Long-term exposure of austenitic steels and nickel-based alloys in lignite-biomass cofiring

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Abstract
The aim of this study was to assess the long-term impact that the addition of biomass provokes on superheater materials exposed to fireside corrosion environments. Alloys covering a broad range of commercially available materials were investigated. Their corrosion kinetics under different corrosive deposits and atmospheres was evaluated, and their corrosion products analyzed to deepen understanding of the underlying corrosion mechanisms. Therefore, three nickel-based alloys and three austenitic steels containing 20–24 wt.% Cr were tested at 650°C for 7,000 hr. The long-term exposure shows new mechanistic aspects of Type II hot corrosion that were revealed by accelerated material depletion. The formation of Ni–NiS eutectic and the formation of a Cr depleted zone close to the substrate corrosion product interface are indicative of the breakaway occurrence. Differences in the corrosion behavior are related to the balance of Ni, Mo, Co, and Cr and can serve as the material selection argument. The evaluation concluded with the finding that alloys presenting Mo and Ni might be preferentially used in fireside corrosion in the presence of biomass, whereas the use of austenitic steels suffer less corrosion if no biomass is present in the corrosive atmosphere.

KEYWORDS
austenitic steels, biomass cofiring, hot corrosion, lignite firing, Ni-based alloys

1 | INTRODUCTION
Conventional coal-fired power plants are still important to guarantee the supply of base-load energy without the fluctuations of alternative sources, such as wind and solar power. In an effort to reduce consumption of fossil fuels and greenhouse gas CO₂ emissions, cofiring of biomass has become a key option in coal-fired power plants. Although cofiring reduces the CO₂ footprint of the plant, it also introduces higher amounts of corrosive species such as chlorine into the system. Metallic components of energy conversion systems such as heat exchangers are affected by the fireside corrosion that such corrosive hot combustion gases and salt deposits provoke. At the same time, the efficiency level of the plant has to remain high, and thus have higher operation temperatures compared to pure biomass or waste incineration plants.

From the materials perspective, using biomass as a secondary fuel increases the demand on the corrosion resistance of the superheater tubes. So, materials used at the actual systems might not respond adequately to the new environments mainly due to higher corrosion rates, losses in wall thickness, and therefore accelerated mechanical failure.⁴⁻¹,²
The corrosion phenomena expected in such cases are commonly referred to as fireside corrosion. This is a Type II hot corrosion and occurs in the temperature range of 550–750°C, well below the melting point of pure sodium sulfate. The molten species are usually formed between sulfates of metallic substrate elements and the salt deposit. In Type II hot corrosion, hardly any depletion of protective elements of the substrate material is observed and usually corrosion is manifested in the form of pits.\[3\] The corrosion intensity usually increases up to around 700°C before going down again as per the typical “bell-shaped” function of the temperature.\[4\] This temperature range matches with the operation temperature requirements for cofiring systems.

Depending on fuel chemistry, different compounds are present in the deposits on the metallic surfaces of the components.\[5\] Especially damaging are the ionic compounds, such as sodium/potassium sulfates and chlorides. The reaction of these salts with protective metal oxide scales initiates the dissolution of the scales by a mechanism called fluxing.\[6\] Molten salts are widely studied, but solid salts can also cause scale fluxing if a chemical reaction is forming eutectic phases. Molten salts also facilitate fast transportation of corrosive species in the salt layer to reaction sites. So the presence of the salts, formation of protective oxide scales and only fluxing of transient scales takes place. This slow period at some point turns into much faster corrosion kinetics usually named as propagation. In the propagation stage, the salt or its aggressive corrosion products react with the scale to penetrate it and dissolve further elements of the substrate into the salt deposit.

The scientific community agrees that hot corrosion attack can be divided into two stages: incubation and propagation.\[4,7–9\] The initially observed slow corrosion is defined as the incubation stage. In this period, besides the presence of the salts, formation of protective oxide scales and only fluxing of transient scales takes place. This slow period at some point turns into much faster corrosion kinetics usually named as propagation. In the propagation stage, the salt or its aggressive corrosion products react with the scale to penetrate it and dissolve further elements of the substrate into the salt deposit.

In this study, the presence of HCl and KCl as typical biomass combustion products on fireside corrosion were studied up to 7,000 hr. These results were compared to typical lignite salt deposits and flue gases without such contaminants. This research question is highly relevant for actual plants because tubes subjected to increased corrosive attack require time consuming and costly unforeseen replacements of nickel-based alloys and steels.

## 2 | EXPERIMENTAL

Samples were machined from superheater tubes into 4-mm thick round shaped cuboids. The convex part represents the original tube’s outer surface (see Figure 1). This part was investigated in this study and was only cleaned in an ultrasonic bath using acetone to remove any surface contaminants such as lubricants from the mill to ensure “ready to use” surface conditions before exposure.

The nominal compositions and the main phases present in the microstructure of the tested austenitic steels and nickel-based alloys are shown in Table 1.

Samples of 15 × 10 × 4 mm were buried in alumina crucibles filled with the tested ashes, as shown in Figure 1. The crucibles were introduced in a horizontal furnace. The furnace’s hot zone was set at 650 ± 5°C in accordance with ISO norms for high-temperature corrosion tests.\[17,18\] The flux of corrosive gas was maintained at ~6 L/hr. All gases were neutralized after testing by passage through potassium hydroxide solution.

Exposures under simulated lignite (brown coal) combustion conditions were obtained by using [L] labeled exposure (see Table 2). The [L + B] condition serves to simulate the effect of simultaneous lignite and biomass combustion. Such cofiring leads to increased amounts of chlorides and alkali metals, therefore, KCl and HCl were added to the [L] conditions.

Throughout testing, samples were removed after different exposure times up to a cumulative exposure time of 7,000 hr. No ashes were added or replaced during the exposures. The samples’ cross-sections were taken from the middle of the sample (indicated in Figure 1) ground, and polished by using water-free solutions. Such a location represents the highest corrosion rates, according to previous studies at our laboratory (work to be published). Sample dimensions were recorded before and after exposure to assess their metal loss. Measurements of the cross-section were taken with light optical microscopy after exposure at different sample locations. The deepest metal loss was calculated by subtraction of the measurements before and after exposure. The microstructure and composition of the cross-sections were further characterized by scanning electron microscopy (SEM) combined with energy-dispersive X-ray.
spectroscopy (Philips XL40). Element distribution maps with a spatial resolution close to 1 μm were obtained by electron probe microanalysis (EPMA; JXA 8100, JEOL) using excitation energy of 15 kV.

3 | RESULTS

3.1 | Corrosion kinetics

According to the light microscopy analysis, corrosion progresses in three different stages. These stages are shown in Figure 2 for the case of DMV C 263 and DMV 304 HCu. These alloys are shown to represent a nickel-based alloy and an austenitic steel case. Due to the similarities of the appearance of the cross-sections, the other tested alloys are not shown. These three corrosion stages occur at different exposure times depending on the alloy composition.

Initially, corrosion forms pits in which oxides of the scale are dissolved, and the corrosion front at the interface with the substrate is easily identified by the presence of segregated sulfides (Figure 2a). More advanced corrosion shows high Ni dissolution effects in the form of Ni₃S₂–NiSO₄–NiO droplets

| TABLE 1 | Nominal composition of the tested alloys (wt. %) |
| Wt.% | Ni | Fe | Cr | Co | Mo | Cu | Ti | Al | C | Mn | Si | Nb | W | B | Microstructure<sup>10–16</sup> |
|-------|-----|-----|-----|-----|----|----|----|----|----|----|----|----|----|----|-------------------|
| DMV 304 HCu | 8.83 | 68.01 | 18.64 | 3.01 | 0.10 | 0.72 | 0.23 | 0.43 | MC, M₂₃C₆, γ, α-Cu |
| Sanicro 25 | 25.36 | 43.23 | 22.35 | 1.44 | 2.98 | 0.023 | 0.064 | 0.51 | 0.18 | 0.49 | 3.37 | MC, M₂₃C₆, γ, α-Cu |
| DMV 310 N | 21.02 | 52.14 | 24.68 | 0.06 | 1.21 | 0.44 | 0.45 | MC, M₂₃C₆, γ, α-Cu |
| DMV 617 | 55.01 | 0.8 | 21.8 | 11.9 | 8.7 | 0.01 | 0.45 | 1.15 | 0.065 | 0.03 | 0.08 | 0.005 | M₂₃C₆, γ, γ' |
| DMV C 263 | 50.72 | 0.44 | 20.00 | 19.80 | 5.90 | 2.4 | 0.45 | 0.05 | 0.15 | 0.09 | 0.003 | M₂₃C₆, γ, γ', G, η |
| Alloy 740 | 48.79 | 0.23 | 24.67 | 20.20 | 0.50 | <0.01 | 1.80 | 0.93 | 0.03 | 0.30 | 0.51 | 2.02 | 0.003 | M₂₃C₆, γ, γ', G, η |

| TABLE 2 | Experimental conditions (atmosphere in vol. %, salt deposit in wt. %) |
| Label | Atmosphere | Salt deposit composition |
|-------|-------------|--------------------------|
| [L] | N₂–20%H₂O–15%CO₂–4%O₂–0.25%SO₂ | 15% Na₂SO₄–1%K₂SO₄–60%CaSO₄–bal. Al₂O₃ |
| [L + B] | N₂–20%H₂O–15%CO₂–4%O₂–0.25%SO₂ + 500ppm HCl | 15% Na₂SO₄–1%K₂SO₄–60%CaSO₄–1%KCl–bal. Al₂O₃ |

**FIGURE 2** Light microscopy of DMV C 263 after (a) 500 hr (incubation), (b) 1,000 hr (propagation), and (c) 7,000 hr (breakaway) and (d) DMV 304 HCu after 500 hr (incubation), (e) 1,000 hr (breakaway), and (f) 7,000 hr (breakaway) of exposure under [L] conditions. Note the different magnifications [Color figure can be viewed at wileyonlinelibrary.com]
above the pits (Figure 2b). This transition stage can also be identified by the absence of sulfides in the corrosion front of the pit. The final corrosion stage (breakaway) shows the formation of a continuous corrosion layer instead of the previously observed pits and droplets (Figure 2c,e,f). This corrosion layer consists of superimposed oxides of substrate elements and NiS as scale. The three corrosion stages can also be identified in the metal loss evolution. Incubation and propagation stages show a moderate metal loss, while breakaway stages accelerate the metal loss (see Figure 3).

Metal loss of Ni-based alloys is compared in Figure 3a,b for lignite combustion with and without biomass cofiring and for the austenitic steels in Figure 3c,d, respectively. Whereas generally, the performance of the different alloys shows the same trend in the two different exposure environments, their rate is strongly influenced by the presence of chlorides in gas and solid forms. Generally, nickel-based alloys show a higher resistance, whereas austenitic steels perform worse in chlorides containing environment.

Among the nickel-based alloys, the highest corrosion resistant one is the alloy 740 and the least protective, DMV 617. Alloy 740 does not show breakaway after 7,000 hr in co-combustion, whereas breakaway was observed for the same alloy upon \([L]\) combustion. A comparison with Table 1 shows that alloys with rather low Cr and high Co and Mo contents are more prone to severe metal loss. This finding is well in line with the literature. Cr is known to have beneficial effects in hot corrosion protection, whereas Co and Mo show the opposite behavior.\cite{4,19,20} Co is known to form low melting eutectics, while Mo increases acidic fluxing.\cite{8} Alloy DMV 617 shows breakaway after 5,000 hr of exposure. DMV C 263 with similar Cr but less Co and

![Graphs showing metal loss vs. time for different alloys under different conditions.](https://example.com/image.png)

**Figure 3** Evolution of maximal metal loss at 650°C of Ni-based alloys under [L] (a), [L + B] conditions (b), austenitic steels under [L] (c) and [L + B] (d) conditions representing lignite combustion and cofiring of lignite and co-combustion, respectively. Full symbols indicate stages before breakaway and empty symbols breakaway stages [Color figure can be viewed at wileyonlinelibrary.com]
Mo than DMV 617 shows a lower corrosion rate but breakaway occurs after 5,000 hr. Alloy 740 shows the lowest corrosion rates. This alloy presents the highest Co content among the tested Ni-based alloys, but is counter-balanced by the highest Cr content (25 wt.%) and almost no Mo.

Figures 3c,d show the metal loss of the tested three austenitic steels. DMV 304 HCu is the steel showing the highest corrosion rate and the shortest time to breakaway. It contains the lowest Cr quantity (around 18 wt.%). Sanicro 25 and DMV 310 N with a higher Cr content (22 and 25 wt.%, respectively) exhibit similar metal loss rates. Contrarily to the nickel-based alloys, austenitic steels exposed to [L + B] conditions show a higher metal loss compared to [L] conditions. This can be evidenced by the higher metal loss of DMV 304 HCu but also because Sanicro 25 shows breakaway effects (after 7,000 hr of exposure) only when exposed to [L + B] conditions. Therefore, it is obvious that the addition of biomass to lignite combustion is detrimental for iron-rich materials.

3.2 Corrosion scales and products

3.2.1 Nickel-base alloys

Figure 4 shows the light optical microscopy cross-sections of DMV 617 showing the highest metal loss rates in both [L] and [L + B] conditions.

Breakaway of DMV 617 is manifested by the formation of droplets or nodules containing Ni-Ni$_3$S$_2$ at their core. The fact of forming nodules is already indicative of the formation of a molten phase. The sequence of Figure 4 reveals, as previously described, that the breakaway is retarded in the presence of biomass. The first nodules of Ni-NiS are present in [L + B] conditions after 5,000 hr of exposure (Figure 4g,h), whereas in [L] conditions, they are present even after 1,000 hr of exposure (Figure 4b).

The SEM and elemental distribution map of DMV 617 exposed in [L + B] conditions before and after breakaway are shown in Figure 5. This figure strongly helps to understand the corrosion mechanisms. Before breakaway, corrosion occurs mainly by dissolution of Ni and Co, and their reprecipitation in the outer ash deposit is observed. This corrosion stage corresponds already with the propagation stages described in the introduction. The fluxing and reprecipitation in the outer part of matrix elements is possible due to the presence of Na and K oxides and sulfates that react with the oxide scale, which is formed on this alloy when exposed to elevated temperatures. The combination of these elements forms the core of the pits (compare simultaneous location of Cr, Na, and K in Figure 5a). Chromia is not protective and reacts with K, forming low melting temperature potassium chromates. The nonprotective chromia permits the transport of sulfur species into the substrate. The highest amount of the sulfur is localized at the reaction layer close to the substrate (bottom of the pit) and not in the core of the pit. This demonstrates that the ionic molten phases’ formed in the core of the pit’s main function is the fast

![Figure 4](https://example.com/figure4.png)

Figure 4 Light microscopy cross-section images of DMV 617 Ni-based alloys exposed to 650°C under [L] conditions at (a) 500 hr, (b) 1,000 hr, (c) 5,000 hr, and (d) 7,000 hr and under [L + B] conditions after (e) 500 hr, (f) 1,000 hr, (g) 5,000 hr, and (h) 7,000 hr [Color figure can be viewed at wileyonlinelibrary.com]
transport of corrosive species and substrate materials in opposite directions. At this stage, the corrosion mechanism neither shows strong sulphidation nor main element depletion, which are typical characteristics of Type I hot corrosion.

The cross-section of Figure 5b represents the zone close to the previously mentioned Ni-rich nodules. This figure was obtained after 7,000 hr of DMV 617 under [L + B] conditions, that is to say, after its breakaway. The main difference with Figure 5a is the presence of higher Ni amounts in the salt deposit in the form of NiSO$_4$ and NiO. The decomposition of the Ni–Ni$_3$S$_2$ eutectic at a higher pO$_2$ might be responsible for the separated presence of sulfates and oxides around the
nodule. Such nodule formation has already been described in Grégoire et al.\cite{21} for pure Ni substrates and was associated with the shielding effect of Na$_2$SO$_4$ that decreases both pO$_2$ and pSO$_3$ at the sample. The other nickel-based alloys show the same type of attack and are not shown.

### 3.2.2 | Austenitic steels

The cross-section of the interface between the corrosion product and the substrate steel is shown in Figure 6 for the DMV 304 HCu steel. This steel is shown because it presents the worst attack among the tested austenitic steels. High porosity is observed in the subsurface zone after exposure to [L + B] conditions (with biomass addition). This porosity is more pronounced at lower exposure times and seems to decrease as the exposure time increases. Similar pore formation has been typically observed in other active oxidation cases\cite{22,23} in the presence of Cl. Their presence has been related to the evaporation of substrate elements due to the formation of volatile chloride species, especially iron chlorides.

The distribution of the porosity changes from being randomly located within metal grains to being concentrated at the grain boundaries as the exposure time increases. Location of such pores at grain boundaries has been observed also in the presence of chlorides before.\cite{24,25} When samples were exposed to [L] conditions (lignite firing without biomass addition), the only corrosion effect in this area is the internal sulphidation (Figure 6).

A similar effect has been observed for DMV 310 N (see Figure 7). Even if the alloy presents a much lower metal loss than DMV 304 HCu it shows the same effects at the corrosion layer/substrate interface. The porosity formed under [L + B] conditions decreases as the exposure time increases. At long exposure times, mostly sulphidation is observed at the same interface attacking the steel grain boundaries.

### 4 | DISCUSSION

#### 4.1 | Corrosion mechanism

Corrosion proceeds notably differently at initial stages in [L] and [L + B] exposures (lignite without and with biomass addition).
In [L] conditions, the sulfates-basically attack the main corrosion mechanism. As shown in Figure 8, the corrosive species quantity in the gas phase, \(\text{SO}_3\), is not affected by the presence of the salt deposit. The situation is different for the combined fuel case. Chlorides are less stable than sulfates in flue gas conditions similar to [L + B].\(^{[26]}\) At the gas-deposit boundary, chlorides will dissociate to form sulfates, and consequently, the released chlorine species will increase the Cl-activity. Therefore, in [L + B] conditions, corrosion is initially dominated by Cl and thus active oxidation.\(^{[27]}\) As the exposure time increases, active oxidation becomes less important, and the main corrosion mechanism becomes related to the presence of sulfur-containing species (hot corrosion).

To describe the initial formation of volatile chlorides and oxochlorides, the so-called quasi-stability diagrams were calculated. Such diagrams were developed in Schwalm and colleagues\(^{[28,29]}\) and combine traditional stability diagrams with the formation of volatile species above \(10^{-4}\) atm vapor pressure. As a rule of thumb, a pressure value of \(10^{-4}\) atm is used for a critical vapor
pressure or partial pressure above which significant evaporation of gaseous metal chlorides occurs, leading to significant metal consumption by corrosion, and thus, to a decrease of the bearing cross-section of the material.\[30\] The quasi-stability diagram of Figure 8 shows critical chloride formation with the substrate elements. This diagram helps to understand the initial corrosion behavior of exposures under [L + B] conditions. It is difficult to predict the Cl$_2$ activity at the substrate scale interface, only the conditions at the gas-deposit boundary could be calculated. It has to be noted that even if the main chlorine species in the corrosive gas is HCl at the metal oxide interface, the expected corrosive species is Cl$_2$, and Figure 9 has been calculated accordingly. In

**FIGURE 9** Metal–O–Cl “quasi-stability” diagram at 650°C for [L + B] based on solid and volatile phases with lines for the critical partial pressure of the volatile species of 10$^{-4}$ atm. Corrosive atmosphere conditions with salt addition and without salt addition are indicated in the gray and black box, respectively [Color figure can be viewed at wileyonlinelibrary.com]

**FIGURE 10** Superposed Ni and Cr phase stability diagram in Na$_2$SO$_4$ containing atmosphere at different O$_2$ and SO$_3$ partial pressures at 650°C. Blue circle localizes [L] gas activities. Golden colored zone determines the conditions to form Ni–Ni$_3$S$_2$ eutectic phases [Color figure can be viewed at wileyonlinelibrary.com]
line with the current results, the diagram validates the fact that alloys containing higher Ni and Mo amounts show better protection against chloride attack, and that the formation of pores at the grain boundaries is related to the presence of chromium as carbides, which is more prone to chloride attack. Similar observations have been also reported in Schimtt.\[31\]

As the exposure time increases, active oxidation becomes less important due to the volatility of reactants and products. The main corrosion mechanism changes from active oxidation to hot corrosion due to the predominance of sulfur-containing species. But at this stage, active oxidation has already depleted the presence of Cr and Fe from the substrate/scale interface (Figures 6 and 7c). This subsequent effect of active oxidation and hot corrosion provokes breakaway of austenitic steels at earlier exposure times in [L + B] conditions. When Fe and Cr are depleted in the subsurface zone, it becomes Ni and Mo richer, making the surface more susceptible to sulphidation, which provokes breakaway of austenitic steels and Ni-based alloys. On the basis of the phase stability diagram (Figure 10), under the [L] condition, both NiSO₄, which can promote hot corrosion, and Cr₂O₃, which is protective, can be formed. However, activities of corrosive gas species are subjected to high modifications during corrosion reactions and through the formed scale. The formation of Ni₃S₂–Ni eutectic becomes possible if the O₂ partial pressure decreases through the scale or under the deposits. The processes occurring at the alloy/salt interface are controlled by local conditions rather than the gas phase.

### 4.2 Critical Cr content

Table 3 shows the quantitative analysis of Cr present at different locations of the tested material substrate. Values with * indicate measurements of cases in breakaway (when continuous Ni–NiS layer is observed). Values with interface indication were obtained at the substrate area close to the corrosion layer, whereas values with matrix indication were obtained at the substrate far from the corrosion layer (where no corrosion effects are observed). Figure 11 shows as an example, the points analyzed for the alloy 740 in [L] condition.

It seems that the alloy’s initial Cr reservoir is not directly related to the breakaway. DMV 310 N and alloy 740 have similar Cr contents at the interface after 7,000 hr of exposure under [L] condition but only the latest presents breakaway. The Cr/Ni ratio seems to be a very helpful value to describe breakaway occurrence. Values at the interface below 0.5 present breakaway, whereas values above 0.8 present local breakaway and values above 1.1 show protective behavior.

The measured values reveal that contrarily to the textbook description of Type II hot corrosion, depletion of protective elements is observed. Values can be correlated with a critical Cr content but differently to the Type I hot corrosion, depletion of such protective elements is not diffusion induced. Microstructural characterization in the outermost zone of the substrates reveals significant modifications during corrosion experiments. A deeper

| Austenitic steel | Ni-based alloy |
|------------------|---------------|
|                  |               |
| **At.%**         | **Ni**        |
| **Cr/Ni**        | **Cr**        |
| **DMV 304 HCu**  | **Interface** |
| [L]              | 0.3          |
| [L + B]          | 0.4*         |
| **DMV 310 N**    | **Cr**        |
| [L]              | 1.1          |
| [L + B]          | 0.4          |
| **Sanicro 25**   | **Matrix**    |
| [L]              | 2.3          |
| [L + B]          | 1.4          |
| **DMV C 263**    | **Cr**        |
| [L]              | 20.3         |
| [L + B]          | 26.7         |
| **DMV 617**      | **Cr**        |
| [L]              | 20.9         |
| [L + B]          | 27           |
| **Alloy 740**    | **Cr**        |
| [L]              | 21.8         |
| [L + B]          | 21.9         |

**Note:** Values with * are obtained at locations presenting breakaway corrosion.
microstructural study is required, but this is beyond the scope of the actual results.

5 | CONCLUSIONS

As a rule of thumb, Cr has the strongest effect on corrosion resistance. Co is rather detrimental when sulphation prevails, but its negative effect can be compensated by sufficient Cr. All alloys show breakaway corrosion after extended exposures. The breakaway is identified by the formation of a continuous Ni–NiS layer in the scale. At the same time, microstructural changes of the substrate revealed important Cr depletion in the interface close to the scale. These two important effects are responsible for the observed accelerated corrosion, and are for the first time observed in Type II hot corrosion. Corrosive behavior for such long times has not been reported before. The time to get breakaway situations was correlated with the alloy composition as well as gas and salt environments. In lignite combustion, Ni-based alloys suffer stronger metal loss than austenitic steels. On the contrary, biomass addition to lignite provokes a higher deterioration of austenitic steels than to Ni-based alloys due to initial active oxidation by chlorine.

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