Original Article

Mechanical and microstructural behavior of C-Mn steel weld deposits with varying titanium contents

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A B S T R A C T

The effect of micro-alloying on the microstructure of C-Mn steel weld metals has been extensively studied in the past and the effect of titanium, in particular, has motivated several investigations, due to its essential contribution to the occurrence of acicular ferrite. However, there are cases where a substantial amount of acicular ferrite present in the last pass is not directly related to higher impact energy, since other parameters such as the proportion of the transformed phases by reheating, occurrence of micro-phases and non-metallic inclusions also influence the toughness of weld metal. Based on this scenario, the current work complements previous investigation and proposes an alternative methodology for evaluation of the behaviour of microstructure and impact toughness of C-Mn steel weld metals containing varying titanium contents, in the range of 6 and 255 ppm, based on comparison with the microstructural characteristics observed at the Charpy-V notch, where impact toughness was measured.

The weld metal microstructure was characterized at the top bead and at the mid-thickness by Optical (OM), Electron Microscopy (SEM) and electron backscattered diffraction (EBSD). Vickers microhardness tests were performed at the same positions where metallographic examination was done. Charpy-V impact tests employing test samples removed from the center line and at mid-thickness of the weld deposits were carried out in order to obtain test temperatures corresponding to impact energy of 100 joules.

The results evidenced that the increase of titanium content promoted a substantial amount of acicular ferrite and the refinement of microstructure at the top bead. At the Charpy-V notch location, the same behavior was observed, the difference being relative to the lower amount of acicular ferrite. Optimum impact toughness was attained for 28 ppm Ti, although the metallographic examination revealed that the higher amount of refined acicular ferrite presenting a lower effective grain size and increased frequency of high angle boundaries was observed for 255 ppm Ti. From these findings, a methodology involving OM, SEM and EBSD for an adequate characterization of the microstructure at the Charpy-V notch location is applied, in order to evaluate the influence of reheating, microstructure, effective...
grain size, frequency of high angle boundaries, presence of MA constituents and inclusions. The analysis revealed that the behavior of impact toughness is associated with the presence of the M-A constituents occurring at the Charpy-V notch for 255 ppm Ti.

The obtained results indicate the need for a more stringent procedure for an explanation of the behaviour of the impact properties of C-Mn steel weld metals, due to the role of the thermal cycle build-up of multi-layer weldments.

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1. Introduction

It is well known that acicular ferrite nucleated at intragranular inclusions results in a fine-grained interlocking arrangement of ferritic plates providing high tensile strength and excellent impact toughness [1–5], being, therefore, a desirable microstructural constituent in C-Mn steel weld metals.

In this regard, several works have been conducted in the last decades [1–25] in order to identify the potential of the alloying elements on the formation of acicular ferrite. From the findings, it was observed that the micro-alloying also exert an important effect. In particular, the influence of titanium deserves special attention due to its effective influence on the formation of acicular ferrite [2,3,7–18,22–24]. Evans research [2,13,8–18] showed a significant impact of Ti at around 20–30 ppm Ti, followed by a slight decline in toughness at intermediate Ti levels, at about 100–200 ppm and higher Ti levels, the impact toughness increased again.

Although acicular ferrite exerts a considerable effect on the impact toughness of C-Mn weld metals containing titanium due to its fine grain size and interlocking structure with high angle boundaries that can act as an obstacle for crack propagation, thereby improving the toughness [1,2,6,25–28], multi-pass weldments present a complex behaviour and some other parameters must be considered when analysing the mechanical properties [29]. Besides changes in the amount of acicular ferrite, the influence of reheating, MA constituents and inclusions are important, because they also exert a considerable effect [29–36], which can be beneficial or deleterious depending on the chemical composition and microstructure of the studied material [36]. However, it is important to emphasize that a weld metal with high amount of refined acicular ferrite can control more effectively some of the other parameters such as inclusions and MA constituents. Indeed, acicular ferrite refining the microstructure contributes to the improvement on the M–A size and distribution, which determines the level of M–A-induced brittleness [25]. A higher titanium content induces a smaller and more dispersive M–A constituents and weld metal with higher impact toughness [25]. In regard to the presence of inclusions, increasing Ti content contributes to the adequate inclusion size distribution, density and chemistry [25,26], which decisively assist the formation of acicular ferrite [26].

Although an increased fraction of reheated zone is usually associated with improvements of mechanical properties [35], it is still difficult to foresee these results [33,37] since it is dependent of a interaction between chemical composition and welding procedure. The limited number of studies, according to the author’s knowledge, does not allow to reach a conclusive mechanism concerning the microstructural changes associated with the presence of Ti and the impact toughness of the reheated region of the multi-pass weld metal deposits.

As already presented in a previous work [7], the discussion regarding the relationship between acicular ferrite and impact toughness is very complex and it is still open at this time. The association of impact toughness with acicular ferrite considering the top bead is not a reliable procedure, even for a single pass weld deposits [36,38,39].

Recent research [35] reported that toughness strongly depends on the multi-pass weld microstructure of the investigated material, which contains several sources of inhomogeneity such as inter-dendritic segregation and the effective grain size [30,35] can also be a significant factor contributing to explain the strong deviations of local impact toughness values.

This work complements previous investigations conducted by Evans studying the influence of titanium, in the range of 6 and 255 ppm, on the microstructure and mechanical properties of C-Mn weld deposits [13], where the higher amount of acicular ferrite observed at the top bead was not related to the optimum impact toughness. Based on these evidences, an alternative methodology for evaluation of the microstructure of C-Mn steel weld metals is proposed, incorporating the effect of reheating, MA constituents and inclusions, in order to allow a better understanding of the behaviour of the impact toughness.

2. Experimental procedure

2.1. Material

Ten samples presenting basic composition 0.07C-1.50Mn-0.30Si-0.012V-0.002Nb with different amounts of titanium (Table 1), were received for metallographic examination.

These samples were prepared according to the International Standard ISO 2560 [40], with three beads per layer, leading to a total of 24 weld runs.

All weld deposits were obtained by manual metal arc welding, in the flat position, using 200 °C preheat. Direct current (positive electrode) was employed, the current being 170 A, the voltage 21 V and the nominal heat input of 1.5 kJ/mm for each welding pass. The efficiency factor used was 0.7. For these conditions, the cooling time between 800 and 500 °C calculated according to EN 1011-2 Annex D [41] was 6 s, which is similar to the result obtained experimentally (7 s) in other work using the same welding procedure [42].
More informations about experimental conditions are described in the previous work [13].

Fig. 1 shows macrographs of two selected specimens.

2.2. Mechanical tests

Database containing Charpy-V notch results obtained in previous work [13] was received in order to obtain full transition curves. As described in the original work [13], approximately 35 standard Charpy-V test specimens (10 × 10 × 55 mm) were removed from the center line of weld deposits at mid-thickness (Fig. 2). Tests were conducted at temperatures varying from −90 to −20 °C. The test temperatures corresponding to impact energy of 100 joules were plotted with varying titanium content. The average and standard deviation were obtained by using Origin software.

Vickers microhardness tests (500 gf load) were performed on samples obtained from the centerline of weld deposits, as showed in Fig. 3. The measurements were done in three parallel lines separated by 0.25 mm from top surface to the root of the weld metals and a spacing of 1.0 mm, as showed in Fig. 3. The behavior of microhardness with varying titanium content was plotted. The average and standard deviation were obtained by using Origin software.

2.3. Metallographic examination

Metallographic examination of the weld metals was performed in specimens removed from the center line at the positions showed in Fig. 4, by optical microscopy (OM), scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) and electron backscattered diffraction (EBSD).

Quantitative measurements of the percentage of the columnar region, at the position relative to the Charpy-V notch, was done directly on the OM screen, to verify the
influence of a different number of passes on the toughness. Microstructural constituents were evaluated using the counting technique point to point, with a 10 × 10 grid on the OM screen. A minimum of 1,000 points were considered for each weld metal, at a nominal magnification of 1,000 ×. The samples were prepared using the conventional procedure of grinding and polishing, followed by nital 2% etching.

Besides, the occurrence of M-A constituent was also observed by OM using Le Pera reagent and by SEM using a two-stage electrolytic etching method [43].

Quantitative analysis of micro-phases was performed using a counting technique point to point, with a 10 × 10 grid on the SEM screen. 1000 points, at least, were considered for each weld metal, at a nominal magnification of 2,500 ×. The images were analyzed using Image J software, followed by calculation of the area fraction of the micro-phases. An analysis was conducted with both etchants, nital 2% and selective, to separate M-A constituents as described below.

The two-stage selective method used to separate M-A constituents can be summarized as follows [43]:

Stage 1—The electrolyte for the first etch was 5 g ethylene diamine tetra acetic acid (EDTA), 0.5 g sodium fluoride (NaF) and 100 ml distilled water. Etching condition was 3V, 3s. In this case, ferrite is etched, and M-A constituents and carbides remain unetched.

Stage 2—The electrolyte for the second etch was 5 g picric acid, 25 g sodium hydroxide (NaOH) and 100 ml distilled water. Etching condition was 6V, 60s. This second etching resulted in carbides being preferentially etched.

After the application of a sequence presented above, can be easily identified by SEM [28].

Unetched samples were examined by SEM with EDS, so as to obtain the distribution, size and composition of inclusions of some selected compositions.

Electron Back Scattering Diffraction (EBSD) maps were obtained by SEM operating at 20 kV and step size of 0.3 μm. High-angle grain boundaries (HAGBs, misorientation >15°) [28] were distinguished by the scalar misorientation between the adjacent pixels. Effective grain size (EGS) of the grains with HAGBs was measured using the line-intercept method.

For EBSD characterization, 0.25 μm colloidal silica suspension was also used for automatic polishing without further etching.

3. Results

3.1. Mechanical tests

Fig. 5 shows the behaviour of microhardness, where a complex trend is indicated with a peak of 16 ppm Ti and a continuous increase above 80 ppm Ti.

Fig. 6 shows the transitions curves for different titanium contents. From these results, the Charpy-V test temperature corresponding to absorbed energy of 100 were obtained and showed in Fig. 7, where it is observed that the best results being obtained at around 28 ppm Ti.
3.2. Metalographic examination

The relative proportions of the columnar and reheated regions corresponding to the notch location in Charpy-V specimens (Fig. 8), revealed the predominance (50%) of the fine grained reheated region (FGRR) in all weld deposits.

Figs. 9 and 10 show the potent effect of Ti for the increase of acicular ferrite (Fig. 9) and refinement of the microstructure (Fig. 10). This refinement makes the clarification of microstructural constituents more difficult, mainly at the Charpy-V notch position, where the influence of reheating is more intense. Consequently, higher magnification than that usually applied and recommended by IIW [44] is used to allow a more precise characterization.

EBSD analysis confirmed changes of morphology and effective grain size (EGS) of the acicular ferrite (Fig. 11). Also, Fig. 12 shows the grain boundary misorientation profile, where as expected for acicular ferrite [28,45-47], the effect of titanium on increasing high angle boundaries (HAB) is seen.

Optical examination of the coarse grain reheated region (CGRR) revealed the same tendency observed in the columnar region. In the fine grain reheated region (FGR), the microstructure was composed primarily of polygonal ferrite (Fig. 13). EBSD analysis confirmed the predominance of polygonal ferrite and, additionally, showed the refinement of microstructure for 255 ppm Ti (Fig. 14). It is also noted that, although presenting a larger EGS, the presence of polygonal ferrite in the fine grain reheated region showed a much higher fraction of HAB (Fig. 15).

Fig. 16 shows the occurrence of micro-phases at different positions of weld deposits. Although Le Pera reagent has been extensively used for evaluation of M-A constituents in heat affected zone (HAZ) [48-55], it cannot provide the same useful result when analyzing weld metals, due to the smaller size of the M-A in this region of the welded joint.
Fig. 10 – Influence of Ti on the microstructure at the columnar region of weld metals for two selected samples (OM). Etching: nital 2%.

Fig. 11 – Characteristics of the microstructure at the columnar region for top bead and mid-thickness for two selected samples (EBSD). Nominal magnification: 1,500x.

Consequently, to distinguish M-A constituents from other micro-phases, an alternative electrolytic etching technique, proposed by Ikawa et al. [43], was applied and the samples were observed by the SEM at a higher magnification (Fig. 17). The preferential occurrence of massive M-A constituent was observed in the top bead and a predominance of carbides at the mid-thickness as consequence of reheating (Fig. 16). Quantitative analysis indicated a low volume fraction (<5%) of refined M-A constituents even in the top bead (Fig. 18).
Fig. 12 – Grain boundary misorientation distribution of the columnar region of weld metal at mid-thickness for two selected samples.

Fig. 19 shows the inclusions observed in some selected samples, where it is noted that smaller inclusions are observed for 255 ppm Ti (average size 28 ppm Ti – 0.474; average size 255 ppm Ti – 0.460). Inclusions considered as nucleants for acicular ferrite contain titanium and have size in the range of 0.4 μm to 1 μm, which is adequate for this purpose [9,26,56].

4. Discussion

This work access weld metals examined previously [13], under a different approach considering the microstructural distribution at the Charpy notch path to evaluate the correlation between microstructure and toughness incorporating the effect of multiple passes, usually applied when working with the SMAW process on thick plate compared with the top bead.

Table 1 shows the chemical composition of the weld metals where it is noted that oxygen content decreased progressively due to the deoxidation potential of titanium, as expected.

Fig. 13 – Influence of Ti on the microstructure of the reheated regions of weld metals at the top bead (OM). Etching: nital 2%.

Fig. 14 – Influence of Ti on the characteristics of FGRR of weld deposits (EBSD). Nominal magnification: 1,500x.
As stated by Koseki and Thewlis [6], in low aluminum weld metals, oxygen is available for titanium oxide formation, the titanium deoxidation prevailing over that of silicon and manganese even though the amount of titanium addition is small. As will be discussed later, it contributed to the formation of inclusions which are crucial for nucleation of acicular ferrite [6,24,25,56,57,59].

According to Loder et al. [22], the addition of titanium and manganese is essential to the production of acicular ferrite. Although Beidokhti et al. [60] stated that increasing titanium content increased the recovery of manganese and hardenability of the weld metal, which encourages grain boundary nucleation of bainite with greater frequency than intragranular nucleation of acicular ferrite, it is known that an appropriate balance between Ti and Mn favors a microstructure with high amount of acicular ferrite. In this respect, Evans studied the influence of titanium in weld metals containing Mn [14] and the results showed that, in the range of 1.35–1.80% Mn, the addition of around 200–250 ppm Ti does not alter significantly the mechanical properties. Recently, other investigation [9] confirmed that Mn has an effect on the suppressing the transformation temperature, but the temperature did not change dramatically with varying Ti content.

The effect of niobium and vanadium was studied by Evans [20,21] and the results showed that no significant effect was observed in the range observed in the present work. Particularly, in regard to the vanadium, Evans concluded that in the as-welded condition the notch toughness was marginally improved at intermediate vanadium contents when the manganese level was 1% and above [21]. In addition, the response of the two elements was found to be essentially insensitive to variations in manganese content [14].

Considering the above discussed, the variations observed in the chemical composition do not compromise the analysis performed in the present work, since no significant changes on the properties are expected.

Fig. 9 shows the effect of Ti on the microstructure of the deposits, where it is confirmed that similar results for the top bead where obtained as those observed earlier [13]. It is important to observe the lower amount of acicular ferrite at the Charpy-V notch, in agreement with other works [61,62], where the reduction of acicular ferrite in the reheated region is due to the smaller prior austenite equiaxed grains in comparison to larger columnar grains observed at the columnar region [63].

Fig. 20 shows that the potential effect of Ti on impact toughness reported in the present work is in agreement with other works studying the same effect of micro-alloying on the Charpy-V impact toughness of C-Mn weld deposits [7,64–66].

![Graph](image1.png)

Fig. 15 – Grain boundary misorientation distribution of the fine grain reheated region of weld metals containing 28 and 255 ppm Ti.

![Images](image2.png)

Fig. 16 – Influence of Ti on the micro-phases at the columnar region of weld metals. Etching: nital 2%.
Although the impact toughness is dependent on several factors, it is recognized the potent effect of acicular ferrite due to its fine-grained interlocking structure to prevent brittle crack propagation by cleavage. The high misorientation angle boundaries and high density of dislocations of acicular ferrite are effective for high strength and high toughness [67]. However, different values of toughness can be observed for the same amount of acicular ferrite, depending on the Ti content, as shown in Fig. 21. These results indicate that other factors are contributing to the impact toughness.

As observed before, although a superior amount of acicular ferrite is desirable for good impact toughness, it does not explain the results shown in Fig. 21. It confirms that the association of microstructure and impact toughness based on the analysis at Charpy-V notch position is more appropriate. In this respect, it is imperative to emphasize that specimens for impact Charpy-V tests are composed of several regions subjected to reheating (Fig. 8) and, consequently, many aspects must be taken into account when considering mechanical properties, mainly microstructure, grain size,
reheating, micro-phases and inclusions. First of all, the precise characterization of the microstructure is critical for discussion of the impact results.

In this respect, Fig. 10 shows that, even with higher magnification, the precise interpretation of refined microstructures can be a very complex task, leading to a high variability of the results. In order to resolve the microstructure appropriately, the EBSD technique was applied as a complementary tool (Fig. 11). Recently, this technique has been considered as an interesting alternative [28,45,68,69] to overcome the optical microscopy deficiency. The high definition provided by the EBSD technique, especially regarding the grain boundaries, is beneficial to separate acicular ferrite and bainite (ferrite with the second phase). In regard to the evaluation of MA constituents and inclusions, SEM analysis (Figs. 16 and 19) is more suitable for this task. Therefore, it is believed that the combination of the OM, SEM and EBSD techniques provides the best methodology to study C-Mn steel weld metals when a refined microstructure is present.

When accurate characterization of the microstructure is obtained, the evaluation of the impact toughness can be done, based on the following evidence:

a) Reheating—This parameter is not so representative in this work since the same proportion of recrystallization was obtained for all weld deposits;

b) Microstructure—Higher amount of AF was observed for 255 ppm (Fig. 9). EBSD results confirm this tendency showing that more refined microstructure (Fig. 11) presents the higher frequency of high angle boundaries (HAB), which can effectively force the propagation of cleavage cracks to deviate or stop [26]. The same behavior was noted for the refined grain reheated region (Figs. 14 and 15), where polygonal ferrite is predominant (Fig. 14);

c) No metallic Inclusions—It is known that non-metallic inclusions can exert two opposing effects on impact toughness. One of them is that the inclusions act as initiation sites for both ductile and cleavage fracture. The other is that they can assist in the formation of acicular ferrite. It was observed that increasing Ti content contributed to the inclusions adequate to support the formation of refined acicular ferrite, in agreement with other works [3,6,22,24,26,70].

d) MA constituents—Fig. 18 shows the behavior of micro-phases, where it is noted that increasing titanium content increased the volume fraction of micro-phases and MA constituents at the top bead. However, it is essential to mention that the same behavior was not observed at the Charpy-V notch, where the decomposition of the MA constituent is expected [7] due to the multiple welding passes (Fig. 16).

Based on the above discussed, the results indicate that 255 ppm Ti should induce a higher impact toughness. Indeed, the higher amount of acicular ferrite, more refined microstructure (Fig. 11) having a higher frequency of HAB (Fig. 12) and smaller Ti-containing inclusions (Fig. 19) due to the increased number of nucleation sites for acicular ferrite [71], are representative of superior impact toughness [25,28,71]. However, as showed in Figs. 6 and 7, the best results were obtained for lower Ti contents, notably, 28 ppm Ti, as also verified in other investigations [2,13,14,22].

In this respect, an additional evaluation by SEM revealed the presence of MA constituents along the Charpy-V notch for 255 ppm Ti (Fig. 22).

Although its effect is not entirely understood [72,73], it is reported that the occurrence of M-A constituent can be critical since it can deteriorate the impact toughness, even in weld metals containing an increased proportion of acicular ferrite [36,74]. In general, the critical effect is observed when MA constituents are decorating the previous austenite grain size [25,73,75–77], but even smaller and more dispersive MA constituents can exert a deleterious effect depending on their size, morphology, amount and distribution.

Considering the results discussed in the present work, the difference observed on impact toughness is due to the occurrence of M-A constituents at the Charpy-V notch location (Fig. 22).
These findings reinforce the effect of M-A constituent on the impact toughness and confirm that the mid-thickness cannot be considered as representative of the entire test piece and a more embracing analysis must be conducted for each weld metal.

The most adequate procedure must consider the metallographic examination by OM, SEM and EBSD, involving the entire position where mechanical properties are being measured.

5. Conclusions

The main conclusions are:

a) Ti promotes a substantial increase of the amount of acicular ferrite, but it is not continuous;
b) The best impact toughness was observed at 28 ppm Ti;
c) Impact toughness of C-Mn-Ti weld metals are related to the balance between acicular ferrite and MA constituents present at the Charpy-V notch location;
d) Micro-phases present along the Charpy-V notch are crucial for impact toughness of weld metals and;
e) The association of OM, SEM and EBSD techniques is an interesting methodology for metallographic examination of refined microstructures of weld metals;

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