CORROSION IN THE METALLIC SHELL OF HOT STOVES

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Abstract

This study proposes to verify and clarify the main corrosion mechanisms in industrial regenerators or hot stoves, widely used in the steel industry, indicating actions for its monitoring and control. The corrosion mechanisms in the upper parts, internally, are associated with the formation of siderite ($FeCO_3$) and sulfur, which also favors stress corrosion and occurrence of cracks. Different corrosion mechanisms may be associated in the lower parts of the regenerators and may be accelerated by the presence of flue gas condensate and sulfur. Practical actions were proposed for the correct measurement and control of good operating conditions of the regenerators.

Keywords: *corrosion, hot stoves, sulfur, fuel gas quality*

1. INTRODUCTION

The Brazilian steel industry, in similarity with other parts of the world in this segment, has evolved rapidly in its operational results using applied technology and technical-scientific knowledge to overcome the not so recent challenges of competitiveness, excess capacity, cost reduction and environmental issues, the latter being well contextualized by Fernandes [1], such as carbon dioxide emissions and energy efficiency. Within these industrial plants, in blast furnaces, the regenerators have a useful life cycle between 30 and 40 years and play an important operational and financial role for the proper functioning of these reactors.

The understanding of the main mechanisms of corrosion of the metallic casing of the regenerators is of utmost importance so that operation and maintenance engineers and technicians can periodically monitor these components and define the best strategies for conservation and use of this equipment, to avoid failures or prolonged and early interruptions of production for revamps that have a relevant impact on the master plan of the companies in the steel segment. As the few works related to regenerators have focused mainly on refractories or stress corrosion mechanisms [2,3,4], the objective of the current paper is to present an overview of the corrosion mechanisms of the metal casing and correlate them to the process conditions and failures commonly found in this equipment.

Hot stoves or Cowpers regenerators, which in this paper will be referred to only as regenerators, are widely used equipment for heating the air used in blast furnaces, operationally reaching temperatures between 1,150 and 1,200 ºC of blast air. To perform their function, the regenerators work both as pressure vessels and heat exchangers in alternating cycles to air and gas. Each regenerator is formed by a metallic casing, normally made of structural steel, refractory materials, and a set of special valves, with the function of facilitating heat exchange in a low-pressure system (normally between 3 to 5 bar, gauge pressure).

As it can be seen in Figure 1, a burner, which can be of ceramic or metallic type, combusts a mixture of blast furnace, coke oven gas or natural gas with air in the combustion chamber. Then, the combustion gases transfer energy and heat the stacking of the checker chamber (refractory bricks whose stacking generates many smaller diameter ducts to maximize the heat exchange area), before exiting through the smoke channel. In the air cycle, combustion is stopped, the smoke channel is also closed and then the blowing air supply is opened and heated by the previously heated checker chamber and directed to the blast furnace.

Typically, a blast furnace has a configuration of three to four regenerators, which work in alternation in these gas/blow cycles, producing a repeatability of thermal and mechanical oscillations in the entire assembly during the life of the equipment [5-7].

Typical flue gas compositions as a function of the percentage of oxygen in the air used in combustion and main regenerator operation parameters can be seen in Table 1.

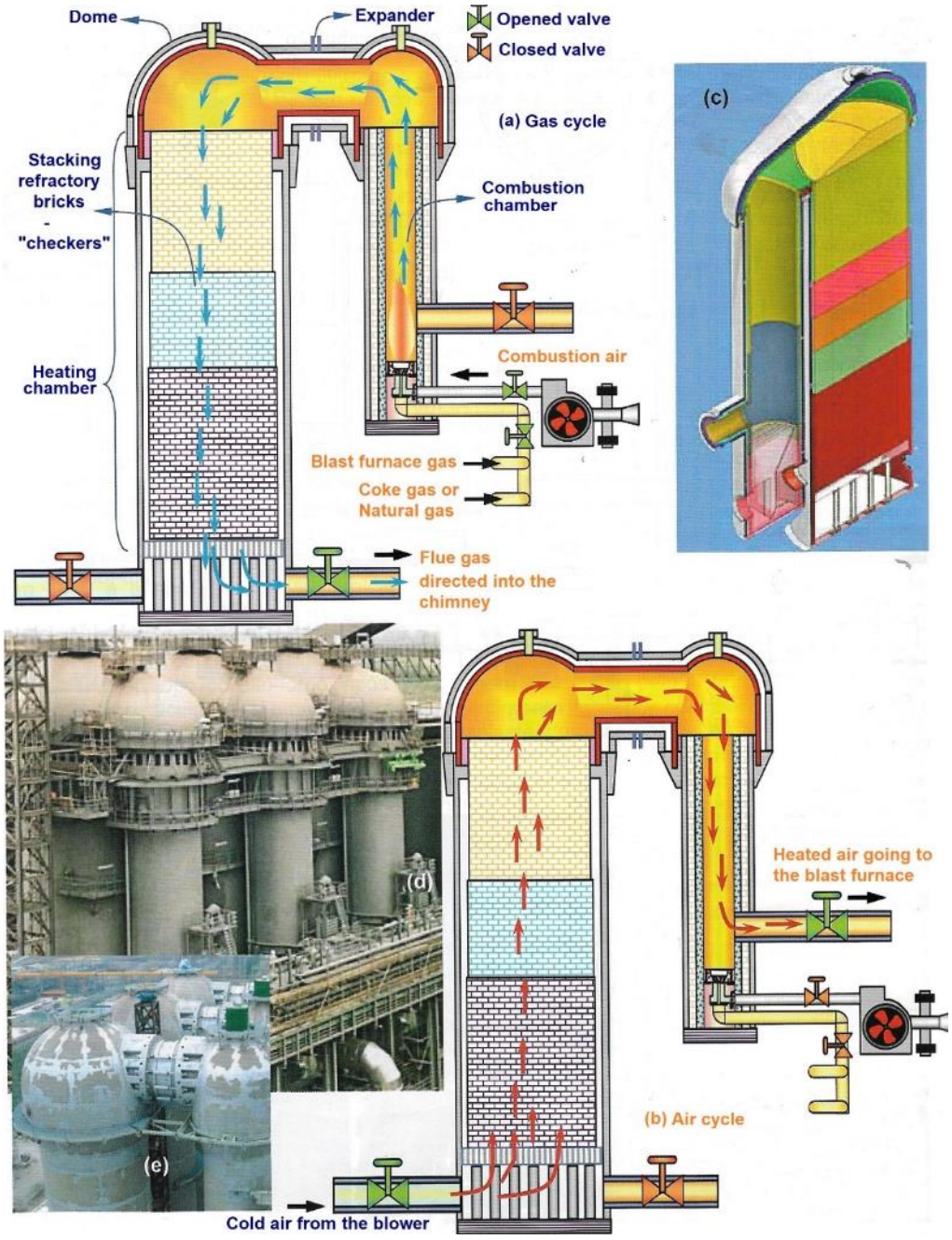


Figure 1. Cowper regenerator with external combustion chamber in two stages of operation: a) cycle with gas or heating, b) cycle with wind or blowing, c) another option for the dome, d and e) images of industrial installations, adapted from [5].

Table 1. Operating conditions of a regenerator and flue gas composition

	Unit	Normal air	22% O_2	23% O_2	24% O_2	25% O_2
O_2 in air+O_2	Vol.-%	20.7	22.0	23.0	24.0	25.0
Air	Vol.-%	100.0	98.3	97.0	95.8	94.5
O_2	Vol.-%	0	229.4	392.2	547.4	695.4
O_2 flow	Nm^3	1412	1445	1469	1491	1512
Flame temperature	ºC	17.0	17.5	17.9	18.2	18.6
Flue gas flow(C6)	kNm^3/h	31.8	31.6	31.5	31.4	31.3
Power	MW	18.0	18.6	19.0	19.4	19.7
Air+O_2 flow (C6)	kNm^3/h	14.0	13.6	13.2	12.9	12.6
Flue gas flow	kNm^3/h	28.4	28.4	28.4	28.4	28.4
Flue gas/(BFG+NG)	-	1.68	1.63	1.59	1.56	1.53
Flue gas analysis						
CO_2	Vol. %	24.1	24.8	25.4	25.9	26.4
N_2	Vol. %	66.5	65.5	64.8	64.1	63.4
O_2	Vol. %	0.7	0.7	0.7	0.7	0.7
H_2O (as vapour)	Vol. %	8.7	9.0	9.1	9.3	9.5
Fire time C6	Vol. %	105.9	102.1	99.6	97.3	95.2

2. EXPERIMENTAL

To remove the sample from the top, a 63.5 mm diameter (2 ½) STARRET rapid steel cup saw and a magnetic base drilling machine with variable rotation were used. The equipment was done at a relatively lower speed aiming to preserve the microstructural characteristics of the steel.

For thickness measurement by ultrasound, it was used equipment TM510-SE internal reference standard PT016 ME (similar to current NBR16616 of 07/2017), measuring head angle of 0º and grease as coupling. Imaging and analysis of the oxide layers were performed in a ZEISS EVO MA10 Scanning Electron Microscope.

3. CORROSION IN REGENERATORS

Schaub et al. [2] divided the attack on the metal casing of regenerators mainly into two corrosion groups. The first mechanism of attack to the metallic casing leads to a reduction in the thickness of the metallic casing of the regenerator. Figure 2 and Table 2 shows an example of a field ultrasonic measurement of the lower region of a regenerator's metallic casing with thickness reduction (uniform corrosion), the plate being 26 years old and 20 mm thick in the original design. In point 2, there was the greatest loss of thickness in the period, a 63% reduction of the original thickness of the plate, or 0.48 mm/year. Points 2 to 4 presented the highest steel corrosion rates. At point 12, the lowest thickness loss of only 0.06 mm/year occurred.

Corrosion on metallic casings in Cowpers type regenerators in structural steels, such as ASTM 242 and similar steels built in the 80's and earlier, have an average corrosion rate expected in design in a range of 0.35-0.40 mm/year (depending on the design and the standard of the country of origin used for

pressure vessels - source: author), especially keeping the working and environmental conditions to which this equipment is submitted similar to the design.

These differences in corrosion rates can be explained due to the presence or not of flue gas condensate in some parts of the internal face of the plate, which eventually crosses an eventual failure of the refractory lining and, when condensation occurs, forms an electrolyte, even if temporary during return in operation after shutdowns, for example.

The lower regions of the regenerators (Fig.2 and 3) are also the ones with the lowest temperature of the shell, where the moisture of the combustion gases from the mixture of fuel gases and combustion air can be partially in liquid phase, especially on the cold face of the refractory and its interface with the shell. For this, it is sufficient that the flue gas temperature be lower than the gas dew temperature, which according to Kassas et al. [3] has been estimated at 180°C.

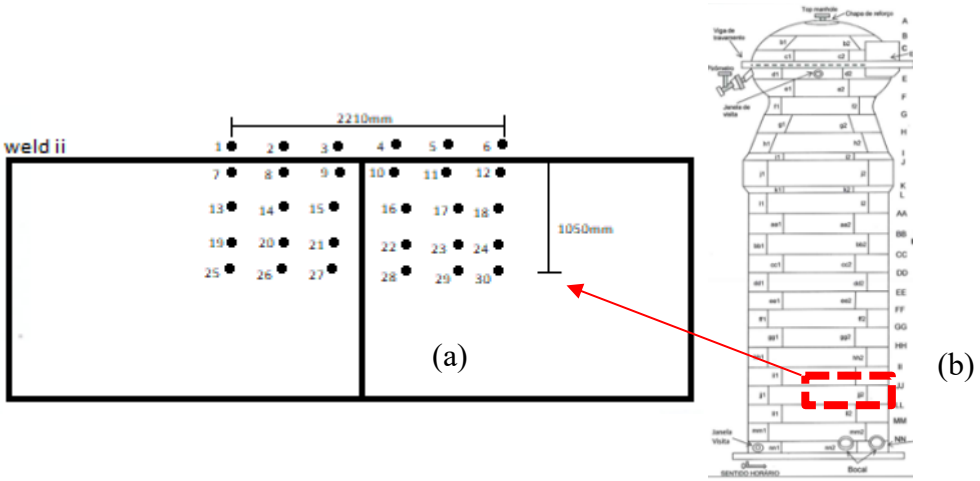


Figure 2. Loss of thickness of the metallic housing of a regenerator measured by ultrasound (a) detailed view and (b) general view of hot stove

Table 2. Ultrasonic thickness measurements as already detailed in the experimental part

Point	Thickness (mm)	Point	Thickness (mm)
1	11.2	16	17.8
2	7.4	17	17.1
3	8.4	18	10.3
4	9.5	19	12.3
5	13.7	20	10.3
6	11.2	21	16.8
7	11.5	22	16.8
8	11.3	23	13.3
9	11.1	24	12.4
10	15.6	25	13.2
11	17.3	26	11.4
12	18.4	27	15.8

13	10.8	28	16.7
14	11.8	29	17.3
15	17.3	30	16.8

The corrosion of carbon steel in environments containing CO_2 generates a corrosion product called siderite ($FeCO_3$) that can form a passive layer or not, depending on the chemical conditions of the electrolyte such as pH, carbonate concentrations [8,9] and oxygen saturation, among other factors. The reactions involved in this mechanism, where ad means adsorbed compound, are:

$$CO_{2(ad)}+H_2O_{(l)} \leftrightarrow H_2CO_{3(ad)} \quad (1)$$

$$H_2CO_{3(ad)}+e^- \leftrightarrow H^+_{(ad)}+HCO_3^-{}_{(ad)} \quad (2)$$

$$Fe^{2+}_{(aq)} + HCO_3^-{}_{(aq)} \leftrightarrow FeCO_{3(s)} \quad (3)$$

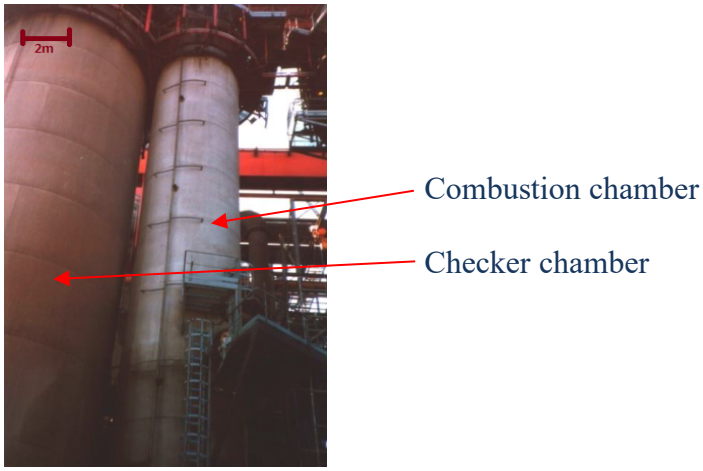


Figure 3. External plating of the checkers chamber (left) and combustion chamber (lower parts) for a regenerator with external combustion chamber

Corrosion due to the presence of sulfur [10-13] usually occurs in small, localized regions on the metal surface, forming pits. It occurs on carbon steel and on metals or alloys that passivate or form films of corrosion products on the metal surface. Behind the flue gas condensate electrolyte, an electrochemical cell of the localized type initiated with small anodic and large cathodic areas is formed, thus constituting a system that locally allows and/or favors material penetration.

It can be stated that, the lower the pH the higher the probability of sulfur corrosion. The formation and/or precipitation of ferrous sulfide (FeS) in the vicinity of the pitting regions can be explained by the increased concentration of OH^- ions that promote, consequently, the increase of the pH value and the alkalinization of the reaction [12]:

$$SH^-_{(aq)} + OH^-_{(aq)} \rightarrow H_2O_{(l)} + S^{2-}{}_{(aq.)} \quad (4)$$

The concentrations of $[Fe^{2+}]$ and $[S^{2-}]$ reach the saturation limit of these ions in the medium and promote the precipitation of ferrous sulfide based on the reaction [11]:

$$Fe^{2+}_{(aq)} + S^{2-}_{(aq)} \rightarrow FeS_{(s)} \quad (5)$$

Zimer [14] states that the effect of H_2S on the corrosion of carbon steel also consists of the galvanic pile formed between the cementite and ferrite phases. The heterogeneous deposition of FeS on the steel surface does not produce an efficient passivation layer. In this case, the following reactions can occur [14]:

$$Fe_{(s)}+H_2S_{(aq)}+H_2O_{(l)} \leftrightarrow FeSH^-_{(ad)}+H_3O^+_{(aq)} \quad (6)$$

$$FeSH^-_{(ad)} \leftrightarrow Fe(SH)_{(ad)}+e^- \quad (7)$$

$$Fe(SH)_{(ad)} \leftrightarrow FeSH^+_{(aq)}+e^- \quad (8)$$

$$FeSH^+_{(aq)} \leftrightarrow FeS_{1-x}(s)+xSH^-_{(aq)}+(1-x)H^+_{(aq)} \quad (9)$$

$$FeSH^+_{(aq)}+H_3O^+_{(aq)} \leftrightarrow Fe^{2+}_{(aq)}+H_2S_{(s)}+H_2O_{(l)} \quad (10)$$

In the upper parts of the regenerators (Fig. 4), there is the chemical or dry oxidation of the metal housing first externally with the industrial atmosphere (air contaminated with NO_2 and SO_2) and then internally with the combustion gases (CO_2, CO, O_2, and H_2O), with the kinetics most favored by the higher temperatures.

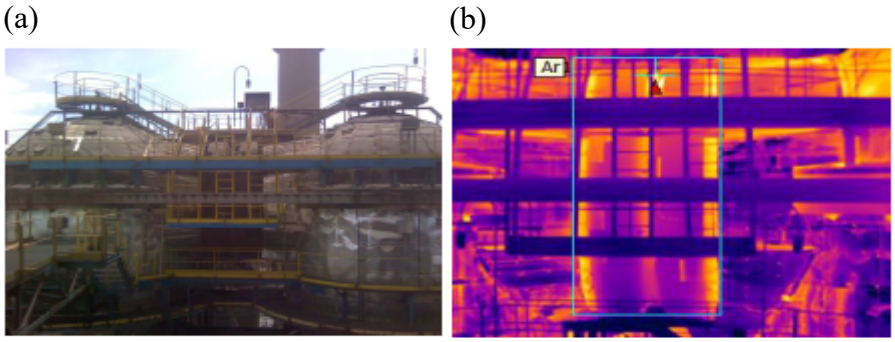


Figure 4. The interconnection between the combustion chamber and the dome (upper parts) for a regenerator with external combustion chamber: (a) general view and (b) thermographic detail view

The main reactions acting in the high temperature accelerated atmospheric corrosion reaction are:

$$4Fe_{(s)}+3O_{2(g)} \leftrightarrow 2Fe_2O_{3(s)}\,(\text{Temp.} < 400\ ^oC) \quad (11)$$

$$SO_{2(g)}+1/2O_{2(g)} \leftrightarrow SO_{3(g)} \quad (12)$$

$$SO_{2(g)}+H_2O_{(l)} \leftrightarrow H_2SO_{3(l)} \quad (13)$$

$$SO_{3(g)}+H_2O_{(l)} \leftrightarrow H_2SO_{4(l)} \quad (14)$$

$$2Fe_{(s)}+2H_2SO_{3(g)} \leftrightarrow FeS_{(s)}+FeSO_{4(l)}+H_2O_{(l)} \quad (15)$$

$$2Fe_{(s)}+H_2SO_{4(l)}+O_{2(g)} \leftrightarrow FeSO_{4(l)}+2H_2O_{(l)} \quad (16)$$

$$2FeSO_{4(l)}+\tfrac{1}{2}\,O2(g)+H_2SO_{4(l)} \leftrightarrow Fe_2(SO_4)_{3(l)}+H_2O_{(l)} \quad (17)$$

$$2Fe_{(s)}+3NO_{2(g)} \leftrightarrow Fe_2O_{3(s)}+3NO_{(g)} \quad (18)$$

The reactions of the steel in the higher regions with the internal environment of the regenerator are [15,16]:

$$4Fe_{(s)}+3CO_{2(g)} \leftrightarrow 2Fe_2O_{3(s)}+3CO_{(g)} \quad (19)$$

$$Fe_{(s)}+CO_{2(g)}+H_2O_{(g)} \leftrightarrow FeCO_{3(s)}+H_{2(g)} \quad (20)$$

Considering the presence of NO_2 and SO_2 from coke oven gas combustion typically with the presence of H_2S and NH_3 [15,16], one has:

$$H_2S_{(g)} + 3/2O_{2(g)} \leftrightarrow SO_{2(g)} + H_2O_{(g)} \quad (21)$$

$$SO_{2(g)} + 1/2O_{2(g)} \leftrightarrow SO_{3(g)} \quad (22)$$

$$SO_{2(g)} + H_2O_{(g)} \leftrightarrow H_2SO_{3(l)} \quad (23)$$

$$SO_{3(g)} + H_2O_{(g)} \leftrightarrow H_2SO_{4(l)} \quad (24)$$

$$2Fe_{(s)} + 2H_2SO_{3(l)} \leftrightarrow FeS_{(s)} + FeSO_{4(s)} + H_2O_{(g)} \quad (25)$$

$$2Fe_{(s)} + H_2SO_{4(l)} + O_{2(g)} \leftrightarrow FeSO_{4(l)} + 2H_2O_{(g)} \quad (26)$$

$$2FeSO_{4(s)} + ½\ O_{2(g)} + H_2SO_{4(l)} \leftrightarrow Fe_2(SO_4)_{(s)} + H_2O_{(g)} \quad (27)$$

$$NH_{3(g)} + 3/2O_{2(g)} \leftrightarrow NO_{2(g)} + H_2O_{(g)} \quad (28)$$

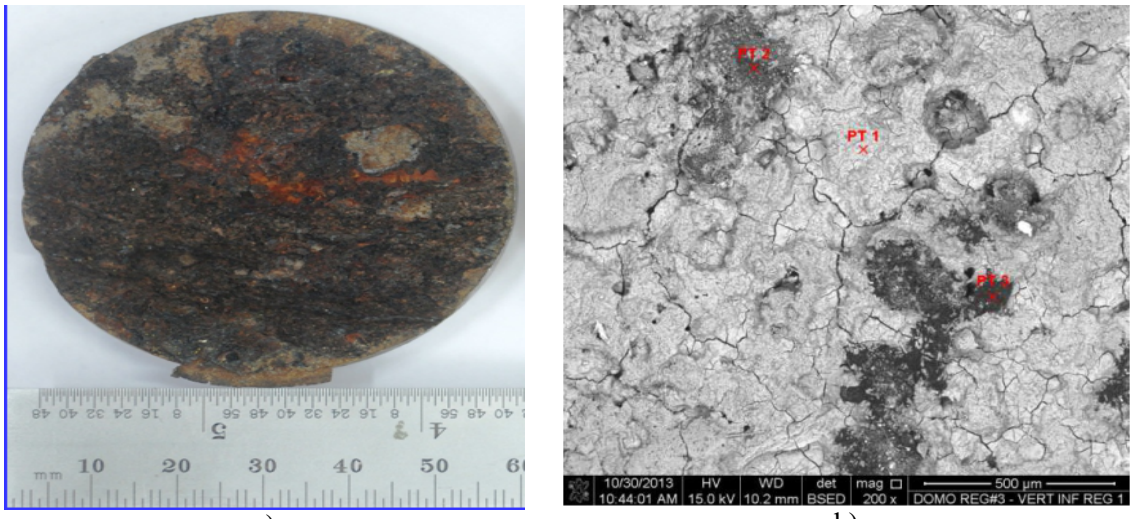
$$N_{2(g)} + 2O_{2(g)} \leftrightarrow 2NO_{2(g)} \quad (29)$$

$$2Fe_{(s)} + 3NO_{2(g)} \leftrightarrow Fe_2O_{3(s)} + 3NO_{(g)} \quad (30)$$

$$3NO_{2(g)} + H_2O_{(l)} \leftrightarrow 2HNO_{3(aq)} + NO_{(g)} \quad (31)$$

$$Fe_{(s)} + 2HNO_{3(aq)} \leftrightarrow Fe(NO_3)_2(s) + H_{2(g)} \quad (32)$$

A sample was taken from the metal casing of a regenerator in the dome part, showing the uniform corrosion appearance of the inner surface of the sample at Fig.5 (a), and by scanning electron microscopy, the micrograph shows different phases of corrosion products and fouling, referenced by points 1, 2 and 3 in Fig. 5 (b).



a) b)

Figure 5. Macro (a) and micrographic (b) visualization of the oxidation products of the upper part of the metallic housing of a regenerator

As a complement to the analysis and based on the selected points in Fig. 5 (b), the qualitative chemical composition of each phase of the corrosion products is presented in Fig. 6. The formation of siderite ($FeCO_3$) and sulfides (Fig. 6 a and 6 b) can be verified, correlating these oxidation compounds for the first time in the literature with this type of equipment. In Fig. 6 (b), the presence of sulfides and oxides of iron, aluminum and chromium can be visualized. Fig. 6 (c), at point 1, indicates the presence of sulfides and oxides of Al, Si, P, and Fe.

The spectrum of Fig. 6 (c), referring to point 2 indicates the presence of sulfides, iron carbonate, potassium and calcium, and oxides of aluminum, silicon, and phosphorus. The spectrum of Fig. 6 (c), point 3, shows high carbon content, but without the presence of oxygen and iron, and the presence of Si, Al, and Na.

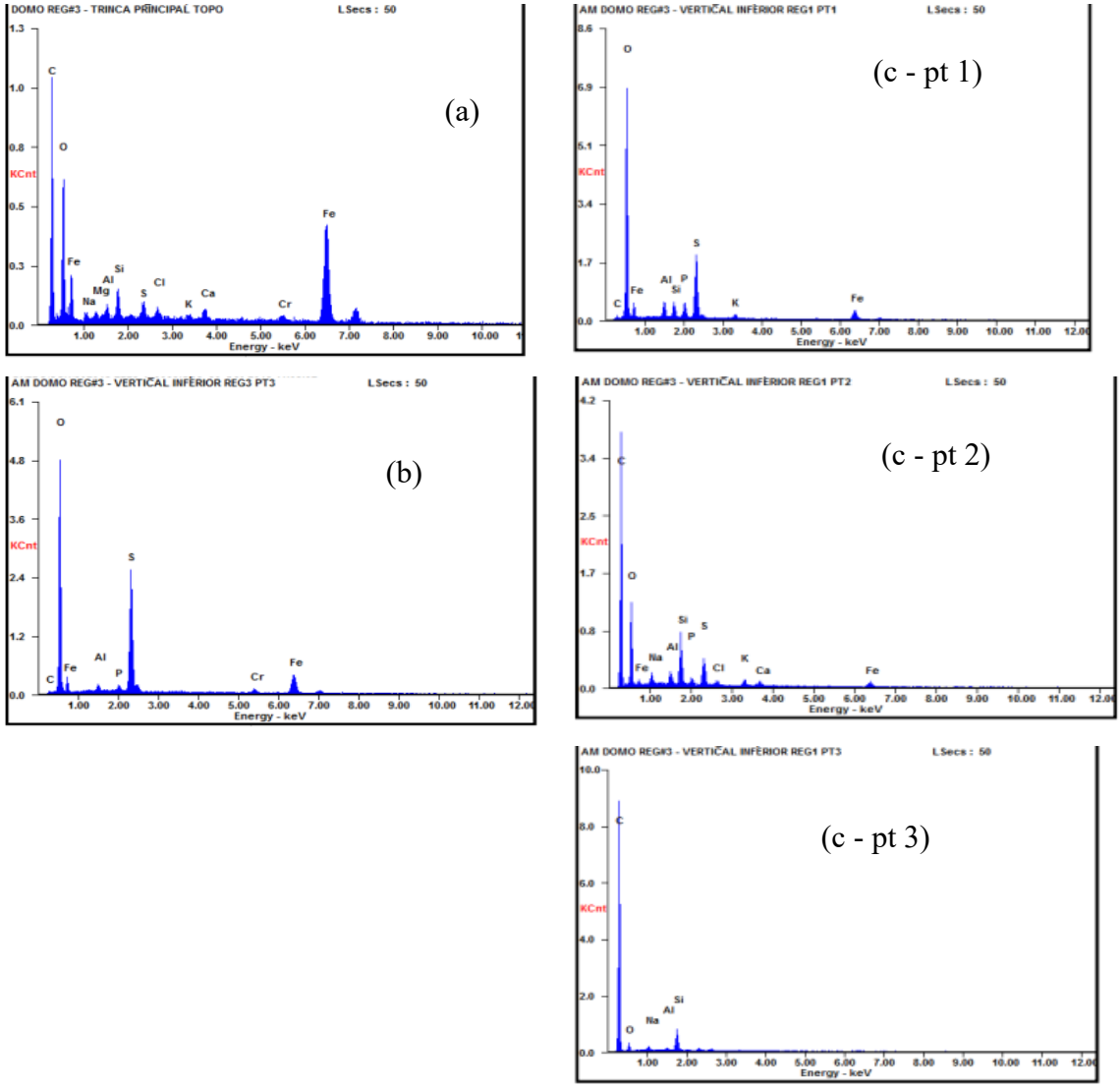
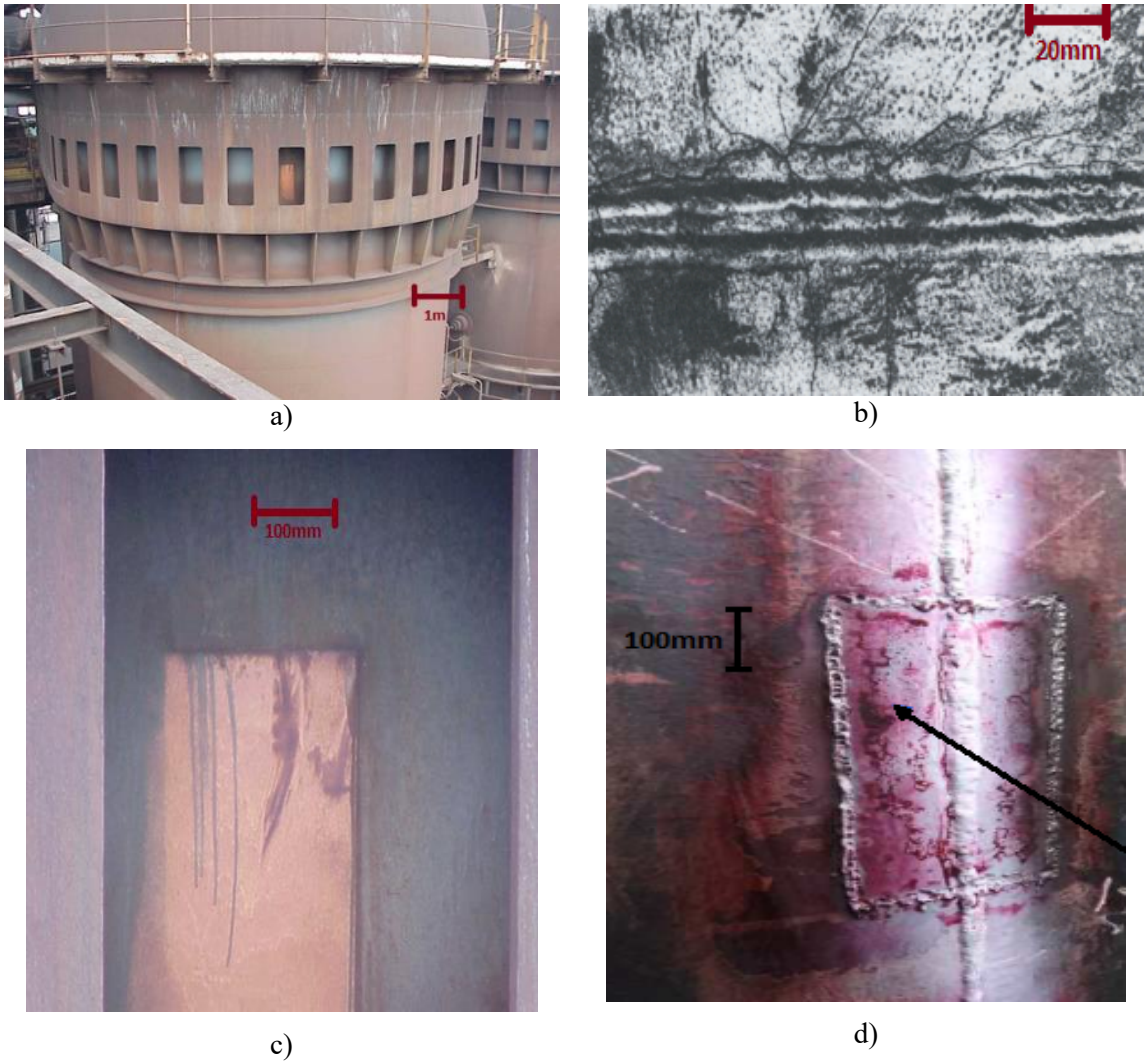


Figure 6. Energy dispersive spectroscopy results of oxidation products of the internal part of the regenerators:(a and b) typical results from upper parts, (c) points 1,2 and 3 showed at figure (6b)

The second group of corrosive attacks to regenerator metal casings classified by Schaub et al. [2] are stress corrosion cracking (SCC), as shown in Fig. 7 and very common in regenerators, especially near weld regions in the thermally affected zones (TAZs).

The cyclic operation for heating the air to be heated has its effect on the temperature oscillation on the regenerators' casing, generating mechanical stresses on the equipment's casing and the corrosiveness of the environment, mainly internal, predominantly acid, with the presence of sulfur and CO_2, has its influence on the interface of the reactions with the metallic casing surface and its corrosion products.



a) b) c) d)

Figure 7. Typical occurrences of cracking in the metal casing of regenerators caused by SCC: (a) general view, (b, c, and d) detailed view.

When the siderite ($FeCO_3$) layer collapses, either by mechanical stress or chemical dissolution, it can lead to exposure of the steel substrate and promote localized corrosion such as pitting corrosion [7,9], as can be seen in Fig. 8.

As pitting corrosion generates stress concentrators, cracks can be nucleated and propagated by different mechanisms.

In the 1980s, stress corrosion cracking (SCC) was identified to occur when regenerators unprotected from coatings were operated at dome temperatures exceeding 1,300°C and at high pressures [4]. Studies have revealed that the formation of NO_x from nitrogen in air increases exponentially when the temperature is above 1300°C [4]. Therefore, the formation of nitric acid (HNO_3) occurs, which is a very aggressive condensate produced in the electrolyte in contact with the regenerator housing, attacking especially areas of high tensile stress concentrations, such as: the thermally affected zones (TAZs) adjacent to the housing weld seams, and the weld seams themselves. This leads, over time, to progressive corrosion damage, continued crack formation, and ultimately to the failure of the regenerator as a pressure vessel. The main reactions involved in this mechanism are equations (29), (30), (31) and (32).

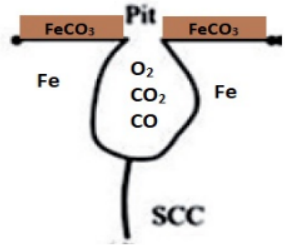


Figure 8. Mechanism of SCC formation after siderite ($FeCO_3$) removal and pitting formation, adapted from [13]

In general, the amount of NO_x generation increases dramatically between 1,450 and 1,500°C [4]. In terms of regenerator operation, the increased NO_x generation leads to a greater possibility of SCC in the metal casing, and therefore the dome temperature must be controlled to limit the phenomenon [4]. The more lagging ceramic burners installed in conventional regenerators usually have low mixing property of fuel gas and combustion air, compromising this control.

Because of this, when the regenerator is operated with temperature higher than 1400°C, even if below the theoretical range, localized regions of high temperature are generated in the combustion chamber resulting in a large amount of NO_x, as shown in Fig. 9. For this reason, the dome temperature is limited to 1400°C.

The mechanism of stress cracking in the presence of regenerator sulfur involves SO_2 from flue gas condensate, which forms sulfurous acid (H_2SO_3) that can be oxidized and form H_2SO_4, particularly when O_2 is present, as verified in equations (25), (26), and (27). As the condensate medium becomes acidic, generation of atomic hydrogen produced near the pitting and/or crack regions can occur by cathodic reduction of hydrogen ions.

Part of the generated atomic hydrogen, due to its small dimension, can penetrate through a pitting or a crack in the crystalline structure of the steel, going to lodge near the disagreements of the atomic structure, micro-cracks, inclusions, or voids. The presence of dissolved atomic hydrogen in the ferritic or martensitic matrix facilitates the movement of disagreements in the crystal structure of the material (4,7). Atomic hydrogen can be adsorbed on the crack surface, decreasing its surface energy, and contributing to crack propagation [15].

There are several factors that affect the likelihood of stress corrosion occurrence in metallic materials, such as H_2S concentration, temperature, total pressure, pH, exposure time, total applied stress, material properties (chemical composition, tensile strength, hardness, heat treatment, microstructures, etc.), and impurities and defects that may exist [17, 18]. MR 0175-2000 for carbon steel establishes requirements for selection of materials in the presence of H_2S.

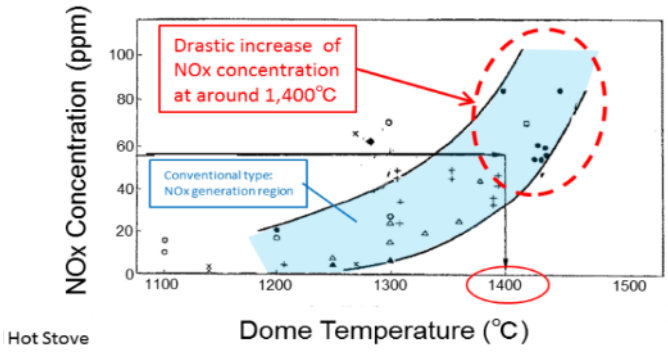


Figure 9. NO_x concentrations from combustion in metal burners, adapted from [4]

There are still gaps to be further investigated for better proof of the corrosion mechanisms of the regenerator casing, such as the use of advanced X-ray photoelectron spectroscopy (XPS) and glow discharge optical emission spectroscopy (GDOES) techniques for characterization of the oxides, nitrides and sulfides formed in the corrosion reactions and elemental analysis of composition profile. The study of the electrical properties of the regenerator casings, measurement of resistance/impedance, potentials, and evaluation of the influence of grounding on the electrical properties of this equipment are also relevant and the investigation of the particulate content of the gases used, the SO_x content of the flue gases and its influence on the corrosion kinetics of different structural or low alloy steels also deserves investigation.

4. CONCLUSIONS

This paper proposed to present and discuss the main corrosion mechanisms associated with the metallic casing of regenerators.

Uniform or generalized corrosion was observed in the regenerators. The greatest thickness loss observed over a 26-year period in a studied regenerator was a 63% reduction of the original plate thickness, i.e., a corrosion rate of 0.48 mm/year. The lowest thickness loss recorded corresponded to a corrosion rate of only 0.06 mm/year.

In the upper parts of the regenerators, internally, the corrosion mechanisms of the metal casing are present with the formation of siderite ($FeCO_3$), sulfides and that both favor stress corrosion.

Different mechanisms of metallic casing corrosion can be associated in the lower parts of the regenerators, being favored by the presence of electrolyte formed by the flue gas condensate.

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