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Examination of copper corrosion specimens from ABM 45, package 5

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Summary

In the field test ABM 45 (Alternative Buffer Materials), different types of bentonite clay were exposed to ground water and heated for five years in the Äspö Hard Rock Laboratory. Copper specimens were installed in the clay in order to measure corrosion during the exposure. The amount of corrosion was quantified by weight-loss measurements and the corrosion morphology was examined by light optical microscopy. The average corrosion depths of the copper specimens ranged from 2.3 µm to 5 µm, with a mean value of 3.2 µm. The results are in good agreement with the corrosion measured in LOT (Long-term test of buffer materials), a series of field experiments very similar to ABM. As for the corrosion morphology, surface defects were counted and measured on both a corroded specimen and uncorroded reference specimens after film removal. A statistical comparison revealed no significant difference between the depth of the deepest defects (8–30 µm) found on the corroded and reference specimens, suggesting that these are not related to the corrosion. This view is further supported by microscopic inspection of the defects, which seems to be caused by mechanical damage or wear. The defect surface density (number of defects deeper than 6 µm per unit area) were of the same order of magnitude on the corroded and reference specimens after film removal, but much lower on a reference specimen before removal of the corrosion film. It is suggested that the method of film removal had an influence on the surface topography and caused some localised corrosion.

Sammanfattning

I fältförsöket ABM 45 (alternativa buffertmaterial) exponerades olika typer av bentonitlera för grundvatten och uppvärmning under fem år i Äspölaboratoriet. Kopparprover installerades i leran för att mäta korrosion under exponeringen. Det genomsnittliga korrosionsdjupet kvantifierades genom gravimetrisk analys (mass-förlust) och korrosionsmorfologin undersöktes med ljusoptisk mikroskopi. Det genomsnittliga korrosionsdjupet för kopparproverna varierade från 2,3 µm till 5 µm, med ett medelvärde av 3,2 µm. Resultaten är i god överensstämmelse med den korrosion som uppmätts i LOT, en serie fältförsök vars utformning är väldigt snarlika ABM. När det gäller korrosionsmorfologin, räknades och mättes defekter och gropar på både ett korroderat prov och icke-exponerade referensprover efter att korrosionsprodukter avlägsnats från provytorna. En statistisk jämförelse visade ingen signifikant skillnad mellan de djupaste defekterna (8–30 µm) som återfanns på såväl korroderade prover som referensprover. Detta tyder på att groparna inte är relaterade till den korrosion som skett under exponeringen i Äspölaboratoriet. Denna slutsats stöds även av mikroskopisk inspektion av provernas ytor, vilka uppvisar spår av mekanisk påverkan och slitage.

Contents

1 Introduction

The field experiment ABM 45 (Alternative Buffer Materials package 4, 5, and 6) consisted of three packages, each containing a number of ring-shaped bentonite blocks mounted around a cylindrical 1 kW heat source made of steel. In package 5 the clay was heated to 80 °C for the first four and a half years, and between 150 °C and 250 °C during the last six months of the exposure. The packages were installed in boreholes at ca 420 m depth in the Äspö Hard Rock Laboratory. The aim of the test was to study and compare the alteration of different bentonite qualities due to ground water saturation, heating and interaction with corroding metals. Further experimental details of the ABM series can be found in Sandén et al. (2018).

In some of the bentonite blocks, copper specimens were installed with the aim of quantifying corrosion over the exposure period. Cylindrical copper specimens with a height of 25 mm and a diameter of 10 mm were made from oxygen free phosphorous doped (OFP) copper from SKB's canister laboratory in Oskarshamn, see Figure 1-1. Before being installed in the bentonite blocks, the specimens were washed with dilute hydrochloric acid and deionized water and weighed using analytical scales. The ABM 45 series contained in total 20 copper specimens of which four were saved as reference specimens and stored under dry indoor conditions at room temperature in order to avoid extensive corrosion. The 16 remaining specimens were divided equally between the ABM 4 and 5 packages. Package 5 was retrieved during the autumn of 2017 and had then been exposed for more than five years. Although the package contained eight copper specimens, only four were found during the retrieval. In addition to the specimens found at retrieval and analysed herein, further specimens have later been found in pieces of bentonite sent to external laboratories. These specimens will be analysed in a separate report.The specimens found during retrieval were identified as Specimen 9 (mounted in Asha 505 bentonite clay), and the Specimens 13, 15, and 16 (mounted in MX-80 clay). After retrieval, the copper specimens were kept in sealed plastic bags inside an inert-gas glove box to protect them from further corrosion.

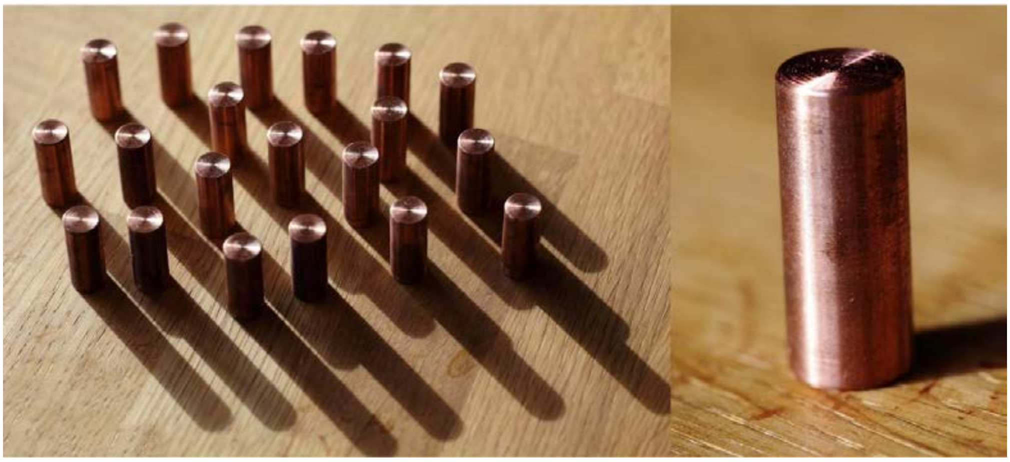

Figure 1-1. *Copper specimens before the ABM exposure.*

2 Examination of corrosion

The copper specimens from ABM package 5 were washed with deionised water in order to remove any remaining bentonite clay stuck to the surface. Gravimetric analysis was performed for all specimens by removing the corrosion film with chemical methods, and the topography of the underlying copper surface was examined with light optical microscopy.

2.1 Gravimetric analysis

Gravimetric analysis, or mass-loss analysis, was used to quantify the extent of corrosion that had occurred to the test specimens. The corroded specimens were chemically cleaned, removing the corrosion products by pickling. Repeated pickling is typically needed to remove all corrosion products. The specimen is weighed after each cleaning treatment and the procedure is repeated until the mass-loss between two treatments is very small, and comparable to mass-loss on an unexposed reference specimen. Both the metal and the corrosion products determine what chemical cleaning solution is suitable to use. Ideally, removal of the corrosion products is done in a way that doesn't affect the underlying base metal. The effect on the base metal is verified by use of the reference specimen during the cleaning procedure.

The standard SS-EN ISO 8407:2014 lists solutions that are recommended for removing corrosion products from different metals. In a test like ABM 45, the initial (and major) oxidant is expected to be molecular oxygen, present in the porosity of the bentonite clay and in the initially air-filled gaps in the experiment, e.g. between the central heater and the bentonite rings. In the longer term (several months), when the environment has become anoxic (Smart et al. 2015), and when the sulphide-containing ground water has had time to diffuse through the dense clay (years), the composition of the corrosion film is expected to change into a mixture of copper oxides as well as stoichiometric and non-stoichiometric Cu_xS phases, e.g. Cu_2S (chalcocite). On even longer time-scales, only copper sulphides are expected, since the oxide film is unstable in anoxic sulphide solutions (Smith et al. 2007). From experience (possibly due to the complex composition of the corrosion film), it is known that it is not always enough to use the 50 % HCl solution, as recommended by the ISO standard, but alternating pickling with HCl and NH_4OH solutions is needed to remove the corrosion film completely. In this work the pickling was carried out firstly with a duration of 1 minute, then with alternating 2 minute periods of immersion in an ultrasonic bath with HCl (37 wt% diluted with deionized H_2O with a ratio 1:1) and NH_4OH (20 wt%) until the pickling was considered to be complete. Each immersion in the two different pickling solutions was counted as one pickling period, i.e. 2 minutes in HCl + 2 minutes in NH_4OH is counted as only 2 minutes in the figures contained in the appendices.

Plotting the weight-loss of a corroded specimen after repeated cleaning treatments versus the number of cleaning cycles (or time units) gives a principle graph as shown in Figure 2-1, which is used to determine when the corrosion products have been removed from the specimen surfaces. The line AB corresponds to the removal of corrosion products. The cleaning procedure is repeated until three points on the nearly horizontal line BC are achieved. Up to point B there are still corrosion products left on the surface, while the line BC measures the extent of corrosion of the base metal during cleaning. The mass-loss is extrapolated to point D on the y-axis, which gives the mass-loss caused by corrosion during the experiment. The difference between C and D corresponds to the weight-loss of the base metal during the cleaning cycles.

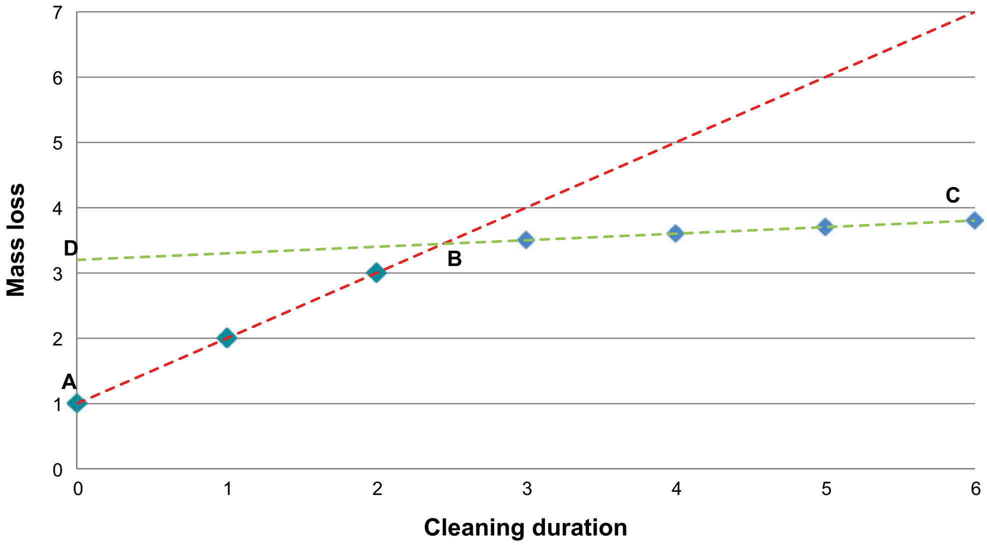

Figure 2-1. *Principle plot of gravimetric analysis according to SS-EN ISO 8407:2014.*

2.2 Examination of defects and surface morphology

A light optical microscope with calibrated focus was used to determine the depth of all types of defects found on the surfaces of the specimens. The depth was measured by first focusing on the highest point of a defect and setting the focus to zero. The focus was then adjusted to the deepest point of the same defect and the depth was determined using the calibration marks of the focus adjuster. This method of defect or pit depth measurement is described in the ASTM standard G46-94 (ASTM 2013). Since the copper specimens were cylindrical, the mantle surfaces were scanned for defects along the circumference of the cylinder. Eight scans were made by rotating the cylindrical specimens 45 degrees about the cylinder axis between each examination. The five deepest defects found were measured and documented by photography. Although it is discussed further below that the defects found seemed to be of different origins, when discussing unspecified local surface features we refer to all of these as *defects* in this report. Surface defects were counted and measured by careful manual examination of a rectangular area marked on the specimen. All defects deeper than 6 µm in the marked area were measured and documented. The defect surface density was evaluated as the number of defects deeper than 6 µm divided by the area of the examined surface. This method is described in the ASTM standard G46-94.

3 Results

The ABM 45 package 5 was retrieved during the autumn of 2017 and the copper specimens were placed in sealed plastic bags and stored in an inert gas glove box in order to protect them from further corrosion. In the beginning of 2018 the four corroded specimens found during retrieval (numbered 9, 13, 15, and 16, see Figure 3-1) were sent to Swerea KIMAB in Kista, together with two reference specimens (numbered 18 and 19), prepared in the same way as the corrosion specimens but never exposed to the bentonite-groundwater environment.

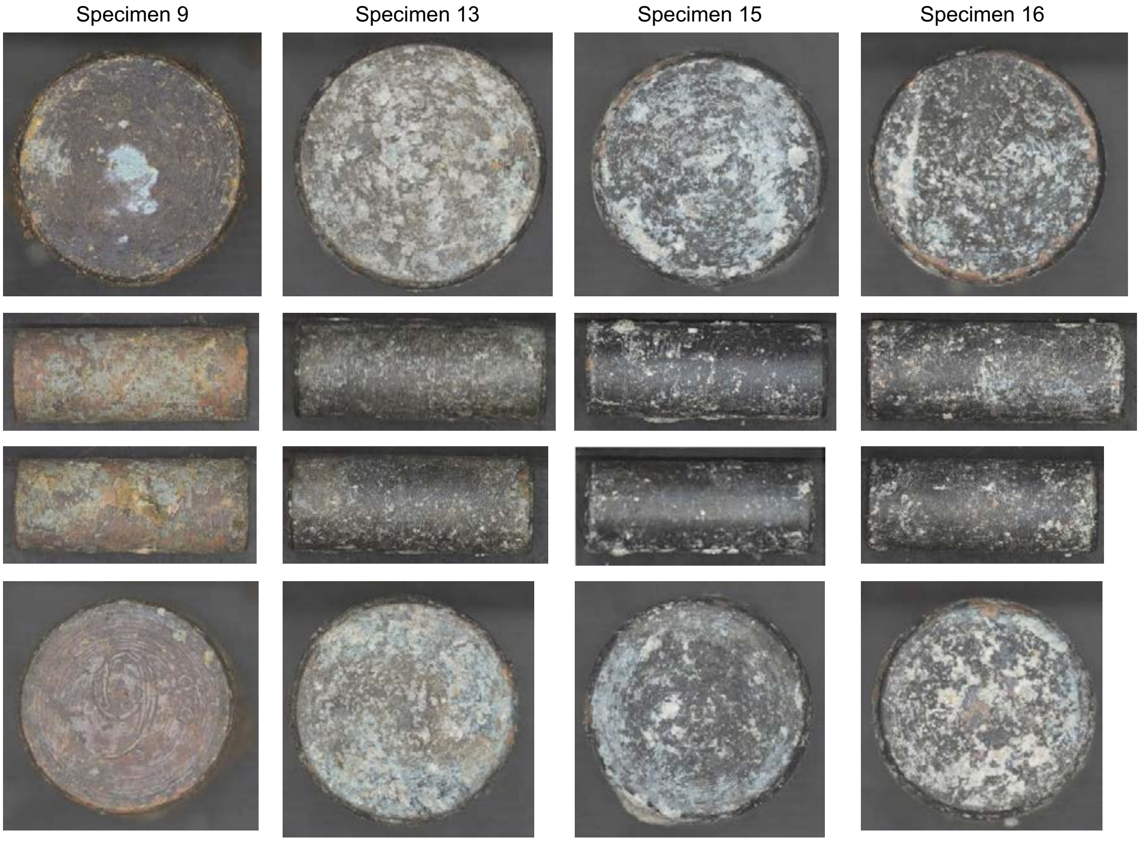

Figure 3-1. *The corroded copper specimens from ABM 45 package 5. The images show the appearance of the specimens prior to cleaning. Some bentonite clay is still attached to the surfaces. Top row: end surface of the specimens; Middle two rows: representative images of the side surfaces of the specimens; Bottom row: opposite end surface of the specimens.*

3.1 Results of gravimetric analysis

The corroded Specimens 9, 13, 15, 16 and the reference Specimen 18 were cleaned in order to remove the corrosion products on their surfaces. The results are summarised in Table 3-1, further details are given in the Appendix.

Table 3-1. Results from gravimetric analysis.

Specimen ID	Mass-loss		Average corrosion depth
	[g]	$[g/m^2]$	[µm]
9	0.0425	44.8334	5.00
13	0.0193	20.3688	2.27
15	0.0247	25.9774	2.90
16	0.0231	24.4957	2.73
Mean	0.0274	28.9188	3.23
Standard deviation	0.0103	10.8719	1.21
18 (reference)	0.0051	5.4151	0.60

The mass-loss of the corroded specimens corresponds to average corrosion depths of ca 2.3–5 µm, with a mean value of 3.2 µm. Some dark spots (possibly Cu_2S) remained on the surfaces of the corroded copper specimens after cleaning (see Figure 3-2), however, the results of the cleaning procedure indicated that the cleaning was complete (see Appendix).

The mass-loss of the reference Specimen 18 corresponds to an average corrosion depth of 0.6 µm, which is markedly lower than for the exposed specimens. The reference specimens were covered by a dark brown oxide layer that was removed during the cleaning (pickling) treatment (see Figure 3-2), which explains the mass-loss of the reference specimen.

3.2 Results from microscopy

For the exposed Specimen 9 and the reference Specimen 18, the mantle surface was examined in the microscope in order to find the five deepest surface defects after removal of the corrosion film. For reference Specimen 19, the same examination was also made although the corrosion film was not removed. The defects found were documented by photography (Figures 3-3 to 3-5). As seen in the photos there are parallel ridges in the copper surface. These ridges are ca 10 µm high and are due to machining durning preparation of the specimens (Figure 3-6). Defects were counted and measured according to the standard method described in Section 2.2. The results are summarised in Table 3-2 and analysed statistically in Figure 3-7. The results show that the depth of the five deepest defects are similar in all specimens but that defect densities are higher in the exposed specimen subjected to the cleaning procedure (Specimen 9) and in the reference specimen subjected to the cleaning procedure (Specimen 18).

Specimen 9, pickled

Specimen 13, pickled

Specimen 15, pickled

Specimen 16, pickled

Specimen 18, (reference) pickled

Specimen 19, (reference) not pickled

***Figure 3-2.** Corroded copper specimens after pickling. Reference specimens pickled and not pickled.*

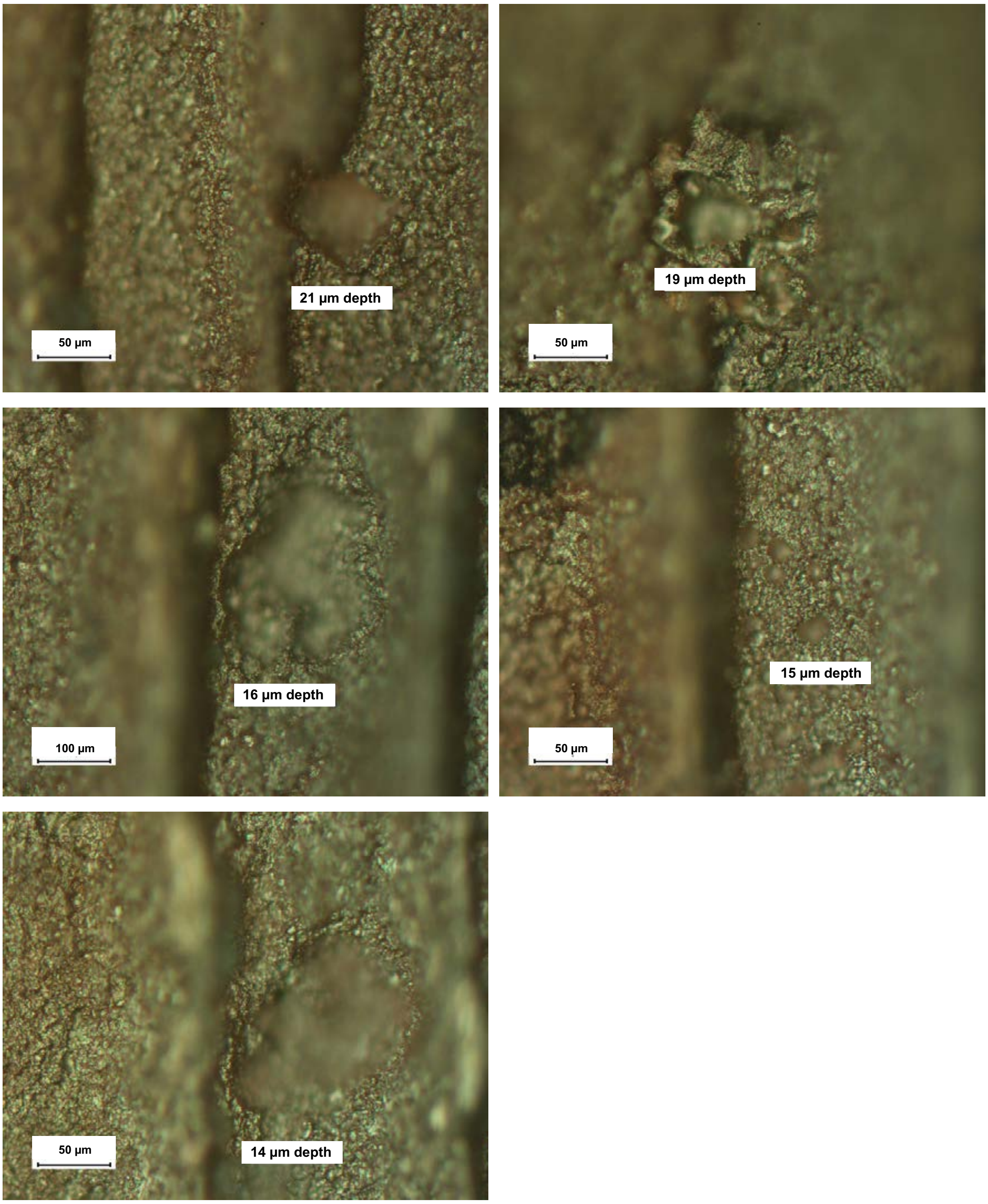

Figure 3-3. *Microscope images of the five deepest defects found on Specimen 9.*

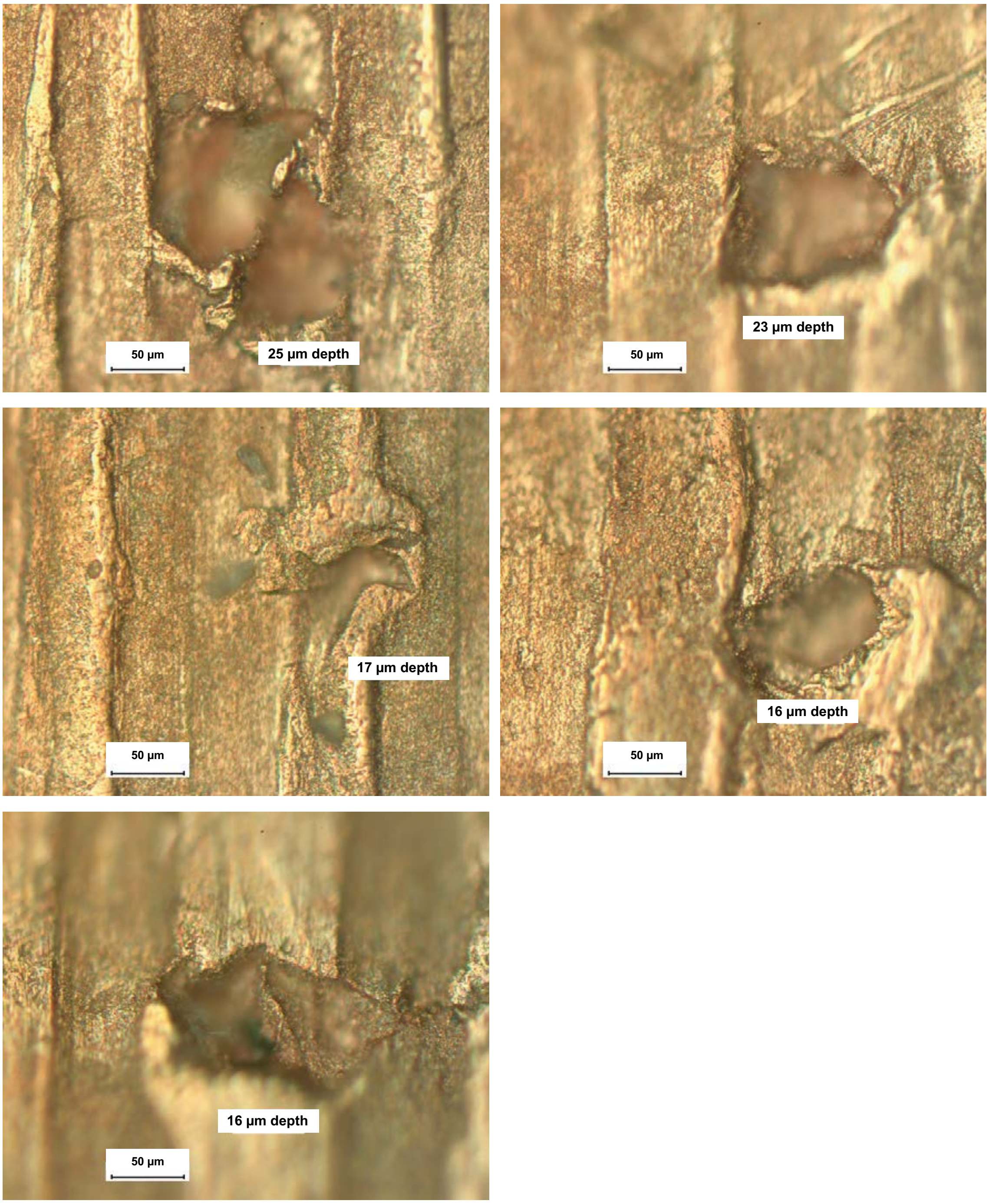

Figure 3-4. *Microscope images of the five deepest defects found on Specimen 18.*

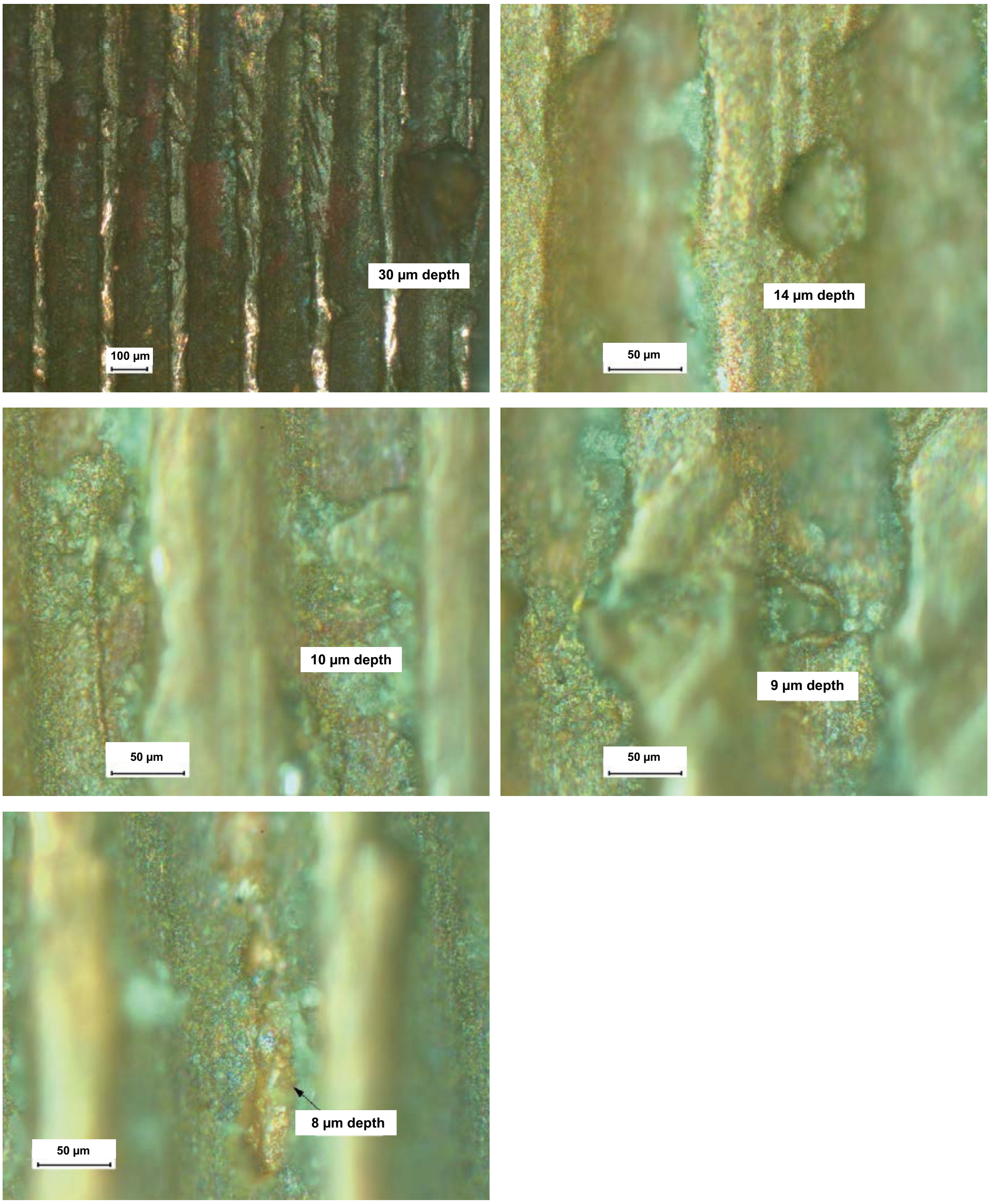

Figure 3-5. *Microscope images of the five deepest defects found on Specimen 19.*

Table 3-2. Results from evaluation of surface topography according to the ASTM-standard G46-94.

Specimen ID	Five deepest defects [µm]					Average corrosion depth from weight loss [µm]	Defect density		
							Area [cm^2]	Number of defects	Defect density [defects/ cm^2]
Corroded Specimen 9, after cleaning	21	19	16	15	14	5.00	0.5	102	204
Reference 18, after cleaning	25	23	17	16	16	0.60	0.25	75	300
Reference 19, with oxide layer	30	14	10	9	8	–	1	5	5

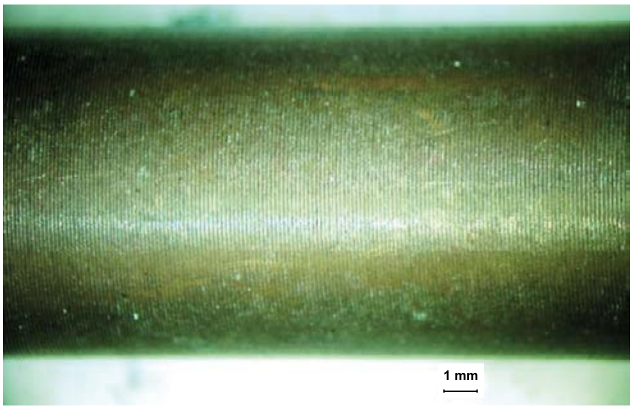

Figure 3-6. *Reference Specimen 19. Machining marks on the specimen were found to be ca 10 µm deep.*

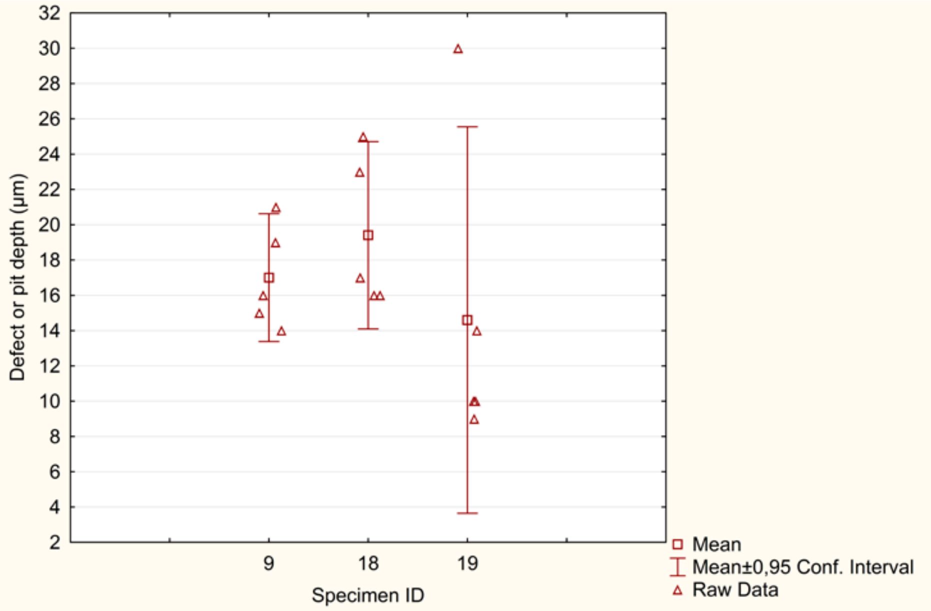

Figure 3-7. *Mean plot of the deepest defects found on Specimen 9 (corroded specimen, after cleaning) and reference Specimens 18 (after cleaning) and 19 (oxide still present) (Table 3-2). The mutual overlap between the mean values and the two standard deviations (95 % confidence intervals) of the corroded specimen and the references mean that there is no significant difference between the five deepest defects on the specimens.*

4 Discussion

From the appearance of the corroded copper specimens (Figure 3-1) a difference may be noted between the specimen mounted in Asha bentonite (Specimen 9) and the specimens in MX-80 (Specimens 13, 15, 16). The reason for this apparent difference in composition and/or morphology of the corrosion film is currently unknown. The corrosion products will be analysed on additional copper specimens found after retrieval, as discussed in the introduction. Although some bentonite clay is visible as light patches at the surfaces in Figure 3-1, it is also possible that some of the light areas were formations of some Cu(II)-hydroxy corrosion product. In the LOT series, which contained only MX-80 bentonite, it was confirmed that molecular oxygen was the major oxidant causing the corrosion observed (Wersin 2013). At the copper surface, mainly Cu_2O and some $Cu_2(OH)_3Cl$ were identified with XRD. While particles rich in Cu and S were found at the copper-clay interface, elemental analysis by EDS showed in general very little sulphur (Karnland et al. 2009). Due to similar experimental configurations and dimensions it is reasonable to assume that the corrosion scenarios were similar in ABM and LOT.

The average corrosion depths of the copper specimens from ABM package 5 ranged between 2.3 µm and 5 µm, with a mean value of 3.2 µm. This is in good agreement with the field experiments LOT/S1, LOT/A0, and LOT/A2 that were also performed in the Äspö HRL (Karnland et al. 2000, 2009, 2011). In the LOT series the average corrosion depth ranged from 1.5 µm to 4.8 µm, with a mean value of 3.4 µm. The packages of the LOT series had the same dimensions as the ABM series, i.e. the compacted bentonite rings were 17 cm thick (difference between outer and inner diameter) and there was initially an air-filled gap of a few mm between the copper heater and the clay. While the ABM package 5 was kept at 80 °C for four and a half year and then heated further to 150 to 250 °C for the last six months, the corrosion specimens of the LOT series were heated to between 30 and 80 °C and the experimental durations ranged from one to six years. A possible explanation of the variation in measured corrosion depth within the ABM (and LOT) series could be heterogeneous saturation of the clay, which affects the diffusion of O_2.

The deepest defect found on the corroded Specimen 9 was 21 µm, while the deepest defect found on the reference specimen was 25 µm (Table 3-2). The average of the five deepest defects found on the corroded Specimen 9 were 17 µm, while the corresponding average defect depth for the reference specimen was 19.4 µm. A comparison between the deepest defects found on the corroded specimens with the defects found on the references shows that there is a mutual overlap of the confidence intervals (95 %) and the mean values of the deepest defects found on the specimens (Figure 3-7). This means that there is no significant difference between the deepest defects found on the specimen surfaces, which suggests that these are not related to corrosion during the experimental exposure.

In principle, since the reference Specimen 18 was slightly corroded, the deepest defects analysed could have been caused by corrosion. However, this would mean that the same pit depths were formed after a mass-loss corresponding to 0.6 µm corrosion (Reference 18) as for 5 µm corrosion (Specimen 9). The conclusion that the deepest defects found are not related to corrosion of the copper surfaces is further supported by the morphology and shape of some of the defects shown in Figure 3-4, suggesting that mechanical damage or wear is the likely origin of the topography observed. For example, the 17 µm deep defect found on Reference 18 shows signs of a directed mechanical damage on the surface. It may further be noted that defects of similar dimensions and shapes have been reported for both machined and polished copper surfaces, and before as well as after corrosion exposure (Högberg et al. 2017, Gordon et al. 2018).

The occurance of defects deeper than 6 µm was of the same order of magnitude for both the corroded and reference specimens after film removal, ca 10^2 defects/cm^2. This is markedly different to the reference specimen before film removal, which had only five defects/cm^2. One possible explanation could be that the oxide film was covering shallow defects before its removal. However, it has also recently been reported that aerobic acid cleaning of both corroded copper specimens and reference specimens can cause some localised corrosion (Gordon et al. 2018). In that study it was found that while film removal using cycles of alkaline (20 wt% NH_4OH) and acid (37 wt% HCl) exposure (as used herein) had a pronounced effect on the topography of the copper surface, whilst using only the alkaline solution did not affect the copper surface significantly, as compared with uncorroded reference specimens. Given the similarity in defect density found on the exposed and reference specimens subjected to the cleaning procedures, it seems plausible that the film removal caused some localised corrosion of the copper Specimens 9 and 18.

From this comparison, it seems that localised corrosion did not occur during the exposure in the Äspö HRL, however, the number of data is small and the presence of defects and machining marks on the specimens makes them less suitable for evaluation of topographical surface changes during exposure. It is important to note though, that literature studies of copper corrosion in aerobic and saline environments suggest that passivation of copper (a prerequisite for pitting corrosion) require concentrations of carbonate and chloride that are far from the composition of the Äspö ground water (King and Lilja 2014).

5 Conclusions

From the gravimetric analysis it can be concluded that the metal loss corresponds to average corrosion depths of 2.3 µm to 5 µm, with a mean value of 3.2 µm. These values are similar to earlier results obtained from the LOT series, in which corrosion ranged from 1.5 to 4.8 µm, with a mean value of 3.4 µm.

Microscopic examination of defects after film removal revealed no significant difference between the deepest (8–30 µm) defects found on corroded or reference specimens, suggesting that these are not related to corrosion. This conclusion is further supported by microscopic inspection of the copper surfaces, which shows signs of mechanical damage and wear.

A higher defect surface density was found on the corroded specimen after removal of the corrosion products compared to a reference specimen stored in indoor atmospheric conditions. However, the defect density on the corroded specimen was of the same order of magnitude as that on an unexposed reference specimen after removal of the corrosion product, suggesting that the cleaning procedure, rather than the actual experimental exposure, can cause localised corrosion of copper.

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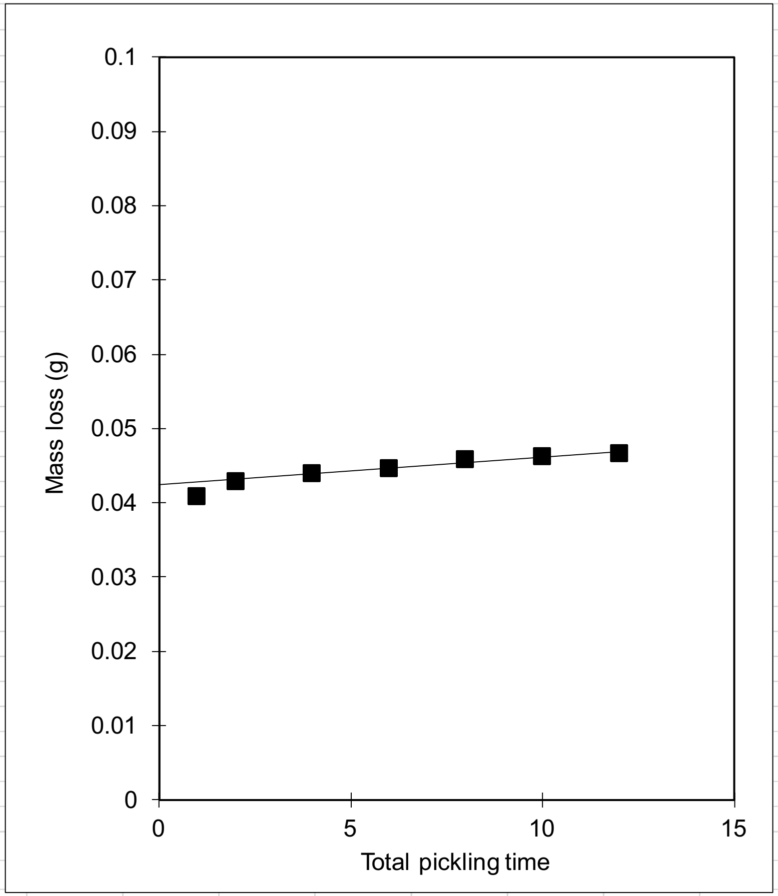
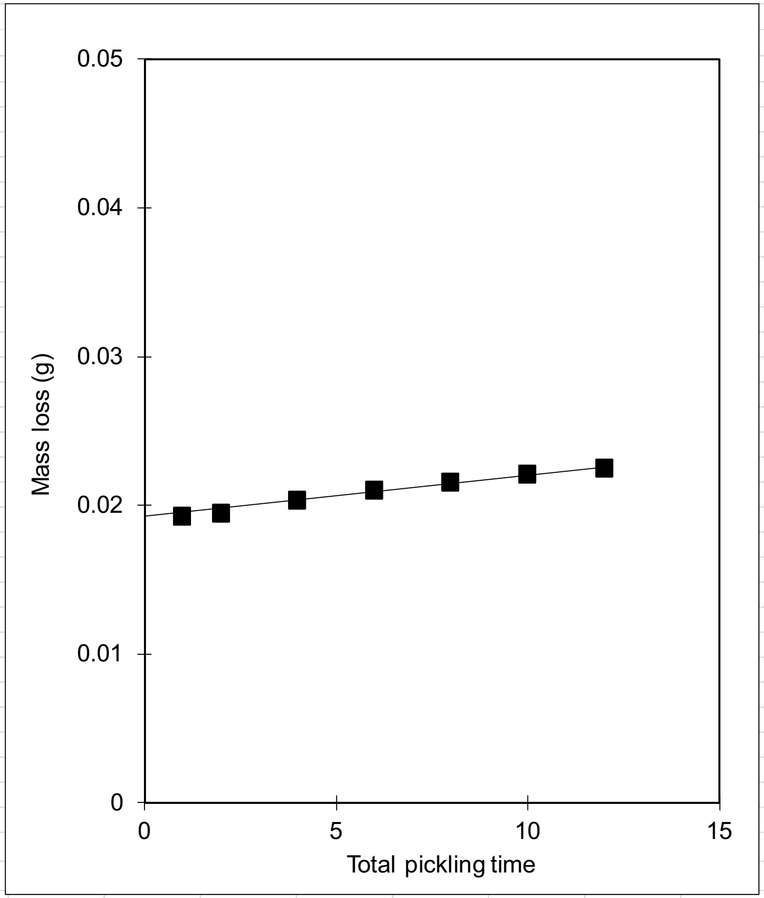
Appendix

Raw data from the gravimetric analysis of the corroded Specimens 9, 13, 15, 16, and the unexposed reference Specimen 18. The gravimetric analysis was carried out in accordance with the standard SS-EN ISO 8407:2014.

	Material				**Specimen**
	Cu				9
Pickling no.	**Mass (g)**	**Pickling (min)**	**Mass-loss (g)**	**Point incl. diagram?**	**Mass (g)**
0	17.5573	0			
1	17.51651	1	0.0408	0	0.0000
2	17.51448	2	0.0428	0	0.0000
3	17.51344	4	0.0439	1	0.0439
4	17.51275	6	0.0446	1	0.0446
5	17.51148	8	0.0458	1	0.0458
6	17.51108	10	0.0462	1	0.0462
7	17.51063	12	0.0467	1	0.0467
	Sx	Sy	Sx2	Sy2	Sxy
	40	0.22712	360	0.010322	1.83154
	Intercept with Y-axis			0.042508	
		Slope		0.000365	
		r		0.978288	
		Area	0.000948133	m^2	
		Densitet	8.96	g/cm^3	
		Corrosion:	44.83339	g/m^2	



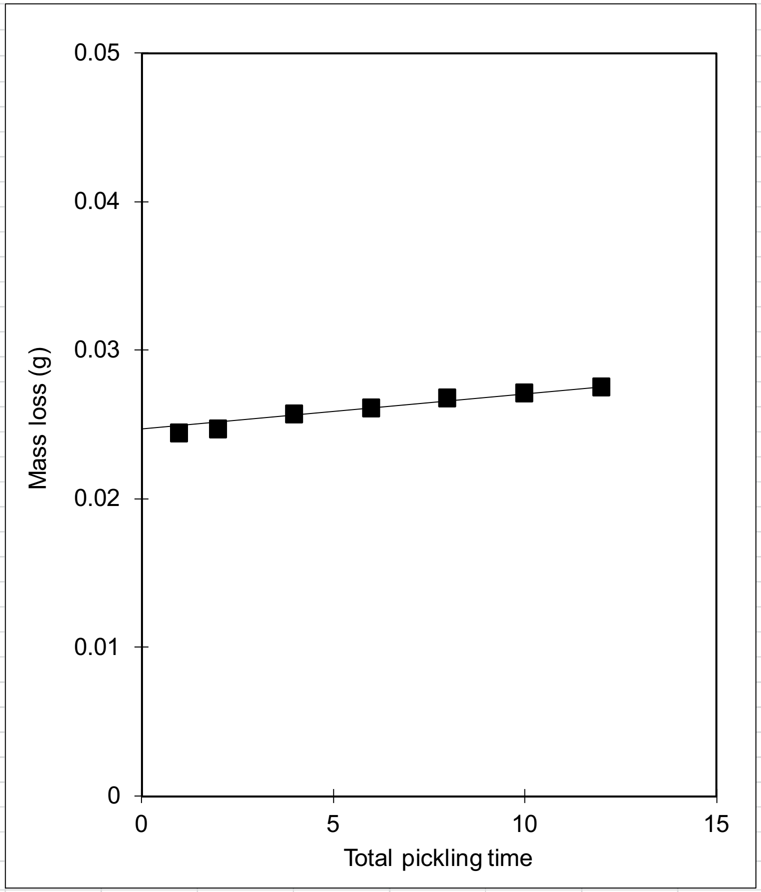
	Material				**Specimen**
	Cu				13
Pickling no.	**Mass (g)**	**Pickling (min)**	**Mass-loss (g)**	**Point incl. diagram?**	**Mass (g)**
0	17.4831	0			
1	17.4639	1	0.0192	0	0.0000
2	17.46368	2	0.0194	0	0.0000
3	17.46284	4	0.0203	1	0.0203
4	17.46214	6	0.0210	1	0.0210
5	17.46158	8	0.0215	1	0.0215
6	17.46104	10	0.0221	1	0.0221
7	17.46065	12	0.0224	1	0.0224
	Sx	Sy	Sx2	Sy2	Sxy
	40	0.10725	360	0.002304	0.86896
	Intercept with Y-axis			0.019258	
		Slope		0.000274	
		r		0.995016	
		Area	0.000945464	m^2	
		Densitet	8.96	g/cm^3	
		Corrosion:	20.36883	g/m^2	



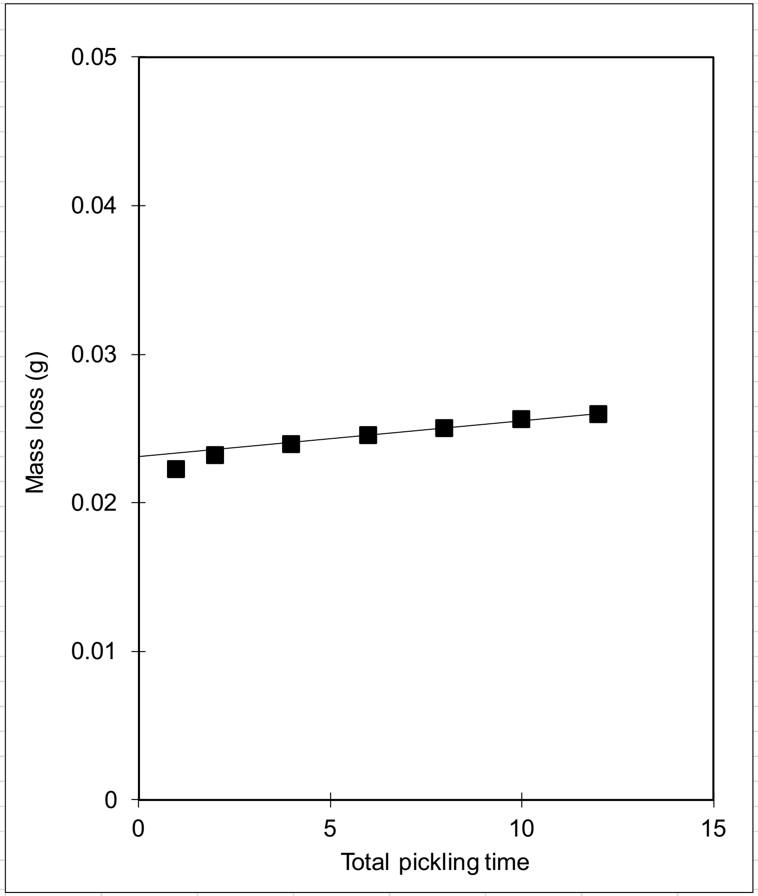
	Material				**Specimen**
	Cu				15
Pickling no.	**Mass (g)**	**Pickling (min)**	**Mass-loss (g)**	**Point incl. diagram?**	**Mass (g)**
0	17.6508	0			
1	17.62644	1	0.0244	0	0.0000
2	17.62619	2	0.0246	0	0.0000
3	17.62516	4	0.0256	1	0.0256
4	17.62474	6	0.0261	1	0.0261
5	17.6241	8	0.0267	1	0.0267
6	17.62372	10	0.0271	1	0.0271
7	17.62332	12	0.0275	1	0.0275

Sx	Sy	Sx2	Sy2	Sxy
40	0.13296	360	0.003538	1.07308

Intercept with Y-axis	0.024712	
Slope	0.000235	
r	0.995798	
Area	0.000951288	m^2
Densitet	8.96	g/cm^3
Corrosion:	25.97741	g/m^2



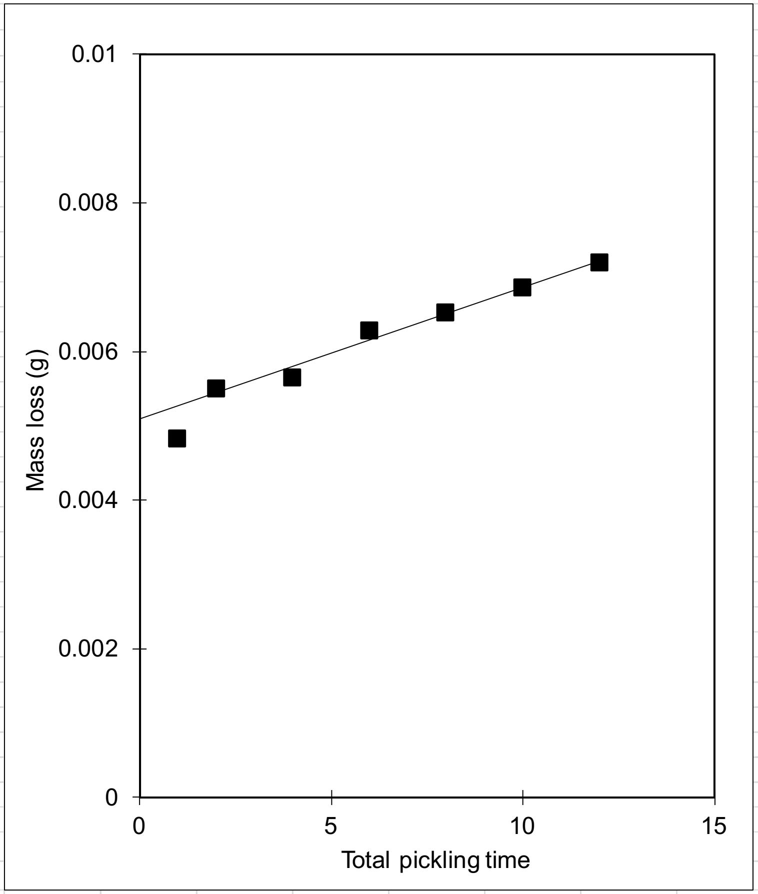
	Material				**Specimen**
	Cu				16
Pickling no.	**Mass (g)**	**Pickling (min)**	**Mass-loss (g)**	**Point incl. diagram?**	**Mass (g)**
0	17.5012	0			
1	17.47894	1	0.0223	0	0.0000
2	17.478	2	0.0232	0	0.0000
3	17.47731	4	0.0239	0	0.0000
4	17.47667	6	0.0245	1	0.0245
5	17.47619	8	0.0250	1	0.0250
6	17.47563	10	0.0256	1	0.0256
7	17.47528	12	0.0259	1	0.0259
	Sx	Sy	Sx2	Sy2	Sxy
	36	0.10103	344	0.002553	0.914
	Intercept with Y-axis			0.023129	
		Slope		0.000236	
		r		0.996253	
		Area	0.000944206	m^2	
		Densitet	8.96	g/cm^3	
		Corrosion:	24.49570	g/m^2	



	Material				**Specimen**
	Cu				18 (Pickling reference)
Pickling no.	**Mass (g)**	**Pickling (min)**	**Mass-loss (g)**	**Point incl. diagram?**	**Mass (g)**
0	17.3825	0			
1	17.37768	1	0.0048	0	0.0000
2	17.37701	2	0.0055	1	0.0055
3	17.37686	4	0.0056	1	0.0056
4	17.37622	6	0.0063	1	0.0063
5	17.37598	8	0.0065	1	0.0065
6	17.37564	10	0.0069	1	0.0069
7	17.37531	12	0.0072	1	0.0072

Sx	Sy	Sx2	Sy2	Sxy
42	0.03798	364	0.000243	0.27826

Intercept with Y-axis	0.00509	
Slope	0.000177	
r	0.990082	
Area	0.000939965	m^2
Densitet	8,96	g/cm^3
Corrosion:	5.41510	g/m^2



Arkitektkopia AB, Bromma, 2019

SKB is responsible for managing spent nuclear fuel and radioactive waste produced by the Swedish nuclear power plants such that man and the environment are protected in the near and distant future.

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