Effects of nitrogen element on microstructure and pitting corrosion resistance of duplex stainless steel welded joints

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Abstract

The microstructure, element distribution and pitting corrosion resistance of Gas Tungsten arc welding (GTAW) duplex stainless steel welded joints under 100% argon (Ar) and 98% Ar + 2% nitrogen (N2) shielding gas were studied by optical microscopy (OM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and electrochemical corrosion methods. These results show that the addition of N2 to the shielding gas promotes the formation of austenite (γ) phase in the weld joint. Also, the austenite phase distribution is dendritic and the ferrite (δ) and austenite phases in the heat-affected zone are no longer banded structures. Moreover, the austenite phase in the welded joint is enriched in nickel (Ni) and nitrogen (N) elements, while the ferrite phase is enriched in chromium (Cr) and molybdenum (Mo) elements. The pitting corrosion index (PREN) of the austenite/ferrite (δ/α) phase boundary is lower than that of δ and γ phases, which leads to their preferential corrosion. The appearance of chromium nitride (Cr2N) decreases the corrosion resistance of the weld seam and the heat affected zone. N2 in the 98% Ar + 2% N2 shielding gas can restrain Cr2N precipitation, increase the pitting corrosion index of secondary austenite structure (γ2), and improve the pitting corrosion resistance of welded joints.

1. Introduction

Duplex stainless steel constituted by ferrite and austenite phase structures has the same good mechanical properties as the austenitic stainless steel and the same corrosion resistance properties as the ferritic stainless steel. They are increasingly used in various fields of petrochemical industry, such as the submarine pipelines and riser system in the oil and gas industry, sea water cooling heat exchanger tube and shell, pressure vessels, boilers, water desalination and nuclear power equipment [1–3]. Welding is an irreplaceable thermal processing process in the manufacturing process of petrochemical equipment. The phase distribution and proportion of weld seam (WM) and heat-affected zone (HAZ) are different from those of the base metal [4, 5]. In order to maintain the excellent performance of the base material, the ideal microstructure state of weld joint is to maintain the ratio of two-phase microstructure (ferrite and austenite) at 1:1. Gas Tungsten arc welding (GTAW) is one of the best welding methods, but its low welding efficiency limits its wide applications [6, 7]. Currently, there are limited literatures about the effect of nitrogen element on the weld microstructure and corrosion of duplex stainless steel.

Heating and cooling are essential during the welding process of the weld seam and the heat-affected zone. During the solidification of the metal, γ and γ2 will precipitate from the ferrite phase [8–12]. In addition, Cr2N, metallic carbide (M23C6) and other phases may also precipitate, which will reduce the mechanical properties and corrosion resistance of the welded joints [13]. When multilayer and multi-pass welding is carried out, the most obvious microstructure change in the HAZ is the precipitation of γ2 [10, 14, 15]. The γ2 not only changes the mechanical property of duplex stainless steel but also increases its local corrosion tendency [13]. Chen et al [16] proposed that the decrease of corrosion resistance of duplex stainless steel welded joints is due to the presence of...
2. Experimental procedure

2.1. Welding procedure

2205 duplex stainless steel plate with 300 mm × 75 mm × 10 mm (length × width × height) was adopted. V-shaped groove with 60° was selected. Welding root gap was 2–3 mm and blunt edge was 2 mm. The welding wire with the diameter of 1.2 mm ER2209 was selected as welding materials. Compared with the base material, the content of Ni and copper (Cu) in the welding material is higher, which is beneficial to the formation of austenite phase in the weld seam [23]. The main chemical composition is shown in Table 1. Before welding, we used special stainless steel grinding wheel to polish groove and edge using 280#, 400#, 600#, 800#, 1000#, 1200#, and acetone to clean plate surface, remove oxide film, oil stains, impurities, etc. In GTAW test, pure Ar and 98% Ar + 2% N2 mixed gas, with a gas flow of 20 l min⁻¹, were used to protect the root and surface of the whole welding process. The welding joint and welding process were shown in Figure 1.

Figure 1. Schematic diagram of welding joints (left) and welding process (right).

\[ E = \eta \frac{U}{V} \]  

\( I \) stands for welding current, \( U \) stands for welding voltage, \( V \) represents welding speed and \( \eta \) represents the thermal efficiency of welding (GTAW: 0.65). The calculated average thermal input value and basic mechanical properties of duplex stainless-steel welded joints are shown in Table 2.

2.2. Microstructure characterization

Taking the weld center as the symmetry axis, the 14 mm × 10 mm × 10 mm samples were cut from D1-D2 test plate along the direction of vertical weld seam. Firstly, ground with 280#, 400#, 600#, 800#, 1000#, 1200#, 1500#, and 2000# and polished to mirror surface. The surface was cleaned using the ultrasonic standard method and 95% ethanol. After that, samples were etched using Vickers etchant for 15 s and then washed by deionized water. The content of Ni and copper (Cu) in the welding material is higher, which is beneficial to the formation of austenite phase in the weld seam [23].

Table 1. Chemical composition of Duplex stainless steel 2205 and E2209T1-1 flux cored wire (mass fraction, %).

| Test materials              | C    | Si   | Mn   | P   | S   | Cr  | Ni  | Mo  | N   | Cu  |
|----------------------------|------|------|------|-----|-----|-----|-----|-----|-----|-----|
| Standard value of 2205 board max | 0.03 | 1.00 | 2.00 | 0.030 | 0.020 | 22–23 | 4.5–6.5 | 3–3.5 | 0.14–0.2 | —   |
| Measured value of 2205 plates avg | 0.024 | 0.56 | 1.39 | 0.013 | 0.002 | 22.35 | 5.66 | 3.22 | 0.16 | 0.08 |
| ER2209 standard max        | 0.04 | 1.00 | 0.5–2.0 | 0.04 | 0.03 | 21–24 | 7.5–10 | 2.5–4 | 0.08–0.2 | 0.50 |
| ER2209 measured values avg | 0.021 | 0.66 | 1.07 | 0.012 | 0.020 | 22.72 | 9.39 | 4.32 | 0.18 | 0.15 |

\( \gamma_{2} \). \( \text{Cr}_2\text{N} \) is one of the most common precipitates in the welding process. The precipitation of \( \text{Cr}_2\text{N} \) will lead to poor Cr and local corrosion [17, 18]. Nitrogen element is added to duplex stainless steel in order to promote \( \gamma \) phase transformation and further improve its mechanical properties and corrosion resistance [19–21]. Adding nitrogen element into the shielding gas is a relatively effective economic method [18, 20] to maintain the ideal ratio of two-phase microstructure and performance of the WM and HAZ of duplex stainless steel. So far, some progresses have been made in the research of nitrogen element on the microstructure and properties of duplex stainless-steel joints. Westin and Johansson et al. [19] pointed out that excessive \( \delta \) phase content in WM accompanied by the precipitation of \( \text{Cr}_2\text{N} \) would lead to point corrosion, while adding nitrogen element into the shielding gas could significantly improve its corrosion resistance. Tsuge et al. [22] proposed that in welding thermal simulation experiment of duplex stainless steel, the increase of nitrogen content would increase the amount of dissolved nitrogen in austenite phase, which would significantly improve resistance to point corrosion and stress corrosion. Kim et al. [18] proposed that the addition of nitrogen in argon gas can reduce the production of \( \text{Cr}_2\text{N} \) and improve the corrosion resistance, while the addition of nitrogen to the shielding gas has not studied the distribution of the two-phase structure of the elements in the weld and the thermal effect.

In this paper, ferrite and austenite two-phase structures and element distribution of GTAW welded joints containing 100% Ar and 98% Ar + 2% N2 shielding gas components were studied by OM, SEM, EDS and constant potential polarization methods, and the corrosion resistance of welded joints was also studied.
Table 2. Welding process parameters and basic mechanical properties of duplex stainless–steel welded joints.

| Test plate | Shielding gas     | Welding current/A | Welding voltage/V | Welding speed/cm · min⁻¹ | Heat input/KJ · cm⁻¹ | Yield strength Rₚ₀.₂/MPa | Tensile strength Rₘ/MPa | Microhardness of WM (HV) |
|------------|-------------------|-------------------|------------------|--------------------------|----------------------|--------------------------|--------------------------|--------------------------|
| BM         | —                 | —                 | —                | —                        | —                    | 656                      | 790                      | 244.2                    |
| D1         | 100%Ar            | 140               | 13               | 12                       | 5.9                  | 575                      | 802                      | 252.1                    |
| D2         | 98%Ar + 2%N₂      | 140               | 13               | 12                       | 5.9                  | 624                      | 782                      | 238.6                    |
1500#, and 2000#, followed by polishing. Then use 10% oxalic acid solution to electrolyze for 15–20 s with 0.4 A DC. Finally, the microstructure of different austenite phases and fine precipitates (such as Cr₂N) were observed by metallographic microscope. Metallographic microscope and pro-image software were used to calculate the ferrite content in different areas. The 10 images with 500x in each area from the WM, HAZ and BM areas were collected for gray scale transformation, binarization, phase extraction and calculation. Finally, the ferrite content average value was calculated.

2.3. Electrochemical testing
In order to evaluate the effect of nitrogen element on the pitting corrosion performance of the welded joint, a three-electrode electrochemical corrosion test was conducted at PARSTAT 2273 electrochemical workstation. The weld specimen was used as the working electrode (WE), the platinum electrode (Pt) as the reverse electrode (CE), and the saturated calamine electrode (SCE) as the reference electrode. Taking the weld center as the symmetry axis, samples were cut from D1–D2 test plate in the direction of vertical weld in order to ensure that the welded joint includes the weld, the heat-affected zone and the base material, and the test area is not less than 1 cm². In order to ensure the stability of the test results, three samples were prepared for each welding method. The samples were ground with 180-2000#, sandpaper mechanically polished to mirror brightness, washed with deionized water, washed with anhydrous ethanol, and dried in a desiccating agent. The 3.5% NaCl solution was selected as the electrochemical corrosion solution, and the critical pitting temperature value (CPT) was determined at constant potential.

Firstly, −1.3 V was applied to the sample to remove the oxide film, and then the corrosion potential was stabilized by running at an open potential for 30 min, and then 700 mV (versus SCE) constant potential was applied to the sample. After 60 s, the temperature was controlled by microcomputer program. The test solution in the constant temperature tank was linearly heated at 1 °C min⁻¹, and the current-time data were recorded at the point frequency of 1 Hz. The corresponding temperature when the current density continuously exceeds 100 μA cm⁻² is the CPT. The higher the CPT value, the better the pitting resistance. The measured potential range of the potentiometric polarization curve was −0.3–1.3 V (versus SCE), the amplitude of ac disturbance voltage was 10 mV, and the scanning rate was 1 mV S⁻¹. The difference value of Epit-Ecorr was used to evaluate the pitting resistance. The material with a larger Epit-Ecorr value was more likely to form a stable passivation film [24], and the pitting resistance performance was better.

3. Results and discussion
3.1. Microstructure characterization
Figure 2 shows the metallographic structure of the welded joints of biphasic stainless steel under 100% Ar and 98% Ar + 2% N₂ gas. The white area is austenite phase, and the gray area is ferritic phase. During the solidification process of weld microstructure, grain boundary austenite (GBA), Widmanstatten-like austenite (WA), intra-austenite (IGA) and secondary austenite can be observed according to different austenite precipitation mechanism and morphology. Intracrustalline austenite is usually wrapped in fault or sediment in the small place nucleation. Compared with other austenitic phases, intracrustalline austenite has higher lattice intracrustalline diffusion activation energy. However, its formation needs more too cold degree as the driving
force. Since the lattice diffusion rate is much slower than the grain boundary diffusion rate, the growth of intracrystalline austenite is restricted, which leads to grain re
refinement\[15\]. The nitrogen in ferrite has low solubility and is in the state of oversaturation, and the solidification process in the welding pool is short, which leads to the precipitation of Cr$_2$N in the grain boundary and inside the ferrite. The Cr$_2$N phases precipitated at grain boundary play a pinning role for the migrated phase boundary, and the appearance of Cr$_2$N becomes a key factor to control the ferrite volume and the formation of intracrystalline austenite\[25, 26\]. However, Cr$_2$N and $\gamma_2$ are often associated, and the chromium deficiency around nitride promotes the formation of secondary austenite with not only chromium deficiency but also nitrogen deficiency, which explains the reason why the weld resistance to pitting corrosion of duplex stainless steel is poor relative to the base material.

Figure 3 shows the EBSD image of the welded joint of duplex stainless steel. Figure 3(a) The base metal is a typical striped structure with ferrite and austenite phases. The ferrite is blue, the austenite is yellow and the Cr$_2$N is red. It can be found that the Cr$_2$N is distributed in the WM and the HAZ. The Eulerian diagram as shown in figure 3(b) illustrates that a ferrite/austenite band consists of several grains with different orientations. The polar diagrams as shown figures 3(c), (d) shows that most ferrite grains show a rolling deformation component \{001\} $\langle 110 \rangle$, while austenite grains show a random orientation [26]. In the heat affected zone, the banded structure gradually disappeared and the equiaxed grains were formed. The orientation of the coarse ferrite grains was the same as that of the banded ferrite in the parent material, while the austenite was precipitated along the ferrite boundary or within the ferrite grains.

Table 3. Ferrite content in weld metal, fusion zone and heat affected zone (%).

| Test plate | Shielding gas           | Weld seam | Heat affected zone | Base metal |
|------------|-------------------------|-----------|--------------------|------------|
| D1         | 100% Ar of GTAW         | 53.2      | 68.2               | 48.7       |
| D2         | 98% Ar + 2% N$_2$ of GTAW | 44.8      | 65.7               | 48.7       |

Table 4. Mass percentage of different chemical elements in ferrite and Austenite phases of GTAW joint in 100% Ar protection (%).

| Area                  | Phase                          | Cr     | Mo    | Ni    | N      | PREN  |
|-----------------------|--------------------------------|--------|-------|-------|--------|-------|
| Weld                  | ferrite                        | 21.74  | 3.52  | 8.02  | 0.082  | 34.67 |
|                       | austenitic                     | 21.04  | 2.37  | 8.65  | 0.120  | 30.78 |
|                       | ferrite/austenite phase boundary| 20.50  | 3.12  | 5.45  | 0.102  | 32.43 |
| Heat affected zone    | ferrite                        | 20.96  | 3.88  | 5.45  | 0.115  | 35.60 |
|                       | austenitic                     | 20.84  | 2.57  | 5.46  | 0.142  | 31.59 |
|                       | ferrite/austenite phase boundary| 20.19  | 2.64  | 4.30  | 0.123  | 30.87 |
| Base metal            | ferrite                        | 22.68  | 3.73  | 4.51  | 0.121  | 36.92 |
|                       | austenitic                     | 21.88  | 3.55  | 7.13  | 0.144  | 35.90 |
|                       | ferrite/austenite phase boundary| 21.37  | 3.17  | 3.65  | 0.128  | 33.88 |
while the austenite phase content increased, and the ferrite content in weld seam decreased from 53.2% to 44.8%. N₂ in shielding gas promoted the conversion of ferrite to austenite during solidification and increased the austenite phase ratio. In the heat-affected area, when the peak temperature of HAZ was close to 1350 °C, ferrite phase and a small amount of liquid phase co-existed, and then HAZ cooled rapidly, leading to a small amount of austenite [12].

### 3.2. Allocation of elements in different phases
Thermodynamic calculation shows that the solidification mode of weld and base metal is ferrite-austenite solidification, and the weld microstructure cannot be composed entirely of ferrite phase [13]. In the weld seam and heat affected zone, ferrite is the main solidification phase due to the rapid cooling after welding. Westin and Hertzman et al [27] studied the element distribution of LDX2101 DSS welds and found that metal elements were prone to segregation at solidified dendrites. Generally, when duplex stainless steel solidifies, ferrite grains in the weld seam grow along the epitaxial boundary of the molten ferrite boundary to form dendrites along the direction with a large temperature gradient, which will lead to segregation of metal elements. Tables 4, 5 shows that the content of different chemical components in ferrite and austenite phases in the weld seam and heat affected zone of D1 and D2 welding test plates, respectively. Ferrite stabilizing elements such as Cr and Mo are enriched in the ferrite phase, while austenite stabilizing elements such as Ni and N are enriched in the austenite phase, and the content of elements in weld seam is not as uniform as that in base metal. The contents of Cr and Mo elements in the ferrite phase are lower than those in the ferrite phase. The contents of Cr and Mo elements at the ferrite/austenite phase boundary in weld seam and heat affected zone with different shielding gas are lowest. The corrosion index formula calculation results show that the ferrite and austenite phase PREN values in the base metal are close, while the ferrite/austenite phase boundary and the austenite phase PREN values in the weld seam and heat affected zone are low, which will result in that the pitting corrosion occurs preferentially at these locations. Pitting resistance equivalent number (PREN) = wt% Cr + 3.3 wt% Mo + 16 wt% N.
3.3. Analysis of corrosion resistance

Figure 4 shows the potential-constant polarization curves of the biphasic stainless steel welded joint and base metal in 3.5% NaCl solution under 100% Ar and 98% Ar + 2% N2 shielding gas. It can be found that CPT of three samples are 45.2 °C, 47.3 °C, and 56.7 °C, respectively, and the CPT value of base metal is highest. No corrosion occurred on the surface of the sample in the initial heating stage. When the solution temperature continuously increased to the CPT, the current density sharply increased and the pitting corrosion occurred. During the GTAW process, 2% N2 was added to the Ar shielding gas, which significantly increased the CPT of the weld joint metal.

![Figure 5. Potential polarization curve of the welded joint in 3.5% NaCl solution.](image)

| Test plate | $E_{corr}$/V | $E_{pit}$/V | $E_{pit}-E_{corr}$/V |
|------------|-------------|-------------|----------------------|
| D1         | -0.24       | 0.54        | 0.78                 |
| D2         | -0.15       | 0.66        | 0.81                 |
| BM         | -0.11       | 1.01        | 1.12                 |

![Figure 6. The distribution of corrosion pits in welded joints.](image)

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Figure 5 shows the potentiodynamic polarization curve of the welded joint and the base metal in 3.5% NaCl solution at 50 °C (equivalent to the CPT). The average values of $E_{corr}$, $E_{pit}$ and $E_{pit}-E_{corr}$ obtained are listed in table 6. The $E_{pit}-E_{corr}$ difference value is used to evaluate the pitting corrosion resistance of the welded joint of the test samples. The $E_{pit}-E_{corr}$ difference value of the GTAW joint with 98% Ar + 2% N$_2$ shielding gas is 0.81 V. The pitting potential is $-0.15$ V, second only to the base metal. Therefore, the point corrosion resistance of the GTAW joint with 98% Ar + 2% N$_2$ shielding gas is better than that with 100% Ar shielding gas.

The distribution of corrosion pits in welded joints after the dynamic polarization curves test is shown in figure 6. The corrosion pits in the welded joints are more than those in the base metal, and the corrosion pits in the GTAW joints with 98% Ar + 2% N$_2$ shielding gas are less than those in the GTAW joints with 100% Ar shielding gas. The corrosion pits mainly distribute the ferrite/austenite phase boundary and within the austenite phase. Tables 4, 5 show that the PREN of ferrite and austenite phase boundary are lower than that of ferrite or austenite phase in WM and HAZ. This explains why the pitting corrosion is more likely to occur at the ferrite/austenite phase boundary.

4. Conclusions

(1) The addition of N$_2$ into the shielding gas promotes the formation of austenite in the GTAW weld seam and heat affected zone. The austenite content in the GTAW welded joint with 98% Ar + 2% N$_2$ shielding gas is higher than that in the GTAW welded joint with 100% Ar shielding gas.

(2) The banded structure in heat affected zone disappears and equiaxed ferrite grains are formed. The grain orientations of these ferrite grains are consistent with those of banded ferrite in the base metal, while austenite precipitates along the ferrite boundary or within the ferrite grains with different grain orientations.

(3) Austenite phases in the weld seam, the heat affected zone and the base metal all enrich Ni and N elements, while ferrite phase enriches Cr and Mo elements. Cr$_2$N in the weld seam and heat affected zone precipitates at ferrite grain boundary and inside the ferrite.

(4) The increase of N$_2$ in 98% Ar + 2% N$_2$ shielding gas controls the two-phase ratio and limits the precipitation of $\gamma_2$. The pitting corrosion resistance of the welded joint was improved, but lower than that of the base metal. The PREN of the ferrite/austenite phase boundary are lower than that of ferrite and austenite phase in weld seam and heat affected zone, which will result in that the pitting corrosion occurs preferentially at the ferrite/austenite phase boundary.

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