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Plastic behavior, damage mechanisms and crack propagation resistance in high-toughness Dual-Phase steels

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My personal ethical viewpoint on the purpose of this study

One of the final purposes of this study, if not a personal achievement and a teamwork for the progress of science, is the common aim of the automotive industry to decrease the vehicles weight so as to achieve the supposedly ecological goal of consuming less fuel. Sure, given the colossal number of vehicles worldwide, decreasing the vehicles weight has a compelling impact on the amount of carbon dioxide released in the atmosphere and constitutes a work that must be pursued. And as regards the often claimed ecological electric or fuel cell driven cars, although commendable because potentially much better regarding their life cycle analysis, the carbon dioxide emitted and other issues related to production are far from negligible. Replacing each and every internal combustion car by a somewhat lighter electric car or fuel cell driven car, all things equal otherwise, would keep the automotive sector among the big polluters.

Facing the climate emergency, such changes are not sufficient in order to remain well below the 2°C, and preferably below the (very unlikely) 1.5°C global warming threshold of temperature increase since pre-industrial levels, set by the COP21. A deep systemic change of economy and society functioning seems essential to me. The change must act on a large variety of domains with a priority to the most impacting (e.g. nutrition and land use, energy production, transport at all levels, insulation, heating and cooling, data storage, overall consumption, degrowth, carbon capture and storage...).

Focusing on car issues to stick to the topic at hand, this can translate into combining 'soft' solutions (e.g. the decrease of vehicles weight, cleaner engines, the transition to an automobile fleet of electric and fuel cell driven vehicles) to a drastic reduction of their number, which is, at the time of writing this thesis, a failure of monumental proportions. The number of cars keeps on increasing (data in the EU [1]) and, in general, the average car user is very much attached to its car(s), even for trips that can easily be performed by public transport. Almost half of cars in Brussels perform intra-Brussels trips. 25% of the trips are shorter than 1 km, only 20% of the trips are longer

than 10 km (data from Brussels [2]). In my ideal world, cars are used in case of emergencies or in a shared way for occasional trips. Of course, given the magnitude of the necessary changes for a world with less cars, and given the bewildering current dependence of the masses of the people on the car established in the space of a century, a period of transition is necessary to make it concrete. Several possibilities that could help this goal follow, without being exhaustive.

The reduction of the number of cars can go through the progressive end of private cars. One step towards the end of private cars is the development of the offer and network of shared cars (following the business models of Cambio, Zen Car, Zipcar, DriveNow...). This could in turn be coupled to an application that optimizes the use of the shared vehicles, making use of the data about the daily movements of its users. That would help avoid acquiring a car for a very short daily use. Indeed, the average car is in use (i.e. fulfilling its primary purpose) only 3 to 4% of the time and stays parked at home for about 80% of the time (data from the United Kingdom [3]). Also, as approximately 14.4% of the vehicles on the roads are commercial vehicles (data in the EU [1]), an application similar to that for the shared car network based on daily use data could be developed to optimize the delivery system, focusing furthermore on maximizing the filling. Indeed, in many instances, commercial vehicles and trucks are empty, ranging from 12% to almost 40% of the time spent on the road (data in the EU [4]).

Talking about data, autonomous cars may flood the market in a few decades. They will probably be more reliable, effective, fast and safe. However, the amount of data they will create and the need to back it up (for reasons of functioning in the short-term and for longer term insurance and legal purposes) worries me due to the carbon dioxide emissions it would generate (combined with the progressing internet of things and the larger amount of data overall).

Providing incentives to follow a particular behavior delivers better results than prohibiting the opposite behavior. Such incentives are to improve the public transport system by increasing their comfort to compete with the comfort of a private car, increasing frequency and developing the network. Another important incentive is to render them financially competitive and thus cheap compared to cars. The cost associated to tax benefits on company

cars borne by the Belgian State, and thereby any taxpayer even though it is mostly the affluent who benefit from this advantage, amounts to at least €2.3 and probably around €4 billions per year [5, 6, 7]. This scheme promotes road travel and impedes public transport use. Putting an end to the favorable tax treatment for company cars would leave a convenient investment margin for cheap and quality public transport. My naive guess for which the company car system is conserved is on the one hand the attachment of the beneficiaries to this privilege and on the other hand that it allows for a higher economic activity related to a higher mobility. This constitutes one of many examples where greed for a high and short-term comfort and economy win over sustainability, a typical tragedy of the commons. On the contrary, the tax on cars should be increased. Of course, it must be performed in a thoughtful manner in order to avoid penalizing mostly people with reduced incomes. This remains applicable, and of paramount importance, regardless of the measure taken.

The layout of the roads could additionally be modified so as to facilitate and secure the riding of bicycles and other modes of soft mobility (skateboards, scooters, monowheels...) and give them a higher priority compared to cars. In addition, car trips should be simultaneously rendered less comfortable. For instance, in line with reducing the proportion allocated to cars for a given road in favor of soft mobility, some roads within cities could be completely blocked to road traffic. They could then be transformed into better living places and/or into roads entirely dedicated to bicycles. This is possible and can work very well as demonstrated for instance by Houten, a city in the Netherlands. It was designed so that getting around by bicycle is faster than by car. The bicycle roads and amenities moreover offer a great comfort to their users.

Other examples of measures are the introduction of limits on taking the car alone on portions where alternatives exist or the introduction of limits on large cars (when not needed). The second would moreover have a positive impact on road safety as a large difference in vehicle weight leads to more severe accidents [8].

The implementation of the restrictive measures must (unfortunately in view of the emergency) not be too abrupt. The reason is that, as shown by history, or simply by the behavior of children towards their parents, a privilege once

granted inevitably gives rise to strong resistance once suppressed. To facilitate acceptance of the measures, sometimes limiting what is considered freedom, concepts of behavioral science could be applied on a large scale. Policies should be designed keeping in mind that small changes in the context can lead to large impacts on people's choices. Techniques of benevolent manipulation could be used to achieve a more eco-friendly behavior, such as priming, framing, giving people a choice between two eco-friendly alternatives, setting eco-friendly default options (case in which the inertia of current mobility could be fought through the inertia of the default options), etc. For more information about cognitive biases, I recommend reading 'Thinking fast and slow' from Daniel Kahneman, and 'Nudge' from Richard Thaler and Cass Sunstein.

On top of the carbon dioxide emissions, massive car use inflicts welfare costs to society by exacerbating air pollution. The emissions of NO_x and fine solid particles are responsible for many diseases and premature deaths (data from the European Environment Agency [9]). The pollutants are inhaled by pedestrians and by cyclists, especially when forced to breathe close to the exhausts in cities still primarily designed for cars (e.g. 70% of the public space is dedicated to cars in Brussels [2]), even though car users should also feel concerned about it [10]. Health issues put aside, working for a field responsible for a large amount of the carbon dioxide emissions while striving personally for a more ecological behavior is worth considering. I wish not to participate in lifestyles that are harmful to the Earth's balance. A major concern of which is of course the anthropogenic climate crisis that will, and already did, cause plentiful animal suffering, human misery and, entering the Holocene mass extinction, the waste of millions of years of evolution.

These few paragraphs made clear that I am opposed to the massive use of cars. Yet, according to forecasts for future car sales, one should not expect a rapid decrease in number of vehicles (data from the U.S. Energy Information Administration [11]). Despite the above, my hope is that what follows is quality science and will be enjoyed by the readers. I moreover hope that the conclusions of this thesis, relatively general and fundamental, could at some point be applied to other fields than the automotive or aeronautical fields, ideally for a more sustainable application such as the railway sector, recycling or reuse. Moreover, an additional hope is that the skills I have developed

and acquired during my PhD will serve in the future to work towards the realization of sustainable applications.

The thesis counts among its objectives to decrease the vehicles weight and increase their strength, these few pages have the objective of strengthening the weight of the words in relation to the consequences of the massive use of cars. The comfort of a car, in my opinion, does not justify the importance of negative externalities that result from the use made out of it nowadays.

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Chapter 1

Introduction

1.1 General context

For their interesting mechanical properties at reasonable costs compared to other metallic alloys, steels are since a long time produced massively worldwide and are used in applications as various as civil and mechanical engineering, domestic devices, pipelines, art, ... In particular, the automotive industry, which is of interest in the present work, abundantly consumes steels. The use of steel sheets indeed enables a combination of interesting formability properties, high levels of strength and crashworthiness, at affordable cost.

In the last decades, environmental standards have rightly become more severe, although the vast majority of efforts remain to be provided. A step towards the reduction of fuel consumption is to decrease vehicle weight. As the structural part of vehicles (the body-in-white) represents on average 40% of the total vehicle weight, reducing the mass of the structure is an impactful way of reducing vehicle weight. The total vehicle weight is actually continuously increasing due to comfort equipments (such as seats, air conditioning and numerous innovative useful and less essential electronic options), together with a vehicle size increase. Another reason of the weight increase is an increase in car size. Furthermore, safety requirements are becoming stricter as well.

For meeting today's requirements, by carefully controlling chemical composition and thermomechanical treatment, Advanced High-Strength Steels (AHSS) are processed (*Bleck, 1996; Bouaziz et al., 2013*). These AHSS include Dual-Phase steels, complex-phase steels, martensitic steels, ferrite-bainite steels, transformation-induced plasticity steels (TRIP) and very

effective though costly steels such as twinning-induced plasticity steels (TWIP).

Two fundamental properties for formability and energy absorption are strength and ductility. Figure 1.1 shows how steels compare to one another with respect to elongation (quantifying ductility) and tensile strength (quantifying strength). This Figure 1.1 clearly illustrates that strength and ductility are antagonist properties.

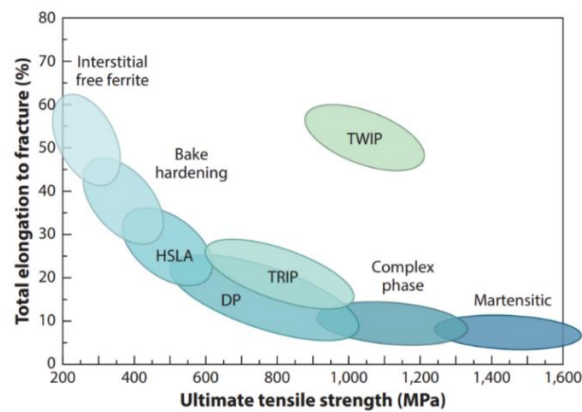


Figure 1.1 Total elongation to failure as a function of ultimate tensile strength for various steel grades. (Tasan et al., 2015)

The steels of interest for the present work are Dual-Phase steels, nowadays extensively used in the automotive industry as evidenced by Figure 1.2.

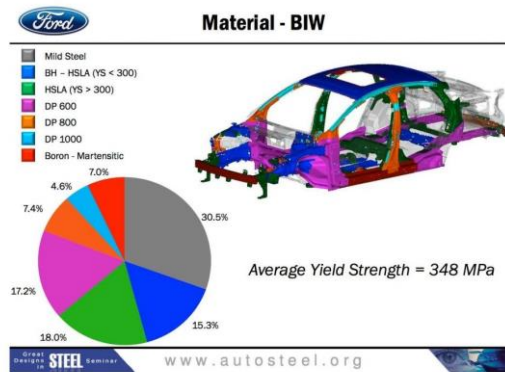


Figure 1.2 Illustration of the use of Dual-Phase steels in the automotive structure. (image taken from www.autosteel.org)

1.2 Motivation and objectives

In addition to strength and ductility, other important mechanical properties are resistance to fatigue, joining capability and fracture toughness. The latter, which quantifies the ability to resist to the propagation of an existing crack, is often overlooked. The scientific literature dealing with fracture toughness of Dual-Phase steels is, although not non-existent, modest for the time being. Moreover, the few studies often report relatively low levels of fracture toughness (e.g. *Lacroix et al., 2008*). Therefore, improving fracture toughness of Dual-Phase steels is crucial to extend their use in structural components that must resist cracking. Furthermore, a sufficiently high fracture toughness is required for some forming operations. For instance, small cracks may initiate on the edges during the cutting operations of Dual-Phase steels sheets, which is often performed by shearing. The cracks occasionally propagate during stamping or other subsequent forming steps implying deformation. Figure 1.3 illustrates such a case. A high fracture toughness is needed to mitigate these issues and withal, increase the recyclability and reusability of deteriorated steels.

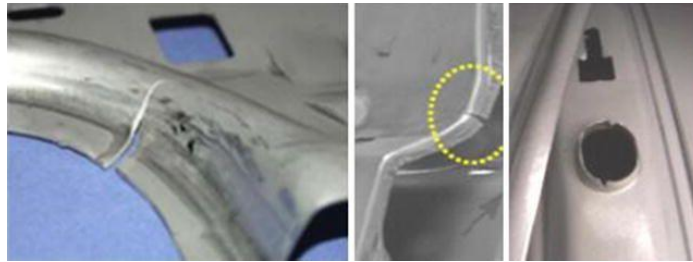


Figure 1.3 Example of edge cracking. (Casellas *et al.*, 2016)

Recently, Dual-Phase steels with a lamellar microstructure, called ‘Thomas fibers’ (Kim and Thomas, 1981), have been re-investigated showing some very promising fracture behavior compared to classical Dual-Phase steels. In particular, Pierman (2013) showed in her thesis that ‘Thomas fibers’ Dual-Phase steels were able to reach surprisingly high levels of fracture toughness. To illustrate this, Figure 1.4 shows a collection of J_{Ic} and of essential work of fracture w_e (quantifying fracture toughness) as a function of the fracture strain ε_f (quantifying the deformation before failure) for various steels (Dual-Phase steels, TRIP steels, CrMo steels, stainless steels) as well as of other metals (Al alloys, brass, zinc, bronze) in order to set a wide basis of comparison to discuss the potential of the ‘Thomas fibers’ Dual-Phase steels. The values are taken from the literature (Guan *et al.*, 2011; Lacroix *et al.*, 2008; Mao *et al.*, 1992; Pardoën *et al.*, 2004; Pierman, 2013). The bolder dots appearing in red represent ‘Thomas fibers’ Dual-Phase steels addressed in this thesis and the bolder dots appearing in blue represent ‘Thomas fibers’ Dual-Phase steels addressed by (Pierman, 2013). The origin of the very high values for Dual-Phase steels constitutes the subject of the present thesis. Only a few materials outperform or match the ‘Thomas fibers’ Dual-Phase steels, those are stainless steels and TRIP steels, known for their high cracking resistance but also for much more expensive compositions in alloying elements.

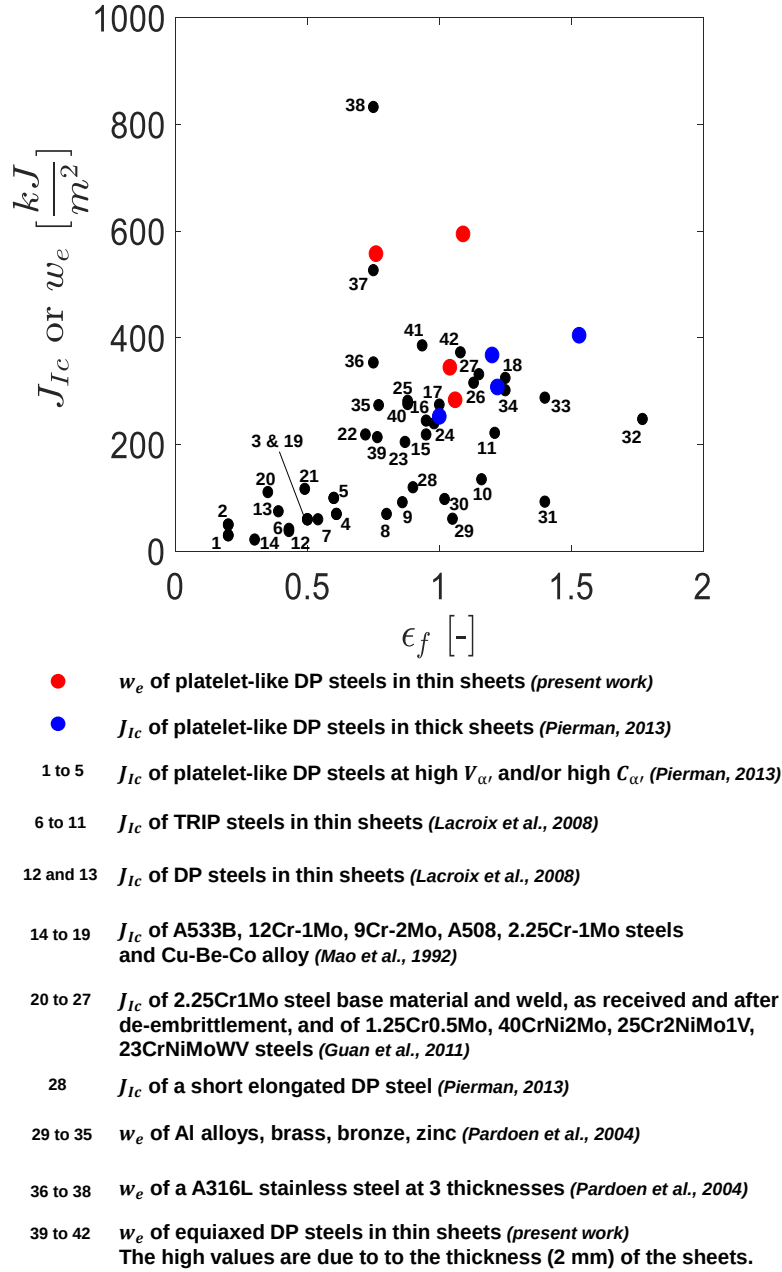


Figure 1.4 Initiation fracture toughness J_{Ic} or essential work of fracture w_e as a function of fracture strain ϵ_f for various steels (Dual-Phase steels, TRIP steels, CrMo steels, stainless steels) but also other metals (Al alloys, brass, zinc, bronze). Some ‘Thomas fibers’ Dual-Phase steels (red and blue bold dots) compete with the toughest steels.

Given the possibility of a high fracture toughness in Dual-Phase steels, the goal of this thesis is to investigate the potential of ‘Thomas fibers’ Dual-Phase steels and explore the origin of the good behavior. More precisely, the fundamental scientific objective of this thesis is to elucidate the relationship between microstructure and cracking resistance. This requires an understanding of the fundamental mechanisms governing damage behavior, which depends on the local plastic behavior. In particular, the role of the morphology on the plastic behavior, damage behavior and crack propagation resistance is a main interest of this work.

Therefore, classical and ‘Thomas fibers’ microstructures were processed with varying martensite volume fraction and thickness. Damage and cracking resistance were systematically characterized on top of the traditional elastoplastic properties. Computational modeling enabled to gain insight into the role of microstructure morphology on mechanical properties of Dual-Phase steels through a systematic investigation of the effect of particle shape and orientation on the effective plastic behavior, on the local mechanical fields of Dual-Phase steels and on damage behavior. This was performed by taking the mean behavior of ferrite and martensite into account without going into the details of the phase structure.

1.3 Outline of the thesis

In addition to this introductory chapter, six chapters will contribute to the scientific plan of action to achieve the objectives.

Chapter 2 provides background information about Dual-Phase steels. It browses first the important aspects of Dual-Phase steels metallurgy, their specific mechanical properties and the importance of heterogeneities. Afterwards, the main numerical tools used for the modeling and the main experimental findings about plasticity, damage behavior and crack propagation resistance that are relevant in the framework of this work are reviewed.

Chapter 3 presents the Dual-Phase steels used in this study. They are model materials, generated in a way to master the microstructural parameters. Details about the materials, the processing, the microstructural characterization and the elastoplastic properties are presented.

Chapter 4 deals with the modeling of the plastic behavior of Dual-Phase steels. A micromechanical model based on elementary cells is developed to study the impact of second phase morphology and orientation at single grain and multi-grain levels, and on the local fields.

Chapter 5 is dedicated to the experimental characterization of fracture. Damage is observed post-mortem on tensile and DENT specimens. Crack propagation resistance is investigated mainly through the use of the essential work of fracture method. The hole expansion ratio is also quantified.

Chapter 6 is a brief chapter presenting preliminary results about the modeling of damage through a cellular automaton approach.

Chapter 7 summarizes the main results, and in particular, how Dual-Phase steels microstructures affect the plastic behavior, the damage mechanisms and the crack propagation resistance. Research perspectives are proposed at last.

Appendix A provides additional information about the processing of the materials. Appendices B, C and D are complementary to Chapter 4 providing additional information and results about the modeling of plastic behavior. Appendix B roughly assesses the impact of heterogeneities, martensite interconnectivity and internal stresses on the plastic behavior. Appendices C and D analyze slightly further the local fields following deformation. Appendix E provides supporting data relative to the essential work of fracture study.

Chapter 2

Theoretical background

Chapter 2 aims at giving the necessary knowledge about Dual-Phase steels for the understanding of the following chapters. Section 2.1 presents the key aspects of the metallurgy of Dual-Phase steels with information about the microstructure and processing. The relationship is then made with the mechanical properties, from plasticity (Section 2.2) to crack propagation resistance (Section 2.4) through damage behavior (Section 2.3). Throughout, experimental observations and modeling tools are presented. A more exhaustive state of the art about Dual-Phase steels is also available in (*Tasan et al., 2015*).

2.1 General features about the metallurgy and mechanical behavior of Dual-Phase steels

2.1.1 Ferrite and martensite phases

As illustrated in Figure 2.1(a), a Dual-Phase steel is intrinsically a composite material, comprising two phases, the martensite denoted α' and the ferrite denoted α . Retained austenite and bainite may be present in small proportions as well. Most often, the matrix is ferritic with a distribution of more or less isolated martensite grains. Martensite is hard compared to ferrite, and is responsible for a good part of the large hardening. On the other hand, the martensite is much less ductile than ferrite, see Figure 2.1(b). The ductility decreases when martensite carbon content increases, which can even lead to failure before necking during a tensile test (*Roumina et al., 2013*). Tempering is used in martensitic steels to increase ductility.

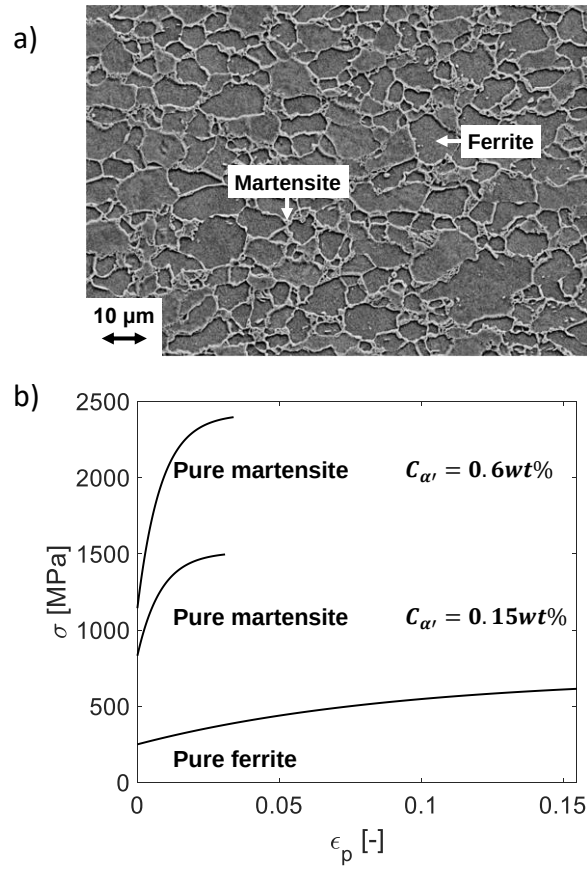


Figure 2.1 a) Micrograph showing the phases present in a typical Dual-Phase steel microstructure. b) Typical hardening behavior for the ferrite α and martensite α' phases up to necking.

Information about crystal structure is needed to understand the origin of the advantages of Dual-Phase steels. Ferrite, a stable allotrope of iron at room temperature, has a body centered cubic (BCC) crystal packing. The martensite phase generally exhibits a body centered tetragonal (BCT) structure transformed from austenite, which has a face centered cubic (FCC) structure.

Martensite formation results from quenching to room temperature that leads to a displacive transformation of the austenite, which is a diffusionless shear mechanism (Bhadeshia, 2006). The carbon is trapped in the martensite and its presence induces a lattice distortion. Moreover, the austenite-to-martensite transformation causes a volume expansion (Moyer

and Ansell, 1975). The distortion, together with the volume expansion, causes the accumulation of dislocations in the as-quenched microstructure (Krauss, 2001), which leads to a gradient in the properties (Brands et al., 2016).

The carbon solubility in ferrite is low, equal at most to 0.02wt.%, and even much less at room temperature. This small amount of carbon is sufficient however to cause the Lüders band phenomenon and the discontinuous yielding of pure ferrite, a phenomenon that disappears in Dual-Phase steels for a reason that will be described later. In Dual-Phase steels, the carbon balance is found in the martensite phase.

2.1.2 Processing of Dual-Phase steels

The composition of Dual-Phase steels is principally based on the presence of carbon (0.05 to 0.2 wt.%) and manganese (typically of the order of 1 to 3 wt.%), both of which stabilize austenite. Carbon's presence moreover increases martensite strength, and manganese leads to solid-solution strengthening within ferrite. Other alloying elements may be found, though in smaller quantities than in the majority of AHSS. The targeted alloying can serve different purposes, e.g. chromium or molybdenum can be added to hinder the formation of pearlite and bainite, silicon to accelerate ferrite transformation, vanadium and niobium to increase precipitation strengthening and refine the microstructure. The smaller quantities of alloying elements limits the cost of Dual-Phase steels and thereby renders them interesting from an industrial perspective.

In order to generate the common equiaxed Dual-Phase steel microstructure, an intercritical annealing of the initial cold-rolled ferrite-pearlite steel is typically applied in the ferritic-austenitic domain, followed by water quenching below the martensite start temperature M_s to induce the transformation of austenite into martensite. Figure 2.2 shows the typical equiaxed microstructure, together with the schematic thermal history. Martensite particles appear in light grey and ferrite in dark grey.

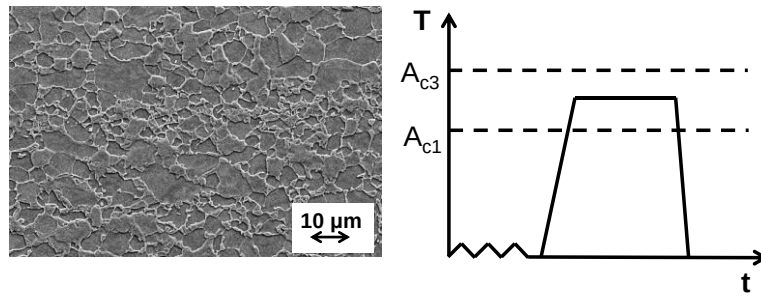


Figure 2.2 Equiaxed microstructure and schematic associated heat treatment.

In addition, Figure 2.3 shows a TTT diagram (Time-Temperature-Transformation diagram) illustrating schematically the temperatures and cooling rates to be achieved in order to obtain a Dual-Phase steel, for a given steel composition given schematically as well in the relevant part of the Fe-C phase diagram.

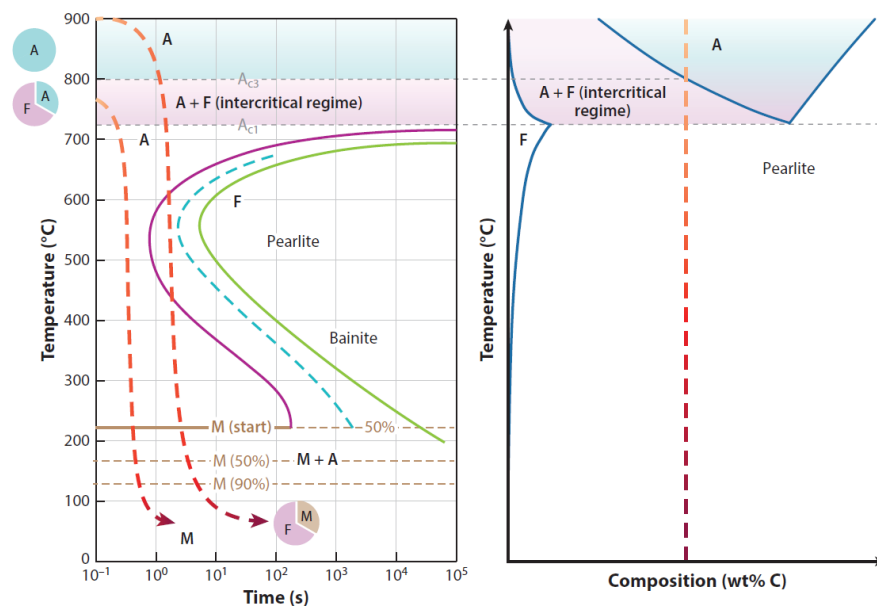


Figure 2.3 TTT (Temperature-Time-Transformation) diagram for steels and corresponding Fe-C phase diagram.

The low coherency between the crystal packing of ferrite and austenite resulting from their different crystal structure (see Section 2.1.1), in addition to the volume expansion during austenite formation, leads to a large

activation energy for austenite nucleation within a ferrite grain (*Becker and Hornbogen, 1979*). Therefore, heterogeneous nucleation, for which austenite nucleates on defects such as along the grains boundaries, phase boundaries (e.g. ferrite-cementite interfaces where carbon is available), at the ferrite triple junctions or at the dislocation cells boundaries, is favored over homogeneous nucleation.

Hence, during a conventional intercritical annealing, the nucleation and growth of the new austenite grains occurs at defects, the distribution of which influences the final Dual-Phase steel microstructure. Several morphologies can thus be obtained depending on the initial microstructure on which the heat treatment is applied.

In the context of the automotive industry, the heat treatment is classically applied on a cold-rolled sheet of steel, as a step of a continuous casting process. In such a case, the microstructural defects are finely distributed and dispersed. The recrystallization of the cold-rolled microstructure during heating then leads to the characteristic microstructure with a fine dispersion of grains of a few microns of ferrite and martensite (*Delincé et al., 2006; Delincé, 2008; Jacques et al., 2006; Landron et al., 2011; Mazinani and Poole, 2007*). The austenite nucleation step is illustrated in Figure 2.4, in which nucleation at the dislocation cells boundaries is represented. Indeed, cold-rolling leads to the multiplication of dislocations that organize into fine dislocation cells within the grains, which are preferential nucleation sites for austenite formation.

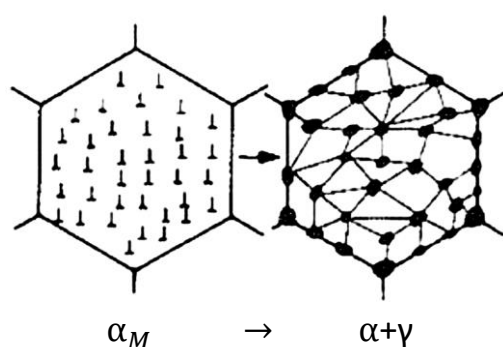


Figure 2.4 Schematic of austenite nucleation on a cold-rolled steel. The austenite nucleates on the dislocation cell walls.

Regarding the mechanisms of austenite formation, starting with a ferrite-pearlite microstructure, the rate of austenite growth decreases throughout the formation (*Speich et al., 1981; Yi et al., 1985*). At first, heterogeneous nucleation of austenite occurs almost instantaneously and is followed by a rapid austenite growth at the expense of the pearlite, until its complete dissolution. Due to carbon availability from cementite, the newly formed austenite nuclei located at the pearlite grow faster than the nuclei at ferrite-ferrite grain boundaries. The latter rely on carbon diffusion from carbon-rich areas through the ferrite matrix up to the austenite-ferrite interface. After the pearlite dissolution, the austenite continues its growth within the surrounding ferrite. Again, due to carbon availability, the rate of growth is slower and is determined by carbon diffusion inside the austenite. At last, the equilibration of ferrite and austenite is even slower, the rate of which is determined by manganese diffusion in austenite. The austenite volume fraction depends on the temperature during the annealing.

Alternatively, a few studies considered another morphological arrangement of ferrite and martensite, a microstructure commonly called as ‘Thomas fibers’, in tribute to the first author who, to our knowledge, has generated this microstructure (*Kim and Thomas, 1981*). In the present case, the microstructures result from a two-stage heat treatment. At first, a full austenitization followed by water quenching takes place. Secondly, an intercritical annealing is applied to the resulting fully martensitic microstructure. The reversion of martensite to austenite induces its nucleation and formation at grain boundaries and at the interfaces of the martensite laths. Figure 2.5 shows the austenite nucleation spatial distribution starting from a fully martensitic microstructure. As opposed to Figure 2.4, few dislocations are present at the stage of intercritical annealing due to the preceding full austenitization.

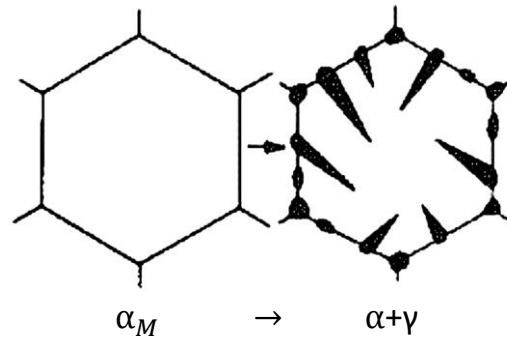


Figure 2.5 Schematic of austenite nucleation on a martensitic steel. The austenite nucleates on the grain boundaries along the martensite laths, the dislocation density being low due to the full austenitization.

A final water quenching brings about the particular microstructure, on which more details will follow. Figure 2.6 shows a representative ‘Thomas fibers’ microstructure, together with the schematic thermal history. This kind of microstructure has been shown to lead to excellent fracture toughness levels by (Pierman, 2013) and constitutes the core motivation of this thesis.

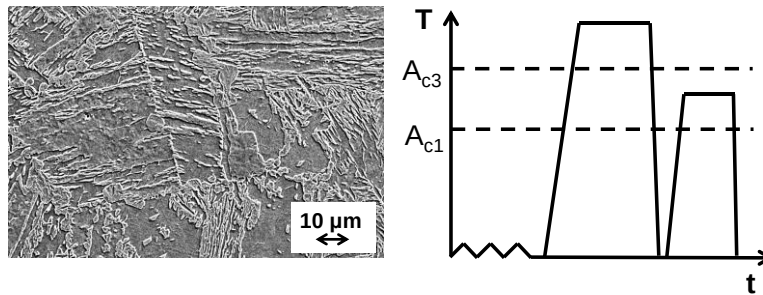


Figure 2.6 ‘Thomas fibers’ microstructure and schematic associated heat treatment.

In addition to austenite formation, other metallurgical mechanisms can take place during the heat treatment: recovery, recrystallization, diffusion, carbide precipitation, pearlite spheroidization. If, and to what extent these phenomena occur and interact influences the final microstructure and depends on the processing parameters: heating rate, annealing temperature and time, cold-rolling reduction (Azizi-Alizamini *et al.*, 2010; Huang *et al.*, 2004; Peranio *et al.*, 2010; Yang *et al.*, 1985).

For instance, if the heating rate is low enough, ferrite recrystallizes prior to austenite nucleation, leading to a random austenite distribution during the

annealing and ultimately to an equiaxed microstructure. In case of a high heating rate, ferrite recrystallizes later, and austenite exhibits a similar spatial distribution as the elongated pearlite, which eventually leads to microstructural banding. The initial microstructure and composition are also important. For instance, recrystallization does not occur in low-carbon martensitic steels (*Bhadeshia, 2006*).

Furthermore, in case of rapid quenching, almost only martensite and ferrite are present in the final Dual-Phase microstructure. Indeed, retained austenite generally remains in proportions between 2 and 9% (*Speich, 1981*). However, other phase transformations are possible during cooling depending on the cooling rate (and on austenite hardenability, which depends on composition), namely the formation of epitaxial ferrite (*Huppi et al., 1981; Speich, 1981*) and bainite. At intermediate temperatures, bainite may form in the austenite in case of a slower cooling, see the TTT diagram in Figure 2.3.

2.1.3 Characteristic mechanical properties of Dual-Phase steels

The good compromise between strength and ductility of Dual-Phase steels results from the very different and complementary properties of the two constitutive phases. The deformation incompatibility between these constitutive phases is responsible for the generation of large back stresses, which dominates the work-hardening response (*Brown and Clarke, 1975*). The different properties confer to Dual-Phase steels the characteristic mechanical properties of a continuous flow curve and a high initial work-hardening rate. In addition, Dual-Phase steels have a moderate to high yield strength depending on martensite content and interesting bake-hardening properties (*Bleck, 1996; Bouaziz et al., 2013; Rachid, 1981; Sakaki et al., 1983; Speich and Miller, 1979*).

a) Continuous flow curve

High-strength low-alloy steels generally present Lüders bands giving rise to a Lüders plateau in the tensile stress-strain response, whereas Dual-Phase steels exhibit a continuous flow curve. A stress-strain curve resulting from a tensile test involving a Lüders band is schematically illustrated in Figure 2.7.

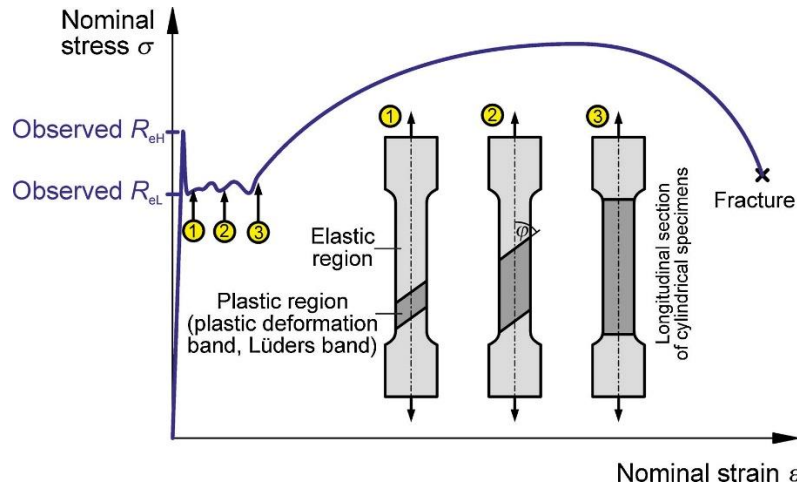


Figure 2.7 Schematic of a stress-strain curve in the presence of a Lüders band in the tensile sample and illustration of the corresponding plastic deformation band (Schwab and Ruff, 2013).

The Lüders plateau is due to the formation of Cottrell atmospheres. In such atmospheres, dislocations are pinned as a result of their interactions with diffusive carbon and their glide, necessary for plastic deformation accommodation, is hindered. Larger stresses are needed to overcome the pinning obstacles. After elasticity, this leads to a rather constant stress level over some deformation (Lüders plateau) with a heterogeneous strain pattern in the material (Lüders bands). This phenomenon occurs until all dislocations have been liberated from Cottrell atmospheres.

The continuous yielding of Dual-Phase steels is probably the result of the presence of the residual stresses and a high density of mobile dislocations within the ferrite region around the martensite phase (Korzekwa *et al.*, 1984). Indeed, geometrically necessary dislocations (GNDs) are produced at ferrite-martensite interfaces to accommodate the deformation due to the austenite-to-martensite transformation. They are necessary for lattice continuity to be kept in ferrite that plastically deforms because of the volume expansion of forming martensite. The near interface zones have the highest dislocation density, the latter steadily decreases when moving away from the interfaces (Sakaki *et al.*, 1983; Tanaka *et al.*, 1979). As opposed to the pinned dislocations in Cottrell atmospheres, the now mobile dislocations can move and no yield drop phenomenon is observed. More precisely, zones with the highest dislocation density near interfaces yield first at low applied stress

explaining the relatively low yield stress of Dual-Phase steels. The zones in the middle of the ferrite grains yield last. The yielding occurs in a continuous manner between the two regions, explaining the continuity of the flow curve. (Tian *et al.*, 2020) moreover argue that the ferrite phase is responsible for the early yielding of the Dual-Phase due to a smaller critical resolved shear stress.

b) High initial work-hardening rate

Compared to other steels, Dual-Phase steels exhibit a high work-hardening rate at small strains (Davies and Magee, 1979). It is partly due to the back stress present to meet the conditions for plastic compatibility between ferrite and martensite (Gerbase *et al.*, 1979), and to the heterogeneous yielding in ferrite that also contributes to the Bauschinger effect (Sarosiak and Owen, 1984).

2.1.4 Importance of microstructure heterogeneities

The mechanical behavior of Dual-Phase steels is the result of an interplay of mechanisms, most of them dictated by various parameters that are sources of inhomogeneities as illustrated in Figure 2.8. Even though the different mechanisms and characteristics are interconnected (e.g. an increase in martensite volume fraction is generally accompanied by changes in morphology and connectivity), the inhomogeneities have identifiable origins. For instance, the stress and strain distributions can be due to chemical inhomogeneities. Besides the difference in composition of the phases, there may be composition gradients within the phases or differences in carbon - or other chemical element - content between different particles, or zones in the microstructure. The martensite carbon content, which influences the local hardness, is especially heterogeneous when the annealing temperature is close to the A_{c1} temperature due to undissolved cementite, controlled by Mn diffusion (Scott *et al.*, 2018). Furthermore, stress and strain inhomogeneities within each phase are present in Dual-Phase steels resulting from processing. For instance, ferrite grains have a higher hardness at the ferrite-martensite boundary compared to their very center (Kadkhodapour *et al.*, 2011a; Ramazani *et al.*, 2013a) due to martensite expansion. Brands

et al. (2016) observed an exponential increase in hardness when approaching the martensite particle using nanoindentation. *Yan et al.* (2015) also observed severely localized strains, even within ferrite grains. Moreover, orientation gradients are also higher at ferrite-martensite interfaces compared to ferrite-ferrite interfaces (*Calcagnotto et al.*, 2010b). In addition to the stress and strain distributions due to processing, a strain distribution builds up within ferrite grains with plastic deformation (*Liao et al.*, 2017). Again, the highest strains are located at ferrite-martensite interfaces (*Karlsson and Sundström*, 1974). Inhomogeneities in strain and stress fields are also due to microstructural heterogeneities. For instance, (*Zarei et al.*, 2016) attribute the stress and strain distributions partly to crystallographic orientation and shape of the grains. An increase in martensite volume fraction also increases the inhomogeneity of the plastic deformation process (*Liao et al.*, 2017). More examples of microstructural heterogeneities in Dual-Phase steels are among other things morphology, the different shapes and sizes of martensite particles and ferrite grains, the orientation of non equiaxed particles or their spatial distribution, which sometimes leads to anisotropic mechanical properties (*Liao et al.*, 2017).

Heterogeneities have repeatedly been shown to play an important role on controlling the damage accumulation and cracking initiation process in materials, see e.g. in Al alloys (*Hannard et al.*, 2016) or on fracture toughness of bone (*Nalla et al.* 2005).

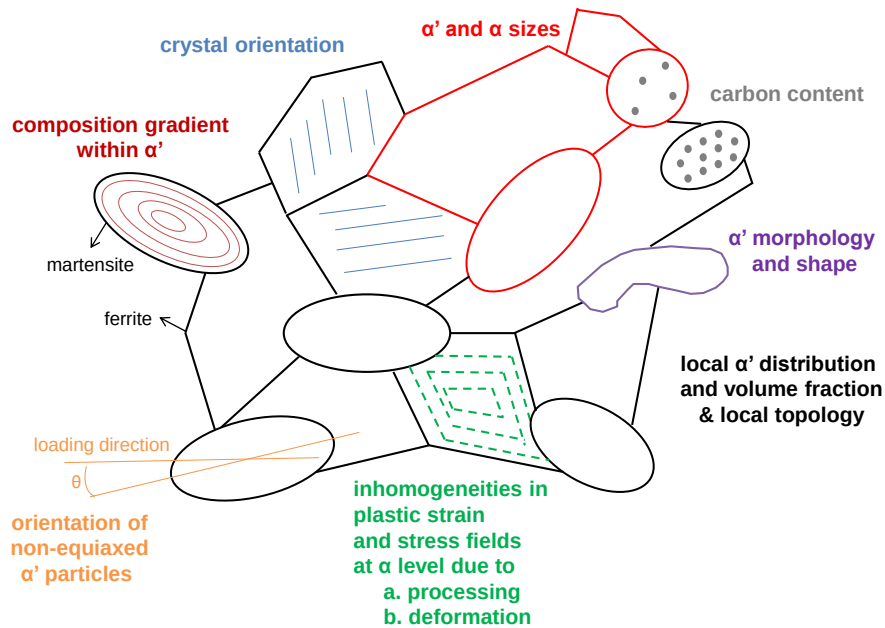


Figure 2.8 Heterogeneities of diverse origins impact the mechanical behavior of Dual-Phase steels.

2.2 Plastic behavior of Dual-Phase steels

2.2.1 Experimental evidences

Experimental investigations reported in the literature mainly focused on the impact of martensite volume fraction, ferrite grain size and carbon content. An increase in martensite volume fraction leads to higher yield and tensile strengths (*Chang and Preban, 1985; Davies, 1978; Kumar et al., 2008; Lai et al., 2015; Lai et al., 2016; Marder, 1982; Mazinani and Poole, 2007; Pierman et al., 2014; Ramos et al., 1979; Sarosiek and Owen, 1984; Speich and Miller, 1979*), with a larger impact on tensile strength than yield strength (*Ramos et al., 1979*), generally to the detriment of uniform elongation. Grain refinement strengthens the material, with a larger impact on yield strength than tensile strength (*Chang and Preban, 1985*), generally at the expense of uniform deformation, but not always (*Calcagnotto et al., 2010a*). Martensite carbon content has a more limited influence on the plastic behavior than the

latter two parameters (*Davies, 1978*). Increasing carbon content generally increases strength and decreases uniform deformation (*Ramos et al., 1979; Speich and Miller, 1979*). However, in (*Pierman et al., 2014*), an increase in carbon content increases uniform deformation at low martensite volume fraction (of around 15% to 30%). An overview on microstructure evolution during processing and on the link with mechanical properties in Dual-Phase steels can be found in (*Tasan et al., 2015*).

In contrast, much less studies have addressed the role of particle morphology on the elastoplastic behavior of Dual-Phase steels, even though significant effects have been demonstrated. For instance, martensite banding is now known to be detrimental to damage resistance (*Bag et al., 1999*). In rolled steels, *Sun and Pugh (2002)* measured a higher strength in the rolling direction and a lower total elongation in the case of a fibrous microstructure of an as-rolled steel, whereas *Sarwar and Priestner (1996)* observed both an increase in strength and ductility when increasing the aspect ratio of the grains. The increase in strength is confirmed by the work of *Zhang et al. (2016)* in the case of martensite alignment along the rolling direction. *Kim and Thomas (1981)* proposed the alternative heat treatment for Dual-Phase steels leading to the ‘Thomas fibers’ microstructure. Such an arrangement has been shown to recurrently have positive effects on the plastic behavior (*Das and Chattopadhyay, 2009; Tomita, 1990*).

In a recent study, *Pierman et al. (2014)* systematically investigated the relative contributions of martensite volume fraction, carbon content and second phase morphology in model Dual-Phase steels. They found that, at high martensite volume fraction (60%), steels with a ‘Thomas fibers’ microstructure present lower strength but higher ductility than Dual-Phase steels with equiaxed microstructure at the same volume fraction and martensite carbon content.

2.2.2 Computational results about the plasticity of Dual-Phase steels

A micromechanical approach taking into account explicitly the composite character of Dual-Phase steels is useful to provide quantitative information about the influence of the different microstructural features on the effective mechanical behavior. Semi-analytical homogenization models based on

Eshelby's isolated inclusion solution have been repeatedly used to predict the properties of Dual-Phase steels. For instance, *Delincé et al. (2007)*, *Mazinani and Poole (2007)* and *Pierman et al. (2014)* have shown, among other things, that accounting for martensite plastic behavior is essential to accurately predict the overall mechanical response. However, mean field models provide only average mechanical field values. For detailed predictions of the local stress and strain fields, direct Finite Element (FE) analysis on representative microstructures constitutes the most reliable approach. This requires a simplification of the microstructure, an example of which is shown for a Dual-Phase steel in Figure 2.9, with its corresponding unit cell (*Lai et al., 2016*).

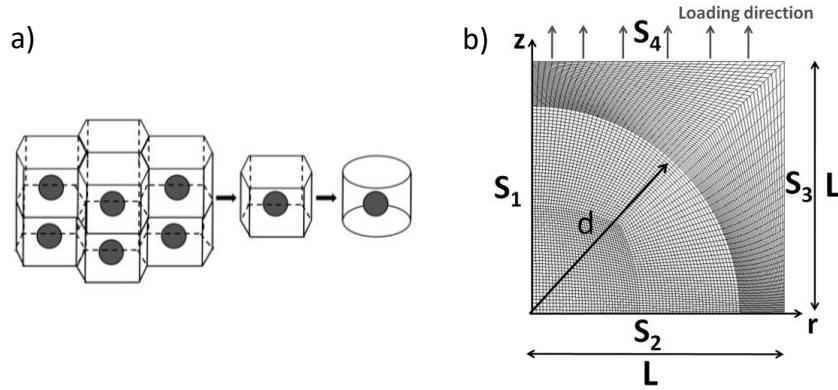


Figure 2.9 a) Representation of a simplified Dual-Phase steel microstructure with a ferritic matrix and spherical martensite particle and, b) corresponding periodic axisymmetric unit cell (images taken from (*Lai et al., 2016*)).

FE calculations can be performed on simple unit cells such as in (*Delannay et al., 2006; Lai et al., 2016; Mahnken et al., 2009*) assuming a periodic microstructure. FE simulations on random Representative Volume Elements (RVEs) containing many particles can be used to better account for microstructure complexity. Realistic 2D RVEs can be directly built based on SEM micrographs to capture the distribution of martensite as well as morphology variation (*Abid et al., 2015; Choi et al., 2009*), but generally tend to underestimate the strength. This can be addressed by considering 3D RVEs (*Amirmaleki et al., 2016; Bong et al., 2017; Paul and Kumar, 2012*) at the expense of a larger computational cost. Examples of generated 3D RVEs for Dual-Phase steels are given in Figure 2.10.

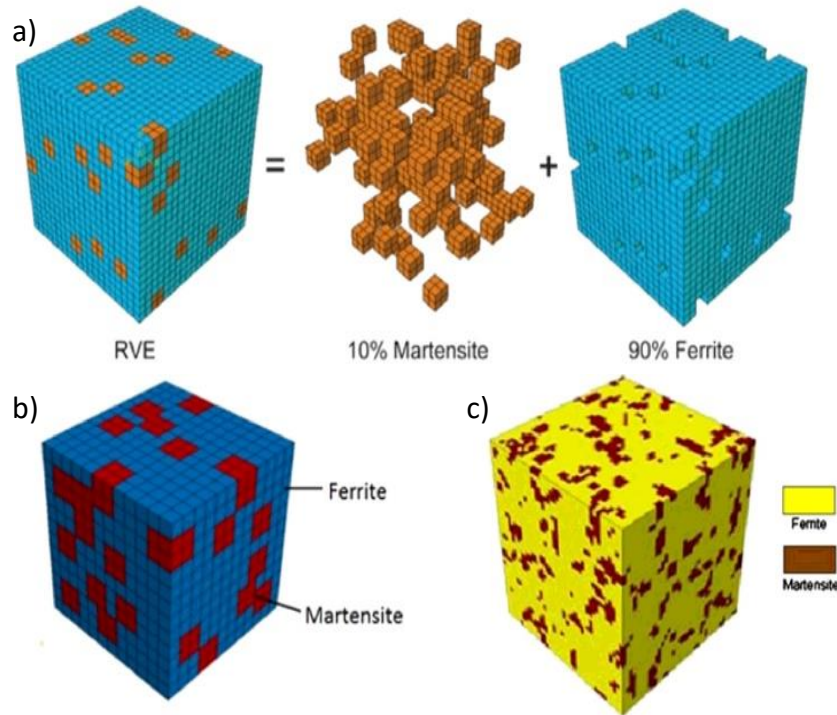


Figure 2.10 Examples of generated 3D RVEs corresponding to real Dual-Phase steels from: a) (Uthaisangsuk et al., 2011), b) (Paul, 2013), and c) (Ramazani et al., 2013b) (images taken from (Amirmaleki et al., 2016)).

By fully resolving the local stress and strain fields in the microstructure, these simulations can provide detailed insight in the process of plastic localization and damage evolution, see (de Geus et al., 2015; Kadkhodapour et al., 2011b; Sun et al., 2009; Tasan et al., 2014; Zarei et al., 2016). For instance, high stress concentrations near martensite islands can lead to interfacial debonding or fracture of the hard phase; high local stress triaxiality favors void growth; and local imperfections can be the precursors of instability in the form of shear bands (see Section 2.3).

Only few of these computational studies addressed the role of second phase morphology. Ramazani et al. (2012) used FE simulations on 2D RVEs to investigate the effect of martensite bands on the effective properties of Dual-Phase steels. They found that decreasing the aspect ratio of the bands leads to larger uniform elongation. Pagenkopf et al. (2016) considered equiaxed, elongated and flattened martensite in 3D RVEs loaded in directions

parallel and perpendicular to the elongated particles, and concluded that the elongated and the flat martensite morphology lead to a higher strength compared to spherical particles. *Abid et al. (2017)* considered 2D RVEs with equiaxed and elongated martensite particles, predicting an increase of strength and ductility with the elongated morphology.

The main disadvantage of full-field simulations on realistic microstructures is the large computational cost, especially for 3D simulations. Also, such simulations, in general, do not allow a systematic investigation of the effect of single parameters (such as individual particle orientation) as these are coupled to one another, depending on the location of the particle in the RVE. As an alternative, an original two-step approach has been proposed by *Ognedal et al. (2014)* in the context of a study of void nucleation and growth in mineral filled PVC. In this approach, simplified unit cells were assembled to represent a staggered array of spherical particles, reducing the computational cost compared to an RVE. This approach has been recently extended by *Hatami et al. (2017)* for application to multiphase TRIP steels. A similar approach will be adopted in Chapter 4 to model Dual-Phase steels with elongated and flat martensite. The use of elementary cells has proven successful in the study of Dual-Phase steels (*Al-Abbasi and Nemes, 2003; Al-Abbasi and Nemes, 2007; Hüper et al., 1999; Lai et al., 2015*).

2.3 Damage mechanisms in Dual-Phase steels

Ductile fracture can occur through plastic localization by necking and shear banding, or through a process of microvoid nucleation, growth and coalescence. The focus is put in this work on the second mechanism as it is the main failure mode in Dual-Phase steels at room temperature. Figure 2.11 illustrates schematically the several stages of the damage process. Following microvoids nucleation at critical spots in the microstructure, the voids grow as a result of plastic deformation. The void growth mechanism continues steadily up to a point at which a localization of plasticity occurs within the ligament between two or more neighboring voids. This plastic localization is called the onset of coalescence, the coalescence being the merging of these voids. Once several pairs of voids have coalesced, a crack forms, leading

eventually to its catastrophic failure by propagation through the entire specimen or structural component.

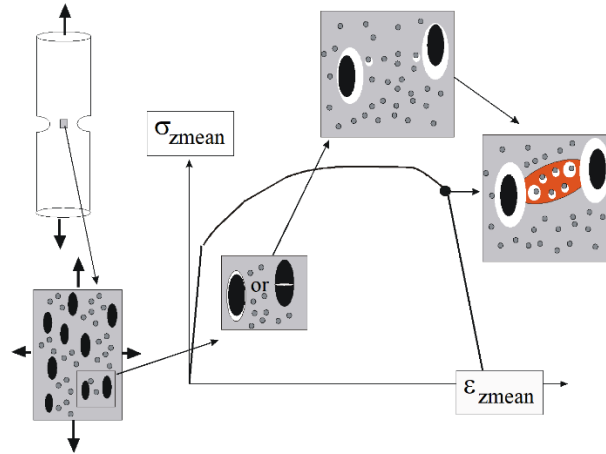


Figure 2.11 Schematics representing the damage process by microvoid nucleation, growth and coalescence in relation to the macroscopic loading. (image taken from (Pineau and Pardoën, 2007))

Each stage of the process has been visualized experimentally. The need to understand and accurately predict this three-stage process has led to numerous studies in the past decades for many ductile metals, Dual-Phase steels included. The damage behavior depends on the geometrical distribution of voids, stemming from the heterogeneous distribution of the weak spots, and on the local loading conditions: stress triaxiality, stress level, loading direction, Lode angle parameter... Each stage is presented in this section, with a particular focus on Dual-Phase steels.

2.3.1 Experimental evidences of damage in Dual-Phase steels

a) Void nucleation

Void nucleation starts before the ultimate tensile strength is reached (Asik *et al.*, 2019). Detailed experimental studies have shown that microvoid nucleation in Dual-Phase steels generally takes place either due to ferrite-martensite interface decohesion (rather at low martensite volume fraction), or by hard particle brittle fracture (rather at high martensite volume fraction) (Asik *et al.*, 2019; Ghadbeigi *et al.*, 2010; Lai *et al.*, 2015; Tasan *et al.*, 2009) (or more rarely, at inclusions (Asik *et al.*, 2019)). An

example of each two cases is shown in Figure 2.12 for ‘Thomas fibers’ Dual-Phase steels. A particular subcase of interface decohesion that frequently occurs is microvoid nucleation taking place between two closely spaced particles (*Avramovic-Cingara et al., 2009a; Avramovic-Cingara et al., 2009b; Sarwar and Priestner, 1996; Steinbrunner et al., 1988; Su and Gurland, 1987*) as deformation localizes and the stress concentrations reach high values at such spots.

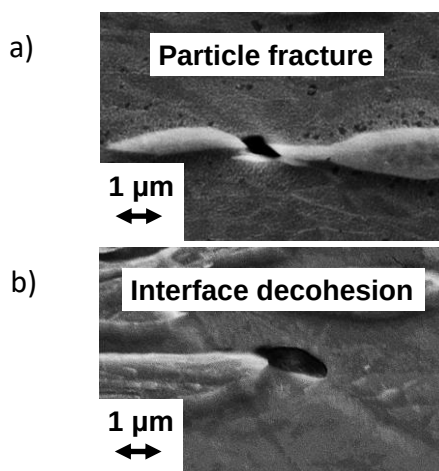


Figure 2.12 a) Void nucleated by particle fracture and b) void nucleated by interface decohesion in ‘Thomas fibers’ Dual-Phase steels.

Which mechanism effectively takes place depends on microstructural parameters. Decohesion is the most broadly observed mechanism (*Avramovic-Cingara et al., 2009a and b; Gerbase et al., 1979; He et al., 1984; Kim and Thomas, 1981; Korzekwa et al., 1980; Landron et al., 2010; Marder, 1982; Sarwar and Priestner, 1996; Speich and Miller, 1979; Steinbrunner et al., 1988; Su and Gurland, 1987; Szewczyk and Gurland, 1982*). This is explained by the deformation mismatch between the two phases (*Sarosiek et al., 1984*). Voids nucleate preferentially at the interfaces on grain boundaries (*Su and Gurland, 1987; Saeidi et al., 2016*), on the interface perpendicular to the tensile axis (*Avramovic-Cingara, 2009a and b*) and on interfaces that are close to a neighboring phase.

Particle size distribution is a dominant microstructural feature controlling void nucleation. Voids nucleate with more ease and thus firstly on large

particles than on small particles, as the likelihood of containing internal defects (for particle fracture) or interfacial defects (for decohesion) that could lead to void nucleation increases gradually with particle size (*Dighe et al., 2002; Gurland, 1972; Hannard et al., 2016; Lewandowski et al., 1989*). Moreover, larger particles can house larger defects, more prone to initiate breaking.

Much more generally than in the case of a pronounced particle size distribution, void nucleation takes place from small to high strains (*Avramovic-Cingara et al., 2009a; Landron et al., 2010; Maire et al., 2008; Szewczyk and Gurland, 1982*), in a succession of many discrete events, as shown in Figure 2.13 for three Dual-Phase steels. Figure 2.13 moreover shows that nucleation behavior heavily depends on microstructure morphology as the three specimens have the same martensite volume fraction equal to 60%.

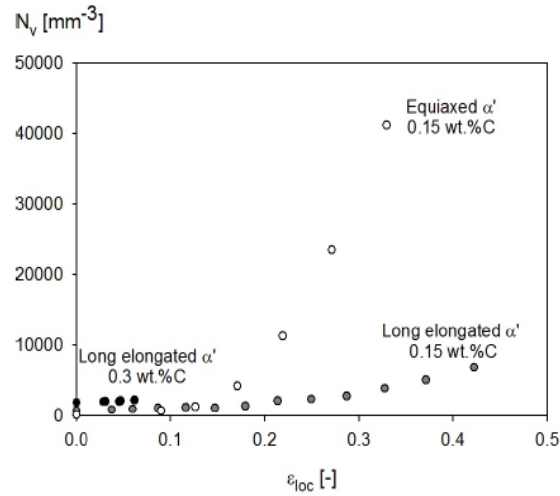


Figure 2.13 Number of voids as a function of local strain during a tensile test for three Dual-Phase steel microstructures. (image taken from (*Pierman, 2013*))

The discontinuous inhomogeneous process of void nucleation is in general heavily affected by microstructure and crystallography that dictate the local stress and strain fields that in turn dictate the load transfer in the particles (*Bugat et al., 1999*). For instance, (*Lassance et al., 2007*) have shown the

importance of the impact of particle shape and orientation with respect to loading on void nucleation.

b) Void growth and coalescence

As found for a myriad of materials (e.g. *Becker et al. 1988; Huber et al., 2005; Pardoen et al., 1998*), including steels (e.g. *Decamp et al., 1997; Jablovskov et al., 2001; Marini et al., 1985; Pineau, 1992*), fracture strain ε_f decreases exponentially with stress triaxiality T . The fracture strain is defined from the reduction of cross-section area as

$$\varepsilon_f = \ln\left(\frac{A_0}{A_f}\right) \quad 2.1$$

where A_0 and A_f are respectively the initial and final projected area of the fracture surface. And stress triaxiality is defined as

$$T = \frac{\sigma_h}{\sigma_{eq}} \quad 2.2$$

where σ_h is the hydrostatic stress and σ_{eq} is the von Mises equivalent stress, that are defined as

$$\sigma_h = \frac{\sigma_{11} + \sigma_{22} + \sigma_{33}}{3}, \quad 2.3$$

$$\sigma_{eq} = \sqrt{\frac{1}{2}[(\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2] + 3(\sigma_{12}^2 + \sigma_{23}^2 + \sigma_{31}^2)}. \quad 2.4$$

The stress triaxiality reflects the importance of the hydrostatic component compared to the deviatoric one. For instance, a stress triaxiality equal to -1/3 corresponds to uniaxial compression, 0 to pure shear, 0.6 to plane strain, 1/3 to uniaxial tensile loading, 2/3 to biaxial tensile loading and ∞ to a pure triaxial loading. In practice, stress triaxiality increases with increasing notch radius in the case of notched specimens. The limit case of a fine notch is the tip of a crack at which the stress triaxiality is equal to 3 or more in the plain strain regime.

Figure 2.14 illustrates schematically the exponential decay of fracture strain with stress triaxiality and its relationship with notch radius.

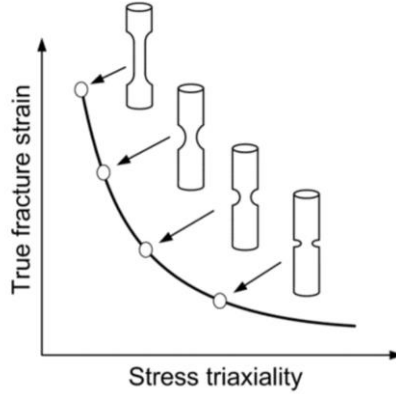


Figure 2.14 Variation of fracture strain as a function of stress triaxiality (image taken from (Han et al., 2016))

Void growth evolution has been observed and quantified in a large variety of materials. Using synchrotron microtomography, (Landron et al., 2011; Maire et al., 2008; Pierman, 2013) visualized in 3D voids in Dual-Phase steels. An example is shown in Figure 2.15. The number of voids is the highest in the necking region, where the highest stress triaxiality is found.

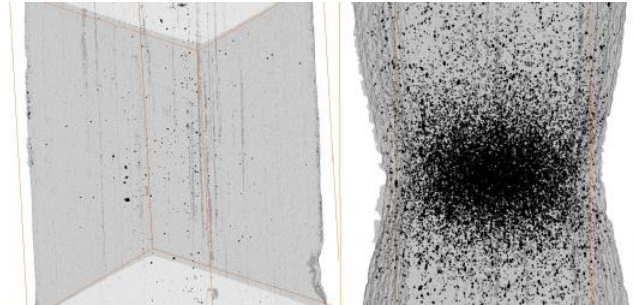


Figure 2.15 Example of a 3D representation of voids within a Dual-Phase steel sample before a tensile test and just before fracture. (image taken from (Maire et al., 2008))

To quantify void growth, (Landron et al., 2011) measured the average diameter of the N largest cavities with N equal to 20 and 50 and compared it to the average diameter of all voids and to the diameter of three voids chosen arbitrarily. Figure 2.16 shows their results for a Dual-Phase steel.

These results imply that void growth does not occur homogeneously and depends on void size. Additionally, it corroborates the fact that nucleation occurs discontinuously at different stages of deformation.

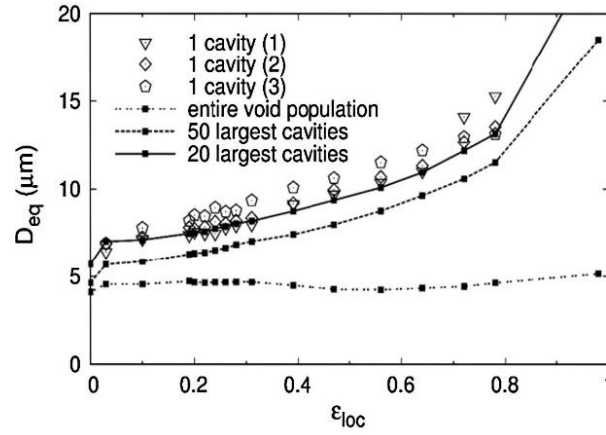


Figure 2.16 Variation of average void diameter for different numbers of examined voids as a function of local strain in a Dual-Phase steel. (image taken from (Landron et al., 2011))

This finding is supported by the work of (Pierman, 2013) that also proves that damage evolution depends on microstructure morphology, as shown in Figure 2.17.

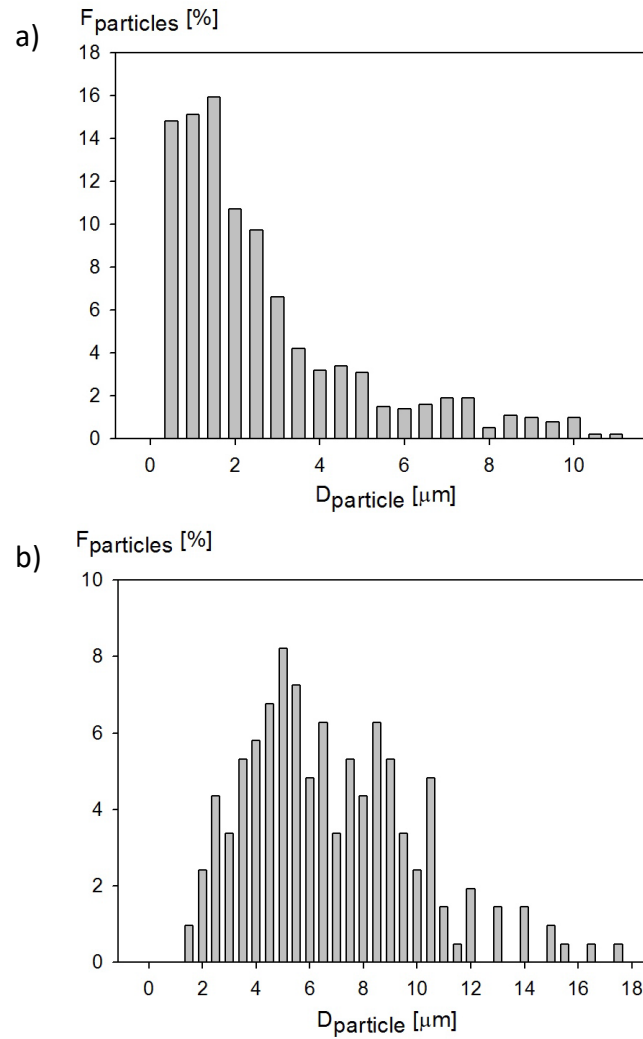


Figure 2.17 Voids distribution as a function of diameter in a) a 'Thomas fibers' Dual-Phase steel and b) an equiaxed Dual-Phase steel. (images taken from (Pierman, 2013))

c) Effect of microstructural parameters on damage behavior in Dual-Phase steels

Increasing the **martensite volume fraction** is generally accompanied by a decrease in fracture strain. This occurs because of an increase in void nucleation sites, a decrease of relative void spacing, and an increase of stress triaxiality in the more confined ferrite (Pierman *et al.*, 2014; Speich and Miller,

1979). Moreover, the strengthening of the steels when increasing martensite volume fraction also leads to a larger propensity for strain localization (*Hou et al., 2019*).

Decreasing martensite and ferrite **grain size** increases the post-necking deformation. The reason is that when decreasing grain size, interface decohesion becomes a more favorable nucleation mechanism, enabling thereby a more ductile deformation (*Chandiran et al., 2019*).

Decreasing the martensite **phase hardness** - by changing its composition, particularly its carbon content, or by tempering - increases fracture strain (*Azuma et al., 2012; Mazinani and Poole, 2007; Pierman et al., 2014; Speich and Miller, 1979; Tomota et al., 1977*). In Dual-Phase steels, martensite (and ferrite) hardness dictates the degree of incompatibility. A larger phase contrast leads to a higher incompatibility, thus higher interface stresses and a higher stress triaxiality. This in turn leads to an earlier decohesion and a faster void growth (*de Geus et al., 2016; Landron et al., 2010*). Reducing the phase contrast without sacrificing the plastic properties is possible by taking advantage of the composite behavior of the Dual-Phase steels, i.e. by tuning the behavior of the independent phases. (*Tian et al., 2020*), through micropillar compression experiments on either ferrite or martensite, measured very different phase properties between two Dual-Phase steels having the same ultimate tensile strength. They also found that an increased local strain hardening capability can delay global damage accumulation.

Regarding **morphology**, martensite interconnectivity (*Kunio et al., 1975*) and banded microstructures (*Avramovic-Cingara et al., 2009b; Ramazani et al., 2014; Tasan et al., 2010*) both negatively affect fracture strain. *Yan et al. (2015)* observed, using a coupling of microstructure-independent micro-strain pattern maps and pattern-independent microstructure maps, that martensite morphology and percolation mostly impact damage nucleation; *Asik et al. (2019)* observed that voids nucleate more inhomogeneously along the rolling direction, as cold-rolling induces a more pronounced band formation. Furthermore, related to the preceding, *Jafari et al. (2016)* found, using SEM and crystal-plasticity finite element modeling, that the homogeneity of the **martensite dispersion** highly affects the damage and strain localization in Dual-Phase steels. The strain distribution

inhomogeneity increases with the martensite dispersion inhomogeneity. The regions in which strain has localized coalesce together and form the macroscopic shear band. On the contrary, a more homogeneous martensite dispersion maintains a more homogeneous strain distribution, preventing thereby the appearance of early shear bands.

In addition to the microstructural parameters, the loading scenario and sample preparation also influence the local mechanical fields, and thus damage behavior. *Kahziz et al. (2016)* observed, using laminography, a different edge fracture behavior between a machined sample and a punched sample of the same Dual-Phase steel. In a machined sample, damage starts in the bulk of the sample, whereas it starts at the edge in a punched sample.

2.3.2 Damage models

Ductile fracture can be predicted using micromechanical models describing the nucleation, growth and coalescence of voids (*Pineau and Pardoën, 2007*). Numerous direct finite element calculations on elementary unit cells with periodic boundary conditions have been performed (*Koplik and Needleman, 1988; Pardoën and Hutchinson, 2000; Yerra et al., 2013*). Several semi-analytical models have also been proposed combining a growth model with a coalescence model, see for example (*Tekoglu and Pardoën, 2010*). Some models for damage prediction are presented in this section for the three stages of the damage process.

a) Void nucleation models

Several models have been proposed for the nucleation of voids, relying either on a critical stress, a critical strain or a critical energy, which values strongly depend on the case at hand. The critical stress in the presented stress-based models is denoted σ_c . A possible void nucleation criterion is the attainment of a critical stress by the maximum principal stress, in the bulk or at the interface whether the case is particle fracture or interface decohesion.

Among the commonly used models, two of them deserve mentioning. The Argon criterion (*Argon and Safoglu, 1975*), within a continuum approach of plasticity, highlights the importance of hydrostatic pressure on nucleation.

$$\sigma_c = \sigma_h + \sigma_{eq}. \quad 2.5$$

The Beremin group suggested a model that, by extending the Eshelby theory to plasticity, also considers the heterogeneities in deformation between matrix and particle (*Beremin, 1981*). Particle shape is accounted for through the use of a shape parameter k .

$$\sigma_c = \sigma_I + k(\sigma_{eq} - \sigma_0) \quad 2.6$$

where σ_I is the maximum principal stress, σ_{eq} is the average equivalent von Mises stress and σ_0 is the matrix yield stress.

The statistical aspects of void nucleation can be taken into account using the Weibull approach. For instance, (*Hannard et al., 2016*) considered particles in Al alloys as brittle volumes, whose breaking occurs when the maximum principal stress attains a critical stress associated to a specific family or size of particle. Then, the probability of fracture P for a given local maximum principal stress σ_I and particle volume V is expressed as

$$P(\sigma_I) = 1 - \exp \left[-\frac{V}{V_0} \left(\frac{\sigma}{\sigma_u} \right)^m \right] \quad 2.7$$

where V_0 is the reference particle volume and where σ_u and m are the distribution parameters. The use of a Weibull distribution also works for interface decohesion (*Babout et al., 2004*).

b) Void growth model

The very first models about void growth involved a single isolated void in an infinite perfectly plastic material. For example, (*McClintock, 1968*) expressed the growth of a cylindrical void in the case of an axisymmetric loading. Rice and Tracey (*Rice and Tracey, 1969*) expressed the more realistic case of a spherical void in an infinite medium with axisymmetric loading.

$$\frac{dR}{R} = \alpha \exp \left(\frac{3}{2} T \right) d\bar{\varepsilon}_{eq} \quad 2.8$$

where R is the void radius, α is equal to 0.283 as first proposed in (*Rice and Tracey, 1969*) or to 0.427 in (*Huang, 1991*), and $d\bar{\varepsilon}_{eq}$ is the equivalent plastic strain increment.

In order to assess the impact of void growth on the mechanical behavior, (Gurson, 1977) derived the expression of the yield surface for a J_2 material containing spherical or cylindrical voids with volume fraction f . The Gurson model has been revisited numerous times and various extensions are able to account for microstructural or material characteristics such as void size (Wen *et al.*, 2005), void shape (e.g. Gologanu *et al.* 1993), and many others. An extensively used extension of the Gurson model is the Gurson-Tvergaard-Needleman (GTN) model (Tvergaard and Needleman, 1984). In this framework, and applied to Dual-Phase steels, (Balan *et al.*, 2015) recently proposed a void nucleation law and (Santos *et al.*, 2019) identified the parameters for the GTN model based on an experimental analysis.

Similarly to stress triaxiality, the dimensionless Lode angle parameter $\bar{\theta}$, ranging from -1 to 1, has a significant impact on void growth as shown for example by (Bai and Wierzbicki, 2008). It is defined as (Beese and Mohr, 2011)

$$\bar{\theta} = 1 - \frac{2}{\pi} \arccos \left(\frac{3\sqrt{3}}{2} \frac{J_3}{\sqrt{J_2^3}} \right) \quad 2.9$$

where J_2 is the second invariant of the deviatoric Cauchy stress tensor $\mathbf{s}$:

$$J_2 = \frac{1}{2} \mathbf{s} \cdot \mathbf{s} = \frac{1}{6} [(\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2] + \sigma_{12}^2 + \sigma_{23}^2 + \sigma_{31}^2, \quad 2.10$$

and J_3 is the normalized third invariant of the deviatoric Cauchy stress tensor

$$J_3 = \det(\mathbf{s}) = \frac{1}{3} s_{ij} s_{jk} s_{ki} = \begin{vmatrix} \sigma_{11} - \frac{I_1}{3} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} - \frac{I_1}{3} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} - \frac{I_1}{3} \end{vmatrix} \quad 2.11$$

where I_1 is the first stress invariant equal to the trace of the stress tensor

$$I_1 = \sigma_{11} + \sigma_{22} + \sigma_{33}. \quad 2.12$$

The Lode angle parameter describes the loading mode. Characteristic values for an intuitive interpretation of the Lode angle parameter, which gives an information on the magnitude of the second principal stress σ_{II} with respect to the maximum and minimum principal stresses σ_I and σ_{III} , are

- $\bar{\theta} = -1$ corresponding to $\sigma_{II} = \sigma_I$. This occurs in the case of an equibiaxial tension or uniaxial compression.
- $\bar{\theta} = 0$ corresponding to $\sigma_{II} = \frac{\sigma_I + \sigma_{III}}{2}$. This occurs in the case of pure shear.
- $\bar{\theta} = 1$ corresponding to $\sigma_{II} = \sigma_{III}$. This occurs in the case of uniaxial tension.

c) Void coalescence models

The simplest model to predict fracture strain is the use of a critical void radius without taking the onset of coalescence and coalescence explicitly into account. A simple geometrical criterion for the onset of void coalescence has been proposed by (*Brown and Embury, 1973*) for spherical voids at first. Following this criterion, the point of onset of coalescence is reached when a connection between two neighboring voids through two 45° microshear bands with respect to the main loading direction becomes possible, as shown in Figure 2.18. The criterion can be generalized for any pair of ellipsoidal voids (*Pardoen et al., 1998*) and is expressed as

$$\chi\sqrt{1+W^2} = 1 \quad 2.13$$

where χ is the current relative void spacing and W the current void aspect ratio. The criterion proved useful on several occasions, leading to acceptable predictions (*Koplik and Needleman, 1988; Pineau and Pardoen, 2007*).

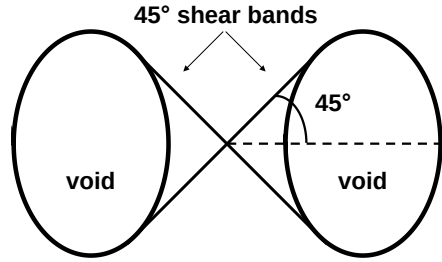


Figure 2.18 Brown and Embury geometrical criterion: the point of onset of coalescence is reached when a connection between two voids through two 45° shear bands becomes possible.

Based on the same parameter of relative distance, a famous and more complex criterion for the onset of coalescence that deserves mentioning is the Thomason criterion (*Thomason, 1985*). Following this criterion, the coalescence occurs when

$$\frac{\sigma_n}{\sigma_0} = (1 - \chi^2) \left[\alpha \left(\frac{1 - \chi}{\chi W} \right)^2 + 1.24 \frac{1}{\sqrt{\chi}} \right] \quad 2.14$$

where σ_n is the stress normal to the localization plane and where α depends on the strain hardening exponent through $\alpha(n) = 0.1 + 0.217n + 4.83n^2$, see (*Pardoen and Hutchinson, 2000*).

d) Limitation of classical damage models to periodic and homogeneous void distributions

Nevertheless, all preceding approaches suffer from an important limitation. Those methods lack to take explicitly into account the effect of microstructure once the nucleation sites are determined. Quite often, the models are even periodic. Moreover, and perhaps more importantly, they do not account for the statistical aspects related to microstructural heterogeneities. A way to overcome this is the use of a cellular automaton approach as performed in (*Hannard et al., 2016*).

2.4 Resistance to crack propagation

2.4.1 Generalities

The fracture toughness is generally quantified by the value of the stress intensity factor at cracking initiation in mode I , K_{IC} , or alternatively by the energy release rate at cracking initiation in mode I , G_{IC} , using the Linear Elastic Fracture Mechanics (LEFM) framework. In the event of a plasticity zone size larger than a fraction of the crack length, LEFM assumptions are not met and the Non Linear Fracture Mechanics framework must be used. The critical value of the J integral defined by *Rice (1968)* at cracking initiation in mode I , J_{IC} , is preferred. The fracture toughness is usually defined as the plane strain value required for specimens much thicker than the plastic zone size.

The resistance to ductile tearing (after initiation) is not an intrinsic material parameter. Indeed, the work dissipated in the plastic zone varies with crack advance, which leads to the JR curves (e.g. *Gao et al, 1998; Lam et al., 2003*). In addition, cracking resistance depends on loading conditions and geometry. This is called the constraint effect and geometry dependence of the JR curves, see Figure 2.19 (*Zhu and Jang, 2001*).

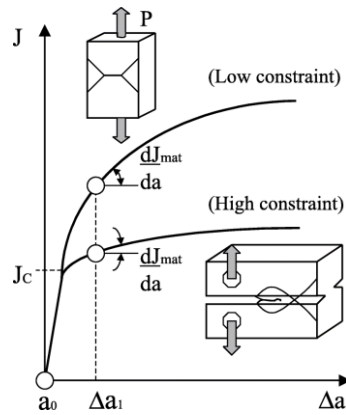


Figure 2.19 Tearing resistance depends on extrinsic parameters such as geometry, dimensions and loading conditions. (image taken from (Zhu and Jang, 2001))

The methods and sample dimensions for the measurement of K_{Ic} , G_{Ic} and J_{Ic} have been standardized. However, the standards impose a minimum thickness to generate plane strain results that is not suitable if one wishes to measure the resistance to the propagation of a crack in thin sheets. As sheet thickness decreases, the stress state evolves from plane strain to plane stress. In the near plane stress regime, the level of stress triaxiality is much lower than in plane strain, allowing larger strains in the fracture process zone (FPZ). Hence, the fracture toughness is higher. Furthermore, necking can develop near the crack, bringing an additional contribution to the fracture toughness (Pardoen *et al.*, 2004). Figure 2.20 illustrates schematically the evolution of fracture toughness with specimen thickness.

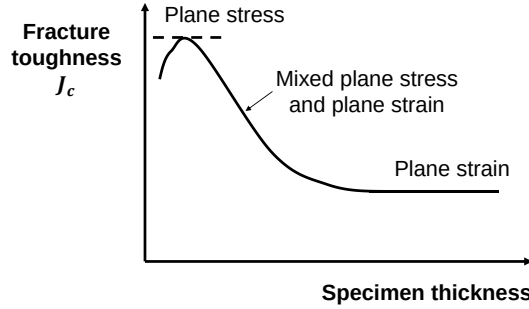


Figure 2.20 Evolution of fracture toughness with specimen thickness.

The Essential Work of Fracture (EWF) method, proposed by *Cotterell and Reddel (1977)* is an appropriate tool for the quantification of the crack propagation resistance in thin sheets of ductile materials.

2.4.2 The Essential Work of Fracture (EWF) method

Two zones can be distinguished in front of the crack tip: (i) a fracture process zone (FPZ) in which fracture occurs and (ii) a diffuse plastic zone (DPZ) in which plasticity occurs without material separation. For all ductile materials, the DPZ size is large compared to the FPZ size. The total work of fracture, W_f , may be written as the sum of the plastic work spent in the DPZ written W_p and of the work spent in the FPZ written W_e :

$$W_f = W_e + W_p \quad 2.15$$

Double-Edge Notched Tension (DENT) specimens are used for their symmetry and confinement of the plastic zone within the sample between the notches. Figure 2.21 shows a schematic of a DENT specimen, illustrating the locations of the FPZ and DPZ and their approximate relationship in terms of size.

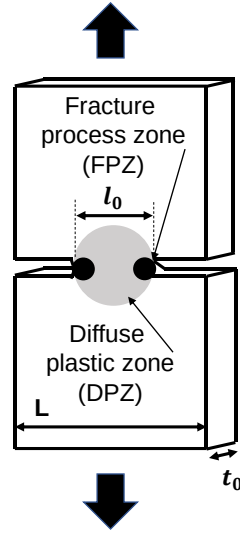


Figure 2.21 Schematic of DENT sample and illustration of the diffuse plastic zone (DPZ) and fracture process zone (FPZ). (inspired from (Martin et al., 2012))

The EWF method is based on dimensional considerations: (i) the volume of the DPZ and thus W_p is proportional to $t_0 l_0^2$, l_0 being the initial length of the ligament and t_0 the initial thickness of the sheet, as illustrated in Figure 2.21, while (ii) the volume of the FPZ and thus W_e is proportional to the part of material in which the crack propagates, which is proportional to $t_0 l_0$ as the crack propagates along the ligament. An assumption of the EWF method is thus the proportionality between plastic work and plastic zone size. This assumption is correct as long as the stress state remains in plane stress.

$$W_p \propto t_0 l_0^2 \quad 2.16$$

$$W_e \propto t_0 l_0 \quad 2.17$$

From these dimensional considerations, the total work of fracture W_f can be expressed as

$$W_f = l_0 t_0 w_e + \alpha l_0^2 t_0 w_p \quad 2.18$$

where w_e is the work spent per unit area of cracking within the FPZ and is called the essential work of fracture; w_p is the average plastic work spent in the DPZ per unit volume and α is a factor characterizing the shape of the DPZ.

The specific work of fracture w_f is obtained dividing W_f by the initial ligament area $l_0 t_0$, giving

$$w_f = \frac{W_f}{l_0 t_0} = w_e + \alpha l_0 w_p. \quad 2.19$$

In practice, DENT samples with various ligament lengths are deformed in tension until fracture. The measurement of the total energy for the separation of each sample provides W_f at a given l_0 and is computed as

$$W_f = \int F du \quad 2.20$$

where F is the force and u the displacement.

Based on Equation 2.19, since the method relies on the proportionality to l_0 , a linear regression on the values of w_f as a function of l_0 provides w_e as the constant term.

Figure 2.22 visually summarizes the complete methodology.

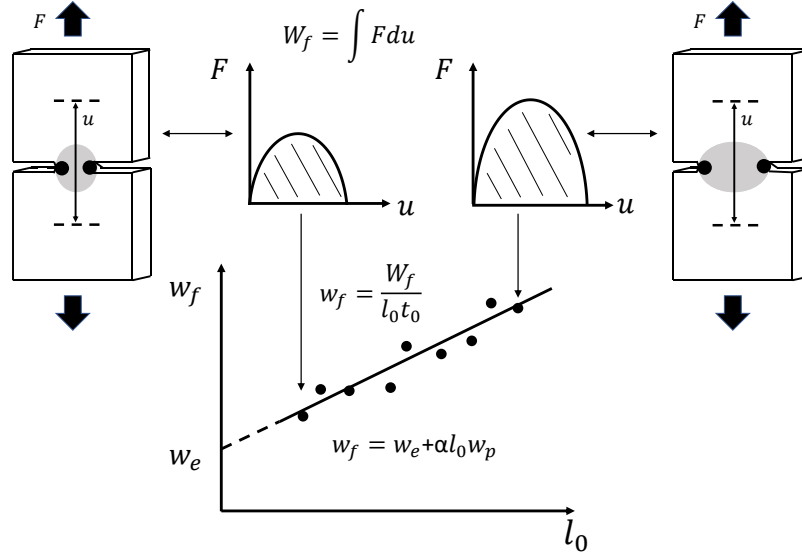


Figure 2.22 Illustration of the EWF method. (inspired from (Martin et al., 2012))

The EWF w_e characterizes the cracking resistance of the material, in opposition to w_p that depends on the geometry of the sample and on the way the loading is applied. The essential work of fracture w_e is not an intrinsic material property though as it depends on the sheet thickness. This is because when considering fracture of thin sheets, the material at the crack tip undergoes necking. This work of necking is included in the w_e term.

A further extension of the EWF method has been proposed to take into account the effect of sheet thickness. Indeed, w_e can be further separated in the contribution of an intrinsic work spent for damage and material separation taking place within the FPZ per unit area of crack advance, noted Γ_0 , and in the contribution of an average work spent in plastic deformation within the neck per unit area of crack advance, noted Γ_n (Cotterell et al., 2005; Pardoen et al., 2002; Pardoen et al., 2004; Pineau et Pardoen, 2007).

$$w_e = \Gamma_0 + \Gamma_n = \Gamma_0 + \beta t_0 w_n \quad 2.21$$

where w_n is an average work spent in necking at the crack tip per unit volume prior to material separation and β a factor characterizing the shape of the necking zone.

The work of necking may in some cases be the largest contribution to the cracking resistance (*Pardoen et al., 2004*). Besides, w_n depends on t_0 , hence cracking resistance depends on thickness. Finally, Γ_n and Γ_0 are not independent as a high damage resistance goes hand in hand with a higher thickness reduction in the neck before separation (*Pardoen et al., 2002; Pardoen et al., 2004*).

Combining Equations 2.19 and 2.21, the characteristic equation of the extended EWF method enabling to additionally separate the effect of sheet thickness becomes

$$w_f = \frac{W_f}{l_0 t_0} = \Gamma_0 + \beta t_0 w_n + \alpha l_0 w_p. \quad 2.22$$

By measuring the total work of fracture at different thicknesses and ligament lengths, the three contributions can be isolated. Figure 2.23 illustrates the procedure to extend the EWF method to take into account the effect of thickness.

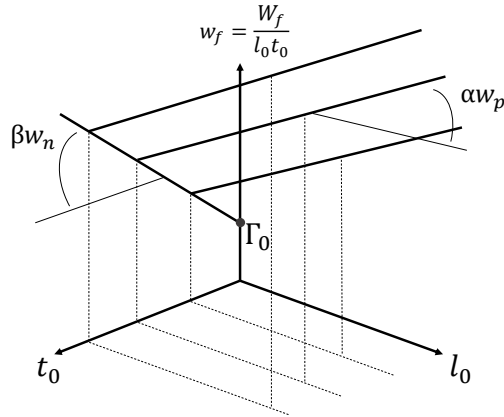


Figure 2.23 Extension of the EWF method to take into account the effect of sheet thickness. (inspired from (*Pardoen et al., 2004*))

The confinement of the plastic zone, the ligament in a state of plane stress and the complete yielding of the ligament before cracking initiation are

prerequisites for the method to be valid. The conditions of validity that must be met have been determined (e.g. *Cotterell and Reddel, 1977; Mai and Pilko, 1979; Mai and Cotterell, 1986; Mai and Powell, 1991; Mai, 1993; Marchal et al., 1997*) and translate into restrictions on the ligament length. The ligament length l_0 must be in an interval between a lower bound of admissible ligament length l_{min} and upper bound l_{max} .

l_{min} ensures the ligament to behave in plane stress and hence a cracking process under plane stress conditions. Below a critical length, the stress state changes as the FPZs at the two crack tips can interact leading to larger stress triaxialities. The stress state would then depend on ligament length (which would lead to different essential work of fracture values). The transition to the plane stress regime, as observed experimentally on numerous materials, takes place at a ligament length between three and five times the plate thickness t_0 . Also, by integrating from crack initiation to final separation, the work spent for initiation and the work spent in propagation are both accounted for in the integral. The work spent during the steady-state regime of propagation is of interest only, unlike the work spent during the transient regime related to the crack initiation process. Fortunately, the steady-state regime of propagation is reached when the crack advance is approximately equal to the sheet thickness. Hence, the transient regime only mildly affects the total work in case of a sufficiently long ligament length, which is another reason for setting a minimum l_0 .

l_{max} first ensures the confinement of the plastic zone avoiding it to reach the edges of the specimen by imposing l_0 smaller than $L/3$, L being the specimen width. l_{max} also ensures the complete yielding of the ligament before cracking initiation. A disconnection of the plastic zones at the crack tips would render the EWF method not applicable. Hence, l_0 must also be smaller than the size of the plane stress plastic zone r_p , which can be computed using linear elastic fracture mechanics and/or estimated as (*Marchal et al., 1997*)

$$r_p = \frac{1}{\pi} \left(\frac{K}{\sigma_y} \right)^2 \approx \frac{1}{\pi} \frac{E w_e}{\sigma_y^2}. \quad 2.23$$

Overall, the restrictions on l_0 are expressed as

$$l_{min} = (3 \dots 5)t_0 < l_0 < \min(L/3; r_p) = l_{max}. \quad 2.24$$

The method is suitable for any ductile material, therefore it is also much utilized in polymer science (e.g. *Bárány et al., 2010; Martinez et al., 2009*).

2.4.3 Fracture toughness of Dual-Phase steels

The fracture toughness of Dual-Phase steels has not been much investigated. Different authors have assessed the resistance to impacts of Dual-Phase steels through Charpy impact tests (*Bag et al., 1999; Bag et al., 2001; Calcagnotto et al., 2010a; Chao et al., 2007; Kang and Kwon, 1987*). This test provides an information about the dynamic fracture toughness. Regarding the quasi-static fracture toughness, *Bag et al. (2001)* performed three point bending tests on chevron-notched Dual-Phase steels specimens leading to a maximum K_{Ic} equal to $138 \text{ MPa}\sqrt{\text{m}}$ while *Bayram et al. (1999)* and *Alaneme (2011)* performed tensile tests on circumferentially notched specimens leading to a maximum K_{Ic} of $56.4 \text{ MPa}\sqrt{\text{m}}$. This corresponds to a J_{Ic} of around 90 kJ/m^2 and 15 kJ/m^2 , respectively, using the relationship

$$J_{Ic} = \frac{K_{Ic}^2}{E} \quad 2.25$$

where E is the Young's modulus.

The essential work of fracture method has been applied to a few Dual-Phase steels. *Casellas et al. (2016)* measured an essential work of fracture of 138 kJ/m^2 for a 1.4 mm thick Dual-Phase steel, *Gutiérrez et al. (2012)* a maximum value of 184 kJ/m^2 for a 1.5 mm thick sheet, *Lacroix et al. (2008)* a value around 50 kJ/m^2 for a 0.9 mm sheet and *Sahoo et al. (2018)* a value of 371 kJ/m^2 for a 1.53 mm sheet.

There is a large disparity in the latter values due to the difference in the steels properties and microstructure. All steels exhibit an equiaxed microstructure but (*Gutiérrez et al., 2012*) studied a Dual-Phase steel with a martensitic matrix, whereas the matrix was ferritic for the others. It moreover exhibited banding in the direction of rolling. A higher martensite volume fraction can

lead to a higher resistance to cracking partly because the crack is then forced to propagate through the interconnected hard phase (*Samei et al., 2019*). Furthermore, increasing martensite volume fraction for a given composition reduces the phase contrast, which delays damage appearance. The ultimate tensile strength ranged from 590 MPa for (*Sahoo et al., 2019*) to around 1000 MPa for (*Casellas et al., 2016*), the carbon content ranged from 0.09C for (*Sahoo et al., 2018*) to around 0.15C for the others.

However, seeing that the essential work of fracture is higher than the fracture toughness J_{Ic} for a given material due to the additional contributions and the different stress state, the values are seldom high, and are far from the astonishing values found by *Pierman (2013)* in some ‘Thomas fibers’ Dual-Phase steels (see Chapter 1).

Chapter 3

Materials, processing, microstructure characterization and elastoplastic properties

In this chapter, the as-received material is first described, together with the processing stages and the resulting microstructures. Basic mechanical properties are finally described.

3.1 As-received material

The different investigated microstructures all result from the same initial as-received ferrite-pearlite steel batch, supplied by ArcelorMittal Maizières Research SA. The chemical composition is given in Table 3.1.

C	Mn	Si	Cr	Al	P	S
0.045	2	0.3	0.4	0.028	0.013	0.0025

Table 3.1 Chemical composition (in wt.%) of the as-received steel grade.

12 mm thick and 120 mm wide plates were provided by ArcelorMittal, following a hot rolling after casting. Before receiving the slabs, they were also held during eight hours at 1200°C for a homogenized chemical composition and finally air-cooled. In order to study the mechanical behavior of thin sheets, the plates were hot-rolled again down to a thickness of 5 mm. This was performed by heating the plates at 1250°C in a muffle furnace for one hour, followed by rolling in one or two passes. A chemical or mechanical pickling step subsequently took place in order to get rid of the remaining oxide. In the case of a chemical pickling, the sheets were bathed for four hours (or more) in a pickle liquor, made of 500 ml of water, 800 ml of HCl and

200 ml of HF. A brush then helped in the removal of the oxide crust. In the case of a mechanical pickling, a sandblaster (Laborex) was used.

Thickness being a parameter of interest, samples with two different thicknesses have been processed, namely 1 mm and 2 mm thick sheets, by a successive cold rolling followed by thermal treatments. A plate thickness of 0.5 mm was additionally considered for essential work of fracture tests, see Chapter 5. The practical details of the thermal treatments will be articulated later in Section 3.2 .

3.2 Processing

In order to achieve predetermined microstructural values for the model microstructures, a trial and error study was first undertaken to fix the heat treatment parameters. To do so judiciously, information was first sought about the temperatures at which austenite and to a lesser extent martensite are formed. Indeed, the way to obtain a 'Thomas fibers' microstructure is a first step heating in the fully austenitic region above the austenite end temperature Ac_3 followed by a heating above the austenite start transformation temperature Ac_1 during the second step of the heat treatment, both steps ending with a water quenching to obtain martensite starting from the martensite start temperature M_s (see Figure 2.6 in Chapter 2).

The determination of the transformation temperatures relevant for ferrite-martensite Dual-Phase steels, namely Ac_1 , Ac_3 , M_s and M_f was carried out using phenomenological formulae based on composition (Andrews, 1965) and dilatometry. In addition, the temperature during the heat treatments was acquired using a thermocouple. More details are given in Appendix A.

3.2.1 Thermal treatments

Two distinct morphologies of the martensite will be studied, namely an equiaxed microstructure and a 'Thomas fibers' microstructure (Kim and

Thomas, 1981). Such microstructures result from two kinds of heat treatments (see Chapter 2).

- **'Thomas fibers' microstructures:** A two-step heat treatment is applied for the processing of 'Thomas fibers' microstructures. The first step aims at generating a fully martensitic microstructure. In all cases, the austenitization is performed at 1100°C for 17 minutes (time in furnace) in a muffle furnace and ends with water quenching. The second step is an intercritical annealing at a temperature depending on the desired martensite volume fraction. It is performed in a fluidized bed furnace bringing fast heating. The second step lasts for 7 minutes (time in furnace) and likewise ends with water quenching to ensure martensite formation. Dual-Phase steels with martensite volume fractions of either 19% or 36% were processed, for which the temperature of the second step was 760°C and 785°C, respectively.
- **Equiaxed microstructures:** A one-step heat treatment is applied for the processing of the equiaxed microstructures. The sheets are brought to a temperature between A_{c1} and A_{c3} depending on the desired martensite volume fraction. This is performed in the same fluidized bed furnace and was ended by water quenching. The step lasts for 7 minutes (time in furnace). For purposes of comparison with the 'Thomas fibers' microstructures, equiaxed Dual-Phase steels with the same two martensite volume fractions have been processed, namely 19% and 36%, for which the temperature of the step was 735°C and 785°C, respectively.

All microstructures are labeled according to their microstructure morphology. The name of the samples writes xVytz with x being either e or p for equiaxed or 'Thomas fibers', respectively, y being the martensite volume fraction in percent, and z being the sheet thickness in mm.

As a summary, Table 3.2 provides the times and temperatures of each step of the heat treatments for all investigated thin sheets microstructures.

	<i>Austenitization</i>	<i>Intercritical annealing</i>
eV19t1 & eV19t2	/	735°C – 7 min
eV36t1 & eV36t2	/	785°C – 7 min
pV19t1 & pV19t2	1100°C – 17 min	760°C – 7 min
pV36t1 & pV36t2	1100°C – 17 min	785°C – 7 min

Table 3.2 Temperature and duration of the applied thermal treatments, each step being ended by water quenching.

3.2.2 Frame for limiting warping during heat treatment

Thin sheets can warp during quenching due to the generation of internal stress gradients. This effect is enhanced when the area of the sheet increases, when the thickness decreases and when the temperature before quenching increases. In order to perform accurate essential work of fracture measurements, the warping of the plates should be avoided. In order to limit warping, an external frame has been designed and machined as shown in Figure 3.1. The sample is inserted in this frame made out of stainless steel to enable multiple uses. Furthermore, it was designed with the intention of limiting thermal inertia for the most homogeneous cooling possible implying that the mass of the frame must not be too large. Furthermore, the frame center is material free as it is the most critical zone, in which the ligament of the EWF samples is machined.

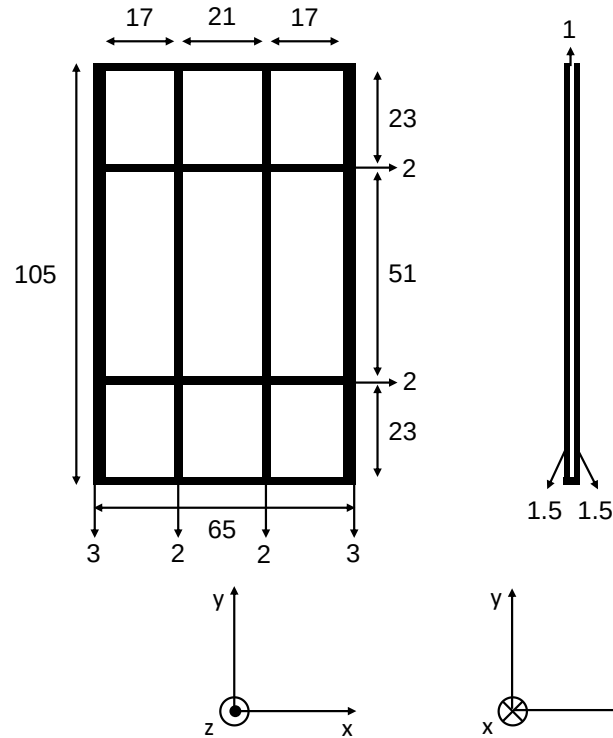


Figure 3.1 Dimensions of the external frame used for the processing of the 1 mm thick DENT samples.

3.3 Microstructure characterization

3.3.1 General procedure for microstructure characterization

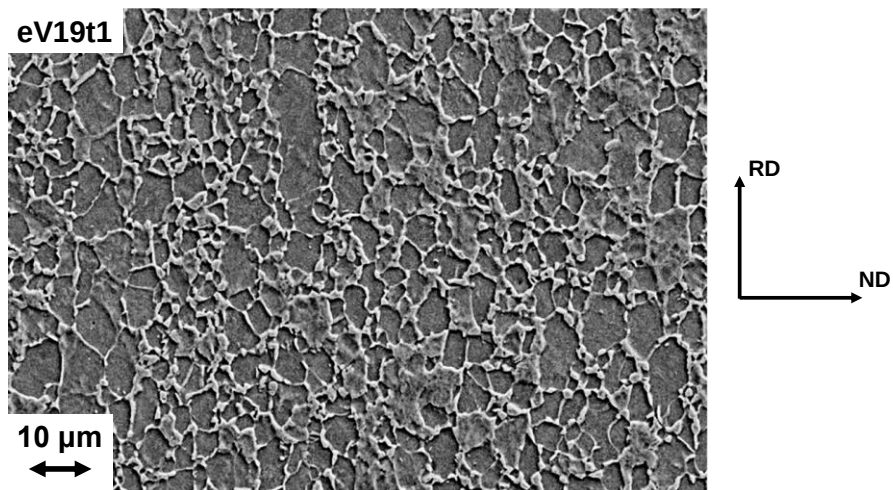
Samples were first prepared so as to be mounted for grinding and polishing. Samples were hot-mounted in a conductive resin. The mounted samples were subsequently mechanically ground with water lubricated SiC papers, with grits ranging from 320 at first to 1200 at last. Finally, the samples were mechanically polished using diamond paste. The size of the diamond particles decreased from 9 μm (or 6 μm) to 1 μm . Each individual grinding or polishing step took between three and fifteen minutes depending on microstructure and initial surface state. To check the quality of the polishing state, an optical microscope was used.

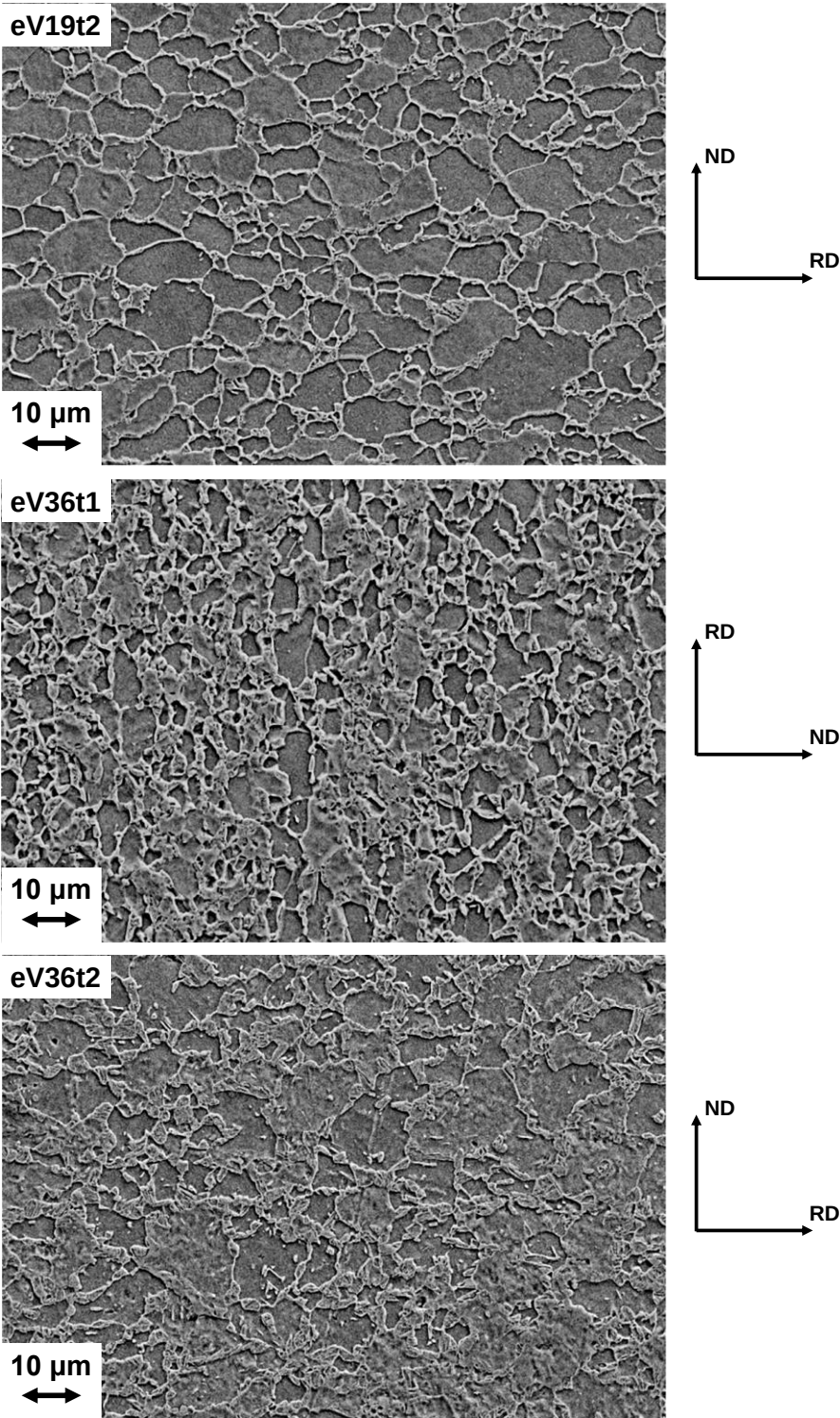
Prior to observation, the polished samples were etched by a Nital solution (2% of nitric acid and 98% of ethanol) in order to reveal the ferrite and martensite, as the Nital solution preferentially etches the carbon-poor phases. Etching duration was about 15 seconds.

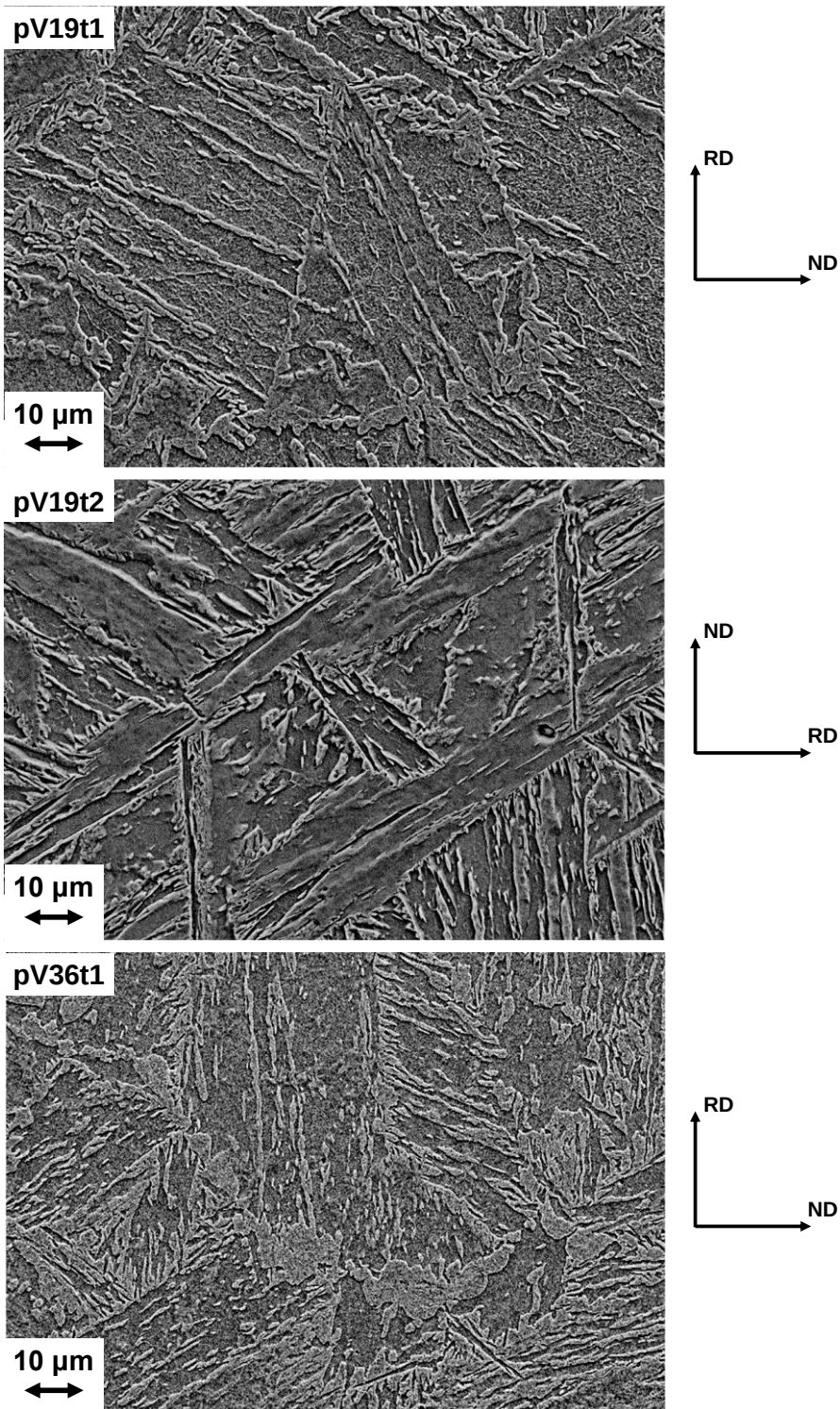
To characterize the Dual-Phase steels at the scale of the microstructure, a field emission gun scanning electron microscope was used. The latter was generally operated at 15 kV under secondary electrons mode.

3.3.2 Investigated microstructures

Figure 3.2 is a panel of SEM micrographs corresponding to each of the investigated microstructures.







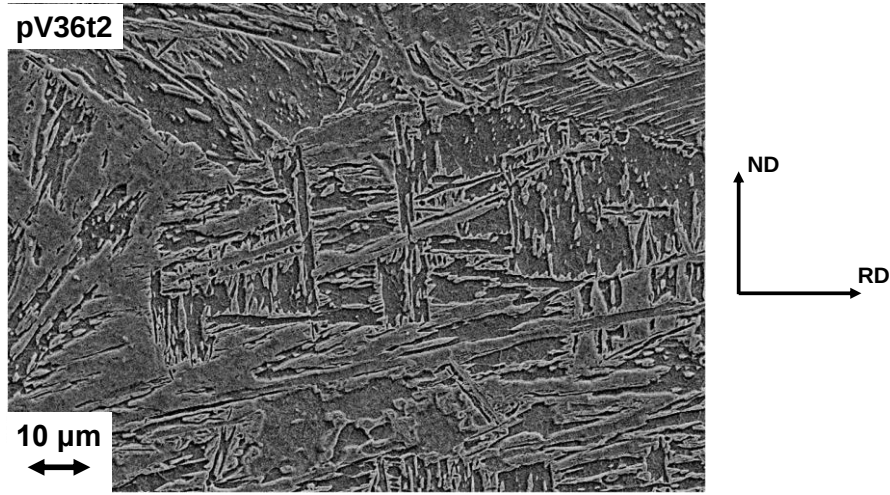


Figure 3.2 SEM micrographs of the 8 investigated microstructures.

Microstructural banding occurs in the equiaxed microstructures, as attested by the presence of fine-grained and coarse-grained bands. The grains in the coarse-grained bands are roughly twice as large as the grains in the fine-grained bands, which are of the order of 10 μm . This banding is probably the result of a banded microstructure in the initial ferrite-pearlite steel, due to chemical segregation.

The grain size in the equiaxed microstructures is slightly larger for the steels with 19% of martensite than with 36%. Furthermore, the 1 mm thick sheets exhibit a smaller grain size than the 2 mm thick sheets. The ferrite grain density is around 1.47 times higher in the case of the equiaxed microstructures with 19% of martensite and around 2.1 times higher in the case of the equiaxed microstructures with 36% of martensite (computed from Figure 3.2). The refinement is possibly due to a higher reduction of thickness during cold-rolling. To quantify the reduction of thickness, the reduction rate is used. It is defined as

$$\text{reduction rate} = \frac{t_0 - t_f}{t_0} \quad 3.1$$

where t_0 and t_f are the initial and the final thickness of the sheet, respectively.

As a hot-rolling from 12 mm down to 5 mm was performed for all microstructures, the cold-rolling reduction rate varies for 1 mm and 2 mm thick sheets. It is equal to 80% for 1 mm and to 60% for 2 mm. Of course, a higher reduction rate implies a higher dislocation and defect density. The initial microstructure before recrystallization is somewhat different, the density in grain nucleation sites for phase transformation is higher. In the case of the 'Thomas fibers' microstructure, the full austenitization erases the effects of cold-rolling on the dislocation density.

Figure 3.3 shows a micrograph of a pV36t1 steel at a lower magnification to illustrate the size of a martensite packet, which is of the order of 100 μm .

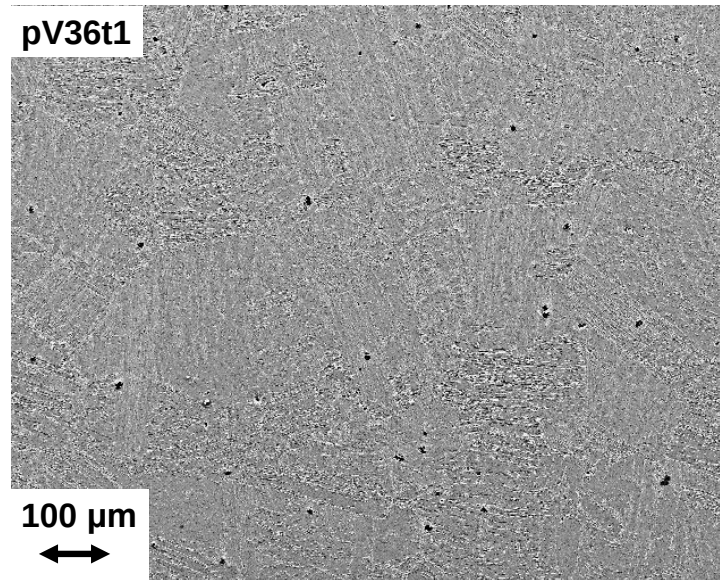


Figure 3.3 Illustration of the martensite packet size in a pV36t1 steel.

Table 3.3 provides the measured martensite volume fraction, estimated using the intercept method as well as the martensite carbon content $C_{\alpha'}$. The intercept method was performed on $113 \times 76 \mu\text{m}^2$ micrographs. On each micrograph, 6 lines were systematically drawn through the image in the same way; and 7 images were used per microstructure. The images were moreover taken sufficiently far away from each other to avoid the influence of local changes in martensite volume fraction. The measurements were performed by hand; even though the quantification is not precise, the intercept method

is very informative. The martensite carbon content $C_{\alpha'}$ is computed making the hypothesis that carbon finds itself entirely in martensite, such that

$$C_{\alpha'} = \frac{C_{tot}}{V_{\alpha'}} \quad 3.2$$

where C_{tot} is the total carbon content, and $V_{\alpha'}$ is the martensite volume fraction. Hence, for both morphologies, the martensite carbon content decreases when martensite volume fraction increases as the global composition remains constant.

	$V_{\alpha'}$	$C_{\alpha'}$
eV19t1	19	0.24
eV19t2	20	0.23
eV36t1	36	0.13
eV36t2	34	0.13
pV19t1	19	0.24
pV19t2	22	0.20
pV36t1	36	0.13
pV36t2	36	0.13

Table 3.3 Martensite volume fraction and martensite carbon content of the eight investigated microstructures.

For comparison reasons, the microstructures eV19t1, eV19t2, pV19t1 and pV19t2 are considered to contain 19% of martensite. Similarly, the microstructures eV36t1, eV36t2, pV36t1 and pV36t2 are considered to contain 36% of martensite.

3.3.3 3D microstructure characterization

Traditionally, a SEM micrograph is used to observe the microstructure of a material. However, a single 2D image is not sufficient for a quantitative description of the microstructure growing in the three directions of space. The effect of morphology on the mechanical behavior being of interest, it is key to be certain of the shape of the martensite particles. As a consequence, a 3D microstructure characterization was undertaken on a sample of a 10 mm thick sheet taken from (Pierman, 2013). It was performed by means

of a serial sectioning procedure, in which Vickers indents were used on the one hand for the positioning and on the other hand for the estimation of material thickness removal at each step. Indeed, knowing the geometry of the Vickers indent, the height of it, h , can be measured based on the indent's base projected area A_{proj} using Equations 3.3 and 3.4, whose parameters are relative to the schematic of the Vickers indent geometry shown in Figure 3.4.

$$A_{proj} = a^2 = \frac{d^2}{2} \quad 3.3$$

$$\tan(68^\circ) = \frac{a}{2h} \quad 3.4$$

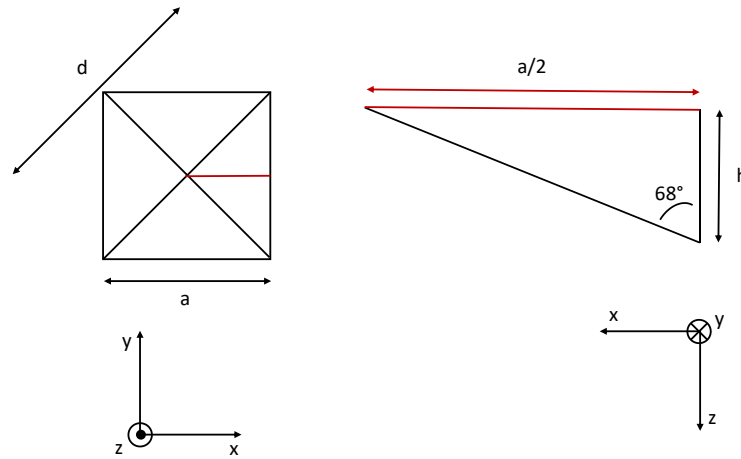


Figure 3.4 Geometry of the Vickers indent used for the 3D microstructure characterization.

The procedure revealed that the martensite particles of the 'Thomas fibers' microstructures are platelets. Even though heterogeneous, the estimated size of the platelets is around $1 \times 10 \times 20 \mu\text{m}^3$. A superposition of 2D images at two different angles as well as a corresponding schematic of the microstructure are available in Figure 3.5.

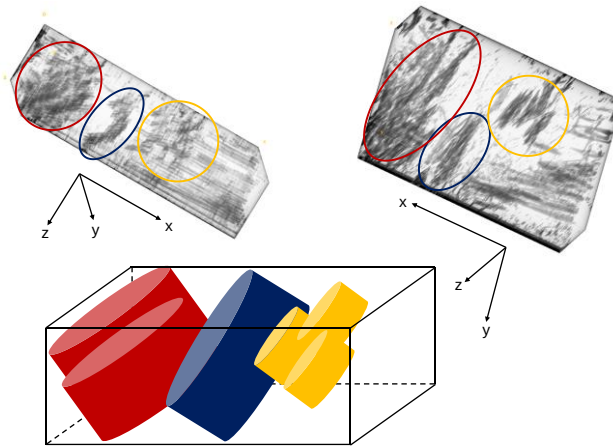


Figure 3.5 Superposition of 2D SEM micrographs at two different angles and schematic representation of the 3D microstructure. The colors of the martensite platelets on the schematic correspond to the encircled platelets in the superposition.

As a remark, the platelet-like morphology explains the ‘p’ used in the microstructure nomenclature for the ‘Thomas fibers’ microstructure.

3.4 Mechanical testing

3.4.1 Hardness tests

Micro-hardness measurements were performed. Using a Vickers tip, the load applied was 100 g for a dwell time of 10 s. For statistical accuracy, the measurement was performed ten times on each microstructure. Table 3.4 provides the micro-hardness values for the eight investigated steels.

	HV		HV
eV19t1	208 ± 3	eV19t2	197 ± 6
eV36t1	257 ± 11	eV36t2	255 ± 12
pV19t1	215 ± 15	pV19t2	224 ± 18
pV36t1	213 ± 13	pV36t2	248 ± 17

Table 3.4 Micro-hardness of the investigated steels.

3.4.2 Uniaxial tensile tests

For more complete information about the mechanical behavior, uniaxial tensile tests were conducted. The tests were performed at controlled room temperature and humidity of 23°C and 49%, respectively, under displacement control at constant crosshead velocity of 1 mm/min. The tensile loading was aligned with the rolling direction. An extensometer was used to measure strain with an initial gauge length at least equal to 20 mm.

3.4.3 Elastoplastic properties

Figure 3.6 shows the stress-strain curves of the investigated steels. Full lines represent a 1 mm thick specimen, dotted lines represent a 2 mm thick specimen.

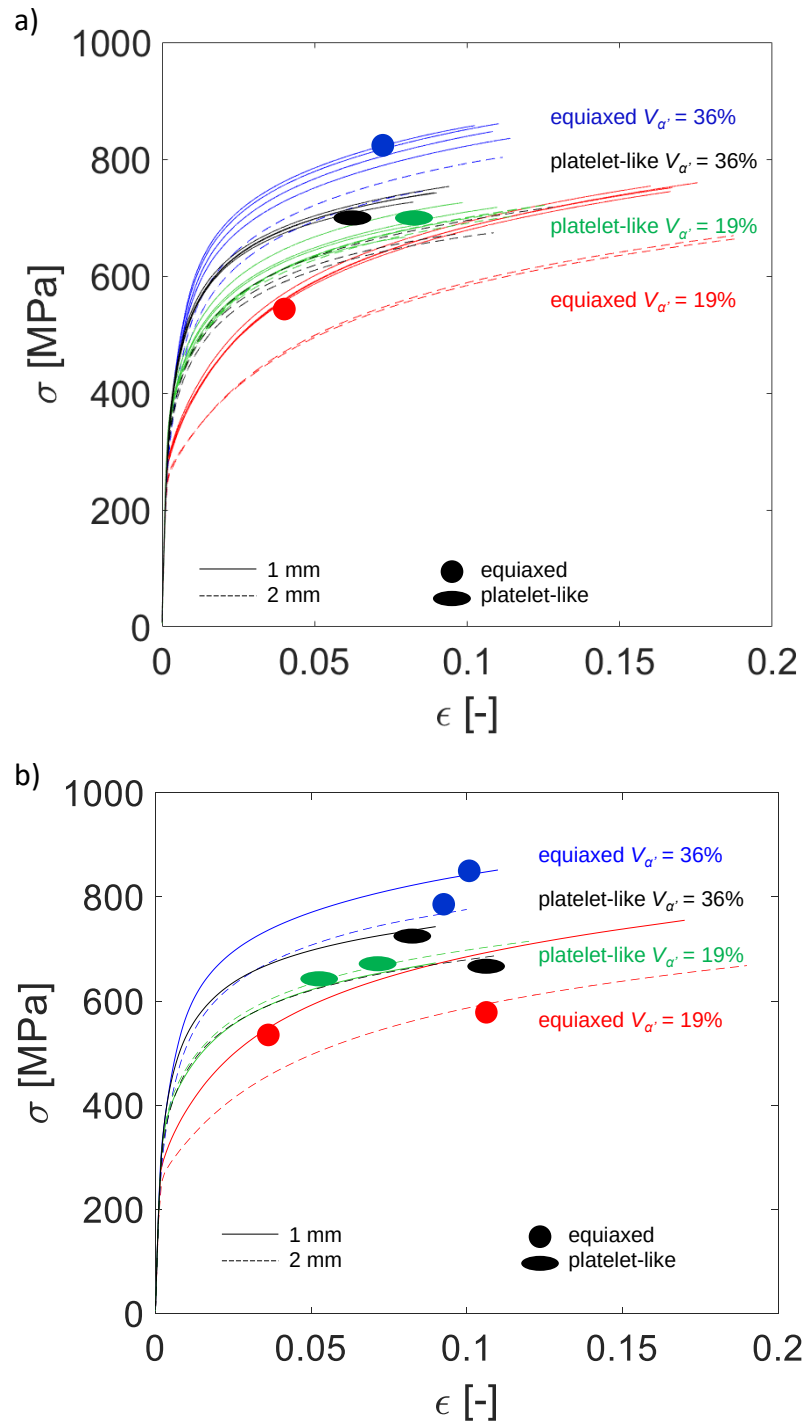


Figure 3.6 Experimental true stress-true strain curves of the investigated steels. a) All curves, b) mean curves.

The characteristic values of yield stress, σ_0 , true ultimate tensile strength, σ_u , and true strain at necking, ε_u , were determined in the following way. The yield stress is computed by taking the intersection of the experimental true stress-true strain curve with a straight line with a slope of 210 GPa starting at a true strain ε equal to 0.002. The onset of necking is taken at the maximum of the nominal stress-strain curves. The values are computed on each curve independently; the mean and standard deviation are taken afterwards. Finally, true fracture strain, ε_f , is measured post-mortem on the broken samples. It is quantified as defined in Section 2.3.1. Finally, the fracture stress, σ_f , is calculated as the ratio between the force at fracture F_f and the final projected area of the fracture surface A_f .

Table 3.5 summarizes the measured tensile properties and micro-hardness values for the eight investigated steels.

	σ_0 [MPa]	ε_u [-]	σ_u [MPa]	ε_f [-]	σ_f [MPa]
eV19t1	311 ± 4	0.17 ± 0.01	752 ± 6	0.765 ± 0.007	1497 ± 52
eV36t1	417 ± 9	0.11 ± 0.01	854 ± 13	0.88 ± 0.096	1953 ± 163
pV19t1	396 ± 10	0.09 ± 0.01	700 ± 27	1.04 ± 0.023	1765 ± 175
pV36t1	415 ± 7	0.09 ± 0.005	741 ± 11	1.06 ± 0.12	1984 ± 98
eV19t2	274 ± 4	0.19 ± 0.0002	666 ± 4	0.935 ± 0.025	1561 ± 37
eV36t2	367 ± 9	0.1 ± 0.02	775 ± 40	1.08 ± 0.156	2117 ± 235
pV19t2	385 ± 2.5	0.12 ± 0.005	716 ± 9	0.76 ± 0.04	1618 ± 137
pV36t2	369 ± 5	0.11 ± 0.02	688 ± 26	1.09 ± 0.09	1820 ± 110

Table 3.5 Tensile properties of the investigated steels.

The 1 mm thick sheets exhibit a higher strength than the 2 mm thick sheets at same martensite volume fraction. The hardening is possibly due to the refinement effect between the two thicknesses (see Section 3.3.2).

Finally, the smaller difference between 1 mm and 2 mm for the equiaxed Dual-Phase steels with 36% of martensite compared to the 19% of martensite is maybe due to the larger contribution of martensite volume fraction to hardness compared to grain size. The correspondence between tensile properties and hardness values is not systematic. The scatter in hardness is possibly related to the microstructural banding, as observed in Figure 3.2.

Chapter 4

Impact of second phase morphology and orientation on the plastic behavior of Dual-Phase steels

The general objective of Chapter 4 is to gain insight into the role of microstructure morphology on mechanical properties of Dual-Phase steels through computational modeling. More precisely, focus is put on microstructures similar to those considered by *Pierman (2013)* and *Pierman et al. (2014)*. Chapter 4 presents a systematic investigation of the effect of particle shape and orientation on both the effective plastic behavior and on the local mechanical fields of Dual-Phase steels. We developed a two-level homogenization approach involving FE calculations on unit cells, in the spirit of the approach of *Ognedal et al. (2014)*. The first level is the one of a pseudo-grain in which the martensite particles are all oriented in the same direction. The second level is the macroscopic level, consisting of a collection of grains with different particle orientations. The focus is on the impact of particle shape and orientation on the flow stress, uniform elongation, strength-ductility product and back stress. Local fields within pseudo-grains are investigated in terms of local stress triaxiality, maximum principal stress and plastic strain distribution, which control damage evolution. Although the focus is on Dual-Phase steels, some of the results are of interest for other classes of metallic alloys such as various α/β Ti alloys or metal matrix composites with short fibers.

Chapter 4 is organized as follows. The modeling approach and averaging procedure are described in Section 4.1 . Results of the parametric study are given in Section 4.2 , at both grain level (where all particles have the same orientation) and at macroscopic level (where grains with particles of different

orientations are considered). The model and its results are discussed in Section 4.3 , before concluding.

4.1 Model description

4.1.1 Two-level modeling approach

The modeling strategy is schematically represented in Figure 4.1. The microstructure of interest is made of martensitic particles with a platelet shape. In such microstructures resulting from the two-stage heat treatment explained in Chapter 2, the martensite particles forming within a specific parent martensite packet exhibit the same shape and orientation. Therefore, the microstructure is virtually decomposed into a set of “grains” made of matrix and of one specific particle shape and orientation relative to a macroscopic coordinate system.

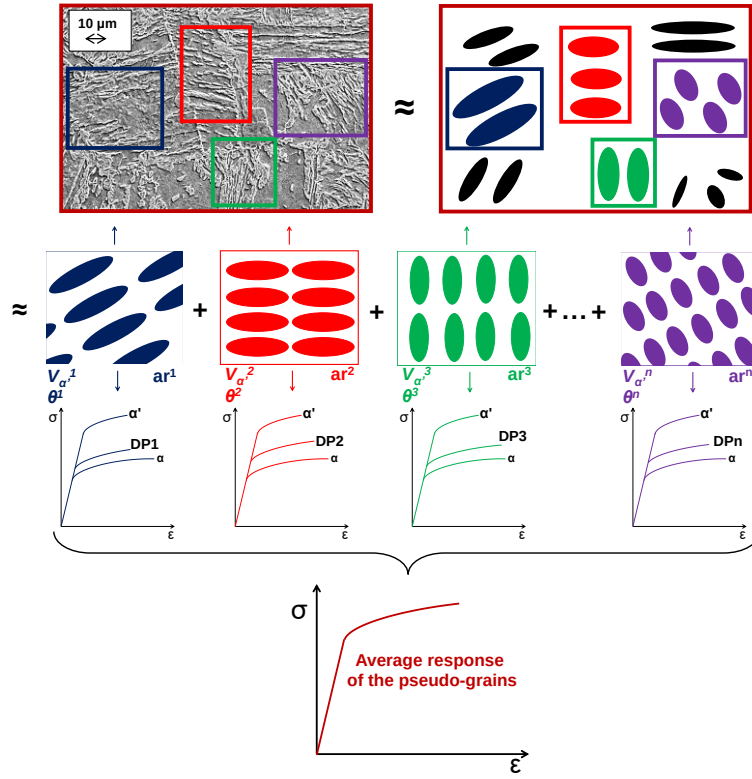


Figure 4.1 Computational approach for a generic Dual-Phase microstructure.

The two-level computational strategy is based on the generation of a wide range of unit cells, each one representing a grain, in which the microstructure is assumed periodic, consisting of a regular array of spheroids (Figure 4.1). Within each cell, three parameters are independently varied, namely the particle aspect ratio (ar), orientation (θ) and martensite volume fraction ($V_{\alpha'}$). Each unit cell is uniaxially deformed in direction 1 of the macroscopic reference system. The effective response of the aggregate of grains is obtained by averaging the stress over all unit cells. In the averaging procedure, a weight is attributed to the response of each cell depending on its importance in the distribution of orientations. This method has the advantage of being fully 3D without being too computationally intensive. More details about the definition of the parameters, the FE procedures and the averaging procedure are given hereunder.

4.1.2 Unit cell description

Owing to the periodicity of the microstructure within each pseudo-grain, each unit cell consists of a parallelepiped box containing a single spheroidal particle surrounded by matrix material. The main directions of the particle are thus aligned with the edges of the cell. In view of FE calculations using periodic boundary conditions, it is much more convenient to always consider particles to be aligned with the cell edges, and, instead, to vary the loading direction relative to the spheroid axes. The unit cell is described by three parameters:

- **Aspect ratio “ ar ”.** The aspect ratio of the spheroidal particle is defined as the ratio of the long axis to the short axis in the case of a prolate spheroid ($ar > 1$), and as the ratio of the short axis to the long axis in the case of an oblate spheroid ($ar < 1$). Prolate spheroids were considered in addition to oblate particles that better represent the physical reality of platelet-like particles in order to increase the generality of the modeling results. The unit cell is given the same aspect ratio as the particle. In other words, the effect of the relative particle spacing between particles at constant aspect ratio is not investigated. Note also that the considered particle stackings are not isotropic, even in the case of spherical particles, which are aligned in a cubic array. The ratio of the cell edges can then be deduced from the volume fraction and aspect ratio of the particle (the actual size of the cell is not relevant since the constitutive models used in this work do not include any internal length). Four aspect ratios have been selected: (i) $ar = 1$ (spherical particles); (ii) $ar = 0.2$ (platelet-like particles); (iii) $ar = 2$ and $ar = 6$ (fiber-like particles).
- **Orientation angle “ θ ”.** Uniaxial loading of the unit cell is performed at varying angles relative to the spheroid axes in order to determine the effect of particle orientation. As shown in Figure 4.2, θ is defined as the angle between the macroscopic loading direction and the direction of the particle revolution axis. In the case of an oblate spheroid, the revolution axis is the short axis while it is the long axis

for a prolate spheroid. Angles θ equal to 0° , 30° , 45° , 60° and 90° have been considered. φ is kept equal to 0° .

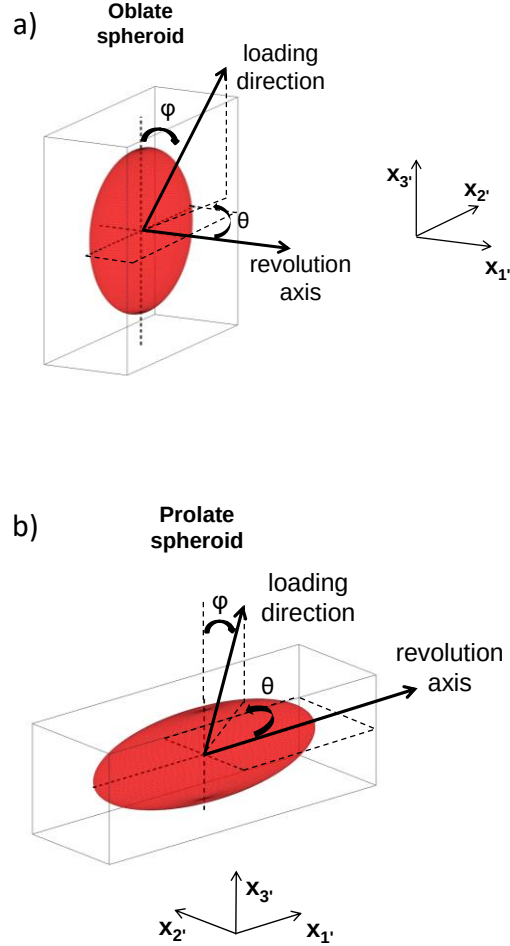


Figure 4.2 Definition of loading direction θ relative to the revolution axis of the spheroid in the case of a) an oblate spheroidal particle, b) a prolate spheroidal particle.

- **Martensite volume fraction " $V_{\alpha'}$ ".** $V_{\alpha'}$ is varied by changing the size of the particle at constant cell size. For low to moderate volume fraction of martensite (up to 50%), we assumed a martensite particle within a ferrite matrix. The role of martensite as a particle phase becomes however questionable at larger volume fractions, where martensite connectivity arises. In order to take this

percolation effect into account, the roles of matrix and particles are reversed in the numerical simulation at martensite volume fractions larger than 50%, with the martensite being treated as the matrix and the ferrite as the particle. The considered values for the martensite volume fraction are $V_{\alpha'} = 10\%$, 20%, 30%, 45% (martensite particle) and 55% and 70% (ferrite particle).

The effective response of the k^{th} unit cell ($k = 1, \dots, n$) is calculated using the FE method. Periodic boundary conditions are imposed and expressed as

$$u_{i'}^+ - u_{i'}^- = \varepsilon_{i'j'}^{(k)} (x_{j'}^+ - x_{j'}^-) \quad 4.1$$

where $\mathbf{u}$ is the displacement vector on the cell boundary, $\mathbf{x}$ refers to the position of the nodes on the boundary and superscripts + and - indicate corresponding points on either side of the unit cell. According to a classical result in homogenization theory, the strain tensor $\varepsilon^{(k)}$ then coincides with the volume average of the microscopic strain field within the unit cell. In Equation 4.1, the primed indices denote components in a *local* coordinate system with axes aligned with the edges of the unit cell. The effective stress applied to the unit cell, $\sigma^{(k)}$ is then calculated as the volume average of the local stress field within the unit cell.

The unit cell is uniaxially loaded in tension in the macroscopic direction 1, which makes an angle θ with the particle revolution axis. In a strain-driven approach, this means that only the component $\varepsilon_{11}^{(k)}$ is known a priori (unprimed indices indicate components in the macroscopic coordinate system). The other components of strain $\varepsilon_{ij}^{(k)}$ are determined iteratively in such a way that the stress state $\sigma_{ij}^{(k)}$ is uniaxial, with $\sigma_{11}^{(k)}$ the only non-zero component. All unit cells are thus subject to the same macroscopic strain $\varepsilon_{11}^{(k)} = \varepsilon_{11}$, whereas the other components $\varepsilon_{ij}^{(k)}$, as well as the stress $\sigma_{11}^{(k)}$, differ from cell to cell. Components of strain (stress) in the macroscopic and local coordinate systems are related by the usual transformation rules for second-order tensors.

Periodic FE meshes with second-order tetrahedra elements were generated using the software Netgen (Schöberl, 1997) and the FE simulations were

carried out using the FE software Abaqus (ABAQUS, 2012) using C3D10M elements. The periodic condition (Equation 4.1) and the transformation rules between local and macroscopic coordinate systems were implemented through the use of the keyword EQUATION in ABAQUS, together with fictitious nodes undergoing a displacement representing the a priori unknown components of macroscopic strain. The number of elements in the meshes for a spherical particle, prolate particle and oblate particle were approximately 7500, 67000 and 62000, respectively. Unit cell calculations have been performed for every combination of the parameters mentioned above. This database can certainly be extended in future works to other parameters.

4.1.3 Phase behavior

The elastic response of ferrite and martensite is assumed linear and isotropic with Young's modulus $E = 200$ GPa and Poisson's ratio $\nu = 0.3$, identical for both phases. Classical J_2 flow theory is used to describe the plastic flow behavior of both ferrite and martensite.

The hardening behavior of martensite is described by an exponential law:

$$\sigma_{y,\alpha'} = \sigma_{y0,\alpha'} + k_{\alpha'}(1 - \exp(-p n_{\alpha'})) \quad 4.2$$

where $\sigma_{y,\alpha'}$ is the current yield strength, p the accumulated plastic strain, and $n_{\alpha'}$ the hardening exponent. The initial yield strength $\sigma_{y0,\alpha'}$ and hardening parameter $k_{\alpha'}$ (in MPa) depend on the martensite carbon content $C_{\alpha'}$ (in wt.%) according to

$$\sigma_{y0,\alpha'} = 300 + 1000C_{\alpha'}^{1/3} \quad 4.3$$

and

$$k_{\alpha'} = (1/n_{\alpha'}) \left[a + \frac{bC_{\alpha'}}{1 + (C_{\alpha'}/C_0)^q} \right] \quad 4.4$$

where $a = 33.103$ MPa, $b = 36.104$ MPa, $C_0 = 0.7$, $q = 1.45$ and $n_{\alpha'} = 120$. The hardening model (Equations 4.2 to 4.4) and parameter values are borrowed from *Pierman et al. (2014)*. Best-fit parameters were obtained

based on experimental tensile testing data for fully martensitic bulk samples with varying carbon contents. A comparison with experimental curves can be found in that paper. All results shown in the following were obtained for a martensite carbon content $C_{\alpha'}$ equal to 0.15 wt.%.

The hardening law of ferrite is expressed by Voce's law supplemented by a stage-IV hardening regime:

$$\sigma_{y,\alpha} = \sigma_{y0,\alpha} + (\theta_{\alpha}/\beta)(1 - \exp(-\beta p)) \quad \text{for } \sigma_{y,\alpha} \leq \sigma_y^{tr}, \quad 4.5$$

$$\sigma_{y,\alpha} = \sigma_y^{tr} + \theta_{IV}(p - \varepsilon^{tr}) \quad \text{for } \sigma_{y,\alpha} \geq \sigma_y^{tr} \quad 4.6$$

where σ_y^{tr} and ε^{tr} are respectively the flow stress and the plastic strain at the transition between stage-III and stage-IV hardening and are given by

$$\sigma_y^{tr} = \sigma_{y0,\alpha} + \frac{\theta_{\alpha} - \theta_{IV}}{\beta}, \quad 4.7$$

$$\varepsilon^{tr} = \frac{1}{\beta} \ln(\theta_{\alpha}/\theta_{IV}), \quad 4.8$$

and where $\sigma_{y0,\alpha}$ is the initial yield strength of ferrite, θ_{α} is the initial work-hardening rate, β is the dynamic recovery coefficient and θ_{IV} is the hardening rate during stage-IV. The values used for the ferrite parameters were taken from (Lai *et al.*, 2015), who identified them by fitting his experimental data: $\sigma_{y0,\alpha} = 250$ MPa; $\theta_{\alpha} = 4900$ MPa; $\beta = 11$; $\theta_{IV} = 100$ MPa.

4.1.4 Effective properties

4.1.4.1 Averaging over particle orientations distribution

The effective response of the steel is obtained by averaging the stress in the direction of macroscopic loading predicted by a series of unit cells corresponding to various particle orientations. In this study, we assumed that the martensite volume fraction and particle aspect ratio is the same in all unit cells in order to represent one hypothetical model microstructure for each combination of parameters.

We define σ_{mean} as the macroscopic stress in the direction of loading, averaged over all particle orientations. For a uniform distribution of particle orientations, averaging of the macroscopic stress component σ_{11} of each grain over each particle orientation expresses as:

$$\sigma_{mean} = (1/4\pi) \int_0^{2\pi} \int_0^\pi \sigma_{11}(\varphi, \theta) \sin\theta d\theta d\varphi, \quad 4.9$$

where $\sigma_{11}(\varphi, \theta)$ represents the average stress in a unit cell in which the macroscopic loading direction relative to the spheroid principal axes is described by the two angles (φ, θ) , see Figure 4.2. The periodicity of the unit cell implies that the particles are arranged in a rectangular packing. Therefore, the effective response of a unit cell is not exactly transversely isotropic, since the distance between the particles differs with the angle φ . However, to limit the computational cost, we only considered the case $\varphi = 0^\circ$ and assumed transverse isotropy, i.e.:

$$\sigma_{mean} \approx \frac{1}{2} \int_0^\pi \sigma_{11}(\theta) \sin\theta d\theta. \quad 4.10$$

In practice, simulations have been carried out for a limited number of angles only (see Section 4.1.2). A weight was attributed to each orientation in order to represent a uniform distribution of angles. The integral in Equation 4.10 is approximated as:

$$\begin{aligned} \sigma_{mean} &= (1/2) \cdot 2 \left[\int_{0^\circ}^{15^\circ} \sigma_{11}(0^\circ) \sin(\theta) d\theta + \int_{15^\circ}^{37.5^\circ} \sigma_{11}(30^\circ) \sin(\theta) d\theta \right. \\ &+ \int_{37.5^\circ}^{52.5^\circ} \sigma_{11}(45^\circ) \sin(\theta) d\theta + \int_{52.5^\circ}^{75^\circ} \sigma_{11}(60^\circ) \sin(\theta) d\theta \\ &\left. + \int_{75^\circ}^{90^\circ} \sigma_{11}(90^\circ) \sin(\theta) d\theta \right]. \end{aligned} \quad 4.11$$

4.1.4.2 Determination of the mechanical properties

Mechanical properties are calculated at both grain level and aggregate level. Investigating mechanical properties at the level of a single unit cell is interesting for two reasons: (i) to help understanding the trends for the

aggregate response; (ii) to gain insight into the limit case of a Dual-Phase steel with strong morphological anisotropy.

a. Flow stress at different levels of plastic deformation and at the onset of necking. All values are given in terms of true stress and true (logarithmic) strain. The yield strength $\sigma_{0.2}$ is taken as the stress at 0.2% of plastic strain. Likewise, the flow stresses σ_1 and σ_5 are defined at 1% and 5% of plastic strain. The tensile strength σ_u and the associated uniform elongation ε_u are estimated using the Considère criterion

$$\frac{d\sigma}{d\varepsilon} = \sigma. \quad 4.12$$

b. Back stress. The contrasted phase properties lead to the development of heterogeneous plastic strains and hence back stresses. Back stresses are responsible for the Bauschinger effect, which consists in a reduction of the effective flow stress during reverse loading. Reverse loading simulations were systematically conducted following a 10% pre-straining, and the following measure of back stress was used (*Moan and Embury, 1979*):

$$\sigma_b = \frac{\sigma_{ref} - |\sigma_{be}|}{2}, \quad 4.13$$

in which σ_{be} is the reloading yield strength at 0.2%, called the back extrapolated stress, and σ_{ref} is the maximum value of stress reached in monotonic loading prior to unloading, called the reference stress, see Figure 4.3. Back stresses contribute to the strain hardening capability.

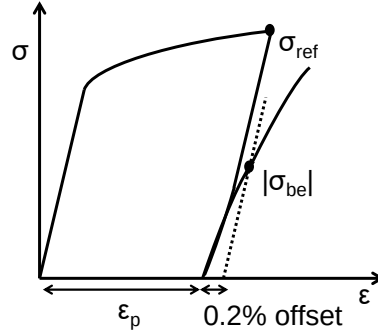


Figure 4.3 Definition of the parameters used to define the magnitude of the back stress.

4.1.5 Extraction of local fields

The analysis focuses on three local field quantities, namely the accumulated plastic strain p , the maximum principal stress σ_I and the stress triaxiality T . The latter is defined as

$$T = \frac{\sigma_h}{\sigma_{eq}} \quad 4.14$$

where σ_h is the hydrostatic stress and σ_{eq} is the von Mises equivalent stress. These three parameters are known to be the primary elements controlling the process of damage. A damage indicator D , motivated by the Rice and Tracey void growth law (Rice and Tracey, 1969), is calculated as

$$D = \int_0^p \exp((3/2) T) dp'. \quad 4.15$$

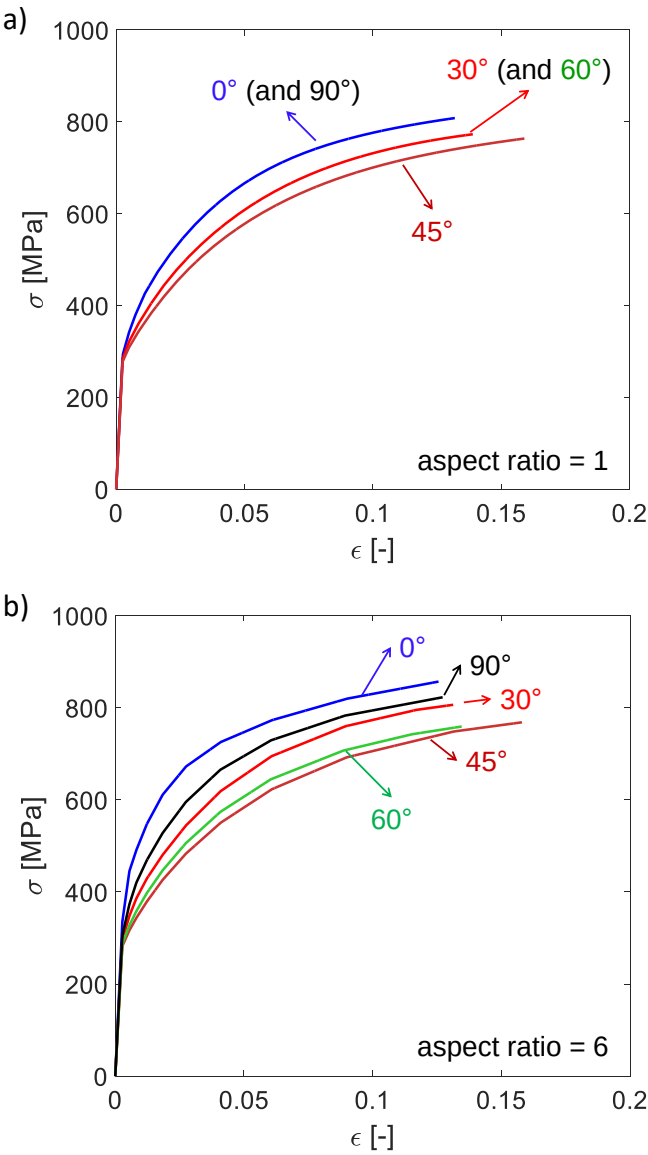
The magnitude of these parameters can be evaluated at all points in the cell, but we will in particular look at the pole of the particle on the revolution axis where they can attain especially high values due to stress and strain concentration. It is worth noting that the Lode angle parameter, which is not investigated here, can also largely influence damage behavior (Bai and Wierzbicki, 2008; Erice and Gálvez, 2014; Pineau et al., 2016; Xue, 2007) (see Appendix D).

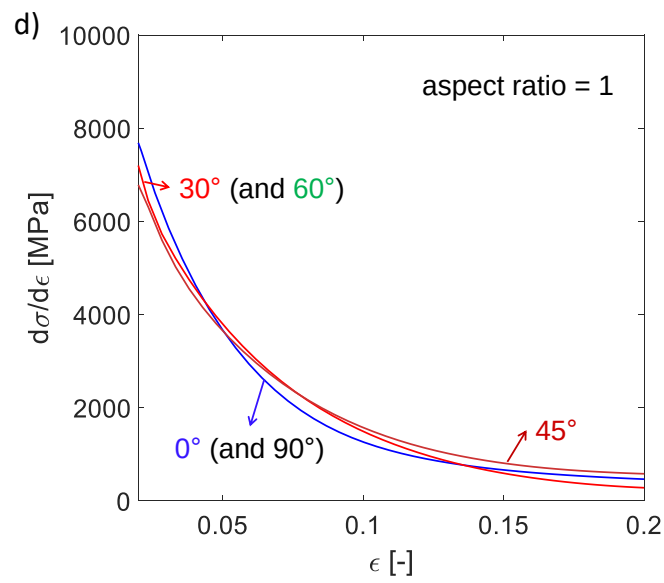
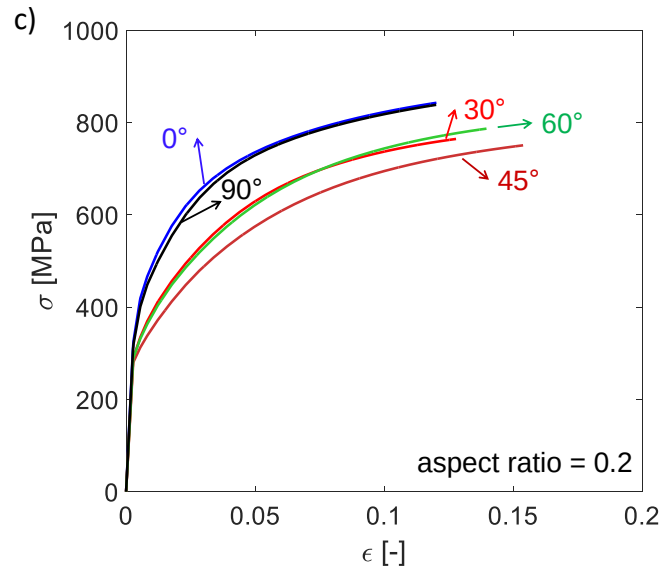
4.2 Parameter study

4.2.1 Effect of particle orientation on mechanical properties at grain level

4.2.1.1 Stress-strain response

The predicted stress-strain curves of unit cells corresponding to different loading orientations are given in Figure 4.4 up to the onset of necking together with the variation of the hardening rate as a function of strain. Figure 4.4(a) shows that for spherical martensite particles, the hardening capacity decreases when increasing the angle θ from 0° to 45° . Once the threshold value of 45° is reached, increasing further θ up to 90° leads to the opposite trend. The difference between the responses comes from the non-isotropic distribution of particles as explained in Section 4.1.2 .





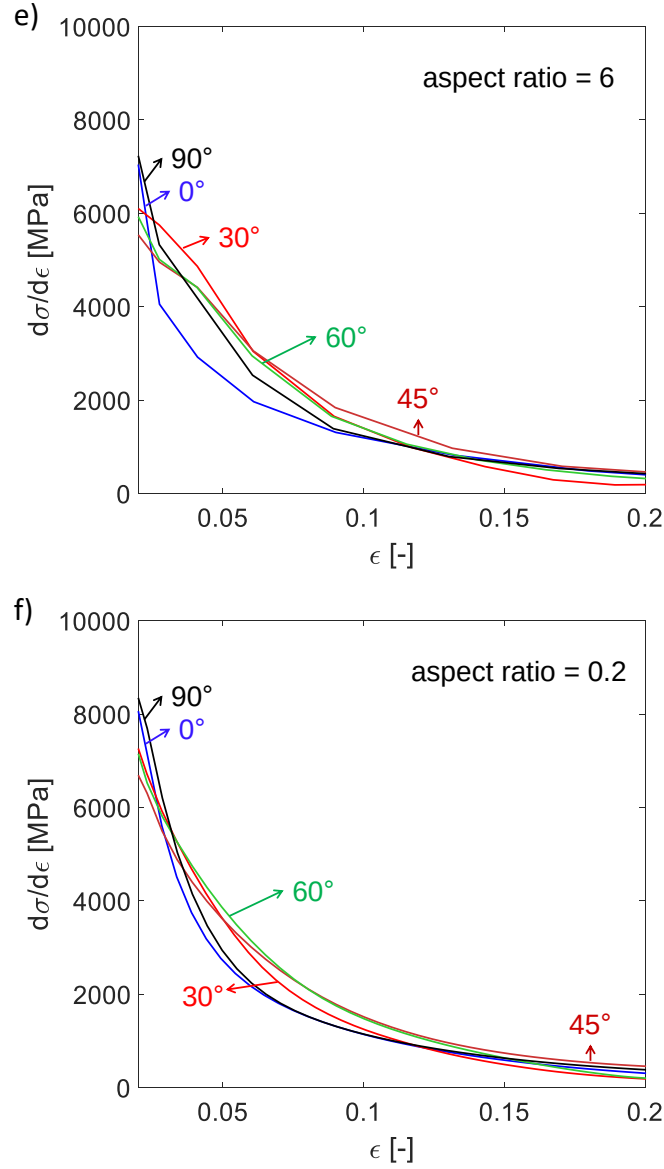


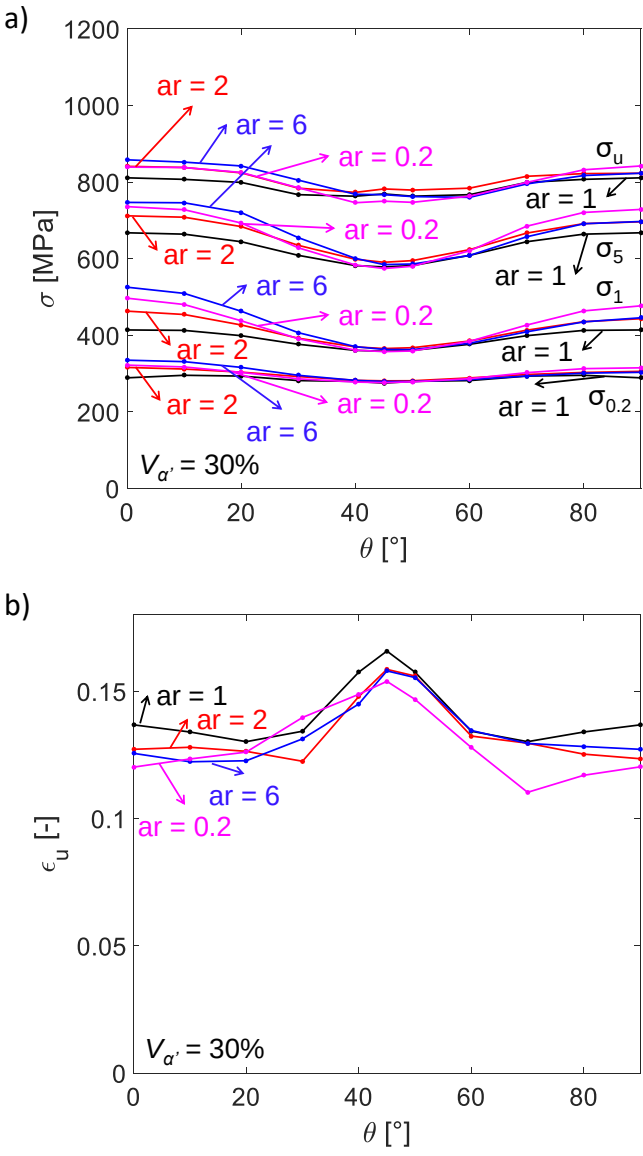
Figure 4.4 Stress-strain response and derivative of stress with respect to strain for various loading directions θ in the case of elementary cells with $V_{\alpha'} = 30\%$ and involving: a) and d) spherical martensite particle, b) and e) prolate martensite particle, c) and f) oblate martensite particle.

Figure 4.4(b) and Figure 4.4(c), for the case of prolate and oblate particles, respectively, show that the same trend applies to non-spherical particles, i.e. that the 0° and 90° orientations lead to a larger strain hardening capacity

than the 30° and 60° responses which in turn have a larger strain hardening capacity than the 45° response. Besides, the behaviors for θ equal to 0° and 90° (or 30° and 60°) do not coincide as in the case of spherical particles, due to a reduced symmetry of the cells when the particle shape is not spherical. The importance of the difference between these responses varies with aspect ratio and martensite volume fraction.

4.2.1.2 Strength and ductility

Figure 4.5 shows the variations of the yield strength $\sigma_{0.2}$, the tensile strength σ_u , as well as σ_1 and σ_5 reference strength levels, and ε_u as a function of θ . Figure 4.5(a) and Figure 4.5(b) are for a ferritic matrix ($V_{\alpha'} = 30\%$) and Figure 4.5(c) and Figure 4.5 (d) for a martensitic matrix ($V_{\alpha'} = 70\%$). In agreement with the results of Figure 4.4, the hardening capacity decreases from 0° to 45° and increases again up to 90°. Ductility is the highest at 45° and the strength is the lowest. Furthermore, spherical particles almost always exhibit the lowest strength and the highest ductility for a ferrite matrix. The higher strength in the 0° and 90° orientations compared to ~45° is due to a higher hardening rate around 0° and 90° orientations at small strains, and the higher ductility around 45° is due to a higher hardening rate in the ~45° orientation at higher strains, see Figure 4.4(d) to Figure 4.4 (f). Departing from an aspect ratio of 1 increases the strength and decreases the ductility. The effect of the aspect ratio is opposite when martensite plays the role of matrix. But, in both cases, the effect of orientation increases when departing from an aspect ratio of 1. These results show that orientation effects can lead to grain to grain strength differences, sometimes up to more than 20%. And, in the case of a martensitic matrix, the ductility ε_u almost doubles between 0° and 45° for very prolate or very oblate ferrite particles.



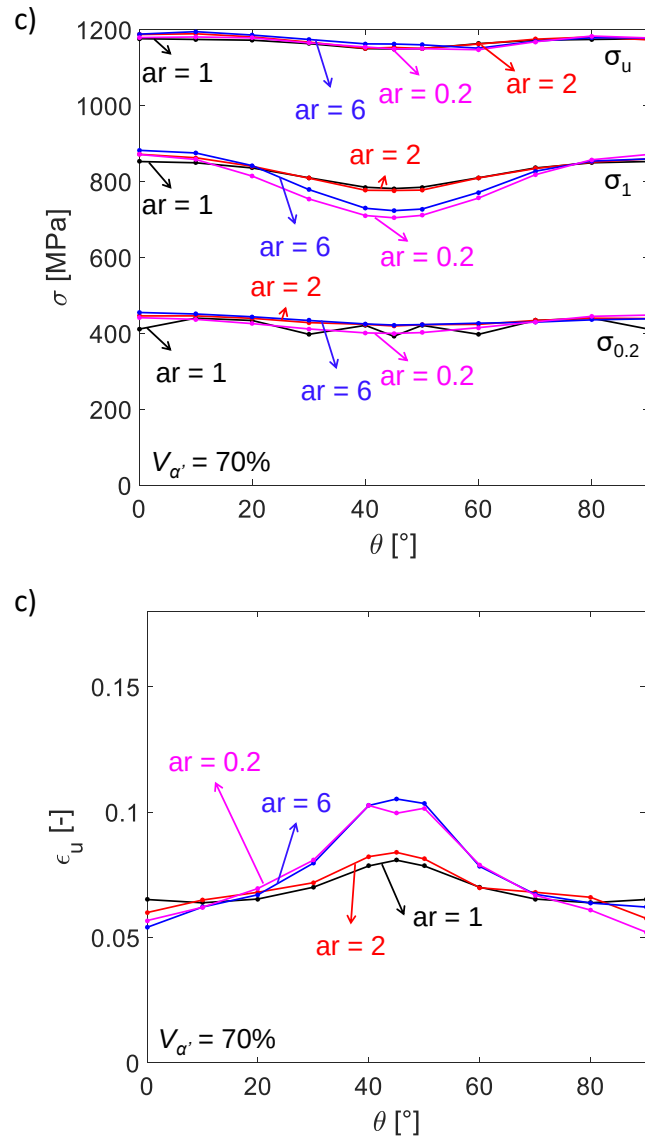


Figure 4.5 Summary of the main flow properties for elementary unit cells as a function of loading direction θ . a) Yield strength at various strains for a ferritic matrix, b) uniform deformation for a ferritic matrix, c) yield strength at various strains for a martensitic matrix, d) uniform deformation for a martensitic matrix.

4.2.1.3 Magnitude of the back stress

Figure 4.6 shows the back stress σ_b generated by a pre-strain of 0.1 as a function of the orientation for three particle aspect ratios. In the case of a ferritic matrix (Figure 4.6(a)), the magnitude of the back stress is significantly amplified in non-equiaxed particles, especially near the 0° and 90° orientations. This means that internal stresses are larger for oblate and prolate particles, because the absence of sharp particle tips in equiaxed microstructures and a lower confinement of the ferrite matrix in the vicinity of the tips lead to more homogeneous stress distribution. The larger kinematic hardening contribution (i.e. large back stresses) probably explains the larger strain hardening capacity at small strains. In the case of a martensitic matrix (Figure 4.6(b)), the magnitude of the back stress is larger for equiaxed particles for most orientations, explaining the higher strain hardening capacity at small strains in the equiaxed microstructure steels. Here again, the variation of back stress with orientation is more significant for non-equiaxed particles. However, the order of magnitude of the back stress is about the same for both a ferritic and martensitic matrix. But, compared to the yield stress, its amplitude is much lower in the case of a martensitic matrix. A smaller back stress is expected for a hard matrix embedding softer particles. The reason is that the hard matrix then contributes more to deformation, thereby decreasing the plastic deformation mismatch, and thus the ensuing back stress.

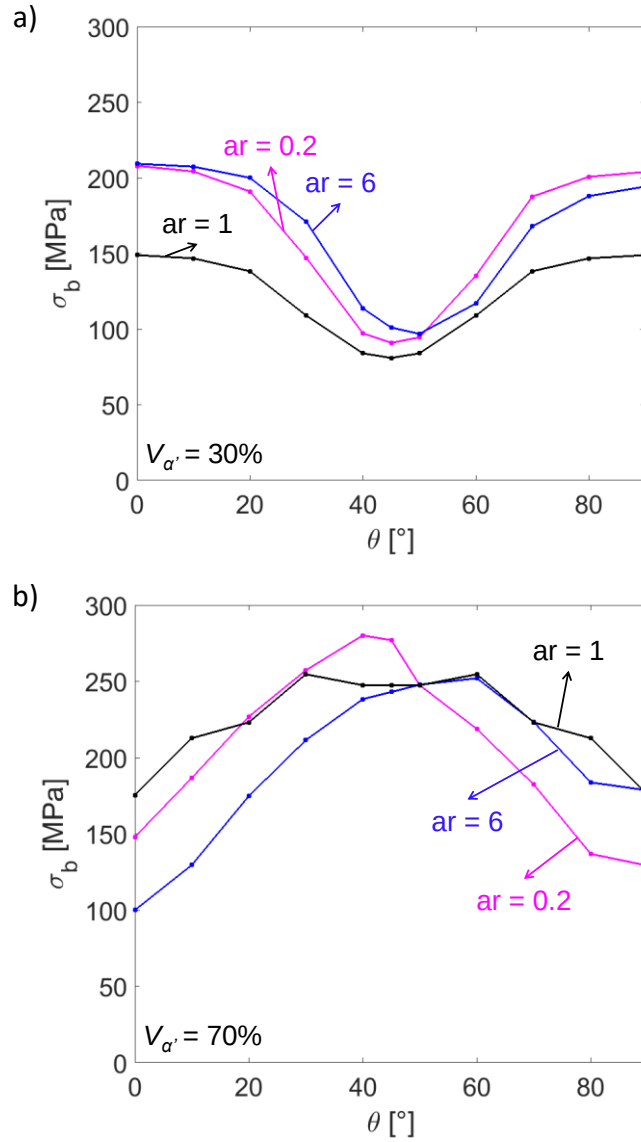
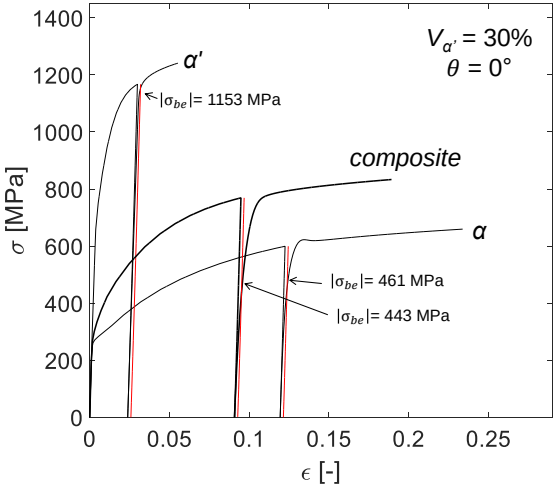


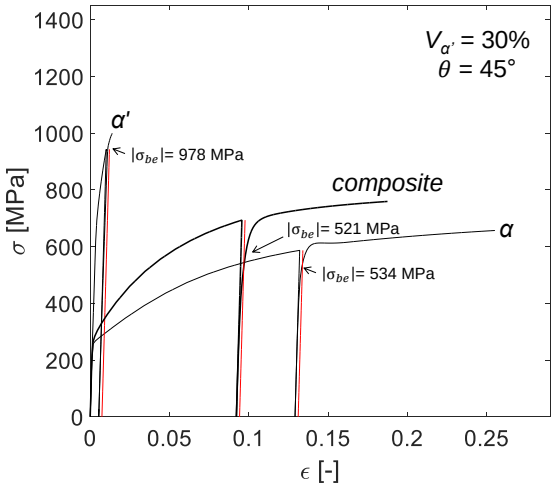
Figure 4.6 Effect of the loading orientation and particle aspect ratio on the magnitude of the back stress for a) a ferritic matrix, b) a martensitic matrix.

In addition to Figure 4.6, Figure 4.7 shows that there is less (resp. more) back stress at 45° than at 0° when the particle phase is made of hard martensite (resp. the softer ferrite). The effect on the macroscopic curve is more pronounced in the case of a ferritic matrix ($V_{\alpha'} = 30\%$). This is because back stress essentially builds up in the softer ferrite, which contribution is low in the case of the martensitic matrix ($V_{\alpha'} = 70\%$).

a)



b)



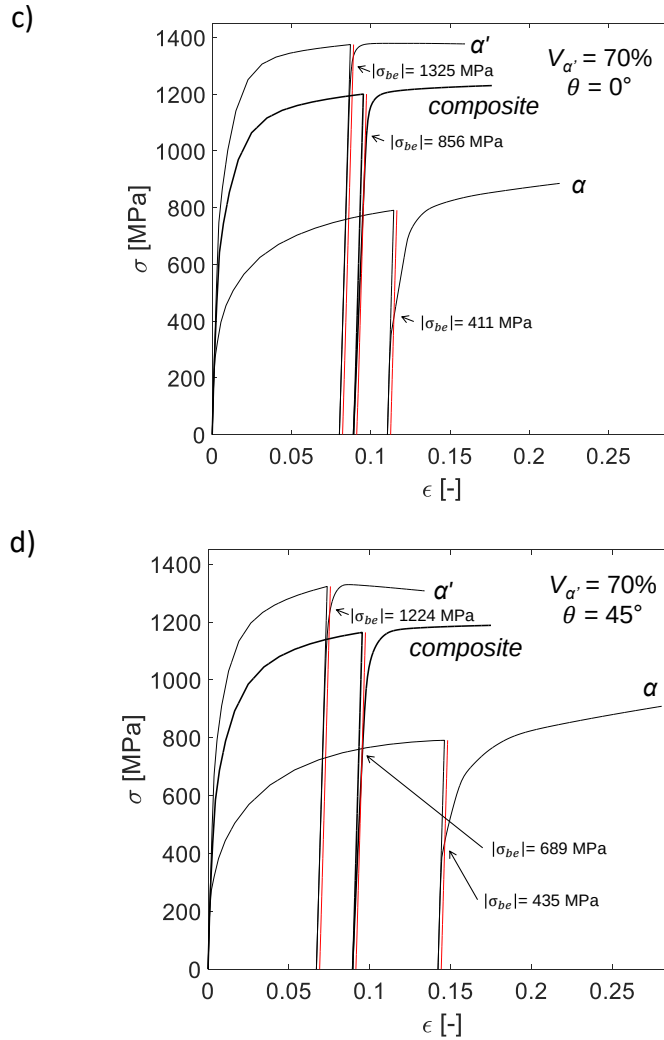


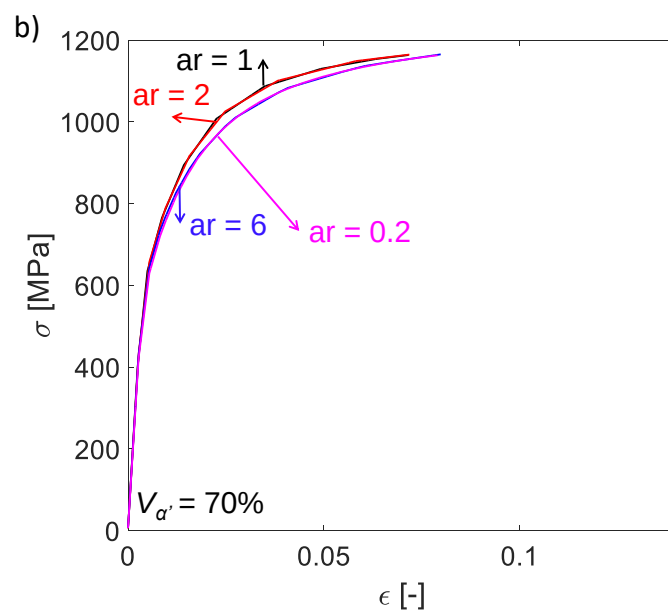
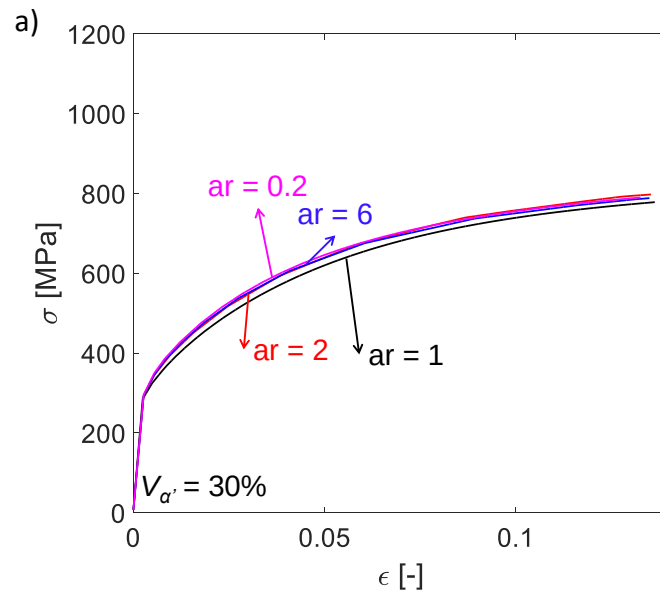
Figure 4.7 Back stress level at an overall prestrain of 0.1 in the ferrite phase, martensite phase and full unit cell for a) 30% $V_{\alpha'}$ and a 0° θ , b) 30% $V_{\alpha'}$ and a 45° θ , c) 70% $V_{\alpha'}$ and a 0° θ , d) 70% $V_{\alpha'}$ and a 45° θ .

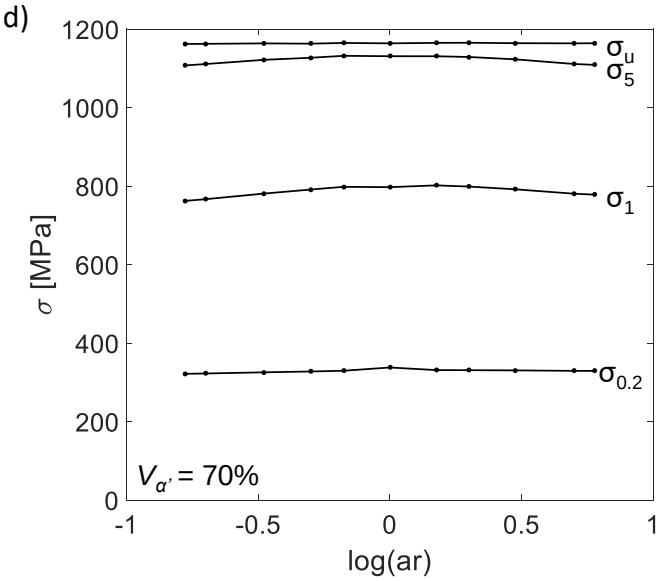
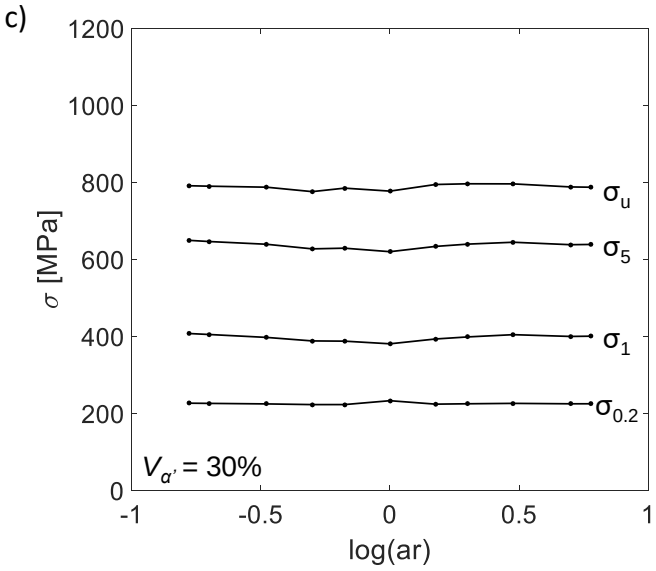
4.2.2 Impact of martensite volume fraction and aspect ratio on aggregate response

The response of, on the one hand, an aggregate of several grains with different martensite lath orientations in a ferritic matrix ($V_{\alpha'} = 30\%$), and, on the other hand, of ferrite particles embedded inside a martensitic matrix

($V_{\alpha'} = 70\%$), are addressed in Figure 4.8 for different aspect ratios. Figure 4.8(a) and Figure 4.8(b) show the stress-strain response for the ferritic and martensitic matrix, respectively. Figure 4.8(c) and Figure 4.8(d) show the magnitude of the flow stress at different strains for the ferritic and martensitic matrix, respectively. Figure 4.8(e) and Figure 4.8(f) show the effect of particle aspect ratio on the ductility and on the $\sigma_u \varepsilon_u$ product, respectively. The morphology of the second phase has some effects on the aggregate behavior:

- For a ferritic matrix, the strain hardening capacity of the Dual-Phase steels increases at small deformation when the martensite particle aspect ratio differs from 1. A decrease of the strain hardening capacity at small deformation with increasing particle aspect ratio is observed in the case of a martensitic matrix. This reversed trend compared to a ferritic matrix is observed due to the fact that martensite then accommodates a larger part of the deformation. Indeed, the ferrite becomes more spatially confined when the aspect ratio differs from unity, especially around the regions of high curvature at the spheroid tips. Hence, the ferrite phase is forced to follow the deformation of the martensite matrix and the deformation mismatch thus decreases.
- Figure 4.8(c) to Figure 4.8(f) show that very prolate particles lead to a behavior similar to very oblate particles, hence particle morphology acts roughly through the ratio between the length of the longest to the shortest axis.
- Figure 4.8(e) shows that the uniform elongation varies inversely to the strength regarding the effect of aspect ratio. This leads to a relatively constant product $\sigma_u \varepsilon_u$ in the case of a ferritic matrix as seen in Figure 4.8(f). In the case of a martensitic matrix however, an increase of the $\sigma_u \varepsilon_u$ product by about 10% is found when departing from the spherical shape. This is due to the larger variation of uniform elongation with orientation for very prolate or very oblate particles, which doubles between 0° and 45° , see Figure 4.5(d).





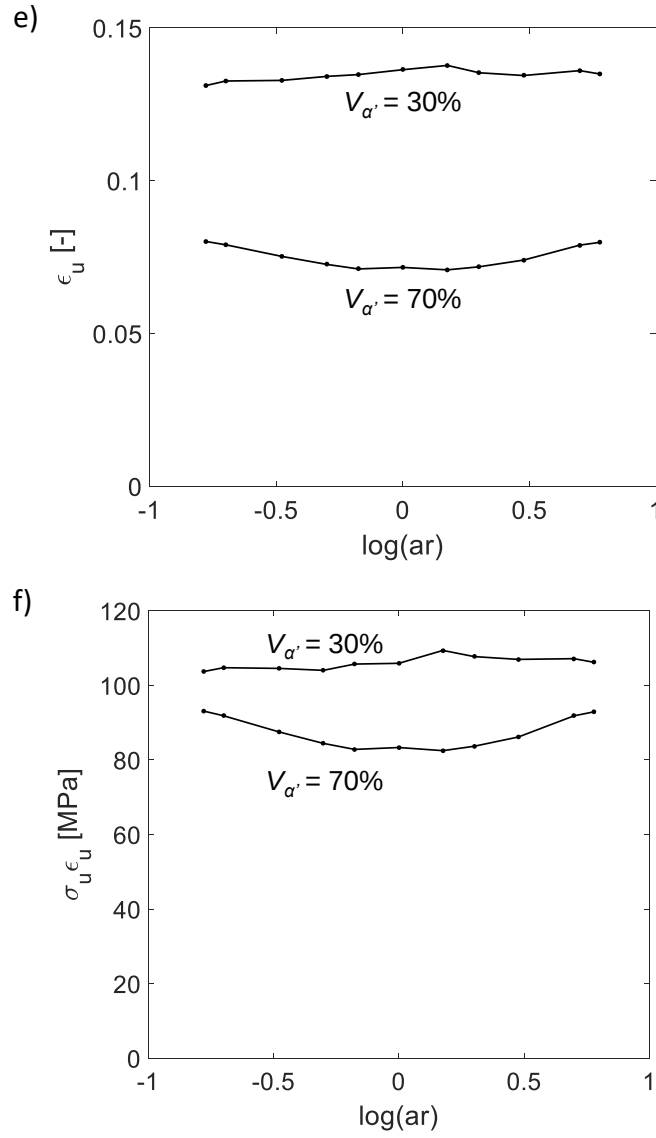


Figure 4.8 Effect of the particle aspect ratio on the response of Dual-Phase steels with a uniform distribution of particle orientations in terms of a) uniaxial stress-strain behavior for a ferritic matrix, b) uniaxial stress-strain behavior for a martensitic matrix, c) flow stress at different strains for a ferritic matrix, d) flow stress at different strains for a martensitic matrix, e) uniform deformation, f) product $\sigma_u \epsilon_u$.

The most important conclusion at this stage of the analysis is that the impact of martensite morphology on the aggregate hardening behavior is small (see

discussion) when the distribution of second phase arrangement and spatial arrangement is isotropic while much larger effects are found at the “grain” level with aligned particles. All these results were obtained for a martensite carbon content equal to 0.15 wt.%. Calculations have been repeated for a martensite carbon content equal to 0.60 wt.%, showing a slightly larger but still limited effect.

4.2.3 Local fields analysis

Figure 4.9 shows contour plots of the local accumulated plastic strain p and of the maximum principal stress σ_I in deformed unit cells for several aspect ratios and identical martensite volume fraction and orientation ($V_{\alpha'} = 30\%$; $\theta = 0^\circ$). The contour plots correspond to a macroscopic strain of 0.1. One eighth of a particle is present at every corner of the cell. Extreme differences in the local values can be found within one unit cell. The ferrite-martensite interface is systematically undergoing the largest plastic strains on the soft ferrite side, and, in the particular case of a spherical martensite particle at 0° , the center of the ferrite ligament in the direction of loading also undergoes large strains.

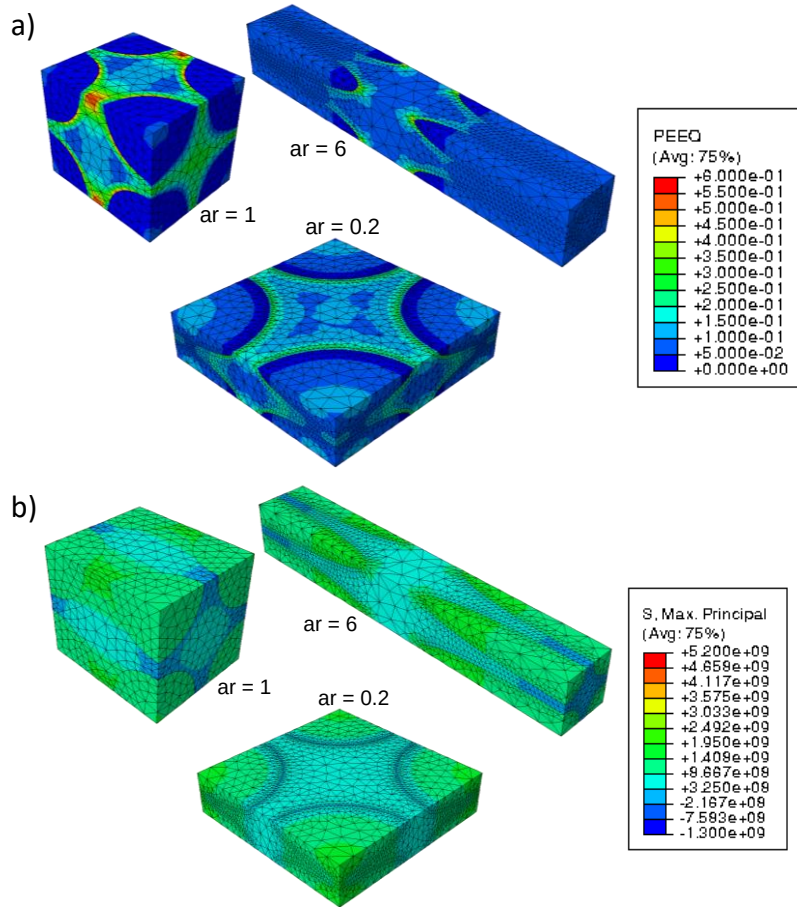
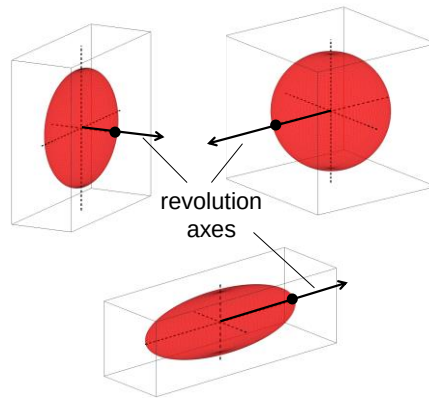


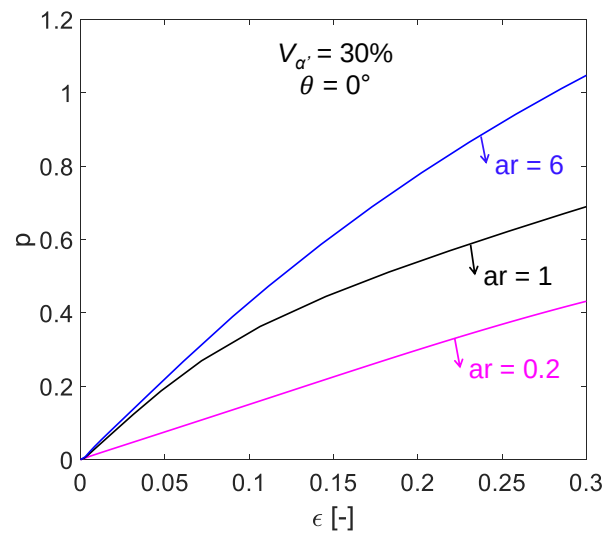
Figure 4.9 Contour plots for unit cells at a global strain of 0.1 with $V_{\alpha'}$ of 30%, θ of 0° of a) accumulated plastic strain and b) maximum principal stress (in Pa).

The variation of p at the particle pole located on the revolution axis (see Figure 4.10(a)) is shown in Figure 4.10(b) and Figure 4.10(c) as a function of the macroscopic applied strain. Figure 4.10(b) is for $\theta = 0^\circ$ and Figure 4.10(c) is for $\theta = 90^\circ$. For the 0° or 90° orientations, p is the highest for a prolate particle and the lowest for an oblate particle, as a result of the difference of particle curvature: the curvature is the highest for a prolate particle with sharp particle tip compared to the smoother curvature of an oblate particle pole.

a)



b)



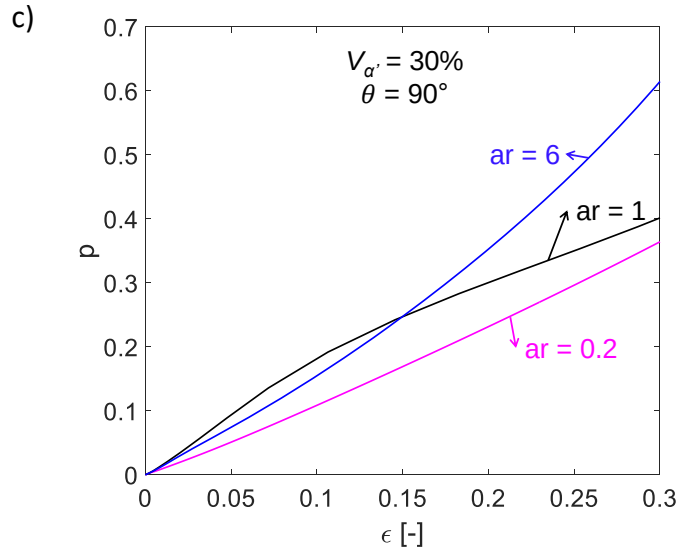
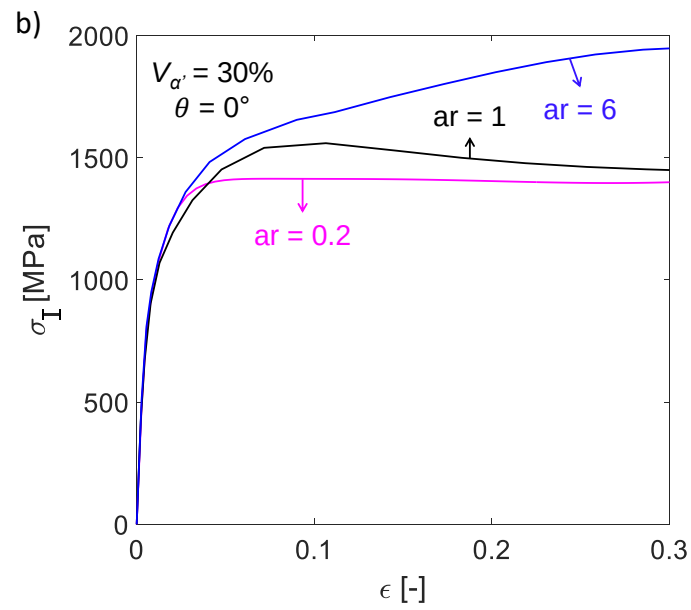
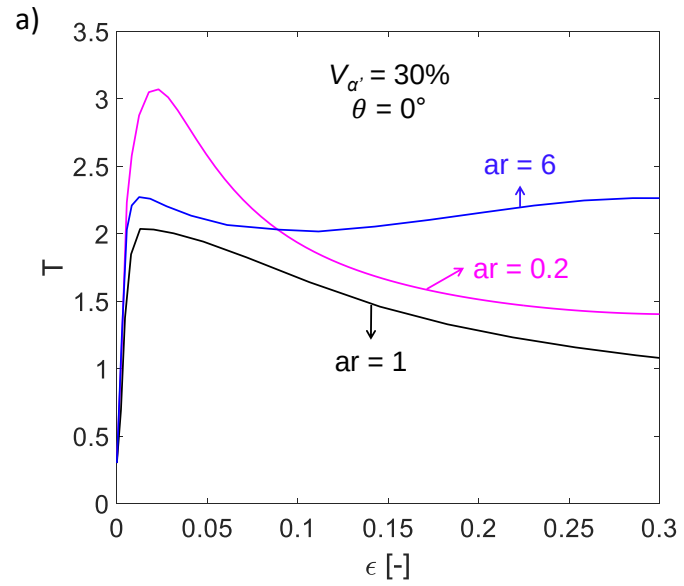
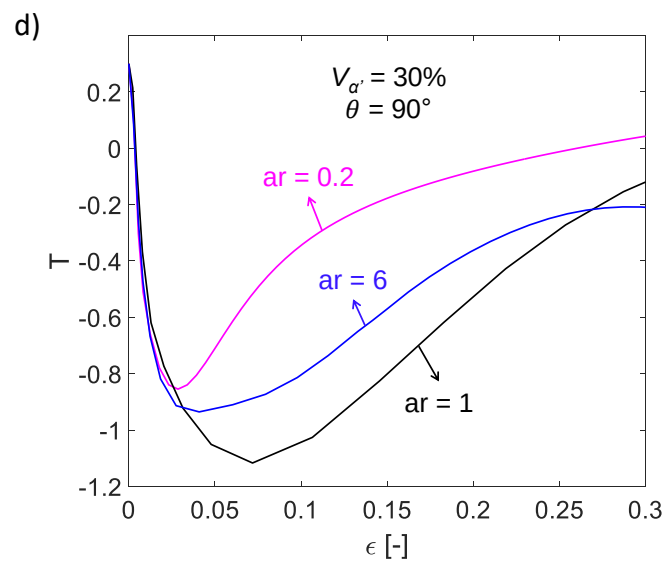
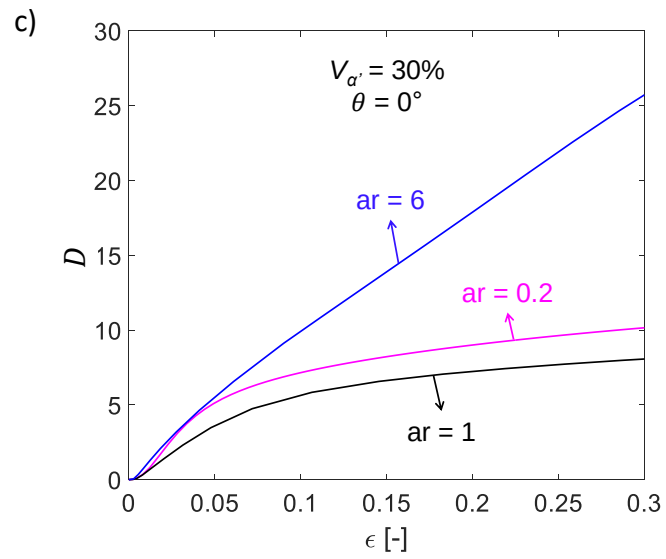


Figure 4.10 Local equivalent strain at the pole of the particle located on the revolution axis as shown in a) as a function of global strain for different aspect ratios ($V_{\alpha'} = 30\%$) for: b) $\theta = 0^\circ$, c) $\theta = 90^\circ$.

The variation of the stress triaxiality, maximum principal stress and damage indicator D are represented as a function of the macroscopic strain ϵ for different aspect ratios in Figure 4.11. The results are given at the pole following the logic of Figure 4.10. The stress triaxiality is positive for 0° since the pole region is in tension and it is negative for 90° due to compression. The maximum principal stress follows the same trend, apart from a first increase at 90° at very small strains within the elastic regime. Even if p is relatively similar for both angles, the pole is a critical damage spot at 0° where D reaches high values.





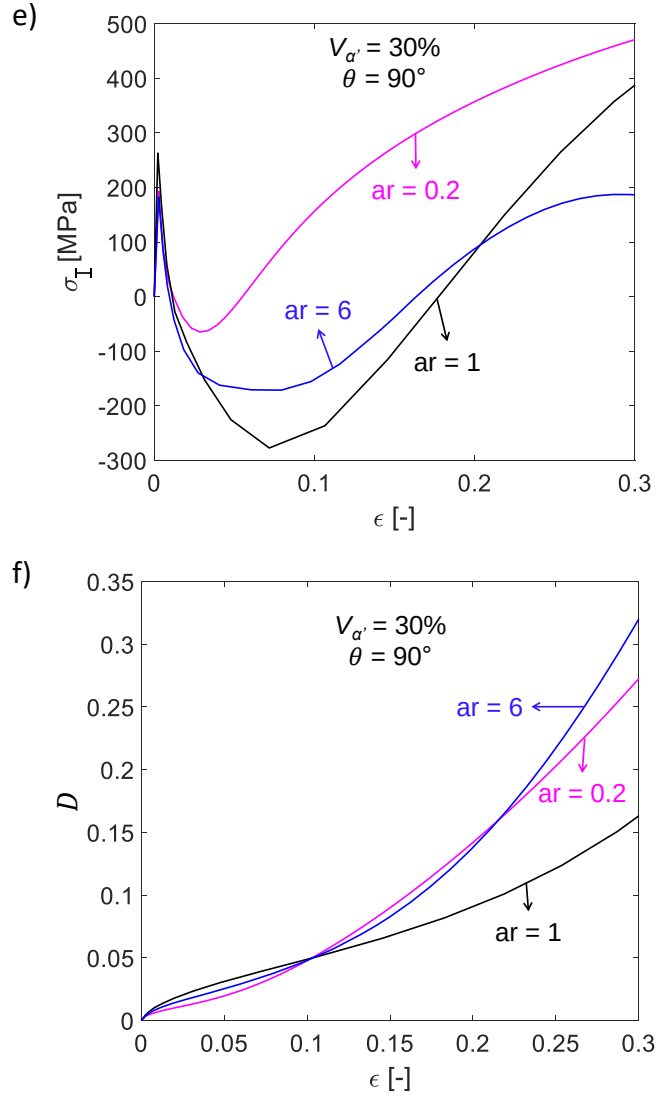


Figure 4.11 Evolution with strain of a) the stress triaxiality for $\theta = 0^\circ$, b) the maximum principal stress for $\theta = 0^\circ$, c) the damage indicator D for $\theta = 0^\circ$, d) the stress triaxiality for $\theta = 90^\circ$, e) the maximum principal stress for $\theta = 90^\circ$, f) the damage indicator D for $\theta = 90^\circ$.

4.3 Discussion

An important conclusion from Section 4.2 is that second phase morphology plays a minor role on the overall elastoplastic material behavior in the case

of a uniform distribution of orientations. In contrast, experimental trends indicate a positive effect of an elongated second phase morphology on strength and ductility, e.g. in (*Das and Chattopadhyay, 2009*) or (*Pierman et al., 2014*). The main reason for which the present model does not capture this trend is probably related to the absence of any internal length in the analysis. By changing morphology from an equiaxed to a platelet-like microstructure, the absolute spacing between particles decreases (refinement effect), which may impact the plastic flow behavior through a larger contribution of geometrically necessary dislocations accumulating near interfaces. The impact of microstructure refinement on the behavior of Dual-Phase steels has been addressed for instance by *Delincé et al. (2007)* showing the possibility to attain an optimum strength-ductility balance when playing with both martensitic volume fraction and ferrite grain size. The refinement effect can also directly impact the magnitude of the residual stresses generated during processing (*Kadkhodapour et al., 2011a; Ramazani et al., 2013a*), which were neglected in this study as well. Now, the present results show that Dual-Phase microstructures with preferential alignment of elongated second phase particles can lead to significant effects on the hardening behavior. This could potentially be useful in applications where the main loading direction is fixed and in which one could beneficially use some controlled particle morphology anisotropy.

Other interesting conclusions can be drawn from the results shown in Section 4.2, regarding the local fields and their impact on damage and fracture toughness. The results of Figure 4.10 and Figure 4.11 suggest that void nucleation by interface decohesion (mainly dictated by the amplitude of the maximum interface normal stress and accumulated plastic strain), and void growth (mainly dictated by stress triaxiality and accumulated plastic strain) are both expected to preferentially proceed at the particle pole located along the direction of loading (for $\theta = 0^\circ$ or $\theta = 90^\circ$). Indeed, the ferrite regions near the interface typically exhibit the largest strains due to the abrupt change in properties (see Figure 4.9). This explains why damage often initiates by interface decohesion in Dual-Phase steels as shown in Figure 4.12 and in many literature papers (e.g. *Landron et al., 2010; Kadkhodapour et al., 2011c*), and in particular at the poles of the elongated particles.

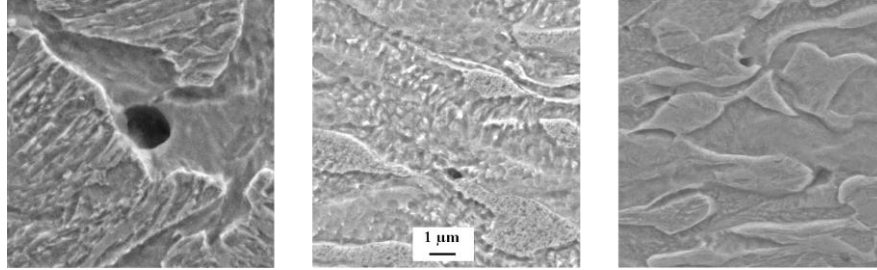


Figure 4.12 Void nucleation by decohesion in Dual-Phase steels. (taken from *Pierman (2013)*)

Moreover, and this is at the core of the objective of the present study, Figure 4.10 and Figure 4.11 show that large differences in local quantities (p , T , σ_I , D) occur at a given pole depending on the aspect ratio and orientation of the particles. A first reason is that stress and strain states at a given location in the matrix near the interface depend on the curvature of the particle (see Section 4.2.3). Second, the relative confinement of the plasticity in the matrix at the particle tip also changes with the aspect ratio. Third, the solicitation at a given point changes with orientation. For instance, under loading at 0° , the ferrite at the pole along the revolution axis undergoes large tensile stresses compensating for the small martensite deformation; under loading at 90° , it will be compressed against the martensite, which resists contraction in the direction perpendicular to the tensile loading. For this pole, 0° will lead to early decohesion due to a high interface normal stress and large accumulated plastic strain and accelerated void growth due to a high stress triaxiality and accumulated plastic strain. At 90° , damage will not develop at the pole, protected by a negative maximum principal stress and a negative stress triaxiality.

The previous considerations suggest that, in an aggregate of grains with elongated/flat particles of different orientations, the variation of local fields from grain to grain will lead to a very heterogeneous damage behavior at this scale, with early nucleation of voids and accelerated void growth in some grains compared to others, in turn leading to early onset of void coalescence in the highly damaged grains. However, fracture will need a second level of coalescence in between the highly damaged grains. This suggests that two length scales are at play during the fracture process: one

associated to particle spacing (as discussed above) and one associated to the martensite packet size following the first step of the heat treatment. The need for large-scale void coalescence between grains at the second length scale could lead to large fracture strains as demonstrated by (*Pierman, 2013*) for Dual-Phase steels with platelet-like microstructure. Regarding fracture toughness, the critical J-integral at cracking scales with the internal length scale dominating the failure process, e.g. in (*Pardoen and Hutchinson, 2003*) or (*Pineau et al., 2016*). A similar two-stage process was for instance recently addressed by *Hannard et al. (2018)* on Al alloys and could also be the reason for the high fracture toughness as also found experimentally by (*Pierman, 2013*). In metallurgical practice, increasing the time and/or temperature of the first step of the heat treatment for a platelet-like microstructure would lead to a larger martensite packet size and, potentially, if the reasoning above is valid, to a higher resistance to cracking as quantified both by the fracture strain and fracture toughness.

At last, in addition to the aforementioned absence of internal length, a few other limitations of the model should however be kept in mind as there are no direct experimental comparisons that can be made at this stage. Some limitations are intrinsic to the model. For instance, the grains are all assumed to be subject to macroscopically uniaxial tension, driven by the same macroscopic strain. In other words, we made an iso-strain assumption in direction 1, and an iso-stress (zero-stress) assumption regarding all the other components of stress (in the macroscopic coordinate system). This assumption was needed to solve each unit cell problem independently, and thereby to reduce the computational cost. An alternative strategy could be to consider a true iso-strain assumption, in which case the iterations on the a priori unknown values of the macroscopic strain components should be carried out after averaging the stress response over all unit cells. This would thus require the simultaneous numerical solution of all the unit cells problems, which would dramatically increase the computational cost of the simulation. However, it is not clear whether a true iso-strain simulation would be more realistic than the adopted strategy. A probably more accurate approach would be to adopt a self-consistent scheme to partition the macroscopic strain among the unit cells. In a self-consistent scheme, the average strain of each unit cell would be determined by treating it as an isolated inclusion in an infinite medium having the a-priori unknown

properties of the whole aggregate. Such an approach would also bring about a large computational cost. Moreover, since periodic conditions are used in each unit cell, the local fields at interfaces between zones with particles oriented differently are not accounted for. The model also does not capture long range effects such as shear band development at meso or macro-scale. Furthermore, some aspects are not taken into account in this study, although they can be addressed in the framework of the model. For instance, the previously mentioned initial accumulation of dislocations at interfaces due to austenite-to-martensite transformation and the associated residual stress and gradient of hardness were not taken into account in this analysis. The presence of residual stress resulting from processing can be simulated by subjecting the unit cells to inhomogeneous thermal dilation mimicking the transformation strain, e.g. (Deng, 2009; Lingamanaik and Chen, 2011) (see Appendix B). The effect of chemical inhomogeneities on the mechanical properties could also be easily incorporated by using spatially dependent hardening properties. The effect of martensite connectivity could be investigated at any low to moderate volume fraction, for example by considering percolating star-like network (e.g. Bugat *et al.*, 1999) of martensite within the unit cell (see Appendix B). For instance, Landron *et al.* (2012) observed islands of interconnected martensite in a steel with a martensite volume fraction equal to 11% using holotomography. In order to account for the refinement effect, a strain gradient plasticity model could be adopted, see e.g. (Mazzoni-Leduc *et al.*, 2008; Pardoën and Massart, 2012). Finally, one could consider using crystal plasticity theory to describe the ferrite and martensite constitutive behavior, instead of J_2 plasticity theory. Choi *et al.* (2013) and Pagenkopf *et al.* (2016) have compared FE calculations with an isotropic model and a crystal plasticity model, respectively, and found that the computed local stress fields were significantly different. Varying the crystallographic orientations from grain to grain would introduce even more heterogeneities, with probable impact on the damage behavior. However, there is no reason to expect that the use of more refined constitutive models would change the conclusion that particle shape has a limited impact on the elastoplastic behavior in the case of uniform orientation distribution and absence of change of the internal microstructural length.

4.4 Conclusions and perspectives

Early works addressing the modeling of the link between microstructure and mechanical properties of Dual-Phase steels focused mainly on martensite volume fraction and on carbon content and rarely on particle morphology. The aim of this work was to shed light on the impact of particle morphology and orientation on the plastic behavior and damage of Dual-Phase steels in order to ultimately help in the prediction of mechanical properties and guide the processing of improved steels. The conclusions may nevertheless be applied to other metals comprising two phases of different hardness.

The modeling strategy is based on a two-level unit cell computational approach, which constitutes an efficient tool to study local and effective behavior while avoiding computationally intensive full FE simulations on multiparticle RVEs. The main findings are the following:

- Regardless of the value of the particle aspect ratio, unit cells loaded at a 45° angle relative to the revolution axis exhibit the highest uniform elongation and lowest tensile strength. Loading along 0° or a 90° orientation leads to the opposite trend.
- The effect of the loading angle on the mechanical properties (i.e. the anisotropy) increases as the aspect ratio of the particle differs from 1. In the case of a ferritic matrix (martensite volume fractions lower than 50%), the tensile strength increases and the uniform elongation decreases as the aspect ratio departs from 1. In the case of a martensitic matrix (martensite volume fractions larger than 50%), the opposite trend is observed.
- Even though the impact of particle aspect ratio is significant at a given loading angle, the particle shape has relatively limited impact on the effective behavior of an aggregate of grains with a random distribution of particle orientations. The effects found experimentally are presumably related to a microstructure refinement effect.

These results suggest that producing microstructures with preferential orientation of the second phase (for example by cold rolling) could be used to act on both ductility and strength. Despite having a small effect on plastic behavior of the aggregate, the morphology, orientation and martensite volume fraction largely influence the local mechanical fields and, thereby, lead to important stress and strain inhomogeneities, which control the damage process. We anticipate that damage in Dual-Phase steels with 'Thomas fibers' microstructures develop in a two-stage process, first in some grains with specific orientation leading to fast local coalescence events, followed by larger-scale coalescence between damaged grains. This two-stage process might be the reason for the good cracking resistance of Dual-Phase steels with a platelet-like microstructure.

Chapter 5

Damage and cracking resistance characterization of platelet-like Dual-Phase steels

5.1 Introduction

The cracking resistance of materials is directly related to the damage behavior, and the damage mechanisms are very much connected to several microstructural features.

The objective of this chapter is to unravel the mechanisms governing damage and leading to the superior cracking resistance of ‘Thomas fibers’ microstructures compared to traditional equiaxed microstructures. In order to do so, both types of morphologies were systematically investigated and compared in terms of damage and cracking resistance.

In addition to the thin sheet microstructures described in Chapter 3, thick Dual-Phase steels with a platelet-like morphology from an earlier study (*Pierman, 2013; Pierman et al., 2014*) will also be considered in this chapter. The latter are described in Section 5.2 that also presents the experimental procedures to quantify the crack propagation resistance and addresses the procedure used to characterize the damage mechanisms. The results for all microstructures are described in Section 5.3 and discussed in Section 5.4 before concluding.

5.2 Materials and experimental procedures

5.2.1 Materials and processing

In addition to the thin sheets investigated in this study (see Chapter 3), three grades, given in Table 5.1, were used in an earlier study (*Pierman, 2013; Pierman et al., 2014*) to generate four Dual-Phase steels in the form of 10 mm thick samples. Given their similar microstructures compared to the thin sheets studied in this thesis (see Chapter 3), relevant results about the thick version are recalled, based on the work of (*Pierman, 2013*).

wt.%	C	Mn	Si	Cr	Al	P	S
Alloy 1	0.023	2.05	0.31	0.405	0.038	0.012	0.003
Alloy 2	0.045	2	0.3	0.4	0.028	0.013	0.0025
Alloy 3	0.088	2.07	0.305	0.435	0.036	0.0025	0.0025

Table 5.1 Chemical composition (in wt.%) of the as-received steel grades used for the bulk samples.

The heat treatment conditions were not identical to the ones for thin sheets. Different thermal conditions were applied in order to generate similar microstructures as in thin sheet form. The heat treatments conditions used to generate thick sheets made of four different ‘Thomas fibers’ microstructures are given in Table 5.2. The heat treatments took place as previously in a resistive furnace and a fluidized bed furnace. The name of the samples writes ‘bV_yC_z’ with ‘b’ standing for bulk, ‘y’ being the martensite volume fraction in percent, and ‘z’ being the martensite carbon content in percent, considering that all the carbon is located in the martensite. More details about the materials in bulk form can be found in (*Pierman, 2013; Pierman et al., 2014*).

	<i>Austenitization</i>	<i>Intercritical annealing</i>
bV17C0.15 (alloy 1)	1100°C – 15 min	720°C – 20 min
bV21C0.22 (alloy 2)	1100°C – 15 min	735°C-20 min
bV14C0.63 (alloy 3)	1100°C – 15 min	702°C-1380 min
bV32C0.15 (alloy 2)	1100°C – 15 min	760°C-20 min

Table 5.2 Temperature and duration of the applied thermal treatments for thick sheets, each step being ended by water quenching at room temperature.

5.2.2 Microstructures

Figure 5.1 is a panel of SEM micrographs corresponding to the thick sheets microstructures. They are very similar to the thin sheets microstructures, with, however, even finer platelets.

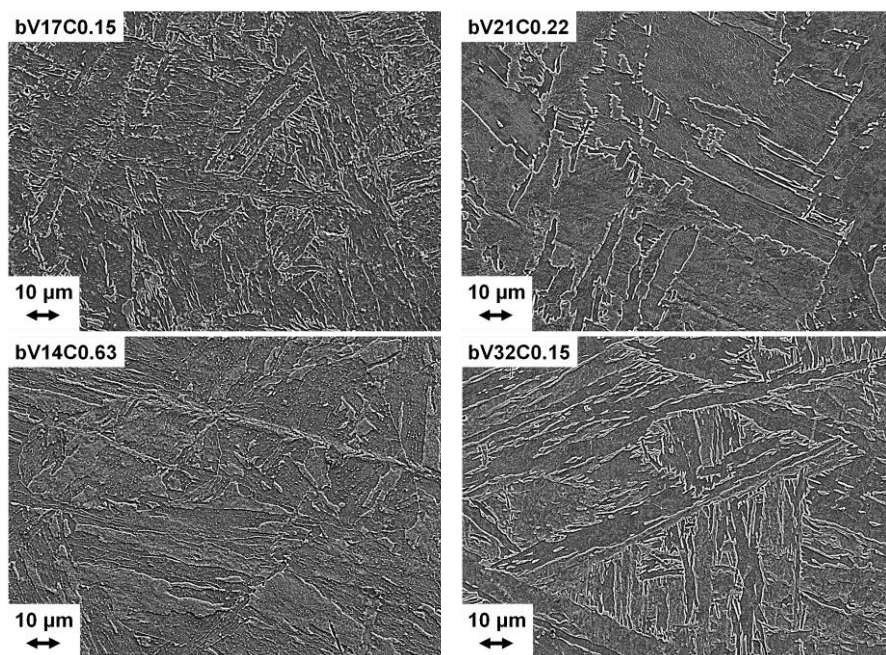


Figure 5.1 SEM micrographs of the thick sheets microstructures.

5.2.3 EWF test procedure

The EWF method (whose concept is explained in Section 2.4.2) was applied on all thin sheets microstructures. In this work, DENT specimens with a length of 100 mm, and a width of 50 mm of the eight thin sheets microstructures were tested parallel to the rolling direction. In order to take into account the effect of the sheet thickness on the crack propagation resistance, one additional thickness equal to 0.5 mm, obtained by cold-rolling as well, was addressed for the platelet-like microstructures and for the equiaxed microstructure with 36% of martensite. The same heat treatments as for pV19t1 and pV19t2, pV36t1 and pV36t2, and eV36t1 and eV36t2 were applied.

In order to fulfill the validity requirements, the ligament length was selected to range from 5 mm to 17.5 mm for the 1 mm thick sheets, from 5 mm to 15 mm for the 0.5 mm thick sheets, and from 9 mm to 15 mm for the 2 mm thick sheets. Electrical discharge machining (Agie-Charmille CUT20-P) was first used to produce the notches with an opening of around 250 to 300 μm . In order to produce a precrack, a razor blade was used at the notch tip, leading to a notch tip radius between 50 and 100 μm . The optical micrograph in Figure 5.2 shows a typical precrack. The use of a razor blade for precracking can affect the microstructure through local work hardening and local damage, which in turn can affect the crack initiation energy. However, as the work dissipated during initiation only mildly contributes to the total work when the ligament length is sufficiently long, the use of a razor blade does not significantly impact the essential work of fracture. Nonetheless, the precrack tip radius must be inferior to the CTOD of the material to ensure a measurement that is not dependent on the initial opening. This has been verified to be the case in thick sheets samples (see later) by *Pierman (2013)* and assumed to be the case in thin sheets samples of comparable microstructure, which thus avoided the time-consuming fatigue precracking.

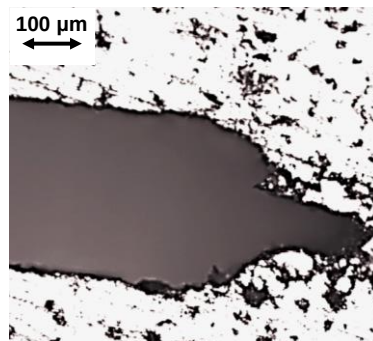


Figure 5.2 Typical precrack introduced using a razor blade.

The specimens used for the EWF method were deformed in tension until completion of cracking using a screw driven universal testing machine (Zwick-Roell 50 kN) at a crosshead velocity of 1 mm/min. Displacements were measured by means of an extensometer with an initial gauge length between 25 and 50 mm. The standard deviation of the w_e values is calculated from the least square principles. For a linear regression $y = ax + b$, the standard deviation s_b of the intercept for $x = 0$ is equal to (*Martin et al., 2012*)

$$s_b = \left[\frac{\sum_{i=1}^n (y_i - \hat{y}_i)^2 \sum_{i=1}^n x_i^2}{n(n-2) \sum_{i=1}^n (x_i - \bar{x})^2} \right]^{1/2} \quad (5.1)$$

where n is the number of (x_i, y_i) data, $\bar{x}$ is the mean of the x values, and $\hat{y}_i$ are the values of y_i on the regression line at the corresponding value of x_i (i.e. fitted y_i values).

The essential work can be further separated into the contribution of necking βw_n and of damage Γ_0 by a linear regression on the thickness (see Equation 2.21). As the error associated to each (x_i, y_i) point is different, the weight attributed to each point in the linear regression depends on its reliability. The best-fit value of the slope, used for βw_n in the regression of the essential work of fracture on the thickness, is given by

$$\text{slope} = \frac{\left(\sum_{i=1}^n \frac{x_i}{s_b^2} \right) \left(\sum_{i=1}^n \frac{y_i}{s_b^2} \right) - \left(\sum_{i=1}^n \frac{x_i y_i}{s_b^2} \right) \left(\sum_{i=1}^n \frac{1}{s_b^2} \right)}{\left(\sum_{i=1}^n \frac{x_i}{s_b^2} \right)^2 - \left(\sum_{i=1}^n \frac{x_i^2}{s_b^2} \right) \left(\sum_{i=1}^n \frac{1}{s_b^2} \right)}. \quad (5.2)$$

The intercept of the linear regression, used here for Γ_0 , is given by

$$\text{intercept} = \frac{\left(\sum_{i=1}^n \frac{x_i}{s_b^2} \right) \left(\sum_{i=1}^n \frac{x_i y_i}{s_b^2} \right) - \left(\sum_{i=1}^n \frac{y_i}{s_b^2} \right) \left(\sum_{i=1}^n \frac{x_i^2}{s_b^2} \right)}{\left(\sum_{i=1}^n \frac{x_i}{s_b^2} \right)^2 - \left(\sum_{i=1}^n \frac{x_i^2}{s_b^2} \right) \left(\sum_{i=1}^n \frac{1}{s_b^2} \right)}. \quad (5.3)$$

The error associated to the slope is given by

$$\text{error}_{\text{slope}} = \sqrt{\frac{\left(\sum_{i=1}^n \frac{1}{s_b^2} \right)}{\left(\sum_{i=1}^n \frac{x_i}{s_b^2} \right) \left(\sum_{i=1}^n \frac{1}{s_b^2} \right) - \left(\sum_{i=1}^n \frac{x_i^2}{s_b^2} \right)^2}}. \quad (5.4)$$

And, finally, the error associated to the intercept is given by

$$\text{error}_{\text{intercept}} = \sqrt{\frac{\left(\sum_{i=1}^n \frac{x_i^2}{s_b^2} \right)}{\left(\sum_{i=1}^n \frac{x_i}{s_b^2} \right) \left(\sum_{i=1}^n \frac{1}{s_b^2} \right) - \left(\sum_{i=1}^n \frac{x_i^2}{s_b^2} \right)^2}}. \quad (5.5)$$

5.2.4 Determination of the CTOD

The Crack Tip Opening Displacement (CTOD) at initiation δ_c , which is the critical opening at which the crack starts propagating, was measured by *Pierman (2013)* using the methodology proposed by (*Pardoen and Delannay, 2000*). It consists in measuring the difference between the total crack opening after blunting and the opening due to crack propagation. Following the measurement of this value for different crack extensions, an extrapolation for a crack extension equal to zero leads to the δ_c . δ_c is related to the fracture toughness J_{Ic} through the following relationship

$$\delta_c = d \frac{J_{Ic}}{\sigma_0} \quad 5.6$$

where d is the Shih factor (*Shih and German, 1981*). This method was applied to the thick specimens of *Pierman (2013)* to estimate J_{Ic} .

5.2.5 Hole expansion tests

Hole expansion tests were performed to assess the stretch flangeability of eV19t1, eV36t1, pV19t1 and pV36t1 thin sheets. The specimens were first punched with holes of 10 mm diameter and a clearance of 12%. The specimens were then clamped with a force of 150 kN in a blank holder and the holes were expanded by a 60° conical punch moving at a speed of 0.8 mm/s. Figure 5.3 provides a schematic of the hole expansion ratio test. The tests were stopped as soon as a crack through the thickness of the sample was detected. For each sample, the inner diameters of the expanded hole were measured in two directions perpendicular to each other, while avoiding cracks. Using the average diameter of the expanded hole $\overline{D_h}$, the hole expansion ratio λ expressed in percent was calculated as

$$\lambda = 100 \frac{\overline{D_h} - D_0}{D_0} \quad 5.7$$

where D_0 is the initial hole diameter. The hole expansion ratios are the result of an averaging on seven specimens.

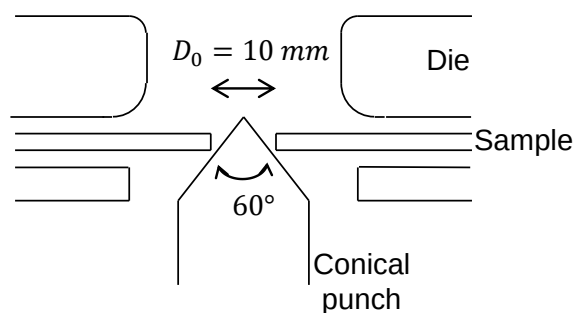


Figure 5.3 Hole expansion ratio test – principle.

5.2.6 Damage characterization

The broken uniaxial tension and DENT specimens were prepared for damage characterization starting by SEM observation of the fracture surfaces. The samples were then cut at mid-width (Struers Accutom-5) and mounted to allow transverse observations on sections below the fracture surface as shown in Figure 5.4. The surface was polished down to $1\text{ }\mu\text{m}$, followed by an OPA polishing step of 20 min and by an ultrasonic bath (BRANSON 1200) of 10 min to remove the potential residues. OPA was occasionally sufficient to reveal the microstructure using SEM. In case of a too thick passivation layer, the sample was etched using a Nital solution.

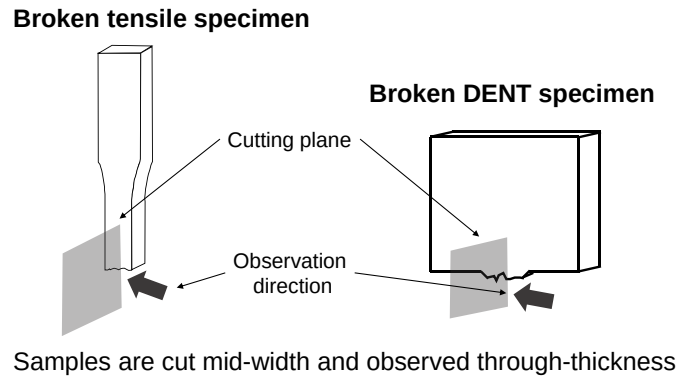


Figure 5.4 Schematic representation of the damage characterization procedure in broken uniaxial tensile and DENT specimens.

5.3 Results

5.3.1 Uniaxial tensile test results

Following the procedure explained in Section 3.4.2 for uniaxial testing, Figure 5.5(a) recalls the mean true stress – true strain curves of the thin sheets of equiaxed and platelet-like Dual-Phase steels obtained as an average of 2 to 5 replicas (it is an average of all curves, which can be found in Section 3.4.3). Full lines represent 1 mm thick specimens; dotted lines represent 2 mm thick specimens. Figure 5.5(b) shows representative stress-strain curves of the thick sheets steels up to the onset of necking. The original curves can be found in (*Pierman et al., 2014*) and in (*Pierman, 2013*).

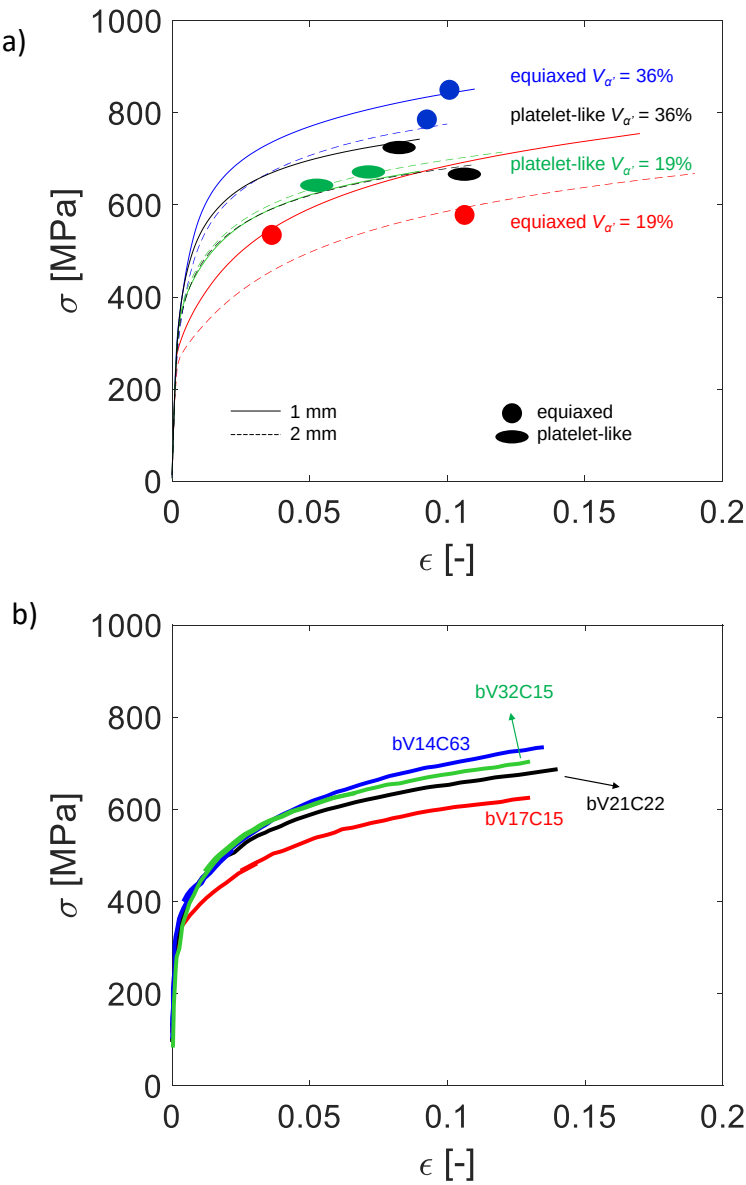


Figure 5.5 Experimental true stress-true strain curves of the steels for the a) thin sheets, and b) thick sheets. (data taken from (Pierman et al., 2014))

Table 5.3 summarizes the main mechanical properties of the thin sheet microstructures extracted from these curves as well as ϵ_f , σ_f and the product $\epsilon_f \sigma_f$. The values available for the thick sheets microstructures are added in Table 5.3 as well.

	σ_0 [MPa]	ϵ_u [-]	σ_u [MPa]	ϵ_f [-]	σ_f [MPa]	$\epsilon_f \sigma_f$ [MPa]
eV19t1	311 ± 4	0.17 ± 0.01	752 ± 6	0.765 ± 0.007	1497 ± 52	1075 ± 160
eV36t1	417 ± 9	0.11 ± 0.01	854 ± 13	0.88 ± 0.096	1953 ± 163	1733 ± 319
pV19t1	396 ± 10	0.09 ± 0.01	700 ± 27	1.04 ± 0.023	1765 ± 175	1852 ± 212
pV36t1	415 ± 7	0.09 ± 0.005	741 ± 11	1.06 ± 0.12	1984 ± 98	2105 ± 343
eV19t2	274 ± 4	0.19 ± 0.0002	666 ± 4	0.935 ± 0.025	1561 ± 37	1460 ± 75
eV36t2	367 ± 9	0.1 ± 0.02	775 ± 40	1.08 ± 0.156	2117 ± 235	2264 ± 422
pV19t2	385 ± 2.5	0.12 ± 0.005	716 ± 9	0.76 ± 0.04	1618 ± 137	1239 ± 173
pV36t2	369 ± 5	0.11 ± 0.02	688 ± 26	1.09 ± 0.09	1820 ± 110	1982 ± 271
bV17C15	342 ± 2	0.13 ± 0.013	626 ± 10	1.54 ± 0.1		
bV21C22	345 ± 10	0.14 ± 0.03	687 ± 20	1.2 ± 0.15		
bV14C63	494 ± 25	0.135 ± 0.005	735 ± 10	1 ± 0.04		
bV32C15	351 ± 75	0.13 ± 0.01	704 ± 20	1.21 ± 0.21		

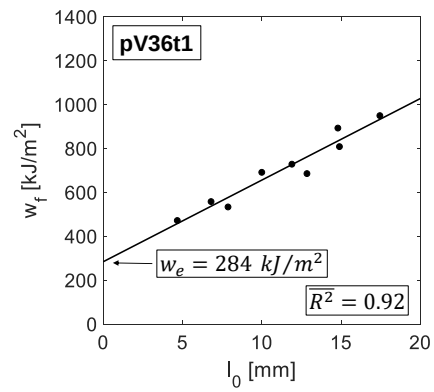
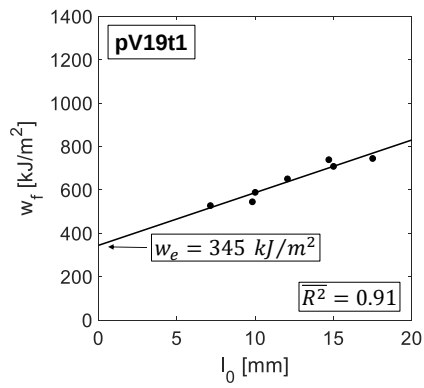
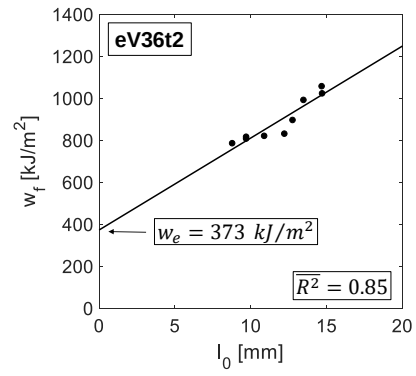
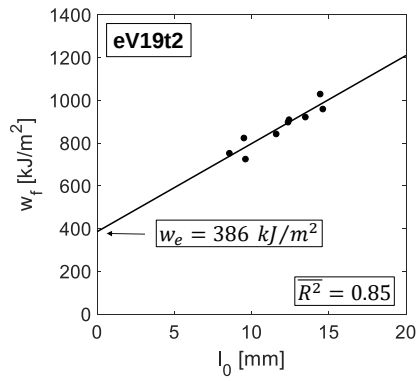
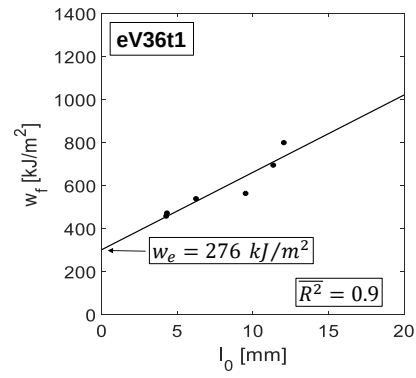
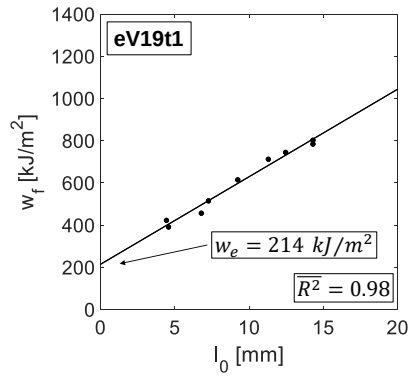
Table 5.3 Tensile properties of the steels.

The hardening at small strains is higher in a platelet-like microstructure compared to an equiaxed microstructure at a martensite volume fraction of 19%, but the opposite is found at 36% of martensite. The fracture strain is higher for the platelet-like microstructure, except for the eV19t2 - pV19t2 case. The yield and tensile stresses increase with martensite volume fraction. The 1 mm thick sheets generally exhibit a strength around 10% higher compared to the 2 mm thick sheets at the same martensite volume fraction for equiaxed microstructures, showing that it is difficult to reproduce exactly identical microstructures with different thicknesses.

5.3.2 Essential work of fracture

The load - displacement curves of the DENT specimens are given in Appendix E. When normalized by the maximum force F_{max} and maximum displacement u_{max} , the curves superpose very well, which is a good indication of the validity of the method as it indicates that the mechanisms remain the same when varying the ligament length. 2D digital image correlation was performed as an additional validity check of the EWF method. As needed for a valid result, the plastic zone is confined between the two notches and the entire ligament deforms plastically during the test (see Appendix E for more information).

Figure 5.6 shows the variation of w_f with respect to l_0 and the linear regressions used to determine the essential work of fracture values, together with the regression coefficient $\overline{R^2}$. Some results must be interpreted with care, namely the cases pV19t2, pV36t0.5 and eV36t1. Indeed, in these cases, either the regression coefficient is small and/or the number of samples is more limited and/or the samples with the largest ligament length are missing.



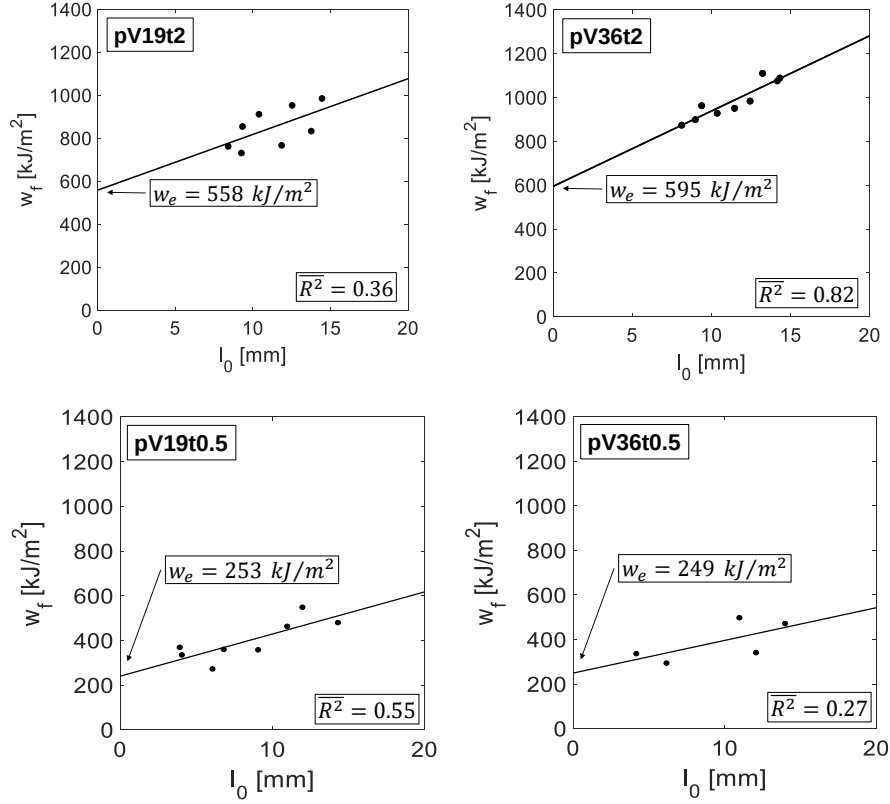


Figure 5.6 w_f as a function of l_0 , together with the w_e and the quality of the linear regressions as quantified by R^2 .

Figure 5.7 shows the variation of w_e as a function of the thickness for the different microstructures. As expected, the essential work of fracture significantly increases with thickness for a given martensite volume fraction and morphology.

For a given martensite volume fraction and thickness, the essential work of fracture of the platelet-like microstructure is significantly larger than for the corresponding equiaxed morphology. As a remark, the values for a platelet-like microstructure and a thickness equal to 1 mm and 2 mm also correspond to the ones indicated in Figure 1.4 with red bullets.

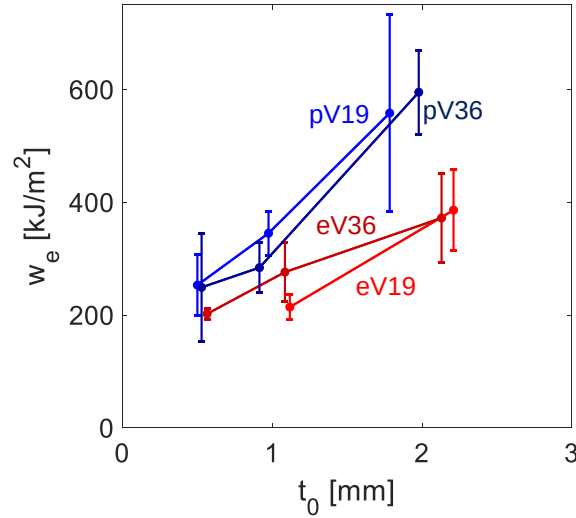


Figure 5.7 Essential work of fracture w_e as a function of initial sheet thickness t_0 .

Based on Equation 2.21 and using Equations 5.2 to 5.5 for the linear regression on the thickness, Table 5.4 provides separately the contribution of necking βw_n and of damage Γ_0 to the essential work of fracture, together with the associated errors. This separation is only semi-quantitative in view of the limited number of thicknesses investigated but the main trends are worth exploring.

	Γ_0 [kJ/m ²]	βw_n [kJ/m ³]
eV19	38 ± 86	157 ± 69
eV36	137 ± 29	115 ± 46
pV19	140 ± 98	215 ± 111
pV36	56 ± 87	267 ± 72

Table 5.4 Γ_0 and βw_n of equiaxed and platelet-like microstructures.

Table 5.4 shows that the βw_n values are higher in platelet-like microstructures. This means that the work of necking increases a lot with thickness in platelet-like microstructures. The error on Γ_0 is too large to allow any quantitative conclusions to be taken. This error propagates from the error already significant on some w_e values. In addition, the small differences between the alloys as a function of the thickness also contributes to the dispersion.

5.3.3 Fracture toughness of thick sheets

The fracture toughness J_{Ic} of the four thick sheets microstructures was determined by *Pierman (2013)* from the measurement of the δ_c using Equation 5.6. Table 5.5 gathers the measured crack tip opening displacement, the Shih factor, the yield strength and the estimated J_{Ic} . In order to assess the error associated to the J_{Ic} , the experimental error due to the measurement of the critical opening is estimated to be around 50 microns and the error of the yield stress is the standard deviation following the uniaxial tensile tests. The error associated to the J_{Ic} is then computed using standard error propagation. These results confirm the higher cracking resistance of Dual-Phase steels with platelet-like α' morphology as the level of cracking resistance is higher than what is commonly encountered in the literature for Dual-Phase steels (see Section 2.4.3).

	$\delta_c [\mu m]$	$d [-]$	$\sigma_0 [MPa]$	$J_{Ic} [kJ/m^2]$
bV17C15	800 ± 50	0.68	342 ± 2	402 ± 21
bV21C22	850 ± 50	0.75	345 ± 10	391 ± 26
bV14C63	612 ± 50	0.7	494 ± 25	432 ± 42
bV32C15	592 ± 50	0.71	351 ± 75	351 ± 67

Table 5.5 Fracture toughness J_{Ic} and values of the parameters used for the determination of J_{Ic} for the four thick sheets microstructures.

5.3.4 Hole expansion tests

Figure 5.8 provides the hole expansion ratio values for the eV19t1, eV36t1, pV19t1, pV36t1 steels as a function of the essential work of fracture. The values of the hole expansion ratio for the steels exhibiting a platelet-like microstructure are higher compared to the steels with an equiaxed microstructure, as expected from the higher cracking resistance of platelet-like Dual-Phase steels.

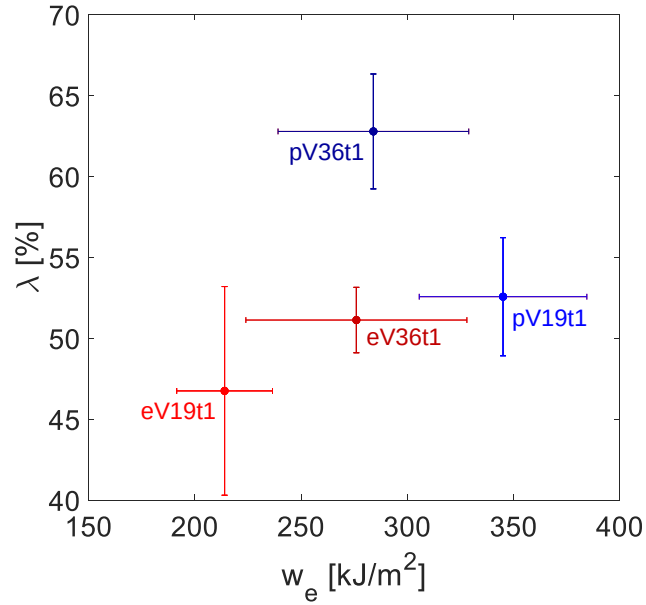


Figure 5.8 Hole expansion ratio values λ as a function essential work of fracture for the eV19t1, eV36t1, pV19t1 and pV36t1 steels.

5.3.5 Damage characterization

5.3.5.1 Fractography

Figure 5.9 shows SEM micrographs of the fracture surfaces of uniaxial tension specimens of the eight thin sheets microstructures.

The fracture surface of the thin sheet steels pV19t1 and pV36t1 deformed in uniaxial tension exhibit a slant mode. This explains the distorted shape of the dimples for these two conditions. The steels eV19t1, eV19t2, eV36t1, eV36t2, pV19t2 and pV36t2 break by a flat fracture mode.

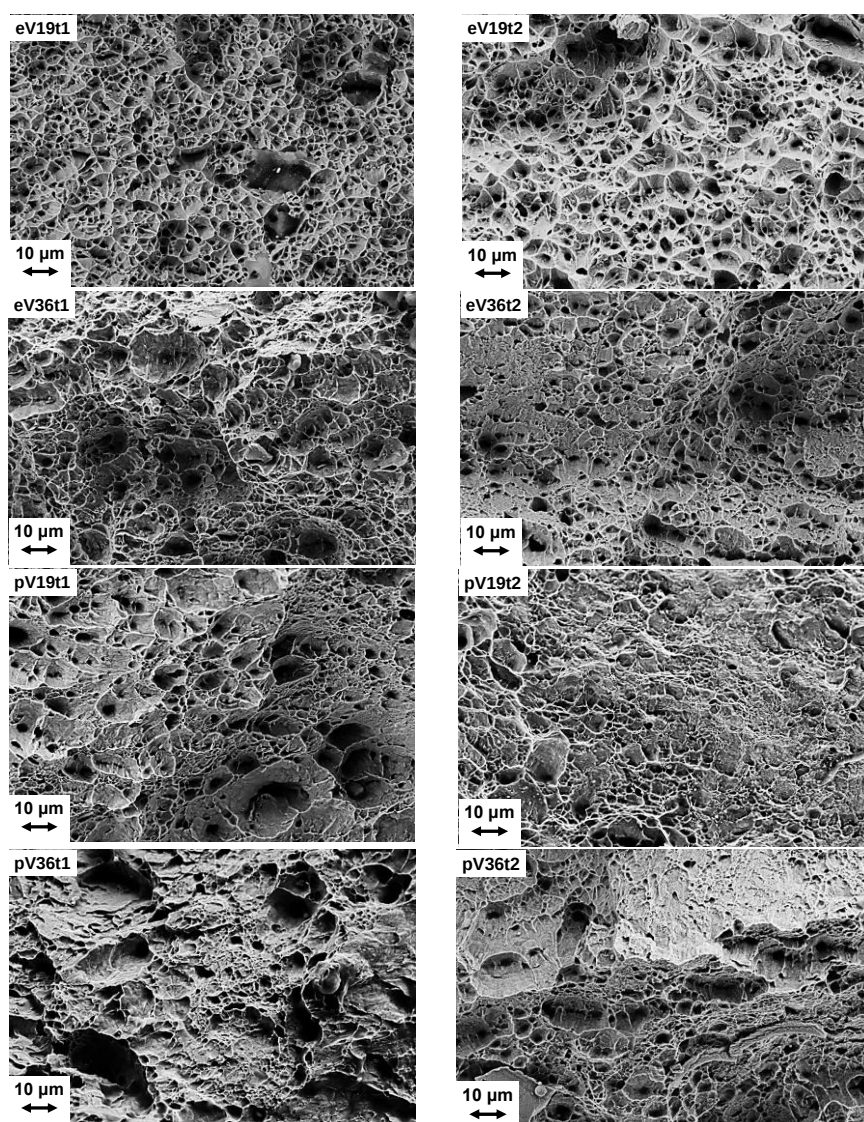


Figure 5.9 Fracture surfaces of uniaxial tension specimens for the eight thin sheets microstructures.

The SEM micrographs exhibit classical dimples showing that all eight microstructures fail by ductile fracture through a process of nucleation, growth and coalescence of primary voids. There are significant differences though between the equiaxed and the platelet-like microstructures. Equiaxed microstructures exhibit homogeneous dimple sizes. The size of the dimples is smaller for the 1 mm thickness, corroborating the smaller grain size of the 1 mm thick sheets microstructures (see Chapter 3). In platelet-like

microstructures, some flat zones with fewer dimples are present, in which voids tend to have a parabolic shape indicating a local shearing mechanism (e.g. *Barsoum and Faleskog, 2007*). Void sizes are thus less homogeneous compared to equiaxed microstructures. In DENT specimens, for a given material, the micrographs are very similar to the fracture surfaces of tensile specimens and indicate a ductile mode of crack propagation as well. This is shown in Figure 5.10, which is a panel of fracture surfaces on DENT specimens obtained by SEM corresponding to the eight thin sheets microstructures.

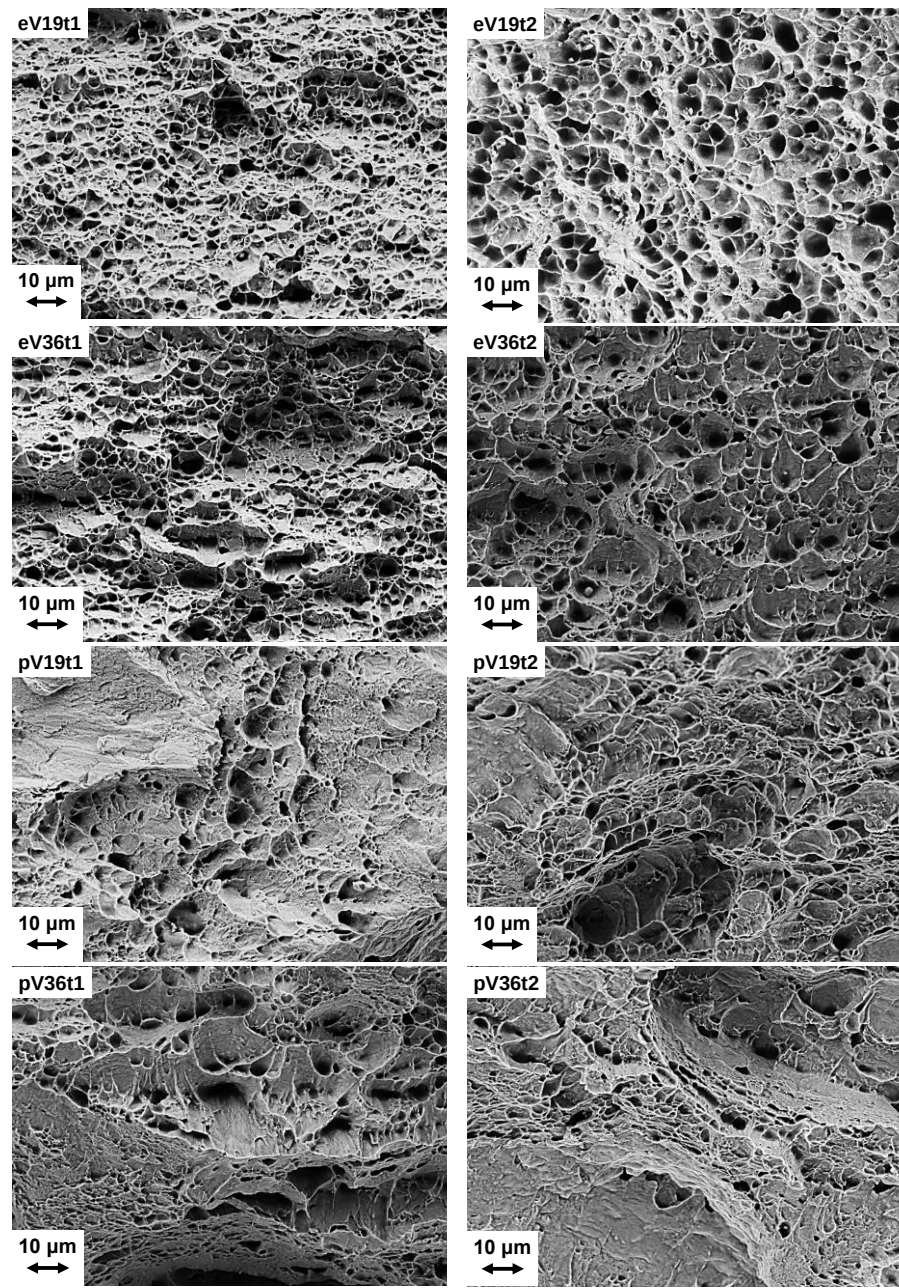


Figure 5.10 Fracture surfaces of DENT specimens for the eight thin sheets microstructures.

The thickness reduction factor r_f and the average equivalent fracture strain $\varepsilon_{f,eq}$ of the DENT specimens for the eight thin sheets microstructures are given in Table 5.6. They are computed as

$$r_f = \frac{t_0 - t_f}{t_0}, \quad (5.8)$$

$$\varepsilon_{f,eq} = \frac{2}{\sqrt{3}} \ln \left(\frac{t_0}{t_f} \right) \quad (5.9)$$

where t_0 is the initial thickness of the DENT specimen and t_f is the thickness after necking during crack propagation in the DENT specimen.

	r_f [-]	$\varepsilon_{f,eq}$ [-]
eV19t1	0.52	0.84
pV19t1	0.61	1.07
eV36t1	0.62	1.11
pV36t1	0.61	1.09
eV19t2	0.44	0.67
pV19t2	0.53	0.87
eV36t2	0.48	0.76
pV36t2	0.59	1.04

Table 5.6. Thickness reduction factor r_f and average equivalent fracture strain $\varepsilon_{f,eq}$ of the DENT specimens for the eight thin sheets microstructures.

Table 5.6 shows that the thickness reduction and the fracture strain are higher in platelet-like microstructures compared to equiaxed microstructures, except for the steels with 36% of martensite and a sheet thickness equal to 1 mm. The eV36t1 steel moreover corresponds to the especially high essential work of fracture in equiaxed microstructures.

Figure 5.11 shows a comparison of macro fracture surfaces between an equiaxed (eV19t1) and a platelet-like (pV19t1) microstructure. It shows that the platelet-like microstructure exhibits a rougher and more heterogeneous fracture surface than the equiaxed microstructure.

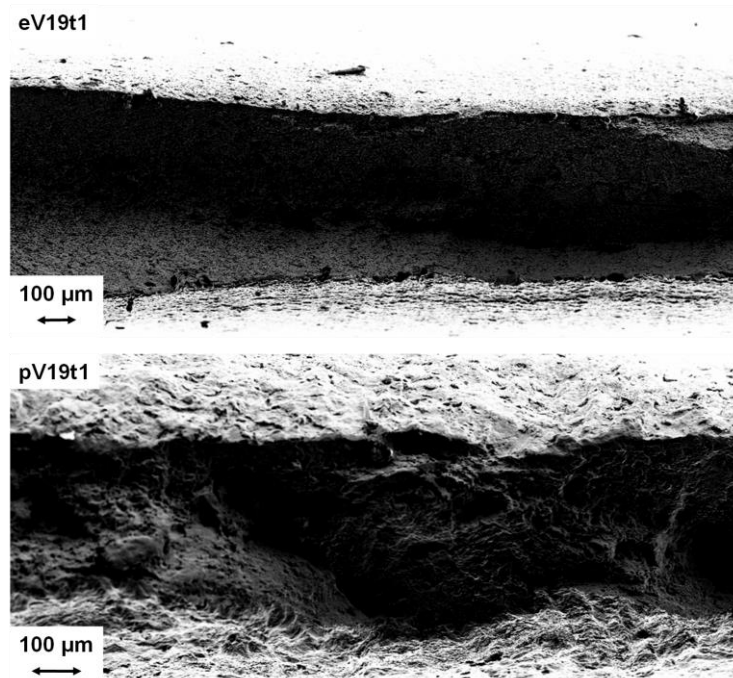


Figure 5.11 Fracture surfaces at the macro-scale of a eV19t1 DENT specimen and of a pV19t1 DENT specimen.

5.3.5.2 Damage characterization

Figure 5.12 shows a comparison of the transverse sections extracted below the fracture surface for an equiaxed and a platelet-like microstructure. Platelet-like microstructures show a much lower density of voids than equiaxed microstructures (a similar observation was made by (Pierman, 2013)). Even just below the fracture surface where the strains are the highest, the number of voids remains small. This shows that the platelet-like microstructure is much more resistant to void nucleation with a much smaller volume fraction of voids due to both a smaller number of voids and a smaller size. Indeed, the voids are generally of sub-micron size, with dimensions around a few hundreds of nanometers. This is a very important and partly unexpected finding of this study. The voids in equiaxed microstructures are significantly elongated in the direction of loading indicating an early nucleation.

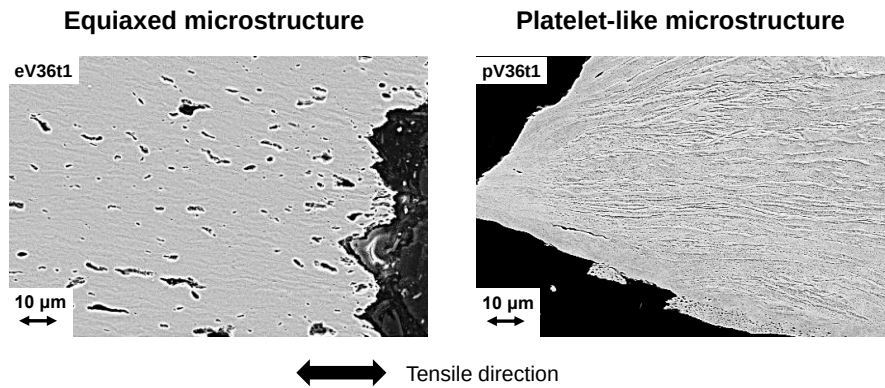


Figure 5.12 SEM micrograph of a transverse section of a uniaxial tension specimen of an equiaxed microstructure (eV36t1) and of a platelet-like microstructure (pV36t1).

Figure 5.13 shows that both mechanisms of nucleation by ferrite-martensite interface decohesion and martensite particle fracture occur in the investigated steels. However, depending on the microstructure, one mechanism dominates over the other. In equiaxed microstructures, voids nucleate primarily by particle breaking. Nonetheless, on many occasions, the voids develop along ferrite-martensite interfaces, as observed as well by (*Pierman, 2013*), due to the stress concentrations at the interfaces. In platelet-like microstructures, voids nucleate primarily by interface decohesion at the tip of a particle, on the interface perpendicular to the loading direction (as the particle is aligned with the loading), see Chapter 4. Just as it is the case for equiaxed microstructures, voids nucleate more often in zones in which the martensite density is large as these zones are more spatially, and thus mechanically, constrained. A particular case of constrained zone is the ferrite between two closely spaced martensite particles. This is besides what has been modeled in Chapter 4 due to the periodicity of the unit cells. Void nucleation by particle failure occurs far in the plastic regime. Indeed, high distortions are often visible. The voids grow in the direction of loading. Similarly to the case of equiaxed microstructures, the void grows by decohesion when a martensite particle is nearby in the direction of growth.

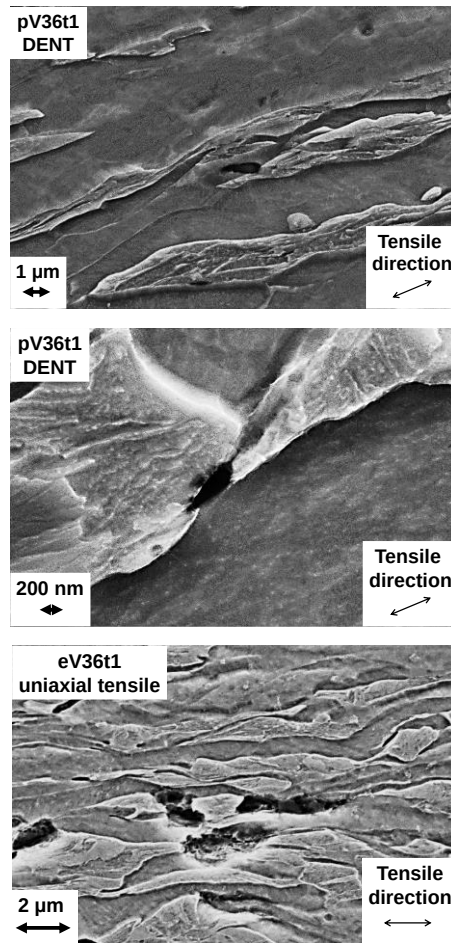


Figure 5.13 SEM micrographs of transverse sections right below the fracture surface showing: interface decohesion in a pV36t1 DENT specimen, particle breaking in a pV36t1 DENT specimen, interface decohesion and particle breaking in a eV36t1 uniaxial tension specimen.

An important final finding for platelet-like microstructures is shown and schematized in Figure 5.14. The martensite platelet particles are randomly oriented away from the fracture surface (Figure 5.14(b)). But, when moving towards the fracture surface, there is a progressive alignment of the particles with the loading direction. Below the fracture surface, the particles are almost completely aligned in the tensile direction as shown in Figure 5.14(c). Figure 5.14(d) also shows that in the event of a region fracturing under large shear, in addition to aligning with the tensile direction, the particles rotate in the shear direction.

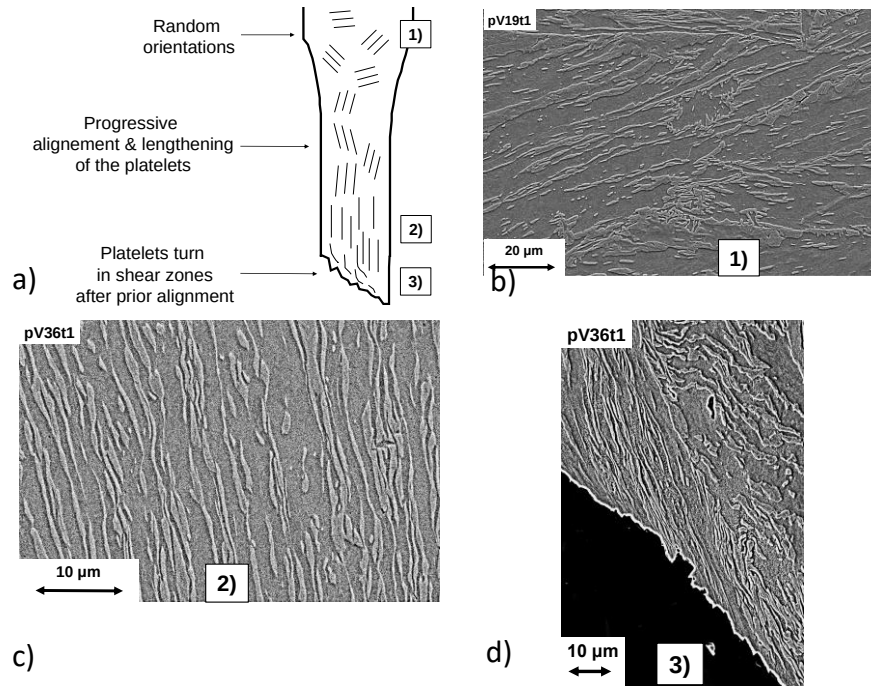


Figure 5.14 Schematic illustration in a uniaxial tension specimen of the alignment of martensite particles with loading direction in a platelet-like microstructure (a), and examples of corresponding SEM micrographs: away from the fracture surface (b), close from the fracture surface (c), and in a shear zone (d).

5.4 Discussion

The platelet-like microstructure leads to a larger cracking resistance compared to the equiaxed microstructure and the effect is amplified for thicker plates. This finding is confirmed using the hole expansion test, which depends on the resistance to cracking from preexisting edge defects. When looking at the behavior under uniaxial tension, there is obviously no correlation to be expected between w_e or J_{Ic} and the product of uniform elongation and tensile strength as uniform elongation quantifies resistance to plastic localization and not cracking resistance.

However, there is a direct correlation between the deformation capacity of the steels quantified through the average equivalent fracture strain $\varepsilon_{f,eq}$ and the cracking resistance quantified through the essential work of fracture w_e . In order to support this correlation, Figure 5.15 provides the ratio of the

essential work of fracture in a platelet-like microstructure and in an equiaxed microstructure, for a fixed martensite volume fraction and sheet thickness as a function of the square of the corresponding ratio of the average equivalent fracture strains in DENT specimens. It is the square of the ratio that is taken because the energy dissipated scales with the product of deformation and stress, the latter increasing with the former (at very large strains, stress varies almost linearly with strain such that $w_e \propto \epsilon_f \sigma_f \propto \epsilon_f^2$). The errors are computed using standard error propagation.

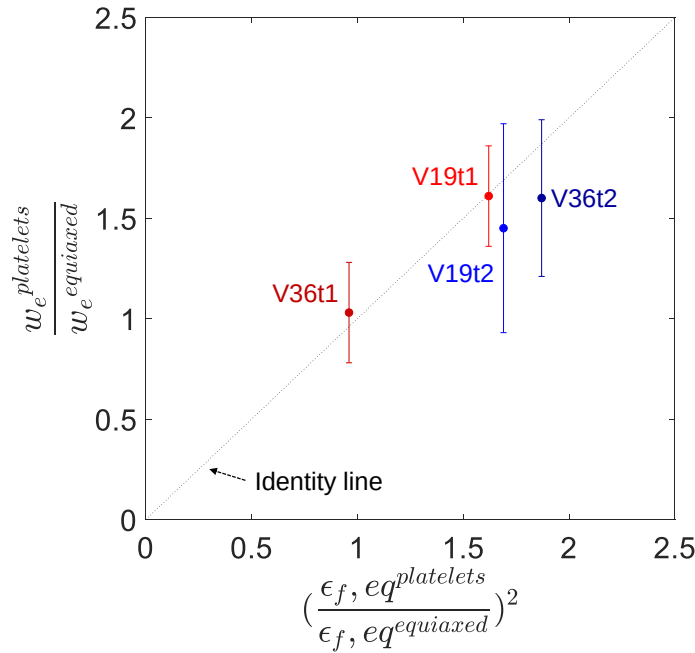


Figure 5.15 Ratio of the essential works of fracture in a platelet-like microstructure and in an equiaxed microstructure as a function of the square of the corresponding ratio of the average equivalent fracture strains in DENT specimens, for a given martensite volume fraction and sheet thickness.

The main reason explaining the better cracking resistance in platelet-like microstructures is thus their ability to reach high thickness reduction during crack propagation, and thus high stresses contributing to the dissipated energy. This is in turn the result of the smaller propensity of the platelet-like microstructure to damage, both in number and size of voids.

One of the origins of the excellent void nucleation resistance of the platelet-like microstructures is related to the mechanism of particle

alignment. The alignment of the martensite particles with the loading direction decreases the fraction of ferrite-martensite interface area in the direction perpendicular to the main loading. Since voids nucleate mainly by interface decohesion in platelet-like microstructures, the resulting configuration is the least propitious for damage as the weakest spots for void nucleation are on the interfaces that are perpendicular to loading. The alignment possibly also leads to an increase of strain hardening at large strains, after the reorientation of the particles, increasing even more the dissipated energy.

This alignment is made possible by the reorientation of the martensite particles owing to the shearing of the softer ferrite phase. But the alignment is also partly due to the ability of the particles to deform to large strains without breaking. Much earlier void nucleation would of course not allow this mechanism to be at play. The additional rotation of the particles in shear zones (see Figure 5.14) reveals how far this rotation and alignment with the load mechanism can go, despite the high hardness of the particles. This ability to accommodate a high deformation is most probably due to the small section of the martensite particles in platelet-like microstructures. A final reason, directly related to the first one, for which only small voids are observed even at high strains, is the slow rate of void growth. Indeed, the voids in platelet-like microstructures have a size of a few hundreds of nanometers only. Strain gradient plasticity arguments prove that the growth rate of sub-micron-sized voids can be much lower than the one of large voids (*Liu et al, 2003; Tvergaard and Niordson, 2004; Liu et al., 2005; Borg et al., 2008*).

A possible second order effect for the large cracking resistance of the platelet-like microstructures that also ensues from the analysis of the damage mechanisms is the existence of a large characteristic length associated to the cracking process. Seeing that the particles are oriented randomly prior to straining, zones in which the particles are unfavorably oriented with respect to loading undergo large strains during deformation. Conversely, zones in which particles are favorably oriented undergo less severe deformation. The latter zones thus contribute to a lesser extent to the energy dissipated than zones in which particles are unfavorably oriented. The resulting heterogeneous deformation potential leads to a heterogeneous

damage accumulation at the scale of the zones with similar initial martensite platelet orientation. Such zones correspond to the martensite packets arising from the first step of the heat treatment and are of the order of 100 μm . Based on this, the two-stage crack propagation process proposed in Chapter 4 and schematically described in Figure 5.16, can also contribute to the high cracking resistance. The two stages are: (i) a first level intragrain crack propagation by void coalescence associated to the scale of particle spacing, of the order of one or a few microns, and (ii) a second level intergrain crack percolation associated to the scale of the martensite packet size. This two-stage cracking process can be the origin of the rougher fracture surfaces in platelet-like microstructures compared to the equiaxed microstructures (see Figure 5.11). The size of the voids in Figure 5.16 is obviously only schematic, as their real dimensions barely reach the micrometer.

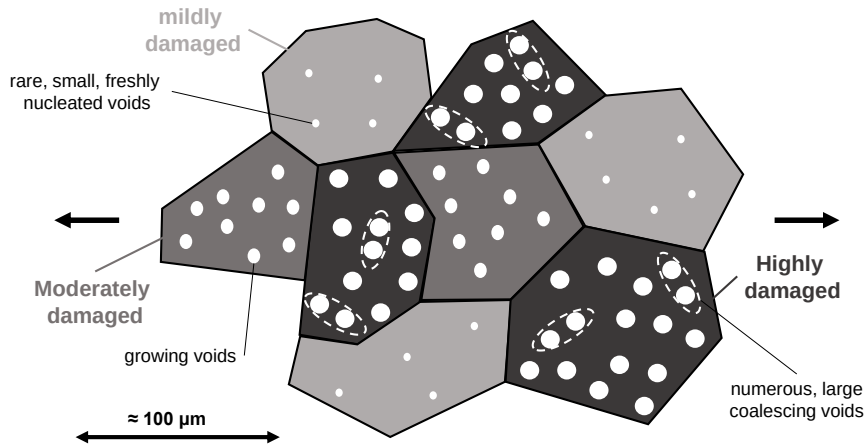


Figure 5.16 Schematic illustration of the heterogeneous damage accumulation in platelet-like microstructures.

The cracking behavior of the steels and the link with an internal length can be, at least partially, rationalized by an analytical model, that will be qualitatively discussed. Based on Equation 2.21 and on the detailed analysis of the work spent for damage given in (*Pardoen et al., 2004*), the essential work of fracture can be expressed as

$$w_e = \Gamma_0 + \Gamma_n = \sigma_0 X_0 F_2(n, f_0) + \beta w_n(n, \sigma_0, \varepsilon_f) t_0 \quad 5.10$$

where σ_0 is the yield stress, X_0 a length related to microstructure that governs the cracking process, and F_2 a function that depends on strain hardening exponent n and initial void volume fraction f_0 .

Figure 5.7 shows that, in all cases, the essential work of fracture w_e increases with initial sheet thickness t_0 . In general, w_e increases with thickness at small thicknesses because of a higher energy dissipated in necking Γ_n , reaches a maximum and then decreases again to a steady value corresponding to the plane strain work of fracture. The steels investigated here are in the range of thicknesses in which w_e increases linearly with t_0 . The work dissipated in necking not only depends on initial thickness but also on βw_n , which is a term taking into account the shape and size of the necking zone (through β) and amount of work spent per unit volume (through w_n). The term βw_n , which quantifies the impact of thickness variation on the energy dissipated, is systematically larger for platelet-like microstructures compared to equiaxed microstructures. It implies a preponderant contribution of necking in platelet-like microstructures and is undoubtedly a reason for the higher essential work of fracture in platelet-like microstructures. It nicely corroborates the analysis of Table 5.6, as when the necking contribution increases, so does the thickness reduction factor and the average equivalent fracture strain. This is taken into account through the factor β that is thus larger for the platelet-like microstructures. Furthermore, as the average equivalent fracture strain $\varepsilon_{f,eq}$ is in most cases larger for the platelet-like microstructures compared to the equiaxed microstructures owing to the alignment mechanism, the plane strain $\varepsilon_f \sigma_f$ product is also larger. And as the $\varepsilon_f \sigma_f$ product is a good indicator of w_n , the term w_n is thus in most cases larger in platelet-like microstructures as well.

Focusing now on the work spent for intrinsic damage Γ_0 , a few plausible interpretations are proposed even though the associated errors are high. The platelet-like microstructures outperform the eV19 case regarding their Γ_0 . This is in line with the previous observations since the Γ_0 also depends on the fracture strain. It is furthermore in agreement with the internal length argument, quantified here through X_0 . However, the eV36 case leads to a surprisingly large Γ_0 , with the eV36t1 series having an especially large essential work of fracture w_e . Consequently, even though the platelet-like

microstructures outperform the equiaxed microstructures regarding their w_e , the I_0 of the equiaxed steels with 36% of martensite is higher. A possible reason is the higher strength of the steels (mostly eV36t1). This would compensate for the lower value of X_0 in equiaxed microstructures compared to platelet-like microstructures. This higher strength of the equiaxed steels with 36% of martensite is maybe due to a higher martensite interconnectivity, leading to a higher deformation accommodation by the hard martensite.

The reasoning above about the analytical model however suffers from some limitations. In addition to the uncertainties related to the experimental procedures, microstructural differences that influence strain hardening and fracture resistance complicate interpretation. For instance, in the analysis of the effect of thickness, as a consequence of controlling the microstructures for martensite volume fraction only, the grain size is smaller at a 1 mm thickness than at 2 mm in equiaxed microstructures, probably because of a higher reduction rate during cold-rolling. The cold-rolling reduction rate is equal to 80% for a 1 mm sheet thickness and to 60% for a 2 mm sheet thickness. This refinement effect in equiaxed microstructures causes the difference in strain hardening between the 1 and 2 mm thick sheets at same martensite volume fraction. Hence, when thickness increases, the yield stress in equiaxed microstructures decreases because of the larger grain size. In platelet-like microstructures, when increasing sheet thickness, the size of the primary austenite grains, and thus of the martensite packets, decreases as a result of a slower heat diffusion during the austenitization. Furthermore, for both microstructure morphologies, the stress triaxiality in the plastic zone in front of the crack tip increases as plane strain contribution increases when going from a thin sheet to a thick sheet. A lower yield stress, a larger stress triaxiality and, as argued here, a smaller martensite packet size, all decrease cracking resistance. Hence, the work spent for damage (thus excluding the necking contribution) is expected to be somewhat lower at higher thickness. In the analysis of the effect of martensite volume fraction, one must keep in mind that the global carbon content remains constant, which leads to a softer martensite when its volume fraction increases. This is a possible reason for the comparable strength of the pV19t2 and pV36t2 steels.

As future research, to further characterize the damage and cracking mechanisms, in addition to what is possible with the fracture surfaces and profiles, mechanical testing coupled to DIC at the level of the microstructure could be performed in platelet-like microstructures in order to observe the heterogeneous straining. In-situ experiments with X-ray tomography on platelet-like microstructures would furthermore enable to better confirm that the heterogeneous straining leads to a heterogeneous damage accumulation. Platelet-like Dual-Phase steels with an even larger martensite packet size than what was considered in this study could be developed in order to verify if it leads to an enhanced cracking resistance, or better yet, assess the martensite packet size impact on cracking resistance. A larger experimental campaign of hole expansion ratio tests could be performed in order to be able to link the stretch-flangeability to the essential work of fracture or to other material parameters.

5.5 Conclusions

Dual-Phase steels exhibiting a platelet-like microstructure can have a surprisingly high cracking resistance. This was verified to be true both in bulk samples and in thin sheets using different methods: the CTOD method, the EWF method and hole expansion ratio tests. The results were systematically compared to traditional equiaxed microstructures to put an emphasis on the importance of the morphology on fracture resistance.

A high cracking resistance combined to the good compromise of strength and ductility is useful for the automotive industry, and furthermore for the recycling industry. The aim was to make known those impressive values for their usefulness as well as to contribute to the understanding of the mechanisms governing fracture resistance in such Dual-Phase steels.

The main reason for the high cracking resistance is the ability of the platelet-like microstructures to deform to large strains in the fracture process zone during crack propagation, as attested by the average equivalent fracture strain $\varepsilon_{f,eq}$. This ability is due to the high void nucleation resistance of such microstructures compared to equiaxed microstructures, which is in turn due to the reorientation of the martensite platelets in the direction of loading. The reorientation occurs owing to the rotation of the martensite

platelets, due to the shearing of the ferrite, and to the deformation capacity of the platelets themselves. A second order effect for the high cracking resistance is a possible two-stage crack propagation process involving martensite packet scale in addition to particle spacing. Finally, cracking behavior has been qualitatively discussed based on an existing analytical model involving a limited number of material parameters. The model supports the large necking contribution and enabled to make a link with the internal length associated to the microstructure.

Chapter 6

Damage modeling using a cellular automaton approach

The research presented in this chapter was performed in close collaboration with Florent Hannard. The objective of the chapter is to introduce a model to predict the fracture strain of Dual-Phase steels based on physical grounds. The model is based on a cellular automaton approach and aims at taking into account the statistical aspects related to complex microstructural heterogeneous aspects.

The model is described first. Preliminary semi-quantitative results are then presented. Finally, a section offers some perspectives in order to enhance the capabilities of the model and to make it more predictive.

6.1 Generalities about a cellular automaton

A cellular automaton is a computational tool dealing with a discrete system. It is discrete both in space and time. It comprises a finite number of elementary units whose states evolve over discrete time steps. The evolution of the state of an elementary unit depends on the state of the units in the direct neighborhood (involving the unit itself) following a set of rules, excluding thereby long range influences. The rules are applied iteratively at every time step.

Cellular automata are used in a large variety of scientific fields. Comprised of a large number of units, in some instances, a cellular automaton can generate complex patterns of behavior based sometimes on remarkably uncomplicated rules.

6.2 Description of the damage model

6.2.1 General computational strategy

A cellular automaton approach based model is applied to several model microstructures. An important benefit of a cellular automaton approach compared to traditional damage models such as the Gurson model (*Gurson, 1977*), is the capability of introducing a large number of different particles in the microstructure and thereby of taking into account the statistical aspects related to the different possible heterogeneities, enabling more realistic RVEs, without the intensiveness of a finite element model, but with a coarser description of the mechanical fields.

In this work, the stress and strain fields at each step of deformation in the model are drawn from the unit cell finite element calculations performed in Chapter 4, at a finite number of points. The points constitute the discrete spatial distribution and the deformation steps constitute the discrete time steps. Therefore, part of the heterogeneity comes from the choice of the parameters of the model used for the description of the elastoplastic behavior (see Chapter 4). As a recall, those are the aspect ratio, the martensite volume fraction and carbon content, and the orientation of the particle with respect to loading.

The steps of nucleation, growth and coalescence of voids are dealt with simultaneously, although separately for each particle/void. No interaction between voids takes place before the stage of coalescence. The three steps of damage are treated in an analytical way. A critical stress distribution is used for void nucleation, the Rice and Tracey model is used for the growth of the voids, and finally the Brown and Embury criterion is used for the detection of coalescence.

Figure 6.1 provides a general overview of the approach followed for damage modeling in the form of a flowchart. The microstructure, which is given as an input to the cellular automaton and which is made up of the local mechanical fields taken from the unit cell calculations, is deformed in a strain-controlled manner. At each deformation step, the state of damage is updated. The nucleation criterion is checked at each particle that has not yet nucleated a

void. If the criterion is fulfilled, a void is nucleated. The Rice and Tracey model is used to predict the evolution of the void radius. And if two neighboring voids meet the Brown and Embury criterion, they are considered to have coalesced for the remaining of the simulation. The starting version of the cellular automaton has been borrowed from the work of *Hannard et al. (2016)*, who also largely contributed to the development of the version of the cellular automaton presented in this chapter.

Compared to the model in (*Hannard et al., 2016*), one of the main novelties, which complicates significantly the framework, is the connection to the unit cell calculations all along the deformation process, which provides accurate results for the local fields. This remains true until void nucleation. This means that after void nucleation, the second phase particle is assumed not to affect the void evolution. An important hypothesis of the model is the absence of damage-plasticity coupling. Hence, the local stress relaxation brought about by the presence of voids and by the loss of stress carrying capacity of the particles after void nucleation is not taken into account. Other differences compared to the model in (*Hannard et al., 2016*) are a few extra simplifications. For instance, the volume of the particle was taken into account for the nucleation step in the work by (*Hannard et al., 2016*). The voids nucleated were moreover penny-shaped, which required the extended form of the Rice and Tracey model for void growth and the extended form of the Thomason model for the detection of coalescence, which is more accurate than the Brown and Embury criterion.

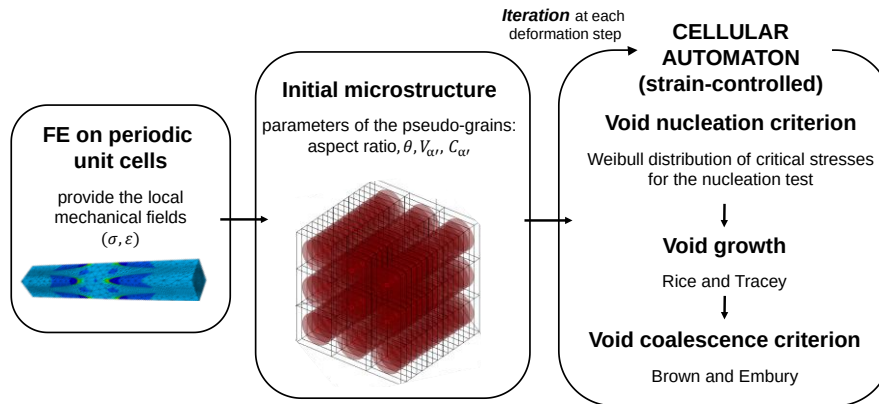


Figure 6.1 Flowchart of the cellular automaton approach model for damage modeling.

6.2.2 Generation of the microstructure and the corresponding local stress-strain fields

In order to generate the microstructure and to assign the values of the stress and strain fields for each deformation step, unit cells derived from the finite element calculations performed in Chapter 4 are virtually stacked to form a cube. One cube represents a grain made of many martensite particles with a similar orientation and shape. The shape, the orientation, the martensite volume fraction and the martensite carbon content can only have a few discrete values for which FE calculations are available. Points are distributed inside the cube assuming a homogeneous value for the microstructural parameters. Each point of the cellular automaton is connected to its reference FE cell calculation results and is associated to a specific evolution of the damage indicator D , of the stress triaxiality and of the equivalent plastic strain as a function of macroscopic strain.

In practice, a limited number of points within the unit cells are selected within the ferrite phase, at which the values of stress and strain are taken from the calculations. A mapping from the local values at the selected points is then performed. Figure 6.2 shows the location of the points relatively to the particle for a single cell in the case of an oblate particle. The points in only one eighth of the cell are shown due to symmetry. In addition to the points of (initially) fixed position displayed on the image, there are the points at which the maximum principal stress, the equivalent plastic strain and the damage indicator D have the highest values.

The unit cell calculations being limited to a strain equal to 0.35 (see Chapter 4), and fracture occurring at strains larger than 0.35, the local fields were extrapolated to larger strains. The extrapolation was performed linearly. The subsequent mapping was performed on the points of (initially) fixed position (i.e. the points shown in Figure 6.2).

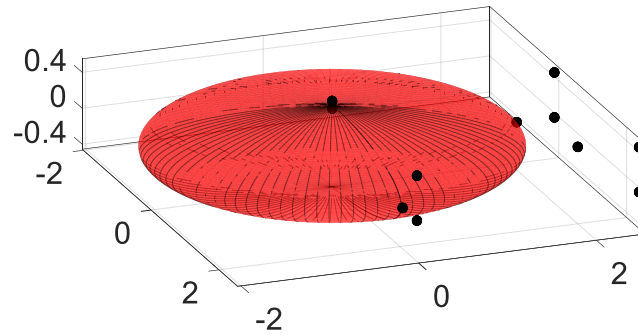


Figure 6.2 Locations of the points (of initially fixed location) in the unit cells, whose values of stress and strain are transferred to the damage model in the cellular automaton. The red spheroid represents the martensite particle.

The local stress and strain values are all taken in the ferrite phase because voids essentially nucleate by interface decohesion and mainly grow in the soft phase in platelet-like microstructures (see Chapter 5). The extension to particle breaking is however possible.

6.2.3 Damage equations and implementation

The nucleation, growth and coalescence stages of the voids are described using basic analytical micromechanical models, the interest being here on addressing a large number of particles instead of working with very precise damage rules. The equations associated to each stage constitute the set of rules that must be verified at every time step.

6.2.3.1 Void nucleation

The nucleated void is assumed to be spherical. Its initial volume V_{nucl} is equal to a fraction of the particle volume fixed at this time as equal to 1/20. The size of the initial void volume is chosen small as voids nucleate in reality with

a size much smaller than the particles in platelet-like microstructures (see Chapter 5).

A void nucleates when the maximum principal stress in the ferrite reaches a critical value. It is assumed that one particle can lead to the nucleation of at most one void. The critical stress for void nucleation at each nucleation site is assumed to follow a Weibull distribution, whose cumulative distribution function writes

$$P(\sigma_{interface}^{max}) = 1 - \exp\left(-\left(\frac{\sigma_c^{interface}}{\sigma_w}\right)^m\right). \quad 6.1$$

Isolating the critical stress above which nucleation occurs, one gets

$$\sigma_c^{interface} = \sigma_w[-\log(1 - P)]^{1/m}. \quad 6.2$$

For an equiaxed microstructure, the values used for the parameters of the distribution are $\sigma_w = 2143 \text{ MPa}$ and $m = 24$. For a platelet-like microstructure, the values are $\sigma_w = 3198 \text{ MPa}$ and $m = 15$. The procedure for choosing these parameters values is explained hereafter.

Hence, in addition to the parameters of the plasticity model, another heterogeneity is introduced through the distribution of critical stress for void nucleation over the different particles, which leads to a non-simultaneous nucleation process.

Choice of σ_w and m values for both types of microstructures

The damage nucleation mechanisms being different in case of a platelet-like microstructure and in case of an equiaxed microstructure (see Chapter 5); the parameters of the Weibull distribution are different. In order to calibrate the parameters, the damage evolution on one equiaxed microstructure (eV36t1) and one platelet-like microstructure (pV36t1) was characterized.

For this purpose, the number of voids per unit surface was counted post mortem in each case at the center of the sample, on a transverse section. The counting of void density was performed for different levels of deformation ε_i , as illustrated in Figure 6.3.

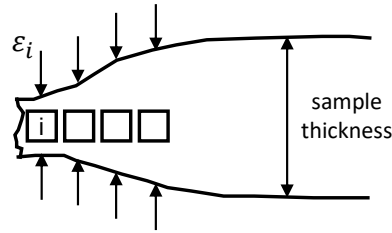


Figure 6.3 Schematic of the damage characterization mapping approach based on which the void density is estimated: at the center of the sample, on a transverse section, for different levels of strain ε_i .

The number of particles per unit surface was quantified on SEM micrographs. The number of voids normalized by the total number of particles can be computed as a function of strain, together with the void density for a given strain ε_i .

It was hypothesized that the number of voids normalized by the total number of particles is equal to the probability P appearing in the Weibull distribution that a particle nucleates one void (for a given level of deformation). Figure 6.4 shows the evolution of the probability P as a function of strain for the two morphologies.

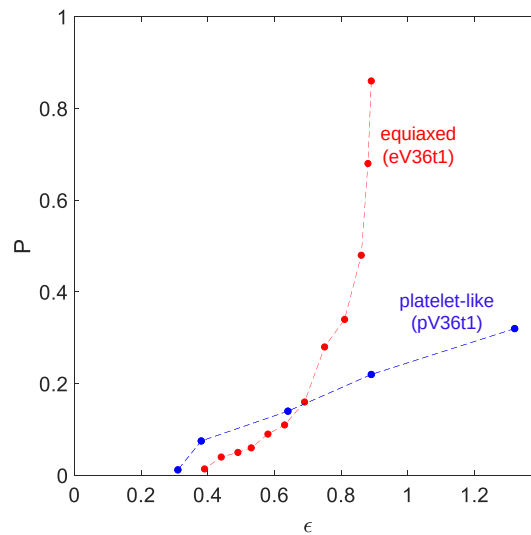


Figure 6.4 Probability P of a particle leading to a void as a function of strain for a platelet-like microstructure and for an equiaxed microstructure.

The nucleation criterion is based on the largest value of the local maximum principal stress. Hence, the local maximum principal stress corresponding to the macroscopic deformation is a field parameter to be extracted from unit cell calculations. In order to calibrate the parameters of the Weibull distribution, a unit cell with an aspect ratio equal to one was taken for the equiaxed microstructure and an aspect ratio of 0.2 for the platelet-like microstructure. Unit cells with a martensite volume fraction equal to 30% and a martensite carbon content equal to 0.15wt.% were taken as they come close to the test materials. An angle of 30° was taken for an aspect ratio of 1 and an angle of 0° was taken for an aspect ratio of 0.2.

Since the experimental data for P correspond to macroscopic strains larger than 0.35 (which is the value at which the unit cell calculations were limited in Chapter 4), an extrapolation of the local maximum principal stress was needed after this value up to the fracture strains of the studied steels. In order to do so, as the mapping after extrapolation is performed on the points of (initially) fixed position, the largest value of the local maximum principal stresses at those points was first taken at every deformation step. The obtained values of maximum principal stress σ_{max}^{princ} were then fitted as a function of macroscopic strain ε_{macro} using a power law of the form

$$\sigma_{max}^{princ} = a\varepsilon_{macro}^b + c. \quad (6.3)$$

An example is shown in Figure 6.5 for the platelet-like morphology. The black curve represents the data extracted from the unit cells, i.e. the largest value of maximum principal stress among the (initially) fixed position points for every deformation step, and the blue curve is the fitted and extrapolated data using the power law.

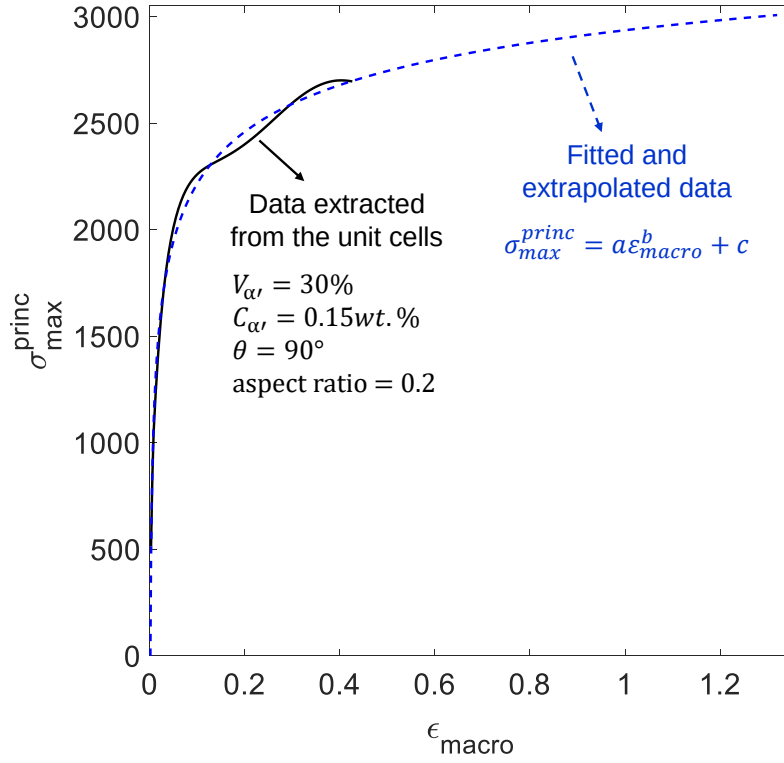


Figure 6.5 Largest value of maximum principal stress for the platelet-like morphology as a function of macroscopic strain extracted from the unit cells (full black curve) and fitted and extrapolated using a power law (dashed blue curve).

Using the values of σ_{max}^{princ} corresponding to the macroscopic strains at which P is available, this leads to the relationship between P and σ_{max}^{princ} given in Figure 6.6, in which the values of σ_w and m for both morphologies are also indicated. The last five values were not taken in the case of the equiaxed microstructure as the most relevant measurements for void nucleation are for lower strains.

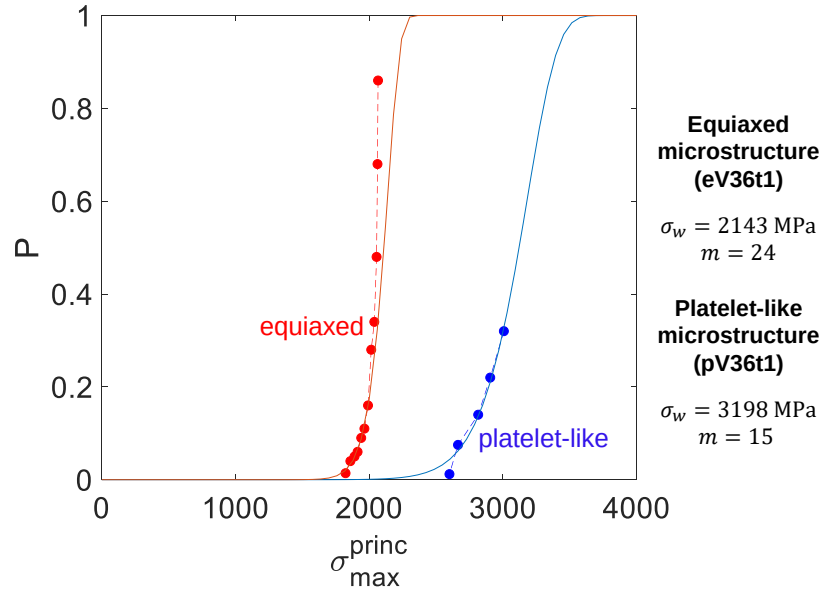


Figure 6.6 Probability P of a particle leading to a void as a function of maximum principal stress for a platelet-like microstructure and for an equiaxed microstructure, and corresponding σ_w and m values of the fitted Weibull distribution.

The parameter σ_w can be assimilated to the void nucleation stress (here, the maximum value of the local maximum principal stress at which nucleation occurs). A possible reason for the higher void nucleation stress in the case of a platelet-like microstructure is the particle size. Martensite particles in equiaxed microstructures being coarser than in platelet-like microstructures, they exhibit a lower nucleation stress (which eventually leads to a lower fracture strain).

The parameter m provides information about the transition rate around the void nucleation stress. A lower value of m involves earlier void nucleation, but larger stresses must be reached for all particles to nucleate a void (case of the platelet-like microstructure); while for a larger value of m , all voids nucleate at a stress closer to σ_w (case of the equiaxed microstructure).

6.2.3.2 Void growth

The linearized form of the Rice and Tracey model (*Rice and Tracey, 1969*) describes void growth as

$$\frac{\Delta R}{R} = \alpha \exp\left(\frac{3}{2}T\right) \Delta \varepsilon^p \quad 6.4$$

where R is the void radius, ΔR the difference in the void radius between two time steps and $\Delta \varepsilon^p$ the difference in local equivalent plastic strain between the two time steps. The value of α is the one re-evaluated by (Huang, 1991), equal to 0.427 when $T > 1$ and to $0.427 T^{1/4}$ when $T < 1$.

6.2.3.3 Void coalescence

The geometric Brown and Embury criterion (see Section 2.3.2) is used for the onset of void coalescence. When two voids coalesce, no additional void is created, meaning only the volume of the void due to nucleation and growth is involved in the total volume fraction of void F_v , as illustrated in Figure 6.7 (assuming voids 1 and 2 fulfill the Brown and Embury criterion).

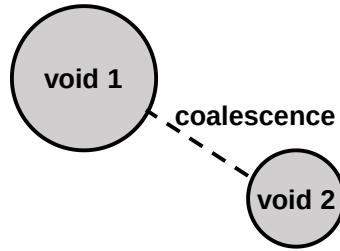


Figure 6.7 When coalescence takes place, only the volume of the voids due to growth (in grey) is counted in the volume fraction of void F_v .

6.2.4 Visualization

For the sake of visualization, Figure 6.8 shows a 2D view of a microstructure with disk particles at various strains. It corresponds to a martensite volume fraction V_α , equal to 30%, a martensite carbon content C_α , equal to 0.15wt.% and an orientation θ equal to 45° . 135 ($3 \times 3 \times 15$) particles are present in the microstructure.

Each virtual box represents one unit cell. The microstructure is at first undeformed (Figure 6.8(a)), deforms with the applied strain (Figure 6.8(b)) until the nucleation of the first voids (Figure 6.8(c)). The first voids nucleate in light green. At larger strains, voids grow and start coalescing, in which case

they share the same color (Figure 6.8(d)). This is why several colors appear in Figure 6.8(d). Each color (different than light grey) corresponds to one microcrack, a microcrack being composed of voids that have coalesced together.

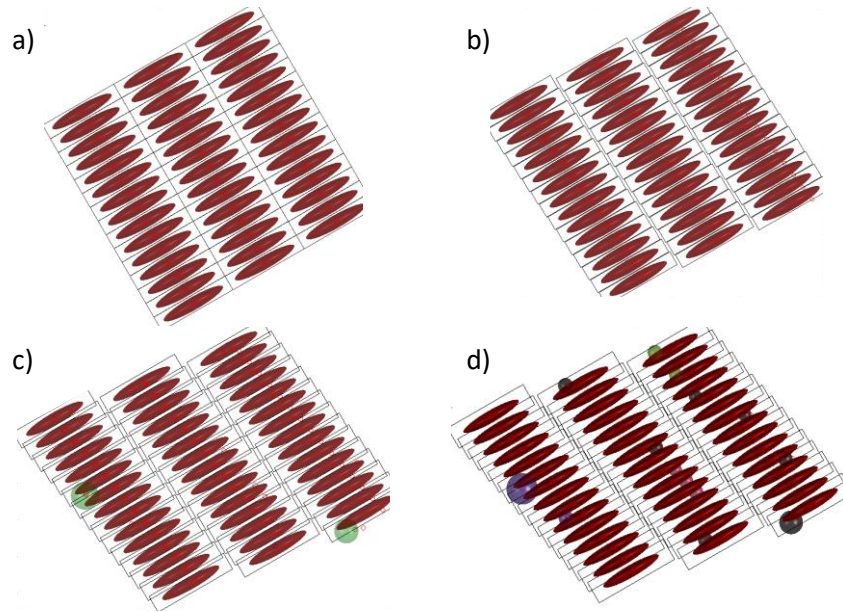


Figure 6.8 Visualization of the microstructure at different strains showing (a) the initial microstructure, (b) the deformed microstructure at small strain, (c) the nucleation of the first voids and (d) the growth and coalescence of voids. The aspect ratio is equal to 0.2. $V_{\alpha'}$ is fixed to 30%, $C_{\alpha'}$ to 0.15wt.% and θ to 45° .

6.3 Results and discussion

After a reference case with spherical particles, the impact of martensite carbon content, martensite volume fraction, orientation and aspect ratio was assessed at grain level. Therefore, oblate particles, having an aspect ratio equal to 0.2, were stacked to form a cube of 625 particles ($5 \times 5 \times 25$ particles) and spherical particles were stacked to form a cube of 512 particles ($8 \times 8 \times 8$). The chapter ends with proposals for improving the model.

6.3.1 The case of spherical particles

A microstructure with spherical particles was investigated first. Figure 6.9(a) shows the proportion of particles that have led to a void as a function of macroscopic strain for different orientations: 0° , 30° and 45° . Figure 6.9(b) shows the total volume fraction of void F_v as a function of macroscopic strain for the different orientations. A dotted line is plotted at a $0.2 F_v$, as it is considered that the material is broken at a void volume fraction of 20%. Results are given for a martensite volume fraction $V_{\alpha'}$ equal to 30% and a martensite carbon content $C_{\alpha'}$ equal to 0.15wt.%.

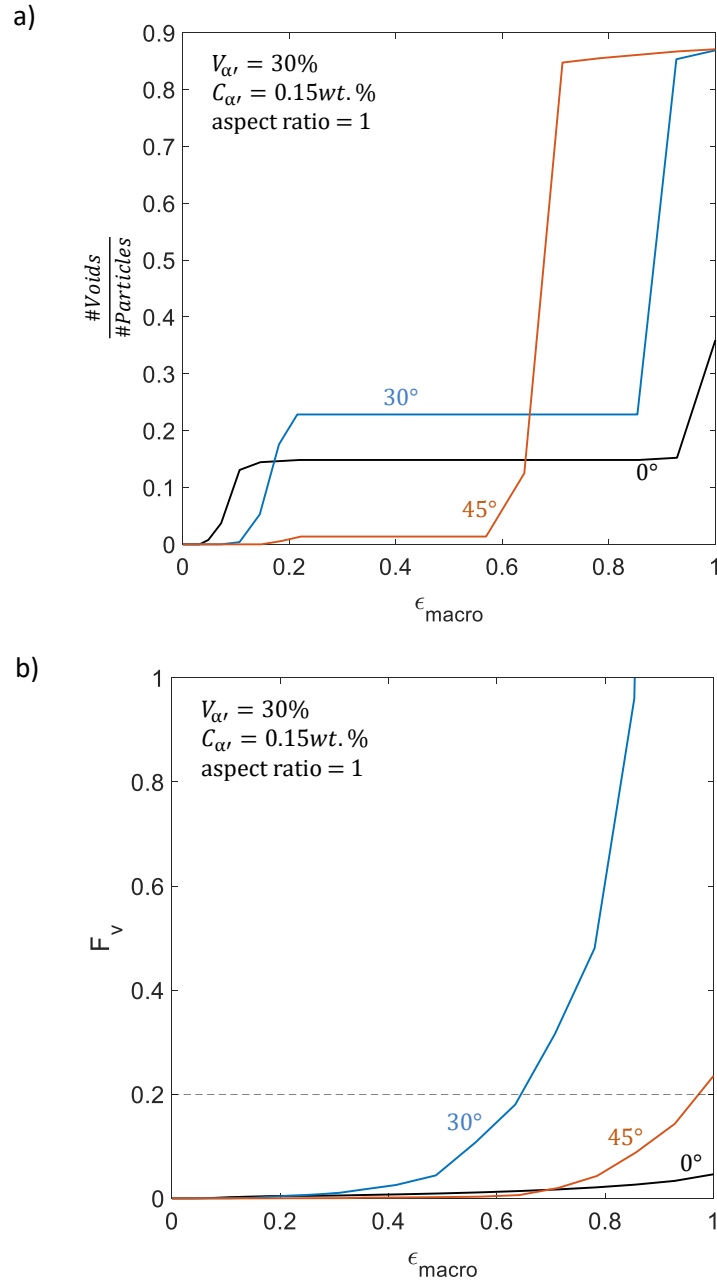


Figure 6.9 a) Proportion of particles leading to a void as a function of macroscopic strain and b) total void volume fraction as a function of macroscopic strain. Results are given for θ equal to 0° , 30° and 45° . The aspect ratio is equal to 1. $V_{\alpha'}$ is fixed to 30% and $C_{\alpha'}$ to 0.15wt.%.

The differences between the different angles emerge from the anisotropy of the cubic stacking of the unit cells (see Chapter 4). A $0^\circ \theta$ leads to an earlier void nucleation. However, the local fields do not lead to a large damage growth. A $30^\circ \theta$ is the worst-case scenario as voids nucleate at relatively low strains, in greater number and they grow faster.

6.3.2 Impact of martensite carbon content

Figure 6.10 illustrates the difference in damage behavior between a martensite carbon content $C_{\alpha'}$ equal to 0.15wt.% (full line) and a $C_{\alpha'}$ equal to 0.60wt.% (dashed line). Figure 6.10(a) shows the proportion of particles that have led to a void and Figure 6.10(b) shows the total volume fraction of void F_v , both as a function of macroscopic strain. Results are given for a martensite volume fraction $V_{\alpha'}$ equal to 30%, an aspect ratio equal to 0.2 and an orientation θ equal to 90° , which is the orientation observed experimentally after particle alignment.

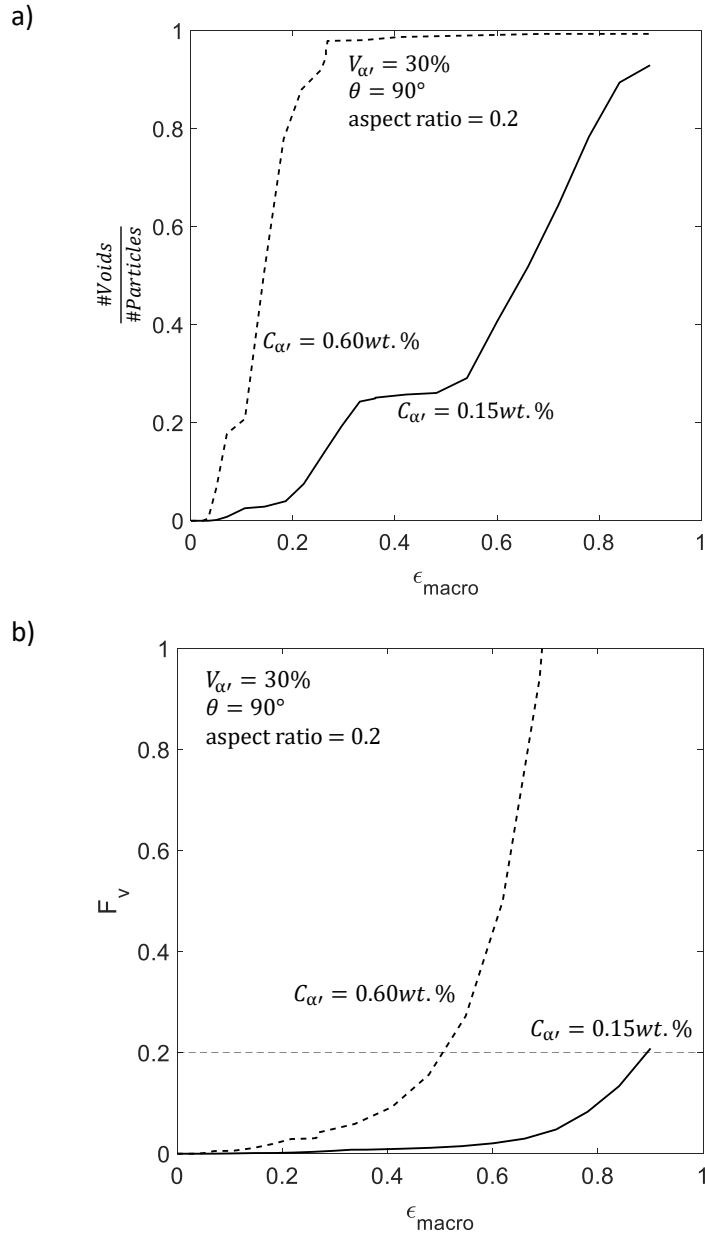


Figure 6.10 Comparison between $C_{\alpha'}$ equal to 0.15wt.% and to 0.60wt.% in terms of a) the proportion of particles leading to a void and b) the total void volume fraction, both plotted as a function of macroscopic strain. $V_{\alpha'}$ is fixed to 30%, θ to 90° and the aspect ratio to 0.2.

Both the proportion of particles leading to a void and F_v are higher for a $C_{\alpha'}$ equal to 0.60wt.%. Voids nucleate at smaller strain and grow faster when

carbon content is larger. Assessing the impact of carbon content is equivalent to assessing the impact of phase contrast. As observed experimentally (*Shen et al., 1986; Mazinani and Poole, 2007*), increasing carbon content leads to a larger number of voids. This is due to the fact that increasing the hardness of the martensite particles (through carbon content increase) decreases their ductility. Yield and tensile stress increase due to kinematic hardening. This implies a less efficient load transfer, thereby a less coherent ferrite-martensite interface with a higher strain mismatch and higher interface stresses and stress triaxiality. Altogether, this accelerates void nucleation and growth.

A high carbon content leads to an earlier void coalescence, as shown in Figure 6.11, and thus to an overall decrease of fracture resistance. Figure 6.11(a) shows the number of coalescence events and Figure 6.11(b) shows the number of microcracks, both as a function of macroscopic strain for the two martensite carbon contents $C_{\alpha'}$. A microcrack occurs as soon as two voids coalesce together.

The number of coalescence events is much higher, at all strains, for a carbon content equal to 0.60wt.%. All particles (625) led to a void and all voids coalesced together at a strain lower than 0.3. The number of microcracks increases first when the voids start coalescing, reaches a maximum and then decreases afterwards when the large voids coalesce together. When all voids have coalesced together, a single crack remains. In line with the number of coalescence events, the first microcracks appear sooner for a $C_{\alpha'}$ of 0.60wt.% compared to 0.15wt.%, their number also increases faster, the maximum is reached at lower strain, as well as the attainment of the final crack once all voids have coalesced together.

A general feature of the present version of the model is the early coalescence of voids compared to what is observed experimentally. Quite often, voids that nucleate directly coalesce with the large crack at moderate strains.

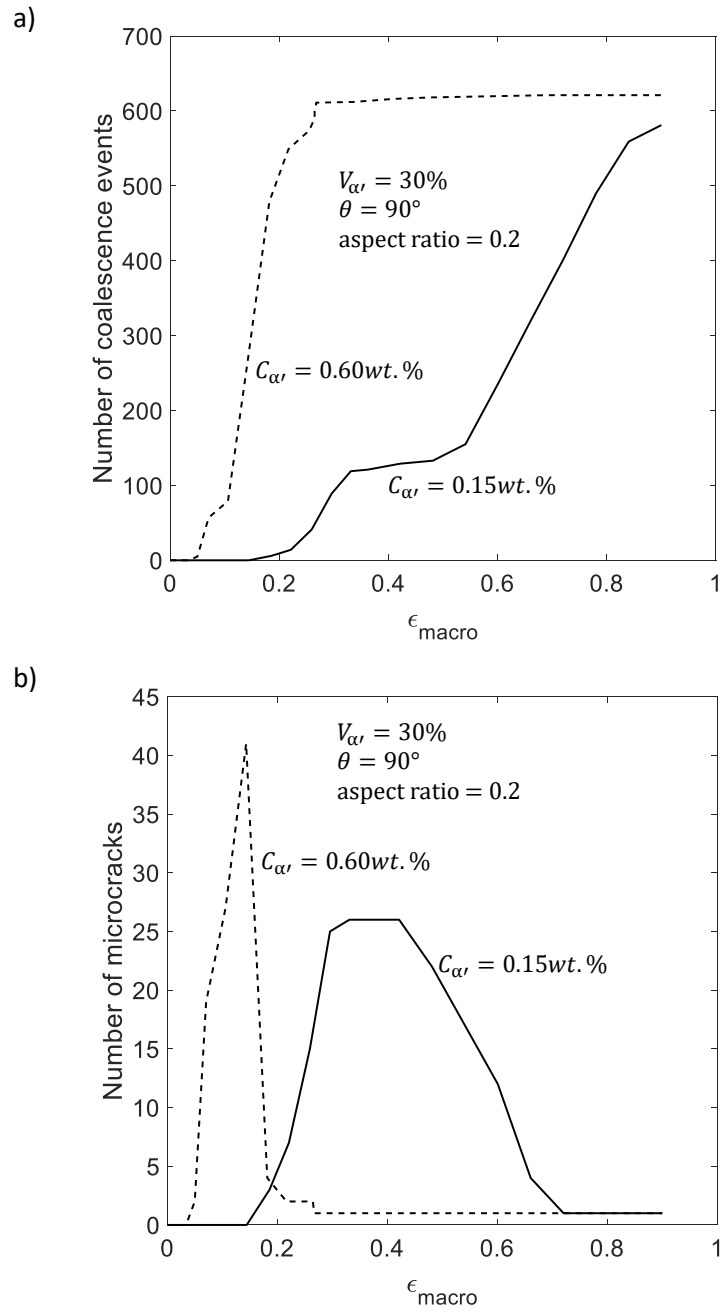
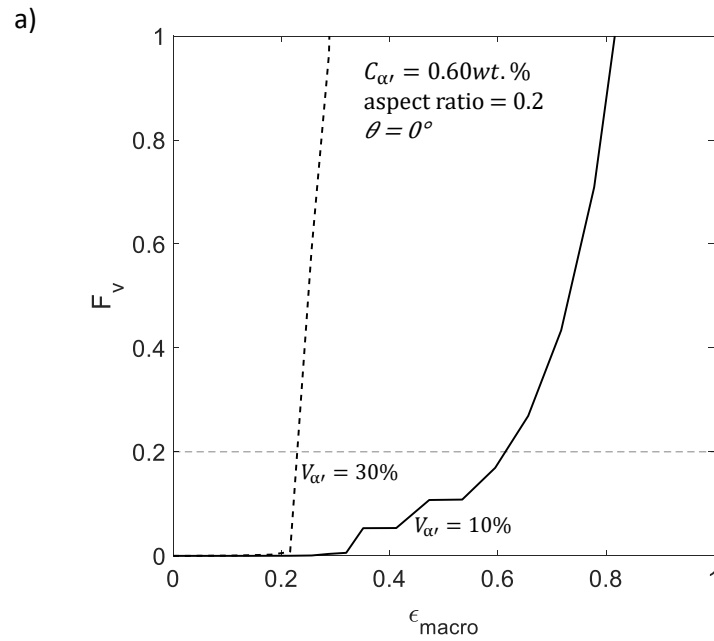


Figure 6.11 Comparison between $C_{\alpha'}$ equal to 0.15wt.% and to 0.60wt.% in terms of a) the number of coalescence events and b) the number of microcracks, both plotted as a function of macroscopic strain. $V_{\alpha'}$ is fixed to 30%, θ to 90° and the aspect ratio to 0.2.

6.3.3 Impact of martensite volume fraction

In order to assess the impact of martensite volume fraction, Figure 6.12 shows, for different orientations (0° , 45° and 90°), the void volume fraction F_v as a function of macroscopic strain. On each graph, the dashed line curve corresponds to a $V_{\alpha'}$ equal to 30% and the full line curve to a $V_{\alpha'}$ equal to 10%. The martensite carbon content $C_{\alpha'}$ is fixed to 0.60wt.% and the aspect ratio to 0.2.



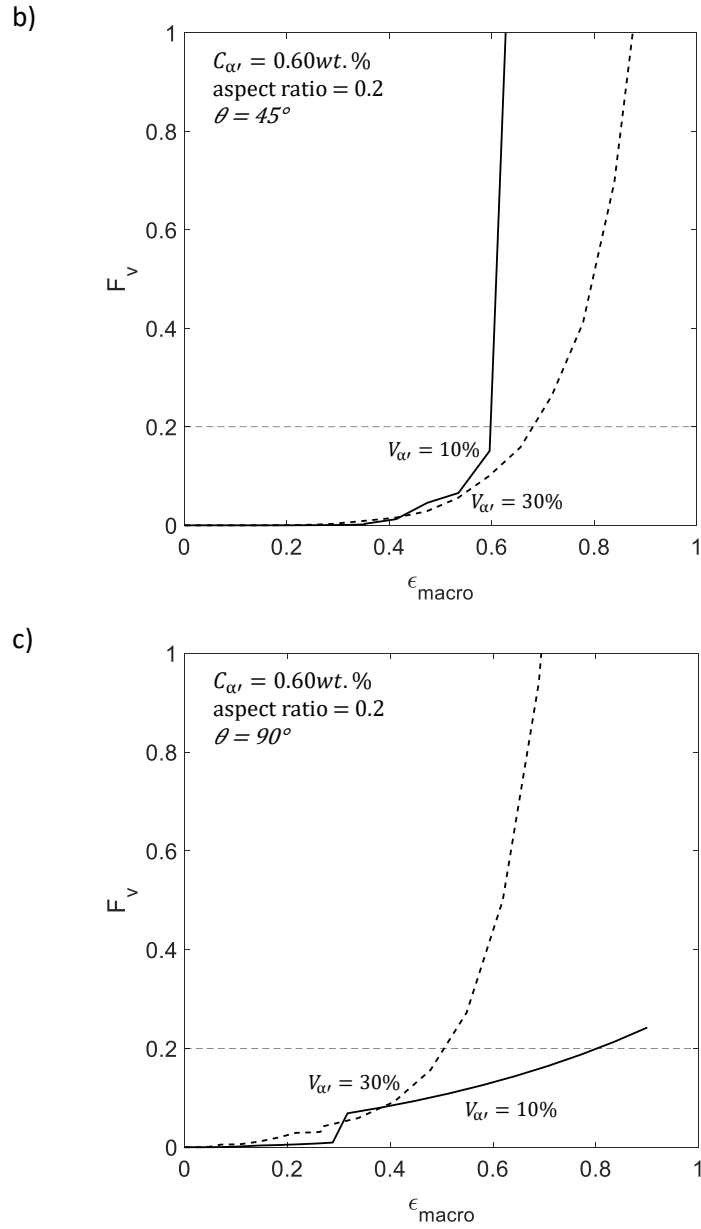


Figure 6.12 Comparison between the variation of the total void volume fraction F_v as a function of macroscopic strain for martensite volume fractions $V_{\alpha'}$ equal to 10% (full line curve) and 30% (dashed line curve), for θ equal to a) 0° , b) 45° , c) 90° . $C_{\alpha'}$ is fixed to 0.60wt.% and the aspect ratio to 0.2.

The impact of martensite volume fraction $V_{\alpha'}$ depends on orientation. For instance, the void volume fraction increases faster for 30% of martensite

compared to 10% for θ equal to 90° and even more at 0° (and 30°). For θ equal to intermediate value 45° (and 60°), the same is true at low strain, but not at large strains.

When martensite volume fraction increases, the particle relative spacing decreases. The plasticity zones at the tips then overlap, which may increase local strain and stress triaxiality. This leads to more favorable damage nucleation sites and accelerated void growth.

Care must be taken at strains larger than 0.35 as the local fields were then extrapolated. However, the different behavior at 45° (and 60°) at large strains may perhaps be explained by the fact that when increasing the martensite volume fraction, the martensite accommodates a larger part of the deformation, which decreases the strain mismatch between both phases. This would delay nucleation and decrease the void growth rate.

6.3.4 Impact of orientation

Figure 6.13 shows results at various orientations θ , namely 0° , 30° , 45° , 60° and 90° . The martensite volume fraction $V_{\alpha'}$ is equal to 30%, the martensite carbon content $C_{\alpha'}$ to 0.15wt.% and the aspect ratio to 0.2. Figure 6.13(a) shows the variation of the proportion of particles leading to a void as a function of macroscopic strain for different orientations. Figure 6.13(b) compares the void volume fraction for different orientations.

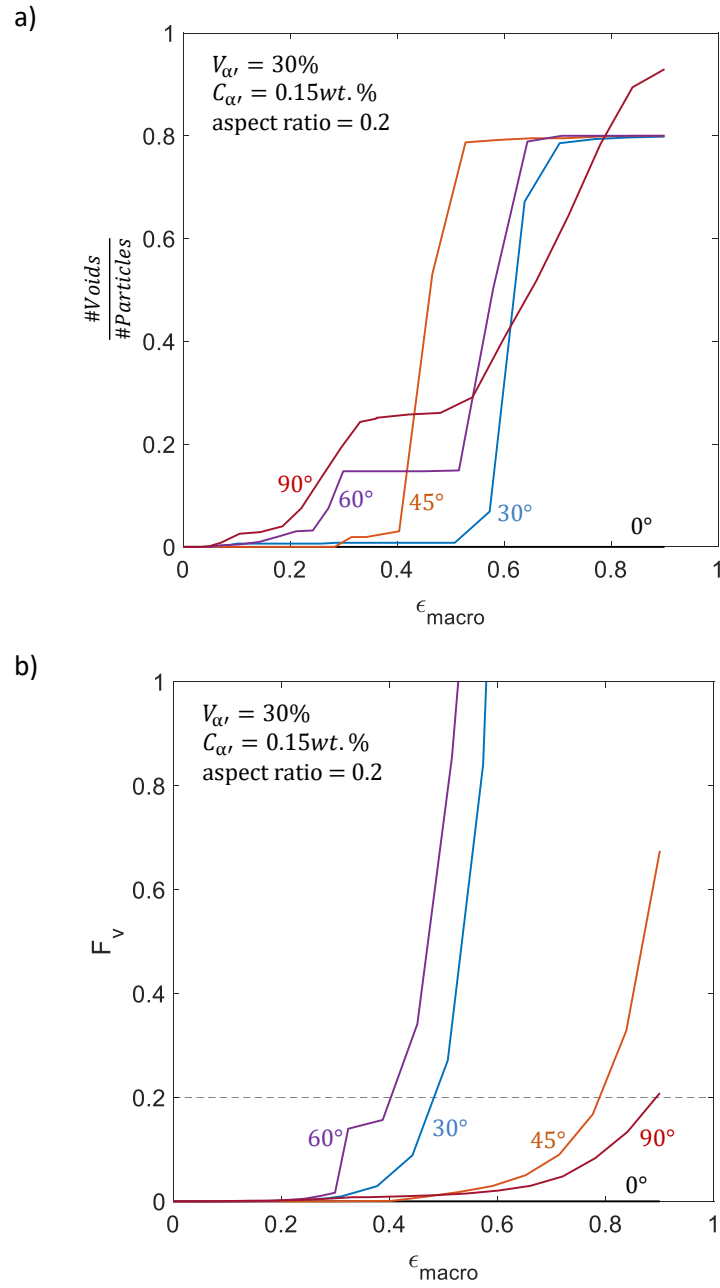


Figure 6.13 Comparison between different orientations in terms of a) proportion of particles leading to a void and b) total void volume fraction. $V_{\alpha'}$ is fixed to 30%, $C_{\alpha'}$ to 0.15wt.%, and the aspect ratio to 0.2.

Overall, the number of nucleated voids is the lowest at 0° and the highest at 90° , with an almost monotonous increase between the two orientations (see Figure 6.13(a)). This is especially true at strains below 0.4; at strains larger than 0.35, the local fields were extrapolated, which could lead to nonrealistic results. The increase in nucleated voids when going from 0° to 90° is due to the fact that the stress and strain concentrations are progressively moving towards the tip of the particle that has a high curvature (see Chapter 4). The number of voids is even equal to 0 for 0° .

Between the different orientations, the order in the number of voids is not maintained when looking at the void volume fraction. For instance, even though the voids appear early at 90° (which is not observed experimentally), it corresponds to a damage resistant configuration (see Figure 6.13(b)). The reason is that void nucleation is driven by the maximum principal stress, but void growth is driven by accumulated plastic strain and stress triaxiality, that are thus higher for instance at 30° (few voids nucleate but F_v is high) than at 90° (many voids nucleate but F_v is low).

The results corroborate the conclusion of Chapter 4 in that they predict that damage behavior depends on orientation, which leads to a heterogeneous accumulation of damage.

6.3.5 Impact of particle aspect ratio

In order to assess the impact of aspect ratio, Figure 6.14 compares results between a microstructure containing spherical particles and a microstructure containing disk particles. Figure 6.14(a) shows the proportion of particles leading to a void, and Figure 6.14(b) shows the variation of the total void volume fraction as a function of macroscopic strain. $V_{\alpha'}$ is fixed to 30% and $C_{\alpha'}$ to 0.15wt.%. Orientations equal to 0° and 30° were taken for an aspect ratio equal to one. They are compared to orientations equal to 90° and to 60° for an aspect ratio of 0.2. The reason of this choice is that 90° is equivalent to 0° in the case of an aspect ratio of one (and 30° to 60°). Moreover, the 90° orientation is of interest as it is the one observed experimentally after particle alignment.

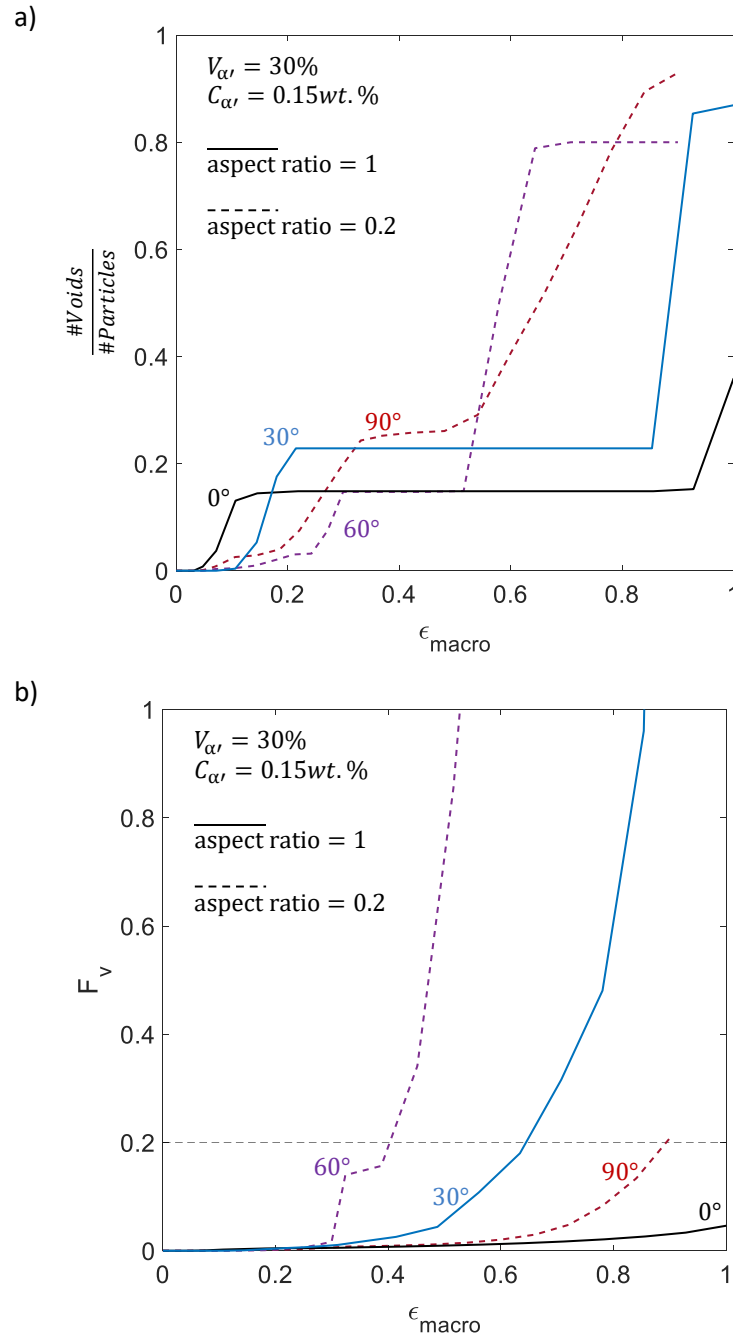


Figure 6.14 Comparison between a microstructure with spherical particles and disk particles in terms of a) the proportion of particles leading to a void and of b) the total void volume fraction. $V_{\alpha'}$ is fixed to 30%, $C_{\alpha'}$ to 0.15wt.%, and $\theta = 0^\circ$ and 30° for an aspect ratio of 1 and $\theta = 90^\circ$ and 60° for an aspect ratio of 0.2.

Voids start nucleating at lower strains in the case of a microstructure with spherical particles, due to the lower stress needed for nucleation. However, a larger amount of voids does not automatically translate into larger void volume fraction, which greatly depends on the growth rate. The void volume fraction is larger in the case of the microstructure with disk particles (when compared to the corresponding angle with spherical particles) because of a larger void growth rate. The larger void growth rate is in part due to the higher particle curvature at the tips in the case of a microstructure with disk particles. Moreover, the local stress and stress triaxiality are higher in case of disk particles when nucleation occurs, as nucleation takes place at a larger maximum principal stress than in the case of spherical particles (see the fitting of the Weibull distribution parameters). Hence, the void volume fraction is ultimately larger.

This behavior is coherent from the model point of view. However, it does not correspond to what is observed experimentally, namely that platelet-like microstructures are more resistant to damage than are equiaxed microstructures (see Chapter 5). An explanation is that in real equiaxed microstructures, particles are not smooth and perfectly spherical but exhibit protrusions around which stress and strain concentrate. Another possible explanation is the different size of the voids at nucleation between an equiaxed microstructure (large voids) and a platelet-like microstructure (small voids), which is not taken into account in the present version of the model. Several other reasons for the model's inability to capture the experimentally observed difference between the two morphologies are discussed in Section 6.3.6 .

6.3.6 Perspectives

Several experimental observations are not captured by the model, the latter being limited by the quality of the information fed to it. In the following, shortcomings of the present version of the model are assessed and ideas to enhance its predictability are proposed.

As stress and strain values are taken from the unit cells, some limitations of which were discussed in Chapter 4, the present model is largely influenced by them. In the unit cell calculations, when the loading direction is not

aligned with an axis of the particle, the unit cell distorts heavily with applied deformation, with no noticeable strain in the martensite particles, as shown in Figure 6.15 ($V_{\alpha'} = 30\%$; $C_{\alpha'} = 0.15\text{wt.}\%$, aspect ratio = 0.2; $\theta = 45^\circ$).

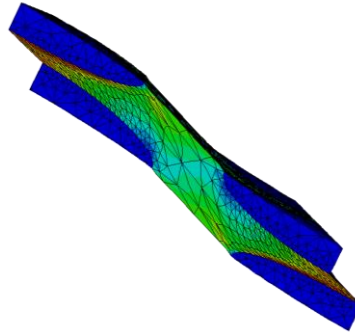


Figure 6.15 Distorted unit cell ($V_{\alpha'} = 30\%$; $C_{\alpha'} = 0.15\text{wt.}\%$, aspect ratio = 0.2 ; $\theta = 45^\circ$).

Experimentally however, it was observed that the particles, rotating with the matrix, deform to line up entirely with the loading direction (see Chapter 5). The complete alignment occurs before void nucleation. A possible reason for this difference between the results of the unit cell calculations and the experiments is that the martensite is modeled as a too hard entity compared to ferrite. Another plausible reason is the absence of any size effect in the model, which would be relevant seeing the small thickness of the platelet-like particles. It was argued that the alignment contributes to the damage resistance of the platelet-like microstructures, through a resistant to damage configuration with rare and small damage occurrences. The model on the other hand considers many voids that grow to reach much larger sizes than what is observed experimentally. What is more, occasional shearing occurred during the tensile tests, which is not captured by unit cells calculations with periodic conditions.

Altogether, those issues prevent the model from being predictive. For a better predictability, the phenomena effectively occurring must be accounted for. Ideally, plasticity and damage should be coupled. This would be possible in the framework of the model if prior calculations were run in unit cells in which damage is taken into account. Moreover, simulations at different stress triaxialities should be performed as stress triaxiality increases

substantially when plasticity localizes, which inevitably occurs during a tensile test (of a ductile material). It is from such a test that the fracture strain, that the model ultimately tempts to predict, is computed. In the present case, the stress triaxiality always corresponds to the one of a pure uniaxial tensile loading, equal to $1/3$.

Based on the available unit cell calculations, a possible way of accounting for the alignment of the particles is to progressively shift from the starting orientation to the aligned orientation by switching between the angles of the unit cell calculations. For instance, if the initial θ is equal to 45° , after a given deformation, the unit cell calculation used would correspond to a 60° θ and finally to a 90° θ . The accurate strain at which the switching must occur remains to be defined. As the martensite particles reorient during straining, the macroscopic strain at which the switching would occur could be the one for which a threshold particle angle with respect to loading is reached. Figure 6.16(a) shows a schematic illustrating the particle angle and Figure 6.16(b) shows the evolution of the particle angle with macroscopic strain in the case of a prolate particle for different initial orientations. $V_{\alpha'}$ is fixed to 30% and $C_{\alpha'}$ to 0.15wt.%.

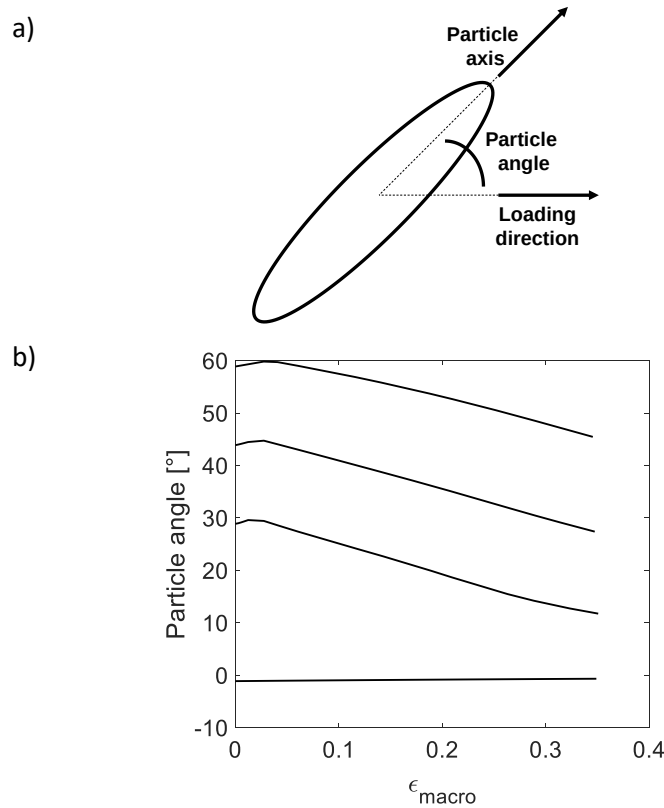


Figure 6.16 (a) Schematic illustration of the particle angle and (b) evolution of the prolate particle angle with macroscopic strain for different initial orientations.

An important advantage of using a damage model based on a cellular automaton approach is the possibility of taking into account several microstructural parameters at once, which is missing in the present version of the model. A key addition to the model is thus the capability of generating a microstructure with several regions, each of which would have a different set of microstructural parameters. Varying the orientation in each region would for instance enable to model the platelet-like microstructure more realistically, since it is composed of regions of different particle orientations. However, using the available unit cell calculations, when considering a microstructure made of various regions with different orientations, the local fields at the region edges will not accurately represent the physical reality. Indeed, the local fields correspond to a periodic distribution of particles, which ceases to apply at the edges.

Finer model improvements would be to consider more complete and accurate micromechanical models for each step of the damage process.

Regarding void nucleation, the critical nucleation stress distribution, that could be based on the more physical maximum normal stress rather than the maximum principal stress, can vary with the microstructural parameters. For instance, *Landron et al. (2010)* have proposed a model for the critical stress by decohesion that depends both on martensite volume fraction and martensite hardness (and thus on martensite carbon content). It increases up to a martensite volume fraction equal to 50% and decreases afterwards.

The number of void nucleation sites is an influential parameter in damage behavior as an increase in the number of voids decreases void spacing. For now, the possibility of one void per particle only has been implemented, whatever the case, even though the sites number changes with martensite particle density and morphology. For instance, in platelet-like microstructures, the particle tips in the loading direction are the most probable sites for nucleation, which equates to at most two voids per particle. In equiaxed microstructures, real particles are non spherical and exhibit various protrusions that could be damage nucleation sites. An additional parameter influencing relative void spacing is void size, which also directly impacts void volume fraction. In order to correctly capture the experimental results, the void size at nucleation should be the one observed experimentally. Therefore, very small voids should nucleate in the case of the microstructure with disk particles compared to the microstructure with spherical particles. This would confer a higher damage resistance to the platelet-like morphology.

Finally, it has been observed in Chapter 5 that voids nucleate both by interface decohesion and by particle breaking, the dominating mechanism depending on the initial microstructure. The extension to both nucleation mechanisms, feasible in the framework of the model, would therefore be a beneficial improvement.

Regarding void growth, as seen in Chapter 5, the voids are much smaller in platelet-like microstructures compared to equiaxed microstructures. As sub-micron voids have a limited growth rate (*Liu et al., 2003; Tvergaard and Niordson, 2004; Liu et al., 2005; Borg et al., 2008*), the void growth model

could take into account void size. (*Liu et al., 2005*) found that the void growth rate of sub-micron size voids at large strain is proportional to the square of the mean stress, which contrasts with the exponential growth predicted by the Rice and Tracey model. This would again accentuate the superior damage resistance of the platelet-like morphology and solve the issue of the premature large void increase. The void growth model could additionally take into account void shape in case of particle breaking as this latter void nucleation mechanism leads to penny-shaped voids.

Regarding the onset of void coalescence, the most important parameter governing coalescence is the void spacing that the Brown and Embury criterion already accounts for. However, coalescence occurs too rapidly for it to be realistic. Perhaps, the more accurate Thomason criterion (*Thomason, 1985*) could solve this issue.

With a realistic void growth rate and coalescence rate, the addition of a *fracture criterion* would enable predicting fracture strain. A possible criterion would be the attainment of the maximum in the curve of the number of microcracks. Indeed, once that maximum attained, one can assume the beginning of the formation of a large crack coalescing with all voids and leading to final failure.

The proposed perspectives essentially boil down to providing the most realistic local stress and strain fields possible and tuning the damage evolution laws. Finally, as a more general perspective, the model can be extended to cracking resistance by providing it with realistic local fields that occur at the tip of a crack.

Chapter 7

Conclusions & Perspectives

The objective of the thesis is to investigate the mechanical behavior of Dual-Phase steels with a platelet-like microstructure. Dual-Phase steels are generally relatively successful in attaining an interesting compromise between strength and ductility, in spite of a simple and inexpensive composition. The project was born from previous (but recent) results, namely that Dual-Phase steels exhibiting a ‘Thomas fibers’ microstructure could potentially reach high levels of fracture toughness (*Pierman, 2013*). A high fracture toughness is a crucial mechanical property on top of strength and ductility to extend the use of Dual-Phase steels in structural components that must resist cracking, or for some forming operations.

Seeing the possibility of a high fracture toughness in ‘Thomas fibers’ Dual-Phase steels compared to traditional equiaxed Dual-Phase steels, the main objective of this thesis was to unravel the microstructural mechanisms leading to the high fracture resistance. In order to achieve this objective, mechanical tests and micromechanical modeling were both carried out. Cracking resistance being linked to damage, which is influenced by plasticity, this work sweeps across these different aspects of mechanical behavior. This closing Chapter 7 recalls the major findings along with the general methodologies that have led to them, and is ended by a proposal of future works.

Metallurgy and metallography

As a first step of an experimental study, model microstructures were developed and their elastoplastic properties characterized. Steels were developed at two controlled martensite volume fractions and two controlled thin sheet thicknesses, but at identical global composition. Furthermore, the

two types of morphologies were investigated for a reference purpose. A 3D microstructural characterization revealed the platelet-like form of the martensite particles in 'Thomas fibers' microstructures.

Modeling of plastic behavior

A two-level micromechanical model was proposed based on FE simulations on periodic unit cells representing grains. The first level is the one of a grain in which the martensite particles are similarly oriented. The second level is the macroscopic level, representing an aggregate of randomly oriented grains. The way morphology is changed is through the aspect ratio. Spherical particles were selected to represent the equiaxed microstructure and disk particles were selected to represent the platelet-like microstructure. The findings are the following. At grain level, particle aspect ratio and orientation significantly influence the plastic response. Unit cells loaded at angles equal to 0° or 90° with respect to the revolution axis exhibit the highest tensile strengths and lowest uniform deformations; the opposite is found for angles around 45° . The impact of orientation moreover increases as the aspect ratio departs from one. In contrast, particle aspect ratio has limited impact on the effective behavior of an isotropic aggregate of randomly oriented grains. Particle aspect ratio and orientation considerably affect local mechanical fields, and hence grain damage and fracture behavior. The presence of randomly oriented grains in the microstructure leads to a heterogeneous damage accumulation at grain scale. Based on this and the need of a high scale at which energy must be dissipated for a large fracture toughness, a two-stage crack propagation process was suggested to explain the high cracking resistance observed in platelet-like microstructures: (i) a first level intragrain crack propagation associated to the scale of particle spacing and (ii) a second level intergrain crack propagation associated to the scale of a martensite packet size. Figure 7.1 illustrates the two-stage process.

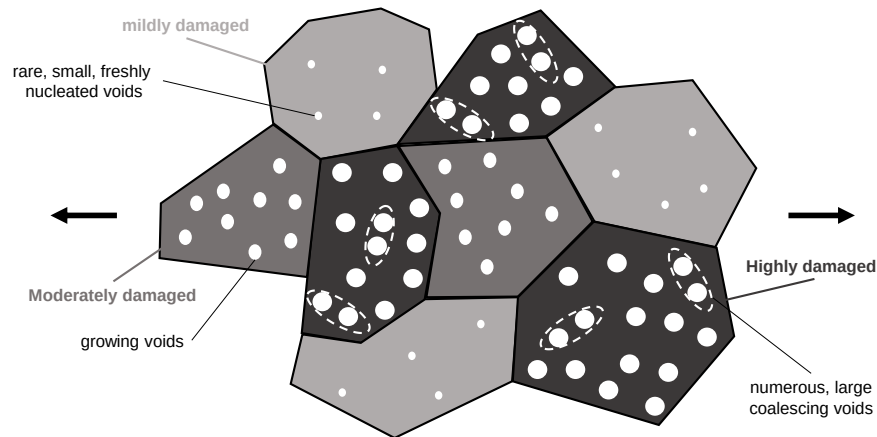


Figure 7.1 Schematic illustration of the heterogeneous damage accumulation in platelet-like microstructures.

Fracture characterization

The essential work of fracture method was systematically applied and revealed a higher cracking resistance of platelet-like microstructures in thin sheets, confirming the previous results in thick sheets. This conclusion is also supported by hole expansion tests. As deduced from the EWF study and shown in Figure 7.2, a reason for the higher resistance in thin sheets is the larger contribution of necking in platelet-like microstructures. This is in turn due to the larger deformation capacity of the platelet-like Dual-Phase steels, as demonstrated by the larger average equivalent fracture strain.

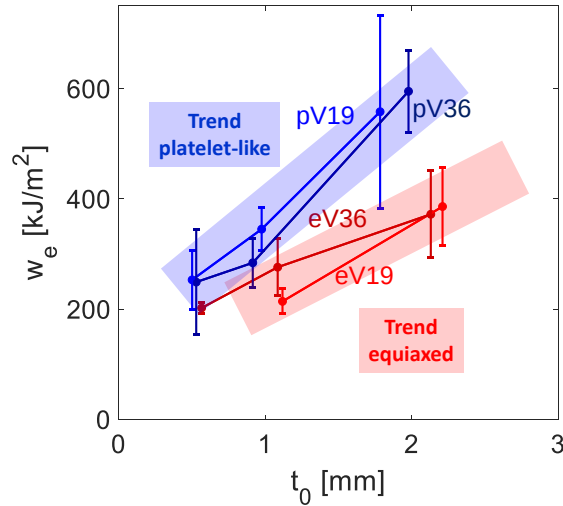


Figure 7.2 Essential work of fracture as a function of initial sheet thickness. The essential work of fracture is larger in platelet-like microstructures and increases more rapidly with thickness, i.e. the necking contribution is more important.

The subsequent damage analysis of transverse sections of DENT and uniaxial tensile specimens showed very few and very small damage occurrences in platelet-like microstructures compared to large and numerous voids in equiaxed microstructures. The analysis furthermore led to an additional possible reason for the high nucleation resistance, namely the minimization of nucleation driving force through an alignment of the martensite particles with the direction of loading as recalled in the schematic of Figure 7.3. Furthermore, the martensite packets that are initially parallel to the loading direction deform less, supporting the heterogeneity in the behavior between the differently oriented martensite packets.

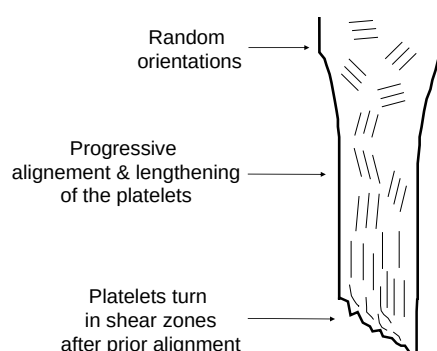


Figure 7.3 Schematic illustration in a uniaxial tension specimen of the alignment of martensite particles with loading direction in a platelet-like microstructure.

Modeling of damage behavior

Damage behavior was modeled through the use of a cellular automaton approach model, to which the data extracted from the elastoplastic analysis were fed. The benefit of a cellular automaton approach, enabling to incorporate a large number of different particles distributed differently in the microstructure, is the possibility of taking into account the statistical aspects related to the microstructural heterogeneities, while limiting the complexity of the damage evolution equations. The latter are the attainment of a critical stress given through a Weibull distribution for void nucleation, Rice and Tracey for void growth and Brown and Embury for void coalescence.

The model, although it still needs to be improved, predicts a large variation in damage behavior when changing the orientation of the particles with respect to loading, supporting once more the heterogeneous damage accumulation.

Perspectives

The approach used for plastic behavior can be extended to other parameters. For instance, the effects of particle relative spacing and of martensite interconnectivity can be assessed by varying the mesh of the unit cells. The

impact of the refinement effect can be addressed through strain-gradient plasticity and the impact of crystallography using crystal plasticity. The intraphase heterogeneities (e.g. chemical heterogeneities) could be taken into account through a spatially dependent hardening behavior.

The approach could also be applied to other two-phase (or more) materials such as third generation steels that exhibit a lamellar microstructure or even to other classes of metallic alloys such as α/β Ti alloys. Such materials could be developed and tested to verify the generality of the conclusions.

Furthermore, given the difference in heat treatments between a conventional equiaxed and a platelet-like microstructure, the microtexture of the ferritic matrix probably varies from one morphology type to the other. As the crystal misorientation distribution influences the local mechanical fields, a thorough characterization of the ferrite phase would bring more insight into the plastic behavior. This could potentially help in explaining the large cracking resistance as the local plastic behavior of the ferrite matrix determines the degree to which the martensite particles will have to deform to align with the loading direction, the alignment being a key feature of the high-toughness platelet-like Dual-Phase steels.

Another possible key feature affecting the mechanical behavior is the microstructural banding in the direction of rolling observable in the equiaxed microstructures (see Figure 3.2) that would appear even more clearly in the initial ferrite-pearlite microstructure. An additional characterization of the ferrite-pearlite microstructure would therefore be useful to assess the degree of banding. Microstructural banding is the result of an underlying chemical segregation, which affects the final mechanical properties. Characterizing the initial carbides spatial distribution and the segregation, especially of carbon and manganese as both elements increase martensite hardness and thereby increase the local phase contrast, could further clarify the final behavior. This also holds for the platelet-like microstructures even though microstructural banding is not readily visible due to the complete austenitization during the first step of the heat treatment.

Mechanical tests involving non-proportional loading paths could be performed in order to determine the behavior of the platelets for more general loading conditions found in real-life applications other than uniaxial loading.

The ultimate aim of the cellular automaton model for damage is to quantitatively predict fracture strain. The perspectives of it have been extensively discussed within Chapter 6. As a recall, the most important issues that remain to be resolved for a proper prediction of fracture strain in platelet-like Dual-Phase steels are: (i) having accurate values of local fields available, which involves taking into account the alignment of the particles and the stress triaxiality evolution, and (ii) using accurate damage evolution laws, which involves a void growth model depending on void size. Further possible heterogeneities are a distribution of martensite particle sizes and of void nucleation stresses.

A subsequent ambition following the understanding of the mechanisms is to predict in a quantitative manner the cracking resistance based on the microstructural parameters. Additionally, an integration of several physics-based models could achieve a multicriteria optimization that takes into account cracking resistance in addition to strength and ductility.

Appendix A

Heat treatments tuning

In order to achieve predetermined microstructural values for the model microstructures, a trial and error study was first undertaken to fix the heat treatment parameters. To do so judiciously, information was first sought about the temperatures at which austenite and to a lesser extent martensite are formed.

Various phenomenological formulae based on composition exist to give an approximate idea of the start and end transformation temperatures. Among them, the *Andrew's formulae* for Ac_1 , Ac_3 , M_s and the *Steven & Haynes formula* for B_s are commonly used (*Steven and Haynes, 1956; Andrews, 1965*):

$$Ac_1[^\circ C] = 723 - 10.7 Mn - 16.9 Ni + 29.1 Si + 16.9 Cr + 6.38 W + 290 As \quad A.1$$

$$Ac_3[^\circ C] = 910 - 203 \sqrt{C} - 15.2 Ni + 44.7 Si + 104 V + 31.5 Mo + 13.1 W - 30 Mn - 11 Cr - 20 Cu + 700 P + 400 Al + 120 As + 400 Ti \quad A.2$$

$$M_s[^\circ C] = 539 - 423 C - 30.4 Mn - 17.7 Ni - 12.1 Cr - 11 Si - 7.5 Mo \quad A.3$$

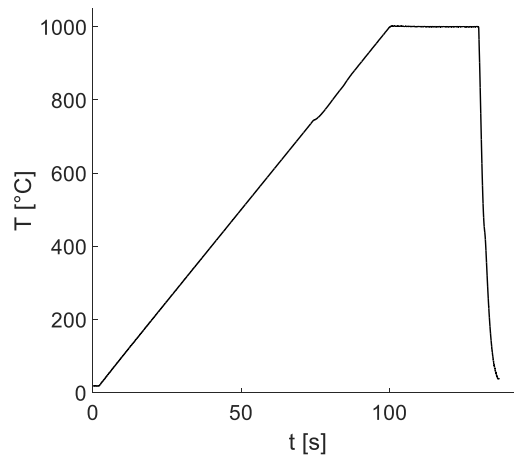
$$B_s[^\circ C] = 830 - 270 C - 90 Mn - 37 Ni - 70 Cr - 83 Mo \quad A.4$$

However, the given formulae do not consider factors other than composition that can also play a role on kinetics, such as defect density, grain size, plate thickness... For this reason, an experimental determination of the start and end transformation temperatures for the investigated steel grade was performed using dilatometry.

A.1 Dilatometry

The determination of the transformation temperatures was performed by dilatometry. Heat treatments were applied by induction heating and helium cooling.

4x10x1 mm³ samples were heat treated following schedules as shown in Figure A.1. Samples are first heated up to 1000°C followed by a ~ 210 °C/s cooling, as estimated by the dilatometry test data. The aim was to determine the transformation temperatures relevant for ferrite-martensitic Dual-Phase steels, namely A_{c1} , A_{c3} , M_s and M_f .



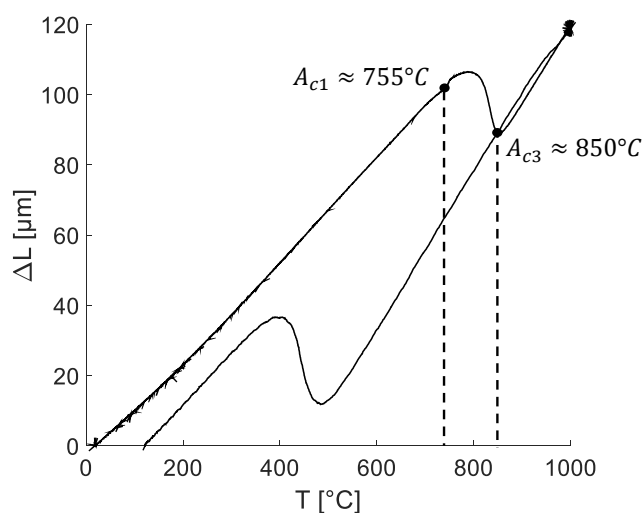


Figure A.2 Dilatation curve of the investigated steel grade corresponding to the heat treatment given in Figure A.1.

Even though the aim was to measure the martensite start and end transformation temperatures M_s and M_f , the metallographic characterization of the dilatometry test sample showed that no martensite was formed. Figure A.3 shows that the formed phase was bainite.

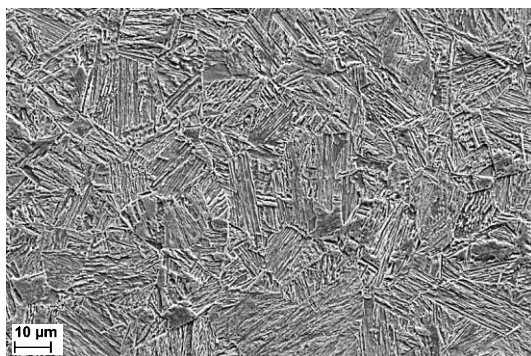


Figure A.3 A 100% bainitic microstructure ensuing from the heat treatment given in Figure A.1.

This implies that the ~ 210 °C/s quenching cooling rate was not sufficiently fast for martensite formation. Another proof that the cooling rate was not sufficiently high is that the transformation took place at a higher temperature than what would be expected for a martensite transformation.

Table A.1 provides transformation temperatures of the studied grade, obtained using the phenomenological formulae and using dilatometry.

	<i>Phenomenologic formulae</i>	<i>Dilatometry test</i>
Ac_1 [°C]	717	755 ± 40
Ac_3 [°C]	836	850 ± 15
M_s [°C]	451	/
B_s [°C]	610	550 ± 70

Table A.1 Start and end transformation temperatures using phenomenological formulae and dilatometry.

The phenomenological formulae given by Equations A.1 to A.4 provide realistic results, giving values in agreement with the dilatometry test mean and standard deviation. As a matter of fact, the Ac_1 temperature is at most equal to 735°C. Indeed, as was proved in Chapter 3, a metallographic observation revealed martensite after a heating up to 735°C.

A.2 Temperature acquisition

To make sure the cooling rate of water quenching is sufficiently high for martensite formation and to gain information on the heat treatments, a thermocouple K coupled to a data acquisition device (Agilent) was used to determine more precisely the temperature during the heat treatments, heating and quenching included. The results are relatively reproducible. An example of water quenching is given in Figure A.4.

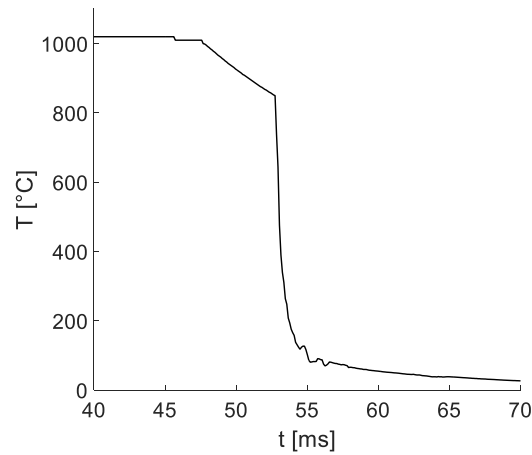


Figure A.4 Temperature acquisition during a water quenching.

Water quenching has been shown to lead to martensite formation. For instance, a heating up to 1000°C maintained for 30 min followed by a water quenching leads to a 100% martensite microstructure as shown in the micrograph of Figure A.5.

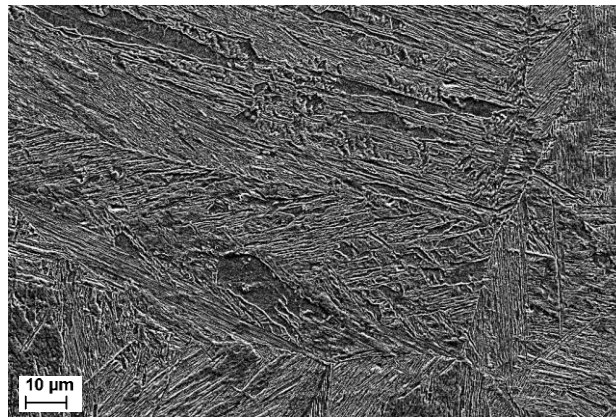


Figure A.5 A 100% martensitic microstructure obtained through a water quenching at room temperature.

From the data of Figure A.4, the cooling rate during the water quenching is around 735 °C/s. The first decreasing slope is due to the time spent in air starting from the removal of the sample from the furnace and ending with its introduction in water; the cooling rate then corresponds to ~ 30 °C/s.

Appendix B

Impact of heterogeneities, martensite interconnectivity and internal stresses on the plastic behavior of Dual-Phase steels

The stress-strain curves predicted with the model of Chapter 4 have been compared to the experimental curves, see Figure B.1. The predicted curve corresponds to the aggregate response computed using an aspect ratio equal to 1 (spherical shape), a martensite volume fraction equal to 19% and a martensite carbon content equal to 0.24wt.%. The experimental curve corresponds to a pV19t2 steel. The model does not capture the elastoplastic transition and the small plastic strain response. The conclusion would remain the same with a disk particle as the aspect ratio has only a negligible impact on the aggregate response (see Chapter 4).

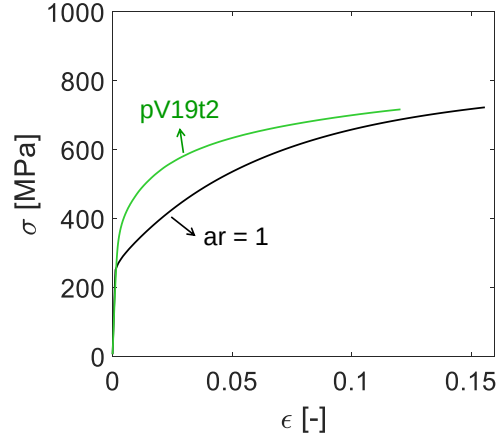


Figure B.1 Experimental stress-strain response of a pV19t2 steel and predicted stress-strain response of a steel with $ar = 1$, $V_{\alpha'} = 19\%$ and $C_{\alpha'} = 0.24\text{wt.}\%$.

Various limitations of the model, that have been stated at the end of Chapter 4, can explain this shortcoming. The aim of the present Appendix B is to assess some of these limitations in order to identify the most impactful features. The selected limitations are: (i) the heterogeneities of other microstructural parameters than orientation, (ii) the effect of a percolating network of martensite even at low martensite volume fractions, (iii) the presence of initial residual stress.

B.1 Impact of heterogeneities

At first, the goal is to investigate the magnitude of the effects that can be expected when taking into account a heterogeneous distribution in microstructural parameters. This is performed by defining an aggregate response as an average of several responses, the average depending on other parameters than solely orientation, unlike what is performed in Chapter 4. The case of a distribution in martensite volume fraction is presented because of the large impact of martensite volume fraction on effective properties compared to the carbon content and morphology. Figure B.2 shows results for an overall martensite volume fraction of 19%. The martensite carbon content and the aspect ratio are kept fixed to 0.3wt.% and 0.2 respectively. Figure B.2(a) shows four distributions: (i) a homogeneous 19%, (ii) a discrete version of a Gaussian type distribution with

a mean equal to 19%, (iii) an extreme case of a scattered distribution and (iv) an even more extreme case.

Figure B.2 shows the predicted stress-strain curves corresponding to the four distributions. The effect of microstructural heterogeneity on plastic behavior is quite small. Even in the extreme cases, distribution effects lead to differences in strength of about 5% at 0.1 of strain as shown in Figure B.2(b). Such modest effects are most probably not the origin of the discrepancy when comparing experimental and predicted curves.

The same reasoning may be applied to a distribution of martensite carbon content or of second phase aspect ratio. The impact is then negligible as the influence of the latter parameters on effective properties is modest compared to the influence of martensite volume fraction.

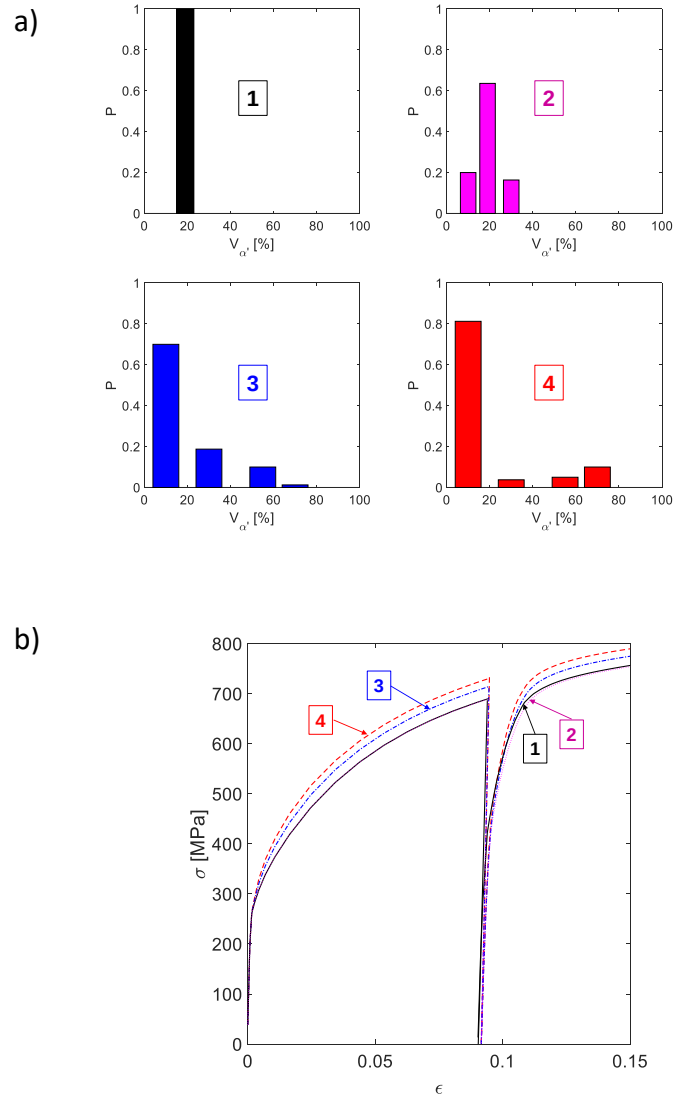


Figure B.2 Distribution effects only have a negligible influence on the global plastic behavior in realistic distributions. a) Four distributions of martensite volume fraction for a 19% average and b) the corresponding stress-strain responses for $C_{\alpha'} = 0.3\text{wt.}\%$ and $ar = 0.2$.

B.2 Impact of martensite interconnectivity

In Dual-Phase steels, martensite interconnectivity already occurs at low martensite volume fractions. For instance, using holotomography, (*Landron*

et al., 2012) observed islands of interconnected martensite in a steel with a martensite volume fraction equal to 11%.

Moreover, Figure B.2 showed that there is an effect of interconnectivity observed when introducing unit cells with a percolating network of martensite (i.e. unit cells with a ferritic particle). Indeed, the two extreme cases 3 and 4 involved, in the average, unit cells with a high martensite volume fraction, with interconnected martensite. It led to a higher strain hardening despite the small proportion of cells with interconnected martensite overall (to guarantee the 19% martensite volume fraction average). Hence, in order to quantify the effect of a percolating martensite on plastic behavior, simulations were launched for a star-like network shown in Figure B.3.

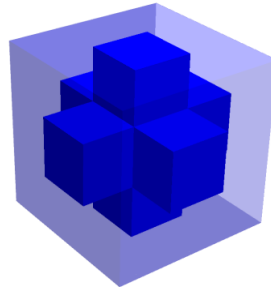


Figure B.3 Illustration of the star-like network.

Figure B.4 shows the stress-strain response of the aggregate response with a star-like network in addition to the pV19t2 experimental stress-strain response and to the predicted curve for an aggregate response with $V_{\alpha'} = 19\%$, $C_{\alpha'} = 0.24\text{wt.}\%$ and $ar = 1$. The aggregate response of an interconnected network is closer to the experimental curve. The strength is higher and the elastoplastic transition slightly sharper as interconnected martensite accommodates a larger part of the overall deformation. The prediction is not fully correct though, suggesting that another key feature is still lacking.

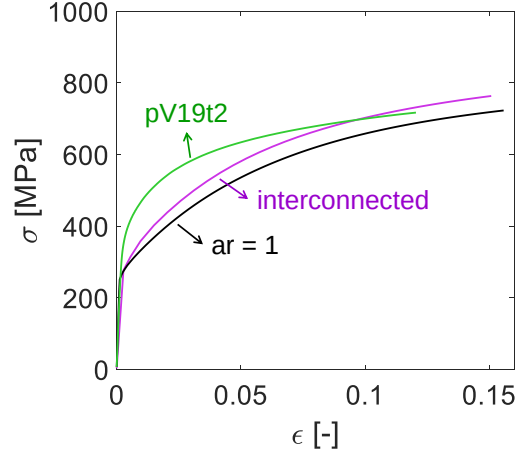


Figure B.4 Experimental stress-strain response of a pV19t2 steel, predicted stress-strain response of a steel with $ar = 1$, $V_{\alpha'} = 19\%$ and $C_{\alpha'} = 0.24\text{wt.}\%$, predicted stress-strain response of a steel with a star like network, $V_{\alpha'} = 19\%$ and $C_{\alpha'} = 0.24\text{wt.}\%$.

B.3 Impact of internal stress

Regardless of the microstructural details of Dual-Phase steels, the last step of the heat treatment is always a water quenching step. During this step, the austenite-to-martensite transformation is accompanied by a volume expansion, leading to the generation of internal stress. Indeed, the austenite has a more compact FCC arrangement than the BCC martensite (or the even less compact BCT arrangement depending on the carbon content). The volume expansion can be quantified on the basis of the atomic packing factors (APF) corresponding to the crystal packings. The APF is defined as

$$APF = \frac{N_{particle} V_{particle}}{V_{unit\ cell}} \quad B.1$$

where $N_{particle}$ is the number of particles in the unit cell, $V_{particle}$ is the volume of the individual particles, and $V_{unit\ cell}$ is the volume of the unit cell.

Hence, particles being iron in all cases, the volume expansion corresponding to the austenite-to-martensite transformation can be derived from the ratio of the APFs.

$$\frac{V_{BCC}}{V_{FCC}} = \frac{APF_{FCC}}{APF_{BCC}} = \frac{0.74}{0.68} \approx 1.09. \quad \text{B. 2}$$

This roughly corresponds to a 9% volume expansion.

An artificial way of mimicking the volume expansion associated to the phase transformation is used to introduce residual stresses and draw a rough idea of their impact: heating the unit cell prior to loading, giving the martensite particle a thermal expansion coefficient and keeping it to zero for the ferrite matrix. The linear thermal expansion coefficient α and the volumetric thermal expansion coefficient β are expressed as

$$\alpha = \frac{\Delta L}{L_0 \Delta T}, \quad \text{B. 3}$$

$$\beta = \frac{\Delta V}{V_0 \Delta T}, \quad \text{B. 4}$$

where $\Delta L/L_0$ is the fractional change in length, $\Delta V/V_0$ is the fractional change in volume, and ΔT is the change in temperature. One has

$$\beta \approx 3\alpha \quad \text{B. 5}$$

when neglecting the terms of second and third order and making the hypothesis of isotropy.

The values used for the computations are $\alpha = 5 \cdot 10^{-5} \frac{m}{m.K}$ for martensite and 0 for ferrite, $\Delta T = 600^\circ C$. (A density of 8000 kg/m³, a specific heat of 450 J/kg.K for both phases, a conductivity of 55 W/m.K for martensite and 0 for ferrite were additionally taken.) This leads to an expansion of 9%.

$$\frac{\Delta V}{V_0} = \beta \Delta T = 15 \cdot 10^{-5} \cdot 600 = 0.09. \quad \text{B. 6}$$

Figure B.5 shows the predicted stress-strain response with internal stress (spherical shape) in addition to the pV19t2 experimental stress-strain response, the predicted curve without internal stress (spherical shape) and the predicted response with a star-like network. With internal stress, the strain hardening capacity is higher, especially at the elastoplastic transition and at small strains, when internal stresses are taken into account. The

predicted strain hardening at small strains is much larger when internal stresses are present in the microstructure prior to loading (see Chapter 2). The reason is the following. The apparent yield stress is always lower in presence of initial residual stresses. Indeed, because the sum of all residual stresses is equal to 0, some regions with forward stress components compared to the loading will initiate plastically at lower applied stress than in the absence of internal stress. The plastic transition will take longer with some regions with backward internal stress components remaining elastic up to larger loads. These “elastic” regions contribute to an apparently larger strain hardening.

Figure B.5 shows that taking into account the residual stresses due to thermal expansion brings the predicted mean stress-strain response closer from the experimental stress-strain curves. In particular, the elastoplastic transition is much better predicted.

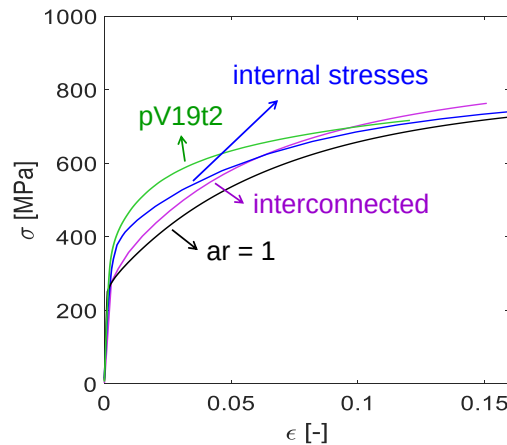


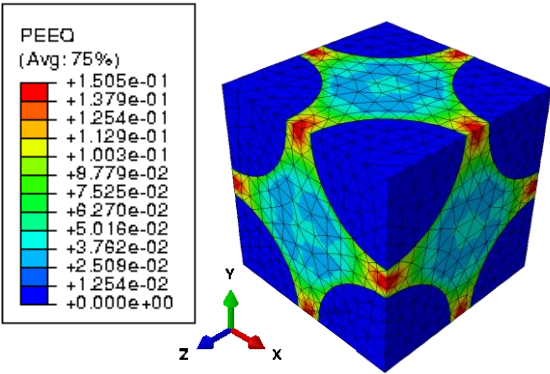
Figure B.5 Experimental stress-strain response of a pV19t2 steel, predicted stress-strain response of a steel with $ar = 1$, $V_{\alpha'} = 19\%$ and $C_{\alpha'} = 0.24\text{wt.}\%$, predicted stress-strain response of a steel with a star like network, $V_{\alpha'} = 19\%$ and $C_{\alpha'} = 0.24\text{wt.}\%$ and predicted stress-strain response of a steel with $ar = 1$, $V_{\alpha'} = 19\%$ and $C_{\alpha'} = 0.24\text{wt.}\%$ after thermal expansion of martensite.

Hence, accounting for the initial residual stresses seems to be the only way to capture the elastoplastic transition properly.

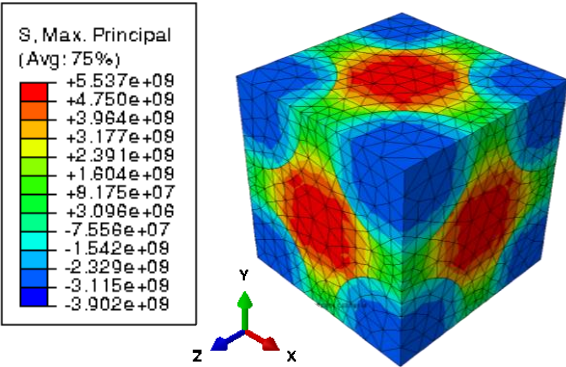
In order to visualize the effect of the phase transformation on the local fields, Figure B.6 shows contour plots of different mechanical properties following the martensite thermal expansion for $V_{\alpha'} = 30\%$, $C_{\alpha'} = 0.15\text{wt.}\%$ and a 12% volume expansion. Figure B.6(a) shows the accumulated plastic strain. The most confined ferrite, found where the accumulation plastic strain is the largest, is located right in the middle of the unit cell's edges, trapped between the martensite particles 'tips'. The deformation zones in front of both tips overlap. This occurs on every edge as the expansion is isotropic. There is no accumulated plastic strain within particles as the nature of the expansion is thermal. Figure B.6(b) shows the maximum principal stress. Martensite particles are in a compression state because of their expansion in a confined ferrite matrix. The ferrite away from the martensite particles is in a state of traction in order to balance the compressive stresses arising from the expansion. Figure B.6(c) shows the deformation in the x direction (as given by the reference system). In this direction, the ferrite between the particles is compressed by the expanding martensite. The same holds for the y and z directions as the expansion is isotropic. This corroborates the conclusion drawn from Figure B.6(a). Figure B.6(d) shows the stress in the x direction and supports the same conclusion. Again, the same holds for the y and z directions. Furthermore, tensile stresses occur away from martensite in order to guarantee a balance of residual stresses.

The latter compressed zones, in the case of isotropic hardening, are the zones that yield last and that contribute to the high strain hardening.

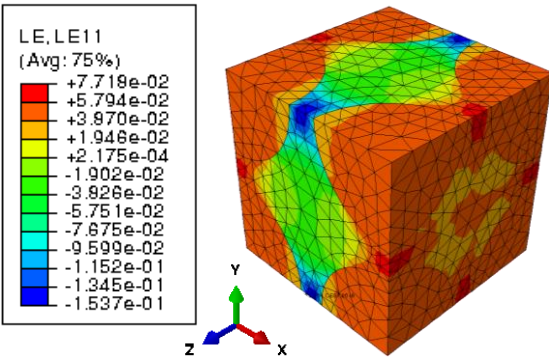
a)



b)



c)



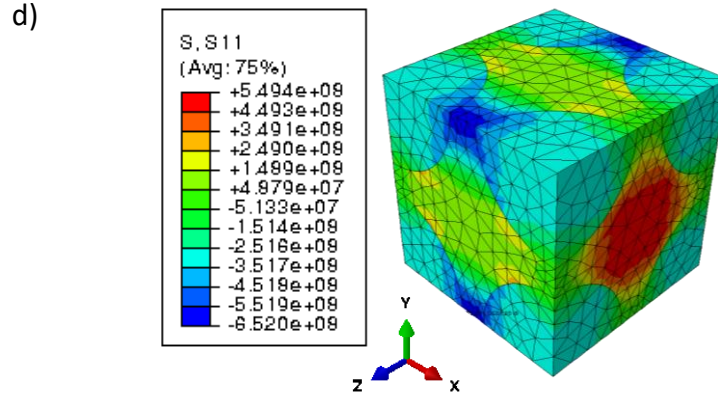


Figure B.6 Contour plots for unit cells after martensite thermal expansion with $V_{\alpha'} = 30\%$, $C_{\alpha'} = 0.15\text{wt.}\%$ and $a_r = 1$ showing a) accumulated plastic strain, b) maximum principal stress, c) strain in direction x, d) stress in direction x.

To conclude, despite the rudimentary nature of the calculations in this Appendix B, they demonstrate that proper account of martensite interconnectivity and residual stresses is needed to accurately predict the stress-strain curves. Heterogeneities however only mildly impact effective plastic behavior. This agrees with (Abid et al., 2015).

Appendix C

Micro plastic localization

The analysis of the local deformation patterns brings an additional explanation for the change in behavior when changing the orientation of the loading with respect to the microstructure unit cell observed in Chapter 4.

Figure C.1 shows contour plots of accumulated plastic strain with the loading orientation at 0° and 45° with $V_{\alpha'} = 30\%$, $C_{\alpha'} = 0.15\text{wt.}\%$, $ar = 1$ and $\varepsilon_{macro} = 0.1$. Strain localizes in both cases between the hard martensite particles in the direction of loading. This leads to a deformation band (i.e. a region whose form is close to that of a band in which the strains are higher) passing through the center of the particles and the ferrite matrix joining them. The location of the band between the hard particles in the direction of loading (shown in grey in Figure C.1) is due to the higher confinement of the ferrite. Therefore, strain localization is more pronounced at 0° compared to 45° because of a higher spatial confinement in the direction of loading at 0° . This leads to a larger proportion of ferrite, hence of soft phase, participating to a larger extent to the macroscopic deformation at 45° , justifying the lower strength at 45° compared to 0° , see Chapter 4.

The strains in ferrite are the highest right in the middle of the two particles in the direction of loading because the two plastic regions in front of the particle 'tips' overlap at this spot. In an equivalent way, higher strains are also present at 45° where the two plastic regions (oriented at 45°) overlap.

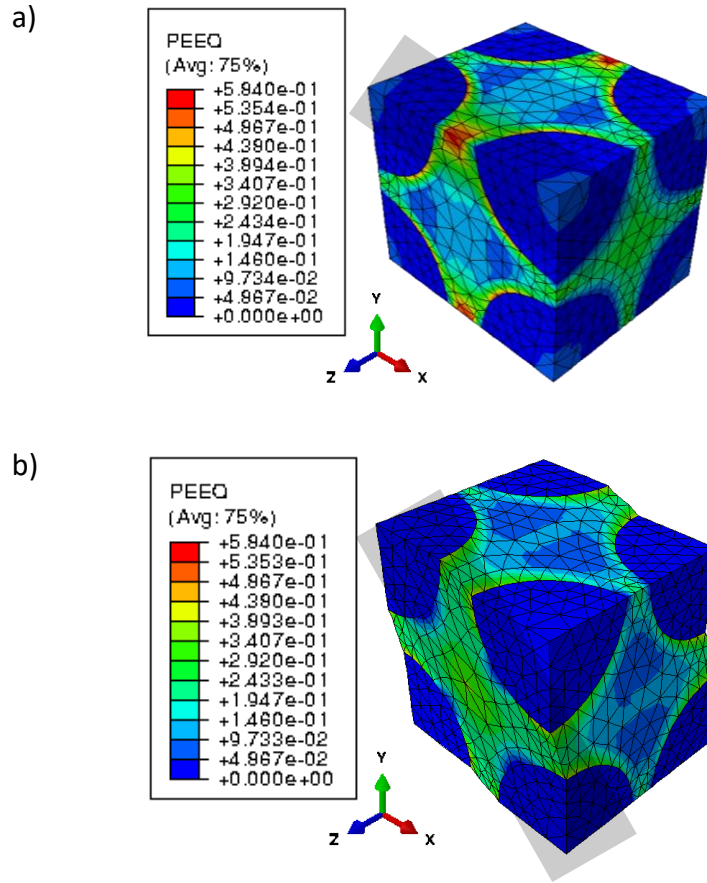


Figure C.1 Contour plots for unit cells with $V_{\alpha'} = 30\%$, $C_{\alpha'} = 0.15\%$, $ar = 1$ and $\varepsilon_{macro} = 0.1$ at a) 0° and b) 45° .

A similar reasoning based on the analysis of the local fields can be applied to a martensitic matrix, explaining again the variation of strength and ductility with orientation. However, the deformation bands are no longer aligned with the direction of loading. The reason is that in a ferritic matrix, the hard phase can follow more easily the deformation of the then freer ferrite, i.e. move as one block, as shown by the almost zero strain in martensite in Figure C.1. With a martensitic matrix, martensite is forced to accommodate a much larger part of the deformation since it is interconnected. It is therefore in regions in which the hard martensite matrix is the furthest away from ferrite particles that it will participate the most to the deformation as there is then less or no soft ferrite to accommodate the deformation in the direction of loading.

Appendix D

Lode angle parameter at particle pole

We calculated the local value of the Lode angle in the deformed unit cell in addition to the variation of the local equivalent strain, stress triaxiality, maximum principal stress and damage indicator D (see Section 4.2.3). The Lode angle is indeed an important parameter in ductile fracture. Figure D.1 shows the variation of the Lode angle parameter $\bar{\theta}$ at the particle pole located on the revolution axis (see Figure 4.10(a)) as a function of the macroscopic applied strain. Figure D.1(a) is for $\theta = 0^\circ$ and Figure D.1(b) is for $\theta = 90^\circ$.

The results partly confirm the conclusions of Section 4.2.3 . At 0° , the Lode angle is close to 1 meaning the point at the pole of the particle undergoes essentially tension. Stresses other than tensile (shear) increase with macroscopic strain. This increase is larger as the curvature gets larger. At 90° , the Lode angle is lower. A negative Lode angle indicates compression stresses, the compression being the largest in the case of a prolate particle at the particle pole.

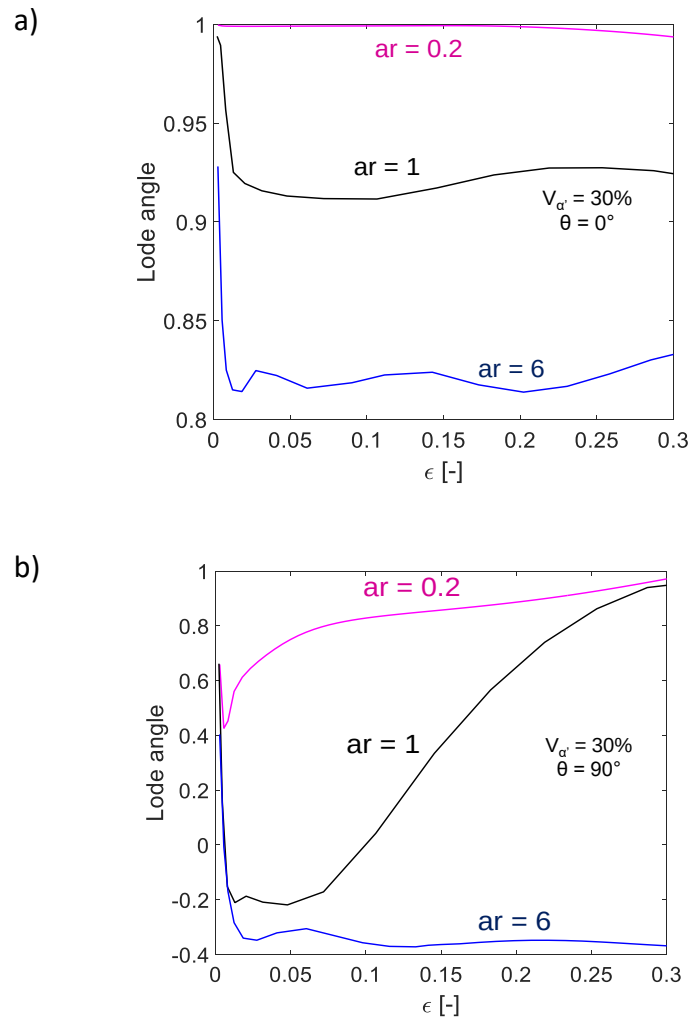


Figure D.1 Evolution with strain of the Lode angle for a) $\theta = 0^\circ$ and b) $\theta = 90^\circ$.

Appendix E

Supporting data – EWF study

Figure E.1(a) shows the force F - displacement u curves at different ligament lengths for the case eV19t1, and Figure E.1(b) shows the force normalized by the maximum force as a function of the displacement normalized by the maximum displacement for the same material. The same overall shape of the curves in Figure E.1(a) and the fact that they are superimposed in Figure E.1(b) indicates that the mechanisms at play are similar. This holds for all other materials tested.

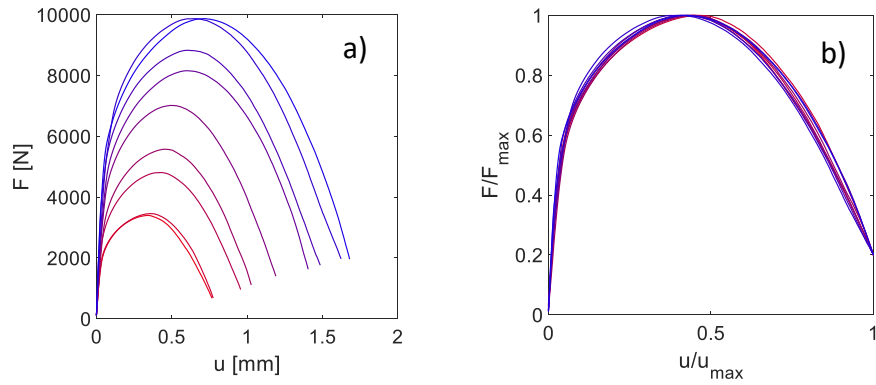
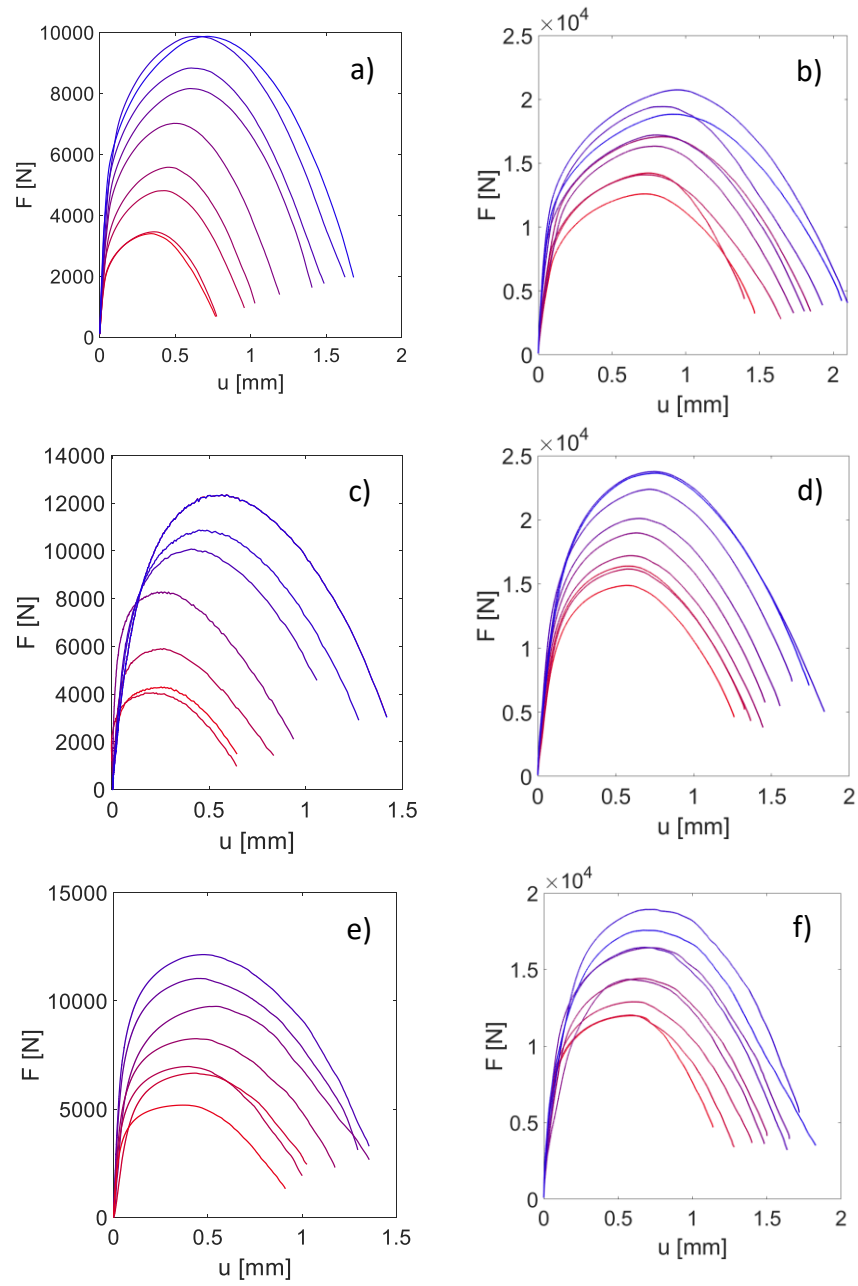


Figure E.1 a) $F - u$ curves at various l_0 for the eV19t1 microstructure and b) $F/F_{\max} - u/u_{\max}$ curves at various l_0 for the eV19t1 microstructure.

Figure E.2 shows the load - displacement curves of all the DENT specimens.



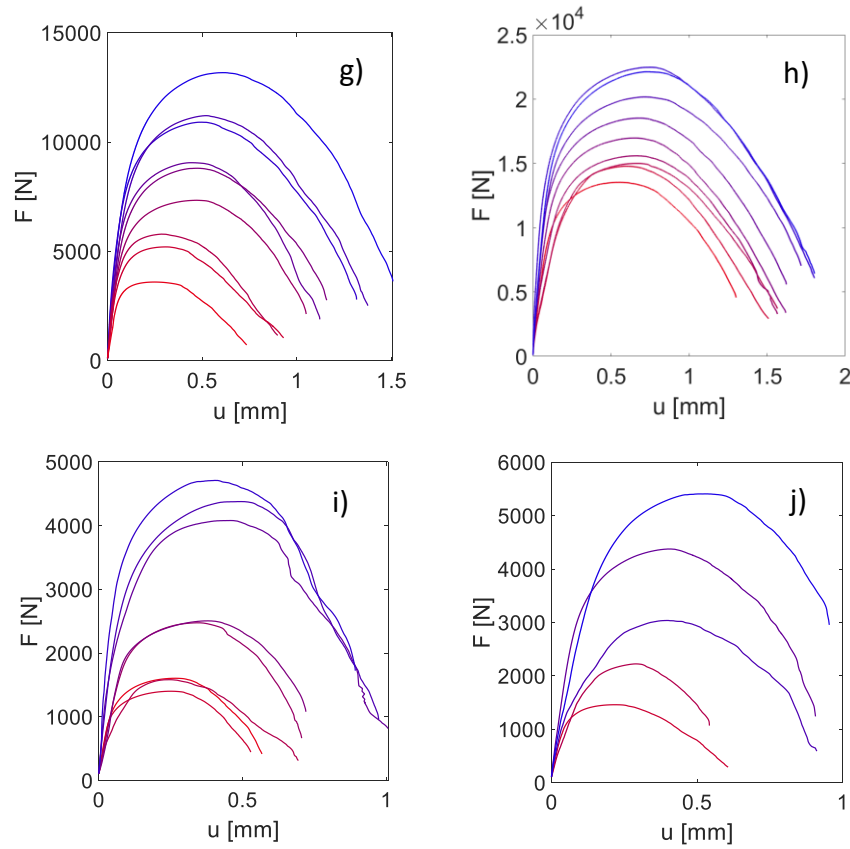


Figure E.2 Load – displacement curves of all DENT specimens: a) eV19t1, b) eV19t2, c) eV36t1, d) eV36t2, e) pV19t1, f) pV19t2, g) pV36t1, h) pV36t2, i) pV19t05, j) pV36t05.

Characterization of the plastic zone by digital image correlation

2D digital image correlation was performed as an additional validity check of the EWF method. The pictures were taken using LIMESS-12 MPixels cameras and the correlation was performed using the software Istra4D. Strain fields of the most critical case, i.e. the sample of the hardest material with the longest ligament (eV36t1 with an initial ligament length of 15 mm), are shown in Figure E.3. As needed for a valid result, the plastic zone is confined between the two notches and the whole ligament deforms plastically during the test. The DPZ and FPZ are visible. A reproducible sequence is observed on every sample. At first, the deformation occurs at the tips only. The entire DPZ forms afterwards (see Figure E.3(a)). A strain concentration along the ligament due to necking then takes place, with invariably a higher strain in

the FPZ (see Figure E.3(b)). Finally, the two FPZs overlap and total separation occurs.

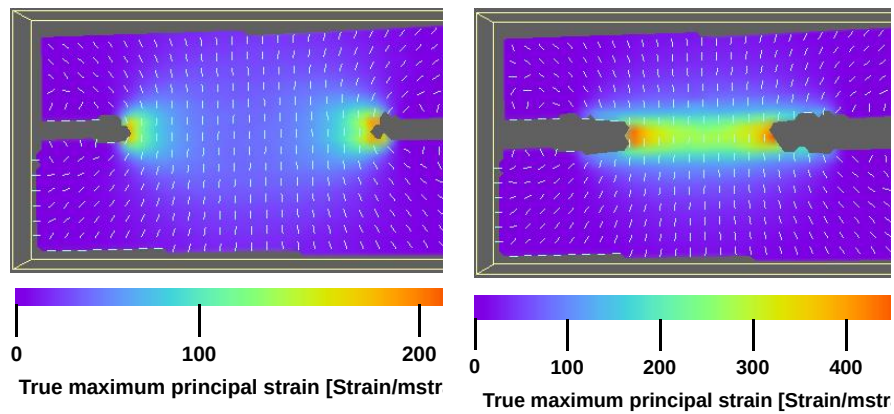


Figure E.3 True maximum principal strain fields at the forming of the DPZ and during crack propagation.

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