

Scale Analysis in the Framework of Optimization of Energy Production at the Soultz-sous-Forêts EGS Site

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ABSTRACT

In order to increase the production of energy at the Soultz-sous-Forêts power plant (Rhine Graben, France), a prototype heat exchanger in which 10% of the whole water flow was diverted has been tested for decreasing the temperature from 70°C to 40°C. After 3 months of operation, it was opened for sampling of scales (deposited in the tubes and water boxes) and study of corrosion. The prototype heat exchanger has been designed with 6 different metallurgies (1.4539, 1.4547, 1.4462, 1.4410, 2.4858, TiGr.2) as described by Ravier et al. (2019). Due to high service conditions (150°C and 25 bar, high salinity brine) and the risk of scale formation, all the geothermal plants in operation in the Rhine Graben are designed with shell and tube heat exchangers. The prototype heat exchanger was designed according to this technology. The alloy 1.4410 is the steel alloy currently used in the heat exchangers of the Soultz industrial power plant. The other metallurgies were already tested in extreme conditions in Iceland (Karlsdottir et al., 2014; 2015) and gave good results. The aim of this work is to present chemistry data about scales related to the different metallurgies, acquired with Inductively Coupled Plasma-Mass Spectrometry (ICP-MS; reference sample), X-ray Fluorescence (XRF; 6 metallurgies tested during 3 months) and Scanning Electron Microscopy coupled with an Energy Dispersive Spectrometer (SEM-EDS; all samples). The reference sample (obtained in normal industrial conditions in contact with 1.4410 alloy) contains mainly Pb (70%), Sb, As, S, Na, Fe and Ca. In Ti metallurgy, small spheres bearing Ti (probably TiO₂) are found in the scales at both the contact with tube and with the fluid whatever the temperature tested; when the temperature is lowered, the amount of Ti in the scales, obtained by XRF, decreases. As and Sb tend to increase in scales when the temperature decreases, whatever the metallurgy, while Pb and Cu tend to decrease, or decrease and then increase, depending on the metallurgy. According to XRF data, the chemistry of scales reflects only partly that of the metallurgy with which they are in contact. Constant features are encountered by SEM-EDS in all the scale samples at 60°C whatever the metallurgy: 1) granular matrix in contact with the fluid, containing Pb, S, As, Sb, 2) NaCl cubes either as big square patches embedded in the granular matrix or as small cubes protruding out of the surface (either in contact with the tube or with the brine). The scales for which a section could be obtained show a thickness of approximately 30-40µm over the 3-month test, that is to say about 10-13µm per month.

1. INTRODUCTION

The European H2020 MEET project “Multidisciplinary and multi-context demonstration of EGS Exploration and Exploitation Techniques” is applied to different geological conditions and aims to demonstrate the geothermal potential of Europe from real projects in relevant industrial environment for attracting investors. Thus, to enable the development and the market penetration of the EGS geothermal energy, demonstrating the feasibility and the upscaling of EGS in different geological conditions in Europe is to be performed by enhancing the heat use and/or by producing electricity in various geological contexts (Dalmais et al., 2019). In that framework, the Soultz-sous-Forêts power plant, located in the Upper Rhine Graben (France), is tested for enhancing energy production. It produces electricity thanks to an Organic Rankine Cycle (ORC). In order to increase the production of heat up to 20-35%, the temperature of the reinjected fluid is to be lowered. As a consequence, a test has been performed from January to April 2019 with a prototype heat exchanger into which 10% of the total flow was diverted. In that framework, the main goal at Soultz power plant is to improve its thermal power production and optimise the fractured granitic reservoir exploitation while minimising environmental issues. Three deep wells (Figure 1) reach a depth of about 5000m among which one is used as a production well (GPK-2) while reinjection is performed through the two others (GPK-3 and GPK-4). The deep wells and the fracture network in which the geothermal brine circulates can be seen in Figure 1. The temperature of the geothermal fluid produced at GPK-2 deep well (about 5000m deep) is around 150°C. At present, the fluid is reinjected at a temperature close to 70°C in GPK-3 and GPK-4. The temperature of reinjection was tested at a 40°C temperature for 3 months (January to April 2019) in the prototype heat exchanger presented in Figure 2. It was designed to cool down the brine to 40°C with 4 passes. Ravier et al. (2019) and Seibel et al. (2020) indicate the conditions of temperature decrease and the kind of operations that were performed at Soultz from January to April 2019 to test the small-scale heat exchanger.

Before temperature lowering, small amounts of scales are produced during the operation of the power plant despite the use of corrosion and scaling inhibitors. They constitute the reference sample obtained from the last water box just before the reinjection line in 2017 (SLZ_SOL_2017_15). As a consequence of temperature lowering, more scales might be produced. The aim of this study is to compare the mineralogy and chemistry of the scales, from the reference state (before lowering of reinjection temperature) to those obtained with various metallurgies after three months of continuous geothermal fluid circulation in similar temperature conditions and at lower temperatures.

2. MATERIAL AND METHODS

The Soultz geothermal brine is highly saline, with major parameters given in Table 1. Its chemical composition is given in Table 2.

Well	Depth (m)	T bottom °C	T Sampling °C	pH	Conductivity at 25°C μS/cm	Alk. meq/l
GPK2	5000	200	37	4.98	127	2.75

Table 1 – Major parameters of the Soultz brine sampled in GPK2 geothermal well (Data from Sanjuan et al., 2016).

Na mg/l	K mg/l	Ca mg/l	Mg mg/l	Cl mg/l	SO ₄ mg/l	NO ₃ mg/l	TIC mg/l	TOC mg/l	SiO ₂ mg/l	Cl/Br wt	TDS g/l
28,140	3195	7225	131	58,559	157	<0.5	35.3	1.0	201	271	99

Table 2 – Chemical composition of the Soultz brine sampled in GPK2 geothermal well (Data from Sanjuan et al., 2016).

To ensure the best resistance to corrosion, a small exchanger with six different metallurgies (1.4539, 1.4547, 1.4462, 1.4410, 2.4858, TiGr.2) were tested as exposed by Ravier et al. (2019) and Seibel et al. (2020). Scales were recovered from each of them and were analysed with ICP-MS (only the reference, 1.4410 Super Duplex SAF 2507 alloy), XRF (the 6 metallurgies tested at the 3 new temperature conditions) and SEM-EDS for all of them. SEM-EDS analyses were performed for the 60°C temperature in order to compare the effect of metallurgy on the chemical composition of scales and at decreasing temperatures (60, 50 and 40°C) for Ti Gr.2 metallurgy.

2.1. Material

The reference metallurgy used at Soultz in the current heat exchangers is Super Duplex 1.4410, as in Rittershoffen, also located in the Rhine Graben. It was selected for tubes and end plates, because of its corrosion resistance, availability on tubes and plates and its feasible welding procedure. This choice was also done for its good operating performances in other plants using a similar stainless-steel grade in the same brine conditions (Ravier et al., 2016). The reference scale sample was extracted from the coldest water box, just before the reinjection line.

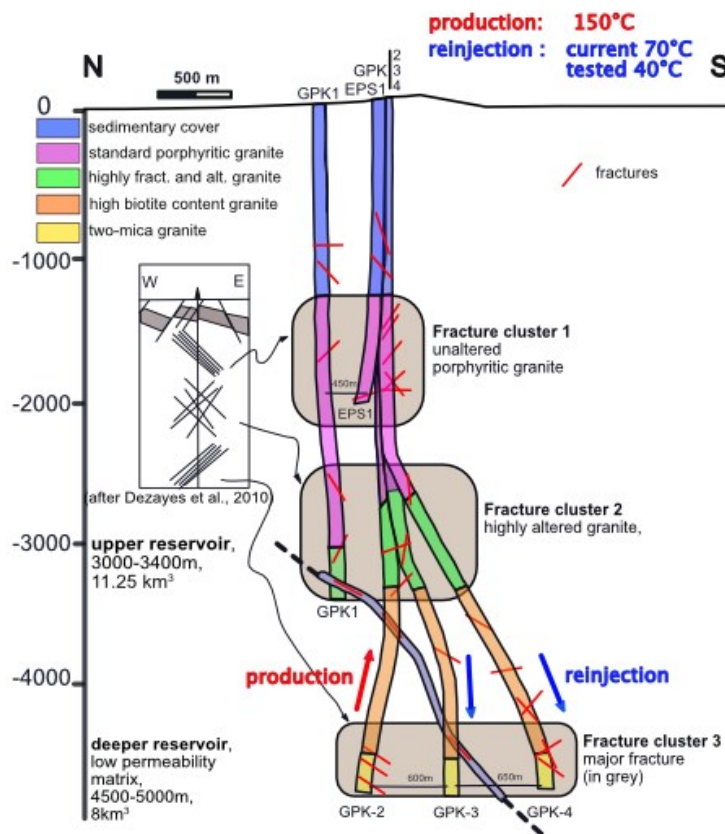


Figure 1- The Soultz-sous-Forêts fractured granitic reservoir and the 3 exploited drillholes (GPK-2, GPK-3 and GPK-4), modified after Meller and Ledésert (2017) for the N-S cross section.

The chemical composition of each metallurgy is given in Table 3.

Alloy	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Fe	Ti	Co	N	O	H
1.4539	≤0.02	≤0.7	≤2.0	≤0.03	≤0.01	19.0-21.0	4.0-5.0	24.0-26.0	1.2-2.0	Rest	-	-	-	-	-
1.4547	≤0.02	≤0.7	≤1.0	≤0.03	≤0.01	19.5-20.5	6.0-7.0	17.5-18.5	0.5-1.0	Rest	-	-	0.18-0.25	-	-
1.4462	≤0.03	≤1.0	≤2.0	≤0.035	≤0.015	21.0-23.0	2.5-3.5	4.5-6.5	-	Rest	-	-	0.10-0.22	-	-
1.4410	≤0.03	≤1.0	≤2.0	≤0.035	≤0.015	24.0-26.0	3.0-4.5	6.0-8.0	-	Rest	-	-	0.24-0.35	-	-
2.4858	≤0.025	≤0.5	≤1.0	≤0.025	≤0.015	19.5-23.5	2.5-3.5	38,0-46.0	1.5-3.0	Rest	0.6-0.12	≤1.0	0.24-0.35	-	-
3.7035	≤0.08	-	-	-	-	-	-	-	-	≤0.25	Rest	-	≤0.06	≤0.013	≤0.2

Table 3 – Elementary concentrations of the tested metallurgies.

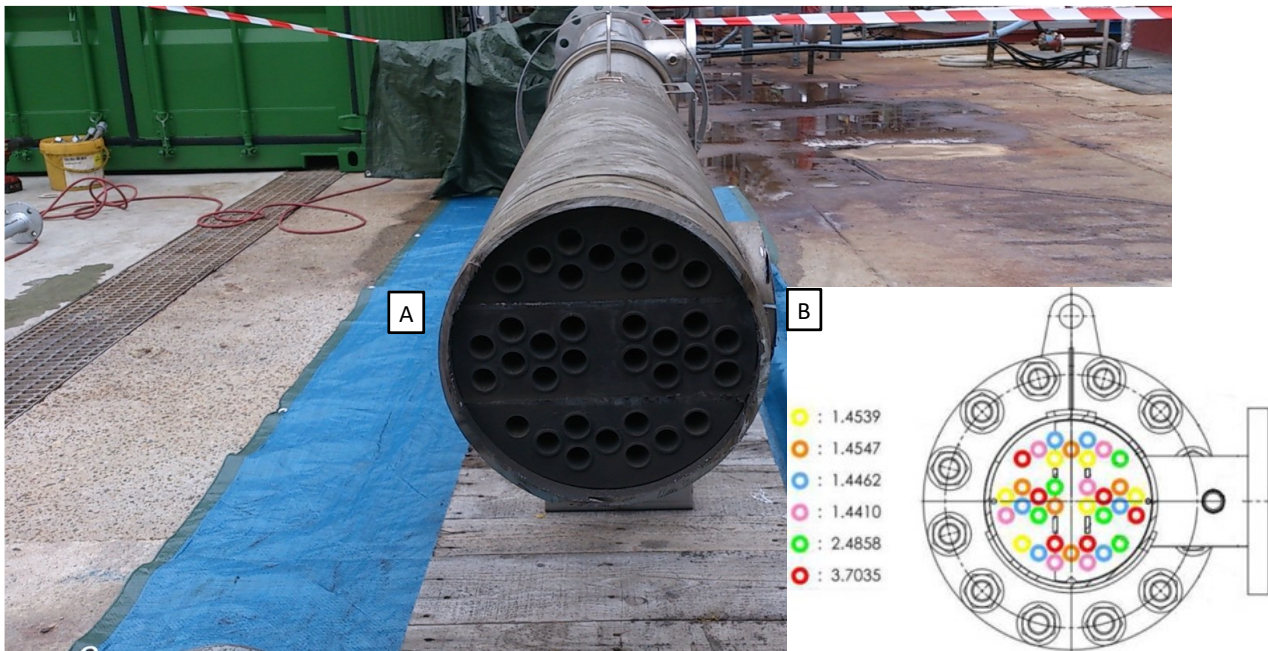


Figure 2- The prototype heat exchanger (A), opened and dismantled in april 2019 (after a 3-month test) for sampling of scales in the small tubes and study of metal corrosion. The design with the 6 metallurgies (B) is from Ravier et al. (2019).

Pros and cons of those tested metallurgies are given in Table 4.

Sample reference	Alloy classification	Metallurgy	Pros	Cons
A	1.4539	904 L	Mo addition. Good general corrosion resistance.	Susceptibility to pitting
B	1.4547	254 SMO	High resistance to general corrosion. Even in supercritical geothermal steam (Karlisdottir et al., 2014; 2015).	Expensive
C	1.4462	DX 2205	High resistance against general corrosion. Positively tested in a corrosion skid at Soultz.	-
D	1.4410	SDX 2507	Designed for service in highly corrosive conditions. More resistant against chloride than other duplex steels, so currently used at Soultz and Rittershoffen (heat exchangers). Good results in very corrosive and acid environments (Iceland; Karlisdottir et al., 2014; 2015)	-
F	2.4858	Alloy 825	High Ni alloy. Commonly used when risk of severe corrosion issues. Operates in temperatures up to 1200°C. Excellent resistance to localised corrosion and against stress corrosion cracking in chloride solution.	-
K	3.7035	Ti Gr.2	Excellent cold formability and weldability. Excellent corrosion resistance. Excellent resistance to high oxidization. Previously tested at Soultz in a corrosion skid and in laboratory.	-

Table 4 – Pros and cons of each tested metallurgy (after data in Ravier et al., 2019 and Seibel et al., 2020).

Scales occur as black deposits on the metal surfaces exposed to the geothermal fluid flow (Figure 3). Scales were extracted after opening and dismantling of the prototype heat exchanger (cutting shell and tubes along their length): they were removed as chips or

as powder (after scratching the surface with a Teflon spatula) depending on their adherence to the metal. Several grams of reference scales were sampled in the current industrial conditions and ICP-MS could be performed. However, for the 3-month test, only very small amounts (sometimes only around 100mg) were recovered. In those conditions, only XRF was performed on the samples for bulk analysis and SEM-EDS analyses were made to obtain chemistry data at local scale on millimeter-long fragments showing the face in contact with the tube and that in contact with the geothermal brine, and when possible the section.



Figure 3- Example of a tube showing the black scales deposited on the internal metal surface.

2.2. Methods

ICP-MS, XRF and SEM-EDS were used to characterise the samples.

The reference sample, SLZ_SOL_2017_15 was extracted from the water box of the last heat exchanger, just before reinjection line. Coming from the Soutz current industrial heat exchanger, the amount of material was large enough for ICP-MS analysis. It was performed using DIN EN ISO 17204-2 procedure, after microwave digestion according to E704 procedure.

Because of the very small amount of scales deposited during the 3-months circulation test in the small-scale test heat exchanger, XRF was used on 0.1g samples to have a first rough estimation of the chemical composition in order to be able to compare them according to the metallurgy on which they grew. A test was first performed in order to make sure of the validity of the measurements on such very small amounts of material and results have been compared to same analysis done with 1 g of scales sample.

The XRF apparatus is a Spectro xSORT. It was calibrated to fit the special matrix of the scales with a high content of Pb. Based on the calibration, a profile was made which was used for all the measurements. Compared to the ICP-MS, it provides a rougher estimation of the elementary composition, in which S cannot be detected because of an overlay of signals with that related to Pb. Moreover, light elements like Na cannot be detected, the first detected element being Mg. The analysis was performed three times on each sample in order to test the homogeneity. Hence, the results are provided as composition ranges together with uncertainty on the measurement. A test was first performed in order to make sure of the validity of the measurements on such very small amounts of material. When the XRF results are compared to the composition obtained with the ICP-MS, it appears that S is missing while it represents about 10 wt% of the sample, because of superimposition of its main XRF peak with that of Pb. The Scanning Electron Microscope (SEM) is a Zeiss GeminiSEM 300. It is coupled with a Bruker Energy Dispersive Spectrometer (EDS).

The data obtained on the reference sample and small samples obtained for each metallurgy at a mean temperature of 60°C are given in the Results section. A comparison is also provided for Ti Gr.2 for the 3 tested temperatures (approximately 60°C, 50°C and 40°C).

3. RESULTS AND DISCUSSION

Results are provided for the three analysis methods used for the characterization of scales.

3.1. Reference sample: ICP-MS analysis

The reference sample (SLZ_SOL_2017_15) analyzed by ICP-MS gave the results presented in Table 5, mainly showing Pb, Sb, As, S, Na, Fe and Ca together with elements present as traces (Cu, Si, K and Ba).

Elements	Concentration in mg/kg	Mass fraction (wt%)
Pb	400 000	70.56
Sb	51 500	9.08
As	39 900	7.04
S	37 200	6.56
Na	14 200	2.50
Fe	7 010	1.24
Ca	6 040	1.07
Cu	5 350	0.94
Si	2 350	0.41
K	2 000	0.35
Ba	1 330	0.23

Table 5 – Elementary concentrations in the reference sample SLZ_SOL_2017_15, obtained after circulation at a temperature around 65-75°C, in an equipment made of Super Duplex 1.4410. Results are given in concentration and in mass fraction for comparison with XRF data. The main elements found in the reference scale sample are, in decreasing order: Pb, Sb, As, S and Na.

3.2. Various metallurgies: XRF analyses of scales

Results of XRF analyses are given in percentages in Figures 4, 5 and 6 for the six tested metallurgies, at a temperature respectively close to 60°C, 50°C and 40°C.

The scales are mainly composed of Pb (75 to 87 wt%), Sb (7 to 17 wt%) and As (3.5 to 6 wt%). Others metals are present as traces (< 1 wt%), except for Ti in the Titane grade tube, which is the major element. Cu is found in all scales, whatever the metallurgies, whether they contain Cu (A, B, F) or not (C, D, K). Cr is found only in scales formed in C, D and F while it enters also in the composition of A and B (around 20%). Fe is found only in scales formed in C while it is obviously present in all steels (A to F). Mo which is found in great abundance in steel A occurs only in scales formed in contact with F. Ti is found only in scales formed in contact with Ti Gr.2.

The scale composition observed in the exchanger prototype is similar to that of the reference sample, in particular for major elements Pb, As and Sb. In contrast, trace elements composition of the scale issued from the prototype exchanger are different from the reference sample. It important to keep in mind that XRF analysis does not allow detecting S, Na, Ca, Ba and K. Thus, a detailed comparison of the scales issued from the MEET experience and the reference deposit cannot be performed using data of XRF analysis but should be done with the results acquired by ICP-MS analysis or EDS.

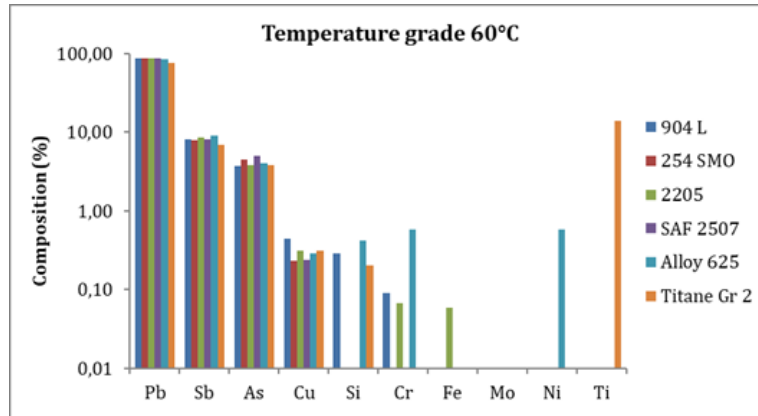


Figure 4- Pb, Sb and As concentration of the scales obtained after a 3 month circulation test in the prototype exchanger, for a temperature around 60°C.

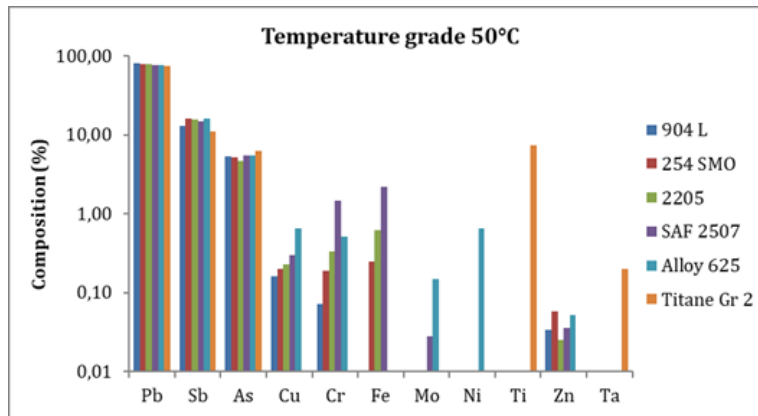


Figure 5- Pb, Sb and As concentration of the scales obtained after a 3 month circulation test in the prototype exchanger, for a temperature around 50°C.

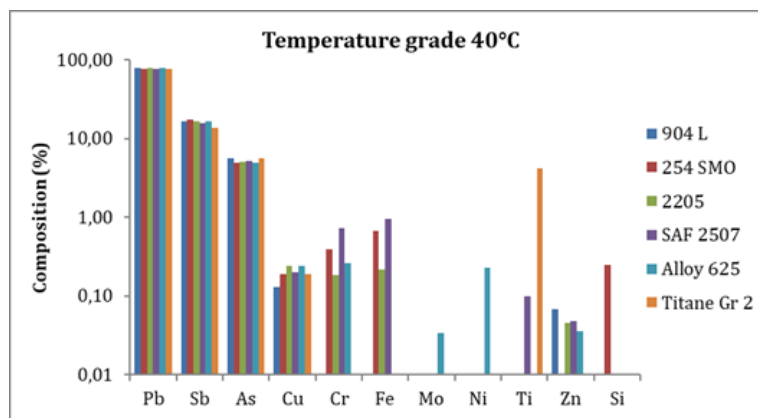


Figure 6- Pb, Sb and As concentration of the scales obtained after a 3 month circulation test in the prototype exchanger, for a temperature around 40°C.

When comparing the composition of the scales obtained from the different metallurgies for the same temperature grade, only small variations may be observed (Figures 4, 5 and 6). Otherwise, the chemical composition, and in particular the concentration of the major elements Pb, Sb and As, is very similar for the different metallurgies.

Concentration of Pb, As and Sb show an evolution with the temperature, which is observed for all the 6 metallurgies used for the prototype exchanger. Thus, Pb has the highest content in the scales deposited at 60°C, while the concentration decreases with lower temperature. As and Sb show the opposite trend, with the lowest concentration at the highest temperature and an increase of their content in the scales formed at 50°C and 40°C. This observation is consistent with Mouchot et al. (2018), where an increase of Pb, As and Sb concentration is observed in the scale of the coldest heat exchanger (70°C – 80°C) of the geothermal plant of Rittershoffen. Metal scales forming in geothermal installation may be the results of i) thermodynamic effects or ii) electrochemical corrosion. In the first case, minerals precipitate due to the variation of temperature and pressure in geothermal installations, inducing fluids oversaturated conditions. In the second case, the aggressive environment corresponding to the geothermal brine (high Cl concentration and CO₂ micro-degassing processes), coupled to hydrodynamic turbulence effects, may induce chemical corrosion of the metal surface. The electrochemical corrosion of casing and surface installation triggers the formation of iron oxides, which induces the deposition of an As and Sb enriched layer (Scheiber et al., 2015). Scheiber et al. (2012) showed that typical scales at Soultz-sous-Forêts powerplant present Pb-sulfides, as well as others mixed sulfides, mainly made of Fe, Sb, Cu and As. When observing the data collected from the scales of the prototype heat exchanger, it may be suggested that the variation of Pb, As and Sb concentration with the temperature decrease is due to the control of thermodynamic effects. However, at this stage of preliminary results, it is difficult to adjudicate about the processes involved in the formation of the observed deposit and to differentiate the influence of thermodynamic from corrosion effects. In order to better investigate this issue, it will be important to characterize the mineralogical form of the scales, and in particular to differentiate metal-sulfides deposit from native metal formation. This should be performed by X-ray diffraction in the forthcoming months, provided the amount of sales deposited during the test is high enough. The evolution of PbS, As₂S₃ and Sb₂S₃ versus Pb(s), As(s) and Sb(s) with the temperature might allow to better understand the scale formation mechanisms.

3.3. SEM-EDS analyses of scales precipitated on the various metallurgies at a temperature of about 60°C

SEM-EDS performed on scale samples allows to distinguish the major elements present in the samples as shown in the following figures.

3.3.1 Reference sample (SLZ_SOL_2017_15) obtained in 2017 in a 1.4410 water box, for a temperature around 65°C-75°C.

Figure 7 and 8 show data for the reference sample (SLZ_SOL_2017_15). It is made of a fine-grained matrix including “feather-like” crystals. The elements present in the fluid precipitated in the 1.4410 water box as kinds of “feathers” containing Pb, S, As, Sb, probably as sulfides. Traces of NaCl are also encountered in the vicinity of these precipitates. The same elements, together with little Cu, are found in the very fine grain matrix of this reference sample (Figure 8).

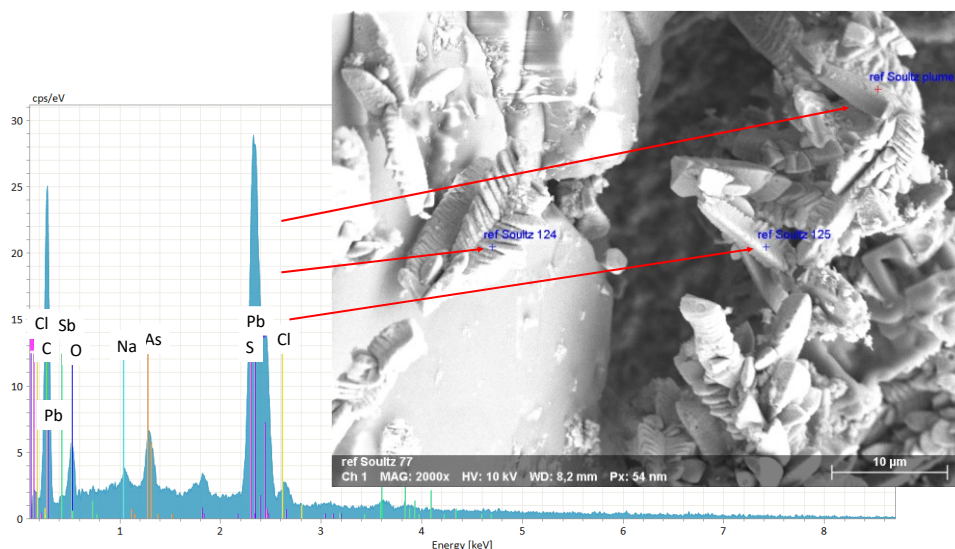


Figure 7 – SEM-EDS analysis of crystals in the reference sample showing Pb, S, As, Sb, probably as sulfides. Traces of NaCl are also encountered in the vicinity of these precipitates.

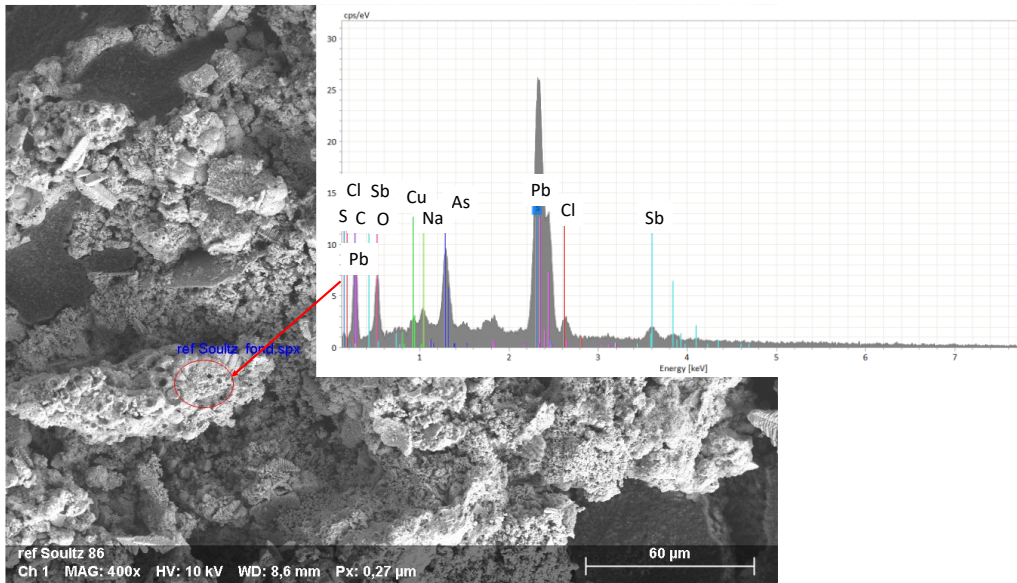


Figure 8- SEM-EDS analysis of the fine-grained matrix of the reference sample showing the same elements as the crystals visible in figure 4, together with Cu.

3.3.2 Sample 1D, 1.4410 steel, 60°C

Figure 9 shows the thickness of the scale while figures 10 and 11 provide SEM-EDX analyses of sample 1.4110 steel at 60°C.

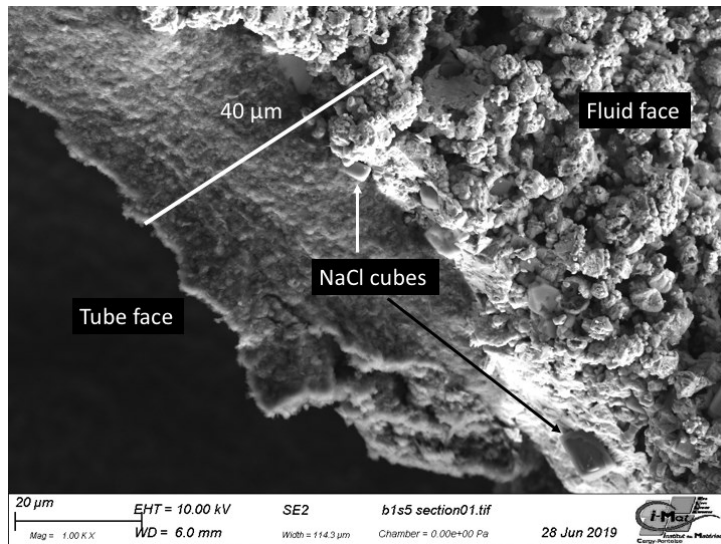


Figure 9: Sample 1D: 1.4410 steel, 60°C. The deposit shows a thickness of approximately 40 µm, made of two layers: a very fine-grained matrix in contact with the metal and a more granular one in contact with the fluid, including NaCl cubes.

It can be noted that “feather-shape” crystals observed in the reference sample have never been encountered in sample 1D even though they were produced in quite similar conditions: same metallurgy (1.4410 steel) and same temperature range (around 60°C for the prototype heat exchanger versus 65-75°C for reference). Figures 10 and 11 show structures encountered in contact with the fluid and with the tube respectively. However, the exposure time of the piece of material in which the reference sample was taken was much longer than for the sample 1D. The shape of the surface on which the samples were taken and the speed of the brine on these surfaces differ too, and represents some changing parameters which could explain the absence of “feather-shape” crystals in the sample 1D.

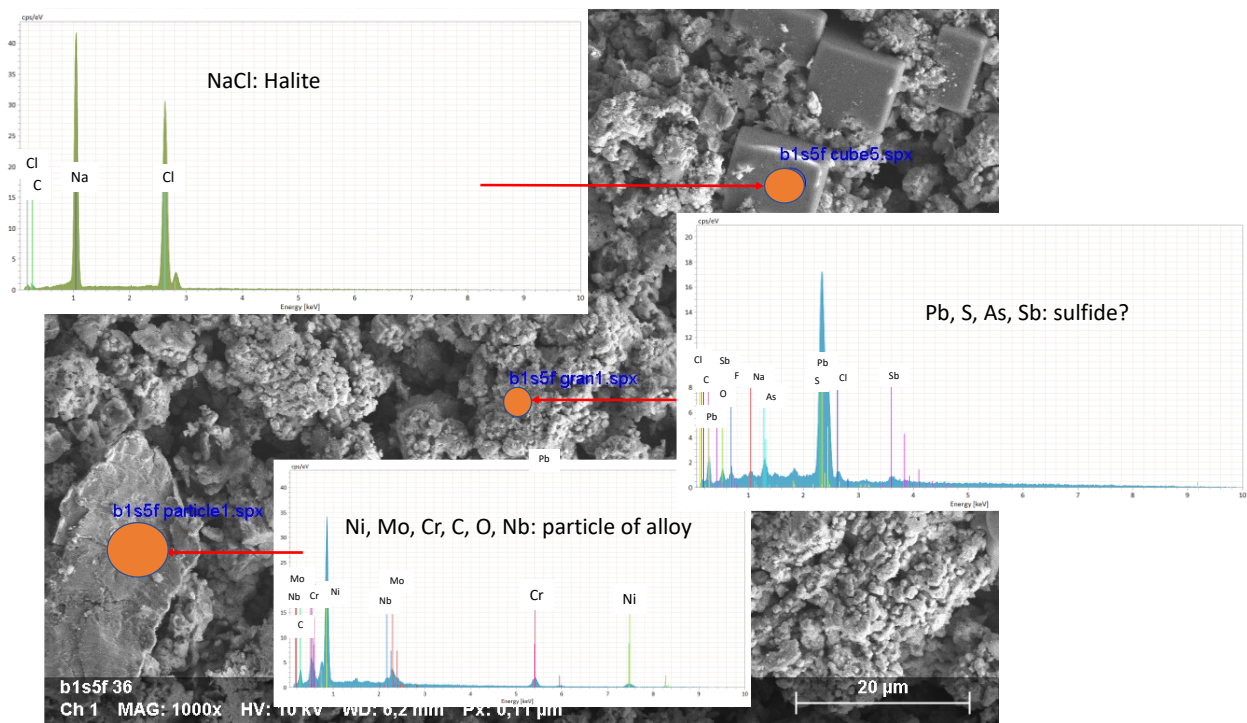


Figure 10: Sample 1D: 1.4410 steel, 60°C, surface in contact with the fluid. Halite cubes are embedded in, and protrude from, a granular matrix probably made of sulfide of Pb, As and Sb. Particles of alloy are also found at the surface of scale in contact with the fluid.

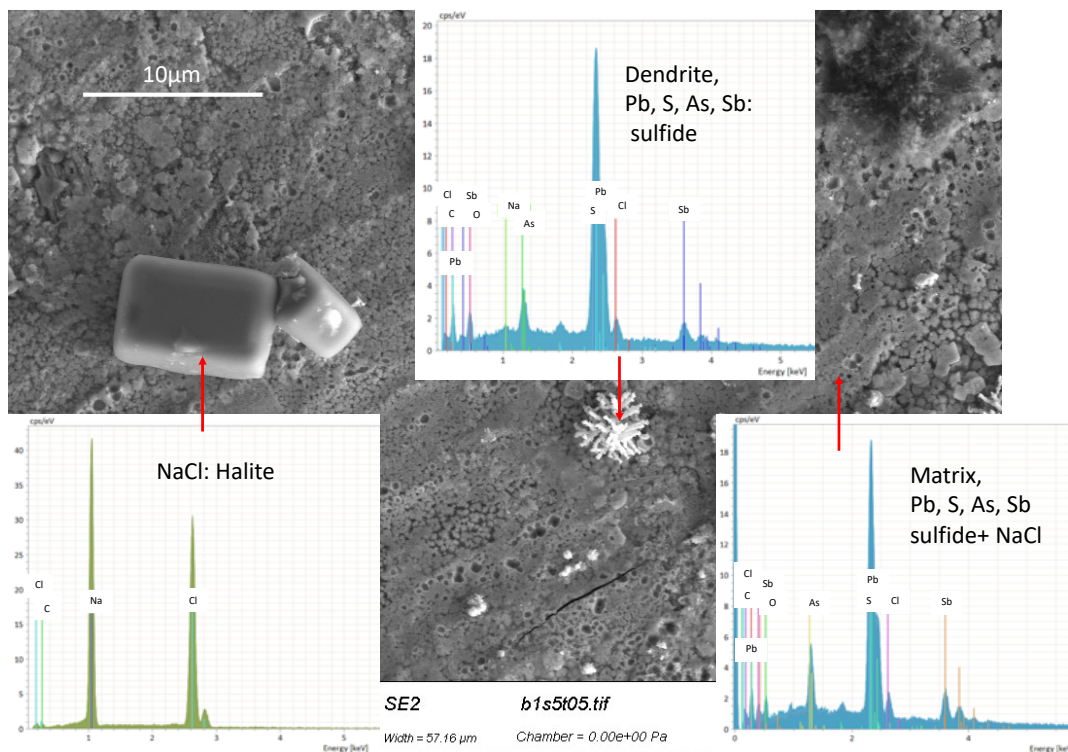


Figure 11: Sample 1D: 1.4410 steel, 60°C, surface in contact with the metal tube. Cubes of halite and dendrites of Pb, As, Sb sulfides are visible on a matrix of Pb, As, Sb sulfides and halite.

3.3.3 Sample 1A Bis, 1.4539, 60°C

The thickness of the deposit is about 30 µm. The face of the deposit in contact with the geothermal fluid is shown in Figure 12.

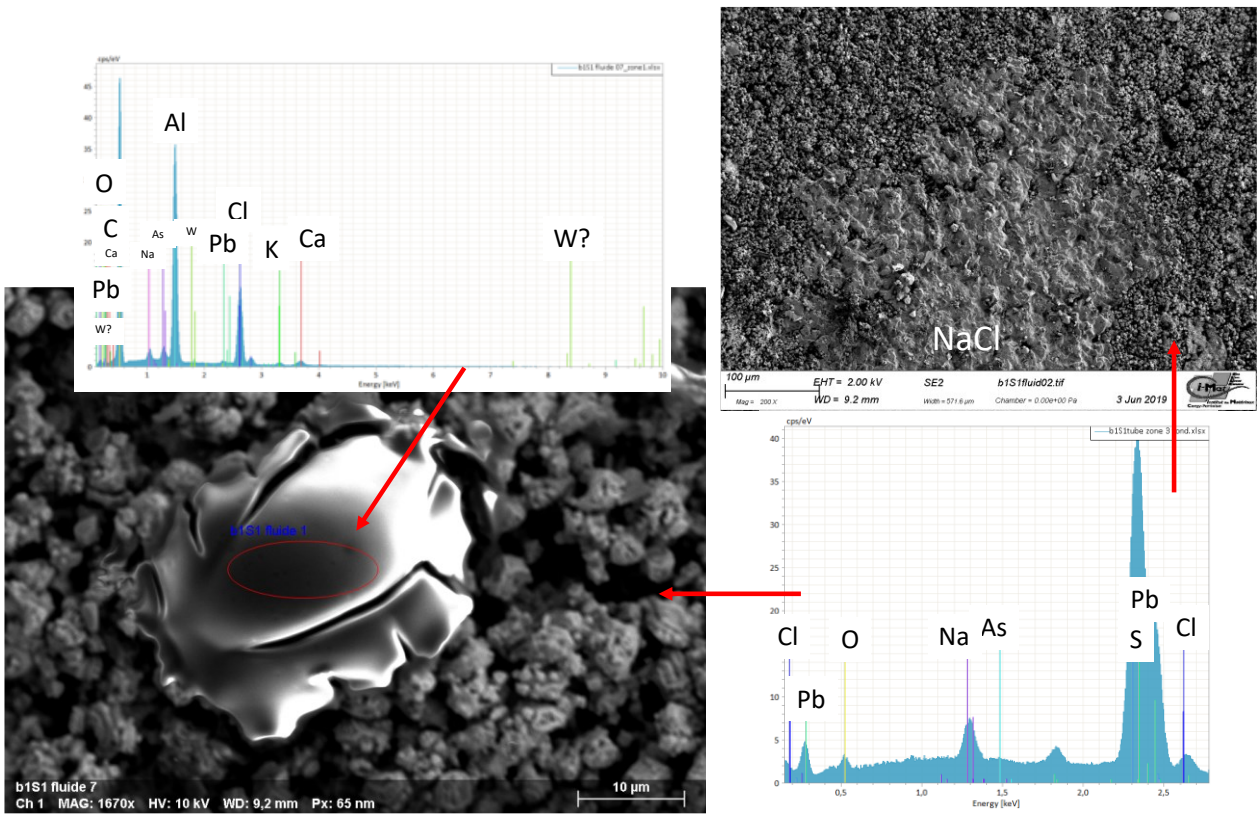


Figure 12: Sample 1A Bis: 1.4539, 60°C, surface in contact with the fluid. Halite zones are surrounded by a granular matrix of Pb and As sulfide on which are found Al-bearing rounded shapes (probably oxides).

The face of the scale in contact with the tube is shown in Figure 13.

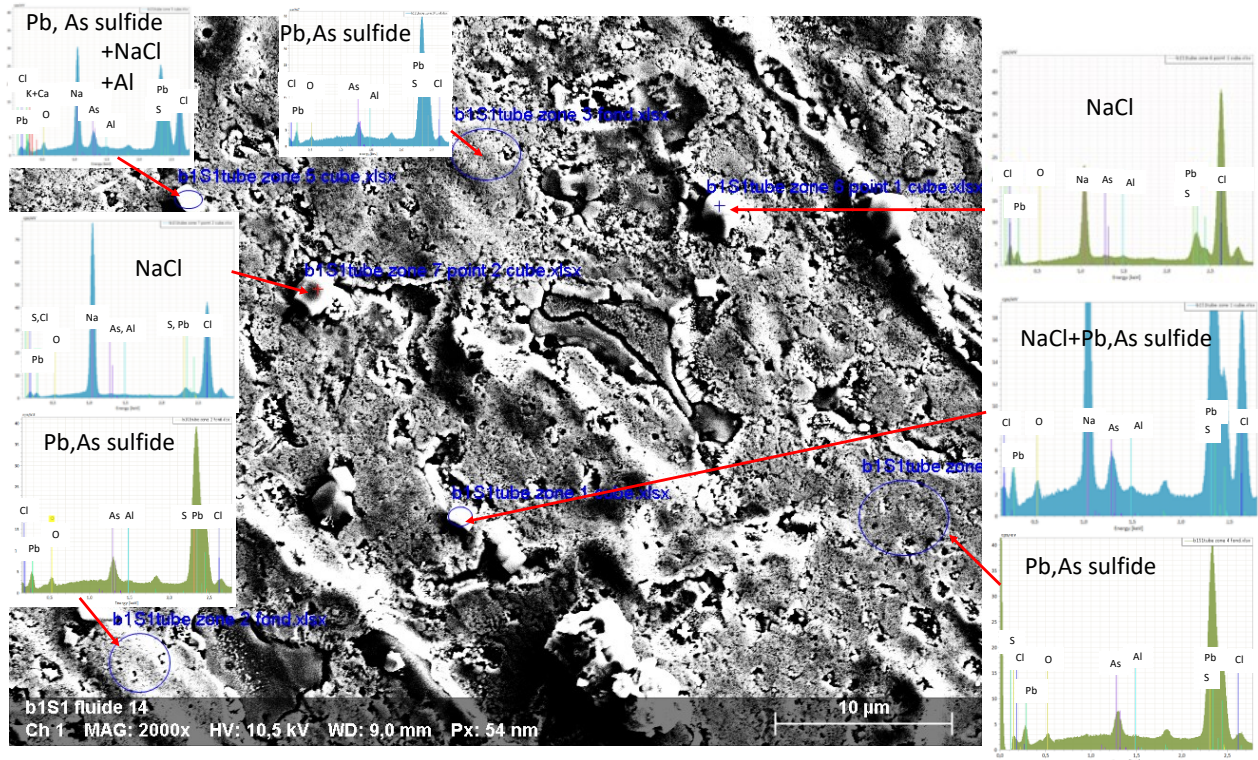


Figure 13: Sample 1A Bis: 1.4539, 60°C, surface in contact with the tube. Small halite cubes are protruding from a matrix of Pb and As sulfide. As they are very small (ca 1-2 µm), the analysis of NaCl cubes also samples the matrix, resulting in a mixing of elements.

3.3.4 Sample 1B, 1.4547, 60°C

The thickness of the deposit is about 30 µm. The face of the deposit in contact with fluid shows large halite cubes (200 µm and more) embedded within the granular Pb, S, As, Sb matrix (Figure 14).

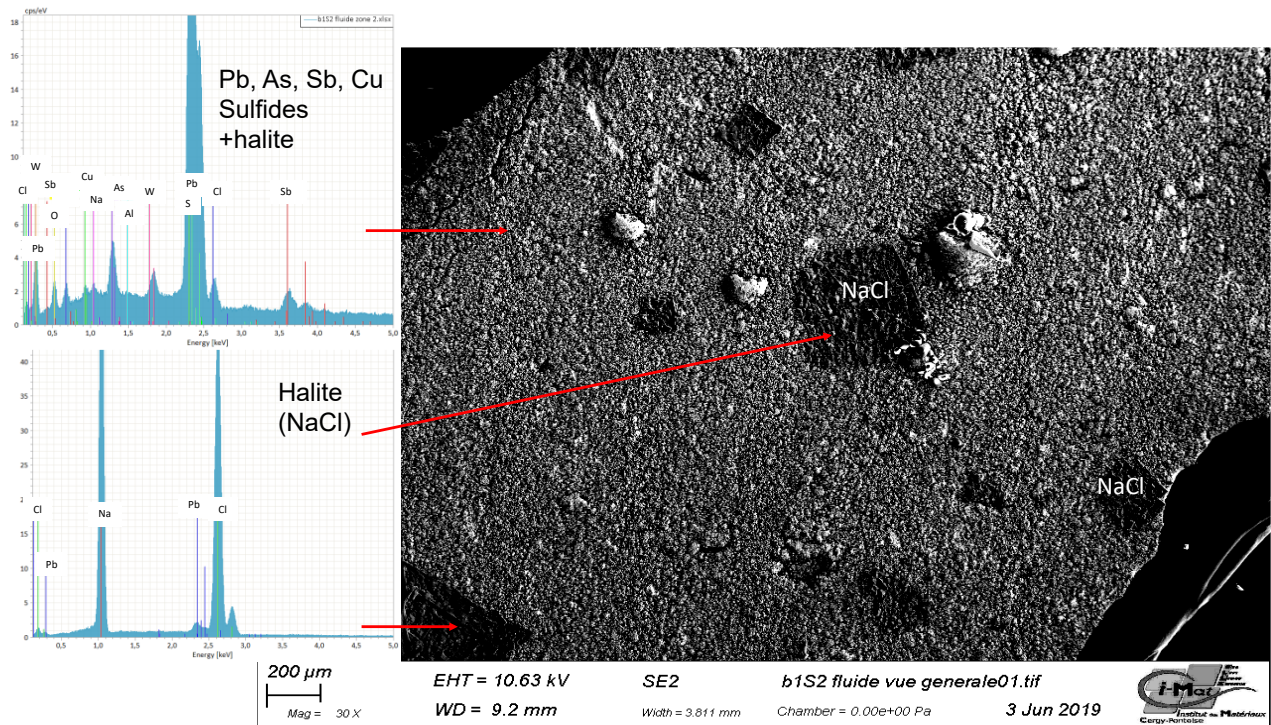


Figure 14: Sample 1B = 1.4547, 60°C, surface in contact with the fluid. Halite cubes are embedded in a Pb, As, Sb, Cu sulfide matrix.

3.3.5 Sample 1C, 1.4462, 60°C

As in samples 1A Bis, 1B and 1D, the surface of scale in contact with the fluid (Figure 15) shows wide square patches of halite (NaCl) embedded in a granular matrix made of Pb, S, As and Sb always showing traces of halite.

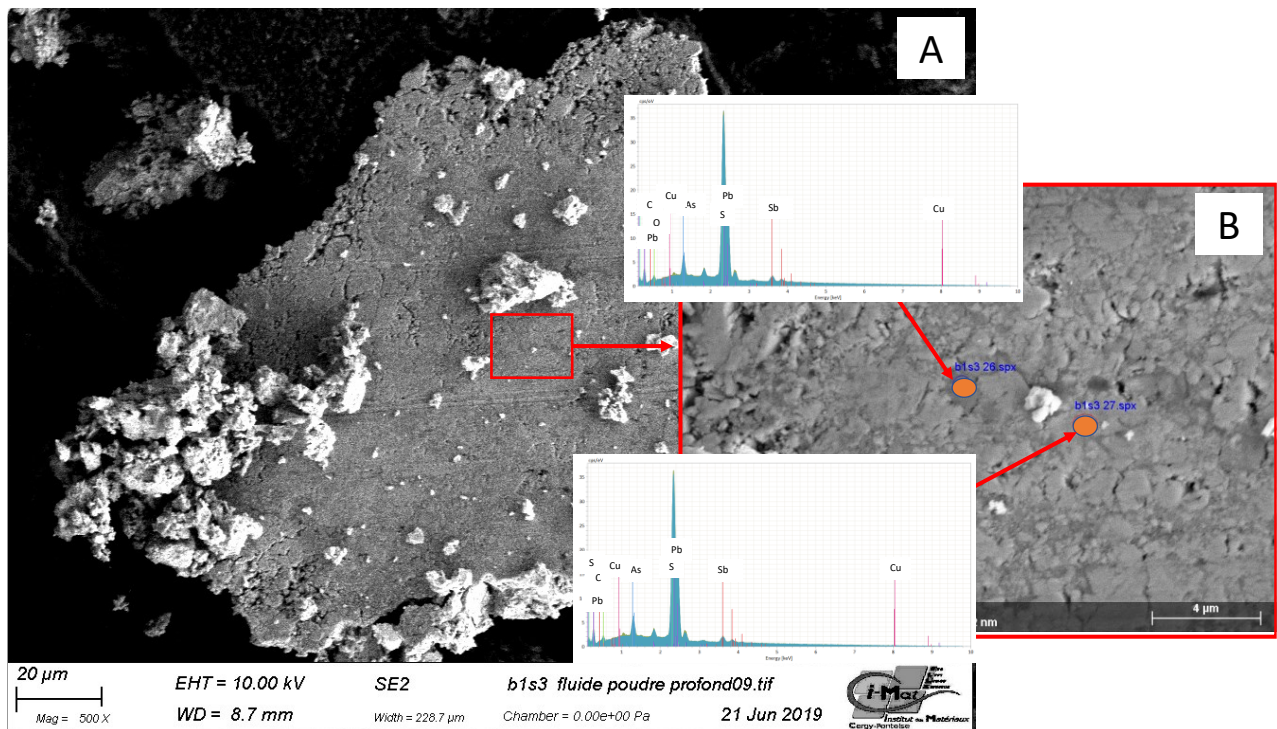


Figure 15: Sample 1C = 1.4547, 60°C, surface in contact with the tube. The big red rectangle is a zoom of the small one. The matrix contains Pb, S, As and Sb.

3.3.6 Sample 1F, 2.4858, 60°C

The surface of scales in contact with the tube of metallurgy 2.4858 at about 60°C (Figure 16) shows numerous round patches that were observed only on few surfaces (sample B, Figure 15) and not on the others (Figures 11, 13 or 15).

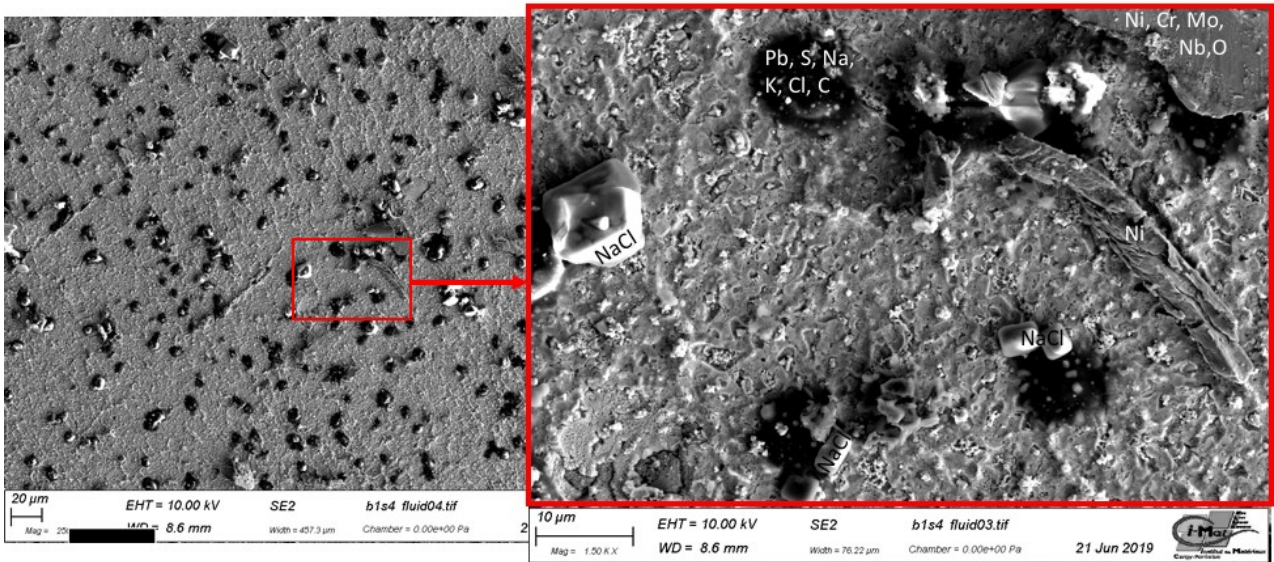


Figure 16: Sample 1F = 2.4858, 60°C, surface in contact with the tube. The big red rectangle is a zoom of the small one. The matrix contains Pb, S, As and Sb. It supports small cubes of NaCl found together with numerous round patches containing C and fragments of metal from the tubes bearing Ni, Cr, Mo, Nb and O.

3.3.7 Comparison of scales deposited on Ti Gr.2 at 60°C, 50°C and 40°C (samples 1K, 2K and 3K respectively).

The surface of scale in contact with the brine in Ti Gr.2 tubes is compared for the 3 temperatures (Figure 17): 60°C (sample 1K), 50°C (sample 2K), 40°C (sample 3K). All of them show tiny balls bearing Ti and O (Probably TiO₂). Similar balls are found in contact with the Ti Gr. 2 tube (Figure 18).

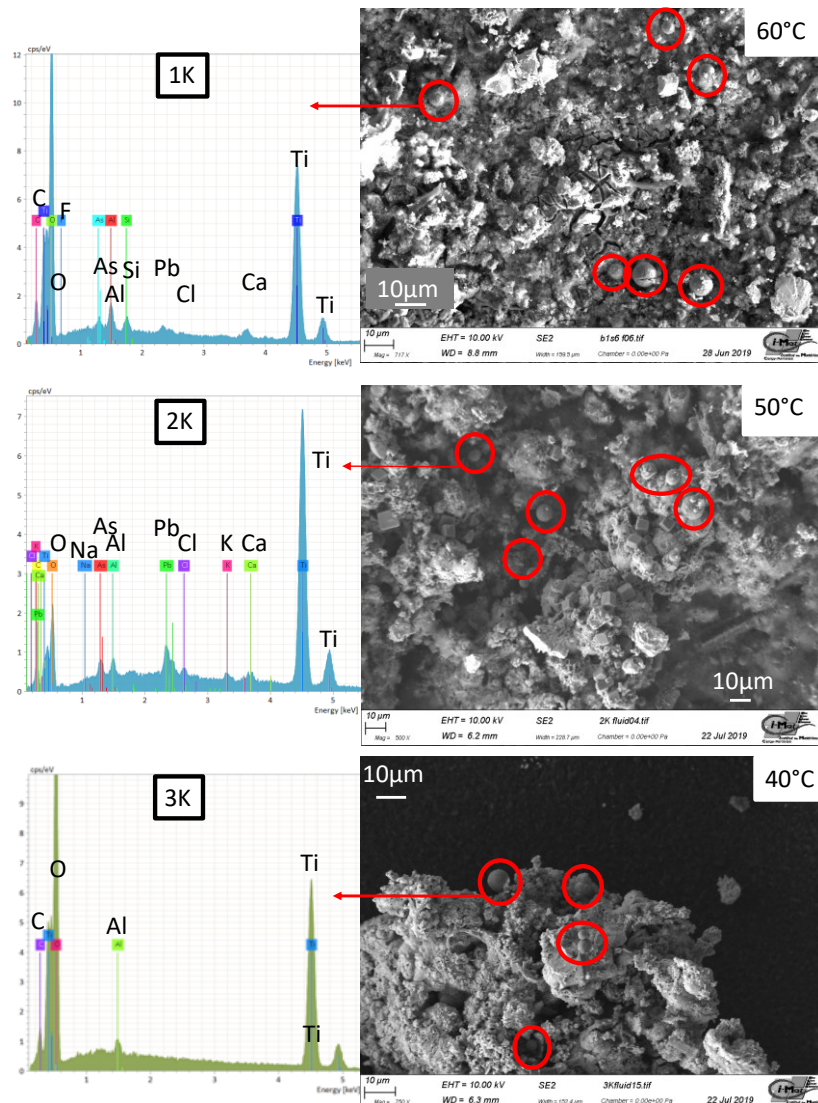


Figure 18: Samples 1K, 2K and 3K = Ti Gr.2, at 60°C, 50°C and 40°C, surface in contact with the geothermal fluid, SEM-EDS. Small balls bearing Ti (surrounded by red circles) are found at the 3 temperatures.

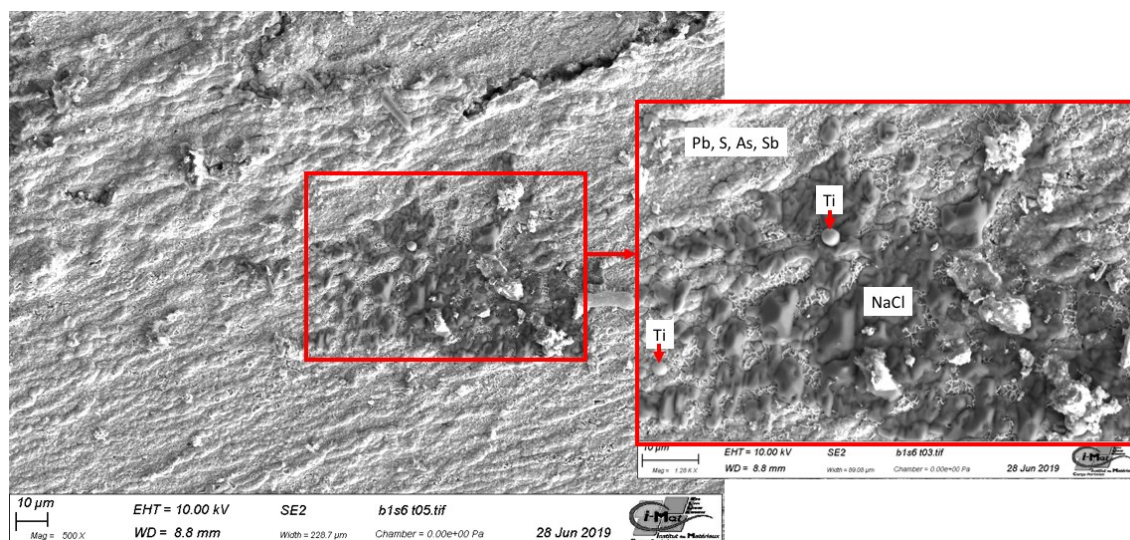


Figure 19: Sample 1K = Ti Gr.2, at 60°C, surface in contact with the tube, SEM-EDS, showing small balls bearing Ti, as on the surface in contact with the brine (Figure 18).

4. CONCLUSION

Constant features among the scales obtained in contact with the 6 metallurgies are: 1) NaCl crystals found either as large square patches or as small cubic crystals, 2) a granular matrix bearing mostly Pb, S, As, Sb, 3) small fragments of metal from the tubes. In addition, for the 3 samples precipitated at 60° C for which it was possible to have a section, the thickness of the deposit is 30 to 40µm which, if the precipitation was homogeneous with time, represents an average thickness of 10 to 13 µm per month. This figure is important for the operation of the power plant. It will be compared with the thickness of deposits that precipitated at lower temperatures to check whether the decrease of temperature plays a role on the amount of deposit, as assumed before the 3-month test performed in 2019.

The scales are mainly composed of Pb, As, and Sb. Other metals are present as traces. The composition of the scale only shows very slight variations within the different metallurgies. On the opposite, Pb, As and Sb concentrations vary with temperature: Pb content decreases with decreasing temperature, while Sb and As increase. Additional analysis should be performed on the scales, to better characterise the geochemical and mineralogical composition. The presence of Pb, As and Sb deposit in metal-sulfide minerals or on the form of native metal will be an important information to differentiate the processes triggering the scale formation, i.e. thermodynamic effects or electrochemical corrosion.

Variations are observed in the amount of metals present in the scales when the temperature decreases: amounts of Pb and Cu decrease from 60°C to 50°C and then remain constant or increase from 50°C to 40°C. On the contrary, amounts of As and Sb increase when the temperature decreases.

Concerning Ti metallurgy, one notes the decrease in the amount of Ti in the scales when the temperatures is lowered (Figure 4, 5 and 6, from around 11% to 4%). However, the same tiny balls bearing Ti and O (probably TiO₂) are found at both the surface in contact with the fluid and that in contact with the metal tube, whatever the temperature.

This characterisation of scales is a key feature for the optimisation of the heat exchanger: the thinner the scales, the better the heat exchange. In addition, a good knowledge of scales allows a better formulation of scale inhibitors, and potentially a better identification of the cleaning process to implement.

There is no real impact of the metallurgy on the composition of scales except for Ti: Ti is only found in scales formed in contact with TiGr.2 tubes. Cu seems to be brought (at least for some part) by the fluid.

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