Impact of crack propagation path and inclusion elements on fracture toughness and micro-surface characteristics of welded pipes in DWTT

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Abstract
Despite fundamental differences between the characteristics of base and weld metal and higher accuracies obtainable using original-size specimens in drop-weight tear test (DWTT), there still exists a research gap in fracture surface examinations of DWTT welded specimens. This study investigates the microscopic characteristics of the fracture surface of spirally welded API X65 steel with chevron notch (CN) and pressed notch (PN) DWTT specimens. Microstructures of different sub-zones were investigated, including, weld metal (WM), heat affected zone (HAZ), and base metal (BM). Scanning electron microscopy (SEM) investigations revealed the numerous inclusions in the WM that cause stress concentration. Micro-cracks are formed at the beginning of the fracture process when the energy level is high; nevertheless, in the shear fracture area, where energy is reduced, micro-cracks were not observed. Inverse fracture was located in the HAZ and BM in the PN and CN specimens, respectively. Comparison of the weight percentages of inclusion elements obtained by energy-dispersive x-ray spectroscopy (EDS) with API 5L standard target values showed that in most inclusions, Mn, Ti, and S were higher than standard values. After metallurgical and mechanical investigations, a framework for a new prospect has been introduced to conduct statistical analyses, based on which, the confidence intervals of the weight percentages of inclusion elements were carefully determined. Changes in the weight percentages of constituent inclusion elements in the WM are smaller compared to the BM and HAZ that would be reasons for better mechanical properties of the WM compared to the BM. Considering the impact of inclusions on weldability and toughness, industrial guidelines are presented to carefully control the elements within the confidence intervals of this research, during welding to minimize the formation of inclusions, in turn, reducing the formation and growth of micro-cracks, and significantly improving mechanical properties.

1. Introduction

API steels, which are commonly used in the pipeline industry, have high strength and excellent toughness. Therefore, to provide a deeper understanding of the role of constituents and processing parameters, basic material properties, and the resulting mechanical behavior of these steels and pipes made of them, three main types of researches have been conducted focusing on alloy contents and fracture behavior [1–5], weld properties [6–8], and fracture and corrosion evaluations [9, 10].

Among the first type of research, Bakaloğlu [11] investigated the impacts of rolling processing parameters, including finish rolling temperature, thickness reduction percentage, inter-pass time, and cooling rate, on the
microstructure and mechanical properties of X52 high-strength low-alloy (HSLA) steel through tensile and Charpy impact tests and optical and electron microscopy. However, the focus is placed on steel plates, instead of welded pipes and a more comprehensive study would be required to generalize the results to pipe steels, accurately. Zhao et al. [12] examined the static and dynamic continuous cooling transformation diagrams of low carbon pipeline steels with different carbon and alloy contents and concluded that their behavior is greatly affected by alloy content and hot deformation. Nevertheless, they only focused on alloy contents that promote the development of acicular ferrite and suppress polygonal ferrites in the BM. In order to provide an accurate understanding of the performance of pipeline steel, further investigation of the weld metal would be required. Korczak [13] presented the composition of steel with optimum alloy content and investigated the microstructure evolution of steel plates in the thermo-mechanical rolling of pipeline plates and their respective cracking resistance. Although the author claims to have provided a composition with optimal alloy contents, few details have been presented on the optimization process, and a more detailed analysis would be required to assess the precise alloy composition to provide optimal fracture response. Sun et al. [14] studied bimetallic hot compression tests of X65/316L by investigating bimetallic bonding mechanisms, including microstructure evolution, element diffusion, and macroscopic deformations and proposed a diffusion model to estimate element concentrations in the metallurgical bonding transition zone. Although the presented model is claimed to predict Fe, Ni, and Cr concentrations with relatively high accuracy, an inclusive study of other elements and inclusions would be required to precisely estimate the behavior of the bimetallic clad pipes, especially in fracture.

Due to the necessity of applying welds in pipelines, the second class of studies have focused on the microstructure and mechanical properties of pipeline weld metal (WM) in contrast to the base metal (BM) [15–19]. For instance, Motohashi et al. [20] investigated the toughness, strength, and hardness of X80 pipelines weld joints considering weld metal composition and grain sizes, and welding conditions, from a microstructural viewpoint. However, their investigations were primarily focused on overmatching of welded joints to guarantee the behavior of X80 pipes in earthquakes, and impact tests were performed using Charpy v-notches solely on weld metal in the HAZ. Huang et al. [21] performed finite element simulations focusing on the effects of in-service welding temperature field on HAZ dimensions in gas pipelines. The numerical simulations studied the pressure and flow rate of the cooling media (methane gas) as determinants of the heat transfer coefficient for in-service welding of S355J2G3 high strength structural steel pipes, without focusing on the chemical composition and changes in the microstructure and compositions of the weld region. Most recently, Kumar et al. [22] investigated the metallurgical characteristic and microstructure evolution of Ti-6Al-4V alloy in TIG welding. The study mainly focused on the analysis of metallurgical phases, hardness, and residual stress, without considering their implications on weld strength and fracture toughness.

To investigate fracture behavior of pipeline steels, more recently, the third class of research studies have focused on the different fundamental laboratory methods and techniques for evaluating fracture properties, which correspond to full-scale fracture tests. Asghari et al. [23] proposed a novel linear model to estimate fracture toughness based on yield strength and Charpy impact energy, in the base, seam weld, and girth weld metals of API X65 line pipe steel. Although the predicted fracture toughness values obtained from the linear model were claimed to match experimental measurement with a mean error value of 4.4%, however, the model was only applied to three specimens within the normal range and needs to be tested using other specimens for accuracy outside of this range. Bohle et al. [24] studied the effects of nickel and molybdenum alloy additions during submerged arc welding (SAW) on the fracture toughness of API HSLA-70 steel at various, including extreme sub-zero temperatures. Their investigations were concentrated only on Mo and Ni compositions in the WM, and WM impact tests were only performed using Charpy v-notches. Beidokhti et al. [25] studied API 5L-X70 pipeline steel, considering the effect of Ti addition during SAW on WM microstructure and toughness, and only one type of impact test was performed using Charpy v-notches.

Among the applied methods for measuring impact toughness, standard Charpy tests are commonly used, especially in industrial applications. The drop-weight tear test (DWTT) has recently showed the potentials to reveal more realistic findings related to ductile fracture resistance compared to the Charpy test. Sha et al. [26], based on research on delamination in the BM of API X80 pipeline steels using DWTT, explained that this is due to the use of specimens with original size and a longer fracture path, able to attain steady-state fracture resistance. Due to the significance of investigating fracture surface characteristics, researchers have tried to explain the correlation between surface characteristics and, among others, microstructure and mechanical properties. Consequently, studies have been conducted on the fracture of DWTT specimens, to investigate the effects of notch shape, thickness, temperature, microstructures, and mechanical properties. Fang et al. [27] presented a regression-based model to study the dynamic cracking behavior and crack-tip-opening-angle (CTOA) of API X70 line pipe steel PN DWTT specimens to estimate the effect of thickness on ductile fracture resistance. However, they only studied the BM and concluded that the critical CTOA is constant and thickness-independent in the steady-state regime. Zhao et al. [28] investigated the effects of fracture surface microstructure and properties of API X80 steel in PN DWTT. Although they paid special attention to the effects of
microstructure on crack and fracture in DWTT tests, only the BM was studied. Despite the numerous researches
investigating fracture surface and properties of WM in Charpy tests, a limited number of research studies have
been performed based on DWTT, most of which were limited to the fracture surface of pipeline BM and few
have included the fracture surface of WM specimens in DWTT, despite the difference in mechanical and
fracture properties of the WM and its microstructure with those of BM. On the other hand, anisotropy and the
presence of inclusions in the weld metal result in the degradation of the mechanical properties. For the
first time, Majidi-Jirandehi and Hashemi [29] investigated the fracture surface of spiral seam weld in API X65 pipeline steel
using chevron-notch (CN) DWTT specimens; however, they only performed macroscopic characterization of
the fracture surfaces, and further detailed microscopic evaluations need to be carried out. Hashemi and
Mohammadyani [30] performed Charpy and Vickers hardness experiments on the BM, WM, and HAZ of
spirally-welded API X65 pipeline, and reported the minimum Charpy fracture energy (160 J) and maximum
hardness level (218 HV) in WM. Majidi-Jirandehi and Hashemi [3] measured the fracture energy of WM using
DWTT specimens with PN and CN and showed that the fracture energy of WM is lower than that of the BM,
without detailed microscopic surface characterization.

Therefore, the study of the fracture surface of the weld metal, especially its microstructure and composition,
is critical and can significantly enhance the understanding of the properties of the fracture surface.
Consequently, in the current study, microscopic fracture surface characteristics of API X65 spiral welded seam is
investigated in the middle of the specimens and alongside the crack propagation path using chevron notch (CN)
and pressed notch (PN) DWTT specimens. The remainder of the paper is presented as follows. First, the
specifications of the test materials and the experimental procedure are described. Then, the fracture morphology
and characteristics of the fracture surfaces are discussed. After that, microstructure of the fracture surface along
the crack path from the WM into the HAZ and BM zones is investigated. Next, SEM fractography is performed
on the surface in three areas, including cleavage fracture, shear fracture, and inverse fracture, and results of EDS
analyses conducted on the inclusions are presented. Finally, the mean difference of weight percentage of
inclusion elements between WM, HAZ, and BM inclusions were analyzed using analysis of variance (ANOVA)
and Tukey’s tests, to determine the zones in which mean differences are statistically significant. At the end,
concluding remarks are presented including implications and guidelines on the impact of inclusions on weld-
ability and toughness, to help control the elements within the confidence intervals obtained in this research, in
the welding procedure, to reduce the formation and growth of micro-cracks, and significantly improve
mechanical properties.

2. Material and experiment

API steels specifications and weld area characteristics have been standardized by the American Petroleum
Institute (API) [31, 32]. As indicated by Reip et al [33] and Shim et al [34], API steels have desirable mechanical
properties, among which are high toughness and strength, and also good weld-ability. The measured material
properties, hardness, Charpy, and DWTT energy of API X65 steel are presented in [35–37].
2.1. Specifications of test material

The steel under investigation in this research was spirally welded API X65 machined from an actual pipeline with 14.3mm wall thickness and 1219 mm outside diameter. Table 1 lists the chemical composition of the BM and WM were according to target values specified by API 5L standard \[32\]. The measured material properties, hardness, Charpy, and DWTT energy of API X65 steel are given in table 2.

| Table 1. Chemical analysis of WM, BM using emission spectroscopy \[30\]. |
|---------------------------------|-----------------|-----------------|----------------- |
| Element | Weight% in WM | Weight% in BM | API 5L X65 |
|---------|----------------|----------------|-------------|
| Fe      | Base           | Base           | —            |
| C       | 0.073          | 0.072          | 0.220 (max) |
| Mn      | 1.370          | 1.450          | 1.450 (max) |
| P       | 0.010          | 0.008          | 0.025 (max) |
| S       | 0.003          | 0.002          | 0.015 (max) |
| Ti      | 0.008          | 0.015          | 0.06 (max)  |
| Si      | 0.246          | 0.201          | —            |
| Nb      | 0.028          | 0.047          | —            |
| Cr      | 0.127          | 0.174          | —            |
| Mo      | 0.017          | 0.240          | —            |
| V       | 0.003          | 0.050          | —            |
| Ni      | 0.002          | 0.009          | —            |
| Cu      | 0.031          | 0.008          | —            |
| Al      | 0.014          | 0.023          | —            |

| Table 2. Material properties of API X65 steel. |
|-----------------------------------------------|
| YM (GPa) | YS (MPa) | TS (MPa) | Hardness (HV10) | Charpy energy (J) | DWTT energy (J) | References |
|----------|----------|----------|------------------|-------------------|-------------------|-------------|
|          |          |          | BM | HAZ | WM | BM | HAZ | WM | BM | HAZ | WM |
| 210      | 490      | 552      | 211 | 208 | 218 | 264 | 190 | 160 | 264 | 190 | 160 | \[30\] |
| —        | 510      | —        | —   | —   | —   | 266 | —   | —   | —   | —   | —   | —   | \[35\] |
| —        | 46505    | —        | —   | —   | —   | 286 | —   | —   | —   | —   | —   | —   | \[35\] |
| —        | 469      | —        | —   | —   | —   | 126 | —   | —   | —   | —   | —   | 5915 | \[36\] |
| —        | 461      | —        | —   | —   | —   | 7600 | —   | —   | —   | 7600 | —   | —   | \[37\] |
| —        | —        | —        | —   | —   | —   | —   | —   | —   | —   | —   | —   | 7085 | CN = 5890 PN = 6211 | \[3\] |

YM: Young’s modulus, YS: yield strength, TS: tensile strength, BM: Base metal, HAZ: Heat affected zone, WM: Weld metal.

Figure 2. Schematics of DWTT test specimens with pressed notch and Chevron notch, along with hammer and supports.
2.2. Experimental procedure

Specimens were cut by flanging from the original spiral welded pipe (figure 1(a)) and were then flattened. The specimens were machined in accordance with the dimensions metered in API 5L3 standard (305 × 76.2 × pipe thickness mm) [38]. Specimen notches were aligned to the spiral seam of the pipeline, according to figure 1(a).

The DWTTs were conducted with a 30kJ impact machine. After DWTTs, the samples were prepared for metallography and imaging with a scanning electron microscope (SEM). The fracture surfaces of the specimens were divided into complete SEM specimens, as shown in figures 1(b), (c). This division procedure is based on macroscopic characteristics of the fracture surface. The standard form of test samples is shown in figure 2. In addition, the dimensions of the samples, hammers, and supports are shown in table 3, in which, S is the distance between the two supports, Rh is the hammer radius, Rs is the support radius, a is groove depth, and L, W and t are the sample length, width, and thickness, respectively. All experiments were performed at ambient temperature and with an impact velocity of 6.3 m s⁻¹.

3. Result and discussion

3.1. Fracture morphology

Figure 3 shows the fracture surfaces of specimens after DWTT on the API X65 steel pipe. As can be seen in figure 3, both specimens show cleavage fracture directly under the notch, followed immediately by a shear fracture. The shear fracture surface occupies the majority of the fracture area and has spread throughout the total width of the specimen. Longitudinal delamination, roughly parallel to the fracture growth direction, longitudinal beach marks, and V-shaped beach marks, is only seen in the shear fracture area of CN specimens.
| Specimen | Length of cleavage fracture (mm) | Length of shear fracture (mm) | Length of inverse fracture (mm) | Maximum crack path deviation (mm) | Number of delamination | Maximum thickness (mm) | Minimum thickness (mm) |
|----------|---------------------------------|------------------------------|-------------------------------|---------------------------------|------------------------|-----------------------|-----------------------|
| CN       | 16                              | 40                           | 16                            | 18                              | 10                     | 18                    | 12                    |
| PN       | 10                              | 45                           | 18                            | 12                              | 0                      | 20                    | 13                    |
In both PN and CN specimens, shear lips and inverse fracture can be seen in the hammer impacted zone. Moreover, the CN specimen has a smaller inverse fracture area compared to the PN specimen (the inverse fracture is 18mm and 16 mm in figures 3(a) and (b), respectively).

A summary of the characteristics of the fracture surfaces of the PN and CN specimens is shown in table 4.

As shown in table 4, the crack path deviation in the CN specimen (18mm) is higher than that of the PN specimen (12mm). Since the width of the seam weld was 20 mm, the crack path entered the HAZ and BM in the CN specimen. As can be seen in figure 4, the thickness variation in the PN specimen has been more significant than that of the CN specimen.

It should be noted that the minimum and maximum thicknesses of the CN specimen are 12 and 18mm, respectively, and 13 and 20 mm in the PN specimen, respectively. Because the value of DWTT fracture energy in PN specimens was higher than CN specimens and part of this energy, which is called 'initiation fracture energy', causes plastic deformations. The notch root of the CN specimen is sharper than the PN specimen. This resulted in higher strain energy stored at the notch root, thus larger plastic deformations in the PN specimen.

3.2. Percent shear area

The fracture area evaluated in the determination of the shear area percentage is calculated according to API 5L3 [38]. The percentage of the shear area, including inverse fracture and the percentage shear area not including inverse fracture, is shown in the following equations [39]:

![Figure 4. Specimen thickness versus distance from the notch.](image)

![Figure 5. Microstructure of API X65 (a) BM, (b) WM.](image)
Table 5. Percent shear area of PN and CN spiral weld of API X65.

| Specimen | Percent shear area | \( A \) (mm) | \( B \) (mm) | \( A' \) (mm) | \( B' \) (mm) |
|----------|--------------------|---------------|---------------|----------------|----------------|
| CN       | Not including inverse fracture | 99.4 | 2.1 | 0 | 0 |
|          | Including inverse fracture     | 99.4 | 2.2 | 0 | 0 |
| PN       | 100                 | 97.4 | 0 | 5.1 | 4.2 |

Figure 6. SEM of the PN specimen, (I) cleavage fracture area, (II), (III) shear fracture area, (IV) boundary between shear and inverse fracture areas, (V) inverse fracture area, (a) area A in part I, (b) area B in part I, (c) voids in part I and the inclusions therein, (d) area C shown in part III, (e) area E shown in part (d), (f) area D shown in part IV, (g) shear lips at the end of the PN specimen, (h) area F shown in part (g), (i) cleavage fracture accompanied by a ductile fracture from the hammer impacted zone, (k) inverse fracture area and the micro-cracks observed in it.
Table 6. EDS analysis of inclusions and impurities.

| Element       | S   | Mg  | Ti  | Fe  | Al  | Si  | Ca  | Mn  | O   | K   | Cl  | Na  | Cr  | C   |
|---------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Inclusion B figure 6(c) | 0.63 | 0.59 | 1.19 | 7.78 | 8.59 | 11.30 | 3.01 | 15.71 | 50.47 | —   | —   | —   | —   | —   |
| Inclusion A figure 6(e) | 0.20 | 1.48 | 1.94 | 4.50 | 12.87 | 10.69 | 8.28 | 7.41 | 52.64 | —   | —   | —   | —   | —   |
| Impurity A figure 6(h) | 6.98 | 2.62 | 0.25 | 28.25 | 22.07 | 0.17 | 6.54 | 3.59 | 29.33 | —   | —   | —   | —   | —   |
| Impurity B figure 6(h) | 16.29 | 0.67 | 0.30 | 39.51 | 6.43 | 0.12 | 4.41 | 20.91 | 11.36 | —   | —   | —   | —   | —   |
| Weight percentage |     |     |     |     |     |     |     |     |     |     |     |     |     |     |
| Inclusion A figure 7(a) | 0.42 | 0.20 | —   | 2.81 | 0.09 | 0.07 | 0.28 | 0.16 | 25.10 | 1.46 | 1.55 | 2.22 | 0.14 | 65.51 |
| Inclusion B figure 7(b) | 0.38 | 1.00 | 4.88 | 4.75 | 10.22 | 8.05 | —   | 17.79 | 52.83 | —   | —   | —   | —   | 0.11 |
| CN           |     |     |     |     |     |     |     |     |     |     |     |     |     |     |
| Inclusion A figure 7(d) | 4.51 | 1.08 | 0.23 | 64.85 | 7.47 | 0.64 | 8.93 | 1.02 | 11.8 | —   | —   | —   | —   | —   |
| Inclusion B figure 7(e) | 13.84 | 0.94 | 3.12 | 18.19 | 9.41 | 0.75 | 33.60 | 1.61 | 18.54 | —   | —   | —   | —   | —   |
| Impurity A figure 7(g) | 12.28 | 0.46 | 1.24 | 6.54 | 21.87 | 1.91 | 24.60 | 0.89 | 30.20 | —   | —   | —   | —   | —   |
| Impurity A figure 7(k) | 4.92 | 11.30 | 0.91 | 3.06 | 24.99 | 0.39 | 6.50 | 1.05 | 46.88 | —   | —   | —   | —   | —   |
percent shear area including inverse fracture = \frac{(71 - 2t) - (3/4)(AB + A'B')}{(71 - 2t)t} \times 100 \quad (1)

percent shear area not including inverse fracture = \frac{(71 - 2t) - (3/4)(AB)}{(71 - 2t)t} \times 100 \quad (2)

where t, A, and A' are thickness, and the width of the initial cleavage fracture and inverse fracture (at the ‘one-t’ line beneath the notch), respectively. Similarly, B and B' are lengths of the initial cleavage fracture and inverse fracture (in between the ‘two-t’ line), respectively.

The delaminations in the tested specimen are parallel to the fracture surface. According to the API 5L3 standard, this type of delamination should not be included in the percent shear area [38]. The percentage shear area of DWTT on PN and CN spirally-welded API X65 specimens is shown in table 5, where t is a full plate thickness (14.3mm).

Several observations of abnormal fractures have been reported in DWTT specimens and can be categorized into three types, according to the sources of cleavage fracture[40]. Many methods have been suggested to reduce the inverse fracture area (cleavage fracture area close to the hammer impacted zone, known as abnormal fracture appearance), such as correcting the notch from pressed to a sharp root notched (such as CN), using roller support in the DWTT machine and back-slotted specimens.

In the CN specimen, the percent shear area including (equation (1)) and not including (equation (2)) inverse fracture area was equal (99%). Because using the CN can reduce the inverse fracture, while percent shear area measured not including inverse fracture (100%) was higher than that measured, including the inverse fracture (97%) in the PN specimen. On the other hand, using a blunted notch in the specimen can increase the inverse fracture area in WM. High shear area percentages (99% and 100% for CN and PN, respectively) indicate the excellent ductility of the spiral weld of API X65. However, the total fracture energy of WM (5890J and 6211J) for CN and PN, respectively is less than that of BM (7085J), and fracture properties of WM should be given more attention.

3.3. Microstructure of the fracture surface

Figure 5 shows optical microscopy images of the steel used in the present study (API X65). Since the crack path was digressed from the WM into the BM in the DWTT specimens, investigation of sub-zone microstructures in the fracture surface is of great importance.

The presence of acicular ferrite in the BM zone (figure 5(a)) increases the toughness, whereas Widmanstatten ferrite in the WM zone (figure 5(b)) can reduce the toughness. Propagation of cracks is deflected as they cross an acicular ferrite microstructure, hence, improving toughness [41]. Investigations of the mechanical properties of boundary ferrites with co-axial coarse grains at the center of WM show that boundary ferrite is detrimental to toughness [28, 41]. The structure of the steel has been characterized in the present study (API X65) using optical microscopy, in accordance with other similar studies, although EDS analyses are performed to obtain the weight percentages of inclusion elements. This point has been added to the manuscript and further references have been provided.

3.4. SEM Fractography

3.4.1. PN specimen

To conduct a complete analysis, the surface of the PN specimen is divided into three areas (see figure 1(b)), cleavage fracture, shear fracture, and the inverse fracture area. An overview of these three fracture areas is shown in figure 6 (parts I to V).

Figures 6(a) and (b) show areas A and B of figure 6(I), respectively. Figure 6a shows the edge of the specimen beneath the PN, where the shear fracture has occurred, while figure 6b shows the initial cleavage fracture. This type of fracture is the verification condition of the DWTT based on the API 5L3 standard [38]. As can be seen, micro-cracks are present in both areas and also voids exist to add to the micro-cracks. Larger voids are formed as a result of smaller voids converging.

The main reason for the formation of small voids was inclusions. Micro-cracks in the cleavage fracture area (figure 6b) are deeper and larger compared to those in the shear fracture area (figure 6a). Crack path propagation in the PN specimen was almost straight and entered HAZ at the end of the fractured specimen. Figures 6(I), (II), and (II) are located in WM. Figure 6c demonstrates the voids in the initial zone of the fracture surface (figure 6I) with inclusions present in the center. Energy Dispersive x-ray Spectroscopy (EDS) analysis of these inclusions is shown in table 6, which verifies the presence of oxide inclusions. As indicated by table 6, the weight percentages of Mn, Ti, and S in inclusion B of figure 6(c) were remarkably higher than the target values specified by API 5L. It is well-known that sulfur and oxide inclusions reduce weld-ability and toughness.
As mentioned above, WM mainly consisted of boundary ferrite, which diminishes the mechanical properties. Inclusions in WM also cause stress concentration around them. On the other hand, at the beginning of the fracture process, the hammer impact force is very high, causing the formation of micro-cracks.

Figure 6(d) shows area C of figure 6(III), which shows the edge of the sample, in which micro-cracks do not exist, unlike the areas shown in figures 6(a), (b). This is because steady crack growth has occurred in the shear fracture area, and the amount of energy is reduced to below the level enough for superficial voids to join and
form micro-cracks. However, voids and inclusions are also observed in the shear fracture. An example of these voids is shown in figure 6(e), which was acquired from area E in figure 6(d). EDS analysis of the inclusion shown in figure 6(e) (point A) is presented in table 6, which shows the presence of iron oxide inclusion. This type of inclusion can affect the toughness of the WM. Coarse grains of WM microstructure and numerous inclusions have a significant effect on toughness, and hence, crack propagation occurs easier. Similar to the previous inclusion, the weight percentages of Mn, Ti, and S in inclusion A of figure 6(e) were also higher than the target values specified by API 5L [30].

Figure 6(f) shows area D in figure 6(IV), which was obtained from the beginning of the inverse fracture area and shows the cleavage fracture and shear fracture areas next to each other. Figure 6(g) shows the shear lips at the end of the specimen. Area F in figure 6(g) can be seen in figure 6(h), where ductile dimple mode and the impurities therein are clearly visible. The EDS analysis of points A and B in figure 6(h) are presented in table 6. The inverse fracture area in the PN specimen enters the HAZ, and therefore its weight percentage of iron is high. In these inclusions, the weight percentages of Mn and S were higher than the target values specified by API 5L.

The SEM from the hammer impacted zone of the PN specimen (figure 6(IV)) mostly shows the joint presence of ductile and cleavage fractures (figure 6(i)); however, in this area, the cleavage fractures are more common than ductile dimples (figure 6(k)). The inverse fracture was affected by strain hardening [39]. Hammering exerts much force on the specimen, increasing the dislocation density and creating conditions that facilitate the cracking of the specimen.

In the PN specimen, inverse fracture was visible in the HAZ, where Widmanstatten ferrites that create stress concentration and provide conditions to facilitate the cracks adjacent to cleavage plates. The density and size of micro-cracks in the inverse fracture area are less than those of the initial cleavage fracture (beneath the notch) since less energy is consumed to crack propagation in the inverse fracture area than in the initial cleavage fracture.

3.4.2. CN specimen

Similar to the PN specimen, the fracture surface of the CN specimen is divided into three areas, cleavage fracture (figure 7(I)), shear fracture (figures 7(II) to (IV)), and inverse fracture (figure 7(V)).

Figure 7(a) shows area A of figure 7(I) in greater detail. As shown in this figure, cleavage fracture and micro-cracks are among the characteristics of this area. The EDS analysis of inclusion in the cleavage fracture (point A in figure 7(a)) is shown in table 6, indicating the presence of iron carbide (cementite) and iron oxide compounds.

Iron carbide is usually brittle and can act as a stress concentration point that helps fracture growth and crack formations. Because carbides are usually in needle form, they have a high-stress concentration. Figure 7(b) shows the SEM image of area H in figure 7(a). As observed, cleavage fracture areas are surrounded by ductile dimples. EDS analysis of inclusions found in ductile dimple (point B in figure 7(b)) is also shown in table 6, indicating the presence of silicon and aluminum oxide precipitates. In other points of this area, similar precipitates have been found, and interestingly, that they have high contents of aluminum, silicon, and magnesium. Some of these elements were probably remnants of the welding procedure. Considering all the mentioned points, it should be noted that these precipitates do not change the type of fracture, they only create stress concentration points and micro-cracks.

Also, this area is located in WM that mainly consisted of boundary ferrite that reduces the toughness. If the impact force is high, conditions are suitable for cleavage fracture growth. Consequently, around these inclusions, the cleavage plates or micro-cracks were formed. In general, like similar inclusions found in WM, the weight percentage of Mn, Ti, and S of these inclusions were very higher than the target values specified by API 5L [30].

The shear fracture area (figures 7(II), (III), (IV)) is located after the primary cleavage fracture area (figures 7(I)) in the CN specimen. At the start of the shear fracture, some longitudinal beach marks that are shown in figures 7(II) and 7(c) (area B of figure 7(II)) can be observed. The walls of the longitudinal beach marks are formed irregularly, and larger magnification of them demonstrated cleavage planes that are shown in figures 7(d) and (e). Results of EDS analyses on inclusions in this area (inclusions A and B in figure 7(d) and (e)) are presented in table 6. Considering that in the shear fracture area, the path of fracture deviates to the HAZ, the amount of iron detected in the analysis is noticeably higher. In the shear fracture area, shown in figures 7(III) and IV, longitudinal delamination is aligned almost parallel to the fracture growth path. Figure 7(f) shows the end of one of these delamination areas (area C in figure 7(III)). Images with further close-ups of this delamination are shown in figure 7(g) (area D in figure 7(III)) and 7(h) (area E in figures 7(III) and 7(g)). These figures show that the interiors of delamination areas have a cleavage plane, though ductile dimples surround them. EDS analysis of impurity A shown in figure 7(g) is presented in table 6, reporting the presence of aluminum oxide and silicon precipitates. Such precipitates were detected in other areas as mentioned before, and as pointed out there, they cannot change the type of fracture. However, the impurities, along with the HAZ anisotropy, have been
Table 7. Mean differences in the percentages of inclusion elements in WM, HAZ, and BM and confidence intervals of the weight percentage of inclusion elements.

| Element | Mean ± SD | p-value | Confidence intervals |
|---------|-----------|---------|----------------------|
|         | BM       | HAZ     | WM       | BM | HAZ | WM |
| O       | 43.84 ± 7.50 | 24.99 ± 11.02 | 42.66 ± 10.27 | 0.005 | 25.20–62.48 | 14.79–35.19 | 36.13–49.14 |
| Mg      | 4.36 ± 3.49 | 2.19 ± 0.40 | 0.67 ± 0.15 | 0.053 | 2.57–10.97 |
| Al      | 22.82 ± 3.06 | 14 ± 3.18 | 6.77 ± 1.92 | 0.006 | 9.62–36.02 | — | 2.37–10.97 |
| Si      | 1.30 ± 0.45 | 2.32 ± 1.62 | 4.60 ± 1.31 | 0.370 | 36.13–49.14 |
| S       | 4.43 ± 2.37 | 9.07 ± 2.12 | 0.49 ± 0.15 | <0.001 | 3.86–14.28 | 0.16–0.81 |
| Ca      | 15.40 ± 4.45 | 14.09 ± 4.14 | 4.63 ± 1.52 | 0.027 | 3.95–24.24 | 1.28–7.98 |
| Ti      | 0.67 ± 0.33 | 1.20 ± 0.41 | 4.30 ± 1.77 | 0.265 | — | — |
| Mn      | 0.55 ± 0.28 | 7 ± 3.46 | 13.09 ± 4.05 | 0.254 | — | — |
| Fe      | 6.60 ± 4.20 | 25.10 ± 5.45 | 18.04 ± 3 | 0.356 | — | — |

SD: Standard Deviation. *: Based on the ANOVA test. Based on the Tukey test; #: significantly different from BM, #: significantly different from WM zone.

identified as the cause of delamination, because the high weight percentage of sulfur in the impurity can reduce the toughness.

SEM images of the inverse fracture area in the CN specimen (figure 7(V)) are very similar in comparison with the PN specimen. Nevertheless, in the CN specimen, micro-cracks were not observed in the inverse fracture area, because this zone of the specimen was in the BM, resisting the formation of micro-cracks. At the end of the specimen, shear lips were formed as well as an inverse fracture area which can be seen in figure 7(f); ductile dimple mode is a sign of shear lips. The start of the inverse fracture area (area G in figure 7(V)), which includes the cleavage plane, is shown in figure 7(k). In the current study, the inverse fracture area of the WM specimen in CN is smaller than the PN, and delamination was only seen in CN specimens. The EDS analysis of impurity that can be seen in the inverse fracture area (inclusion A shown in figure 7(k)) is presented in table 6. Since, at the end of the specimen, the fracture path entered the BM, table 6 shows the weight percentage of BM impurity. In this impurity, the weight percentage of sulfur, aluminum, and magnesium is very high that can assist the formation of cleavage plates.

3.5. The mean differences of inclusion elements

In the previous sections of this study, inclusions were investigated from a combined metallurgical and mechanical point of view. However, in this section, a new prospect has been provided to investigate the phenomena from a more careful statistical viewpoint. For the evaluation of the mean difference of inclusion elements, 30 sample points were used in the analysis of variance (ANOVA) and Tukey’s tests. The ANOVA statistical test enables us to detect the mean difference of weight percentage of elements between WM, HAZ, and BM inclusions (or impurities). Tukey’s multiple comparison test allows us to determine the zones in which mean differences are statistically significant. The result of the ANOVA and Tukey tests are presented in table 7.

As can be seen from table 7, the mean values of weight percentages of O, S, Ca, and Al were statistically significant in the inclusions of WM, heat affected and BM zones (P.value<0.05), while the mean values of weight percentages of Mg, Si, Ti, Mn, and Fe did not differ significantly. According to Tukey’s test (table 7), it was found that the mean of the weight percentages of O in the HAZ inclusions differed significantly from the inclusions of the WM and BM zones. However, the mean of the weight percentage of Al in the inclusions of the WM and BM zones and also the percentage of S and Ca in the inclusions of the WM and HAZ differed significantly.

The 95% confidence interval of O, Al, S, and Ca have also been determined in the mentioned zones. According to the confidence intervals of table 7, it is possible to estimate the zones in welded API X65 in which there exists an inclusion. For example, if the weight percentages of O, Al, S, and Ca are in the range of 36.13–49.14, 2.57–10.97, 0.16–0.81, and 1.28–7.98, respectively, then the inclusion is probably located in the WM.

The values mentioned above can be regarded as guidelines for related industries to be used in order to minimize the formation of inclusions by changing welding parameters, according to the weight percentage of inclusion elements in different zones (i.e. WM, HAZ, and BM). On the other hand, the effects of these elements in altering the mechanical properties should not be ignored.

According to table 7 for elements that are statistically significant (O, Al, S, and Ca), the confidence interval in the weld is smaller than that in other regions. In fact, in the WM, the variations in the weight percentages of the constituent elements of the inclusions are smaller, improving the mechanical properties in the WM, including yield strength and tensile strength.
The weld toughness can be controlled by the finest details in the microstructure. Each of the alloying elements also affects the amount, shape, and type of regions and phases, which in turn will affect the toughness and tensile strength of the WM. In the steel under study in the current research, precise control of alloying elements has resulted in an increase in the tensile and yield strength of WM compared to the BM. As mentioned before, the different microstructure of the BM and the WM has caused the weld failure energy to be less than that of the BM \[29\]. In addition, by further examining the fracture surface of the BM and WM, it was revealed that the number and size of inclusions formed in the weld are more than those in the BM, which results in a decrease in fracture energy.

Future studies can focus on developing a regression equation between the percentage of inclusion elements and mechanical properties by means of thorough investigations. However, this was not the subject of this study and in the course of the present study, the mechanical properties of the base metal and the weld zone are assumed constant according to the available literature.

4. Conclusion

In the current study, the fracture surface point of the specimens containing spiral welded seam API X65 steel with Chevron and pressed notches have been investigated. For this purpose, specimens were fractured by DWTT, then SEM and EDS analyses were performed. Also, the weight percentages of inclusion elements were analyzed statistically. The results are summarized as follows:

- The thickness variation in the PN has been greater than that of the CN specimen. Using blunted notch increased specimen thickness and inverse fracture area. Although shear area percentages in both specimens were high, representing the excellent ductility of welded API X65.

- SEM images of different sub-zones from WM to HAZ and BM showed the inclusions in fracture surfaces. Where the hammer impacted the specimen, hence, high energy levels, WM microstructure, and inclusions caused the formation of micro-cracks due to stress concentration beneath the notch (unlike the shear fracture area). Micro-cracks formed in the inverse fracture of the PN specimen (unlike the CN specimen). The size of micro-cracks in the PN cleavage fracture area was greater and of higher density than those of micro-cracks in the inverse fracture area. The delamination interiors have a cleavage plane, in the CN specimen. Delamination and V-shaped marks are formed only in areas with Ferrite structure. One of the factors reducing the fracture energy of the WM compared to the BM is the lack of these marks.

- Through EDS analyses, the weight percentages of inclusion elements were obtained and compared with target values specified by API 5L standard. In most inclusions, Mn, Ti, and S percentages were higher than standard values. Also, some inclusions had great values of aluminum, silicon, and magnesium that were probably left during the welding procedure.

- The mean values of the weight percentages of O in the HAZ inclusions differed significantly from the inclusions of the WM and BM zones. Moreover, the mean values of the weight percentage of Al in the inclusions of the WM and BM zones and the S and Ca in the inclusions of the WM and HAZ differed significantly, according to statistical analysis (ANOVA and Tukey’s tests). Changes in the constituent elements of the WM inclusions are smaller compared to the BM and HAZ that would be one of the reasons for better mechanical properties of the WM compared to the BM. Weld microstructure greatly reduces the fracture energy due to the formation of Widmanstatten structure and due to stress concentration. Acicular ferrite in the BM increases the fracture energy by changing in the direction of the crack growth.

- The 95% confidence interval of the weight percentage of O, Al, S, and Ca in the inclusions (30 sample points) were determined. Related industries can minimize the formation of the inclusions by considering these confidence intervals, therefore, improving mechanical properties (especially toughness) of the welded zone where the inclusions are higher.

- Reducing the number of inclusions reduces the formation and growth of micro-cracks, which the studies of engineers and manufacturers have always sought to achieve.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Conflict of interest statement

The authors declare that they have no conflict of interest.

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