Influence of Zirconium on Microstructure and Toughness of Low-Alloy Steel Weld Metals

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The influence of zirconium on microstructure and toughness of low-alloy steel weld metal was studied. Weld metals with different zirconium contents were obtained adding iron-zirconium alloy in the welding flux formulation. Weld metal chemical composition proved that zirconium was able to be transferred from the flux to the weld metal. The addition of zirconium refined the weld metal microstructure, increasing the acicular ferrite content. Weld metal toughness, determined by means of impact Charpy-V tests, showed that the zirconium addition is beneficial up to a content of 0.005 wt.%. Above this level, zirconium was not able to produce further microstructure refinement, although the toughness was reduced, possibly due to the formation of microconstituent such as the martensite-austenite constituent (M-A), which is considered to be deleterious to the weld metal toughness.

Keywords	low-alloy weld metal, martensite-austenite constitu-
	ent (M-A), submerged arc welding, toughness, zirco-
	nium

1. Introduction

Generally, weld metal of steel with a microstructure formed predominantly by acicular ferrite (AF) has high mechanical properties for both notch toughness and strength (Ref 1-5). This is attributed to the very fine grain size of the acicular ferrite (1-3 μ m) as well as its high boundary angle and high dislocation density, reducing crack propagation. However, the predominance of acicular ferrite in the microstructure is not the only factor determining the high toughness of the weld metals. Investigations (Ref 3-6) have shown that microphases such as the martensite-austenite constituent (M-A), can influence strongly the toughness of weld metals for carbon/manganese low- and medium-alloy steels.

Zirconium is extensively used for steel refinement because of its strong affinity to oxygen and sulfur, which reduces the volumetric fraction of nonmetallic inclusion and improves both toughness and cold work properties during steel manufacture. It was shown the simultaneous addition of zirconium and bo-

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ron modify weld metal microstructure similarly to when titanium and boron are added to the weld metal (Ref 7).

API 5L-X70 steel combines high resistance and toughness properties, and is extensively used for gas and oil pipelines. Welding this steel needs the use of consumables capable to confer to the weld metal similar mechanical properties to those of the base metal. In this work, the aim was to investigate the influence of zirconium addition on weld metal impact toughness when welding API 5L-X70 steel grade by submerged arc welding process.

2. Materials and Experimental Procedures

Submerged-arc with two pass double-sided welding technique was used on 11.5 mm thick API 5L-X70 steel. The wire was EA-3, according to AWS A5.23. Base metal and wire chemical composition are given in Table 1.

A semibasic flux (basicity index, ~1.2) formulation was used, the composition of which is shown in Table 2. Flux was agglomerated with neutral sodium silicate and baked at 750 °C for 1 h. Prior to agglomeration, a powder alloy of ironzirconium (45% of zirconium) was added to the flux compounds. Fluxes were identified according to the iron-zirconium amount (wt.%) added during flux manufacturing: ZR00 (no iron-zirconium addition), ZR03 (0.3% of iron-zirconium addition), ZR06 (0.6% iron-zirconium addition), ZR09 (0.9% ironzirconium addition), and ZR12 (1.2% iron-zirconium addition). Data for the welding procedure are shown in Table 3.

Quantitative analysis of microstructure constituents formed in the second weld bead was performed using optical microscopy with $500 \times$ magnification in 30 different regions in the

Table 1 Chemical composition (wt.%) for API 5L-X70 steel and EA-3 wire

 С	Mn	Si	Р	S	Cu	Ni	V	Ti	Nb	Мо
0.10	1.29	0.18	0.018	0.007	0.017	0.014	0.049	0.013	0.043	(a)
0.10	1.80	0.15	0.018	0.009	0.15	(a)	(a)	(a)	(a)	0.50

Table 2 Chemical composition of the semi basic flux used (wt.%)

Al ₂ O ₃	MgO	MnO	CaF ₂	SiO ₂	Fe + 45% Si
31	20	10	20	15	4

Table 3Welding procedure for double-sided submergedarc welding

Welding parameter	1st bead	2nd bead	
Welding current	600 A, CC+	700 A, CC+	
Arc voltage	32 V	32 V	
Travel speed	35 cm/min	32 cm/min	
Heat input	3.29 kJ/mm	4.20 kJ/mm	
Wire diameter	3.18 mm	3.18 mm	
Stickout	25.4 mm	25.4 mm	
Pre-heating	25 °C	120 °C max, T	

cross section of the weld metals, perpendicular to the welding direction. For each region a reticulate of 500 points was used. This gave a total of 1500 points analyzed. This procedure follows that described in IIW/IIS Doc. IX-1533-88 (Ref 6).

Weld metal toughness was measured by means of reduced impact Charpy-V specimens $(55 \times 10 \times 6.7 \text{ mm})$ positioned at the center of the second bead and 1.6 mm from the plate. Three specimens were tested at -20, 0, and +20 °C.

For samples ZR00, ZR06, and ZR12, the semiquantitative chemical composition of inclusions was determined by means of energy dispersive spectroscopy (EDS) analysis coupled in a scanning electronic microscope. Five measurements per sample were carried out.

3. Results and Discussion

Welded joint from submerged arc weld has a complex microstructure. From macrostructure analysis different zones can be observed, as shown in Fig. 1. In this scheme it can be seen the base metal heat-affected zone (HAZ) provoked by the local heating above the austenitization temperature. Figure 1 also shows the weld metal coarse grained and fine-grained reheated zones (RHZ-CG and RHZ-FG) formed as a consequence of the heat during the deposition of the second weld bead and regions of columnar grains. The first bead columnar region suffered a type of stress relieve heat treatment, usually considered beneficial to toughness. The as-welded second bead columnar region is considered to offer the lowest toughness values (Ref 1), and will be considered in the present work.

Table 4 shows the weld metals chemical composition (in wt.%). A progressive increase of zirconium content can be observed, and it became evident that the addition of ironzirconium alloy powder to the flux was an effective method to add zirconium to the weld metal. All other elements in the weld metals were considered to be constant, which means that possible changes in weld metal microstructure and toughness will be attributed to the zirconium addition.

Figure 2 shows a typical weld metal microstructure of the columnar region of the second bead. The microstructure constituents were classified according to IIW/IIS Doc. IX-1533-88 (Ref 6) after etching with Nital 2% as acicular ferrite (AF), polygonal ferrite along grain boundaries [PF(G)], polygonal

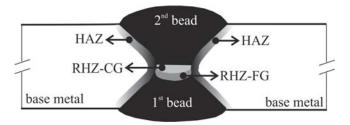


Fig. 1 Schematic representation of a double-sided weld joint obtained with the submerged arc welding processes

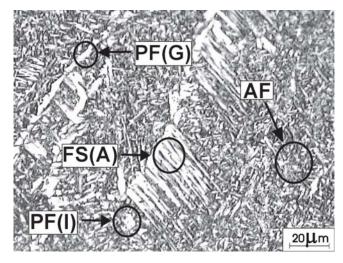


Fig. 2 Typical weld metal microstructure for the second bead (sample ZR06)

Table 4Weld metal chemical composition (wt.%)

Weld metal	С	Mn	Si	Р	S	Nb	Мо	N	Zr	0
ZR00	0.07	1.62	0.09	0.023	0.007	0.025	0.12	0.007	0.000	0.0444
ZR03	0.09	1.62	0.08	0.023	0.006	0.027	0.09	0.006	0.002	0.0558
ZR06	0.08	1.46	0.10	0.025	0.008	0.017	0.16	0.007	0.005	0.0525
ZR09	0.07	1.35	0.08	0.018	0.008	0.019	0.15	0.011	0.008	0.0526
ZR12	0.07	1.63	0.13	0.018	0.008	0.019	0.14	0.007	0.011	0.0495

ferrite inside the grain [PF(I)], and ferrite with aligned second phase [FS(A)].

Table 5 shows the zirconium addition increases the amount of weld metal acicular ferrite. For example, acicular ferrite increased from 68% (sample ZR00, without zirconium addition) to 83% (sample ZR06, with 0.0005% of zirconium). Further addition of zirconium (samples ZR09 and ZR12) could not increase weld metal acicular ferrite content.

Table 6 shows the semiquantitative chemical analysis obtained by EDS technique at weld metal inclusions. The presence of considerable high zirconium content at the inclusions compared with the zirconium content of the weld metal (Table 4) is evidence that zirconium lies preferably at inclusions than in solid solution. Considering that zirconium is much more reactive with oxygen than iron (Ref 8), and the relative high contents of weld metal oxygen obtained (Table 4), it is supposed that zirconium is present into the weld metal as ZrO_2 . This is an indication that zirconium is a strong nonmetallic inclusion former with high potential to nucleate acicular ferrite.

Table 5Weld metal quantitative optical metallographyresults

Constituents formed in the columnar region of the second weld bead

Weld		Consti	tuent, %	
metal	AF	PF(G)	PF(I)	FS(A)
ZR00	68	11	18	3
ZR03	70	9	19	2
ZR06	83	4	12	1
ZR09	78	7	15	0
ZR12	78	4	18	0

Table 6Semiquantitative chemical analysis (wt.%)obtained by EDS at weld metal inclusions

Sample	Al	Ti	Mn	Si	Zr
ZR00	3.8	0.4	28.3	11.0	
ZR06	6.6	0.2	25.0	10.8	1.9
ZR12	6.7	0.4	23.5	11.2	3.5

The role of nonmetallic inclusions in weld metal in the formation of acicular ferrite is reported in the literature (Ref 8-10).

Figure 3 shows the results of impact Charpy-V energy for the three tested temperatures (-20, 0, and 20 °C). It can be observed that the addition of 0.005% of zirconium (sample ZR06) promoted a significant increase in the values of Charpy-V energy. For higher zirconium content (samples ZR09 and ZR12) the Charpy-V energy dropped to almost the same level as the sample without zirconium (ZR00). The following explanation is suggested: samples with low or without zirconium content (samples ZR00 and ZR03) exhibited a low fraction of acicular ferrite. For 0.005% of zirconium, the highest fraction of acicular ferrite was obtained, the optimum toughness was obtained. For zirconium above 0.005%, despite a high amount of acicular ferrite, other factor may have contributed to the fall of weld metal toughness, probably the formation of the constituent martensite-austenite (M-A), as mentioned by others investigations when strong reactive compounds are added to the weld pool (Ref 4, 7, 10, 11).

4. Conclusions

From the results presented in this work, it is possible to draw the following conclusions:

- Iron-zirconium powder alloy was effective in transferring zirconium from the flux to the weld metal.
- Zirconium has shown to be effective to promote the formation of acicular ferrite.

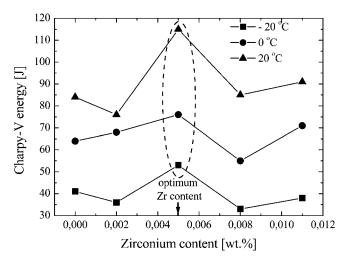


Fig. 3 Charpy-V energy of the weld metals at different temperatures $(-20, 0, +20 \ ^{\circ}\text{C})$ as a function of zirconium content

• The higher Charpy-V energy was obtained with 0.005% of zirconium in weld metals.

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