ELCOGAS IGCC POWER PLANT IN SPAIN: EFFECT OF THE GASIFIER ENVIRONMENT ON THE HIGH ALLOY STEEL PERFORMANCE

A.M. Lancha*, M. Alvarez de Lara*, D. Gómez-Briceño*, P. Coca**

* CIEMAT. Avda. Complutense, 22. 28040-Madrid (Spain)
**ELCOGAS, S.A., C. T. GICC Puertollano. Carretera Calzada de Calatrava a Puertollano, km. 27. 13500-Puertollano, Ciudad Real (Spain)

Abstract

The ELCOGAS power plant in Puertollano (Ciudad Real, Spain), with a gross electrical output of 335 MWe, is the world’s largest capacity Integrated Gasification in Combined Cycle (IGCC) power plant. ELCOGAS is a Spanish joint stock company whose shareholders are European utilities and capital good suppliers from European countries.

The gasification is based on the PRENFLO pressurized entrained-flow process with dry fuel dust feeding. The high-pressure (HP) evaporators, located in the same pressure vessel of the gasifier, are one of the most critical components in terms of materials in the gasifier. The corrosion performance of the austenitic Sanicro 28 alloy tubing exposed to the aggressive raw gas in the HP evaporators is being evaluated. For this purpose, destructive examination of calibrated test samples, installed at various accessible locations and removed after different exposure periods, is being performed. Corrosion results of Sanicro 28 samples with more than 11200 operating
hours are available and show, in general, low corrosion metal losses in the high
temperature gasifier atmosphere.

**Key words:** Sanicro 28, coal gasification environment, high temperature corrosion

1. INTRODUCTION

The ELCOGAS power plant in Puertollano (Ciudad Real, Spain), with a gross
electrical output of 335 MWe, is the world’s largest capacity Integrated Gasification in
Combined Cycle (IGCC) power plant. ELCOGAS is a Spanish joint stock company
whose shareholders are utilities from Spain, France, United Kingdom, Italy and
Portugal, and capital good suppliers from Spain and Germany. The power plant
consists basically in three islands jointly designed and integrated into the process:
the gasification island, the air separation island and the combined cycle island.

The gasification island is composed essentially of the gasification unit that is based
on the PRENFLO process (Pressurized Entrained Flow) with dry fuel dust feeding.
The gas is produced by the reaction of coal with oxygen at high temperatures (up to
1600ºC). The whole system is pressurized at 25 bar. The plant is operating with a
50% mixture of local high ash coal and high sulfur petroleum coke. The ash content
of the coal is higher than 45 wt. % and the sulfur content of the petroleum coke
higher than 5 wt. %. The resulting coal gas is rich in CO and H₂, which, as such, has
low oxygen partial pressure and potentially high carbon activity. However, the
presence of H₂S, COS, HCl, ash particles, etc. in the raw gas is the major cause of
concern with regard to the maintenance and integrity of the structural components,
since this raw gas is very aggressive towards materials in terms of corrosion by sulfidation/oxidation during operation and downtime corrosion during shut-downs and start-ups. The plant is operating in such a way to reduce the risk of downtime corrosion by flushing with nitrogen during shutdowns.

The high-pressure (HP) evaporators, located in the same pressure vessel as the gasifier, are critical components in terms of materials in the gasifier. They consist in some Sanicro 28 tubing banks that experience metal temperatures up to 400°C. The corrosion performance of the austenitic Sanicro 28 alloy tubing exposed to the aggressive raw gas is being evaluated. For this purpose, destructive examination of calibrated test samples installed at various accessible locations in the gasifier and removed after different exposure periods is being performed. Corrosion results of alloy 28 samples with more than 11200 operating hours are available and are presented in this paper.

2. EXPERIMENTAL

The raw gas composition can be seen in Table I and a simplified scheme of the gasifier and the high pressure heat exchangers is shown in Fig. 1. The locations of the test panels (A, B and C) and the thermal conditions estimated in each panel are also indicated in this figure. The test panels consist in some test samples, which are small lengths of accurately measured boiler tubing, welded together to form one straight length of tubing. The test panels are then welded to two neighboring tubes (welds made along the fins) in order to protect test samples when extracting them.
Panels A and C are installed in the HP membrane wall and each one is constituted by one panel of OD 38 mm finned circular tubes with 5.6 mm wall thickness. The estimated metal temperatures of the hot side of these tubes, the side facing inwards the center of the gasifier, are 359ºC and 334ºC respectively. No information on the thermal conditions of the cold side of the tubes installed in panels A and C is available. Panels B consist in four panels, located at 90º positions around the quench pipe. They are made of finned circular tubes of OD 57 mm and 7.1 mm wall thickness. The hot and cold sides of these test samples are respectively facing towards the center of the gasifier and towards the HP membrane wall. The estimated metal temperatures at the hot and cold sides of the B tube samples are 375ºC and 339ºC respectively.

Up to now samples with 3000, 4100, 7000, 8300 and 11200 operating hours have been examined. Results of samples up to 7000 operating hours have been previously described [1]. Three samples with 8300 operating hours have been studied, one for each test panel A, B and C, and a sample from a panel B with 11200 operating hours has been examined. After exposure, the samples were cross-sectioned and polished for microscopic analysis using both optical and scanning electron microscopy (SEM). In order to determine the gas-side corrosion, maximum metal losses were measured at many locations around the 360º of circumference of each sample, in all the zones where material degradation was observed in the micrographs taken at a magnification of x500. Corrosion scale analyses were performed using mainly the EDS attachment to the SEM. In addition, AUGER analyses in depth were carried out at the external surface of the samples to
determine the composition of the corrosion scale layers. Depth composition profiling was obtained by sputtering with an Ar ion gun.

3. RESULTS

Visual Examination

Up to 7000 operating hours mainly colored deposits (gray-white-orange-brownish) were observed at both sides of samples from the panels B and at the hot side of samples from the panels A and C. Cold sides of these last samples showed dark coloration [1]. However, after 8300 and 11200 operating hours all the samples, independently of the panel A, B and C, showed only a gray coloration at both sides.

SEM/EDS Examination

In order to determine the morphology and composition of the corrosion scales, the external surface of all the samples, at the hot and cold sides, was examined by SEM/EDS.

After 8300 and 11200 operating hours samples from panels B, both hot and cold sides, and the hot side of the sample from panel A (8300 operating hours) showed an outer layer of aluminosilicates of K and Ca, with a typical spherical morphology, embedded in a sulfide matrix, Fig. 2(a). In addition, a layer with different morphology, as a nano-belt like morphology, formed mainly by S and Ge with minor Sb and As was detected under the outer layer in many locations of these samples, Fig. 2(b).
These characteristics were also observed in the examined samples up to 7000 operating hours [1].

Cold side of the sample from the panel A and both sides of the sample from the panel C, both with 8300 operating hours, showed different appearance at the external surface, Fig. 3. Scale spallation occurs and three layers of cracked corrosion scales can be differentiated. The most external layer (A) is composed basically by aluminosilicates. The intermediate layer (B) is formed mainly by Fe and Ni sulfides, containing also Ge and As. The internal layer, in contact with the base material seems to be a Cr oxide layer with some Fe(Ni) sulfides.

On the other hand, in general, higher concentrations of As could be observed at the cold sides of the samples from the panels A and C, while higher concentrations of Ge were detected at the hotter sides: both sides of the samples from the panels B and the hot side of samples from the panel A.

**AUGER Analysis**

Auger analyses were carried out at the external surface of all the samples (hot and cold sides). As this technique is a surface analysis technique, previously, the most external deposits were mechanically removed from the samples.

No significant differences were observed in the composition of the inner scale layers in all the samples, neither at the hot nor cold sides. An example of the AUGER results is presented in Fig. 4 that corresponds to the hot side of the sample from a
panel B with 11200 operating hours. In general, the outside layer contains Al, Si, Ca and K, together with some S, Fe and Ni, but without Cr. Below this outside layer, the concentration of S increases, reaching a maximum, pointing out a sulfide layer. An inner oxide layer (containing some sulfide) is observed at the interface metal/scale.

Metallography

The damage detected in the samples after 8300 and 11200 operating hours consisted in a shallow wastage accompanied by some localized corrosion. Similar characteristics were observed in the examined samples up to 7000 operating hours [1]. However, it is worth pointing out that the cold side of samples with 8300 operating hours from panels A and C were clearly more degraded by pitting and wastage than the rest of the sample sides, Figs. 5(a) and (b). In addition, an area with pitting was also detected at the hot side of the sample from a panel B with 11200 operating hours, Fig. 5(c). On the other hand, it is worth standing out that multitude of cracks with different orientations were detected in the corrosion scale layers in all the samples, with 8300 and 11200 operating hours.

A dark gray layer (2-6 μm thickness) in contact with the base material was generally seen in all metallographic samples. However, in some pitted areas this layer was not observed. Above the dark gray layer, the presence of corrosion scales with lighter coloration was observed. The typical aluminosilicates spheres were embedded in this last layer. In some locations, alternate dark and light gray layers were observed what could indicate different operation periods or environmental changes during operation.
Quantitative results of the gas side corrosion are shown in Table II. For comparison, quantitative results of samples up to 7000 operating hours are also included. The results are average values between hot and cold side in each sample. In spite of the fact that in some cases maximum values of metal losses are clearly higher than average values, if only average values are considered no significant differences can be established related to the behavior of the samples in the different test panels. These results are presented graphically in Fig. 6. Average values show parabolic corrosion kinetics in all panels and only a slight increase of the metal loss values are observed for the higher operating hours. However, maximum metal loss values in panel C, that are related to the presence of pits, seem to exhibit a linear corrosion kinetic.

**Analyses in damaged areas**

EDS analysis and X-Ray mapping were performed in the degraded areas detected in the metallographic examination. As an example Fig. 7 shows the obtained results in a zone of a sample from a panel B (8300 operating hours, hot side). The presence of spherical aluminosilicates in the outer deposits can be differentiated. The scales of light gray coloration detected in the metallographic examination correspond to sulfides, being the outer ones mainly of Fe and Ni, and the inner ones almost exclusively of Fe. The dark gray layer in contact with the base material is clearly rich in Cr and contains some S. This points out to a Cr oxide layer containing some sulfides (mainly Fe sulfides). In addition, significant amounts of Ge are detected. On the other hand, cracks at the interface scale/base material and perpendicular to this interface, at the corrosion products inside the base material, can be observed.
Fig. 8 shows the composition and scale cracks detected in an area with the alternate layers described in the metallographic examination. Partial detachment of the corrosion scales parallel to the interfaces oxides+sulfides/sulfides can be seen. In addition, several cracks perpendicular to the base material exist in the sulfides layer (light gray layer). Dual layer scales formed in laboratory during tests including aqueous corrosion and subsequent re-exposure to syngas at high temperature are reported in the literature [2]. A composite scale with alternating iron sulfide and chromium oxide layers is observed in these tests.

Finally, it is worth emphasizing that no Cl has been detected in all of the damaged zones analyzed.

4. DISCUSSION

The raw gas environment from coal gasification is rich in CO and H₂. The main corrosive impurity in fuel is sulfur, which during gasification produces H₂S and COS. Most coals contain also minor quantities of chlorine that produces HCl in the syngas. The hot raw gas is very aggressive and corrosion of metal components by oxidation/sulfidation at elevated temperatures during the operation, or by Down Time Corrosion (DTC) when the plant is cooled down can occur. This last type of corrosion results from either acidic condensation from the syngas or hygroscopic deposits when the system is opened to the atmosphere [2,3].
No significant differences were found, neither quantitative nor qualitative, in the observed degradation, on both hot and cold sides, for any test samples from the different test panels A, B and C up to 7000 operating hours [1]. However, after 8300 operating hours the presence of more and larger areas degraded by pitting at the cold side of the samples from the panels A and C, that are the colder sides of all the panels, seems to indicate processes of Down Time Corrosion (DTC) during start-up and shut-downs. The scale spallation observed in these sides is also in accordance with this type of corrosion. However, an area with pits has also been observed at the hot side of the sample from panel B with 11200 operating hours. The fact that Cl has not been detected in the damaged zones by pits could be due to the volatility of the chloride species during the operation at high temperature.

In accordance with the literature, increased corrosion by pitting is generally found when samples are allowed to cool down to room temperature, specially in environments with relatively high steam contents. Macroscopically such samples are characterized by extensive scale spallation [2,3]. The exposure to moisture during downtime tends to concentrate chlorides and aqueous corrosion products at the scale-metal interface that can cause spallation of the chromium oxide rich scales. Thus, protective parabolic corrosion kinetics can change to more linear rates due to repeated scale spallation [2]. Frequent scale spallation could be the major cause of accelerated corrosion rather than aqueous corrosion during downtime alone [3]. Moreover, according to some authors [4] the presence of sulfides on the scale and sulfide channels in the scale cause high susceptibility to dew-point corrosion during down-times of the plant.
In the present work, considering the average metal loss values, parabolic corrosion kinetics has been observed in all the panels, see Fig. 6. Parabolic kinetics describe high temperature corrosion where the rate determining step is diffusion [5]. This is associated with internal penetration into the substrate of corrosive species, such as oxygen and sulfur, to form the most stable precipitate possible. Internal penetration occurs only if the reactive component of the substrate diffuses outwardly more slowly than the gaseous species diffuses inwardly. Other authors have also been reported parabolic corrosion kinetics of Sanicro 28 in coal gasification atmospheres, especially after long term exposure, and lower metal losses than other austenitic materials [3]. This has been attributed to the Mo content in the alloy.

A trend to linear corrosion kinetic seems to exist, specially in panel C, considering maximum metal loss values related to the presence of pits. Linear rate kinetics are most often associated with materials that do not form protective scales or whose scales are highly porous, crack-prone, or poorly adherent thus experiencing a relative constant surface area to the corrosive environment [5]. At this point, it is worth standing out that in some isolated areas with pits of the examined samples in this work, the adherent layer in contact with the base material composed by chromium oxide/iron sulfide has not been detected.

On the other hand, the temperature effect in the corrosion of Sanicro 28 can be seen in Fig. 9 where the corrosion rate of Sanicro 28 non-cooled sample tubes versus temperature from the German demonstration plant based on the PRENFLO process [6] is represented. No significant changes of corrosion values are observed in the temperature range of 320º-400ºC, temperature effect becomes important from 450ºC.
The maximum and average metal losses values corresponding to all examined ELCOGAS samples, tested in the panels A, B and C where the metal temperature range varies from 334ºC-375ºC, are also included in this figure and agrees with the PRENFLO data.

As regards the Sanicro 28 metal losses in gasification power plants, the existing data from the literature are limited and their comparison is quite difficult since they have been obtained in very diverse conditions, such as, gasification process, gas composition, chloride presence, downtime corrosion periods, cooled or non-cooled samples, fuel used, etc. Fig. 10 summarizes all Sanicro 28 metal loss available data, showing a wide dispersion because of their different origin [3,6,7]. Despite this dispersion, higher corrosion metal losses are observed at high temperatures. Obtained metal loss values from ELCOGAS sample tubes up to 11200 operating hours, showing in general lower metal loss values than other data, are also included in this figure.

Aluminosilicates showing their characteristic morphology of spherical particles, and embedded in Fe and Ni sulfide matrix have been observed in the outer layer of the corrosion scales. Under this one, another sulfide layer with different morphology, as a nano-belt like morphology, formed mainly by Ge and with lower concentrations of Sb has been detected in many locations of the samples from panels B, both sides, and at the hot side of the sample from panel A. In addition, As has been observed in the sulfide layers, though it can not be affirmed being associated with S. It must be pointed out that in the present work, despite heterogeneity of corrosion products, higher concentrations of As could be seen on sample tube cooler surfaces, i. e. on
cooler surfaces of sample tubes from panels A and C, and higher amounts of Ge were observed at the hotter surfaces, i.e. both sides of samples from panels B.

In contact with the base material a very regular, homogeneous an adherent layer, which consists mainly in Cr oxide but also with Fe sulfides, has been observed. Fig. 11 shows a general scheme, which resumes the scale corrosion composition observed in all the examined sample tubes. This composition is typical of corrosion mechanism called type B corrosion in the literature [4,5]. Most of corrosion scales observed in alloys coming from gasification power plants respond to this corrosion mechanism.

5. CONCLUSIONS

- The corrosion metal losses detected in the Sanicro 28 samples up to 11200 operating hours are in general low enough to provide acceptable long term service of this material in the gasifier atmosphere of the ELCOGAS IGCC plant.

- In spite of metal loss average values are no significantly different among the panels, higher maximum metal loss values related to the presence of pits were observed at the cold sides of samples from panels A and C. In these locations, the metal temperatures are lower and, therefore, the possibility of down time corrosion is higher.

- The scale composition is characterized by oxidation of chromium in the alloy under an outward growing iron/nickel sulfide scale.
6. REFERENCES


Table I.- Raw gas composition (% vol. wet)

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Table II.- Maximum metal loss (µm) and average metal loss ± standard deviation (µm) detected in the samples.

| Operating hours | Panel A | | Panel B | | Panel C | |
|-----------------|---------|----------------|---------|---------|---------|
|                 | Maximum Value | Average Value | Maximum Value | Average Value | Maximum Value | Average Value |
| 3000            | 20      | 13 ± 4         | 16      | 13 ± 2  | 20      | 14 ± 4       |
| 4100            | 24      | 14 ± 4         | 23      | 14 ± 4  | 26      | 17 ± 4       |
| 7000            | --      | --             | 30      | 16 ± 6  | --      | --           |
| 8300            | 30      | 16 ± 5         | 30      | 17 ± 4  | 60      | 19 ± 9       |
| 11200           | --      | --             | 34      | 18 ± 6  | --      | --           |
Figure Captions

**Fig. 1.** Gasifier and high pressure exchangers. Location and photographs of the test panels.

**Fig. 2.** SEM/EDS examination of the sample with 11200 operating hours from a panel B, cold side.

**Fig. 3.** SEM/EDS examination of a sample with 8300 operating hours from the panel C, cold side.

**Fig. 4.** AUGER analyses on the sample with 11200 operating hours from a panel B, hot side.
(a) All elements detected normalized to 100%.
(b) All elements detected normalized to 100% but only represented Fe, Cr, Ni, Mo, O and S.

**Fig. 5.** Metallographic cross-sections of samples.
(a) 8300 operating hours. Panel A, cold side
(b) 8300 operating hours. Panel C, cold side
(c) 11200 operating hours. Panel B, hot side

**Fig. 6.** Average ± standard deviation and maximum values of corrosion losses of Sanicro 28 samples in panels A, B and C.

**Fig. 7.** X-Ray mapping and EDS analyses on a sample with 8300 operating hours from a panel B, hot side.

**Fig. 8.** EDS analyses on the sample with 11200 operating hours from a panel B, cold side.

**Fig. 9.** Temperature effect in the corrosion of Sanicro 28.
Fig. 10.- Corrosion loss versus time for Sanicro 28.

Fig. 11.- Composition of the corrosion scales detected in the examined samples.
Fig. 1. - Gasifier and high pressure exchangers. Location and photographs of the test panels.
Fig. 2.- SEM/EDS examination of the sample with 11200 operating hours from a panel B, cold side.
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Fig. 3.- SEM/EDS examination of a sample with 8300 operating hours from the panel C, cold side.
Fig. 4.- AUGER analyses of the sample with 11200 operating hours from a panel B, hot side.
(a) All elements detected normalized to 100%.
(b) All elements detected normalized to 100% but only represented Fe, Cr, Ni, Mo, O and S.
(a) 8300 operating hours. Panel C, cold side

(b) 8300 operating hours. Panel C, cold side

(c) 11200 operating hours. Panel B, hot side

Fig. 5.- Metallographic cross-sections of samples.
Fig. 6.- Average ± standard deviation and maximum values of corrosion losses of Sanicro 28 samples in panels A, B and C.
### Table 1: Elemental Analysis

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**Fig. 7.** X-Ray mapping and EDS analyses on a sample with 8300 operating hours from a panel B, hot side.
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<td>At %</td>
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<td>0.3</td>
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<td>43.5</td>
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<tr>
<td>B</td>
<td>--</td>
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<td>1.6</td>
<td>36.0</td>
<td>7.2</td>
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<td></td>
<td>--</td>
<td>36.0</td>
<td>1.9</td>
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<tr>
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<td>Wt %</td>
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Fig. 8.- EDS analysis on the sample with 11200 operating hours from a panel B, cold side.
Fig. 9.- Temperature effect in the corrosion of Sanicro 28.
Fig. 10.—Corrosion loss versus time for Sanicro 28.
Fig. 11.- Composition of the corrosion scales detected in the examined samples.

$\text{(Fe,Ni)S + Al, Si, Ca, K + Ge, As, Sb} \approx 2-6 \mu m$