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**Manufactura, Soldadura y Materiales**

**Microstructure and pitting corrosion in GMAW Weld of 2304 lean duplex steel under heat input effect, considering the formation of different HAZ sub-regions**

***Microerstructura y corrosión por picadura en soldadura GMAW de acero lean duplex 2304 bajo el efecto del calor de entrada, considerando la formación de diferentes subregiones de la ZAC***

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**Abstract:**

**Problem:** The welding of duplex steels, depending on the input energy of the process, gives it structural changes that can lead to sensitization to pitting corrosion.

**Objetive (s):** To stablish the effect of the heat input (1,5 kJ/mm and 2,5 kJ/mm) on the microstructure and pitting corrosion in GMAW of 2304 lean duplex steel, considering the formation of sub-regions in the HAZ, associated with the shape of the weld bead.

**Methodology:** The ferrite and austenite fractions were evaluated for the three HAZ sub-regions, by EDS the phase’s composition was determined. Pitting corrosion was evaluated by immersion test in ferric chloride, and the Pitting Resistance Equivalent Number (PREN) was determined.

**Results and discussion:** The results obtained reflected that three subregions are formed in the HAZ with differences in their width and in the ferrite’s grain size. Ferrite, grain boundary austenite, Widmanstätten austenite, and intragranular austenite were observed in the HAZ. The phases fractions in HAZ did not suffer significant changes with the heat input, tending to increase slightly the austenite as the heat input increases.

**Conclusions:** Pits appear in the HAZ, starting in the widest sub-region. Pitting corrosion increased with the augment of the heat input, as the HAZ area in the sample grows.

***Resumen:***

***Problemática:*** *La soldadura de aceros dúplex, en función de la energía de entrada del proceso, le imprime cambios estructurales que pueden conducir a la sensibilización a la corrosión por picadura.*

***Objetivo (s):*** *Establecer el efecto del aporte de calor (1,5 kJ / mm y 2,5 kJ / mm) sobre la microestructura y corrosión por picaduras en GMAW de acero lean dúplex 2304, considerando la formación de subregiones en la ZAT, asociada con la forma del cordón de soldadura.*

***Metodología:*** *El diagrama de equilibrio de fases se obtuvo a partir de la composición del metal base. Se evaluaron las fracciones de ferrita y austenita para las tres subregiones HAZ. Mediante EDS se determinó la composición de la fase. La corrosión por picaduras se evaluó mediante una prueba de inmersión en cloruro férrico y se determinó el Número equivalente de resistencia a las picaduras (PREN).*

***Resultados y discusión:*** *Los resultados obtenidos reflejan que se forman tres subregiones en la ZAT con diferencias en su ancho y en el tamaño de grano de la ferrita. En la ZAT se observaron ferrita, austenita de límite de grano, austenita de Widmanstätten y austenita intragranular. Las fracciones de fases en ZAT no sufrieron cambios significativos con el aporte de calor, tendiendo a aumentar ligeramente la austenita a medida que aumenta el aporte de calor.*

***Conclusiones:*** *Los poros aparecen en la ZAT, comenzando en la subregión más amplia. La corrosión por picaduras aumentó con el aumento de la entrada de calor, a medida que aumenta el área de ZAT en la muestra.*

**Keywords:** Lean duplex steel. Heat Affected Zone. Microstructure. Pitting corrosion.

***Palabras Clave:*** *Aceros lean dúplex. Zona Afectada por el Calor. Microestructura. Corrosión por picadura.*

**1. Introduction**

Duplex stainless steels present their best properties for a ferrite/austenite ratio of 50:50, which is achieved by the correct combination of chemical composition and thermal history. Along with the ferrite/austenite rate, other phases that affect the steel’s properties may appear in the microstructure, which also depends on the chemical composition of the steel and the holding time at certain temperature ranges [1, 2]. This indicates that the correct understanding of the performance of these steels is a complex problem, since in the manufacture of the mechanical structure, as well as in the service, there are factors that can diminish their performance.

When in service, one of the most frequent degradation of stainless steels is related to pitting corrosion; which relates to the composition, thermal history, and environment. This causes many investigations to focus their studies on this aspect [2, 3].

A high volume of stainless steels production is used in the manufacture of components by welding, which subject the material to severe thermal cycles of heating and cooling, altering the microstructure and properties and, consequently, the in-service performance.

Due to the diversity of factors that influence the thermal history in the welding process (physical properties of the steel, thickness of the plate, heat input, thermal efficiency of the process, etc.), there is no general rule to predict the optimal conditions to obtain a weld which guarantees the best in-service performance. This explains the high interest shown in the literature about the welding of these steels [1, 3-8].

The Heat Affected Zone (HAZ) attracts the greatest interest within the welding joint of duplex steels, since it is subjected to the most severe thermal cycles, appearing different types of austenite and altering the ratio of this phase with the ferrite. Multiple works about thermal cycles simulation by physical tests Gleeble [1, 9], numerical methods [7, 8, 10], and studies of HAZ on real welds are reported [4-6].

All the reported studies consider the formation of a single HAZ for a given heat input. However, when a high argon mixture is used in the GMAW, the weld bead acquires a shape with inflection of the fusion line [11], leading to the formation of different sub-regions in the HAZ. The formation of these sub-regions in the HAZ in lean duplex steels, associated with the shape of the weld bead, has not been considered in previous studies.

In this context, the objective of the present work is to study the effect of the heat input (1,5 kJ/mm and 2,5 kJ/mm) on the microstructure and pitting corrosion in GMAW of 2304 lean duplex steel with a high argon mixture, considering the formation of sub-regions in the HAZ, associated with the shape of the weld bead.

**2. Methodology**

**2.1 Obtaining weld samples**

As base material was used 2304 lean duplex stainless steel, whose compositions is presented in Table 1. The table also shows the chemical composition of Sandvik wire 22.8.3.L (equivalent to AWS ER2209), used as consumable.

Table 1. Chemical composition of base metal and consumable electrode, (mass percent).

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Material | C | Si | Mn | Cr | Ni | Mo | N | P | S |
| Lean duplex 2304 | 0.027 | 0.34 | 1.38 | 23.09 | 4.96 | 0.18 | 0.12 | 0.026 | 0.0042 |
| Sandvik 22.8.3.L | 0.02 | 0.50 | 1.60 | 23.00 | 9.00 | 3.20 | 0.16 | ≤0.02 | ≤0.025 |

To obtain samples, the beads on plate welds were made by GMAW on 300 mm x 150 mm x 9.5 mm plates of 2304 lean duplex steels. DC current with inverted polarity (DC (+)) was used, with a mixture of 98 % Ar and 2 % CO2, with a flow of 18 L/min and a 1.2 mm diameter wire. The contact tip to work distance was 19 mm. The welds were made with two heat input levels (1.5 kJ/mm and 2.5 kJ/mm). The welding current and arc voltage values were kept approximately constant (180 A and 24 V). To define the welding speed value, corresponding to each predetermined energy value, a thermal process efficiency was considered equal to 85 % (Q=ISUa/VS; IS - welding current; Ua - arc voltage; VS - welding speed and  - thermal efficiency of the process) [11, 12]. The maximum speed, 8.29 m/h (2.3 mm/s), was obtained for the minimum energy and the minimum speed 4.98 m/h (1.38 mm/s) for the maximum energy.

**2.2 Metallographic observation and pitting corrosion test**

The weld beads on plates were cut transversally to prepare the metallographic samples. Sample preparation was performed, according to ASTM E3 [13], using diamond paste for polishing. The chemical etching was performed with modified Behara (35 ml of H2O, 5 ml of HCl and 0,18 g of K2S2O5) for about 30 s. Electrolytic etching was also performed with 20 % NaOH solution, with a voltage of 5 V, for approximately 35 s.

The austenite and ferrite fraction percentage in the HAZ and in the base metal was determined by image processing with the image J software. Average values were obtained from 20 optical microscopy images of samples, etched with modified Behara. Phases chemical compositions were determined by EDS.

The pitting corrosion tests were carried out, in accordance with ASTM G48 standard, method A [4, 14]. The weld beads on plates were cut transversally to obtain samples for corrosion test (6 specimens for each heat input level). All surfaces of the samples were prepared with 1200 abrasive paper.

The tests were carried out by immersion in ferric chloride (100 g FeCl3·6H2O in 900 mlH2O) at 23 oC for two exposure times, 24 h and 72 h (3 samples of each heat input level were tested for each exposure time). For the test, the specimens were cleaned, weighed, placed in a glass cradle and immersed in the test solution. The test beaker was covered with a watch glass. After the test, the specimens were cleaned and weighted. The total surface area of each sample was determined by geometric calculations. The mass loss corrosion rate was determined as the ratio of the mass loss and the samples surface area.

**3. Results and Discussion**

**3.1 Heat Affected Zone (HAZ) formation**

The macrographs of the welds (Figure 1) shown that the geometry of the fusion line does not correspond to an ellipsoid, as assumed in the numerical simulation studies [7, 8, 12, 15]. The HAZ has different sub regions: A- Sub region below the center area of the weld bead, B- The widest sub region is the one close to inflexion of the fusion line, C- Sub region, situated near the intersection of the fusion line with the upper edge of the plate (Figure 1).

The HAZ width is ruled by the heat flow. Theoretical models of heat transfer allow certain interpretations to be made from analytical calculations of the thermal cycles, giving criterion on the holding time at certain temperature ranges of a given point of the HAZ (between 800 oC and 500 oC or between 1200 oC and 800 oC), and cooling rates in those ranges. The critical plate thickness (Tabla 2) was calculated by the Equation (1). It was observed that the critical thickness is high in relation to the plate thickness, this means that the 2D model (which considers a two-dimensional heat flow with uniform temperature distribution in the thickness of the plate at each instant of time) may be used [16, 17].



Figure 1. Welds Macrographs (immersion etching in modified Beraha's solution). a) and b) 2304 lean duplex steel welds with heat input 1.5 kJ/mm and 2.5 kJ/mm, respectively.

1/2 (1)

Where: dc- Critical thickness (mm); Q- Heat input (J/mm); c- Specific heat (J/(mm3 oC)); To- Initial temperature (oC)

Table 2. Cooling times and cooling rates calculated by the Rykalin 2D model.

|  |  |  |
| --- | --- | --- |
| Parameter | 1.5 kJ/mm | 2.5 kJ/mm |
| dc, mm | 21.91 | 28.29 |
| r, (mm)  | 5.43 | 9.06 |
| t8/5, (s)  | 45.05 | 130.69 |
| t12/8, (s)  | 15.99 | 44.42 |
| φ8/5, (oC s-1) | 6.66 | 2.30 |
| φ12/8, (oC s-1) | 25.02 | 9.00 |

Table 2 also shows the results of cooling times between 800 oC and 500 oC and between 1200 oC and 800 oC (t8/5 and t12/8), cooling rates, and the distance from the center of the weld to the point of maximum temperature, which were determined by Equations (2), (3), (4) [16, 17]. Data used in the calculations were: d= 9,5 mm; To= 25 oC; c=0,005304 J mm-3 oC-1; = 0,022 Js-1mm-1 oC-1 [12] and Tp = 1350 oC (Figure 1). Figure 2 shows the thermal cycles determined by Equation (5) [16, 17]. Table 2 and Figure 3 show that with increasing heat input, the cooling is slower and the HAZ width increases (Figures 1 and 3).

 - (2)

 (3)

 (4)

 (5)

Where: Q- Heat input (J/mm); λ- Thermal conductivity (J/(mm3 oC)); c- Specific heat (J/(mm3 oC)); d- Plate thickness (mm); To- Initial temperature (oC); Δt- Cooling time in the temperature range of interest (t8/5 or t12/8) (T1>T2) (s); φ- Cooling rate between temperatures T1 and T2 (oC/s) Tp- Peak temperature (oC); T- Temperature that reaches the given point at the given instant of time “t” (oC) t- Time (s)



Figure 2. Thermal cycles for different heats input, according to the Rykalin 2D model

Theoretical models explain a general behavior. They consider the formation of a homogeneous HAZ, without the formation of sub-regions. The sub-regions formation (Figures 1 and 3) is caused by differences in heat dissipation, related to the weld bead shape.



Figure 3. HAZ sub-regions (immersion etching in modified Beraha's solution). a) and b) Sub-regions below the center area of the weld bead (A- in figure 2), welded with heat input of 1.5 kJ/mm and 2.5 kJ/mm, respectively; c) and d) are the widest sub-regions (B- in figure 2), welded with heat input of 1.5 kJ/mm and 2.5 kJ/mm, respectively; e) and f) sub-regions, situated near the intersection of the fusion line with the upper edge of the plate (C- in figure 2), welded with heat input of 1.5 kJ/mm and 2.5 kJ/mm, respectively

**3.2 HAZ Microstructure**

The base metal microstructure is shown in Figure 4. Two micro-constituents are observed, the austenite phase () (white) and the dark gray ferrite phase ().The grains orientation is observed, as a result of the hot rolling process. Table 3 shows the base metal ferrite phase percentage.

In the HAZ, regardless of the heat input values (cooling time values), significant changes in the microstructure are evidenced in comparison with the base metal (Figures 3 and 4). Ferrite (), grain boundary austenite (GBA), Widmanstätten austenite (WA), and intragranular austenite (IGA) are observed. The microstructure is similar in the three HAZ sub-regions (Figure 3).



Figure 5. Optical microscopy images (Electrolytic etching in 20 % NaOH solution). a) Base metal, b) Widest HAZ sub-region, welded with heat input 1.5 kJ/mm (cooling time (t12/8)- 15 s), c) Widest HAZ sub-region, welded with heat input 2.5 kJ/mm (c) (cooling time (t12/8)- 41.8 s)

Table 3. Ferrite fraction percentage in the base metal and in the HAZ (the rest for 100 % corresponds to austenite fraction).

|  |  |  |
| --- | --- | --- |
| Base metal | HAZ sub-region | Heat input |
| ZAT1.5 kJ/mm | ZAT2.5 kJ/mm |
| 58.50 | A | 78.27  | 77.74  |
| B | 80.23  | 79.76  |
| C | 83.08  | 82.10  |

The most visible effect of heat input (cooling time) on the microstructure is the ferrite grain growth (Figures 3 and 4) as the cooling time (the heat input) increases. This finding agrees with the findings of various authors [1, 7, 10]. The ferrite grain growth is the result of a longer holding time above the solubilization temperature.

The austenite fraction in the HAZ is significantly low compared to the base metal (Table 3). It tends to increase slightly with the increase of heat input (cooling time). Due to a longer cooling time when the heat input is greater the transformation of ferrite into austenite is favored, since it aids the diffusion of austenite stabilizing elements, such as nickel and nitrogen. It is observed that the ferrite fraction practically does not change with heat input variation (Table 3). Contrary to the result of the present work, Yang et al [1] obtained through simulation with Gleeble, that the increase in heat input tends to increase the austenite fraction in the HAZ microstructure. It is emphasized that the aforementioned work deals with a wider heat input range variation than the present work, while the Gleeble simulates more homogeneous conditions in the volume of the sample compared to a real weld, like the present work. The slight increase in the austenite fraction with increasing heat input, observed in the present work, agrees with the results reported by several authors [7, 10, 18]. Xavier et al [7] attributed such behavior to the effect of the initial ferrite grain size on the austenite reforming. Morales et al [10] relates the behavior of the different austenitic morphologies fraction with the total austenite fraction and with the t12/8, showing that for slow cooling (high heat input) the GBA decreases, while the IGA and the WA grows. These authors also established a relationship between the ferrite grain size and the austenitic fraction. Thus, in the present work, the increase in heat input may have a double effect on the ferrite into austenite transformation: it has favored the transformation by increasing the holding time in the transformation temperature ranges, facilitating the chemical elements diffusion; while it has favored the growth of the initial ferrite grain size that also plays a role in the transformation.

In Figure 3 and Table 3 it is evidenced that the HAZ width, the ferrite grains size, and the phases fraction in the HAZ sub-regions of the same weld, show more differences than those manifested by the heat input variation. This fact confirms that the HAZ of a real weld is heterogeneous, subjected in different sub-regions to different thermal cycles.

Table 4 shows the phases chemical compositions obtained by EDS. It is observed that the chemical elements have been distributed in the phases. In the base metal, the nickel and manganese contents are higher in the austenite. In the HAZ, due to the short holding times at the solubilization temperatures of the ferrite and the high cooling rates (Table 2 Figure 3), the elements diffusion is limited, favoring their distribution without equilibrium.

Table 4. Phase’s chemical composition (mass percent) and PREN values.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Sample | phase | Cr | Mn | Fe | Ni | PREN |
| MB 2304 | δ | 23.16 | 1.11 | 8.04 | 3.31 | 24.66 |
| γ | 21.00 | 1.56 | 64.99 | 5.69 | 24.60 |
| ZAT 2304; 1.5 kJ/mm | δ | 23.80 | 1.44 | 67.43 | 3.93 | 25.30 |
| GBA | 22.54 | 1.14 | 68.83 | 3.05 | 26.14 |
| WA | 21.79 | 1.76 | 70.02 | 3.38 | 25.39 |
| IGA | 22.38 | 1.19 | 8.78 | 3.97 | 25.98 |
| ZAT 2304; 2.5 kJ/mm | δ | 23.15 | 1.45 | 68.17 | 3.19 | 24.65 |
| GBA | 21.90 | 1.92 | 68.33 | 3.24 | 25.5 |
| WA | 21.08 | 2.06 | 66.47 | 3.28 | 24.68 |
| IGA | 22.94 | 1.60 | 69.40 | 3.84 | 26.54 |

The presence of sigma phase in the HAZ is unlikely under the heating and cooling conditions of the present work (Table 2 and Figure 2) since, as stated by several authors such as Xavier et al [7], this phase appears under the effect of several heating cycles, as in multi- pass welding. The mentioned authors; as well as Henrik Sieurin and Rolf Sandström [5], showed that at a heat input range from 0.6 kJ/mm to 2.6 kJ/mm (within this range are the heat input values of the present work), the sigma phase formation is unlikely, since the cooling rates are very fast compared to those required, as reported by Kotecki and Siewert [19]. Also, Nowacki and Łukojć [4] stated that there is no sigma phase presence in the HAZ, in a study that considers relatively similar heat input levels to the present work, 1,6 kJ/mm and 2,2 kJ/mm. Pardal et al [20] stated that increasing the grain size decreases the possibility of undesirable phases formation, since they tend to precipitate at the grain boundary; thus, the grain growth in the HAZ with heat input increase is not totally disadvantageous.

The nitrides formation in the HAZ for high cooling rates in welding of duplex steels is reported by several authors [1, 2, 10]. The last aforementioned authors found the very scarce precipitation of the Cr2N in thin foils of samples from the simulated HAZ with heat input equal 1.5 kJ/cm. In consequence, in the cooling conditions of the present work (Table 2), the formation of nitrides in the ferrite is probable, but very scarce.

**3.3 Pitting corrosion behavior**

Table 5 shows the average values of mass loss corrosion rate. According to standard ASTM G48, the samples undergo pitting corrosion (mass loss corrosion rates  0,001 mg/mm2) [14].

Table 5. Mass loss corrosion rate.

|  |  |  |
| --- | --- | --- |
| Heat input, kJ/mm  | Test time, h | Mass loss corrosion rate, mg/mm2 |
| 1.5 | 24 | 0.0599 |
| 2.5 | 0.0623 |
| 1.5 | 72 | 0.1693 |
| 2.5 | 0.2546 |

Pitting develops intensely in the HAZ (Figure 6), starting in the widest sub-region. In the sub-region, below the center of the weld bead no pits appear.

Several authors agree on the onset of pitting corrosion in the HAZ [22, 23]. When the heat input increases, the width of the HAZ increases (Table 2 and Figure 4), consequently, pitting corrosion increases (Table 5).

Elsaady et al, Soares et al and Mesquita et al [21-23] demonstrate that the pits begin in the ferrite, when the molybdenum concentration is low. On the other hand, Ogawa and Koseki [3] stated that the nitrogen dissolved in austenite protects it from pitting corrosion, which appear more likely in ferrite. In the present work, the presence of nitrogen in the base metals (Table 1), which dissolves favorably in the austenite, guaranties the pitting resistance of this phase.

A generalized criterion to evaluate pitting corrosion resistance is the Pitting Resistance Equivalent Number (PREN30 =% Cr + 3.3 × (% Mo) + 30 × (% N)), as reported by several authors [1, 2]. According to Yang et al [1], the nitrogen solubility in austenite is high, so this phase is assigned the total content of this element in the alloy (according to Table 1 it would be 0,12 % for 2304 lean duplex steel); while ferrite is assigned 0,05 %, which is the solubility limit of this phase. On the basis of the phases’ composition, assigning for the nitrogen the values mentioned above, the PREN was determined (Table 4). The PREN values of the phases in the HAZ are similar (Table 4), which does not mean that the pitting performance is similar, since the ferrite is less resistant to pitting than austenite.

The heat input showed no significant influence on the PREN of the phases in the HAZ (Table 4). This confirms that the pitting corrosion increase, when heat input increases (Table 5), is a consequence of the HAZ width increase (Table 2 and Figure 3).



Figure 6. Macrograph of a sample tested for pitting corrosion (heat input – 2.5 kJ/mm, time of corrosion test - 72 h).

**4. Conclusions**

When the heat input increase from 1.5 kJ/mm to 2.5 kJ/mm in the GMAW process of 2304 lean duplex steels, the HAZ width and the ferrite grain size increases due to the cooling rate decrease (8/5= 6.66 oC s-1 at heat input 1.5 kJ/mm and 8/5= 2.30 oC s-1 at heat input 2,5 kJ/mm). Three sub-regions are observed in the HAZ: one in the center, below the weld bead, another, the widest, near the fusion line inflection, and the third, near the intersection of the fusion line with the upper edge of the plate.

The austenite/ferrite ratio was altered in comparison to the base metal. It does not show significant differences under heat input effect, with slight tendency to increase the austenite fraction when it increases. The HAZ microstructure is constituted by a ferritic matrix with grain boundary austenite (GBA), Widmanstätten austenite (WA) and intragranular austenite (IGA), in correspondence with the high cooling rates. Greater differences in phase fraction and grain size are perceived when comparing HAZ sub-regions in the same weld, than what is evidenced by the heat input variation.

The lean duplex steels welds undergo pitting corrosion in the ferric chloride immersion test, which increases with the extension of the test time. The pits appear in the HAZ beginning in the widest sub-region. The heat input increase does not cause significant changes in the PREN of the phases in the HAZ, which confirms that the pitting corrosion is ruled by the width of region with altered phase fraction ratio.

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