

## On-site weld quality assessment and qualification for stainless steels tanks

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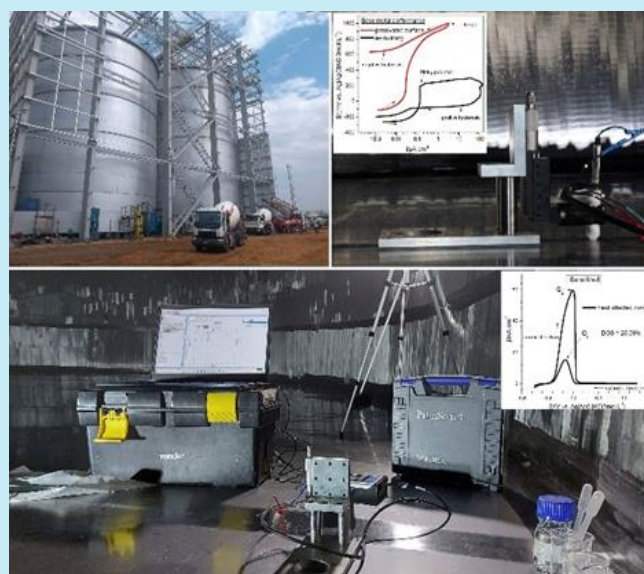
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**ABSTRACT:** Stainless steel tanks are frequently degraded by localized corrosion in bioprocess industries. A case study of 2101 duplex stainless steel tank allowed to apply a portable electrochemical microcell system (PassivityScan) for *on-site* weld inspection and corrosion monitoring from the manufacturing until 12 months of operation. During the tank manufacturing, the double loop electrochemical potentiokinetic reactivation technique was applied to measure the sensitization degree on the welded regions. The manufactured tank was submitted to the passivation treatment to improve the passivation properties and corrosion resistance. *On-site* cyclic polarization tests were performed and confirmed the increase of passivation level of treated surface. The passivation level was measured again after 12 months of operation and detected a lower passivation level compared to the initial passivated surface, indicating that the passivation treatment lost its effect after that period. Therefore, an acceptance criterion to passivation level was empirically determined. PassivityScan is an advanced device to qualify the welds and the passivated surfaces, and useful for corrosion monitoring and the reliability maintenance.



## 1. Introduction

Stainless steel is widely used to build the bioprocessing equipment and facilities due to its natural capacity to generate and recover a passive film on the surface, characterized as a thin layer of chromium oxide ( $\text{Cr}_2\text{O}_3$ ) that becomes the steel surface with high electrical resistance, almost inert and then, adequate to aseptic and anticorrosive applications. The electrical resistance of the passive film is dependent on the steel chemical composition and particularly on the content of chromium (Cr), molybdenum (Mo) and nickel (Ni). Besides the chemical composition, the stainless steel grade to be selected must consider the characteristic of the particular industrial process. The most used stainless steels in bioprocessing applications are from austenitic (304L and 316L) and duplex (2205, 2101 and 2304) groups, because they have showed adequate performance in processes in which hygienic characteristic and anticorrosive properties are required (Guilherme *et al.*, 2022).

The American Society of Mechanical Engineers: Bioprocessing Equipment (ASME BPE, 2019) standard aims to define requirements to the project and construction of the equipment and facilities to the bioprocessing industry, such as pharma and life science, food grade products, as well as any industrial sector where high level of hygienic and asepsis requirements are established. The welding process, the surface finishing and the chemical passivation treatment are considered by ASME BPE as special processes to obtain a hygienic surface and, therefore, the standard has specific requirements to each process.

In the field of welding, the ASME BPE defines physical and metallurgical requirements to achieve an aseptic and corrosion resistant welded joint. The equipment design needs to specify the average roughness ( $R_a$ ) of the surface finishing, which can be obtained by mechanically polishing or electropolishing processes. The surface in contact with the aseptic product also needs to be submitted to the chemical passivation treatment because this procedure removes the impurities and contaminants from the surface, providing a passive film with high corrosion resistance.

The ASME BPE recommends the use of electrochemical techniques to field services as an *in situ* advanced inspection, being specifically suggested the application of the electrochemical impedance spectroscopy (EIS) to assess the rouge contamination and the cyclic potentiodynamic polarization (CPP) to

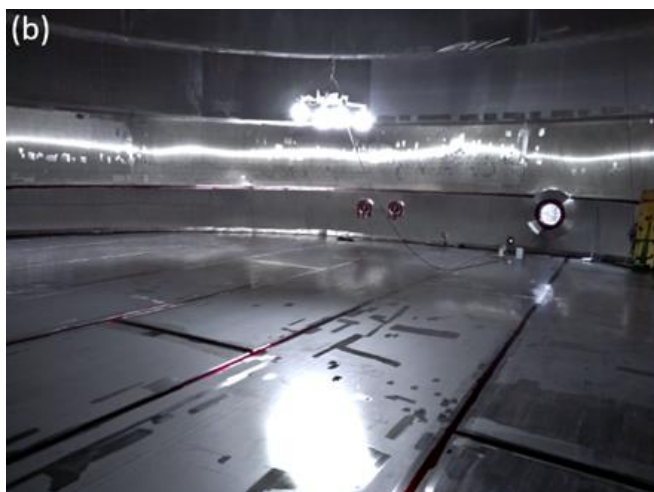
assess the level of surface passivation. However, the ASME BPE mentions that the development of an electrochemical tool to *in situ* services is still ongoing. On the other hand, recent works (Guilherme *et al.*, 2019a; 2021a; b; c; d) have showed a portable electrochemical microcell, named PassivityScan, that can apply electrochemical techniques in *on-site* surface inspection of the tanks and pipelines.

This work aims at showing the results obtained by PassivityScan in advanced *on-site* inspection and how it is useful to the reliability maintenance. For this purpose, results from *on-site* metallurgical integrity and passivation properties inspections are presented and discussed. It is important to highlight that the *on-site* surface inspections were performed in three steps of the tank life cycle: as built, after chemical passivation treatment and after 12 uninterrupted months of operation.

## 2. Study of case

The study of case shows the results of the *on-site* inspection of an aseptic stainless steel tank where advanced electrochemical techniques were applied. The inspections were made in the following steps of the tank life cycle as built, after surface chemical passivation and after 12 uninterrupted months of operation. The LDX 2101 lean duplex was used as base metal to construct the tank that was designed to storage NFC orange juice in an industrial cold chamber that works at around 1–3 °C. Figure 1 shows a general view of the tanks during its manufacturing and the tank inside view regarding the bottom plate and inner wall that were the objective of the surface inspections.





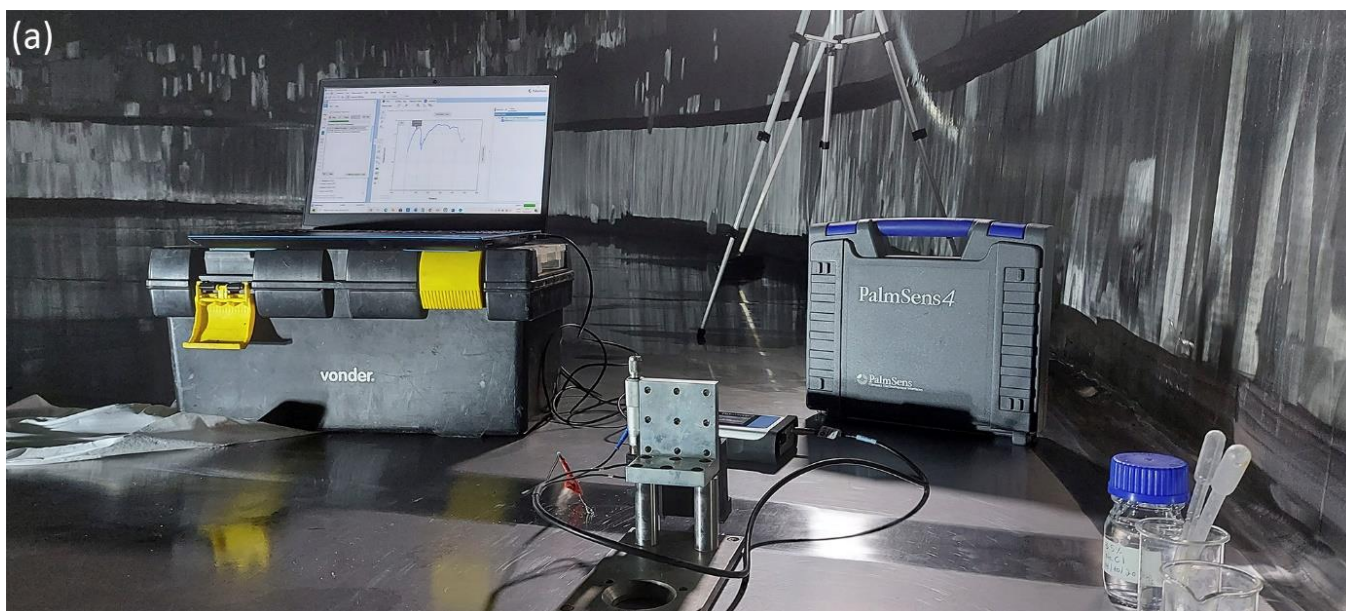
**Figure 1.** Construction site of the industrial cold chamber where it is showed the tanks. **(a)** In building process; **(b)** Internal face of tank showing the bottom plate and shell.

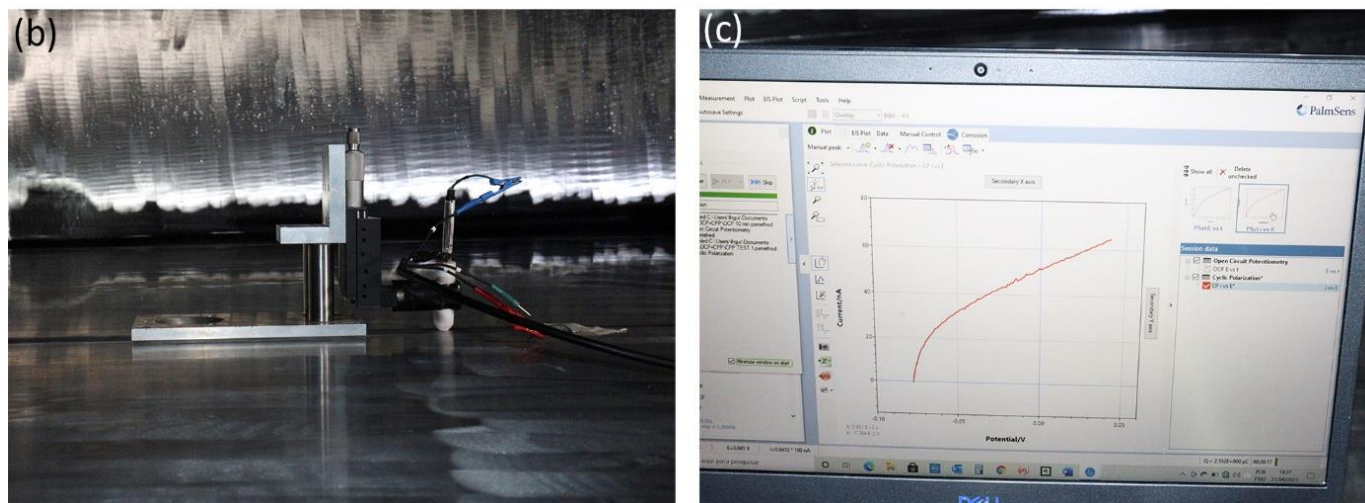
The welding of the tank parts was made using a semiautomatic tungsten inert gas (TIG) process (TIP TIG) by applying a double side synchronous welding technique, which means that two welders attack the joint simultaneously on opposite sides, and the maximum heat input was regulated as  $2.5 \text{ kJ mm}^{-1}$  to generate an appropriate weld metallurgy (Guilherme *et al.*, 2021c; Huang, 2015; Reccagni *et al.*, 2019; Wang *et al.*, 2021). The surface finishing was obtained by

mechanically grinding with an average surface roughness,  $R_a < 0.76 \mu\text{m}$ , and this parameter was controlled by a digital profilometer. After the surface finishing was concluded, a chemical treatment according to ASTM A-380 using a based nitric acid aqueous solution (12 v/v%) was performed to improve the passivation property of the inner surface of the tank.

The PassivityScan is a portable system capable of making electrochemical tests directly on the industrial site to evaluate equipment and facilities. It works as a typical three-electrodes microcell that was designed to be used in *on-site* inspection services (Guilherme *et al.*, 2019a). PassivityScan is robust and versatile to be used in all positions because its O-ring with diameter of 1.0 mm (scanned surface area is  $0.008 \text{ cm}^2$ ) inserted on the bottom of the cell gets a well coupling with the surface, avoiding crevice corrosion, noise, or any external influence. The coupling of the microcell O-ring with the tank bottom surface can be seen in Fig. 2.

The construction site inspection management considered *on-site* electrochemical techniques in the Quality Control Inspection & Test Plan, and the aim was to assess the metallurgical integrity of welds by double loop electrochemical potentiokinetic reactivation technique (DL-EPR tests) and to certify the passivation level after chemical passivation treatment (by CPP tests), as described in Tab. 1.





**Figure 2.** PassivityScan being used to measure the passivation level of the tank's internal surface. (a) PassivityScan's overview; (b) view of support and mini-cell; (c) Cyclic potentiodynamic polarization measurement on going.

**Table 1.** Goals of using PassivityScan in the integrity inspection and passivation.

Tank conditions	Objective	Technique	Performance parameters	Criteria
as built	metallurgical integrity of welds	on-site DL-EPR	$i_a$ , $i_r$ , $Q_a$ , $Q_r$	DOS < 1
after chemical passivation (ASTM A-380)	passivation level	on-site cyclic polarization	$E_{corr}$ , $E_{pit}$ , $E_{prot}$ passivation level	$E_{prot} - E_{corr} > 350$ mV

The degree of sensitization criteria is based on the literature (Deng *et al.*, 2010). The passivation criterion is an empirical criterion.

The DL-EPR technique was applied to measure the degree of sensitization (DOS) on the weld regions, and, for that, the electrochemical potential was scanned first in the anodic direction, from  $-500$  mV to  $+300$  mV vs. Ag/AgCl/KCl  $3 \text{ mol L}^{-1}$ , where the polarization scan was reversed, and scanned back to  $-500$  mV vs. Ag/AgCl/KCl  $3 \text{ mol L}^{-1}$ . A sweep rate of  $1.67 \text{ mV s}^{-1}$  was used for the tests. The criteria for the degree of sensitization consider the limit value to the DOS ratio of 1%, according to previous study (Deng *et al.*, 2010).

Cyclic potentiodynamic polarization tests were carried out to measure the passivation level integrity in view of ensure the passive film resistance at all over the surface (Guilherme *et al.*, 2019b), and the procedure was performed in 3.5% wt. NaCl solution to evaluate the pitting corrosion resistance. After stabilization of the open circuit potential (OCP) ( $\sim 5$  min), an anodic polarization scan was performed at a sweep rate of  $1.67 \text{ mV s}^{-1}$ . The anodic scan was reversed after it reaches one of the criteria: (i) current density of  $1 \text{ mA cm}^{-2}$  or (ii) potential of 1 V. Finally, the samples were scanned in the cathodic direction to a potential of  $-200$  mV vs. OCP.

The passive region (or passivation level) takes into consideration the electrochemical parameters from the CPP curves in order to evaluate the resistance of the material to localized corrosion:  $E_{corr}$ ,  $E_{pit}$ ,  $E_{prot}$ . If  $E_{prot}$  is nobler than  $E_{corr}$  there is a potential range where the passive film is stable and no localized corrosion such as pits, crevice or crack initiation, will initiate or grow. The literature called this region as perfect passivity and the ASME BPE named it as passivation level, and the difference between  $E_{prot} - E_{corr}$  indicates the amount of passivation level (ASME BPE, 2019; Esmailzadeh *et al.*, 2018). An empirical criterion for passivation level of 350 mV was determined.

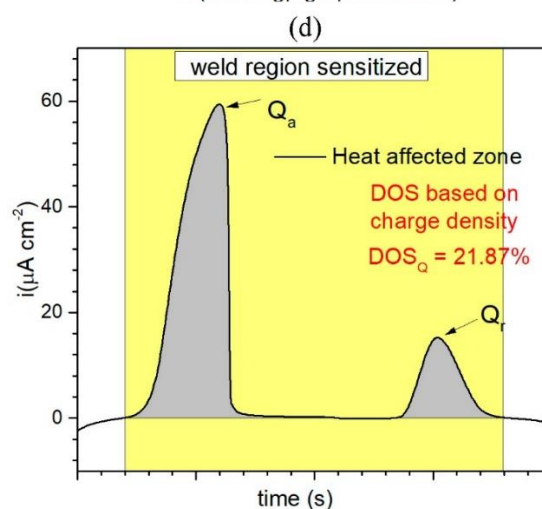
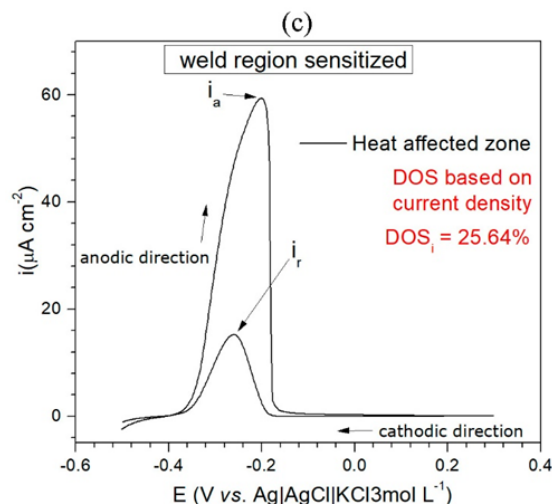
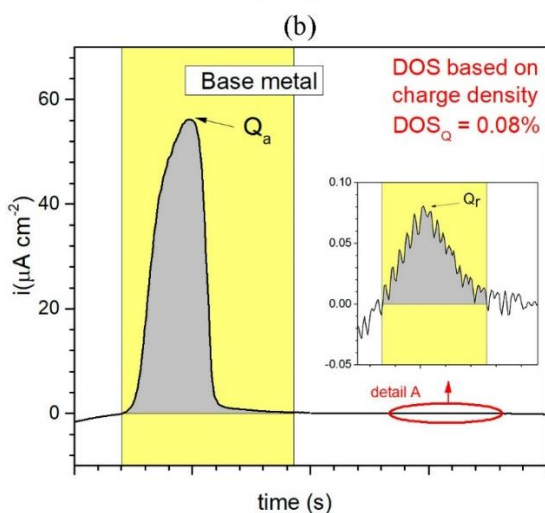
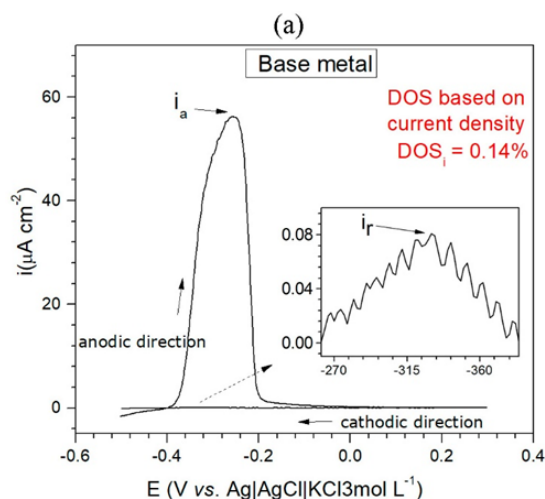
The DL-EPR technique was tested at 90 surface points throughout 270 linear m of weld regions and the CPP technique was tested at 125 surface points on bottom plate and shell related to a total superficial area of  $375 \text{ m}^2$ .

Table 1 describes the electrochemical techniques applied to assess the tank welds and surface and the respective performance parameters and criterion of acceptance. It is important to emphasize that DL-EPR technique was applied to assess the metallurgical features of welds and figure out if the weld region has

Cr-depleted zones, whereas CPP technique assessed the resistance of passive film. Bear this in mind, the criterion of acceptance to the DL-EPR was based on the literature (Deng *et al.*, 2010); nevertheless, there was not a established criterion of acceptance to the CPP technique and/or passivation level ( $E_{\text{prot}}-E_{\text{corr}}$ ). Considering that it is important to have a minimum range of passive region in order to obtain a reliable material for engineering applications, it was empirically defined the criterion of acceptance of 350 mV.

### 3. Results and discussion

Figure 3 presents typical DL-EPR curves of the nonsensitized base metal and sensitized weld heat affected zone (HAZ) and their DOS values that were calculated using two different data: (i) activation and reactivation current densities ( $i_a$  and  $i_r$ ) (Ebrahimi *et al.*, 2011; Hong *et al.*, 2013), and (ii) activation and reactivation charge densities ( $Q_a$  and  $Q_r$ ) (ASTM G108, 2004).



**Figure 3.** On-site DL-EPR curves calculating DOS based on current density [ $(i_r/i_a) \times 100\%$ ] and charge density [ $(Q_r/Q_a) \times 100\%$ ] to not sensitized base metal (a)  $DOS_i = 0.14\%$ ; (b)  $DOS_Q = 0.08\%$ ; and to heat affected zone of sensitized weld surface; (c)  $DOS_i = 25.64\%$ ; (d)  $DOS_Q = 21.87\%$ .

First, the current of the reactivation peak ( $i_r$ ) and the peak activation current density ( $i_a$ ) were graphically determined using Origin v.9 software and the DOS was figured out as the ratio  $(i_r/i_a) \times 100\%$ . Second, the typical DOS curve (potential vs. current density) was converted into current density vs. time, following the charge determination by calculating the integrate of the gray area observed in Fig. 3b and d. It was done using integrate tool in Origin v.9 software and then the DOS was measured as the ratio  $(Q_r/Q_a) \times 100\%$ .

The degree of sensitization is a powerful criterion of acceptance to approve stainless steel tanks in manufacturing phase or maintenance projects, taking into account that it is extremely important to avoid operation fails caused by improper welding processes.

All the same, the DOS might be determined from the current density or the charge density, as shown in Fig. 3, and it was noted a soft difference between the DOS results. For practical applications, it is considered

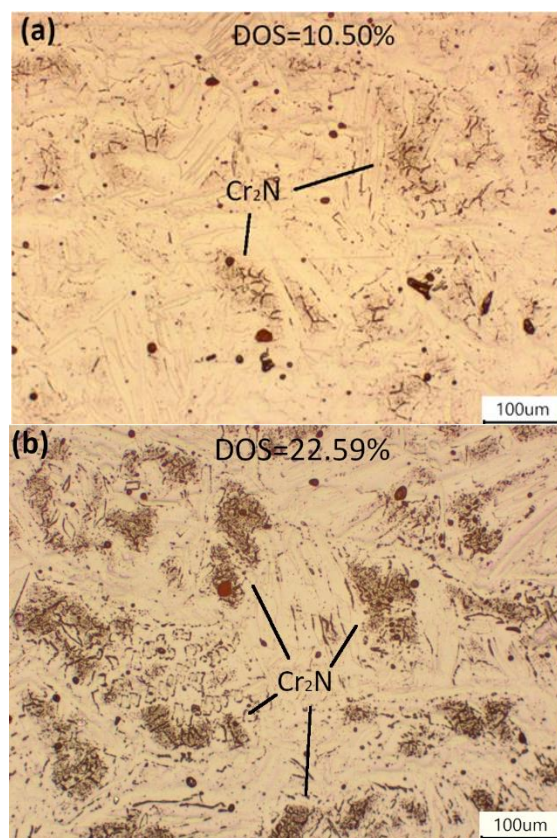
the higher value in view of reducing the risk to failure. Table 2 illustrates how the results are shown in the metallurgical integrity report.

**Table 2.** *On-site* DL-EPR tests results.

Welded joint number	Surface site	DOS based in activation and reactivation current density			DOS based in activation and reactivation charge densities		
		$i_r$ ( $\mu\text{A cm}^{-2}$ )	$i_a$ ( $\mu\text{A cm}^{-2}$ )	DOS $i_r/i_a$ (%)	$Q_r$ ( $\text{mC cm}^{-2}$ )	$Q_a$ ( $\text{mC cm}^{-2}$ )	DOS $Q_r/Q_a$ (%)
base metal	base metal	0.08	56.23	0.14	3.21	3890.21	0.08
weld-01	weld	0.002	1.50	0.10	0.09	124.45	0.07
	fusion line	0.004	3.00	0.12	0.44	401.56	0.11
	HAZ	0.003	2.10	0.13	0.27	345.47	0.08
weld-18	weld	4.79	45.20	10.60 <sup>#</sup>	233.20	2486.15	9.38 <sup>#</sup>
	fusion line	15.23	59.40	25.64 <sup>#</sup>	874.40	3998.60	21.87 <sup>#</sup>
	HAZ	10.27	55.80	18.40 <sup>#</sup>	507.76	3254.87	15.60 <sup>#</sup>

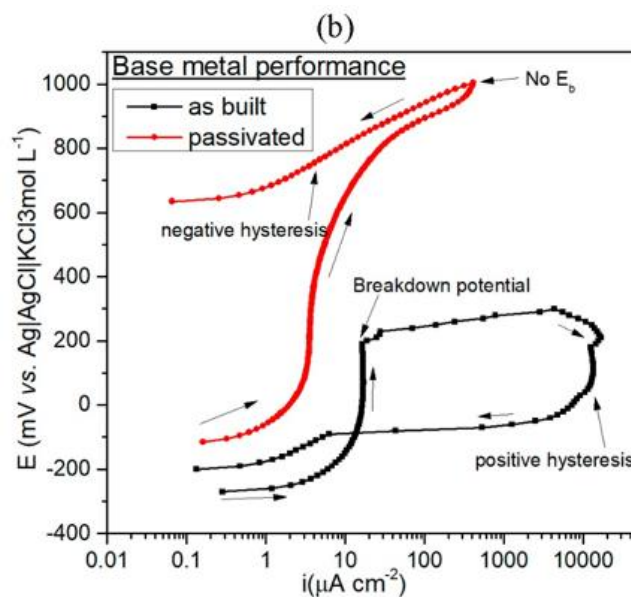
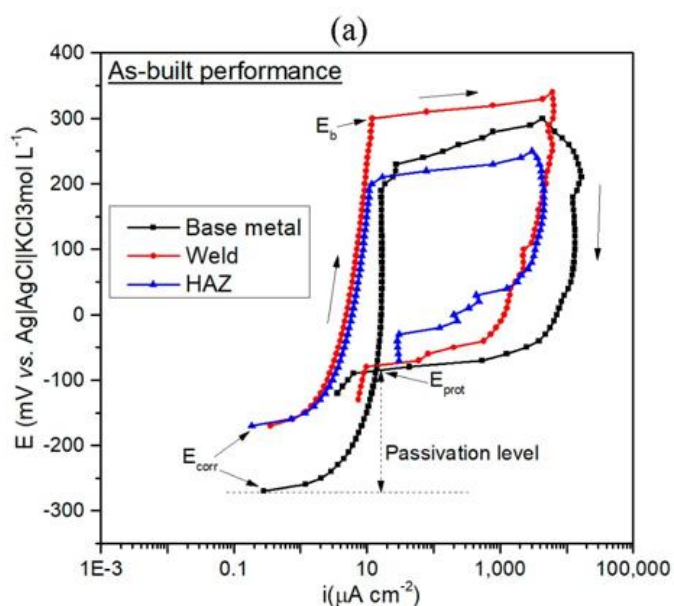
<sup>#</sup>Inspected surface with DOS > 1 has submitted to the repair protocol.

It is to note that these *on-site* DL-EPR measurements were useful to optimize the parameters of the Welding Procedure Specification (WPS) to produce high-performance welds due to the welding energy control and consequently to obtain a microstructure free of sensitization (DOS < 1) (Hong *et al.*, 2013). After that, while the tanks were assembled, *on-site* DL-EPR tests were made to assess and control the compliance of the metallurgical integrity of the welded joints. Eight inspected areas in a total of 90 were reprovved based on the criteria of DOS ≤ 1, which represent a failed level of 8.9%. Each reprovved area was double-checked by field metallography performed after electrolytic etching using a 10% (w/v) oxalic acid aqueous solution to attack the microstructure. It was also observed the preferential corrosion attack of Cr-depleted regions around Cr-carbides and Cr-nitrides, which promote increased DL-EPR values, corroborating the *on-site* DL-EPR measurements (Guilherme *et al.*, 2019b). Figure 4 shows photomicrographs from sensitized surfaces obtained by field metallography, where the morphology of the Cr-depleted zones was noticed. All reprovved welds in the *on-site* DL-EPR inspection were submitted to the repair protocol.



**Figure 4.** Field metallography of the sensitized surface with (a) DOS = 10.50% and (b) DOS = 22.59%. The Cr<sub>2</sub>N regions were responsible for the high level of the degree of sensitization and these regions are highlighted in the figures.

Cyclic potentiodynamic polarization curves in 3.5 wt% NaCl solution are presented in Fig. 5. All CPP curves showed a passive behavior and the electrochemical parameters used to quantify the passivation level were corrosion potential ( $E_{\text{corr}}$ ), pitting potential ( $E_{\text{pit}}$ ), protection potential ( $E_{\text{prot}}$ ), and the passivation level was calculated as  $E_{\text{prot}} - E_{\text{corr}}$ , which represents the *perfect* passive range (Kelly *et al.*, 2002). Figure 5a shows the CPP curves of the finished surface of as-built conditions (black curve), where a passive level of around 180 mV was obtained, and it is highlighted that the weld metal (blue curve) always had a higher performance due to the chemical composition of filler metal ER2209. In contrast, weld HAZ (red curve) showed the lowest performance due to the metallurgical features of this region. Figure 5b compares the passive film resistance between as-built condition (just mechanically polished) and chemical passivated surface (mechanically polished plus chemical treatment), with the latter achieving 4× superior performance (from 185 to 750 mV). It is important to highlight the passivation level obtained from as-built surfaces registered a pitting potential and presented a positive hysteresis after it, which means that the nucleated pits continued to grow until reaching the protection potential. On the other hand, the CPP measurements after chemical passivation treatment did not register a pitting or breakdown potential and demonstrated a negative hysteresis when the scan current density was reversed, which means that the passive film was not degraded by pitting corrosion in this test condition (Kelly *et al.*, 2002).



**Figure 5.** On-site cyclic polarization curves of (a) inspected regions such as base metal, weld metal, and heat affected zone and (b) comparison of performance between chemically passivated and not passivated surfaces.

The electrochemical parameters to assess the surface performance regarding passivation property are shown in Tab. 3 for the three life cycles of the tank: as-built, after chemical passivation treatment and after 12 uninterrupted months of operation. First, it is worth emphasizing that the as-built surfaces did not obtain satisfactory corrosion electrochemical parameters regarding the passivation properties. When compared to the passivated surface, the as-built surfaces demonstrated the lower corrosion potential and a premature pitting potential, which result in a low level of passivation based on the criteria  $E_{\text{prot}} - E_{\text{corr}}$ . The passivated surfaces shown the best performance in electrochemical corrosion tests with emphasis about the pitting potential: pit was not generated. It means that the passivated surfaces are more resistance than as-built surfaces against localized and pitting corrosion. The hypothesis is related to the complete removal of free iron on the surface after mechanical polishing, and the chemical passivation promotes the richer passive film in chromium (ASME BPE, 2019).

All the same, it is important to point out that in the most cases the manufactured tanks are included in the industrial process without chemical passivation treatment and it is a fault considering that the corrosion resistance and aseptic properties are significant increased by this treatment. In addition, the cost to apply the passivation treatment is inconsiderable when compared to the total investment in a new tank or the

cost to repair corrosion degradation. Finally, it was noted that after 12 months of operation the passivation properties were reduced when compared to the passivated surface, and it may be concerned to the

industrial environment that the tank works in term of cleaning solutions used to sterilize it, corrosive elements present in bioprocessing and biofilm formation.

**Table 3.** Passivation level assessment in different periods of the tank life cycle as built, after chemical passivation, and after 12 uninterrupted months of operation.

Construction phase	Inspection site	$E_{corr}$ (mV)	$E_{prot}$ (mV)	$E_{pit}$ (mV)	Passivation level (mV)
As-built	base metal	-270	-85	+230	185
	weld metal	-170	-80	+300	90
	HAZ	-180	-30	+198	150
passivated surface (ASTM A-380)	base metal	-115	+635	+1000 <sup>1</sup>	750
	weld metal	+70	+680	+1000 <sup>1</sup>	610
	HAZ	+140	+660	+1000 <sup>1</sup>	520
after 12 months of operation	base metal	-185	91	+345	311
	weld metal	-130	204	+389	334
	HAZ	-169	128	+255	297

<sup>1</sup> $E_{pit}=1000$  mV indicates that stable pit nucleation and grow did not occur.

Looking at Fig. 5a, one may have the impression that different values of current density were used for the reversal of the scanning direction of potentials in the different regions of the weld. To clarify this, the readers are invited to see the Appendix.

#### 4. Conclusions

The results from CPP measurements demonstrated that the surface finishing with  $R_a = 0.76 \mu\text{m}$  had a passive behavior, and a low passivation level was obtained after polishing process. The surface chemical passivation treatment significantly increased the passivation level. The surface passivation level was reduced after 12 uninterrupted months of tank operation. The surface chemical passivation, applied according to ASTM A-380 restores the passivation level.

The *on-site* surface advanced inspection can be useful to reliability maintenance of managing the assets and facilities that operate in corrosive or aseptic processes. The application of the welding procedure specification permitted the obtention of a proper weld microstructure to be corrosive resistant and perform the weld quality control during tank manufacture. The CPP measurements gave the surface passivation level of the tank critical parts and qualify the compliance of the surface after chemical passivation treatment. These measurements also supported the assessment of the tank inner surface in contact with food grade product and allow demonstrating the passivation level decreased as a function of time. Bear it in mind, an acceptance criterion to passivation level was

determined for practical application, and this is an important data manage to the reliability maintenance. Based on passivation level parameter is defined the correct moment to restore the tank by chemical passivation treatment.

#### Authors' contribution

**Conceptualization:** Guilherme, L. H.; Benedetti, A. V.  
**Data curation:** Guilherme, L. H.  
**Formal Analysis:** Guilherme, L. H.  
**Funding acquisition:** Guilherme, L. H.; Benedetti, A. V.  
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**Project administration:** Guilherme, L. H.; Benedetti, A. V.  
**Resources:** Guilherme, L. H.; Fugivara, C. S.  
**Software:** Not applicable.  
**Supervision:** Benedetti, A. V.  
**Validation:** Guilherme, L. H.; Fugivara, C. S.; Benedetti, A. V.  
**Visualization:** Guilherme, L. H.; Benedetti, A. V.  
**Writing – original draft:** Guilherme, L. H.  
**Writing – review & editing:** Guilherme, L. H.; Fugivara, C. S.; Benedetti, A. V.

#### Data availability statement

The data will be available upon request.

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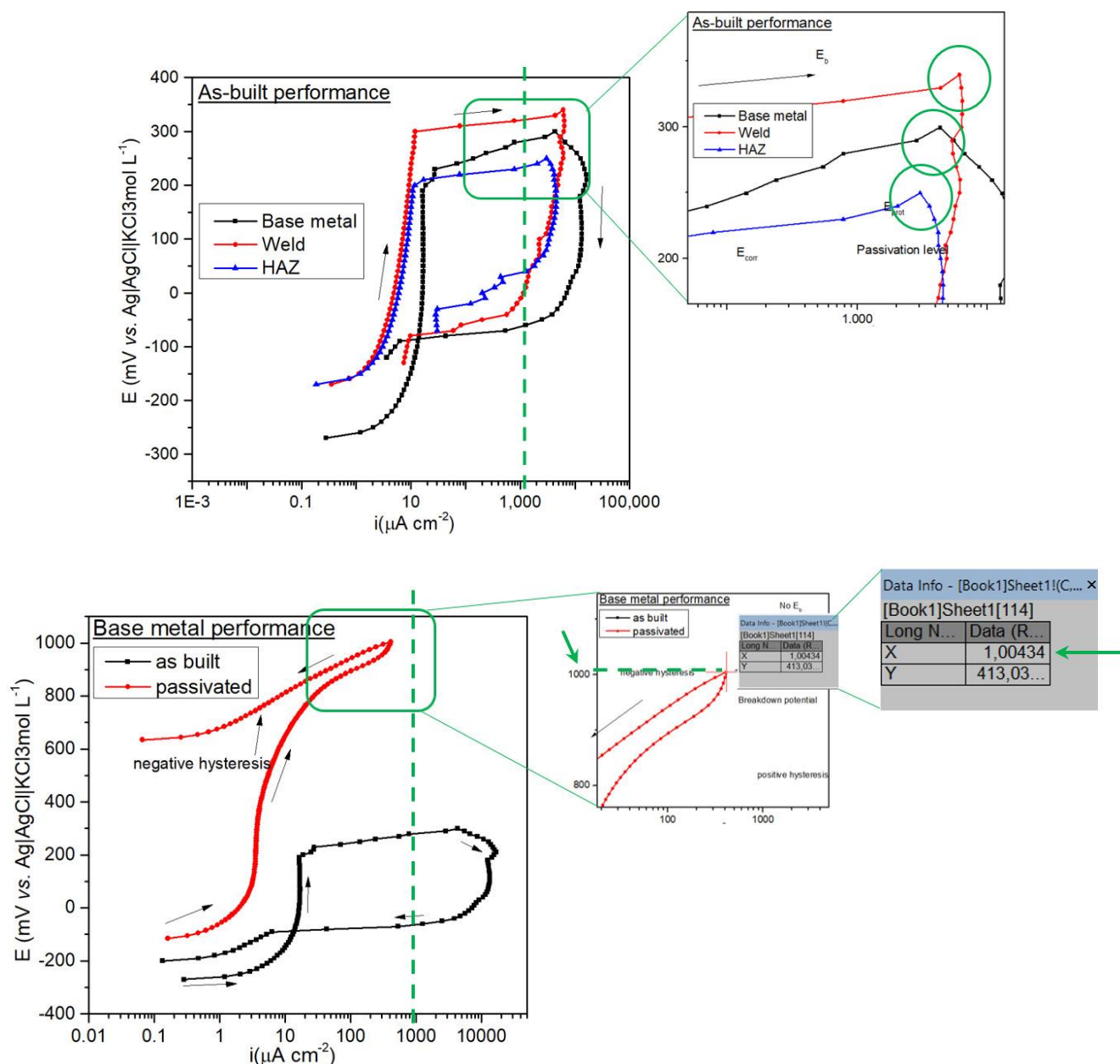
Not applicable.

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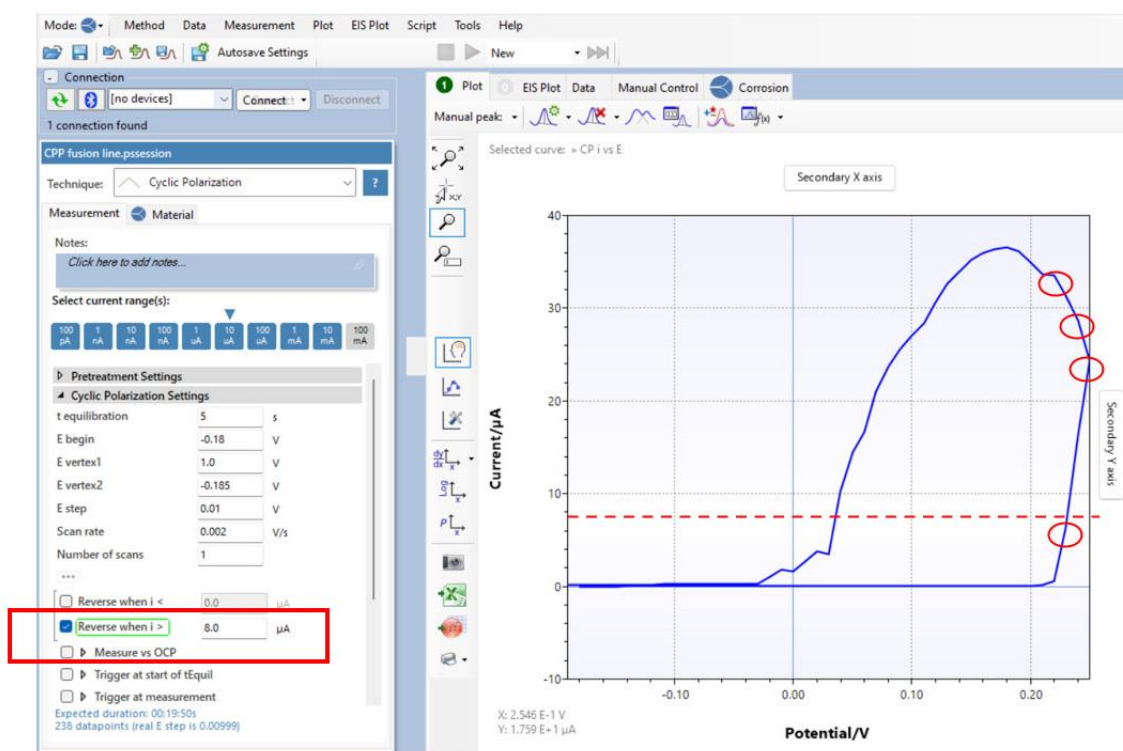
## Appendix

To clarify the current limit of  $1 \text{ mA cm}^{-2}$ , the curves were plotted as line+symbol and each symbol refers to individual measurements during the tests (Fig. A1). It is important to emphasize that the current limit was  $1,000 \text{ mA cm}^{-2}$  (it corresponds to limit the current to  $8 \text{ }\mu\text{A}$  in the data set of the PalmSens Pstrace software), however, when the sample reached and exceeded this value normally the material performance increased the current density a little bit more before beginning the decrease of current density. The green line represents the limit of the current density, and the circles show the last measurements before exceeding the limit.



**Figure A1.** CPP curves showing the limit criterion to reverse the potential scan in current density of  $1 \text{ mA cm}^{-2}$  (attained before achieving 1 V) or potential of 1 V versus reference electrode (attained before achieving  $1 \text{ mA cm}^{-2}$ ).

In Fig. A2 it is possible to observe the PalmSens Pstrace software screen where it is showed the CPP curve in axis of current vs. potential and in the left it is possible to see the current limit criterion (8  $\mu\text{A}$ ).



**Figure A2.** Cyclic polarization curve of fusion line showing a graphic of current ( $\mu\text{A}$ ) vs. potential (V) where the scan is reversed at the first point exceeding 8  $\mu\text{A}$ .