# Practical Aspects of the Electrochemical Potentiodynamic Reactivation Technique Applied in Welded Joint of Superduplex Stainless Steel

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### Abstract

Electrochemical Potentiodynamic Reactivation with Double Loop (DL-EPR) technique has been used for detection of deleterious phases in stainless steels. However, the use of DL-EPR to evaluate the corrosion resistance of the welded metal of superduplex stainless steel needs a systematic study. The major issue is to establish experimental conditions to identify a small reduction in corrosion resistance due to precipitation of intermetallic deleterious phases (phases and  $\sigma$  and  $\chi$  mainly). The present work summarizes DL-EPR results using a portable device specially designed to work with real welded joints. This cell was employed in two joints of superduplex stainless steel welded with low and high heat input. The phase detected was characterized by scanning electron microscopy (SEM) and Electron Backscatter Diffraction (EBSD). Finally, it will be presented correlations between parameters obtained by DL-EPR tests and the susceptibility to localized corrosion.

#### Introduction

Duplex and super duplex stainless steel (DSS and SDSS) are basically Fe-Cr-Ni alloy with a matrix composed of delta ferrite ( $\delta$  - Body Centered Cubic, BCC) and austenite ( $\gamma$  - Face Centered Cubic, FCC), usually in a ratio near 1:1. The main alloying elements present are chromium (Cr), nickel (Ni) and molybdenum (Mo), but depending on the alloy it can have additions of nitrogen (N), copper (Cu), silicon (Si) and tungsten (W). These classes of stainless steels are susceptible to important microstructure transformation when submitted to ranges of temperature between 350°C and 1000°C [1]. The presence of intermetallic phases decreases the corrosion resistance of the DSS and SDSS due to the creation of adjacent regions depleted in Cr and Mo, susceptible to localized corrosion [2]. The most critical situation for DSS and SDSS occur during welding, where a volume of the weld metal will be subjected constantly within the range of temperatures between 350°C and 1000°C. Depending on the exposure time precipitation of undesired intermetallic phases may happen. The residence time within the range of critical temperatures depend on the heat input and multiple thermal cycles of welding. Concerning the DL-EPR, some standards are used to identify the decrease of corrosion resistance. Číhal et al [3] presented a methodology to evaluate localized corrosion and this procedure becomes the basis of the standard ISO 12732. The methodology consist in the anodic scanning from open circuit potential (E<sub>ocp</sub>) to 700 mV of overvoltage with a reverse cathodic scan until E<sub>ocp</sub>. Depending of the microstructural condition two or more current peaks occur during the anodic and cathodic scan (I<sub>a</sub> and I<sub>r</sub>, respectively). The value of the relation  $I_r/I_a$  and/or the area under the curves  $(Q_r/Q_a)$  represent a quantitative parameter that can be used to identify the susceptibility to localized corrosion of DSS an SDSS. The objective of this work is to evaluate the DL-EPR and its applicability for detection localized corrosion of a superduplex stainless steel welded with two different heat inputs.

## Experimental

Two different welded joints were prepared using two heat inputs. The process and values of heat input used in the welding are presented in table 1.

Table 1: Welding parameters						
	Process					
Sample	Pass			Heat input [kJ.mm <sup>-1</sup> ]		
	Root	Filling	Finishing			
Low heat input (LH)	GTAW	GTAW	GTAW	$\approx 0.8$		
High heat input (HH)	GTAW	GTAW	GTAW	$\approx 3.0$		

After welding, samples were obtained for DL-EPR and microstructure tests. The identification and quantification of the  $\delta$  and  $\gamma$  and of the intermetallic phases were performed using SEM with EBSD techniques. Table 2 shows information about SEM/EBSD.

Table 2: SEM/EBSD parameters				
Work Distance (mm)	12-18			
Spot Size (µm)	0.55-0.59			
Step Size (µm)	0.6-0.7			
voltage (kV)	20			

The electrochemical tests were performed with a three-electrode cell using HCl  $3mol.l^{-1}$ : The reference and counter electrode were platinum and the work electrode the welded joint. An AUTOLAB PGSTATION was used imposing a scan rate of 0.56 mV.s<sup>-1</sup> from E<sub>ocp</sub> to  $\approx$  + 300 mV, as suggested by ISO 12732.

#### **Results and Discussion**

The SEM/EBSD results are presented in tables 3 and 4, for the samples welded with low and high heat input, respectively.

Table 3: Quantification of the phases on surface of the welded joint with LI				
Grains analyzed	7761			
Total area analyzed ( $\mu m^2$ )	30224,00			
Percentage of grain identified	$96{,}24\pm0{,}08$			
% γ	$63,\!29 \pm 7,\!16$			
% δ	$36,71 \pm 5,01$			
Table 4: Quantification of the phases on surface of the welded joint with HI				
Grains analyzed	27411			
Total area analyzed ( $\mu m^2$ )	134933,60			
Percentage of grain identified	$92,06\pm2,52$			
% δ	$6,\!84 \pm 2,\!52$			
% γ	$76,42 \pm 3,06$			
% χ	$7{,}18\pm3{,}05$			
% σ	$1,62 \pm 0,26$			

The DL-EPR tests are present in the table 5 and figure presents the cell used to polarize the pipe.

Table 5: $Q_r/Q_a$ Values obtained after tests			
Sample	$Q_r/Q_a$		
LI (≈ 0,8 kJ/mm)	$0,30\pm0,08$		
HI ( $\approx$ 3,0 kJ/mm)	$0,53\pm0,05$		



(b) Figure 1: DL-EPR device

### Conclusions

The DL-EPR technique, carried out with the portable device, was able to detect the presence of intermetallic deleterious phases. However, as it will be emphasized in the paper, the microstructure characterization is essential for the correct interpretation of electrochemical test.

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