Effect of Heat Treatment on the Microstructure of Duplex Stainless Steel Welds

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Abstract
Duplex stainless steels (DSS) are gaining in popularity due to their characteristic features, excellent mechanical properties, and corrosion resistance. The microstructure of DSSs consists of ferrite up to 50 %, and the rest is built up from austenite. The ferritic microstructure can cause chromium-nitride precipitation because the nitrogen solubility in the ferrite phase is very low below 700 °C. Our research showed that electrochemical etching is an acceptable process for revealing chromium-nitrides. Additionally, our research points out that chromium-nitride acts as a secondary austenite nucleation site.

Keywords: heat treatment, duplex stainless steels, austenite, electrochemical etching, nitrogen.

1. Introduction
 Duplex stainless steels provide a high-strength alternative to design engineers within the family of stainless steels. However, the welding of duplex steels requires extreme attention and precise observance of technological variables due to the formation of many possible non-equilibrium transformations [1, 2]. To maintain the appropriate 1:1 austenite (γ):ferrite (δ) phase ratio and alloy content, a usually nickel-over alloyed filler material or nitrogen-containing shielding gas, is recommended for the welding of duplex steels [3–4].

Duplex steels are also alloyed with nitrogen (N) because N is an austenite-forming element, which is more soluble in austenite than in ferrite [5]. Therefore, when the delta-ferrite cools from 1100 °C, it becomes supersaturated in nitrogen, resulting in chromium nitride precipitations (Cr₂N). Chemical composition, cooling rate, and ferrite grain size are factors that determine which precipitates appear. Nitrogen-containing shielding gas also plays a significant role in the formation of the austenite phase, so less nitrogen remains in the ferrite phase, and, as a result, less Cr₂N is formed. Secondary austenite (γ₂) is rapidly formed by various temperature-dependent mechanisms [6]. At temperatures between 600 and 800 °C, where diffusion is faster, Widmannstätten-type austenitic phases are formed [6]. Although the N content of the γ₂ phase is higher than that of ferrite, its Cr and Ni contents remain substantially below that of austenite formed from ferrite [6]. In the temperature range of 700–900 °C, another variant of γ₂ appears at the δ/γ phase boundary, which is lower in terms of Cr content [6]. In the present study, we investigated the formation of chromium nitride and secondary austenite phases in the case of TIG welding without filler metal and during post-weld heat treatment.

2. Materials and methods

2.1. Base materials
 The base material used for both tungsten inert gas welding (TIG welding) and heat treatment was a conventional X2CrNiMoN22-5-3 (1.4462) duplex steel in the form of a 6 mm thick plate. The chemical composition given by the manufacturer is shown in Table 1.
Figure 1. Possible chromium nitride precipitates inside the ferrite grains in the heat-affected zone. The arc energy was 0.25 kJ/mm, and the shielding gas was argon.
gases used. These nitride precipitates were present inside the ferrite grains and, to a large extent, in the heat-affected zone (Figure 2).

Oxalic acid electrochemical etching, on the other hand, dissolves chromium nitride precipitates, so it was not possible to accurately measure their composition in the present research.

3.2. Microstructure after heat treatment

The heat treatment experiment was performed to achieve the formation of secondary austenite. It can be seen from Figure 3 that the heat treatment applied resulted in an increase in the austenite content in both the weld metal and the heat-affected zone. This is due to the appearance of secondary austenite precipitates in the ferrite particles at the site of the previously observed nitride precipitates. It can also be seen that the formation of secondary austenite started inside the ferrite particles.

Regarding the change in the amount of austenite (Figure 4), it can be observed that the austenite content decreases with increasing arc energy in the case of TIG-welded samples in which pure argon was used as the shielding gas.

The addition of 6 % nitrogen to the shielding gas increased the austenite content of the weld metal because nitrogen is a strong austenite former. In this case, the austenite content of the weld metal is 10% higher than the austenite content of the base materials ~50%.

After the heat treatment of the sample welded with argon shielding gas, the austenite content also increased to 50 %. The increase in the austenite content is the result of the formation of γ2 in the ferrite grains, in which phase the Cr2N precipitates that

Figure 2. Possible chromium nitride precipitates inside the ferrite grains in the heat-affected zone. The arc energy was 0.25 kJ/mm, and the shielding gas was argon

Figure 3. Microstructure images of the sample welded with argon and the lowest arc energy after heat treatment; (a) weld metal and (b) heat-affected zone

Figure 4. The austenite content of the weld metal as a function of arc energy, in the case of a sample, welded with both shielding gases and in the case of post-weld heat treatment.
were present before the heat treatment act as a nucleation site for the formation of the secondary austenite. As the proportion of secondary austenite is high due to heat treatment, we can conclude that γ₂ also plays a very important role in actual, multi-pass welding designs, where it can significantly increase the austenite content of the weld metal during reheating of the previously welded passes [9].

Comparing the results obtained with color etching (Figure 5. a) and electrochemical etching (Figure 5. b), it can be concluded that chromium nitride precipitates led to the formation of secondary austenite in the ferritic heat-affected zone, which is in agreement with the results of other researchers [14].

The formation of secondary austenite in the heat-affected zone and in the weld metal can also occur during the multi-pass welding of duplex steels when the austenite content can be significantly increased [15].

4. Conclusion

In our research, we performed TIG welding of conventional duplex steel using pure argon and nitrogen-containing shielding gases. Samples welded with pure argon were post-weld heat-treated in molten salt. The applicability of oxalic acid electrochemical etching to the detection of chromium nitride precipitates was also investigated. In addition, we investigated how these precipitates are converted to secondary austenite during heat treatment. Based on our results, it can be stated that oxalic acid electrochemical etching is a suitable method for the detection of chromium nitride precipitates. Chromium nitride precipitates were visible in the heat-affected zone, and after heat treatment, secondary austenite particles formed at the site of the nitride precipitates, which increased the austenite content of the weld metal.

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