Failure analysis of AISI 310S plate in an inert gas generator used in off-shore oil platform

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In this work a failure analysis was conducted in a mirror plate made of austenitic stainless steel AISI 310. The mirror is part of the inert gas generator, which is a combustion chamber dedicated to produce inert gas atmosphere (N₂ + CO₂) to be used during the oil transfer in off-shore platforms. The mirror failed in two distinct regions. The first crack was observed in a region where the temperatures reached were sufficiently high to provoke intense sigma phase precipitation. Other cracks were observed in a region subject to seawater refrigeration, named “cold zone”. These two failures were mitigated in order to propose solutions to avoid similar failures in the new equipment.

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1. Introduction

The oil transfer operation in off-shore platforms must be carried out under inert gas atmosphere. The inert gas generator (IGG) is the equipment used to produce this inert atmosphere, consisting basically of CO₂ and N₂. This inert mixture is obtained by a complete combustion of air in a chamber. The burner is inserted in the chamber through a mirror plate made of AISI 310S stainless steel.

Modern oil platform are equipped with two IGG’s, which work intermittently, i.e., while one of them is operating the other is stopped. The inert gas production is necessary only during the oil transfer.

Fig. 1 shows the mirror of AISI 310S plate with 22.0 mm of thickness, which has prematurely failed in zone A and in one B near the border with zone A. The burner (not shown) is inserted in the circular hole in the center of the plate. Zone A is the high temperature zone, since it is very close to the burner. As the distance to the burner increases the temperature of the plate under operation decreases. There is also a gradient of temperatures through the thickness, i.e., the inner surface is heated to higher temperatures than the outer surface. It is also reported that the outer surface of region B is continuously refrigerated with sea water.

This work is focused on the failure analysis of the cracks observed in regions A and B of the mirror. The equipment has worked intermittently for 3 years.

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2. Experimental

The failed mirror was taken off the equipment and cut for analysis. Fig. 2 shows the slice of the mirror used in this work. Samples were collected from locations R1, R2, R3, R4, R5, R6 and R7. The cracks investigated were found in R2 and R4. The main actions conducted in the failure analysis were:

– Chemical analysis of the material. Cr, Mn, Ni, Si and P were analyzed by optical emission technique, while C and S were analyzed by combustion analysis.
Observation of cracks in zones A and B with stereomicroscope. Scanning electron microscopy (SEM) analysis of cracks in zones A and B. The crack surface of the failure in zone A was oxidized due to the high temperature exposure, and had to be cleaned with Clarck’s reagent (1000 mL HCl + 20 g SnCl₂ + 20 g Sb₂O₃) before SEM.

Laboratorial tests with the same material of the mirror to evaluate the effect of high temperature exposure on toughness. Charpy impact specimens were machined, solution treated and aged in the range 600–800 °C for periods of time up to 200 h. The specimens were tested at room temperature.

Microstructural analysis in samples collected in regions R1, R4 and R7.

Measurement of the hardness profile through in cross sections of regions R1, R3, R4, R5, R6 and R7.

Microstructural analysis was conducted in light optical microscope with specimens prepared by electrolytic etch with 10% NaOH solution (3 V, 15 s) or 10% acid oxalic solution (8 V, 60 s). Vickers hardness was carried out with load of 30 kgf, and 5 measurements per distance were considered to plot the hardness profiles.

Fig. 3. Cracks in the hot zone: (a) principal crack; (b) secondary cracks.
3. Results

3.1. Crack in the hot zone (region R2)

The steel contains 0.054%C–1.766%Mn–0.605%Si–19.41%Ni–24.65%Cr–0.0015%S–0.016%P (wt.%). This chemical composition fits to the AISI 310S (UNS S31003) specification [1].

Fig. 3a and b shows the crack observed in the high temperature zone, precisely in region R2 of zone A. The principal crack was nucleated in the inner side and propagated through the thickness, reaching the outer side in a small extension (not shown). Secondary cracks of about 0.5 mm are observed near and parallel to the principal crack (Fig. 3b).

The SEM analysis of the surface crack of region R2 is presented in Fig. 4a–d. Fig. 4a shows a general view, with secondary cracks transverse to the plane of primary fracture. These cracks may be associated to the accommodation of transverse strains induced by the plastic deformation of the primary fracture [2].

The surface fracture has dimples denoting a ductile behavior. However, as shown in Fig. 4b–d, striations typical of fatigue propagation are also observed. This is an indication that thermal fatigue has occurred due to the intermittent operation of the IGG. Surface secondary cracks in region A (Fig. 3b) are also an evidence of fatigue initiation.

Fig. 5a–c shows the microstructures in the cross section of region R2, near the external surface, in the middle thickness and near the internal surface of region R2, respectively. These micrographs were obtained with specimens prepared with electrolytic etching with NaOH solution, which is recommended to reveal sigma phase in austenitic steels [3]. The high temperatures experimented under service promoted the sigma phase precipitation in the hot zone. This precipitation is more pronounced in the inner face than in the outer face, due to the temperature gradient through the thickness.

The hardness profile obtained in regions of the hot and the cold zones are shown in Fig. 6. The regions of the hot zone (R1 and R3) are harder than the cold zone (R4, R5 and R7), due to the sigma phase precipitation. In the hot zones, the hardness increases from the outer to the inner face due to the intensification of sigma precipitation as shown in Fig. 5a–c.

Chvátalová et al. [4] showed interesting thermodynamic calculations of sigma phase formation in Fe–Ni–Cr systems. Fig. 7 shows the plot of the amount of phases versus temperature in AISI 310S obtained with ThermoCalc® using equilibrium thermodynamic data. Sigma phase and chromium carbides (Cr23C6) may precipitate when the steel is aged from 600 °C to 900 °C. Although the equilibrium amount of sigma decreases with the increase of temperature, a previous work has shown that the precipitation of this phase was faster and more intense at 800 °C than at 700 °C and 600 °C [5]. At 600 °C the carbide precipitation was much more intense than sigma phase in specimens aged for 200 h. However, the increase of exposure time at this temperature must may favors the sigma phase precipitation. The IGG investigated in this study has worked intermittently for 3 years, which means that exposure time was much longer than 200 h.
Specimens of the AISI 310S plate were cut and machined for Charpy impact tests. These specimens were aged at 600 °C, 700 °C and 800 °C for 100 and 200 h. The results of impact toughness are shown in Table 1. For such small aging periods of time significant precipitation of sigma phase is observed at 700 °C (Fig. 8a) and 800 °C (Fig. 8b). At 600 °C intergranular chromium carbides precipitated abundantly, but sigma phase was not observed with NaOH etching. The intergranular precipi-

Fig. 5. Microstructures of the material in the cross section of region R2: (a) near external surface; (b) middle thickness; (c) near internal surface.
The precipitation of sigma phase in austenitic steel may involve a mechanism $\gamma \rightarrow M_23C_6 \rightarrow \sigma$ [5]. The results show that sigma precipitation is responsible for a severe decrease of toughness, although intergranular carbides precipitation have also an embrittlement effect.

![Hardness profiles obtained at different regions (R1, R3, R4, R5 and R7).](image)

![Thermodynamic prediction of the phase amounts in function of temperature using Thermocalc software and considering the steel composition.](image)

### Table 1
Toughness (impact energy) at 22 °C of specimens of AISI 310S steel aged at 600, 700 and 800 °C.

<table>
<thead>
<tr>
<th>Heat treatment</th>
<th>Impact energy (J)</th>
<th>Microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solution treatment</td>
<td>217</td>
<td>Austenite ($\gamma$)</td>
</tr>
<tr>
<td>600 °C/100 h</td>
<td>165</td>
<td>$\gamma + M_23C_6$</td>
</tr>
<tr>
<td>600 °C/200 h</td>
<td>122</td>
<td>$\gamma + M_23C_6$</td>
</tr>
<tr>
<td>700 °C/100 h</td>
<td>119</td>
<td>$\gamma + M_23C_6 + \sigma$</td>
</tr>
<tr>
<td>700 °C/200 h</td>
<td>116</td>
<td>$\gamma + M_23C_6 + \sigma$</td>
</tr>
<tr>
<td>800 °C/100 h</td>
<td>55</td>
<td>$\gamma + M_23C_6 + \sigma$</td>
</tr>
<tr>
<td>800 °C/200 h</td>
<td>23</td>
<td>$\gamma + M_23C_6 + \sigma$</td>
</tr>
</tbody>
</table>
Fig. 8. Sigma phase precipitation in specimens of the steel aged at (a) 700 °C for 200 h and (b) 800 °C for 200 h.

Fig. 9. Secondary cracks propagation in region R2 where intense sigma phase precipitation is observed.
Fig. 9 shows a secondary crack propagating through the grain boundaries in a region of sigma precipitation. The thermal fatigue crack in the hot zone was facilitated by the intense sigma phase precipitation which reduces the toughness and increases the hardness of the material. According to Liu et al. [6] the precipitation of sigma phase in austenitic stainless steel reduces the creep-fatigue life.

3.2. Cracks in the cold zone (region R4)

Fig. 10a shows the crack of region R4. Differently from the cracks of the hot zone, the cracks of the cold zone initiate in the external face of the plate. Fig. 10b shows the branched path of the cracks, typical of stress corrosion cracking (SCC). The region R4 is refrigerated with sea water during operation, which gives the environmental conditions necessary to SCC. EDS analysis in the crack tip (Fig. 11) shows peaks of Cl, Ca and S, which confirms SCC in seawater.

The microstructures of regions R4 and R7 revealed with 10% acid oxalic etching is shown in Fig. 12a and b. The material is sensitized due to chromium carbide precipitation. Sigma phase was not detected with NaOH etching. The temperature in region R7 never exceeds 100 °C, which means that the material was purchased with Cr$_{23}$C$_6$ carbides in the microstructure. This is not a problem for high temperature applications, since intergranular chromium carbides improve the creep resistance, and high carbon austenitic steels are usually selected to high temperature services [7]. In the fabrication process the mirror plate was probably hot worked, and the Cr$_{23}$C$_6$ carbides precipitated during slow cooling. A final solution treatment was not performed.

The influence of Cr$_{23}$C$_6$ precipitation on the SCC susceptibility is an interesting discussion. Although Muralleedharan et al. [8] have found a positive effect of intergranular chromium carbides, the majority of the research works on this matter have
Fig. 11. EDS spectra of the crack tip showing Cl, Ca and S peaks.

Fig. 12. Microstructure of intergranular chromium carbides, revealed with 10% oxalic acid solution (electrolytic, 8 V, 60 s): (a) region R4 and (b) region R7.
concluded that the sensitization decreases the SCC resistance of austenitic stainless steels [9–11]. Bhaumik et al. [12] attributed the failure by SCC in austenitic stainless steels tubes to the sensitization in the heat affected zone.

4. Conclusions and solutions proposed

Two cracks were observed in the inert gas generator mirror plate of AISI 310 steel. The main conclusions of the failure analysis are:

- Intense sigma phase precipitation observed in the hot zone caused hardening and severe embrittlement.
- The cracking in the hot zone of the mirror presented fatigue striations. Since the equipment works intermittently, the mirror plate was subjected to large temperature variations which promoted thermal fatigue initiation and propagation.
- Intense carbide precipitation was observed in the cold zone, suggesting that the material was purchased in a sensitized condition. Part of this region was refrigerated by seawater, which caused the nucleation and propagation of stress corrosion cracks (SCC).

The solution proposed to avoid cracks in the hot zone is to use ceramic lining in the mirror in the inside surface of the chamber. This would minimize the temperature gradient across the thickness and the temperature variation during the intermittent operation. The ceramic lining would also reduce the sigma phase formation [3], enhancing the life of the mirror.

The solution proposed to avoid the failure in the cold zone is to use treated water to refrigerate the mirror, instead of seawater which contains high Cl− concentration. The material must be purchased in the solution treated condition, since the intergranular chromium carbides (Cr23C6) seem to increase the susceptibility to SCC.

References