Microstructural and Micromechanical Effects of Cold Roll-forming on High Strength Dual Phase Steels

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In this work correlation between the 1000 MPa dual phase (DP) steel microstructure and the strain gained after roll-forming process have been studied by both microstructural and micromechanical analysis. The scanning electron microscope (SEM) inspection in the bent area reveals changes in the ferrite-martensitic microstructure. The plastic deformation of DP steels originates defects at the edges of bent sheet make them partly responsible for the damage caused. In addition, electron backscatter diffraction (EBSD) measurements have been carried out for an in-depth characterization after roll-forming. A high density of misorientation of the crystal lattice within the ferrite strained grains is observed, mainly concentrated in the ferrite/martensite grain boundaries. Furthermore, the ultramicrohardness tests exhibit little dependence between mechanical parameters and the material properties.

Keywords: dual phase steel, roll-forming, EBSD, ultramicrohardness, microstructure

1. Introduction

High strength low alloy (HSLA) steels, specifically dual phase (DP) steels, are widely used in the automotive industry due to the necessity of improving fuel efficiency1-3, with its subsequent energy saving and environmental protection4. In addition, these DP steels have been developed to increase the highly demanding collision safety standards5 in the vehicle body frame parts like bumper beam, door beam and panel reinforcement.

DP steels are characterized by a microstructure consisting of about 75-85% ferrite phase with the remainder being a mixture of the martensite and lower amounts of other such as bainite and retained austenite6-7. Such a structure leads to different unique properties, as high tensile strength, low yield strength ratio followed by continuous yielding behavior (no-yield point elongation), high work-hardening rate at early stages of plastic deformation as well as good ductility8-10. Furthermore, the absence of the yield point elongation provides DP steels with a high crash resistance, good formability and excellent surface finish11.

Roll-forming is a bending process in which a sheet metal is continuously and gradually formed into a profile with a desired cross-section along the transverse direction by passing through a series of rolls arranged in tandem12. It can be performed at high speeds and therefore has been considered, in recent years, very interesting for the manufacturing of automobile components. However, during roll-forming the microstructure of the sheet metal can undergo serious damage. The outer surface remains stretched consequently the inner surface is compressed. Thus, these outer surfaces, highly deformed, are preferential nucleation sites, growth and coalescence of voids and/or cracks13,14.

Several studies4,7,15-19 have been conducted on the DP steels behaviour under formability tests, as bending, stamping or cup drawing. Luo & Wierzbicki15 analysed the failure behaviour of DP steels during stretch-bending operations and summarized the typical damage accumulation in this area. Wu-rong et al.13 found that DP steels show competitive formability after cup drawing tests, despite encountering consistent shear cracks on the damage surfaces. Wang & Wei16 showed that in stretch-bending tests not only latitudinal but also longitudinal cracks were propagated along DP steels in a mixed propagation mode, i.e. along ferrite-martensite interfaces and/or also across martensite grains. Subsequently, Wang et al.4 discussed that the fracture mode experiences a transition from shear fracture to necking before fracture. Mishra & Thuillier17 concluded that bending area of maximum strain is not highly localised, but more uniform.

However, to the author’s best knowledge no studies have been reported on the DP steels evolution in cold roll-forming processes.

In this work, microstructural and micromechanical study have been performed in order to go deep in the understanding of the behaviour between the microstructure and the deformation during cold roll-forming processes and provides overall responses to the automobile manufacturing industry, which is a subject of main concern, from both industry sector, in ever increasing new designs for car structural components, and also in academic research.

2. Experimental Procedure

The chemical composition of the commercial high strength dual phase DP1000 used is given in Table 1. The 1.5mm thick DP1000 sheet was roll-forming in hat-section geometry profile by Autotech Engineering (AIE, Gestamp R&D). The roll-forming direction was parallel to the rolling direction of the sheet and performed at a feed rate of 50 m/min. Hat-section was formed by 6-stages, in which
the arrangements of the roll bend angles were varied over 15º → 30º → 45º → 60º → 75º → 90º.

Specimens were taken from a DP1000 sheet after being processed revealing the LT-ST plane (Figure 1).

The specimens analysed are presented in Figure 2. One of specimens was taken from an area free from any strain - blank specimen-. Three specimens were taken from the bent area characterized by three distinctive regions: the outer edge, the middle zone and the inner edge.

Metallographic specimens were prepared according to standard procedures. They were mounted in an epoxy resin, ground down through successive grades of SiC paper to 2000 grade, degreased with alkaline cleaner and rinsed in tap water followed by deionized water and finally polished with diamond paste of 3 and 1 µm.

For scanning electron microscopy (SEM) inspections, the specimens were etched with 2% Nital solution for 15 s, given that Nital preferentially etches ferrite and outlines their grain boundaries leaving martensite undissolved.

In order to calculate the volume fraction of ferrite and martensite phases, metallographic analysis were conducted by using an image software analysis. Using a commercial imaging software (Analysisdocu®) the martensite and ferrite phases were automatically distinguished by adjusting the contrast and colour, so martensite grains turn black, while ferrite grains turn white.

Finally, for Electron Back Scatter Diffraction (EBSD) analysis, the specimens were mounted in epoxy resin, ground in SiC and polished with diamond paste of 3 and 1 µm as it was above described. After this stage the specimens were automatically polished with colloidal silica for 30 s. Then, the specimens were rinsed in distilled water, soaked in ethanol and dried in direct warm air. Immediately after, the specimens were etched with 2% Nital solution for 15 s.

SEM images and EBSD scans were carried out in a field emission gun scanning electron microscopy (FEG-SEM) JSM6500F JEOL equipped with energy-dispersive spectroscopy (EDS) facilities. A statistically relevant area of 322 x 257 µm was scanned using step size of 0.1 µm. The EBSD raw data were post-processed in detail by making use of the HKL Analysis Software. Grain boundaries were characterized by a misorientation larger than 5º between among neighbouring measurements points.

Several parameters on the basis of EBSD, such as the image quality (IQ), the inverse pole figure (IPF) and the kernel average misorientation (KAM) maps are powerful tools in order to characterize the material. However, the similarity between the crystalline structure of the microconstituents in dual-phase steels -ferrite (bcc) and martensite (bct, with a low tetragonality)- limits the software in order to distinguish between both phases, specifically when ferrite is highly strained. In this work, it has been indexed ferrite as the main phase. Consequently, the Kikuchi diffraction patterns that differ from the ferrite crystalline structure (bcc) will be considered not indexed, given as a dark point and related to strained ferrite, martensite phase or grain boundaries.

Hardness measurements were performed by a NanoTest 550 Micro Materials Ltd nanoindenter, using a Berkovich diamond tip. Tests were carried out at two indentation depths of 500 nm and 5000 nm. The local hardness and elastic modulus results were estimated from the loading and unloading curves using the standard Oliver-Pharr methodology.

3. Results

3.1. SEM characterization

Figure 3 clearly shows that the microstructure of DP1000 steel is comprised by harden island-shaped martensite inclusions randomly distributed (bright contrast) and a soft ferrite matrix (dark contrast). Figure 3a reveals the microstructure corresponding to the blank specimen. This DP1000 specimen analysed is characterized by a 26.5 vol.% of martensite phase. Ferrite grains size range from 1.2 µm to 5 µm, approximately.

The SEM images gathered in Figure 3b-d correspond to the microstructure in the bent area; outer edge, middle zone and inner edge, respectively. The microstructure corresponding to the outer edge in the bent area, Figure 3b, shows elongated ferrite grains, with the long axis parallel to the bending direction. The length of these ferrite grains vary

| Chemical composition of DP1000 dual phase steel (wt.%) |
|-----------------|-------|------|-----|-----|-----|-----|-----|-----|-----|
| C    | Mn   | Si   | Mo  | Cr  | Nb  | Ni  | V   | Fe  |
| DP1000| 0.067| 2.29 | 0.38| 0.25| 0.020| 0.037| 0.025| <0.02| Balanced |

Figure 1. DP1000 specimen (a) Hat-section profile; (b) Scheme main directions.

Figure 2. Details of DP1000 specimen (a) selected areas to analysis; (b) representative studied regions in the bent area.

Table 1. Chemical composition of DP1000 dual phase steel (wt.%).
from 3µm to 10 µm, approximately. Conversely, the martensitic islands appear not to be deformed, but it is noteworthy the presence of some voids (A). These voids appear preferentially localized around the martensite particles as result of the stress concentration and deformation mismatch\textsuperscript{22,23}. Other voids initiation (B) appears in the ferrite/martensite grain boundary and are considered a normal separation of both phases\textsuperscript{22}.

Moving throughout the bent area, towards the inner edge, the appearance of the ferrite grains is less elongated and the presence of voids disappears. The microstructure corresponding to the middle region of the bent area (Figure 3c) is comprised by ferrite grains apparently not deformed, keeping similar microstructure as in the blank specimen (Figure 3a). Finally, at the compressed inner edge of the bent specimen, Figure 3d, the ferrite grains vary their size and morphology into smaller and more equiaxed grains. In this region voids are not distinguished.

3.2. EBSD measurements

EBSD enables an exhaustive study related to the crystallographic orientations based on the Kikuchi diffraction patterns from the surface of the specimens\textsuperscript{24-26}. Besides lattice rotation and lattice strain at the surface of very small volumes of material can be measured\textsuperscript{11,27}.

Figure 4 depicts representative EBSD image quality maps (IQ) corresponding to the studied areas, where ferrite has been indexed as the main phase. The IQ maps describe the grain structure of the different phases\textsuperscript{24}. Due to overlapping diffraction signals, coming from two grains at a grain boundary, the contrast in the Kikuchi pattern faint or is even inexistent. As a consequence, a low value in the IQ map leads to a dark grain boundary or as a noticeable dark grain, constituting the martensite phase or highly strained ferrite\textsuperscript{20}.

Figure 3. SEM images of DP1000 specimens etched with 2% Nital at 3000x (a) blank area; (b) outer bent edge; (c) middle bent zone; (d) inner bent edge.
These maps reveal the very low quality of the EBSD patterns as black regions. Figure 4b-d present a noticeable difference in dark regions regarding the IQ map in the blank specimen (Figure 4a). These dark areas suggest extremely strained ferrite phase as a result of a high level of residual stresses due to the bending process. Also, martensite islands could appear as black.

Furthermore IQ maps have been successfully used to determine the volume fraction of the microstructural constituents of several alloys, such as AHSS steels and aluminium alloys. In the present work, the volume fraction of the phases varies depending on the region analysed, that is, blank specimen or bent regions.

Figure 5 show the volume fraction calculated as a function of these areas. The volume fraction for high IQ values, related to the ferrite phase, is higher than the volume fraction for the so-called low IQ values, related to grain boundaries, strained ferrite and martensite. It is noteworthy that the average volume fraction of the low IQ values, related to the non-indexed areas (grain boundaries, martensite and deformed ferrite) slightly increases regarding the blank specimens. This is consequence of the increase of the strained ferrite at the extremely deformed regions (outer and inner edges). A volume fraction of the low IQ values about 25% has been found in the outer and inner bent areas, while in the blank specimen the volume fraction is about 16%. In the middle area is observed a similar low IQ value to the blank specimen coincidently with the neutral line which separates the areas submitted to tensile and compressive strains in the outer and inner areas respectively.

Misorientation profiles, associated to selected ferrite grains in blank specimens (1, 2) and bent specimens (3, 4), are depicted in Figures 6e-h. These histograms reveal the misorientation distribution within each grain. For instance, Figure 6e and f gather the analysis performed in the areas labelled as 1 and 2 in the blank specimen. These histograms show significant changes of the misorientation distribution. While in Figure 6e, no differences in the orientation of the lattice are perceived, in Figure 6f there is an abrupt increase of the misorientation suggesting the proximity to low IQ values that in this case, blank specimen, should correspond to the proximity of a martensite phase.

The histograms corresponding to selected grains within the bent area are shown in Figure 6g and h. In particular these histograms are associated to specific grains in the outer edge (3) and inner edge (4), respectively. Such values could be consequence either of the presence of martensite next to these grains or highly strained ferrite grains.

Figure 7 depicts the representative EBSD KAM maps, corresponding to the areas above studied. The misorientation between a data point and its neighbours is analysed by the KAM maps. Furthermore, these maps indicate the dislocation density and the strain distribution on individual measurement points. In this work KAM scans were measured calculating each point by considering up to its 3rd neighbours and...
Figure 6. IPF maps of DP1000 specimens in (a) blank area; (b) outer bent edge; (c) middle bent zone; (d) inner bent edge; (e)-(h) misorientation profiles of the selected grains taken from the same areas.
without taking into account any misorientation higher than 5°, since this value was chosen to elucidate between grain boundaries and internal misorientation.

The illustrated colour typed code determines the degree of local crystalline misorientation within each grain. Blue colour corresponds to the grains without any misorientation, while red colour would correspond to the maximum. As it can be seen in Figure 7a, the blank specimen shows low misorientation. This specimen is characterized by low KAM values -green colour- visible at the ferrite-martensite interfaces. However, it is clearly observed that the KAM distribution shifts to higher values as the plastic deformation proceeds.

Figure 7b-d reveal an evident increase of the local crystalline misorientation, higher areas coloured in green. In these figures, it is seen that the plastic deformation occurs in the outer and inner edges regarding the blank specimen, varying substantially within the ferrite grain as result of the deformation. In particular, note that the ferrite with the highest level of misorientation -green to red colour- tends to concentrate at the ferrite/martensite grain boundaries.

3.3. Ultramicrohardness

Figure 8 shows an optical micrograph depicting representative ultramicrohardness indentations displayed in the DP1000 steel. Higher magnifications of several selected indentations, corresponding to two different indentation depths of 500 nm and 5000 nm, are shown in Figure 9a and b, respectively. These SEM images show that the higher the indentation depth, the larger the Berkovich indentation. In addition, it is clearly observed that each indentation encompass both ferrite and martensite phases.

Figure 9c presents a typical load versus displacement curve at the indentation depths of 5000 nm, respectively. The curve exhibits a parabolic behaviour in the loading section and a power-law behavior in the unloading one. The elastic recovery exhibited during the unloading section is small, as expected in metal alloys. Figure 10 shows the hardness obtained for the DP1000 blank and bent specimens at the two analysed indentation depths of 500 nm and 5000 nm. This depicts that the values obtained at lower indentation depth are slightly higher. This response is classically observed for the known indentation size effect.

Figure 8. Light-optical microscope visualization of a representative Berkovich indentation in DP1000.
Particularly, in dual-phase steels these size effects usually result from the strain gradient induced by several sources related to the microstructure -natural hardening of ferrite and reinforcement by martensite-, the applied deformation field and/or the specimen size. As shown in the literature, nanohardness measurements in dual-phase steels (e.g. with 26% content of martensite) show different intrinsic hardness values, i.e. $2.96 \pm 0.47$ GPa for ferrite, and $6.99 \pm 5.75$ GPa for martensite. Therefore, current results obtained at an indentation depth of 500 nm show an average hardness value dominated by the martensite phase. This might be a direct consequence of the relationship between the indentation size and the martensite grain size.

On the other hand, it is observed that the hardness values corresponding to the bent specimens are slightly higher than the blank specimen, regardless the indentation depth, suggesting a work-hardening after the roll-forming process. Furthermore, it can be noticed that the hardness measured in the bent area also depends on the specific region evaluated. The values corresponding to specimens with severe deformation -outer and inner edges- appear slightly higher than in the middle region.

### 4. Discussion

The deformation behaviour of dual phase steels is considered to be quite complex. However, despite the lack of understanding of the interactions between the present constituents and their influence on mechanical properties some generalization has been made in the literature. In general, localized plastic strain is resulting from the incompatible deformation between the soft ferritic matrix and the harder martensite phase, which have dissimilar properties. This plastic deformation process is initially related to the dislocation density in the ferrite phase. In the low strain range -during the first stages deformation- the work-hardening rate is considerably high as a consequence of rapid dislocation multiplication and the back stresses resulting from the strain incompatibility.

Then, when the ferrite phase reaches its maximum strained capacity (in the high strain range), ferrite matrix transfers strain across the ferrite-martensite interface, leading to the onset of a plastic deformation in the martensite grains. Subsequently, the work-hardening rate of the dual phase steel diminishes.
Ashby stated that the compatible deformation of a soft matrix which contains hard particles requires the generation of plasticity gradients, such as statistically stored (SSDs) and geometrically necessary dislocations (GNDs), within the more deformable phase. The SSDs are created by simple work hardening of ferrite, while the GNDs emerge from the necessity to maintain both ferrite and martensite in contact during plastic deformation. This theory has been widely used to explain dislocation movements and deformation of dual-phase steels.

Speich & Miller ascertained this statement in dual-phase steels. Also, the original Bergström dislocation theory, but adjusted for dual-phase steels, is employed in order to justify the plastic deformation process in these steels. However, according to this theory, the deformation is considered to be entirely supported by the ferrite phase, conversely to several authors who consider plastic deformation in martensite.

In this work, SEM images (Figure 3) confirm that the ultrafine grained DP1000 steel deformation mainly takes place in the ferrite phase. As a result, an elongated ferritic microstructure is observed in the outer edges from the bent area and compressed ferrite grains in the inner edges.

Moreover, the presence of several voids in the specimen after the roll-forming process is also worth to note, specifically in the outer edges (Figure 3b). It is well known that the initiation of voids occurs at ferrite/martensite grain boundaries, martensite/martensite grain boundaries and in ferrite/martensite ones.

In addition, it is known that the distribution of void nucleation sites is dependent on the microstructure but their growth and coalescence is controlled by local stress state. In this sense, the localization of the voids formed in the outer edge and their shape suggest that they initiate and grow parallel to the bending direction.

The IQ maps report significant differences of the evolution of the microstructure during the deformation process. They indicate the degree of distortion of the crystal lattices in the diffraction patterns, revealing the location of the grain boundaries and the martensite phase. Therefore, the low IQ values in the bent area -outer and inner edges- suggest a severe deformation in ferrite due to the plastic stresses appeared predominantly within the ferritic matrix and in the vicinity of the ferrite/martensite boundaries.

On the other hand, as it is shown in the misorientation profiles (Figure 6e-h), high values of misorientation degree are concentrated within the ferrite grains which are surrounded by martensite. In particular, the maximum degree of this variation is found close to the ferrite/martensite boundaries.

Qualitatively, the previously results in IQ maps are in good agreement with the results displayed in the KAM maps. The misorientation differs to a great extent as the plastic deformation increases. In general, the blank specimens reveal a weak misorientation within the grains. Whilst in the bent area the plastic strain is mainly localized next to ferrite/martensite boundaries.

In this work, hardness measurements indicate that the plastic deformation resulting from the roll-forming process applied on the specimens have small effects in the surface hardness. That suggests dependence among the mechanical parameters (depth, hardness) and the material properties, such as the work-hardening resulting from the plastic flow deformation.

5. Conclusions

In the present work the microstructure and micromechanical analysis of ultrafine grained DP1000 steel have been performed for two different representative areas of a constant hat-section profile obtained by means of continuous roll-forming process with a high feed rate with the aim of providing responses to the behaviour of this high strength low alloy steel for the automotive industry.

Meanwhile in not deformed specimens the microstructure of the DP steel reveals weak misorientation within the ferrite grains, the microstructure corresponding to the bent area exhibits variations depending on the particular analysed region. The specimens from the edges of the bent specimen -outer and inner- exhibit a severe plastic deformation mainly localized within the ferritic matrix and in the vicinity of the ferrite/martensite grain boundaries. As a result, high misorientation is noticed, suggesting a large density of dislocation in these deformed zones. Stretched ferrite grains are found in the outer edges whereas compressed ferrite grains are observed in the inner edge. The presence of several voids in the outer edge also indicates evident damage resulting from the plastic deformation undergone in the roll forming process.

The hardness measurements appear to indicate that the plastic deformation possess certain effects on the DP steel surface hardness.

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