

Effect of post-welding sensitization on the degree of sensitization of the welding zones of AISI 304 resistance spot welding joints studied by using an electrochemical minicell

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Abstract

In resistance spot welding (RSW) joints of austenitic stainless steel (ASS), a small-scale electrochemical cell (minicell) was used for assessing individually, on each of the three welding zones, of size less than 1000 μm (fusion zone (FZ), heat affected zone (HAZ), and base metal (BM)), the combined effect of a RSW process and post-welding sensitization on the degree of sensitization (DOS). The results show that the three welding zones have different microstructures that make each of them respond differently to post-welding sensitization. The DOS varies with post-welding sensitization time in all three welding zones, but it varies at a different rate in each welding zone (the highest rate in the FZ). This variation is due to the fact that when the DOS reaches a certain level, which is observed when plotting the reactivation charge (Q_r) versus the post-welding sensitization time, a microstructural regeneration occurs. The observation that the BM of the RSW joint without post-welding sensitization has no reactivation confirms that reactivation is only possible when the DOS reaches a certain threshold.

Keywords

Resistance spot welding process; Austenitic stainless steel; Degree of sensitization; Electrochemical minicell

1. Introduction

Resistance spot welding (RSW) is widely employed for joining metal sheets in the manufacturing of automobiles, trucks trailers, buses, recreational vehicles, office furniture and appliances, railway vehicles, airplane structures and also for aeronautical and space applications [1]. In the automotive industry, RSW, because of its low cost, high speed and high degree of adaptability for automation in mass production, is the predominant process in sheet metal joining [2–9].

Austenitic stainless steels (ASSs), in view of their good combination of (i) energy-absorption ability, due to a face-centered-cubic lattice structure of austenite stabilized at room temperature by nickel alloy additions, and (ii) corrosion resistance, due to chromium alloy additions that allow the formation of a protective layer based on chromium oxide, can be considered as an interesting option for vehicle body manufacturing in the automotive industry [10,11]. Andersson et al. [12] pointed out that crash performance and energy absorbing capability of a specific component in automotive vehicles is a combination of geometry and material properties and that, since ASSs combine excellent formability with high strength, can be press-formed into components with high stiffness and high energy absorbing capability. Capelli et al. [13] and Friesen and Cunat [14] agreed that ASSs combine high strain rate sensitivity with high formability capability (that, in turn, allows straighter designs), which promotes crushing instead of buckling and, consequently, improves the energy absorption capability. The NGV (Next Generation Vehicle) Project showed that ASSs can be used to improve safety and sustainability in structural automotive systems while reducing both weight and costs [15]. In addition to the automotive industry, RSW of ASS, given its good performance, is extensively used in metro and railway car body manufacturing [16–18].

The heat associated with welding processes may cause, in ASSs, a phenomenon known as sensitization which leads to precipitation, along critical zones, of chromium-rich phases whose adjacent zones are then chromium depleted zones and therefore more susceptible to corrosion attack [19,20]. RSW has the advantage over other welding processes of having rapid heating/cooling cycles [3,21,22], which limits the thermal exposure time and, therefore, can reduce the formation of the aforementioned chromium-rich phases [23,24].

Even though the degree of sensitization (DOS) associated with RSW process is low [25], the RSW joint may be subjected to high temperature service conditions [15,26] where the temperature range in which sensitization occurs is reached and, hence, it is necessary to assess the combined effect of RSW process and post-welding sensitization, induced by subsequent service conditions, on the DOS [25]. In addition, the effect of corrosion can be critical in an already small-sized welded joint such as a RSW joint [27].

Electrochemical potentiokinetic reactivation (EPR) is a quasi non-destructive test that is used mainly to assess the DOS, and which is more sensitive, faster, and more precise than the conventional corrosion tests [28,29].

RSW is a fusion welding process, in which a weld nugget is formed from the solidification of the molten metal after a heating by Joule effect [30]. Thus, as a result of the process, three weld zones can be distinguished in the RSW joint [31]: (i) fusion zone (FZ), also called weld nugget; (ii) heat affected zone (HAZ); and (iii) base metal (BM). Since the maximum dimensions of the FZ are limited by the face diameter of the contact electrodes (i.e., by the electrode-sheet contact area) and by the thickness of the overlapped sheets, and since the HAZ is narrow due to the fact that RSW is a low heat input process compared to other welding processes [32,33] such as gas metal arc welding (GMAW) process [34], and that ASSs have low thermal conductivity [35] compared to carbon steel [36,37], a conventional large-scale EPR test may not be able to isolate specific results from each of the three small-sized individual zones of the RSW joint.

The majority of microelectrochemical methods employed to study localized corrosion on small areas [38–42] use microcapillary-based droplet cells that have the advantage of the small size (in the micrometer range) of the exposed working area [43–45]. These capillary microcells have some drawbacks [46] and in some cases it is not required to use areas as small as those provided by microcapillaries [47]. Thus, ad hoc small-scale electrochemical cell known as “minicell”, different to the capillary-based microcell and which has been successfully utilized in the study of welded joints of ASSs [47–50], can be applied to RSW joints given that it has study sizes in the range of 200–1000 μm [49].

In this work, a small-scale electrochemical cell (minicell) was used for assessing individually, on each of the three small-sized zones of the welded joint, the combined effect of RSW process and post-welding sensitization, induced by subsequent service conditions, on the DOS, with the aim of overcoming the limitations of employing large-scale EPR tests for assessing the DOS in small-sized welding zones.

2. Experimental Procedure

2.1. Materials

Table 1 shows the chemical composition of the AISI 304 ASS sheets (with 0.8 mm thickness) welded by RSW process and Table 2 summarizes their mechanical properties.

Table 1. Chemical composition of the AISI 304 ASS sheets (wt. %).

C	S	P	Al	Co	W	Si	Mn
0.08	0.002	0.019	0.003	0.17	0.03	0.426	1.153
Ti	V	Cu	Nb	Mo	Cr	Ni	Fe
0.004	0.05	0.39	0.02	0.36	18.03	8.74	Bal.

Table 2. Mechanical properties of the AISI 304 ASS sheets.

Tensile strength (MPa)	Yield strength (MPa)	Microhardness (HV, 100g)	Total elongation (%)
675	290	162	70

2.2. RSW procedure

The AISI 304 ASS sheets were welded with a single-phase alternating current (AC) 50 Hz equipment by using water-cooled truncated cone Resistance Welding Manufacturing Alliance (RWMA) Group A Class 2 electrodes with 4.5 mm face diameter [51]. The controlled parameters in RSW process were welding current (fixed at 5 kA), welding time (fixed at 0.20 s), and electrode force (fixed at 1500 N).

As a result of the fusion welding process, the RSW joint has three small-sized welding zones with different microstructures that can respond in different ways to post-welding sensitization: (i) FZ (weld nugget), which is a cast dendritic microstructure of austenite with delta-ferrite in interdendritic regions (Fig. 1); (ii) HAZ, whose microstructure depends on the heat generated in the RSW process which may remove signs of prior cold work (Fig. 2); and (iii) BM, which is not affected by the welding heat input, and shows an austenitic structure with signs of prior cold work such as delta-ferrite bands oriented in the rolling direction (Fig. 3) [19,25].

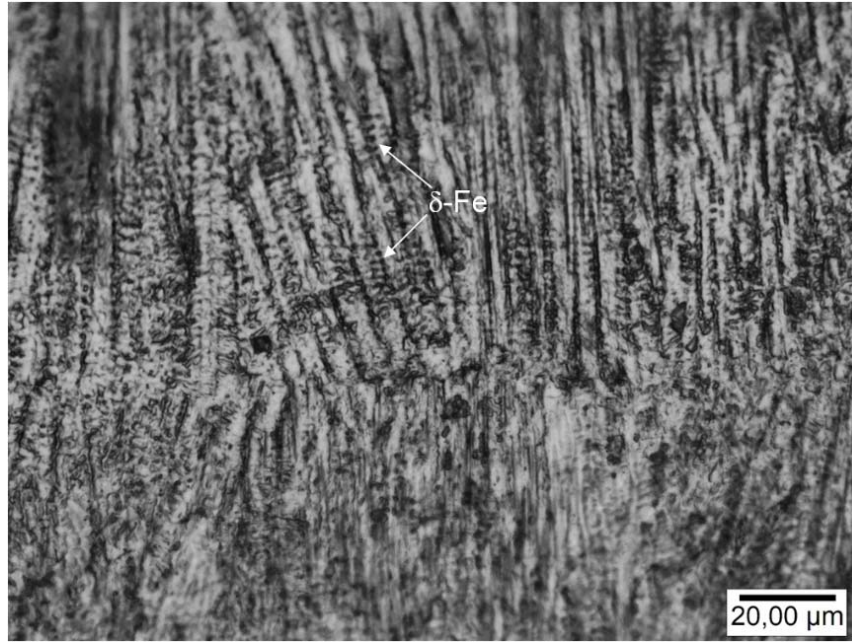


Fig. 1. Optical micrograph of the FZ of a RSW joint (without post-welding sensitization), which has a cast dendritic microstructure of austenite with delta-ferrite (δ -Fe) in interdendritic regions (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A) [52].

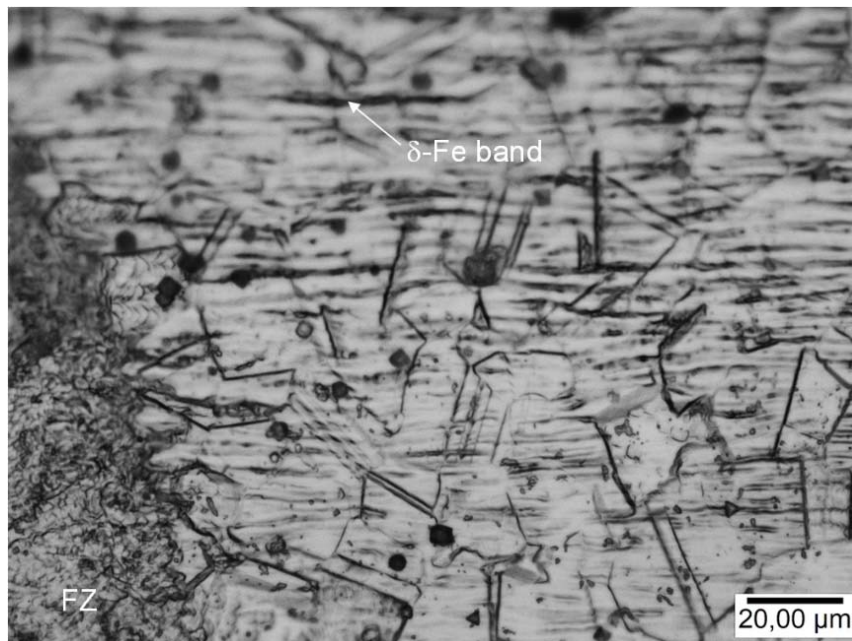


Fig. 2. Optical micrograph of the HAZ of a RSW joint (without post-welding sensitization), in which signs of prior cold work (delta-ferrite (δ -Fe) bands oriented in the rolling direction) are partially removed by the effect of the welding heat input (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A) [52].

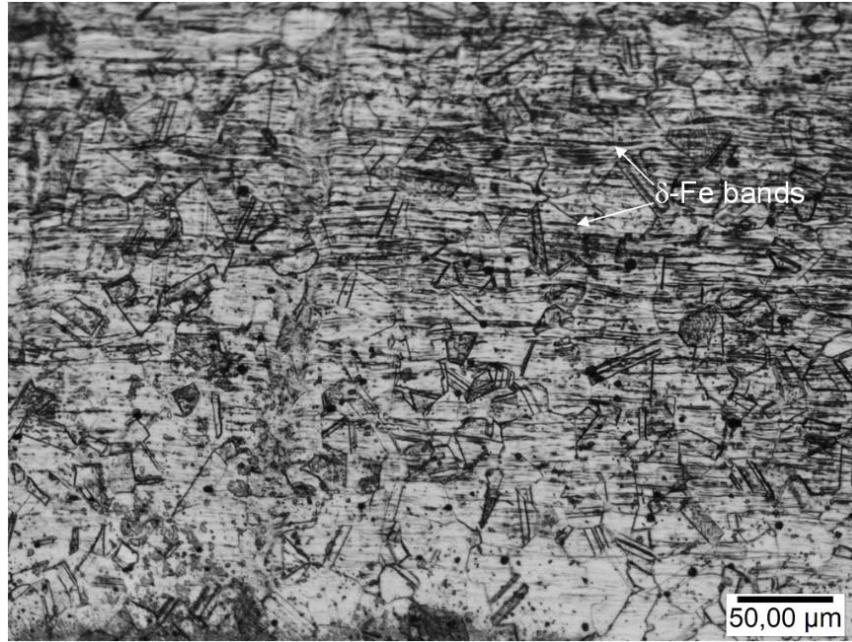


Fig. 3. Optical micrograph of the BM of a RSW joint (without post-welding sensitization), which shows an austenitic structure with signs of prior cold work such as delta-ferrite (δ -Fe) bands oriented in the rolling direction (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A) [52].

2.3. Post-welding sensitization procedure

Each of the twelve post-welding sensitization conditions [25] were applied on a different RSW joint, considering the RSW joint without post-weld sensitization as the reference state. Thus, a sample was obtained for each state: sample No. 0 corresponds to the RSW joint without post-weld sensitization and samples Nos. 1 to 12 correspond to RSW joints subjected to different post-weld sensitization conditions (Table 3).

Table 3. The twelve different states of RSW + post-welding sensitization compared to reference state. A sample was obtained for each state and three small-scale EPR tests were performed, by using a minicell, on each of the samples (one small-scale EPR test on each of the welding zones, i.e., FZ, HAZ and BM).

RSW condition	Post-welding sensitization conditions		Sample No.
	Temperature (°C)	Time (min)	
Welding current = 5 kA Welding time = 0.20 s Electrode force = 1500 N	--	--	0 ^a
	675	15	1
		30	2
		45	3
		60	4
		120	5
		240	6
	750	15	7
		30	8
		45	9
		60	10
		120	11
		240	12

^a Sample No. 0 corresponds to the reference state and has no post-welding sensitization. FZ, HAZ and BM shown respectively in Fig.1, Fig. 2, and Fig. 3, were obtained from sample No. 0.

2.4. Small-scale EPR test

2.4.1. Minicell set-up

The sample **to be studied** was clamped on a micrometer table. Thus, it was possible to move the sample to the desired position with respect to the tip of the **minicell, so** that the tip was located exactly on top of the weld zone to be studied in each case (Fig. 4A).

The minicell used (Fig. 4B) was made in polymethyl methacrylate (PMMA). It **consisted** of two parts: the lid and the body. The electrolyte flowed in the minicell through the upper inlet (5 mm in diameter) and left through the lower outlet (1 mm in diameter). Thus, the reduction in section **led** to a suction effect that continuously **renovated** the chemical species of the working area and **allowed** a continuous flow of fresh electrolyte over the working electrode area, and the disturbing phenomenon of possible

bubbling **was** then lessened. The platinum counter electrode (0.2 mm wire) **was** positioned between the working and the reference electrode. The same reference electrode as for large-scale experiments **was** used (Saturated Calomel Electrode (SCE)), and **was** located at a distance (ca. 9 mm) **from** the working electrode (this positioning is aimed at obtaining the best possible electrical signals) [47,49,50].

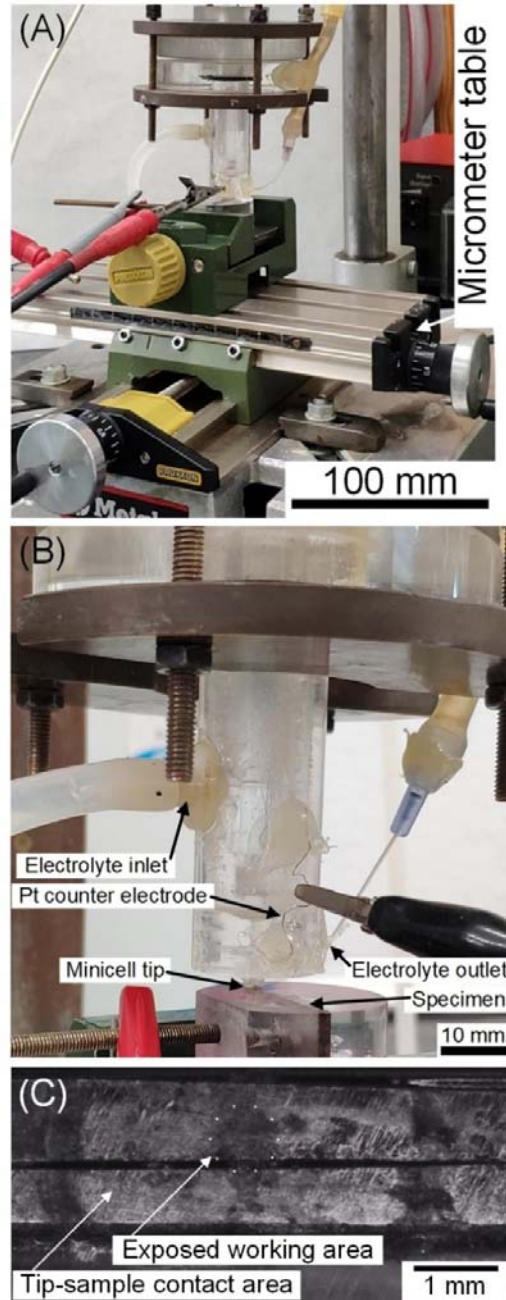


Fig. 4. (A) Micrometer table on which the sample was clamped. (B) Minicell used in the small-scale EPR tests. (C) Optical macrograph taken in the BM of a RSW joint after a small-scale EPR test to show the size of the exposed working area.

2.4.2. Small-scale EPR test procedure

EPR test was performed following ASTM Standard G-108 [53] but, given that the results were found to be dependent on the degree of surface preparation, an electrochemical conditioning, which allows a degree of surface preparation (No. 600 emery paper) less severe than the established by the aforementioned standard, was conducted [47]. The electrolyte was 0.5 M H₂SO₄ + 0.01 M KSCN and the test temperature is 30 °C ± 1. The experimental procedure of the test was the following: a **holding time** of 5 min at open circuit (OC) potential, deaerated, an anodic attack at –220 mV(SCE) for 2 min, a **holding time** of 2 min at VOC, a cathodic cleaning at –600 mV(SCE) for 1 min and a **holding time** of 5 min at VOC. Passivation was carried out by applying 200 mV(SCE) for 2 min (a longer time would give rise to a more stable passive layer, which would prevent certain EPR curves from having a passive zone where the current density is not exactly constant, but it would also lead to a lack of reactivation in other EPR curves). The reactivation scan starts at 200 mV until 100 mV below the VOC at a rate of 100 mV/min.

As shown above (Fig 4C), the exposed working area studied in each of the small-scale EPR tests has a value of approximately 0.005 cm² (ca. 0.8 mm in diameter).

Three small-scale EPR tests were conducted, by using the minicell, on each of the thirteen samples Nos. 0 to 12 (one small-scale EPR test on each of the welding zones, i.e., FZ, HAZ and BM) (Fig. 5A). The repeatability of the tests was assessed by repeating the small-scale EPR test several times in the same welding zone and verifying that the differences between the EPR curves obtained were not significant (Fig. 5B).

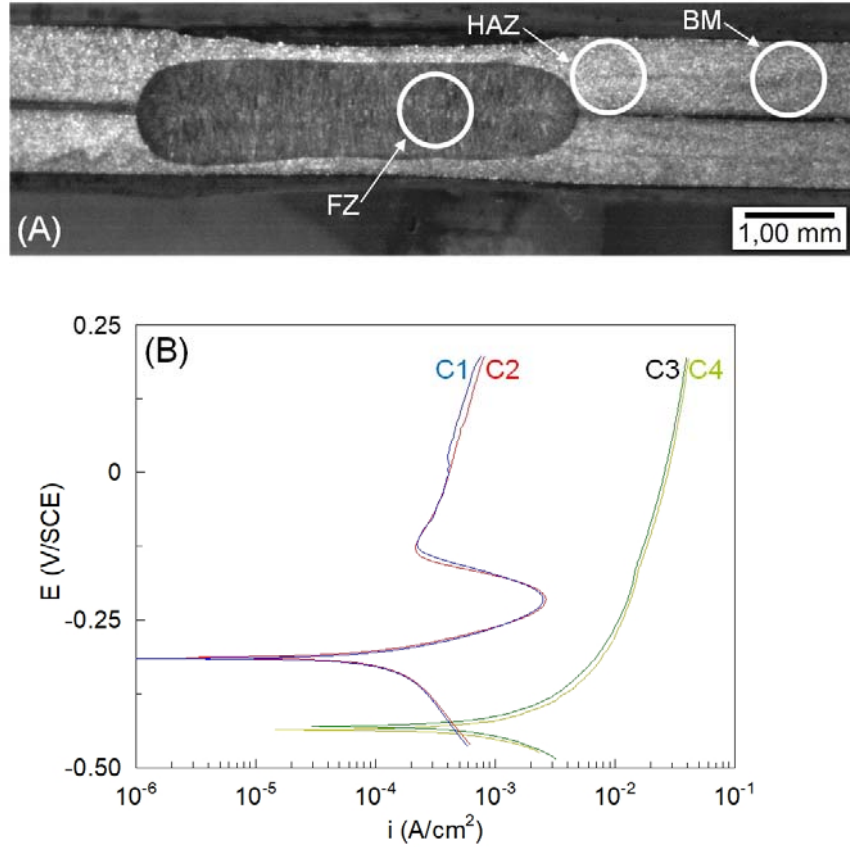


Fig. 5. (A) Location of the small-scale EPR tests in each of the welding zones. The exposed working area is indicated by a circle 0.005 cm² in area and 0.8 mm in diameter. in the small-scale EPR tests. The RSW joint shown corresponds to sample No. 10 (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A) [52]. (B) Assessment of repeatability of the small-scale EPR tests: curves obtained from the BM in samples No. 1 (C1 and C2) and No. 0 (C3 and C4).

The EPR test parameter Q_r (reactivation charge) (Fig. 6), given its correspondence with the DOS [54], is used to assess such a DOS [55].

Certain EPR small-scale EPR curves show local increases of current density that may be due to the **formation** of crevice corrosion at the interface between the minicell tip and the sample, but this phenomenon occurs mainly in the passive zone of the EPR curve, outside the reactivation hump (Figs. 9C, 10D and 12B) and also, as pointed out by De Tiedra [56], does not affect significantly the reactivation charge Q_r (Fig. 6).

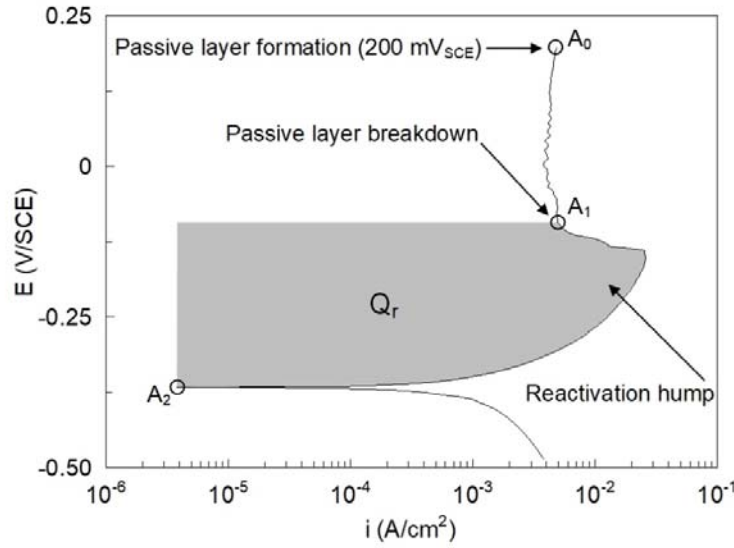


Fig. 6. In each EPR curve, the reactivation charge Q_r is determined between point A_1 and point A_2 (reactivation hump). Passive layer formation occurs at 200 mV_{sce} (A_0) and the passive layer (passive zone between A_0 and A_1) limits the flow of current until its localized breakdown (A_1) causes the current density to increase significantly. The small-scale EPR curve shown is obtained from the FZ of sample No. 11.

3. Results and Discussion

The results obtained from the small-scale EPR curves (Figs. 7-12) are analyzed in terms of the Q_r parameter (Figs. 13 and 14) [55]. As shown in Fig. 6, the Q_r parameter increases as the current density through the passive layer increases, which occurs when the passive layer is broken or weakened at the zones where chromium-rich phases precipitate.

The DOS values are, in general, higher in the post-welding sensitization at higher temperature (750 °C) [57] (Figs. 13 and 14), which is consistent with the results of Yu et al. [58]. According to these authors, DOS increases as sensitization temperature increases, due to the fact that the rate of chromium diffusion increases when the temperature increases.

As it can be seen in Figs. 7-14, the DOS experiences a variation with post-welding sensitization time which is due to the fact that when the DOS reaches a certain level, a microstructural regeneration, during which the DOS decreases with sensitization time, takes place [49,50]. After this microstructural regeneration, the microstructure undergoes a further increase in DOS with sensitization time until the level at which microstructural regeneration occurs is reached again. These results are consistent with those of Matula et al. [55], according to which the EPR test is capable of detecting modifications in the chromium distribution, not only the depletion but also the rehomogenization [55].

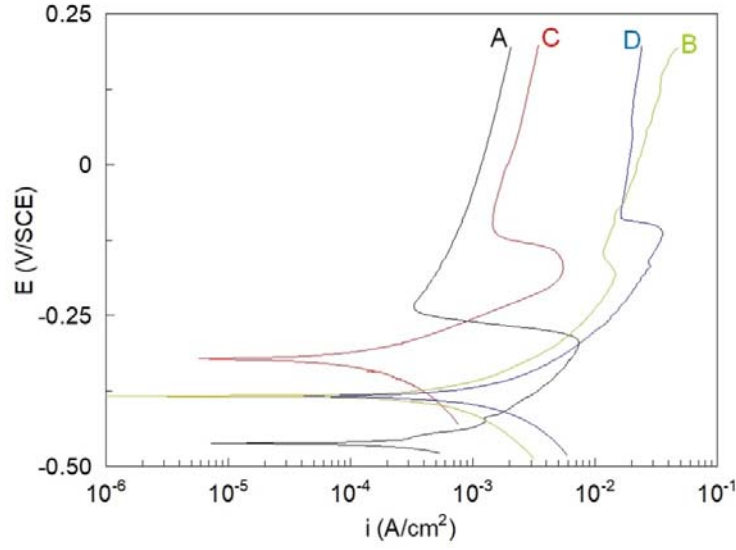


Fig. 7. Small-scale EPR curves obtained from the FZ of: (A) sample No. 0 ($Q_r = 0.52144 \text{ C/cm}^2$); (B) sample No. 1 ($Q_r = 1.2165 \text{ C/cm}^2$); (C) sample No. 4 ($Q_r = 0.37101 \text{ C/cm}^2$); and (D) sample No. 6 ($Q_r = 3.4622 \text{ C/cm}^2$).

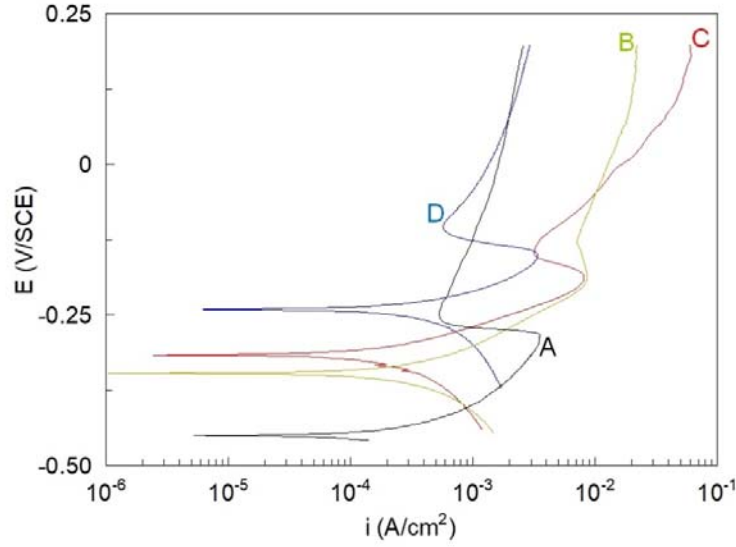


Fig. 8. Small-scale EPR curves obtained from the HAZ of: (A) sample No. 0 ($Q_r = 0.23861 \text{ C/cm}^2$); (B) sample No. 1 ($Q_r = 0.69272 \text{ C/cm}^2$); (C) sample No. 4 ($Q_r = 0.41732 \text{ C/cm}^2$); and (D) sample No. 6 ($Q_r = 0.1602 \text{ C/cm}^2$).

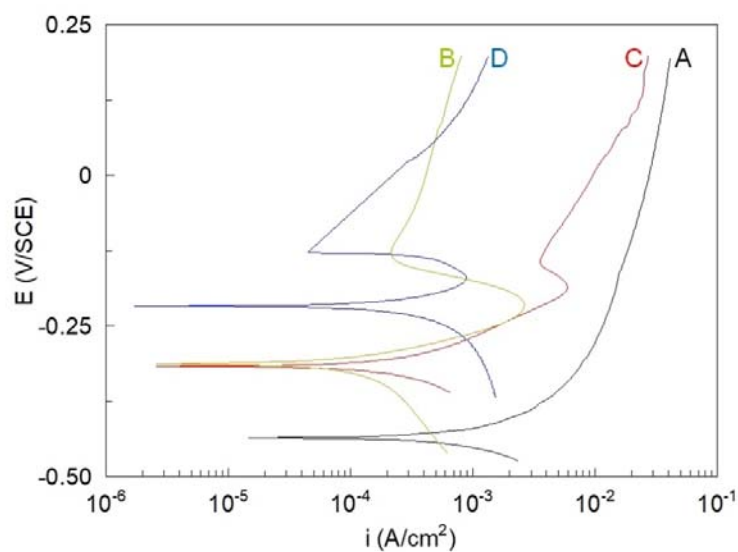


Fig. 9. Small-scale EPR curves obtained from the BM of: (A) sample No. 0 ($Q_r = 0 C/cm^2$, i.e., there is no reactivation); (B) sample No. 1 ($Q_r = 0.14747 C/cm^2$); (C) sample No. 4 ($Q_r = 0.32757 C/cm^2$); and (D) sample No. 6 ($Q_r = 0.03397 C/cm^2$).

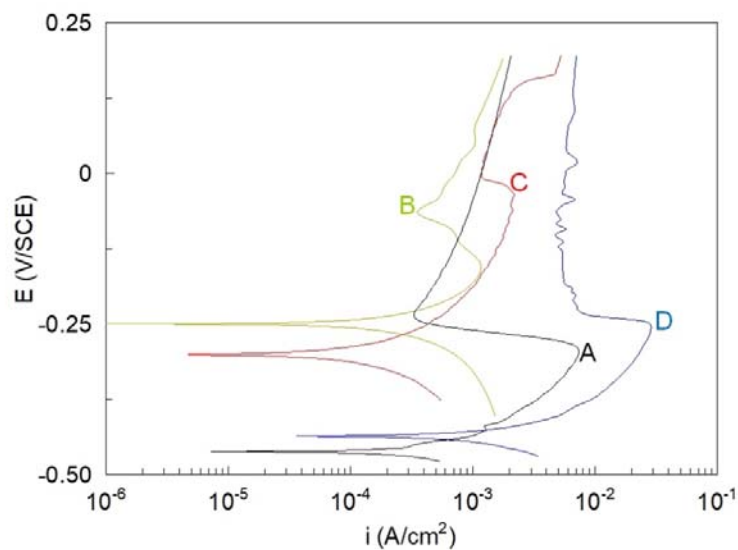


Fig. 10. Small-scale EPR curves obtained from the FZ of: (A) sample No. 0 ($Q_r = 0.52144 C/cm^2$); (B) sample No. 7 ($Q_r = 0.094963 C/cm^2$); (C) sample No. 10 ($Q_r = 0.25427 C/cm^2$); and (D) sample No. 12 ($Q_r = 2.1729 C/cm^2$).

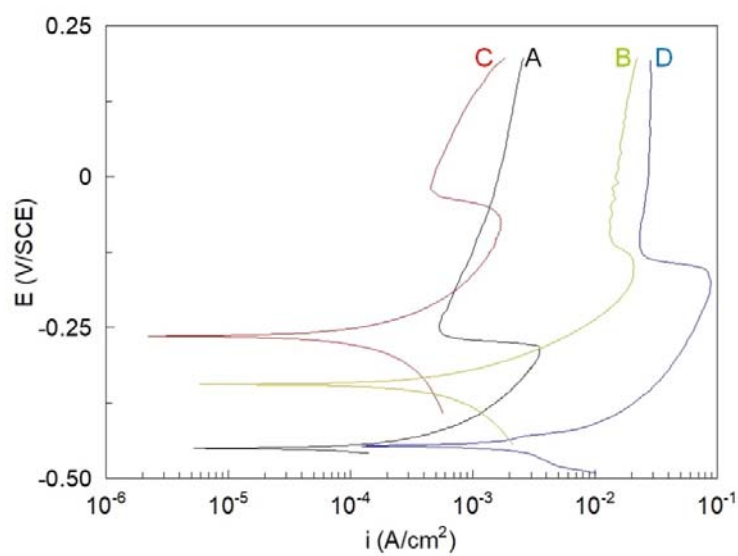


Fig. 11. Small-scale EPR curves obtained from the HAZ of: (A) sample No. 0 ($Q_r = 0.23861 \text{ C/cm}^2$); (B) sample No. 7 ($Q_r = 1.9035 \text{ C/cm}^2$); (C) sample No. 10 ($Q_r = 0.16359 \text{ C/cm}^2$); and (D) sample No. 12 ($Q_r = 10.345 \text{ C/cm}^2$).

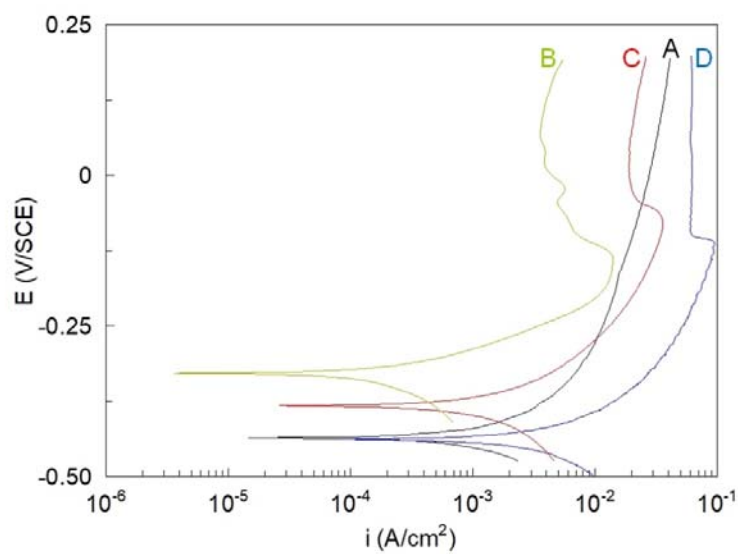


Fig. 12. Small-scale EPR curves obtained from the BM of: (A) sample No. 0 ($Q_r = 0 \text{ C/cm}^2$, i.e., there is no reactivation); (B) sample No. 7 ($Q_r = 1.1744 \text{ C/cm}^2$); (C) sample No. 10 ($Q_r = 4.2562 \text{ C/cm}^2$); and (D) sample No. 12 ($Q_r = 11.143 \text{ C/cm}^2$).

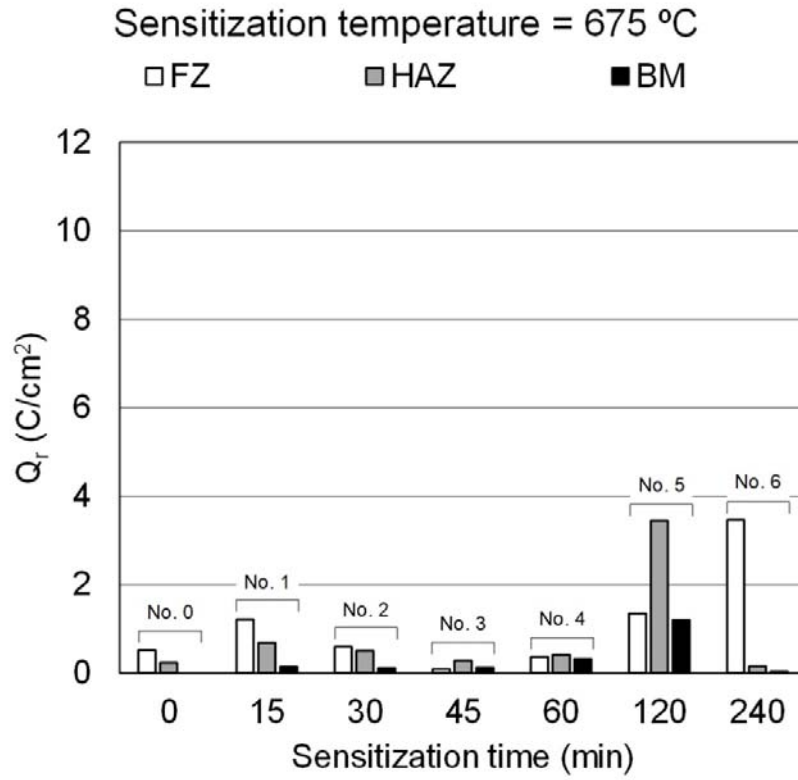


Fig. 13. Reactivation charge, Q_r (C/cm²), obtained from each of the welding zones (FZ, HAZ and BM) of the RSW joint without post-weld sensitization (sample No. 0, which has no reactivation in the BM, i.e., with $Q_r = 0$ C/cm²) and of the six RSW joints subjected to post-weld sensitization at 675 °C (samples Nos. 1 to 6).

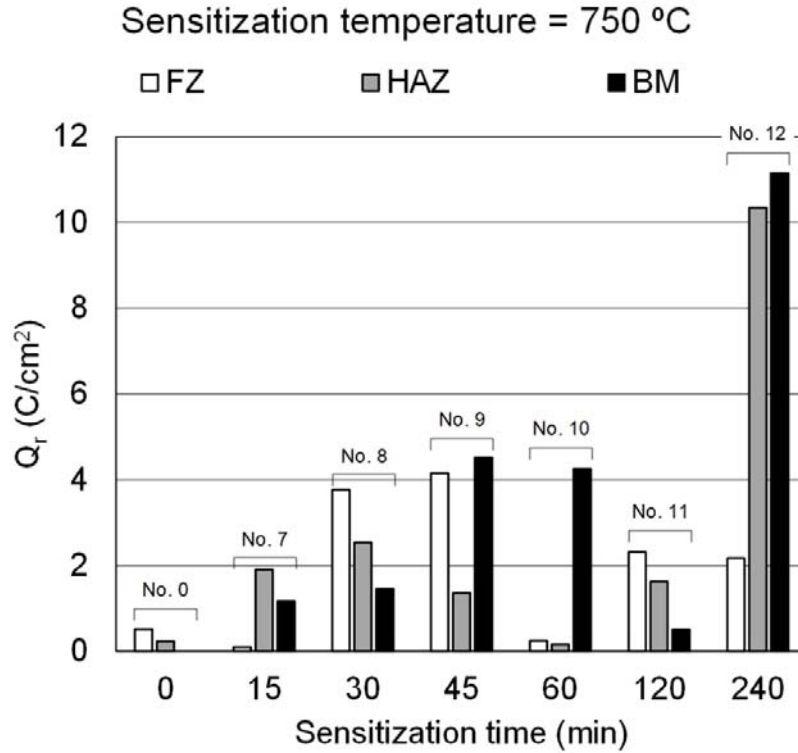


Fig. 14. Reactivation charge, Q_r (C/cm^2), obtained from each of the welding zones (FZ, HAZ and BM) of the RSW joint without post-weld sensitization (sample No. 0, which has no reactivation in the BM, i.e., with $Q_r = 0$ C/cm^2) and of the six RSW joints subjected to post-weld sensitization at 750 °C (samples Nos. 7 to 12).

In FZ, the post-welding sensitization causes the precipitation of chromium (ferrite-stabilizing element) rich phases along dendritic boundaries [19] (Fig. 17), which have a high content of delta-ferrite due to the rapid cooling [59,60] associated with the RSW process [61,62]. This type of sensitization is associated with interdendritic corrosion (IDC). The variation, of the DOS in FZ, with post-weld sensitization time, shown in Figs. 13 and 14, is in accordance with that of the IDC, shown in the Figs. 1, 15 (A) to (C) and 16 (A) to (C).

In both HAZ and BM, the post-welding sensitization causes the precipitation of chromium-rich phases [19,20,35,63–65], which takes place along: (i) austenite/delta-ferrite interfaces, twins and slip bands inside austenitic grains (this type of sensitization is associated with transgranular corrosion (TGC)); and (ii) and grain boundaries (this type of sensitization is associated with intergranular corrosion (IGC)). The variation, of the DOS in HAZ and BM, with post-weld sensitization time, shown in Figs. 13 and 14, is in accordance with that of the TGC and IGC, shown in Figs. 2, 15 (D) to (F) and 16 (D) to (F) for HAZ and in Figs. 3, 15 (G) to (I) and 16 (G) to (I) for BM. It is also observed that, in both HAZ and BM, the variation over sensitization time is more significant for TGC than for IGC [25].

As it can be seen in Figs. 13 to 16, the fact that the three welding zones (FZ, HAZ and BM) have different microstructures makes each of them respond differently to post-welding sensitization:

although the DOS varies with sensitization time in all three welding zones, it varies at a different rate in each welding zone.

As it can be observed in Figs. 13 and 14, the variation rate of the DOS with post-welding sensitization time is higher in the FZ than in HAZ and BM. This phenomenon is caused by the fact that the kinetics of microstructural regeneration is faster in FZ than in HAZ and BM [25,66], which may be due to two causes. Firstly, the chemical segregation associated with the solidification processes [67,68], where the last fraction of liquid that solidifies enriches in ferrite-stabilizing elements such as chromium, and thus, the interdendritic regions can be expected to be zones for preferential delta-ferrite formation [69,70] (this chemical segregation makes the rate of diffusion higher since concentration gradient is, according to Fick's law, the driving force of diffusion [71]). Secondly, diffusion takes place faster along grain boundaries than into the grain [72,73] and, as it can be seen in Figs. 1, 2, 3, 15 and 16, grain boundary area is larger in the dendritic-grain microstructure of FZ than in the equiaxed-grain microstructure of HAZ and BM.

Finally, the fact that BM (without thermal effects due to welding process) of sample No. 0 (without post-welding sensitization) has no reactivation (EPR curve A in Fig. 9 and Fig. 12) confirms that the lack of reactivation occurs when the DOS does not reach a certain reactivation threshold [19].

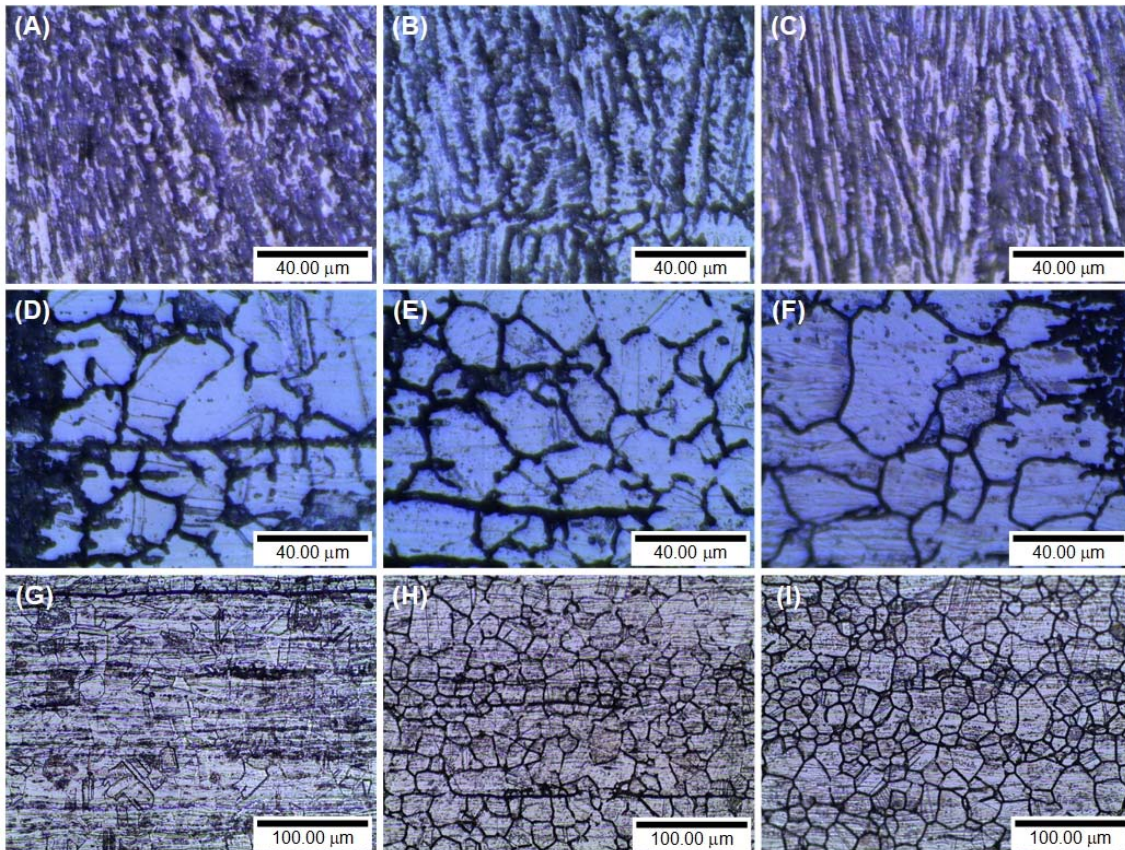


Fig. 15. Optical micrographs (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A [52]) of: (A) FZ of sample No. 1; (B) FZ of sample No. 4; (C) FZ of sample No. 6; (D) HAZ of sample No. 1. (E) HAZ of sample No. 4; (F) HAZ of sample No. 6; (G) BM of sample No. 1; (H) BM of sample No. 4; (C) BM of sample No. 6.

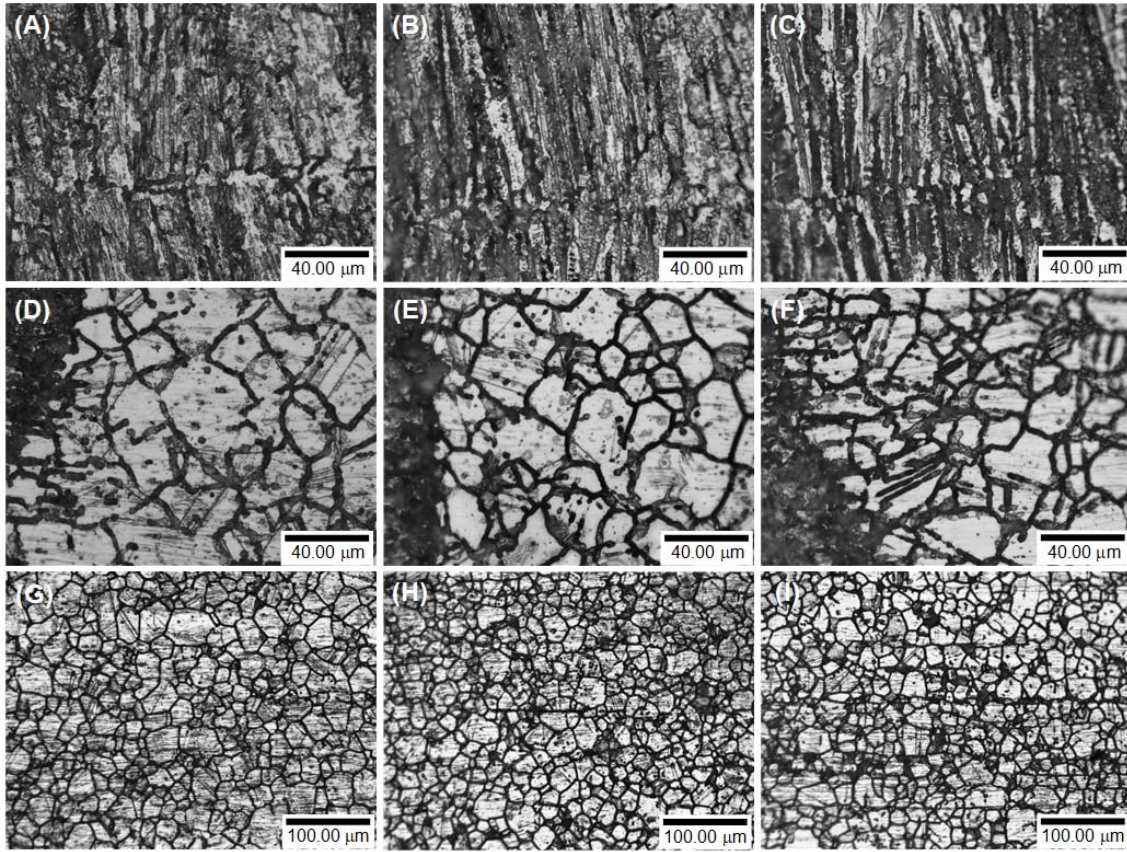


Fig. 16. Optical micrographs (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A [52]) of: (A) FZ of sample No. 7; (B) FZ of sample No. 10; (C) FZ of sample No. 12; (D) HAZ of sample No. 7. (E) HAZ of sample No. 10; (F) HAZ of sample No. 12; (G) BM of sample No. 7; (H) BM of sample No. 10; (C) BM of sample No. 12.

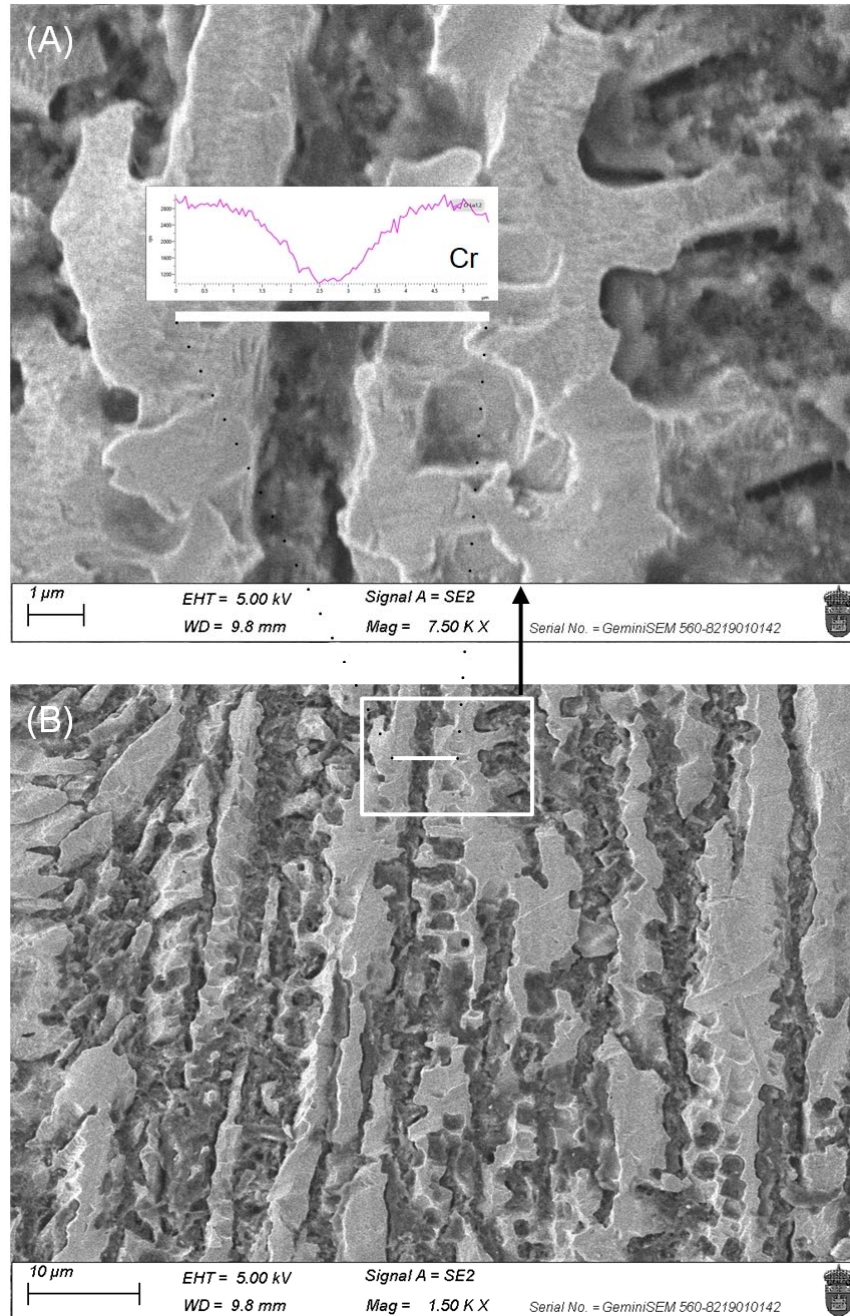


Fig. 17. (A) EDX line scan between two dendrites for Cr content in the FZ of sample No. 12; although the electrolytic etching with oxalic acid, according to ASTM A262-15(2021), dissolves the chromium-rich phases that had precipitated along interdendritic regions, the chromium depleted zones, adjacent to the chromium-rich phases, are shown. (B) SEM micrograph of the line scan location.

Conclusions

In this work, a small-scale electrochemical cell (minicell) is used for assessing individually, on each of the three small-sized zones (of size less than 1000 µm) of the welded joint, the combined effect of RSW process and post-welding sensitization, induced by subsequent service conditions, on the DOS. The **main** conclusions are:

- The three weld zones (FZ, HAZ and BM) in the RSW joint have different microstructures that makes each of them respond differently to post-welding sensitization: although the DOS varies with post-welding sensitization time in all three weld zones, it varies at a different rate in each weld zone.
- The variation of the DOS with post-welding sensitization time observed is due to the change when the DOS reaches a certain level (which can be observed when plotting the reactivation charge (Q_r) versus the post-welding sensitization time) and a microstructural regeneration occurs.
- The variation rate of the DOS with post-welding sensitization time is higher in the FZ than in HAZ and BM. This phenomenon may be due to kinetics of microstructural regeneration are faster in FZ than in HAZ and BM.
- The base metal (without thermal effects due to welding process or post-welding sensitization) has no reactivation appears to confirm that reactivation of the RSW weld microstructures only occurs when the DOS reaches a certain reactivation threshold.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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Figure captions

Fig. 1. Optical micrograph of the FZ of a RSW joint (without post-welding sensitization), which has a cast dendritic microstructure of austenite with delta-ferrite (δ -Fe) in interdendritic regions (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A) [52].

Fig. 2. Optical micrograph of the HAZ of a RSW joint (without post-welding sensitization), in which signs of prior cold work (delta-ferrite (δ -Fe) bands oriented in the rolling direction) are partially removed by the effect of the welding heat input (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A) [52].

Fig. 3. Optical micrograph of the BM of a RSW joint (without post-welding sensitization), which shows an austenitic structure with signs of prior cold work such as delta-ferrite (δ -Fe) bands oriented in the rolling direction (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A) [52].

Fig. 4. (A) Micrometer table on which the sample was clamped. (B) Minicell used in the small-scale EPR tests. (C) Optical macrograph taken in the BM of a RSW joint after a small-scale EPR test to show the size of the exposed working area.

Fig. 5. (A) Location of the small-scale EPR tests in each of the welding zones. The exposed working area is indicated by a circle 0.005 cm² in area and 0.8 mm in diameter. in the small-scale EPR tests. The RSW joint shown corresponds to sample No. 10 (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A) [52]. (B) Assessment of repeatability of the small-scale EPR tests: curves obtained from the BM in samples No. 1 (C1 and C2) and No. 0 (C3 and C4).

Fig. 6. In each EPR curve, the reactivation charge Q_r is determined between point A₁ and point A₂ (reactivation hump). Passive layer formation occurs at 200 mV_{sec} (A₀) and the passive layer (passive zone between A₀ and A₁) limits the flow of current until its localized breakdown (A₁) causes the current density to increase significantly. The small-scale EPR curve shown is obtained from the FZ of sample No. 11.

Fig. 7. Small-scale EPR curves obtained from the FZ of: (A) sample No. 0 ($Q_r = 0.52144$ C/cm²); (B) sample No. 1 ($Q_r = 1.2165$ C/cm²); (C) sample No. 4 ($Q_r = 0.37101$ C/cm²); and (D) sample No. 6 ($Q_r = 3.4622$ C/cm²).

Fig. 8. Small-scale EPR curves obtained from the HAZ of: (A) sample No. 0 ($Q_r = 0.23861$ C/cm²); (B) sample No. 1 ($Q_r = 0.69272$ C/cm²); (C) sample No. 4 ($Q_r = 0.41732$ C/cm²); and (D) sample No. 6 ($Q_r = 0.1602$ C/cm²).

Fig. 9. Small-scale EPR curves obtained from the BM of: (A) sample No. 0 ($Q_r = 0$ C/cm², i.e., there is no reactivation); (B) sample No. 1 ($Q_r = 0.14747$ C/cm²); (C) sample No. 4 ($Q_r = 0.32757$ C/cm²); and (D) sample No. 6 ($Q_r = 0.03397$ C/cm²).

Fig. 10. Small-scale EPR curves obtained from the FZ of: (A) sample No. 0 ($Q_r = 0.52144$ C/cm²); (B) sample No. 7 ($Q_r = 0.094963$ C/cm²); (C) sample No. 10 ($Q_r = 0.25427$ C/cm²); and (D) sample No. 12 ($Q_r = 2.1729$ C/cm²).

Fig. 11. Small-scale EPR curves obtained from the HAZ of: (A) sample No. 0 ($Q_r = 0.23861$ C/cm²); (B) sample No. 7 ($Q_r = 1.9035$ C/cm²); (C) sample No. 10 ($Q_r = 0.16359$ C/cm²); and (D) sample No. 12 ($Q_r = 10.345$ C/cm²).

Fig. 12. Small-scale EPR curves obtained from the BM of: (A) sample No. 0 ($Q_r = 0$ C/cm², i.e., there is no reactivation); (B) sample No. 7 ($Q_r = 1.1744$ C/cm²); (C) sample No. 10 ($Q_r = 4.2562$ C/cm²); and (D) sample No. 12 ($Q_r = 11.143$ C/cm²).

Fig. 13. Reactivation charge, Q_r (C/cm²), obtained from each of the welding zones (FZ, HAZ and BM) of the RSW joint without post-weld sensitization (sample No. 0, which has no reactivation in the BM, i.e., with $Q_r = 0$ C/cm²) and of the six RSW joints subjected to post-weld sensitization at 675 °C (samples Nos. 1 to 6).

Fig. 14. Reactivation charge, Q_r (C/cm²), obtained from each of the welding zones (FZ, HAZ and BM) of the RSW joint without post-weld sensitization (sample No. 0, which has no reactivation in the BM, i.e., with $Q_r = 0$ C/cm²) and of the six RSW joints subjected to post-weld sensitization at 750 °C (samples Nos. 7 to 12).

Fig. 15. Optical micrographs (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A [52]) of: (A) FZ of sample No. 1; (B) FZ of sample No. 4; (C) FZ of sample No. 6; (D) HAZ of sample No. 1. (E) HAZ of sample No. 4; (F) HAZ of sample No. 6; (G) BM of sample No. 1; (H) BM of sample No. 4; (C) BM of sample No. 6.

Fig. 16. Optical micrographs (electrolytic etching with oxalic acid according to ASTM A262-15(2021) Practice A [52]) of: (A) FZ of sample No. 7; (B) FZ of sample No. 10; (C) FZ of sample No. 12; (D) HAZ of sample No. 7. (E) HAZ of sample No. 10; (F) HAZ of sample No. 12; (G) BM of sample No. 7; (H) BM of sample No. 10; (C) BM of sample No. 12.

Fig. 17. (A) EDX line scan between two dendrites for Cr content in the FZ of sample No. 12; although the electrolytic etching with oxalic acid, according to ASTM A262-15(2021), dissolves the chromium-rich phases that had precipitated along interdendritic regions, the chromium depleted zones, adjacent to the chromium-rich phases, are shown. (B) SEM micrograph of the line scan location.