Microstructural effects on fracture toughness of ultra-high strength dual phase sheet steels

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ABSTRACT

The influence of microstructure on the fracture toughness of two industrially processed 1000 MPa dual-phase (DP) steel grades is investigated. Crack initiation and propagation resistance are evaluated by means of the essential work of fracture (EWF) methodology and the main damage and fracture mechanisms are investigated. The results are discussed in terms of the proportion and distribution of the different microstructural constituents, which is assessed by scanning electron microscopy (SEM), high-resolution electron backscatter diffraction (HR-EBSD) and nanoindentation hardness measurements. The investigations show that the strain-induced transformation of retained austenite to martensite (TRIP effect), may be detrimental to cracking resistance, even though it increases tensile properties. This phenomenon is attributed to a ‘brittle’ network effect generated by the presence of hard fresh martensite islands in the fracture process zone. The connectivity of the hard secondary phases and the proportion of soft phase (ferrite) also have a major role in fracture toughness. The DP steel with the larger volume fraction of ferrite and homogeneously distributed martensite islands shows significantly higher crack propagation resistance. The contribution of necking to the ductile fracture process is evaluated by means of thickness measurements in fractured DENT specimens and the correlation between the specific essential work of fracture (wE) and tensile properties is investigated. It is concluded that the global formability and cracking resistance of high strength DP steels can be balanced through microstructural tailoring.

1. Introduction

Dual Phase (DP) steels are one of the most extensively used Advanced High Strength Steels (AHSS) for body in white automobile components [1]. Their excellent compromise between high strength and good formability has contributed to their widespread implementation in passenger’s car, allowing vehicle weight reduction and enhancing occupant’s safety [1,2]. These steels belong to the first generation of AHSS family and are characterized by showing high strength, low yield strength to tensile strength (YS/TS) ratio, high strain hardening and high ductility compared to other high strength low-alloy steels [1–3]. Such unique combination of mechanical properties is closely linked to their particular microstructure, basically consisting of a soft ferritic matrix with hard martensite islands embedded. In some cases, other secondary phases, such as bainite or retained austenite, can also be present in different proportions [4,5].

The relationship between microstructure and strength/ductility properties of DP steels has been the focus of extensive research in the last decades [4–14]. Strength level is primarily dictated by the martensite volume fraction; the higher the martensite/ferrite ratio the higher the yield strength and the ultimate tensile strength (typical martensite volume fractions are in the range of 20–50% for tensile strengths of 600–1200 MPa) [5–9]. Generally, such increase of strength is accompanied by a decrease of elongation, both uniform and total [6–8]. Other factors, such as the grain size [10,11], the hardness of the microstructural constituents [12] or their morphology and distribution [13,14] also have influence on mechanical behaviour. Their high strain hardening is caused by the strain gradients between the soft matrix (ferrite)
and the hard secondary phases (martensite) during forming, which generate dislocations pile-ups (geometrically necessary dislocations) in the ferritic matrix to accommodate the plastic incompatibility between the two phases [15]. Such deformation mechanism contributes to increase the work-hardening rate and to delay the onset of localized necking, thus improving the formability. Strain hardening behaviour is mostly influenced by the volume fraction, hardness, size and distribution of martensite [1,5–7,10,13]. Steels containing retained austenite also have an additional contribution to strain hardening due to the strain-induced transformation of retained austenite to martensite (Transformation Induced Plasticity, or TRIP, effect) in the initial stages of deformation [1,4].

Owing to their great ductility and strain-hardenability, DP steels are especially suitable for cold forming operations involving large deformations (deep drawing, stretching, hydroforming, etc.). However, in spite of their good formability, several cracking problems during forming, related to their limited edge formability or hole expansion capacity, have been reported [16–18]. The development of DP steel grades with increasingly higher strength (current grades can reach up to 1200 MPa [19,20]) has incremented the frequency of occurrence of these fractures, being a real concern to automotive part manufacturers. Thus, cracking resistance has become a critical factor limiting the applicability of high strength DP steels.

Even though many works have addressed the influence of microstructure on edge formability (or stretch flangeability) of DP steels [12, 21–26], it is still being a topic of debate. Their rather low edge fracture resistance is generally attributed to the heterogeneous microstructure and the differences in hardness between the microstructural constituents [21–25]. Nevertheless, the identification of a singular microstructural parameter controlling the stretch flangeability of DP steels is very complex, since it depends on the interaction of different microstructural features (martensite volume fraction, hardness, size, morphology, distribution of the phases, etc.) [26]. Furthermore, stretch flangeability is standardly measured by means of the Hole Expansion Test (HET) [27]. The value obtained from this test is the Hole Expansion Ratio (HER), which represents the material’s ability to resist crack formation from a punched hole. The HER is not a material property and depends on the hole preparation method [22,28], which sometimes makes difficult to establish clear relationships between microstructure and edge cracking resistance.

Fracture toughness, measured in the frame of fracture mechanics, has shown to be a determinant property governing AHSS edge fracture resistance [17,26,29–35]. Hence, understanding the effects of microstructure on fracture toughness is essential to develop DP microstructures with improved cracking resistance. However, few studies have been conducted on the relation microstructure-fracture toughness of DP steels [30,36–38]. Tkach et al. [36] noticed an increase of Kc with increasing the martensite volume fraction in a 0.06C–1.8Cr–1.6Ni–0.6Mo steel with different heat treatments. They attributed this effect to the higher toughness of the martensite respect to the ferrite and to the lower carbon content of martensite when applying higher annealing temperatures. Fonstein et al. [30] showed that the crack propagation resistance of a cold rolled DP steel was significantly enhanced replacing part of the martensite by bainite. Bayram et al. [37] found that microstructures showing fine needle-like martensite homogeneously distributed in the ferrite matrix exhibited higher Kc than those with equiaxed grains of ferrite and martensite. Lacroix et al. [38] studied the fracture toughness of different TRIP steels, dual and DP steels and observed that a decrease in the connectivity of martensite grains significantly increased the crack initiation resistance (JC).

The present paper aims at better understanding the influence of microstructure on the crack initiation and propagation resistance of high strength DP steels. For this purpose, the fracture toughness and the fracture mechanisms of two industrially processed 1000 MPa DP steel sheets are investigated and correlated to their microstructural features. Fracture toughness is evaluated by means of the essential work of fracture (EWF) methodology [39]. The method allows to readily evaluate the plane stress fracture toughness of thin ductile sheets, separating experimentally the energy consumed during the tearing process in two terms: the energy dissipated in the fracture process zone (FPZ), called specific essential work of fracture (wε), and the non-essential plastic work (wε) dissipated in an outer region of the fracture. The first term, wε, is an average resistance value for the complete fracture (i.e. it contains energetic contributions from crack initiation and crack propagation resistance) and it is a suitable parameter to measure the ductile tearing resistance of thin sheets [38–43]. It is important noting that, even though wε is independent of specimen geometry and in-plane dimensions [43], plane stress fracture toughness has an additional extrinsic contribution from the necking developed at the crack tip [38–42]. Therefore, it is not strictly a material property but a constant for a given sheet thickness. The non-essential plastic work is a geometry dependent parameter and, thus, cannot be considered a material property. The EWF method has been used in several works to assess the cracking resistance of AHSS sheets [32–35,38,42,44–46]. The mechanical properties and tensile fracture resistance of the two investigated DP steels are evaluated through conventional tensile tests and local fracture strain measurements. Microstructural characteristics are investigated by means of scanning electron microscopy (SEM), high-resolution electron backscatter diffraction (HR-EBSD) analysis and nanoindentation hardness measurements. The results can help to guide microstructural design of high strength DP steels with optimum balance between strength, ductility and fracture resistance.

2. Materials and methods

2.1. Materials

2.1.1. Chemical composition and material processing

Two 1000 MPa DP steels were processed via hot dip galvanizing line (HDGL) at industrial scale. The slabs, obtained by continuous casting, were reheated to a temperature of 1200 °C and hot rolled to a thickness of 3.4 mm. After pickling, the steels were cold rolled to the final thickness (1.35–1.4 mm) and annealed on the HDGL.

A schematic representation of the time-temperature cycles of the HDGL is given in Fig. 1. DP1000-A was annealed in fully austenitic range. Ferrite was formed during cooling to the overaging temperature. While overaging, stabilization as well as further transformation of the remaining austenite to bainite takes place. After overaging, the steel was dipped in liquid zinc bath. Fresh martensite was formed from remaining austenite during final cooling. DP1000-B was soaked in the intercritical range to have a certain amount of ferrite and austenite in the microstructure. At the end of this soaking step, the steel strip was cooled down to the overaging temperature before dipping in a liquid zinc bath. During this overaging step, a main part of austenite is transformed to bainite. At the exit of the zinc bath, the remaining austenite is transformed to martensite during the final cooling. The chemical compositions of the two DP steels are given in Table 1.

2.1.2. Microstructural characterization

Microstructural analysis was performed by means of SEM and HR-EBSD. After mechanical grinding and polishing, SEM samples were electro-polished. HR-EBSD samples were mechanically polished to mirror surface finish with a 0.05 μm colloidal silica suspension. The HR-EBSD measurements were performed at 20 kV with a step size of 0.15 μm. The analysed areas were 311 × 231 μm². The percentage of different phases, as well as the average grain size of ferrite and martensite, was determined by a combination of image analysis, SEM and optical micrographs and EBSD measurements. The amount of retained austenite was validated by means of magnetization saturation measurements [47].

Fig. 1 shows the resulting SEM micrographs. HR-EBSD results and volume fraction and size of the different microstructural constituents are
shown in Fig. 2 and Table 2, respectively. Fig. 2 shows the obtained inverse pole figure (IPF) map, the phase map and the mean angular deviation (MAD) map.

The microstructure of DP1000-A consists of a matrix containing ferrite ($\alpha$) and bainite/tempered martensite ($\alpha_{b}$) with some dispersed martensite and fresh martensite/retained austenite (M/RA) islands. The SEM images reveal a great presence of carbide precipitates within B/TM grains (Fig. 1 a). DP1000-B has a ferritic-bainitic matrix with a slightly lower amount of martensite islands ($\approx$ 27%), which are homogeneously distributed. Contrary to the observed in DP1000-A, carbide precipitation is hardly seen in bainitic (B) areas (Fig. 1 b).

Fig. 2 b shows the HR-EBSD phase constitution map differentiating the BCC phase (ferrite) in blue and the FCC phase (austenite) in yellow. The SEM images reveal a great presence of carbide precipitates within B/TM grains (Fig. 1 a). DP1000-A has a ferritic-bainitic matrix with a slightly lower amount of martensite islands ($\approx$ 27%), which are homogeneously distributed. Contrary to the observed in DP1000-A, carbide precipitation is hardly seen in bainitic (B) areas (Fig. 1 b).

Table 1

| Steel grade | C  | Si  | Mn  | Cr  | B  | Al  | Ti  |
|-------------|----|-----|-----|-----|----|-----|-----|
| DP1000-A    | 0.15 | <0.5 | >2.3 | <0.7 | <0.003 | <0.05 | <0.0060 |
| DP1000-B    | 0.08 | 0.26 | >2.6 | 0.31 | 0.0018 | 0.16 | 0.0372 |

Previous works reported that EBSD measurements may underestimate the volume fraction of retained austenite [48,49]. On the other hand, magnetic measurements provide a more reliable quantification of retained austenite content [48].

The martensite volume fraction was determined from SEM micrographs. However, the differentiation between ferrite and bainite in SEM images was sometimes difficult, especially in DP1000-B. Therefore, the amount of ferrite phase was obtained from Nital etched optical micrographs. The MAD colour map (Fig. 2 c) was also used as a qualitative indicator of the ferrite fraction. The MAD indicates the degree of misfit between the theoretical and the measured Kikuchi bands. The lower the MAD, the better the match and the higher the indexing quality or the image quality (IQ). The IQ analysis has been often used in literature to differentiate ferrite from bainite or martensite [50–52]. Since bainite and martensite present higher dislocation density than ferrite, the quality of the diffraction pattern is affected by the larger lattice distortion [50–52], leading to lower IQ or higher MAD. However, the differentiation between bainite and martensite is more difficult since the differences in misorientation due to lattice distortions are not so evident. In this work, the MAD analysis was used to provide a general idea on the presence of ferrite in the two investigated DP steels. In Fig. 2 c, the blue colour indicates a good degree of pattern quality and, thus, it can be associated to the ferrite phase. On the other hand, green colour suggests a deviation of the measured patterns for BCC phase from the theoretical Fe-BCC patterns. These deviations can be associated with the presence of bainite and/or martensite. The MAD colour maps reveal a greater presence of ferrite in DP1000-B compared to DP1000-A, as observed in optical micrographs.

Both steels present very similar grain size distribution for both ferrite and martensite constituents, as shown in Table 2.
2.2. Experimental methods

2.2.1. Uniaxial tensile properties

Conventional tensile tests according to ISO 6892–1 [53] were performed to obtain the tensile properties of the two investigated DP steels for the transverse orientation. Tensile specimens with a width of 20 mm and a parallel length \( L_c \) of 120 mm were used (specimen type 2 described in ISO 6892–1, Annex B). An initial gauge length of 80 mm was used for strain measurements. The strain rate during the test was \( 2.5 \times 10^{-4} \, \text{s}^{-1} \) until the end of the elastic deformation, then \( 6.7 \times 10^{-3} \, \text{s}^{-1} \) until the failure of the sample. Three specimens per material were tested.

The instantaneous strain hardening exponent \( n_i \) and the strain hardening rate \( \theta \) were calculated according to Equations (1) and (2):

\[
n_i = \frac{d}{d \varepsilon} \left( \ln \sigma \right)
\]

\[
\theta = \frac{d\sigma}{d\varepsilon}
\]

where \( \sigma \) and \( \varepsilon \) are the true stress and the true strain respectively.

The true fracture strain \( \varepsilon_f \), derived from the reduction of area at fracture was also evaluated for all the specimens. \( \varepsilon_f \) is used as a measure of the material’s fracture resistance and it has been recently proposed as a relative index of local formability [54]. It is given by Equation (3):

\[
\varepsilon_f = \ln \left( \frac{A_i}{A_f} \right)
\]

where \( A_0 \) is the initial cross-section area and \( A_f \) is the area at fracture. The area at fracture was measured from the fracture surface of tensile specimens according to ASTM E8 [55] by using a stereo-light microscope.

To evaluate the evolution of retained austenite-to-martensite transformation with deformation, magnetization saturation measurements were performed to obtain the volume fraction of retained austenite and the fraction of martensite. The results are shown in Table 2.

Table 2

| Steel    | Ferrite (\( \alpha \)) | Bainite/Tempered martensite (\( \alpha_b \)) | Martensite (\( \alpha' \)) | Retained Austenite (\( \gamma \)) | Grain size [\( \mu \text{m} \)] |
|----------|------------------------|---------------------------------------------|-----------------------------|-----------------------------------|---------------------------------|
| DP1000-A | 23 ± 2                 | 39 ± 1                                      | 32 ± 1                      | 6.0 ± 0.4                         | 1.8 ± 0.4                      |
|          |                        |                                             |                             |                                   | 0.6 ± 0.4                      |
|          |                        |                                             |                             |                                   | 1.2 ± 0.4                      |
| DP1000-B | 33 ± 2                 | 37 ± 4                                      | 27 ± 1                      | 2.0 ± 0.3                         | 1.7 ± 1.3                      |
|          |                        |                                             |                             |                                   | 0.6 ± 0.4                      |

\( a \) Results from magnetization saturation measurements.

\[ \text{Fig. 2. EBSD results for DP1000-A (left) and DP1000-B (Right). a) IPF map, b) phase map and c) MAD map. GB: grain boundary.} \]
were performed in pre-strained uniaxial tensile samples. Five pre-strain levels were selected between zero and uniform strain ($\varepsilon_u$). For each pre-strain level, two specimens were tested.

2.2.2. Nanoindentation

Nanoindentation measurements were performed using an iNano nanoindenter (Nanomechanics). A Berkovich indenter was used. The experimental procedure for sample preparation was the same as described for EBSD measurements. In each sample, three matrices of 25 x 25 indentations were performed. An indentation separation of 6 μm was chosen, covering an area of 150 x 150 μm². The indentations were performed at an applied load of 20 mN. Hardness values were evaluated using the Oliver and Pharr methodology [56].

2.2.3. Essential work of fracture tests

EWF tests were performed using rectangular Double Edge Notched Tension (DENT) specimens of 240 x 55 mm (Fig. 3a). The specimens were machined with the notches aligned to the rolling direction (T-L configuration). Starter notches with notch radius $\rho = 150$ μm were machined by electrical discharge machining (EDM). Then, in order to avoid the influence of notch radius on fracture toughness results, fatigue pre-cracks were nucleated at the notch root (Fig. 3b) following the recommendations of the ASTM E1820 [57]. Fatigue pre-cracking was conducted under load (P) control in a resonance fatigue machine. Tests were run at room temperature at a constant axial load ratio, $R = P_{min}/P_{max} = 0.1$ (tension–tension). The $\Delta K$ ($K_{max} - K_{min}$) was kept below 0.3 $K_o$, where $K_{max}$ and $K_{min}$ are, respectively, the maximum and minimum stress intensity factor applied and $K_o$ is the linear elastic fracture toughness at crack initiation. This condition was verified after the test. The cracks were extended about $1.5$ mm per side. The crack growth was tracked by visual inspection and the final crack length was measured with the aid of an optical microscope. Due to the manual monitoring of the crack growth and the difficulty in propagating the two cracks in a perfectly symmetrical manner, the final crack length usually presented some variations from one side of the specimen to the other (maximum deviation ±0.3 mm).

Five different initial ligament lengths ($l_0$) ranging from 6 to 14 mm were used and 3 specimens per ligament length were tested. The specimens were tested up to fracture at a constant cross-head speed of 1 mm/min. The load-line displacement was measured by means of the video extensometer using initial extensometer marks separated 50 mm. The specific total work of fracture ($w_f$) for each specimen was obtained by integrating the energy under the load vs load-line displacement curve and dividing by the initial cross-section area:

$$w_f = \frac{1}{l_0h_0} \int_0^u P \, du$$

where $l_0$ is the initial ligament length, $h_0$ is the sheet thickness, $P$ is the load, $u$ the load-line displacement and $w_f$ the displacement at fracture (Fig. 3c). The specific essential work of fracture, $w_e$, was then obtained by linear extrapolation of $w_f$ vs $l_0$ data to ligament zero (Fig. 3d). The slope of the linear regression represents the non-essential plastic work ($w_p$) multiplied by a geometry factor ($\beta$). The fracture toughness at crack initiation, $W_f$, was obtained from an average of specific work of fracture initiation ($w_f^I$) values for different ligament lengths. $w_f^I$ is calculated by integration of the area under load-displacement curves up to the point of crack growth initiation (Fig. 3c) according to Equation (7):

$$w_f^I = \frac{1}{l_0h_0} \int_0^{u_i} P \, du$$

where $u_i$ is the load-line displacement at crack growth initiation (Fig. 3c). As observed in Fig. 3d, $w_f^I$ remains constant with the ligament length [34,42]. For the determination of the crack growth initiation, a high-resolution video camera synchronized with the testing machine was used. Generally, the crack growth was first initiated in one of the two sides of the specimen. $u_i$ corresponds to the displacement when the first crack starts to grow. $w_f^I$ was calculated for two specimens of each ligament length.

2.2.4. Fracture surface analysis of DENT specimens

The fracture surface of DENT specimens was investigated by optical microscopy and SEM. To quantify the contribution of necking to the ductile fracture process, thickness strain measurements were performed in different positions of the fracture surface by using an optical microscope and a digital image analysis software (Fig. 4a and b). The true fracture thickness strain ($\varepsilon_{f3}$) was then calculated as follows:

$$\varepsilon_{f3} = \ln\left(\frac{t_f}{t_0}\right)$$

where $t_0$ is the initial sheet thickness and $t_f$ is the thickness at fracture measured from the fracture surface. The evolution of $\varepsilon_{f3}$ as a function of the crack advance was evaluated and two terms were identified: the fracture thickness strain at crack tip ($\varepsilon_{f3, tp}$) and the steady-state fracture thickness strain ($\varepsilon_{f3, ss}$) (Fig. 4c).

A fractographic analysis of the fracture surfaces of broken DENT specimens...
specimens was performed by means of SEM. Void size and distribution and main fracture mechanisms were investigated. The crack path was investigated by SEM observation of DENT specimens tested and stopped after a small amount of crack extension. Metallographic samples were obtained from an area close to the crack tip, polished and etched with a 2% Nital solution to reveal the microstructure.

3. Results

3.1. Tensile properties

Fig. 5 shows the engineering and true stress-strain curves for the two studied DP steels. True curves are represented up to the uniform elongation (solid lines) and extrapolated to the true fracture strain ($\varepsilon_f$). The true stress at fracture is obtained by dividing the load at fracture by the final cross-section area, $A_f$. Mechanical properties are given in Table 3. The DP1000-A shows slightly superior strength and elongation values than DP1000-B. DP1000-A also exhibits higher $\varepsilon_f$ and UTSxTE. The strain hardening behaviour is shown in Fig. 6. Both DP steels are characterized by a high initial strain hardening rate that rapidly decreases with increasing strain, which is typical from DP microstructures. From the onset of the plastic region ($\varepsilon \approx 0.02$), DP1000-A shows higher strain hardening rate and strain hardening exponent ($n$) than DP1000-B. Even though DP1000-B has higher initial $n$, it continuously decreases from the beginning of deformation. On the other hand, in DP1000-A, the strain hardening exponent remains constant at the initial plastic deformation stage ($\varepsilon \approx 0.02-0.03$) and then linearly decreases up to the true uniform strain. The greater strain hardening behaviour of DP1000-A is probably related to the transformation of retained austenite to martensite, which takes place at the early stages of deformation (see insert in Fig. 6a).

3.2. Nanohardness measurements

Fig. 7 shows SEM images of the nanoindentation arrays and the resulting nanohardness mappings. Due to the very fine grain size distribution, most of the indentations were located in areas containing more than one single phase, which made difficult the evaluation of the hardness for each individual microstructural constituent. Nevertheless, the results provide an illustrative picture of the hardness distribution in the two studied DP steels. Hardness histograms are presented in Fig. 8. It shows that both steels present a normal distribution of hardness data. The values of hardness can be roughly divided into three groups: $3.25 < H < 4.25$ GPa, $4.25 \leq H < 4.75$ GPa and $H \geq 4.75$ GPa, which might be associated to the ferrite, bainite and martensite phases, respectively. Such range of hardness values for ferrite and martensite are in good agreement with results from the literature [12].

Even though both steels present quite similar distribution of hard phases ($4.75 \leq H < 5.5$ GPa), DP1000-A shows a larger presence of very high hardness values ($H > 5.5$ GPa, colour red in Fig. 7). These hard phases showing the highest hardness values may be associated with the carbon enriched fresh martensite/retained austenite islands. The distribution of ferrite (green) and bainite (yellow) in both steels is in concordance with the microstructural description shown in Section 2.1.2., i.e. DP1000-B shows a higher fraction of ferrite than DP1000-A. For the intermediate hardness values, i.e. $4.25 \leq H < 4.75$ GPa,
associated with bainite, the histograms in Fig. 8 show that steel DP1000-A has a larger proportion of hardness values in the high hardness range than steel DP1000-B. This could be related to the higher amount of carbides observed in the bainite/tempered martensite areas of steel DP1000-A (Fig. 1). Even though differences in average hardness, are small, DP1000-A ($H = 4.6 \pm 0.3$ GPa) shows slightly greater hardness than DP1000-B ($H = 4.4 \pm 0.3$). Applying a t-distribution analysis, it can be asserted with a 99% confidence level that both hardness values are significantly different.

### 3.3. Fracture toughness

#### 3.3.1. Essential work of fracture

Fig. 9 shows the load vs load-line displacement curves obtained from EWF tests. The values of specific work of fracture ($\text{w}_f$) and specific work of fracture initiation ($\text{w}_f^i$) are plotted as a function of the ligament length.
As observed in Fig. 9, for similar ligament length, DP1000-B shows greater maximum load and displacement at fracture than DP1000-A. This results in higher $w_f$ and, consequently, higher $w_e$ ($w_e = 286 \pm 17 \text{ kJ/m}^2$ for DP1000-B and $149 \pm 21 \text{ kJ/m}^2$ for DP1000-A). DP1000-B also presents higher fracture toughness at crack initiation, $w_e$, and greater energetic contribution from crack propagation after initiation ($w_e/w_e^i = 1.3$ and 1.6 for DP1000-A and DP1000-B, respectively).

The similar slope of the $w_f$ vs $l_o$ data regression indicates that both steels have almost identical non-essential plastic work, $\beta w_p$. The values of $\beta w_p$ obtained for DP1000-A and DP1000-B are $24 \pm 2$ and $23 \pm 1 \text{ MJ/m}^3$ respectively.

3.3.2. Fracture thickness strain measurements in DENT specimens

Fig. 11 shows the evolution of $\varepsilon_{3f}$ as a function of the distance from the crack tip. The plotted $\varepsilon_{3f}$ values are the average of two specimens. The steel DP1000-B exhibits a higher degree of necking both at crack initiation ($\varepsilon_{3f}^{tip}$) and during crack propagation ($\varepsilon_{3f}^{SS}$). In both steels the crack tip neck stabilizes at a distance of approximately 0.4 mm from the crack tip, reaching a steady fracture strain of $\varepsilon_{3f}^{SS} = 0.12$ and 0.16 for DP1000-A and DP1000-B, respectively.

3.3.3. Investigation of crack path

Fig. 12 shows SEM micrographs taken from an area adjacent to the crack advance. Fig. 12a–d shows the crack propagation path for DP1000-A and DP1000-B. In both steels, the crack preferentially propagates bordering the martensite grains. DP1000-A shows a straighter crack propagation path, favoured by a greater presence of martensite grains and fresh martensite blocks (M/RA) along the crack path. On the other hand, in DP1000-B the crack follows a zig-zag path, which indicates lower connectivity of the hard secondary phases, caused by a more homogeneous distribution of martensite islands. As illustrated in Fig. 12e and f, void nucleation results mainly from decohesion of martensite grains and decohesion of the ferrite-martensite and/or fresh martensite-bainite interface.

3.3.4. Fractographic analysis

Fig. 13 and Fig. 14 show the SEM micrographs of the fracture surface of DENT specimens for the two studied DP steels. Three different regions are identified in the fracture surface: Zone I, Zone II and Zone III. The Zone I corresponds to the flat triangular region ahead of the crack tip (surrounded by a white line in Figs. 13a and 14a), where crack initiation takes place. Due to the higher stress triaxiality in the centre of the plate thickness, the crack initiates following a normal plane respect to the principal tensile stress. After the formation of this initial triangular area, the growing crack rapidly tilts $45^\circ$ respect to the loading axis and a slant (or shear-induced) fracture occurs. This mode of fracture is commonly observed in thin ductile sheets [58], and it is associated to the formation of shear deformation bands produced by the variation of stress...
triaxiality through the specimen thickness (triaxiality is lower in the outer free-surfaces of the specimen) [59]. Hence, the stress state in the crack front evolves from plane strain at crack initiation to a biaxial (or plane stress) state during propagation. Zone II and Zone III correspond to different locations in the shear-induced fracture. Zone II is in a region close to the crack tip whereas Zone III is in the stable crack propagation area. The size of the flat triangular region for the two studied DP steels is given in Table 4.

The fracture surface of both steels present a dimpled appearance, typical of a ductile fracture mechanism of void nucleation, growth and coalescence. DP1000-A presents a bimodal dimple size distribution with large (≈7 μm) and small (2–3 μm) secondary voids. Some dispersed tearing cracks can be also observed (Fig. 13b). In the slant fracture area (Zone II and Zone III), a few smooth zones, where dimples are hardly visible, are detected (indicated by arrows in Fig. 13c and d). These smooth areas suggest that friction between the two fracture surfaces has occurred during the slant fracture process [60,61]. Three different void sizes are distinguished in DP1000-B: small (2–3 μm), large (10–12 μm) and very large (18–30 μm) voids. Table 4 shows the average void size distribution for the three different fractures zone investigated. In general, because of the high stress triaxiality, Zone I shows larger spherical voids and dimples elongated in the loading direction [61,62]. In Zone II

Fig. 11. Fracture thickness strain ($\varepsilon_{3f}$) as a function of the distance from crack tip.

Fig. 12. SEM micrographs from a region close to the crack tip. DP1000-A (upper row) and DP1000-B (middle row). a, c) Crack propagation path (2500× magnification). b, d) Closer view of the crack propagation path (6500× magnification). e, f) Void nucleation (white arrows) by decohesion of martensite grains and ferrite-martensite or fresh martensite (M/RA)-bainite interface. DP1000-A (e) and DP1000-B (f).
and Zone III (slant fracture), voids are smaller and oriented at 45° respect to crack advance direction, especially in Zone II. This coalescence mode of small secondary voids is often called void sheeting.

Small voids are predominant in both steels. As shown in Fig. 12 and reported in previous studies [38,63], these voids are primarily generated by martensite cracking or interface decohesion between harder and softer particles. Also, some inclusions were observed within the dimples, which acted as void initiation sites. In the case of DP1000-A, few small (≤2 μm) spherical oxide inclusions were present (Fig. 15a). In DP1000-B, larger voids are mainly nucleated at coarse TiN (∼3–4 μm)

Fig. 13. DP1000-A. SEM micrographs of the fracture surface of a DENT specimen. a) General image of the fracture surface (50× magnification). Closer view (1000× magnification) of: b) Zone I, c) Zone II and d) Zone III.

Fig. 14. DP1000-B. SEM micrographs of the fracture surface of a DENT specimen. a) General image of the fracture surface (50× magnification). Closer view (1000× magnification) of: b) Zone I, c) Zone II and d) Zone III.
precipitates by particle fragmentation and/or decohesion of the interface particle/matrix [64] (Fig. 15b).

4. Discussion

4.1. Influence of microstructure on uniaxial tensile properties

Fig. 5 shows that the tensile properties of the two investigated DP steels are quite similar in terms of strength and ductility. However, small differences can be discerned and discussed on the basis of the distribution of their microstructural constituents. The steel having a lower amount of ferrite (DP1000-A) shows slightly higher yield strength (YS). This is in good agreement with observations in previous studies and it is related to the lower dislocation density present in ferrite/ferrite interfaces compared to ferrite/bainite or ferrite/martensite boundaries [65,66]. The lower density of dislocations facilitates the free movement of existing dislocations due to the lower interaction between them [66]. When increasing the volume fraction of hard phases, the dislocation density is increased due to the major presence of ferrite/bainite or ferrite/martensite interfaces, leading to lower dislocations mobility and thus to higher YS. The differences in the bainite phase, with a higher amount of carbides for DP1000-A than for DP1000-B could also contribute to the higher YS.

Chang et al. [65] showed that yield strength of DP steels is mainly governed by ferrite characteristics and independent of the martensite volume fraction since during yielding the plastic deformation of martensite is practically negligible. On the other hand, they found that tensile strength (UTS) depends on the amount of martensite. This is confirmed for the steels investigated in this work. DP1000-A, which has higher martensite volume fraction, shows slightly higher UTS. This steel also has an additional influence of the strain-induced transformation of retained austenite to martensite (TRIP effect). The larger fraction of retained austenite present in DP1000-A (6%) compared to DP1000-B (2%) and the TRIP effect significantly improves the strain hardening behaviour (Fig. 6) and contributes to increasing strength and elongation [4]. As shown in Fig. 5, the true fracture strain ($\varepsilon_f$) is also slightly increased. The beneficial influence of TRIP effect on mechanical properties is associated to the formation of additional mobile dislocations in ferrite and bainite in the vicinity of newly formed strain-induced martensite, which increases strain hardening and delays the onset of necking [67,68].

4.2. Microstructural effects on fracture toughness

The large differences in crack initiation and propagation resistance (Fig. 10) also can be explained by the higher volume fraction of retained austenite present in DP1000-A. Contrary to the observed in uniaxial tensile properties, the strain-induced transformation of retained austenite to martensite may have a detrimental effect on fracture toughness. Jacques et al. [69] observed that in TRIP-assisted steels, due to the greater stress triaxiality in the front of the crack tip, most of the retained austenite present in the fracture process zone transforms to martensite before crack initiation. As a consequence, the newly formed martensite is distributed in the form of a “brittle” network along the fracture process zone. This is corroborated in Fig. 12a and b, where the presence of fresh martensite grains along the crack path is confirmed. Therefore, in DP1000-A, the transformation of retained austenite to martensite generates a continuous network of high strength bainite/-tempered martensite (harder than the one in DP1000-B due to the higher amount of carbides) and untempered fresh martensite that increases the connectivity of the hard secondary phases (Fig. 7a) and triggers unstable crack propagation [38,69-71]. On the other hand, in DP1000-B the hard martensite islands are more “isolatedly” distributed (Fig. 7b), which facilitate crack propagation through the soft ferrite phase (Fig. 12c and d). Such crack propagation mode, enables larger plastic deformation and increases crack propagation resistance, giving rise to higher fracture toughness.

In summary, the results show that the fracture toughness of the two 1000 MPa DP steels investigated in this work primarily depends on two main factors: the presence of C-enriched fresh martensite/retained austenite islands and the connectivity of the hard secondary phases. It is shown that even a moderate amount of fresh martensite/retained austenite islands may have a negative influence on fracture resistance. The presence of a lower proportion of ferrite in the matrix has also shown to have a deleterious effect on cracking resistance of ultrahigh strength DP steels. These observations show that, depending on the application, cracking resistance can be enhanced at the cost of elongation and strength by reducing the connectivity and volume fraction of residual austenite/martensite and increasing the ferrite fraction.

4.3. Necking contribution to plane stress fracture toughness

As pointed out before, plane stress ductile fracture has an important energetic contribution from necking [38-42]. Accordingly, the specific essential work of fracture ($w_f$) developed in the fracture process zone, can be expressed as the sum of two contributions [41]:

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Table 4

| Steel  | Void type | Zone I | Zone II | Zone III | Area of flat fracture (µm²) |
|--------|-----------|--------|---------|----------|---------------------------|
| DP1000-A | Small    | 2.6    | 1.7     | 1.9      | 186.43        |
|        | Large     | 6.9    | 6.7     | 6.9      | 328.98        |
| DP1000-B | Small    | 2.7    | 2.2     | 2.2      | 186.43        |
|        | Large     | 12.3   | 9.4     | 10.3     | 328.98        |
|        | Very large | 30.7  | 17.0     | 18.5     | 328.98        |
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Fig. 15. a) Microvoid nucleated at Al₂O₃ inclusion in DP1000-A (Secondary electrons, 2000× magnification). b) Microvoid nucleated at TiN precipitate in DP1000-B (Secondary electrons, 2000× magnification) and detail of the fragmented TiN particle (InLens image, 5000× magnification).
\[ w_k = \Gamma_0 + \Gamma_n \]  

(9)

where \( \Gamma_0 \) is the energy spent on the creation of new surfaces at the front of the crack tip (intrinsic fracture resistance) and \( \Gamma_n \) is the work of necking, which depends on sheet thickness. This explains the thickness dependence of \( w_k \) (and of plane stress fracture toughness in general) and states the importance of comparing fracture toughness values for materials of similar thickness, as the selected in the present study.

The contribution of necking to the ductile fracture process is illustrated in Fig. 11. The lower values of true thickness strain both at crack initiation (\( 2\varepsilon_f \)) and during crack propagation (\( 2\varepsilon_f \)) coincides with the lower fracture toughness at crack initiation (\( w_k \)) and specific essential work of fracture (\( w_k \)). When most of the necking work is developed at crack initiation and the neck is rapidly stabilized, the contribution to the total crack propagation resistance is limited, such as the case of DP1000-A (\( 2\varepsilon_f \)). On the other hand, as observed in DP1000-B, if the neck developed at the crack tip progressively increases during the crack advance, the crack propagation resistance is significantly increased (\( 2\varepsilon_f \)). This confirms that one of the main contributions to the crack propagation resistance of high strength steel sheets comes from necking [42].

4.4. Damage and fracture mechanisms

The two investigated DP steels presented a ductile fracture mechanism of void nucleation, growth and coalescence. The decohesion of martensite grains and ferrite-martensite or martensite-bainite interfaces were identified as the main void nucleation mechanisms (Fig. 12c and f). In DP1000-A, the greater presence of fresh strain-induced martensite in a mainly bainitic matrix promotes rapid void nucleation and coalescence, which explains the prevalence of small dimples and tearing cracks in the fracture surface (Fig. 13). DP1000-B showed a more ductile fracture appearance with a greater number of larger dimples. This can be ascribed to the lower hardness of martensite islands and the main proportion of ferrite in the matrix.

The influence of the coarse TiN precipitates (Fig. 15b) on the fracture toughness of DP1000-B is not clear and should be studied in further detail. Previous works have reported that coarse TiN particles may act as cleavage initiation sites in steels and have a detrimental effect on toughness [72-74]. Nevertheless, in DP1000-B, these precipitates serve as preferential sites for primary ductile void growth and seem not to have a major influence on cracking resistance. Even though no clear evidence of the effect of TiN precipitates on the fracture behaviour is shown in this work, in general, it is recommended to limit the presence of these particles, since they can also have a negative impact on stretch formability [75].

4.5. Relationship between fracture toughness and tensile properties

It is evident that fracture toughness has become a relevant property to understand the cracking resistance of thin AHSS sheets [17,26, 29-35]. Nevertheless, fracture mechanics testing of thin ductile plates is not straightforward and, therefore, toughness is often inferred from tensile properties. For instance, the product of UTSxTE, which represents a combination of the material’s strength and ductility, is usually used as a toughness indicator [66]. However, several works have shown that this practice can lead to misleading conclusions on the fracture resistance of AHSS [38,42,71]. As shown in the present work, it is especially risky to assume a direct relation between uniaxial tensile properties and cracking resistance on materials containing retained austenite since the TRIP effect, whereas can effectively improve strength and elongation, may have a negative influence on fracture toughness [38,69,71].

Fig. 16 shows the relation between fracture toughness and uniaxial tensile parameters for different DP and complex phase (CP) steels of similar thickness. No clear correlation can be established between \( w_k \) and tensile strength (YS or UTS) or the UTSxTE product. This evidences again that UTSxTE is not a good indicator of fracture toughness. Especially good linear correlation is observed between \( w_k \) and strain hardening exponent (\( n \)); \( w_k \) linearly decreases with increasing the \( n \) value. This can sound contradictory since, usually, higher \( n \) is associated with greater formability. However, as aforementioned, strain hardening mechanisms of AHSS are closely related to microstructure inhomogeneity and strain gradients between soft and hard microstructural constituents, which promotes nucleation and coalescence of microvoids. In fracture toughness testing, due to the large stress triaxiality present in the front of the crack tip and the high plastic localization, this void growth and coalescence process is accelerated and rapidly contribute to macroscopic fracture [23].

These are key factors to keep in mind when developing new AHSS microstructures with an optimum balance between formability and cracking resistance. For example, it is interesting to note that, although previous works showed that DP steels have lower crack propagation resistance than CP ones [33], DP microstructures can be designed to attain high fracture toughness comparable to CP steels of similar strength (Fig. 16) at expenses of global formability (strain hardening and elongation).

5. Conclusions

The fracture behaviour of two commercial 1000 MPa DP steels processed at industrial scale has been characterized and the influence of microstructural constituents on fracture response has been investigated. From the investigations performed and the obtained results, the following conclusions can be drawn:

- The mechanical properties of DP1000-A in terms of strength and ductility are slightly superior to DP1000-B. The lower amount of ferrite in the matrix leads to an increase in yield strength, whereas the higher martensite volume fraction increases UTS. The presence of a moderated amount of retained austenite (\(<6\%) and the strain-induced transformation of austenite to martensite (TRIP effect) contributes to increasing strain-hardening and elongation (both uniform and total).
- Contrary to the observed in uniaxial tensile properties, the presence of fresh martensite/retained austenite (M/RA) islands has a negative effect on cracking resistance of DP1000-A. It is attributed to the formation of a “brittle” network of hard phases that favours rapid crack propagation.
- The superior crack initiation and propagation resistance of DP1000-B are ascribed to three main factors: 1) lower volume fraction of fresh martensite/retained austenite, 2) a higher proportion of ferrite in the matrix and 3) lower connectivity of the hard secondary phases.
- The two DP steels show a ductile fracture mechanism of void growth and coalescence. The main void nucleation mechanisms were identified as: 1) decohesion of martensite grains, 2) decohesion of the ferrite-martensite interface, 3) decohesion of bainite-fresh martensite (M/RA) interface and 4) void nucleation at inclusions (aluminium oxides in DP1000-A and titanium nitrides in DP1000-B). Further investigations are suggested to understand the role of coarse TiN particles on fracture toughness.
- The fracture toughness of high strength DP steel sheets has a significant contribution from necking. The steel showing a higher degree of necking at crack initiation and propagation (DP1000-B) exhibits greater crack initiation and propagation resistance. The differences between crack initiation and crack propagation resistance also can be explained by the evolution of necking during crack propagation. When the neck developed at the crack tip progressively increases during crack propagation, a significant increase on crack propagation resistance is observed.
- Fracture toughness cannot be directly inferred from tensile properties. From the comparison of results obtained in this work with data
from the literature, it is shown that $w_e$ does not correlate with YS and UTS. Contrary to the usual perception, fracture toughness does not increase with increasing UTSxTE and $n$ value, which are generally used as indicators of toughness and formability. Rather, on the contrary, a negative relationship was observed between fracture toughness and $n$ value. The inverse proportionality between $w_e$ and strain hardening exponent is related to the nature of strain-hardening mechanisms of AHSS, which are mainly governed by microstructural inhomogeneities that favour local failure.

These results provide a better understanding of the role of the different microstructural constituents on cracking resistance of high strength DP steels and can serve as a guide for new material development.

CRediT authorship contribution statement

D. Frómeta: Conceptualization, Methodology, Writing - original draft, Investigation. N. Cuadrado: Investigation. J. Rehrl: Investigation, Resources. C. Suppan: Investigation, Resources. T. Dieudonné: Investigation, Resources. P. Dietsch: Investigation, Resources. J. Calvo: Writing - review & editing. D. Casellas: Writing - review & editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.msea.2020.140631.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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