



# Corrosion and scaling studies – project results and lessons learned

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28.01.2022, Online

# Overview

Within the **MEET project** a significant part of the research work performed was the conduction and subsequent analysis of various in-situ corrosion and scaling tests performed to find a suitable material choice for heat exchangers used in the Organic Rankine Cycle power plants developed in the project as well as to evaluate brine transport solutions:

- › Coupon testing of various material candidates
  - › Stainless steels, nickel alloys, titanium, carbon steel and even plastic
  - › In different environments across Europe
- › Corrosion evaluation of heat exchanger plates used during ORC demonstration sites
- › Corrosion evaluation of polymer coated joints for transportation of hot geothermal brine
- › Characterization of scales deposited on various material candidates in 2 tested exchangers at Soultz

During this presentation we will discuss the various in-situ tests performed, some of their results as well as to summarize some of our lessons learned during the project.



# Coupon corrosion testing

To support Enogia with material selection of geothermal heat exchangers within their ORC power plant in different geothermal areas, in-situ corrosion testing of coupons (U-bend and tensile tests) were performed at various sites, using various materials.

## Materials:

- Stainless and duplex steels: 316L, 904L, 2205, 2507, 254SMO
- Nickel alloy: Inconel 625
- Titanium: Ti gr. 2
- And even plastic: PVF

## Sites:

- Reykjanes and Grásteinn (Volcanic sites in Iceland)
- Cazaux and Chaunoy (Sedimentary sites in France)
- Soultz-sous-Forêts (Granitic site in France)

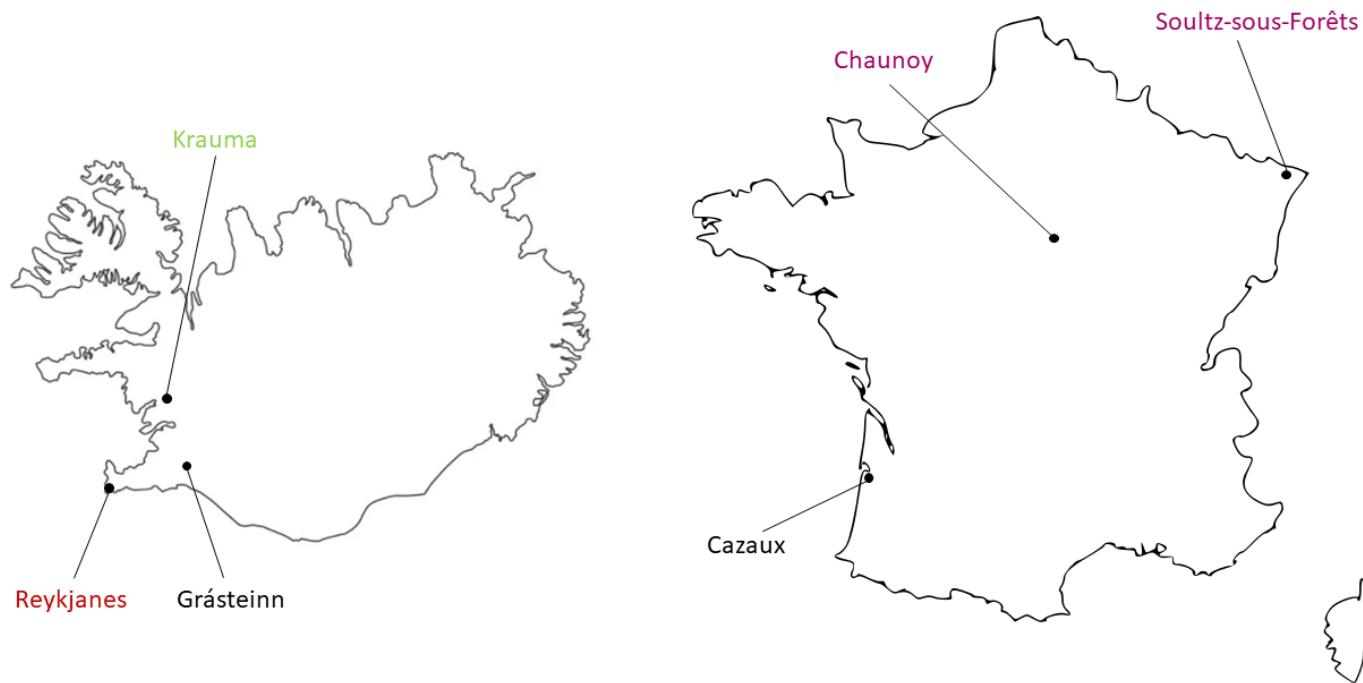


# Coupon corrosion testing

The initial corrosion tests were performed at Reykjanes, Grásteinn and Cazaux.

- All the previously mentioned materials were tested
- Using corrosion coupons, U-bend samples and tensile samples

The results from these tests were used to advise Enogia on the material choice for their heat exchangers to be used during the ORC demonstrations of the project.



Initial coupon testing

Initial coupon testing and ORC demo

ORC demo and second coupon testing

ORC demo



# Initial coupon corrosion testing - Reykjanes



The samples from the Reykjanes testing; a) the U-bend samples, b) the rest of the samples and c) all the samples after they were removed from the holders.

At Reykjanes, all metal material candidates were tested in a geothermal steam at 200 °C and 18 Bar for 1 month, in a special testing pressure vessel operated at the Reykjanes power plant by HS Orka.

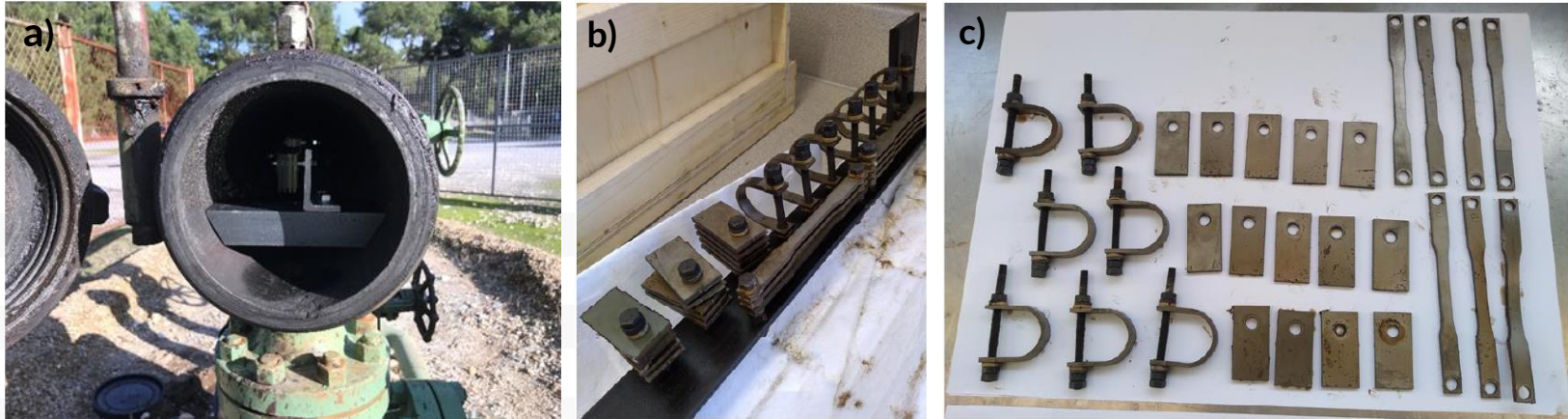
Material	Pits	Cracks	Wide slanting pits/cracks	Inclusions	Voids	Inclusions/voids in horizontal formation
316L	xxx	x	n/a	x	x	x
2205	xx	x	x	x	x	n/a
904L	x	n/a	xx	x	x	xx
2507	xx	xx	x	x	x	x
Alloy 625	(x)	xx	xx	xx	x	x
254 SMO	x	x	x	x	x	xx
Ti Gr.2	x	n/a	n/a	n/a	n/a	n/a

Corrosion effects and defects in the materials after testing at Reykjanes.

(x) – minor indication, x – minor defect, xx – considerable defect, xxx – significant defect



# Initial coupon corrosion testing - Cazaux



The samples from the Cazaux testing; a) the “pig-trap” where the samples were tested, b) the samples on the sample rack after testing, c) all the samples after they were removed from the sample rack.

At Cazaux, all material candidates apart from titanium were tested in an oil-geothermal brine mixture at 95 °C and 17 Bar for 3 months, in a “pig-trap” at the Cazaux oil field operated by Vermilion.

Material	Pits	Cracks	Wide slanting pits/cracks	Inclusions	Voids	Inclusions/voids in horizontal formation
316L	x	x	n/a	x	x	x
2205	xx	xxx	x	x	x	x
904L	x	(x)	xx	x	x	n/a
2507	x	xx	x	x	xx	x
Alloy 625	x	xx	xx	xx	x	x
254 SMO	x	n/a	xx	x	x	n/a

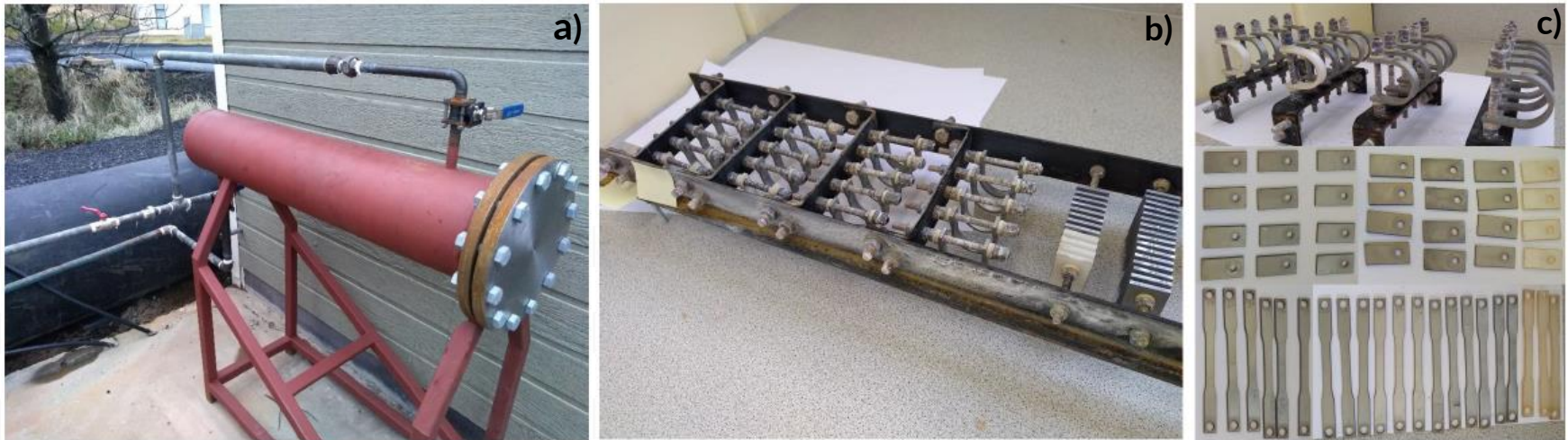
Corrosion effects and defects in the materials after testing at Cazaux.

(x) – minor indication, x – minor defect, xx – considerable defect, xxx – significant defect





# Initial coupon corrosion testing - Grásteinn



The samples from the Grásteinn testing; a) the pressure vessel where the samples were tested, b) the samples on the sample rack after testing, c) all the samples after they were removed from the sample rack.

At Grásteinn, all material candidates apart from titanium were tested in an low-temperature geothermal brine at 96 °C and 1 Bar for 3 months, in a pressure vessel at the site.

Material	Pits	Cracks	Wide slanting pits/cracks	Inclusions	Voids	Inclusions/voids in horizontal formation
316L	x	x	x	x	x	xx
2205	xx	x	x	x	x	x
904L	x	n/a	x	x	x	n/a
2507	xx	xx	xx	x	x	xx
Alloy 625	x	xx	xx	xx	x	x
254 SMO	x	x	x	x	x	n/a

Corrosion effects and defects in the materials after testing at Grásteinn.

(x) – minor indication, x – minor defect, xx – considerable defect, xxx – significant defect



# Initial coupon corrosion testing –results

Based on the overall results from the initial coupon testing, the tested metals were ranked for their corrosion performance. The three best materials were:

254SMO

904L

Ti Gr. 2

Reykjanes		Cazaux		Grásteinn	
Ranking no.	Material	Ranking no.	Material	Ranking no.	Material
1	Ti Gr.2	1-2	254 SMO	1	904L
2-3	904L	1-2	904L	2	254 SMO
2-3	254 SMO	3-4	Alloy 625	3-4	316L
4	Alloy 625	3-4	316L	3-4	Alloy 625
5	2205	5	2507	5	2205
6	316L	6	2205	6	2507
7	2507				



Warping of plastic samples after testing.

PVF was an initial material candidate as plastic heat exchangers are often used in severely corrosive conditions. However, due to warping, the plastic material was discarded as a potential candidate.



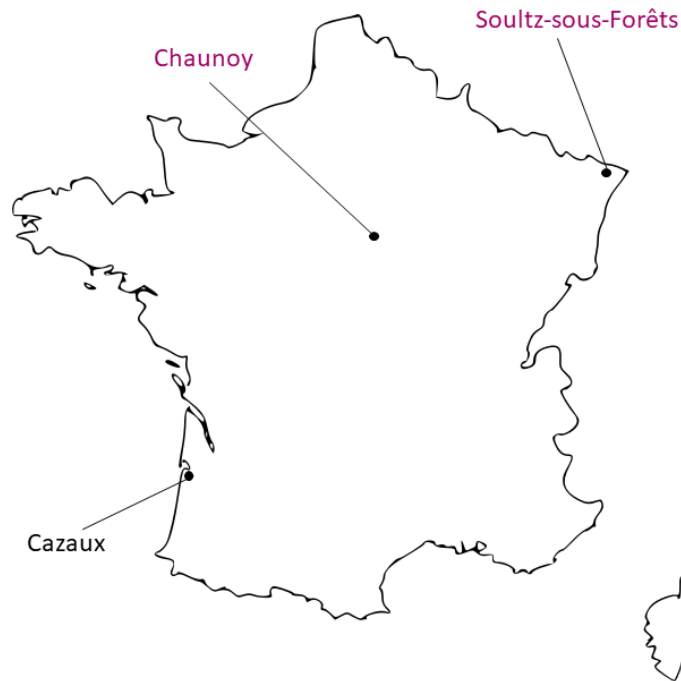
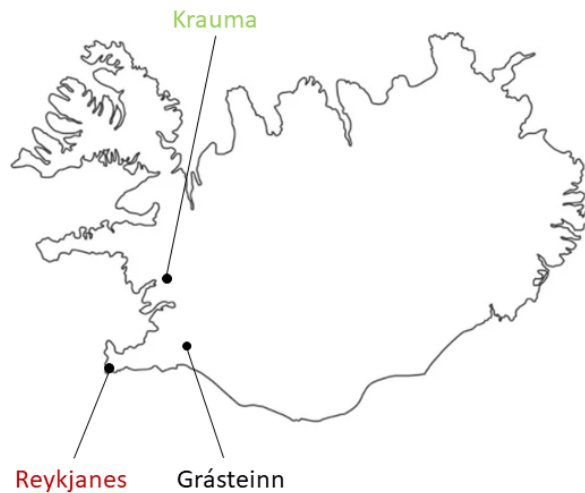
# Plate heat exchanger evaluation and secondary coupon testing

Later in the project, three heat exchanger from ORC demonstrations at three different sites were analysed:

- Chaunoy (Ti gr. 2) - Sedimentary
- Grásteinn (254SMO) - Volcanic
- Soultz-sous-Forêts (254SMO) - Granitic

Additionally, secondary coupon testing was performed at:

- Chaunoy
- Soultz-sous-Forêts



Initial coupon testing

Initial coupon testing and ORC demo

ORC demo and second coupon testing

ORC demo

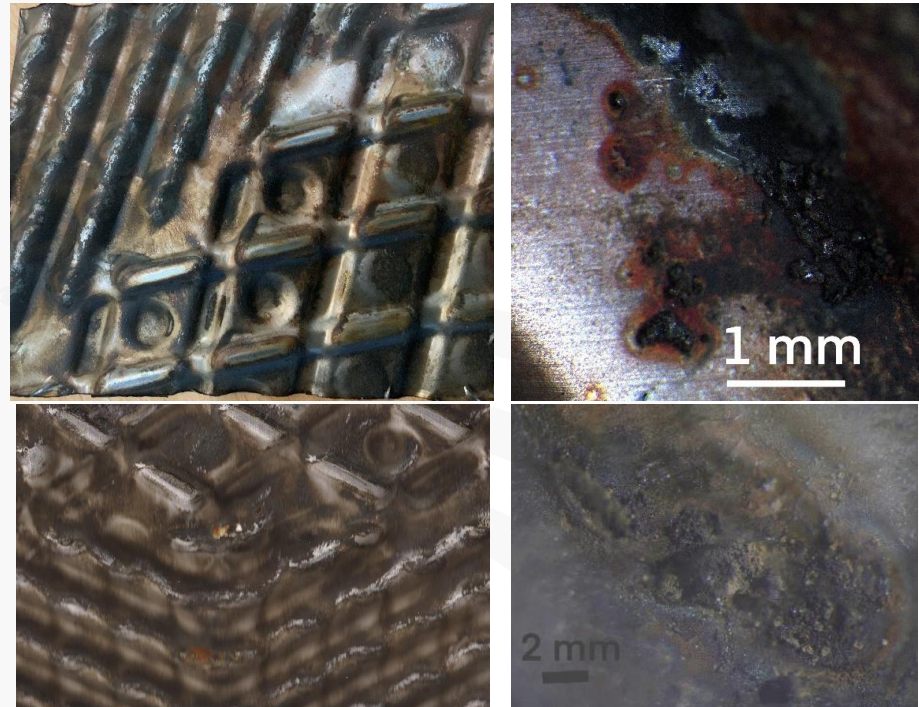


# Plate heat exchanger analysis - Grásteinn

Three plates were removed from the ORC heat exchanger at Grásteinn and analysed macro- and microscopically.



Dismantling of heat exchanger.



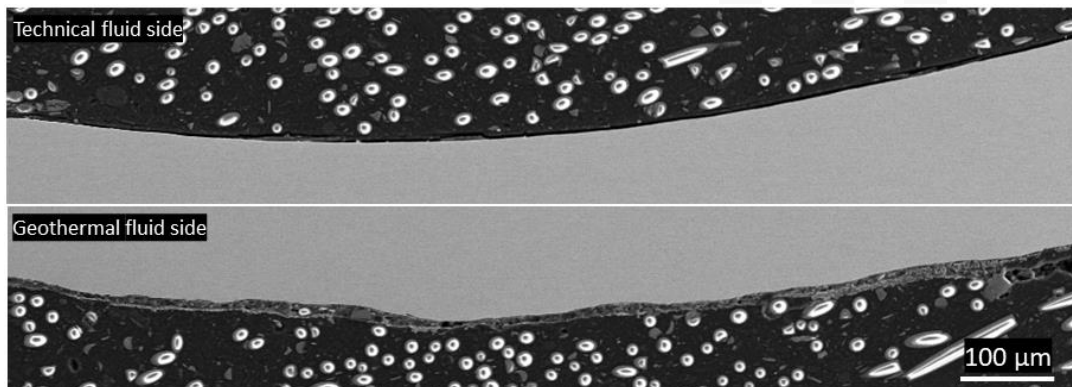
Corrosion indicators observed on the surface of the HX plates.

# Plate heat exchanger analysis - Grásteinn

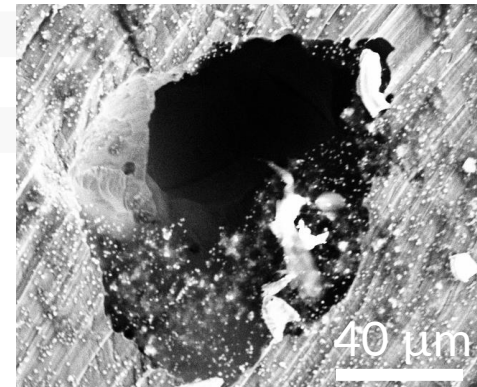
Corrosion was observed on the heat exchanger plates, both in the form of pits, increased surface roughness and sub-surface cracking. Although the material did not perform as well as expected, a few things should be kept in mind:

- The areas analysed were the ones observed to be most affected, in most areas, the heat exchanger was unaffected by the exposure.
- Frequent starts and stops of the ORC led to varying exposure conditions which might have resulted in increased levels of local corrosion
- The defects observed were of a small magnitude (few to tens of  $\mu\text{m}$ )

However, this raises the question; does the increased cost of 254SMO justify itself in low-temperature volcanic environment when compared to cheaper materials such as 316L?



The geothermal fluid side had a notable increase in surface roughness.

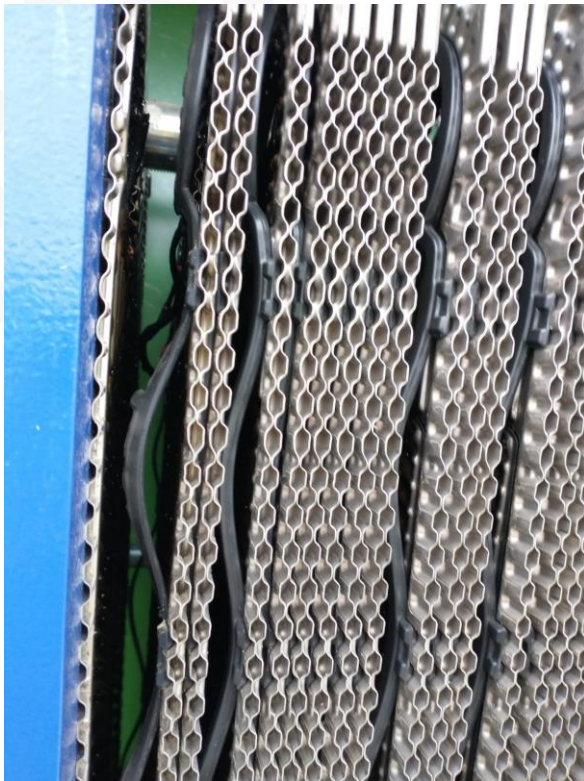


A pit observed on one of the plates.

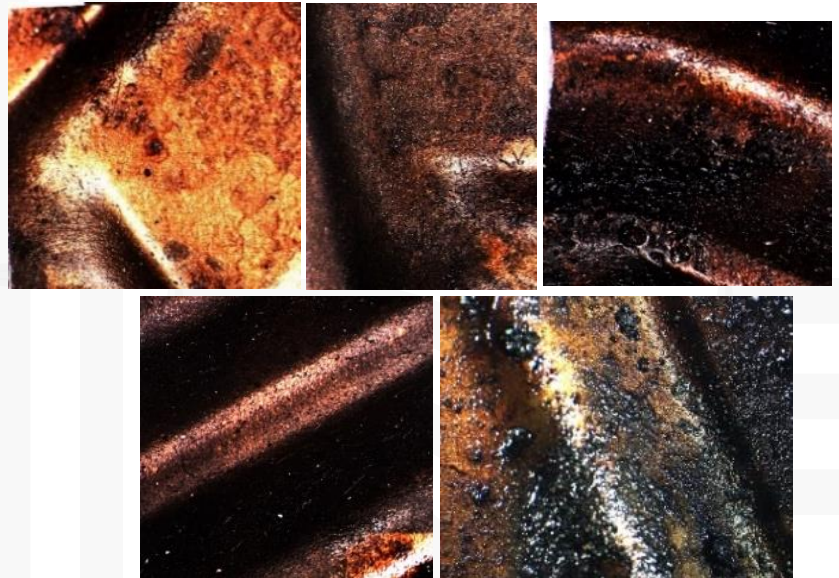


# Plate heat exchanger analysis - Chaunoy

Four plates were removed from the ORC heat exchanger at Chaunoy and analysed macro- and microscopically.



Dismantling of heat exchanger.



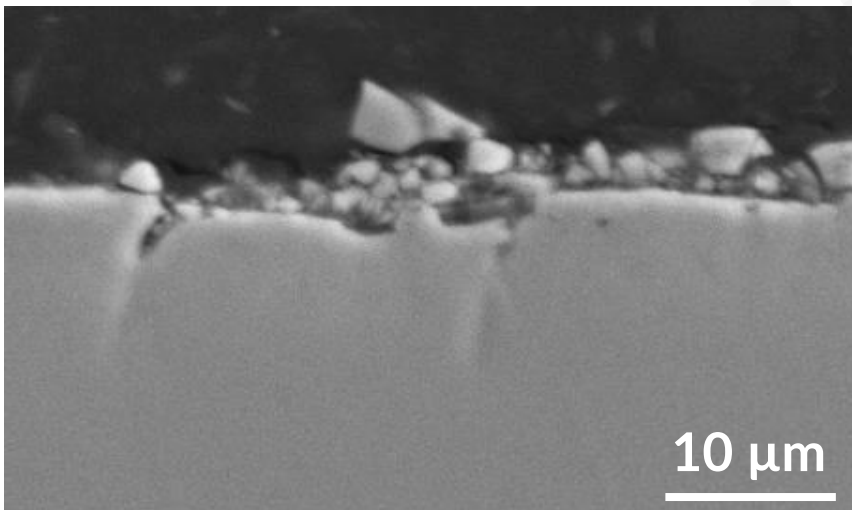
5 mm

Optical microscopy observed very few corrosion indicators, mostly oil deposits.

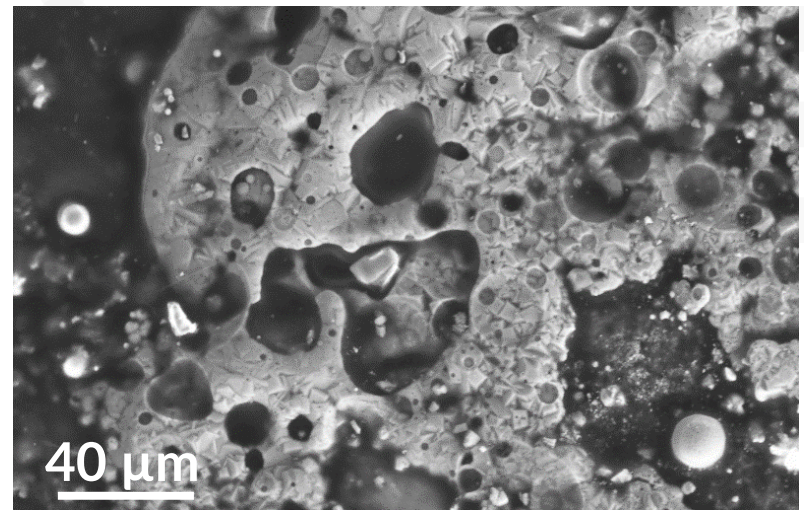
# Plate heat exchanger analysis - Chaunoy

Considerably less corrosion was observed for the heat exchanger plates in the Ti. gr. 2 heat exchanger after the Chaunoy demonstration in comparison to the 254SMO heat exchanger at Grásteinn. The main results acquired were:

- Deposits from the oil-geothermal water mixture covered most of the exposed plates
  - Mostly Fe-S compounds
- Underneath the Fe-S deposits, some indicators of crevice corrosion were seen, although quite minor
- In general the heat exchanger plates performed very well.



Crevice corrosion indicators underneath the Fe-S deposits.



Fe-S deposits on the surface of the heat exchanger plates.



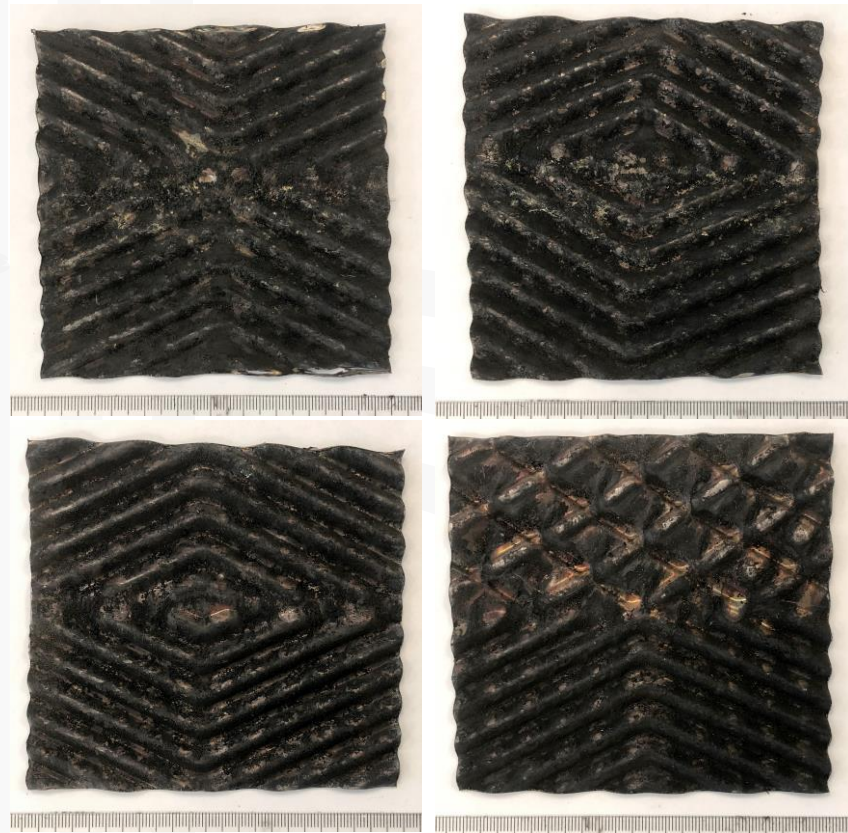
# Heat exchanger analysis - Soultz-sous-Forêts

Three plates were removed from the ORC heat exchanger at Soultz and analysed.

Due to the naturally occurring radioactivity of the scales from Soultz, the samples were initially analysed at Orano SAS, in France, before being chemically cleaned and shipped to IceTec.



Dismantling of heat exchanger.



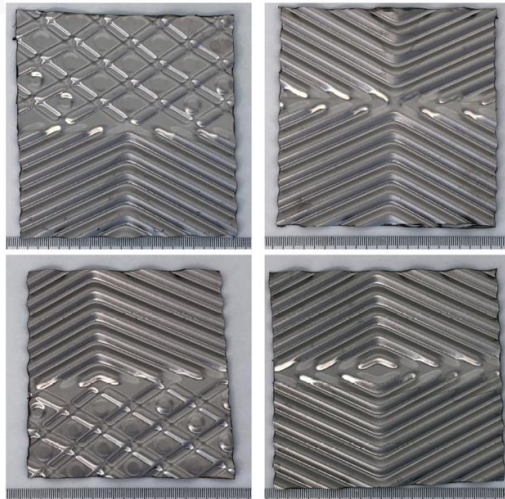
Some of the gathered samples.



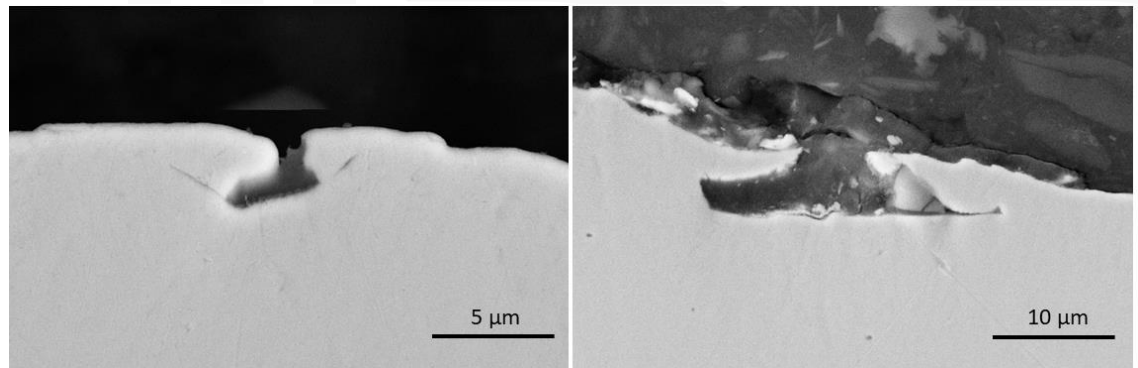
# Heat exchanger analysis - Soultz-sous-Forêts

For both the surface analysis of “uncleaned” and “cleaned” plates as well as the cross-sectional analysis of the “cleaned” plates, insignificant corrosion was observed.

- The 254SMO materials seems to resist the corrosive geothermal fluid at Soultz very well
- Defects such as cracks and pits were found in some samples, however they were very small ( $< 5 \mu\text{m}$  in depth) as well as few in number.
- However, the corrosion resistance of the material does not dictate the usability of the heat exchanger, as the scaling formed on the plates was hard to clean off and caused the heat exchanger to be unusable for a later demonstration.

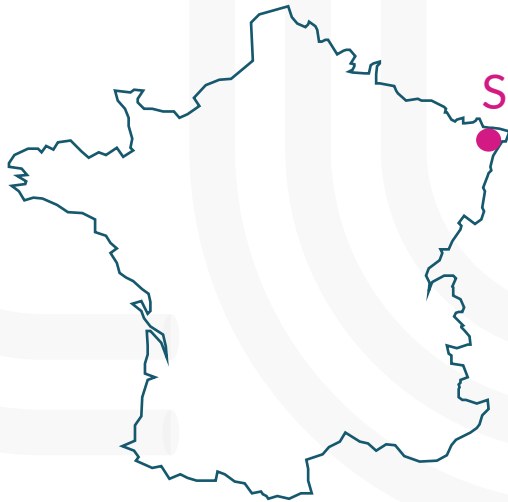


The exposed heat exchanger samples after chemical cleaning.



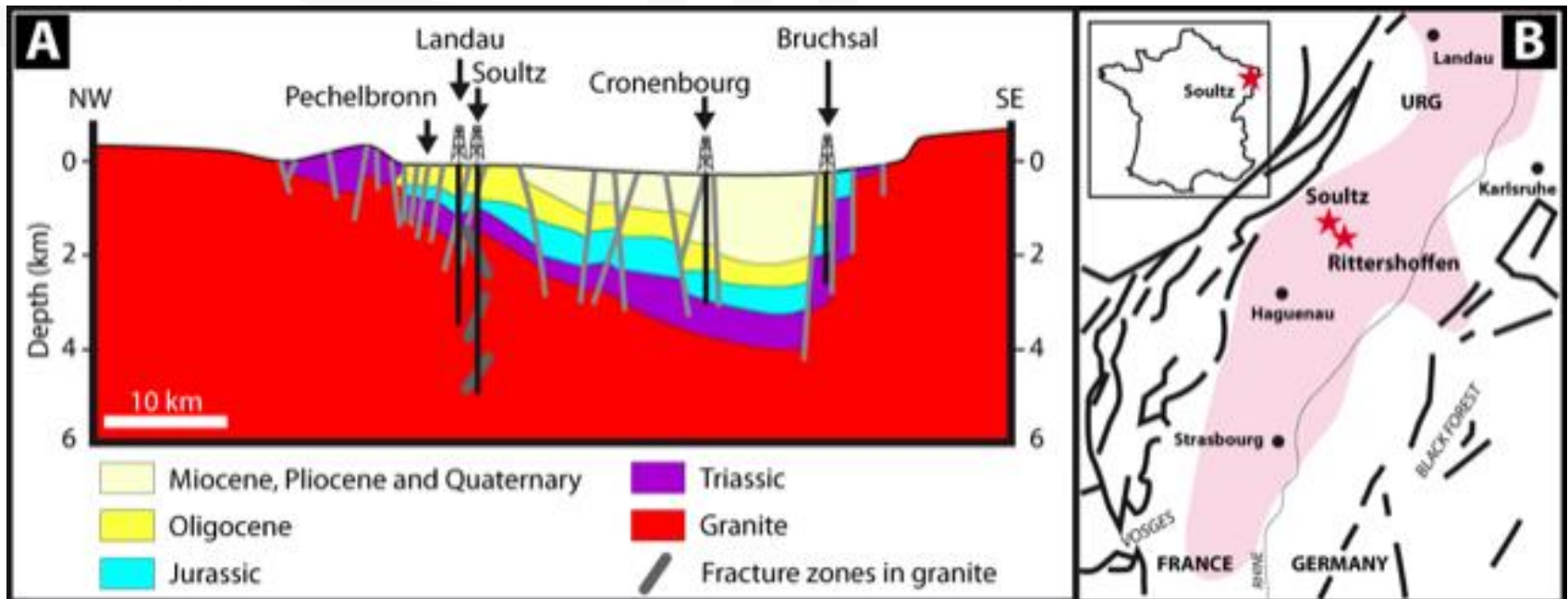
Some of the largest defects found in the cross-sectional samples.

# Scaling in heat exchangers at Soultz-sous-Forêts



Soultz-sous-Forêts

Upper Rhine Graben: large thermal anomaly  
Fractured granite basement  
3 wells down to 5.000m  
Brine with salinity ca 100g/L (mostly Na and Cl)  
Temperature around 160°C



# Scaling in heat exchangers at Soultz-sous-Forêts

2 tested heat exchangers : **A**- 3 months in 2019, **B**- 6 months in 2021

Materials tested:

- \*Metals: 304L, **904L**, **254SMO**, **2205**, **2507**, Alloy 825, **Ti Gr.2**
- \*Coated metals
- \*Glass fiber

Aims:

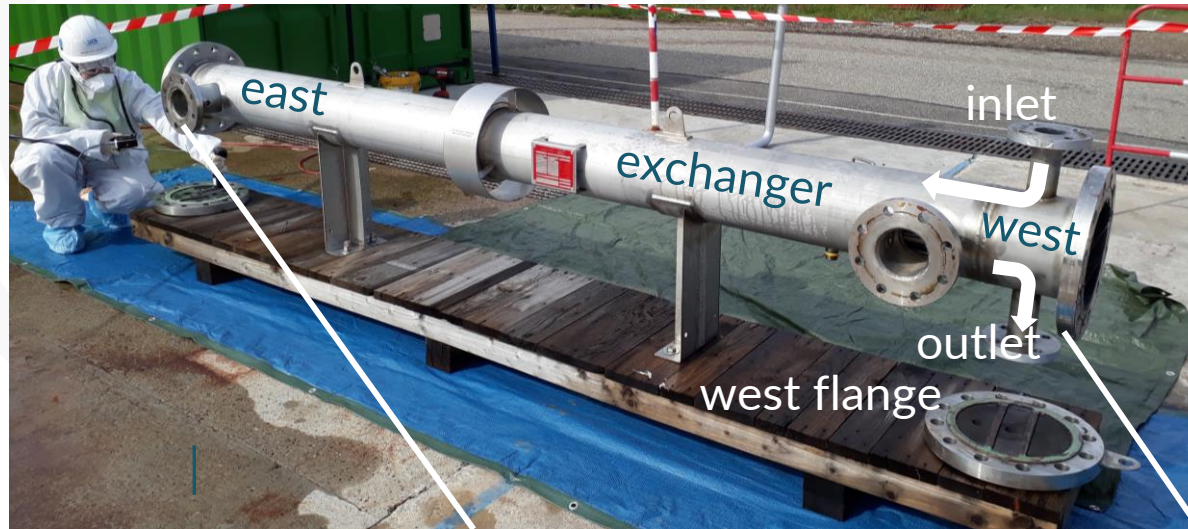
- \*test various materials
  - 1- in current industrial conditions (**test B**) with reinjection at 70°C
  - 2- at lower injection temperature (40°C) to increase energy production (**test A**)
- \*Characterize the scales and check if differences with the materials
- \*See if influence of temperature on scaling



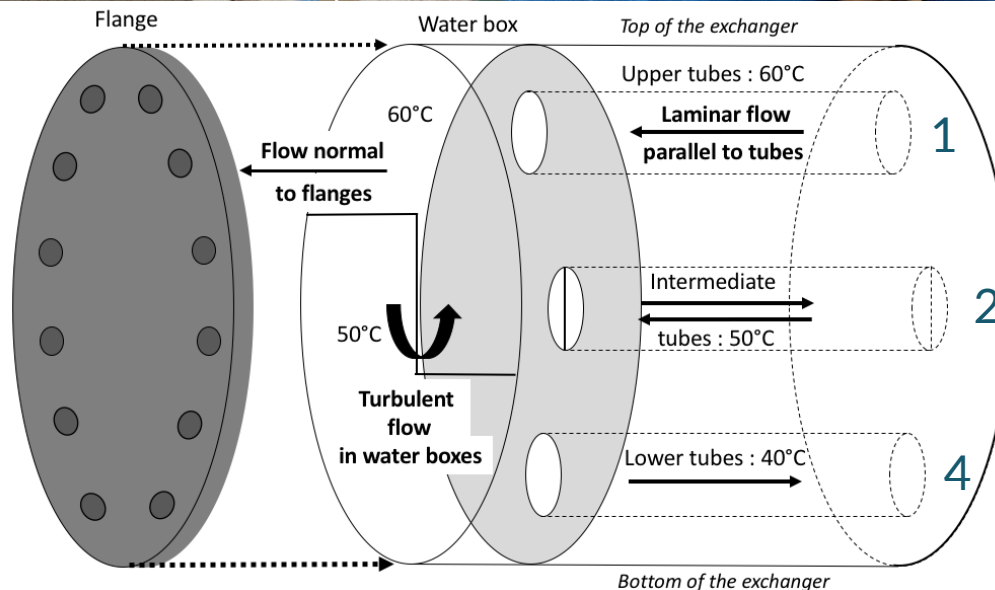
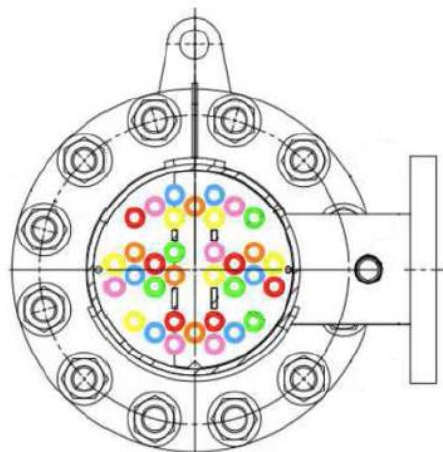


# Scaling in heat exchangers at Soultz-sous-Forêts

test A (3 months, 2019): design of the prototype heat exchanger



- 904L
- 254SMO
- 2205
- 2507
- Alloy 825
- Ti Gr.2



Cooling with  
4 passes

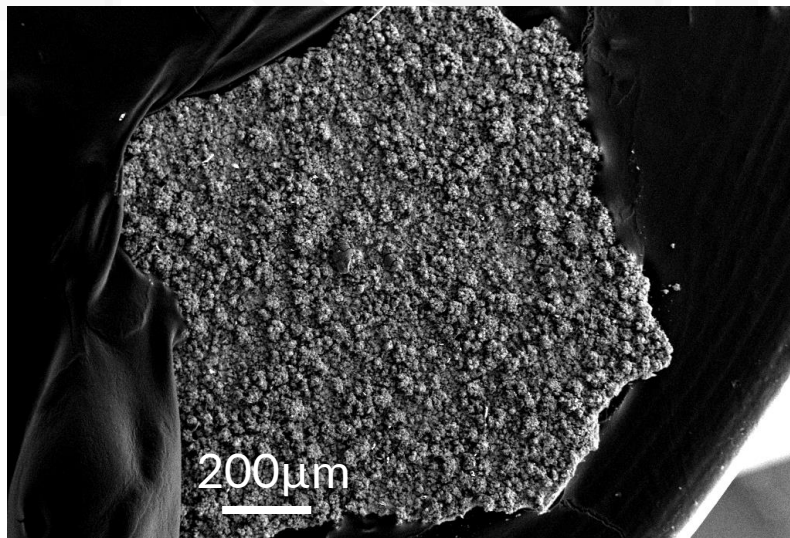




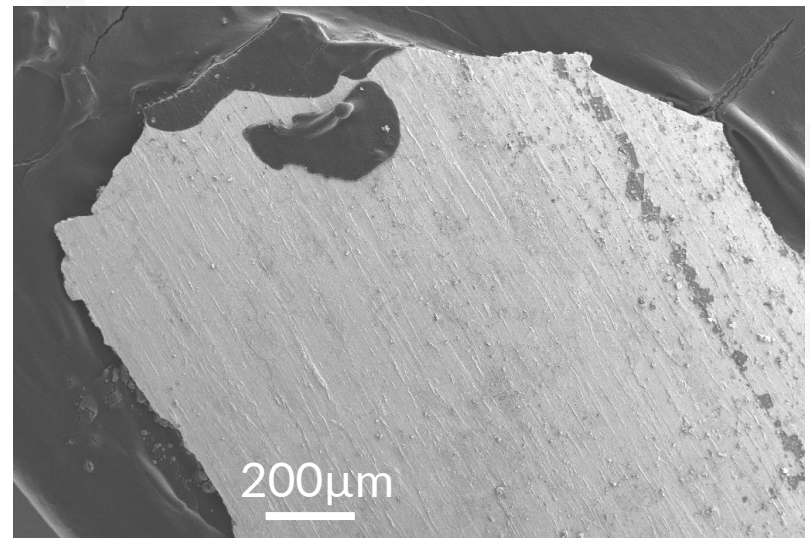
# Scaling in heat exchangers at Soultz-sous-Forêts



## Scanning electron microscopy (SEM)



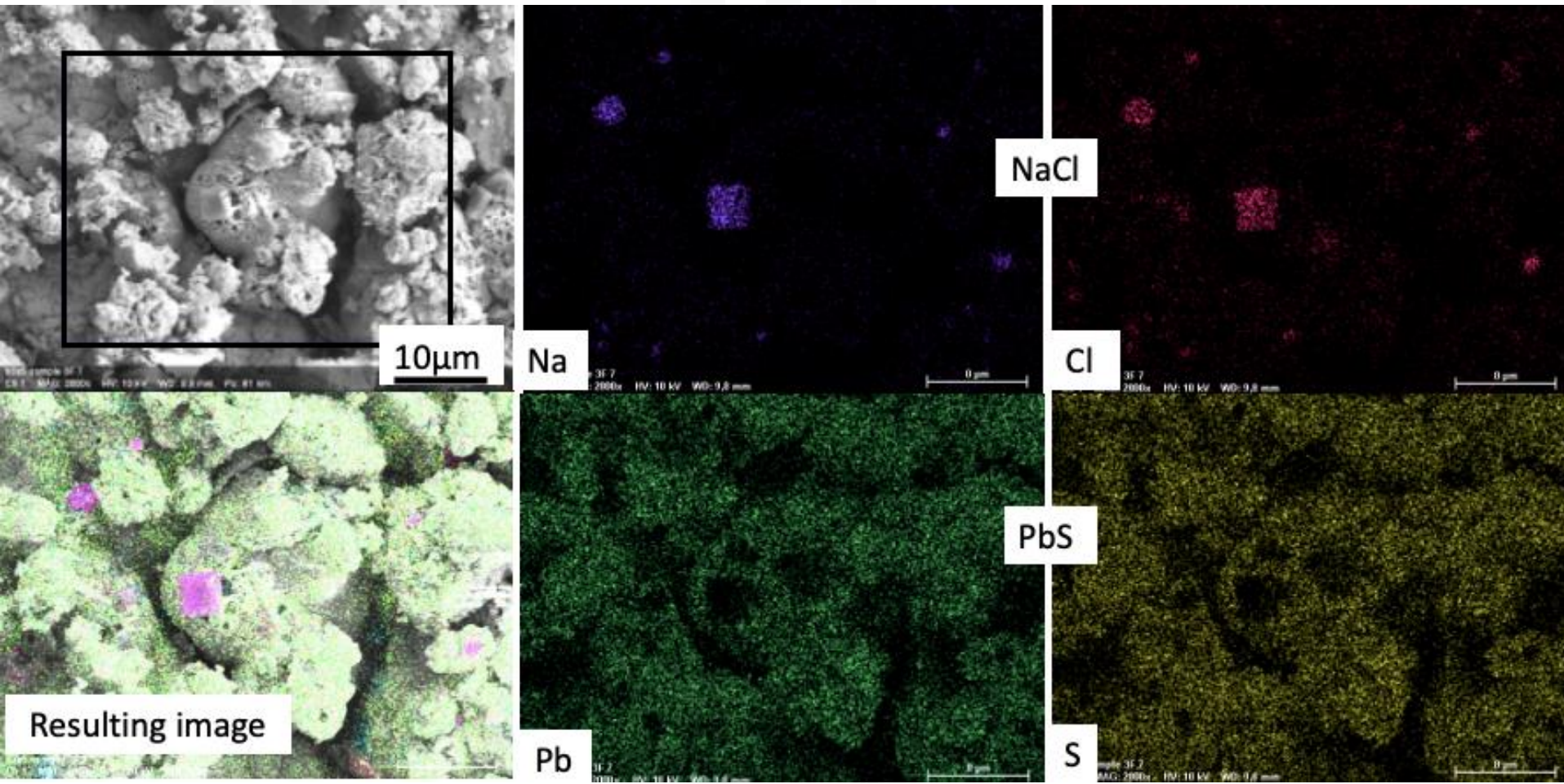
Rough fluid side



Smooth metal side

# Scaling in heat exchangers at Soultz-sous-Forêts

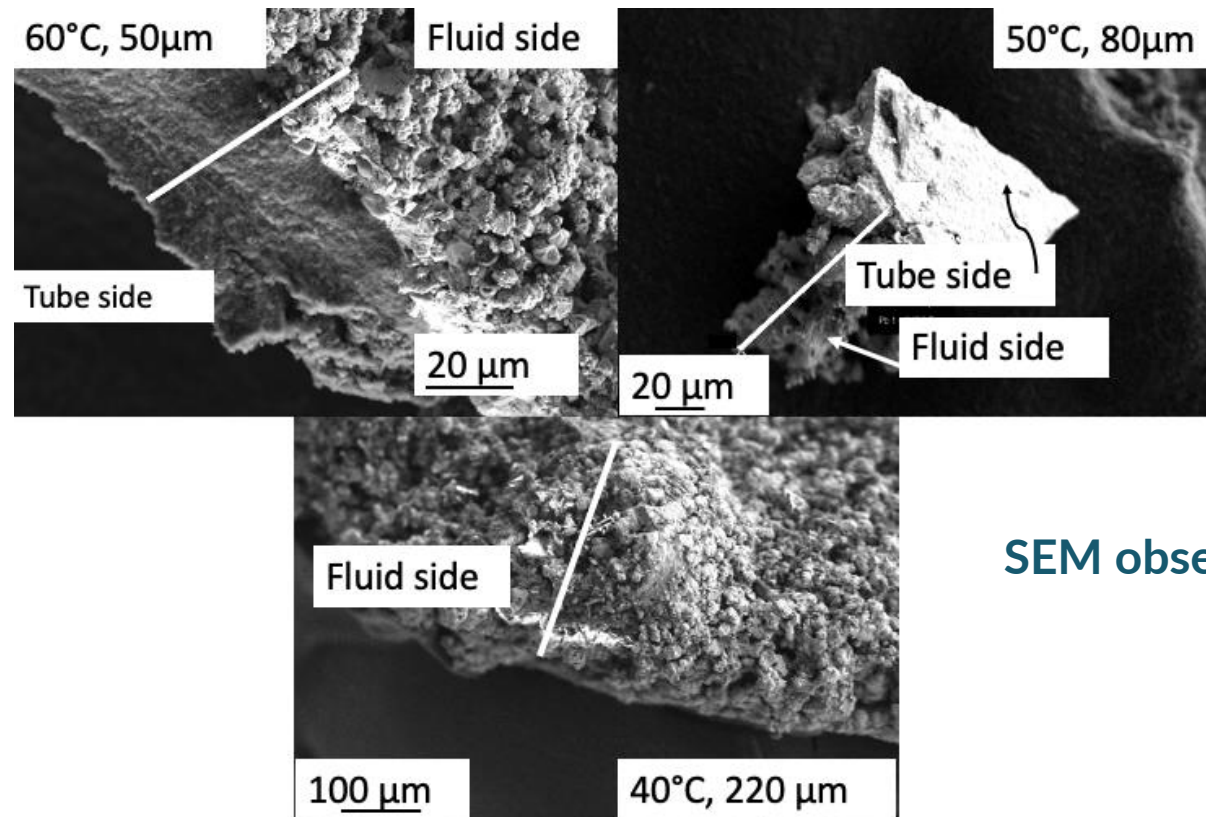
SEM coupled with Energy Dispersive Spectrometry (SEM-EDS): galena and halite





# Scaling in heat exchangers at Soultz-sous-Forêts

- \* Same mineralogical composition (galena and halite) whatever the metal
- \* Same mineralogy as in normal operation conditions (70°C reinjection)
- \* Likely increased thickness of scales when the temperature decreases (60°C → 40°C)

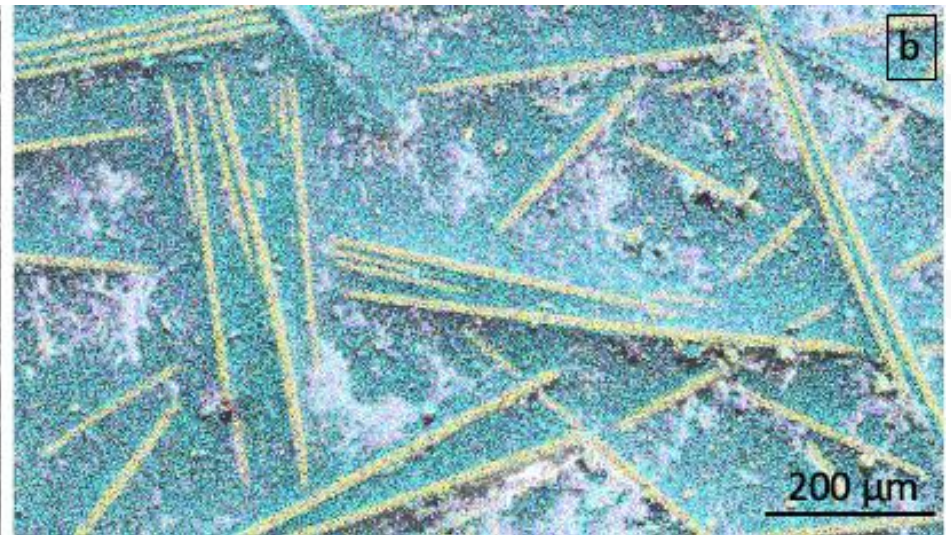
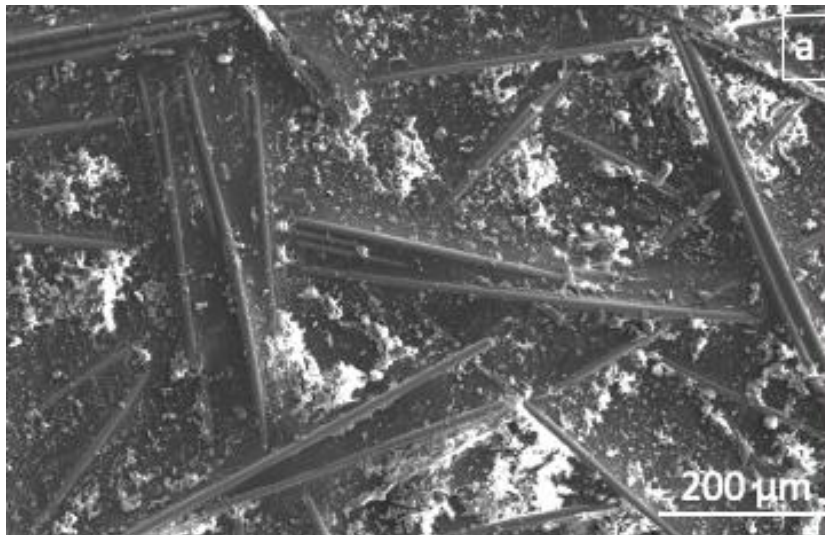


SEM observation

# Scaling in heat exchangers at Soultz-sous-Forêts

test B (6 months, 2021)

- \* Same mineralogical composition (galena and halite) as test A whatever the metal
- \* Same mineralogical composition on metals, coated metals and fiberglass
- \* Fiberglass: inside surface of the pipe after cleaning (SEM)



➔ Difficult to clean because of fibres not fully embedded in epoxy



# Take home message about scaling

## \*test various materials

- 1- in current industrial conditions (test B) with reinjection at 70°C
  - 2- at lower injection temperature (40°C) to increase energy production (test A)
- Similar alloys and different materials (coated alloys and fiberglass): same scaling
  - Fiberglass not favourable to cleaning when fibres are not well embedded in epoxy

## \*Characterize the scales and check if differences with the materials:

no difference in mineralogy

## \*influence of temperature on scaling :

- -no influence on mineralogy
- - likely influence on deposit thickness: ==> when temperatures decreases, thickness increases





# Thank you for your attention.

For more information see Geosciences MEET Special Issue:  
[https://www.mdpi.com/journal/geosciences/special\\_issues/meet\\_project](https://www.mdpi.com/journal/geosciences/special_issues/meet_project)





*Article*

## **Study of Corrosion Resistance Properties of Heat Exchanger Metals in Two Different Geothermal Environments**

Svava Davíðsdóttir <sup>1</sup>, Baldur Geir Gunnarsson <sup>1</sup>, Kjartan Björgvin Kristjánsson <sup>1</sup>, Béatrice A. Ledésert <sup>2</sup> and Dagur Ingi Ólafsson <sup>1,\*</sup>

*Article*

## **Scaling in a Geothermal Heat Exchanger at Soultz-Sous-Forêts (Upper Rhine Graben, France): A XRD and SEM-EDS Characterization of Sulfide Precipitates**

Béatrice A. Ledésert <sup>1,\*</sup> , Ronan L. Hébert <sup>1</sup> , Justine Mouchot <sup>2</sup>, Clio Bosia <sup>2</sup>, Guillaume Ravier <sup>2</sup>, Olivier Seibel <sup>2</sup>, Éléonore Dalmais <sup>2</sup>, Mariannick Ledésert <sup>3,†</sup>, Ghislain Trullenque <sup>4</sup>, Xavier Sengelen <sup>1</sup> and Albert Genter <sup>2</sup>

