Influence of Dissolved Oxygen on DL-EPR Behavior of SUS329J4L Duplex Stainless Steel after Convection Cooling

by

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Sigma phase precipitates in duplex stainless steel during slow cooling, degrading the corrosion resistance and the notch toughness. The double loop electrochemical potentiokinetic reactivation (DL-EPR) method in a solution of 0.5 M H₂SO₄ + 0.01 M KSCN + 0.5 M NaCl is known to be good for detecting sigma phase. However, the influence of dissolved oxygen (DO) in solution had not been elucidated, and on-site application of DL-EPR had not been carried out. In this study, SUS329J4L stainless steel was continuously cooled at different time constants by convection using nitrogen, and its DL-EPR behavior was measured in solution with different DO concentrations. A cathodic current partially appeared at potentials nobler than the corrosion potential due to the reduction of oxygen. Elimination of DO suppressed the cathodic reaction, resulting in adequate sensitivity for detection of precipitated sigma phase. The detection sensitivity was almost the same as that for laboratory samples in 700 mL/cm² solution and simulated small structures exposed to 20 mL/cm² solution, and with almost the same high sensitivity as the immersion corrosion test.

Key words:
Duplex stainless steel, sigma phase, electrochemical potentiokinetic reactivation (EPR), time constant, dissolved oxygen, detection sensitivity

1 Introduction

Austenitic (γ) - ferritic (α) duplex stainless steels are widely used in mechanical structures and machines because of their excellent mechanical properties and high corrosion resistance. However, the precipitation of sigma (σ) phase in such steels greatly reduces both the impact toughness and corrosion resistance. The σ phase has a 30-atom tetragonal crystal lattice with two inner quasi-hexagonal atomic layers enriched with chromium (Cr) and molybdenum (Mo) atoms. In general, cleavage fracture occurs more readily in σ phase and prior corrosion occurs in the vicinity to this phase.

The σ phase precipitates in duplex stainless steel during both isothermal aging between 973 ~ 1173 K and continuous cooling from 1338 K. In our previous study, XRD analysis showed that the amount of σ phase in SUS329J4L steel cooled at 0.02 K/s from 1073 to 773 K was 22.9%. During isothermal aging, carbides (M₃C₆) form at the γ/α boundary and then grow into the neighboring γ phase. This brings about a newly formed γ phase, which pushes the γ/α interface slightly towards the σ phase side. Then, at the new γ/σ interface, σ phase nucleates and grows into the α phase with continuous formation of γ' phase in the vicinity of the σ phase. The same precipitation process is expected to also occur with slow and continuous cooling from a near solution annealing temperature.

Davison et al. studied the degradation of Charpy impact energy of 2205 duplex stainless steel air cooled from 1338 K and its corrosion resistance in ferric chloride solution. In our previous study, during Charpy impact tests, ductile, transient and brittle fractures were found to occur orderly in SUS329J4L steel when increasing the time constant in convection cooling. Because of the delicate control required for a consistent cooling rate from a high temperature, convection cooling is more practical for fabricating mill-products or machines. As for convection cooling, the authors proposed a parameter of the time constant in the lumped capacitance method to characterize the cooling intensity.

To minimize the manufacture and maintenance costs of quality duplex steel concerning cooling intensity, a nondestructive and effective method that can be performed on-site to detect precipitated σ phase is essential. The traditional methods, however, namely X-ray diffraction, metallographic examination, Charpy impact testing and ferric chloride corrosion testing, cannot be performed on-site or are invasive. A method that may possibly be applied on-site to large machinery is the electrochemical potentiokinetic reactivation (EPR) method.
using a mounted mini-electrochemical cell. The response of the double loop EPR (DL-EPR) ratio to intermetallic compounds, σ phase, carbides and γ phase, in the vicinity of Cr- or Mo-depleted regions has been reported in several studies. Lopez et al. obtained a DL-EPR ratio that was proportional to σ phase content in the duplex stainless steel UNS S31803 in a solution of 2.0 M H\textsubscript{2}SO\textsubscript{4} + 0.01 M KSCN + 0.5 M NaCl. We also recently developed a modified DL-EPR method to detect σ phase in SUS329J4L steel, that had been continuously cooled from 1323 K, in a solution of 0.5 M H\textsubscript{2}SO\textsubscript{4} + 0.01 M KSCN + 0.5 M NaCl. EPR test results depend not only on the chemical composition of the Cr- or Mo-depleted regions but also on the solution composition. For instance, Cr\textsubscript{4}+ in the solution activates the dissolution of a stronger passive film on steels containing higher amounts of Cr or Mo. Such findings have only been studied under laboratory conditions in deaerated solutions. However, dissolved oxygen (DO) in the solution is also expected to influence the test reaction because it generally strengthens the passive film. As complete exclusion of DO from the solution on-site can be difficult, unless the solution is handled carefully, the detection sensitivity of the DL-EPR test might be influenced by unwelcome DO. Because the details including quantification of the influence of DO on the EPR ratio has not been fully elucidated, it is necessary to assess the application of the test to real structures or machines.

Therefore, in this study, the modified DL-EPR test by using solutions with various DO concentrations was applied to SUS329J4L duplex stainless steel cooled by convection and the reactivation behavior corresponding to the precipitated σ phase was observed. Next, the validity of the modified test was confirmed by applying it to several samples in on-site simulations and by comparing it with immersion corrosion rates and Charpy impact test results.

2 Experimental
2.1 Material, heat treatment and samples

A commercially available SUS329J4L duplex stainless steel bar with the following chemical composition was used: 0.029 mass% C; 0.43% Si; 0.73% Mn; 0.038% P; <0.005% S; 24.19% Cr; 5.68% Ni; 2.68% Mo; 0.14% N; and the balance Fe. It was cut into samples measuring 20 mm x 20 mm x 5 mm. The samples were kept in a vacuum furnace at 1323 K for 1.8 ks, then cooled at various intensities by controlling the pressure of nitrogen as a cooling flux.

Several cooling curves are shown in Fig. 1, where \( T \) is the temperature at time \( t \) and \( T_1 \) is the initial temperature of 1323 K. The dimensionless temperature \( (T-T_0)/(T-T_1) \) is also shown on the right Y axis. The curves show exponential relationships between the temperature, and therefore the dimensionless temperature also, and cooling time \( \tau \). The cooling intensity for several samples from 730 K or lower was enhanced by faster fan rotation, which does not affect the σ phase formation. As the final temperature of the nitrogen (\( T \)) was 323 K, for each curve, the time taken to cool to 929 K is 0.5 \( \tau \), \( \tau \) being the time constant, as indicated in the graph. The time constant \( \tau \) can also be physically determined from the specific heat, density, characteristic length of the sample and the heat transfer coefficient of the cooling gas. It is reasonable to use the time constant to express the cooling intensity rather than to use an instantaneous cooling rate at a particular temperature or an average cooling rate of a particular temperature range. The time constants of the curves in Fig. 1 were 445, 1795, 3115, and 5500 s, corresponding to average cooling rates of about 1.0, 0.3, 0.2, 0.1 K/s, respectively, between 1073 and 773 K.

![Fig. 1 Temperature and dimensionless temperature - time curves of SUS329J4L steel cooled with convection of nitrogen gas at different fan rotations and the determination of half of the time constant (0.5\( \tau \)) at 929 K.](image)

In preparation for the EPR measurements, the samples were polished with 2000 grit paper and ultrasonically cleaned in acetone. These samples were used in two set-ups, one under normal laboratory conditions and the other under simulated on-site conditions, as shown in Fig. 2 (a, b), and the samples referred to as laboratory samples (L.S.) and simulated small structures (S.S.S.), respectively. All the surface of the L.S. was sealed with silicone sealant, except for a 10 mm x 10 mm polished area, and immersed in 700 mL of solution. The S.S.S. were attached to a minicell by epoxy resin and manicure, with a polished area of about 100 mm\(^2\) exposed to about 20 mL of solution in an acrylic cylinder with an inner diameter (\( \phi \)) of 16 mm mounted as shown in Fig. 2b.

A third set-up shown in Fig. 2 (c), for simulated large structures (S.L.S.), was established. A 260 mm long steel pipe with a 125 mm inner diameter and a 250 mm outer diameter was cooled by convection and then machined to reduce its outer diameter to 230 mm for one third of its length, 188 mm for another third and 165 mm for the remaining third. At positions 1, 2 and 3 in Fig. 2 (c), a minicell was mounted using the same...
materials and dimensions as for S.S.S, and also with an exposed polished area of 100 mm\(^2\), albeit curved.

EPR measurements of S.S.S and S.L.S. are different from the conventional laboratory conditions, represented by L.S., in that there was much less solution and therefore limited space for diffusion of the dissolved metal ions and oxygen or the dispersion of corrosion products. Furthermore, the temperature of the solution was not controlled precisely.

After keeping the potential at the open circuit potential (OCP) for 300 s, the sample surface was reduced by dynamic cathodic polarization from OCP to -0.55 V or cathodic polarization at -1.0 V for 60 s to remove the residual passive film. After keeping the potential again at the OCP for 300 s and confirming the OCP value to be near -300 mV, indicating a bare surface \(30\), the potential was swept at 0.833 mV/s from -0.55 V to 0.3 V (or 0.35 V), and then swept back to -0.55 V. In the DL-EPR curves in Fig. 4, \(i_a\) is the peak activation current, and \(i_i\) is the peak reactivation current. The EPR ratio can be expressed as either \(i_a/i_i\) or the charge ratio from the reactivation and activation reactions. Umemura reported the validity of their good correlation to the depleted zone in sensitized SUS304 steel \(31\). In this study here, the EPR ratio was expressed as \(i_a/i_i\), corresponding to the dissolution from regions in the vicinity of \(\sigma\) phase.

3 Results and Discussion

3.1 Dissolved oxygen concentration with gas bubbling

Fig. 5(a) shows cathodic polarization curves obtained using a platinum (Pt) plate in 0.5 M NaCl aqueous solutions streamed with nitrogen, air and oxygen. All the values were corrected with respect to the Cl\(^-\) concentration. In general, the DO concentration in aqueous solution does not correlate with the pH of the solution. In this study, a saturated calomel electrode (SCE) was used as a reference. The current density corresponds to the cathodic reaction rate of DO on the Pt surface, which is controlled by the diffusion rate of the oxygen from the bulk solution. It is well known that the saturated DO concentration in pure water (without NaCl) at 303 K is 7.53 mass ppm \(32\), or 6.29 ppm after correction in 0.5 M NaCl aqueous solution. The corrected value corresponds well with the DO meter reading (6.16 ppm). Thus, the DO concentration of 6.16 ppm in 0.5 M NaCl solution can be taken as corresponding to the limited current density at -0.5 V vs. SCE with air bubbling. Based on this, the DO concentrations when
oxygen and nitrogen were bubbled into the solution were 29.57 and 0.04 ppm, respectively.

![Graph](image)

**Fig. 5** Cathodic polarization curves on platinum (Pt) plate obtained in 0.5 M NaCl aqueous solutions with different gas bubbling (a) and the dissolved oxygen concentration (mass ppm) obtained from the cathodic current density on Pt plate at -0.5 V (vs.SCE) and from a dissolved oxygen meter.

### 3.2 EPR polarization curves of laboratory samples

In previous XRD analysis, the amount of σ phase in samples with the same composition as used in this study that had been continuously cooled with $\tau = 3115$ s (0.2 K/s vs. 1073 K) and 5500 s (0.1 K/s) was 5.9% and 13.4% respectively, but no σ phase was detected when $\tau$ was 1795 s (0.3 K/s) or smaller. On the other hand, the Charpy absorbed energy of the sub-size samples at room temperature continually decreased with the time constant from $\tau = 445$ s (1.0 K/s) to 28000 s (0.02 K/s). As almost the same amount of absorbed energy was measured when the sample had been cooled at $\tau = 445$ s as when water quenched (WQ), the σ phase has a tendency to precipitate when cooling intensity is weak, such as when $\tau = 445$ s as in this study. Table 1 shows the chemical composition ratios of the γ, α, γ′ and σ phases as analyzed by EDX in the sample cooled at $\tau = 28000$ s. More Cr and Mo are contained in the σ phase than in the neighboring γ′ phase. This indicates the existence of a Cr- or Mo-depleted region in the vicinity of the σ phase.

Table 1 Chemical composition ratios (mass%) of phases in SUS329J4L steel after cooling at $\tau = 28000$ s, analyzed by EDX.

|   | Fe  | Cr  | Ni  | Mo  |
|---|-----|-----|-----|-----|
| γ | 64.93 | 24.14 | 8.59 | 2.34 |
| α | 62.68 | 29.63 | 4.47 | 3.22 |
| γ′ | 67.95 | 25.61 | 4.37 | 2.07 |
| σ | 58.37 | 30.94 | 4.54 | 6.13 |

Fig. 6 shows a series of polarization curves obtained from the L.S. with $\tau = 1795$ and 5500 s. Their EPR ratios have been clearly influenced by DO. The solution has been confirmed as good for sensitive detection of precipitated σ phase in the SUS329J4L steel. Two activation current peaks near the potential of -0.2 V correspond to start of dissolution from the α and γ phases in forward polarization. In backward polarization, the reactivation current at almost the same potentials indicates the dissolution from Cr- or Mo-depleted regions in the vicinity of σ phase. In general, a larger $i_r/i_a$ ratio indicates more precipitation of σ phase. On the other hand, cathodic currents appeared in both the forward and backward polarization even at potentials nobler than the corrosion potential ($E_{corr}$), marked “▲”, when air or oxygen was bubbled into the solution. Such cathodic currents did not appear with nitrogen bubbling. Therefore, the cathodic current results from the dissolved oxygen and its reduction.

![Graph](image)

**Fig. 6** Polarization curves of steel cooled with time constants (τ) of 1795 and 5500 s. L.S. and N.S. with τ = 5500 s. L.S. and N.S. with τ = 1795 s. EPR polarization with different DO concentrations of samples were tested (Fig. 6 (b)).

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**Fig. 7** Polarization curves of steel cooled with time constants (τ) of 1795 and 5500 s. L.S. and N.S. with τ = 5500 s. L.S. and N.S. with τ = 1795 s. EPR polarization with different DO concentrations of samples were tested (Fig. 6 (b)).
oxygen reduction rate depends not only on the applied potential but also on the surface state, which should be different in the forward and backward polarizations. It is speculated that more DO in the solution can play a role in fixing the preferentially eluted region in the vicinity of σ phase by the formation of a metal oxide or metal hydroxide passive film. However, in Fig. 6, the EPR ratio \( i_2/i_1 \) of sample cooled with \( \tau = 1795 \) s is near zero, without the appearance of reactivation current regardless of DO concentration. When \( \tau = 5500 \) s, the influence of DO concentration on the EPR ratio was apparent, with higher EPR ratio at lower DO concentration. The higher EPR ratio in lower DO concentration is probably due to the difficulty in repassivation in the preferential elution region in the vicinity of the σ phase.

Fig. 7 Speculated inner anode and cathode polarization curves corresponding to the cathodic currents at potentials nobler than the corrosion potential of cooled steels in solution bubbled with nitrogen, air and oxygen.

Fig. 8 shows several surface morphologies after EPR polarization with different DO concentrations of samples cooled with \( \tau = 1795 \) and 5500 s. Much more σ phase precipitated at the slower cooling rate. With either cooling rate, the dissolution around the σ phase was severer in the deaerated than the aerated solution, which corresponds well with the results shown in Fig. 6.

3.3 EPR polarization curves of simulated small structures

Fig. 9 shows polarization curves of S.S.S. obtained in 0.5 M H\(_2\)SO\(_4\) + 0.01 M KSCN + 0.5 M NaCl solutions exposed to the air (a) and bubbled with nitrogen (b, c). Blue lines marked ‘‘*’’are cathodic currents.

According to the above analysis, DO in the solution promotes the appearance of cathodic current in both the forward and backward polarizations. Fig. 10 shows schematically steel surfaces with passive film just before the reactivation reaction (i.e., a: O\(_2\) bubbling; b: N\(_2\) bubbling), and when elution occurred at γ’ regions in the vicinity of the σ phase after the reactivation reaction (i.e., c). The compactness as well as the thickness of a film can affect corrosion resistance. It has been reported that films are thinner on phases with a higher than lower Cr content.\(^{36,37}\) DO can effectively promote the formation of a compact passive film on the γ’ regions near the σ phase, which should have resulted in a smaller EPR ratio and thus decreased the detective sensitivity of σ phase precipitation. Therefore, for on-site detection of the σ phase by DL-EPR, the deaeration of solution should be strictly controlled.
3.4 Comparison of corrosion rate, absorbed energy and EPR ratio at various time constants

Fig. 11(a) is a plot of L.S. and S.S.S. EPR ratios, obtained when the solution was bubbled with nitrogen, against cooling time constants. Both types of sample give almost the same plot. This means that the effectiveness of the modified EPR method was not compromised by the amount of solution from 700 to 20 mL/cm². Note that the EPR ratio at τ = 1795 s did not appear in this figure because there was no reactivation peak, perhaps as a result of unclarified precipitation of σ phase at that cooling intensity. Fig. 11(b) shows the corrosion rates and absorbed energies of samples cooled with τ ranging from 4.6 to 31500 s. The corrosion rates were obtained from S.S.S. samples by the ferric chloride test (6.0 wt% FeCl₃ solution; 24 hrs at 313±2 K), while the absorbed energies were measured by the Charpy impact test (sub-size sample of 55 mm x 10 mm x 7.5 mm with a V-notch machined from S.L.S. sample; room temperature). The absorbed energy curve can be divided into ductile, transient and brittle zones. The loss of the ductility is due to the increased amount brittle σ phase during cooling. The σ phase easily cracks and produces stress concentration, which can result in cleavage fracture in the α phase. The absorbed energy dropped down to one third at τ = 1000 s. In contrast, the corrosion rate remained low until it started to rise sharply from 4.0 mdd at τ = 1000 s. Therefore, the absorbed energy showed relatively higher sensitivity to responses by the σ phase. On the other hand, the EPR ratio for L.S. and S.S.S. samples started rising sharply from 0.05% at τ = 1000 s (Fig. 11(a)). This suggests that the modified DL-EPR method has almost the same sensitivity as immersion corrosion in regard to detection of σ phase precipitation in duplex steel. This can be attributed to almost identical dissolution behaviors from the Cr- or Mo-depleted zone in the vicinity of the σ phase.

Fig. 12 shows on-site polarization curves of convection cooled S.L.S., at all three depths shown in Fig. 2(c), in 0.5 M H₂SO₄ + 0.01 M KSCN + 0.5 M NaCl solution bubbled with nitrogen. All three curves are very similar, with no appearance of reactivation current. Therefore, this large structure can be judged as safe because no harmful σ phase precipitation was detected.

Fig. 12 Polarization curves of simulated large structure at positions No.1, 2, 3 obtained in nitrogen bubbled 0.5 M H₂SO₄ + 0.01 M KSCN + 0.5 M NaCl solution.

4 Conclusions

To enhance the sensitivity of detecting σ phase in duplex stainless steel, the influence of dissolved oxygen (DO) concentration in an aqueous solution of 0.5 M H₂SO₄ + 0.01 M KSCN + 0.5 M NaCl on the DL-EPR behavior of SUS329J4L samples, cooled by convection at a range of time constants, was investigated using a modified procedure. The following results were obtained.

(1) DO in the solution greatly influenced the polarization behavior of SUS329J4L steel in the DL-EPR measurements. The DO promotes the appearance of cathodic current in both the forward and backward polarizations. This would have strengthened the passive film on the steel surface.

(2) Elimination of DO can help to promote the reactivation reaction by weakening the passive film on Cr- or Mo-depleted regions. Therefore, in the DL-EPR test, the detection sensitivity for the presence of σ phase in SUS329J4L steel can be enhanced by purging DO from the solution by bubbling nitrogen through it.
was not compromised by the amount of solution from 700 to which can result in cleavage fracture in the α phase. Contrast, the corrosion rate remained low until it started to rise. Energy showed relatively higher sensitivity to responses by the.

Fig. 11(a) Relationship between time constant τ and EPR ratio of L.S.

EPR ratio at various time constants. The absorbed energies were measured by the.

2 \text{K})$, while the absorbed energy curve can be divided into ductile, transient and brittle zones. The loss of the ductility is judged as safe because no harmful σ phase precipitation was almost the same when the sample was SUS329J4L steel as when it was a laboratory sample exposed to 20 mL/cm$^2$ solution. The detection sensitivity of the DL-EPR test for the presence of σ phase was almost the same when the sample was SUS329J4L steel as when it was a laboratory sample in 700 mL/cm$^2$ solution or a simulated small structure exposed to 20 mL/cm$^2$ solution. The detection sensitivity of the DL-EPR test is comparable to that of the immersion corrosion test, but slightly lower than that of the Charpy impact test.

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