with high orientation gradient. The step size as well as the depth thickness is set to 100 nm.](image)
The analysis of the grain substructures from the previous EBSD scans are based on a two-dimensional observation. The FIB-EBSD technique allows the reconstruction of the PDZ in three-dimensions. Figure 4.21a shows the 3D Kernel map from the reconstructed volume of Figure 4.7. The 3D-KAM map was elaborated by averaging the misorientation angle of each kernel voxel with respect to its 26 nearest neighbours (first neighbours). In order to visualize the geometry of the regions with high KAM values, the voxels with KAM angles higher than 4° are displayed in Figure 4.21b. This isosurface map allows observing the connection among the regions with equal misorientation, i.e. it visualizes the orientation gradients and therefore reveals the grain substructures geometry. Based on Figure 4.21b, the voids are surrounded by the dislocation structures which are inter-connected to other regions with high orientation gradients located in the grain interior. These regions with high KAM angles are aligned along planes but also follow a curved morphology. For instance, the region located close to void 1 and the corner of the TiN particle show a peculiar geometry. The intersection of this grain substructure with the surface plane has a similar geometry to the closed loops observed in the 2D EBSD map in Figure 4.20a. However, in 3D mapping this substructure is closed below the surface, with a shape similar to the shape of a bowl (cf. Figure 4.21c).

4.6 Discussion

4.6.1 Void initiation on TiN particles
In order to understand the micromechanisms of void initiation on TiN particles, it is necessary to introduce some mechanical characteristics of these particles. Titanium nitride has a B1 (NaCl)-type structure which is characterized by an inherent brittleness, as most of the refractory compounds. This brittleness is reflected in the fragmentation of these particles when they are subjected to plastic deformation, as has been reported in other investigations [6-8]. On the other hand, it is claimed that the interfaces between these particles and the ferrite matrix have a significant strength [4] and, therefore, this interfacial bond strength provides a significant opposition to the initiation of voids by interface separation.

In this investigation, SEM fractography demonstrates the relevance of TiN in the ductile failure process of IF steels by acting as initiation sites for voids. Although other types of particles are also found (Al₂O₃ inclusions), TiN precipitates represent the majority of the total population of second phase particles. This predominance is also perceived from the particles attached to the walls of the deeper dimples on the fracture.
surface. Other authors also identified TiN as void initiation sites during deformation of IF steels [6, 11]. In contrast to the observations of Imlau et al. [20], no clear voids were identified in grain interiors, grain boundaries or triple junctions without the presence of particles. Such interpretations are difficult to assert, due to factors such as the probability of removing the particles during the metallographic sample preparation.

Based on the metallographic observation in samples with different levels of strain, it was concluded that there is not a unique void initiation mechanism exclusively acting on the TiN precipitates during the entire deformation history. Both, fragmentation and interface debonding mechanisms occur during tensile deformation although the dominance of each one alternates depending on the amount of deformation. In summary, three successive stages are distinguished in the failure micromechanisms of TiN particles: (i) dominance of particle fragmentation, (ii) dominance of interface debonding and (iii) broad void growth.

4.6.2 Particle fragmentation

The fragmentation of TiN particles mainly occurs during the first stages of plastic deformation and predominantly affects particles with dimensions larger than the average size. The preference of larger TiN particles to fracture has also been reported on maraging steels [7, 8]. This manifestation of length-scale influence is attributed to the greater probability of finding, or creating, crack initiation sites in larger volumes of the particles [7]. In summary, the smaller the TiN particle, the larger the critical stress to fragment it.

Throughout the stage of particle fragmentation predominance, the ferrite matrix only displays a limited extent of substructure formation. Well-defined dense dislocation walls start appearing at 15% deformation and similar structures occur in the particle deformation zone (cf. Figure 4.15). It could be concluded that the rate reduction of the number of fragmented particles with the strain is related to the particle deformation zone. At low level of deformation, when a homogeneous crystal orientation within the grains is preserved, the stresses generated by the deformation are directly transmitted to the TiN particles. When the stresses increase, the weaker particles start to breakup whereby the larger particles have higher probabilities to be fractured. With the progress of the deformation, larger stresses are required to fracture the remaining particles. Then a critical state is reached at which the stresses created at strain incompatibility regions, such as particles and grain boundaries, start being relieved by plastic relaxation. This relaxation is achieved by crystal rotation in regions close to the particle, giving rise to the formation of the particle deformation zone [12]. Correspondingly, these crystal
rotations contribute to inhibit the increment of stresses required to continue fracturing the remaining TiN particles.

### 4.6.3 Particle deformation zone

It was already mentioned that the formation of a deformation zone around particles is acting as a mechanism of stress relaxation for strain incompatibilities produced by the interaction between a particle and the surrounding ductile matrix. The magnitude of crystal rotation within the deformation zone increases as deformation continues and can achieve rotations as high as 40°. At low strain levels, most of the particle deformation zone is formed by long-range orientation gradients which are characterized by their low point-to-point, but accumulative misorientation gradients along a trajectory away from the particle [21]. However, in addition to the long-range orientation gradients, abrupt rotations of which the magnitude depends on the strain level become more frequent in the proximities of the particles at higher strain levels. These sharp boundaries correspond to the DDW delimiting the subgrain boundaries as also observed by Thomas et al. [22] in IF steels and by Delannay et al. [21] in an aluminium alloy. Both the DDW and the long-range orientation gradient contribute to the orientation contrast of the particle deformation zones.

In contrast to the dependency of the misorientation angle magnitude on the amount of tensile deformation, it was found that the size of the deformation zones remains almost constant and is of the order of 2 to 4 µm for all tensile engineering strain values, which is comparable to the TiN particle size. This similarity of sizes has also been observed by Humphreys [12] on non-deformable particles embedded in aluminium or copper single crystals.

The rotation is mainly concentrated in zones where the strain incompatibility is more critical, such as the regions aligned with the tensile direction, the grain boundaries and the particle corners. Evidence of the latter is provided by the high misorientation values observed in the corners by means of the EBSD measurements (cf. Figure 4.16b, Figure 4.17b and Figure 4.18b).

### 4.6.4 Particle-matrix debonding

As a result of the extensive deformation in the particle deformation zone, the plastic relaxation is exhausted and stresses are increasing, especially in zones with higher plastic localization like the particle corners. At this stage, a second form of stress relaxation occurs: void nucleation. If the void initiation criteria are satisfied for particle-
matrix debonding, such as the critical stress and energy criteria [23], then a void will be nucleated.

The highest increase of void nucleation by particle debonding occurs between 15% and 25%, which coincides with the transition of grain substructures created close to particles. In this case, the matrix surrounding the particle relaxes the stress condition on the interface by plastic deformation and only when the matrix cannot accommodate more deformation, the stresses at the interface reach the critical condition for decohesion.

4.7 Conclusions

The scarce amount of particles in IF steels provide the opportunity of studying the strain partitioning between the individual TiN particles and the ferrite matrix without influence of other particles in the surrounding.

Void initiation at TiN particles in IF steel sheets, subjected to uniaxial tension, has been experimentally investigated by OM, SEM and EBSD on samples deformed to different tensile engineering strains. Based on the above discussions, the following conclusions can be drawn:

- TiN precipitates were identified as void initiation sites during tensile deformation of IF steels. These particles can initiate voids by either separation of the particle-matrix interface or by particle fragmentation. Both mechanisms may be active on the same particle, which implies that one mechanism does not exclude the other.
- The failure mechanism of TiN particles during tensile deformation can be divided in three consecutive stages: (i) predominance of particle fragmentation, (ii) predominance of particle-matrix debonding and (iii) extensive void growth.
- Particle fragmentation mainly occurs at low deformation (up to $e=15-25\%$) and is more frequent among the larger particles. The development of the particle deformation zone reduces the void initiation rate by this mechanism, probably due to the relaxation of stresses at the particle.
- Particle-matrix debonding preferentially occurs at particle corners and is preceded by a certain amount of crystal rotation.
- The average value of the maximum GND density along the PDZ is $2.2\cdot10^{17}$ m$^{-2}$, whereas in the grain interior, away from the particle, the average value is only $2.7\cdot10^{16}$ m$^{-2}$.
If both void initiation mechanisms are compared in terms of fracture resistance, particle-matrix decohesion exhibits the higher resistance. The reasons are the lower initial void volume which also reduces the amount of void growth.

References


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Chapter 5

Failure micromechanisms in DP steels

The failure micromechanisms of DP600 steels with different heat treatments are studied in this chapter using microscopic techniques. In the first part of this chapter the initial microstructure of the as-cold rolled DP steel is studied together with the microstructure after the heat treatment. The mechanical properties of the different DP steels are investigated by tensile tests and expanded by analysis of the geometry of the broken samples. The preferential sites for void initiation in current Dual Phase (DP) steels are mainly associated to the martensite. Finally, it is observed that shear bands are responsible for the final stage of fracture by acting as nucleation sites for cracks.

5.1 Introduction

Although the concept of the duplex microstructure of the DP steels can be traced back to 1937, the intensive research of these steels and their applications to the automotive industry was developed during the seventies, as part of the solution to decrease the vehicle weight [1, 2]. Formability has been one of the priorities on the development of DP steels [3] whereby the fracture mechanisms are one of the principal factors of concern.

The void initiation mechanisms of DP steels have been extensively reported by several research groups and were associated with either non-metallic inclusions or martensite. In the former case, the typical inclusions observed are aluminium oxides [4], manganese sulphides [4, 5] and in some instances titanium nitrides [6]. However, the amount of inclusions in modern DP steels is extremely low and it has been frequently claimed that the voids nucleating in those inclusions are usually stable and have no
impact on the fracture process [7]. These assumptions have nowadays been confirmed by most of the investigations performed on fracture of DP steels. Therefore the void nucleation in inclusions is usually considered as a secondary damage process, whereas martensite-related void initiation is the dominant process [4, 7-12].

Void initiation at martensite principally occurs by either the fragmentation of the martensite or decohesion at the ferrite-martensite interface. A third mechanism was proposed by Steinbruner et al. [8] based on observations of separated martensitic regions. This separation was attributed to (i) the splitting of adjacent martensitic regions or to (ii) the localization of the deformation within the martensite.

The first two initiation mechanisms have different characteristics. Martensite fragmentation is known to occur at strains as low as 3% [4], 5% [8, 9] or even at the onset of plastic deformation [10], whereas decohesion of the ferrite-martensite interface takes place at higher strains [8]. The preferential occurrence of the void initiation mechanisms depends on several factors, such as the carbon content, structure, morphology or distribution of the martensite. Avramovic-Cingara et al. [4] reported that lower carbon content within martensite promotes the mechanism of void initiation by fragmentation because of the higher plasticity of the martensite. It was proposed that the extensive deformation of martensite allows reaching the critical condition for its fracture at lower global strains whereas a martensite with higher carbon content enhances the integrity of martensite and is prone to decohesion from the ferrite [11]. The observations of Avramovic-Cingara et al. [4] were made based on two DP steels with different carbon content but with similar volume fraction and distribution of martensite. The application of tempering produces changes on the structure of the martensite and, as a consequence, has an influence on the void initiation mechanisms of the DP steel. Stevenson [13] concluded that tempering reduces the incidence of martensite fragmentation and favours ferrite-martensite interface decohesion. With regard to the influence of the martensite morphology, He et al. [9] found that a coarse structure leads to a fragmentation of the martensite at lower strains followed by decohesion of the ferrite-martensite interface at higher strains. In the case of a finer structure, the ferrite-martensite separation is the dominant mechanism.

The aforementioned microstructural factors have their influence on the void activity of DP steels due to modifications produced on the strain partitioning between martensite and ferrite [14]. The strain partitioning among the martensite and ferrite of DP steels has been directly proved by in-situ tensile tests coupled with digital image correlation or micro grid techniques [15-17]. A common point in this type of
experiments is the proof of plastic deformation of martensite and the heterogeneous nature of the deformation between ferrite and martensite.

Although the void initiation mechanisms are well documented, the final stage of fracture has received less attention. In most cases, it is assumed that void coalescence takes place after a high amount of void initiation and growth. In some specific cases, cleavage has been proposed as a secondary mechanism, which interrupts the void coalescence process and produces the final failure of the material [18]. Independently of the participation of an additional fracture micromechanism, the majority of the experimental studies have commonly shown a relatively low amount of void fraction at the moment of fracture. Quantitative studies based on image analysis on the cross-section of broken tensile samples from different research groups have reported a maximum area fraction of voids between 0.7 and 1.4% close to the fracture surface [6, 4, 18, 19]. These low percentages are also found in void volume fraction measurements. Maire et al. [20] conducted in-situ tensile test experiments using X-ray microtomography and observed that the maximum void volume fraction is smaller than 2%.

The present chapter is dedicated to the analysis of the different stages of failure evolution in DP steels and considers the microstructural origin with regard to the impact on the macroscopic scale. In order to achieve this goal, the different microstructural constituents are first characterized in terms of crystallography and chemistry for the different heat treated DP steels described in Chapter 3. Afterwards the mechanical properties are investigated with a special emphasis on the properties related to deformations from the onset of necking until fracture. Once the macroscopic evolution of fracture is revisited, the mechanisms of fracture occurring within the microstructure are analysed in terms of the ferrite grains deformation, the void initiation mechanisms and the statistical quantification of the void activity. The conducted experimental observations allows establishing the mechanisms of failure on DP steels and clarifying the last stage of the fracture process which is related to the link between the macro- and meso-scale fracture mechanism.

5.2 Initial microstructure

The microstructure of the as-cold rolled material prior to the heat treatments is composed of ferrite and pearlite, cf. Figure 5.1. The ferrite grains have an elongated geometry oriented along the rolling direction (RD). Because of the cold rolling conditions, grain substructures are commonly observed within the ferrite grains (cf. Figure 5.1b, c) and the pearlite colonies form bands aligned along the sheet rolling direction. (cf. Figure 5.1a). The total area fraction of 16% ± 2.4% pearlite was measured
based on 13 different optical micrographs. The SEM micrographs at relatively high magnifications (cf. Figure 5.1c) displayed the lamellar structure of the pearlite colonies formed by fragmented cementite platelets of different lengths and the substructure of the deformed ferrite grains is revealed.

Figure 5.1: Optical micrographs displaying (a) the preferential alignment of the pearlite in bands along the rolling direction, and (b) the grain substructure patterns within the elongated ferrite grains. A high magnification SEM micrograph of the pearlite structure is shown in (c). In all cases, the samples are etched with Nital 2%.

The resulting microstructures after the different heat treatments are displayed in Figure 5.2. The microstructures of all DP steels are a mixture of ferrite and other constituents such as martensite and bainite. It is important to emphasize that no pearlite formation was observed in the DP steels. A detailed analysis about the nature of the constituents is made in the following paragraphs using the dilatometer results. In some cases, manganese sulphides and aluminium oxide inclusions were located inside the martensite bands. The reason for martensite banding is the interdendritic segregation of solute substitutional elements, e.g. manganese, during slab solidification of casting [21]. This microsegregation is aligned in bands parallel to the rolling plane during the hot and
cold rolling processes. As a consequence, alternating concentration gradients of solute elements are produced through the material thickness. Manganese is known to decrease the Ar$_1$ temperature and, during cooling from the austenitic temperature range, the epitaxial ferrite starts first growing in low Mn content regions. During this process, the carbon is rejected via diffusion towards regions with lower Ar$_1$ (high Mn content) where martensite eventually forms along these bands. At certain conditions, such as higher Mn and C concentrations, martensite or even austenite retention may occur at slow cooling rates.

As the martensite banding partly depends on the solid-to-solid phase transformation kinetics [21], the heat treatments have an effect on the distribution of the martensite. For example, the slow heating from room temperature to the intercritical ($A_1$-$A_3$) temperature region and the high reheating temperatures reduce the amount and the thickness of the martensitic bands (cf. Fig. 5.2b, d).

![Figure 5.2: Optical micrographs of the DP steel specimens varying in the heat treatment: (a) Reference (Ref). (b) Slow heating (SH). (c) Slow cooling (SC). (d) High temperature (HT). The samples were etched with Nital 2%. The darker regions correspond to the martensite and the bainite while the ferrite appears as the brighter phase. For details of the heat treatment, please refer to Table 3.3.](image-url)
Chapter 5

Table 5.1 displays the results of the quantitative microstructural characterization of the heat treated steels. The grain sizes in all cases are significantly affected by the heat treatments whereby the extreme values correspond to the slowly heated DP steel with the largest GIL (10.3 μm) and to the smallest grains of the HT DP steel (4.2 μm).

Table 5.1: Microstructural characterization of the DP steels. The Grain Intercept Length, GIL, and some martensite features of the DP steel samples were calculated based on OM micrographs. The Vickers microhardness measurements were made using a test load of 1000 gf.

<table>
<thead>
<tr>
<th></th>
<th>Ref</th>
<th>SH</th>
<th>SC</th>
<th>HT</th>
</tr>
</thead>
<tbody>
<tr>
<td>GIL along RD, μm</td>
<td>7.4</td>
<td>10.3</td>
<td>6.8</td>
<td>4.2</td>
</tr>
<tr>
<td>±1.5</td>
<td>±2</td>
<td>±1.2</td>
<td>±0.7</td>
<td></td>
</tr>
<tr>
<td>Martensite/Bainite area fraction, %</td>
<td>10.3</td>
<td>10.0</td>
<td>7.5</td>
<td>10.0</td>
</tr>
<tr>
<td>Individual martensite/bainite area, μm²</td>
<td>±2</td>
<td>±1.8</td>
<td>±0.5</td>
<td>±1.2</td>
</tr>
<tr>
<td>Martensite/Bainite nearest neighbour distance, μm</td>
<td>4.1</td>
<td>4.5</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td>±0.5</td>
<td>±0.6</td>
<td>±0.3</td>
<td>±0.4</td>
<td></td>
</tr>
<tr>
<td>Martensite/Bainite nearest neighbour distance, μm</td>
<td>3.2</td>
<td>3.3</td>
<td>3.9</td>
<td>2.9</td>
</tr>
<tr>
<td>±0.1</td>
<td>±0.1</td>
<td>±0.1</td>
<td>±0.2</td>
<td></td>
</tr>
<tr>
<td>Bainitic ferrite lath width, μm</td>
<td>0.14</td>
<td>0.25</td>
<td>0.36</td>
<td>0.43</td>
</tr>
<tr>
<td>±0.05</td>
<td>±0.12</td>
<td>±0.11</td>
<td>±0.18</td>
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</tr>
<tr>
<td>Vickers microhardness, HV₁₀₀₀</td>
<td>214</td>
<td>201</td>
<td>170</td>
<td>220</td>
</tr>
<tr>
<td>±5.1</td>
<td>±2.8</td>
<td>±4.1</td>
<td>±5.7</td>
<td></td>
</tr>
</tbody>
</table>

In the case of the reference and SC specimens, the values of both steels exhibit a slight difference that is lower than the standard deviation of the measurements. The area fraction, area and minimum nearest neighbour distance of the other constituents of the microstructure, i.e. martensite and bainite, present also differences due to the heat treatments. The reference, the SH and the HT specimens have similar values of martensite/bainite area fraction (~10%) while the SC DP steel is the only specimen with markedly lower fraction of martensite and bainite (7.5%). Although the martensite/bainite fraction is the lowest, the average area of the martensite islands of the SC specimen is the largest (5 μm²) in comparison to the other DP steels. This is also accompanied by a larger separation between the martensite products, as can be observed by the relatively larger value (3.9 μm) of the average nearest neighbour distance. In contrast, the martensite products of the HT DP steel have the lowest nearest neighbour distance (2.9 μm) and are in average the smallest with 4 μm² average individual area, although this martensite/bainite size is slightly lower than the corresponding one of the reference DP steel. In the case of the bainite, the average bainitic ferrite lath width was determined from SEM micrographs of the initial microstructure and it was found that
the bainite of the HT DP steel has the wider bainitic ferrite laths (0.43 µm) among the rest of the DP steels.

The overall changes produced by the different intercritical heat treatments have an impact on the Vickers microhardness, which is considered as an indication of the mechanical properties. In this case, the slowly cooled DP steel displays the lowest microhardness value by a significant difference from the other DP steels.

In addition to the GIL, the crystal orientation distribution (texture) of the ferrite grains was determined using EBSD (Electron Backscatter Diffraction). Figure 5.3 represents the orientation distribution function, ODF, in the $\phi_2 = 45^\circ$ section of Euler space for all DP steels. In general, all four steels show comparable textures, both in terms of intensity and in terms of orientation components.

Figure 5.3: ODF of the (a) reference, (b) slow heated, (c) slow cooled and (d) high temperature DP steels represented in the $\phi_2 = 45^\circ$ section of the Euler space. The texture was calculated based on EBSD data.
The textures are weaker than the annealing texture of the IF steel (see Chapter 4) and they are composed of $\alpha$- and $\gamma$-fibres. However, the texture exhibits a slight deviation from the theoretical $\gamma$-fibre whereby a particular enhancement of the (554)$\{225\}$ component instead of the (111)$\{1\ 1\ 2\}$ component is observed. These texture characteristics are typical for the ferrite grains in DP steels, as other researchers have also reported [22, 23]. Other components were also detected in the texture of the steels, such as the Goss and Cube orientations, although the corresponding intensities are weak and vary from sample to sample.

In order to identify the type of constituents, such as martensite, bainite or retained austenite, dilatometric analysis in combination with SEM and EBSD maps are used to complement the information from optical microscopy. The dilatometric curves of the samples employed to simulate the different intercritical heat treatments are displayed in Figure 5.4. In addition to the dilatometric curves, SEM micrographs and EBSD maps of the DP steels are also included.

The thermal expansion of the ferrite during the heating of the reference DP steel (cf. Figure 5.4a) displays a linear behaviour until 730°C. The $A_c_1$ temperature, marks the beginning of the ferrite-to-austenite transformation and, by continue increasing the temperature, a volume contraction of the sample is observed. The contraction is the consequence of the continuous ferrite-to-austenite transformation and occurs during the final part of the heating stage as well as during the isothermal holding at 780°C. Afterwards, during the cooling of the sample, the thermal contraction follows also a linear behaviour although the linearity is slightly deviated; first within the region between 600 and 700°C and second, at temperatures lower than 200°C. The first deviation suggests both the formation of bainite [24] and the epitaxial growth of ferrite [25], while the second one is attributed to the martensite transformation. The occurrence of the bainitic and martensite transformations is corroborated by SEM micrographs and EBSD maps. The non-indexed regions in the IPF-map of Figure 5.4a (black coloured in the IPF map) correspond to martensite, denoted as $\alpha'$. The reason of this low indexation rate is the poor pattern quality of martensite produced by the high density of lattice defects, such as dislocations [26]. Bainite is composed of bainitic ferrite plates separated by blocks of martensite. This configuration is similar to the bainite of low carbon steels subjected to a quenching and partitioning heat treatment, as Santofimia et al. [25] observed in samples with large partitioning times. Although the epitaxial ferrite was not identified in the SEM micrographs, its existence was supported by the difference in martensite content between a dilatometer sample cooled at 210°C/s and the reference DP steel.
Failure micromechanisms in DP steels

Despite the fact that the reference and the slow heated (SH) DP steels have markedly different ferrite grain sizes; the type and amount of the other microstructure constituents are similar. The dilatometric curve of the SH specimen is comparable to the corresponding curve of the reference DP steel, showing a slight linear deviation between 600 and 700°C and at lower temperatures. SEM and EBSD data show that the microstructure of the SH DP steel is also composed of ferrite, martensite and bainite. In the case of bainite, alternating plates of bainitic ferrite and martensite blocks are also observed (cf. the ND IPF map of Figure 5.4a).

![Figure 5.4: Dilatometric curves of the intercritical heat treatments applied to (a) the Reference, (b) the Slowly Heated, (c) the Slowly Cooled and (d) the High Temperature DP steels. Each curve is accompanied of a SEM micrograph and a ND IPF map of the martensite or bainite of the corresponding DP steels. The segments of the dilatometric curves enclosed by a circle in (c) and (d) highlight the occurrence of bainitic transformation.](image)

The DP steel after slow cooling displays more differences with respect to the reference DP steel. The segments of the dilatometric curves corresponding to the heating
and isothermal holding of the SC specimen are the same as in the case of the reference DP steel (cf. Figure 5.4c). However, during the cooling, a large volume expansion is observed between 600 and 700°C, indicating that a significant amount of austenite has transformed to bainite. Some martensitic regions are still observed in the EBSD maps either as islands located at the ferrite grain boundaries or as blocks within the bainite, although the sizes are smaller than in the reference DP steel. In some bainite grains, similar to the ones observed in the SEM micrograph of Figure 5.4c, traces of carbide precipitation are observed. Interestingly, the EBSD maps revealed the existence of a small amount (~ 0.2%) of retained austenite (cf. Figure 5.4c -the grain shown in the ND IPF map and marked as $\gamma$). The retained austenite grains have similar size to those of the martensitic islands and they are mostly located at the ferrite grain boundaries.

Similarly to the SC specimen, the high temperature (HT) DP steel also displays a significant phase transformation during cooling in the range between 600 and 700°C, as can be seen in the dilatometric curve of Figure 5.4d. Nevertheless, the resulting microstructural constituents are significantly different from the rest of the DP steels. Although the microstructure is also composed of bainitic ferrite and martensite, the size of the bainite packets is in average larger than in the other DP steels and comparable to the ferrite grain size (cf. Figure 5.4d SEM and EBSD images). As in the case of the SC DP steel, the HT specimen also contains a small amount (~ 0.04%) of retained austenite with an average size of ~0.3 μm.

The partition of chemical elements within the microstructural constituents is another factor affecting the mechanical properties of DP steels. Electron Probe Microscopic analysis (EPMA) is used to examine the partitioning of carbon, manganese, silicon and chromium. Figure 5.5 displays an example of the EPMA analysis performed on the slowly cooled DP steel. In the case of the carbon distribution along the EPMA line (cf. Figure 5.5a), concentration gradients are clearly observed with concentration maxima, that can reach values as high as five times the nominal carbon content of the as-received cold rolled sheet (0.092%). The regions with carbon content lower than the nominal amount are regarded as ferrite whereas the peaks of carbon are attributed to either martensite, bainite or retained austenite. The other chemical elements, like chromium and silicon are more homogeneously distributed among the microstructural constituents (cf. Figure 5.5b). The manganese content varies between 1.5% and 2.5% where the higher values well coincide with the carbon peaks. Nonetheless, the peaks of manganese are found to reach values as high as 7.5%, as can be observed in Figure 5.5b. The reason of this high concentration of Mn is the presence of elongated manganese sulphide (MnS) stringers, which are usually oriented along the rolling direction. The
SEM micrograph of the region where the EPMA was performed (cf. Figure 5.5c) confirms the presence of these inclusions.

**Figure 5.5**: EPMA profile of the SC DP steel. (a) Carbon distribution along the EPMA line together with an indication of the bulk carbon content (gray line) of the as-received material (Chapter 3). (b) Manganese, chromium and silicon distribution along the EPMA line. The error bars correspond to the counting error of the technique. (c) SEM micrograph of the region where the EPMA line (black arrow) was performed. A manganese sulphide (MnS) inclusion is crossed by the EPMA line.

In general, all DP steels show similar concentration gradients along the microstructure although the relative amount differs. Table 5.2 displays the average percentage of each element based on the EPMA lines as well as the average maximum carbon values (concentration peaks) for all DP steels. The homogeneity of the silicon and chromium concentrations along the EPMA lines can be estimated by the low standard deviation of those elements, which are of the same order of magnitude as the estimated measurement error (0.01%). The higher scattering of the manganese data is attributed to both the MnS inclusions as well as the concentration gradients of the
Chapter 5

microstructural constituents. Based on the standard deviation one can conclude that the carbon content distribution is the most heterogeneous element along the EPMA lines due to the different carbon content of the ferrite and the martensite or bainite. It is important to highlight that the variations of the carbon content are larger than the instrumental error (0.01%) and are even similar to the average values. Among the DP steels, the SC specimen shows the minimum carbon average value. This comparison is in contrast with the maximum carbon peaks of 0.48%C measured in the same DP steel. This behaviour suggests that although the SC specimen contains smaller martensite or bainite fractions (cf. Table 5.1), these constituents have a higher carbon concentration in comparison to the other DP steels, which is in agreement with the observations made on the EBSD analysis where even retained austenite is observed as a result of carbon stabilization.

Table 5.2: Average chemical content along the EPMA measurements together with their standard deviation. The maximum carbon peaks is the average value of the maximum amount of carbon along each EPMA line.

<table>
<thead>
<tr>
<th></th>
<th>Ref</th>
<th>SH</th>
<th>SC</th>
<th>HT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon content, %</td>
<td>0.11</td>
<td>0.13</td>
<td>0.09</td>
<td>0.1</td>
</tr>
<tr>
<td>±0.09</td>
<td>±0.14</td>
<td>±0.11</td>
<td>±0.08</td>
<td></td>
</tr>
<tr>
<td>Maximum carbon peaks, %</td>
<td>0.33</td>
<td>0.37</td>
<td>0.48</td>
<td>0.31</td>
</tr>
<tr>
<td>±0.04</td>
<td>±0.03</td>
<td>±0.09</td>
<td>±0.09</td>
<td></td>
</tr>
<tr>
<td>Manganese content, %</td>
<td>1.85</td>
<td>1.85</td>
<td>1.88</td>
<td>1.84</td>
</tr>
<tr>
<td>±0.21</td>
<td>±0.1</td>
<td>±0.51</td>
<td>±0.26</td>
<td></td>
</tr>
<tr>
<td>Silicon content, %</td>
<td>±0.01</td>
<td>±0.01</td>
<td>±0.02</td>
<td>±0.01</td>
</tr>
<tr>
<td>Chromium content, %</td>
<td>0.59</td>
<td>0.59</td>
<td>0.59</td>
<td>0.58</td>
</tr>
<tr>
<td>±0.02</td>
<td>±0.02</td>
<td>±0.03</td>
<td>±0.02</td>
<td></td>
</tr>
</tbody>
</table>

In addition to the microstructural constituents, DP steels also contain inclusions which were identified to consist primarily of two types. The elongated inclusions, which were frequently observed in the microstructure (cf. Figure 5.5c and Figure 5.6a and c), were identified by EDS as manganese sulphides (MnS) stringers. The MnS stringers are usually located within and along the segregation bands. Another characteristic of the MnS stringers is that some of them are found fragmented in smaller pieces along their length, possibly as a consequence of the rolling process. The second type of inclusions recognized in the material are the aluminium oxides (Al₂O₃). Figure 5.6a shows a SEM micrograph of this type of inclusion and Figure 5.6c displays the EDS spectrum of an alumina inclusion. Similar to the IF steel of the previous chapter, the shape of these
Inclusions is ellipsoidal and, in some of the cases, the Al$_2$O$_3$ are observed together with the MnS stringers; e.g. the SEM micrograph of Figure 5.6a. However, the Al$_2$O$_3$ inclusions are fewer and smaller in comparison to the MnS.

The populations of both types of inclusions are expected to be the same among the DP steels. The reason is that the initial material is common to all the DP steels and moreover these inclusions remain unaffected by the subsequent heat treatments.

![SEM micrograph of the Reference DP steel which contains two types of particles. X-ray EDS analysis identified the inclusions as (b) Al$_2$O$_3$ and (c) MnS.](image)

In summary, the different heat treatments mainly affected the content and structure of the second phase, i.e. martensite and bainite. With regard to the ferrite, the principal consequence of the heat treatments was observed on variations of the grain size, whereas the texture only showed minor differences among the DP steels. The overall changes in the microstructure of the specimens have an influence on the Vickers microhardness, especially in the slow cooled DP steel. A further analysis on the relation between the mechanical properties and the microstructure of the different heat treated DP steels based on the tensile tests is made in the following section.

### 5.3 Mechanical properties

The tensile stress-strain curves of all DP steels are displayed on Figure 5.7a, and a summary of the principal properties acquired from the tensile tests is given in Table 5.3. All of the curves showed a continuous yielding behaviour, which is one of the characteristics of DP steels. The ultimate tensile strength (UTS) of the reference (Ref), the slow-heated (SH) and the high-temperature (HT) dual phase steels is above 600 MPa, whereas the slow-cooled (SC) showed the lowest strength which is even below the established standard value for the DP600 steel grade. It is important to highlight that the
decreasing order followed by the tensile strength of the DP steels is the same as the Vickers microhardness values of Table 5.1; i.e. the higher Vickers microhardness and UTS for the HT DP steel and the lowest corresponding values to the SC DP steel.

In terms of ductility, all steels display similar elongation at \( UTS \) of \( \sim 13\% \) and at fracture \( (\sim 17\%) \), although the elongation at fracture of the reference material is slightly higher (18.2\%) than the other DP steels.

The incorporation of the fracture strain and stress values based on the fracture surface into true stress-strain curves gives additional information on the deformation behaviour of the DP steels, as can be seen in Figure 5.7b. The solid portion of the curves represents the true stress and the logarithmic strain calculated from the tensile test data using the uniform deformation region. The dashed line corresponds to a linear interpolation between the beginning of the necking and the final fracture point. The most remarkable difference between the data represented in Figure 5.7a and b is that the SC DP steel seems to undergo larger post-necking plasticity in comparison to the other steels. In addition, all DP steels display comparable true fracture stress \( (\sigma_F) \) values, an observation which contrasts to the different values of the ultimate tensile strength displayed in Figure 5.7a. The reason for this discrepancy is attributed to the higher macroscopic deformation localization (necking) of the slow cooled DP steel after the uniform deformation stage. After the samples are deformed beyond the corresponding strain at \( UTS \), localization of the deformation starts taking place at a reduced region of the sample while the surrounding regions remain almost unaltered. As a consequence of the longitudinal deformation within the localized zone, the transversal section initiates to decrease and thus the associated stress increases on that area. In the case of the SC DP
steel, this localization is more pronounced, i.e. the transversal section reaches a lower area than in the other specimens, and fracture occurred at local stresses similar to the other steels even if macroscopically the engineering stresses at fracture are dissimilar. Based on the previous explanation, one parameter which can be used as an indicative of the degree of strain localization on tensile samples is the ratio of the reduction of area at fracture, RA, to the deformation at UTS (cf. Table 5.3). Based on this parameter, it can be noticed that the SC specimen has the highest ratio (in average 4.8), indicating a higher degree of strain localization after the necking onset in comparison to the other DP steels. The HT DP steel is the second specimen with the highest degree of strain localization (an average value of 4.3) and has also the highest true fracture stress due to the relatively larger reduction of area attained at fracture.

<table>
<thead>
<tr>
<th>Ref</th>
<th>SH</th>
<th>SC</th>
<th>HT</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2% Offset yield strength, MPa (S0.2)</td>
<td>248</td>
<td>241</td>
<td>214</td>
</tr>
<tr>
<td>±2.7</td>
<td>±6.6</td>
<td>±2.76</td>
<td>±7.4</td>
</tr>
<tr>
<td>Ultimate Tensile Strength, MPa (UTS)</td>
<td>723</td>
<td>693</td>
<td>568</td>
</tr>
<tr>
<td>±7.1</td>
<td>±2.8</td>
<td>±3.8</td>
<td>±7.3</td>
</tr>
<tr>
<td>Deformation at UTS, %</td>
<td>13.4</td>
<td>13.3</td>
<td>13.7</td>
</tr>
<tr>
<td>±0.3</td>
<td>±0.2</td>
<td>±0.2</td>
<td>±0.6</td>
</tr>
<tr>
<td>Deformation at Fracture, %</td>
<td>18.2</td>
<td>17.9</td>
<td>17.6</td>
</tr>
<tr>
<td>±0.6</td>
<td>±0.9</td>
<td>±0.3</td>
<td>±0.8</td>
</tr>
<tr>
<td>Reduction of Area at Fracture, % (RA)</td>
<td>50</td>
<td>49</td>
<td>65</td>
</tr>
<tr>
<td>±1.3</td>
<td>±3</td>
<td>±0.9</td>
<td>±2.1</td>
</tr>
<tr>
<td>Ratio of Reduction of area at fracture to deformation at UTS</td>
<td>3.7</td>
<td>3.7</td>
<td>4.8</td>
</tr>
<tr>
<td>±0.06</td>
<td>±0.2</td>
<td>±0.08</td>
<td>±0.3</td>
</tr>
<tr>
<td>True fracture strain (εF)</td>
<td>0.69</td>
<td>0.68</td>
<td>1.06</td>
</tr>
<tr>
<td>±0.03</td>
<td>±0.06</td>
<td>±0.03</td>
<td>±0.04</td>
</tr>
<tr>
<td>True fracture stress, MPa (σF)</td>
<td>1236</td>
<td>1169</td>
<td>1260</td>
</tr>
<tr>
<td>±27</td>
<td>±63</td>
<td>±14</td>
<td>±37</td>
</tr>
</tbody>
</table>

The strain localization is a consequence of the plastic instabilities occurring during deformation, which in turn depends mainly on the strain hardening behaviour of the
Chapter 5

material [27]. The strain hardening $\Theta$ (equation 3.2 in Chapter 3) for all the DP steels as a function of the true stress is displayed in Figure 5.8a. The limits of the curve correspond to the yield strength ($R_{0.2}$) as a lower limit, whereas the upper limit is determined by the ultimate tensile strength (UTS), which signifies the end of the uniform deformation. The larger values for the initial strain hardening of the specimens correspond, in decreasing order, to the HT DP steel, followed by the reference and slowly heated specimens and, finally, to the SC sheet. In the particular case of the SC DP steel, the strain hardening curve can be decomposed into two sections where the initial part displays the highest decreasing rate among all DP steels and, in contrast, the second part exhibits the lowest decrease of the hardening rate (cf. Figure 5.8a). A similar behaviour is observed in the other DP steels: the highest decreasing rate of the strain hardening occurs at the yield strength. However, the transition between the different hardening rates is smoother and more diffused for the Reference, the SC and the HT specimens in comparison to the slowly cooled DP steel.

The strain hardening behaviour of the DP steels was also analysed using the differential Crussard-Jaoult analysis, $D_{C-J}$, in which the logarithm of the strain hardening rate, $\Theta$, is plotted as a function of the logarithm of true plastic strain [28]. This type of analysis offers the advantage of typically delineating three stages of the strain hardening behaviour of DP steels [29, 30], which can be associated to the substructures created within the microstructure during deformation [31]. Figure 5.8b presents the strain hardening behaviour of the DP steels by the $D_{C-J}$ method. This representation also

![Graphs showing strain hardening behaviour for DP steels](image)
reveals an obvious difference in the hardening behaviour between the SC DP steel and the other three specimens. The distinction lies in the presence of three regions in the hardening curve of the SC DP steel while the other steels display similar curves with only two slightly different stages (cf. Figure 5.8b). Lawson et al. [29] observed a similar effect of the cooling rate on the hardening behaviour where the three regions of the strain hardening curve become more prominent as the cooling rate in the intercritical annealing decreases. On the other hand, they also showed experimental evidence that the three strain hardening stages give the impression to collapse into one single region at high enough cooling rates (~1000 °C/s [29]). In this case the Ref, the SH and the HT DP steels represent an intermediate case, where two strain hardening stages are still present although with a diffuse demarcation between the remaining stages. The common point among all DP steels is the similar decrease of the strain hardening rate in stage III, which occurs within the same strain range. This suggests that similar deformation mechanisms are taking place at the end of the uniform deformation.

In summary, the microstructural differences created by slowing down the heating rate (SH) or by increasing the intercritical soaking temperature (HT) with respect to the reference DP steel have a weak influence on the strain hardening behaviour in comparison to reducing the cooling rate (SC) after the intercritical annealing. Additional information about the peculiar mechanical behaviour of the SC specimen is obtained from the fracture surfaces of the tensile specimens as discussed in the following subsection.

5.3.1 Macroscopic failure mechanisms and fractography

The macroscopic mechanisms of fracture of the DP steels differ from the IF steel in the last stages of deformation. Although the DP steel samples also went through diffuse necking after uniform deformation, the location of the necking occurred randomly along the tensile direction instead of taking place in the middle length of the tensile sample, as in the case of the IF steel. Most frequently, the diffuse necking started close to the fillets (rounded section which connects the grip to the strain gage sections) of the tensile sample. Furthermore, the final failure occurs within the region of the diffuse necking and without the appearance of localized necking. As a consequence of the absence of localized necking, the fracture surface of all samples is roughly oriented perpendicular to the tensile direction, or in other words, parallel to the TD-ND plane of the rolled sheet (cf. Figure 5.9a). The details of the longitudinal cross-section of all DP steels near the fracture surface in Figure 5.9b reveal that the fracture profile forms a zigzag pattern. Each segment of such pattern is oriented approximately ±45° with respect to the tensile
direction, which is parallel to the rolling direction of the samples. In this chapter, the term *point of fracture* is used hereafter to refer to the intersection of two segments of the zigzag geometry of the fracture profile. It is important to mention that this term is applied based on the cross-section images of the broken samples (ND-RD plane) since these points are in reality the intersection of linear features of the fracture surface with the cross-section plane of the sample, as can be inferred from the scheme of Figure 5.9a. Apart from the slowly cooled DP steel, the fracture profiles of the broken samples display more than one point of fracture without any preference of the location along the thickness direction. Particularly noteworthy is the fact that the SC DP steel always shows only one point of fracture which is located in the middle thickness of the sample. In addition, the high area reduction reported in Table 5.3 for this DP steel is more discernable than in the case of the other materials.

Figure 5.9: (a) Schematic representation of the fracture surface of all DP steels. (b) OM micrographs of the broken sample profiles (c) together with the respective SEM fractographs of the DP steels. The OM profiles correspond to the middle width of the tensile samples. The arrows indicate the points of fracture; i.e. the intersection of two segments of the zigzag geometry.

One common characteristic of all DP steels is the thickness variation of the fracture surface along the width of the tensile samples. The minimum thickness in all cases is located in the middle width, whereas the maximum value corresponds to the edges of the sample. This observation implies that the samples produce a deformation gradient along the transverse direction after the onset of necking (end of the uniform
deformation), whereby the maximum thickness strain occurs in the mid-width of the sample.

Figure 5.10: SEM fractographs of the DP steels showing typical features of the fracture surface. a) Reference DP steel. b) Slow Heated DP steel. c) Slow Cooled DP steel. d) High Temperature DP steel.

One first approach used to identify the differences in the failure micromechanisms of the DP steels is based on the characteristics of the fracture surfaces. Figure 5.10 displays the fractographs of the DP steels at relatively high magnification. Similar to the IF steel, the fracture surfaces of all DP steels exhibit typical features of ductile failure with a dimpled topography. Based on the size and distribution of the dimples, two different populations are distinguished for all specimens. In one case, the dimples are larger, deeper and elongated along the transverse direction of the tensile samples. The interior of the dimple walls exhibits serpentine glide; i.e. marks associated to the deformation process [32]. In contrast to the fracture surface of the IF steel, particles are seldom observed in the interior of the dimples. The second type of dimples population is characterized by smaller sizes and they are shallower. They cover most of the fracture surface of the broken sample. It is important to mention that no cleavage facets were
observed on the fracture surfaces of any of the DP steels, as in the case of Ahmad et al. [18] where a mixture of cleavage and ductile failure mechanisms was reported.

In general, the SEM fractographs reveal similar failure mechanisms in all DP steels. No specific detail was observed on the fracture surface which can be used to make a distinction between the materials in terms of failure mechanisms. A further understanding of the voids development and its relation to the microstructure is accomplished by analysing the cross-section of the tensile samples.

### 5.4 Failure micromechanisms

The analysis of the fracture surface only provides information about the last stage of a series of events occurring within the microstructure during the deformation history. In order to obtain a deeper understanding of the evolution of the failure micromechanisms, the microstructure of the DP steels was analyzed at three different deformation stages: 

1. the onset of necking,
2. deformation previous to the final failure and
3. after fracture.

Therefore, tensile samples were deformed at different strains in order to obtain these three conditions for each of the DP steels. Figure 5.11 displays a scheme summarizing the three types of employed tensile samples as well as the zones which were analysed.

The samples deformed up to the onset of necking are of interest because those conditions correspond to the last stage of uniform deformation before macroscopic strain localization takes place. The strain at which the onset of necking occurs is similar for all DP steels (deformation at UTS in Table 5.3) and is approximately equal to $e=12.5\%$.

In the case of the samples with conditions ii) and iii), the region away from the necked region exhibits similar characteristics as to the last stage of uniform deformation. These regions were also analyzed and compared to the sample deformed up to the onset of necking.
The analysis performed in the regions of interest for each of the samples were intended first to study the deformation of the microstructure and second for the characterization of the void initiation mechanisms and its quantification.

### 5.4.1 Heterogeneous deformation of the microstructure

Based on the results obtained from the IF steels (Chapter 4), it was inferred that the local deformation of the ferrite grains serves to accommodate the strain incompatibilities between the matrix and the particles. In the case of DP steels, a similar behaviour takes place between the ferrite grains and the martensite/bainite islands. However, the large fraction of particles produces an interaction between the particle deformation zones and the ensuing development of grain substructures. Therefore, EBSD analysis was performed in order to study the development of the grain substructures of the ferrite matrix during the fracture process from the onset of necking until the final failure of the tensile sample. All EBSD maps were cleaned up using a single iteration of the “Grain Dilation” algorithm defined by the TSL-OIM® software. The grain tolerance angle was set to 10° and pixel clusters with more than 50 points were considered grains.

It is usually assumed that ferrite in DP and IF steels deforms in a similar way. However both steels develop different substructures during deformation as pointed out by Dillien et al. [33]. They observed that the deformed microstructure of a DP steel consisted of only large-range orientation gradients and, in contrast to IF steels, the absence of dense dislocation walls (DDW). Contrary to those results, Ohtani et al. [34] reported the formation of well-defined DDW within grains as a product of plastic deformation. In this dissertation, both large- and short-range orientation gradients are observed in the deformed samples of all DP steels. One example of grain substructures created during deformation is shown in the crystal orientation maps of Figure 5.12 and Figure 5.13. Both figures correspond to the SH DP steel deformed up to 12.5% and to a strain previous to fracture, respectively. As can be observed in the kernel average misorientation (KAM) map of the sample with uniform deformation (Figure 5.12b), DDW are distributed over the entire grain interiors. One of the principal characteristics of the grain substructures is that the DDW with higher misorientation angle are located in the proximities of the martensite, giving the impression that they emanate from the martensite. This observation strengthens the hypothesis of the strain heterogeneity occurring on the interface between the ferrite grains and the martensite where the ferrite grains create substructures in order to accommodate the strain incompatibility. The Geometrically Necessary Density of Dislocations (GND) map of Figure 5.12c illustrates similar behaviour of the ferrite during deformation; i.e. grain substructures distributed
within the grains where some of the DDW have a high content of GND density close to $10^{18} \text{ m}^{-2}$.

Based on the crystal orientation maps of Figure 5.13, it was estimated that the amount of grain substructures within the necked region is higher than within the uniform deformation zone. Although the GND density content of the grain substructures are also close to $10^{18} \text{ m}^{-2}$ as in the uniform deformation region, the amount of the substructures with that higher dislocation content is higher; cf. Figure 5.13c. In addition, the grains of the microstructure are more elongated along the tensile direction.

The deformation of all DP steels is similar to the HT DP steel described previously although the individual quantitative characteristics for each specimen are slightly different. In order to compare the evolution of the substructures between the different DP steels, the average $SOS$ and $KAM$ parameters (Appendix A) were calculated based on the crystal orientation maps, which were measured at different stages of deformation of the DP steels. The results are summarized in the plot of Figure 5.14. Similar to the results from IF steels, the large scatter of both $KAM$ and $SOS$ parameters (cf. to the error bars on Figure 5.14) are associated to the different deformation of each individual grain. However, it is possible to discern for all DP steels that the regions close to the fracture surface and within the necking zone generate larger long-range orientation gradients,
Failure micromechanisms in DP steels

based on the $SOS$ parameter, and larger short-range orientation gradients, $(KAM$ parameter), in comparison to the regions with uniform deformation.

Figure 5.13: Heated DP steel deformed up to necking. (a) ND inverse pole figure. (b) Kernel average misorientation map using the first closest neighbours. (c) GND map with color-code given in logarithmic scale. The step size of the map is 0.1 μm.

It was surprising to observe that the necked region showed higher short- and long-range orientation gradients than the broken specimens. This effect is attributed to (i) the large scatter of the $SOS$ and $KAM$ parameters between grains affecting the average value and (ii) the fragmentation of the grains at large strains. At some point of the deformation of the samples, the grain substructures produce fragmentation of the original grain which becomes more severe at higher strains. Close to the fracture surface, some grain substructures boundaries produce crystal rotations with misorientation angles similar to the values of the original grain boundaries. This similarity makes it difficult to reconstruct the original grain based on the crystal orientations because some grain substructure boundaries with higher misorientation angle (higher than 10°) are considered as grain boundaries. As a consequence, the KAM values corresponding to these grain substructure boundaries (which are confounded by grain boundaries) are excluded from the average grain KAM, which produces a decrease of the value of this parameter. In the case of the average SOS, the lower values on the region closer to the fracture surface are attributed to the partition of the original grain.

Table 5.4 displays the average grain GND density content for the DP steels corresponding to the regions with uniform deformation and necking. The data related to the uniform deformation region correspond to an interrupted tensile test deformed up to $\varepsilon = 0.118$. Regarding to the GND density in the necking region, the data were acquired
from samples deformed at strains close to fracture where the maximum deformation and stress within the necked region are also displayed in Table 5.4. The values for the zones close to the fracture surface are not included due to the severe plastic deformation which makes it difficult to reconstruct the grains. As a general result, the average GND density fraction is increased twice between the onset of necking and just before final failure.

**Table 5.4: Average GND density per grain for all of the DP steels.**

<table>
<thead>
<tr>
<th>GND, m⁻²</th>
<th>Ref</th>
<th>SH</th>
<th>SC</th>
<th>HT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uniform Deformation (ε = 0.118)</td>
<td>1.8·10¹⁵</td>
<td>2.5·10¹⁵</td>
<td>2.2·10¹⁵</td>
<td>2.9·10¹⁵</td>
</tr>
<tr>
<td>Necking</td>
<td>3.6·10¹⁵</td>
<td>4.1·10¹⁵</td>
<td>3.7·10¹⁵</td>
<td>3.7·10¹⁵</td>
</tr>
<tr>
<td>ε = 0.63</td>
<td>ε = 0.71</td>
<td>ε = 0.84</td>
<td>ε = 0.75</td>
<td></td>
</tr>
<tr>
<td>σ=1233 MPa</td>
<td>σ=1269 MPa</td>
<td>σ=1194 MPa</td>
<td>σ=1375 MPa</td>
<td></td>
</tr>
</tbody>
</table>

**5.4.2 Void initiation sites**

In general, two void nucleation sites were observed in the cross-section of all DP steels: the inclusions and the martensite/bainite phase. A significant difference exists between both void initiation sites. As the content of inclusions such as Al₂O₃ and MnS is extremely low, the number of voids nucleated on those sites is also extremely low in comparison to the voids nucleated at the martensite/bainite phase. As indicated in the introduction of this chapter, other researchers have also reported similar observations [4, 7-12]. Moreover, as all DP steels were heat treated from a common as-cold rolled sheet, the bulk chemistry as well as the type and amount of inclusions are expected to be the same for all the specimens. Thus, any difference in the fracture process between the DP steels cannot be attributed to the inclusions.
Based on the observations made on the cross sections of the tensile samples deformed up to 12.5%, it was found that the void nucleation occurs previous to the initiation of necking (cf. Figure 5.15).

The identified mechanisms of void initiation related to martensite/bainite can further be classified as internal fragmentation of the martensite/bainite or interface decohesion of the martensite/bainite from the ferrite matrix. The term internal fragmentation is applied to the cases where martensite or bainite is observed in both sides of the voids along the tensile direction although the exact mechanisms are not really clear. The problem is ascribed to the difficulty in the discrimination between

Figure 5.15: SEM micrographs representing some examples of void initiation in all DP steels by constituent fragmentation (Fr) or by ferrite-martensite interface decohesion (ID). The micrographs corresponds to the (a) Reference, (b) SH, (c) SC and (d) HT DP specimens. The samples were deformed up to 12.5%, prepared for metallography and slightly etched with Nital 2%. Martensite appears as light gray colour while the ferrite has a darker gray colour.
different martensite islands. For instance, the geometry of the martensite islands from both sides of the void in Figure 5.15b suggests that two different martensite islands were separated during deformation instead of the fragmentation of a single martensite island. A different situation is observed twice in the central area of Figure 5.15d where the thickness of the martensite islands on both sides of the void are of similar size. This situation suggests that the fragmentation of the elongated martensite island occurs in two points along a single martensite island, although the other mechanism is not discarded. Whether the separation of two adjacent constituent islands occurs or whether the fragmentation of one constituent island is taking place remains unknown. However, the voids in both situations inherit the thickness of the martensite at the point where the void was nucleated.

In the case of the interface decohesion, the voids were observed to nucleate either at the intersection of a ferrite grain boundary with the martensite (e.g. Figure 5.15a) or on the interface between a single ferrite grain and the martensite (e.g. Figure 5.15c and d). In both situations, the void area is in general smaller than in the case of internal fragmentation, and this size difference is due to the thickness of the voids. As in the case of voids nucleated at TiN particles (Chapter 4), the growth of the voids occurs only along the tensile direction, whereas the thickness and the width of the voids keep the same length as at the instant of the nucleation of the void. The thickness, or width, of the void nucleated by interface decohesion usually occurs along a small region of the martensite/bainite interface (e.g. Figure 5.15c) instead of the entire thickness of the martensite/bainite. Therefore, although the voids from both initiation mechanisms may grow in similar lengths along the tensile direction, the voids nucleated by fragmentation create in average a larger void area in comparison to the voids nucleated along the ferrite-martensite/bainite interface due to the difference on the void thickness and width.

The amount of voids created at $e=12.5\%$ is low and they are uniformly distributed over the cross-section of the DP steels. As the voids have limited growth at this deformation stage, the dimensions of the voids are confined to the microstructure scale; i.e. the thickness of the voids is equal or smaller than the thickness of the martensite/bainite where the void was initiated and, in addition, the length of the voids along the deformation direction is similar to the thickness of the voids.

As it was mentioned in the previous section, the tensile samples of all DP steels developed macroscopic deformation localization at strains beyond the ultimate tensile strength; i.e. necking. The inspection of the region out of the necked zone in the broken and necked tensile samples showed that equivalent void initiation mechanisms took place as in the samples deformed up to $e=12.5\%$. This similarity is not surprising as the
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The necked region accommodates most of the deformation once the necking commences while the rest of the tensile sample does not undergo any additional deformation. This is the reason why the zone away of the necked region preserves the characteristics of the last stage of uniform deformation.

Additional information on the void initiation sites was obtained from the crystal orientation maps by EBSD. Figure 5.16a display the SEM micrograph of a region in the Reference DP steel containing three voids in the microstructure. The martensite or bainite cannot solely be distinguished from the SEM micrograph because the sample was not chemically etched with Nital, in order to avoid a detrimental effect on the quality of the EBSD patterns. However, the band contrast (BC) map of Figure 5.16d allows identifying the microstructural constituents without being disturbed by grain substructures. For instance, the large region with the $\langle 001 \rangle$ direction parallel to the ND (red region enclosed by the ellipse in Figure 5.16b) is composed of bainite (bainitic ferrite with martensite laths) grains.

![Figure 5.16: Region containing three voids corresponding to the sample deformed to 12.5% of the Reference DP steel. (a) SEM image. (b) ND inverse pole figure combined with band contrast map. (c) Kernel average misorientation map using the first closest neighbours. (d) Band contrast map. F: ferrite. B: bainite. The step size of the map is 0.1 $\mu$m.](image)

In addition, the KAM map of Figure 5.16c also displays the orientation gradients produced by the bainitic transformation although the deformation of the microstructure has also an additive influence. The three voids are located in the boundary between grains of bainite (below the voids) and ferrite (above the voids). Furthermore, void 2 is situated between two martensite islands, which are also located along the ferrite-bainite
boundary. This situation suggests the occurrence of martensite fragmentation as mechanism for void initiation. However, based on the few indexed points on both martensite islands, two different crystal orientations on both sides of the void were observed which suggests decohesion between different martensite islands rather than fragmentation of the same martensite island. In the case of void 1, martensite is recognized on the right side of the void whereas bainitic ferrite is distinguished on the other side.

![Figure 5.17: Region containing four voids corresponding to the sample deformed 12.5% of the Slow Cooled DP steel. (a) SEM image. (b) ND inverse pole figure combined with band contrast map. (c) Kernel average misorientation map using the first closest neighbours. (d) Band contrast map. F: ferrite. B: bainite. The step size of the map is 50 nm.](image)

The coarser structure of the bainite for the SC DP steel allows a better identification of the initiation sites in comparison to the finer structure of the Reference DP steel. The SEM image of Figure 5.17a shows four voids in a region of the sample deformed up to 12.5% of the SC DP steel. The crystal orientation maps of the corresponding region demonstrate that all voids are surrounded by bainitic regions. The bainitic ferrite on both sides of the voids has in all cases different orientations, which
Failure micromechanisms in DP steels supports the idea of decohesion of the bainite packets rather than fragmentation or fracture of the bainite.

Similar situations are observed in the orientation maps of all DP steels deformed up to the limit of the macroscopic uniform deformation. It can be said in general that void initiation occurs by the separation of the interfaces of martensite, bainite packets and ferrite.

In contrast to the uniform deformation region of the tensile sample, the necked region suffers the highest critical conditions along the whole tensile sample in terms of local stress and strain. The micrographs in Figure 5.18 correspond to the necked region of samples which were deformed just one step previous to final fracture (cf. Figure

![SEM micrographs of the tensile sample deformed up to almost fracture and corresponding to the central region of the necking. Samples etched with Nital 2%. (a) Reference, (b) SH, (c) SC and (d) HT specimens. The black arrows point out the voids influenced by the plastic flow of the ferrite.](image-url)
5.11b). In terms of void characteristics, the principal differences between the necked region and the uniform deformation zone are the larger sizes and the increased amount of voids. The enlargement of the voids is mainly the result of void growth which shows a unidirectional character. The enlargement of the voids mainly occurs along the tensile direction as a consequence of influence of tensile deformation over void growth. Nevertheless, in some cases the void growth direction has deviated from the tensile direction and is controlled by the heterogeneity of the microstructure as well as the direction of the local deformation of the ferrite. One example of this is indicated by an arrow in Figure 5.18d where the growth of the void follows the curved path imposed by the ferrite plastic flow.

Similar to the uniform deformation region, the extent of the void thickness in the necked zone is less or equal to the thickness of the martensite where the void was nucleated. This observation reinforces the unidirectional character of the void growth under tensile deformation where the directions perpendicular to the tensile direction show no void growth. However, the void thickness is in average larger within the necked region than in the uniform deformation zones, and is not a consequence of void growth but rather of void nucleation. As in the case of the IF steel, void nucleation is a continuous process during the deformation history whereby the nucleation of voids occurs at larger martensite/bainite sizes and hence creating voids with larger thickness as the deformation increases.

So far the void process described in the previous paragraphs is confined to the scale of the microstructural constituents; in other words, the void initiation is linked to a single martensite/bainite island and there is no interaction between the voids during their growth. This is the case for the majority of the voids during the entire deformation history. However, coalescence of voids is observed in some limited cases, and one of these examples is the void pointed at in Figure 5.18c. The two coalesced voids are distinguishable from each other whereby the coalescence occurs as a consequence of the void growth.
In addition, another type of voids is observed within the region of higher deformation of the samples. The extension of this type of voids is in most of the cases larger than several grains; for example, the voids marked by an arrow in Figure 5.18a and b. The characteristics of these voids are their higher aspect ratio values and their inclination of approximately 45° with respect to the tensile direction. Unlike the case of void coalescence, the individual voids are not distinguished from each other on their coalescence path. These voids are hereafter referred to as cracks with the purpose of differentiating them from the voids growing along the local plastic flow. On the one hand, the inclination of these voids along the grains is not affected by the different crystal orientations of the grains and therefore seems to be independent of the grains. On the other hand, the extremes of the voids coincide with the grain boundaries. Consequently, though the inclination and trajectory of the voids are not influenced by the grains, the ending of the voids is demarked by the grain boundaries.

The broken tensile samples also show this type of cracks although the dimensions of these voids are larger and cross several grains. The majority of the cracks are situated close to the fracture surface and one example of this type of cracks on the Reference DP steel is displayed in Figure 5.19. It can clearly be seen in the micrograph of Figure 5.19a that the
enclosed crack has the same orientation as the fracture surface indicated by the arrow. In fact, the crack seems to be the continuation of the fracture surface pointing towards the interior of the sample. It is important to highlight that the crack is directly connected to a “point of fracture” (cf. Figure 5.9b). The fracture surface and the crack are separated by a portion of material, although it is unknown if they are interconnected underneath the surface of observation. At higher magnification (cf. Figure 5.19b), the crack crosses several ferrite and martensite/bainite grains without modifying the path. Although it is possible to observe the occurrence of coalescence of the crack with voids, the major segment of the crack seems to be constituted of only one element. The void enclosed by a circle in Figure 5.19b is aligned with the crack, although it is separated by a portion of material from the crack. The interconnection between both the crack and the void is possible underneath the surface of observation. A closer inspection of the lower part of the void (cf. Figure 5.19c) allows observing that the ferrite matrix follows a curved path, in which the two sides of the microstructure, separated by the crack, have almost an horizontal flow but the region in between is curved towards the direction of the crack and the void; in other words, the ferrite flow resembles a ‘S’ shape.

Figure 5.20: (a) SEM micrograph located close to the fracture surface of the broken sample for the Reference DP steel. The ND IPF map in (b) corresponds to the region enclosed by the upper rectangle in (a). The BC map in (c) corresponds to the area enclosed by the rectangle located in the lower part of (a). The step size of the EBSD maps is 50 nm.
The SEM micrograph of Figure 5.20a shows a region of the Reference DP steel containing a crack, which was also scanned by EBSD. The ND IPF map in Figure 5.20b corresponds to the region enclosed by the rectangle located in the upper part of the micrograph in Figure 5.20a. The indexation of the map is poor in the proximity of the cracks due to the high amount of deformation. However, it is possible to observe the ‘S’ morphology of the grains at the extremes of the cracks where a segment of the grains is oriented towards the direction of the cracks; e.g. the grain below the dashed red line in Figure 5.20b. The BC map in Figure 5.20c allows observing the highly partitioned and fine substructures created along the trajectory of the cracks. Although the aligned cracks are separated by a portion of material, a large amount of grain substructure is observed, interconnecting the aligned cracks and oriented along the same direction as the crack.

The characteristics of the substructures observed along and close to the cracks, which resembles an ‘S’ shape, is typical of shear bands; i.e. plastic instabilities created by extensive shear strain localization (cf. Chapter 2).

Figure 5.21 shows an EBSD scan made close to the fracture edge of a tensile sample of the SC DP steel. The length of the biggest void observed in the scan has a length of about 20 μm. The void seems to nucleate on the martensite band together with the other
larger voids. Shear bands are observed to initiate on the corners of the largest void and the ‘S’ shape effect is observed in the grains. Particularly noteworthy is the fact that the cracks are observed within the shear bands. Whether these cracks are nucleated within the shear bands or whether the cracks are the result of propagation from the largest void which is hidden by the two dimensional limitation of the scan remains to be determined. Nevertheless, these cracks and the shear bands are linked to each other. Another important observation is that these cracks have inherited the spatial orientation of the shear bands, which is ~45° with respect to the tensile direction. It is essential to mention that this angle corresponds to the macroscopic orientation of the fracture edge (cf. Figure 5.9b). The trajectory of the shear band is identified due to grain partitioning, whereas the trajectory independency of the crystal orientation is confirmed by observing the propagation of the shear band through several grains without changing trajectory. The limits of the cracks do not look to be set by grain boundaries.

Therefore it is postulated that the stress concentration of larger voids gives rise to the nucleation of shear bands and afterwards to the nucleation of cracks within them. This mechanism is assumed to trigger the void coalescence process, which is responsible for the final failure. In the case of the reference, HT and SC DP steels, shear bands are also observed in the points of fracture.

5.4.3 Quantitative analysis by void mapping

In contrast to the low amount of voids in the IF steel, the DP steels display a higher amount of voids heterogeneously distributed along the cross-section of the broken tensile samples. In order to acquire a quantitative description of the void activity and its relation to the local stress and strain states along the deformed samples, the 2D spatial distribution of the voids was obtained together with the individual characteristics of the voids using contiguous SEM micrographs. This approach is referred to as void mapping.

The distribution of the revealed voids within the cross-section of the broken samples is displayed on the void maps of Figure 5.22, where the size of the points was set proportional to the area of the detected voids. Based on those maps, it is possible to observe an increasing number of voids along the tensile direction with a maximum concentration close to the fracture edge. The location of this maximum coincides with the minimum thickness of the sample due to the necking formation. It is also common to find cluster(s) of voids aligned along the tensile direction in all the samples within the whole investigated area, which can be mainly attributed to two different reasons. In the first case, the voids are not interconnected to each other but rather are nucleated in martensite islands located along the segregation bands. In the other case, other voids are also nucleated along the segregation bands but different from the first case, these bands
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contain broken MnS stringers. Due to the low content of MnS inclusions, this second case represents a low fraction of the total amount of voids.

Figure 5.22: Void maps showing the spatial distribution of the voids on the cross-section of the broken samples for the a) Reference, b) SH, c) SC and d) HT dual phase steels. The size of the points in the maps is proportional to the void size.

Based on the data from the void maps, the highest total void area fraction (TVAF) of 0.39% corresponds to the reference DP steel while the SC DP steel exhibits the lower value with 0.21% TVAF. The HT and the SH DP steels show values of 0.38% and 0.35%, respectively. However the different necking geometries and the ensuing strain gradients created within the necked region of the DP steels make it difficult to directly compare the void nucleation behaviour between the steels. In order to evaluate the different void nucleation behaviour of the samples, the results of the maps are normalized with respect to the true strain and stress. Both the strain and the stress along the tensile direction of the samples were estimated from the sample profiles and afterwards combined with the data of the void maps. These data were used to analyze the evolution of the void area fraction and the number of void initiation sites per area as a function of the local strain and stress.
The graphs of Figure 5.23 display the void area fraction and the number of void initiation sites as a function of the true strain and true stress along the tensile direction.

Previous to the description of the results for the behaviour of the void area fraction and the number of voids per area, it is important to mention the relevance of both parameters in view of the void evolution process. The void area fraction represents the overall void growth activity within the sample. However, the void growth embraces the initial area of the void at its nucleation, the growth produced by the deformation of the material and the growth of the cracks close to the fracture surface. Thus, the void nucleation is also implicitly accounted for by the void area fraction. Conversely, the number of voids per unit area is a parameter describing the void nucleation activity without including information on the void growth.

Both void related parameters increase as the local strain rises along the tensile direction whereby the maximum value of the void parameters is located close to the
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fracture surface. This general observation is in agreement with other results [8, 4, 18]. It is important to highlight that all local values of void area fraction are below 1%, even in the proximity of the fracture surface. In the case of the SC DP steel, the void area fraction starts increasing at higher strains in comparison to the other three DP steels (Figure 5.23a). The number of void initiation sites in Figure 5.23c for the same DP steel also follows a similar behaviour than for the void area fraction.

A different situation is observed if the void parameters are plotted against local stresses, (cf. Figure 5.23b and d), where all DP steels display similar void activity behaviour. Interestingly, the quantification of the void initiation sites per area (cf. Figure 5.23d) follows the same trend for all steels; in other words, the correlation between the number of voids nucleated and the local stresses is similar among all DP steels. Despite of the differences between the intercritical heat treatments, this result suggests that void initiation activities for all DP steels behave in a similar manner under the same stress values.

In comparison to the previous void initiation activity results, slightly larger differences among the DP steels are observed on the correlation between the void area fraction and the estimated local stresses (Figure 5.23b). As void area fraction embraces information of both void nucleation and growth, the observed differences between the steels are attributed to the variations of the void growth behaviour.

![Figure 5.24: Variation of the void thickness, i.e. extension of the voids along the ND of the samples, as a function of (a) true strain and (b) true stress. The data is based on the void maps of Figure 5.22.](image)

One method to discern whether the larger martensite islands are prone to void initiation at lower strains is by analyzing the void thickness evolution. As the growth of the voids occurs along the tensile direction, then it is expected that the thickness of the
voids (dimension along the ND) has no contribution to the void growth and gives information of the martensite thickness where the void was nucleated. The graphs in Figure 5.24 display the void thickness as a function of the strain and stress.

Figure 5.24: Variation of the void thickness as a function of strain and stress.

Figure 5.25: Variation of the void length along the tensile direction as a function of (a) true strain and (b) true stress. The data is based on the void maps of Figure 5.22.

As already mentioned, the void area fraction contains information of both void initiation and growth. The length of the voids is another parameter, which contains useful information for both void formation mechanisms. The advantage of the void length is that the data of the void thickness is excluded and thus the thickness of the martensite where the voids nucleated is also ignored. Then the void length reflects better the growth of the voids as the deformation occurs along that direction. Figure 5.25 shows the evolution of void length as a function of the strain and stress. In both cases, the growth of the voids increases with the strain and stress although, contrary to the stress dependency of the number of voids, the void length of all DP steels shows a more uniform behaviour with strain.

5.5 Discussions

In essence, the micromechanisms of fracture observed on all DP steels of this dissertation are typical for ductile failure in metals; i.e. void nucleation, growth and coalescence. The void initiation sites are related to either the intermetallic inclusions or the martensite. Several researchers have discarded the relevance of the voids created at the inclusions among the fracture mechanisms due to their small amount and stability of the voids during deformation. In addition, the DP steels used in this research have in common the same base material and thus the type and amount of inclusions are the same. Therefore, the differences in the mechanical properties and the fracture characteristics
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between the DP steels are principally a consequence of the microstructure and not of the inclusions.

5.5.1 The heat treatments and the initial microstructures

The variations of the intercritical heat treatments of the as-cold rolled sheet steel influence some characteristics of the microstructure, which in turn modify the mechanical properties of the heat treated DP steels.

The heat treatment used as a reference for the other specimens (Ref. DP steel) produces a typical DP steel microstructure composed of ferrite and martensite. The martensite is found in three different morphologies: (i) well delimited martensite islands, (ii) distributed in bands along RD and (iii) aggregates of alternating plates of ferrite and martensite (bainite). In the first case, the shapes of the martensite islands vary from circular up to elongated plates. The martensite is located mainly along the ferrite grain boundaries, although it is also common to find martensite islands in the interior of the grains. The martensite bands are the result of the Mn segregation during the thermo-mechanical processing of the steel and because of this most of the MnS stringers are located within the martensite bands. In the last case, the aggregates of ferrite and martensite plates are in agreement with the definition of bainite given by Bhadeshia [35]: “Both upper and lower bainite consist of aggregates of plates of ferrite, separated by untransformed austenite, martensite or cementite”. In all specimens, the martensite has lath morphology based on the relatively low carbon content (cf. EPMA results from Table 5.2) and the high dislocation density within the laths (non-indexed regions of the EBSD maps in Figure 5.4) [26, 36].

The slowdown of the heating rate during the intercritical heat treatment mainly affects the distribution of the martensite (less banding) and increases the size of the ferrite grains. Both microstructural effects are a consequence of the austenite formation and its interaction with the recrystallization and grain growth of the deformed ferrite. During the heating stage of the heat treatment, the occurrence of the first austenite nuclei takes place at or inside the pearlite colonies. In addition, subsequent austenite nucleation sites occur at the ferrite grain boundaries and a competition starts between the austenite growths from both nucleation sites. However, the recrystallization of the ferrite plays an important role on the austenite nucleation at the ferrite grain boundaries because the moving boundaries from the recrystallization process inhibit or hinder the nucleation of austenite [37]. On the one hand, if the heating rate is slow enough to complete the recrystallization of ferrite before the material reaches the Ac₁ temperature, then austenite nuclei are expected to develop at both the grain boundaries and the
pearlite colonies. Moreover, some ferrite grain growth may occur before austenite starts nucleating. On the other hand, if the recrystallization of ferrite is still in progress when the ferrite-to-austenite transformation is starting, then the first austenite nuclei will continue growing along the length and thickness of the former pearlite colonies and only few austenite nuclei will start appearing at the ferrite grain boundaries when recrystallization has finished. This explains why the martensite bands of the SH DP steel are thinner and more discontinuous as well as more martensite islands are observed at the grain boundaries in comparison to the reference DP steel. Other characteristics of the microstructure, such as total martensite/bainite content or carbon partitioning, of the SH specimen are similar to the reference DP steel.

The martensite and bainite fraction in the DP steel is mainly affected by decreasing the cooling rate during the intercritical heat treatment. In a more general sense, the slow cooling rate affects both the carbon partitioning in the microstructure and the austenite-ferrite transformation. Whereas most of the austenite created during the intercritical holding is transformed to martensite in the reference DP steel, the cooling rate of the SC specimen promotes the growth of epitaxial ferrite and produces a coarser ferritic bainite in comparison to the other DP steels. This transformation to ferrite promotes the diffusion of carbon towards inner zones of the former austenite grain and produces higher carbon concentration in those regions. If the concentrations are high enough then it gives rise to retained austenite (EBSD data from Figure 5.4c) or formation of carbides (SEM micrograph from Figure 5.4c). Due to this carbon partitioning, the carbon content in the martensite is in average higher in comparison to the martensite of other DP steels. Hence, the SC DP steel displays a higher ferrite fraction and its martensite contains higher carbon content in comparison to the other DP steels.

The HT DP steel is characterized with similar martensite/bainite content as the reference DP steel but with a finer microstructure as well as more circular shapes of the second phase islands. The higher intercritical holding temperature allows the nucleation and growth of more austenite than in the reference specimen. However, as the cooling rate is not high enough to transform all austenite in martensite, as in the case of the reference DP steel, some transformations at high and intermediate temperatures occur; i.e. epitaxial ferrite growth and bainite formation. Similar to the SC specimen, the bainitic ferrite areas are larger and more elongated than the corresponding ones in the reference DP steel.
5.5.2 The failure micromechanisms of DP steels

Based only on the tensile test curves, one can conclude that the deformation evolution of the DP steels has a similar behaviour; i.e. the necking onset and even the final fracture of all specimens occur at similar engineering strains whereby the difference is in the stress displayed during the deformation process. It is important to mention that the typical parameters from tensile test data, such as tensile strength and deformation at fracture, give an incomplete description of the mechanical properties at fracture because of the post-necking deformation of the samples and the ensuing macroscopic localization of the deformation. When the tensile fracture stresses are calculated using the cross-section area at fracture, the values are not solely increased in comparison to the ultimate tensile strength but, in addition, the values of the true stresses at fracture for all DP steels are similar. Moreover, contrary to the data from tensile stress-strain curve, the deformation at fracture measured on the sample really shows a difference between the fracture strains of the different DP steels. These findings bear some similarities to the results of Saeglitz and Krauss [38] where the fracture stress at ductile fracture have similar values of around 4000 MPa for samples with different carbon contents and different tempering conditions. This is a first indication that the principal aspects of the fracture process for the DP steels in this study are controlled by the stress conditions rather than by strain.

Then, if the fracture conditions are controlled by stress and have similar fracture stress values, which factors produce the differences in the mechanical properties among the DP steels? Krauss [7] emphasizes the role of strain hardening when the fracture stresses are similar, such as the DP steels used in this study. Specimens with low average strain hardening rate (e.g. SC DP steel) reaches the Considère condition (equation 3.2 in Chapter 3) at relatively low stresses, and therefore, the material commences to localize the deformation. As the stress reached at the necking onset is still lower than the critical fracture stress necessary for the final fracture, then the material requires relatively extensive necking deformation to achieve the required critical stress conditions for fracture. In contrast, materials with higher strain hardening rates display higher ultimate tensile strength when the plastic deformation localization commences and therefore, less post-necking deformation is needed for reaching the stress required for final failure.

The previous paragraphs concern the fracture mechanisms on a macroscopic scale; however these observations are also congruent to the microstructural behaviour. Despite the noticeable different evolution of the void nucleation activity for all DP steels as a function of the true strain, it is surprisingly observed a similar behaviour for all DP
steels in the evolution of number of voids per area when plotted as a function of true stress (cf. Figure 5.23d). Therefore, this evidence suggests that the void nucleation evolution during deformation is controlled by the local stress conditions of the specimens and is similar for all DP steels, despite having different microstructures due to the variations on the heat treatments.

The SC DP steel showed similar elongation at failure like the other specimens, although the tensile strength was the lowest and the necking was more severe. Conversely, this steel also displayed the lowest rate of void nucleation as a function of true strain (cf. Figure 5.23b) in comparison to the other DP steels. The reason of this apparent void resistance is attributed to the soft ferrite matrix which accommodates the strain incompatibilities around the martensite islands. Because the strength of the martensite islands is relatively high due to the higher carbon content and dislocation density content, most of the strain is accommodated in the ferrite matrix. This localized strain has two different sources, (i) the imposed deformation from the tensile test and (ii) the accommodation of the strain heterogeneities due to the larger volume of the martensite phase. As the deformation of the ferrite phase increases, a threshold value is reached at which the second phase starts deforming plastically. Afterwards, void initiation will eventually occur once the local conditions of the martensite or at the martensite-ferrite boundary attain a critical value. The internal heterogeneities within the microstructure produce a variation in the location of void initiation. For example, variations in the carbon content of the martensite or the crystallographic orientation of the surrounded ferrite grains can trigger easily strain localisation and, as a consequence, rising the local stresses for void initiation. The void area fraction is an indication of the damage of the material where not only the void initiation sites are considered but also the growth of the voids is included in this parameter. The number of initiation sites per area is a parameter which only considers the void initiation activity within a certain region without the influence of void growth.

Despite the fact that certain characteristics of the microstructure were different between the DP steels, it is important to highlight that similar mechanisms of void initiation and growth were observed among the samples.

5.5.3 Shear bands and the final failure propagation

The reason of the low void activity at the final stage of fracture in modern DP steels is a phenomenon which is not well understood. The low amount and small size of the voids present underneath the fracture surface are not characteristic of the typical void coalescence mechanisms where extensive void volume fraction occurs prior to fracture.
In some specific cases, the low levels of void area fraction are attributed to the competition between different failure micromechanisms. One example is the experiment conducted by Ahmad et al. [18]. They proposed that cleavage is the predominant mechanism for producing the final and sudden failure of the steel and is responsible for interrupting the ductile failure mechanisms. Evidence of this hypothesis was found by observation of cleavage facets on the fracture surface. In the present dissertation, however, no typical features of cleavage mode are observed on the fracture surface of the tensile samples. Moreover, the fracture surface for all DP steels exhibits a dimple topology, indicative of a ductile fracture process. Therefore, most probably another mechanism is responsible for the final rupture.

One of the principal results of this dissertation is the observation of cracks within shear bands (cf. Figure 5.21). In order to avoid confusions, the definition of crack employed to designate the cavities observed in Figure 5.19 is exclusively used based on geometry; in other words, the term crack is used for cavities with high aspect ratio, reduced void volume and large surface area. This term is not intended to designate cavities which are related to the cleavage fracture mechanisms.

The idea of void nucleation occurring within shear bands is similar to state that void initiation and growth could evolve under a shear deformation field. This relation is in contradiction with most of the damage mechanics concepts. As mentioned in Chapter 2, most of the void growth equations are based on the deviatoric stress tensor where the shear stress components are not involved. However, void growth may occur with an expansion of surface area without the volume expansion of the cavity, as the Gurson model states.

Based on experimental evidences, the observation of shear bands in conjunction with void coalescence was also mentioned in the studies of Avramovic-Cingara et al. [4, 6]. They explained that this combination is the result of a possible nucleation of a secondary population of voids within the shear band connecting the primary voids; i.e. void sheet coalescence. The formation of voids within shear bands has also been observed in other metals although in dynamic conditions [39].

Based on the previous discussions, it is proposed that the detrimental aspect of voids on the DP steels is not directly due to the total null-carrying capacity of the voids but rather to the stress concentration generated around the voids and the ensuing strain localization taking place in the void vicinity. The strain localizations, which are reflected as shear bands, lead eventually to the formation of cracks within the shear bands. Then it is very likely that the small dimples observed on the fracture surface are the result of the cracks nucleated along the shear bands.
The difference in the coalescence mechanism of the DP steels relies on the number of points of fracture (Figure 5.9b). Each point of fracture signifies the onset of shear band propagation and the associated nucleation of cracks along it. The reference, the HT and the SH DP steels display several points of fracture randomly distributed along the thickness. Such behaviour is different from the one of the slow cooled DP steel, which exhibits a preference to create only one point of fracture in the middle thickness of the tensile sample. This localization in the slow cooled material suggests that the middle thickness region develops shear bands in earlier stages than other locations of the sheet thickness. Whereas in the other DP steels, the onset site of the coalescence mechanism is determined by a stochastic process instead of a preferential site.

5.6 Conclusions

In this chapter, the failure micromechanisms together with the microstructure characterization and the mechanical properties of four DP600 steels with different heat treatments were investigated. It was found that the effects of the heat treatments variations on the microstructure mainly affected the local carbon content distribution as well as the structure of the second-phase constituents (bainite), which were composed of alternate laths of bainitic ferrite and martensite. In the case of the mechanical properties, the principal parameters influenced by the heat treatments were the tensile strength, the work hardening behaviour and the post-necking characteristics; specially for the case of the slow cooled DP steel. In spite of the different mechanical properties, the deformation and fracture micromechanisms were alike among the specimens. In a first place, the strain incompatibilities of the microstructural constituents were mainly accommodated by the ferrite grains. Once the ferrite grains were not able to accommodate further deformation and the second phase constituents commenced to deform, void initiation process started taking place. Voids were principally initiated in two sites within the microstructure; (i) along the ferrite-martensite/bainite interface and (ii) within the bainite. In addition, the occurrence of void nucleation is a continuous process taking place during the deformation history of the material. The results of this investigation suggest that the evolution of void initiation has a similar behaviour for all DP steels and which is controlled by local stress conditions. Nevertheless, the void area fraction do not exceed one percent within the proximities of the fracture surface. Finally, it was concluded that the detrimental effect of voids is not their null carry capacity but rather the stress concentration created around the void which gives rise to shear bands, which is a form of microstructural strain localization. Eventually, cracks are nucleated along the shear bands and are responsible for the final failure of the material.
References

Chapter 5


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Failure micromechanisms in DP steels


Chapter 6

AA6016 aluminium alloys

The failure micromechanisms of two AA6016 aluminium alloys with different magnesium content after uniaxial tensile tests are studied in this chapter. SEM fractographs of both materials suggest two types of failure mechanisms. The first is characterized by a sequence of events, i.e. void initiation, growth and coalescence on intermetallic constituents, whereas the second one can be identified as ductile intergranular failure. The second type of failure is observed more often in the aluminium alloy with higher magnesium content. In both aluminium alloys, secondary cracks were found close to the fracture surface on the cross-section plane of the tensile sample. Crystal orientation maps show that the secondary cracks were formed either by an intergranular failure mechanism or by local plastic instabilities such as shear bands which subsequently progress into cracks. The higher magnesium content gives higher strength but reduces the ductility by weakening the grain boundaries.

6.1 Introduction

From a microstructural point of view, the fracture of the AA6XXX aluminium alloy series may occur in an intergranular [1-4] or transgranular [3-7] mode. On the one hand, the intergranular mode should be avoided because of its detrimental effects on the ductility and fracture toughness of the aluminium alloys. The occurrence of intergranular ductile failure has been mainly attributed to (i) the presence of particles along grain boundaries or (ii) formation of precipitation free zones, (PFZs). In the first case, the variety of particles reported to induce intergranular fracture are precipitates, dispersoids or impurities such as sodium, calcium or strontium [2]. In the other case, the PFZs are a soft region located close to grain boundaries which act as a deformation
localization site. The formation of precipitation free zones is associated with the intensive diffusion of alloying elements (in AA6XXX series Mg) from the grain boundaries vicinity to the bulk of the grains.

On the other hand, the micromechanisms of transgranular ductile failure of aluminium alloys, particularly on the AA6XXX series, consist usually of a sequence of void nucleation, growth and coalescence triggered by (or in the vicinity of) coarse second phase particles. These particles are intermetallics mainly containing iron; they have dimensions in average larger than 1 μm and are known as constituents. These constituents are reported to have a detrimental effect on the mechanical properties [8]. In the case of ductile failure, the brittleness of these intermetallics and their weak adhesion to the Al matrix turn them into suitable sites for void nucleation, which can be initiated even at relatively low strains [6].

However, a combination of both failure modes is often found in aluminium. The microstructural variables, which affect the transition from transgranular to intergranular failure have been reported to be the size of the precipitate free zones [9], the distribution of coarse particles, the slip band spacing during deformation [1, 3] and the segregation of precipitates or trace impurities [2] along grain boundaries.

The AA6XXX aluminium alloy series are heat treatable and their higher strength is based on precipitation hardening of Mg-Si precipitates. The influence of magnesium on the strength of these aluminium alloys was studied by Caceres et al. [10] on alloys in as-cast conditions where increasing the amount of magnesium leads to higher tensile strength but reduces the elongation at fracture.

The objective of this chapter is to analyse the origin of void nucleation of two AA6016 aluminium alloys with different magnesium content subjected to tensile deformation. Fractography studies together with the use of electron backscatter diffraction (EBSD) technique on the cross-section of tensile samples were used to clarify the mechanisms by which void initiation occurs.

6.2 Initial Microstructure

The microstructure of both AA6016 aluminium alloys of which the chemical composition is displayed in Table 3.4 is formed by a similar population of plate-like intermetallic constituents, embedded in a recrystallized aluminium matrix; as can be seen in the micrographs of Figure 6.1. Table 6.1 shows the principal characteristics of the aluminium matrix and the constituents for the aluminium alloys studied on this chapter. The Grain Intercept Length, GIL, and the geometrical characteristics of the constituents were calculated based on light optical microscopy images, whereas the
Vickers microhardness measurements were made using a load of 300 gf. The grains of the aluminium matrix are slightly pancaked with the longest direction oriented along the sheet rolling direction (RD) and the shortest length aligned along ND. The lengths of the grains are similar between both aluminium alloys; for example, the grain intercept length along RD is $27.6 \pm 4.8 \mu m$ for the 4Mg alloy and $23.6 \pm 4 \mu m$ for the 6Mg alloy.

![Figure 6.1: Optical micrograph of the aluminium alloys type (a) 4Mg and (b) 6Mg. The elongated inclusions correspond to the intermetallic constituents.](image)

Based on the characterization of the intermetallic particles such as area fraction, particle area and distance between particles (cf. Table 6.1), a similarity can be observed between the intermetallic particle populations of both aluminium alloys. This fact indicates that the differences in the mechanical properties or failure micromechanisms between both alloys are not a direct consequence of the intermetallic constituents.

### Table 6.1: Microstructural characterization of the AA6016 aluminium alloys.

<table>
<thead>
<tr>
<th></th>
<th>Type 4Mg</th>
<th>Type 6Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>GIL along RD, $\mu m$</td>
<td>27.6</td>
<td>23.6</td>
</tr>
<tr>
<td></td>
<td>$\pm 4.8$</td>
<td>$\pm 4.0$</td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>0.8</td>
</tr>
<tr>
<td>Area fraction of the constituent particles, %</td>
<td>$\pm 0.5$</td>
<td>$\pm 0.4$</td>
</tr>
<tr>
<td>Individual constituent area, $\mu m^2$</td>
<td>9.5</td>
<td>8.3</td>
</tr>
<tr>
<td></td>
<td>$\pm 3.7$</td>
<td>$\pm 3.6$</td>
</tr>
<tr>
<td>Constituent nearest neighbour distance, $\mu m$</td>
<td>14.6</td>
<td>14.8</td>
</tr>
<tr>
<td></td>
<td>$\pm 2.5$</td>
<td>$\pm 3.4$</td>
</tr>
<tr>
<td>Vickers microhardness, HV$_{300}$</td>
<td>74.7</td>
<td>93.8</td>
</tr>
<tr>
<td></td>
<td>$\pm 2.6$</td>
<td>$\pm 2.6$</td>
</tr>
</tbody>
</table>
The crystallographic texture of both alloys is typical for recrystallized sheet aluminium alloys [11] where the cube orientation appears as the strongest component; as can be seen in Figure 6.2. The 6Mg aluminium alloy has a larger intensity of the cube component in comparison to the 4Mg alloy with 2.3 orders of magnitude.

![Figure 6.2: ODF of the 4Mg and 6Mg AA6016 aluminium alloys represented on three different $\phi_2$ sections. The ODF were experimentally obtained by X-ray diffraction.](image)

The intermetallic constituents observed in both alloys are Fe-containing intermetallics identified as $\alpha$-AlFeMnSi and $\beta$-AlFeSi particles. Figure 6.3 shows SEM

![Figure 6.3: Energy Dispersive X-ray spectrum (EDS) of the particle shown on the SEM micrograph. (a) $\beta$-AlFeSi intermetallic particle. (b) $\alpha$-AlFeMnSi intermetallic particle.](image)
micrographs of both intermetallics together with the X-ray spectrum of the particles. The area fraction of the constituents is close to 1% for both alloys. It is important to emphasize that the particle population is similar between both aluminium alloys based on the analysis from the SEM micrographs in backscatter mode (cf. Chapter 2). The average values of the principal parameters obtained from the image analysis of the constituents are given in Table 6.1.

6.3 Mechanical properties

The engineering stress-strain curves of both aluminium alloys are shown in Figure 6.4 while Table 6.2 summarizes the principal mechanical properties. As was expected [10], the aluminium alloy with the higher magnesium content, type 6Mg, showed an ultimate strength, which is approximately 16 MPa higher than the strength of the 4Mg alloy. The Vickers microhardness shows a similar difference. In contrast, the elongation at fracture of the 6Mg is inferior compared to the 4Mg alloy. These differences can be attributed to the strengthening of the aluminium matrix where the alloy with higher magnesium content is assumed to create more hardening precipitation particles.

Assuming that all magnesium content is located in the form of stable Mg$_2$Si precipitates, the aluminium alloy 6Mg contains close to 0.95 wt. % of Mg$_2$Si precipitates. This value is significantly larger than the estimated amount of 0.63 wt. % for the aluminium 4Mg and hence the difference can be correlated to the higher strength observed on the 6Mg type.
Table 6.2: Mechanical properties of the two AA6016 aluminium alloys based on tensile tests. The main value of each parameter is calculated from the average of three different tests and is accompanied by its standard deviation.

<table>
<thead>
<tr>
<th>Property</th>
<th>Type 4Mg</th>
<th>Type 6Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultimate Tensile Strength, MPa (UTS)</td>
<td>246.6 ± 3.0</td>
<td>262.7 ± 1.8</td>
</tr>
<tr>
<td>Deformation at UTS, %</td>
<td>23.2 ± 0.6</td>
<td>21.6 ± 0.3</td>
</tr>
<tr>
<td>Deformation at Fracture, %</td>
<td>27.7 ± 0.3</td>
<td>26.1 ± 0.4</td>
</tr>
<tr>
<td>Reduction of Area at Fracture, % (RA)</td>
<td>50.2 ± 0.9</td>
<td>38.3 ± 2.7</td>
</tr>
<tr>
<td>True fracture strain, % ((\varepsilon_F))</td>
<td>69.7 ± 1.7</td>
<td>48.3 ± 4.4</td>
</tr>
<tr>
<td>True fracture stress, MPa ((\sigma_F))</td>
<td>365 ± 4</td>
<td>387 ± 18</td>
</tr>
<tr>
<td>R value along RD ((R_{0°}))</td>
<td>0.69 ± 0.1</td>
<td>0.7 ± 0.1</td>
</tr>
<tr>
<td>Average R value ((\bar{R}))</td>
<td>0.59</td>
<td>0.58</td>
</tr>
<tr>
<td>(\Delta R)</td>
<td>0.12</td>
<td>0.40</td>
</tr>
</tbody>
</table>

6.3.1 Macroscopic failure mechanisms and fractography

The deformation and failure mechanisms in both AA6016 aluminium alloys develop in three consecutive stages. During tensile tests, initially the deformation of the samples occurs uniformly along the gauge length (stage I). When the ultimate tensile strength (UTS) is achieved and the deformation starts to localize, the materials reach stage II, and finally, end with fracture in stage III.

SEM fractographs of both aluminium alloys reveal the existence of two different fracture topologies (cf. Figure 6.5). The features observed in Figure 6.5a are more characteristic for the fracture surfaces observed in sample 6Mg. Polyhedral shapes of which the size is similar to the grain size of the material are the main characteristic of the fractured surface. The polyhedral facets show traces of shallow dimples which are generally smaller than 1 \(\mu m\). This type of fracture surface is usually considered as intergranular ductile failure, whereby the plastic deformation takes place before grain boundary decohesion occurs. The shallow dimples suggest the presence of small void initiation sites at grain boundaries, possibly caused by small particles.

Another fracture surface observed in some regions of the samples shows a typical ductile failure based on initiation, growth and coalescence of voids. The intermetallic particles are often found to be responsible for void initiation in sample 4Mg (Figure 6.5b) and even some of these particles remain in the bottom of the deeper dimples. In several cases, shallower dimple populations are located on the border of the deep dimples which are assumed to be the product of void sheet coalescence. Both types of fracture surfaces appear in both materials although the alloy type 6Mg qualitatively exhibits more zones similar to Figure 6.5a than alloy type 4Mg.
6.4 Failure micromechanisms

6.4.1 Void initiation sites

SEM observation of the sample cross-section was made on two regions; one within the zone away from the fracture edge, where uniform deformation is assumed to occur, and a second region which is close to the fracture edge. In the uniform deformation region of both alloys, voids are commonly initiated by either (i) particle-matrix decohesion or (ii) particle fragmentation. In the first case, the nucleated voids are oriented along the tensile axis with a thickness equal to the thickness of the particle. The voids can be located on one or both sides of the particle. In case of fragmentation, the particle is split in two or more parts.

Some voids are also observed without any particles around. It is believed, however, that the metallographic preparation was responsible for removing the particles from these voids. Not all particles initiate voids and it is particularly noteworthy that large particles are more prone to initiate voids than small particles, as can be observed in the SEM micrographs of Figure 6.6. Within the uniform deformation region of the tensile
samples, both alloys show no appreciable difference between them with respect to the void formation mechanisms.

In the region close to the fracture edge of both aluminium alloys, another form of void initiation is observed, which is referred to as crack formation due to geometry. These cracks are roughly oriented at 45° with respect to the tensile direction and they can be observed either with or without constituent particles. Some examples are shown in Figure 6.7. Based on optical micrographs, 11 cracks were counted close to the fracture edge in one of the tensile specimens of type 6Mg, while only 3 cracks were found in type 4Mg. In the former case, the further crack was localized 825 μm away from the fracture edge while the maximum distance from the fracture edge in the case of 4Mg was 545 μm.

**Figure 6.6:** SEM micrographs of broken tensile specimens made within the uniform elongation zone of the aluminium alloys type (a) 6Mg and (b) 4Mg. In both alloys, the constituent particles are observed to be fragmented or debonded from the matrix. Also voids without particle in the vicinity are observed.

**Figure 6.7:** SEM micrographs of broken tensile specimens close to the fracture surface of alloys type (a) 6Mg and (b) 4Mg. In the case of (b), the cracks are extended following the constituent particles.
Figure 6.8: Inverse pole figure maps (IPF), (a) and (c), and SEM micrographs, (b) and (d), of a zone close to the fracture edge of AA6016 type 6Mg. Three different cracks are found in these maps with their origin along grain boundaries. The misorientation profile chart in (e) corresponds to the line AB traced in the IPF map of c). These curves correspond to the misorientation angle with respect to point A (P2O), the point-to-point misorientation (P2P) and the misorientation angle with respect to the average crystal orientation considering the two adjacent grains as one (P2Av).

The nature of some cracks and voids was investigated in both alloys by EBSD technique. Figure 6.8 illustrates two cases of cracks located close to the fracture edge of the 6Mg alloy. In one of the cases (cf. Figure 6.8a and b), two cracks are observed along
the grain boundaries. The largest crack, crack 1, is located in the grain boundary of three grains, two grains from one side and one grain on the other side. The misorientation angles between grain 1 and 2 is 35.4° and between grain 1 and 3 is 55.1°. The average crystal orientation of grains 1, 2 and 3 was used for the above measurement. The cracks in the grain boundary formed between grain 1 and 2 are thicker, which allows assuming that the crack initiates in the grain boundary 1-2 and further propagates to the boundary of grains 1 and 3.

The crack appears to be stopped in the triple junction of grains 1, 3 and 4. In the case of the crack created between grains 5 and 6, the misorientation angle is 46.7°. Grains 1 and 6 have similar orientations with a misorientation angle of only 4.8°.

A different case is observed in Figure 6.8c and d where a crack is observed among two grains with similar crystal orientations. The misorientation among the average crystal orientation of both regions is 16.9° which is close to the transition value between high and low angle grain boundaries.

A misorientation line profile along both regions (AB) was extracted from the EBSD map of Figure 6.8c and is displayed in Figure 6.8e. It is observed that the regions closer to the crack exhibit an orientation gradient, which can be compared from the different types of misorientation profiles. The orientation gradient is a product of plastic deformation and shows that the deformation increases in the vicinity of the crack by a gradual formation of Geometrically Necessary Density of Dislocations, GND. Therefore, the grain boundary separation is not limited to high angle grain boundaries, but may take place on low angle grain boundaries too.

Figure 6.9a and b show cracks situated between two grains in material 4Mg which form a misorientation angle of 24.4° between their average crystal orientations. This case is similar to the cracks observed on type 6Mg. Figure 6.9c shows an EBSD scan from a zone containing a constituent particle which is debonded from the matrix at both ends. The particle seems to be rotated towards the tensile axis and gives the impression that it tries to align itself with the elongation direction of the grain (i.e. tensile axis). The particle is found inside a grain which contains a dense dislocation wall associated to a misorientation angle as high as 30°. The type of microbands observed in this grain is indicative of a strong deformation in the matrix.

The smooth orientation gradients found close to intergranular cracks suggest some deformation of the grain boundary region before decohesion which is in agreement with the observations of Evenson et al. [1] from Laue back reflection X-ray patterns on the fracture facets.
6.5 Discussions

6.5.1 The competition between transgranular and intergranular fracture

The SEM fractographs show typical features of both intergranular and transgranular fracture. However, it is not possible to assert whether the void initiation has an intergranular or transgranular origin. When voids or cracks have reached a certain size, their propagation represents the next step to final failure. In this case, the propagation can also be achieved by void-sheet coalescence, which is considered as transgranular, or as intergranular if it follows the grain boundaries. Therefore, it is not possible to discriminate between the initiation and the propagation failure mechanism based solely on the fracture surface inspection. Complementary information about the initiation mode is obtained from the cross-section of the broken tensile specimens by examining the voids (or cracks) created below the fracture surface. Then the existence
was observed of cavities with higher aspect ratio and oriented approximately 45° with respect to the tensile direction. Similar to the case of DP steels from the previous chapter (Chapter 5), these cavities are referred to as cracks due to their geometrical characteristics. It is important to mention that the applied term excludes the occurrence of cleavage.

The nucleation of cracks occurs only in the proximities of the fracture surface and is not observed within the uniform deformation region of the tensile sample. This fact indicates that crack initiation takes place after the end of the uniform deformation stage where the deformation starts localizing and higher local stresses are generated.

The EBSD mapping of the regions surrounding the cracks gives the advantage of both, determining the origin of cracks along grain boundaries and proving the existence of deformation close to these cracks by revealing the formation of grain substructures. Therefore it can be said that cracks in both alloys occur along grain boundaries and are accompanied of plastic deformation. However, failure by dimple rupture mechanism from constituent particles is also taking place as it is observed by fractography. Initiation and growth of the voids is possible to be observed in the cross-section of the tensile samples but void coalescence could not be observed.

The intergranular failure and the dimple rupture mechanism are in competition in these alloys, although the former mechanism should be avoided due to its low energy failure process. The higher amount of cracks and the larger intergranular fracture surface area found in the aluminium alloy with higher magnesium content, type 6Mg, suggests that the intergranular mechanism increases with higher magnesium content and the concomitant higher amount of Mg$_2$Si precipitates. The aluminium alloy 6Mg is estimated to contain 0.95 wt % of Mg$_2$Si precipitates, which is larger than the alloy 4Mg with a content of 0.63 wt. %. The action of the magnesium is not only attributed to the precipitation enrichment of the grain boundary regions but also to the hardening of the aluminium matrix by the creation of more precipitates. A harder matrix implies a larger stress on regions of plastic strain incompatibilities such as grain boundaries and the particle-matrix interface. It is believed that local stresses along grain boundaries reach a critical value which triggers the separation.

### 6.6 Conclusions

The study of the void formation during fracture of AA6016 aluminium alloys with variable Mg content showed that the amount of Mg mainly affects the mechanical properties of the aluminium matrix due to the variation on the number of Mg$_2$Si precipitates. The increase of precipitates content increases the tensile strength and the
microhardness of the alloy. However the strengthening of the matrix is accompanied with a decrease in the ductility. Based on the assessment of the fracture surface and secondary cracks, it was demonstrated that the higher magnesium content increases the regions of intergranular ductile failure and the number of cracks occurring along grain boundaries. Nevertheless some regions also failed by void initiation, growth and coalescence of constituent particles. This transition is attributed to the segregation of grain boundary precipitates and additionally to the high level of local stresses on grain boundaries as a consequence of the matrix strengthening.

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Chapter 7

Modelling of DP steels

The present chapter is focused on modelling the evolution of local stresses of the DP steel constituents during tensile deformation. In order to simulate the mechanical behaviour of DP steels, a micromechanical model is developed by incorporating a physical-based work hardening model into a crystal plasticity framework for each of the microstructural constituents. Among the material parameters employed by the model, the crystallography of martensite and the Geometrically Necessary Density of Dislocations, GND, of the ferritic matrix are taken into account for the model development. Finally, the model is calibrated and afterwards validated on experimental data of DP steels submitted to different annealing treatments.

7.1 Introduction

The simulation of the deformation behaviour of DP steels has been extensively investigated since the introduction of this type of steels [1-7]. Several models have been proposed to simulate, to analyze and to predict the mechanical behaviour of DP steels. The different approaches that have been suggested vary from using empirical equations such as the Holloman, Voce or Ludwig equations [1, 2] up to the robust Representative Volume Element (RVE) by Finite Element Method (FEM) simulations [4, 5] where selected characteristics of the microstructure are incorporated.

One of the purposes in modelling the mechanical properties of DP steel microstructures is to estimate the work hardening behaviour for each of the microstructural constituents, which is difficult to obtain experimentally. Some models assume that martensite only deforms elastically although this assumption conflicts with experimental observations where martensite actually deforms plastically [8]. A majority of modern models accept the deformation of martensite by either assuming a perfectly
plastic behaviour [6] or using a different hardening law than for ferrite. In the last case, the challenge is to find the correct parameters for the work-hardening equations.

In the better state-of-the-art models, the principal microstructural features representing the martensite are the martensite size, the initial dislocation density and the carbon content. Hatem et al. [7] incorporated the crystallography of the martensite in the FEM simulations.

An additional challenge is the strain partitioning between martensite and ferrite due to the different mechanical response of both phases and their interaction. Bouaziz and Buessler [9] proposed a model based on the equal energy developed by both phases; the so-called IsoW assumption. Tjahjanto et al. [10] observed that the solution given by the IsoW assumption in a crystal plasticity framework gives similar results than robust FEM simulations. However, the great advantage of the IsoW approach over the FEM is the less computational effort required by the first.

The present chapter is focused on modelling the evolution of local stresses of the DP steel constituents during tensile deformation. The objective is not limited to simulate the average mechanical response of the martensite/bainite constituents but rather to predict the range of stress variation of the constituents produced by the crystallographic texture of both martensite/bainite inclusions and the ferrite matrix in which they are embedded. In order to achieve this goal, the mechanical behaviour of DP steels is modelled by assembling a physically-based work hardening model into a crystal plasticity framework for each of the microstructural constituents. Following the approach proposed by Hatem et al. [7], the crystallography of martensite is taken into account for the model development. Finally, the model is calibrated and subsequently validated on experimental data of DP steels with different heat treatments.

7.2 Homogenization scheme of the DP steel microstructure

The Representative Volume Element (RVE) of the DP steels is assumed to be composed of several microstructural aggregates, which are individually composed by ferrite grains surrounding the “second phase” inclusions. In this chapter, the term inclusion is applied in a mechanical sense to designate a delimited region of the material with different mechanical properties with respect to the surrounding matrix. These inclusions are the transformation products of the parent austenite grains created during the intercritical heat treatments of the DP steels and are composed of alternated laths of martensite and bainitic ferrite. A schematic illustration of the components at the different length scales of the RVE is shown in Figure 7.1.
The present model is based on crystal plasticity as described in the subsequent sections. A modified physical-based model developed by Pantleon [11] for the work hardening behaviour is incorporated into a crystal plasticity framework. For the sake of simplicity, the elastic deformation contribution has not been incorporated into the model.

### 7.2.1 Work hardening model

The critical resolved shear stress for each of the slip systems of either the ferrite grains, the bainitic ferrite laths or the martensite blocks is considered to be composed of two principal contributions. In the first place, the stress component $\tau_{0}$, hereafter referred to as static yield stress, represents the strengthening produced by the lattice friction and the chemical elements in solid solution. The second component is related to the flow stress due to the interaction between dislocations on all slip systems. Considering both contributions, the resulting critical resolved shear stress $\tau^{(s)}$ of the slip system $s$ is given by

$$
\tau^{(s)} = \tau_{0} + \alpha \mu b \sqrt{\sum_{s=1}^{\text{NSS}} \rho^{(s)}} \tag{7.1}
$$

where $\mu$ is the shear modulus, $b$ is the magnitude of the Burgers vector, NSS is the number of active slip systems and $\alpha$ is a factor that characterizes the interaction between dislocation which depends on the strain rate and temperature.

The shear stress $\tau_{0}$ of equation (7.1) can further be subdivided into two other expressions: one accounting for the lattice friction and the strengthening contribution of chemical elements in solid solution, $\tau_{SS}$, and the other one is the solid solution
strengthening by carbon atoms, $\tau_C$. The term $\tau_{SS}$, expressed in MPa, is calculated using the following empirical equation

$$\tau_{SS} = 77 + 80 \cdot \text{Mn} + 750 \cdot \text{P} + 60 \cdot \text{Si} + 80 \cdot \text{Cu} + 45 \cdot \text{Ni} + 60 \cdot \text{Cr} + 11 \cdot \text{Mo} \quad (7.2)$$

where the element content is given in mass percentage. The empirical formula in equation (7.2) has also been employed by other models [12-14] in order to simulate the mechanical response of ferrite, martensite and bainite.

In the case of the strengthening contribution of carbon, the stress $\tau_C$ is calculated using different formulae for ferrite and for martensite. The strengthening of the ferrite grains due to carbon is calculated using the same equation as in [13] and [14]

$$\tau_C = 5000 \cdot C_f \quad (7.3)$$

Whereas in the case of the martensite blocks, the following formula is used [12]

$$\tau_C = 3065 \cdot C_m - 161 \quad (7.4)$$

It is important to mention that the equations (7.2), (7.3) and (7.4) were initially formulated for the calculation of the macroscopic flow stress without a crystal plasticity framework and therefore the equations need to be adapted for individual slip systems rather than for the macroscopic material behaviour. In order to adjust those equations, it is considered that the static yield stress on each slip system is proportional to the sum of both shear stresses $\tau_{SS}$ and $\tau_C$. This assumption gives rise to the following equation

$$\tau_0 = \left( \frac{\tau_{SS} + \tau_C}{C_1} \right) \quad (7.5)$$

where the constant $C_1$ is a proportionality constant which has to be determined.

On the one hand, the static yield stress controls the macroscopic yield stress of the material and remains constant during the entire deformation process. The work-hardening behaviour, on the other hand, is controlled by the accumulation and interaction of dislocations, which is described by the second term of equation (7.1). The work hardening term depends on the definition of the evolution of dislocations density during the deformation process and to this purpose the model proposed by Pantleon [11] is employed in this dissertation. Based on the classic model of Kocks and Mecking [15], Pantleon analysed the evolution of dislocation density on the slip system $s$ by discriminating between dislocations of opposite sign ($\rho_\alpha^{+s}$, $\rho_\gamma^{-s}$) and opposite fluxes ($\overline{\gamma}^{+s}$, $\overline{\gamma}^{-s}$). The flux $\overline{\gamma}^{+s}$ represents the set of mobile dislocations of positive Burgers
vectors moving to the right whereas \( j^{(e)} \) corresponds to the flux of dislocations of negative sign moving to the left [16]. Then, the evolution of the dislocation density due to storage and annihilation by dynamic recovery mechanisms is described for dislocations of opposite signs as

\[
\frac{d\rho^{(s)}}{dt} = \left( \frac{1}{\lambda} - 2y\rho^{(s)}_\perp \right) j^{(s)} - 2y\rho^{(s)} j^{(s)} \quad (7.6)
\]

\[
\frac{d\rho^{(s)}}{dt} = \left( \frac{1}{\lambda} - 2y\rho^{(s)}_\perp \right) j^{(s)} - 2y\rho^{(s)} j^{(s)} \quad (7.7)
\]

The above equations establish that the density of immobile dislocations of one sign increases as mobile dislocations moving along one direction are trapped after travelling a mean free path \( \lambda \). However, a fraction of those mobile dislocations annihilates the same number of immobilized dislocations of opposite sign \( \rho^{(s)}_\perp, \rho^{(s)}_\parallel \) by passing within an annihilation distance \( y \). As a consequence, the net storage of immobile dislocations is equal to the total flux of arrived mobile dislocations minus the amount of annihilated immobile dislocations of opposite sign. This net storage effect is represented by the difference within the first term of equations (7.6) and (7.7). The second term in the above equations directly represents the immobile dislocation density \( \rho^{(s)}_\perp, \rho^{(s)}_\parallel \) which was annihilated by the flux of moving dislocations of opposite sign; in other words, the second term represents the dynamic recovery mechanisms. In order to integrate equations (7.6) and (7.7), additional conservation equations are needed.

On the one hand, the total flux of dislocations of both signs is given by the sum of dislocations moving in either direction and is responsible for the shear strain \( \gamma^{(s)} \) on the slip system \( s \). The flux balance is expressed as

\[
j^{(s)} = j^{(s)} + j^{(s)} = \frac{1}{b} \frac{d\gamma^{(s)}}{dt} \quad (7.8)
\]

On the other hand, the bias of the dislocation flux is the result of the difference between opposite fluxes and is given by

\[
\Delta j^{(s)} = j^{(s)} - j^{(s)} \quad (7.9)
\]

In a similar manner, the total dislocation density for a particular slip system is given by the sum of dislocation densities of opposite sign.
\[ \rho^{(s)} = \rho^{(s)}_{\perp} + \rho^{(s)}_{\uparrow} \]  
(7.10)

Whereas the excess of dislocations of one sign is determined by

\[ \Delta \rho^{(s)} = \rho^{(s)}_{\perp} - \rho^{(s)}_{\uparrow} \]  
(7.11)

The combination of the equations (7.6), (7.7), (7.8), (7.9) and (7.11) into the derivative form of equation (7.10) produces the following formula

\[ \frac{d \rho^{(s)}}{d\gamma^{(s)}} = \frac{1}{2} \frac{2 \gamma}{b} \rho^{(s)} + 2 \gamma \Delta \rho^{(s)} \frac{d \Delta \rho^{(s)}}{d\gamma^{(s)}} \]  
(7.12)

At this point, it is worthwhile to analyze the effect of the excess dislocation density \( \Delta \rho^{(s)} \) in the crystal lattice, or better known as the Geometrically Necessary Density of Dislocations (GND). The excess of dislocations of one sign (non-redundant dislocations) produces a local elastic distortion into the crystal lattice, which is characterized by the crystal curvature tensor. In the case of a single slip system, the curvature can be represented by a scalar \( \kappa \) that characterizes the gradient of the crystal orientation along the slip direction (unit rad/m). In this case, the relation between the dislocation density excess along the slip system \( s \) and the crystal curvature is established by Ashby [17] through the following expression

\[ \Delta \rho^{(s)}_{\kappa} = \frac{\kappa^{(s)}}{b} \]  
(7.13)

The evolution and distribution of misorientations across dislocation substructures during plastic deformation has been extensively studied by several researchers, among whom Hansen [18] presents a state-of-the-art review on this topic. Based on the formation mechanisms, the grain substructures can be classified in Geometrically Necessary Boundaries (GNB) or Incidental Dislocation Boundaries (IDB). The IDBs are formed due exclusively to statistical fluctuations of the dislocation fluxes, whereas the GNBs are produced by deterministic processes in addition to the statistical fluctuations [19]. As a consequence, the GNB creates higher misorientation angles across their boundaries in comparison to the IDB. The frequency distribution of the normalized misorientation angle of both types of dislocation boundary can be separately described by a Rayleigh distribution [20] at any plastic strain, which is also a consequence of the scaling hypothesis [21]. Based on Pantleon [11], the average misorientation angle for GNB, \( \bar{\theta}_{\text{GNB}} \), as a function of the shear strain is given by the following equation
The constants $C_2^*$ and $\sigma_{imb}^*$ are respectively related to the statistical fluctuations and the activation imbalance processes of the GNB formation and evolution [19]. In the case of the IDBs, the constant $\sigma_{imb}^*$ is equal to zero due to its stochastic nature.

In this dissertation, only the misorientation angles associated to GNBs are considered for their contribution to the GND density distribution. It is assumed that the crystal curvature has a similar distribution and evolution during deformation than the average misorientation angle of the GNBs. This assumption implies that the width of the GNBs is considered to be constant during the entire deformation process. Therefore, the dependency of the crystal curvature along the slip system $s$ on the shear strain along the same slip system is established by the following expression.

The inclusion of equation (7.15) into (7.12) produces the final form of the equation describing the evolution of dislocation density with respect to the shear strain on a specific slip system.

The first term of the equation corresponds to the hardening produced by the accumulation of dislocations (dislocation storage). The second term represents a softening process produced by the annihilation of dislocations by dynamic recovery. The third term involves an extra hardening component produced by the excess of dislocations of one sign and the ensuing inhibition of the annihilation process. As Pantleon [11] mentioned, the GND density evolution term produces a constant work hardening term at high strains that simulates stage IV and which is not possible to obtain using the classical Kocks-Mecking model.

The accumulation of dislocations during the deformation process is controlled by the changes in the mean free path. In the case of the ferrite grains, the mean free path $\bar{\lambda}$ incorporates two different contributions. First, as grain boundaries represent an obstacle for dislocation movement, the grain size ($d_g$) is included as part of the mean free path.
The second component is attributed to the formation of dislocation structures during deformation where the mean free path is reduced as the dislocation density increases. Therefore, the mean free path is calculated as follows

\[ \frac{1}{\lambda} = \frac{1}{d_g} + \frac{1}{\beta} \sqrt{\sum_{s=1}^{NSS} \rho_s^{(s)}} \] (7.17)

where \( \beta \) is a parameter accounting for the details of the dislocation structures.

It is important to highlight that equation (7.17) employs the total dislocation density over all slip systems instead of using only dislocations of the same slip system. This assumption is justified based on the idea that immobilized dislocations from any slip system act as obstacles for the dislocation flux on any given slip plane.

A similar equation is used to calculate the mean free path in the case of martensite although instead of using the grain diameter \( d_g \), another more appropriated length is employed. Further details are given in section 7.2.3.

The present model for describing the work-hardening behaviour of the slip systems of martensite and ferrite mainly consists of parameters characterizing the chemical content of the phases and other measurable microstructural features. The work hardening model contains in total five fitting parameters that are related to the static yield stress \( (C_1) \), the mean free path \( (\beta) \), the annihilation distance \( (\gamma) \) and the GND evolution \( (C_2, \sigma_{imb}^2) \). The increment of shear strain for each of the slip systems is determined from a crystal plasticity framework, described in the next section.

### 7.2.2 Crystal plasticity framework

The previously described work-hardening model allows obtaining the resolved shear stress, in addition to the dislocation content, based on the activity of the slip systems. This model will be coupled to a crystal plasticity framework, which allows deriving, for a given local velocity gradient, the shear rates of the different slip systems of a single crystal entity (either a ferrite grain or a martensite lath). The Taylor-Bishop-Hill method [22] is applied to this purpose and the method also allows calculating the local deviatoric Cauchy stress tensor and the rotation of the crystal lattice for a given local velocity gradient.

Based on the kinematical relations ruling the slip activity of a single crystal system, the local velocity gradient \( \mathbf{I} \) is composed of the shear rate of each of the slip systems and the rotation rate of the crystal lattice \( \mathbf{\omega}^L \). The equation relating the previous tensorial relation is given by
Modelling of DP steels

\[ l = \sum_{s=1}^{NSS} K^{(s)} \gamma^{(s)} + \omega^{L} \]  
\( \text{(7.18)} \)

Where the tensor \( K^{(s)} \) is a crystallographic factor equal to the dyadic product between the unit vector \( \hat{b}^{(s)} \), which is parallel to the slip direction of the slip system \( s \), and the unit vector \( \hat{n}^{(s)} \), which is perpendicular to the slip plane. In other words,

\[ K^{(s)} = \hat{b}^{(s)} \otimes \hat{n}^{(s)} \]  
\( \text{(7.19)} \)

Because the local velocity gradient tensor is imposed, the primary challenge of equation (7.18) is two-fold, \( (i) \) to resolve which slip systems are active and \( (ii) \) to calculate the shear rate \( \gamma^{(s)} \) of the active slip systems.

As the spin rate is another unknown, it is convenient to decompose the local velocity gradient of equation (7.18) into its symmetric and antisymmetric components; where both of them can be written separately as

\[ d = \sum_{s=1}^{NSS} \frac{1}{2} \left( K^{(s)} + K^{(s)T} \right) \gamma^{(s)} \]  
\( \text{(7.20)} \)

\[ w = \sum_{s=1}^{NSS} \frac{1}{2} \left( K^{(s)} - K^{(s)T} \right) \gamma^{(s)} + \omega^{L} \]  
\( \text{(7.21)} \)

The local strain rate tensor \( d \) and the local spin tensor \( w \) are a function of the slip rate on the slip system under consideration; whereas in the expression for the \( d \), the rotation rate of the crystal lattice \( \omega^{L} \) has vanished (equation (7.20)) due to the antisymmetric nature of that tensor.

As the strain rate tensor contains six components, equation (7.20) results in a set of the same number of linear equations. However, the number of unknowns in these equations is equal to the number of slip systems of the crystal lattice; e.g. a total of 24 variables are found in the case of BCC steel if the \( \{110\}\langle111\rangle \) and \( \{112\}\langle111\rangle \) slip systems are considered. Moreover, as plastic deformation occurs without volume change, the trace of the deformation rate tensor must be equal to zero \( (d_{11}+d_{22}+d_{33}=0) \). This extra equation reduces the total number of linear equations from six to five. The infinite number of solutions resulting from the five equations in (7.20) can be reduced to a few solutions only by introducing an objective function to minimize. As originally suggested by Taylor [23], an energy criterion is added to the set of equations where the frictional power dissipation in the slip systems must be minimal. In other words, the following equation must be satisfied
\[
P = \sum_{s=1}^{\text{NSS}} \tau^{(s)} \left| \dot{\gamma}^{(s)} \right| \rightarrow \text{min} \quad (7.22)
\]

As all involved equations have a linear character, the problem can be solved by linear programming, where the objective function to minimize is given by (7.22) and is subjected to the constraints of the set of equations in (7.20). The simplex method [24] is used to obtain the set of shear rates that minimizes the plastic power dissipation where a maximum of five slip systems are necessary to accommodate the plastic deformation.

The calculation of the local deviatoric Cauchy stress tensor \( \mathbf{S}' \) is performed once the active slip systems are known based on the resolved shear stress of the crystal system. It is important to mention that the resolved shear stresses of all slip systems of the same crystal are equal in the present model. The equations to calculate the components of the stress tensor \( \mathbf{S}' \) are given by the projections of the tensor \( \mathbf{S} \) onto the active slip systems

\[
\text{sign}(\dot{\gamma}^{(s)}) \tau^{(s)} = \frac{1}{2} \left( \mathbf{K}^{(s)} + \mathbf{K}^{(s)T} \right) : \mathbf{S}' \quad (7.23)
\]

The solution to the set of the linear equations derived from (7.23) gives the resulting stress tensor submitted to the prescribed conditions of the local velocity gradient.

Finally, the reorientation of the crystal lattice is calculated based on equation (7.21) by the following expression

\[
\mathbf{\omega}^L = \mathbf{w} - \sum_{s=1}^{\text{NSS}} \frac{1}{2} \left( \mathbf{K}^{(s)} - \mathbf{K}^{(s)T} \right) \dot{\gamma}^{(s)} \quad (7.24)
\]

The resulting components of the tensor \( \mathbf{\omega}^L \) are used to obtain the necessary rotation to update the orientation of the crystal after each strain increment.

The Taylor-Bishop-Hill method is applied directly for the deformation behaviour of the ferrite grains and of the second phase inclusions. However, some extra considerations are made for describing the crystal plasticity of the martensite, which are given in the following section.

### 7.2.3 Second phase inclusions

As mentioned in Chapter 5.2, the martensite of the DP steels studied in this dissertation is observed in three different morphologies: (i) as martensite bands, (ii) as martensite islands and (iii) within bainite. In the last case, the martensite laths are located between bainitic ferrite which also has lath morphology. In contrast to the stiff
behaviour of the pure martensite islands, the combination of a relatively soft phase, bainitic ferrite, and a harder phase, martensite, results in a broad range of mechanical response upon loading. Therefore, the overall response of the bainite is controlled by the individual strength, structure and volume proportion of its microstructural components: bainitic ferrite and martensite. In turn, the microstructural features of both components are controlled by the heat treatment, as is pointed out in section 5.2. Therefore, in the third case, bainite is considered as the second phase in the present model.

In this case the 2nd phase has a sheaf morphology and these sheaves are composed of both bainitic ferrite ($\alpha_b$) and martensite ($\alpha'$), which are the product of a solid-to-solid phase transformation from a parent austenite grain. A crystallographic orientation relation exists between the parent austenite grain and its products where the most commonly accepted orientation relationships (OR) are the Kurdjumov-Sachs (K-S) and the Nishiyama-Wasserman ORs [25]. In both cases, it is generally assumed that a close-packed $\{110\}_\alpha$ plane of the bainitic ferrite or the martensite (BCC structure) is parallel to a $\{111\}_\gamma$ plane of the parent austenite (FCC structure). The difference between both ORs is that the K-S OR considers a $\{111\}_\alpha$ direction of the products parallel to a $\{110\}_\gamma$ of the austenite whereas the N-W OR proposes a $\{110\}_\alpha$ direction parallel to a $\{112\}_\gamma$ direction of the austenite. The resultant variants produced by both ORs only differ by a rotation of 5.25° around the axis perpendicular to the common close-packed planes. However, it has been experimentally observed that the OR between the parent austenite and its BCC products is not exact to either ORs but lies between the N-W and K-S OR [26-28]. As both orientation relationships are in reality close to each other, the N-W OR is adopted on the determination of the variants of bainitic ferrite and martensite. This preference is based on the lower number of generated variants of the N-W OR in comparison to the 24 variants of the K-S OR. This choice considerably reduces the computational effort in the model simulations.

The entire set of crystallographic variants of the N-W OR is considered to appear after the transformation from the parent austenite and each of these orientations is used as input for the crystal plasticity model describing the local response of the sheaf of bainitic ferrite and martensite. The crystal orientation $g_{\alpha_b,\alpha'}^\gamma$ of each of the variants $\nu$ is obtained by applying the following transformation

$$g_{\alpha_b,\alpha'}^\gamma = T_{NW}^\nu g_{\gamma}$$ (7.25)
Chapter 7

Where $g_\gamma$ is the orientation of the parent austenite grain and $T_{NW}^v$ is the orientation transformation relating the parent austenite grain to the variant $v$. The corresponding transformations of the N-W OR are grouped and given as quaternions [29] in Table 7.1.

Table 7.1: 12 crystallographic variants corresponding to the N-W orientation relationship. The data are based on [30]

<table>
<thead>
<tr>
<th>$v$</th>
<th>Parallel planes</th>
<th>Parallel direction</th>
<th>Quaternion representation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[2\overline{1}T]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.674; 0.214, 0.628, 0.325}</td>
</tr>
<tr>
<td>2</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[1\overline{2}T]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.247; -0.293, -0.596, -0.706}</td>
</tr>
<tr>
<td>3</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[\overline{1}T2]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.921; -0.078, 0.032, -0.381}</td>
</tr>
<tr>
<td>4</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[\overline{2}1T]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.674; 0.214, -0.628, -0.325}</td>
</tr>
<tr>
<td>5</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[1\overline{2}T]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.247; -0.293, 0.596, 0.706}</td>
</tr>
<tr>
<td>6</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[\overline{1}T2]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.921; -0.078, -0.032, 0.381}</td>
</tr>
<tr>
<td>7</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[21T]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.325; 0.628, 0.214, 0.674}</td>
</tr>
<tr>
<td>8</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[\overline{1}2T]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.706; 0.596, 0.293, -0.247}</td>
</tr>
<tr>
<td>9</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[\overline{1}12]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.381; -0.032, 0.078, -0.921}</td>
</tr>
<tr>
<td>10</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[2\overline{1}1]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.628; 0.325, -0.214, 0.674}</td>
</tr>
<tr>
<td>11</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[\overline{1}21]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.032; -0.381, 0.078, 0.921}</td>
</tr>
<tr>
<td>12</td>
<td>(111)$<em>\gamma$ $(011)</em>\alpha$</td>
<td>$[\overline{1}12]<em>\gamma$ $[0\overline{1}1]</em>\alpha$</td>
<td>{0.596; 0.706, -0.293, -0.247}</td>
</tr>
</tbody>
</table>

The hardening rule followed by the slip systems of each of the variants is the same as the one proposed for the ferrite grains and established by equation (7.16). Regarding the term which represents the dislocation storage mechanism, the mean free path for martensite is also partly considered to be controlled by the dislocation structures as obstacles. The other factor contributing to the mean free path term is the block size of the martensite $d_m$ [31] and the bainitic ferrite lath size $d_b$ [32]. Then, the mean free path for both cases is given by the following expression

$$\frac{1}{\lambda} = \frac{1}{d_{m,b}} + \frac{1}{\beta} \sqrt{\sum_{s=1}^{N_{SS}} \rho^{(s)}}$$

(7.26)
The overall mechanical response of the inclusion composed of bainitic ferrite and martensite originating from a common parent austenite grain is obtained using the full-constraint assumption. This means that the same velocity gradient is imposed on all of the products and its variants. In addition, if it is assumed that the length and width of the bainitic ferrite laths are equal to the corresponding dimensions of the martensite laths and the only difference is their lath size, then the corresponding deviatoric Cauchy stress tensor of the second phase inclusion $\mathbf{S}_i'$ is given by the weighted sum of the stresses of the bainitic ferrite $\mathbf{S}_b'$ and of the martensite $\mathbf{S}_m'$ laths

$$
\mathbf{S}_i' = \frac{1}{(d_b + d_m)N^v} \sum_{n=1}^{N^v} \left( d_b \mathbf{S}_b^{(n)} + d_m \mathbf{S}_m^{(n)} \right)
$$

(7.27)

where $N^v$ is the total number of the crystallographic variants given by the N-W OR and thus equal to 12.

The modelling framework employed to simulate the mechanical behaviour of the second phase of the DP steels takes into account several microstructural factors contributing to its strength such as crystal orientation, characteristic lengths, transformation dislocation density content, carbon content and morphological structure.

### 7.2.4 Ferrite-martensite homogenization scheme

It is well-known that the interaction between ferrite and martensite is one of the principal factors influencing the plastic deformation behaviour of DP steels. Assuming that martensite deforms as much as ferrite, i.e. full-constraint or iso-strain approach, represents a stiff and unrealistic approach given the importance of the interaction between both phases. Several homogenization schemes have been proposed, from different approaches based on the rule of mixtures [33] to robust RVE Finite Element [34] methodologies. In this dissertation, the strain partitioning between the ferrite grains and the second phase inclusions is simulated based on three different approaches: (i) an iso-strain partitioning scheme, (ii) an iso-stress partitioning scheme and (iii) a rule of mixture based on equal plastic power dissipation referred to as IsoW assumption [9].

In all homogenization schemes, the RVE of the model is considered to be composed of several components, which are referred to as DP units. Each DP unit is represented by a ferrite grain of certain crystal orientation enclosing a second phase inclusion. As mentioned in the previous section, the inclusion of one DP unit is composed of crystallographic variant products of one common parent austenite grain. The deviatoric Cauchy stress tensor $\mathbf{S}_U'$ and the corresponding macroscopic strain rate...
\( d_U \) of each DP unit are calculated based on a mean field approach where the following expressions allows calculating the stress (strain) of the DP unit by averaging the microscopic stresses (strains) of its constituents:

\[
S'_U = f_v S'_i + (1 - f_v) S'_f \\
d_U = f_v d_i + (1 - f_v) d_f
\]

where \( S'_i \) and \( S'_f \) are respectively the deviatoric stress tensors of the second phase inclusion and the ferrite grain, \( d_i \) and \( d_f \) are the strain rate of the inclusion and the ferrite grain and \( f_v \) is the total volume fraction of the material occupied by the inclusions. As observed in equations (7.28) and (7.29), it is assumed that equal numbers of inclusions and ferrite grains are present within the RVE and, in addition, the fraction of an inclusion within any DP unit is made equal to the total fraction of inclusions of the entire RVE. The postulation of equations (7.28) and (7.29) are insufficient to completely describe the mechanical behaviour of the RVE unit and its components because some additional assumptions related to strain partitioning between the inclusion and the matrix need to be incorporated in the model.

**Iso-strain assumption**

One of the simplest assumptions on the strain partitioning between two phases of different mechanical properties is the iso-strain condition. This methodology proposes that both phases undergo the same prescribed strain rate in order to ensure strain compatibility between the inclusion and the matrix. Moreover, based on equation (7.29), the homogenised strain rate of the DP unit is also equal to the strain rate in each of the constituents. The general equations describing the iso-strain conditions are given by

\[
d_U = d_i = d_f
\]

However, the stress equilibrium between the inclusion and the matrix is ignored by the iso-strain assumption and this violation results in an upper-bound limit response of the real mechanical behaviour of the DP unit.

**Iso-stress assumption**

Whereas the iso-strain assumption represents the upper-bound limit mechanical response, the lower-bound limit is controlled by the iso-stress condition; i.e. the stress tensor of the inclusion and the matrix are assumed to be equal. Such condition ensures the stress equilibrium within the DP unit although, in turn, the strain compatibility is
violated. As the Taylor-Bishop-Hill method used for describing the crystal plasticity
behaviour of the phases uses as input the velocity gradient, some modifications are
applied to simulate the iso-stress condition.

The strain rate directions are considered to be equal for the inclusion and the matrix
and the parameter $\chi$ is proposed to describe the strain partitioning between the
constituents of the DP unit as expressed in the following equation

$$d_i = \chi \; d_f$$

The parameter $\chi$ is determined in an iterative fashion until the equality between the
von Mises stresses of the inclusion and the matrix is ensured,

$$\sigma'_{viMi} = \sigma'_{viMf}$$

In case that the von Mises stress within the inclusion is larger than the stress of the
matrix even if the inclusion undergoes no deformation ($\chi = 0$) then it is assumed that the
inclusion remains undeformed and its stress mirrors the stress of the matrix. This
situation is expected at low deformations where the inclusion stress is several orders of
magnitude larger than the stresses of the matrix.

The difference of the previously proposed methodology with respect to the original
iso-stress assumption is that the corresponding components of the stress tensors are not
necessary equal in equation (7.32), and therefore the stress equilibrium is not strictly
guaranteed.

The root finding algorithm of Van Wijngaarden-Dekker-Brent [24] is used to
determine the value of $\chi$ that satisfies the condition given by equation (7.32).

*IsoW assumption.*

The iso-strain and the iso-stress assumptions represent respectively the upper- and
lower-bound limits of the actual strain partitioning during plastic deformation of a
particle-matrix material [33]. One intermediate approach which does not require the
addition of fitting parameters is the phenomenological IsoW assumption. Bouaziz and
Buessler [9] proposed that the strain partitioning between inclusion and matrix is
dictated by an iso-energy condition; i.e. the plastic power dissipation should be equal for
each of the constituents. This criterion was originally implemented for simulating the
work-hardening behaviour of composite materials during uni-axial deformation and in
order to implement it in a crystal plasticity framework, one additional assumption is
required. It is assumed that the strain rate of the inclusion and the matrix are
proportional to each other by a factor $\chi$: 
The parameter $\chi$, similarly to the proposed iso-stress condition, determines the strain partitioning between the inclusion and the matrix and, in this particular case, the parameter range is expected to be between zero and one due to the higher strength of the martensite in comparison to the ferrite grains.

The IsoW assumption is established by assuming that the plastic power dissipation per volume unit of the inclusion and the matrix are equal, which is expressed by the following equation:

$$ S_i' : d_i = S_f' : d_f $$

(7.34)

Similar to the iso-stress assumption, the root finding algorithm of Van Wijngaarden-Dekker-Brent [24] is employed to determine the value of $\chi$ that satisfies the isoW condition of equation (7.34).

The three different schemes are applied to describe the strain partitioning within the DP units and are compared between each other for better simulation of the experimental data as well as computational functionality.

7.2.5 General Homogenization scheme

The homogenization method adopted to describe the strain partitioning among the DP units is the full-constraint assumption. Each of the DP units is assumed to follow the same velocity gradient as the one imposed to the entire RVE. This condition is expressed as:

$$ l_U = L_{RVE} $$

(7.35)

The deformation rate necessary as input for all strain partitioning homogenization schemes is calculated from the velocity gradient as follows:

$$ d_U = \frac{1}{2} (l_U + l_U^T) $$

(7.36)

The global response of the RVE is given by the contribution of each and all of the DP units constituting the RVE. Therefore, the corresponding macroscopic deviatoric Cauchy stress is expressed as:

$$ S'_{RVE} = \frac{1}{n_E} \sum_{i=1}^{n_E} S'_U_i $$

(7.37)
7.3 Boundary conditions: Uniform deformation of tensile test

In order to allow a comparison between the simulated mechanical response of the DP steels with the corresponding experimental data, the boundary conditions of the tensile tests need to be correctly implied in the model. The constraint conditions of a tensile test sample consist mainly of a macro-stress with the normal stress component along the tensile direction as the only non-zero component. For example, if the tensile axis is along the component $x_1$, then the only non-zero component is $\sigma_{11}$. The present model is based on the Taylor-Bishop-Hill crystal plasticity method and therefore the imposed conditions are based on velocity gradient tensors rather than stress conditions which are more convenient for simulating uni-axial elongation. Thus, the problem relies on determining the strain conditions which lead to the stress-based tensile test constraints. One solution is proposed by Bunge et al. [35] where the average Taylor factor $\bar{M}$ is set as a function of the strain tensor and the one which minimizes the Taylor factor corresponds to the real tensile test conditions. The minimization problem is then reduced to one dimension because the global velocity gradient tensor, given in equation (7.38), is defined as a function of the $l_{11}$ component (which is imposed) and the contraction ratio $q$, which remains to be determined.

$$L_{RVE} = \begin{pmatrix} 1 & 0 & 0 \\ 0 & -q & 0 \\ 0 & 0 & q-1 \end{pmatrix} \begin{pmatrix} l_{11} \end{pmatrix}$$  \hspace{1cm} (7.38)

This method is based on the minimization of plastic power dissipation energy per unit volume of the system, which under certain conditions is proportional to the average Taylor factor. One of the conditions for this proportionality is that the critical resolved shear stress is equal over all crystallites and on all slip planes. However, the incorporation of work-hardening behaviour makes the grains to harden differently and, as a consequence, the minimum average Taylor factor does not coincide anymore with the minimum of the plastic dissipation energy for the same $q$ value.

A similar methodology is followed for the current tensile test simulations although instead of using a minimization criterion for the Taylor Factor, a root finding procedure is employed to determine the condition at which the two deviatoric stress components perpendicular to the tensile direction ($\sigma'_{22}$ and $\sigma'_{33}$) are equal. The justification for this is based on the following explanation. The stress state obtained by the Taylor-Bishop-Hill crystal plasticity method is a linear combination of shear stresses along different slip systems; in other words, no hydrostratic stresses are obtained within the volume.
Therefore the resultant stress tensor is a deviatoric stress; i.e. a stress tensor without the hydrostatic component and defined by equation (7.39).

\[
S'_{RVE} = S_{RVE} - I \cdot \text{trace}(S_{RVE})
\]  
(7.39)

where \(\text{trace}(S_{RVE})\) is a constant equivalent to the sum of the main diagonal elements of the stress tensor. The subsequent property of the deviatoric stress \(S'_{RVE}\) is that its trace is equal to zero. This condition implies that no deviatoric stress can satisfy the condition of only one non-zero component in the main diagonal which is the condition for tensile stresses. So, it is obvious that the tensile test constraint condition has to be formulated for deviatoric stresses. The hydrostatic component is a diagonal matrix with its components all equal to the same constant, \(\text{trace}(S)\). This matrix is afterwards subtracted from the stress tensor in order to obtain the deviatoric stress and thus all components of the principal diagonal of the deviatoric stress are subtracted with the same constant. Therefore the corresponding deviatoric tensor from the tensile test constraint condition is a diagonal matrix with \(\sigma'_{22}\) equal to \(\sigma'_{33}\) and both components are equal to the negative half of \(\sigma'_{11}\) as given in equation (7.40).

\[
S'_{RVE} = \begin{pmatrix} 1 & 0 & 0 \\ 0 & -0.5 & 0 \\ 0 & 0 & -0.5 \end{pmatrix} \sigma'_{11}
\]  
(7.40)

Hereby, the strain tensor related to the real tensile test conditions for each strain increment is determined by finding the contraction ratio \(q\) at which the difference between \(\sigma'_{22}\) and \(\sigma'_{33}\) is equal to zero. These are the boundary conditions applied to the simulations and the root finding algorithm employed to determine the corresponding \(q\) value is based on the Van Wijngaarden-Dekker-Brent approach [24].

In order to illustrate and verify the method employed to determine the uni-axial tensile conditions, Figure 7.2 displays the simulated values of the stress component along the tensile direction \(\sigma'_{11}\), the difference between the deviatoric Cauchy stress components \(\sigma'_{22}\) and \(\sigma'_{33}\) and the plastic dissipation energy per volume unit \((W = S'_{RVE} : D_{RVE})\) as a function of the contraction ratio \(q\). These values are calculated using the IsoW scheme, the experimental data of the Reference DP steel and a strain increment along the tensile direction equal to 0.01. As can be observed on the graph, the minimum of the plastic dissipation energy correspond to the same \(q\) value at which the difference between \(\sigma'_{22}\) and \(\sigma'_{33}\) is zero. It is also important to mention that the stress
component along the tensile direction, $\sigma'_{11}$, is not necessary minimum at that $q$ value, which is in contradiction to the methodology suggested by Li et al. [36].

$$r \text{ value}$$

![Figure 7.2: Example for the determination of the contraction ratio $q$ for tensile test conditions. The contraction ratio $q$ at which $\sigma'_{22}$ is equal to $\sigma'_{33}$ coincides with the minimum of the plastic dissipation power.](image)

### 7.4 Identification of microstructural and experimental parameters

The value of the model parameters related to microstructural characteristics of all DP steels are listed in Table 7.2. Some of the parameters are taken from the microstructural characterization made on the DP steels in Chapter 5. This is the case of the martensite volume fraction, the ferrite grain size, the bainitic ferrite lath width and the martensite carbon content. The rest of the microstructural parameters are extracted from different literature sources. For instance, the carbon content in the bainitic ferrite is assumed to be equal to the ferrite phase value, which is taken from similar DP steel composition from the studies by Thomser et al. [14]. The lath width of the martensite is taken from the experimental studies by Morito et al. [31] in a Fe-0.2C-2Mn alloy. The initial dislocation density content of the ferrite grains is assumed to be equal for all DP steels and, and from the work of Bouquerel et al. [37], an initial value of $3 \cdot 10^{12} \text{ [m}^{-2}\text{]}$ is proposed. This dislocation density content was originally estimated by Antoine et al. [38] using TEM-based analysis in an undeformed IF steel. In the case of the initial dislocation density contents within the martensite and the bainitic ferrite laths, the empirical expression suggested by Takahashi and Bhadeshia [39] is employed. This
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expression estimates the dislocation density of the product phases resulting from a solid-to-solid phase transformation and is given by

\[
\log \rho_m = 9.2848 + \frac{6880}{T} - \frac{1780360}{T^2}
\]  

(7.41)

where \( T \) is either the martensite or the bainite transformation temperature expressed in Kelvin. The equation has been quoted to be valid only within the temperature range of 300-650 [°C].

<table>
<thead>
<tr>
<th>Microstructural parameter</th>
<th>Source</th>
<th>Ref</th>
<th>SH</th>
<th>SC</th>
<th>HT</th>
</tr>
</thead>
<tbody>
<tr>
<td>( f_V ) Martensite/Bainite area fraction, %</td>
<td>Table 5.1</td>
<td>10.3</td>
<td>10.0</td>
<td>7.5</td>
<td>10.0</td>
</tr>
<tr>
<td>( d_g ) Ferrite grain size, ( \mu m )</td>
<td>Table 5.1</td>
<td>7.4</td>
<td>10.3</td>
<td>6.8</td>
<td>4.2</td>
</tr>
<tr>
<td>( d_b ) Bainitic ferrite lath width, ( \mu m )</td>
<td>Table 5.1</td>
<td>0.14</td>
<td>0.25</td>
<td>0.36</td>
<td>0.43</td>
</tr>
<tr>
<td>( d_m ) Martensite lath width, ( \mu m )</td>
<td>[31]</td>
<td></td>
<td>0.23</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( C_f ) Ferrite Carbon content, %</td>
<td>[14]</td>
<td></td>
<td>0.007</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( C_m ) Bainitic ferrite carbon content, %</td>
<td>[14]</td>
<td></td>
<td>0.007</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( C_m ) Martensite carbon content, %</td>
<td>Table 5.2</td>
<td>0.33</td>
<td>0.37</td>
<td>0.48</td>
<td>0.31</td>
</tr>
<tr>
<td>( \rho_{f,0} ) Initial ( \rho ) for ferrite grains, ( m^{-2} )</td>
<td>[37]</td>
<td></td>
<td>3·10^{12}</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \rho_{m,0} ) Initial ( \rho ) for bainitic ferrite laths, ( m^{-2} )</td>
<td>Eq. (7.41)</td>
<td></td>
<td>4.5·10^{14}</td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \rho_{m,0} ) Initial ( \rho ) for martensite laths, ( m^{-2} )</td>
<td>Eq. (7.41)</td>
<td></td>
<td>7.4·10^{15}</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

As mentioned in Chapter 5.2, the martensite transformation temperature for all DP steels is observed at around 200 [°C] whereas the bainitic starting transformation temperature occurs between 600 and 700 [°C]. As both temperature are out of the temperature range of equation (7.41), the extreme values are used; in other words, the
transformation temperature of the martensitic and bainitic transformation are assumed to be 300 and 650 [°C], respectively. The corresponding initial dislocation density contents of both transformations are given in Table 7.2.

The active slip systems considered for the crystal plasticity framework of ferrite and martensite were the family systems \{110\}\{111\} and \{112\}\{111\}. This choice is justified by experimental observations at room temperature [40] as well as the small difference produced on simulation results for full-constraint models when the slip system \{123\}\{111\} is added [41]. The ratio of the critical resolved shear stress on the \{112\} and the \{110\} glide planes was assumed to be equal to one.

The input data for the crystal plasticity model consist of crystallographic data of both the ferrite grains and of the inclusions. In the case of the ferrite grains, the initial texture data for all DP steels is discretized from the ODF calculated from the crystal orientation maps in Chapter 5 (cf. Figure 5.3). In the case of the bainitic ferrite and the martensite, the texture used as input corresponds to the parent austenite grains. Although there is no available data of the austenite formed during the intercritical heat treatments of the DP steels of this study, the texture of a TRIP steel with similar thermo-mechanical processing history is used to this purpose. The data corresponds to heat treatment experiments on a TRIP steel grade sheet heated up to 800 [°C] where the texture was acquired in-situ using high-energy synchrotron radiation [42]. The ODF of the austenite phase of the TRIP steel is shown in Figure 7.3.

![Figure 7.3: ODF of austenite at intercritical annealing (800 °C) of a TRIP grade steel represented on three different \(\varphi_2\) sections. The ODF was experimentally obtained by X-ray Synchrotron Radiation experiments [42].](image-url)
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The discretization of both the ferrite and the austenite textures in a set of individual orientations that can be fed to the model is made by routines of the open source software MTEX for texture analysis [43]. In total, 28 orientations are used to represent the parent austenite texture and 70 orientations for representing the ferrite textures. As each DP unit is composed of a ferrite grain and a second phase inclusion, which is characterized by the parent austenite orientation, the simulations considers all possible combinations of ferrite and parent austenite orientations which gives a total of 1960 (28x70) DP units per simulation.

7.5 Calibration of the model

There are in total five model parameters to be identified: the constant of the initial critical resolved shear stress $C_1$ from equation (7.5), the shape parameter $\beta$ and annihilation distance $\gamma$ from equation (7.16) and the parameters $C_2$ and $\sigma_{imb}^2$ of the same equation controlling the evolution of GND along individual slip systems during the shear deformation. As can be observed, these parameters correspond exclusively to the work-hardening equations and this means that the other model stages, such as the crystal plasticity or the inclusion-matrix strain partitioning scheme, do not have any fitting parameters. However, a set of fitting parameters is determined for each of the strain partitioning schemes which implies that the calibration procedure is performed for the iso-strain, the iso-stress and the IsoW conditions.

The calibration of the model is performed on the experimental data of the reference DP steel and is constituted of two consecutive stages. The first part consists in adjusting the parameter $C_1$ of the model in order to represent the yield stress of the material. The yield stress $S_{0.2}$ is influenced exclusively by the constant contained within the static yield stress $\tau_0$. The fitting procedure is based on the Van Wijngaarden-Dekker-Brent root-finding algorithm [24].

In the second part of the calibration the remaining four parameters related to the work-hardening of the slip systems are adjusted. The data available for this adjustment are the experimental true stress-strain and work-hardening curves between the initiation of the plastic deformation up to the necking onset of the sample, the GND at 12.5% and at a strain close to fracture (advanced necking condition), and the strain and stress at fracture and at the advanced necking condition. Table 7.3 shows the strain, stress and GND values corresponding to the conditions after necking onset of the Reference DP steel. The four model parameters are fitted using the downhill simplex method [24], whereby the function to minimize is given by the sum of the square relative errors between the experiments and the simulation results. As it is mentioned in the following
sections, it is not possible to satisfy all experimental conditions. Therefore, different weights are used to each of the square relative errors in order to give priority to some of the experimental conditions as well as to compensate the influence of the different nature of the data on the relative errors. Therefore, the final minimization function employed for the simulations with a strain increment of 0.01 is given by the following expression.

\[
    f_{\text{min}} = 5 \cdot e_{TC} + 10 \cdot e_{WA} + 2 \cdot e_{GND}
\]

where \(e_{TC}\), \(e_{WH}\), \(e_{IA}\) and \(e_{GND}\) are the square relative errors of the tensile test curve along the uniform deformation, the work-hardening behaviour up to the necking onset, the individual stress-strain values calculated by image analysis and given in Table 7.3 and the average GND of the ferrite grains at certain strains (cf. Table 7.3), respectively.

\[7.42\]

Table 7.3 Experimental data of the reference DP steel corresponding to the conditions after uniform deformation.

<table>
<thead>
<tr>
<th>Experimental parameter</th>
<th>Source</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\Delta \rho_U)</td>
<td>GND at (\varepsilon = 0.118), m(^{-2})</td>
<td>Table 5.4</td>
</tr>
<tr>
<td>(\Delta \rho_N)</td>
<td>GND at Necking, m(^{-2})</td>
<td>Table 5.4</td>
</tr>
<tr>
<td>(\varepsilon_N)</td>
<td>Strain at Necking, mm/mm</td>
<td>Table 5.4</td>
</tr>
<tr>
<td>(\sigma_N)</td>
<td>Stress at Necking, MPa</td>
<td>Table 5.4</td>
</tr>
<tr>
<td>(\varepsilon_F)</td>
<td>Strain at Fracture, mm/mm</td>
<td>Table 5.3</td>
</tr>
<tr>
<td>(\sigma_F)</td>
<td>Stress at Fracture, MPa</td>
<td>Table 5.3</td>
</tr>
</tbody>
</table>

Although the second part of the calibration is in charge of simultaneously fitting four parameters, in contrast to the single parameter fitted on the first part of the calibration, the advantage is that each fitting parameter has a higher influence on the calibration depending on the nature of the experimental data (e.g. tensile tests or GND). For instance, the shape parameter \(\beta\) and the annihilation distance \(y\) have more influence on the fitting of the simulated flow curve to the experimental true stress-strain curve.
within the uniform deformation, whereas the fitting of the simulated GND evolution to the experimental data acquired by EBSD is more sensitive to the other two parameters ($C_2$ and $\sigma_{imb}^2$).

7.6 Simulation results and discussions

7.6.1 Calibration results of the Reference DP steel simulations

The corresponding model parameters for the three different strain partitioning schemes which better fit the experimental data of the Reference DP steel are given in Table 7.4. As can be observed, the order of magnitude of the model parameters is similar between the different strain partitioning schemes and are also in agreement with the literature or its physical interpretation. For instance, the shape parameter $\beta$ has similar values to the corresponding parameter in the model of Bouquerel et al. [37] for ferrite and bainitic ferrite of TRIP. In the model of Bouquerel et al. the parameter $k$ is the multiplicative inverse of $\beta$. In the case of the annihilation distance $y$, the value is relatively higher (around five times) in comparison to the model of Bouquerel et al. [37] although it is still in accord to the scale of its physical meaning where the annihilation distance should be in the range of the atomic scale. Although the definition of the constants describing the GND evolution during deformation is different as originally formulated by Pantleon [11], the magnitude of both constants ($C_2$ and $\sigma_{imb}^2$) have similar order of magnitude than the data reported in [11].

Table 7.4: Model fitting parameters for the different matrix-inclusion strain partitioning schemes after calibration on the experimental data of the Reference DP steel.

<table>
<thead>
<tr>
<th></th>
<th>Iso-strain</th>
<th>Iso-stress</th>
<th>IsoW</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compositional constant ($C_1$)</td>
<td>6.073</td>
<td>2.862</td>
<td>6.073</td>
</tr>
<tr>
<td>Shape parameter ($\beta$)</td>
<td>44.4</td>
<td>53.4</td>
<td>51.7</td>
</tr>
<tr>
<td>Annihilation distance $y$, nm</td>
<td>4.3</td>
<td>3.6</td>
<td>3.8</td>
</tr>
<tr>
<td>$C_2$, $\mu$m$^{-2}$</td>
<td>0.015</td>
<td>0.013</td>
<td>0.016</td>
</tr>
<tr>
<td>$\sigma_{imb}^2$, $\mu$m$^{-2}$</td>
<td>0.306</td>
<td>0.286</td>
<td>0.261</td>
</tr>
</tbody>
</table>
The simulation results of the models using the parameters set of Table 7.4 is displayed in Figure 7.4 and compared with the experimental uniaxial stress-strain data of the reference DP steel. The three approaches are able to simulate the macroscopic stress-strain curve of the tensile test experiments. In general, the difference is minimal between the model and the experimental curves within the range of uniform deformation. However, the models show an underestimation of the stresses close to and at the fracture point. This common discrepancy of all strain partitioning schemes is partly attributed to the origin of the experimental data which is based on the image analysis of the samples geometry. Although the strain (stress) conditions after the onset of necking are assumed to behave as independent slabs along the tensile samples with each slab having uniform strain (stress) conditions along the thickness and the width direction, the real conditions exhibits a more heterogeneous nature where the middle-thickness and middle-width region of the sample suffers the more severe strain (stress) conditions. The strain-stress curve of the uniform deformation region is experimentally calculated from initial geometric conditions as well as assuming homogeneous deformation,
whereas the image analysis employed to obtain the strain-stress values close to fracture is performed in the middle-width of the samples.

Despite the difference between the simulations and the experimental data are small for the macroscopic strain-stress curves, the major discrepancy among the strain partitioning schemes is observed in the mechanical behaviour of the microstructural constituents. As expected, the iso-strain conditions lead to the highest stresses within the inclusions whereas the iso-stress conditions exhibit the opposite with the lowest stresses within the inclusions. In addition, the stress within the DP units is not high enough to reach the yield stress of most of the inclusions and therefore no plastic deformation is produced in the majority of the inclusions, even at high strain values. In addition, this means that all plastic deformation of the DP steel is solely absorbed by the ferrite matrix. The IsoW scheme shows results which are intermediate to the iso-strain and the iso-stress assumptions. It is interesting to observe that the inclusion stress displays values close to the iso-strain conditions, albeit with the principal difference that the total deformation of the inclusion is lower.

The principal advantage of the present model is that it allows analysing the variation of stress within the inclusions based first on the different crystal orientations (texture) of the parent austenite and second on the interaction between the inclusions and the ferrite matrix with different combination of crystal orientations for both constituents. Figure 7.5a shows the average von Mises stress (thick lines) predicted by the models as a function of the macroscopic tensile deformation together with the minimum and the maximum inclusion stresses (thin lines) for all DP units which is represented as a difference in Figure 7.5b. A common aspect among the three strain partitioning schemes is that the variation of inclusion stress is minimum at the beginning of the plastic deformation, increases and finally remains almost constant at high strain values. Contrary to the average inclusion stress values, the iso-stress condition displays the largest stress variation, especially between the beginning of deformation and 0.25 of the tensile true strain. At this point it is important to comment that the iso-stress condition is only applied to the strain partitioning of the inclusion and the matrix of the individual DP units and not to the interaction between the DP units. Therefore, although the von Mises stress between the inclusion and the matrix is the same, the stress between the DP units is different and is the principal reason of the stress variation of the inclusions. In addition, as previously mentioned the inclusions undergo no plastic deformation in this specific case and the stress experienced by the inclusions is exactly the same as the matrix stress. As a consequence, the nature of this large stress variation of the iso-stress scheme is solely ascribed to the deformation of the different
crystallographic variants of the ferrite matrix. Comparing the three strain partitioning schemes, the IsoW approach displays consistently intermediate values between the iso-stress and iso-strain conditions. However, the IsoW condition exhibits the more similar behaviour to the iso-strain scheme in terms of the inclusion stress behaviour during deformation.

Another important difference between the models is revealed by the ratio between the inclusion and the matrix strains and stresses and their evolution during deformation, as can be seen in Figure 7.6. Both ratios represent respectively the average strain and stress partitioning between the inclusion and the ferrite matrix. As expected, the strain and stress partitioning of the IsoW condition is observed between the iso-strain and iso-stress schemes. The constant ratio equal to one between the matrix and inclusion von Mises strains (cf. Figure 7.6a) confirms the imposed condition of the simulation in the iso-strain scheme. The stress partitioning of this scheme exhibits the largest ratio at the beginning of the deformation (cf. Figure 7.6b), which is decreased afterwards as the deformation proceeds. A similar stress partitioning behaviour is observed for the IsoW condition although with values slightly lower than in the iso-strain condition. In contrast to the IsoW and the iso-strain, the stress ratio of the iso-stress scheme is equal to one during the entire deformation process, whereas the strain partitioning is equal to zero due to the higher yield stress of the inclusion in comparison to the ferrite strength at all strains; in other words, the inclusion remains plastically undeformed.
The IsoW scheme is expected to better simulate the experimental strain partitioning between the ferrite matrix and the inclusion. In this aspect, the strain and stress ratio evolution for the IsoW condition, cf. Figure 7.6, are in agreement with other theoretical studies [2] where the strain ratio inclusion/matrix increases as deformation proceeds, while the corresponding stress ratio continuously decreases. To the purpose of comparison with experimental data, Figure 7.7a displays another representation of the strain partitioning behaviour between the inclusion and the matrix. The model results of Figure 7.7a are compared with the digital image correlation experiments (cf. Figure 7.7b) performed on a DP600 steel [8]. It is interesting to observe that the martensite strain of the experimental data has half the values of the iso-strain line, very much in agreement with the IsoW model predictions. However, a major discrepancy exists with regard to the strain partitioning of the ferrite where the model underestimates the deformation of the matrix. As observed on the EBSD data of the DP steels in Chapter 5.4.1, the ferrite matrix suffers a larger deformation in the proximities of the martensite in comparison to the core of the grain which represents the real strain partitioning condition between both constituents. In the case of the IsoW scheme as well as in the iso-strain and iso-stress conditions, the ferrite grains are assumed to accommodate the deformation of the strain partitioning in a homogeneous fashion in contrast to the real heterogeneous behaviour which may produce larger average strains of the ferrite matrix. Therefore, the fact that the homogeneity assumption does not reflect the physical reality provides a possible explanation for the underestimation of the average ferrite matrix strain.
With regard to the GND data, Figure 7.8 displays the evolution of the GND density during tensile deformation for the three assumptions together with the experimental data obtained by EBSD. The three models exhibit similar behaviour for this parameter which is in accord to the similarity of the model parameters for the GND density evolution equation among the three assumptions. The experimental GND data at the strain close to fracture is well predicted by the three models although a common underestimation is observed for the GND value at the end of uniform deformation. This deviation from experimental data is attributed to the questionable assumption of homogenous strain within the grain as well as to the experimental error on the calculation of the GND data. However, the advantage about calibrating the model to experimental GND values is to have an approximation of the order of magnitude of the GND data.

An additional experimental parameter which is used to further evaluate the models and is not employed in the calibration procedure is the $R$ value evolution estimated from
the image analysis performed on the profile geometry of the broken tensile samples. It is important to mention that the $R$ values calculated from the simulations are not the same as the instantaneous $r$ values employed for the boundary conditions. In order to compare the Lankford parameter of the experimental data with the simulation results, the following accumulative equation is employed:

$$ R = \frac{\sum_{\varepsilon_{11}=0}^{\varepsilon_{11}} \Delta \varepsilon_{22}(\varepsilon_{11})}{\sum_{\varepsilon_{11}=0}^{\varepsilon_{11}} \Delta \varepsilon_{33}(\varepsilon_{11})} $$

(7.43)

where $\Delta \varepsilon_{22}$ and $\Delta \varepsilon_{33}$ are the strain increments along the width and thickness directions of the tensile test as a function of the tensile true strain $\varepsilon_{11}$, respectively.

Figure 7.9 shows both the experimental and simulated evolution of the $R$ value along the macroscopic tensile deformation. The first important observation is that the three strain partitioning schemes give similar results of the $R$ value evolution, where the IsoW and the iso-strain condition show only slight differences. In fact, the parameter which has more influence on the $R$ value is the number of DP units used in the calculation which is modified by increasing (or decreasing) the number of crystal orientations to represent the texture of the ferrite and the parent austenite grains. The best agreement with the experimental data is observed for the highest number of DP units (11940 DP units) where a total of 60 different crystal orientations are used for the parent austenite grains and 199 for the ferrite grains. However, the main inconvenience about using large number of DP units is the huge computational time. In addition, the $R$ value is the only parameter largely affected by the number of DP units whereas the other parameters, such as the macroscopic strain-stress curves or the inclusion stresses, exhibit smaller differences. A second observation is that...
the $R$ value of the DP steels has in general a decreasing behaviour during deformation which is essentially attributed to the texture evolution, i.e. the crystal rotations, of both the ferrite and the martensite/bainite constituents. Moreover, the decreasing $R$ value can exclusively be described by the ferrite grains texture. The previous statement is formulated based on the iso-stress scheme because the ferrite grains in those simulations represent the only constituent to deform plastically.

The work-hardening model exhibits a good agreement with the experimental data including the tensile stress-strain behaviour of the DP steel together with the GND density evolution of the ferrite grains. It is important to emphasize that the $R$ value is also satisfactorily predicted when a high number of DP units is included despite the fact that this parameter is not employed in the calibration procedure. In the particular case of the inclusion-matrix strain partitioning, the IsoW scheme correctly simulates the strain partitioning of the inclusions by comparing them with experimental data based on digital image correlation [8]. This agreement gives some reliability on the inclusion stress and the ensuing stress variation among the inclusions of the RVE.

The purpose of the present model is also to serve as a predictive tool for different microstructure parameters and therefore its predictability is validated with the other DP steel grades in the following section.

### 7.6.2 Validation of the model

As shown in Table 7.2, the principal differences between the microstructural features of the studied DP steels are given by the martensite volume fraction, the ferrite grain size, the bainitic ferrite lath width and the martensite carbon content. These characteristics of the microstructure are believed to be the main responsible of the difference on the mechanical properties of the DP steel grades and are incorporated in the model as microstructural input parameters. One of the purposes of the present model is to simulate the mechanical response of the DP steel grades based exclusively on its microstructural characteristics and without modifying the fitting parameters. Figure 7.10 shows the comparison between the experimental and the simulated tensile curves of the SH, SC and HT DP steel grades. It is important to notice that the simulations are made employing the same five model fitting parameters used for the Reference DP steel (cf. Table 7.4) and only differ in the microstructural input parameters given in Table 7.2. In addition, the simulation of the Reference DP steel (cf. Figure 7.4c) is also included in the graphs of Figure 7.10 in order to compare the behaviour of the other simulations.
In the case of the simulation of the SH DP steel (cf. Figure 7.10a), a decrease of the strength of this steel is observed in comparison to the simulation of the reference DP steel. This tendency is also in agreement with the experimental observations although the simulations show some overestimation of the experimental curve. A similar situation occurs for the simulation of the SC DP steel (cf. Figure 7.10b), where the influence of the microstructural parameters on the model performance is able to predict the experimental tendency with respect to the reference DP steel but shows an overestimation when compared to its corresponding experimental behaviour.

In contrast to the previous DP steel grades, the simulation of the HT DP steel displays an opposite mechanical response in comparison to its corresponding experimental tensile curve when both are compared to the reference DP steel; i.e. the experimental data exhibit higher strength as compared to the reference DP steel whereas the simulation produces lower values (cf. Figure 7.10c). This difference between the simulations and the experimental data is principally attributed to the insufficient sensitivity of the model to the microstructural input parameters; especially with regard to the characteristic lengths of the
microstructural constituents. This can be observed by the smaller deviations of the simulations from the reference DP steel stress-strain curve, for which the data were calibrated. The present curves were produced by decreasing or increasing mainly one, and not a combination, of the microstructural parameters. For example, the softening factor of the SH and the SC grades is principally a consequence of the larger ferrite grain size and of the larger bainitic ferrite lath width, respectively. However, the lack of correspondence for the HT grade may be attributed to the combination of both the softening effect, produced by a larger bainitic ferrite lath width, together with the hardening effect based on the reduction of the ferrite grain size with respect to the microstructure of the reference DP steel.

The competition between both effects is not satisfactorily predicted by the work hardening model of the simulations and this lack of reliability can be attributed to the relative simplicity of the model. For instance, Delince et al. [6] incorporated a term related to the grain boundary hardening on the work hardening equation in order to include the back stress contribution. Nevertheless, this addition has the inconvenience of increasing the number of microstructural parameters which are difficult to acquire experimentally and thus need to be calibrated. As a final remark, the variation of only one microstructural parameter of the model allows obtaining a correct trend on the relative response of the material, albeit with some differences compared to the experimental data. The following section explores the influence of the bainitic ferrite lath width on the macroscopic mechanical properties and the strain partitioning between matrix and inclusion.

7.6.3 Effect of the bainitic ferrite lath width

Figure 7.11 shows the effect of the bainitic ferrite lath width on the tensile stress-strain curve of the DP steels. The range of the tensile stress-strain curves are limited to the uniform plastic deformation stage where the maximum strain allowed is calculated using Considère’s criterion (equation 3.2). The microstructural parameters other than the bainitic ferrite lath width are kept constant on the corresponding simulation results of Figure 7.11 and taken from the Reference DP steel parameters (cf. Table 7.2). As expected, the increase of the lath width leads to a lower strength of the DP steel grades together with an increment of the uniform plastic deformation region.

The bainitic ferrite lath width mainly influences the mechanical properties of the inclusions by accommodating part of the plastic deformation, and as a consequence, relaxing the stresses of the martensite laths. This effect is corroborated in the plot of
Figure 7.12a where the average von Mises stress-strain behaviour of the inclusions is displayed for different lath width values.

By decreasing the lath width, the aggregate of martensite and bainitic ferrite increases its strength mainly due to the combination of two effects: (i) the reduction in the volume of the bainitic ferrite (softer phase), as can be observed on equation 7.27, and (ii) the hardening effect of decreasing the mean free path of the bainitic ferrite (equation 7.26). Regarding the variability of stresses for the inclusions of each simulation, Figure 7.12b displays the difference of the von Mises stress between the inclusions with the maximum and the minimum stress value. As can be observed, this stress difference is larger for smaller lath widths and reaches a maximum difference of 500 MPa for a lath width of 0.05 μm at the end of the uniform plastic deformation stage.

Figure 7.11: Simulated tensile stress-strain curves for different bainitic ferrite lath widths going from 0.05 μm up to 0.5 μm in steps of 0.05 μm.

Figure 7.12: (a) Average von Mises stress-strain curves of the inclusions from the simulations for different bainitic ferrite lath widths. (b) Plot displaying the variability of the von Mises stresses of the inclusions by showing the difference between the maximum and minimum stress value of the inclusions for different lath widths.
The influence of the inclusion stress variation on the void initiation evolution during plastic deformation is related to the premature achievement of the critical fracture stress, despite the lower average inclusion stress value. This results in the nucleation of voids at lower strain values for DP steel with smaller bainitic ferrite laths than for larger ferrite lath widths.

The inconvenience of void nucleation at early deformation stages is related to their growth and the ensuing local stress concentration. The premature voids suffer larger growth in comparison to those nucleated at higher deformation due to its exposure to larger plastic strains. Those voids also start creating stress concentrations around them and, as previously discussed in section 5.5.3, eventually shear plastic localization is created around the voids and leads to the final failure stage.

7.7 Conclusion

In this chapter, a physical-based model is assembled in order to simulate the mechanical response of DP steels taking into account a number of relevant microstructural parameters. The model incorporates the work hardening model reported by Pantleon [11] and includes this model in a crystal plasticity framework. One of the principal advantages of this approach is the use of the same work-hardening model for all of the microstructural constituents of the steel in addition to the utilization of the same value for the fitting parameters after its calibration. Moreover, the model allows obtaining the variation of the stress and strain for each of the microstructural constituents based only on the crystallographic texture of the ferrite and the parent austenite.

A satisfactory agreement is observed between the simulations using the present model and the experimental data of the Reference DP steel studied in Chapter 5. Unfortunately, the predictability of the model is still limited for different DP steel with varying microstructural parameters although it is possible to observe the appropriate trends.

Finally, the effect of the bainitic ferrite lath width on the mechanical properties of DP steel was studied. It was shown that the smaller lath widths produce higher average stresses in the particle inclusions in addition to a larger stress variation.
References


Modelling of DP steels


A better understanding of the micromechanics of fracture and their relation to damage can lead to benefits for the formability of automotive sheet metals and hence produce an improved reliability of the final component performance. For instance, by predicting the fracture limits of the materials based on their microstructure evolution during deformation, it is possible to improve material and forming process selection for complex component designs as well as to prevent the ensuing failures during vehicle life service.

The purpose of the current dissertation was to investigate the micromechanisms of failure occurring during plastic deformation of some relevant sheet metal materials commonly employed in the automotive industry: Interstitial Free (IF) steels, Dual Phase (DP) steels and AA6016 aluminium alloys. All of these materials exhibited a common feature on the microstructural scale during the fracture process: the local deformation of the metal matrix (either ferrite or aluminium grains) in order to compensate the strain heterogeneities created by the presence of harder particles (inclusions or second phase constituents) and the subsequent generation of voids.

One clear example of the relation between local matrix deformation due to strain heterogeneities and the failure micromechanisms was the evolution of the particle deformation zone (PDZ) in IF steel. In Chapter 4 it was demonstrated that microstructural voids are nucleated on the TiN particles by either particle fragmentation or particle-matrix debonding. Whereas the former mechanism occurs at relatively low strain values and displayed a preference for larger particles, the decohesion of the TiN particles from the matrix takes place after some degree of local deformation has developed at the interface. This local deformation constitutes the PDZ and is characterized by the discrete and/or continuous crystal rotation of the lattice. One of the
relevant findings achieved in Chapter 4 is that the void nucleation mechanism on the well-bonded and rigid TiN particles depends on the stress relaxation of the strain incompatibilities between particle and matrix, allowing switching from fragmentation to interfacial debonding of the particles. This is attributed to the limitation of the internal stresses within the particle due to the relaxation effect of the PDZ, which helps to accommodate the deformation at the strain incompatibilities and therefore to reduce the probability of particle fragmentation. However, as local deformation increases within the PDZ, the interfacial stress reaches a critical condition at which debonding takes place.

The DP steels also develop similar void nucleation mechanisms as in the case of IF steels (Chapter 5), although the voids are related to the second phase constituents; i.e. martensite and bainite. Although the interface decohesion between the ferrite and the other constituents was comparable to the IF steel, the apparent fragmentation of the bainite/martensite have the particularity that this mechanism consists in the separation of bainite/martensite packets. Nevertheless, both mechanisms are also accompanied by local deformation of the surrounding ferrite matrix in order to reduce strain incompatibilities.

In addition to the mechanisms of interfacial decohesion and particle fragmentation, the AA6016 aluminium alloys studied in Chapter 6 develop a third void initiation mechanism, which is not found in the IF nor DP steels, at deformations close to fracture. The intergranular separation of the aluminium matrix was characterized by strain localization along the grain boundaries and was observed to be more common in the aluminium alloy with higher magnesium content. The predominance of this type of void initiation mechanism was attributed to the contribution of two factors: (i) the formation of precipitation free zones along grain boundaries; and (ii) to the relatively high level of local stresses generated in these regions as a result of the matrix strengthening and the ensuing reduction of the stress relaxation in strain incompatibility regions.

As a general conclusion from the experimental observations on the three material types, it can be stated that voids are initiated at sites of microstructural strain incompatibilities once the local stresses reach the critical conditions. The magnitude of these local stresses depends on the relaxation degree that could be achieved by the accommodation of plastic deformation of the surrounding matrix.

Despite the relative high number of void nucleation sites available in DP steels (Chapter 5), the maximum void area fraction close to the fracture edge is lower than 1%.
This apparent contradiction is the result of two combined effects: (i) the initial dimensions of the voids inherited from the nucleation process and, (ii) the local stress-strain conditions developed during tensile deformation. These phenomena were also observed to occur in IF steel (Chapter 4) and in the AA6016 aluminium alloy (Chapter 6). The growth of voids then occurs in a stable mode.

One of the most significant findings of this dissertation is the relation between shear bands and meso-scale cracks in DP steels (Chapter 5). At strain values close to fracture, the presence of shear bands was commonly observed in all DP steels and identified by EBSD in terms of their inclination, their path independence from the grain morphology and their detrimental effect on the image quality of the EBSD patterns. In addition, two principal characteristics of the shear bands are highlighted from the experimental observations: (i) shear bands seem to emanate from voids originally initiated from the martensite/bainite islands during previous deformation processes; and (ii) the existence of cracks within and oriented along the shear bands. Based on this evidence, it was suggested that shear bands have their origin from the grain substructures generated around martensite/bainite related voids due to the generation of stress concentration in these regions. Eventually, cracks are later developed within and along shear bands; at this moment the propagation of the cracks commences in an unstable manner and its path is almost unaffected by microstructural features because of the high strain localization nature of shear bands. The previous described mechanisms represent the void coalescence stage of the fracture micromechanisms and mark the transition of the crack propagation from the meso- to the macro-scale.

*Therefore, the detrimental effect of voids is not their null carrying capacity but rather the stress concentration created around them that eventually gives rise to strain localization in the form of shear bands and the ensuing nucleation of meso-scale cracks.*

In order to correctly simulate and predict the damage evolution of the microstructure of automotive metal sheets, two principal phenomena need to be properly addressed by models: (i) the continuous nucleation of voids during the deformation history of the material and, (ii) the development of grain substructures with the intention of predicting the formation of shear bands. The proposed model for DP steels described in Chapter 7 represents an initial attempt to achieve these goals. In the first place, the continuous nucleation of voids during deformation is a consequence of the heterogeneous local stress conditions within the microstructure due to the combination of the different local boundary conditions and the constituent properties, such as crystal
Chapter 8

orientation. The model allowed estimating the variation of the stress for each of the microstructural constituents based only on the crystallographic texture of the ferrite and the parent austenite. This approach allows capturing the scattered and dispersed nature of the microstructure, instead of only utilizing average quantities of the microstructure. With regard to the evolution of the grain substructure, the employed work-hardening model incorporated the influence of different types of dislocation densities, whereby the evolution of the Geometrically Necessary Density of Dislocations (GND) is described by a statistical-based equation and calibrated with experimental data.

The intention of the proposed model is to serve as a basis for the development of new and improved modelling tools for materials design, using as fundamental approach the microstructure-based damage control of metals.
Appendix A

Crystal orientations

A.1 Crystal orientations descriptors

The crystallographic orientation $g$ can be represented by different mathematical expressions and its choice only depends on the purpose and use which are going to be performed with these descriptors. For example, some descriptors may be more suitable to perform mathematical operations, but its physical interpretation is difficult to visualize at first sight. One common characteristic and requirement of these descriptors is that all of them need at least three independent parameters to fully describe orientations. This requirement hinges on the fact that the degree of freedom to define a crystallographic orientation is three (Euler’s rotation theorem).

Due to geometric symmetries of the crystal lattice and/or the applied mechanical processes, it is possible to obtain two or more crystallographic orientations which are numerically different but physically (or statistically, in the case of sample symmetries) represent the same crystal orientation. These equivalent orientations are related to each other by the different elements of the symmetric orientation group.

Another important concept related to crystallographic orientations is misorientation. The misorientation, $\Delta g$, between two crystallographic orientations is defined in a passive rotation concept as the rotation to transform tensor quantities (vector, stress or strain) from one set of crystals axes $g_A$ to other set $g_B$. In other words, the misorientation represents the orientation difference between two crystal orientations $A$ and $B$. From this definition, it can be highlighted that the usual frame of misorientations is in crystal coordinates and therefore the sample coordinate system has no relevance as it has for crystallographic orientations. Similar to physical and statistical symmetries on crystal orientations, the misorientations also manifest two different types of symmetries which are necessary to be applied in order to obtain all the equivalent solutions. These symmetries are the crystal and the switching symmetries.

As all the equivalent solutions physically represents the same misorientation, it is a common practice to chose, from among all possible solutions, the one with the smallest
Appendix A

misorientation angle to represent the orientation difference. In fact, it is common to find in literature [1] that while the whole set of equivalent orientation differences are referred to as misorientations, those with the smallest misorientation angles are known as disorientations.

The principal orientation and misorientation descriptors used in materials science are given in Table A.1 together with a brief summary of their properties.

<table>
<thead>
<tr>
<th>Representation</th>
<th>Properties, advantages and drawbacks</th>
</tr>
</thead>
</table>
| **Orientation Matrix** | The orientation matrix are proper orthogonal tensors of degree 3; i.e. \( g^T = g^{-1} \) and determinant(\( g \)) = +1
Both the rows and columns are unit vectors

\[
g = \begin{pmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{pmatrix}
\]
| The cross product between 2 rows or 2 columns give the third row or column
| Mathematical tool for calculation of other descriptors of orientation
| Contains 6 non-independent terms
| Difficult visualization of the physical meaning |
| **Miller indices or ideal orientations** | For specific indices and cubic materials, dot product between the Miller direction and plane is equal or close to zero.
For non-specific indices, the negative sign is not written
\((hkl)[uvw]\)
(specific indices)
| Easy physical visualization. In rolled products, the Miller direction is parallel to rolling direction and the Miller plane is perpendicular to the normal direction
| The 24 cubic crystal symmetries are obtained by permuting the indices and/or changing the signs
\|\{hkl\}<uvw>\)
(non-specific indices)
| The indexes are rounded to the nearest low index
| No suitable to perform orientation operations |
| **Euler angles** | Singularity at \( \phi = 0 \): \( \varphi_1 \) and \( \varphi_2 \) become linearly dependent parameters
\( \varphi_1, \phi, \varphi_2 \)
| Minimum number of independent parameters
| Difficult to apply mathematical operations
| Convenient for calculation of ODF by spherical harmonics |
| **Angle-Axis** | On misorientation, \( \omega \) is the misorientation angle
\( \omega \ (\hat{n}_1, \hat{n}_2, \hat{n}_3) \)
| Useful for misorientation concept
| No convenient for mathematical operations |
| **Rodrigues vector** | On misorientation, the magnitude of the Rodrigues vector is equal to the tangent of the angle divided by two

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Crystal orientations

| \( \mathbf{\bar{p}} = (\rho_1, \rho_2, \rho_3) \) | Minimum number of independent parameters
| | Useful for misorientation concept
| **Quaternions** | Its magnitude is equal to +1.
| \( q = \{q_0; q_1, q_2, q_3\} \) | +q represents the same orientation as \(-q\)
| = \{q_0; \mathbf{q}\} | Easy to normalize
| | Simple arithmetic operation to calculate misorientation angle

A.2 Quaternions

The quaternion representation [2] for crystal orientations allows performing more efficient mathematical operations between crystal orientations as well as keeping an easy visualization. In fact, this descriptor is a subgroup of quaternions and the more appropriate name should be *positive unit quaternions*. The space of this subgroup is localized on the “surface” of a hemi-hypersphere of four dimensions with ratio equal to one [3, 4]. The positive half of the hypersphere is usually employed due to the physical symmetry on the hyperplane with respect to the first constant component.

The expressions of the four elements of the quaternion \( q \) as a function of the Euler angles, the angle-axis and the orientation matrix representation are:

\[
\begin{align*}
q_0 &= \cos \frac{\phi}{2} \cos \frac{\varphi_1 + \varphi_2}{2} = \cos \frac{\omega}{2} = \frac{\sqrt{a_{11} + a_{22} + a_{33} + 1}}{2} \\
q_1 &= \sin \frac{\phi}{2} \cos \frac{\varphi_1 - \varphi_2}{2} = \sin \frac{\omega}{2} \mathbf{n}_1 = \frac{a_{23} - a_{32}}{2\sqrt{a_{11} + a_{22} + a_{33} + 1}} \\
q_2 &= \sin \frac{\phi}{2} \sin \frac{\varphi_1 - \varphi_2}{2} = \sin \frac{\omega}{2} \mathbf{n}_2 = \frac{a_{31} - a_{13}}{2\sqrt{a_{11} + a_{22} + a_{33} + 1}} \\
q_3 &= \cos \frac{\phi}{2} \sin \frac{\varphi_1 + \varphi_2}{2} = \sin \frac{\omega}{2} \mathbf{n}_3 = \frac{a_{12} - a_{21}}{2\sqrt{a_{11} + a_{22} + a_{33} + 1}}
\end{align*}
\]

(A.1)

Based on equation (A.1), the component \( q_0 \) contains information only about the misorientation angle while the other three components, usually known as *quaternion vector*, are proportional to the misorientation axis weighted by the misorientation angle. This is the reason the quaternion vector is proportional to the Rodrigues-Frank vector \( \mathbf{\bar{p}} \) and in fact, can easily be calculated by dividing the quaternion vector \( \mathbf{q} \) by the scalar component \( q_0 \).
Appendix A

A.3 Crystal orientation averaging

In some tasks concerning microtexture data analysis, it is useful to obtain the average or representative value for a collection of crystallographic orientations, e.g. crystallographic orientations data belonging to grains or subgrains [1] or as part of cleaning procedures for slightly deformed material containing cells or subgrains with misorientations ~0.5-3° [3]. However the acquisition of such crystallographic orientation average is not an easy issue. One of the reasons is because the average cannot be calculated by a simple arithmetic average of the three Euler angles nor of any other parameters from the other orientation descriptors. The mathematical reason of this incapability is related to the distortion of all the descriptor spaces [3].

The average orientation of crystal orientation data set is defined as the orientation which is “closer” to all and each component of the set. The significance of this “closeness” is related to the concept of misorientation and thus, the average orientation is the one which minimize an objective function containing the misorientations of the average orientation with respect to each component of the data set [5]. Mathematically, this concept is represented by the following equation

\[
\min \left\{ \sum_{i=1}^{n} f(\omega(g_{av}, g_i)) \right\}
\]  

(A.2)

where \(\omega\) represents the misorientation angle between the proposed average orientation, \(g_{av}\), and the \(i\)-th orientation, \(g_i\), of the data set constituted by a total of \(n\) crystallographic orientations. The objective function to minimize is represented by \(f\).

In addition to the distortion of the descriptors space, there is another issue which complicates crystallographic averaging: the crystal symmetry. As mentioned in the beginning of Appendix A, two crystallographic orientations may have totally different descriptor parameters but represent the same orientation. One example is the orientations expressed by the Euler angles \((\phi_1=35^\circ, \phi=45^\circ, \phi_2=0^\circ)\) and \((\phi_1=55^\circ, \phi=90^\circ, \phi_2=45^\circ)\) which represents the same orientation in cubic crystals and is known as the copper component. A proposed solution to this problem is to simply apply one set of crystal symmetry operators for each of the orientations on the data set and chose the one with the minimum misorientation angle. The second set of symmetry operators is not required since it is only necessary the misorientation angle. The acquisition of the grain average is an iterative process where a crystallographic orientation average is proposed and tested until equation (A.2) is satisfied with the desired accuracy [6]. However this method results in being computer-time consuming, especially with a high number of crystallographic orientations on the data set.
The average orientation is employed for several calculations on this thesis; for instance, the GROD map and the SOS parameter. The algorithm employed to determine the average orientation of a dataset (grain) is displayed in Figure A.1, which is based on methodologies using quaternion averaging [3, 7, 8]. The developed method consists firstly in choosing the orientation with the highest Confident Index (CI) among the orientations of the selected grain (cluster data) as an initial guess for the average orientation \( q_{av} \). Afterwards, the crystal symmetry equivalent of each of the cluster orientations, \( q_i^e \), which produces the minimum misorientation angle \( \omega \) with respect to the proposed average orientation is selected and stored for the subsequent calculations. The average misorientation angle, \( \omega_{av1} \), between each of the equivalent orientations of the cluster and the average orientation is computed and compared with the previous misorientation angle, \( \omega_{av0} \). On the first trial, the misorientation angle \( \omega_{av0} \) is set to the highest possible angle; for instance, 180°. If the difference between both misorientation angles is lower than a defined tolerance (set in 0.01°), then the proposed average orientation is selected and the algorithm finishes. In case the tolerance was not satisfied, a new proposed average orientation is calculated according to the following equation [3]:

\[
\omega_{av1} = 2 \cos^{-1} \left( \frac{1}{n} \sum_{i=1}^{n} q_i^{e} \cdot q_i \right)
\]

Figure A.1: Schematic representation of the algorithm employed to calculate the average orientation from an orientation cluster.
A.4 Parameters employed on the characterization of plastic deformation by EBSD

Two main parameters are employed for the characterization of the plastic deformation: the KAM and the SOS parameters. Both parameters are associated to the KAM and GROD maps.

On the one hand, the *Kernel Average Misorientation*, KAM, is a parameter employed to characterize the short range orientation gradients based on local misorientation data within grains. The KAM is computed from a cluster of KAM units within the grain. The magnitude of this KAM unit is calculated by averaging the misorientation angles of the point of interest (kernel) with respect to all its neighbouring pixels (Figure A.2). In contrast to the definition established by TSL software [9], the calculation of the KAM unit excludes neighbouring pixels which do not belong to the same grain as the kernel pixel, instead of using a misorientation angle threshold as an exclusion criterion. The motivation to this modification is that grain substructures (*dense dislocation walls*, for example) may reach high misorientation angles, but a path within the grain still exists that connects these regions through low misorientation angles. Therefore this method depends on the effectiveness of the grain reconstruction, although includes on the calculation the influence of the grain substructures on the short range orientation gradients.

For the KAM map, each pixel is coloured according to the magnitude of the KAM unit. In the case of the KAM parameter for a single grain, the following equation is used for its calculation:

\[
q_{av} = \frac{1}{n} \sum_{i=1}^{n} q_i
\]

\[
q_{av} = \frac{1}{\sum_{i=1}^{n} q_i}
\]

(A.3)

A.4 Parameters employed on the characterization of plastic deformation by EBSD

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On the one hand, the *Kernel Average Misorientation*, KAM, is a parameter employed to characterize the short range orientation gradients based on local misorientation data within grains. The KAM is computed from a cluster of KAM units within the grain. The magnitude of this KAM unit is calculated by averaging the misorientation angles of the point of interest (kernel) with respect to all its neighbouring pixels (Figure A.2). In contrast to the definition established by TSL software [9], the calculation of the KAM unit excludes neighbouring pixels which do not belong to the same grain as the kernel pixel, instead of using a misorientation angle threshold as an exclusion criterion. The motivation to this modification is that grain substructures (*dense dislocation walls*, for example) may reach high misorientation angles, but a path within the grain still exists that connects these regions through low misorientation angles. Therefore this method depends on the effectiveness of the grain reconstruction, although includes on the calculation the influence of the grain substructures on the short range orientation gradients.

For the KAM map, each pixel is coloured according to the magnitude of the KAM unit. In the case of the KAM parameter for a single grain, the following equation is used for its calculation:

\[
KAM = \frac{1}{n} \sum_{i=1}^{n} KAM^U_i
\]

(A.4)
Where $n$ is the total number of pixels within the grain of interest and $\text{KAM}_i$ is the KAM unit of the $i$th pixel.

In order to reduce the effect of long range orientation gradients and of regions without crystal rotations, a lower misorientation angle threshold was incorporated where the calculation of the KAM parameter of the grain only includes KAM units with values higher than this threshold. A similar procedure was adopted by Delanay et al. [10] to characterize grain substructures in cold rolled aluminium.

On the other hand, the *Scalar Orientation Spread*, SOS, is used to characterize the long range orientation gradients within grains by accounting for the orientation spread around a reference crystal orientation [11]. On the present thesis, the average orientation of all the pixels constituting the grain is employed as the reference orientation for the SOS. The equation used to calculate this parameter for a particular grain is given by

$$\text{SOS} = \frac{1}{n} \sum_{i=1}^{n} \omega_i \left( g_{\text{av}}, g_i \right)$$

(A.5)

Where $\omega_i$ represents the misorientation angle between the average orientation of the grain, $g_{\text{av}}$, and the orientation of the $i$th pixel, $g_i$, and $n$ is the total number of pixels within the grain.

In similarity to the KAM parameter, the SOS also uses an angle threshold to exclude misorientation angles of the pixels which are below of this limit value. The justification is to better reflect the effect of the orientations which are more misoriented from the average orientation due to the long range orientation gradient effect.

The map associated to the SOS is the *Grain Reference Orientation Deviation* map which each pixel is coloured depending on its misorientation angle with respect of the average orientation of the grain. An example of a GROD map is shown in Figure A.3.

![Figure A.3: Example of a colour-coded GROD map of an IF steel ferrite grain deformed up to 35%. The associated SOS is equal to 6.2°.](image_url)
Appendix A

References

Appendix B

Geometrically Necessary Density of Dislocations by EBSD

B.1 Theory: Nye’s Relation

Assuming compatibility for a deformed body on a continuum dislocation theory framework [1], then the total distortion tensor, $\mathbf{\beta}^T$, derived from the displacement vector field must satisfy the following continuity condition for any closed path or circuit within the body:

$$\nabla \times \mathbf{\beta}^T = 0 \quad (B.1)$$

The total distortion tensor can be linearly decomposed into the elastic, $\mathbf{\beta}^e$, and plastic, $\mathbf{\beta}^p$, distortions [1], which turns the equation (B.1) into

$$\nabla \times \mathbf{\beta}^e = -\nabla \times \mathbf{\beta}^p \quad (B.2)$$

Whereas the total distortion tensor is characterized by being a compatible field, the elastic and plastic tensors are incompatible and therefore the curl of both tensors is usually different from zero.

The plastic distortion tensor, which represents the “eigenstrain” caused by the presence of dislocations within the volume [2], is associated to the net burgers vector $\mathbf{B}$ of multiple dislocations crossing through the surface $S$ by the following equation:

$$\mathbf{B}^p = \int_C (\nabla \times \mathbf{\beta}^p) \cdot \hat{n} dS \quad (B.3)$$

where $C$ refers to the circuit delimiting the surface $S$ and the unit vector $\hat{n}$ is normal to that surface. The correlation between the right-side terms of equation (B.3) is based on the Stokes theorem for second-rank tensors.

Regarding to the aforementioned burgers vector, Nye [3] introduced the dislocation density tensor $\mathbf{\alpha}$ which is a second-rank tensor specifying the state of dislocations of the region. Then, the net burger vector of the dislocations piercing through $S$ can be described by the dislocation density tensor using the area integral:
Appendix B

\[ \mathbf{\bar{B}} = \int_S (\mathbf{a} \cdot \mathbf{n}) dS \]  \hspace{1cm} (B.4)

The combination of equations (B.3) and (B.4) led to the conclusion that, for any circuit, the dislocation density tensor is equivalent to the curl of the plastic distortion tensor

\[ \mathbf{a} = \nabla \times \mathbf{\beta}^p \]  \hspace{1cm} (B.5)

as well as to the elastic distortion tensor if the equality given in equation (B.2) is considered

\[ \mathbf{a} = -\nabla \times \mathbf{\beta}^e \]  \hspace{1cm} (B.6)

As Kroner [1] pointed it out, equation (B.5) is the basic definition of the dislocation density tensor in the linearized continuum theory of elastoplasticity. However, equation (B.6) is not a definition but it contains the postulate that the body remains perfectly connected (no cracks) during the elastoplastic deformation.

The elastic distortion tensor can be described as the sum of a symmetric, \( \mathbf{\varepsilon}^e \), and an anti-symmetric, \( \mathbf{\omega} \), tensor

\[ \mathbf{\beta}^e = \mathbf{\varepsilon}^e + \mathbf{\omega} \]  \hspace{1cm} (B.7)

The symmetric component \( \mathbf{\varepsilon}^e \) represents the elastic strain or stretching of the lattice, whereas the tensor \( \mathbf{\omega} \) describes the rotation of the lattice orientation on a volume element.

Therefore, the substitution of equation (B.7) into equation (B.6) results in

\[ \mathbf{a} = -\nabla \times (\mathbf{\varepsilon}^e + \mathbf{\omega}) \]  \hspace{1cm} (B.8)

The right side of the previous equation contains elastic strain gradient as well as rotation gradient terms. The gradient terms of the elastic stretches are commonly neglected in most of the analysis of crystal curvature using EBSD [4-8]. One of the justifications is that if elastic strains (gradients) were large, then the stored energy would be large and would be reduced by any recovery process [8]. The same line of thinking was followed in this thesis and all elastic strain gradient terms are considered close to zero; in other words,

\[ \mathbf{a} = -\nabla \times \mathbf{\omega} \]  \hspace{1cm} (B.9)

Due to a different sign convention, Kroner’s definition of the dislocation density tensor differs to Nye’s definition [5] by the following relation:

\[ \mathbf{a}_{\text{Nye}} = -\mathbf{a}_{\text{Kroner}}^T \]  \hspace{1cm} (B.10)

In order to deduce the same equation as Nye [3], the previous convention defined by Kroner [1] is modified to Nye’s convention, resulting in a modification of equation (B.9)
In the case of the rotation tensor, their gradient terms are associated to the curvature of the crystal lattice structure. The general form of the rotation or spin tensor expressed in its matrix form is

\[
\omega = \begin{pmatrix}
0 & -\theta_3 & \theta_2 \\
\theta_3 & 0 & -\theta_1 \\
-\theta_2 & \theta_1 & 0
\end{pmatrix}
\] (B.12)

The terms \(\theta_1, \theta_2\) and \(\theta_3\) are the components of the vector \(\vec{\theta}\) which direction is parallel with the axis of rotation and its magnitude is equal to the rotation angle expressed in radians. As a consequence, the matrix form of the curl of the rotation tensor would be:

\[
\nabla \times \omega = \begin{pmatrix}
-\frac{\partial \theta_2}{\partial x_2} & -\frac{\partial \theta_1}{\partial x_2} & \frac{\partial \theta_1}{\partial x_3} \\
\frac{\partial \theta_2}{\partial x_1} & -\frac{\partial \theta_1}{\partial x_1} & -\frac{\partial \theta_2}{\partial x_3} \\
\frac{\partial \theta_3}{\partial x_1} & \frac{\partial \theta_3}{\partial x_2} & -\frac{\partial \theta_1}{\partial x_1} - \frac{\partial \theta_2}{\partial x_2}
\end{pmatrix}
\] (B.13)

or,

\[
\nabla \times \omega = \nabla \vec{\theta} - \left(\nabla \cdot \vec{\theta}\right) I
\] (B.14)

Nye [3] introduced the second-rank tensor \(\kappa\) in order to describe the curvature of the lattice; i.e. the lattice rotation gradient. The Nye’s curvature tensor is defined as

\[
\kappa = \nabla \vec{\theta}
\] (B.15)

or, in matrix form,

\[
\kappa = \begin{pmatrix}
\frac{\partial \theta_1}{\partial x_1} & \frac{\partial \theta_1}{\partial x_2} & \frac{\partial \theta_1}{\partial x_3} \\
\frac{\partial \theta_2}{\partial x_1} & \frac{\partial \theta_2}{\partial x_2} & \frac{\partial \theta_2}{\partial x_3} \\
\frac{\partial \theta_3}{\partial x_1} & \frac{\partial \theta_3}{\partial x_2} & \frac{\partial \theta_3}{\partial x_3}
\end{pmatrix}
\] (B.16)

It is important to highlight that each of the columns of the curvature tensor corresponds to the orientation gradient of the lattice structure along different direction. The units of the curvature tensor terms are radians per length unit.
Finally, the incorporation of the curvature tensor into equations (B.14) and (B.11) gives the well known equations formulated by Nye [3]

\[
\alpha = \kappa^T - I \text{ trace}(\kappa) \quad \text{(B.17)}
\]

\[
\kappa = \alpha^T - \frac{1}{2} I \text{ trace}(\alpha) \quad \text{(B.18)}
\]

The physical meaning of Nye’s equation is that correlates non-redundant density of dislocations within the volume to the curvature (orientation gradient) of the crystal lattice [9]. This dislocation density, referred to as \textit{Geometrically Necessary Density of Dislocations}, is the result of trapped dislocations during the plastic deformation process. Besides the dislocations with a non-redundant effect, dislocations exist which produce a net redundant effect within the reference volume. This dislocation density is commonly known as \textit{Statistically-Stored Dislocations}, SSD, and has no effect on the lattice curvature of a predefined volume.

**B.2 GND extraction based on EBSD data**

The algorithm to extract the GND from EBSD data was implemented in a C language code. The program uses the EBSD output file containing the spatial coordinates of each pixel, its crystal orientation and the grain number.

\textit{Curvature tensor}

Each of the columns of the curvature tensor \( \kappa \), meaning the orientation gradient along each direction, is acquired from a cluster of neighbouring pixels and the kernel (cf. Figure B.1).

![Figure B.1: Representation of the pixels cluster (gray colour boxes) employed to calculate the orientation gradient at the kernel pixel along different directions.](image)
As EBSD was only performed on 2D sections, it was only possible to determine two of the columns of the curvature tensor. A progressive application of masks is applied to each of the pixels cluster (cf. Figure B.2), where the magnitude of the misorientation angle was the factor controlling the robustness of the masks. This methodology is employed in order to improve the accuracy of the curvature tensor due to the angular resolution of the EBSD data. Further details of this procedure are given elsewhere [10].

![Figure B.2: Representation of the different types of masks employed to determine the orientation gradient along the horizontal direction in order to improve the precision of the calculations.](image)

After the application of the masks, a quaternion describing the orientation gradient for each direction is obtained. Based on the conversion formulae for quaternions given in equation (A.1), the misorientation angle, $\theta$, and the associated unit vector, $\hat{n}$, were calculated and introduced on the following equation:

$$
\kappa \approx \begin{bmatrix}
\frac{(\theta n_1)^{q_1}}{\Delta x_1} & \frac{(\theta n_1)^{q_2}}{\Delta x_2} & \frac{(\theta n_1)^{q_3}}{\Delta x_3} \\
\frac{(\theta n_2)^{q_1}}{\Delta x_1} & \frac{(\theta n_2)^{q_2}}{\Delta x_2} & \frac{(\theta n_2)^{q_3}}{\Delta x_3} \\
\frac{(\theta n_3)^{q_1}}{\Delta x_1} & \frac{(\theta n_3)^{q_2}}{\Delta x_2} & \frac{(\theta n_3)^{q_3}}{\Delta x_3}
\end{bmatrix}
$$

(B.19)

The superscripts on the terms of equation (B.19) indicate the quaternion to which correspond the misorientation angle and the unit vector component. The increments $\Delta x$
Appendix B

are the step size of the EBSD scan along the specified direction. Despite all nine components of the curvature tensor are fulfilled in equation (B.19), only six components (two columns) can be calculated.

Dislocation density tensor

The dislocation density tensor $\alpha$ is constituted by the sum of the individual effects of dislocation densities $\rho^{(s)}$ from different dislocation types ($s$), each characterized by its Burgers vector $\mathbf{b}^{(s)}$ and the unit vector $\mathbf{i}^{(s)}$ which is parallel to the dislocation length. Hence, the tensor $\alpha$ is given by:

$$\alpha = \sum_{s=1}^{N} \left( \mathbf{b}^{(s)} \otimes \mathbf{i}^{(s)} \right) \rho^{(s)}$$

where $N$ is the total number of dislocation types.

Nye’s equation

Pantleon [5] demonstrated that by employing equation (B.18), it is possible to make use of the maximum number of data collected from EBSD data. The substitution of equation (B.20) into (B.18) gives place to

$$\kappa = \sum_{s=1}^{N} \left( \mathbf{b}^{(s)} \otimes \mathbf{i}^{(s)} \right)^T - \frac{1}{2} \text{trace} \left( \mathbf{b}^{(s)} \otimes \mathbf{i}^{(s)} \right) \rho^{(s)}$$

Taking into account that only six components of the curvature tensor are available, the previous equation can be decomposed into six equations with a total of $N$ unknowns; i.e. the dislocation densities $\rho$ of the $N$ dislocation types. In the particular case of steel (BCC structure) and for the sake of plasticity, it has been only considered the influence of the edge dislocations along the slip family systems \{110\}, \{111\} and \{112\}, \{111\}. Therefore, the system gives a total of 24 unknowns (slip systems) and only six equations. As exists more unknowns than equations, the system is undetermined and an infinite set of solutions satisfy equation (B.21). Therefore, assuming that the system must minimize the line energy of all dislocation densities and that each dislocation type has the same line energy, the objective function to be minimized results in

$$\min \left( \rho_{\text{GND}} = \sum_{s=1}^{N} \rho^{(s)} \right)$$

As all equations together with the objective function are linear, the simplex method [11] was used to obtain the dislocation components which produce a minimum amount of the GND density.
References

5. W. Pantleon: Resolving the geometrically necessary dislocation content by conventional electron backscattering diffraction, Scripta Materialia, 58, (2008), pp. 994-997
Summary

The principal objective of this thesis is focussed on the identification and characterization of the damage micromechanisms occurring during plastic deformation of three different materials IF steels, DP steels and AA6016 aluminium alloys that have found very wide application in the automotive industry.

Although the three materials studied in this thesis are totally different in chemical composition, microstructure and mechanical properties, they present the same type of problem from a micromechanical point of view: a ductile matrix containing inclusions where both matrix and inclusion possess different mechanical properties. This similarity allows for a generic approach aimed to obtain a better understanding of the ductile failure micromechanisms in modern alloys by comparing and studying three different cases: (i) small rigid inclusions with cubical shape strongly embedded in a ductile matrix (IF steels); (ii) a dense population of hard “inclusions” in a ductile matrix (DP steel); and (iii) brittle inclusions weakly embedded in a ductile matrix of which the strength is controlled by nano-sized precipitates (AA6016 aluminium alloy).

The mechanisms acting during ductile fracture involve different scales. A short survey related to the fracture micromechanisms occurring during plastic deformation of metals is given in this chapter. Firstly, a description of the overall mechanisms of failure occurring at different scales is provided together with the state–of-the-art on ductile fracture modelling. A further review is given on the critical aspects contributing to the stages of failure from a microstructural point of view. Because the plastic deformation is a fundamental part on the study of ductile failure a review of the plastic deformation processes occurring at the microstructural level is presented together with a state-of-the-art report on the use of Electron Backscatter Diffraction (EBSD) for characterization of local deformation.

Chapter 4 is dedicated to the study of the micromechanisms inducing void formation during tensile deformation of an IF steel and the associated evolution of grain substructures. First a description of the initial microstructure is given, which includes the particles characterization followed by the mechanical properties acquired from tensile tests. Afterwards the failure micromechanisms were studied by observation of the
fracture surface and examination of the cross-section of tensile samples. The grain substructures developed at different tensile deformation stages within the ferrite grains were analyzed and a special emphasis was given on the region surrounding the particles. This analysis was complemented with data from the novel FIB-EBSD technique. Although the IF steel has a relatively high purity, the primary void initiation preferentially occurs on the TiN particles embedded in the microstructure.

The failure micromechanisms of DP600 steels with different heat treatments are studied in this chapter using microscopic techniques. In the first part of this chapter the initial microstructure of the as-cold rolled DP steel is studied together with the microstructure after the heat treatment. The mechanical properties of the different DP steels are investigated by tensile tests and expanded by analysis of the geometry of the broken samples. The preferential sites for void initiation in current Dual Phase (DP) steels are mainly associated to the martensite. Finally, it is observed that shear bands are responsible for the final stage of fracture by acting as nucleation sites for cracks.

The failure micromechanisms of two AA6016 aluminium alloys with different magnesium content after uniaxial tensile tests are studied in this chapter. SEM fractographs of both materials suggest two types of failure mechanisms. The first is characterized by a sequence of events, i.e. void initiation, growth and coalescence on intermetallic constituents, whereas the second one can be identified as ductile intergranular failure. The second type of failure is observed more often in the aluminium alloy with higher magnesium content. In both aluminium alloys, secondary cracks were found close to the fracture surface on the cross-section plane of the tensile sample. Crystal orientation maps show that the secondary cracks were formed either by an intergranular failure mechanism or by local plastic instabilities such as shear bands which subsequently progress into cracks. The higher magnesium content gives higher strength but reduces the ductility by weakening the grain boundaries.

Chapter 7 is focused on modelling the evolution of local stresses of the DP steel constituents during tensile deformation. In order to simulate the mechanical behaviour of DP steels, a micromechanical model is developed by incorporating a physical-based work hardening model into a crystal plasticity framework for each of the microstructural constituents. Among the material parameters employed by the model, the crystallography of martensite and the Geometrically Necessary Density of Dislocations, GND, of the ferritic matrix are taken into account for the model development. Finally, the model is calibrated and afterwards validated on experimental data of DP steels with different heat treatments.
In conclusion, the thesis presents an analysis of the damage micromechanisms occurring within the microstructure during plastic deformation.
Samenvatting

Het voornaamste doel van dit proefschrift is gericht op de identificatie en karakterisering van de schade-mechanismen die optreden tijdens plastische vervorming van drie verschillende materialen die zeer breed worden toegepast in de auto-industrie: IF staal, DP staal en AA6016 aluminiumlegeringen. Er wordt hierbij speciaal de nadruk gelegd op structuurbeschadiging op het niveau van de microstructuur.

Hoewel de drie materialen bestudeerd in dit proefschrift totaal verschillend zijn met betrekking tot chemische samenstelling, microstructuur en mechanische eigenschappen, stellen zij hetzelfde type probleem vanuit micromechanisch oogpunt: harde inclusies zijn ingebed in een ductiele matrixfase, waarbij matrix en inclusies sterk verschillende mechanische eigenschappen vertonen. Deze overeenkomst maakt een generieke aanpak mogelijk, gericht op een beter begrip van de breuk-micromechanismen in moderne legeringen. In dit werk worden drie verschillende gevallen bestudeerd en met elkaar vergeleken (i) kleine stijve insluitsels met kubusvormige vorm die sterk ingebed zijn in een vervormbare matrix (IF staal), (ii) een dichte concentratie met harde insluitsels in een ductiele matrix (DP staal), en (iii) brosse insluitsels die zwak ingebed zijn in een ductiele matrix waarvan de sterkte bepaald wordt door nano-precipitataten (AA6016 aluminium).

De mechanismen gerelateerd aan taai breuk kunnen op verschillende lengteschalen beschouwd worden. In hoofdstuk XX wordt een literatuur-overzicht voorgesteld met betrekking tot de breuk-micro-mechanismen die optreden tijdens plastische vervorming van metalen. Vooreerst wordt een algemene beschrijving gegeven van de schade-mechanismen zoals die op verschillende lengthe-schalen worden geobserveerd, waarbij er ook ruim aandacht besteed wordt aan de state-of-the-art met betrekking tot modellering van taai breuk. Nadien worden ook de kritische aspecten besproken die bijdragen aan de opeenvolgende stadia van het schadeproces en dit vanuit microstructureel oogpunt. Omdat plastische vervorming een fundamentele rol speelt bij taai breuk zal er ook uitgebreid ingegaan worden op de plastische vervormings-processen die zich afspelen op microstructureel niveau. Hierbij wordt ook overzicht
beschreven omtrent de actuele stand van zaken met betrekking tot het gebruik van Electron Backscatter Diffractie (EBSD) voor de karakterisering van lokale vervorming.

Hoofdstuk 4 is gewijd aan de studie van de micro-mechanismen die een rol spelen bij het ontstaan van microholtes tijdens rekvervorming van een IF-staal en de bijbehorende ontwikkeling van de vervormings-substructuur. Vooreerst wordt een beschrijving gegeven van de initiële microstructuur, met een gedetailleerde karakterisering van de distributie en morfologie van 2e fazedeeltjes, gevolgd door een studie van de mechanische eigenschappen aan de hand van trekproef-resultaten. Vervolgens worden de breuk-micro-mechanismen bestudeerd door een nauwkeurige observatie van het breukoppervlak en een metallografische studie van de trekproef-monsters. De korrelsubstructuur die ontstaat in de ferrietkorrels in de opeenvolgende fazen tijdens een trekproef werd in detail geanalyseerd, met speciale aandacht voor de vervormde zone in de onmiddellijke omgeving van 2e fazedeeltjes. Deze analyse werd vervolledigd met een 3D microstructurele studie die uitgevoerd met de Focus Ion Beam (FIB-EBSD) techniek. Niettegenstaande de betrekkelijk hoge zuiverheid van IF staal, wordt vastgesteld dat primaire holtes zich vormen aan de TiN deeltjes die ingebed zijn in de ferritische microstructuur.

De schade micromechanismen in dual phase stalen van het type DP600, die gemodificeerd werden met verschillende warmte-behandelingen, werden bestudeerd met behulp van microscopische technieken. In het eerste deel van dit hoofdstuk wordt de microstructuur bestudeerd van de het koudgewalste DP staal, alsook de microstructuur na de warmtebehandeling. De mechanische eigenschappen van de verschillende DP-stalen werd onderzocht aan de hand van trekproeven en een uitgebreide analyse van de geometrie van de monsters na breuk. De preferentiële plaatsen voor holte-initiatie in de onderzochte DP staalsoorten werden voornamelijk geassocieerd met de aanwezigheid van de martensietfaze. Tenslotte wordt opgemerkt dat afschuifbanden verantwoordelijk zijn voor de laatste fase van breuk door als kiemplaatsen te fungeren voor scheuren.

Het breuk-micro-mechanismen van twee AA6016 aluminiumlegeringen met verschillende magnesiumgehalte werden bestudeerd in dit hoofdstuk aan de hand van uni-axiale trekproeven. Een fractografische studie met behulp van SEM beelden suggereert twee soorten faalmechanismen. Het eerste wordt gekenmerkt door een opeenvolging van gebeurtenissen, zijnde initiatie, groei en coalescentie van micro-holtes ontstaan aan intermetallische deeltjes; terwijl het tweede kan worden geïdentificeerd als
ductiel intergranulair falen. Het tweede breuk-mechanisme wordt vaker waargenomen in de aluminium legering met een hoger magnesiumgehalte. In beide aluminiumlegeringen werden secundaire scheuren gevonden dichtbij het breukvlak, zoals geobserveerd op een dwarsdoorsnede van een trek-proef-monster. Oriëntatie-contrast-beelden tonen aan dat de secundaire scheuren ofwel werden gevormd door een intergranular faalmechanisme of door lokale plastische instabiliteiten zoals shear bands die zich vervolgens tot scheuren ontwikkelen. Het hogere gehalte aan magnesium geeft een hogere sterkte, maar vermindert de taaiheid door het verbrossen van de korrelgrenzen.

Hoofdstuk 7 richt zich op het modelleren van de evolutie van lokale spanningen tijdens rekvervorming in de martensiet- en ferrietfase in DP stalen. Om het mechanisch gedrag van DP staalsoorten the simuleren, is een micromechanische model ontwikkeld gebaseerd op een fysisch koud-verstevigings-model rekening houden met een verschillend kristalplastisch gedrag voor elk van de microstructurele bestanddelen. In dit model worden volgende microstructurele toestands-variabelen in aanmerking genomen: de kristallografische oriëntatie van martensiet- en ferriet-korrels, alsook de dichtheid aan Geometrisch Noodzakelijke Dislocaties (GNDs). Het model werd eerst geijkt en nadien gevalideerd op basis van experimentele gegevens van DP staalsoorten die onderworpen werden aan verschillende warmtebehandelingen.
List of Publications

Journal papers


Conference contributions


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Curriculum Vitae

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Born on 29 June 1980 in Distrito Federal, Mexico

1998 - 2003 Mechanical Engineer, Universidad Nacional Autónoma de México (UNAM), Mexico
Thesis title: *Modelling and Simulation of Cold Rolling Process by Slab Analysis*
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Awarded as the best graduated mechanical engineer from the UNAM (Generation 99)

2003 - 2004 Master in Materials Science in the ALFA program “Fray Pedro de Gante”, Gent University, Belgium
Thesis title: *Effect of Lubrication on the Evolution of texture in Cold Rolled Ultra Low Carbon Steel at Different Thickness Reductions*
Daily supervisor: Dr. Ir. Ana Carmen da Costa Reis
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2005 Researcher, Gent University, Belgium
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Research project title: *A study on the mechanism of mechanical anisotropy of API steels*
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2010  
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Research project title: *Crystallographic aspects of the austenite formation and decomposition during processing and forming of low-carbon TRIP-assisted steels*

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