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Characterisation of different Cu-OFP grades using TEM

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Abstract

In a previous metallographic study of oxygen-free phosphorus alloyed copper material some particle-like regions appeared when characterising with TEM. The precipitates were only a few nanometers and it was not possible to conclude if these regions were particles or crystalline defects. Due to the chosen sample preparation methods it was difficult to verify the possible dissolution of phosphorus in these particle-like defects. For this reason new characterisation is carried out using other sample preparation methods that excludes possible phosphorus contamination.

Four different Cu-OFP grades have been investigated. No phosphorus agglomeration has been found in any of the materials. Thermodynamic analysis indicates that some precipitated impurities might still be present, but due to low concentrations it will be practically difficult to find these. In one of the materials sulphur enrichments was found, it is believed to be sulphides of Cu₂S stoichiometry.

The particle-like regions are identified as crystalline defects on the {111} planes in the material. They are probably produced during the sample preparation for TEM investigation. EDS measurements of these defects indicate a minor increase of argon, and the final argon ion milling could be responsible for these defects.

Sammanfattning

I ett föregående arbete av Bergqvist och Gordon (2018) rapporterades partikelliknande områden vid TEM-karakterisering av syrefri fosforlegerad koppar (Cu-OFP). Dessa områden var väldigt små, några nanometer, och det var inte möjligt att kvantifiera om dessa områden var partiklar eller kristallina defekter. Med valda provprepareringsmetoder var det inte möjligt att säkerställa om de låga fosfornivåer som observerades var en konsekvens av eventuell partikel eller en följd av provpreparering.

I detta arbete har fyra olika Cu-OFP legeringar karakteriserats med TEM. Provprenpareringsmetoder har valts i syfte att undvika eventuell fosforkontaminering. Tidigare termodynamisk studie har indikerat att vissa inneslutningar bör finnas i kopparmaterialet, och sulfider har observerats som förväntat.

De partikelliknande områden som observerats i tidigare arbete är i detta arbete identifierat som kristallina defekter på $\{111\}$ plan i atomgittret. Det är troligt att dessa defekter uppstår vid provpreparering med argonpolering.

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1 Introduction

1.1 Impurities and OFP-copper

Oxygen free copper alloyed with phosphorus (OFP-copper) is a nearly pure copper material. OFP-copper is intended to be used as canister material for disposal of spent nuclear fuel in Sweden. The only alloying element is phosphorus that is added in low concentrations, 30–100 wtppm. In this copper material, as well as other alloys, there are impurity elements in minor concentrations, typically a few ppm. The OFP copper grade follows the material standard for oxygen-free high conductivity coppers (OFHC), C10100. This standard specifies the upper limit allowed for many different impurities. According to the SKB application, some additional limits in element concentrations are given (SKB 2010).

The concentrations of impurity elements are low. The copper metal will dissolve many of the impurities, but others might precipitate. The possible chemical reactions have been studied in a previous work for the elements Pb, Zn, Fe, Ag, As, Sb, Te, Bi, Cd, Mn, Ni, Se, Sn, O, and S in copper with phosphorus (Magnusson and Frisk 2014, Magnusson 2017). The impurity can precipitate on its own forming an inclusion in the copper metal. Two typical examples are lead and bismuth, which are often added to copper-based brass alloys in order to raise their machinability. The impurity can also react with the copper metals. This is the case for the chalcogens elements oxygen, sulphur, tellurium and selenium, which all have a very low solubility in copper and forms copper inclusions on Cu_2X stoichiometry. Finally, on some rare occasions the impurity can react with another impurity or phosphorus. Due to the low concentrations of impurities in OFP-copper this is not common. The previous work has indicated that this might be the case for the elements oxygen, iron, and nickel which could possibly react with phosphorus (Magnusson and Frisk 2014, Magnusson 2017).

The strong phosphorus-oxygen reaction has been indicated in a previous work, as a part of the thermodynamic evaluation of the Cu-H-O-S-P system (Magnusson and Frisk 2014). In the more recent work (Magnusson 2017), iron and nickel were found to show high reactivity with phosphorus possibly forming phosphides in copper. Iron showed the highest affinity, and if iron was at the upper limit according to the standard (10 wtppm), iron phosphides was calculated to become stable already at 470 °C.

The high reactivity between iron and phosphorus is the basis for precipitation hardened high conductivity coppers (Lu et al. 2006). The basic idea with these copper materials is to choose an iron to phosphorus atomic relation that follows the precipitating Fe_2P phosphide. If this is made, the concentration of these elements in the matrix will be low and the conductivity will be high. In addition this copper material will have increased strength through precipitation hardening. High resolution TEM indicated 3–10 nm sized precipitates, when aging Cu-0.22Fe-0.06P for 1 hour at 300 °C.

According to the standard, a maximum of 10 wtppm iron can be present in OFP-copper and this will never give any significant phase fraction of phosphides. For this reasons it will be difficult to find iron phosphides or other phosphorus compounds if present in OFP-copper. It can also be noted that iron phosphides, or other phosphorus agglomerations, have not been reported in previous characterisation of Cu-OFP such as TEM-studies (Savolainen 2012), atom probe and TEM study (Thuvander 2015), or TOF-SIMS study (Andersson-Östling et al. 2018).

1.2 Previous work on investigating impurities in Cu-OFP

Previous characterisation work of Cu-OFP by Bergqvist and Gordon (2018) on MiniCan material was based on two different methods for sample preparation, etching with phosphoric acid and focused ion beam (FIB) milling. It was mentioned in their work that they found nanometer small regions giving contrast in the material which appeared to be particles. In addition, these regions showed a minor increase in phosphorus. The sizes of these regions were a few nanometers, which is in agreement with the iron phosphides reported by Lu et al. (2006). However, it was difficult to accurately determine the element concentrations of these particles. The thin foil analysed with TEM typically have a thickness that is around 50 nm. This thickness is much greater than the size of the particle of interest. For this reason the analysed region will mainly be related to the matrix and only a minor part is related to the particle. Due to this dilution it is difficult to conclude the composition of the particle.

An issue with using FIB-prepared samples is the overlap between platinum and phosphorus, as shown in Figure 1-1a. Platinum is sputtered on the surface of the sample as part of the FIB-preparation. By using argon-milling most of the platinum was removed as shown in Figure 1-1b, although it is difficult to exclude the possible platinum interaction.

In order to avoid any platinum contamination, the sample was also prepared using phosphoric acid. The as etched sample gave many phosphides, but also sulphides on the surface of the sample. The etched sample is shown in Figure 1-2, and the corresponding EDS-measurements. These particles were probably formed during the sample preparation and can be seen as 100 nm large particles.

In an attempt to remove the contaminated layer, the samples were argon milled and the results after this treatment can be seen in Figure 1-3. In general, no larger inclusions were seen in the material and it was mainly clean. However, some tiny regions about 5 nanometers in size are present. It can be seen that the dislocations in the material seems to rest on some of these defects. The corresponding EDS-spectra are shown in the figure. The darker regions shows increased phosphorus content but still on low levels (Bergqvist and Gordon 2018).

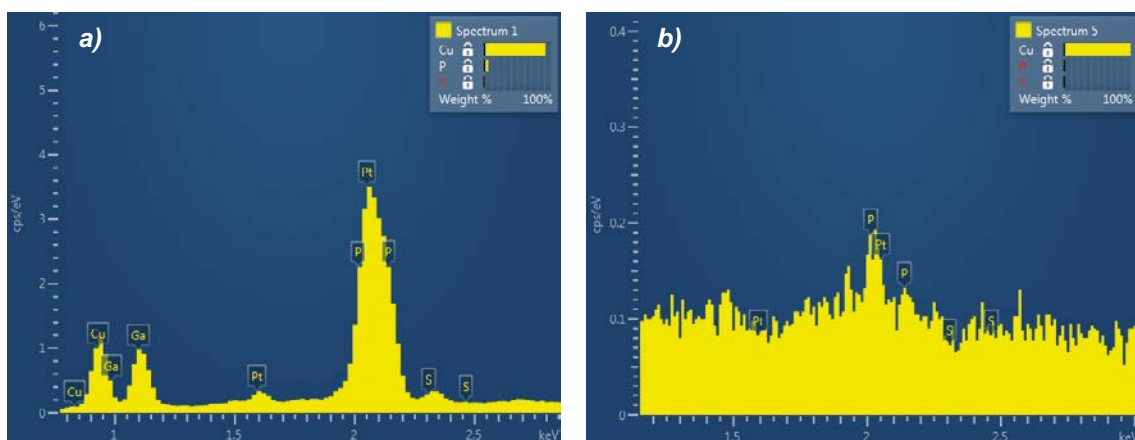


Figure 1-1. EDS diagrams of FIB-prepared samples a) after FIB b) after FIB and argon ion milling.

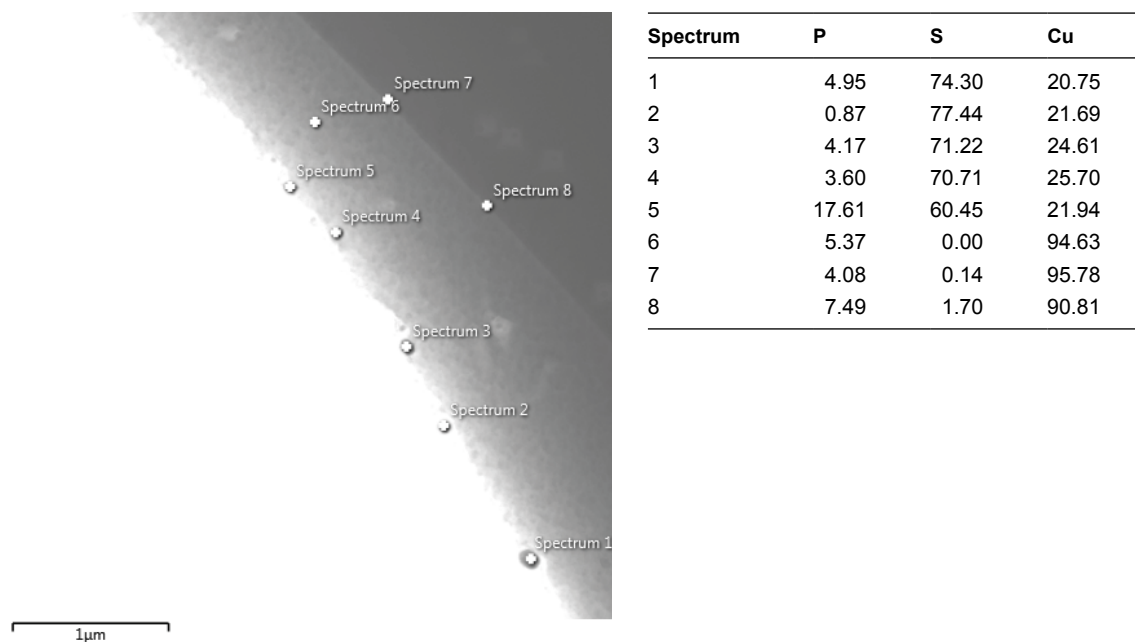
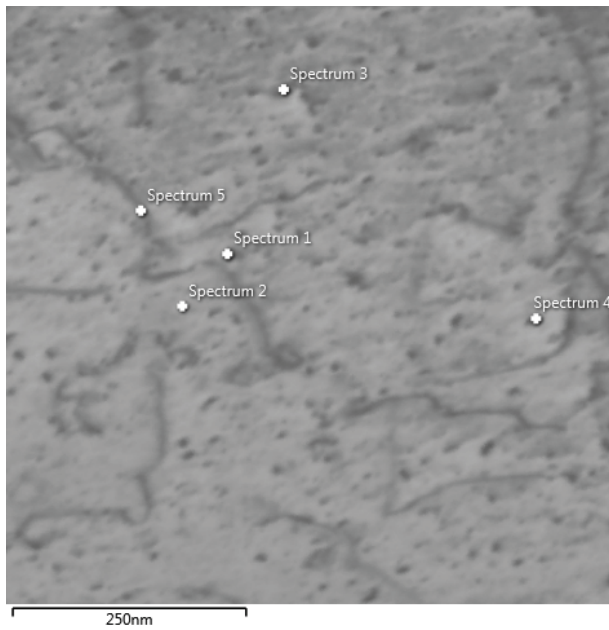


Figure 1-2. Characterised MiniCan material thinned using phosphoric acid. The corresponding EDS measured concentrations are shown in the table, in at%.



Spectrum	P	S	Cu	Site
1	0.16	0.13	99.71	Dark regions
2	0.02	0.10	99.88	Matrix
3	0.19	0.00	99.81	Dark regions
4	0.07	0.06	99.87	Dark regions
5	0.02	0.00	99.98	Dislocation

Figure 1-3. Characterised MiniCan material based on etching in phosphoric acid and then argon milled. Evaluated concentration of elements by EDS is shown as well, with concentrations in at%.

1.3 Motivation and aim with present work

The FIB-preparation gave unwanted platinum contamination. The phosphoric acid gave possible phosphorus contamination. The motivation for this work is to reinvestigate some OFP-copper grades using other sample preparation methods for TEM that will not interfere with the phosphorus element.

In addition, the status of these tiny defects seen in the material is also of interest to understand. In order to clarify the status of the particle-like defects previously observed, additional metallographic work is carried out in this work.

2 Material and methods

2.1 OFP-copper and materials in this study

Four different OFP-copper materials are studied in this work, as summarised in Table 2-1. Both as-received and creep tested copper are included. HOSP-copper is an experimental copper grade with maximised impurity concentrations regarding hydrogen, sulphur and phosphorus. Finally, the same material as used in the previous study (MiniCan) is included in this work as well.

The measured concentrations of hydrogen, oxygen, sulphur and phosphorus are given in Table 2-1. Two measurements were made for the creep tested OFP and HOSP materials, as well as the MiniCan material. It can be seen that the HOSP material has the highest sulphur concentration compared to the other materials.

Table 2-1. Materials studied in this work. The concentration of H, O, S, and P are given as well, together with a reference.

Material	Concentration, in wtppm				Reference
	H	O	S	P	
As recieved OFP	< 0.5	1.1	5	106	Magnusson et al. (2015)
Creep tested OFP, 125 °C, 155 MPa, $t_r = 5395$ h	0.53/0.63	3.1/3.1	3/3	65/66	Wu and Sandström (2015) Internal designation REF50-10
Creep tested HOSP copper, 125 °C, 160 MPa, $t_r = 10$ min	0.45/0.6	7/7.6	11.9/12.6	112/115	Wu and Sandström (2015) Internal designation M2HP-12.
MiniCan 4	0.46	1.7	7	32	Smart and Rance (2009)

2.2 Sample preparation and TEM

Different sample preparation methods for TEM are discussed in this work, and they are summarised below:

- FIB and argon milling: MiniCan material (Bergqvist and Gordon 2018).
- Phosphoric acid and argon milling: MiniCan material (Bergqvist and Gordon 2018).
- Ground and polished: Creep tested Cu-OFP, this work.
- Ground, polished and argon milling: All materials, this work.

In this work, a grinding and polishing method was selected as method for sample preparation. The process can be summarised in the four different steps below:

1. Grinding with silicon carbide paper down to 4 000 grit. The grinding is made with a minor angle in order to fully grind down the lower part of the specimen. The specimen is 2–3 mm in diameter.
2. Polishing with diamond paste with 1 μm grains.
3. Polishing with alumina or quartz with 40 nm grains.
4. Soft argon ion milling with 4–5 kV acceleration voltage.

For one of the analysed samples, the final step (4) was excluded in order to avoid possible contamination from argon. The ground and polished sample can be seen in Figure 2-1.

The TEM examination was performed at 200 kV, in a field emission STEM, JEOL 2100 HR, equipped with an EDS-detector X-max from Oxford Instruments. The electron spot size used for the analysis was 0.7 nm. The thickness of the samples is 40–80 nm.

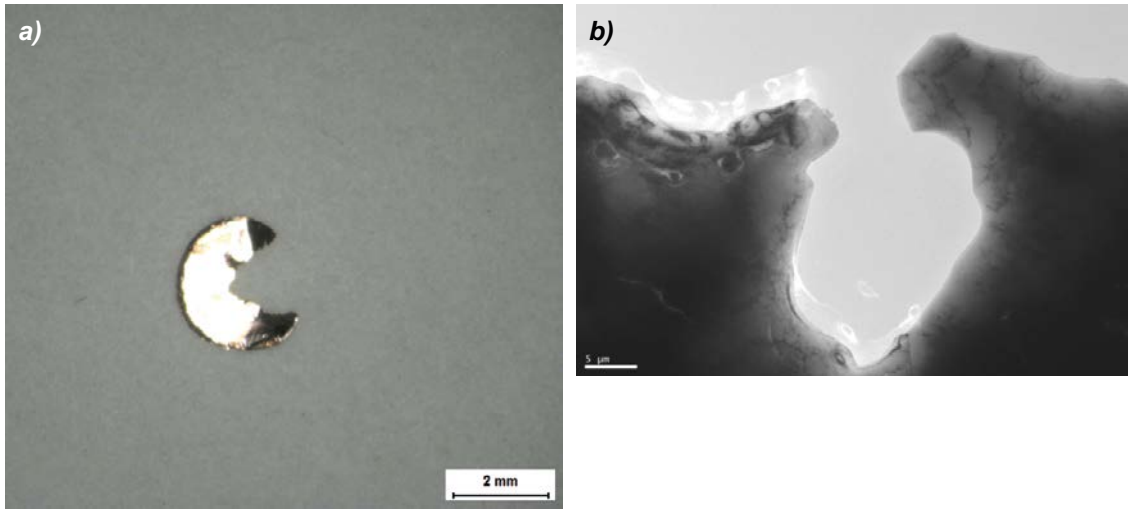


Figure 2-1. a) An example of sample after grinding and polishing. b) Material analysed with TEM in low magnification.

3 Results

3.1 Defects in copper

The darker regions previously observed (Bergqvist and Gordon 2018) as point defects are promoted by tilting the sample so that diffraction occurs. Figure 3-1 presents two images of the same region whereas Figure 3-1b has been tilted to promote diffraction. In this case the as-received Cu-OFP sample is investigated in the ground, polished, and argon-milled state. The dark regions shown in the figure are approximately 5–10 nm in size, and are in agreement with the previous observations, see Figure 1-3. The darker regions seem to be crystalline defects in the material. They give clear diffraction contrast when $\{111\}$ planes are parallel to the incoming electron beam. It is believed that these defects were caused by the sample preparation. This will be further discussed in this work.

Additional attempt to promote the crystalline defects has been made for the MiniCan material, in the ground, polished and argon-milled state. Again, the crystalline defects are highlighted by tilting the sample so that $\{111\}$ planes are diffracted. The defects become black in the bright-field image. The resulting image can be seen in Figure 3-2a, in higher magnification in Figure 3-2b.

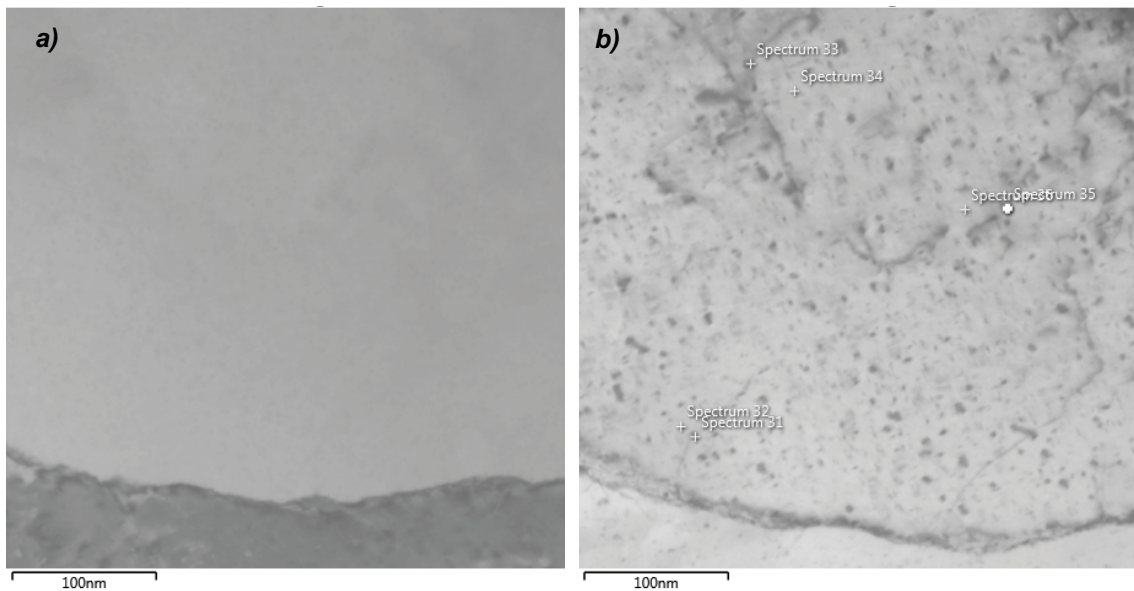


Figure 3-1. As received Cu-OFP is presented in both figures. Both samples show the same region, but b) has been tilted to promote the diffraction.

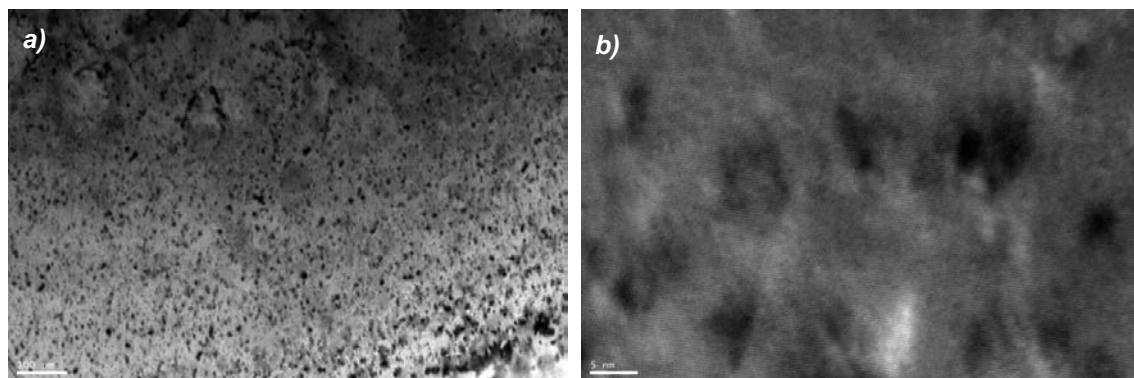


Figure 3-2. a) Bright-field image with sample tilted so that $\{111\}$ planes are shown in black. b) as in a) but greater magnification.

3.2 As received Cu-OFP

The as received Cu-OFP has been analysed and the TEM micrograph is presented in Figure 3-3. The analysed concentrations are also given in this figure. In general, the darker regions in the image do not show any accumulation of phosphorus according to the EDS-measurements. One spectrum shows higher concentration, and that is spectrum 35. This spectrum is shown in Figure 3-4. The position seems to be located at some boundary in the material, which could motivate some increase in elements. But due to the low concentrations it is not likely any precipitated phase.

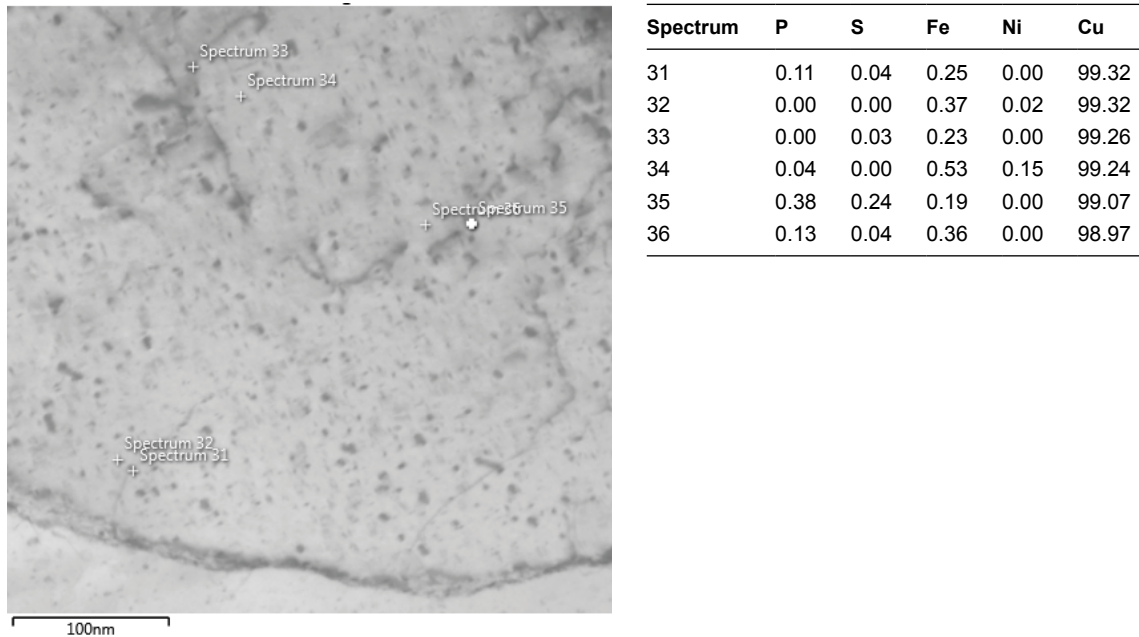


Figure 3-3. Characterised as received Cu-OFP is presented. EDS evaluated concentrations are given in at%, and minor measurements of both Si and Ar are excluded in the table.

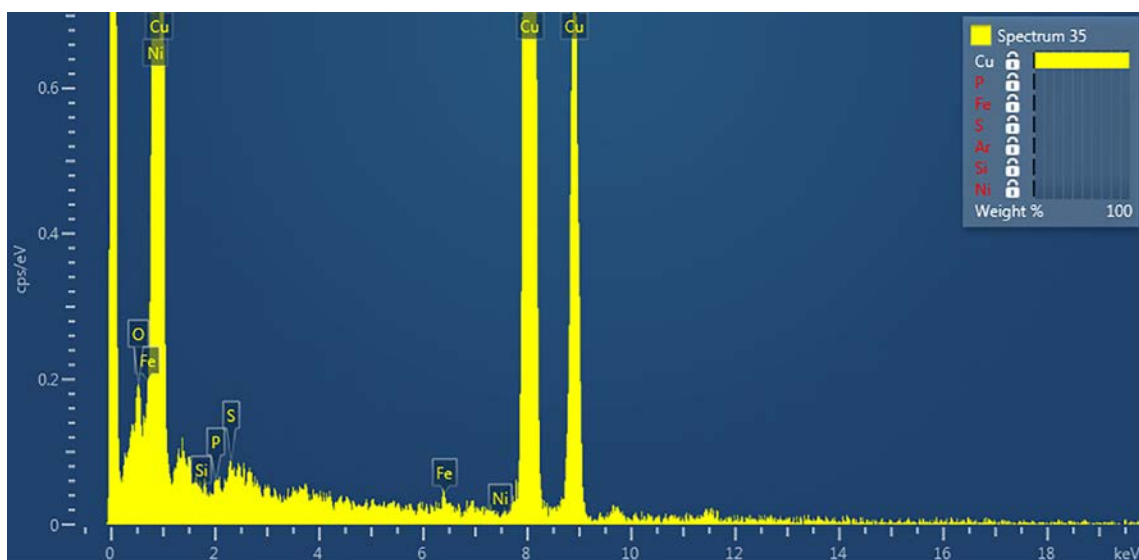
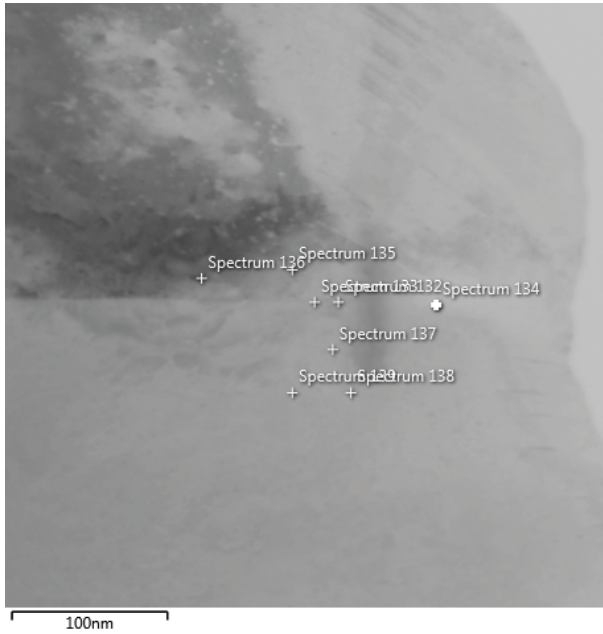


Figure 3-4. EDS spectrum of spectrum 35, as indicated in Figure 3-3.

An additional image is shown in Figure 3-5 together with the measured concentration of elements in the analysis points. Again no accumulation of any elements can be seen, and the measured concentrations are at the detection limits. The spectrum of analysis point 134 is shown in Figure 3-6. This spectrum gave the highest concentrations, and it can be seen that the levels are at the detection limit.



Spectrum	P	S	Fe	Ni	Cu
132	0	0.04	0.23	0.04	98.34
133	0.14	0	0.49	0	97.81
134	0.67	0.63	0.01	0.05	98.63
135	0	0.44	0.23	0.24	98.10
136	0	0	0.42	0	99.33
137	0	0.33	0	0	99.14
138	0	0	0.48	0.22	98.80
139	0.67	0.34	0.11	0.05	98.16

Figure 3-5. Characterised as received Cu-OFP is presented, together with the measured concentrations in the different analysis points. All values are given in at%. Si and Ar are not included in the table.

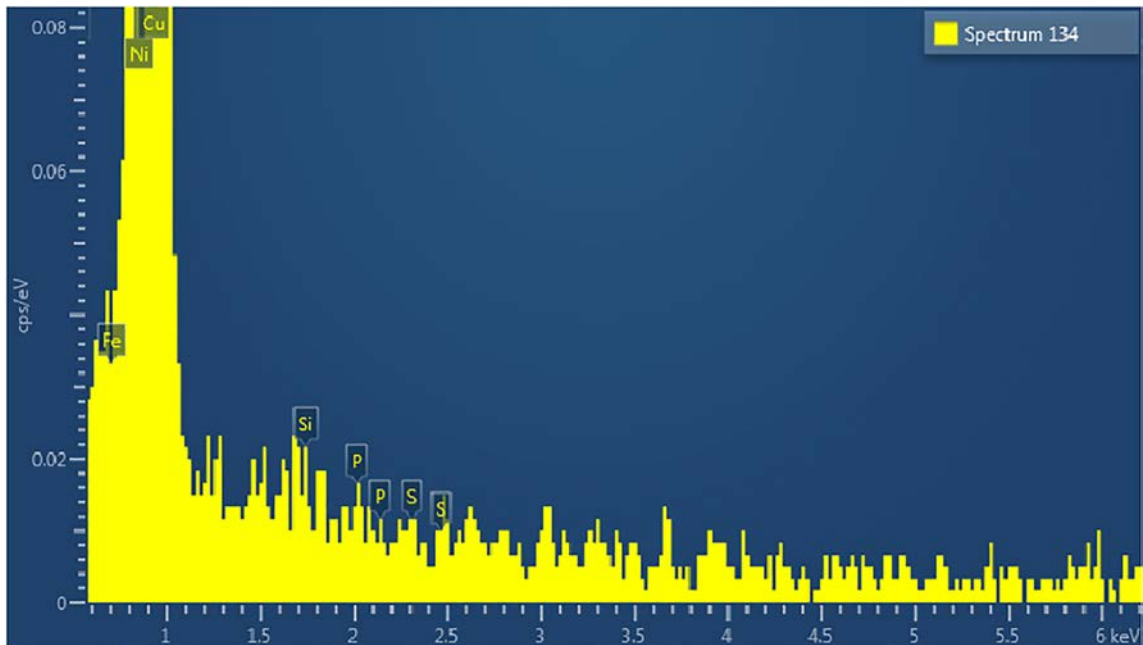


Figure 3-6. Spectrum according to point 134, as shown in Figure 3-5.

3.3 HOSP material

Material HOSP has been characterised with the same method as previously shown for as received Cu-OFP. Two TEM images and their corresponding EDS analysed concentrations are presented in Figure 3-7. For this material an accumulation of sulphur can be seen in the outermost part of the foil. The analysis point with the highest concentration sulphur gives 12.3 at%. Its spectrum is presented in Figure 3-8. The sulphur peak is clearly shown. The analysis point 60 gives also a high concentration of sulphur, 14.74 at% and is most likely a copper sulphide Cu_2S .

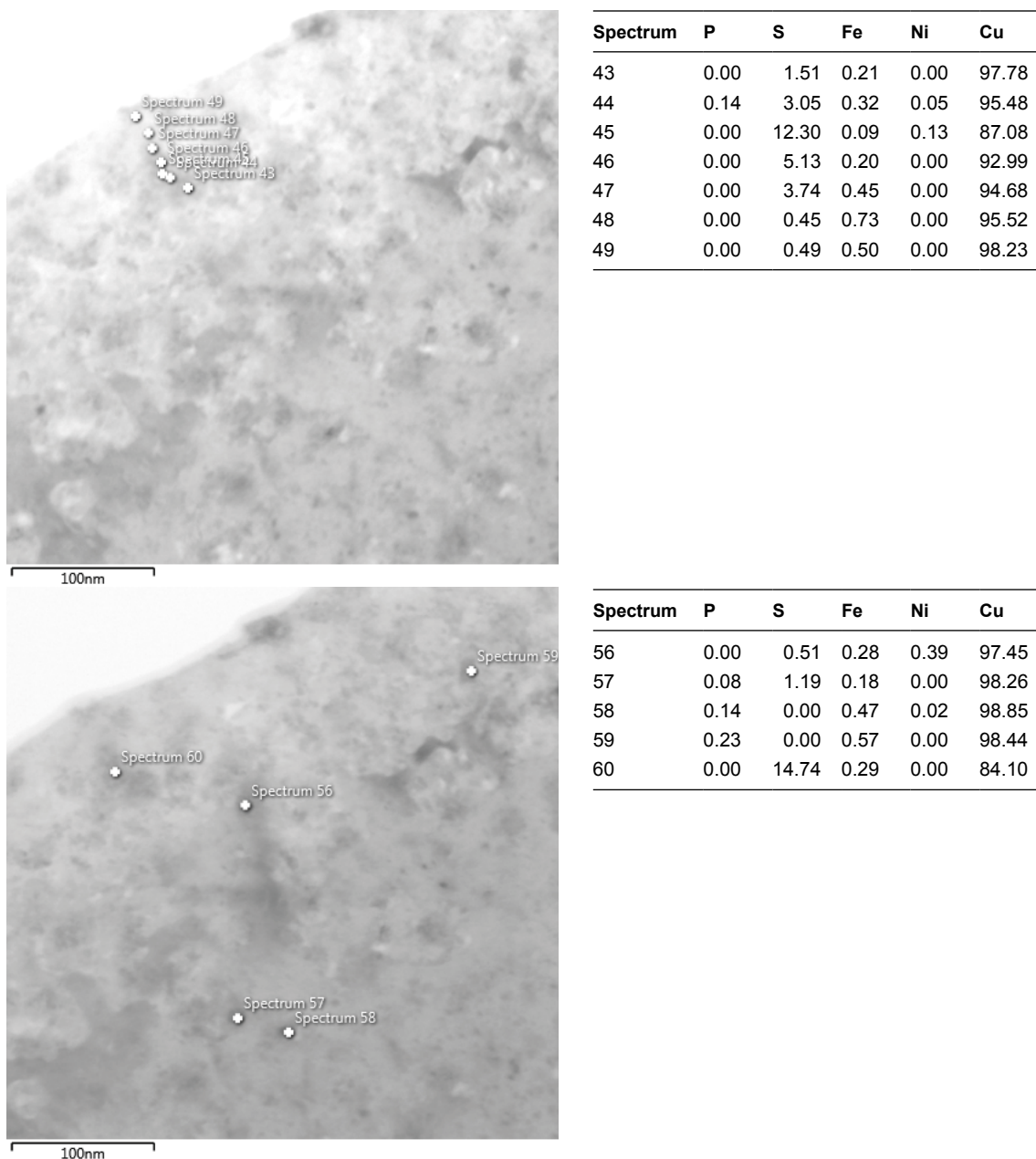


Figure 3-7. Characterised HOSP material is presented in both figures. Measured concentrations are presented as well for the different analysis points. All concentrations are given in at%. Elements Si and Ar are excluded in the tables.

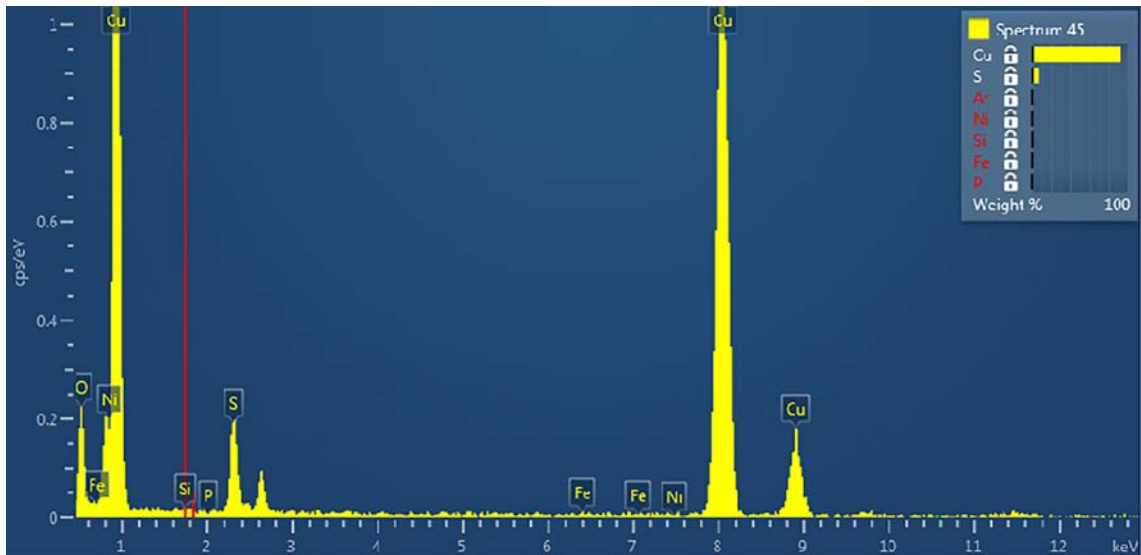


Figure 3-8. EDS spectrum corresponding to analysis point 45, as shown in Figure 3-7.

An additional TEM-figure for the HOSP material is presented in Figure 3-9, with corresponding EDS measurements. Some different analysis points are investigated, as well as an average composition of the whole region. Some darker regions are found but no relation to increased concentration of elements is found.

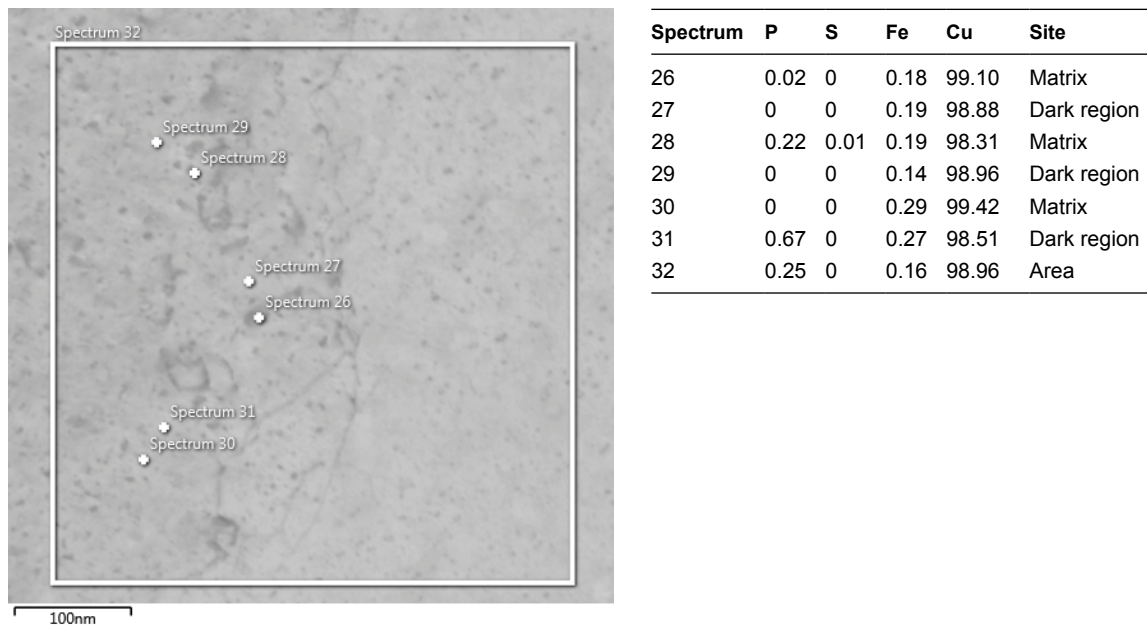


Figure 3-9. Characterised HOSP material is presented together with the positions of the EDS analysis. All concentrations are given in at%. Elements Al, Si and Ar are excluded in the tables.

3.4 MiniCan 4

MiniCan 4 material prepared with the same methods as described above, has been characterised. The material appears clean without any inclusions. The material is shown in Figure 3-10 together with its EDS quantification. A grain boundary is seen in Figure 3-10. The sample is tilted so that the defects appear in the upper grain. The lower grain has other orientation and no defects are seen in that grain.

An additional image for the MiniCan material is presented in Figure 3-11, together with the analysed concentrations. No accumulation of any elements can be found in the material.

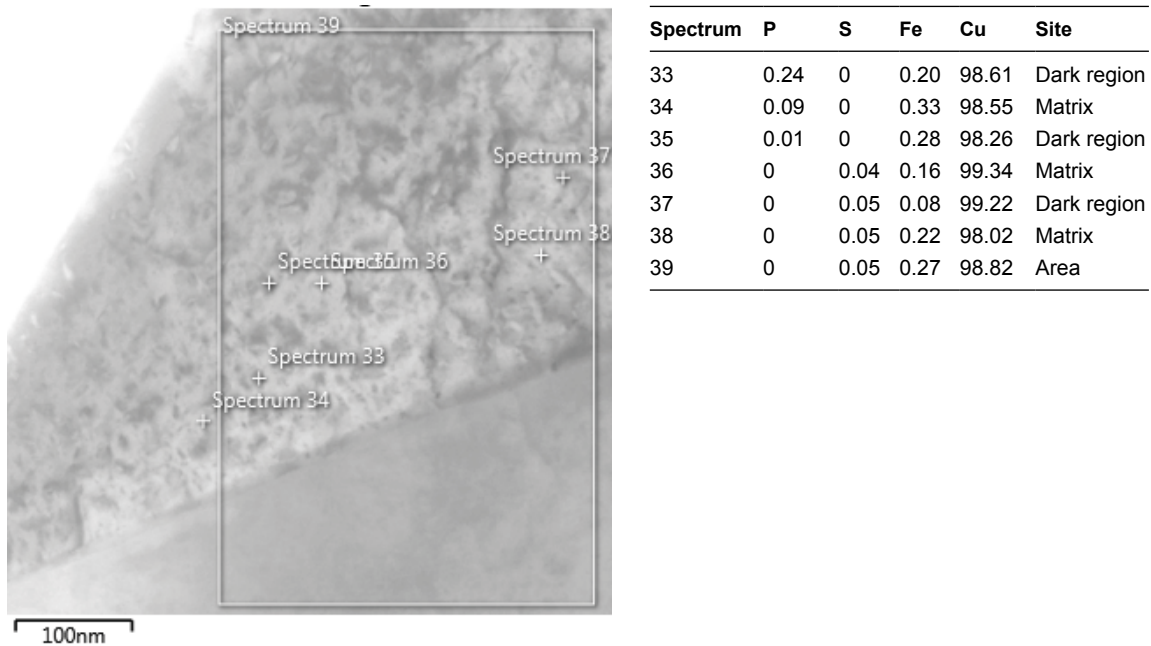


Figure 3-10. TEM image of material MiniCan 4 showing defects in the microstructure and a grain boundary. EDS spectra of analysed regions are presented in Figure 3-10. Values are given in at%. The different sites for measurements are indicated as well.

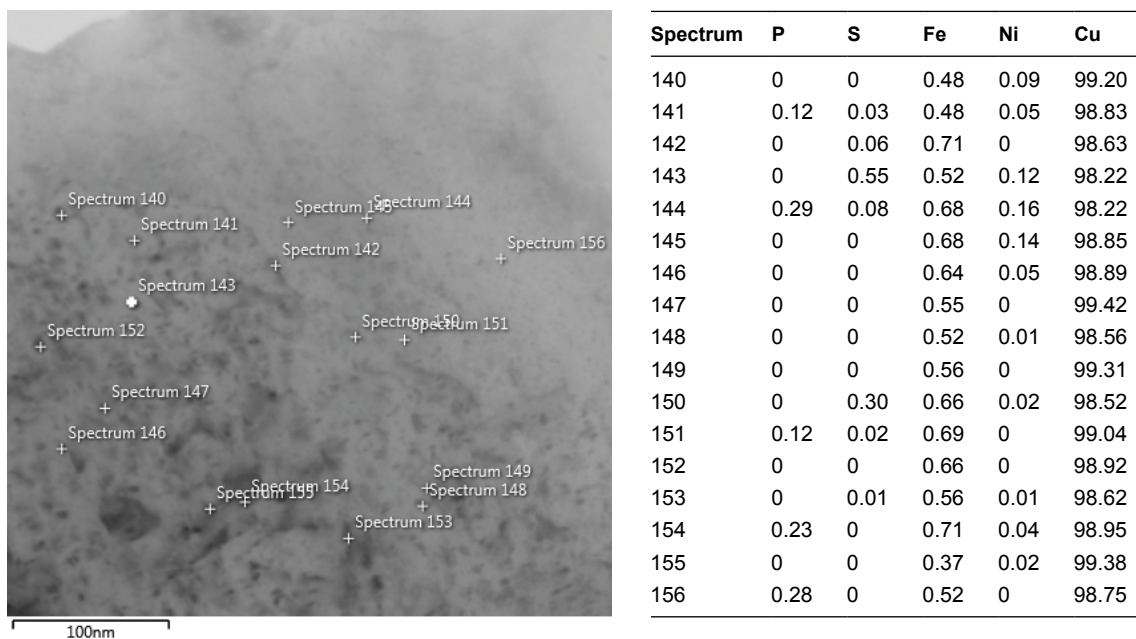
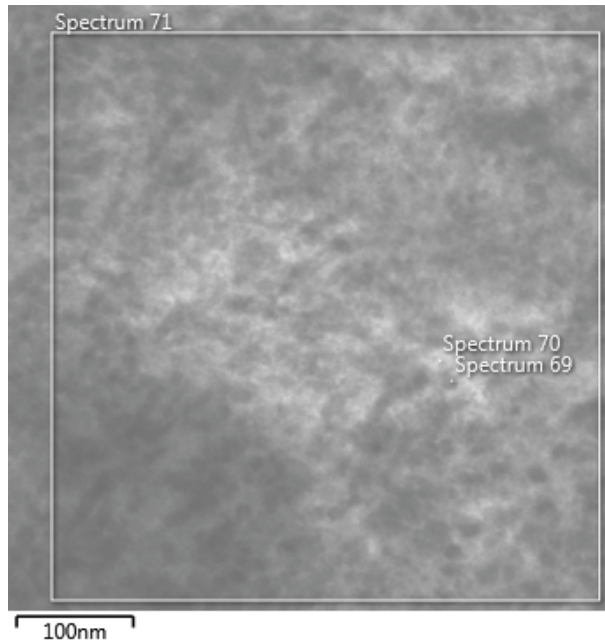


Figure 3-11. Characterised MiniCan 4 material is presented. Measured concentrations in the different analysis points given in Figure 3-11. All values are in at%.

3.5 Creep tested Cu-OFP

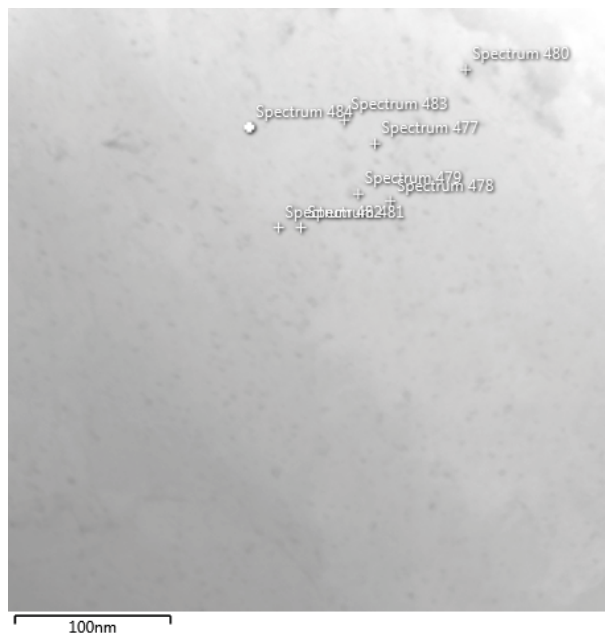
The creep tested OFP-copper has been characterised as well. Material ground and polished, but not argon milled, is shown in Figure 3-12. The image quality is worse for the sample that has not been argon polished. Some darker regions can be seen in this figure as well, although it is difficult to conclude their presence. No agglomeration of any elements is seen in the defects compared to the surrounding matrix.

The same material, but ground, polished and argon milled is presented in Figure 3-13 together with the corresponding analysis points. Again, no accumulation is seen of any elements. In this spectrum oxygen is included which is from the surface oxides. These analysed regions are at the outmost part of the foil giving greater amount of surface oxides.



Spectrum	P	S	Fe	Cu	Site
69	0.11	0.14	0.31	98.25	Dark
70	0	0	0.11	96.28	Light
71	0.02	0	0.31	96.97	Area

Figure 3-12. Characterised creep tested Cu-OFP that has been only ground and polished. The corresponding analysis points are presented as well, with concentrations in at%.



Spectrum	O	P	S	Fe	Ni	Cu
477	6.19	0	0	0.34	0	92.91
478	7.18	0	0.31	0.31	0	92.20
479	5.08	0	0	0.46	0.21	94.25
480	5.70	0	0	0.33	0.24	92.73
481	4.02	0	0	0.52	0	95.45
482	4.33	0	0	0.49	0	94.58
483	5.95	0	0	0	0	94.05
484	4.48	0.32	0.36	0.37	0	94.46

Figure 3-13. Characterised creep tested Cu-OFP. EDS measurements at the analysis points are presented as well, with concentration given in at%.

4 Discussion

The previous TEM study by Bergqvist and Gordon (2018) showed that there were particle-like regions in the material. Due to the sample preparation methods, using FIB or phosphoric acid, it was difficult to verify that these defects did not include phosphorus. Phosphides, for instance including iron, are well known to exist for precipitation hardened high conductivity copper materials (Lu et al. 2006). The previous thermodynamic evaluation indicated that iron phosphides can become stable even for ppm-levels of iron. On the other hand, the low concentration of impurities will give a low phase fraction of precipitates. By evaluating the area fraction of defects that were found in the different materials any phosphides can directly be ruled out due to low concentrations of impurities.

The sample preparation method used in this work, based on grinding and polishing, did not give any contamination that interferes with phosphorus measurements. For this reason it is concluded, also from the EDS-measurements, that the defects do not include phosphorus. The thickness of the analysed sample is approximately 40 nm. If a phosphide is present, for instance 4 nm in size, one could expect that 1/10 of the analysis should correspond to the phosphide. Lu et al. (2006) reported 3–10 nm of Fe₃P phosphides in strengthened high conductivity coppers. This phosphide has 33 at% phosphorus, which should in total yield approximately 3 at% phosphorus in the analysed regions. This high concentration has not been found in any analysis point in any of the materials.

However, the sample preparation did still influence the material. The argon milling could be the cause for these tiny defects in the material. Some argon, or possibly silver, was also detected in the EDS-spectra. It is difficult to separate between silver and argon due to overlapping spectra. Silver is an impurity element which can take quite high concentration in OFP-copper, up to 25 wtppm. But due to the many defects they cannot be related to ppm-levels of silver either. The defects are oriented on the {111} planes. Crystalline defects have previously been reported for sample preparation of copper using relative low energy of argon milling. Similar defects have been reported by De Mol van Otterloo et al. (1995), and De Hosson (1977), when using argon milling. The presence of defects occupying {111} planes has also been verified by annealing experiments, for instance using copper with helium at 400 °C (Wei et al. 2010).

Sulphides have been found for one of the materials, the HOSP material where sulphur has deliberately been increased. More sulphur will give more sulphides and these were found in the HOSP material. Sulphides in OFP-copper has been reported previously for instance by Bergqvist and Gordon (2018), and on friction stir welded Cu-OFP by Savolainen (2012).

5 Conclusions

In this study no inclusions containing phosphorus have been found. The low concentration of phosphorus seems to be homogeneously distributed in the material. The thermodynamic information indicates that some of the impurity elements could be present as inclusions in the material, but the low concentration of impurities makes it practically difficult to find any of these.

Inclusions containing sulphur has been found for the HOSP material, and it is believed to be Cu_2S type of sulphide. Sulphides are most likely present in the other copper grades as well, but due to higher concentration of sulphur in HOSP material it is easier finding them.

Crystalline defects, appearing on the $\{111\}$ planes have been found. It is believed that these defects are due to sample preparation. The presence of argon indicates that the final argon ion milling could be responsible. This is also in agreement with some references from literature.

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SKB's (Svensk Kärnbränslehantering AB) publications can be found at www.skb.com/publications. SKBdoc documents will be submitted upon request to document@skb.se.

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