In-situ micro-cantilever bending experiments were executed and analyzed in a detailed and quantitative manner to evaluate the effect of texture and post-welding heat treatment on the local fracture toughness of spot welds. In particular the objective is to explore by these in-situ experiments the structure-property relationship in advanced high strength steel resistance spot welds. An interesting finding is that, through a switch from single to double pulse scheme the texture of martensite formed in the fusion zone becomes responsible for a significantly higher fracture toughness of the area in front of the pre-crack. In addition, we found that the paint baking heat treatment also results in a higher fracture toughness through the tempering of the martensitic microstructure. A quantitative correlation is made between the macro-scale mechanical performance and micro-scale notched cantilever bending tests.

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7.1 Introduction

The design of AHSS was an attempt to push the limits of the strength and toughness of structural steels for engineering applications in automotive industries. As already discussed in Chapter 1 and 2, the chemical composition of AHSS is sophisticatedly designed to produce a multiphase microstructure through a precisely controlled heating and cooling processes. AHSS enable decreasing the vehicle weight for improved fuel economy and reduced impact to the environment whilst improving crash energy absorption for better protection (greener and safer cars). Recently, the third generation of AHSS have been introduced to the automotive industry providing even better strength-ductility combinations compared to the traditional (1st generation) high strength steels and AHSS [1]. However, the problems associated with the RSW of this new generation of steels have remained unsolved.

Extensive research work has been carried out on the simultaneous enhancement of the strength and fracture toughness of bulk martensitic microstructure via grain refinement through severe plastic deformation [2], microalloying [3] and thermal treatments [4–7]. Attempts were made also to modify the texture of steel to enhance its fracture properties via thermomechanical processing steps [8–10]. However, the microstructure and texture modification of a resistance spot weld come with special limitations in terms of sample size and economic efficiency in a mass-scale production line. Modifications in the weld scheme are one of the few limited measures to alter the microstructural and textural evolution of the weld nugget without compromising process efficiency in time and energy.

It was already shown experimentally (see chapter 3) that applying a double pulse weld scheme can effectively enhance the cross-tension properties of the spot weld. In particular, it was demonstrated that the double pulse welding subdivides the primary nugget into two weld zones. The outer layer in front of the pre-crack with an equiaxed structure of PAG and the inner layer with a typical columnar structure result from the solidification process. The change in cross-tension of spot welds with a change in weld scheme can arise from change in either strength or fracture toughness of the weld nugget. Whilst the strength of the microstructure can be easily related to its hardness, the fracture toughness evaluation is still a big challenge in the case of small-scale samples of resistance spot welds. Several computational and analytical models have been designed to predict the cross-tension strength mainly based on the local mechanical properties of the weld such as hardness and fracture toughness [11–14]. However, unfortunately, experiments concentrating on the fracture toughness of RSW are scantily available as the conventional standard test methods fail in the evaluation of the local fracture toughness of different weld zones due to the geometrical constraints of spot welds. This is more problematic in the case of double pulse welded samples in which the thin outer layer at the weld edge controls the crack initiation and propagation during cross-tension test. One may even conclude that the studies so far, lack a deeper understanding of the mechanical
response of the weld zones as the direct measurement of mechanical properties of different regions of spot welds is hardly possible due to limitation of small sizes. To bridge this discrepancy notched micro-cantilever beam bending seems a feasible technique to evaluate the fracture toughness of weld zones.

The present chapter concentrates on manipulating of the texture in the weld nugget by changing in the weld scheme as a controlling design tool so as to achieve the desired mechanical performance. In-situ micro-cantilever bending is used to reveal the effect of texture change on the local fracture toughness in high strength steel resistance spot weld. The detailed information about the microstructure and micro-fracture toughness was used to make a bridge to the mechanical performance of the weld at a macro-scale. In addition, the effect of paint baking (PB) cycle as a conventional manufacturing process in automotive industries on the mechanical response of the weld has been studied.

7.2 Experimental

The material used in this study was 1.5 mm thick cold rolled and galvanized 3rd generation 1 GPa AHSS (VDA239-100 CR700Y980TDH) with a multi-phase microstructure composed of ferrite, martensite, bainite and retained austenite. Resistance spot welds were produced using a 1000 Hz MFDC pedestal welding machine with constant current regulation. Welding electrodes (F1 16-20-5.5) and single pulse weld scheme were taken from the VDEh SEP1220-2 welding standard [15]. An electrode force of 4.5 kN was applied. Two weld schedules were applied in order to study the effect of pulse scheme on the mechanical performance and microstructural evolution of resistance spot welds. For the single pulse welding, 550 ms of squeeze time followed by a 380 ms welding time and a 300 ms of holding time with welding current of 6.4 kA was applied. For the alternative double pulse welding a non-standard procedure was selected. During this welding process, two 380 ms weld pulses with equal current level as the single pulse weld, and a cooling time of 80 ms in between were applied. Effect of post-heat treatment on the mechanical properties of the resistance spot welds were investigated by heating the welds at 180 °C for 20 min (oven heating). The heat treatment is equivalent to the PB process carried out in automotive industries during which the painted car body structures are subjected to an elevated-temperature baking process.

The OIM characterization was carried out via collecting and indexing electron back-scatter diffraction patterns using a Philips ESEM-XL30 SEM equipped with a field emission gun operating at 20 kV. Nanoindentation tests were performed using an MTS XP Nano-indenter XP, equipped with a Berkovich indenter. Minimum of 10 indentations were conducted for each weld at the constant maximum load of 30 mN. The micro-cantilevers with a nominal length of 15 µm, a thickness of 4.5 µm and a width of 5 µm were milled in the selected region using the procedure described in chapter 5 (Figure 7-1a).
Finite element analysis (FEA) was performed to make a correlation between the beam stiffness and crack size. Cantilevers with different notch to thickness ratios (a/t) were simulated in the elastic regime without considering plastic deformation or crack propagation. Another set of simulations were used to understand the plastic deformation response of micro-cantilevers. The indenter and the cantilever were considered as semi spherical infinitely stiff body and homogenous deformable solid, respectively. The notched micro-cantilevers were modelled according to the dimensions used in the actual experiments. One end face of the cantilever was fixed against all rotations and displacements. Utilizing displacement mode control simulation, the indenter pressed the end of the cantilever toward the opposite Y-axis, and the reaction force between the indenter and the sample was acquired until the maximum force was reached (the onset of crack initiation). Based on the response of each micro cantilever, the associated mechanical properties were regenerated to capture the experimental load displacement curves. The mesh sensitivity of the results was checked and the optimum mesh size was selected. C3D8 element type was utilized and mesh refinement was generated on the notch tip to attain accurate stress/strain field (Figure 7-1b).

For transmission electron microscopy (TEM) characterization, FIB slicing was employed to prepare lamellas in a dual beam FIB/SEM microscope (Lyra, Tescan). In the case of bent cantilevers, the samples were milled from both sides until 1 µm thick and then gently transferred to a three-post copper grid using a lift-out manipulator. Subsequently, the samples were further thinned by focused ion beam to 100 nm for TEM observations with a JEOL 2010-FEG transmission electron microscope.

![Figure 7-1](image-url) (a) In-situ bending of FIB-milled micro-cantilever and (b) the geometries and corresponding mesh used in 3D finite element simulations.

### 7.3 Results and discussion

#### 7.3.1 Mechanical properties

Figure 7-2 shows the cross-section of the single and double pulse welds. A martensitic structure with a typical columnar structure of PAGs resulting from rapid solidification of the weld is obtained for the single pulse weld (Figure 7-2a). Similar to the DP resistance spot welds, the FZ of the double pulse is composed of two
different martensitic microstructures: the outer layer with equiaxed PAGs and inner core composed of columnar PAGs (Figure 7-2b).

Figure 7-2 Cross-section of the resistance spot welds made by (a) single pulse welding and (b) double pulse welding.

Figure 7-3 Cross-tension properties of four different welds. Cross-section of failed samples: (b) SingleP, (c) SingleP-PB, (d) DoubleP and (e) DoubleP-PB.

The CTS and energy absorption capability of the welds are shown in Figure 7-3a. The weld size for all single and double pulse samples is ~ 5 mm that rules out the effect of nugget size on the mechanical properties. As clearly shown, double pulse welding can significantly enhance the mechanical properties of the welds (DoubleP), doubling the maximum load and 4.5 times higher of energy absorption capability compared to the single pulse welds (SingleP). Besides, the PB treatment leads to largely enhanced mechanical performances, both the maximum load and energy absorption capability of the welds (SingleP-PB vs. SingleP, DoubleP-PB vs. DoubleP). The double pulse weld with PB (DoubleP-PB) shows the highest maximum load and
energy absorption capability among the four samples. The failure modes of the welds are shown in Figure 7-3b-e. SingleP fails in the IF mode as the crack directly propagates through the fusion zone (Figure 7-3b). The failure mode changes to the PIF mode with the PB treated single pulse weld (SingleP-PB) as the crack firstly propagates into the fusion zone and then redirected towards the sheet thickness (Figure 7-3c). DoubleP weld also fails in the PIF mode, however the plug ratio is much larger compared to SingleP-PB sample (Figure 7-3d). DoubleP-PB that shows the best mechanical performance fails in the PF mode as the failure occurs outside the weld and the fusion zone remains intact after the test (Figure 7-3e).

Fracture surfaces at the faying surface of interfacial and partial interfacial failure exhibit semi-cleavage fracture as well as small areas of ductile fracture characterized by dimples for SingleP, SingleP-PB and DoubleP samples shown by arrows (Figure 7-4a-c). As observed, the fraction of the area failed in ductile manner is becoming larger with applying PB. Besides, the dimpled area becomes even larger by changing the weld scheme from single to double (Figure 7-4c). In the case of DoubleP-PB sample, the failure occurs by shear in the HAZ outside the weld and is characterized by mixed intergranular brittle and elongated dimpled ductile fracture (Figure 7-4d).

Figure 7-4 Fracture surface of (a) SingleP, (b) SingleP-PB, (c) DoubleP and (d) DoubleP-PB (the insert shows the location of SEM observation).
7.3.2 Effect of PB

Figure 7-5 shows the TEM images of the martensitic structure formed in the fusion zone of paint baked and unbaked single pulse welds. As shown in Figure 7-5a and b the structure of the SingleP sample is composed of fully lath martensitic structure. In the case of paint baked single pulse sample (SingleP-PB), transition ε carbides are detected inside the lath of martensite (Figure 7-5c, d). As can be seen, paint baking at 180 °C for 20 min leads to low-temperature tempering of the martensitic structure of the weld. The first stage of tempering of martensite is associated with the segregation of carbon atoms to dislocations and interstitial sites and occurs at the temperature below 100 °C. It is followed by the precipitation of transition carbides that happens at temperatures between 80 °C and 200 °C [16]. Low temperature and short period of time used for the paint bake process cannot lead to subsequent stages of tempering including decomposition of austenite, recovery and formation of coarse cementite particles as they are considered to occur only at higher temperature and longer time. It is noteworthy that similar precipitation behavior is expected for the paint baked double pulse (DoubleP-PB) sample (not presented here).

Figure 7-5 TEM bright field images of the martensitic structure and corresponding SAED pattern for the SingleP (a, b) and SingleP-PB (c, d).
7.3.3 Effect of weld scheme

In the resistance spot weld, the sharp pre-crack at the faying surface of two overlap sheets acts as an intrinsic crack at the weld nugget. The microstructural and mechanical characteristics of the area in front of the pre-crack are crucial as the pre-crack tends to start and propagates through this zone. In order to study the effect of the weld scheme on the crystallographic features of martensite and to make a structure-property relationship, the weld was polished down from the top surface until the half of the nugget at the faying surface of two sheets. Large area OIM scan data for the single pulse and double pulse welds in front of the pre-crack are shown in Figure 7-6 and Figure 7-7, respectively. The schematic image of the weld and the location of the scanned area in front of the pre-crack are also indicated. The plane of observation is parallel to the plane of the pre-crack that propagates into the fusion zone during the loading. Both the weld samples exhibit a typical lath structure of martensite. The columnar boundaries of PAGs can be clearly distinguished in the martensitic structure in the IPF map of single pulse weld Figure 7-6a. As can be seen clearly, the double pulse weld scheme transforms the initial columnar structure in the outer layer of the fusion zone to the martensitic structure with equiaxed PAGs (Figure 7-7a).

Figure 7-6 (a) Normal direction IPF, (b) CPP packet and (c) Bain packet maps of SingleP weld. (d) Calculated IPF texture in normal direction together with the schematic image of the scanned area.
Generally reported of K-S OR [17] was used to study the crystallographic features of the martensite formed during RSW. As explained in chapter 3, based on the K-S OR, a single PAG transforms to 24 different variants of martensite. These variants are classified into four close-packed plane (CPP) packets, which are composed of six variants keeping their \{110\} planes parallel to one of the four \{111\} planes. Figure 7-6b and 7-7b illustrate the map of CPP packets of the two samples in four different colors. 24 variants can also be classified by three different Bain groups as there are three different options for Bain axes. The 24 variants of martensite grouped into three different Bain packets are shown in three different colors in Figure 7-6c and 7-7c for the single and double pulse welds, respectively. Each Bain packet is composed of variants of martensite sharing low angle boundary, whereas every different Bain packets are separated by high angle boundaries. In both CPP packet and Bain packet maps, PAGBs are shown by black lines. The quantitative analysis of the microstructure of single and double pulse welds is listed in Table 7-1. As illustrated, the PAGs size becomes coarser by applying double pulse weld scheme.
CPP and Bain packet sizes are also smaller for the single pulse weld, although the difference is not as significant as PAG size. Overall, single pulse welding leads to finer microstructure compared to the double pulse welding.

<table>
<thead>
<tr>
<th></th>
<th>PAG diameter (µm)</th>
<th>CPP packet (µm)</th>
<th>Bain packet (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SingleP weld</td>
<td>46.4 ± 24.4</td>
<td>6.6 ± 1.4</td>
<td>5.3</td>
</tr>
<tr>
<td>DoubleP weld</td>
<td>87.3 ± 42.3</td>
<td>8.3 ± 1.3</td>
<td>6.3</td>
</tr>
</tbody>
</table>

Figure 7-8 shows the IPF map of crack propagation path into the FZ captured from the cross-section of the weld that corresponds to the cleavage fracture of single pulse weld. The PAGBs are shown in black lines in the IPF map. The insert shows the location of the scanned area in front of the pre-crack indicated with an open arrow. It confirms the transgranular fracture of the single pulse weld, i.e. without delamination of the structure from the prior austenite grain boundaries but largely deflected at PAGBs. Ductile fracture associated with bending and rotation of martensite blocks is also observed indicated by dashed circles. Hypothetically, as the fracture surface characterization reveals a transgranular cleavage fracture, coarsening of the martensitic microstructure of double pulse weld should deteriorate the mechanical performance of the weld, which is against the obtained cross-tension results.

The orientation of grains in the two samples with respect to the normal direction shows a significant difference between the texture of the solidification structure for the single pulse weld (Figure 7-6d) and thermo-mechanically processed structure of the double pulse weld (Figure 7-7d). The SingleP weld shows a stronger <001>//ND, whereas the dominant texture changes to <011>//ND in the case of the DoubleP weld. Orientation distribution function (ODF) was estimated by statistical kernel density estimation method [18] of OIM data collected with respect to the pre-crack direction. Figure 7-9a and b show the ODF section of the Euler space at 45° for the single and double pulse welds, respectively. As noticed from Figure 7-9a, the major component for the martensitic structure formed in the single pulse weld resulting from the rapid solidification of RSW shows maximum intensities at (001)[110] and (001)[110]
rotated cube texture. Applying the second pulse decreases the intensity of the rotated cube component while a texture with the major components at Goss (110)[001] and rotated Goss (110)[110] is developed (Figure 7-9b).

![Graph showing ODF at φ2=45° for the martensitic structure of (a) single pulse and (b) double pulse weld.](image)

**Figure 7-9** ODF at φ2=45° for the martensitic structure of (a) single pulse and (b) double pulse weld.

![Graph showing normal direction IPF map of reconstructed PAGs of (a) single pulse and (b) double pulse weld. The corresponding ODF textures corresponding to (a) and (b).](image)

**Figure 7-10** The normal direction IPF map of reconstructed PAGs of (a) the single pulse and (b) double pulse weld. (c) and (d) ODF textures corresponding to (a) and (b).

The texture development during RSW was further analyzed by the orientation of reconstructed PAGs. The normal direction IPF maps of PAGs for single and double pulse welds are shown in Figure 7-10a and b, respectively. Their corresponding ODF at φ2 = 45° are shown in Figure 7-10c and d. As shown in Figure 7-10c, the PAG structure of the single pulse weld shows a very strong texture with dominant component of cube {100}<001> texture. A more random texture is obtained for the
PAGs of the double pulse weld, although the major texture component is still cube. Cube texture is considered as the dominant component of the recrystallized austenite [19]. However, the prior austenite and also martensite microstructure of the single pulse weld resulted from the rapid solidification of the weld and the occurrence of recrystallization of austenite cannot justify the Cube texture of PAGs.

Table 7-2 Orientation of the grains numbered in Figure 7-10a and misorientation angle between their <001> and $\nabla T_{\text{max}}$.

<table>
<thead>
<tr>
<th>Grain number</th>
<th>Grain Orientation with respect to pre-crack</th>
<th>Pinched off?</th>
<th>Misorientation angle between &lt;001&gt; and $\nabla T_{\text{max}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>$(1\overline{1}27)\langle 29\overline{2}1\rangle$</td>
<td>No</td>
<td>5.2</td>
</tr>
<tr>
<td>2</td>
<td>$(3\overline{1}8\overline{2}3)\langle 28\overline{5}3\rangle$</td>
<td>No</td>
<td>8</td>
</tr>
<tr>
<td>3</td>
<td>$(1\overline{3}4)\langle 2\overline{1}5\overline{9}\rangle$</td>
<td>Yes</td>
<td>40</td>
</tr>
<tr>
<td>4</td>
<td>$(\overline{3}1\overline{2}5)\langle 4\overline{1}2\overline{5}\rangle$</td>
<td>No</td>
<td>3.6</td>
</tr>
<tr>
<td>5</td>
<td>$(15\overline{1}6\overline{4})\langle 12\overline{7}17\rangle$</td>
<td>Yes</td>
<td>51.9</td>
</tr>
<tr>
<td>6</td>
<td>$(\overline{1}1\overline{4})\langle 1\overline{5}1\rangle$</td>
<td>No</td>
<td>17.5</td>
</tr>
<tr>
<td>7</td>
<td>$(13\overline{5}2\overline{6})\langle 13\overline{1}3\overline{4}\rangle$</td>
<td>Yes</td>
<td>45</td>
</tr>
</tbody>
</table>

RSW with a stationary heat source leads to the formation of elongated grains with a texture highly dependent on the maximum thermal gradient ($\nabla T_{\text{max}}$). Table 7-2 shows the orientation of the grains numbered in Figure 7-10a with respect to the direction of the pre-crack. Assuming that $\nabla T_{\text{max}}$ lies in the plane of the section and its direction is parallel to the pre-crack from the center of the weld towards its edge, the minimum misorientation between <001> and $\nabla T_{\text{max}}$ was calculated. It is shown that the grains with their <001> direction making an angle smaller than 20° with the $\nabla T_{\text{max}}$ direction are allowed to grow across the weld nugget. In contrast, those grains that their <001> direction is misoriented with larger angles relative to $\nabla T_{\text{max}}$ are mostly pinched off and blocked by the neighbour fast growing grains. It is generally accepted that the major axes of the columnar grains during the solidification lies along the direction of $\nabla T_{\text{max}}$. In the case of metals of cubic lattice structures the grains that their <001> axes are mostly parallel to the maximum heat flow grow rapidly since they fulfill the favored crystal growth direction, whereas the other grains that are badly misoriented in this respect are stifled [20]. Careful examination of the orientation of grains with small angle between <001> and $\nabla T_{\text{max}}$ (i.e. grain number 1, 2, 4 and 6) reveals that their indices are close to the ideal cube {100}<001> texture. Thus, it can be inferred that the Cube texture of the austenite for the single pulse weld results from the solidification texture that tends to align the <001> axes of the columnar grains parallel to the maximum heat flow. The dominant cube texture of the double pulse weld may result from the recrystallization process that occurs once the second pulse is applied leading to equiaxed grains of prior austenite phase. Another possibility for the Cube texture of double pulse sample can be partial re-melting of the outer layer. In this case, partial re-melting of the PAGBs results in the transformation of columnar grains to the equiaxed ones. Thus, the structure keeps
its original texture without formation of new grains. As shown, the structure is more randomized compared to the single pulse weld as also shows a minor texture component around (113)[110] and Brass (110)[112]. Brass texture is considered as one of the main components of deformed austenite [21] and its minor presence with small deviation from ideal component may indicate the partial deformation of austenite under the pressure of the electrodes for the double pulse weld.

The variants of martensite proposed by K-S OR can be produced by 90° rotation of prior austenite about each of 24 <112> axes. However, Bain transformation can readily estimate the orientation of the martensite products, although it never happens in reality. Based on Bain transformation, 3 variants of martensite are obtained by 45° rotation around each of three <100> axes. Both single and double pulse welds show cube texture of PAGs. The rotation of 45° around <100> axes for the cube texture leads to the formation of rotated cube, Goss and rotated Goss martensite products, which is in correspondence with the ODF textures of Figure 7-9. The single pulse weld shows a stronger texture around the rotated cube component that is a typical texture in the as-cast columnar structures. It was already shown that columnar grains in the as-cast strips show pronounced rotated cube texture [22,23] and also they form a strong texture of <001> parallel to the normal direction in the cast slabs [24]. Waterschoot et al. [25] also showed an obvious variant selection in favor of the rotated cube instead of Goss and rotated Goss in the bcc structure transformed from cube texture of prior austenite. Transformation texture of the double pulse weld shows a stronger texture around rotated Goss and Goss components. It is known that the Goss orientation is formed by the shear texture of bcc materials [10]. Wittridge et al. [26] also showed that the presence of the Goss component in the bcc transformation structure is an indication that the prior austenite is subjected to shear deformation. The rotated Goss component is also known to be dominant after plane strain compression [27]. In the case of the double pulse weld, the structure is subjected to a larger plastic deformation applied by the electrodes. As a result the intensity of shear and compression texture components are intensified, leading to a stronger texture of Goss and rotated Goss compared to the single pulse weld at which the rotated cube orientation is the major component of the transformation texture from cube orientation of austenite.

7.3.4 Micromechanical properties

To examine the effect of the texture development and also post-heat treatment on the cross-tension properties of the resistance spot welds, micromechanical tests in front of the pre-crack were performed to obtain a deeper insight into the failure mechanism of the welds. Nanoindentation tests were carried out to evaluate the tensile properties of the region in front of the pre-crack. The reversed algorithm developed by Dao et al. [28] was used to extract the yield strength and strain hardening exponent. The extracted yield strength and work hardening exponent $n$ of the zone in front of the pre-crack for four different welds are shown in Figure 7-11.
The BM was also indented to verify the accuracy of the extracted yield strength and $n$ value compared with the corresponding properties measured by macromechanical standardized tensile test (red bars). As shown in Figure 7-11, there is a good agreement between the yield strength of the 3rd generation steel obtained from standard tensile test and the extracted value using the nanoindentation technique. The $n$ value is slightly overestimated by the nanoindentation technique; however, it can be still used for the comparative study of the different weld samples. The change in weld scheme and PB heat treatment make no big difference on the yield strength of the welds, although the SingleP sample shows slightly higher average yield strength compared to the other samples. Accordingly, insignificant change is observed for the $n$ values (0.040–0.055) of the welds. The data obtained using nanoindentation technique illustrates that the weld scheme and also post-heat treatment lead to minor change in the tensile properties of the weld in front of the pre-crack.

![Figure 7-11 Tensile properties of four different weld samples obtained using nanoindentation. The red bars show the tensile properties of the BM obtained from standard tensile test.](image)

In order to evaluate the local fracture toughness of the microstructure formed in front of the pre-crack, micro-cantilever bending was carried out. Figure 7-12 shows the experimental load-displacement curves of the weld samples. As already shown (see section 7.3.3), the change in the weld scheme results in change in the developed texture of martensite in the weld fusion zone. However, the difficulty of the micro-cantilever bending is that the process is a localized test making an evaluation of the global mechanical properties questionable. To overcome this problem and minimize the effect of anisotropy on the measured fracture toughness, the cantilevers were milled and notched inside the grains with an orientation representative of the texture of the whole microstructure. In the case of single pulse welds, the dominant texture is <001>//ND, whereas the major texture for the double pulse samples is <011>//ND. The inserts in the graphs of Figure 7-12 show the
approximate location of the milled cantilevers for each sample. It should be noted that the cantilever sizes have been drawn over-exaggerating, just for indication. As shown, the bending of all cantilevers is associated with considerable plastic deformation after yielding. Three different stages can be observed during the bending of the cantilevers.

All the samples show strain hardening effects with increasing load upon displacement after yielding (stage I). Then the maximum load is obtained, which is also characterized by the onset of crack growth, followed by the force plateau (stage II). This stage is followed by the gradual decrease in load vs displacement (stage III). Load-displacement curves displayed in Figure 7-12 provide preliminary insight on the fracture properties of different samples as the bending of SingleP cantilever is accompanied by a limited strain hardening after yielding and also the extension of the force plateau is negligible. The maximum load value is the lowest for this sample, whereas the DoubleP-PB cantilever shows the highest maximum load.

![Figure 7-12 Load-displacement curves of the micro-cantilever bending test for SingleP (a), SingleP-PB (b), DoubleP (c) and DoubleP-PB (d) samples. The inserts show the approximate location of the milled cantilevers for each sample. Cantilever sizes have been drawn exaggeratedly for better indication.](image)

Linear-elastic fracture mechanics (LEFM) was used to evaluate the conditional critical stress intensity \( (K_{IQ}) \) values for the bended samples using Eq. 5-7. As any small change in the geometrical dimensions such as bending length or beam thickness alters the force levels in the load-displacement curves, the respective stress intensity factor graphs normalized with the sample geometry were plotted against displacement using Eq. 5-7 and Eq. 5-8 as shown in Figure 7-13 without considering crack propagation. The inserts in Figure 7-13 show the notch tip after the loading of
cantilevers. A very rough fracture surface is observed for the tested cantilevers in front of the notch showing a ductile fracture behavior. However, compared to other samples, the SingleP cantilever exhibits a more homogeneous and flat fracture surface. Using LEFM the conditional fracture toughness value was calculated as 7.16, 7.52, 7.58 and 11.26 MPa m$^{1/2}$ for SingleP, SingleP-PB, DoubleP and DoubleP-PB welds, respectively. The plastic zone in front of the notch can be estimated using Irvin approximation [29]: $t_1 = \frac{K_{LEFM}^2}{3\pi\sigma_Y}$. ASTM standard [30] also sets restrictions for the sample dimension as the thickness must be larger than 2.5 ($\frac{K_{LEFM}^2}{\sigma_Y}$). For the sample thickness smaller than $t_1$, the measured fracture toughness corresponds to the plane-stress fracture toughness, while for the samples thicker than $t_1$, plane-strain fracture toughness is measured. The calculated plastic zone thickness $t_1$ for SingleP, SingleP-PB, DoubleP and DoubleP-PB samples is 1.6, 1.98, 1.98 and 4.57 µm, respectively. However, the critical sample thickness proposed by the standard is not fulfilled as it is calculated as 39, 46, 46 and 107 µm, correspondingly. Since the geometrical constraints are not met and also all the tested samples behave in a ductile manner, LEFM cannot be used to derive the valid fracture properties but it can provide the lower limit for the fracture toughness. However, the requirements for the measurement of plane-strain fracture toughness are fulfilled, thus, the results are presented as conditional fracture toughness.

![Stress intensity factor graphs corresponding to the load-displacement curves of Figure 7-12.](image)

**Figure 7-13** Stress intensity factor graphs corresponding to the load-displacement curves of Figure 7-12.

### 7.3.4.1 Crack tip opening displacement (CTOD) approach

In order to determine CTOD, it is needed to measure the crack mouth opening displacement (CMOD), according to Eq.5-9 in chapter 5. The CMOD was determined
using in-situ SEM observation. The load-CMOD graphs are plotted in Figure 7-14 for the bent samples as it can be a measure to evaluate the crack blunting and propagation behavior. As observed, the end of the force plateau for the SingleP sample is associated with the lowest CMOD compared to the other sample. In contrast, the DoubleP-PB shows the highest CMOD value at the end of force plateau. The end of force plateau is considered as the point when the crack nucleates, and with further displacement the crack grows with gradually decreasing load. Hence, it can be inferred that the notch is blunted more before the crack onset in the case of DoubleP-PB sample leading to larger CMOD value. The $K_{Q}$ values for the SingleP, SingleP-PB, DoubleP and DoubleP-PB were calculated using Eq. 5-9 and Eq. 5-10 as 9.44, 11.91, 12.34 and 16.03 MPa m^{1/2}, respectively.

![Figure 7-14 Load-CMOD graphs for (a) SingleP, (b) SingleP-PB, (c) DoubleP and (d) DoubleP-PB samples.](image_url)

#### 7.3.4.2 J-integral approach

For the sake of comparison and to position the results of the previous section in context we explored another approach, i.e. the $J$-integral method to characterize the fracture toughness of the materials with large-scale yielding. As presented in Figure 7-12, partial unloading segments were applied after specific interval of 500 nm. Partial unloading enables to track the crack extension during the loading of micro-cantilevers by measuring the beam stiffness. The crack propagation decreases the ligament size and thus leads to the reduction in the bending stiffness. Figure 7-15a
shows the change in stiffness as a function of the number of unloading segment for DoubleP-PB sample as an example. The sample shows increase in bending stiffness until the 4th unloading segment (maximum load) corresponding to the strain hardening stage. Thus, it can be reasonably assumed that no crack propagation occurs until the load-displacement curve attains the maximum load at the 4th unloading step. Then, there are small reductions in the stiffness of the beam until the 7th unloading part indicating the occurrence of crack propagation but with slower rate. And finally, the beam stiffness decreases steadily until the last unloading step that corresponds to the stable crack growth stage. The simulated beam stiffness normalized to the maximum stiffness value is plotted in Figure 7-15b. In the case of DoubleP-PB sample, the initial notch size $a_0/t$ is 0.58. The decrease in stiffness in percent for every $a/t > 0.58$ is calculated by dividing the stiffness of every unloading step by the stiffness value of $a_0/t = 0.58$. Thus, it is possible to calculate the crack size in different unloading segments from the relative decrease in stiffness using the graph in Figure 7-15b.

![Graph of beam stiffness](image)

**Figure 7-15** Measured beam stiffness for every unloading segment for DoubleP-PB cantilever (a). Stiffness plot for different $a/t$ ratios obtained from FEA (b).

Figure 7-16 represents the measured crack extension size for every unloading step. Two distinct stages can be identified. The first stage after the crack propagation is associated with a slow crack growth, i.e. a crack blunting stage. The second stage
is called stable crack growth and the crack growth rate is higher, which is also associated with gradual decrease in the load. The measured crack sizes in Figure 7-16 show that the SingleP sample has the largest crack size (~0.85 µm) after bending as opposed to the DoubleP-PB sample with the smallest crack size (~0.45 µm). Furthermore, the final crack size in the DoubleP sample is slightly larger than that of the SingleP-PB cantilever.

![Figure 7-16 Crack extension versus unloading step for the SingleP (a), SingleP-PB (b), DoubleP (c) and DoubleP-PB (d) samples.](image)

The calculated $J$ values for each unloading segment against crack size were calculated by Eq. 5-12 and plotted in Figure 7-17. SingleP sample shows the lowest critical $J$ value as opposed to the DoubleP-PB sample with the highest $J_c$ value. A small rise in $J_c$ is observed for the DoubleP sample compared to the SingleP-PB sample.

The conditional fracture toughness values measured using three methods, namely LEFM, CTOD and $J$-integral are summarized in Figure 7-18. LEFM provides only the lower bound of the fracture toughness for ductile materials. CTOD also delivers lower values for the fracture toughness compared to $J$-integral method. Nonetheless, the trend of the fracture toughness values for all the samples using three different methods are quite similar. SingleP shows the lowest fracture toughness, whereas the DoubleP-PB sample reaches the highest. Furthermore,
DoubleP sample exhibits a very small increase in fracture toughness compared to the SingleP-PB sample.

Figure 7-17 J-\(\Delta a\) curves for SingleP (a), SingleP-PB (b), DoubleP (c) and DoubleP-PB (d) cantilevers.

Figure 7-18 Measured conditional fracture toughness values using LEFM, CTOD and J-integral methods.

As generally accepted, the strength and to a certain extent also the toughness of martensite are improved by the refinement of the structure. PAG, CPP and Bain packets with high angle grain boundaries are effective against crack propagation and finer structure of these units can enhance the fracture toughness of martensite.
3rd generation AHSS resistance spot weld

However, double pulse welds with coarser martensitic units show higher fracture toughness implying the strong effect of texture on the mechanical response of the microstructure. Generally, cleavage fracture occurs along the \{001\} planes in the bcc materials as they provide an easy path for crack to propagate [33]. It is widely reported that the rotated cube component has a detrimental effect on the fracture properties of steel [9,18,34,35].

It was shown that decrease in the intensity of the rotated cube texture by appropriate thermo-mechanical process can enhance the fracture toughness of the structure. In contrast, the grains with orientation of <011>//ND play a key role to improve the fracture properties as \{011\} plane is one of the main slip planes of the bcc structure for plastic deformation. These planes contribute to the nucleation and coalesce of micro-voids and increase the amount of plastic deformation and absorbed energy during fracture. Hence, the major Goss and rotated Goss texture components of the double pulse weld in front of the pre-crack can be the main factor that results in higher fracture toughness compared to the single pulse sample. In the case of paint baked samples, the tempered martensite formed in front of the pre-crack becomes less strained compared to the fresh martensite of unbaked sample as the transition carbides precipitate inside the laths. It is also considered that the carbon segregation to lattice defect at the initial stage of the tempering may lead to slight increase in hardness [16]. However, no noticeable changes in the hardness of paint baked and unbaked samples are observed in the present study. This can be attributed to the relatively low carbon content of the steel that provides no sufficient carbon for hardening (~ 0.2 wt.%). Softening of martensite by low-temperature tempering is unlikely until the temperature rises up to 200 °C. In fact, low-temperature tempering of the spot welds during PB treatment results in neither significant increase nor decrease in the hardness of the martensitic microstructure, but adds certain amount of improvement to the fracture toughness of the martensite formed in the fusion zone of the weld.

The cracking behavior of SingleP and DoubleP-PB cantilevers with the lowest and highest fracture toughness was further analyzed by TEM as shown in Figure 7-19. The side of the bent cantilevers was further milled using FIB to roughly reach the center of the bent cantilever samples. Different failure behavior can be readily seen from the TEM images. The dashed yellow line lies on the initial crack before stable crack growth, while the red dash line highlights the crack after propagation. As shown, the initial crack is heavily blunted in the DoubleP-PB sample with the highest fracture toughness. In contrast, the crack blunting before crack propagation is much less effective in the case of SingleP sample. Besides, the difference in the final crack size of the two samples can be clearly seen, as the crack size is smaller for the DoubleP-PB cantilever. It should be noted that there is a relatively good agreement between the observed crack size by TEM and the measured crack extension using the beam compliance and FEA in Figure 7-16, although the TEM only shows the crack size in the center of the cantilever and the average crack size through the whole
Chapter 7

thickness might be a bit different. It was already shown by Skogsrud et al. [36] that for the crack lying on the (001) plane, small density of dislocations is emitted from the crack tip making dislocation loops as shown by white arrows in Figure 7-19a. These loops lead to the formation of voids slightly ahead of the tip immediately connected to the crack front upon increase in load. For this crack system, no strong pattern of dislocations is formed in front of the crack and they do not have an easy way to blunt the crack intensively. In the case of crack system lying on the (011), depending on the crack direction, the emitted dislocations can be either parallel or not parallel to the crack tip. If the emitted dislocations are not parallel to the crack front, the deformation will be concentrated in the crack center leading to the difference in crack propagation between the center and edge of the cantilever. In this case, the high amount the dislocation density at the edges leads to the relaxation of the crack tip and higher fracture toughness. If the emitted dislocations are parallel to the crack front, they can be formed on the slip system of \{112\} & \{111\} resulting in blunting of the crack front. In this crack system, the crack propagation is associated with a large density of emitted dislocations ahead of crack front leading to a large plastic deformation needed for failure. It is noteworthy that the blunting effect of the crack in DoubleP-PB arises from two factors. First, the crack is subjected to a larger plastic deformation because of change in the texture and second, low temperature tempering of the microstructure drastically increases the fracture toughness of the structure. These two parameters collectively lead to the smallest crack size associated with heavily blunted crack tip.

![Figure 7-19 TEM examination of the bent cantilever for SingleP (a) and DoubleP-PB (b) samples.](image)

Figure 7-20a and b are a comparison of the obtained load-displacement curves using FEA and corresponding experimental bending test for SingleP and DoubleP-PB samples until the maximum force is reached (the onset of crack initiation), respectively. A good agreement is found between the simulated and experimental results. Figure 7-20c and d show the stress distribution in front of the crack tip at
the onset of crack propagation for SingleP and DoubleP-PB samples, respectively. As it can be observed, the stress required for initiation of cracking in the notch tip of the cantilevers is attained essentially different both in terms of magnitude and distributions. For SingleP, maximum stress area (reaching 3.9 GPa) mostly spreads toward the opposite Y-axis with a narrow pattern, whereas, for DoubleP-PB sample, the stress field for the crack propagation (reaching 5.5 GPa) is expanded along X-axis. The simulation also provides a predictive view on the crack propagation upon further loading. As seen, for the SingleP sample, a larger area in front of notch reaches the required stress for the crack propagation proposing an easier path for the crack to extend with loading. In the case of DoubleP-PB sample, a very confined
area in front of notch shows the highest stress field needed for the crack to propagate. Thus, the crack can only extend through a limited distance and extra plastic deformation is needed for the further propagation. According to Figure 7-20 e and f, the higher equivalent plastic strain in maximum load before crack initiation is attained for DoubleP-PB sample. It is also more widened enabling the notch to hinder the sharp crack initiation, as opposed to the very thin and narrow strain localization in Z-direction for sample1 case.

The CTOD was measured as 95 and 233 nm from in-site SEM observation and using Eq. 5-9 for SingleP and DoubleP-PB, respectively. The corresponding values obtained from simulation are 75 and 145 nm, respectively. The relatively small deviation can be attributed to the errors in the CMOD measurements from SEM image, when it comes to detect the changes in distance at nano-scale. Nevertheless, the simulation also approves larger CTOD and also much stronger crack tip blunting before propagation in the case DoubleP-PB sample with the highest fracture toughness value.

### 7.3.4.3 Final remarks

A connection was found between the micro-mechanical performance of the formed microstructure in the weld nugget and its cross-tension properties. Results obtained from nano-indentation tests revealed that the change in weld scheme and also post-welding heat treatment leads to no significant change in the tensile properties of the weld zone in front of the pre-crack. Although the ductility was not examined in this study, the measured $n$ value from nano-indentation test shows the ability of the microstructure for uniform elongation after yielding and can be used as a rough estimation for the ductility of the weld. As shown, no noticeable change in $n$ value was measured. By contrast, the micro-cantilever bending demonstrated that the double pulse welding and also PB process after welding can effectively enhance the fracture toughness of the martensitic microstructure formed in front of the pre-crack. It is shown that cross-tension properties of the resistance spot weld are governed by the fracture toughness of the microstructure formed in front of the pre-crack. During the cross-tension test, the area in front of the pre-crack is subjected to mode I loading and notched micro-cantilever bending with the same loading configuration can be used to simulate the response the weld to the loading during large-scale mechanical test. The crack is heavily blunted during mode I loading of DoubleP-PB cantilever leading to higher cross-tension maximum load and energy absorption capability. By contrast, the weld microstructure of SingleP shows more brittle behavior during micro-cantilever bending providing an easier way for the crack propagation and also deteriorated cross-tension performance compared to other welds. The failure mode in the cross-tension test is the result of the competition between the shear plastic deformation outside the weld in the heat affected zone or the base metal and crack propagation into the fusion zone. If the fracture toughness in front of the pre-crack is high enough so that the yielding occurs outside the weld.
before crack propagation into the weld, the failure happens in PF mode. Smith [11] derived an equation for the critical weld nugget size to ensure PF failure based on this completion as follows:

\[ D_c = 2.93 \left( \frac{\tau_{CG}}{K_{F,CG}} \right) t^{4/3} \]  \hspace{1cm} (7-1)

where \( \tau_{CG} \) is the shear strength of the coarse-grained heat affected zone where the failure occurs in the PF mode, \( K_{F,CG} \) is the fracture toughness of the weld in front of the pre-crack and \( t \) is the sheet thickness. The yield shear strength of the HAZ was estimated from the obtained data from yield strength in tension of nano-indentation test as \( \tau_{CG} = 0.577 \sigma_{FG} \). Figure 7-21 illustrates the calculated critical weld nugget size (black dots) using Eq. 7-1 for different welding schemes and paint baking treated welds against the measured average weld nugget size (5 ± 0.1 mm). As observed, only for the DoubleP-PB sample, the critical weld nugget size that guarantees the PF mode is smaller than the experimentally measured weld nugget size. In the case of DoubleP sample, the measured value is very close to the critical weld size but still smaller. According to this graph, in the case of SingleP sample, to ensure that the weld fails in PF mode the minimum weld nugget size must be ~ 6.7 mm. To verify the validity of the measurements, extra welds were made with different nugget sizes as shown by red dots in Figure 7-21. The minimum weld nugget size proposed by the standard ANSI/AWS/SAE [15] was made for the DoubleP-PB sample. The weld size is smaller than the previously investigated sample but still slightly larger than the critical weld size. As shown, the DoubleP-PB sample even with the minimum weld nugget size fails in PF mode. In the case of SingleP-PB and DoubleP samples, the possible maximum current was used to make weld with the largest size right before expulsion. Both the weld sizes are larger than the proposed critical weld nugget sizes. DoubleP sample shows a PF as the weld fails from coarse-grained heat affected zone. However, in the case of SingleP-PB on the left side, there is a small penetration of the crack into the fusion zone that leads to PIF mode with a large plug ratio and small damage in weld zone. The small discrepancy between the proposed weld nugget size for PF and the observed failure mode at a larger weld size for SingleP-PB might be attributed to the single micro-mechanical measurement of the fracture toughness. More measurements with more statistical reliability can lead to more accurate prediction of the weld failure. The SingleP weld still shows no PF mode failure in accordance with the proposed critical weld size. It was not possible to make a weld with the nugget size larger than the critical weld size as expulsion occurs at such a large weld current. Nevertheless, the combined nanoindentation and notched micro-cantilever bending techniques seem to provide a comprehensive and deeper insight of the fracture behavior of the resistance spot welds.
Figure 7-21 The proposed critical weld nugget size for four spot weld samples (black dots). The dashed red line shows the average weld nugget size of the investigated samples. For verification, new weld samples with different nugget sizes were examined (red dots) and their failure mode have been shown as inserts to the graph.

7.4 Conclusions

An in-situ micro-cantilever bending method was successfully implemented in scanning electron microscopy to solve the problems associated with the resistance spot welding as a process widely used in mass production lines. The method was used to evaluate the effect of texture modification and post heat treatment on the local fracture toughness of the resistance spot welded 3rd generation high strength steel. Texture of the martensitic microstructure in front of the pre-crack was manipulated via change in weld scheme as double pulse welding changed the texture of the single pulse weld from $<$001$>$/ND to $<$011$>$/ND. Paint baking cycle was also applied at 180 °C for 20 min and as characterized by the precipitation of transition $\varepsilon$ carbides.

Micro-cantilever bending test revealed that change in the texture of martensite and also $\varepsilon$ carbide precipitation enhance the fracture toughness of the weld in front of the pre-crack. It was also shown that there is a direct correlation between the micro-fracture toughness of the weld and its cross-tension strength and energy absorption. Finally, the critical weld nugget size was calculated based on the nanoindentation and micro-cantilever bending results to predict pull-out failure of the weld at different welding parameters. A good agreement was observed between the proposed critical weld size and the experimental evidence of failure modes.

Reference


[31] S. Li, G. Zhu, Y. Kang, Effect of substructure on mechanical properties and fracture behavior of lath martensite in 0.1C-1.1Si-1.7Mn steel, J. Alloys Compd. 675 (2016) 104–115.


