Microstructural and Electrochemical Characterization of Duplex Stainless Steels Friction Stir Welded

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Introduction

Duplex stainless steels (DSSs) balanced with ferrite (α) and austenite (γ) phases exhibit good mechanical property and corrosion resistance as well as better weldability and lower sensitivity to the weld cracking [1]. This enables various types of DSSs application such as chemical and petrochemical industries [2-3].

However, the fusion welding processes associated with the melting and solidification destroy the favorable duplex microstructure of stainless steels, its produces a microstructure consisting of coarse ferrite grains, and both intergranular and intragranular austenite phases in the weld metal and heat affected zone (HAZ) [4]. These changes of the microstructure decrease the corrosion resistance in the weld. To avoid this problem, solid-state joining technologies as friction stir welding (FSW) has attracted considerable attention [5].

The friction stir welding technique is based on a tool to generate heat and deformation [3]. The resultant joint presents different areas as the heat affected zone, termomechanical affected zone and stir zone. The main advantages of this process are the solid state welding, the absence of metal of addiction, and the fast cooling, avoiding precipitation. This process has been studied to applications in oil/gas pipes, aerospace industry and equipments.

The objective of the present study was to evaluate the corrosion resistance of the stir zone of the welded samples in comparison with the base metal. In this work, the corrosion resistance of two FSWed DSS samples (S32205 and S32101) was evaluated using electrochemical impedance spectroscopy (EIS) in a 3.5% NaCl solution.

Experimental

The experiments were performed at the Brazilian Nanotechnology National Laboratory (LNNano - CNPEM). The investigated materials were UNS2205 and UNS S2101 with the nominal chemical compositions shown in **Table 1**.

Duplex	С	Cr	Ni	Mn	Mo	Ν	Fe
S32101	0.02	21.4	1.6	5.1	0.2	0.2	Bal
S32205	0.02	22.5	5.4	1.8	2.8	0.2	Bal

Table 1: Nominal chemical compositions of experimental duplex stainless steels (wt %).

The duplex stainless steel plates of 300 mm (L), 50 mm (W), 12 mm (T) were FSWed in two passes using a NBCP tool of 6.4 mm. The process was performed in the normal direction to the rolling direction in order to compare to the pipeline arc-welding circular joints, under argon atmosphere. The spindle speed was 300 rpm and the welding speed was 100 mm/min.

The samples surfaces were prepared before corrosion tests by grinding with silicon carbide paper up to #4000, polishing with diamond paste up to 1 μ m, and then washed with isopropanol, dried under hot air stream and then immersed in naturally aerated 0.1 mol L⁻¹ NaCl solution at 25 °C. Microstructural observations were performed by optical microscopy (OM) and scanning electron microscopy (SEM) equipped with an energy-dispersiveX-ray spectroscopy (EDS) analysis system.

The electrochemical tests were performed using a three-electrode cell set-up, with a platinum wire and an Ag/AgKCl electrode as counter and reference electrodes respectively.

Potentiodynamic polarization measurements in 3.5% NaCl solution were accomplished using a 1255 Solartron frequency response analyzer coupled to an EG&G 273A potentiostat.

Results and Discussion

Figure 1 show a typical macrostructure of the cross section at different regions of FSWed sample. In the cross-section, the left and right sides of the weld center are related to the retreating (RS) and advancing sides (AS) of the rotating tool, respectively. The microstructure of the welded sample can be classified into two distinct regions including the stir zone (SZ) and, the base metal (BM). The heat-affected zone (HAZ) and the thermo-mechanically affected zone (TMAZ) are not clearly observed.





Figure 1. Macrostructure of S32205 sample showing BM (base metal) and the stir zone (SZ).

Figure 2. Microstructures of FSWed S32205 SS: (a) BM and (b) SZ and S32201 SS samples: (c) BM and (d) SZ.

Figure 2 presents micrographs of the BM and SZ of S32205 SS and S32101 SS samples obtained from the transverse cross-section of the plate. The Figures 2a and 2c displays that white austenite (γ) islands are embedded in a gray matrix of ferrite (α). The SZ consists of fine equiaxed grains with a distorted structure due to the heat effect and mechanical deformation of the material (Figure 2b and 2d). The presence of fine equiaxed grains is more pronounced in the austenite phase. This suggests that the ferrite has a higher diffusion rate than austenite, producing a recrystallization followed by grain growth. Besides, it was not possible to find deleterious phases as sigma, for example, commonly found in samples welded by other processes. The absence of these phases is due to the rapid cooling of the welding, too fast to their nucleation and growth.

Most of phases that use to precipitate in duplex stainless steels during welding decrease the corrosion resistance due to the sensitization of the matrix. Thus, electrochemical tests were

performed in order to evaluate the characteristics of the passive layer formed on the welded samples.

Electrochemical tests were performed in both base metal and stir zone and the results are presented in Figure 3. The total impedance measured at 10 mHz was about 10 times higher for SZ samples than BM samples. S32101 BM sample showed the highest corrosion resistance in aerated and unstirred 3.5% NaCl solution. Complex plane plots showed a capacitive arc which increases according to the following order (S32101 BM > S32205 BM) >>>> (S32101 SZ \cong S32205 SZ). The Bode-phase angle plots showed two time constants for all samples, but the angles phases are higher for BM samples.



Figure 3. Electrochemical impedance spectroscopy (EIS) experiments after 22 h of immersion in 3.5% NaCl solution (a) Nyquist plots (a) and log |Z| and - ϕ vs. log (f) Bode plots.

Conclusions

The FSWed samples did not present deleterious phases due to the rapid cooling of the samples. The microstructures of the stir zone showed the presence of fine equiaxed grains, more pronounced in the austenite phase. This suggests that the ferrite has a higher diffusion rate than austenite, producing a recrystallization followed by grain growth. The electrochemical results indicate the presence of a high stability oxide layer at the base metal of both DSS. However, at the stir zone, both Bode phase curves decrease at lower frequencies. This can be related to the recrystallization process and the consequent unbalanced microstructure. All Bode phase diagrams presented two peaks associated to the duplex oxide layer formed on the surface of stainless steels.

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