

## TESTING OF CORROSION BEHAVIOR OF INCONEL 738 AT HIGH TEMPERATURES IN CARBON DIOXIDE

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### Abstract

Corrosion and material selection are critical challenges in the practical design and operation of power plants, chemical plants, pipeline transportation systems, and wells. All of these are components of the systems used to capture and store carbon dioxide (CCS). To guarantee compatibility between the working medium and the structural material, it is necessary to determine which materials are most appropriate. These materials mostly comprise steels or specific nickel-based alloys. We selected Inconel 738 for our investigation. The corrosion coupons of Inconel 738 were exposed to impure carbon dioxide in the High-Temperature Furnace (HTF) at 750 °C and 900 °C for 250, 500 and 1000 hours. The specimen surface, corrosion layer thickness and composition were evaluated by Scanning Electron Microscope (SEM) with Energy Dispersive Spectroscopy (EDS). Glow Discharge Optical Emission Spectrometry (GD-OES) and gravimetric methods were also used to evaluate the materials. All the specimens were covered with a uniform protective oxide layer, growing with exposure time. The layer contained mainly Cr, O<sub>2</sub> and a minor amount of Ti. At a temperature of 900 °C, the specimen's weight and corrosion layer thickness increased significantly. These measurements yield an evaluation of the quality of the tested materials, as well as an assessment of their corrosion resistance.

**Keywords:** nickel alloy, corrosion, CCS technology, carbon dioxide

### 1. INTRODUCTION

CCS technology is one technique to tackle climate change and lower CO<sub>2</sub> concentrations in the atmosphere. Currently, three basic technologies can be used to separate carbon dioxide from other gases in industrial flue gas: separation of CO<sub>2</sub> from the flue gas produced during the combustion process (post-combustion), separation of CO<sub>2</sub> before the combustion process (pre-combustion), and finally separation of CO<sub>2</sub> after combustion in an oxygen atmosphere without the presence of nitrogen (oxy-fuel combustion) [1].

The high-temperature carbonate loop is a recently developed technology for removing CO<sub>2</sub> from flue gas. This process uses metal oxides (CaO and MgO), which are obtained by thermal decomposition of carbonates, to capture carbon dioxide. The carbonate loop method is commonly based on the utilization of two linked fluid reactors loaded with limestone (CaO). Fluidized bed technology enables easy circulation of the sorbent material and thus ensures contact between the gas phase and the solid phase. The solid phase is formed by limestone and the gas phase by flue gases, containing around 15% carbon dioxide. For the reaction to proceed quickly enough, temperatures of around 550-750 °C are required. The following **equation (1)** describes a process known as carbonation.



The subsequent calcination process is accompanied by the release of carbon dioxide by the decomposition of carbonate formed at temperatures of 850 - 950 °C, according to the following **equation (2)**.



The regenerated calcium oxide can be reused in a subsequent cycle, while the released CO<sub>2</sub> is utilized for downstream processing [1,2].

The purpose of this study was to validate the corrosion resistance and degradation of alloy 738 in simulated conditions of high-temperature coolant of the CCS technology, by measuring the thickness of the surface layer and determining changes in elemental composition on alloy 738 that had been exposed to the corrosive effects of high-temperature carbon dioxide, as well as noting changes in the alloy's surface layer due to exposure.

## 2. EXPERIMENTAL

### 2.1 Material

Nickel alloy 738 was tested in an environment simulating a carbonate loop. The chemical composition of alloy is listed in **Table 1**.

**Table 1** The chemical composition (in wt%) of Inconel 738

Elements	Fe	Ni	Cr	Mo	Ti	Co	Nb	Al
Inconel738	5	Bal.	16.8	1	3.6	7.6	0.9	7.5

### 2.2 Specimens

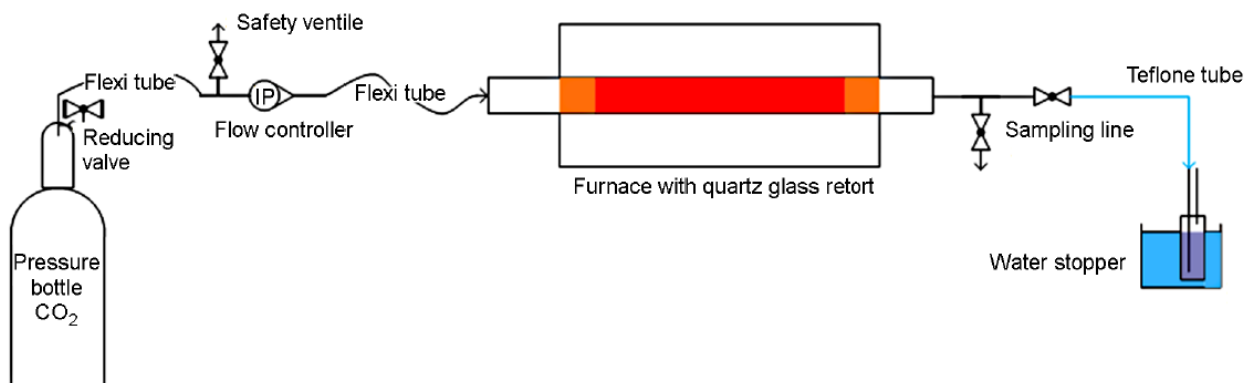
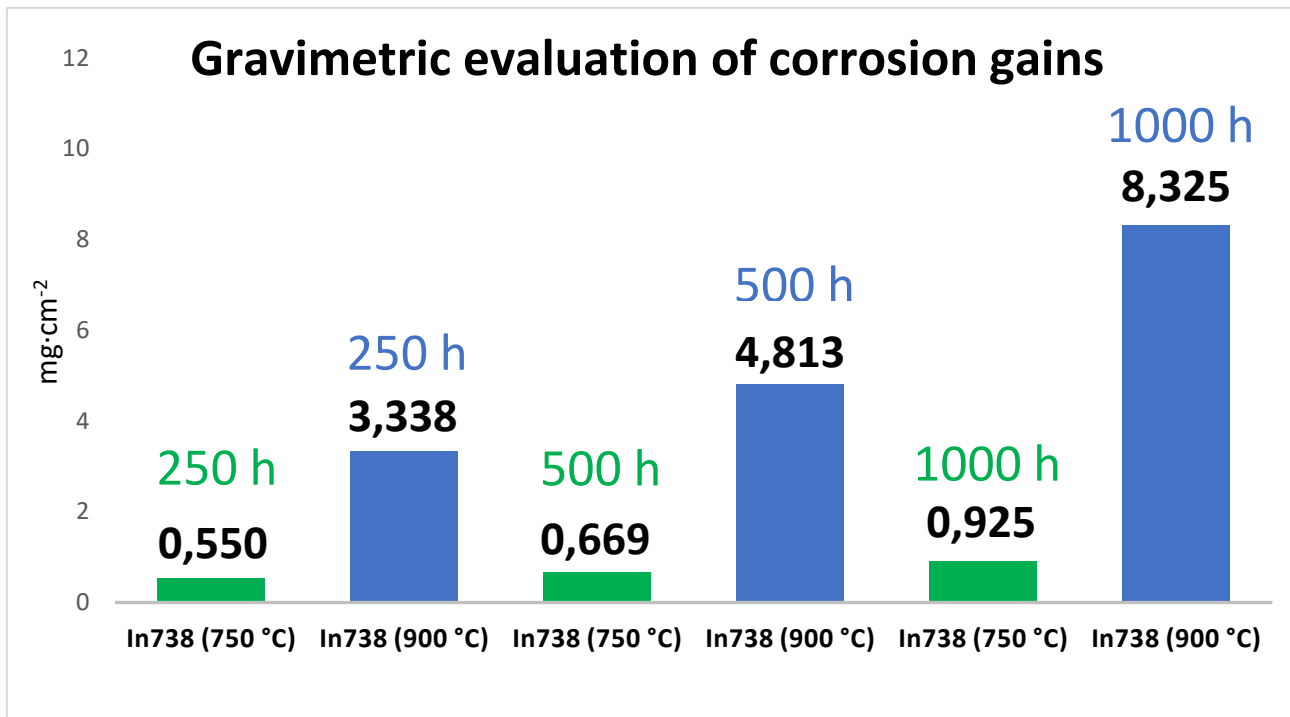
Specimens with grinded surfaces (30 µm grit sandpaper) were evaluated. The proportions of the tested specimens were 40 x10 x 2 mm. Before exposure, all specimens were ultrasonically cleaned with acetone and dried. The specimens were weighed before and after each exposure with an accuracy of 0.00001 g for post-exposure gravimetric evaluation of corrosion gains (**Figure 1**). Gravimetric measurements were performed on Radwag analytical balances XA 110.4Y PLUS series.

### 2.3 Corrosion testing in impure CO<sub>2</sub>

The specimens were subjected to the provided environment for 250, 500, and 1000 hours. The temperature during the experiment was 750 °C, then 900 °C, and the pressure was atmospheric. The desired experimental temperature was achieved gradually with a temperature ramp of 1 °C per minute, and a constant temperature was maintained inside the furnace (**Figure 2**). The cooling of the device was also gradual, with a temperature ramp of 1 °C per minute. The gas flow was approx. 50 cm<sup>3</sup>/min. Gas purity was ≥ 99.9% (Carbon dioxide 3.0, pressure bottle).

After exposure, the samples were metallographically cut in half and ground with 1200 grit sandpaper, and polished with diamond paste (3 and 1 µm). Following this, the oxide layer was analysed and measured in thickness using a Scanning Electron Microscope (SEM, LYRA3 TESCAN) equipped with detectors for imaging Backscatter Electrons (BSE) and Energy Dispersive Spectroscopy (EDS) for chemical analysis.

Glow Discharge Optical Emission Spectrometry (GD-OES), like SEM-EDX, enables an elementary analysis of material. However, this approach also offers information on the amount of carbon in each layer of the sample. This approach allows you to determine the carbonation of a sample of material. Carbonization can lead to damage and increased corrosion. The element analysis was performed using the Horiba JobinYvon GD Profiler 2 atomic emission spectrometer.



**Figure 1** Evaluation of the In738 material's mass increases following exposure to high-temperature CO<sub>2</sub>

**Figure 2** Experimental device schema [3]

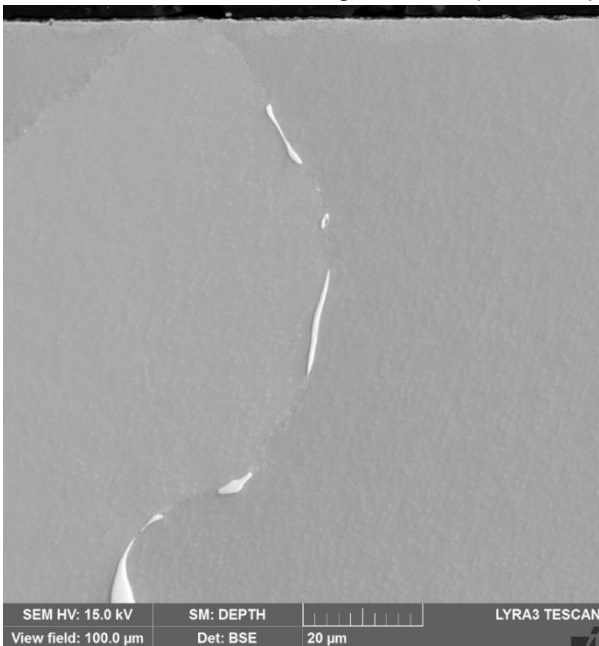
### 3. RESULTS AND DISCUSSION

SEM images of sample cross sections in the initial state and after exposure are showed in **Figures 3-5**. Corrosion attack of material In738 in a high-temperature carbon dioxide environment is shown in **Figure 4** and **Figure 5**. Exposure to 900 °C for 1000 hours resulted in the largest oxide layer, with a rise of 8.3 mg/cm<sup>2</sup>.

**Figure 4** shows a sample that was exposed to high temperature carbon dioxide for 1000 h at 750 °C. The surface of the alloy consists of chromium oxides, the layer was compact and over 3 μm thick. The movement of chromium in a high-temperature carbon dioxide environment is from the core of the sample to the surface of the alloy, where it easily oxidizes and, thanks to this, creates a layer that is resistant and increases the resistance of this alloy. The corrosion products of this alloy consisted mainly of an outer layer of chromium oxides. The highest amount of carbonization was recorded directly on the surface of the oxide layer, further the amount of penetrated carbon significantly decreases with the depth of the material layer, until finally the amount of carbonized material is very small.

**Figure 5** shows In738 after a 1000 h exposure in a high temperature furnace at 900 °C. The oxide layer was compact and is over 12 µm thick. The surface of the alloy was formed by chromium and oxygen, which together form chromium oxides in this alloy, the corrosion layer here extends to a depth of 24 µm.

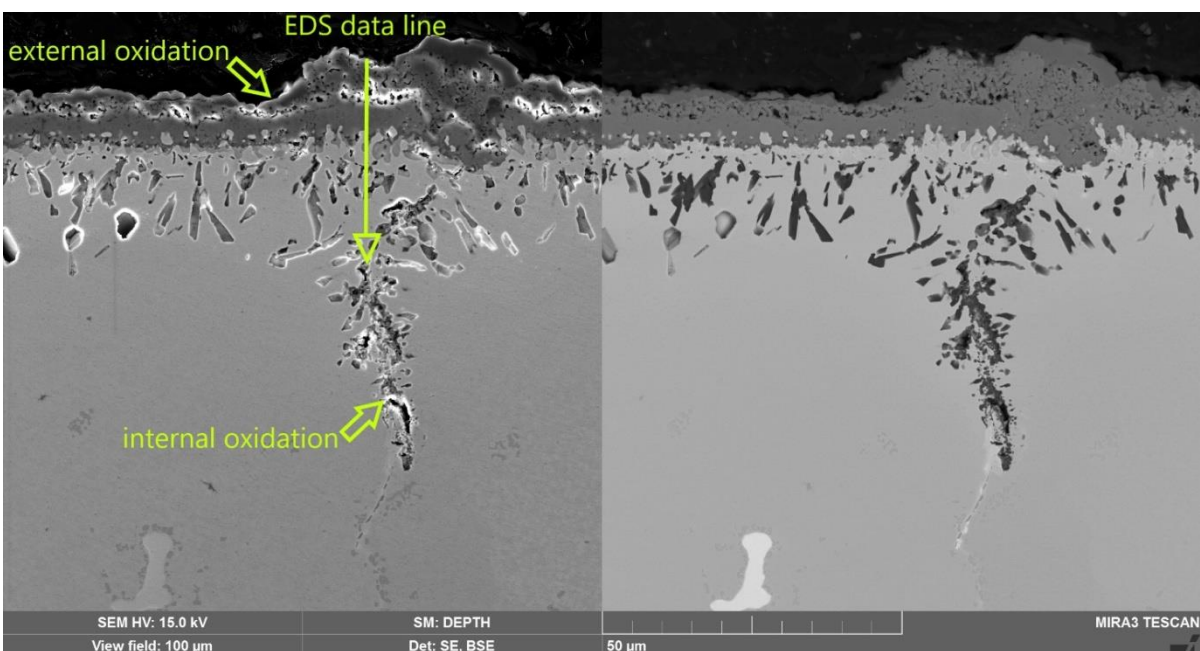
As shown in **Figure 6**, the corrosion products of In738 nickel alloy consisted of an oxidation layer that was formed by chromium oxides to a depth of approximately 10 µm. This layer was formed by the chromium contained in the alloy traveling towards the surface of the sample, where it was exposed to the given environment, which oxidized it. In the next layer, titanium oxides were formed here, reaching a depth of approximately 16 µm, followed by aluminium oxides, which reached a depth of 20 µm, and finally, titanium oxides were detected here again at a depth of 23 µm.



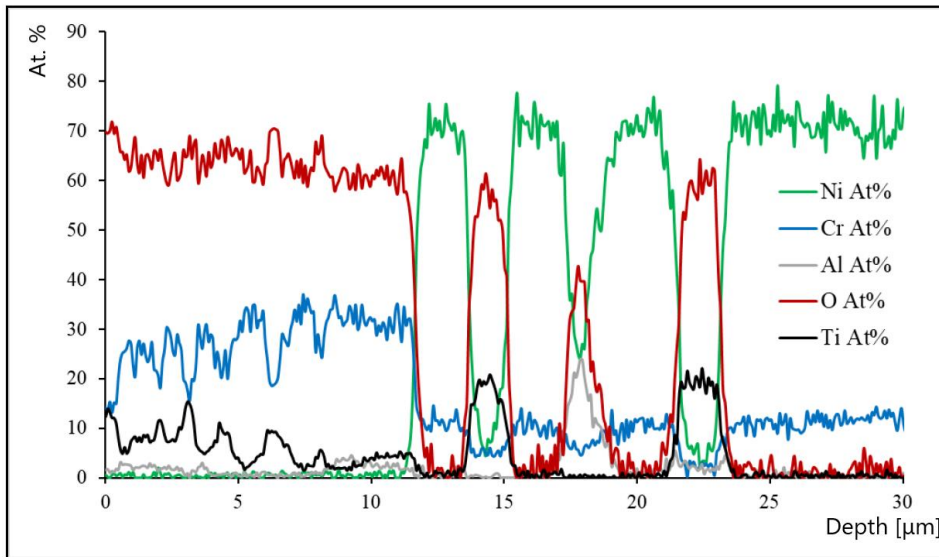
**Figure 3** The initial state of the In738 material



**Figure 4** SEM BSE image of specimen exposure at 750 °C for 1000 h

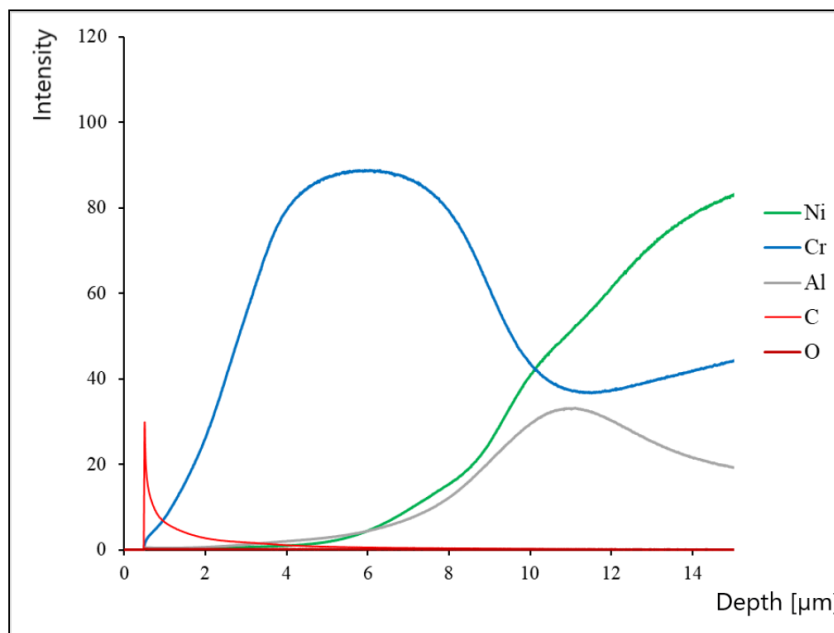


**Figure 5** SEM SE and BSE image of specimen exposure at 900 °C for 1000 h



**Figure 6** Graph of SEM-EDS analysis for In738 (representation of elements in at%) [4]

This material showed very significant formation of chromium oxides due to oxidation in high temperature CO<sub>2</sub>. However, the analysis used showed that there was a low level of carbonation (**Figure 7**). The carbon here extended to a depth of approximately 5 μm. The chromium oxide corrosion layer of Inconel 738 after 1000h exposure was estimated to be 12 μm, with the total corrosion reaching a depth of approximately 23 μm.



**Figure 7** Graph of GD-OES analysis of In738 [4]

The analyses show that the In738 alloy in a high-temperature carbon dioxide environment at 900 °C and atmospheric pressure tends to form higher oxide layers compared to other nickel alloys [4].

According to EDS analysis, the oxide layer was mostly composed of Cr, O<sub>2</sub>, and a trace of Ti. To reduce damage caused by severe corrosion processes in alloys used at high temperatures, the formation of a protective Cr<sub>2</sub>O<sub>3</sub> scale is required. It is known that a minimum Cr content of roughly 20 weight percent is necessary to create a protective, continuous scale on nickel alloys, which limits further assault [5]. Internal corrosion occurred at grain boundaries, in addition to the formation of a passive oxide scale Cr<sub>2</sub>O<sub>3</sub>. The internal

corrosion product was aluminium oxide. Deep interior oxidation can cause fatigue and creep fractures to form [6].

## CONCLUSION

The corrosion coupons of Inconel 738 were exposed to impure carbon dioxide in the High-Temperature Furnace (HTF) at 750 °C and 900 °C for 250, 500 and 1000 hours.

- All the specimens were covered with a uniform protective oxide layer, growing with exposure time. The layer contained mainly Cr, O<sub>2</sub> and a minor amount of Ti. Internal corrosion occurred at grain boundaries, in addition to the formation of a passive oxide scale Cr<sub>2</sub>O<sub>3</sub>.
- At a temperature of 900 °C, the specimen's weight and corrosion layer thickness increased significantly.
- The material has little carbonization, the carbon extended to a depth of around 5 µm.

More tests on the exposed specimens using various techniques are planned to learn more about the material's degradation after exposure.

## ACKNOWLEDGEMENTS

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## REFERENCES

- [1] VIKTORA, S. *Methods of CO<sub>2</sub> separation*. 2019. <https://theses.cz/id/53rgg2/>.
- [2] *Capture of CO<sub>2</sub> from flue gas*. E-learning VŠCHT. (n.d.). <https://e-learning.vscht.cz/course/view.php?id=1105>.
- [3] POLÁČKOVÁ, J., PETRŮ, J., JANÁK, M., BERKA, J., KRAUSOVÁ, A. Materials for use in calcium looping technology for CCS – corrosion processes in high-temperature CO<sub>2</sub>. *Corrosion and Material Protection*. 2017, vol. 61, no. 4, pp. 143–148. <https://doi.org/10.1515/kom-2017-0017>.
- [4] PŘIBYL, A. Degradation of special alloys and steels in a carbon dioxide environment at high temperatures and atmospheric and supercritical pressure. Online. Thesis. Prague: University of Chemistry and Technology in Prague, Faculty of Environmental Technology. 2023. Viewed from: <https://theses.cz/id/xzc5ae/>.
- [5] TRINDADE, V.B., KRUPP, U., WAGENHUBER, E-G., YANG, S., CHRIST, H.J. The different role of alloy grain boundaries on the oxidation mechanisms of Cr-containing steels and Ni-base alloys at high temperatures. In: *Novel Approaches to Improving High Temperature Corrosion Resistance*. Woodhead Publishing. 2008, pp. 80-92. ISBN 9781845692384.
- [6] CABET, C., TERLAIN, A., LETT, P., GUÉTAZ, L., GENTZBITTEL, J-M. High temperature corrosion of structural materials under gas-cooled reactor helium. *Materials and Corrosion*. 2006, vol. 57, pp. 147-153.