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A detailed electrochemical analysis of sensitized AISI 410 martensitic stainless steel

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Abstract

In the present work, the effect of sensitization heat treatment on the intergranular corrosion (IGC) behavior of AISI 410 martensitic stainless steel was investigated using potentiodynamic electrochemical impedance spectroscopy (PDEIS) in conjunction with conventional methods including acid etching test, anodic potentiodynamic polarization, double-loop electrochemical potentiodynamic reactivation (DLEPR) and electrochemical noise (EN). Microstructural studies by scanning electron microscopy revealed that increasing tempering time up to 2h would be followed by more severe localized attack, but desensitization state attributed to specimens undergone prolonged tempering cannot be interpreted based on the standard test method. Susceptibility to IGC via DLEPR experiments indicates differences in the degree of sensitization (DOS) of specimens with varying tempering time, revealing a healing effect arising from prolonged tempering. The EN results elucidated the alloy tendency for active dissolution, but no obvious correlation can be established between the EN results and IGC behavior. The PDEIS data clarified that the charge transfer resistance values related to the AC responses in the middle of transpassive potential region can be utilized to investigate and compare the IGC susceptibility of the alloy. Moreover, the surface morphology of corroded samples under such experimental conditions can be used to qualitatively compare the severity of intergranular attack.

1. Introduction

Martensitic stainless steels (MSSs) possess distinctive and attractive characteristics, making them promising candidates for practical applications (akin to plastic molds [1, 2] and medical devices [3, 4]) that require excellent mechanical properties, satisfactory corrosion resistance, high hardness and good wear resistance [5]. In practice, MSSs are austenitized at the temperatures ranging from 970 to 1100 °C and then oil-quenched [6, 7] so as to acquire martensite phase of BCT crystal structure, but the detrimental impact of such a brittle phase on the mechanical performance of the alloy necessitates implementation of a post-heat treatment known as tempering (i.e. heat treating the alloy in the temperature range of 200–700 °C, followed by oil-quenching) [8].

Despite improvement in mechanical characteristics, tempering can deteriorate corrosion properties of MSS due to the precipitation of chromium carbides at martensite lath interfaces (i.e. sensitization) [9]. In fact, the corrosion resistance of stainless steel depends on the quality of passive layer that is formed upon its surface and the formation of carbides at the martensite lath interfaces would result in the formation of defective passive layer which in turn provokes a localized type of attack known as intergranular corrosion (IGC) [6, 8, 10].

Although there are so many standard tests [11–14] and conventional DC electrochemical techniques [15–17] available to evaluate the IGC behavior of different types of stainless steels (SSs), they are not capable to assess the IGC behavior in both quantitative and qualitative ways simultaneously, to say nothing of their drawbacks. For instance, it has been reported that qualitative tests like oxalic acid etch test preferentially dissolve chromium carbides instead of chromium-depleted zones, making it difficult to differentiate between sensitized and
desensitized states [6]. In terms of double-loop electrochemical potentiodynamic reactivation (DLEPR), previous studies [18] suggest that the type of test solution, its temperature and the test procedure (e.g. potential limit and scan rates) serve a prominent role in accuracy and reliability of DLEPR test results. More importantly, it is salient to mention that according to the literature [19], the fine microstructure of stainless steels can contribute to a sharp gradient of Cr within the grain, which can drastically reduce the required width of chromium-depleted regions for the degree of sensitization (DOS) measurement; so, some errors might be introduced into the DOS values and it would be logical to deduce that reliability of DLEPR results should be assured using other techniques, as will be discussed below. In the case of austenitic stainless steels [20–24] and duplex ones [25], the above mentioned issues has been successfully addressed using potentiodynamic electrochemical impedance spectroscopy (PDEIS) technique which is regarded as a modification of EIS test used to analyze stationary conditions. In other words, PDEIS can be applied to systems that change with time or potential [26] and it has been employed to study the reaction mechanisms and correlate the AC bias response of the electrode/electrolyte interface with the applied overpotential [23, 27–29]. Hence, in light of previous studies, there are not much published research works concerning the investigation of corrosion behavior and passive film properties of martensitic stainless steels using advanced electrochemical techniques (akin to PDEIS test method); so, the aim of the present study is to assess the effect of tempering heat treatment condition on the intergranular corrosion performance of AISI 410 martensitic stainless steels using such a sophisticated technique in conjunction with conventional ones as DLEPR, electrochemical noise, qualitative approach and hardness measurement.

2. Materials and methods

AISI 410 alloy rod, with chemical composition shown in table 1, was cut into cylindrical test samples (10 mm diameter × 10 mm length). The specimens were first subjected to austenitization heat treatment at 980 °C for 1200 s, followed by oil-quenching so as to have martensitic phase. Following that, the as-quenched samples were tempered (sensitized) at 550 °C for different time spans (ranging from 0.5 h to 24 h) and then oil-quenched. Before each electrochemical experiment, the samples were consecutively wet ground sequentially to 3000 grit SiC paper and then polished with 1 μm diamond paste. All the electrochemical experiments were performed using an Ivium A32700 potentiostat, Pt foil counter electrode and each heat treated sample as the working electrode in a solution of 0.5 M H2SO4 at 30 °C. The reported values for each test is the average of at least three individual measurements. In order to investigate anodic behavior of the samples, anodic polarization tests were performed in the potential range from open circuit potential (OCP) to 1.20 VAg/AgCl at a scan rate of 1 mV/s. The localized corrosion behavior of each sample was assessed by double-loop electrochemical potentiodynamic reactivation (DLEPR), electrochemical noise (EN) and potentiodynamic electrochemical impedance spectroscopy (PDEIS) techniques. In all DLEPR tests, the potential was scanned anodically from OCP and reversed at 0.50 VAg/AgCl with potential scan rate of 1.67 mV/s. The degree of sensitization (DOS) values were measured as described in equation (1)

\[
\text{DOS} = \frac{I_r}{I_a} \times 100
\]

where I_r and I_a account for backward and forward scan peak currents, respectively. Electrochemical noise measurement was performed for 40000 s using Ag/AgCl reference electrode and two nominally same electrodes with the same surface area in 0.5 M H2SO4 solution at 30 °C. The working electrode and the ground one were the untempered/tempered samples and the untempered specimen, respectively. Localization index (LI), also known as pitting index, is used as a criterion to investigate the sensitized samples susceptibility to either uniform or localized attack [30–32]. It can take a value between 0 to 1 and samples with LI values less than 0.01 are susceptible to uniform corrosion while those with LI values greater than 0.1 are more prone to localized attack [33]. The LI value can be determined according to equation (2):

\[
\text{LI} = \frac{\text{EN}}{\text{C}_{\text{OCP}}}
\]

\[
\text{C}_{\text{OCP}} = \frac{\text{EN}_{\text{C}1}}{\text{EN}_{\text{C}2}}
\]

where EN is the electrochemical noise, C1 and C2 are the potentials where the noise peaks, respectively.
The oxalic acid etch test (ASTM A763 test standard - practice W [12]) was also employed in order to qualitatively evaluate the specimens. To do so, electro-etching process was conducted at 1 A/cm² for 90 s in a solution of 10 wt.% oxalic acid. The surface studies (including microstructural investigation of etched samples and morphological characterization of the corroded specimens) were performed using a TESCAN-Vega 3 scanning electron microscope (SEM) coupled with energy-dispersive X-ray (EDS) analysis system. The Brinell hardness number (BHN) of the samples were determined by a universal Hardness Tester device (Model: UV1) with an applied load and dwell time of 187.5 kg and 10 s, respectively.

3. Results and discussion

3.1. Hardness and microstructural details

The Brinell hardness test results attributed to the samples that were tempered for different time spans are presented in figure 1. As can be seen, increasing the tempering time leads to decrease in BHN values, which can be associated with the precipitation and growth of chromium carbides in such a way that larger volume fractions of carbides would be precipitated at prolonged tempering time, which is in accordance with previous reports [34]. These findings can be further investigated by the microstructure of tempered samples shown in figure 2.

Microstructural features according to the SEM micrographs of specimens are presented and compared in figure 2. In case of untempered specimen (figure 2(a)), the step structure (i.e. no observable ditches) indicates no susceptibility to intergranular corrosion; however, a dual structure with discontinuous ditch grain boundaries (gb) can be noted based on figure 2(b), which can be related to partial precipitation of carbides at lath interfaces. For the 2h-tempered sample (figure 2(c)), the observation of at least one grain surrounded by ditches corresponds to ditch structure which in turn implies that the alloy is prone to severe chromium carbide-type intergranular attack due to enhanced formation of carbides under such an experimental condition. In samples undergone prolonged tempering (figures 2(d), (e)), the grain boundaries become less visible as the tempering time increases from 5h to 24h, which can be correlated to diffusion of chromium from inner grain to Cr-depleted zones at lath interfaces (i.e. desensitization), making it difficult to recognize a clear distinction between
dual structure and ditch one. Taking it into account that instead of dissolution of Cr-impoverished regions, electro-etching in oxalic acid electrolyte provokes etching of carbides \[6\], one can postulate that in case of desensitized sample, no substantial differences can be made between the qualitative test results and other credible alternatives should be sought in order to assess the IGC behavior of tempered MSS, which will be discussed below.

3.2. Anodic potentiodynamic polarization
The anodic polarization test results attributed to the samples that were tempered for different time spans are presented in figure 3(a) and the corresponding passive current densities are presented in figure 3(b). As seen, all the samples exhibit a typical active-passive behavior in their respective anodic polarization curves in which the transpassive region start potential can be approximately estimated at 0.95 $V_{Ag/AgCl}$ and the passive potential region lies between $-0.25$ and $0.90$ $V_{Ag/AgCl}$. In case of 0.5h and 2h-tempered samples, two anodic peaks of (I) and (II) at overpotentials of $\approx -0.35$ and $0.00$ $V_{Ag/AgCl}$, respectively, are denoted on the graph (figure 3(a)). The former can be attributed to the passive behavior of the inner martensitic bulk while the latter corresponds to passivation of Cr-impoverished zones, as has been reported previously \[6, 35\].

The passive current densities (i.e. the lowest current density in the passive potential range \[36\]) of the polarization curves shown in figure 3(a) are illustrated in figure 3(b). Considering figure 3(b), it seems obvious that increasing the tempering time up to 2 h results in increase in passive current density ($i_{pass}$) values which is indicative of higher susceptibility to IGC due to the formation of more defective passive film. This can be inferred from the decline in internal stress of the sample (i.e. directly proportional to hardness values \[5\] presented in figure 1), leading to creation of less corrosion resistant passive layers upon the surface \[37, 38\]. The decrease in passive current densities in the case of samples that were tempered for prolonged times (more than 2 h) would be related to the desensitization (as discussed earlier) which can counteract the effect of internal stress on passive film formation.

3.3. Double-loop electrochemical potentiodynamic reactivation
Typical DLEPR curves and the corresponding DOS values attributed to the samples that were tempered for various durations are presented in figure 4. As can be seen in figures 4(a), (b), the forward scan of the DLEPR plots behaves in a similar way to that of anodic polarization curves shown in figure 3(a). When it comes to the backward scan, the tempering duration should be regarded as a key factor affecting the electrochemical response of the alloy. In this regard, the DOS values were calculated based on equation (1) and the corresponding results are presented in figure 4(c). As seen, the as-quenched sample would be considered as a non-sensitized specimen as its DOS value is lower than 1%, but the tempered samples, with DOS values exceeding 1%, would be recognized as sensitized ones. The great tendency of tempered alloy to IGC can be inferred from the chemical composition of the alloy (table 1) and its microstructure compared to other types of stainless steels. In other words, high grain boundary density of the MSS resulting from its fine microstructure would pave the way for promoted precipitation of chromium carbides adjacent to the lath interfaces. On the other hand, high C content of the MSS (compared to other grades of stainless steels) would facilitate the formation of such destructive precipitates, leading to severe Cr-depletion during tempering process, which can be held responsible for such
high DOS values. Figure 4(c) also demonstrates that increasing the tempering time up to 2 h leads to increase in DOS values while further increase in tempering time results in an opposite trend in DOS values. The former can be attributed to the formation of depleted regions adjacent to grain boundaries due to chromium carbide precipitation at short tempering times while the latter is related to the desensitization (formation of less defective passive films at grain boundaries) which is due to Cr diffusion from bulk to depleted zones at prolonged tempering times. The fine microstructure of this alloy, which can provide short diffusion paths for chromium, would be regarded as the major cause of healing effect (desensitization) for prolonged tempering processes. Such observations have also been reported in previous studies [8].

3.4. Electrochemical noise measurements
The electrochemical noise results are depicted in figure 5. As can be seen, all of the samples have localization index (LI) greater than 0.1, which indicates their susceptibility to localized attack. In simpler terms, the working electrode cannot be passivated even at prolonged immersion times and the samples remain active in 0.5 M H₂SO₄ electrolyte as long as no oxidation reaction comes to shift the electrode potential in the passive region. Moreover, figure 5 illustrates that the LI value of the specimens remain approximately unchanged as the exposure time increases, which clarifies the active behavior of the alloy, regardless of tempering duration.

The morphology of corroded samples after EN test (figure 6) confirms these findings. As seen, relatively large pits can be observed in SEM micrographs of all samples and the pits are surrounded by non-protective corrosion products in case of tempered specimens. In fact, active dissolution of the alloy during the EN test (as discussed above) can provide a context in which such defective films would be formed upon the surface, provoking localized attack under such experimental conditions. So, one can say that the susceptibility to localized corrosion can be quantitatively determined by LI values, but the result cannot be correlated to IGC behavior of the alloy.
requiring more advanced electrochemical methods (akin to potentiodynamic EIS) for IGC evaluation of the MSS.

For the sake of clarity, the EDS spectra of the resulting corrosion product films and the corresponding chemical composition are also presented in figure 7. As seen, O, Fe and Cr are the major constituents of the
resulting films, indicating the formation of oxides of Fe and Cr under such experimental conditions, which is consistent with previous reports [39]. Moreover, the Cr content of the film on tempered specimens is relatively lower than that of the untempered one (figure 7), which can be inferred from the adverse impact of the chromium carbide precipitates (i.e. due to sensitization heat treatment) on the available Cr contributing to the creation of the corrosion product layer. The detection of S in EDS results can be attributed to the presence of S-containing compound of FeSO₄ (ferrous sulfate) salt in the film, as has been reported previously [39]. Last but not least, it is salient to note that the Au peak in figure 7 is due to the Au-sputtering treatment [40].

3.5. Potentiodynamic electrochemical impedance spectroscopy
The PDEIS responses attributed to samples that were sensitized for different time spans are presented in figure 8. As seen, two types of AC response can be observed as the applied potential increases in the transpassive region.
The AC response for the polarization potentials lower than 1.10 V\text{Ag}/AgCl (figures 8(a)–(c)), there is a depressed semi-circle capacitive loop at high and medium frequencies, which is followed by an incomplete inductive loop at low frequency range. The former is regarded as the common characteristic of passive film dissolution on stainless steels [23, 25] while the latter can be related to the incomplete desorption of passive layer at chromium-depleted regions adjacent to lath interfaces. Moreover, increasing the applied potential from 0.95 to 1.05 V\text{Ag}/AgCl leads to decrease in the semi-circle diameters while the inductive loop starts to be completed. The completion of inductive loop at low frequencies occurs at applied bias voltage of 1.10 V\text{Ag}/AgCl (figure 8(d)) where the passive films that were formed on the surface of Cr-depleted zones would be completely desorbed. As the applied DC bias increases from 1.15 to 1.20 V\text{Ag}/AgCl (figures 8(e), (f)), the AC response of specimens would remain as the same as that were polarized to 1.10 V\text{Ag}/AgCl. Nonetheless, higher applied overvoltages would result in smaller depressed semi-circles followed by slightly decreased inductive loops.

The equivalent circuits that were used for fitting the AC responses obtained in the applied DC bias in the range of 0.95–1.05 V\text{Ag}/AgCl and 1.10–1.20 V\text{Ag}/AgCl are shown in figures 9(a) and (b), respectively. In these equivalent electrical circuits, R_s is the solution resistance, CPE is the constant phase element of double layer [41], R_{ct} is charge transfer resistance, R_P is the polarization resistance and L is the inductance. The fitted values to the experimental EIS data (figure 8) are given in table 2. The value of R_{ct} at applied DC bias where inductive loop is completed (i.e. 1.10 V\text{Ag}/AgCl) can be considered as a criterion in order to evaluate IGC in MSSs, i.e. lower R_{ct} values at this overvoltage imply higher susceptibility to intergranular attack. In this regard, one can say that, increasing the tempering time up to 2 h results in decrease in IGC resistance while further increase in tempering duration (more than 2 h) leads to increase in IGC resistance. These results are in agreement with those of other electrochemical analyses as discussed above. Furthermore, the L and R_P values follow the same trend as R_{ct} ones, which means samples that are more resistant to IGC have higher inductance and polarization resistance values too (more resistance to passive layer desorption). As the overvoltage increases in the transpassive potential region, the n-values decrease due to increased effective surface area, which is in accordance with lower n-values that have been reported for rough surfaces [42, 43].

figures 10 and 11 represent the surface morphologies of the corroded samples after performing PDEIS tests. As can be seen, the uniform corrosion behavior of the samples change to localized attack as the tempering time increases up to 2 h (figures 10(a)–(c) and figures 11(a)–(c)); however, the severity of IGC decreases for longer...
tempering durations (desensitized state in figures 10(d), (e) and figures 11(d), (e)) as a result of the healing effect (i.e. desensitization) occurring due to the restitution of intergranular Cr diffusion to the critical value of Cr content (Cr $\geq$ 12 at.%) [44]. In simpler terms, increased tempering duration would be followed by diffusion of Cr atoms from inner grain to Cr depleted zones at lath interfaces and thereby desensitizing Cr impoverished
Table 2. Fitted values to the PDEIS experimental data (figure 8) attributed to the equivalent electrical circuits elements shown in figure 9.

| Applied voltage (V) | Tempering time (h) | $R_s$(ohm.cm$^2$) | $R_p$(ohm.cm$^2$) | CPE(F.cm$^{-2}$s$^{\alpha-1}$) | $n$ | L(H.cm$^{-2}$) | $R_\text{ct}$(ohm.cm$^2$) | $\chi^2$ |
|--------------------|--------------------|-------------------|-------------------|-----------------------------|-----|----------------|-------------------------|--------|
| 0.95               | 0                  | 4.68              | 5950              | 8.31E-04                    | 0.8632 | —              | —                      | 2.46E-02 |
|                    | 0.5                | 3.64              | 3660              | 8.09E-05                    | 0.8742 | —              | —                      | 8.82E-03 |
|                    | 2                  | 3.56              | 3047              | 2.34E-04                    | 0.8249 | —              | —                      | 9.06E-03 |
|                    | 5                  | 3.35              | 4520              | 9.66E-04                    | 0.8517 | —              | —                      | 4.02E-02 |
|                    | 24                 | 5.35              | 4850              | 1.02E-04                    | 0.8456 | —              | —                      | 2.22E-02 |
| 1.00               | 0                  | 3.59              | 1462              | 1.21E-04                    | 0.8520 | —              | —                      | 1.77E-02 |
|                    | 0.5                | 3.90              | 1158              | 1.67E-04                    | 0.8333 | —              | —                      | 3.01E-02 |
|                    | 2                  | 3.47              | 891               | 2.49E-04                    | 0.8297 | —              | —                      | 1.76E-02 |
|                    | 5                  | 5.24              | 1354              | 1.53E-04                    | 0.8244 | —              | —                      | 2.24E-02 |
|                    | 24                 | 3.32              | 1380              | 1.42E-04                    | 0.8348 | —              | —                      | 5.11E-02 |
| 1.05               | 0                  | 4.65              | 878               | 1.56E-04                    | 0.8310 | —              | —                      | 1.98E-02 |
|                    | 0.5                | 3.63              | 571               | 1.39E-04                    | 0.8242 | —              | —                      | 2.13E-02 |
|                    | 2                  | 5.50              | 502               | 3.11E-04                    | 0.8073 | —              | —                      | 2.41E-02 |
|                    | 5                  | 5.31              | 639               | 1.77E-04                    | 0.8164 | —              | —                      | 4.37E-02 |
|                    | 24                 | 3.97              | 662               | 1.97E-04                    | 0.8032 | —              | —                      | 4.09E-02 |
| 1.10               | 0                  | 3.39              | 471               | 1.68E-04                    | 0.8055 | 990            | 323                     | 1.45E-02 |
|                    | 0.5                | 4.13              | 285               | 1.80E-04                    | 0.7957 | 631            | 223                     | 6.91E-02 |
|                    | 2                  | 5.55              | 262               | 2.99E-04                    | 0.7838 | 594            | 172                     | 4.45E-02 |
|                    | 5                  | 4.06              | 377               | 1.91E-04                    | 0.7978 | 790            | 257                     | 1.22E-02 |
|                    | 24                 | 5.41              | 411               | 1.62E-04                    | 0.7947 | 832            | 277                     | 1.49E-01 |
| 1.15               | 0                  | 5.31              | 366               | 1.35E-04                    | 0.8015 | 788            | 303                     | 9.88E-02 |
|                    | 0.5                | 3.63              | 274               | 1.32E-04                    | 0.7936 | 599            | 254                     | 2.38E-02 |
|                    | 2                  | 5.31              | 213               | 2.21E-04                    | 0.7955 | 479            | 182                     | 4.06E-02 |
|                    | 5                  | 4.72              | 326               | 1.39E-04                    | 0.8045 | 694            | 288                     | 4.67E-02 |
|                    | 24                 | 4.23              | 331               | 1.51E-04                    | 0.7956 | 716            | 290                     | 1.95E-02 |
| 1.20               | 0                  | 3.41              | 353               | 1.34E-04                    | 0.8669 | 769            | 268                     | 4.43E-02 |
|                    | 0.5                | 5.52              | 210               | 2.33E-04                    | 0.7860 | 470            | 171                     | 7.61E-02 |
|                    | 2                  | 4.16              | 193               | 1.48E-04                    | 0.7672 | 444            | 153                     | 3.47E-02 |
|                    | 5                  | 3.97              | 300               | 1.62E-04                    | 0.7621 | 654            | 220                     | 1.19E-01 |
|                    | 24                 | 4.71              | 325               | 1.11E-04                    | 0.7675 | 701            | 249                     | 9.08E-02 |
These findings are in agreement with the EIS test results indicating highest IGC for the samples that were tempered for 2 h.

4. Conclusions

The influence of tempering time on the subsequent intergranular corrosion behavior of AISI 410 MSS was studied. The susceptibility to IGC was measured using conventional techniques and corroborated by implementation of PDEIS method in the transpassive potential region. Generally, increasing tempering time up to 2 h led to decline in IGC resistance of the alloy, while prolonged tempering contributed to improvement in IGC resistance due to the healing effect. It was found that the limitations of acid etching test, DLEPR and EN tests make it difficult to either differentiate between sensitized and desensitized states or assess the IGC quantitatively. Nonetheless, the simultaneous quantitative and qualitative analyses performed by the PDEIS can pave the way for evaluation of IGC behavior. In particular, the charge transfer resistance value of the alloy and its morphology after conducting PDEIS experiments from transpassive start potential to $1.10 \, V_{Ag/AgCl}$ (i.e. the overpotential at which inductive loop related to the desorption of unstable passive film upon Cr-depleted region would be completed) can be used to study IGC susceptibility of the alloy. In this regard, $R_{ct}$ values greater than
470 ohm.cm² are indicative of non-sensitized state while those in the range of 370–470 ohm.cm² can be interpreted as the desensitized condition. Those samples exhibiting Rct values lower than 370 ohm.cm² are prone to IGC and the severity of attack can be further examined using the surface morphology.

**Data availability statement**

All data that support the findings of this study are included within the article (and any supplementary files).

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